

FOOD AND DRUG ADMINISTRATION OFFICE OF REGULATORY AFFAIRS <i>ORA Laboratory Manual Volume IV Section 12</i>	Document Number: IV-12	Revision #: 02 Revision Date: 06/30/2020
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1. Radiation Safety

1.1. Objective

The purpose of this section is to ensure that everyone working in the laboratory environment is aware of the radiation sources which might be present in the laboratory. Common Radiation sources which might be present in the laboratory will be enumerated and in so doing, policies and procedures to ensure that any radiation exposures are kept as low as reasonably achievable will be discussed. Laboratorians may be exposed to both ionizing (i.e. possessing enough energy to strip electrons from atoms) and non-ionizing radiations in the performance of their work. These radiations may be electromagnetic in nature (e.g. radiofrequency, optical, x-rays and gamma rays) or particulate (i.e. alpha and beta particles). The sources of the radiations may be radiation emitting electronic products or radioactive material. Many laboratory operations require the use radioactive material (e.g. Uranyl acetate as a microbiological stain) or radiation emitting electronic products (e.g.

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microwave digesters). The following sections will provide information on the safe use of these products.

This section pertains to all analysts, technicians, microbiologists and engineers who work in a laboratory setting as well as their supervisors and managers. Radiation safety is everyone’s responsibility.

1.2. Assignment

This section will first cover electronic products and provide guidance on their safe use; a similar pattern will be used in the discussion of the radioactive material. It should be noted that unlike radioactive material, electronic products such as x-rays or lasers do not pose a safety hazard when powered off.

1.2.1. Electronic Products

Microwave Digesters use radiofrequency radiation to digest samples. They produce microwaves at a frequency of 2450 Megahertz. These units may resemble a typical microwave oven, however there is no federal performance standard for these units. The biological effects of microwave radiation are thermal in nature (i.e. erythema and cataractogenesis). To prevent exposure to microwaves, follow the user manual and ensure that the door gasket is clean and that the door is not loose prior to use. Interlocks should be checked in accordance with the user manual. Microwave leakage should be checked annually and should not exceed 5mW/cm².

LASERS serve many purposes in the laboratory. They emit electro magnetic radiation ranging from far infrared region to the far ultraviolet region (1000 μm to 180 nm). A federal performance standard does exist for laser products and ANSI Standard Z136.1 address the safe use of lasers. Additionally, ORA has issued a laser safety manual for laboratory users. Laser products are classified by the hazard they present. Class 1 laser products are inherently safe. Class 2 lasers emit visible light and depend on the eye’s aversion reaction (~0.25 seconds) to restrict exposure. The maximum power for a Class 2 laser is 1 mW. Class 3 laser products have a maximum power output of 5 mW. Class 3B lasers have beams whose power ranges between 5 and 500 mW. Class 4 lasers have outputs more than 500 mW. When using Class 3B and Class 4 lasers the use of proper eyewear is mandatory.

It should be noted here that a Class 1 laser product may contain a Class 3B or 4 lasers. If an operation requires that the laser needs to be removed from its protective housing, proper signage and precautions must be taken. The Laser Safety Officer shall be consulted prior to this happening.

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The biological effects from laser exposure are dependent on the wavelength of the optical radiation emitted. The eye is the critical organ, skin can also be affected by the beam. In addition to beam hazards, collateral hazards also exist with the use of high-powered lasers. These include electrical hazards, collateral radiation hazards and laser generated air contaminants (LGAC).

Laser products in the laboratory commonly include:

- A. Laser cutters
- B. Laser ablation units
- C. Laser microscopes
- D. Raman mass spectrometers
- E. Positioning lasers

All users of lasers must pass the ORA Laser safety course, be familiar with the ORA laser safety manual and follow all recommendations in the user manual which accompanied the laser product. Laser safety eyewear should be inspected before use to ensure proper wave length protection and optical density. Contact Laser Safety Officer for information on the ORA Laser Safety Course

Analytical X-ray systems include x-ray fluorescence units (XRF) and x-ray diffraction units (XRD). The radiation emitted by these devices is classified as ionizing. Users of these devices must have taken a radiation safety course such as OTED’s RH 102. All users of these devices shall wear ring and whole-body dosimeters when working with the device. The ring badge should be worn on the dominant hand. Users of these systems should be familiar with the user manual and must check interlocks regularly as described in the manual. The ORA XRF SOP describes the required safety procedures. XRF and XRD units must be tested for leakage at regular intervals, the user manual describes the frequency at which these units must be monitored. Leakage radiation should be no greater than 0.25 mRem/hr at 5 cm from any accessible surface of the analytical x-ray unit.

Scanning Electron Microscopes can produce x-rays and should be monitored on a regular basis – yearly and after maintenance. Additionally, the user should be familiar with the user manual and perform all checks and maintenance in accordance with the manual. Stray radiation from the electron microscope should not exceed 0.5 mRem/hr at 5 cm from any accessible surface of the unit.

Note: analytical x-ray systems and electron microscopes produce low energy x-rays and special detectors are required to survey the units. The individual performing the surveys must be a qualified expert.

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1.2.2. Radioactive Material

Safety regulations pertaining to radioactive material at ORA fall under distinct Nuclear Regulatory Commission (NRC) or Agreement State Licensure types. Each licensure type has unique regulations that specify safety procedures and possession requirements. Therefore, it is crucial for facilities to have an accurate inventory of radioactive material which includes licensure category. For more information, see ORA.006 ORA Radiation Safety Manual.

Facilities may possess radioactive material under one of the following NRC licensure types: 1) NRC Specific License, 2) NRC General License, 3) Exempt Distribution License. Typically, ORA facilities possess radioactive material that fall under General or Exempt Distributed Licenses.

Specifically Licensed Radioactive Material

ORA holds an NRC Broad Scope Type A License that allows the Winchester Engineering and Analytical Center (WEAC) to use loose forms of alpha, beta, and gamma emitters. All ORA locations specifically listed under this license must follow all safety instructions stated in the license (e.g. authorized use, training, disposal). For requests to be added to the license, please contact ORA's Radiation Safety Officer.

In addition to the radioactive material authorized for use at WEAC, ORA's NRC License authorizes the use of tritium labeled Saxitoxin at the following laboratories:

- A. ORA Northeast Laboratory
- B. ORA Pacific Northwest Laboratory
- C. CDER OTR OPQO Division of Pharmaceutical Analysis (CDER-DPA)

Generally Licensed Radioactive Material

NRC Generally Licensed radioactive material typically includes radioactive material contained in a sealed source within a shielded device or capsule. These devices are designed with inherent radiation safety features, so they can be used by persons with limited or no radiation training or experience and are typically found in the following laboratory instruments or supplies:

- D. Electron Capture Detectors (ECD) in Gas Chromatographs (GC) containing Ni-63
- E. Ion Mobility Spectrometers (IMS) containing Ni-63
- F. Liquid Scintillation Counters containing a built-in source (i.e. Co-60, Cs-137, Eu-152)

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- G. X-ray fluorescence units (XRF) containing a gamma emitter (i.e. Cd-109)
- H. Static eliminators containing Po-210
- I. In-vitro laboratory kits containing H-3 or C-14.
- J. Small quantities of Thorium or Uranium (see [10 CFR 40.22](#)) greater than 0.05% by weight but less than 1.5 kg.
- K. Radium in quantities less than 1 microCi (see [10 CFR 31.12](#))

End-users automatically become General Licensees when they purchase these instruments and are required to comply with NRC Regulations (See 10 CFR 31) regarding the registration, leak testing, transfer, reporting, and disposal of these Radioactive materials. In addition to NRC regulations, ORA radiation safety policies that include procuring, utilizing, labeling, storing, surveying, tracking, disposing as well as training and dosimetry requirements must be followed by lab personnel. See ORA.006 ORA Radiation Safety Manual for more details.

Exempt Distributed Licensed Radioactive Material

Some manufacturers hold an NRC Exempt Distribution License and produce radioactive materials that are exempted from licensing requirements. The NRC deems that these products and types of uses do not constitute an unreasonable risk to the common defense or security or to public health and safety and the environment. Radiation safety features are built into the sealed source or device or the amount of radioactive material that can initially be distributed in such a device is restricted. Typical Exempt Distribution radioactive materials in the laboratory include:

- A. Radioactive Check sources containing a gamma emitter (i.e. Cs-137)
- B. Liquid scintillation calibration and quality standards (i.e. H-3 and C-14)
- C. Some types of Ion Mobility Spectrometers containing Ni-63.
- D. Very small quantities of loose radioactive material

Although, there are no formal regulatory requirements for exempt-distributed material, ORA radiation safety policies that include procuring, utilizing, labeling, storing, surveying, tracking, disposing as well as training and dosimetry requirements must be followed by lab personnel. See ORA.006 ORA Radiation Safety Manual for more details.

1.3. Questions

Useful Equations: $c = \lambda\nu$

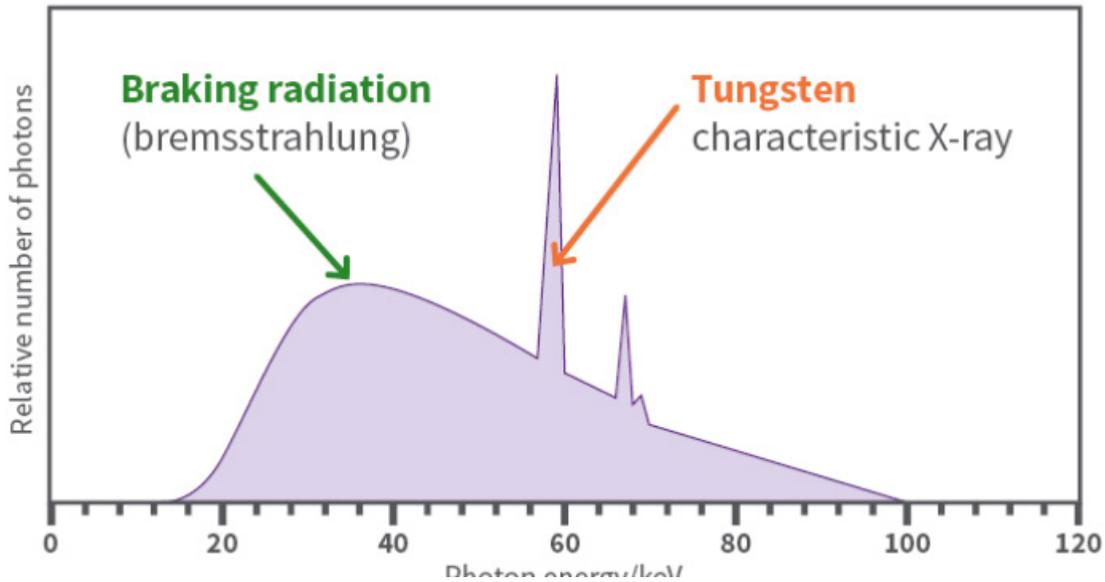
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$$E = hv \quad h=6.6 \times 10^{-27} \text{erg-s}$$

Where E is the energy of a photon

u is its frequency

λ is its wavelength



1. Describe how the two types of x-ray were generated and their utility in the laboratory.

Bonus: What is the maximum energy (kVp) of the x-rays produced.

Answer:

Bremsstrahlung is x-rays are produced whenever electrons are decelerated and give up energy. Characteristic x-rays are produced when an inner shell electron is ejected via a photoelectric interaction with an x-ray and an outer shell electronic fills the void created by the interaction.

Bremsstrahlung is always created when electrons are accelerated and subsequently slowed via an interaction with a target. It is the cause for radiation exposure from SEM's and analytical x-ray systems. Characteristic x-ray can be used to identify the material that was struck by the x-ray. In this case a 100 kVp beam struck a tungsten target.

2. Give an example of a laboratory instrument that uses the following radiations:
Microwaves, Infrared, Visible light, UV, Ionizing radiation

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Answer:

Microwave digester

Raman Spectrometer

Laser, colorimetric analytical tools

Germicidal lamps. Lasers.

Analytical x-ray systems, scanning electron microscopes, radioactive materials

3. **True** or False The FDA inspects laser light shows.
4. **True** or False laser pointers cannot exceed 5 milliwatts, but a green laser pointer owned and used by the FDA may not exceed 1 milliwatt.
5. When working with a Raman Spectrometer with a wave length of 1064 nm what are the biological effects that might be expected.

Answer:

At the specified wavelength, the retina of the eye is of the most concern, followed by the skin. Depending upon the energy of the laser beam injuries could range from minor burns to the skin, to scotomas on the retina, rupture of blood vessels in the choroid layer or blindness.

6. True or **False** When using a class 4 laser, one need not worry about non-specular reflections.
7. True or **False** All Facilities containing radioactive material have a Nuclear Regulatory Commission License
8. Give Examples of Generally Licensed Radioactive Material

Answer:

Electron Capture Detector, Ion Mobility Spectrometer, Static Eliminators, Uranium Acetate

9. Describe the Principles of Time Distance and Shielding

Answer:

Time: Limiting or minimizing the exposure time reduces the dose from radiation sources.

Distance: Dose of radiation decreases dramatically as you increase your distance from the source. Doubling the distance away from the source reduces the dose to one quarter its original value.

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Shielding: Shielding a radiation source will also reduce exposures. Lead or concrete work best for gamma emitters or x-rays. Plastic works best for high energy beta emitters.

10. True or **False** Dosimeters protect against radiation exposure
11. True or **False** Radioactive material always pose an external exposure hazard
12. True of **False** Lead is an effective shield against gamma radiation and beta radiation

2. Radionuclides in Foods Program

2.1. Objective

The purpose of this section is to train analysts in various procedures in the Radionuclides in Foods Program. The food program may include, but not limited to, the following training assignments:

- A. Tritium
- B. Gross alpha
- C. Gross beta
- D. Plutonium 238, 239

2.2. Assignment

The trainer will provide the training in various methodologies as described in the training SOP, e.g. reading, demonstrations, self-study, on the job training. This includes the radiochemical procedures as well as instrument use

Training samples may be reserved food samples (e.g. dried mushrooms, jam, leafy vegetables) or proficiency samples. Initial training covers analyses of analyses of gamma emitting radionuclides and Strontium 90.

2.3. Questions

1. **What is the purpose for using the 400-mL fill line in the analysis of gamma-emitting radionuclides?**

Answer:

The 400-mL fill line is the level to which a plastic sample container is filled in order to maintain a constant geometry for all samples counted on the detector. The detector is also calibrated using a radioactive source in an identical plastic container filled to the 400-mL line.

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2. Describe the separation methods utilized for Sr-90 in the Market Basket Strontium 90 method.

Answer:

In this method the activity of Y-90, the decay product of Sr-90 present in radioactive secular equilibrium with Sr 90 in the sample, is measured. A known quantity of food homogenate is dried and charred at high temperature, and the ash is dissolved in nitric acid. The acid solution is shaken in a separatory funnel with tributylphosphate and strontium and yttrium carrier solutions. Y-90 is differentially dissolved (or separated from Sr-90) in the organic phase. After a series of procedures, Y-90 as oxalate is deposited onto a paper filter and assayed using a beta particle counter. The Y-90 activity is then used to calculate the Sr-90 activity in the original food sample.

3. Describe the sample preparation for the analysis of tritium by liquid scintillation analysis.

Answer:

A weighed, edible portion of sample is transferred to a special glass distillation tube. An identical tube, used as a water trap, is joined to this tube and to a vacuum pump via an adapter. The sample tube is sealed and mildly heated, and the water trap is evacuated using the pump, and chilled. The two tubes are then joined together via the adapter and water vapor from the sample is gradually collected in the cold trap. The collected water is then mixed with liquid scintillator solution (sometimes referred to as a "cocktail") which produces visible light when ionizing radiation (emitted by tritium) passes through it. The light pulses are detected and counted in the liquid scintillation counter.

3. Radiation Detection Instrumentation

3.1. Objective

To familiarize the trainee with the radionuclide laboratory equipment.

3.2. Assignment

The trainer will discuss the location, care, and calibration procedures for equipment found in the radionuclide laboratory. The training exercises include calibration of the instruments defined below. Typical equipment found in the radionuclide laboratory may include the following:

1. low level gas-flow proportional counter,
2. low background beta counter,
3. liquid scintillation counter,

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4. alpha spectrophotometer,
5. ionization chamber,
6. solid state silicon surface barrier detector,
7. high purity germanium detector,
8. sodium iodide detector
9. ICP Mass Spectrometer , and
10. x-ray detector.

3.3. Questions

Describe the theory of operation and its application in the laboratory for the following: a proportional counter, a scintillation counter, and a solid-state detector coupled to a multichannel analyzer

Answer:

A proportional counter consists of a gas-filled chamber containing two electrodes across which an electrical potential is applied. Under the influence of the electric field, ions generated by radiation in the gas are collected, producing an output signal proportional to the energy deposited in the counter by the radiation. Therefore, particle identification and energy measurement are possible for any charged particle. A scintillation counter consists of a medium that produces light when ionizing radiation passes through it, and a detector, usually a photomultiplier tube, which amplifies the light and produces an output signal. The scintillator may be solid, liquid, or gas. Photons, neutrons, and charged particles may be detected using scintillation counters. A solid-state detector consists of a semiconductor crystal across which a bias voltage is applied. Charges generated in the crystal by radiation are collected and produce a voltage pulse proportional to the energy deposited in the crystal. The pulse-height distribution or spectrum may display using a multichannel analyzer (MCA) and consists of a plot of counts versus energy

4. Document History

Revision #	Status* (D, I, R)	Date	Author Name and Title	Approving Official Name and Title
1.2	R	02/06/2012	LMEB	LMEB
1.3	R	02/14/2013	LMEB	LMEB
02	R	06/30/2020	LMEB	LMEB

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* - D: Draft, I: Initial, R: Revision

5. Change History

Revision #	Change
1.2	Table of Contents – deleted Quality Assurance/Quality Control; added Document History Section 12.1 Introduction – revised Section 12.2 C. – Questions 7. and 9. revised; added 10. and 11. Section 12.2 D. – References 1. and 2. updated; added WEAC Radiation Safety Manual SOP Section 12.3 B. – Bullets 1, 2., and 5. revised; added ICPMS Section 12.3 C. – deleted 2. Section 12.3 D. – updated 1. and 2.; added WEAC Radiation Safety Manual SOP Section 12.4 A. and B. – revised Section 12.4 C. - deleted 2. and 5. Section 12.4 D. – updated 9. Section 12.5 A. and B. – revised Section 12.5 C. – updated 9. Section 12.6 – deleted Section 12.7 Answer Key (now 12.6) – added 10. and 11. to 12.2; deleted 2. from 12.3; deleted 2. and 5. from 12.4; deleted 12.6
1.3	Header – Division of Field Science changed to Office of Regulatory Science
02	Section 1: expanded to include different categories of radiation producing devices and radioactive materials

6. Attachments

None