

CALIFORNIA INSTITUTE OF TECHNOLOGY



X-RAY SAFETY MANUAL

2020

A PUBLICATION OF THE CALTECH ENVIRONMENT, HEALTH, AND SAFETY OFFICE
1200 E. CALIFORNIA BOULEVARD
MAIL CODE 25-6
PASADENA, CA 91125
(626) 395-6727

safety@caltech.edu and <http://www.safety.caltech.edu>

X-RAY SAFETY MANUAL

Table of Contents

1.	GENERAL INFORMATION	3
1.1	INTRODUCTION.....	3
1.2	RADIATION SAFETY COMMITTEE.....	4
1.3	INSTITUTE RADIATION SAFETY OFFICER AND STAFF	5
2.	RADIATION PRODUCING MACHINE USE AUTHORIZATION	6
2.1	APPLICATION FOR POSSESSION AND USE	6
2.2	PERMIT AUTHORIZATION.....	6
2.3	USE PERMIT TERMINATION AND NON-COMPLIANCE POLICY	7
3.	UNITS OF EXPOSURE AND DOSE.....	8
4.	BIOLOGICAL EFFECTS OF IONIZING RADIATION.....	9
5.	RADIATION DOSE STANDARDS AND PERSONNEL MONITORING.....	11
5.1	RADIATION DOSE STANDARDS	11
5.2	PERSONNEL MONITORING (EXTERNAL).....	12
6.	RADIATION SURVEY METERS	14
7.	RADIATION PRODUCING MACHINES	16
7.1	DEFINITIONS.....	16
7.2	POLICIES AND PROCEDURES FOR USE OF RADIATION PRODUCING MACHINES.....	16
8.	RADIATION SAFETY FOR X-RAY DIFFRACTION UNITS.....	19
8.1	NATURE OF ANALYTICAL X-RAYS	19
8.2	X-RAY HAZARDS AND BIOLOGICAL EFFECTS	21
8.3	SOURCES OF IONIZING RADIATION	22
8.4	SAFETY PRECAUTIONS AND NOTES	22
9.	EMERGENCY PROCEDURES.....	25

1. GENERAL INFORMATION

1.1 INTRODUCTION

PURPOSE

This manual provides information about the radiation protection program at Caltech. The purpose of this program is to protect Caltech personnel, guests, and property from hazards associated with radiation producing machines.

DEFINITIONS

A radiation producing machine is any device capable of producing ionizing radiation when the associated control devices are operated, except devices which produce radiation only by the use of radioactive material. X-ray diffraction and radiography units, particle accelerators, electron microscopes, tokomaks, and high voltage rectifiers operating above 10 kV all fall into the category of radiation producing machines.

REGULATION

Use of radiation producing machines is regulated by the State. A copy of the California Radiation Control Regulations (CRCR) is available for review in the Environment, Health, and Safety Office (EHS).

Caltech's policies for the use of radiation producing machines are designed to meet the applicable requirements of the CRCR and to ensure the safety of users and other personnel.

REGISTRATION

Radiation producing machines must be registered with the State and a registration fee paid. Registration will be in the name of the Institute, with EHS as the point of contact. EHS must be notified of the acquisition, transfer, or disposal of any radiation producing machine. Some machines, such as electron microscopes, may be exempt from registration. EHS will make that determination based on current State guidelines.

1.2 RADIATION SAFETY COMMITTEE

PURPOSE

The Radiation Safety Committee is appointed by the President of the Institute. The Committee is responsible for ensuring that radioactive materials and radiation-producing devices are used safely and in accordance with state and federal regulations as well as Institute policies.

RESPONSIBILITIES

The Committee is responsible for:

- Formulating general policy governing the use of radioactive material and radiation-producing equipment.
- Reviewing and approving all requests for the use and possession of radioactive material and radiation-producing devices at all Caltech locations.
- Verifying that all individuals authorized to use radioactive material and radiation-producing machines have sufficient training and experience to conduct their duties safely.
- Establishing a program to ensure that all individuals whose duties may require them to work in the vicinity of radioactive material or radiation-producing equipment are properly instructed about all appropriate health and safety matters.
- Advising and keeping current the President and Administrative Officers on matters involving the use of radioactive material and radiation-producing machines.

MEETINGS

The Committee meets once every calendar quarter.

MEMBERSHIP

Radiation Safety Committee members include:

- Faculty members (appointed Division Radiation Safety Officers) with expertise in the use of radioactive material or use of radiation-producing machines.
- The Institute Radiation Safety Officer who also serves as the Executive Officer.
- The Director of the Environment, Health, and Safety Office is a representative of administration.

Other members shall be appointed at the discretion of the President.

SUB-COMMITTEE

As members of the Radiation Safety Committee, Division Radiation Safety Officers (DRSOs) serve as the primary point of contact regarding radiation safety matters within their department. Except for those uses requiring full committee approval (see section 2.2, PERMIT AUTHORIZATION), the DRSO and the Institute Radiation Safety Officer form a sub-committee to review and approve applications and amendments involving routine use of radioactive material and radiation producing machines.

REPORTS AND RECOMMENDATIONS

Reports and recommendations of the Radiation Safety Committee are directed to the Associate Vice President for Facilities and the President, as appropriate.

1.3 INSTITUTE RADIATION SAFETY OFFICER AND STAFF

INSTITUTE RADIATION SAFETY OFFICER

The Institute Radiation Safety Officer (RSO) is appointed by the Associate Vice President for Facilities. In addition to responsibilities as a committee member, the RSO directs the Radiation Safety Program.

RADIATION SAFETY STAFF

Under the direction of the RSO, the staff of EHS:

- Assist in developing general policies for control of radiation.
- Conduct training in the safe use of radioactive materials and radiation-producing machines.
- Evaluate operational techniques and procedures.
- Perform environmental monitoring of areas involving use of radioactivity or radiation.
- Respond to emergencies and investigate accidental doses.
- Issue personnel dosimetry devices.
- Calibrate portable radiation survey instruments.
- Maintain records.
- Aid in completing the application for a permit to use radiation producing machines and provides consulting services.

2. RADIATION PRODUCING MACHINE USE AUTHORIZATION

2.1 APPLICATION FOR POSSESSION AND USE

Faculty members who wish to operate a radiation producing machine must submit an Application for Use of Radiation Producing Machine form to the Radiation Safety Committee (RSC).

Each application must be completed in sufficient detail for RSC evaluation. Applications must include a description of the machine (including maximum operating parameters), description of proposed use, and for machines operating above 100 kV, a description of the facility.

Applications for permits and a Guide for completing them are available from EHS.

2.2 PERMIT AUTHORIZATION

All applications for use of radiation producing machines are reviewed by the Radiation Safety Committee (RSC) or the sub-committee. Upon approval, a numbered permit is issued, listing any special conditions specified by the RSC.

Most applications and amendment requests are approved by the sub-committee. The following require full committee approval:

- Applications for the use of an accelerator or an x-ray unit operating above 100 Kv.
- Applications or amendments which the RSO or the DRSO feel should be brought to the attention of the full committee.

AMENDMENT TO PERMIT

A request for amendment to an approved application is submitted as above on a Request for Amendment to Permit for Use of Radiation Producing Machine form available from the EHS Office. Amendment requests may be made for the following changes:

- Change of location.
- Change in facility design (> 100 kV).
- Change in proposed use.

ANNUAL RENEWAL OF PERMITS Radiation producing machine use permits must be renewed each year before March 31st. Sixty days prior to that date, EHS will send an annual renewal form to each Principal Investigator (PI).

2.3 USE PERMIT TERMINATION AND NON-COMPLIANCE POLICY

USE PERMIT TERMINATION Failure to annually renew a use permit will result in automatic cancellation of the permit. To reactivate a cancelled permit, a new application must be submitted for Committee review and approval.

NON-COMPLIANCE POLICY The following items of non-compliance will be brought to the attention of the DRSO. Either the DRSO or the RSO may recommend review by the full-committee.

- Willful violation of the Institute’s policies or the State regulations regarding the use of radiation producing machines.
- Doses exceeding the Caltech administrative limits (see Section 5.1)
- Improper use of radiation producing machines which results in significantly endangering the safety of personnel or the environment.
- Use of radiation producing machines not authorized by the permit.

Depending on the severity of the incident, the Committee will take appropriate action up to revocation of the permit.

3. UNITS OF EXPOSURE AND DOSE

DEFINITIONS

Dose is a measure of energy deposited by radiation in a material, or of the relative biological damage produced by that amount of energy given the nature of the radiation.

Exposure is a measure of the ionizations produced in air by x-ray or gamma radiation. The term exposure (with its "normal" definition) is sometimes used to mean dose. (e.g. "He received a radiation exposure to his hand.")

EXPOSURE

The unit of radiation exposure in air is the *roentgen (R)*. It is defined as that quantity of gamma or x-radiation causing ionization in air equal to 2.58×10^{-4} coulombs per kilogram. Exposure (R) applies only to absorption of gammas and x-rays in air.

DOSE

The *rad* is a unit of absorbed dose. One rad is equal to an absorbed dose of 100 ergs/gram (1 erg = 6.24×10^{11} eV). The SI unit of absorbed dose is the *Gray (Gy)*. $1 \text{ Gy} = 1 \text{ joule/kilogram} = 100 \text{ rad}$.

An exposure of 1 R results in an absorbed dose of 0.87 rad (in air).

A *quality factor (Q)* is used to compare the biological damage producing potential of various types of radiation, given equal absorbed doses. The effectiveness of radiation in producing damage is related to the energy loss of the radiation per unit path length. The term used to express this is *linear energy transfer (LET)*. Generally, the greater the LET in tissue, the more effective the radiation is in producing damage. The quality factors for radiations frequently encountered are:

<u>Radiation</u>	<u>Q</u>
Gammas and x-rays	1
Beta particles & electrons	1
Alpha particles & fission fragments	20
Neutrons	10

The *rem* is a unit of dose equivalent. The dose equivalent in rem is equal to the absorbed dose in rad multiplied by the quality factor. The SI unit for dose equivalent is the Sievert (*Sv*). $1 \text{ Sv} = 100 \text{ rem}$.

4. BIOLOGICAL EFFECTS OF IONIZING RADIATION

RADIATION HAZARDS	The hazards associated with the absorption of radiation in mammalian systems and tissue are related to both the type of radiation and the nature of the absorbing tissue or organ system.
PHOTONS	Externally, the hazard from low energy (<30 keV) gammas and x-rays is primarily to the skin or the tissues of the eye. Higher energies are more penetrating and therefore a whole body hazard.
MECHANISMS OF DAMAGE	<p>Radiation causes atoms and molecules to become ionized or excited. These ionizations and excitations can result in:</p> <ul style="list-style-type: none">▪ Production of free radicals.▪ Breakage of chemical bonds.▪ Production of new chemical bonds and cross-linkage between macromolecules.▪ Damage to molecules which regulate vital cell processes (e.g. DNA, RNA, and proteins).
TISSUE SENSITIVITY	In general, the radiation sensitivity of a tissue varies directly with the rate of proliferation of its cells and inversely with the degree of differentiation.
EFFECTS OF ACUTE HIGH RADIATION DOSES	<p>A whole body radiation dose of greater than 25 to 50 rem received in a short time results in the clinical "acute radiation syndrome." This syndrome, which is dose related, can result in disruption of the functions of the bone marrow system (>25 rem), the gastrointestinal system (>500 rem), and the central nervous system (>2000 rem). An acute dose over 300 rem can be lethal.</p>
EFFECTS OF LOW RADIATION	<p>There is no disease uniquely associated with low radiation doses.</p> <p>Immediate effects are not seen below doses of 25 rem. Latent effects may appear years after a dose is received. The effect of greatest concern is the development of some form of cancers.</p> <p>The National Academy of Sciences Committee on Biological Effects of Ionizing Radiation (BEIR) issued a report in 1990 entitled "Health Effects of Exposure to Low Levels of Ionizing</p>

Radiation," also known as BEIR V. The following is an excerpt from the Executive Summary of the report:

EFFECTS OF LOW
RADIATION (Continued)

On the basis of the available evidence, the population-weighted average lifetime risk of death from cancer following an acute dose equivalent to all body organs of 0.1 Sv (0.1 Gy of low-LET radiation) is estimated to be 0.8%, although the lifetime risk varies considerably with age at the time of exposure. For low LET radiation, accumulation of the same dose over weeks or months, however, is expected to reduce the lifetime risk appreciably, possibly by a factor of 2 or more. The Committee's estimated risks for males and females are similar. The risk from exposure during childhood is estimated to be about twice as large as the risk for adults, but such estimates of lifetime risk are still highly uncertain due to the limited follow-up of this age group.

The Committee examined in some detail the sources of uncertainty in its risk estimates and concluded that uncertainties due to chance sampling variation in the available epidemiological data are large and more important than potential biases such as those due to differences between various exposed ethnic groups. Due to sampling variation alone, the 90% confidence limits for the Committee's preferred risk models, of increased cancer mortality due to an acute whole body dose of 0.1 Sv to 100,000 males of all ages range from about 500 to 1200 (mean 760); for 100,000 females of all ages, from about 600 to 1200 (mean 810). This increase in lifetime risk is about 4% of the current baseline risk of death due to cancer in the United States. The Committee also estimated lifetime risks with a number of other plausible linear models which were consistent with the mortality data. The estimated lifetime risks projected by these models were within the range of uncertainty given above. The Committee recognizes that its risk estimates become more uncertain when applied to very low doses. Departures from a linear model at low doses, however, could either increase or decrease the risk per unit dose.

Caltech's whole body dose limit for planned exposures is 500 mrem/year (5 mSv/yr). If a Caltech worker were to receive the maximum allowable planned dose each year for twenty years, the total dose received would be 10 rem (0.1 Sv). According to the BEIR V report, the worker's chance of death from cancer would increase by approximately 0.4%. This is fairly small compared to the normal chance of death from cancer in the U. S. of about 20%.

5. RADIATION DOSE STANDARDS AND PERSONNEL MONITORING

5.1 RADIATION DOSE STANDARDS

DOSE STANDARDS Permissible occupational radiation dose levels are set by the California Radiation Control Regulations (CRCR). The levels are based on recommendations by the International Committee on Radiation Protection (ICRP), the National Council on Radiation Protection and Measurements (NCRP), and by federal and state regulatory agencies.

The CRCR limits for adult radiation workers are listed in the table below.

Dose Category	Adult Occupational Dose Limit
Total Effective Dose Equivalent (TEDE)	5 rem/year
Total Organ Dose Equivalent (TODE)	50 rem/year to any individual organ or tissue except the lens of the eye
Eye Dose Equivalent	15 rem/year
Shallow Dose Equivalent	50 rem/year to the skin or any extremity

CALTECH ADMINISTRATIVE LIMIT

The Caltech Radiation Safety Committee has established the general policy that planned radiation doses shall not exceed ten percent (10%) of the above limits.

It should be noted that the above dose limits are permissible *occupational* dose limits. These dose limits are in addition to the background radiation dose or medical radiation dose received by the worker. The average annual background radiation in the U.S. due to natural sources is approximately 300 millirem per year.

MINORS

Radiation dose limits for radiation workers under the age of 18 are 10 percent of those listed above for adult workers.

PREGNANCY

The dose limit to the embryo/fetus of a declared pregnant woman is 0.5 rem. Efforts must also be made to avoid a dose substantially higher than 0.06 rem in one month. A declared pregnant woman means a woman who has voluntarily informed EHS, in writing, of her pregnancy and the estimated date of conception.

ALARA GUIDELINE

In practice, radiation doses should be *As Low As is Reasonably Achievable*. ALARA is a guideline meant to strike a balance between the cost of radiation protection and the health benefit derived from that protection.

It is the responsibility of everyone including radiation workers, principal investigators, radiation safety personnel and the administration to operate within the ALARA guideline. This is achievable by outlining safety procedures to minimize radiation dose by monitoring the workplace environment to control contamination and/or radiation leakage.

5.2 PERSONNEL MONITORING (EXTERNAL)

CRITERIA

The California Radiation Control Regulations require that anyone who is likely to receive a dose in excess of 10% of the maximum permissible occupational dose in any year be monitored for exposure to radiation. Although experience has shown that no one at Caltech falls into this category, the Radiation Safety Committee has determined that personnel dosimeters are to be worn by anyone in the following categories:

- Persons working with radiation producing machines deemed *open system*.
- Persons entering a posted High Radiation Area (>100 mrem/hr).

The Radiation Safety staff will determine when anyone else should be issued a dosimeter.

EXTERNAL MONITORING

Generally, a Thermoluminescent dosimeter (TLD) is used for whole body dose monitoring and it is exchanged either monthly or quarterly.

The Institute Radiation Safety Officer may require special dosimeters such as ion chambers be used in High Radiation Areas (>100 mrem/hr), certain areas be posted with environmental dosimeters, or alarmed electronic dosimeters be installed and used under certain conditions.

Dosimeters are processed commercially and dose reports are generated. These reports are reviewed by the RSO. Any unusual

or excessive doses are investigated by the Radiation Safety staff and, when appropriate, preventive measures are implemented.

PRECAUTIONS

The whole body dosimeter you have been issued is to be worn when in a laboratory or area in which radiation producing machines are used.

It should be worn at the collar or chest level to measure the radiation dose received by the trunk of the body.

The radiation dose recorded by the dosimeters is your occupational dose record. Make sure that this record is valid and accurate by observing the following precautions:

- Do not wear anyone else's dosimeter.
- When not in use, the dosimeters should be left in a place free from radiation, moisture, and high temperatures.
- Do not deliberately expose the dosimeter to radiation.
- Do not tamper with the dosimeter packet.
- If you have reason to believe that a dosimeter has been damaged, lost, or exposed accidentally, notify EHS as soon as possible so a replacement can be issued.

DOSIMETER EXCHANGE

It is the responsibility of individuals to ensure that their dosimeters are exchanged monthly or quarterly, as appropriate. In the case of a dosimeter which has been damaged, lost, or exposed accidentally, the RSO will investigate and make a dose estimate for the period in question.

DOSE HISTORY

Radiation workers interested in reviewing their dosimetry records should contact EHS. A request in writing is required for anyone interested in obtaining a copy of their dosimetry records.

6. RADIATION SURVEY METERS

INTRODUCTION

There are several types of portable radiation survey instruments in use on campus. Various types have different qualities and can therefore have very different detection capabilities.

As a radiation worker, you are expected to be able to use the survey meters in your laboratory. During your initial training, you will learn how to operate the instruments in your lab. You should know their capabilities and limitations and be able to interpret the meter readings.

GEIGER-MULLER DETECTOR

The Geiger-Mueller (G-M) counter is the most common radiation detection instrument on campus. In this type of meter, an ionization in the detector results in a large output pulse that causes meter and audio responses. Because of the inherent characteristics of the detector, all initial ionizing events produce the same size output pulse. *Therefore, the meter does not differentiate among types or energies of radiation.*

Most G-M detectors have a thin mica film "window" at one end. This window is very fragile. Always use the thin end window for detecting particulate radiation (e.g. alpha or beta particles) and low energy photons (e.g. x-rays less than 40 keV). The aluminum side wall should be used only for the detection of penetrating x-rays and gamma radiation.

Very high radiation fields may temporarily overload the detector circuit resulting in a partial or complete loss of meter or audio response. If this happens, remove the meter and yourself from the area and push the reset button or turn the meter off, then back on. The meter should resume normal operation. Always turn on a survey meter before entering an area that might have high radiation fields.

ION CHAMBER

Ionization chambers are suitable for measuring radiation exposure rate or cumulative radiation exposure at high radiation intensities. They are not especially useful at low radiation intensities, x-rays energies (<40 kV), or for detecting small quantities of radioactive material.

CALIBRATION

Most survey meters have scales that read in milliRoentgen per hour (mR/hr) and/or counts per minute (cpm) or counts per second (cps).

There is an important difference in these measurements.

After detector efficiency is taken into consideration, the cpm or cps scales gives an indication of the quantity of radioactivity. This means:

$$\text{Actual rate} = \frac{\text{meter reading}}{\text{efficiency}}$$

The mR/hr scales give an indication of the radiation exposure rate. *Exposure rate measurements are only valid for electromagnetic radiation such as x-rays or gamma radiation.*

EHS calibrates all of the portable radiation survey instruments on campus. We use two general types of calibration procedures -- one for instruments that are used for detection and measurement of particulate radiation, and another for instruments used for detection and measurement of penetrating electromagnetic radiation. The two procedures are explained briefly below so that you will understand the difference.

Survey meters used in biology and chemistry research labs are calibrated for the detection and measurement of particulate radiation. These meters are calibrated using a pulse generator so that the cpm or cps scales read correctly (i.e. one pulse in = one meter count). If the meter reads only in cpm or cps, we may place an additional calibration tag on the instrument giving the mR/hr equivalent of the count rate reading for penetrating electromagnetic radiation.

Survey meters that are used for radiation exposure measurements are calibrated with a comparable radiation source. The mR/hr scale will read correctly when the detector is exposed to electromagnetic radiation greater than 100 keV.

7. RADIATION PRODUCING MACHINES

7.1 DEFINITIONS

A *radiation producing machine* is any device capable of producing ionizing radiation when the associated control devices are operated.

A *closed system* is a radiation producing machine which satisfies the requirement that all areas with exposure rates greater than 0.25 mR/hr are enclosed within an interlocked barrier. All others are considered *open systems*.

7.2 POLICIES AND PROCEDURES FOR USE OF RADIATION PRODUCING MACHINES

AUTHORIZED USERS

Principal Investigators are responsible for ensuring that only authorized users are allowed to operate their machines. Authorized users are those individuals listed on the original application or added using the *New Radiation Worker Form*.

TRAINING REQUIREMENTS

New personnel must be trained by the Principal Investigator (or an experienced qualified operator designated by the PI) and must satisfactorily demonstrate knowledge of operating and safety procedures before independently operating a radiation producing machine.

Depending on prior experience and the type of machine to be operated, new personnel may be required to attend an x-ray safety training session offered by EHS Office and/or complete a short written test to demonstrate knowledge of operating and safety procedures.

POSTING

A sign bearing the radiation symbol and the words *CAUTION X-RAYS* must be posted at the entrance to each laboratory containing a radiation producing machine. The sign must include the name and after hours phone number of the Principal Investigator or designee.

A label bearing the words *CAUTION THIS EQUIPMENT PRODUCES RADIATION WHEN ENERGIZED* (or similar wording) must be affixed to the control device of each machine.

A label bearing the words *CAUTION HIGH INTENSITY X-RAY BEAM* (or similar wording) must be affixed on or near the tube of

an x-ray unit.

An *X-RAY ON* warning light, labeled as to its meaning, shall be located on or near an x-ray tube to indicate when x-rays are being produced.

ACCESS CONTROL

Key control must be maintained for each machine. For older machines which do not have a keyed control device, this requirement may be fulfilled by controlling access to the laboratory.

While any open system is in operation, an operator must be present or the laboratory must be kept locked.

OPERATING PROCEDURES

Operating procedures for each machine must be available near the machine. For open systems, these procedures must specifically include:

- means to control and limit doses to individuals, including controlling access to the area, and
- the use of radiation survey instruments and dosimeters.

SURVEYS

For open systems, the Principal Investigator must provide a radiation survey meter to locate the presence of unwanted radiation and to trace the origin of radiation leaks. The recommended instrument is a Geiger-Mueller survey meter with a thin window probe.

After a change in experimental set-up and before energizing the machine, the operator must visually inspect ports, cameras, shielding, etc.

After the machine is energized, a radiation survey should be performed to check for scattered or leakage radiation.

UNUSED PORTS

All unused x-ray ports must be permanently blocked or be interlocked so that x-ray production is stopped if the port is opened. Material used to block unused ports must be of sufficient density and thickness to attenuate the primary beam to acceptable levels.

SHIELDING

Protective barriers and/or shielding must be used in open x- ray systems to ensure exposure rates in accessible areas are less than 2 mR/hr at 5 cm from any exterior surface. It is important to avoid cracks and gaps in the shielding.

LOG BOOK

For open systems, a log book must be kept which records the following information:

- operator
- data
- voltage
- current
- exposure time
- radiation survey results

DOSIMETRY

Dosimetry requirements will be established by the Institute Radiation Safety Officer upon a review of each facility.

In general, users of electron microscopes or other enclosed systems will not be issued dosimeters.

Users of accelerators, radiographic units, or open system x- ray diffraction units will be issued a personnel dosimeter. This dosimeter should be worn at the collar or chest level while in the facility when the machine is in operation. The dosimeter will only measure scattered radiation; it will not indicate personnel dose from exposure to the primary beam.

EHS may post area dosimeters at various locations in the facility. These dosimeters should not be moved by laboratory personnel.

RADIATION SAFETY SURVEYS

Radiation Safety staff perform inspections of radiation producing machines when first installed, at yearly intervals, and whenever significant changes to the machine, facility, or operating procedures are made.

8. RADIATION SAFETY FOR X-RAY DIFFRACTION UNITS

8.1 NATURE OF ANALYTICAL X-RAYS

Analytical x-ray machines produce intense beams of ionizing radiation that are used for diffraction and fluorescence studies. The most intense part of a beam is that corresponding to the K emission of the target material and is called characteristic radiation. In addition to the characteristic radiation, a continuous radiation spectrum of low intensity is produced ranging from a very low energy to the maximum kV-peak setting. This is referred to as "bremsstrahlung" or white radiation. Undesirable wavelengths may be filtered out using a monochromator.

Figure 8.1 shows the schematic view of an x-ray tube and Figure 8.2 illustrates spectrum of a Mo target.

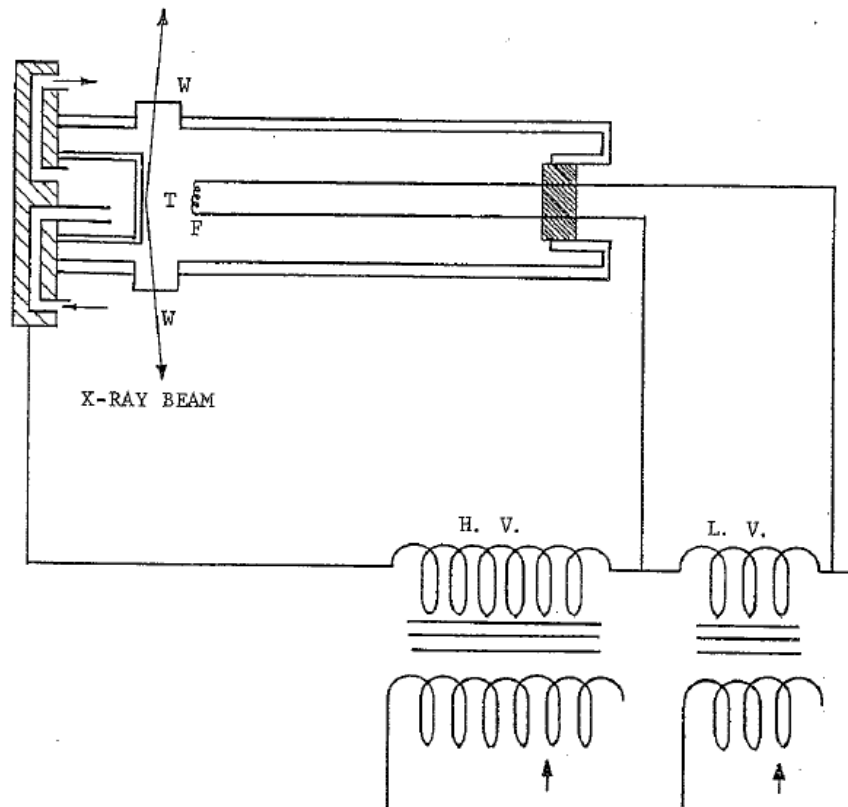


Figure 8.1- Schematic view of x-ray tube and electric circuit.

T: water -cooled target; F: tungsten filament; W: windows
H.V.: high-voltage transformer; L.V.: low voltage transformer.

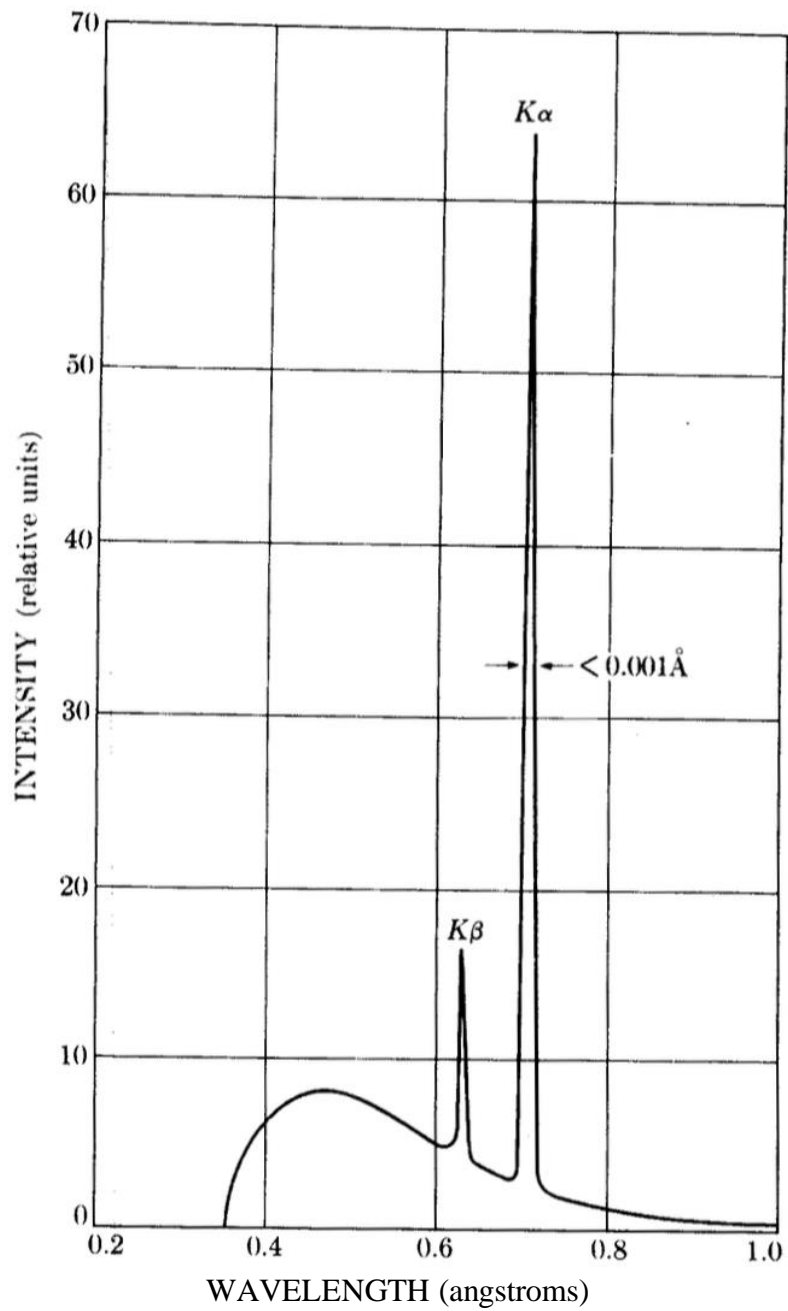


Figure 8.2- Spectrum of a Mo target at 35 kV. Line widths not to scale.

X-ray diffraction wavelengths (λ) are selected so as to roughly correspond to the inter-atomic distances within the sample, and to minimize fluorescence.

Wavelengths commonly used are 1.54 Å (Cu targets), 0.71 Å (Mo targets), and 0.56 Å (Ag targets).

The relationship between wavelength and x-ray photon energy is determined by the equation:

$$E = hc/\lambda$$

where E = energy in ergs ($1 \text{ eV} = 1.6 \times 10^{-12} \text{ erg}$)
 H = Planck's constant = $6.614 \times 10^{-27} \text{ erg-sec}$
 C = velocity of light = $3 \times 10^{10} \text{ cm/sec}$
 λ = wavelength in cm ($1 \text{ Å} = 1 \times 10^{-8} \text{ cm}$)

K_{α} wavelength and energy values for various target materials are listed in the tables below:

Target	Wavelength (Å)	Energy (keV)	Target	Wavelength (Å)	Energy (keV)
Ti	2.75	4.51	Cu	1.54	8.04
Cr	2.29	5.41	Zn	1.44	8.63
Mn	2.10	5.90	Mo	0.71	17.4
Fe	1.94	6.40	Rh	0.61	20.2
Co	1.79	6.93	Pd	0.59	21.1
Ni	1.66	7.47	Ag	0.56	22.1

X-rays emitted from an open, uncollimated port form a cone of about 30°. The x-ray flux can produce a radiation field at one meter on the order of 10,000 R/hr. A collimator reduces the beam size to about 1 millimeter diameter.

8.2 X-RAY HAZARDS AND BIOLOGICAL EFFECTS

X-rays produced by diffraction machines are readily absorbed in the first few millimeters of tissue, and therefore do not contribute any dose to the internal organs of the body. However, the lens of the eye can receive a dose from x-rays of this energy.

Overexposure of lens tissue can lead to the development of lens opacities and cataracts.

Absorbed doses of a few hundred rem may produce a reddening of the skin (erythema) which is transitory in nature. Higher doses -- 10,000 rem and greater -- may produce significant cellular damage resulting in pigment changes and chronic radiation dermatitis. Exposure to erythema doses may not result in immediate skin reddening. The latent period may be from several hours to several days.

(Note: X-rays used for medical diagnosis are about one order of magnitude shorter in wavelength. Diagnostic rays are designed for tissue penetration and are carefully filtered to avoid x-ray damage to the skin caused by the longer, more readily absorbed wavelengths.)

8.3 SOURCES OF IONIZING RADIATION

The primary beam is not the only source of ionizing radiation. Any high voltage discharge is a potential source of x-rays. Faulty high-voltage vacuum-tube rectifiers may emit x-rays of twice the voltage applied to the x-ray tube. Other sources of ionizing radiation are:

- Secondary emissions and scattering from the sample, shielding material, and fluorescent screens.
- Leakage of primary or scattered x-rays through gaps and cracks in shielding.
- Penetration of the primary beam through or scattering from faulty shutters, beam traps, or collimator couplings.

8.4 SAFETY PRECAUTIONS AND NOTES

The shielding, safety equipment, and safety procedures prescribed for x-ray diffraction equipment are applicable only for up to 75 kV- peak x-rays. Additional or greater precautions are necessary for machines operating at higher voltages.

The PI has the primary responsibility for providing a safe working environment by ensuring that equipment is operationally safe and that users understand safety and operating procedures.

The equipment operator is responsible for his own safety and the safety of others when using an analytical x-ray machine.

Prior to removing shielding or working in the sample area, the operator must check both the warning lights and the current (mA) meter on the console. *Never trust a warning light unless it is on!* Always use a survey meter to check that the shutters are actually closed if current is still being supplied to the tube. It is possible for a shutter to be stuck partially open even when the indicator shows that it is shut. *The best way to avoid an accidental exposure is to turn the machine off before working in the sample area.*

Never put any part of the body in the path of the primary beam. Exposure of any part of the body to the collimated beam for even a fraction of a second may result in damage to the exposed tissue.

A person not knowledgeable about x-ray equipment should not attempt to make repairs or remedy malfunctions. If you suspect a machine is malfunctioning, turn it off or unplug it. Place a note on the control panel and inform the PI or his designated representative.

Repairs to the high voltage section must not be made unless the primary leads are disconnected from the high voltage transformer and a signed and dated notice is posted near the x-ray ON switch. Turning off a circuit breaker is not sufficient. Follow appropriate Lockout/ Tagout procedure.

Bare feet are not permitted in the laboratory or around electrical equipment. Even slightly moist skin is an excellent electrical conductor and contact with faulty, ungrounded equipment may result in severe injury or death.

Do not attempt to align x-ray cameras without first consulting an experienced person. Alignment procedures require special training and knowledge.

Special care is required when one power supply is connected to more than one x-ray tube.

EYE PROTECTION

The use of safety glasses or prescription lenses is encouraged when working with analytical x-rays. While glasses cannot be depended upon to provide complete protection to the eyes, they can reduce x-ray exposure. Glass provides about 10 times the protection of plastic

for low energy x-rays. Neither, however, will adequately protect the eye from direct exposure to the primary beam.

FLUORESCENT SCREENS

It is unsafe to inspect an x-ray beam with a fluorescent screen without special precautionary measures. Notify EHS before performing a procedure using a fluorescent screen.

TUBE STATUS INDICATORS

There must be a visual indication located on or near the tube head to indicate when x-rays are being produced. This is usually an assembly consisting of two red bulbs, wired in parallel and labeled *X-RAYS ON*. If one of the lights is burned out, the operator should either replace it before leaving the room, or leave a note on the light assembly indicating that the bulb is burned out. An unlit warning bulb does not necessarily mean that x-rays are not being produced. Always check the control panel.

SAFETY DEVICES

Interlock switches are used to prevent inadvertent access to the x-ray beam. They should not be bypassed. Interlocks should be checked periodically to ensure that they are functioning properly.

Interlocks and other safety devices and warning systems are not foolproof or fail-safe. A safety device should be used as a back-up to minimize the risk of radiation exposure -- never as a substitute for proper procedures and good judgment.

9. EMERGENCY PROCEDURES

RADIATION PRODUCING MACHINES

In the event of an accident or unusual incident involving a radiation producing machine:

1. **TURN OFF THE MACHINE.** If possible, also unplug or shut off the circuit breaker for the machine or follow appropriate Lockout/Tagout procedures.
2. If there is a serious injury or fire, call x5000 or 626-395-5000 and request paramedics or the fire department.
3. Notify the EHS (x6727 or 626-395-6727). If after working hours, call x5000 or 626-395-5000 and have the operator contact an EHS representative.
4. Notify the laboratory supervisor or Principal Investigator.
5. Record information about the incident (e.g. operating voltage and current, exposure time, and distance from radiation source.)