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RECYCLING AND REUSE OF RADIOACTIVE MATERIALS

by

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Bachelor of Science in Radiological Health Physics Lowell Technological Institute 1974

Master of Science in Radiological Science and Protection University of Lowell 1981

A dissertation submitted in partial fulfillment of the requirements for the

Doctor of Philosophy in Radiochemistry

Department of Chemistry College of Science Graduate College

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THE GRADUATE COLLEGE

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ABSTRACT

Recycling and Reuse of Radioactive Material

by

Thomas Joseph O'Dou

Dr. Kenneth Czerwinski, Examination Committee Chair Professor of Chemistry University of Nevada, Las Vegas

The Radiochemistry Program at the University of Nevada, Las Vegas (UNLV) has a Radiation Protection Program that was designed to provide students with the ability to safely work with radioactive materials in quantities that are not available in other academic environments. Requirements for continuous training and supervision make this unique program capable of turning out graduates that have an understanding of contamination and dose control techniques that complement their knowledge of the elements that they work with. The Program has also adopted a radionuclide recovery and reuse program that has provided materials from other universities, government agencies, and private companies for use in experiments.

ACKNOWLEDGEMENTS

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Lastly, some great friends assisted me in activities at the Harry Reid Center, Trevor Low, Michelle Bossler, and Julie Bertoia and the students of the UNLV Radiochemistry program from the beginning. Thank you for your help, friendship, and confidence!

Thomas Joseph O'Dou

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CHAPTER 1

RECOVERY OF RADIOACTIVE MATERIALS FOR REUSE RESEARCH OVERVIEW

1.0 Radionuclides Used in the UNLV Radiochemistry Program

The radiochemistry program at the University of Nevada Las Vegas (UNLV) is a comprehensive, research intensive program that is collaboration between the Departments of Health Physics and Chemistry. The research within the program focuses on the radioelements, primarily the actinides and technetium (Table 1-1). The isotope ⁹⁹Tc, with a half-life of 2.13×10^5 years and beta emission energy (E_{max}) of 293.7 keV is used in a range of studies. Isotopes of thorium, uranium, neptunium, plutonium, americium, and curium have been examined in solutions and solid phases. These research efforts span from basic studies to applied experiments. All this research effort necessitates the usage of radionuclides. Obtaining these radionuclides, and using them in a safe manner, is central to the success and capabilities of the program.

This combination of work with actinides and ⁹⁹Tc and the provision of safety make for a unique learning atmosphere within the PhD radiochemistry program. This combination also provides an opportunity to develop, evaluate, and document issues associated with radionuclide usage in an educational setting. The recycling of radionuclides from in house experiments or outside sources provides a means to preserve a relatively rare resource. Additionally, the ability of students to work with these radioelements necessitates the development of a unique radiation safety program. These novel attributes of the UNLV Radiochemistry program are explored and used as a basis for developing a means of radionuclide reuse and documenting the necessary radiation safety for their use in experiments. This combination of radionuclide usage and applied health physics is exceptional and provides the basis for the unique contribution contained within this document.

Obtaining and using radioactive materials are cost-driven activities. Often it is difficult to obtain suitable concentrations of radioelements. Initially their purchase may be high, if obtainable, and the analysis of their properties may become expensive. Reuse of experimental residue by recovering the radionuclide to an identifiable chemical form provides a route to reduce costs and supply a scarce resource. A component of this project focuses on the recycling and reuse of radioactive materials. This reduces procurement and disposal costs, and minimizes the generation of hazardous waste products that contain radioactive materials.

Another unique aspect of this work was development of the radiation safety program for measurable masses of radionuclides in an academic setting. The vision was a defined program that allowed research use of technetium and actinide isotopes in experiments. These experiments require quantities that have contamination control and serious exposure implications. The program development was facilitated by unprecedented changes to the radioactive materials license, procurement of control equipment, and training. Use of long lived radionuclides by many faculty members, post-doctoral candidates, visiting scientists and students in many laboratories on campus has changed UNLV's image in the research community. The radiation safety described in this work is a central component to the noted and acknowledged radiochemistry capabilities at UNLV.

Nuclide	Half-Life	Specific Activity	Annual Limit on Intake	Emissions
⁹⁹ Tc	2.13x10 ⁵ a	6.27x10 ⁸ Bq/g	2.59x10 ⁷ Bq	β
²³² Th	1.41x10 ¹⁰ a	4.04x10 ³ Bq/g	3.7x10 ¹ Bq	γ, α
233U	1.62x10 ⁵ a	3.5x10 ⁸ Bq/g	1.48x10 ³ Bq	γ, α
235U	7.1x10 ⁸ a	7.9x10 ⁴ Bq/g	1.48x10 ³ Bq	γ, α
238U	4.46x10 ⁹ a	1.25x10 ⁴ Bq/g	1.48x10 ³ Bq	γ, α
²³⁷ Np	2.14x10 ⁶ a	2.61x10 ⁷ Bq/g	1.48x10 ² Bq	γ, α
²³⁹ Pu	2.44x10 ⁴ a	2.27x10 ⁹ Bq/g	2.22x10 ² Bq	γ, α
²⁴¹ Am	4.29x10 ² a	1.28x10 ¹¹ Bq/g	2.22x10 ² Bq	γ, α
²⁴³ Am	7.94x10 ³ a	6.86x10 ⁹ Bq/g	2.22x10 ² Bq	γ, α
²⁴⁴ Cm	1.76x10 ¹ a	$3.08 \mathrm{x} 10^{12} \mathrm{Bq/g}$	3.7x10 ² Bq	γ, α

Table 1-1. Common Radionuclides used in Radiochemistry at UNLV

1.1 Sources for Procurement of Radionuclides for Experiments

In the laboratory the option of using milligram to kilogram quantities of these radionuclides enhances the ability to observe the result of chemical reactions in both the solution and solid phase. Radionuclides that are used in experiments are usually obtained commercially from companies that purchase larger quantities and dispense them in solutions, solids, or plated on materials [1]. The source of those radionuclides is typically government laboratories, research or production reactors, or laboratories with the ability to produce radionuclides using an accelerator [2]. The availability of radionuclides is typically associated with the demand. If the demand is low, it may only be available at times when the need is demonstrated and the conditions are right for production. For the UNLV radiochemistry program, radionuclide needs are often independent of this demand.

For some radionuclides, costs and availability can hinder or prevent experiments. There is no standard for determination of the cost as prices vary widely between vendors. Exploring the availability and pricing options will provide an extensive array of both (Table 1-2). For less commonly used radionuclides, such as transuranic elements, ²³⁷Np, ²³⁹Pu, ²⁴³Am, this expense can increase substantially. Two prime considerations in the cost of the material are the quality of both the compound and the quality of the radionuclide analysis. Activity that is traceable to a standardizing entity [3] such as the National Institute of Standards and Technology (NIST) in the United States or Physikalisch-

Technische Bundesanstalt (PTB) in Europe has a large fee associated with support of the facility. The cost is typically associated with the need for calibration of specialty measurement equipment and specially trained personnel. The compound available for high activity, long lived radionuclides is typically a chemically stable molecule that may be suitable for the intended work. In many cases, chemical methods are applied to change the compound with the intention of making novel applications of radionuclides, or providing a platform from which to synthesize other compounds.

Compound	Radionuclide	Manufacturer/Date	Cost (\$ per MBq)
Uranyl Nitrate Hexahydrate UO ₂ (NO ₃) ₂ ·6H ₂ O	238U	International Bio- analytical Industries, Inc. 2006	\$321.00
Ammonium Pertechnetate NH ₄ [TcO ₄]	99Tc	Oak Ridge National Laboratory 2006	\$1.59
Plutonium Nitrate Pu(NO3)4	²³⁹ Pu	Ekert & Ziegler Isotope Products Laboratory 2006	\$121.89
Neptunium Nitrate NpO(NO3)	²³⁷ Np	Ekert & Ziegler Isotope Products Laboratory 2006	\$70.74
Americium Chloride AmCl ₃	²⁴¹ Am	Ekert & Ziegler Isotope Products Laboratory 2006	\$29,054.00

Table 1-2. Cost of Radioactive Compounds used at UNLV Since 2006

In 2007, an in-house price comparison was done to determine the cost and availability of ²⁴⁴Cm for experimentation. Estimates were received from Oak Ridge National Laboratory and Ekert & Ziegler Laboratories. The cost of 0.5 mg, 1500 MBq, from the national laboratory was \$5682.00. The cost of 1.2 μ g, 3.7 MBq, of this radionuclide from a commercial laboratory was \$2300.00 for a nominal solution (±15%) and \$2860.00 for a calibrated (±2.5%) solution [4]. While the purchase from the national laboratory is attractive from a quantity standpoint (400 times as much for 2 times the price), consideration must be given to the quantity needed for the research and the quality of the product. As a result of this comparison, the decision was to purchase the calibrated solution from the commercial laboratory for the experiments. This solution was provided at a lower cost with a certified analysis. Another benefit of this purchase was a minimization of total activity, enhancing contamination control capabilities.

1.2 Disposal of Radioactive Material

In the United States disposal of radioactive material is a controlled process that provides a high degree of assurance that radioactive wastes will be transferred from a radioactive material licensee to a licensed radioactive waste disposal site. The foundation for the process of controlling waste is in Nuclear Regulatory Commission regulations, Title 10 of the Code of Federal Regulations, Energy [5].

Each of the low level radioactive waste disposal sites in the United States have restrictions on the type of materials that are allowed [6]. For example, the US Ecology Richland Washington facility may not receive mixed waste. Mixed wastes are materials that generate toxic gases, vapors, or fumes, or pyrophoric, hazardous, dangerous, or explosive items. This exclusion of mixed waste is a necessary measure to protect the integrity of the site [7] [8]. The impact on the radiochemistry program is associated with the need to comply with regulation and prevent mixed waste production, as limited disposal is available.

The costs of disposal at commercial sites may be associated with the volume, mass, activity, activity concentration, radionuclides present, and any hazardous chemicals that may require processing before the waste is accepted for burial [9]. The most recent price for disposal of dry low level radioactive waste generated at UNLV in 2009, has been approximately \$1000.00 per 55 gallon (208 L) drum [10]. UNLV uses a 'waste broker' [11] [12] to facilitate the removal of these wastes from the campus and their delivery to a licensed low level radioactive waste site. A waste broker is a commercial service that will collect and combine wastes from other facilities, minimize waste volume through compaction, and deliver the waste to a licensed disposal facility. Use of a broker is important to reduce waste costs, eliminate transportation costs and provide expert consultation regarding the site to be used for disposal.

The waste collected for disposal in the radiochemistry laboratories is non-radioactive waste, non-radioactive hazardous waste, or low level radioactive waste. These wastes may be either solid or liquid. There are currently four listed wastes, F-list (from non-specific sources), K-list (from specific sources), P-list (acutely hazardous discarded commercial chemical products), and U-list (discarded commercial chemical products) [13]. Listed and characteristic hazardous waste require controlled disposal. Treatments such as neutralization or solidification [14] may be necessary to allow their proper burial. This is of course also true of the disposal practices for radioactive wastes that have a hazardous component in order to prevent damage to the waste site from the hazardous waste.

Disposal costs for mixed waste are associated with the removal or neutralization of the hazardous component. If processes are not available to separate these materials, the waste may be solidified or encapsulated to allow disposal. Since each of these processes requires special facilities, resources, and trained personnel, the costs of disposal are high for mixed waste compared to solely radioactive waste. An exact price for disposal of mixed wastes requires a detailed analysis of the radioactive and hazardous components of the waste by a certified laboratory and an evaluation of the processing and disposal costs of the resulting components by the commercial disposal facility. It is therefore prudent to prevent the generation of mixed waste and if it cannot be prevented, its volume and hazardous nature must be minimized to allow removal of the hazard or removal of the radionuclide.

1.3 Use of Radioactive Materials in Experiments

Radioactive materials are used in research to identify the behavior of radionuclides in chemistry [15], identify properties for commercial exploitation [16], evaluate their transport in the human body and the environment [17] [18], and determine safe means for their disposal [19]. Together with the Authorized Radioactive Material User, the researcher decides to use radioactive materials available in storage or from a commercial vendor. The decision will provide two benefits if recycled material is available, the material can be dispensed to the researcher within a short time and the material has no financial drain on the research funds. If the decision is made to purchase radioactive materials, there may be a waiting period while the radio-chemicals are prepared, usually up to 6 weeks. Additionally there is a cost and time component associated with commercial radionuclide procurement. The sequence of obtaining and using radioactive materials in the radiochemistry laboratories at UNLV is explained in Figure 1-1.

The User has the overall responsibility for the radioactive material in their laboratories. The User assigns a laboratory and equipment for the trained researcher to work. A protocol is devised that provides technique, equipment, safety, storage and waste considerations associated with the radionuclide(s) and experiment [20]. Use of the material is documented in a tracking log [21], and the experiment proceeds. While the experiment is in progress, the researcher is responsible to document disposal and the content of residues that may

be useful for recovery. Upon completion of the experiment a recovery analysis may be useful to identify whether recovery of the radionuclides from any residue is beneficial. The availability of these records provides a means to reuse radionuclides within the program.

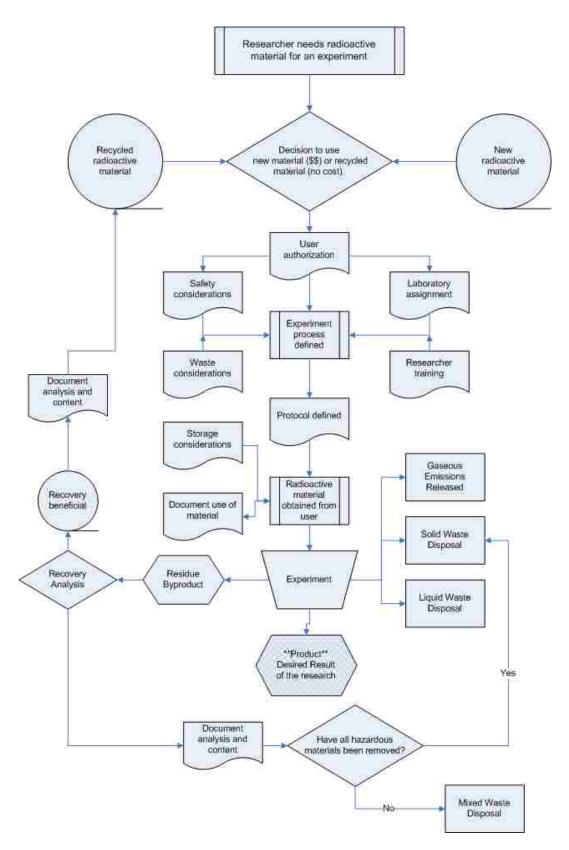


Figure 1-1. Flow Chart for Use of Radioactive Materials in Radiochemistry

1.4 Residue Recycling

Recycling of materials from experiment residue eliminates the concerns for mixed waste generation and provides radionuclides for use in future experiments [22]. Radionuclides used in experiments can be expensive; reuse reduces costs associated with replacement and waste disposal. In Chapter 3, an evaluation method to determine the feasibility of reuse of materials residues generated from laboratory experiments will be discussed. If the residues can be broken down and converted to an identified chemical form with a reasonable procedure, then recovery should be the desired option. Furthermore, the concept of recycling residues to obtain valuable radionuclides can be expanded to industrial application (see Chapter 4). As the Radiochemistry Program grows and expands its use of radionuclides, it is even more desirable to recover material upon completion of their experiments due to increased scarcity of even common uranium compounds. Since the researchers are the most familiar with their experiments, they would be the best suited to propose the appropriate techniques for recovery and provide a description of the final residue.

The products of experiments in radiochemistry laboratories are typically a prepared, well-defined compound plus liquid and solid wastes. The intended product has a known structure and has been characterized by analysis from a host of techniques. The low level solid radioactive waste products, primarily laboratory gloves and other consumables are disposed in the laboratories. The liquid residues contain products of the

experiments that have hazardous and radioactive components (See Chapter 3). These residues often contain reaction byproducts of radionuclides in both liquid and solid forms. Recovery of the radionuclides to an identified chemical form represents a means to reuse these isotopes in experiments.

The decision to recover radioactive materials from experimental products should be based on the value of the radionuclides and ease of separation. Labeling residues based on their expected chemistry provides a starting point for recovery. In planning any recovery, the researcher who produced the material should be consulted. Since recovery often deals with the seldom investigated part of an experiment, the desirability of recovery is not always an easy decision to make. In order to recover radioactive materials from liquid or solid residues of radiochemistry experiments, there must be a series of evaluations to answer the questions that follow:

- Is the radionuclide worthwhile to recover?
- Is the recovery cost efficient?
- Does the material present a hazard?

In many cases, the decision to recover the material can be made with a simple evaluation regarding radionuclide quantity. For example, if the total radionuclide stock is large (kg) compared to the amount in the experimental product (mg or g), then recovery may not be desirable. A more difficult decision will be required when there is valuable material to

recover but separation will be difficult. In this case leveraging established laboratory procedures can facilitate recovery.

Figure 1-2 provides some important considerations in determining recycling feasibility. Costs of recovery are radionuclide and residue specific. Consider the following example for recovery of ²³⁹Pu from a residue. An experiment has created a product that can be broken down with minimal effort and the cost for materials and labor is less than \$500.00. The radioactive compound used in creation of the product cost \$8,000.00. The cost for disposal of the product, a mixed waste, is \$2000.00. Considering a 90% recovery, the value of the material recovered is \$7,200.00. In this example, the financial decision may be either the primary or sole consideration. Therefore, the gains (avoided cost of disposal and value of material recovered = \$2000.00 + \$7200.00compared to losses (cost of recovery = \$500.00) would indicate that recovery is a reasonable decision. If the solution that was created is not well documented then the decision to recover may not be justifiable. Once the administrative evaluation is complete and the residue is determined to be valuable, the ability to define a recovery method is then made. The recovery effort should put the radionuclides in a condition that allows their reuse.

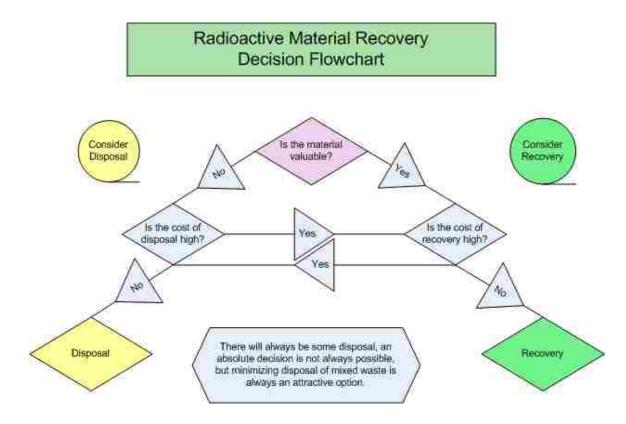


Figure 1-2. Recovery Flowchart for Radioactive Material Used in Research

Products that have been recovered must have an identified chemical form of known purity to be usable in further experiments. Ideally the level of purity should be similar to commercially available material. The material can be characterized with a range of techniques designed to identify chemical concentrations. Within the UNLV program these include atomic emission spectroscopy, mass spectroscopy, UV-Visible spectroscopy, and radiochemical methods [23] [24]. If the experiments will simply involve use of the activity of the radionuclide and the chemical compound is not important, then characterization may only involve simple activity determination. When the product of recovery is available for reuse, the properties of the material must be made available. These properties include chemical form, isotopes, and any impurities. If the final form is a solution then the matrix composition and radionuclide concentration needs to be reported.

1.5 Recovery Methods

There are a number of simple techniques that can be used to separate radionuclides from a mixture of chemicals. These include filtration [25], precipitation [26], evaporation [27], ion exchange [28], and extraction [29]. This section will describe some methods that might be used for recovery of a material of interest. In order to use one of these processes, it is important that the mixture conform to the needs of the method. This may require a pretreatment such as precipitation or solution neutralization. For example, solutions that are known to be destructive to filters may require pH neutralization as a pre-treatment. The recovery method is intended to remove what has been determined to be a valuable quantity of radioactive material based on analysis of a sample aliquot or prior knowledge of the solution.

An example of evaporation as a removal method is described. The rotary evaporator provides a means to enclose the evaporation process to reduce emissions and losses. With evaporation, it is usually the intent to remove a volatile liquid that is the solvent matrix for the radionuclide. Where many solvents or liquids with a low flash point are combined, it may be reasonable to use multiple collection temperatures on a rotovap, Figure 1-3, to allow extraction of reasonably clean solvents. In order to use this method, the residue must have one or more volatile liquids and

the material of interest must be in a chemical form that is less volatile. If the above condition is met, the volatile liquid(s) will be evaporated from the remainder of the solution using a rotary evaporator or similar device.



Figure 1-3 Typical Rotovap Evaporation Apparatus

In cases where the residue matrix is well known, the final evaporative state may be a suitable final product. As an example, for a radionuclide in an acid, the resulting radioelement salt can be a suitable final product. For an organic residue, the final product may require further treatment. As an example, technetium in a hydrocarbon organic residue can be treated by steam reforming to produce the metal [30]. An important consideration for some materials is their volatility in different compounds; enclosed apparatus such as the rotovap will help to prevent loss. Many other techniques for removal of radionuclides from solutions, this is the subject of many chemistry courses too extensive to add to this document. The separation techniques to be used should be researched so that the most appropriate technique for recovery of the materials of concern is used.

1.6 Donated Material

Many organizations have donated radionuclides to the UNLV Radiochemistry Program in the past several years (Table 1-3). Materials obtained were planned for disposal by other radioactive material licensees but were instead transferred to UNLV. These donations have resulted in procurement of many materials useful to the university for very low cost. The methods to receive, license and control these materials are inherent in the UNLV Radiation Safety Program. The savings by the donating licensee was sufficient incentive for a donation. Confirmation of the chemical and radiological purity of the donated samples is necessary prior to finalization of the transaction. The material received in donations has been adequate for reuse. A list of some of the materials in a typical donation received is shown in Table 1-4. The items received were labeled and assigned a number from the radiochemistry stock tracking system. As the materials were received, the quantity and inventory was controlled by continuous communication with the UNLV Radiation Safety Office. Over the years 2006 to 2010 the UNLV Radiation Safety Officer (RSO) understood that the UNLV Radiochemistry Program was not taking the 'waste' products from other facilities; it was taking the facility's excess

materials for reuse rather than having them dispose of useful radionuclides.

The suitability of a donation to provide benefit to the Radiochemistry program must be thoroughly evaluated to prevent receipt of materials that will require costly disposal or cause an unsafe situation. Considerations appropriate for a useful donation are; radionuclide(s), quantity, age and stability of the compound(s), and purity. The documented radionuclides and their activity must provide assurance that the material will be usable. The age should be considered so that there is confidence that there has not been ingrowth of radionuclides that provide an undesirable radiological hazard; the stability of the compound should be such that it remains usable, and the purity should be adequate to provide for reuse. Some complications that interfere with the ability to know the purity are related to the radio-sensitivity of the compound. If there is sufficient activity in the compound, there should be consideration given to radiolytic degradation.

Plutonium was received in the form of two NIST standards that had documentation indicating isotope composition. Each standard contained 250 mg of solid plutonium sulfate. The value of this material to the program is very high as the costs of plutonium for experiments in 2007 had been as much as \$10,000 for 5 ml of liquid containing 1 mg of ²³⁹Pu. Many different uranium compounds were received such as oxide, acetate, formate, nitrate, oxalate, sulfate, and chloride, commonly as the hexavalent oxidation state.

Compound	Radionuclide(s)	Manufacturer	Cost
Plutonium Sulfate Tetrahydrate Pu(SO4)2·4H2O	²³⁸ Pu, ²³⁹ Pu, ²⁴⁰ Pu, ²⁴¹ Pu	National Institute of Standards & Technology	Approximately \$1500.00, the cost of shipping.
Uranyl Nitrate ²³⁵ UO ₂ (NO ₃) ₂ *6H ₂ O	235U	Argonne National Laboratory	No cost
Uranyl Nitrate ²³⁸ UO ₂ (NO ₃) ₂ *6H ₂ O	238U	Many	No cost
Uranyl Acetate	238U	Many	No cost
Uranyl Sulfate	238U	Many	No cost
Thorium Nitrate	²³² Th	Many	No cost

Table 1-3. Radioactive Compounds Donated to UNLV

Table 1-4. A Transfer of Radioactive Compounds from the University of Massachusetts at Amherst in 2009

Manufacturer	Net Uranyl Acetate Mass (g)
Mallinckrodt	297.15
Fisher Lot 772508	55.75
Fisher Lot 745119	16.75
Fisher Lot 731493	13.45
Fisher Lot 770672	24.19
Fisher Lot 745119	21.49
Fisher Lot 725175	15.66
Fisher Lot 772764	105.85
Fisher Lot 741569	112.53
Fisher Lot 725175	112.17
Total Net Mass of Uranyl Acetate (grams):	774.99
Total Uranium (²³⁸ U grams):	441.21
Total ²³⁸ Uranium Activity (Bq):	5.49x10 ⁶

Specific donations sought from other radioactive material licensees across the United States have helped to build a stockpile of uranium, thorium, and plutonium sufficient to provide for research with these actinides for years into the future (Table 1-5). This clearly indicates that there were many facilities that took advantage of the offer for UNLV to utilize their excess. For small facilities, radiological control agencies, universities, and even high schools, it was advantageous for the UNLV Radiochemistry Program to provide this service. There were other facilities that had materials they wanted to donate; however, the inability to ship the material or the lack of a method to ship the material provided a barrier that could not be overcome. In other cases, some recognized a liability that their management considered unacceptable. While the majority of the donors were eager to provide the materials that were needed without stipulation, some of the more stringently regulated agencies were concerned with our ability to provide them with closure to their obligation for the subject materials.

Materials recovered that were typically planned for disposal by other radioactive material licensees may not have a purity that would be expected when purchased from a chemical manufacturer. It is prudent to inspect any donated radioactive compounds to verify that the purity is adequate for the intended experiment. Initially, this may be completed by obtaining and evaluating documentation of any work performed on the subject material by the original licensee. Table 1-5 lists the donations that have been received since donations were first received in 2006. Another risk taken when accepting a donation is the possibility that the material has already been used in experiments and what is received is

actually a residue that may or may not be acceptable for further research. In this situation, the extra evaluative step to provide prevention from receiving a waste product can be important.

Donor	Radionuclide	Compound	Mass (g)
U Mass at Amherst	²³⁸ U	Uranyl Acetate	1138
U Mass at Amherst	238U	Uranyl Nitrate	503
U Mass at Amherst	238U	Mg-Na-Uranyl Acetate	229
Brown University	238U	Uranyl Acetate	305
Brown University	238U	Uranyl Oxalate	10
Brown University	238U	Mg-Uranyl Acetate	25
Brown University	238U	Uranyl Oxide	50
Brown University	²³⁸ U	Uranyl Formate	5
VA E. Colorado	238U	Uranyl Acetate	265
U Texas at Austin	²³⁸ U	Uranyl Nitrate	600
U Texas at Austin	²³⁸ Pu, ²³⁹ Pu, ²⁴⁰ Pu, ²⁴¹ Pu	Plutonium Sulfate	0.5
Cal State Fresno	238U	Uranyl Acetate	900
Cal State Fresno	238U	Uranyl Nitrate	860
Cal State Fresno	238U	Uranyl Sulfate	160
Cal State Fresno	238U	Zn-Uranyl Acetate	50
U Nevada Reno	238U	Uranyl Acetate	1260
U Nevada Reno	238U	Uranium Oxide	2800
U Nevada Reno	²³⁸ U	Uranyl Nitrate	50
U Nevada Reno	²³² Th	Thorium Nitrate	1350
U Nevada Reno	²³² Th	Thorium Chloride	100
New York Med Col	²³⁸ U	Uranyl Nitrate	172.5
Penn State U	²³⁸ U	Uranyl Nitrate	168
Florida Dept. of Radiation Control	238U	Uranyl Nitrate	454
Rensselaer Polytech Institute	238U	Uranyl Acetate	500
Battle Mountain High School	238U	Uranyl Nitrate	50

Table 1-5. Summary of Radioactive Material Donations Received

After the receipt of donations of uranium compounds at UNLV, a company in Florida, International Bio-Analytical Industries, Inc. was

found to be accepting uranium compounds for reuse. This practice continues today [31]. While the reason for the recycling effort on the part of this company may or may not have been as a result of the success of the UNLV recovery, it was not in place before recycling uranyl compounds was found to be useful at UNLV. The donations that have been received were as a result of general inquiries regarding the materials that are considered surplus by radioactive material licensees. The current stockpile of radioactive materials is reasonable to keep uranium research projects well provided for until 2015. In seeking future donations, requests made should be more specific regarding nuclide and available quantities to avoid having material become waste because of non-use and to avoid having surplus material at the time of decommissioning of the program.

1.7 Thesis Preview

In the following chapters novel aspects of material reuse and control inherent to a unique academic program utilizing high levels of radioelement will be described. Chapter 2 is concerned with development of radiation safety for a radiochemistry program. As the levels of activity used in the university program are unique, the radiation safety in the laboratory, training of personnel, surveys, monitoring, and management represent a novel contribution to academic research. Recycling Tc recovered from decontamination of a chemical fume hood is presented in Chapter 3. This effort used a commercial gel based radionuclide removal

agent. The investigation into the utilization of this material has implications for its application to nuclear forensics [32]. A unique application of the gel in the sampling and recovery of materials is shown to be feasible. The use of the gel in collection of samples from contaminated sites for health physics or nuclear forensics applications [33] can be an outcome of this initial research. Methods to break down the gel are discussed; other reactions are identified but not pursued. In Chapter 4 recovery of Tc from experiment waste products is discussed. This required working closely with researchers to identify methods that would not result in unexpected reactions and unsafe conditions. The resulting solutions are useful for moderate activity experiments and free of gross organic impurities. The techniques used, monitoring methods, development of rapid analysis of the waste for activity, and recovery yields are discussed. In chapter 5, analysis of coal ash is completed to determine the feasibility of recovering natural uranium from the ash. This represents an application of the reuse and recovery concepts developed in the laboratory to an industrial setting. The results indicate the same concepts can be applied to residue reuse evaluation.

Chapter 6 provides recommendations for the future of the recovery and reuse of radioactive materials and radiation safety in the UNLV Radiochemistry Program.

Unique contributions are as follows:

• The recovery of radioactive materials from hazardous waste in anticipation of reuse is a novel approach to resolution of two

problems associated with the UNLV Radiochemistry Program; disposal of mixed waste and availability of radionuclides for an academic radiochemistry program.

- The use of a decontamination gel created by a commercial firm as
 a means for recovering spilled radionuclides or sampling high
 activity dispersions of radioactive materials is a new use of this
 material.
- Implementation of a plan for upcoming radiochemistry experiments by each researcher was a precursor to the vision of a complete laboratory experiment tracking system.
- Development of a radiation safety program for use of measurable masses of radionuclides provided an opportunity for development of a program for novel radiochemistry research in an academic setting.

The last section of each chapter is provided with an implementation document. This could be converted to a publishable paper or note in a professional journal. This is provided to accentuate the value of the thesis in programs where the concepts presented are useful.

CHAPTER 2

RADIATION SAFETY FOR A RADIOCHEMISTRY PROGRAM

2.0 Introduction – Design Considerations for Radiochemistry

Radiation safety for the UNLV Radiochemistry Program [34] was developed with the key features used by any academic facility using licensed radioactive materials in the United States and innovative methods to maintain control. Academic programs in the United States are designed with the basic requirements prescribed by regulatory authorities [35] [36] [37]. Prior to the existence of radiochemistry as a degree at UNLV, the license and radiation safety program supported low activity use of short lived radionuclides for biology experiments and long lived radionuclides in low activity solutions, powders, and sealed or encapsulated sources for instrument calibration or environmental analysis. The requirements of the new license would require a combination of the regulatory guidance for academic programs, use of special nuclear materials, a license of broad scope, and a service provider license. Table 2-1 provides a review of controls before and after establishment of the Radiochemistry Program.

The radionuclides and associated activity limits applied for were devised by the Authorized User, the Director of the UNLV Radiochemistry Program. The controls established for these radionuclides were described in the license amendment application.

The development of the radiation safety aspects of the program were completed as needed to support research goals for radiochemistry and meet all appropriate regulatory guidance. There are many unique aspects of this type of program compared to other radiation safety programs [38] for research in the United States [39]. This chapter will discuss some of those differences, the details of work control and the successes and failures observed since the program started using radioactive material.

The fundamental needs of a radiation safety program must consider the goals of licensed entity. The goal of the UNLV Radiochemistry Program is to prepare students for a career at facilities where large quantities of radioactive material may undergo processes that could create a hazardous work environment. The preparation for such a career requires that the student understand the controls required for protecting themselves and others around them while they do their work. Students work with low and moderate activity radioactive material to develop contamination control skills while researching the properties of the materials that they create. Consider the regulatory agencies as shown in Figure 2-1.

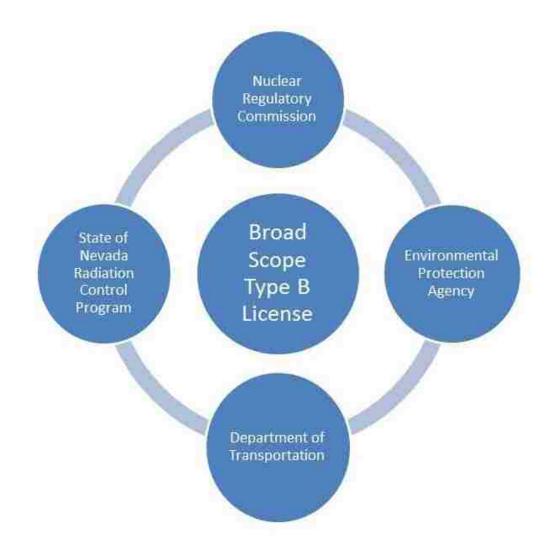


Figure 2-1 Major Regulatory Agencies Involved with UNLV Licensing

Enhancement of the UNLV Radiation Safety Program to allow work with quantities of radioactive material with the potential to cause harm was done at a time when the public image of radiological and nuclear facilities was poor. By public opinion polling in 18 countries in 2005, the IAEA identified that 54% of those asked, were concerned that the risk of nuclear terrorist acts was high because of inadequate protection [40]. The case presented to the State of Nevada regulators to allow use of gram quantities of alpha emitting radionuclides in any form was strong; contamination control, dose control, and public protection were addressed to resolve concerns. Before radioactive material use at UNLV changed to accommodate the Radiochemistry program, contamination control was a minimal aspect. It has now become the most important aspect. The following factors were considered for development of radiation safety for the UNLV Radiochemistry Program:

- control of individual internal and external dose;
- researcher awareness of radiological conditions and controls;
- training to provide researchers with protection techniques;
- management oversight of routine and non-routine experiments;
- protection of members of the campus and the public;
- control and security of radioactive materials;
- appropriate support of area and personnel monitoring programs;
- completeness and retrievability of records;
- radiological control performance indicators;
- special controls for unusual situations or work;
- compliance with regulatory standards.

The preparation for high activity work required license and office operation changes in the UNLV Radiation Safety Office to recognize that this work would be done on campus and controls must be in place when it is. This would be a significant change from the control of short lived radionuclides used in nuclear medicine, or biology research and the use of x-ray machines for imaging with which the UNLV Radiation Safety Office was already familiar.

An amendment to the UNLV radioactive material license was submitted in 2004 that provided significant capability for a new program at a facility that had no demonstrated User experience with high activity dispersible sources of actinides [41]. The license change allowed up to 200 grams of the following radionuclides in any form: ²³⁷Np, ²⁴¹Am, ²⁴³Am, ²⁴⁴Cm, and ²⁴⁸Cm in addition to the previous limits in the license. The approval of this license amendment provided evidence that the State of Nevada had confidence that the UNLV program was capable of controlling the radionuclides listed in addition to the radionuclide inventory that was currently maintained on campus. Nevada is an NRC Agreement State with radiation protection regulations and license specifications in the Nevada Administrative Code (NAC) [42].

The radiochemistry laboratories were initially only housed at UNLV's Harry Reid Center for Environmental Studies (HRC). As the program grew, the laboratories expanded to UNLV's Bigelow Health Science (BHS) building and the Science and Engineering Building (SEB). Original facilities for chemistry laboratories were adequate for radiochemistry program operations.

2.1 Radiation Laboratory Design Hypothesis

Through proper laboratory design, several aspects of the radiochemistry program are enhanced.

- personnel access control;
- isolation of work with different radionuclides;
- isolation of low activity and high activity work;
- multiple levels of contamination control;
- location of instrumentation outside of contaminated areas;

• low background areas for radionuclide analysis.

Laboratories where radioactive materials are used have desired features to allow personnel to control radioactive materials and protect themselves from hazardous materials. In order to ensure personnel who enter the laboratories are prepared for work with hazardous materials, they must be trained to understand methods of control and be provided with security access. Changes were required in laboratories to prevent access to radioactive materials by untrained personnel and to provide radiation detection equipment for contamination control. The most substantial change was to the laboratory layout and the security controls for each laboratory.

- 2.1.1 Challenges in Laboratory Use and Design
 - Two groups inhabited the laboratory areas, one worked with low activity (Bq) of ⁹⁹Tc, ²³⁷Np and ¹²⁹I; the other worked with gram quantities (MBq to GBq) of ⁹⁹Tc and MBq of actinides. Both groups ingressed and egressed their laboratories in the same hallway.
 - Contamination controls were not of interest to the low activity group because their activity was always in solution and their solutions did not cause contamination control concerns.
 - Equipment for contamination control did not provide adequate sensitivity to minimize the probability of activity release from the laboratories.

- The laboratories had many exit points and access and egress from the laboratories did not require security in some cases.
- Equipment was moved from areas of high activity use to areas of low or no activity use without concern for contamination control.
- Fume hoods designed for work with radioactive materials were insufficient in number to allow for the number of projects with radioactive materials.
- Many fume hoods in the program laboratories did not have High Efficiency Particulate Air (HEPA) filtration of exhaust air, minimizing the number of simultaneous experiments.
- Security of radioactive materials in the laboratories was continuously in question because doors to laboratories were left propped open or unlocked to facilitate ease of access.
- Security of radioactive materials was minimal and was controlled by a single user.
- Chemicals were commonly stored in many locations without regard to potential incompatibility hazards.
- There was no organized accountability of personnel access to the laboratories.
- There was no organization of personal protective supplies.
- 2.1.2 Actions Taken to Improve Facility Use and Design

The issue of one group using activity of a radionuclide used in quantities 1×10^6 to 1×10^9 times higher than the group across the hall

was resolved by ensuring that these groups did not have access to each other's laboratories. A proximity card access system was implemented and personnel were informed of their access limitations and the reasons for them. This type of restriction will only have the desired effect for a limited time. The low activity group left UNLV and the issue was resolved.

Contamination controls were required to be implemented at the work site in accordance with the UNLV Radiation Safety Program. To implement this, all laboratories were required to purchase contamination control instrumentation and conduct surveys of work areas. In addition, all personnel have been instructed in how to take smear surveys and how to analyze smears. It is emphasized in training that the primary contamination control measures must be taken at the work area.

Contamination control equipment was provided at the exit from the laboratories to ensure that the hands and feet of workers were sufficiently free of activity to allow release from the area. A revision to this that further enhanced control was a donation from the USEPA of a personnel contamination monitor (PCM-1B). The PCM is not designed to be a primary instrument for release of personnel from the laboratories. It is the final action that is taken to ensure that the primary contamination control actions were effective. When a researcher leaves one of the laboratories, it is necessary that they are radiologically clean and the work area that they just left is not contaminated. In this way, the PCM will not get contaminated and will provide the level of comfort for the

Authorized Users and the radiation safety officer that it was designed to provide.

As more research with higher activity was conducted, the layout of the laboratory bay was changed to provide for one entry point, one controlled exit point, and several emergency exit points from the area that previously had been individual laboratories. Some individual laboratories had their own security and some did not. Figure 2-2 shows the combined layout which became the original laboratories of the UNLV Radiochemistry Program. Proximity card access was decided as the most desirable method of security. With keys, a door could be left unlocked, they also provide a physical vector for contamination transfer to a hand, pocket, or to another person. Marlock cards provide the security but have the same difficulty for contamination transfer in that they require handling. Proximity cards can operate a lock without a person touching them and without touching a surface.

The new layout provided better security, a location for common entry and exit from the area, a contamination control point, a protective clothing storage location, a monitoring station, and an exit to another enclosed location in the building that could serve as a secondary control point should there be an event which caused either loss of control or a requirement for evacuation. Sufficient room and monitoring capability at the exit location provided for the ability to ensure adequate monitoring of equipment before it was moved out of the laboratory bay to other areas where contamination control was not necessary. The use of filtered ventilation on fume hood exhaust was rare in the laboratories, as that was only a feature on two the laboratories. Filtration of exhaust on laboratory fume hoods is provided to minimize release of activity from the hood that might result from vaporization of radioactive solutions or unexpected changes in the physical state of radioactive liquids or solids. The filtration is a safety measure not provided to routinely collect activity that is discharged but to minimize the probability of accidental release of radioactive material from the facility. As the number of experiments requiring controlled ventilation has increased, more chemical fume hoods were provided with high efficiency (HEPA) filters.

In 2010, the new UNLV RSO supervised testing of the HEPA filter systems in use to verify that they indeed met the specifications for HEPA filter systems. A form of testing using dispersed oil particulate (DOP) was used to evaluate the filtration capability of the system. In this testing a known concentration of the DOP is introduced to the exhaust flow upstream of the filter and the measured concentration downstream is compared with the introduced concentration to determine the filtration efficiency. All systems passed the testing.

The number of fume hoods with HEPA filtration systems on their exhaust was significantly increased with the installation of filters on five remaining unfiltered hoods in the older laboratories in June of 2012. Of the significant events that relate to the potential for discharge of radioactive materials to the campus, one event in MSM-165 had potential

for release of an actinide mixture to the campus through an unfiltered fume hood. In this incident, a researcher was heating and stirring a solution in a fume hood that did not have filtered exhaust. The liquid boiled away and activity entrained in the vapor was carried to the back of the hood where it attached to the cooler hood surfaces. Surveys of the spread of contamination in the hood did not reveal evidence that activity was released. This event once again caused a discussion of the use of radioactive materials in fume hoods that did not have filtered ventilation. It was decided at this time that all hoods would be labeled to prevent use for experiments where there was the possibility of release of radioactive material from the experiment. All researchers were made aware of the new labeling and the new policy regarding hood use at the weekly group meeting and through the distribution of Newsletter 29 in Appendix B.

The entry to the radiochemistry facility was provided with proximity card security [43] that enabled direct control of access to the laboratories and would allow exclusion of people who were not qualified or disqualification of person's whose training had gone beyond a year without receiving requalification training. The experience of the initial development of the program indicates that a Radiochemistry Program in an academic setting needs to be independent from non-complementary research and security protocols need to be in place and understood by all researchers who enter the radiochemistry laboratories.

Because of the large quantities of radionuclides held for research by the radiochemistry program, and the way that radionuclides are used, security of the areas where there is a contamination control concern is very important. UNLV is in the center of a city whose most important industry is tourism [44]. There must never be a fear developed in the local population that would cause people to have second thoughts of coming to Las Vegas because of poor security of radioactive materials. The only way to prevent this is to have tight security of all radiochemistry laboratories and important quantities of radioactive materials. It is not acceptable to lose sight of the fact that the Radiochemistry Program is not important to the local population and there must always be focus on the need for detailed documentation that proves the ability of personnel in the program to control radioactive materials. Security for control of radioactive materials has been good and has not changed since the use of a gun safe, for radioactive material storage, procured in 2006.

While security in the program is good, there must continue to be improvement in this area because it is imperative. In the past, events have occurred where someone was allowed to enter controlled laboratories without proper training and without an escort. Until everyone associated with security of any part of the program recognizes the need for control, the program will be at risk. The Nuclear Regulatory Commission issued an order for increased controls for certain radioactive materials licensees in 2005 [45]. This order established controls for radioactive materials based on quantities that are much higher than the levels held by the Radiochemistry Program. At the current time the security of the laboratories is established as the responsibility of

everyone in the program. There are no requirements for increased security based on government regulations or UNLV procedures.

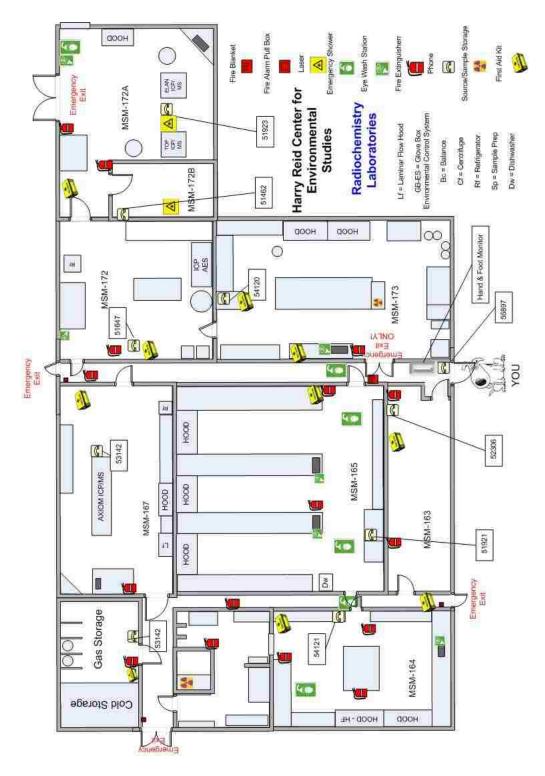


Figure 2-2. Layout of the HRC Laboratories for Radiochemistry

Chemical storage in the laboratories was a concern because the quantity of chemicals tracked by the Risk Management Group was close to limits for some chemicals and many containers of the same chemical were spread throughout the laboratories. A common storage location was decided on, storage cabinets procured and a labeling mechanism devised to allow easy access to the chemical desired. This system minimized time looking for the chemical in all areas of laboratories where dose rates were higher than the chemical storage area. This change was a boost to chemical control and to ensuring personnel dose was ALARA.

When first established, the access to the laboratories was based on the access granted by administration of the Harry Reid Center. Access was granted by need to enter and not on evaluation of the individual's qualification to work in the laboratories with hazardous and radioactive materials. A change specified that all personnel would apply for access and criteria established for required training and permission from the Authorized User for the laboratories. Access requires a magnetic proximity card be programmed to allow access to the bay and to any rooms where the trained researcher required access. Control of access remains related to training and permission.

Redesign of the laboratory layout and control of the hallways allowed a reorganization of the protective clothing, dosimetry, eye-ware, and an employee 'In/Out' status board that are located at the entrance to the laboratories. This establishes a means whereby a researcher can store their personal belongings, collect all of their safety equipment and

ensure that they are ready to do their work before they enter a radioactive materials work area. The entry hallway must be maintained radiologically clean to allow for survey of equipment and personnel before leaving the area. This location also serves as a decontamination area should activity be identified in the release survey.

2.2 Development of the Radiation Safety Culture Hypothesis

The most important aspect of any radiation safety program is a well-founded respect for radioactive materials and radiation producing devices. Training in the fundamental aspects of radionuclide properties and radiation protection principles is the beginning of establishing a safety culture. The Radiation Safety Officer provides for the control of radioactive materials and the radiation safety of the campus by authorizing personnel as Users with the responsibility to supervise the use of radioactive materials. Users must have respect for the requirements of the radioactive material license and work together to keep students, staff, and the public safe.

The safety culture at the Radiochemistry Laboratories is good within the program. As long as the Authorized Users communicate well with each other and with the UNLV Radiation Safety Officer, the programs that are set up will succeed and the research will be unaffected. Two important aspects of the program are attention to detail in contamination control and minimization of waste products that are both hazardous and radioactive. The first, contamination control, will

keep the program in operation by preventing the release of radioactive materials from the confines of the radiochemistry laboratories. The other feature, hazardous waste minimization, is the basis for the work presented in Chapters 3 and 4 of this dissertation. Creation of mixed waste is costly and is unnecessary in a radiochemistry program.

It is essential that researchers in a Radiochemistry Program respect the authority of a Radiation Laboratory Administrator (Authorized User or Laboratory Staff) and the University Radiation Safety Officer and staff. In addition, other members of the organization need to recognize that it would be highly unproductive in the laboratories unless there was respect for radiological controls. Personal dose must be As Low As Reasonably Achievable (ALARA) and the understanding of this concept can be achieved with continuous training through weekly group meetings that emphasize a need for dose and contamination control.

2.2.1 Challenges in Safety Culture Development - Incidents

The safety culture in a program develops based on influences from management, senior researchers, professors, and associate researchers. Whether the program has positive influences depends on the observations that a researcher experiences when working with wellrespected researchers in a group. Constant positive influences of good work practices, contamination control, and license requirements are needed to keep everyone on the right track. The following items challenged the program's method of compliance with license conditions.

On 17 January, 2008, there was a minor spill of ⁹⁹Tc in laboratory MSM-173. This spill caused low level contamination of a small area of the laboratory and the clothing of two researchers. The clothing was collected and disposed, the floor was cleaned and work resumed. The activity spilled caused no additional dose to the researchers but provided indication that contamination in the area was becoming a concern to the UNLV RSO.

On 18 January, 2008, another loss of control, this time associated with handling particles of plutonium from soil in laboratory MSM-165. The particles were found to travel from the work bench to the floor without recognition by the researchers until their post work survey. After the particles were found; the area was isolated, cleaned, and work was allowed to resume in a more controlled manner. It was again recognized that the event was not important from a dose standpoint but indicated an increasing trend of conditions adverse to good contamination control.

On 30 April 2008 a third event occurred in a different laboratory, this time involving a spread of ²³³U from an experiment in a fume hood in MSM-164. This event was also not important from a dose standpoint but was an important event regarding an undetected loss of contamination control. Although surveys were done by the researcher, they were not sufficiently extensive to recognize the contamination outside of the hood. The timeliness and detail of RSO surveys and laboratory staff surveys identified the contamination, the radionuclide, and the source of the

contamination within 48 hours, the area was isolated and the contamination cleaned up in the next 12 hours.

2.2.2 Actions Taken In Response to Incidents

When there is an event that has caused a deviation from the normal conditions of radiological control, it is necessary that the event is evaluated with input from all of the people that were involved and the people with the authority to make changes based on the results of the evaluation. In many industries this evaluation is termed a root cause analysis, the idea to find the underlying event(s) that contributed to the occurrence. In each of the occurrences that contributed to changes in the control of radioactive material, a meeting was held with each of the participants, written statements were obtained by those involved and action taken was reported to the UNLV when it was decided on by the Authorized Users for the area.

A review of all of the events together provides the following set of similarities and differences:

- each event was related to contamination control,
- each event caused contamination spread on the floor,
- there were different personnel involved in each event,
- each event involved different radionuclides, ⁹⁹Tc, ²³⁹Pu, ²³³U,
- each event occurred in different laboratories,
- everyone had different research goals,
- one event was discovered immediately,

- one event was identified when the end of the day survey was done,
- one event was not identified for two days after its occurrence,
- in the ⁹⁹Tc event the material was being moved in an open container,
- in the ²³⁹Pu event, the material was being physically sorted on a bench-top,
- in the ²³³U event, a solution was being bubbled with gas in a fume hood.

In any evaluation to determine the cause, once the facts about the event are collected, a set of questions is generated to identify the most reasonable actions to take for recovery from the event this is the analysis phase which allows the event to be seen from a larger viewpoint than the individual experiences of the participants. With sufficient information about what happened conclusions might be reached that establish the cause and contributing factors. From the analysis of possible causes a determination of actions to prevent recurrence of the event can be made.

The three events discussed in section 2.2.1, changed the course of the program. The changes included:

- training regarding the details of these three occurrences,
- requirements for more detailed contamination control surveys,
- recognition that methods for movement of material requires closed containers,

- work with potential for contamination spread should be done in enclosures such as glove boxes or fume hoods,
- procedures for research involving radioactive materials and requirements for contamination control need to be specified, and
- closer evaluation of work areas where work with solutions, powders or particles was to be done.

As a result of the attention provided to these events in requirements for training, detailed contamination surveys, better material control, and plans for work involving high amounts of activity, the number of events that could be termed incidents was reduced. There were no events with similar magnitude as those identified here.

All researchers need a structured program for controls no matter how much experience they have or how comfortable they are working with radioactive materials. For example, contamination controls are established to prevent the spread of activity from work surfaces. Yet, in the first two years of the program, most researchers did not analyze smears of their work area as they were trained to do. Periodically a point was made at group meetings or training bulletins were issued, yet the spread of contamination continued until there were individual repercussions when it was found that surveys were not done.

It is best to determine a method for dose and contamination control that has minimal effect on a researcher's work and maximum control for the effort that he or she would apply. If a researcher has to spend time associated with contamination control for survey or cleanup, it is more likely if the time required for this action is minimized and demonstrated to be important.

Since the occurrence of these events, there have been other minor losses of contamination control, but they have been recognized by the more frequent and detailed surveys. Laboratory management also has increased the detail of their surveys and ensures immediate cleanup when contamination is found. Even with simple solutions, proper equipment, and continuous reminders, work could be done with radioactive materials in powder and liquid form without doing contamination control surveys.

In March 2010, the number of smears taken in the weekly surveys that were identified in excess of the contamination control limit continued to increase. This was again indicative that researchers were not complying with the requirement to survey their work area each time they used radioactive material. Although surveys were required, documentation of those surveys was not. At this time a change was made in the contamination control program to require documentation of all surveys done by researchers. The surveys were easy to do and document, this provided more assurance that contamination was controlled at the worksite. The efforts since that time have focused on each individual working in the facility, what they are working with, and when they are present in the laboratory. This has increased the number of surveys done, and prevented spread of contamination from the laboratories.

2.3 Training the Participants Hypothesis

A safety culture is evolved in time through recognition that noncompliance with radiation safety rules is detrimental to one's research. The following statements are the hypothesis for this section:

- There must be continuous identification of what the rules are so that everyone has a similar belief in what is needed.
- Understanding the goals of the researchers will provide them with the information they need to be successful.

2.3.1 Initial Development and Evolution of the Training Program

The initial training for radiation safety in the UNLV Radiochemistry Program was established as two hours of radiation fundamentals and regulation. In time, this training developed into a laboratory familiarization and general laboratory safety training. Continuing training was first established with the general distribution of a radiation safety newsletter that provided a simple coverage of several topics to enable a basis for compliance with laboratory rules.

Early in the development of a program for radiation safety in radiochemistry, the characteristics of the radionuclides used will lead to conclusions about the balance for internal and external radiation protection. In this program, the radionuclides used require more focus on protection from intake of radionuclides as external dose rates are typically low throughout the laboratories. Contamination control was the primary concern at the start of the program and continues to be the most significant concern for radiation protection. With initial training for radiation safety and annual refresher training providing the fundamentals of radiation protection in two hours, there was no time provided for other aspects of radiation safety such as the fine points of contamination control, instrument use, radioactive waste collection, responsibilities, security, fume hoods, etc. It was decided to establish a method of expanding the periodic training to include those details. This also provided a way of establishing documented training in many aspects of radiation safety that cannot be covered due to time constraints.

In January of 2007 the first HRC Radiation Laboratory Newsletter was published and distributed to the program participants [46]. The first newsletter was entitled 'Contamination Surveys' and was written in a question and answer format. The introduction provided a reason for the newsletter. "Whenever you work with radioactive materials, there is the possibility that some of the radioactive material evaded your control mechanism and may be spread to other parts of your laboratory or even outside of the facility. While the contamination spread is not likely to be hazardous to anyone, it may be in excess of our license conditions. In order to prevent this type of situation from developing, we take measurements of our work areas with portable instruments or in some cases we take smears for laboratory analysis to identify the level of surface contamination (or absence of it) in our work areas."

This newsletter then asked the question "When are surveys required?" The answer was provided as follows: "Contamination surveys

are desired in each laboratory where radioactive material is used, weekly, or immediately after unsealed radioactive materials are used in an area, whichever is more frequent. If you are using high-energy beta emitters, you should also evaluate your body and clothes for the presence of contamination after each procedure."

That newsletter went on to identify what instruments to use for evaluation of surfaces when using different radionuclides, what a smear is and how to evaluate a surface for loose surface activity, how to know when a surface is contaminated, how to convert from instrument response to activity, how to survey your body, what to do if you identify contamination on the body, and how to prevent spills of radioactive material. The newsletter concept was demonstrated to be a good way to get a lot of information out to the continuously growing group in a reasonable amount of time while allowing the information to be digested on their time. Table 2-2 provides a list of the newsletter topics that were published from January of 2007 to December of 2010. The newsletters were brief and covered a single topic. The intent was to provide a constant reminder of the need for vigilance in the control of the radioactive materials that were the mainstay of the Radiochemistry Program. In June of 2007, a newsletter on contamination control was again published, aimed at assigning responsibility so that all researchers would know who they are responsible to when it came to: who must answer for a loss of contamination control. This newsletter also took a different approach with things to do to maintain control of

contamination. in your work area. Some of these items are provided in Figure 2-3. In some cases, the topic of the monthly newsletter was dictated based on the events that had occurred to put the control of the laboratories in jeopardy. For example, before the publication of HRC-9 "Laboratory Access" in September of 2007, there were a number of times that people without training were found to be in the laboratories without an escort. Publication of the rules on that topic was a way to make it clear what was expected so that future events such as that did not recur.

1. Delineate a work area in your laboratory. Ensure that the area is not subject to high personnel traffic that may cause items to be touched or knocked off of the laboratory bench.

2. Wear protective clothing (gloves and laboratory coat) to prevent the spread of contamination.

3. Complete surveys each time that you use radioactive materials to ensure that no material is spread to other parts of your laboratory. Surveys may be done by direct monitoring for some radionuclides but must be done using smears and liquid scintillation counting for others.

4. Ensure that you monitor any items that are taken out of the contaminated area. Remember, anything in that area is suspected of being contaminated until proven not to be.

5. There must be no food, drink or cosmetics stored or used in any laboratory where there exists a potential for intake of radioactive or hazardous materials.

Figure 2-3. June 2007 Radiation Safety Newsletter Excerpt

Number	Date Published	Newsletter Topic	pp
HRC-1	January, 2007	Contamination Control	4
HRC-2	February, 2007	Dosimetry	3
HRC-3	March, 2007	Training	2
HRC-4	April, 2007	Radioactive Waste	4
HRC-5	May, 2007	Security of Radioactive Materials	2
HRC-6	June, 2007	Contamination Responsibility	2
HRC-7	July, 2007	Transporting Radioactive Materials	2
HRC-8	August, 2007	Using Radiation Detectors	4
HRC-9	September, 2007	Laboratory Access	4
HRC-10	October, 2007	Personnel Contamination	4
HRC-11	November, 2007	Protective Clothing	2
HRC-12	December, 2007	Fume Hoods	2
HRC-13	January, 2008	Postings for Radiation Safety	3
HRC-14	February, 2008	Radioactive Materials Regulations	4
HRC-15	March, 2008	X-Ray Producing Devices	3
HRC-16	April, 2008	Instrument Checks and Calibration	2
HRC-17	May, 2008	Background Radiation	4
HRC-18	June, 2008	Liquid Scintillation Counters	4
HRC-19	July, 2008	Transportation of Rad. Material	5
HRC-20	August, 2008	Emergency Equipment in Laboratory	4
HRC-21	September, 2008	Access to the Radiochemistry Facility	3
HRC-22	October, 2008	New Laboratories in HRC Completed	4
HRC-23	November, 2008	Emergency Preparedness	3
HRC-24	December, 2008	Instruments – Ludlum 3/44-9	5
HRC-25	January, 2009	Instruments – Ludlum 2360/43-93	5
HRC-26	March, 2009	Laboratory Inspections	3
HRC-27	April, 2009	Radioactive Material Inventory	3
HRC-28	May, 2009	Survey Documentation	4
HRC-29	June, 2009	Radiochemistry Fume Hoods	7
HRC-30	July, 2009	Risk and Radiation Exposure	3
HRC-31	August, 2009	Labeling Radioactive Material	3
HRC-32	September, 2009	Safety and in the HRC Laboratories	3
HRC-33	October, 2009	X-Ray Machines	4
HRC-34	November, 2009	Good Work Practices	5
HRC-35	December, 2009	Wearing Dosimetry	2
HRC-37	May, 2010	Authorized User Responsibility	8

Table 2-1. A Listing of Radiation Laboratory Newsletter Topics

Over the years there were events that the whole group learned from even though only a few participated in the laboratory situation. Each occurrence was discussed at the weekly radiochemistry group meeting and in some cases presented to the group by the researcher in charge of the experiment. The events were primarily minor losses of contamination control and chemistry research with undesirable consequences. However, in each of the events, there was no measurable intake of radioactive material, there was no measurable dose to personnel, and there was no release of radioactive material from the radiochemistry laboratories.

Then there were the events that were the result of inattention to detail and lack of basic common sense. These events continue to occur, one such item is the disposal of food containers or food wrappers in the waste containers within the radiological control area. This one item has been continuously brought to the attention of everyone in the Radiochemistry Program for all of the years that there has been organized radiochemistry at UNLV and the only way to minimize its recurrence is continuous reminder.

Those who have poor safety practices and put the health and safety of other people in the laboratories with them at risk must have a life changing experience to enable them to recognize the need for changing their ways [47]. As humans, we change our behavior based on perception and availability of a desire to change to satisfy ourselves or others. For example, if someone recognizes that they did something that upset another deeply and caused them to express emotion, this may

cause a change in behavior. Over the years there were many times that evidence of deviation from good safety practice which occurred in the laboratories was brought to the attention of faculty, staff, and students in a radiochemistry group meeting, there was usually recognition that behavior must be changed. Restriction of access to the laboratories may be useful as a means to help a researcher recognize that change is needed for them to be able to complete their work. Another way of ensuring compliance may simply be related to the amount of activity that a researcher is allowed to work with.

In another example, consider a researcher who is using high activity of a radionuclide with a low Annual Limit on Intake in a laboratory where other people are using much less hazardous materials in quantities that are lower in health significance. If the researcher using the more hazardous material fails to conduct detailed surveys of their work area, he or she will put others working in that laboratory at risk for a potentially serious intake. That researcher might also cause a concern in the cohabitants of the laboratory that their research could be affected by a loss of control on the part of the high activity worker.

The discovery of poor controls is not easy, but once identified and reported to a researcher, the realization that their controls may have cost shut down of the laboratory and affected the ability for anyone working there to accomplish their experiments is a significant emotional experience that may enable them to recognize the need for more diligence in control of radioactive materials in their possession. There are also times when an arrogance associated with someone's feeling that their research is much more important than anyone else's and that they will control their activity any way that they want. This type of attitude can be devastating to a radiochemistry program where all researchers must work together to accomplish their projects. The way to accomplish large projects is to get all personnel involved; the way to minimize nonbeneficial interaction is to ensure that all personnel know what it is.

Over the years there have been many changes in the management of the radiation safety program, the research facility, the Department of Chemistry, and the College of Sciences, but there have been no true changes to the Authorized Users in the radiochemistry program. This consistency in the program and the working relationship between the radiochemistry professors has been the force that has kept the program intact. Control of undesirable behavior in the laboratories is the responsibility of the faculty and staff. It must be recognized and dealt with quickly and forcefully. A solution should be presented to management that will resolve any situation that is detrimental to the work of researchers in the program and allow completion of the work of all researchers, even if they need to be separated. Researchers need a management member capable of resolving conflict, to prevent issues that may cause a loss of control.

2.4 Communication of Program Ideals Hypothesis

In training personnel to work with radiation and other hazards in the radiochemistry laboratories safely, there must be expression that security of the materials is essential, dose must be As Low as Reasonably Achievable (ALARA), and contamination control must prevent radioactive materials from becoming airborne and from leaving the confines of the laboratory. These are not goals of a radiochemistry program; they are essential aspects of the radiation safety program. These simple controls prevent violation of the conditions of the UNLV State of Nevada radioactive materials license so that radiochemistry research may continue at UNLV.

The goals of the program must be less lofty and present a quantifiable level below which is ALARA, above which is unacceptable and requires adverse interaction with a laboratory authority if exceeded. Original contamination control goals were to maintain contamination levels less than 10,000 dpm/100 cm² for beta emitters and 100 dpm/100 cm² for alpha emitters in the continually contaminated areas of laboratories, and maintain contamination levels less than 1000 dpm/100 cm² for beta emitters and 20 dpm/100 cm² for alpha emitters in all other areas of the laboratory. These later goals have since become the contamination control limits for the radiochemistry laboratories. There does not appear to be an allowance of contaminated areas at the current time, even when experiments are in progress. The rationale for these limits is associated with the desire to minimize the occurrence of

contamination in unrestricted areas. If higher levels of contamination were allowed to exist in work areas, then the frequency of observing activity outside of the work area would increase. This would also be the result of lowering the contamination control limit to allow for some increased margin of safety. The basis for these limits is the UNLV Radiation Safety Manual, the foundation for these particular values is an obsolete Nuclear Regulatory Commission document on decommissioning from June 1974, Regulatory Guide 1.86.

For airborne activity, a safety factor of 20 is applied to the Derived Air Concentration (DAC) value for the radionuclide being used. This ensures that airborne activity is below the requirements for use of respiratory protection and any potential exposure will be below the 1.25 mSv per quarter year limit for areas where respiratory protection is not required. Ideally, airborne radioactive materials should never be present in the air space of researchers, but realistically this cannot be prevented.

For personnel, goals should minimize the probability that the annual dose will exceed 10% of the federal and state limits for total effective dose equivalent [48], less than 0.005 Sv per year. The fractions are the same fractions used by regulators to provide a limit for unmonitored workers or workers under the age of 18. The standard of care for control of dose in the Radiochemistry Program is the regulatory limit of 0.05 Sv/year. The goal is to establish a level of dose which fits with the excellence of the radiochemistry program. This level demonstrates that the control of higher amounts of activity is

accomplished in the program without harm to program participants. The essential elements of the program are easily communicated to members of the radiochemistry program and they are recognized as non-deviation requirements by the UNLV Radiation Safety Staff.

2.4.1 Key Indicators and Tracking Performance

As the Radiochemistry Program has grown, incidents have been experienced that put the program in need of actions to minimize the occurrence of events adverse to radiation safety and ensure that control of the laboratories was not at risk. Three incidents in 2008, discussed in section 2.2, demonstrated that the program needed changes and researchers were not as good at control of radioactive material as was considered before the events occurred.

Each of these incidents was the result of inattention to detail associated with the work that was being done. None of these events was found to have caused a release of radioactive materials from the laboratories and there was no intake of radioactive materials by any of students, faculty or staff that occupied the laboratories where these events occurred. Since then, there have been several changes that have resulted in a much more closely guarded program and much better controls. Each event was an experience that changed the behavior of personnel in the program, the potential consequence was the loss of the Radiochemistry Program, the response to each experience was, laboratory closure, area cleanup, verification that there was recovery

from the event, documentation of the event, discussion of ways to prevent such an occurrence in the future and a change to the training program to include discussion of each loss of control.

Several measures of the status of a radiation protection program should be used to ensure that the program is performing in a manner to ensure conditions of the radioactive material license are satisfied. A reasonable example for a radiochemistry program is listed in Table 2-3. The focus for this program is contamination control, prevention of internal exposure, and waste minimization.

Table 2-2. Proposed Performance Indicators for Radiation Safety Programs

Key Indicator
The number of skin contamination events.
The number of personal clothing contamination events.
The number of air samples that indicate an airborne concentration greater than the DAC value for the radionuclides used.
The personnel dose equivalent in excess of 1 mSv in a badging period.
More than 10 cubic feet of waste generated in a month.
The number of spills that required greater than 1 hour to clean.
The number of times a laboratory was closed for contamination control problems.

An increase in the number of events for each category must cause a re-evaluation of the control measures and not the key indicator. For example, if the number of skin contamination events in a month is five, and no events have ever been recorded, then there is an indication of poor contamination control. The action taken is resolution of the bad practice. Performance should show continuous improvement. If an indicator shows a trend toward less control, action should be taken to put the indicator back on the right path.

Each event caused a re-evaluation of control methods and the needed changes for the program to continue into the future. One change that has helped substantially by the contamination events was increased awareness of the need for assistance by the UNLV Radiation Safety Office. Immediately after the first important loss of contamination control, the RSO was asked to change the frequency of the surveys that they completed from quarterly to monthly. Surveys completed by the Laboratory Support Group were done weekly and the number of smears taken in high risk laboratories was increased. This increased surveillance would provide a more timely evaluation by the Radiation Safety Office and a more timely alert should something be missed by the research staff surveys.

In large radiological facilities with an abundance of manpower there is a tracking of key indicators to ensure that everyone is aware of situations that are adverse to a quality radiation safety program. The quality of any program should be a concern to the management of that program and items that are preventable and may cause unnecessary harm (or dose) to participants are tracked so that preventative methods may be tested. Such indicators as identified in Table 2-3 should be tracked at these facilities. The UNLV radiochemistry program has done well without these types of encouragers because there has always been an influence from the faculty that unacceptable behavior will be strongly discouraged and the students in the program have always had a high level of maturity. As the program changes and other influences manifest themselves, the desire to implement such a program of tracking performance of the program may become important to improve the program and demonstrate regulatory compliance.

2.5 Radioactive and Hazardous Waste in Radiochemistry

In the United States radioactive waste is considered an undesirable aspect of nuclear power and any industry where radioactive materials are used. Disposal of unneeded residue or contaminated equipment is required. Early in the history of radioactive waste generation there was not a concern for the volume of waste generated and waste sites were filled to capacity. Poor management practices also caused inappropriate disposal of caustic materials and deterioration of waste containers at licensed disposal sites. Recovery from these practices led to increases in the cost of waste disposal and a need to reduce waste volume generated to reduce cost.

Radiochemists make hazardous wastes that are radioactive. This is normally accepted as the nature of the program. These materials together are known as mixed waste when they are no longer a part of research. The new mantra for radiochemistry students and researchers should be: "If you create a hazardous radioactive product, you will return that product to a non-hazardous, reusable, radioactive product or radioactive waste." There will be times when such an accomplishment is not attainable, but with encouragement of faculty and staff, this can be an advantageous philosophy.

2.5.1 Challenges in the Creation of Mixed Waste

The last three chapters of this dissertation identify the most important control problem that lingers in the program today. Waste is the Achilles heel of nuclear power in the United States and it could be the demise of a radiochemistry program if allowed. If new compounds and new techniques for chemical synthesis and separations that enhance our world can be developed, participants in the program can remove the hazardous components of residues created in the laboratories.

Early in the program (2004-2006) there were issues of personnel throwing potentially radioactive and sometimes radioactive waste in the non-radioactive trash. This problem was mostly eliminated within the main radiochemistry population. However, this issue reappears with new people to the laboratories. A constant reinforcement of this rule prevents lapses of compliance.

Since the start of the radiochemistry program, liquid waste has been created at a slow rate. The volume created is relatively small but the degree of associated hazard is high. Waste is put into plastic containers and stored in satellite accumulation areas. The labeling of the waste is not consistent and the originator of the waste and its chemical constituents are not identified.

One major issue in reuse of waste materials that have been recovered is the purity of the final product. Actions should be taken to purify the material of concern to a specific documented endpoint. The recovery should be complete when the radionuclide is in a final identifiable form. Chemical separation of the radioelements may be beneficial to enhance the desirability of reuse.

2.5.2 Actions Needed to Control Mixed Waste

In order to facilitate reuse of recovered materials, it is recommended that a Recovered Material Registry documenting the process to be developed (Figure 2-4). The registry should contain;

- Chemical components and concentrations
- Mass
- Radionuclide and mass or activity
- Researcher who produced residue material
- Experimental methods used to produce material (can reference publication(s), report(s), or theses).

The usefulness of any recovered material will be based on the need and desire of researchers to modify the chemical compound containing the radionuclide. Other information should be provided for the radionuclide to allow an informed decision to be made. Indication of impurities or any known features of the compound(s) may also be useful.

UNLV Radiochemistry Program Recovered Material				
Stock Material Identification Number				
Inventory Number				
Date of recovery				
Material recovered by				
Volume of solution ml Solution pH				
Radionuclide(1):; Mass (1)g; Moles (1);				
Activity(1)Bq				
Radionuclide(1):; Mass (1)g; Moles (1);				
Activity(1)Bq				
Radionuclide(1):; Mass (1)g; Moles (1);				
Activity(1)Bq				
Chemical compound(s):				
Known Impurities:				
Notes:				

Figure 2-4. Recovered Material Documentation

2.6 Administrative Controls Hypothesis

User interactions, work planning, policies, transportation, surveys and monitoring, evaluation of the program and laboratory management are the main personnel functions that make the radiation safety program in the UNLV Radiochemistry Program functional. Administrative controls are the exercise of authority over radiation safety in the Program by control of resources or equipment, logistics, and personnel management. The design of the laboratories and equipment requires an administrative means of operation or it will not be safe.

2.6.1 Program Control - Administration

The UNLV Radiochemistry Program is controlled by a broad scope type B radioactive materials license under the authority of a Radiation Safety Officer with support of a committee of volunteers known as the Radiation Safety Advisory Committee. The Committee meets quarterly to discuss items of concern to the Radiation Safety Officer and provide him/her with their guidance. This system has been in place for more than ten years and has worked well for the radiation safety program. However, even with support of the committee, the radiation safety staff has not kept up with the demands of the UNLV Radiochemistry Program.

In the past five years there has been a progression of Radiation Safety Officers responsible for the radiochemistry laboratories. Each has had a different idea about how radiochemistry work should be controlled. The most important aspects of control in the operation of these laboratories are to keep the radioactive material secure, minimize dose to personnel, and recover and reuse radioactive materials. Each of the radiation safety personnel that came through the radiochemistry laboratories looked at the controls in a different way and provided some degree of enhancement of the radiological control program. Everybody looks at things different; the support provided by these individuals has improved the radiochemistry laboratories.

The Radiation Safety Office provides the license authority and an independent evaluation capability that cannot be a part of the radiochemistry program so that decisions can be made in favor of safety. The line of authority for the Radiation Safety Office is to the president of the University through a pathway that avoids any research conflict of interest.

2.6.2 User Designation and Support

Authorized Radioactive Material Users are trained faculty members and staff members who are trusted by the Radiation Safety Officer and the Radiation Safety Advisory Committee to control the laboratories where radioactive materials are used and stored. They also control all work within those laboratories in accordance with the conditions of UNLV's radioactive material license, State of Nevada regulations, and Federal Regulations regarding the use of radioactive material. A simple loss of communication between the Authorized Users in the radiochemistry program could cause work to be done without the knowledge of the User on duty and result in someone doing work in a

method that results in serious injury or death to a student or other faculty member. This would be devastating to the researcher's family and friends, the User on duty, and every member of the radiochemistry program.

Authorized Users recognize that the authorization is a privilege granted to a trusted person who will protect the radioactive material license while doing their research. This same degree of trust is provided to the students and staff of an Authorized User who use radioactive materials in research under the authority of that person. The interaction of Users in a single laboratory is where this need is most easily recognized. Consider the following situation; a researcher working for one User takes an action that puts another researcher who works for another User at risk. A correction should be made to prevent the first researcher from causing risk to anyone else in the laboratory. A conflict may arise when the second User instructed his researcher to do it that way.

There must be an ultimate authority within the radiochemistry laboratory management that has the ability to correct this type of situation as it is discovered. The correction is always unpleasant for somebody, but the needs of the program must be fulfilled. Depending on the RSO to take corrective actions in this situation places control of the disagreement in a different management chain. This is undesirable. The RSO provides a needed service to the Radiochemistry Program and communication between the RSO and any person using radioactive materials must never be stifled. Situations that involve deviation from

regulations or even from good work practice by Authorized Users should be resolved by Authorized Users, the ability to resolve all situations like this should reside in a Radiation Safety Committee that has supervisory authority over all work with radioactive materials and radiation producing devices. Only with that authority can these differences of opinion be resolved without a loss of communication.

2.6.3 Transportation Challenges

The shipping and receiving of radioactive material is governed by regulations of the Department of Transportation in the United States [49]. All radioactive materials must be controlled to prevent unauthorized transport from the UNLV campus. In order to support radiochemistry experiments, materials and equipment may require movement to laboratories in other buildings or transport to laboratories in other states. All transporters, either on campus, or off-campus must be properly trained and follow the shipping regulations and guidance provided by the DOT and the International Air Transport Association. Even the most detailed preparation of packages will occasionally fail inspections required by carriers. It is essential that personnel who ship radioactive materials for the Radiochemistry Program receive detailed training to ship radioactive packages and collaborate to ensure that each package is properly classified, packaged, labeled, and marked.

The authority to ship radioactive materials for the Radiochemistry Program must be granted from the highest levels of program

administration. The Radiation Safety Office must work together with the radiochemistry program to ensure that the details of all shipments are properly evaluated and addressed. There are times when a rush to meet deadlines for experiments may cause a desire to ship inappropriately, this must never happen as it will put all operations of the program at risk.

2.6.3.1 Actions to Support Relocation of Materials

Transportation on campus is as important to the program as when a package is moved off-campus through the public. A policy imposed early in the program was for transportation of any materials that could cause an area to be contaminated. The policy was specified in radiochemistry newsletter number 7, and is presented in Appendix B. The basic concepts are:

- A user must be aware of the transfer of radioactive material between buildings and there should be a more senior student or User accompanying the transporter.
- The package must be capable of containing the material and must be labeled to indicate the radionuclides and activity.

2.6.4 Planning Experiments with Radioactive Materials

When work with radioactive materials is considered for any purpose, an evaluation must be done to establish control measures for protection of personnel and the environment. This evaluation may be simple and associated with no personnel risk and no environmental effect, or it may require controls to reduce external dose rates, intake of radionuclides, contamination control measures, and environmental protective measures. The evaluation of radiation work and an organized documentation of that work can be done with the aid of software.

2.6.4.1 Work Planning Challenges

Early in the evolution of the program considerations given to the work did not consider all aspects appropriate to safety although when activity (>1 MBq) was used, a discussion of the work to be done and the need to minimize the probability of airborne activity and surface contamination was held. One of the first set of experiments to use a high activity concentration in solution involved > 10 MBq of ²³⁷Np. A detailed training of the researchers was held to ensure that everyone was aware of the serious nature of using this radionuclide with a total activity greater than 2000 times the ALI by ingestion and 250,000 times the ALI by inhalation.

The dominating challenge associated with work planning is assuring that all Users are satisfied with the planned controls and sufficient communication has occurred so that supervision of the work will be available when the work occurs. A general policy early in the program was the presence of a senior member of the staff or faculty was required before work with radioactive materials could proceed. This challenge has become difficult with the increased number of researchers and more need for User coverage.

2.6.4.2 Actions Taken to Support Work Planning

In 2010, a method was implemented to keep track of the experiments that were planned for each upcoming week. Each researcher is required to submit a "Plan of the Week" form that describes to the group of Users that may be responsible that week, what will be done. The plan requires the identification of radionuclides and activity, the researchers and the laboratory, and the methods that will be used. Before implementation of the plan, the approval of the Authorized User is required.

This method provides a communication between the researcher, the Authorized User, and other Users who may be responsible for the laboratories when the work is done. The form provides a basic description of the work to be done including:

- Starting date and expected end date,
- Name of the researcher,
- Radionuclides and mass or activity,
- Laboratory designation,
- Environmental influences that will be experienced by the material,
- Analyses to be done,
- Allowance for User evaluation notes, and
- User Authorization.

Depending on the application, control measures, interaction of multiple work groups, and degree of liability accepted to do this work; multiple levels of authorization may be required. The level of authorization provided should be commensurate with the degree of risk associated with the work. The degree of risk should consider not only radiation hazards, but also other liabilities in doing the proposed work. For large experiments with high risk, the radiation safety office should be a required authorization.

Issues not presented by the Plan of the Week but require consideration are purchasing of materials, the limits of activity appropriate for the experiments, protective clothing requirements, external and internal monitoring requirements, and surveys to be done before during and after the work.

Upon authorization to begin work, authorization may be made to purchase radionuclides, protective equipment, and other needed supplies. As the work continues, monitoring of the work area provides information related to job progress, interferences to radiation safety measures, the need for more or less protection, or the need to stop work and reconsider the work plan. In the event of high exposure to personnel, loss of contamination control or unanticipated airborne activity, work would be stopped and the cause evaluated. To continue, corrective actions would be taken, plans reviewed, control measures evaluated and assigned, and re-authorization considered.

Upon completion of the work, it is customary to evaluate the work area with a post job survey to identify the degree of hazard that remains or was created as a result of the work. This survey considers surface

contamination, external dose rates, and airborne activity. The post job survey requires review by the radiation safety organization.

2.6.5 Surveys and Monitoring Hypothesis

The purpose of monitoring is multifold. Surveys identify the condition of a work area before, contamination and dose rates they are exposed to during, and the situation that remains after work. Whether it is contamination or dose rate, the condition of the area after work should not be worse than before work was done. With appropriate training and experience in how to do surveys researchers will be able to maintain their areas to ensure minimal personnel contamination, and dose.

2.6.5.1 Challenges to Surveys and Monitoring

Early in the radiochemistry program it was decided to allow contaminated areas to remain in fume hoods or glove boxes in an effort to reduce the amount of time spent cleaning those areas and thus reduce the dose received during the cleaning. The concept seems sound, but the level of contamination will build to the point where it cannot be contained in that area and will spread to areas where it becomes a concern. It may be reasonable to allow a buildup to some factor of 2 to 5 times the contamination control limits but this must be periodically evaluated and cleanup must prevent excess buildup. Areas immediately outside of those areas must be assessed for contamination on a more frequent basis. The most important areas for contamination control in a radiochemistry laboratory are associated with process locations where samples are cut, ground, pulverized, polished, heated, pressed, bubbled or weighed. Each of these operations creates small particles that may be spread around due to the motion of the particles when they are created, heated, or bubbled. Smear surveys alone are not sufficient to ensure a clean area after using one of these processes. In some cases, the process needs a contained area with little air movement to ensure control of the particles created.

2.6.5.2 Considerations for Surveying Radiochemistry Areas

In radiochemistry materials are manipulated in powders and solutions that may have high concentrations of radionuclides. When the process is complete a detailed evaluation of the work area for a spread of contamination is expected. This must be a slow continuous direct survey with an appropriate survey meter and a detailed smear survey to demonstrate the area is clean for the next person who will use it. When activity is found in an area, the researcher is responsible to decontaminate the area.

A program was implemented which made surveying work areas simple and keep the spread of contamination under control. From March of 2010 until the end of the year, hundreds of surveys were performed and documented to control radioactive materials at each of the radioactive material work areas. If you were a researcher in the

laboratories and did surveys, there was probably a time when you thought that all of your actions were deliberate and appropriate and you could not have spread contamination from your work area. However, when you counted your smears, there were surprises that indicated the presence of removable contamination in areas that you considered clean.

The discovery of radioactive materials in a work area immediately after work with radioactive materials is done should never be construed as a loss of control. A loss of control is when activity is found in a work area that a researcher has left contaminated. If radioactive materials could be seen as huge colorful spots as we work, then recognition and cleanup would be easy. However, a contaminated area cannot be found until an appropriate survey is completed. Table 2-4 displays the mass equivalence of activity of radioactive materials that used in the Radiochemistry Program at the contamination limit of 1000 dpm/100 cm² (16.7 Bq/100 cm²). With the typical density of that material, a particle size is also provided.

The approximate limit of our ability to see particles if we have good vision is a particle of contrasting color with a diameter of 0.1 mm (100 micron) [50] at 0.3 meter from the particle. Some people may see better, some not as well. This particle size is based on observation at a specific distance to identify an object of contrasting color. Table 2-4 is a reasonably close approximation of the size of radioactive particles.

Another important aspect to consider when looking at the radiological control aspects of this contamination is that a person will not spend a great deal of time scanning the work area for barely visible particles, they may look like dust particles or just some dirt. As shown in the table, this size particle is much more easily detected with a radiation detector for most radionuclides.

Nuclida	Specific Activity	Density	Particle Volume	Particle Diameter
Nuclide	(Bq/g)	(g/cm ³⁾	(cm ³)	(mm)
⁹⁹ Tc	6.27x10 ⁸	11.5	2.32x10 ⁻⁹	0.0120
²³² Th	4.04x10 ⁴	11.7	3.53x10⁻⁵	0.2980
²³⁸ U	1.25x10 ⁴	19.05	7.04x10 ⁻⁵	0.3750
²³⁵ U	7.93x10 ⁴	19.05	1.11x10 ⁻⁵	0.2020
²³³ U	3.50x10 ⁸	19.05	2.50x10 ⁻⁹	0.0123
²³⁷ Np	2.61x10 ⁷	20.45	3.13x10 ⁻⁸	0.0286
²⁴¹ Pu	4.16x10 ¹²	19.84	2.02x10 ⁻¹³	0.0005
²⁴⁰ Pu	8.38x10 ⁹	19.84	1.00x10 ⁻¹⁰	0.0042
²³⁹ Pu	2.26x10 ⁹	19.84	3.72x10 ⁻¹⁰	0.0065
²³⁸ Pu	6.43x10 ¹¹	19.84	1.31x10 ⁻¹²	0.0099
²⁴³ Am	6.86x10 ⁹	13.6	1.79x10 ⁻¹⁰	0.0051
²⁴¹ Am	1.28x10 ¹¹	13.6	9.62x10 ⁻¹²	0.0019
²⁴⁴ Cm	3.08x10 ¹²	13.51	4.01x10 ⁻¹³	0.0007

Table 2-3. The Size of 16.7 Bq Particles for Common Radionuclides

In Table 2-4 a determination is made of the particle diameter for different radionuclides with the consideration that it is a pure metal with the specific activity and density provided. Considering that the limit of our vision is indeed a particle that has a diameter of 100 micron, an approximation of the activity can be made as shown in Table 2-5. In this table it is considered that the volume of that particle is 1.33x10⁻⁶ cm³ and the activity in Bq, is the activity that is in that volume.

The shaded information in Table 2-5 shows those radionuclides with a specific activity that enables them to be easily detected with a radiation detector even though the particles are not likely to be visible to most people. Consider also that a particle 10% of that size will not be visible, yet it will still have an activity greater than the contamination control limit. This table shows that a smear survey provides what can be found with a slow scan survey using an alpha/beta scintillation detector such as the Ludlum model 43/93. This instrument is typically available in all of the radiochemistry laboratories at the HRC.

Nuclide	Specific Activity (Bq/g)	Density (g/cm ³⁾	Activity (Bq)
⁹⁹ Tc	6.27x10 ⁸	11.5	9.61x10 ³
²³² Th	4.04x10 ⁴	11.7	0.631
²³⁸ U	1.25x10 ⁴	19.05	0.316
²³⁵ U	7.93x10 ⁴	19.05	2.01
²³³ U	3.50x10 ⁸	19.05	8.89x10 ³
²³⁷ Np	2.61x10 ⁷	20.45	7.12x10 ²
²⁴¹ Pu	4.16x10 ¹²	19.84	1.10x10 ⁸
²⁴⁰ Pu	8.38x10 ⁹	19.84	2.22x10 ⁵
²³⁹ Pu	2.26x10 ⁹	19.84	5.98x10 ⁴
²³⁸ Pu	6.43x10 ¹¹	19.84	1.70x10 ⁷
²⁴³ Am	6.86x10 ⁹	13.6	1.24x10 ⁵
²⁴¹ Am	1.28x10 ¹¹	13.6	2.32x10 ⁶
²⁴⁴ Cm	3.08x10 ¹²	13.51	5.55x10 ⁷

Table 2-4. Activity of a Visible Particle for Different Radionuclides

The current series of surveys that are completed in the laboratories to maintain control have evolved over the years so that mistakes can be recognized and action taken to correct unfortunate errors before they affect others in the laboratories. The three levels of contamination control survey protection are good and exist as shown in Table 2-6. Use of a system of contamination control that provides checks by multiple groups provides protection for the program and UNLV.

Survey Type	Frequency	Surveyor(s)
Work space control	Each time work is done in a work area.	Individual Researchers
Laboratory control	Weekly	Laboratory Support Professionals
Program control	Monthly	Radiation Safety Office Personnel

Table 2-5. Contamination Control Survey Frequency

2.6.6 Identifying Non-Compliance

In any large program where radioactive materials are used, there is a license requirement to evaluate the radiation safety program annually. This overall evaluation of the program is a function of the radiation safety office. However, in order to minimize non-compliance situations in the large scale review daily evaluations of compliance are recommended for a radiochemistry program.

2.6.6.1 Challenges to Compliance with License Conditions

Non-compliance with laboratory rules is discovered when adverse situations result. In order to identify non-compliance which might result in harm to personnel, the current Radiation Staff frequently visit the radiochemistry laboratories for the purpose of assessing compliance. Usually it is another researcher that identifies if someone has contaminated an area and did not clean up. This is because the researchers are required to do surveys every time they work.

Early in the program a student used a large UV light and observed the distribution of uranyl compounds on surfaces in laboratory MSM-164 [51]. Even though a person could visually identify the presence of these compounds, the absence of surveys caused contamination of keyboards, phone keypads, door knobs, and bench tops. As the routine laboratory duties are accomplished, a laboratory inspection is done. This includes looking to see what is in the trash, review of the setup of each experiment, observation of how and where samples are stored, determination of what is reasonable for waste that is collected from that area and what notation is expected on the waste inventory form for each laboratory. When items that deviate from good work practices are found, action must be taken to correct the situation, or if it is a task that would require hours, document it, photograph it and let people know about it.

The following items of concern have been found periodically in the radiochemistry laboratories:

- radioactive material found in clean waste,
- untrained personnel in a radiochemistry laboratory,
- sloppy control of radioactive material work areas,
- contaminated equipment (furnaces, pellet press, gloveless box),
- contamination in normally clean areas,
- contaminated PPE in a clean area (laboratory coats),
- unsecured radioactive materials,
- safety glasses in a contaminated work area,
- blocked safety equipment,
- blocked emergency exits,
- fume hoods for radioactive work inoperable,

- argon glove boxes without adequate argon supply, and
- contaminated radioactive material storage areas.

Each of these items could have become an item of non-compliance or caused injury to personnel if it was not discovered and corrective action taken.

2.6.6.2 Actions Taken to Avoid Non-Compliance

A daily review of all laboratories before work with radioactive materials and a walkthrough before leaving for the day are actions that will identify conditions adverse to good radiological controls before they cause harm or become items of non-compliance. This walkthrough should be done by a senior researcher or Authorized User with the specific intent of identifying problems that need correction.

Because of the 'area possessive' nature of researchers, the Authorized User of that area will easily be identified and they will recognize the need to fix items that deviate from proper control. If the need for cleanup or a change to control habits is not identified, it must be easy to cut off access to the laboratories until the offender recognizes the need to clean up the area or provide more control. The real unresolvable difficulty is associated with the people whose time is wasted by the inconsiderate actions of some others.

Every person who has access to the radiochemistry laboratories has the power to violate a regulation that would cause the immediate failure of the Radiochemistry Program. The honesty of every person in

the program and the trust in every person who is allowed access to the laboratories by faculty and staff are two of the main components that fight non-compliance. If at any time, a trusted member of the organization observes a situation adverse to the goals of the program, the Authorized Users must be notified and action must be taken.

2.6.6 Emergency Preparedness

"The ultimate objective of disaster management is to bring the probability that damage will occur from an event as close to zero as is possible" [52]. In radiochemistry or any area where there are a number of hazards, the awareness of risks created by the researcher or others in the laboratories is essential. Hazards and risks that will be dictated by nature or equipment failure are not possible to prevent, but easy to prepare for.

As the Radiochemistry Program grew and the need to prepare for an emergency situation was recognized, the laboratories were stocked with fire extinguishers, first aid kits, absorbent materials, protective clothing, and a laboratory presence so that people recognized there was help if it was needed. The support staff has 40 hour HAZWOPER (Hazardous Waste Operations and Emergency Response) training [53], a confidence in the laboratories that comes from years of experience, and familiarity with the equipment and facilities.

Training of personnel in how to respond to an emergency is extremely important. Drills of postulated situations make people aware of

what actions are best to help people that may have been hurt and take action to return the laboratory spaces to normal after the event. In 2005 the Environmental Health and Safety department conducted a chemical spill drill that had participation from the Clark County Hazmat team. It is always possible that the unexpected occurrence will cause a fire, a spill of hazardous or toxic or radioactive material that will result in some degree of harm to one or more people. Obviously, the frequency and severity of such events must be minimized, but any time that humans are involved in an area such as the radiochemistry laboratories, it is possible that an accident will happen. If personnel are trained and ready for an adverse situation, it might be avoided or the probability of harm as a result of it might be reduced.

The first drill training occurred in November of 2010. The first drill was conducted in December of 2010 and the actions taken by the student responder were excellent. The drill team needed to improve their operations, to more appropriately conduct and evaluate the drill, but drills are held so that everyone learns from the experience. Drills associated with possible undesirable events in the radiochemistry laboratories should be held more frequently to ensure that proper actions will be taken should a real event occur.

2.6.7 Radioactive Material Security

Security of materials that might cause harm to members of the public has become more of a concern since 2001 and the concept of

public terror became an important aspect of life in the United States. Radioactive materials in large quantities may cause a dose to personnel that could result in undesirable effects; small quantities of radioactive materials are ingested, inhaled, and absorbed into our bodies every day. The distinction in the radiochemistry laboratory has to be associated with the fact that the materials used are licensed.

Security of the radioactive material in a radiochemistry program is directly controlled by the radioactive material user. Since the start of the program, the Authorized Users are the only people to have access to the laboratories supply of radioactive material. In radiochemistry at UNLV, access to the material requires access to the building, the laboratory bay, the laboratory containing the storage room, the room where the material is located, and the combination to the safe in which it is stored. Each of these access points requires a method of access controlled by the Authorized User, building management, and the Radiation Safety Officer.

Licensed radioactive materials must be controlled in a manner that prevents the likelihood that they will be uncontrolled when outside if the radiochemistry laboratories. When out of any laboratory posted for control of radioactive materials, they must be properly packaged and labeled so that in case of an emergency, the correct people can be immediately notified to establish control while the emergency is handled. Licensed material is under inventory control at all times. A semi-annual inventory of the materials verifies their location, who has possession of the material, and the condition of their container. For special nuclear material controlled under the Nuclear Material Safety and Safeguards policies and distribution of the material must be controlled for each researcher. Disposal by the researcher is recorded and the disposition of material in milligram amounts is ensured in this manner. Periodic reporting to the Office of Nuclear Materials Safety and Safeguards is required to verify that the material is properly controlled to disposal or transfer.

2.7 Management

The most important aspect of any radiation safety program is support from management of the licensed entity whether it is a university, government facility, or industrial facility. Good management of a Radiochemistry Program comes from recognition of the hazards that will be created and providing for proper protection of the people that will do the work. The important aspects of radiation protection are discussed by the International Commission on Radiological Protection (ICRP) in [54]. These are refined by the National Council on Radiation Protection and Measurements (NCRP) in their reports over the years and discussed in detail in [55]. In the United States, the fundamental principles of radiological protection are incorporated into regulation based on the recommendations of the NCRP.

2.7.1 Management Challenges in Radiochemistry

When using radioactive materials in powder and liquid forms, the primary hazard for actinides is from inhalation and ingestion. The degree

of hazard associated with each experiment can be based on some simple management tools that are derived from the work developed at the University of Massachusetts at Lowell [56]. This work was developed over the years based on the recommendations of the ICRP in several publications, the initial work in ICRP 2 [57] and later, ICRP 30 [58].

As a radiochemistry program grows, the costs associated with operating such a program also must grow. The radiation safety support in time, surveys, evaluation of the laboratories, bioassay sample analysis, and job coverage will grow in proportion to the increase in the number of laboratory spaces and the number of personnel in the program. The following considerations for cost should be revisited at least annually.

- radiation safety and laboratory support manpower
- radiation safety technical support capability
- survey instruments at the laboratories
- supplies and equipment to conduct and analyze smears
- support for bioassay requirements

2.7.2 Actions to Facilitate Management Support

The Radiation Safety Officer (RSO) is the coordinator for all radiation work. Authorized Users are the trusted extension of the RSO to implement requirements of the radioactive material license and protect workers. The person in the position of Radiation Safety Officer has now changed six times in six years (2006 to 2012). A stable program can only be achieved with a stable radiation safety office and the support of upper management. The American Academy of Health Physics published [59] standard qualifications for a university RSO in 2003.

Radiochemistry faculty must be more than capable to control the work and teach the students, and the laboratory manager must understand the issues required to control the laboratories. These features must provide for the control of work with radioactive materials. In doing so, the information needed must be provided in detail for high risk work so that there is clear understanding between the researchers, the Authorized Users, the laboratory managers, and the radiation safety staff. The details of controlling will depend on many factors, the radionuclides, the activity, the methods, and the interaction between the people involved [60]. Before each experiment there must be a detailed analysis of the possible outcome, potential undesirable results, and possible actions that may be needed to recover from those results.

Good radiation safety management is the only acceptable, safe pathway to work with radioactive materials and radiation producing devices. It is also important that corporate management maintain control of a radiation safety program to ensure that the impact of events does not destroy a program or the company. In 2006, just after transitioning to the radiochemistry program two papers were presented at the Health Physics Society annual meeting in Portland Oregon [61] [62]. The first paper provided a discussion of the need for any radiation protection program to have a reporting chain to the highest levels of management of

any corporation or entity which allows the use of radioactive materials. This paper was a discussion of first-hand experience in association with the management of radiation protection organization which had become dysfunctional and a comparison of this with the program described in a paper delivered just six months previous to that time [63]. The second paper outlined the risks associated with managing a radiation safety program in a poster style that questioned people about their actions as RSO should a person be hurt due to accident or program failure. This paper displayed the many risks that must be considered and the responsibility that a large program Radiation Safety Officer accepts. There were no answers provided in this presentation, only questions asked in hope that the reader would acknowledge the many aspects of program control that are necessary.

For example, regarding regulatory compliance - A good radiation safety program provides compliance with all regulations.

- What if you don't have sufficient resources to provide compliance?
- What if you get lucky and appear to comply but have some serious problems that go unnoticed?
- What if an audit finds that you are not in compliance?
- Will management help you achieve compliance or pay the fine?

Every safety program manager must accept that unpleasant things can happen. Unpleasant things happen every day, they just don't necessarily happen to you. When commitment is made to management of

a program for minimizing the probability of harm to workers, the public, or the environment, the greatest achievement is prevention of injury.

2.7.3 Perceptions of Work with Radioactive Materials

The radiochemistry program is in a fish bowl where everyone outside is watching to ensure that no one in the program takes an action that could be perceived as hazardous to personnel outside of the program, even if there is a very low probability of harm. Every participant in the program must recognize the fragility of the program. Participants may be strong in the science, they may be strong in protection, but they are humans in a sea of other humans who do not have the same goals and ambitions. The fear of radioactive materials continues to propagate throughout our population due to ignorance.

The Yucca Mountain Project is a classic example of ignorance preventing a project that is needed for protection of the American people [64]. As the politics has changed over the recent past regarding the desirability for nuclear energy, the need for long term isolation or for used nuclear fuel reprocessing has not been recognized as a responsibility of our government. The failure of the politicians in the state of Nevada to recognize the need to provide education for the people in their state is a serious deviation from science based decision making. Using fear as a way to prevent something that people need to know the truth about is inappropriate.

The actions taken by students in the radiochemistry group, teaching people about science and radiation has been outstanding. The participation in the Health Physics Society, American Nuclear Society, and providing support for the Boy Scouts of America Nuclear Merit Badge program is great. The professionals working with radioactive materials and radiation producing machines, providing teaching, or radioactive products must also participate if we are to grow as a State and country.

2.7.4 Visitors to the Radiochemistry Laboratories

Visitors to the radiochemistry laboratories are very important. They provide the opportunity to highlight laboratory capability and knowledge of personnel in the program. They also present a challenge for program participants to get the visitors out of the laboratory in the same condition that they entered.

During 2009 representatives from a Department of Energy facility that is very important as a funding source, a collaboration source, and as a future employer for our students toured the radiochemistry laboratories. During one of the tours a visitor observed what appeared to them to be less than satisfactory controls. Rather than ask about the situation as it occurred, that person reported to his management that controls at the UNLV radiochemistry laboratories were not very good. All students and faculty are encouraged to question anything that they perceive as inappropriate but training was not provided to visitors to encourage them to express their views so that all learn together.

As a result of that incident, a letter was written to the facility management to indicate that they must bring items of concern to the attention of the staff so that everyone would learn from them. A document was prepared that all future visitors must sign [65]. The document provides five simple statements of training and requires that all visitors read and sign the document. The document is then held for future reference should there be a case similar to what happened as a result of that tour.

The document describes that radioactive materials are used in the area in quantities that require radiological controls to;

- minimize external dose to personnel in the laboratories,
- minimize intake of radioactive materials,
- prevent exposure of visitors to our laboratories,
- prevent exposure of workers who maintain utilities in laboratories, and
- prevent exposure to other members of the public who may come in close proximity to laboratories.

It then provides a number of guidance statements for all personnel who intend to enter the laboratories for the purpose of touring or repairing utilities. The laboratory visitor must read and sign that they understand all of the statements made in the document. This simple action provides the visitor with a comfort that no matter what they see, if in their opinion, it does not look right, they can report it and they will get an answer to their concern. No one does everything right all of the time, but researchers and managers can only try harder if the issues are known. Communication of thoughts to the leaders of the radiochemistry program will ensure that the program continues to grow with minimum adverse influences due to lack of knowledge.

2.8 Implementation

The radiation safety program for radiochemistry at UNLV was structured as previously described. This section describes the actions necessary and the program administrative structure that was beneficial to implementing the program. An important consideration in the establishment of new controls is the need to ensure that researchers have had sufficient training and have confidence in their actions to prevent extreme reactions with chemicals in an area where the event could not be controlled.

2.8.1 Abstract

Incorporation of a Radiochemistry PhD Program into an academic radioactive materials license is challenging and requires strong management support to sustain operation. A radiochemistry program uses long lived, low Annual Limit on Intake (ALI) radionuclides in order to observe chemical reactions and take measurements, over period of time, that would not be possible with short lived radionuclides. A program designed for control of short lived beta emissions may have a decay in storage program and thus have no radioactive waste disposal program; this also is not possible for a radiochemistry program.

Collaboration with scientists from other facilities requires shipping specialists to ensure safe transfer of samples. As events occur, actions must be taken to evaluate the cause and reduce the probability of recurrence and thus protect the program.

2.8.2 Discussion

The University Radiation Safety Officer (RSO) is responsible to the regulator or to a Radiation Safety Committee to establish controls appropriate to maintain exposure to university employees, visitors, and students ALARA. Some factors that are important in that action are as follows:

- Management support to a program using alpha emitting radionuclides requires attention to facility capability, observation of work, review of experiments, accountability of materials, license changes, and detailed surveys to verify appropriate control measures are taken by the researchers.
- Management of the Radiation Safety Office requires technical support to be able to properly evaluate issues that might arise regarding exposure of researchers.
- Low ALI (high toxicity) radionuclides in powder form, not in solution, are required to be controlled in hoods or glove boxes.
- High toxicity radionuclides must be maintained in a controlled state or in solution at all times.

- Locations where radionuclides are used in quantities that represent a potential to cause high dose to researchers require more attention. At least daily visits to these locations are necessary.
- Abnormal events are evaluated via critique. A critique is a method to document the occurrence from the eyes of each participant so that a root cause may be identified and action taken to prevent recurrence of the event by identification of possible solutions in the data gathering process.
- Documentation of contamination control surveys by researchers is essential. A delay in this requirement as a means of minimizing survey burden on the researcher was detrimental to the program and recovery was difficult for a group that was not previously required to document their surveys.

In the beginning years of the UNLV Radiochemistry Program radiation safety support was provided by a Certified Health Physicist (CHP) as RSO with the knowledge that three other CHP's were on campus and could assist if needed. This support ensured appropriate attention to issues required to keep the program in operation. The knowledge and experience of radiation safety personnel must be strong to support this type of program. After leaving the UNLV EHS organization, the former RSO joined the Radiochemistry Program as the Radiation Laboratory Director, the Health Physicist responsible for radiation safety for radiochemistry. This was a good fit for the individual and the laboratory based radiochemistry program.

When mixing chemicals, radioactive or stable, inhalation or ingestion are the most important pathways for exposure. Most long lived alpha emitting radionuclides do not emit gamma radiation, but some do. Therefore, internal exposure is the pathway of interest. Since exposure of the lungs is of primary concern for alpha emitting radionuclides, the inhalation exposure pathway is most important to control. With internal exposure as the most important pathway, prevention of exposure is typically simple: keep the radionuclides in solution or in a situation where intake via inhalation or ingestion is not possible. Two simple techniques to prevent airborne activity are; 1. Keep the activity in solution, 2. Keep the activity controlled in a hood or glove box.

Training is the most important way for a researcher to know the rules and minimize their exposure to radiation; safety training must also provide protection techniques. Supervision is the only way for Authorized Users, Health Physicists, or Radiation Safety Office personnel to identify work practices that may lead to intake of radioactive materials and provide guidance to change them. The authorized user, responsible for radiation protection must be present in the laboratories at all times when research is in progress. Radiation Safety Office personnel should visit the laboratories frequently, at least daily when work is in progress.

When things go wrong there must be learning of how it happened and what can be done to minimize the probability for it to happen again.

In research, actions are taken that will occasionally provide unknown results. These results or other actions may cause an undesirable situation to exist or unplanned exposure of personnel to radiation or other hazards. A critique of the situation should document who was involved, what happened from the perspective of each person, and a discussion of the best ideas to prevent recurrence. Management should then implement the best solution identified.

In the first four years of the radiochemistry program there was reluctance by the Authorized Users and the radiochemistry health physicist to require documentation of contamination control surveys. It was considered more appropriate to enable the researchers to focus on their science and for others to focus on radiation protection. In hindsight, this was inappropriate and did not encourage researchers to do the required surveys. Documentation provides that encouragement and should be required. Surveys done by researchers should be for simple dose control or contamination control. Requiring surveys that are lengthy and take much of their time will cause issues of non-compliance; they will not be done as needed.

2.8.3 Summary

A radiochemistry program has special needs for radiological control that are not provided by radiation protection programs designed for short lived beta emitting radionuclides or those that only require protection from sealed sources. Control of long lived alpha emitting radionuclides

must consider contamination control measures as most important, provide for supervision of all aspects of research, and ensure that there is learning from events that cause undesired consequences.

CHAPTER 3

RECOVERY FROM DECONTAMINATION

3.0 Hypotheses

Radioactive waste production can be reduced and sustainability of the resources of the Radiochemistry Program can be accomplished through recovery of materials from contaminated surfaces. Specifically, resource recovery can be enhanced with use of new decontamination agents. In addition, a new decontamination gel is a useful, protective sampling tool.

3.0.1 Literature Review

This section describes a project which originated with the removal of technetium from surfaces of a research hood [66]. The evaluation of surfaces of the fume hood was made using techniques commonly described for radiological characterization surveys [67] [68] these are direct measurement using gas proportional detectors and indirect measurement using smears. The results of the characterization survey were reported in Operational Radiation Safety [69]. Since the Baker Atomic Weapons test near Bikini Atoll [70] that led to gross contamination of ships and land surfaces, methods have been researched to remove radioactive contamination from surfaces. There are many mechanisms to remove contaminants from surfaces. Reference [71] documents some general considerations from the 1950 era. Decontamination techniques include; washing using surfactants [72],

oils [73], or caustic solutions [74]; abrasion by blasting with CO_2 pellets [75], abrasive foam pellets [76] [77], sand, zeolite; peelable polymer surfaces [78], foams, sugar solutions [79], and many more [80]. Each of these methods fit a specific need; however, every one either causes more radioactive waste than desirable, is time consuming, or is inefficient in decontamination.

The following is a discussion of several available decontamination methods and the rationale for selecting the one used in this research. Most commonly, contaminated surfaces are washed with surfactant compounds (detergents, soluble foaming agents, emulsifiers, etc.) that provide a removal action by lowering the surface tension between the decontaminating liquid and the surface to be cleaned [81]. These compounds are inexpensive, readily available, and easy to use. However, there is typically physical scrubbing required to break the contaminant free from the surface and wiping or rinsing action to remove the solution creates volumes of liquid waste. This is not a desirable option for large scale decontamination of a fume hood because it is time consuming, requires scrubbing in hard to reach areas, and creates liquid waste. The surfactant is good for removal of contaminants from small areas.

An original technique for removal of contamination from ship surfaces after the Bikini Atoll tests was scrubbing with fuel oil. The method was abandoned after several attempts because the removal of contamination was difficult and efficiency was low. A more common use of oils for cleaning is associated with removing surface dirt and dust from

wood surfaces. The wood is not damaged and in fact is protected in the process and the cleaning compound typically provides a pleasant aroma. In the case of large areas of stainless steel and plastic surfaces with baked on contaminants, oil does not assist in removing the contaminant, it typically provides a slick layer that may also require treatment for removal. Oil was not a consideration in this case because it would require physical scrubbing and may leave a contaminant on the hood surface (the oil) that could damage experiment purity.

Blasting surfaces with sand and zeolite have been common for paint removal for many decades. A more modern approach in blasting was introduced in the 1980's using dry ice or abrasive foam pellets. Similar to the hard materials, these abrasives are blasted at the surface to be decontaminated and the contaminant is removed. The major difficulty associated with this process is the displacement of the radioactive contaminant from the controlled state on the surface to the free, loose state in the air. In addition, the abrasive blasting process may cause damage to the surface and other items that it hits. The most promising of these at nuclear plants was the dry ice blasting where it could be used on highly contaminated surfaces in a controlled environment with collection of the airborne activity via filtration. For the situation in a small radiochemistry laboratory, use of abrasive blasting would make the contaminant airborne and simply allow for its collection on other surfaces or in HEPA filtration systems. This technique is not desirable for fume hood decontamination.

A common method in dealing with highly contaminated surfaces is binding the contamination to prevent re-suspension and a high concentration of airborne activity. The contamination is attached to the surface using a sprayable fixative that is not soluble in water and is able to be stripped to remove a large fraction of the contaminant thus reducing the dose rate in the work area. This is frequently used during refueling processes at nuclear power plants where contaminated surfaces from refueling water may reach levels of MBq/cm². When the refueling water is removed from the refuel pool, contamination collects on the sides of the pool. The fixative is then sprayed onto the surface to reduce the amount of activity that would become airborne. After removal of the fixative from the pool walls, it is collected in shielded drums for disposal. The cost of this method is high but it has good decontamination efficiency, controls the spread of contamination and does not require physical scrubbing of the surface.

Another novel technique that was used in a highly contaminated area of an abrasive blasting room at Allied Technology Group facilities in Richland Washington was fixing the contamination with a fine spray of sugar water. The sticky surface provided good attachment of the activity to the surfaces so that they could be easily decontaminated using water and a vacuum cleaner with HEPA filtration that was set up for liquid collection. While the concept appeared sound, the use of sugar caused an unexpected problem, insects. A rapid infestation caused the cleanup to be much more difficult than anticipated and the method was not used again. One aspect of this technique that was desirable was the solubility of the fixative.

3.1 Selection of Technique and Evaluation

From the commonly used decontamination methods, the most desirable properties could be selected as follows:

1. Provides encapsulation of the contaminant.

- 2. Allows for removal of the contaminant from the surface.
- 3. Is collected from the surface by peeling.
- 4. Is in the form of a solid upon removal from the surface.
- 5. Attenuates emissions from radioactive material collected.
- 6. Does not create large quantities of waste.
- 7. Does not cause other problems (insects, mixed waste, etc.)

The search for the most desirable decontamination product identified a new fixative with all of the properties identified above. A small volume of this agent [82] was purchased and tested on some spills at UNLV and tests at other facilities [83] [84] were published. This product had one property not present with other usable decontamination techniques. The decontamination material could be put back into solution and the recovered material extracted and reused. This property was discussed [85] with the developer of the material who indicated that research had not been conducted. After this initial evaluation, the known information about the gel indicated it that it meets all of the most desirable properties of a decontamination method as previously described and more, it provides the opportunity to research recovery of a contaminant for reuse.

3.2 Experience Using the Gel at UNLV

Several events occurred in 2008 that prompted review of currently available decontamination methods, these events are described in a series of communications beginning with the description of a personnel contamination with ⁹⁹Tc [86]. The second event involved a spread of contamination from researchers working with ²³⁹Pu contaminated soil [87]. This was a minor loss of contamination control during the separation of hot particles from soil samples that had been collected from a nuclear weapon accident site [88]. The loss of control was recognized immediately by the researcher and action was taken to control the situation and remove all personnel from the area of the spill. Before any attempt at decontamination, the researcher recovered as many of the particles as possible for continued research. The floors in the laboratories where these two events occurred are designed to be slip resistant. This property is achieved by small hard shards that stick up from the rubber floor. When particles fall onto the floor they are difficult to collect and washing the floor surface will not remove radioactive contamination caught in the floor penetrations.

After initial cleaning which consisted of removal of sand and dust, decontamination gel [89] was applied to the floor in the affected area to immobilize identified contamination and collect as much of the

contamination as possible. It is noted at this time that no special preparation of the surfaces was done other than simple cleaning. The area adjacent to the gel was surveyed by direct measurement with an alpha/beta scintillation probe, no activity was found. The gel was allowed to dry overnight, it was peeled from the floor surface and all activity in the area was removed. Smear and direct measurement surveys were used to verify that the contamination was removed to below acceptable guidelines established by the UNLV Radiation Safety Office. The primary guideline in this case was a surface contamination limit of 20 dpm/100 cm² (0.33 Bq/100 cm²). The mass of ²³⁹Pu associated with the contamination was less than 10 micrograms, a mass that is below recovery considerations.

In an event involving ²³³U contamination of a concrete floor in one of the radiochemistry laboratories [90], the hydrophilic gel was spread on the floor covering the extent of the spill area. Approximately 2 liters of gel was poured from the container and spread over the contaminated floor area, approximately 5 square meters, with a thickness that ranged from 2 to 5 mm. The gel was spread by hand; the surface was not prepared in any way. The contamination on the floor was slightly above the contamination control limits with a maximum alpha emitter surface activity concentration of 20 dpm/100 cm². Considering that an area of 1 meter by 5 meters was contaminated to that level, then a maximum activity, A_{MAX}, for ²³³U, could be determined as follows: A_{MAX} = 100 cm X 500 cm X 20 dpm/100 cm² X 1 Bq/60 dpm and A_{MAX} = 1.7x10⁴ Bq, with an approximate specific activity of 3.5×10^8 Bq/g, the mass associated with this activity is estimated to be 4.8×10^{-5} grams. Recovery was not considered for this material; it was not thought of as an option at the time of these events.

The gel was allowed to dry for 24 hours and then it was peeled off of the floor as a solid. A survey of the floor after decontamination revealed that the floor showed no detectable removable or fixed ²³³U contamination. Removable activity was evaluated by smear surveys. The smears were counted on a Tennelec LB 5100 gas proportional counter. Fixed contamination was evaluated using a Ludlum 2360 rate meter with a 43-93 alpha/beta probe.

If recovery were considered; using the method previously described, the value of that recovery is determined as follows: The value of an uncertified nitrate solution of 48 micrograms of ²³³U is less than \$30.00 [91]. The cost of disposal of waste (2 liters of solid gel) is determined from the fraction of a 55 gallon (207.9L) drum times \$1000.00 per drum. This is about \$10.00. The cost of labor to recover the material would certainly be more than an hour, with a labor rate of \$50.00 per hour. Therefore the cost of recovery is more than the worth of the material plus the cost of waste and recovery is unreasonable.

3.3 Recycling of Tc from a Research Hood Decontamination

In four years of working with ⁹⁹Tc in milligram to gram quantities to make many different compounds and provide the resource for the

generation of several publications, a contamination problem was identified. Work in one of the fume hoods used by the UNLV radiochemistry program was contaminated to a degree that resulted in a significant increase in contamination events outside of the controlled hood as discovered in weekly surveys. Section 3.4 describes some of the chemistry that was done in this hood. It was decided that the hood should be decontaminated when the researchers were away, during the winter break in December 2009. The hood, until just before the winter break, held equipment from years of operation, as shown in Figure 3-1.



Figure 3-1. The Technetium Research Hood Before Cleanup

The ⁹⁹Tc research fume hood is a Kewaunee Supreme Air Fume Hood with open bypass operation with high efficiency particulate air filters in the exhaust train. The hood wall and back surfaces are stainless steel coated with anticorrosion material which is hard and smooth allowing for reasonably good removal of surface contaminants. The 1¼ inch epoxy resin base is the surface where all of the equipment and supplies were placed. The cup drain from this hood has been sealed flush with the level of the base. The sash is coated safety glass and is very easy to clean. Decon gel was not used on the sash, it was easily cleaned with a common cleaning liquid in the laboratory, Radiacwash® is one used in the Radiochemistry Laboratories, but many similar cleaning solutions are adequate for this type of surface.

3.4 Assessment of Previous Use of the Hood

This hood has been the location where many researchers have collected to work with ⁹⁹Tc in mg to gram quantities to investigate the chemistry of technetium. The primary focus of the research done here is on fundamental and applied technetium chemistry [92] [93].

In general, ⁹⁹Tc compounds and associated materials are subjected to a number of environmental influences in attempts to create new compounds or recreate those known to exist in order to characterize their behavior. Over the years, the research in this hood included: heating compounds in tube furnaces, mixing compounds in acids and bases, working with volatile and stable compounds, and working with various gases. Compounds have been spilled, splashed, sprayed, bubbled with gases, and vaporized in this fume hood.

Twelve grams of ⁹⁹Tc in the form of ammonium pertechnetate (NH₄)TcO₄-, or similar forms were used in research projects in this fume

hood. This relates to an activity of ⁹⁹Tc of 5.3 GBq. The experiments included investigation of the applied aspects of technetium chemistry with special emphasis on synthesis, separations, and materials chemistry [94]. Synthetic chemistry focused on metal-metal multiple bonding, oxides and halides. Synthesis and characterizations of (n-Bu₄N)₂Tc₂X₈, Tc₂(O₂CCH₃)₄X₂ (X=Cl or Br), TcO₂, Bi₂Tc₂O₇, Bi₃TcO₈, TcBr₃ and TcBr₄ have been performed. Some recent work included preparation of TcCl₄ from the reaction of ⁹⁹Tc metal with excess chlorine in sealed Pyrex ampules at elevated temperatures [95].

Heating in tube furnaces has introduced thermally hot discharges of air entrained with ⁹⁹Tc in different chemical forms into the exhaust air flow of the hood. The cooler surfaces of the hood and exhaust ducts have caused the Tc to attach to the baffles and plenum surfaces, as the particles contacted those surfaces. It is possible, but difficult to prove, that more than 10% of the activity used in these experiments has been attached to the hood and plenum surfaces or impacted the HEPA filters. Equipment present in the hood over the years has been a rotovap apparatus, mixers, hot plates, tube furnaces, cooling baths, Schlenk lines and associated clamps and racks, vacuum pumps, glassware, contaminated tools and many different gases.

3.5 Initial Assessment of Activity in the Hood

The first step in the decontamination was to conduct a reasonable assessment of the surface activity concentration within accessible areas

of the hood and assess the total activity present in the hood. The results showed an approximate 'loose surface activity concentration' of 20-100 $Bq/100 \text{ cm}^2$ with the highest activity concentration on the sides of the hood, possibly the result of sprays of activity from apparatus. As a result of the magnitude of this activity concentration, it was decided that the decontamination of this hood could provide some valuable information about the distribution of ⁹⁹Tc (the major radioactive contaminant in the hood) and the effectiveness of the selected decontamination compound, Decon GelTM 1101 [89].

The hood surfaces were marked into a grid pattern based on the size of a large area Berthold Xe counter [96] [97]. Smears were taken on several surfaces within the hood and counted on a Tennelec LB-5100 Alpha-Beta Counter [98]. The hand held Berthold LB-122 and the Tennelec LB-5100 were calibrated to the same ⁹⁹Tc source [99]. Decon gel was applied to 15 of the grid squares; five before the initial smear sample results were determined and 10 after the initial smear sample results were reported. The gel was applied before the smears on some of the grids to determine if the gel would remove some or all of the loose activity. This initial test provided reasonable assurance that the gel would be effective at removal of the ⁹⁹Tc from the hood without scrubbing. The activity distribution in the initial survey of the hood is shown in Figure 3-2.

22121		63699 S6	2645	
15979			4407	
	17319 S7		8692	

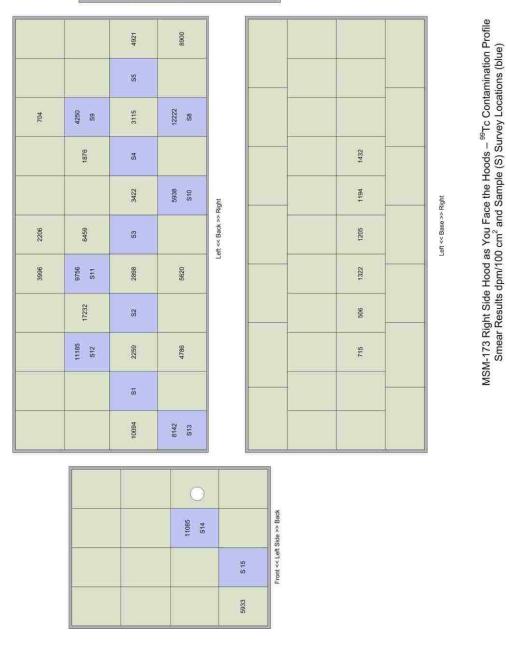


Figure 3-2. Display of the grid system used to evaluate smears, direct readings, and gel samples – Activity units = dpm/smear.

The contamination levels shown in Figure 3-2 are in units of dpm/smear with each smear representing the area of a Berthold LB-122 gas proportional detector, 14 cm by 22 cm (322 cm²). The sensitive area of the Berthold LB122 Xe gas detector is 11.8 cm by 18.9 cm (223 cm²). The Berthold LB122 was selected as the detector for primary measurements on the hood surface. This instrument, in the configuration used, has a sealed xenon detector with a 5 mg/cm² titanium foil window. Using the electroplated ⁹⁹Tc standard, the detection efficiency for ⁹⁹Tc beta emissions was determined to be 0.0635 counts/transformation. The background response rate in the vicinity of the hood was 13.1 counts/second. The response shown in Figure 3-2 is not indicative of a severe contamination problem, since the material was contained in a HEPA filtered fume hood. However, there was an increased frequency of higher than normal activity on smears outside of the hood, which could be attributed to the only source of high ⁹⁹Tc contamination, the hood.

The gel samples were collected from locations S1 through S15 shown in Figure 3-2. Samples S1 through S5 were immediately analyzed for contained activity by direct measurement using the Berthold LB122. Direct measurements were taken on the front and back surfaces of each gel sample and then the samples were dissolved in water for analysis by liquid scintillation counting. These measurements did not reveal information about the depth of activity within the gel because the gel thickness varied between samples. To obtain a more complete assessment of the activity contained within the gel, each of the samples was dissolved in deionized (DI) water ($\geq 18.0 \text{ M}\Omega \text{ cm}^{-1}$). Aliquots of 100 microliters each were taken from the top, middle, and bottom of the beaker containing the solution created by the dissolved gel and analyzed on a Perkin Elmer 2700TR Liquid Scintillation Counter (LSC) [100]. This provided for the determination of the total activity in each gel sample. The liquid scintillation counting results are summarized in Table 3-1.

The results of this analysis indicated that the activity in the gel was distributed evenly in the solution created by dissolving the gel in the DI water. This demonstrates that the activity was present as an even distribution on the surface and not attached particles with high ⁹⁹Tc content.

Location Designation	NET Sample Mass (g)	Activity of Gel Solution Sample 1 (top) (DPM)	Activity of Gel Solution Sample 2 (middle) (DPM)	Activity of Gel Solution Sample 3 (bottom) (DPM)	Average Activity of Gel Aliquots (DPM)	Total Activity in Gel Sample (DPM)	Total Activity in Gel Sample (Bq)
S1	6.02	673.2	691.8	731.8	698.9	559147	9319
S2	7.14	441.8	487.1	441.8	456.9	365520	6092
S3	5.92	397.6	391.5	325.2	371.4	297147	4952
S4	5.10	326.9	320.7	319.7	322.4	257947	4299
S5	8.12	364.4	204.4	246.2	271.7	217333	3622

Table 3-1. Estimate of ⁹⁹Tc activity in gel samples based on LSC analysis

The next planned evaluation was a more detailed smear survey and a direct reading survey to identify the extents of the loose surface activity and the fixed activity on the hood surfaces. From there, the plan was to spread the Decon Gel[™] 1101 onto the surface and allow it to dry. The gel would then be removed in grid squares and the gel squares analyzed by direct measurements using the Berthold LB-122, smears on the gel and on the surface where is was removed would be taken to determine if the gel encapsulated the activity and the fraction of the surface activity that was removed by the gel. Smears of the gel would be analyzed by a gas proportional counting system and the gel in solution would be analyzed using a LSC.

3.6 Data Collection

There data collected from the hood decontamination would answer many questions, including:

- What fraction of the activity on the hood surfaces was collectable using a smear?
- What fraction of the activity on the hood surfaces was removable with the decontamination gel?
- Was the activity collected in the decontamination gel encapsulated?
- What fraction of the activity collected in the decontamination gel was recoverable?

The importance of these questions is as follows:

• The fraction of activity collected by a smear has been a question among radiation protection specialists for many years. The quantification of activity on surfaces has been "estimated over the years by taking a smear of 100 cm², evaluating that smear and reporting the concentration of removable activity on that surface as the activity on the smear per 100 cm². This is incorrect; the actual surface activity is on the average 10 times more.

• The fraction of activity removable with the gel is important as a parameter of the gel that makes it useful. If you have some reason to believe that the gel will effectively remove the contamination, it is a useful decontamination product.

• The fraction of the activity collected that is encapsulated by the gel is important because it determines the desirability to use the gel as a sample collection mechanism. If the gel can collect a sample and hold that sample without the spread of contamination, this is a useful property for movement of the sample as if moving a sealed container.

• The fraction of activity collected by a gel that was recoverable is important because it may be desirable to use the gel as a collector of valuable material, and then recover the material from the gel. This may be by chemical or physical means. Only chemical means were investigated in this research.

In order to answer each of these questions, a plan was devised to do a more detailed evaluation of the activity on the hood surfaces. Direct measurements of accessible areas of the hood before and after application of the gel using the Berthold LB122 would be obtained. A smear survey of all areas of the hood before and after application of the gel would be conducted. Finally, the gel in each individual grid square would be collected for further analysis and possible recovery of the ⁹⁹Tc.

In following the plan, a detailed smear survey of the hood surfaces was conducted. The smears used were the common variety, sticky back, 5 cm diameter, paper smears [101]. The pressure exerted when taking each smear was similar over all of the surfaces surveyed. Figure 3-3 provides a distribution of the loose surface activity in the hood immediately after the initial survey. The back surfaces of the hood are used as examples to demonstrate the survey results and provide some perspective on the effectiveness of the decontamination methods. Although the activity on the back surfaces of the hood is higher than the other areas, activity on all hood surfaces showed similar activity distribution.

The detailed smear survey showed that each of the areas where the decontamination gel was used had less remaining removable activity than those which did not have the gel applied. This result was expected and showed that the gel was effective in removing loose surface activity from the hood surfaces. This survey also showed that there was significant activity in some areas of the hood and much less in others.

The areas of highest activity were toward the back of the hood, following the flow of air, but there were some areas on each side of the hood that were also high and indicate possible sprays or spills that were not affected by air flow.

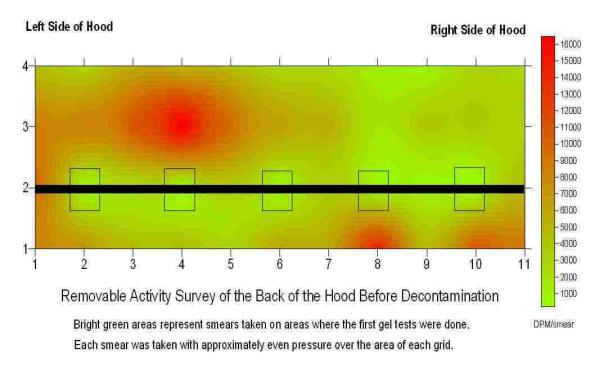


Figure 3-3. Distribution of Removable Activity on the Back Hood Surfaces

The most revealing measurements regarding the total activity in the hood were direct measurements of the hood surfaces. The total activity on the hood surfaces was found to be as much as 100 times more than the removable activity within a grid square. Figure 3-4 shows the approximate distribution of total surface activity concentration on the back surfaces of the hood before decontamination.

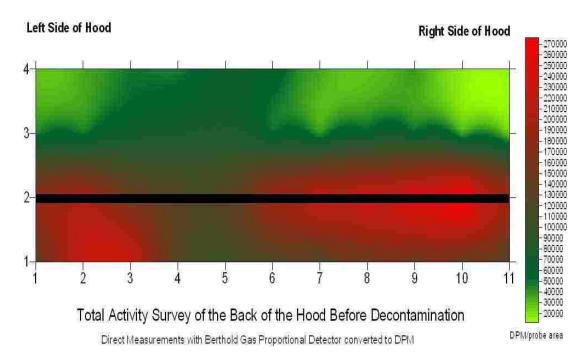


Figure 3-4. Distribution of Total Activity Before Decontamination

When compared to the direct measurement of the surfaces in the same grids as the smears were taken, the distribution of total activity was found to be significant. Figure 3-5 shows a histogram of the fraction of activity that was removed by the smears. The average fraction of activity removed by the smear was calculated to be 14.7% with a median value of 12.8%. Of the 123 readings and smears taken on smooth accessible surfaces, 89% of the smears taken removed between 5% and 30% of the surface activity.

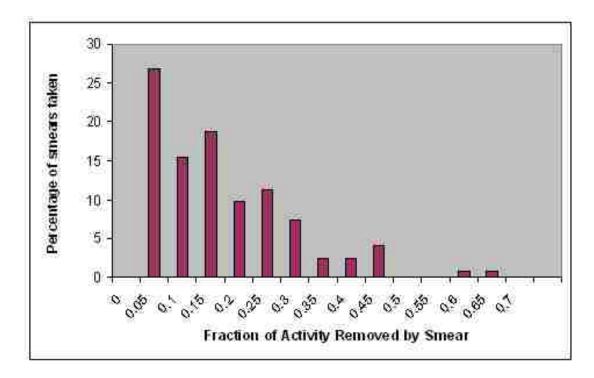


Figure 3-5. Histogram of Fraction of Surface Activity Removed by Smear

3.7 The Decontamination

3.7.1 Decontamination Set-Up

As indicated previously, the decontamination method used DeconGel[™] 1101, a commercially available decontamination product from Cellular Bioengineering, Inc. The gel that was purchased for use in the radiochemistry laboratories at UNLV came in a 20 liter bucket. The gel is blue, viscous, and appears to penetrate into pores and tight places, but is easily removed after it dries. This project is our third large scale use of the gel; the other two uses were for cleanup of a ²³³U spill and a ²³⁹Pu hot particle spill. The gel was effective in both cleanups.

The decontamination gel was spread onto the hood surfaces using a small putty knife. It stuck easily to most surfaces although it tended to drip from the hood ceiling, and collect in lumps from the vertical surfaces of the hood. Even though there was dripping from these surfaces, enough gel stayed on the surface to cause removal of radioactive contamination. The gel was allowed to dry for approximately 24 hours before removal. Figure 3-6 shows the hood surfaces after application of the gel.

After the gel dried onto the hood surfaces, it was cut at the grid lines and peeled from the surface. Each grid square was labeled and placed between two sheets of paper for later analysis. After removal of the



Figure 3-6. The ⁹⁹Technetium Research Hood coated with the gel.

gel, the grids were surveyed by direct frisk using the Berthold LB122 and by taking smears which were evaluated using a low background gas proportional counter.

3.7.2 Results of Decontamination

In the same fashion as before contamination removal, the survey of the hood surfaces was first done by direct measurement, then by smear to prevent further removal of activity prior to the evaluation of total activity. The results from the survey of removable activity are shown in Figure 3-7. The reduction in surface activity due to decontamination using the gel is significant with the removable activity almost completely collected by the gel.

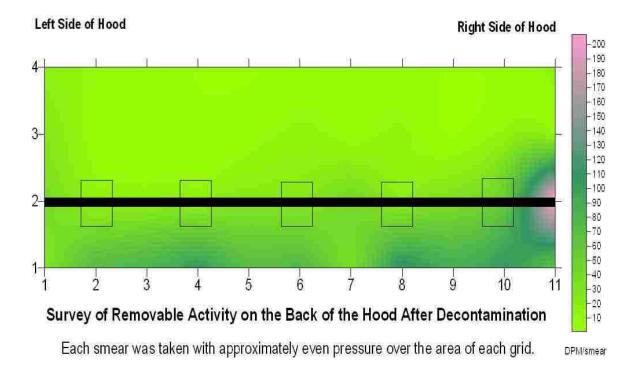


Figure 3-7. Distribution of Removable Activity After Decontamination

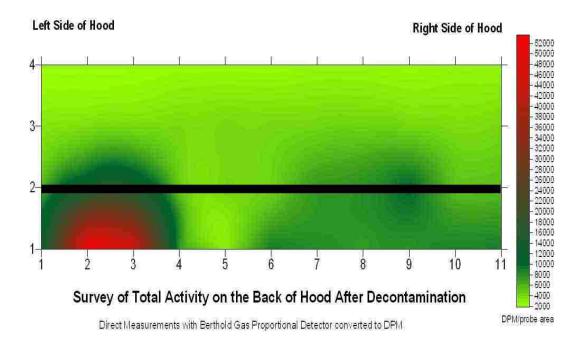


Figure 3-8. Distribution of Total Activity on the Back Surfaces of the Hood After Decontamination

In addition, Figure 3-8 provides a display of the direct measurement survey results. This survey indicates that much of the activity displayed in Figure 3-4 was removed but there is an area of the back of the hood where the activity was not removed. To further evaluate this small area, more decontamination gel was applied to the area. There was some further removal of activity by this second application, but it amounted to less than 5% of the total activity that remained. A third application of decontamination gel to this area did not cause any further reduction in the activity on that surface. This indicates that the activity in these grid squares was much more strongly attached. There was no further attempt to remove the activity.

3.7.3 Decontamination Factors

In radiation safety, the decontamination factor is a measure of the effectiveness of a decontamination process. It is the ratio of the original surface activity (before decontamination) to the remaining surface activity after decontamination: 1,000 and above is usually considered excellent; 100 to 1000 very good and 100 and below are possibly valuable depending on the situation. In this situation, we are interested in the decontamination of the total hood activity which includes elimination of the removable activity and reduction in the fixed activity which has a stronger attachment to the hood surfaces.

The decontamination factors observed for removable activity were determined from a comparison of smear results for the grid squares before the decontamination gel application to after gel removal. These decontamination factors averaged 450 with a range from 2 to more than 7000. The distribution of decontamination factors for removable activity over the hood surfaces is shown in Figure 3-9.

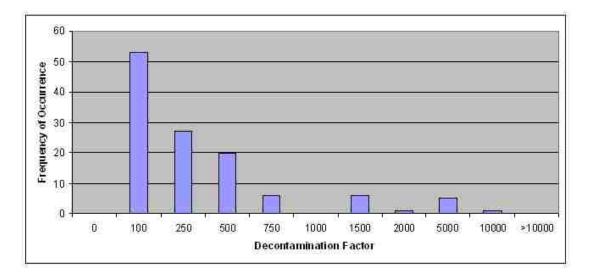


Figure 3-9. Decontamination Factor Distribution for Removable Activity

The decontamination factors for total activity on hood surfaces were determined by comparing the direct measurement of surface activity before the decontamination gel was applied to the direct measurement of surface activity after the decontamination gel was removed. The average decontamination factor was found to be more than 7,000, however, the range of decontamination factors for total activity varied from 2 to more than 100,000. The distribution of decontamination factors for total activity over the hood surfaces is shown in Figure 3-10.

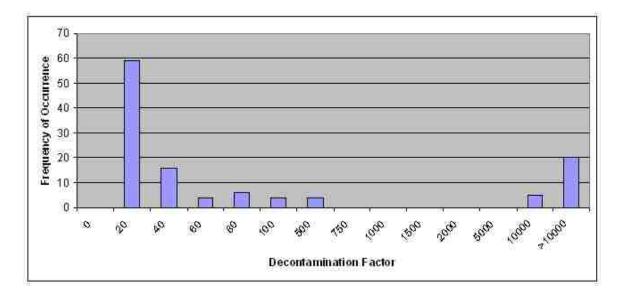


Figure 3-10. Decontamination Factor Distribution for Total Activity

From the survey data, the fraction of the total measured activity in the hood that was collected can be determined by subtracting the total activity remaining after decontamination gel removal from the total activity originally measured and dividing the result by the total activity originally measured.

$$F_{\rm R} = \frac{(A_{\rm T} - A_{\rm F})}{A_{\rm T}}$$

Equation 2-1

Where: F_R = The fraction of the total surface activity removed by the gel

 A_T = The total surface activity before the gel application

 A_F = The total surface activity after the gel removal

The value of the fraction of the total activity removed from the hood by the decontamination gel varied from 51.6% to 100% with a

median value of 95% and a mean value of 93.6%. See Table 3-2 for details of this evaluation.

The total accessible activity in the hood could be estimated from the hood surface area and the average surface activity concentration. Within the hood, there are 4 areas which contribute significantly to the total activity in the hood; the two walls, the base, and the back. Each grid square has an area of 322 cm². The hood contains two walls each with 16 grid squares, a base with 36 grid squares, and a back containing 44 grid squares. The remainder of the inside hood surfaces that was not included in the surveys contain the equivalent of approximately 40 grid squares. The total accessible surface area is approximately: $(16 * 2 + 44 + 36 + 40) * 322 \text{ cm}^2 = 48944 \text{ cm}^2$. With an average total surface activity concentration of 62,500 DPM/probe area, the total activity in the hood before decontamination is estimated to be $3x10^9$ DPM (5.1x10⁷ Bq).

Since approximately 90% of that activity was recovered by the decontamination gel, the collected activity should be approximately $(32 + 44 + 36)/(32 + 44 + 36 + 40) * 0.9 * 5.1x10^7$ Bq = $3.38x10^7$ Bq. The specific activity of ⁹⁹Tc is $6.27x10^8$ Bq/gram; therefore, this represents approximately 53 mg of ⁹⁹Tc.

In order to determine if the ⁹⁹Tc collected by the decontamination gel was encapsulated, smears were taken on the side of the hardened gel that contacted the contaminated surface. This result would be valuable in considering collection of samples of activity from the site of a Radiological Dispersion Device (RDD) [102] explosion. A first responder team could place the decontamination gel in a survey fashion at locations identified as areas of concern by portable instrument. The decontamination gel could be collected after drying without concern for contamination spread and the samples taken to a laboratory for analysis.

Since the back of the hood showed the most significant surface activity concentration, the data from that area will also be used to demonstrate the findings for this evaluation. Each of the gel samples removed from the back was covered with paper and moved into a low background area. The background response measured in this area was 12.6 cps. The response from the decontamination gel was measured with the Berthold LB122 at both sides of each sample. Each smear was taken over the entire surface of the dried gel that contacted the ⁹⁹Tc contaminated surface of the hood and counted on a Berthold low level gas proportional counter. The maximum activity of a smear taken on the dried decontamination gel surface was 2.4 Bq. The maximum fraction removal of activity from a decontamination gel surface after it was removed from the contaminated surface was 2.21x10⁻³. The average fraction of activity removed from the decontamination gel was 3.3x10⁻⁴ with a median fraction of 1.3x10⁻⁴. Some samples indicated that there was no activity available for transfer from the gel. See Table 3-3 for details of this evaluation.

Location	Activity of Gel by Direct	Count Rate on Smears of Gel Side Facing the	Activity in Smear of the Gel Side Facing the	Minimum	Fraction of Activity in Gel Available for
Designation	Response (DPM)	Contamination (CPM)	Contamination (DPM)	Detectable Activity (DPM)	Transfer
0	123978	2.950	(DFM) 7		
B1 B2	123978	2.950	90	2 2	0.00006 0.00052
B2 B3	186340	13.200	51	2	0.00032
B3 B4	114246	5.050	16	2	0.00028
B4 B5	95537	8.370	31	2	0.00032
B5 B6	111506	7.860	28	2	0.00032
B0 B7	112356	3.170	8	2	0.00023
-	137584	11.200	43	2	0.00007
B8 B9			43	2	
	137206	3.020	69	2	0.00006
B10	135411	17.300			0.00051
B11	110466	8.770	32 35	2	0.00029
B12	180671	9.450		2	0.00020
B13	192576	3.310	9	2	0.00005
B14	135033	3.090	8	2	0.00006
B15	122183	1.900	3	2	0.00002
B16	125395	4.890	16	2	0.00012
B17	196167	2.460	5	2	0.00003
B18	221490	7.870	28	2	0.00013
B19	227631	3.260	9	2	0.00004
B20	242372	6.570	23	2	0.00009
B21	278466	0.950	0	2	0.00000
B22	192198	5.950	20	2	0.00011
B23	39411	1.830	2	2	0.00006
B24	41017	2.090	4	2	0.00009
B25	61616	1.140	0	2	0.00000
B26	68041	2.080	4	2	0.00005
B27	72576	1.820	2	2	0.00003
B28	44324	2.580	6	2	0.00013
B29	31946	2.060	3	2	0.00011
B30	49805	3.700	10	2	0.00021
B31	46403	5.880	20	2	0.00043
B32	27694	9.520	36	2	0.00128
B33	17584	7.800	28	2	0.00160
B34	26466	9.880	37	2	0.00140
B35	33080	3.720	11	2	0.00032
B36	47254	7.640	27	2	0.00058
B37	44230	16.800	67	2	0.00151
B38	65112	34.700	144	2	0.00221
B39	60482	7.030	25	2	0.00041
B40	33647	6.730	24	2	0.00070
B41	26561	1.700	2	2	0.00007
B42	37616	1.550	0	2	0.00000
B43	14939	1.390	0	2	0.00000
B44	13143	0.638	0	2	0.00000

Table 3-2. Fraction of Activity in the Gel Available for Transfer

One attractive aspect of the DeconGel[™] 1101 is its ability to collect materials that would otherwise go to disposal, and recover them for purification and conversion to usable forms for reuse in future experiments. Members of the radiochemistry community can utilize these compounds and this would also reduce costs for radioactive waste disposal.

3.7.4 Analysis of Activity in the Gel

Liquid Scintillation Counting (LSC) provides a simple means to determine the activity of a sample of the decontamination gel in solution. For samples that are expected to provide a substantial response, the volume of solution required is a very small fraction of the volume of LSC cocktail required for the analysis. This minimizes the self-absorption of the emissions from the gel, and thus maximizes the detection efficiency of those emissions. Another desirable feature of LSC with emission analysis capability is the discrimination of beta energy. For example, the beta energy spectrum of ⁹⁹Tc has a maximum energy of 293.7 keV with an average energy of 84.6 keV with a 100% beta yield. The beta emission spectra from [103] is shown in Figure 3-11. Using this information and the desire to minimize conflicts with other low energy beta emitters in the liquid scintillation counter, the regions of interest on the liquid scintillation counter were set up as channel A: 0-50 keV, channel B: 50-300 keV, and channel C: 300 – 2000 keV.

In order to improve efficiency, after it was found that there were no low energy interferences, channels A and B could be summed. For ⁹⁹Tc, the expected detection efficiency is 95% to 100% (0.95 to 1 count per transformation). The gel is proven to be useful for decontamination of contaminated surfaces after it is reconstituted with water or ethanol. In the next section experiments are done to determine if radioactive contamination encapsulated in the gel can be recovered.

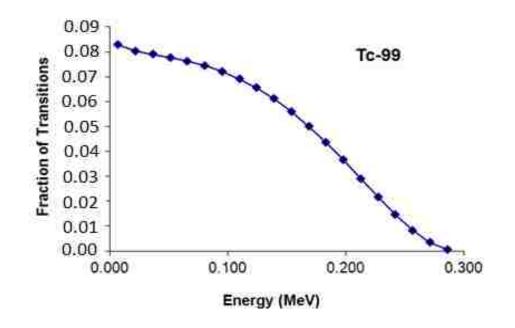


Figure 3-11. Energy and Yield for Beta Emissions from ⁹⁹Tc

3.8 Recovery of Tc from Decontamination Gel

3.8.1 Introduction

The hood decontamination provided an opportunity to evaluate radionuclide recovery from the gel material. Analysis of the gel solution created from the dissolution of the gel in DI water was completed by LSC. The gel has proven to be useful for decontamination of surfaces and after it is reconstituted with water or ethanol it can be reused. In this section, experiments are described that will show radioactive contamination encapsulated in the gel can be recovered.

The tests for materials recovery from decontamination gel have shown that it can be made very dilute or can be broken down into a fluid aqueous solution that is filterable. Attempts at destroying the viscous nature of the gel were made using four types of concentrated acid: nitric, acetic, sulfuric, and hydrochloric. The selected acids may provide for future considerations such as evaluation of acidic gel solutions for corrosive decontamination on metal surfaces. The idea of this set of experiments was primarily to evaluate whether the gel could be broken down to the point where it could be filtered or otherwise treated and recovered for reuse.

3.8.2 Experimental Set-up

In a very basic experiment with the gel, 10 mL of water was added to one gram of the gel, an attempt was made to force the water through a 0.45 micron filter without success. The applied pressure allowed only a drop of liquid to pass though the filter. A second attempt was made with a dilution of one gram of gel in 50 mL of water; a 5 mL aliquot of that sample could be filtered with high pressure on a syringe/filter assembly.

Based on these two simple tests, it seemed that a breakdown of the gel is necessary for recovery of particles in the gel matrix. From the four attempts at breaking down the gel, the method that appeared to have the most reasonable degree of success in a short time was determined to be the nitric acid dilution. The following sections provide more detail.

The gel was first put into water to make it liquid before the addition of the acids. A grid square consisting of a variable mass of the gel in a rectangle the size of the base of a Berthold LB-22 gas proportional detector was dissolved in 100 mL of deionized water. Twelve containers were prepared for each acid test; each container had 5 mL of the gel from a sample collected in decontamination of the fume hood, with known activity. To each sample, increasing volumes of acid were added to allow observation of the effects of the acid on otherwise similar solutions of gel and water with an entrained contaminant. A control sample of the gel for each dilution was also saved.

3.8.3 Experiments with HCl

The hydrochloric acid (HCl) stock was concentrated HCl manufactured by Baker Analyzed, lot number E45048. This acid is a strong mineral acid with uses in cleaning metal surfaces and in production of gelatin. Perhaps its use in this set of experiments would lead to a stronger gel that had the ability to remove some depth of metal

surface in its decontamination of a metal surface. Increasing volume of HCl, varying from 0.1 mL to 1 mL was added to the 5 mL of gel diluted in deionized water. The viscosity of the gel at low HCl concentration was the greatest with only a slightly more fluid indication at the highest HCl concentration. In these experiments, the viscosity was observed as the ability of the gel and acid mixture to flow. A free flowing mixture would be considered less viscous than a slow moving gel. As can be observed in Figure 3-12, the color of the gel changed from blue (control) to light green, to translucent yellow with increased concentration of HCl.



Figure 3-12. Hydrogel Polymer with Varying HCl (The control gel is on the left, acid concentration increases toward the right)

The HCl did not reduce the viscosity of the gel. This was determined by tipping the containers and watching the flow of the gel on the side of the tube. The color change indicates that there was a variation in the compound and was not investigated further. The HCl molar concentration is displayed in Figure 3-13 in a graphical form for each of the containers of gel. This display format was continued for each of the gel and acid mixtures.

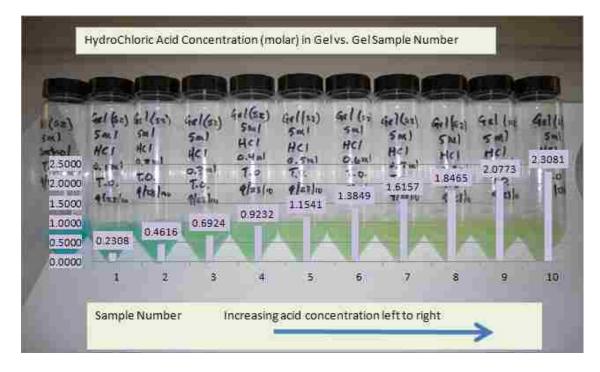


Figure 3-13. Hydrogel Polymer with Varying HCl – The molar concentration of HCl is displayed.

3.8.4 Experiments with H₂SO₄

The sulfuric acid stock was concentrated H_2SO_4 manufactured by Baker Analyzed, lot number G44434. Similar to hydrochloric acid, sulfuric acid is a strong mineral acid, but it is not used in gelatin production. However, its use in corrosive decontamination of metal surfaces may be of interest. Increasing volume of H₂SO₄ varying from 0.1 mL to 1 mL was added to the 5 mL of gel diluted in deionized water. Figure 3-14 displays the color change that occurred with increasing acid concentration from the control sample with no added acid on the left to 1.9 M H₂SO₄ in the far right sample. The gel with the lowest H₂SO₄ concentration flowed more freely than the gel with the highest H₂SO₄. The color change was from blue to green then light green and a more solid yellow at the highest acid concentration than with the addition of HCl. Once again, the color change indicates a change in the compound; the similar viscosity indicates that the gel is still intact.

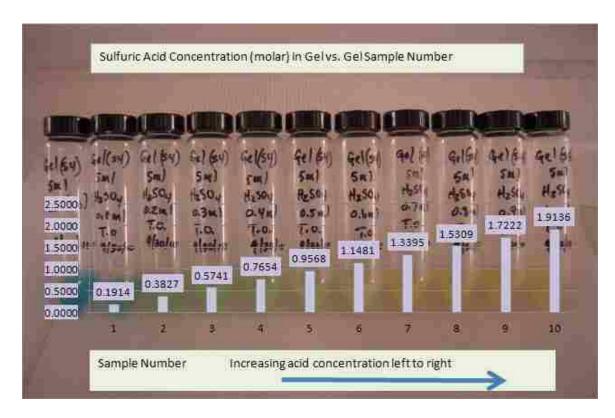


Figure 3-14. Hydrogel Polymer with Varying H_2SO_4 (The control gel is on the left, acid concentration increases toward the right)

3.8.5 Experiments with CH₃COOH

The acetic acid stock was concentrated CH₃COOH from Baker Analyzed, lot number J08N52. The selection of this carboxylic acid was based on its use in production of vinyl acetate and wood glue. It is commonly available in nature and could be an easy and inexpensive method to break down this polymer. The drawback in this acid is its strong corrosive effect on skin. Increasing volume of CH₃COOH varying from 0.1 mL to 1 mL was added to the 5 mL of gel diluted in deionized water. The resulting molarity of the acid in the gel ranged from 0.2795 M to 2.7949 M. The viscosity of the gel did not change with the increased acid concentration as determined by visual observation of the movement of the gel-acid mixture as the tubes were tipped. The color also did not change, as is displayed in Figure 3-15. Since there was no color change or viscosity change with the addition of the acetic acid, it appears that there may have been no change to the hydrogel compound. The rationale for this, as with the reason for an observed change with the addition of the HCl and H_2SO_4 , was not investigated.

3 0000 2 5000 2 5000 1 5000 1 0000 0 5000	11(12) 50 101:00 101:00 10:	fil (53) Smi Retic 0,2-41 T-0 Altofic 0,5554	Sm/ hetic u.Smi T.O tijaojio	(eel (es) Sml Acetic Ac	Gel (15) 5ml Acetic 0.5ml 1:0 4/30/0 13975	Str/Go) Sm 1 Acertic d. Gml T.a - 1.6769	Gal(5) Sm/ Acatic 1.9564 T****	Gel (G) Sml Acetic 0.2 ml 2 2359 (*20)	Gel/(9) 5m) Audic 25154 9 0; 9 0;	Gal 5M Acc 2.794
0.0000	1	2	3	4	5	6	7	8	9	10

Figure 3-15. Hydrogel Polymer with Varying CH_3COOH (The control gel is on the left, acid concentration increases toward the right)

3.8.6 Experiments with HNO₃

The addition of nitric acid in the same fashion as the other acids showed a change of the gel at the higher acid concentration. This corrosive mineral acid is a strong oxidizing agent and has the highest potential as a useful method to break the polymer chains. The change caused observed in the gel and nitric acid mixture was a free flowing liquid. The concentrated nitric acid was prepared by Seastar Chemicals, lot number 1207060. Figure 3-16 displays the samples to which nitric acid was added, increasing acid concentration from left to right. The nitric acid appeared to change the gel to a non-viscous and nearly colorless liquid in less than 30 minutes.

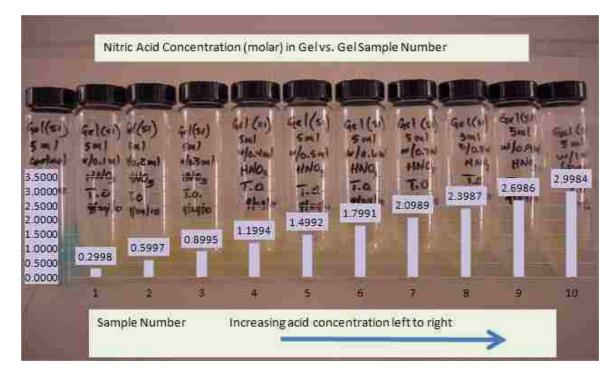


Figure 3-16. Hydrogel Polymer with Varying HNO_3 (The control gel is on the left, acid concentration increases toward the right)

In the case of each acid except the nitric and the acetic, the viscosity became greater. However, only in the case of the addition of concentrated nitric acid did the gel completely breakdown More experimentation was needed to verify that the dissolved gel solution could be manipulated to remove the contaminants. The influence of acid concentration or neutralizing the solution and gel reformation needs to be examined. The color change in the gel with the addition of HNO₃ is shown in figure 3-17.



Figure 3-17. Hydrogel Polymer with Varying HNO_3 (The control gel is on the left, acid concentration increases toward the right) – no scale.

A 10 mL sample of deionized water was added to the acid gel mixture. It demonstrated that the gel was indeed broken down by the addition of the HNO₃ and did not have the original viscosity of the decontamination gel. A 5 mL sample of the liquid was then removed to a syringe and squeezed through a 0.45 micron filter. The gel and nitric acid solution, the diluted gel/nitric acid solution, and the diluted/filtered gel/nitric acid solutions were all sampled and analyzed by LSC. Table 3-4 shows the results from the LSC analyses and indicates that the unfiltered and filtered solutions demonstrate that the ⁹⁹Tc is in solution.

The differences in activity between the filtered and unfiltered samples are not significant. From this information, it is reasonable to believe that extraction techniques typically used to remove these radionuclides from nitric acid would be appropriate to remove them from the acid solution that remained after destruction of the decontamination gel.

3.8.7 Discussion of Recovery Considerations

As shown in section 3.8.6, the nitric acid solutions appeared more fluid and could be pushed through a 0.45 micron filter. In the case of the other acids, the gel either did not change in viscosity or became more viscous. The viscosity was determined by a qualitative observation of the rate of movement of the gel when the test tube holding the gel/acid mixture was tipped to a 60° angle from the bench-top.

From this set of experiments, it was found that nitric acid is useful in changing the polymer to allow filtration. A nitric acid solution in the gel of 1.5M is the lowest concentration at which the liquid is clear. The final step is re-concentration of the ⁹⁹Tc in the solution to see if the polymer is reconstituted. A further experiment could be to determine if the ⁹⁹Tc can be removed from the solution by precipitation with tetrabutylammonium chloride. This was successfully attempted in removal of ⁹⁹Tc from one of the residue recovery experiments. Table 3-4 provides data from the attempt to filter the ⁹⁹Tc from the mixture. This shows that the ⁹⁹Tc was in solution and filtration was not effective at removal.

Table 3-3. Analysis of ⁹⁹Tc in Unfiltered and Filtered Gel/Acid by LSC

The original sample of gel was from decontamination of the			
MSM-173 hood. The solution			Acid/Gel
was approximately 10 grams of		Acid/Gel	Diluted &
gel in 50 mL of deionized water.	Acid/Gel	Diluted	Filtered
Sample Description	Solution Activity Co	oncentration (dpm/0.1 mL)
Tc Gel 0.1 mL Concentrated			
$HNO_3 + H_2O$	315	173	188
Tc Gel 0.2 mL Concentrated			
$HNO_3 + H_2O$	342	191	168
Tc Gel 0.3 mL Concentrated			
$HNO_3 + H_2O$	340	146	142
Tc Gel 0.4 mL Concentrated			
$HNO_3 + H_2O$	376	161	146
Tc Gel 0.5 mL Concentrated			
HNO ₃ + H ₂ O	372	120	128
Tc Gel 0.6 mL Concentrated			
$HNO_3 + H_2O$	358	124	123
Tc Gel 0.7 mL Concentrated			
$HNO_3 + H_2O$	373	125	124
Tc Gel 0.8 mL Concentrated			
$HNO_3 + H_2O$	399	137	135
Tc Gel 0.9 mL Concentrated			
$HNO_3 + H_2O$	386	138	124
Tc Gel 1.0 mL Concentrated			
$HNO_3 + H_2O$	384	145	141

3.9 Considerations for Reuse of the Decontamination Gel

The Material Safety Data Sheet for the gel provides indications that the primary hazardous constituents of the material are ethanol and sodium hydroxide. The gel is relatively benign when compared with many chemicals in the radiochemistry laboratories but handling the gel does require hand protection to prevent drying, cracking, and defatting of the skin. The gel also has a flammability property which may be enhanced when reconstituted with pure ethanol or acetone. The gel dries to a flexible solid, and envelops contaminants to remove them from surfaces. There may or may not be a chemical reaction with the contaminant, depending on its chemical characteristics and the gel is not selective in removal of materials from surfaces. The material used in the decontamination experiments is a hydrogel polymer. Hydrogel polymers can absorb large amounts of water without dissolving due to physical or chemical cross-linkage of the hydrophilic polymer chains [104].

The developers of the decontamination gel indicated that they had not tested it for reuse [85], but based on the MSDS a reconstitution with ethanol or water should allow reuse. Some simple initial experiments by addition of arbitrary amounts of water, ethanol and acetone, and reapplication of this mixture to a contaminated surface in the ⁹⁹Tc hood demonstrated that the gel seems to be reusable after reconstitution. Furthermore, the more volatile the additive, the quicker the gel would dry. This aspect of the gel was not pursued and is certainly an area where future work should be done.

Gels are generally affected by temperature and are observed to 'melt' as the temperature increases and they 'gel' or harden as the temperature decreases. Furthermore, they seem to be incompressible but squeeze through spaces that are large enough for their molecules to pass. Hydrophilic gels will freeze but have a lower freezing point than water. In some cases, the gel may provide several uses for decontamination depending on the mass of material to be collected, the contaminants that may affect the performance of the gel, and the dose rate from radionuclides collected. The gel that was collected in the decontamination of the chemical fume hood contaminated with higher levels of ⁹⁹Tc was not degraded nor did its ability to collect more activity change noticeably after use. It is however possible, that there is a point at which the gel is no longer effective in collection of contamination, but that point was not reached during these experiments. This aspect of the gel is a subject for future work.

There may be a time when the gel could be used many times after a period of decay in storage. For example, if the gel was used for decontamination of surfaces that are contaminated with a short lived radionuclide, the material will be an extremely small mass and will not likely affect the performance of the gel for reuse. Consider ¹²⁵I with a half-life of 60 days, a spill containing only 1 microgram of ¹²⁵I will have an associated activity of 643 MBq. If the surface was clean except for the ¹²⁵I, the gel could be used to decontaminate the surface, stored as a dry solid mass for a time long compared to the half-life, then reconstituted with water and used again for decontamination. Consider that the mass of ¹³¹I in a 30 GBq dose can be calculated from the definition of activity:

 $\mathbf{A} \equiv \boldsymbol{\lambda} \cdot \mathbf{N}$

Equation 2-2

Where: A = activity

 λ = the decay constant for $^{131}\!\mathrm{I}$

N = the number of 131 I atoms

For this situation:

 $30 \text{ GBq} = \ln(2)/(8.05 \text{ days x } 24 \text{ hrs./day x } 3600 \text{ s/h}) \text{ x N}$

N = 3.0×10^{10} Bq / 9.97×10^{-7} s-1 = 3×10^{16} atoms

Once the number of atoms is determined, the following relationship allows determination of the mass of the radionuclide.

$$m = \frac{N}{N_{A} \cdot M(\frac{131}{53}I)}$$

Equation 2-3

Where: m = mass (grams)

N = atoms of 131 I

 N_A = Avogadro's Number (6.02x10²³ atoms/gAw)

 $M^{131}I = 130.9 \text{ g/gAw}$

 $m = 3x10^{16} \text{ atoms}/6.02x10^{23} \text{ atoms}/g-mole * 130.9 g/g-mole}$

= 6.5x10⁻⁶ g

 $m = 6.5 \ \mu g$

As you can see, this mass would be a very small fraction ($<1x10^{-6}$) of the mass of the material spread onto a surface for decontamination which may be on the order of grams per 100 cm². The most significant contaminant that the gel would collect would likely be surface dirt from the area decontaminated. Once the radionuclide collected has decayed, the gel will likely contain primarily non-radioactive contaminants (dirt, grease, etc.) that were on the surface with the radionuclide collected. The gel could be surveyed and disposed of as clean waste, or reconstituted by adding water and reused to collect more contamination. The number of times that the gel could be reused would then be extended by ensuring that the area contaminated for a procedure is cleaned prior to use of radioactive materials on that surface. This type of use in a medical setting where surfaces are required to be clean and short lived radionuclides are common may be a good match. Reuse of the gel for decontamination is most obviously best for use with radionuclides that are easily collected, are typical contaminants around a biology laboratory or medical facility, and have very short half-lives. The radionuclides ¹³¹I, ³²P, and ³⁵S might be ideal candidates for decontamination and reuse of the gel. As with any chemical cleaner, some simple experiments to determine that the compound will be compatible with and collected by the gel should be done.

3.10 Radiation Attenuation by the Gel

A desirable property for use of the gel as a sampling tool is the ability to encapsulate the material that is sampled and not be affected by its emissions. This section provides a discussion of the efficiency with which the decontamination gel is able to attenuate the beta emissions from ⁹⁹Tc. This attenuation would be associated with the constituents of the gel and its mass density thickness. For ⁹⁹Tc, the only published rule that can be associated with the range of beta emissions of the energy emitted is published as equation 6.15 in [105]. This rule applies to all beta emissions with energy greater than 10 keV and less than 2.5 MeV. Therefore, for ⁹⁹Tc emissions, the rule can be stated as follows:

R(beta) mg/cm² = 412 x E^{(1.27-0.0954 * ln(E))}

Equation 2-4

Where:

R is the range of the beta particle in the material in units of mg/cm^2 , and E is the beta particle kinetic energy in MeV.

Using equation 2-4 with the maximum energy of the beta emissions from ⁹⁹Tc, 293.7 keV, the maximum range of beta emissions from ⁹⁹Tc is 75.8 mg/cm². The gel has a density similar to water, so the maximum linear range of beta emissions from ⁹⁹Tc in the gel would be:

 $75.8 \text{ mg/cm}^2 \div 1000 \text{ mg/cm}^3 = 0.0758 \text{ cm or } 0.758 \text{ mm}$ Therefore, a thickness of 1 mm spread on a surface contaminated with ⁹⁹Tc will attenuate all ⁹⁹Tc beta emissions from that surface. An

approximate mass absorption coefficient can be identified for any beta energy [106] in unit density material by the equation:

HVL $(cm) = 0.041 \cdot E^{1.14}$

Equation 2-5

From the Half Value Layer (HVL) for beta particles with kinetic energy E in MeV determined in equation 2-5, the approximate attenuation coefficient in unit density material is given by Equation 2-6.

$$\mu = \frac{17}{E^{1.14}}$$
 for E > (0.1 MeV < E < 4 MeV)

Equation 2-6

Where:

 μ is the linear attenuation coefficient for the beta particle in the material in units of cm⁻¹, and E the beta particle kinetic energy in MeV. Using this relationship, the attenuation coefficient, μ , for the ⁹⁹Tc maximum beta energy emission is 68.7 cm⁻¹. Using this coefficient, the fraction of the emissions from a ⁹⁹Tc contaminated surface that would penetrate 1 mm of the gel covering that surface would be:

I/Io = $e^{-\mu_x}$ = $e^{(-68.7 / \text{cm} *.1 \text{ cm})}$ = 0.001 or 0.1%, where I/Io is the unattenuated fraction of the beta flux incident on the gel.

3.11 Conclusions

This chapter discussed removal of ⁹⁹Tc from the surfaces of a fume hood and subsequently from the gel used. The details of the decontamination provided collection efficiency and breakdown of the gel was determined to be possible with concentrated nitric acid. Decontamination of equipment and facilities is always a concern from many viewpoints. The decontamination should be non-destructive in most cases, the cost should be reasonable and not prevent future work, and the operation must be safe for all participants and future workers. This fume hood decontamination produced acceptable results, the cost was reasonable, there was no detectable exposure of personnel associated with the decontamination, and the degree of hazard for work in this fume hood has been reduced for future workers.

The fraction of surface activity removed with smears was found to average between 10% and 15% for most areas surveyed. The distribution of smear results shows that the range of activity removal for smears was between 5% and 20%. This result confirms the idea that smears remove approximately 10% of the surface activity.

The ability to efficiently decontaminate surfaces without destroying or disabling functions that they perform is a goal of any decontamination operation. In a fume hood that will be used in the future, it is desirable to bring it back into operation as soon as possible. To facilitate this desire, a high decontamination factor is needed. In this case, the decontamination factor was the highest for fixed activity removal because

essentially all of the activity was removed from some of the grid squares. An important consideration here is that more than 90% of the activity on the hood surfaces that was removed by the decontamination gel in one application.

The total activity of ⁹⁹Tc on the accessible surfaces of the hood was estimated to be 3.38x10⁷ Bq (approximately 53 mg of ⁹⁹Tc). The majority of activity in the hood is considered to be under the back baffles, where oils and dusts provide an area for much greater collection of activity because they do not get cleaned. Currently, there is no estimate of activity in these areas.

The smear survey of the gel squares provided reasonable evidence that the decontamination gel encapsulated the majority of the ⁹⁹Tc. The fraction of activity removable from the gel by dry smear was small, averaging less than 3.3x10⁻⁴. This supports the idea that the gel could be used as a sample collection mechanism for materials that may have come from a radiological dispersal device. The samples should be collected in a container such as a bag, bottle, or box. In addition, the material collected in this evaluation did not cause any degradation of the decontamination gel as it was drying. However, some areas where sampling is desired may be wet, may include oils, alcohols or other liquids that would prevent the decontamination gel from drying. Some variations of this type of decontamination gel that would allow it to collect materials without regard for the solubility of the gel may be appropriate for those areas. Results from this study could be used to address matrix issues in a future investigation of the decontamination gel in various environments.

The decontamination gel can be broken down with concentrated nitric acid. A concentration greater than 1 M will remove the cross linking and cause the gel to become a free flowing liquid. This result is valuable in the removal of the encapsulated material from the gel. It is also important to note that for the case of technetium, filtration did not remove the Tc from the liquid. A chemical method such as the precipitation of Tc with tetrabutylammonium would be a reasonable method to remove the Tc from solution. The gel was not broken down with other acids, although this may also be a useful result as perhaps those gels may provide a corrosive decontaminant useful for fixed surface contamination on metal.

3.12 Implementation

A program for recovery of radionuclides from decontamination materials requires consideration of the emission characteristics of the radionuclides to be recovered. In this section consideration is given to implementing a program for use of a decontamination gel for cleanup and recovery of radioactive materials.

3.12.1 Abstract

Decontamination materials that are not hazardous, shield soft emissions, are efficient and transportable, and can be reused are desirable in radiochemistry. A decontamination gel was tested and

results reveal an available gel that could be specialized to allow entrainment of a caustic solution to make the gel more effective for removal of fixed contamination on a solid surface. In addition, the gel may provide a means to remove surface activity from porous surfaces with minimal degradation of the gel, providing reusability.

3.12.2 Discussion

Hydrophilic polymers useful have been shown to be decontamination media and useful for collection of surface samples. A sample of a surface contaminated with radionuclides may be taken by simply applying the gel, allowing it to dry, and collecting the solid sample. The material collected may be a very small fraction of the surface, but analysis by gamma, x-ray fluorescence, of x-ray diffraction spectroscopy may provide a non-destructive analysis of the surface. Further analysis of the material contained within the gel may be made by breaking down the hydrophilic polymer and analyzing the solution by atomic emission or mass spectroscopy.

Collection of the surface samples for containment and ultimate analysis is useful. Breakdown of the polymer through the action of an acidic or basic solution or by thermal destruction may provide a means to recover the material collected. Thus the purpose of the gel as a decontamination agent may be enhanced by using it as a sample collection mechanism and a recovery method. Non-destructive analysis techniques such as gamma spectroscopy, x-ray fluorescence or x-ray

diffraction could be used as needed to evaluate a sample within the matrix of a polymer with known composition.

There are several important considerations regarding use of a decontamination solution that are important to recovery of a useful radioactive compound for analysis. These considerations are:

- The decontamination solution should hold the radioactive compound without bonding with the compound.
- The solution should be removable from the activity without production of an unacceptable amount of waste.
- The removal of the activity from the solution should allow the activity to exist in a usable form for future research.

For a sample collection mechanism, the action taken to remove the activity from the decontamination solution should:

- ✓ Minimize the creation of radioactive waste while maximizing the radionuclide recovery.
- ✓ Allow for a reasonably rapid collection and analysis in relation to the half-life of the radionuclide.
- ✓ Consider techniques for removal of the radionuclide(s) from the solution if needed.
- ✓ Maintain the radionuclide(s) in an unchanging matrix to ensure consistency in analysis.

The neutral nature of the gel is valuable to prevent damage to surfaces and encapsulate activity but removal of activity fixed on surfaces may require consideration of the following:

- ✓ Adding a caustic agent to the gel may make it capable of removing the surface bound activity by breaking chemical bonds that are holding the activity or chemically etching the surface to take some of the surface with the activity.
- ✓ The gel may be re-hydrated with solutions that allow for faster drying, weaker bonding with the activity, or less attachment to a surface.

The re-hydratable nature of the gel used for decontamination allows recovery of activity with several methods using the following concepts:

- The gel polymer could be broken down chemically or thermally.
 Then chemical methods may be useful in removal of the activity.
- The radioactive component may be removed chemically or mechanically (centrifugation, magnetic attraction, or through bonding with another compound that allows other removal methods).

3.12.2 Summary

While the decontamination gel used in decontamination of a highly contaminated fume hood has properties that are attractive for activity collection, some changes may enhance its ability to remove fixed contamination from surfaces or improve the desired properties for use as

a sample collector. The gel used, Decon Gel 1101 from CBI Polymers Inc. provides an off the shelf solution to simple decontamination. There are many aspects of a radiochemistry program that may benefit from this gel, the research to explore these properties may provide for more useful products.

CHAPTER 4

RADIONUCLIDE RECOVERY FROM EXPERIMENT RESIDUE

4.0 Hypothesis

Mixed waste production can be eliminated and radioactive materials recovered for reuse with properly planned experiments and appropriate documentation of wastes created. Sustainability of a Radiochemistry Program can be enhanced by reducing the loss of resources to dispose of waste and recovery of radionuclides from experiment residues.

4.0.1 Literature Review

In this chapter the removal of useful radionuclides from experimental residue to allow reuse is presented as a novel option. Reuse of radionuclides not previously used is a reasonable action because these materials are expensive to use, manage, and dispose [107]. Recovery for reuse is accomplished on a large scale; reprocessing of nuclear fuel [108] [109] and recovery of nuclear fuel from surplus nuclear weapons [110] are two examples. A key feature of reuse of radioactive materials is the savings realized by avoiding disposal of the residue as mixed waste [111]. This is allowed because the residue is in a transition between its original condition and a reusable product, since it is not ready for disposal, the regulation of a radioactive hazardous waste [112] is avoided. This concept is independent of radionuclide, hazardous components, or the expected chemistry to recover materials. As a component of any experiment, the chemistry to return the waste products to a hazard free form should be done. The recovery strategies are commonly used in experiments for separations of radionuclides from mixtures or environmental media. A resource for common extraction techniques for different radionuclides is the V.G. Khlopin Radium Institute [113].

4.1 Constituents of Waste Products

Since the beginning of radiochemistry experiments at UNLV in 2004, many liquid waste products have been received from experiments in the UNLV radiochemistry laboratories. The Laboratory Support Group [114] for the radiochemistry program collects these wastes in order to provide consolidation of like products, recovery of desirable materials, and proper disposal of hazardous wastes in accordance with safety requirements. These products, in general, have a documented content from hazardous waste logs [115] with the following chemicals: TBP (tributyl phosphate), THF (Tetra-hydro Furan), AHA (Aceto Hydroxamic Acid), ethanol, methanol, oxalic acid, sodium hydroxide, ammonium hydroxide, nitric acid, bromine, vacuum pump oil, and radionuclides (²³⁸U, ²³⁹Pu, ²⁴⁴Cm, and ⁹⁹Tc). There were no cases where documentation provided quantitative analysis of chemical content or activity of radionuclides.

Products from past experiments have not been accurately identified and documented due in part on the researcher's desire to focus on the residues of the future and abandon those of experiments from the past. While this allows for the creation of more residues and valued

compounds through more research, a short time spent documenting the content of all products would provide for easier and less costly evaluations in establishing the value of recovery. This documentation would also assist the identification of chemical processes that could be used to return the products to a useable form. Although the primary radionuclides and the chemicals that are used for compound synthesis may be known, the mechanisms to recover radioactive materials from some of these residues are complex, time consuming, and could create a more hazardous situation. The procedure for evaluation of residues derived from this work will identify a method for documentation of the potential for radionuclide recovery.

Radionuclides that are a component part of a solid mass containing metals, soils, and other difficult to dissolve materials, may not be reasonable candidates for radionuclide recovery. This statement is based on the concept that the radioactive material is a very small mass in a mixture or attached to a much larger mass. In this situation, these items might be quickly evaluated and the decision made to contain the hazard and dispose of the material as radioactive waste.

Based on the information available through discussions with the researchers [116], recovery of a usable fraction of radioactive material in residues is possible in many cases. This is most desirable for liquids where a quantity of valuable activity of a radionuclide could be easily recovered. This must be seriously considered when recovery is much less expensive than disposal. Also, from these same discussions with the researchers who generated the most recoverable waste, it was evident that the chemicals that are most commonly used in the experiments comprise a short list. A table of the residues that were processed for this work is shown as Table 4-1.

Residue Number	Radionuclide	Matrix	Volume
12-27-10-1	⁹⁹ Tc	TBP-TcO ₄	100 mL
12-27-10-2	⁹⁹ Tc	Dark liquid – no label	200 mL
12-27-10-3	²³⁹ Pu & ²³⁸ U	n-dodecane	30 mL
12-27-10-4	⁹⁹ Tc	Organic acid	800 mL
12-27-10-5	⁹⁹ Tc	Organic	300 mL
12-27-10-6	⁹⁹ Tc	Organic	25 mL
12-27-10-7	⁹⁹ Tc	Organic	125 mL
12-27-10-8	⁹⁹ Tc	Organic	100 mL
12-27-10-9	⁹⁹ Tc	H ₂ O	400 mL
12-27-10-10	⁹⁹ Tc	Waste	200 mL
12-27-10-11	⁹⁹ Tc	No notation	300 mL
12-27-10-12	⁹⁹ Tc	Orange liquid	800 mL
12-27-10-13	⁹⁹ Tc	Organic	500 mL
12-27-10-14	⁹⁹ Tc	H ₂ O, NO _x	200 mL
12-27-10-15	⁹⁹ Tc (0.5 g)	Organic acid	500 mL
12-27-10-16	⁹⁹ Tc	MeOH, Acid	50 mL
12-27-10-17	⁹⁹ TcO ₄	Sn(II)Cl ₂ & thiourea	450 mL

Table 4-1. Residues from Radiochemistry Experiments

From a labeling aspect, there is a need to provide much better information for the solutions that are turned in for radionuclide recovery or waste. A form such as that illustrated in Figure 4-1 is recommended for documentation of the production of a byproduct of experimentation that has both hazardous and radioactive properties. The form will possibly not fit all situations and will evolve with growth the program. Information important to recovery of materials from the waste and other appropriate hazard data should be indicated by the researcher. This can be accomplished with an in-process waste database.

UNLV Radiochemistry Program				
In-Process Experimental Products				
Products Produced by (Researchers Full Name)				
Date of Storage				
Has a value analysis been completed? (Yes \Box , No \Box)				
Is recovery of radioactive material recommended? (Yes \Box , No \Box)				
Volume of solution mL Solution pH				
Radionuclide (1):; Mass (1)g; Moles (1); Activity(1)Bq				
Radionuclide (2):; Mass (2)g; Moles (2); Activity(2)Bq				
Radionuclide (3):; Mass (3)g; Moles (3); Activity(3)Bq				
Chemical compound(s):				
Chemical compound(s):				
Chemical compound(s):				
Hazardous Components: Flammable Corrosive				
Oxidizer 🗖 Toxic 🗖				
CAS Number of Hazardous components:				
If recovery of radioactive material in this product is not desired, is it possible to remove the hazardous component to allow disposal as radioactive waste? Yes \Box , No \Box)				
Hazardous waste + radioactive waste is mixed waste. If recovery of the radionuclides in this product is not desired and it contains hazardous material that cannot be rendered non-hazardous, then the product may be a mixed waste. Label the material as radioactive and hazardous and notify the UNLV Radiation Safety Office.				

Figure 4-1. Documentation Form for In-Process Products

Protocols are required at UNLV for current work with radioactive materials [117] [118]. Protocols should require documentation of inprocess products, with specification that mixed wastes are only allowed to be made with the permission of the UNLV Radiation Safety Officer so that provisions for their disposal can be made. Specification of ways to minimize the creation of undesirable waste should also be included in the protocol. When properly used, protocol waste specifications should help to prevent the creation of wastes that are dangerous to personnel and the environment or are prohibitively expensive to dispose. In the selection of the waste materials that were to be evaluated for recovery, there was little previous knowledge of the components of the waste. The researcher that created these wastes was consulted for information about the waste and the technique that he recommended for recovery of the ⁹⁹Tc. In all cases, the method recommended was addition of hydrogen peroxide to break down the organic constituents and recovery of the ⁹⁹Tc from the liquid. The liquid in each of the containers was dried in glass beakers and hydrogen peroxide was added slowly as reactions were common.

4.2 Recovery Decisions

The residue matrix documented on waste containers was not useful in determining the best chemical methods which could facilitate returning radionuclides in liquids back to a reusable form. There was no information allowing identification of the quantity of any of the liquids

that were in the samples or the amount of hazardous components created with the residues. Figure 4-2 is typical of the containers of material that were received for disposal.



Figure 4-2. Waste Container from the Accumulation Area

Establishing a basis for recovery with samples that are well documented could be a simple minimum mass-to-recover or a minimum mass concentration to make it worthwhile. However, evaluation of other properties of the material should be a consideration, such as the amount and type of hazardous materials in the residue and the cost of disposal of the waste that will result from the recovery effort. A low residue volume with a high concentration of recoverable material may be quite attractive for recovery. Removal of the activity from the hazardous component of the residue may simply be a filtration and cleaning of the filter as in sample # 12-27-10-6 (Table 4-2). In this case a simple filtration, washing of the filter, and removal of the activity from the filter was all that was required.

Table 4-2 provides the results of analysis of 0.1 mL from each of the waste products that were available for recovery experiments and listed in Table 4-1. The analysis was completed using a LSC, while the volume was small for this analysis; the activity present in each sample was more than adequate to make a reasonable estimate of the ⁹⁹Tc contained in each sample. With this data, the desirability for radionuclide recovery can be more reasonably determined.

Residue #	Volume (mL)	Activity Concentration (MBq/mL)	Total Activity (MBq)	Total ⁹⁹ Tc mass (mg)
12-27-10-1	100	0.00456	0.5	7.28E-01
12-27-10-2	200	0.00054	0.1	1.72E-01
12-27-10-3	30	0.00031	<0.1	1.49E-02
12-27-10-4	800	1.68751	1350.0	2.15E+03
12-27-10-5	300	3.66804	1100.4	1.75E+03
12-27-10-6	25	2.94610	29.5	4.70E+01
12-27-10-7	125	7.93699	992.1	1.58E+03
12-27-10-8	100	0.06937	6.9	1.11E+01
12-27-10-9	400	0.08037	32.1	5.12E+01
12-27-10-10	200	2.48515	497.0	7.92E+02
12-27-10-11	300	0.01224	3.7	5.85E+00
12-27-10-12	800	0.88036	704.3	1.12E+03
12-27-10-13	500	0.74110	370.6	5.91E+02
12-27-10-14	200	0.82096	164.2	2.62E+02
12-27-10-15	500	2.74065	1370.3	2.18E+03
12-27-10-16	50	0.29831	14.9	2.38E+01
12-27-10-17	450	0.29630	133.3	2.13E+02

Table 4-2. Analysis Results for Experiment Residues

4.3 Radionuclide Recovery Method

The researcher who created the waste for these experiments was consulted to ensure that recovery of the radionuclides by chemical methods would not make a more hazardous situation. The samples with the highest activity ratio were selected for simple recovery, first by evaporating the liquids, then attempting physical removal of the radionuclide to an aqueous solution by the addition of H_2O_2 to break out the technetium. After breakdown of the solids to the point where no further reaction was observed, the liquids were removed from the solids with disposable pipettes. The clear liquids were then analyzed for activity content and placed in separate containers. A model Standard Operating Procedure for removal of radionuclides from experimental residue is provided in Appendix D.

Using a modified version of the form depicted in Figure 4-1, a visualization of how the decisions were made with concern for residues noted in Table 4-1. The completion of the data fields in either form contribute to the accuracy of the information presented in Table 4-2. Unless otherwise noted, these materials were collected from the satellite waste accumulation area in the Radiochemistry Laboratories. The methodology of sample liquid dispersal experiments is more completely covered in the Sample Material Analysis section. Tables 4-3 and 4-4 provide the recovery results. The data from this set of experiments is included in Appendix E.

4.4 Recovery Results

The result of experiments done to recover activity in a residue for reuse is presented in Table 4-3. This table lists the original volume of solution, the original radionuclide mass based on LSC analysis of an aliquot of the solution, and the mass recovered, also based on LSC analysis. This table appeared to indicate that higher activity solutions will have a larger loss in the recovery process and thus less recovery. This is also shown in Figure 4-3. As discussed in Appendix D, there are many other methods for recovery of materials from solution, however, some introduce more cost or more hazard into the recovery process.

Sample #	Original Volume (mL)	Estimated ⁹⁹ Tc Mass Available for Recovery (mg)	Estimated ⁹⁹ Tc Mass Recovered from Residue (mg)
12-27-10-4	800	2152	21.6
12-27-10-5	300	1754	168
12-27-10-6	25	47	10
12-27-10-7	125	1582	30.3
12-27-10-10	200	792	77.4
12-27-10-12	800	1122	5.9
12-27-10-13	500	591	31.1
12-27-10-14	200	262	24.7
12-27-10-15	500	2184	13.2
12-27-10-16	50	24	3.8
12-27-10-17	450	213	17.2

Table 4-3. Radioactive Material Recovery from Experiment Residue

Most importantly, the recovery yield of technetium from the waste solutions was very low. This is indicative of the need for solution specific protocols for extraction of radionuclides from residues such as this. In general, these attempts at recovery demonstrate that it is not a difficult process to separate radionuclides from solutions.

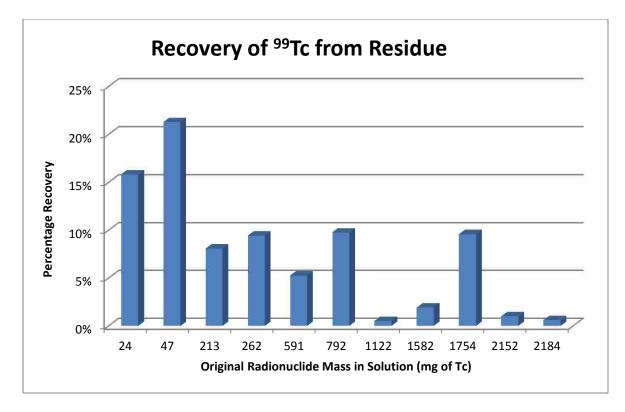


Figure 4-3. Graph of Percent Recovered vs. Original Radionuclide Mass

1-18-87-10-10 VST- 12-27. T.O. Dou Residua 10-12

Figure 4-4 Recovered Solutions

4.5 Simple Material Analysis

A rapid technique for evaluation of the liquids as to their desirability for recovery was evaluated by portable instrument analysis of the filter paper used for determination of the dispersion of the liquid from each sample. A dispersal experiment is conducted where ~100 μ L is pipetted to the center of a 15 cm diameter Whatman #1 filter. The sample color on the filter paper is observed and documented. The dispersion continues to a previously undetermined diameter and the presence or absence of a discernible ring is noted. A visual observation of this technique is noted in Figure 4-5.

Table 4-4 was constructed to indicate the difference in response for each liquid on the filter for a GM detector and a thin window alpha/beta scintillation detector. The response data is listed for each of the residues evaluated. Measurements with each instrument were taken directly over the center of each filter sample at a height above the sample of approximately 1 centimeter. Knowing the radionuclide as ⁹⁹Tc and the approximate average emission energy as 100 keV, the nominal detection efficiency at that location is in the range of 10% for the activity on each filter.

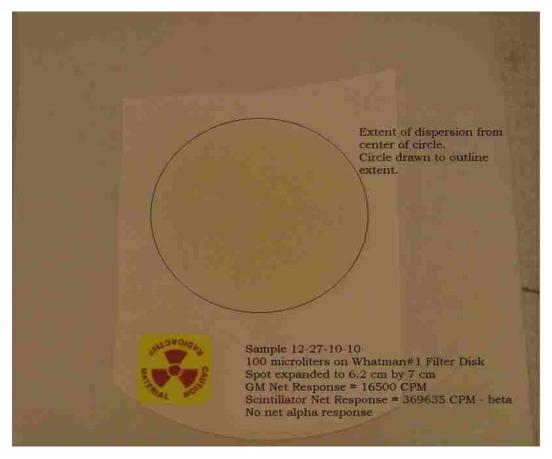


Figure 4-5 Dispersion Filter for Residue # 12-27-10-10

Sample	GM Response	Response Scintillation Detector Response	
Number	(CPM)	Alpha (CPM)	Beta (CPM)
Background	100	2	160
12-27-10-1	200	4	3145
12-27-10-2	130	0	229
12-27-10-3	100	6	196
12-27-10-4	11,000	4	154,395
12-27-10-5	44,000	1	789,244
12-27-10-6	10,000	22	308,254
12-27-10-7	45,000	2	846,860
12-27-10-8	250	2	4,545
12-27-10-9	450	12	12,068
12-27-10-10	17,000	2	369,803
12-27-10-11	220	2	1,378
12-27-10-12	6,500	0	107,624

Table 4-4 demonstrates that a simple direct measurement from a filter may provide adequate information to qualitatively determine whether the residue contains sufficient activity to be considered for recovery. Since the majority of these residues were unknown and unlabeled mixtures, it was incumbent on the researcher to have an evaluative process for initial discrimination.

Sample	GM Response	Scintillation Det	ector Response
Number	(CPM)	LSC (CPM)	Portable Beta (CPM)
Background	100	20	160
12-27-10-1	200	27382	3145
12-27-10-2	130	3245	229
12-27-10-3	100	1866	196
12-27-10-4	11,000	10125070	154,395
12-27-10-5	44,000	22008215	789,244
12-27-10-6	10,000	17676626	308,254
12-27-10-7	45,000	47621954	846,860
12-27-10-8	250	416217	4,545
12-27-10-9	450	482218	12,068
12-27-10-10	17,000	14910872	369,803
12-27-10-11	220	73440	1,378
12-27-10-12	6,500	5282170	107,624

Table 4-5. Comparison of Instrument Response to Residue

The last step to determine the value of the simple analysis technique used was a comparison of the results of the GM (Ludlum Model 3 rate-meter with 44-9 GM probe and Ludlum 2360 ratemeter/scaler with 43-93 alpha/beta scintillation detector response to the LSC response. Table 4-5 provides that comparison. This information provides a simple technique to determine an estimate of the activity concentration of a solution and decision criteria regarding the suitability of that solution for activity recovery. There is not a true calibration of such a technique, but a nominal estimation of the detection efficiency, using the unattenuated efficiency for the detectors used, for the radiation emitted from the activity in the solution will provide a reasonable estimate of the activity on the filter.

4.6 Material Recovered in Experiments

This section describes the recovery of ⁹⁹Tc from the residues available as indicated in Table 4-1. Some of the residues were found to be unsuitable for recovery as noted in Table 4-6, those solutions highlighted had sufficient activity to attempt recovery. In some cases, recovery was attempted but an unrecoverable product was identified or created. The lesson learned from these unsuccessful recoveries is to use only a small fraction of the solution for initial testing with several different methods, then select the best method and use that one for the larger scale recovery.

For each of the residues listed in Table 4-6, the solutions were evaporated in a HEPA filtered fume hood in their original containers, this allowed for a very slow removal of the liquid with no external power or possibility for higher level contamination of glassware or other apparatus. This also minimized the probability that some volatile compounds of Tc would be lost. Since virtually any compound of Tc could be present in the solutions, a quick summary of the volatility of these solutions is presented in [119]. In that document, it is reported that the melting point of Tc₂O₇ is 118°C and its boiling point is 350°C.

Complete volatility of ⁹⁹Tc when calcining an acid solution was attained at 350°C. Mixed oxides have melting points to 1200°C.

Residue #	Volume (mL)	Activity Concentration (MBq/mL)	Total Activity (MBq)	Total ⁹⁹ Tc mass (mg)
12-27-10-1	100	0.00456	0.5	7.28E-01
12-27-10-2	200	0.00054	0.1	1.72E-01
12-27-10-3	30	0.00031	<0.1	1.49E-02
12-27-10-4	800	1.68751	1350.0	2.15E+03
12-27-10-5	300	3.66804	1100.4	1.75E+03
12-27-10-6	25	2.94610	29.5	4.70E+01
12-27-10-7	125	7.93699	992.1	1.58E+03
12-27-10-8	100	0.06937	6.9	1.11E+01
12-27-10-9	400	0.08037	32.1	5.12E+01
12-27-10-10	200	2.48515	497.0	7.92E+02
12-27-10-11	300	0.01224	3.7	5.85E+00
12-27-10-12	800	0.88036	704.3	1.12E+03
12-27-10-13	500	0.74110	370.6	5.91E+02
12-27-10-14	200	0.82096	164.2	2.62E+02
12-27-10-15	500	2.74065	1370.3	2.18E+03
12-27-10-16	50	0.29831	14.9	2.38E+01
12-27-10-17	450	0.29630	133.3	2.13E+02

Table 4-6. Identification of Waste Suitable and for Recovery.

The dried and semi-dried residues were transferred to 250 mL polyethylene containers and a 30% solution of hydrogen peroxide was added in steps to allow transfer of the ⁹⁹Tc to the solution and the creation of ⁹⁹TcO₄⁻. The volume of concentrated H_2O_2 used was typically less than 10 mL. Commonly available 6% solution of by H_2O_2 was used to dilute the product and wash the residue. This was done to minimize impact on the available 30% solution, reducing the cost of the project. The final amount of H_2O_2 added was different for each residue and was

only based on the time required for breakdown of the residue and removal of Tc to a liquid. In some cases this was easily identified as the solid was eliminated, in others it appeared that any reaction had stopped. The approximate amount of H_2O_2 added was 0.1 mL of H_2O_2 per gram of solid residue; this data was not collected for each recovery. The materials that were present in the original residues were unknown. One residue, sample number 12-27-10-9, did not breakdown and was disposed as solid waste.

Some important safety concerns for use of H_2O_2 in high concentrations are that it can give off vapors that can detonate above 70°C at atmospheric pressure. This can then cause a boiling liquid expanding vapor explosion of the remaining liquid. In small volumes, this volatility is less of a concern, but to be safe, samples 12-27-10-4, 12-27-10-5, 12-27-10-12 through 12-27-10-15 and 12-27-10-17 used in these experiments were placed in water baths to maintain the solution at a temperature below 50°C. It should be noted that with a rotovap or other similar equipment, processing time and efficiency could be improved.

Hydrogen peroxide vapors can form sensitive contact explosives with hydrocarbons such as greases [120]. Hazardous reactions ranging from ignition to explosion have been reported with alcohols, ketones, carbolic acids (particularly acetic acid), amines, and elemental phosphorous [121] [122]. Concentrated hydrogen peroxide if spilled on flammable materials such as clothing the material may spontaneously ignite [123].

Considering these potentially hazardous situations and selecting to use 30% H_2O_2 and keep it cool, seems to be an adequate method to recover ⁹⁹Tc from many of the products that have been cast aside for recovery. However, it certainly is not the most efficient. In the situation where the content of the residue is not known, it may be one of the safer methods. For Tc recovery, many of the compounds of Tc, two important aspects are:

- The reaction of Tc(III), Tc(IV) and Tc(V) with hydrogen peroxide in aqueous solutions immediately yields Tc(VII).
- Tc(VII) is easily lost upon evaporation of acid solutions unless a reducing agent is present or the evaporation is conducted at low temperature. [124]

The reaction of hydrogen peroxide with other compounds of Tc in medicine has been shown for example decompose ^{99m}Tc⁻ to pyrophosphate to pertechnetate (99mTcO₄-) [125]. The mass of 99Tc was determined from LSC analysis of a clear solution before and after recovery. The solution before recovery consisted of solvents, acids, and various solids. The effect that the H_2O_2 may have had on the LSC cocktail could have been a cause for underestimate of the 99Tc concentration, however, these samples were analyzed again four months later and the deviation from the original analysis was minimal. The maximum deviation from the original values in the ⁹⁹Tc activity was 7.6% lower and the average deviation was 1.2% lower over seven samples that were re-analyzed.

After recovery, the solutions were transparent and were removed from the solids by pipetting. The purity and cleanliness of the ${}^{99}\text{TcO}_{4}$ in solution was not a concern to this research, other than a more pure solution may be needed for further use of the ${}^{99}\text{Tc}$ solution. The results provide evidence that the recovery of the ${}^{99}\text{Tc}$ from products previously considered as waste products for disposal is possible. The concept that radioactive materials in waste products could be recovered is valid. The purity of the final product will again cause a loss of some of the product but it is reasonable to indicate that recovery efficiency would be improved with the use of other techniques.

Table 4-7 provides the recovery yield of ⁹⁹Tc from the residues processed. The total mass of ⁹⁹Tc recovered was approximately 393.2 mg. The average recovery fraction was 6.21%. The minimum recovery fraction was 0.5% and the maximum 15.8%. The difference in the initial activity determined to be in the residues and the activity determined to have been recovered may have been due to a number of issues such as a higher affinity of the ⁹⁹Tc for another component of the residue. The consistently low yield of recovery appears to indicate that the determination of the activity in the residue was reasonable because there were no unreasonably high estimates of the original activity in the residue. Another reason for the appearance of a low activity yield might be enhanced detection efficiency for the determination of activity in the residue by materials in the residue that may have caused a detectable luminescence. This was not investigated.

The establishment of recovery goals was not possible because the residue compounds were unknown. It appears that a reasonable minimum goal should be on the order of 5%. It also appears that most of the activity was disposed of as solid waste with the hardened organic remains after extraction of the 99 Tc using H₂O₂. In cases where the residue contains a very valuable product it might be reasonable to take further action to remove the product. In cases where the solid completely dissolved in the hydrogen peroxide, other loss mechanisms such as evaporation must have occurred.

Sample #	Recovery Volume (mL)	Estimated ⁹⁹ Tc Mass Recovered from Residue (mg)	Estimated Percentage of the Original ⁹⁹ Tc in Residue that was Recovered (%)
12-27-10-4	800	21.6	1.0%
12-27-10-5	300	168	9.6%
12-27-10-7	10	30.3	1.9%
12-27-10-10	125	77.4	9.8%
12-27-10-12	400	5.9	0.5%
12-27-10-13	200	31.1	5.3%
12-27-10-14	800	24.7	9.5%
12-27-10-15	500	13.2	0.6%
12-27-10-16	200	3.8	15.8%
12-27-10-17	500	17.2	8.1%

Table 4-7. Radioactive Material Recovery Yield from Samples

To identify the degree of cost savings by this small scale research effort, one must consider the cost of disposal for unknown chemical compounds with radioactive components in addition to the cost of recovery using simple methods and minimal analyses costs and the fact that activity was recovered and could be reused. The cost avoidance of disposal of these materials as mixed waste is the real cost savings.

With the information known thus far, this cost avoidance can only be guessed to be significant, and it depends on many factors such as the content of hazardous materials in the waste. Consider an unknown mixed waste consisting of toxic and radioactive characteristics. The process for recovery would require a safety analysis to prevent personnel exposure to, and cause destruction of, the toxic material. Analysis must identify and quantify the hazards and in addition, the hazardous component must be abated or confined. These costs may be substantial for a small waste management program of a research organization. Based on past experience with hazardous waste disposal and information from a simple web search, in the recent past, costs of disposal for mixed waste may exceed \$40.00 per liter.

In this study, the volume of residue collected was almost four liters of waste that had an easily detectable concentration of ⁹⁹Tc. The 393.2 mg recovered can be estimated to be worth approximately \$800.00 at \$2.00 per mg. The materials put into the recovery of the ⁹⁹Tc cost less than \$100.00, so the cost avoidance can be estimated to be close to \$1000 for this simple attempt at recovery of ⁹⁹Tc from the waste residue. Greater cost avoidance with less risk to the researcher would be expected if the content of the residue was known and the chemistry was more accurately designed to remove known compounds of Tc from the waste solutions.

4.7 Safe Practices in Recovery of Radioactive Materials

Whenever something is made that has multiple hazardous constituents, the behavior of that compound or solution is not likely to be well known. If the constituents react with one another at different temperatures or in different atmospheres, the hazard may be evident when those components are first combined. Sometimes incompatible components that are combined in a waste container may ignite or explode immediately, or at some time post mixing.

There are many safety reasons such as prevention of explosions or generation of poisonous or dangerous gases, to investigate the compatibility of chemicals that will be mixed together. If it is unknown what will happen when chemicals react, it is suggested that the chemicals are added together in small quantities first. The Material Safety Data Sheets (MSDS) provide information about the properties of the chemicals, but may not be detailed enough for certain experiments. In addition, the MSDS may not provide sufficient information to ensure continued safety of those working in the laboratory.

Whenever possible, appropriate ventilation and the sash of a fume hood should be used to protect the researcher from potentially dangerous solutions splattering or venting on the face, hands, or any other body part. Personal protective equipment such as safety glasses/goggles or full face shield to protect the face, whole body protection with a laboratory coat and plastic or rubber apron, and hand protection with gloves appropriate for the chemicals that are handled are

some ways to protect researchers working with highly reactive or unknown solutions. Recovery of radionuclides from unknown solution matrices requires an extra effort to be safe. If it is suspected that there may be a hazardous situation created, action must be taken to protect all researchers and the laboratory from damage and consider that contamination control may be an important issue. It is always important to ensure that radioactive material stays in the controlled workspace, therefore it is always important to consider that actions taken to heat, shake, or bubble a solution may cause activity to become airborne or spread from the work area.

The first step to radionuclide recovery is to determine what may be in the solution. The second step is to determine properties of the hazardous components and whether any attempt at recovery is a reasonable thing to do. Next, determine the radionuclides and activity of each in the solution and calculate the approximate mass of radionuclide that is present in the container.

If the analysis of the solution indicates that the radionuclide recovery is feasible, then the next step is to remove all liquid from the residue, if possible. After drying the residue, add the appropriate chemicals to extract the radionuclide of interest from the solid residue into a liquid phase. Finally, collect the liquid phase and determine how much activity (or mass) of radionuclide was recovered.

4.8 Conclusions

The current practices used to document liquid radioactive waste in the Radiochemistry Program are inadequate to identify hazardous materials in the waste and allow safe recovery of radionuclides.

Waste labeling can be effectively used to identify hazardous constituents and methods to make those characteristics harmless. Experimental methods should include waste management techniques.

Mixed waste production can be eliminated in the radiochemistry program by requiring researchers to either recover radioactive materials from their experimental residue or destroy the hazardous components of that residue to enable disposal as radioactive waste.

4.9 Implementation

Recovery of radionuclides from residues of radiochemistry experiments is attractive for two primary reasons; it allows for sustainability of the radiochemistry program through increased availability of materials and it reduces the cost of disposal of radioactive waste. However, a program to allow recovery of radionuclides from hazardous residues of radiochemistry experiments may also present a hazard to the researcher that will complete the recovery. Implementation of a program for radionuclide recovery, and associated precautions and considerations are presented.

4.9.1 Abstract

Radiochemistry research has led to great discoveries of the elements and their isotopes. Recovery with intent to recycle has always been a part of larger programs where the value of radionuclides is high, such as removal of enriched uranium or plutonium from used nuclear fuel. In research laboratories, the financial incentive to recover is not always present, but the desire to reduce waste costs may be the driver that makes recycling feasible. In this document a plan for recovery is proposed for an academic radiochemistry operation. The plan drives hazardous waste compliance and provides a reasonable basis for a financially feasible radionuclide recovery program. Considerations required for incorporating the recovered radionuclides into the accountability system are identified.

4.9.2 Discussion

Mixed waste is an undesirable consequence of radiochemistry due to the need to evaluate the properties of radioelements when subjected to various chemicals in search of the unknown. In some cases, the hazardous component of the waste can be made to be less hazardous or non-hazardous, or the radioactive component of the waste may be recovered for reuse making the waste only hazardous and not radioactive, or in many cases, less radioactive. A radiochemistry program has the unique opportunity to do whatever chemistry is required. Other programs where radioactive materials are used or waste is collected for

disposal, processing of the waste for radionuclide recovery or for concentration of the waste is not allowed.

Academic radiation safety programs provide detailed services for researchers to ensure their protection and the protection of faculty, staff, students, and members of the public. To devise a scheme for that indicates the controls provided, consider that the following questions should be asked by an academic RSO for each of the researchers:

- What am I responsible for?
- ➤ Where is it?
- > Who has it?
- ➤ What are they doing with it?
- > What will they do with it when they are done with it?

The reason for these questions is simple and logical and is based on the premise that radioactive material must be properly controlled to prevent dispersion and maintain dose to all people ALARA. Some important concepts are required to be identified so that hazardous conditions are not created during the recovery.

The method used for recovery of radionuclides from waste residues or for destruction of the hazardous component of those residues is devised by the researcher or the research supervisor based on knowledge of the content of the residue. In the experiments done for radionuclide recovery, the researcher that created the waste was consulted and provided the advice to dry the waste to remove solvents and breakdown

the dried organic residue with hydrogen peroxide to recover the technetium in the waste. While this was not a detailed chemical procedure and it provided poor recovery results for several of the compounds, it was a method that required careful observation of the materials and did not result in a hazardous product. It is however essential that all recovery efforts be closely observed and all appropriate safety measures are taken.

Another important consideration in the recovery of radionuclides is accountability of the radioactive material. A clean way of doing this is subtract the activity in the residue (waste) from the inventory and add in the activity recovered. In this way, any radionuclides that were not recovered from the residue go into the waste that is disposed and only recovered radionuclide(s) are added back into the inventory. In the inventory control system it may be desirable to maintain the recovered material as a separate entity since it may not have the same chemical or radiological makeup as the stock material. The method selected is only important to the completeness of the inventory.

The separation of activity from a mixed waste is not always complete and the destruction of a hazardous material does not ensure that a hazardous component of the waste does not still exist. After removal of as much of the activity possible, the residue should again be evaluated to determine if a hazardous component exists. This may be possible from knowledge of what material went into the waste and what is likely to remain after the operations to recover activity were completed. Advice on disposal of the residue may be sought from a Certified Hazardous Materials Management professional.

The incentive for radionuclide recovery is inherent in several aspects of working with radioactive materials that are not often considered. Some of them are listed here:

- The cost of radioactive solutions is typically high and purchase may be restricted by license.
- The purchase may require several weeks depending on the radionuclide, the chemical compound, and preparation time for the vendor.
- A recovered compound is available when needed and use is only restricted administratively.
- Disposal of mixed waste is costly because waste sites in the United States cannot accept it. Typically a waste site will accept a sample of a waste provisionally to determine if it can be made into an acceptable waste. If it can be processed, a fee is applied to cover that cost and the cost of burial.

4.9.3 Summary

Radionuclide recovery is desirable for radiochemistry programs. The changes to accountability systems are simple and administrative control of waste products is simplified if hazardous components are less hazardous, radionuclides are less concentrated, or radionuclides are completely recovered and the waste is non-hazardous. The measures to be taken to recover radionuclides or make hazardous components nonhazardous should be devised by those who know what constituents went into the residue and what was done to the residue that could have made any chemical changes.

CHAPTER 5

RECOVERY OF URANIUM FROM COAL ASH

5.0 Hypothesis

Samples of coal and ash from a coal fired plant in Oklahoma contain uranium at a sufficient concentration to use that material as a source of uranium for nuclear power generation.

5.0.1 Resource and Criteria

Using the samples provided, identify the concentration of uranium. The economic feasibility of uranium extraction from coal ash occurs at approximately 200 grams of uranium per 900 kg of coal ash [126]. A total of 24 samples of coal ash, 18 samples of coal, and 6 samples of liquid from plant tanks were received from an AES Corporation [127] coal fired generating station in Oklahoma.

5.1 Coal as an Energy Source

Coal is very important to the production of electricity worldwide. According to the World Coal Association, coal fired plants currently provide 41% of global electricity [128]. In 2011 the United States electricity production was 42% by coal [129]. When coal is burned to make steam for an electric plant, ash is produced that contains all of the heavier elements that were present in the coal. According to the US Environmental Protection Agency (EPA), coal combustion generates ash (SiO₂-Al₂O₃-Fe₂O₃-CaO and element impurities) equal to about 10% of the original volume of the coal and typically about 95% of the ash is

initially retained at the site [130]. Typically 70% to 80% of the coal ash is disposed of in dry landfills [131]. The annual production of coal ash in the United States alone of about 90,000,000 kg of ash provides a large waste volume and takes up large land areas for disposal of the ash at each site [132]. The chemical structure of coal ash varies with the type of coal burned. Each type has different elemental constituents that are heated to different temperatures and thus form different fundamental compounds [133].

5.2 Uranium Content of Coal

Uranium is present in the earth's crust and is concentrated in many minerals [134]. The USEPA has published values for uranium and thorium content of coal in 1984 between 1.3 ppm and 3.2 ppm [135]. Natural uranium contains the fissionable isotope ²³⁵U at a natural abundance of 0.71% [136]. The primary interest in uranium is as the fuel source for nuclear power. To be used for nuclear power in commercial light water nuclear reactors in the United States, the concentration of ²³⁵U is increased by enrichment to approximately 2.5 to 4% [137]. Nuclear power has once again become a power source of interest as fossil fuels have more control of the economies of the world; as a result economic outlook for uranium is increasingly positive.

As uranium for use in nuclear reactors is once again considered a valuable commodity, there is interest created by some publications [138] [139] which indicated that the concentration of uranium oxide in coal

ash could be high enough to indicate that recovery is feasible. Values of uranium concentration in coal ash were reported at 281 ppm U, citing a study in Yunnan province China [140]. Most of the available information from these sources cites the coal from this area of China having a uranium concentration of as much as 300 ppm with an average concentration of about 65 ppm. With approximately 90,000,000 kg of ash created each year from burning coal in the United States alone, this could be a tremendous resource for uranium if the mechanism for removal is financially reasonable. At 300 ppm, the amount of uranium in the ash produced is 2.7E4 kg/year, at 2 ppm; it is 180 kg/year. Even though there are richer deposits of uranium such as high grade ore which is 20,000 ppm or greater to low grade ore which is 1000 ppm to 20,000 ppm the higher of these concentrations could be financially acceptable for recovery. However, as shown in this research and in peer reviewed literature including a USGS study [141], the uranium concentration cited in the China coal ash are very rare and indicate a high concentration of uranium in the coal that is burned.

As a result of the publication of this information in trade journals, many people in the coal industry have shown interest in the use of the coal ash created at their plant to produce uranium. This section describes the project.

5.3 Sample Description

Several samples of coal ash, and coal from an AES Corporation power plant in Oklahoma were received for analysis. The coal was labeled as Hartshorne Coal and the samples were from separate piles of coal and coal ash. The initial focus of the project was evaluation of the ash. It is a reasonable consideration that the highest concentration of uranium in these samples would be in the ash because the waste water has a low concentration of the coal before burning and much of the mass of the coal is lost as combustion gasses with the heavier elements becoming more prevalent in the ash. The ash is a waste product at the plant and potentially the most plentiful and available reservoir for uranium recovery.

5.4 Uranium Content of Samples

In order to determine the uranium content of the ash, the most desirable method from a radiochemistry standpoint was high resolution gamma spectroscopy. Coal and its ash are very messy and undesirable to handle in any quantity outside of a closed container. Sample preparation was simple and utilized inexpensive plastic containers as sample holders. The instrument of choice was a high purity germanium detector interfaced through a Canberra DSA 1000 Digital Signal Analyzer. A uranium standard was prepared using uranyl nitrate in a candle gel. The gel maintained the uranium in a homogeneous geometry that remained throughout the project. The standard has similar density characteristics as the ash without the natural radionuclide content. The spectra obtained for both the standard and the coal ash were analyzed using Genie 2000 gamma spectrum analysis software. Gamma spectroscopy is possible for determination of ²³⁸U content because of the immediate short lived daughters of ²³⁸U, ²³⁴Th, ^{234m}Pa, and ²³⁴Pa. The first analyses using this method showed insufficient analysis time to identify the concentrations of uranium present in the samples based on the concentration of the Pa progeny expected to be present in the sample in equilibrium with the ²³⁸U parent. A second analysis proved that this analysis technique was usable but the analysis time must be in excess of 250,000 seconds per sample.

Another method selected that required minimum sample preparation time was x-ray fluorescence analysis of the coal ash. The actual analysis time was short, a few minutes, but since sample preparation and analysis required the Geoscience department at UNLV, it was decided to look into other analysis techniques for further confirmation. This method would ultimately provide a technique that confirmed the results obtained by gamma spectroscopy and mass spectroscopy.

Samples were prepared for analysis by the Thermo iCAP 6000 Duo Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES) by partial digestion of the coal ash with hydrofluoric acid (HF) and leaching with concentrated hydrochloric acid (HCl). This method would allow detection of concentrations of uranium in the low part-per-million (ppm) range. The dilution required for this analysis was approximately 100.

A separate analysis of uranium in coal ash was conducted with the Perkin Elmer Elan DRC II, Inductively Coupled Plasma Mass Spectrometer, and (ICPMS). Due to the sensitivity of the ICPMS, each sample was diluted to enable detection of a concentration of uranium in the part-per-billion (ppb) range. The short sample analysis times allowed for many samples to be analyzed in a reasonable time.

5.4.1 Sample Preparation

In this section the sample preparation for each analysis technique is discussed. The sample preparation may be as simple as placing the sample in a standard geometry, such as is done with gamma spectroscopy. Alternatively, it may require digestion and filtration as was done to prepare the samples for ICP-AES and ICP-MS analysis.

5.4.1.a High Resolution Gamma Spectroscopy

Samples were placed into plastic jars that provided a consistent geometry to standards that were prepared with uranyl nitrate and a suspending gel. In the preparation of the standards, 500 grams of the gel was melted at 200 °C and a known mass of UO₂(NO₃)₂*6H₂O of 1.8 g was added to the melt. This relates to a total uranium activity of 1.06x10⁴ Bq. The activity concentration of the melt was then 2.13x10¹ Bq/g. The net mass of the uranium and gel melt in HRC-185 was 405.42 g and the activity of this standard was therefore 8.62x10³ Bq. Figure 5-1 shows a photo of uranium standard HRC-185.

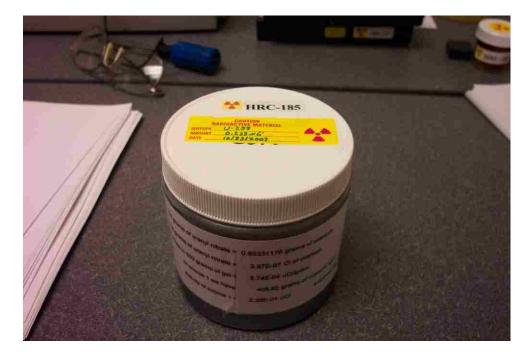


Figure 5-1. Uranium Coal Ash Standard for Gamma Spectroscopy

The standard solution was stirred for 1 hour and the stir bar was removed before allowing the solution to cool and solidify to a consistent, mostly homogeneous solid. A second standard was made with 10% of the activity of the first standard. The jars used in the analysis were the same jars that the standards were made in. The standards were analyzed to provide a spectrum that would be representative of materials that contained uranium. Samples were placed atop an intrinsic germanium detector for analysis. The activity in the standards was sufficient to provide energy and efficiency calibration for the ^{234m}Pa in a short count time. The long count time for analysis at the level of the activity found in the samples prevented further analysis due to demand for use of the instrument by other researchers. 5.4.1.b X-Ray Fluorescence Spectroscopy

One 15 g sample of coal ash that was analyzed using this method was prepared by packing the sample media into a metal container that would present one surface to the x-ray beam for analysis. The sample thickness (greater than 1 cm) would be considered infinite compared to the depth of penetration of the x-ray beam. The PANalytical X-Ray Fluorescence spectrometer was used to analyze the samples. The analysis parameters were selected by the system operator and were not provided with the analysis result. No other XRF analysis of these samples was done.

5.4.1.c Inductively Coupled Plasma Atomic Emission Spectrometry

For analysis using ICP-AES, coal ash samples were first digested then leached [142]. Two mL of hydrofluoric acid (HF) was added to 1 g of coal ash for each sample and allowed to set for 24 hours. A 6 mL sample of concentrated hydrochloric acid (HCl) was added to the partially digested ash to leach out the released uranium. The mixture was allowed to leach for 72 hours prior to dilution for analysis. A total of 100 mL of 5% HNO₃ was added to provide dilution to a concentration more suitable for ICP-AES analysis.

5.4.1.d Inductively Coupled Plasma Mass Spectrometry

Dilution of an aliquot of the solutions prepared for the ICP-AES analysis by 1:1000 was accomplished with 5% HNO_3 then analyzed using ICP-MS.

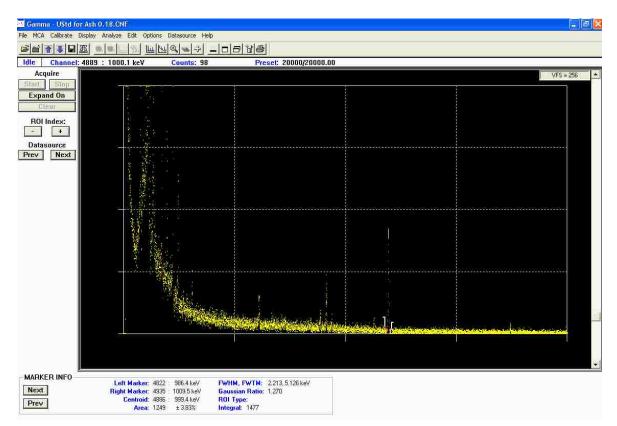
5.4.2 Sample Analysis and Results

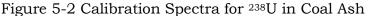
This section presents the results for each analysis technique. The results may be in units of activity as in the case of gamma spectroscopy, or in mass units as reported by the other techniques. In order to compare the results, they were all put into units of grams of uranium per metric ton of coal ash.

5.4.2.a High Resolution Gamma Spectroscopy Results

Coal ash sample number 2 (353.1 grams) was counted on a high purity germanium detector for 50,000 seconds. Coal ash sample number 3 (449.19 grams) was counted on a high purity germanium detector for 5000 seconds. Both of these analyses did not identify the immediate progeny of ²³⁸U; however both spectra did show some of the short lived progeny of ²²²Rn, ²¹⁴Pb and ²¹⁴Bi indicating that perhaps the concentration of ²³⁸U was lower than the detection limit for the analysis.

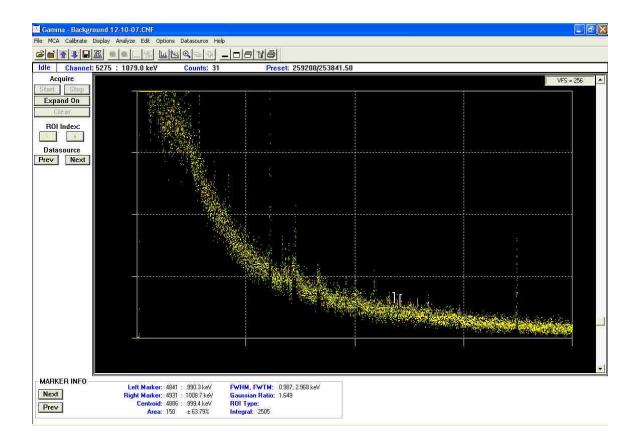
Hartshorne coal sample number 14 (568.1 grams) was counted on a high purity germanium detector for 50,000 seconds. The decay progeny 234m Pa was considered to be in equilibrium with the 238 U parent. The activity identified was 0.107 ± 0.026 Bq of 234m Pa per gram of ash. This relates to a mass concentration of 238 U of 8.61x10⁻⁶ grams/gram of

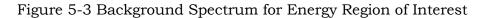




sample (ash). This concentration equates to approximately 7.8 grams of 238 U per ton (900kg) of coal with a 1 sigma uncertainty in the analysis of 1.9 grams/ton (900kg) of coal. The previous analyses were unable to identify the 234m Pa at that level.

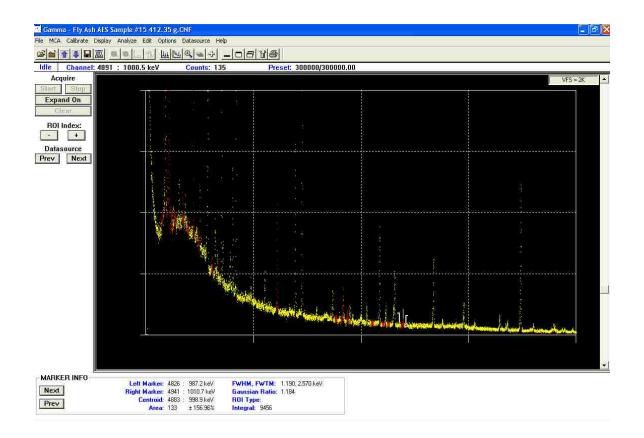
A calibration spectrum was produced using uranyl nitrate in a gel solution that would simulate ²³⁸U in coal ash. The spectrum was collected for 20,000 seconds and the photo-peak of interest for ^{234m}Pa, 1001 keV was easily identifiable in the spectra. This is displayed in Figure 5-2. A long background spectrum was obtained for this calibration to identify any conflicting photo-peaks. There were no conflicting photo-peaks at the energy of concern. This is displayed by Figure 5-2; notice the region of interest markers around the 1001 keV photo-peak in all of the spectra. These are present only for identifying the region and not for quantitative evaluation of the region.





Coal ash sample number 15 (412.35 grams) was again counted on a high purity germanium detector but this time for 300,000 seconds. The decay daughters of ²³⁸U, ²³⁴Th and ^{234m}Pa establish equilibrium within months of separation of the uranium and provide detectable emissions that can be identified as specific to ^{234m}Pa. Since the coal ash is in the form in which it was made, no chemical processes have affected the uranium content of the ash and it is reasonable to consider the immediate progeny to be in equilibrium. The radionuclide of interest in this analysis, ^{234m}Pa is formed from the beta minus decay of ²³⁴Th, the immediate progeny of ²³⁸U. The halflife of ²³⁴Th is 24.1 days and the half-life of ^{234m}Pa is 1.2 minutes. The equilibrium from ²³⁸U is well established within 10 half-lives (241 days) of purification of the uranium. Since purification for the standard batch was conducted many years prior, and the ash is at least moths old, equilibrium of the short lived immediate progeny ²³⁴Th and ^{234m}Pa is reasonable.

The most reasonable photo-peak associated with the determination of ²³⁸U in the sample is the 1001 keV photo-peak from ^{234m}Pa. This peak, although associated with an emission with a low yield (0.837 %), it is in a low background region of the energy spectrum and not affected by other photons with similar energy. Figure 5-4 provides display of the spectrum of Sample #15.





The activity of this radionuclide in the sample was 0.141 ± 0.016 Bq/gram. This relates to a mass concentration of ²³⁸U of 1.13×10^{-5} grams/gram of sample. This concentration equates to approximately 10.3 grams of ²³⁸U per ton (900 kg) of coal ash with a 1 sigma uncertainty in the analysis of 1.1 grams/ton (900 kg) of coal ash. Table 5-1 shows the gamma spectroscopy results for analysis of coal and coal ash from the stockpiles at an AES Corporation plant in Oklahoma.

The results that are presented for coal ash for gamma spectroscopy and the other methods are reasonably consistent with the results of Coal Ash presented in NIST Standard 1633b [143]. Since the coal from this site was only analyzed by gamma spectroscopy, the results presented in Table 5-1 may present a question about the closeness of the concentration of uranium in coal versus the concentration in the coal ash. There is approximately a 10% uncertainty in the analysis as presented. In addition, there is indeed release of uranium from the stack of the facility, a difference in the uranium content of coal ash and bottom ash, and possibly a difference in the type of coal burned to produce the ash that was analyzed and the coal that was analyzed in this work.

A more detailed analysis of naturally occurring radionuclides in coal, fly ash, and bottom ash [144] indicates a distribution between the concentration of natural uranium in coal: bottom ash: fly ash as 1:2:3. This document provides similar information for other naturally occurring radionuclides.

Sample Type	Uranium (Bq/g)	Uranium (ppm)	Uranium (g/900 kg)*
Hartshorne Coal	0.107	8.61	7.82
Coal Ash	0.141	11.3	10.3

Table 5-1 Gamma Spectroscopy Results of Uranium in Coal Ash

* Uranium (g/900 kg) is g of uranium per ton of coal or coal ash as appropriate.

5.4.2.b X-Ray Fluorescence (XRF) Spectroscopy Results

A sample was taken from coal ash sample number 3. The sample mass was 15 grams and it was packed into a container for fluorescence analysis. The analysis results indicated that there was 3.1 ppm ± 1 ppm of uranium in the ash. This equates to approximately 2.8 grams of

uranium per ton of coal ash with a 1 sigma uncertainty in the analysis of approximately 1 gram of uranium per ton (900 kg) of coal ash. Table 5-2 provides the complete element analysis for the coal ash sample.

Element	Value (ppm)	Element	Value (ppm)	Element	Value (ppm)	Element	Value (ppm)
Sc	46.7	Nb	9.3	Rb	59.1	Hf	1.4
V	161	Мо	9.8	Sr	423.3	Pb	22.8
Ni	75.2	Cs	17.4	Y	33.2	Th	14.4
Cu	57.3	Ba	939.8	Zr	113.6	U	3.1
Ga	13.9	La	42.9				

Table 5-2. X-Ray Fluorescence Analysis of Uranium in Coal Ash

The results presented by X-ray Fluorescence analysis are similar to the expected value as identified in published values for coal ash from burning coal in the United States as indicated in NIST standard SRM 2689 [145]. Table 5-3 provides a comparison of some of the elements in the results with the certified values (c) or information concentrations (i) for the NIST standard. It is unknown what standard was used in this analysis, the laboratory did not provide that information. The comparison in Table 5-3 is provided for information.

Constituent	NIST 2689 (ppm)	NIST 1633b (ppm)	Analysis Result
Element			(ppm)
Barium (Ba)	800 (i)	709 (i)	939.8
Cesium (Cs)	11 (i)	11 (i)	17.4
Lead (Pb)	52 (i)	68.2 (c)	22.8
Scandium (Sc)	32 (i)	41 (i)	46.7
Strontium (Sr)	700 (i)	1041 (c)	423.3
Thorium (Th)	25 (i)	25.7 (c)	14.4
Nickel (Ni)	122 (i)	120.6 (c)	75.2
Uranium (U)	Not Reported	8.79 (c)	3.1
Vanadium (V)	Not Reported	295.7 (c)	161

Table 5-3 Comparison of XRF Results with NIST Available Standards

5.4.2.c Inductively Coupled Plasma AES Results

The leaching introduced a dilution factor of 106 as 100 mL of deionized water (DI) and 6 mL of HCl were used for a 1 gram sample of ash. No method blank was used because the solid digestion procedure was sufficient to dissolve the solid and clear yellow solution remained. Filtering of the solution was used to ensure there were no extraneous particulates in the sample that could possibly clog the AES sample introduction tubing or nebulizer. All uranium in expected to have been in solution and have passed through the filter without retention. The Thermo iCAP 6000 was operated with a nebulizer gas flow of 0.51 L/min, an auxiliary gas flow of 0.5 L/min, a coolant gas flow of 12 L/min and a pump speed of 50 rpm. The analysis included 3 replicates for each sample and the wavelength of concern was selected as 367.007 nm. The results indicate a concentration of total uranium as shown in Table 5-4. However, no other analysis of this ash showed any results even close to this concentration of uranium. A review of the analysis indicates that the

results presented in this table were due to an interference peak from other elements in the sample [146]. Figure 5-5 indicates the analytes that might interfere at that wavelength. This wavelength (367.007) was considered to be the most reasonable wavelength to use because other wavelengths for uranium are less significant. The interference from other elements in the sample was significant because of their high concentrations.

	ICP-AES Sample Result		Dilution	Uranium mass per (900 kg)	
Sample	U (ppm)	σ _Y (ppm)	Factor	of ash	
FA-4	3.156	0.084	106	303.76	g
FA-8	2.773	0.125	106	266.9	g
FA-16	2.634	0.123	106	253.52	g
FA-20	2.99	0.057	106	287.78	g
FA-24	3.372	0.1	106	324.55	g

Table 5-4. ICP-AES Results of Uranium in Coal Ash

Since the result indicated was so very high compared to other analysis results (gamma spectroscopy, x-ray fluorescence spectroscopy, and mass spectroscopy) any signal from the uranium in these samples would be masked by the observed signal at the wavelength selected.

Uranium wavelength of Interest (nm)	Element and wavelength of interference (nm)
263.557	Molybdenum 263.553
367.007	Iron at 367.002 Iron at 367.009 Thorium at 367.006
385.466	Barium at 385.476
385.958	Scandium at 385.96
393.203	Scandium at 393.202
409.014	Zirconium at 409.051

Table 5-5 Interference Wavelengths from Sample Constituents

This analysis did not verify the existence of uranium at the concentrations indicated in these samples. In conclusion of this section, ICP-AES is not a good technique to use for the determination of uranium concentrations in coal ash because of interference from naturally occurring analytes present in the ash.

5.4.2.d Inductively Coupled Plasma Mass Spectrometry Results

The dilution of the coal ash sample placed the concentration in the ppb range. The Perkin Elmer SCIEX Elan DRC II mass spectrometer was operated at a nebulizer gas flow of 0.96 L/min, an auxiliary gas flow of 1.2 L/min, a coolant gas flow of 15 L/min and a pump speed of 24 rpm. The lens voltage was 6 V, the analogue stage voltage was -1562 V and the pump stage voltage was 900 V. The monitored masses (M/z) were 234.041, 235.044, and 238.05. There were 10 sweeps per reading, 1 reading per replicate, and 3 replicates per sample.

The results indicated a practical concentration of between 1.7 and 11.4 ppb of uranium. Since the samples were diluted by a factor of 1000, the concentrations of uranium in the coal ash samples were 1.1 to 9.5 ppm, or 1.1 to 9.5 micrograms of uranium per gram of ash. Two samples were excluded from the analysis results because the filters used to remove the solids broke through (FA15 and FA17). The results indicate a range of uranium in coal ash of 1.0 to 8.5 grams of ²³⁸U per (900 kg) of coal ash. This result appears to be consistent with the most common results in the literature, 0.4 to 3.0 ppm in coal, and 0.8 to 30.1 ppm in coal ash. The samples from the scrubber tank at the coal plant did not show any significant difference from the coal ash samples indicating that this tank is not a concentration mechanism from the plant systems. Table 5-6 provides the result of the sample mass spectroscopy analysis with the coal ash sample results indicated in Table 5-7.

Sample			Net Intensity	Concentration	Standard
ID:	Analyte	Mass	(cps)	(ppb)	Deviation (ppb)
B1- Blank	U	238	11318.042	0.455	0.020
FA01	U	238	92776.227	2.827	0.049
FA02	U	238	49599.752	1.329	0.060
FA03	U	238	45189.494	1.175	0.046
FA04	U	238	46880.944	1.234	0.045
FA05	U	238	43634.356	1.122	0.046
FA06	U	238	48110.877	1.277	0.054
FA07	U	238	159180.614	5.131	0.075
FA08	U	238	57045.655	1.587	0.049
FA09	U	238	56386.8	1.564	0.049
FA10	U	238	55461.66	1.532	0.046
FA11	U	238	66611.842	1.919	0.060
FA12	U	238	57002.71	1.585	0.046
FA13	U	238	282733.261	9.418	0.128
FA14	U	238	283678.641	9.451	0.092
FA15	U	238	1171622.894	40.265	0.274
FA16	U	238	167569.231	5.422	0.067
FA17	U	238	482149.831	16.339	0.157
FA18	U	238	193883.179	6.336	0.075
FA19	U	238	156677.984	5.044	0.067
FA20	U	238	166408.023	5.382	0.067
FA21	U	238	158776.976	5.117	0.092
FA22	U	238	128273.136	4.059	0.060
FA23	U	238	153241.641	4.925	0.067
FA24	U	238	150133.964	4.817	0.067
5 ppb	U	238	155388.587	5.000	0.060

 Table 5-4. Mass Spectroscopy Results for Coal Ash Samples

Sample	Net Intensity (cps)	Concentration In Coal Ash	Standard Deviation
B1- Blank	11318.042	(ppm) 0.455	(ppm) 0.020
FA01	92776.227	2.827	0.049
FA02	49599.752	1.329	0.060
FA03	45189.494	1.175	0.046
FA04	46880.944	1.234	0.045
FA05	43634.356	1.122	0.046
FA06	48110.877	1.277	0.054
FA07	159180.614	5.131	0.075
FA08	57045.655	1.587	0.049
FA09	56386.8	1.564	0.049
FA10	55461.66	1.532	0.046
FA11	66611.842	1.919	0.060
FA12	57002.71	1.585	0.046
FA13	282733.261	9.418	0.128
FA14	283678.641	9.451	0.092
FA15	1171622.894	40.265	0.274
FA16	167569.231	5.422	0.067
FA17	482149.831	16.339	0.157
FA18	193883.179	6.336	0.075
FA19	156677.984	5.044	0.067
FA20	166408.023	5.382	0.067
FA21	158776.976	5.117	0.092
FA22	128273.136	4.059	0.060
FA23	153241.641	4.925	0.067
FA24	150133.964	4.817	0.067
5 ppb	155388.587	5.000	0.060

Table 5-5 Uranium Concentration in Coal and Coal Ash

5.5 Conclusions

5.5.1 Gamma Spectroscopy Analysis

After the initial research on the likely radionuclide concentration in the coal and coal ash, it was decided to conduct gamma spectroscopy analysis of these materials. This selection was made because this analysis is non-destructive and the 1.001 MeV photo-peak from ^{234m}Pa is an excellent indicator of the amount of uranium present in the coal because it is considered to be in equilibrium with the uranium. The decay scheme in Table 5-8 identifies the ²³⁸U decay emissions.

Nuclide	Decay Emission	Half-life	Emission Energy MeV (Yield)	Decay product	
238 U	a	4.468x10 ⁹ years	4.151 (20.9%)	²³⁴ Th	
0	u	1.100x10 years	4.198 (79%)	111	
	Ŷ		0.06329 (4.8%)		
	<u> </u>		0.09238 (26%)		
			0.0928 (28%)		
²³⁴ Th	β-	24.10 days	0.086 (2.9%)	^{234m} Pa	
-	β-		0.106 (7.6%)		
	β-		0.107 (19.2%)		
	β-		0.199 (70.3%)		
	β-		1.236 (1%)	234U	
^{234m} Pa	β-	1.18 minutes	2.281 (99%)	2010	
	Ŷ		1.001 (0.87%)	²³⁴ Pa	
²³⁴ Pa	β- (15)	675 have	0.548 to 1.244	234 []	
237Pa	γ	6.75 hours	0.062 to 1.695	²³⁴ U	
²³⁴ U	~		4.722 (28.4%)	²³⁰ Th	
	α	2.455x10 ⁵ years	4.774 (71.4%)		
	γ		0.053 (.123%)		

Table 5-6. Simple Decay Scheme	for ²³⁸ U to Short Lived Progeny
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The alpha emission from ²³⁸U creates ²³⁴Th, this radionuclide decays by beta emission to ^{234m}Pa which decays by gamma emission to the ground state of ²³⁴Pa. This gamma emission is the 1.001 MeV emission which has a photon yield of 0.837%. Because of the short halflife of ²³⁴Th and ^{234m}Pa, these radionuclides are in equilibrium with their parent, ²³⁸U. In equilibrium, the activity of ²³⁸U is the same as the activity of ^{234m}Pa. Therefore, with the determination of the activity of ^{234m}Pa by gamma spectroscopy, the activity of ²³⁸U in the ash is determined. The difficulty that appeared in this analysis was due to the low photon yield associated with the decay of ^{234m}Pa. The analysis time needed to achieve a statistically valid result for the ^{234m}Pa concentration was 300,000 seconds (83.33 hours). While this may be acceptable for single samples, it is not acceptable for 48 samples. So a more rapid analysis technique was sought.

5.5.2 X-Ray Fluorescence Analysis

In XRF analysis, uranium was determined directly as was the determination of many other metals and compounds. With XRF it is not necessary to bring solid samples into solution and then dispose of solution residues, as is the case with all wet-chemical methods. The main prerequisite for exact and reproducible analysis is a plain, homogeneous and clean analysis surface. For analysis of very light elements, e.g. beryllium, boron and carbon, the fluorescence radiation to be analyzed originates from a layer whose thickness is only a few atom layers to a few tenths of micrometer and which strongly depends on the sample material.

5.5.3 ICP/Mass Spectroscopy Analysis

In order to do a more complete analysis of the many samples that were received, even though they could not be traced to a specific location or source of the ash or coal, it was decided to do a detailed analysis of the samples by inductively coupled plasma mass spectrometry. Samples were prepared to have an approximate anticipated uranium

concentration in the ppb range. This was the most detailed of the analysis methods and provided the most comprehensive set of results with reasonable consistency except for two samples that showed signs of cross contamination (FA-15 and FA-17).

5.5.4 Comparison with Viable Sources

Three primary methods confirm the results of uranium analysis in coal and coal ash as between the values of 1.5 grams of uranium per 900 kg of coal ash and 10.3 grams of uranium per 900 kg of coal or coal ash. The coal ash analyzed in this evaluation contains uranium, but not at concentrations that are useful for uranium recovery. At the current time, uranium recovery is viable at approximately 200 grams per 900 kg of coal ash. The results are in the range of many other analysis results of coal and coal as in the United States.

5.6 Implementation

When coal ash deposits are identified as viable for recovery of its components that are identified as valuable, the methods to remove those components need to be identified and optimized. The composition of coal ash is such that recovery of uranium is possible via leaching, filtration, and solvent extraction. IAEA Technical Report Number 359 "Uranium Extraction Technology" provides for methods of removal with extraction efficiency of more than 99%. This is a research field that is periodically evaluated for application of new technologies and implementation is a

large scale operation. This document provides no new concepts for uranium recovery from coal ash.

CHAPTER 6

CONCLUSIONS AND FUTURE WORK

6.1 Radiation Safety for Radiochemistry

An important contribution of this work was creating a radiation safety program for a Radiochemistry Program in an academic setting. There were many challenges as it expanded into the program it is today and the faculty and staff provided many solutions to make controls as good as they could be with the facilities and resources provided.

Radiochemistry researchers should always minimize liquid radioactive waste as it is important to the survival of any radiochemistry program. A program should be established for the minimization of liquid radioactive waste and the elimination of mixed radioactive waste through recovery and reuse. The current system of passing the waste on for disposal regardless of its contents is not desirable from a radiation safety management standpoint. The creation of undesirable waste can be reduced through radiochemistry.

It is recommended that all of the exhaust stacks that allow for removal of potentially contaminated air from radiochemical fume hoods be HEPA filtered and monitored for radioactive material releases. This was a plan many years ago that was put on hold until the Radiochemistry Program used more material or there was an indication that releases from filtered hoods could be significant. At the current time, the Radiochemistry Program has reached that threshold and exhaust from laboratories where radioactive materials are used should be monitored for release. This demonstrates appropriate controls to regulators and answers questions about releases without extensive investigation into events that might have caused them.

In 2006, the UNLV Radiation Safety Advisory Committee decided to seek a Type A radioactive materials license as it was deemed appropriate for the future work with radioactive materials that would be done at UNLV [147]. A Type A license would help UNLV to communicate better among all Users, effect collaboration regarding safety issues, and allow a more detailed evaluation of some of the programs that have significant potential for increased risk to researchers and members of the general public. The pursuit of a Type A license should be restarted to support continued growth of the Radiochemistry Program.

Consistency and expertise is required to efficiently protect personnel using radiochemicals from inhalation and ingestion. The future administration of the radiation safety program at UNLV should consider these qualities for members of the radiation safety staff or for additions to the laboratory support staff.

6.2 Recovery of Materials from Decontamination Solutions

The recovery of radionuclides or other valuable material from a decontamination gel used to remove radioactive contamination from a surface has been demonstrated, but more testing is needed to prove the feasibility. The decontamination gel is a relatively new removal agent that has been shown to be more useful than other products used for

collection of high surface activity concentrations in the past. We use many of them each day such as masking tape and protective film to protect surfaces. However, their use in decontamination of surfaces is limited and typically causes more radioactive waste than is necessary. The gel has a benign nature, is not caustic or toxic and has low flammability. These features alone make it more desirable to use than, for example, the strippable coatings of the past that were used for surface protection or decontamination. These coatings typically had a characteristic hazard or were more difficult to work with, were not reusable, and materials could not be easily recovered.

It has been shown that a decontamination gel could be used to remove technetium from a contaminated surface and a fraction of that material could then be recovered from the gel. Using the gel for decontamination and then recovering the contaminant may be valuable in the case where a material is spilled that has great value, the gel is used to recover the spilled material and some action is then required to remove the material from the gel.

Another feature of the decontamination gel that could be valuable that has not been researched is its value in sampling. The decontamination gel encapsulates materials and appears to encapsulate the hazard associated with materials as it dries. This feature may be useful in the sampling of hazardous or radioactive materials in the environment or the area near a detonated radionuclide dispersion device. In working with the gel for decontamination, it is apparent that its use as a sampling tool could be valuable. Many properties of the decontamination gel have not been fully evaluated but its ability to encapsulate and collect loose surface contaminants is demonstrated.

Additives to a decontaminating hydrogel may increase its effectiveness in removal of fixed contamination from metal surfaces. Mineral acids with corrosive properties may be held on the metal surface with the gel and remove a fraction of the surface over some period of time depending on the concentration and type of acid used.

6.3 Recovery of Materials from Planned Disposal

Recycling of natural resources occurs whether humans participate or not. In the case of radioactive materials for use in a laboratory or for use in a nuclear reactor, the benefits from recycling radionuclides in the form of waste products such as experimental residue, used nuclear fuel, or weapons can be enormous. In general, it results in cost savings to the researcher or the general public, and reduced environmental impact because of a lower, less toxic waste volume. In small scale recycling, the benefits may not be readily apparent, the cost savings may be small and the waste reduction may be small, but the act of decreasing the volume of toxic waste in the environment is desirable. In large recycling operations, such as recovery of usable fuel from previously used fuel bundles, the saving of our natural resources is more obvious.

When work at any facility using radioactive material is completed, the majority of the time, there are materials left over and work must be

done to verify that the facility can be released without the need for special controls. In many cases too much original stock material was procured and the remainder must be disposed of as waste, held in storage for future use, or provided to other people who are doing work with those radionuclides. In the case of the 'UNLV Radionuclide Recovery radionuclides Program', established to obtain for the UNLV Radiochemistry Program, the recovery of kilogram quantities of uranium compounds, kilogram quantities of thorium compounds, and 500 milligrams of plutonium is considered a very successful procurement. The savings from disposal costs on the part of the donors was a success for their programs and the availability of these materials for the Radiochemistry Program will yield many successful research projects in the future.

In order to successfully achieve the procurement of radioactive materials from donors, solid communication and cooperation with the donating organization was fundamental. By continuing this program, the Radiochemistry Program could be successful in the procurement of even more actinides. Consideration should be given to establishing acceptance criteria to prevent receipt of materials that are not usable.

Recovery of radionuclides from planned disposal may require an acceptance criteria be established to prevent a facility from sending a waste product that is not useful. The criteria may simply be that the donated compound be in its original container or that a detailed evaluation of the purity of the compound is provided. These methods

may provide protection against receipt of a waste product. A more formal protection method may be written into a binding contract that requires the donation to be useful. This last criterion may reduce the number of donations as it has legal implications.

6.4 Recovery of Materials from Experiment Residue

The recovery of materials from waste generated in researchers' experiments may have already saved many thousands of dollars in disposal costs simply by destruction of the characteristic hazards of materials that could be classified as mixed radioactive and hazardous waste. Over the years many facilities have devised methods of dealing with a mixed waste problem by focusing on the destruction of the hazardous material, Some of the techniques used were thermal destruction, making the waste into a non-toxic chemical product, neutralizing it, or changing its chemical structure so that it is a more benign radioactive product that can be disposed of with other radioactive wastes.

The work discussed for this research focused on the removal of the radioactive material from the hazard so that the activity might be reused. This process is a partial solution to a mixed waste problem. The radionuclide concentration may be reduced, but the resulting waste product will still contain licensed material and if the hazard was not destroyed, mixed waste still exists. The future of this work is in the work of all research in radiochemistry that uses hazardous material.

Radiochemistry programs should recognize that the creation of a radioactive waste with a hazardous component is undesirable with our current technology. Putting radioactive materials that have hazardous components into storage, in the ground, or in tanks should be prevented and leaving the problem for future generations to solve has never been an appropriate solution. The end result of all future radiochemistry experiments should be a non-hazardous, minimally radioactive waste where the radioactive materials that could be reused have been removed from the residue and put into safe storage.

6.5 Recovery of Uranium from Coal Ash

The coal ash samples analyzed from an AES plant in Oklahoma do not represent ash with economic feasibility to recover uranium. When compared with documents regarding the concentration of uranium in the United States, this material seems to have similar concentrations. The availability of uranium in a form that is easy to process drives the cost that industry is willing to pay. The cost of recovery of uranium in coal ash where the concentration is in the ppm range would require chemical methods that are similar to current methods to extract the uranium, but with a larger volume of feed material. As a result, the cost to recover the uranium from fly ash drives the cost of this uranium to a higher price than that available from mining or leaching at the current time.

Table 2-1 Comparison of Radiological Controls at UNLV Before and After Establishment of the Radiochemistry Program

Radiological Control Procedures Before the Radiochemistry Program was Established	Radiological Controls Established for the Radiochemistry Program	
Type B Radioactive Materials License issued by the State of Nevada – 03-13- 0305-01. Allowance for gram quantities of plutonium and uranium.	Type B Radioactive Materials License issued by the State of Nevada – 03-13- 0305-01. Line item changes to allow gram quantities of technetium, neptunium, americium, and curium.	
Special Nuclear Material, Source Material, and other Radionuclides were allowed for research in any form with low activity limits for other radionuclides (100 microcuries).	Special Nuclear Material, Source Material, and other Radionuclides were allowed for research in any form. Limits were increased for actinides. NMSS limits imposed for control of SNM.	
Smears from all UNLV laboratories using radioactive materials were analyzed by Liquid Scintillation Counting.	Smears analyzed by Liquid Scintillation Counting for laboratories using ³ H, ¹⁴ C, by gas proportional counting for other laboratories.	
Training conducted annually for all groups without regard to their use of radioactive materials.	Training provided specifically to the radiochemistry program participants because of the increased use of actinides and high activity of technetium. Monthly newsletters specifically for radiochemistry.	
Contamination controls established to prevent removal of contamination from work areas.	Radiochemistry contamination controls are established to prevent removal of contamination from laboratories because the number of sources of contamination in laboratories increased.	
Dose rates in laboratories typically not measureable except from sealed sources or radiation producing machines.	Dose rates in radiochemistry laboratories measureable in source storage areas.	
Ventilation from laboratories where radioactive materials are used is not filtered.	New radiochemistry laboratories built with filtered ventilation. Similar controls for Biology established by Planning & Construction Group.	

Radiological Control Procedures Before the Radiochemistry Program was Established	Radiological Controls Established for the Radiochemistry Program	
Visits to most laboratories at least weekly by radiation safety staff.	Radiation Safety staff established to provide for needs of the radiochemistry program.	
Number of personnel contaminations and spills of radioactive material not a concern.	Increased number of spills and contamination spread noticeable from radiochemistry laboratories.	
Concern for inhalation or ingestion intake of radioactive materials is low. There is no formal bioassay program as part of the radioactive materials license.	Concern for inhalation or ingestion intake of radioactive materials increased as the activity of radioactive material used by some researchers approaches and exceeds an Annual Limit on Intake. State mandates formal bioassay program for the radiochemistry program. RSO incorporates program for protocol controls.	
Laboratories are primarily the responsibility of one Authorized Radioactive Material User.	Workload in the radiochemistry laboratories requires many users to work together providing supervision of many projects simultaneously. New controls to ensure coverage are provided in a "Plan of the Week Form".	
Work with radioactive materials was described in an Authorized Users application to become a User and seldom after that.	Work done by researchers in the Radiochemistry Program is reviewed each week to ensure that proper controls are considered by the researcher and the Authorized User as their research supervisor.	

Appendix B - Radiation Safety Training Program Newsletters

This appendix includes the technical content of 37 radiation safety newsletters produced as described in the main text of the dissertation. When published each newsletter was provided an identifying header and a message at the end that included contact information for the Radiation Laboratory Director. When training the instructor must be approachable and available and when people review the information later and questions arise there must be a way to resolve those questions. Indicate time of availability, office location, office phone, cell phone, etc. Make every reasonable effort to ensure that people believe you to be truthful and available to them for more information.

Newsletter 1 – Contamination Surveys

Whenever you work with radioactive materials, there is the possibility that some of the radioactive material evaded your control mechanism and may be spread to other parts of your laboratory or even outside of your laboratory. While the contamination spread is not likely to be hazardous to anyone, it may be in excess of our license conditions.

In order to prevent this type of situation from developing, we take measurements of our work areas with portable instruments or in some cases we take smears for laboratory analysis to identify the level of surface contamination (or absence of it) in our work areas.

When are contamination surveys required?

Contamination surveys are desired in each laboratory where radioactive material is used, weekly, or immediately after unsealed radioactive materials are used in an area, whichever is more frequent. If you are using high-energy beta emitters, you should also evaluate your body and clothes for the presence of contamination after each procedure.

What instrument should I use?

The instrument that you use depends on the radionuclide that you use in your work area. For example if you are using isotopes of Uranium, Plutonium, Neptunium, Curium, or other radionuclides that have strong alpha or beta emissions, you should use a Geiger counter with a thin window to evaluate the surfaces of your work area and the area around it. These radionuclides are beta emitters whose emissions can be detected with portable instruments. If you are using ³H (tritium), ⁶³Ni, or another radionuclide with very low energy emissions, you should consider use of a liquid scintillation counter (LSC) to evaluate smears of the work area and the area around it.

What is a smear and how do I take one?

A smear is a small (2 inch diameter) piece of paper or cloth that is wiped on a potentially radioactive area and then analyzed to determine the activity of radioactive material present in the area smeared. A smear is taken wearing gloves by rubbing the paper over an area of approximately 100 cm^2 (4 inches by 4 inches). If the area is very dry use a drop of water on the smear. Contamination control limits are expressed in units of activity per 100 cm^2 .

How do I know when an area is contaminated?

An area is considered contaminated if there is any detectable radioactive material in that area. It is considered contaminated above limits when the activity found on a smear is greater than the activity indicated in the following table for each type of radioactive material listed.

How do I convert count rate from an instrument to activity?

Area	Alpha Emitters	Beta/Gamma Emitters
Uncontrolled	$11 \text{ dpm}/100 \text{ cm}^2$	$110 \text{ dpm}/100 \text{ cm}^2$
Controlled	$22 \text{ dpm}/100 \text{ cm}^2$	220 dpm/100 cm ²
Restricted	$110 \text{ dpm}/100 \text{ cm}^2$	$1100 \text{ dpm}/100 \text{ cm}^2$

The conversion of count rate to units of activity depends on the detection efficiency of the instrument. Consider the following two examples:

You are using a Geiger counter with a thin window probe such as the Ludlum 44-9 and you observe a count rate of 100 counts per minute above background. If the radionuclide emits a higher energy beta particle such as ³²P, the efficiency is about 15% or 0.15 counts/disintegration. The activity is then 100 cpm/0.15 = 667 dpm. Since the 44-9 probe (frisker probe) has an area of 20 cm², the contamination level is 667 dpm/ 20 cm², or 3333 dpm/100 cm². We always convert the surface activity to dpm/100 cm² for comparison to the limits.

If you are using smears for a low energy beta emitter such as ³H or ¹⁴C, take 100 cm² smears. Each LSC has a certain detection efficiency for ³H and this is noted on your calibration documents. Usually this is around 60% or higher for ³H beta particles. If you have a count rate of 100 cpm for a certain sample (in the ³H channel of course), the activity indicated by the smear = 100 cpm / 0.6 c/d / 100 cm² = 167 dpm/100 cm².

How do I survey for activity on my body?

When surveying yourself for contamination, hold the probe so that the window opening faces the surface that you are surveying. Move the probe slowly over the surface (skin or clothing) at a distance of 1 cm or less from the surface. If the meter starts clicking faster, stop moving the probe and evaluate that area more closely. If the meter indicates a count rate more than 2 times the normal background count rate, there may be contamination present.

What should I do if I identify activity on my body?

In many cases, simple washing with water will remove activity from the skin. For clothing, it may be possible to remove the activity by applying tape to the surface and pulling it off. In some cases the activity may stick to the tape. In many cases where you are working with organic chemicals, the materials may bind to the clothing or skin. Some fraction of the material will then be absorbed into the skin and some will eventually slough off with dead skin or may be removed by further washing.

Notify an authorized user or the RSO if there is contamination on the skin that is difficult to remove or if there is clothing contamination.

The best means of preventing the spread of radioactive materials in laboratories is prevention of spills. The UNLV Radiation Safety Manual provides the following safe work practices:

1. Good housekeeping is required where radionuclides are used. Work areas must be clearly defined and uncluttered.

2. Work surfaces shall be covered to facilitate easy decontamination. Bench coverings shall be changed frequently, i.e., weekly, or whenever the covering is noticeably soiled, torn, or contaminated.

3. Locate work areas away from heavy traffic or doorways.

4. When moving radioactive solutions between approved locations, place the material within covered secondary containers that contain sufficient absorbing material to absorb twice the quantity of liquid. There are many good general rules for radiological control that I have picked up over the years regarding contamination control. Here are a few:

When you are working with liquids and there is a potential for a spill or spray of the liquid, wear a face shield and a plastic apron.

When you are surveying any area with a portable instrument, move the probe very slowly, 1 to 2 inches per second is the fastest that you should move a probe over an area. The faster you move the probe -the less likely that you are to detect the presence of activity on a surface.

In general, you don't have to be concerned about radioactive material being in high airborne concentrations unless you are dealing with higher quantities of unsealed activity (greater than 1 millicurie) or the material is volatile (such as Iodine). If there is a potential for airborne radioactive materials the work should always be done in a ventilated hood (and the vent fan should be ON).

Newsletter 2 - Dosimetry

Radiation Dosimetry is used to determine how much radiation dose we receive during our work with radioactive materials or radiation producing machines. In general, we can wear it to identify the dose that we receive from radiation exposure or to prove that we have not been exposed to radiation higher than background levels.

The dosimetry that is used at UNLV is from a company named Landauer and the type is Luxel.

When is dosimetry required?

It is required that we wear dosimetry when the RSO has determined that it is possible that you could receive in excess of 10% of the Nevada Administrative Code (NAC) limits for radiation dose. All persons <u>working</u> <u>with radioactive material</u> in the laboratories at the HRC are trained in radiation safety and issued dosimetry for 2 month monitoring periods.

Dosimetry is required for work with most radioactive materials at the HRC because the potential exists for someone to receive a dose in excess of 10% of the limits. However, you should be aware that no one working in the HRC in the past 5 years has received a dose in excess of 10% of the limits. Also, it is very unusual for people working in the HRC radiochemistry laboratories to receive any exposure at all!

One great thing that dosimetry provides is a feeling that because we are doing things right, we don't receive measurable dose – and the dosimetry results prove it.

How does the Luxel dosimeter work?

The Luxel optically stimulated luminescence (OSL) dosimeter measures radiation exposure due to x-ray, beta, and gamma radiation using a thin layer of aluminum oxide. After use, the aluminum oxide is stimulated with a laser and produces luminescence in proportion to the amount of radiation dose that the badge received. The Luxel dosimeters have several advantages over the monitors of many other manufacturers, the Luxel dosimeters are:

more durable, water resistant, more sensitive, doses possible down to 1 mrem, more accurate, allow determination of whether or not the exposure was static.

What are the UNLV Occupational Dose Limits?

The annual limits for exposure to ionizing radiation at UNLV are the same as those published by the State of Nevada, 5 rem per year Total Effective Dose Equivalent.

UNLV also has an administrative control level of 500 mrem/year, investigation is required if any person exceeds 83 mrem for the two month monitoring period. This is 10% of the annual limits stated above.

What if I lose my assigned dosimeter?

The Luxel dosimeter is the legal means for determination of your dose due to occupational exposure to ionizing radiation at UNLV. If the RSO does not get your dosimeter back at the end of the monitoring period, a dose estimate is made to determine your dose based on the work that you did or the dose determined from another person doing the same type of work for the same time period.

If you turn in a lost dosimeter report, the RSO will issue you another dosimeter. There is no penalty associated with losing the dosimeter. But, if you lose a dosimeter and do not turn in a lost dosimeter report, the RSO may not issue you a new dosimeter and will put a hold on your records with the registrar until either your dosimeter is returned or a lost dosimetry report is provided so that the RSO can complete your dose records for you.

What if I'm Pregnant?

If you discover that you are pregnant and you declare your pregnancy in writing to the Radiation Safety Officer, RMS will provide two dosimeters to you - one for monitoring you and one for monitoring your future child. The limits for exposure of your unborn child are more restrictive (500 mrem/ 9 months) than your exposure limits. Depending on your job and

past radiation exposure history, the Radiation Safety Officer may recommend that you be reassigned to a job with less exposure. The Radiation Safety Officer keeps exposure records for your badge and for your fetal badge.

How do I get a report of my exposure?

While you are monitored for radiation exposure by UNLV, the UNLV Radiation Safety Office maintains your exposure records. After you are officially hired by another company, your records are maintained by their radiation safety office. Newsletter 3 – Training

One of the most important aspects of radiation safety is training all people that will be working with radioactive materials or radiation producing machines. Without a fundamental knowledge of radiation protection, people would not know how to protect themselves from radiation emissions or prevent the spread of contamination.

When is training required?

It is required that every person that will be exposed to radiation associated with radiation producing machines or radioactive sources at least annually.

What courses are required?

There are three basic courses for radiation safety at the Harry Reid Center. The course sessions that you attend depend on what your job is and whether you will be working with sealed sources of radiation and radiation producing machines, unsealed sources of radioactive material, or working in the laboratories but not working with radioactive materials.

If you are a radioactive material user or assistant researcher and work with radioactive materials in liquid or other unsealed form, you will receive unsealed sources training.

If you work with sealed sources or x-ray machines, you will receive radiation producing devices training.

If you require access to the laboratory areas but do not work with radioactive materials, you need to attend the Basic Radiation Safety course.

How will I know when to attend training?

You will be notified by the Radiation Safety Officer or by the Radiation Laboratory Director when training is required. Failure to attend training after two notifications will result in loss of privileges to use radioactive materials, to operate radiation producing devices, or to enter areas where radiation is produced.

What if I miss the training?

If you do not attend training, you should not enter areas where radioactive materials or radiation producing machines are in use. If you miss the training you should reschedule a session with the Radiation Safety Office.

Is Computer Based Training Allowed?

No, computer based training for initial or re-qualification training for work with radioactive materials or radiation producing machines at the HRC is not allowed. If you take the computer based training provided by the RMS department, you will also have to take the radiation safety course offered by the Radiation Laboratory Director.

Does Training from Other Facilities Count?

No, training at other facilities does not qualify radiation workers at UNLV.

Newsletter 4 – Radioactive Waste

When radioactive materials are used in an unsealed form, radioactive waste is usually created. Typically, this waste is laboratory supplies, protective clothing, pipettes, liquid scintillation samples, and the vials that the radioactive material came in.

We have several important rules for radioactive waste disposal that anyone working with unsealed radioactive materials must know and abide by.

How do I dispose of radioactive waste?

All radioactive wastes are collected by the Radiation Safety Office for disposal. If you have wastes that should be removed from your laboratory simply notify Tom or Trevor, and we will package the waste and call the Radiation Safety Office to schedule a waste pickup. Appropriate and legible records of waste content must be in order and accompany the waste.

Where do the wastes go?

Radioactive wastes from laboratories are transported to the Radiation Protection Laboratory for evaluation, packaging, storage for decay, or eventual transport to a licensed radioactive waste site.

The exact handling, packaging, and destination site for the waste depends on the type of material, radionuclide content, radionuclide concentration, and dose rate from the material.

How do I segregate radioactive waste in my laboratory?

For laboratory disposal, there are basically 7 types of radioactive waste as follows:

- 1. Short-lived solid radioactive waste. This waste consists of materials contaminated with radionuclides that have a short half-life (less than 90 days), such as ³²P, ³⁵S, ¹²⁵I, etc.
- 2. Long-lived radioactive waste is solid waste that contains radionuclides with half-lives greater than 90 days, such as ³H, ¹⁴C, ²³⁹Pu, ²³⁸U, etc.
- 3. Liquid waste can be the remains of experiments that are liquid and contain concentrations of radioactive material. If you will be generating liquid waste you should discuss your process with the Radiation Laboratory Director or the Radiation Safety Officer.
- 4. Mixed waste is radioactive hazardous waste. It contains two components: 1. a radioactive material and 2. a hazardous material. – Do not create a mixed waste without written permission from the Radiation Safety Officer and approval of the Radiation Safety Advisory Committee.
- 5. Radioactive bio-hazard waste. As the name implies this waste is both a radioactive material and a bio-hazard. Before delivery of such waste to the RSO, the bio-hazard must be removed through autoclaving. The RSO should be involved with the autoclave process for radioactive waste. Bags containing the waste and waste records must indicate that it has been autoclaved.
- 6. Radioactive needles, razor blades, broken glass, or other puncture or cutting hazard. Do not include these with any other type of waste package them separately in a labeled sharps container.
- 7. Other waste for other types of waste (those not fitting the definitions above); please discuss options with the Radiation Laboratory Director or the Radiation Safety Officer.

Important Information and Directions

You should use a different container for each type of radioactive waste that you will create. There are many reasons for this, so let me explain a few that are very important:

Do not mix long lived and short lived wastes because short lived wastes are stored in the RPL until they become non-radioactive and can be disposed of in normal trash.

Do not mix hazardous and radioactive waste because mixed waste is approximately 20 times as expensive to dispose of as radioactive waste. Also, special handling of this waste type is required.

Do not throw non-radioactive waste in radioactive trash. All waste in a radioactive waste container is treated as radioactive and this will unnecessarily increase costs of radioactive waste disposal.

Do not place containers of liquids in bags. They might leak and cause a serious spill of radioactive materials onto non-radioactive materials increasing waste volume and associated cost of cleanup.

Do not place sharps (needles, razor blades, broken glass) into any radioactive waste bags. They may cause puncture injury to persons carrying the waste or break through the bags to cause a spill of radioactive material.

How does the RSO keep track of radioactive waste?

Each user of unsealed radioactive sources that creates radioactive waste must document this by using a Waste Disposal Inventory, RSO form number 3 or equivalent method approved by the RSO. Each time material is put into a radioactive waste container, the appropriate waste inventory log should be updated. Records must be legible.

The RSO must take the log from each user as the waste is picked up and provide an accounting of the radioactive material in each radioactive waste container that we send to a licensed radioactive waste site. The waste log from each user must be accurate regarding what radionuclides and the activity of each radionuclide in the waste.

How much does it cost to dispose of radioactive waste?

It really depends on the type of waste but a general figure that could be used to determine the cost of bulk solid radioactive waste disposal is approximately \$1000 to \$2000 per drum including packaging, handling, waste site fees, regulatory agency fees, and transportation charges.

With everyone's help we can reduce the amount of radioactive waste that is generated. Think about what you are throwing away as radioactive. Is it really radioactive?

High activity sources and high concentration waste may significantly increase the cost associated with disposal or transportation or both.

Of course the most important aspect of radioactive waste disposal is associated with its final resting place. Ensure that radioactive waste is properly controlled, properly disposed, and does not represent a hazard to anyone else. Newsletter 5 – Security of Radioactive Materials

Areas where radioactive materials are used or stored must have special security precautions to prevent unauthorized exposure of untrained personnel to radiation and also to prevent theft of the material.

The State of Nevada Radiological Health Office and the Nuclear Regulatory Commission have increased their efforts to ensure that facilities using radioactive materials properly control and secure those materials.

What if the sources are very low in activity?

Many sources of radioactive material at the HRC are very low in activity, however, some are not. The radiation hazards are minimal to trained people, but the degree of regulatory concern is significant. All radioactive sources must be secured when not in use. If we don't live by the rules, we can lose our authorization to use radioactive materials.

How should sources be stored?

When not in use and under the direct supervision of an authorized User of radioactive materials, they must be locked in a secure location.

The UNLV policy for radioactive material security is as follows:

All areas where radioactive materials are used shall be posted with a "Caution Radioactive Materials" sign. All radioactive materials shall only be used by trained personnel. All personnel working in a laboratory where radioactive materials are in use shall be aware of the presence of the material in the laboratory. All radioactive materials not in use shall be in a locked container.

If at any time, radioactive materials are identified as missing from the laboratory, the RSO shall be contacted immediately. Contact numbers for the RSO and Alternate RSO are posted in each laboratory using radioactive materials.

Why is security of low level sources of radiation important?

Although low activity radioactive sources have virtually no potential for health effect, if they are handled correctly, public fear of radiation has caused regulatory agencies to provide enforcement actions that minimize public outcry.

These actions are intended to prevent untrained persons from getting radioactive materials so that fear of perceived health effects is also minimized.

What if sources are missing?

Radioactive material users should always be aware of the status of radioactive materials that have been trusted to them by the RSO and the State. If you identify that one or more radioactive sources are missing, search the area, notify the radioactive material user – if it is gone, the user should call the RSO immediately.

Newsletter 6 – Contamination Control

Radioactive contamination is radioactive material where it is not wanted.

Control of radioactive contamination in the laboratory is the responsibility of each Radioactive Material User at UNLV. In controlling the spread radioactive material we minimize the potential for intake and reduce external dose rates in the laboratory.

Contamination control is discussed here for all personnel who enter the HRC laboratories to enable an understanding of the radioactive material user's responsibility.

Things you should do to maintain control of radioactive material:

- 1. Delineate a work area in your laboratory. Ensure that the area is not subject to high personnel traffic that may cause items to be touched or knocked off of the laboratory bench.
- 2. Wear protective clothing (gloves and laboratory coat) to prevent the spread of contamination.
- 3. Complete surveys each time that you use radioactive materials to ensure that no material is spread to other parts of your laboratory. Surveys may be done by direct monitoring for some radionuclides but must be done using smears and liquid scintillation counting for others.
- 4. Ensure that you monitor any items that are taken out of the contaminated area. Remember, anything in that area is suspected of being contaminated until proven not to be.
- 5. There must be no food, drink or cosmetics stored or used in any laboratory where there exists a potential for intake of radioactive or hazardous materials.
- 6. All containers for radioactive or hazardous materials must be labeled appropriately and should not be food or drink containers.
- 7. Alpha emitting radionuclides and volatile radioactive materials must be maintained in sealed containers to prevent an unknown spread of contamination. These items can escape an open container and cause contamination outside the container if just left open.
- 8. Work areas should be covered with an impermeable material to protect the work bench from getting contaminated from small spills.

- 9. If the technique used for transfer of radioactive materials to your samples causes splashing or splattering you are doing something wrong. Use a technique that prevents these phenomena.
- 10. When transferring materials from your work area to an analysis area or other location, ensure that the material cannot spill. Always use a secondary container with enough absorbent to contain twice the amount of liquid in the container.
- 11. Remember, gloves are not just to protect you from getting materials on your skin. They are also to prevent the spread of contamination from one area to another. If you suspect that your gloves are contaminated, don't touch other items, change your gloves. Only wear your gloves when handling potentially contaminated materials. Take them off to handle materials outside the contaminated area.
- 12. Never leave a container of radioactive material uncovered. Remember Murphy's law, only vials that are uncapped will fall over.
- 13. If you use a detection instrument, have it turned on and adjacent to your work area so that you can check your hands or items moved from the contaminated area to the clean area frequently.
- 14. Keep the laboratory area clean. Cleanliness is good radiological control.

Newsletter 7 – Transporting Radioactive Materials at UNLV

Whenever working with or transporting radioactive materials you must ensure that the samples or sources are protected from damage or spills. The best ways to do this is to provide a secondary container for the material, select a path for transportation that is not crowded or difficult to get through, and ensure that someone else knows that you are transporting radioactive materials in case you need help.

What are the requirements for transporting radioactive materials?

Radioactive materials have different transportation requirements depending on whether they will be transported on campus or off campus. The Department of Transportation has many requirements for transport of radioactive materials on public roads. These have to do with packaging, labeling, security, documentation, emergency procedures, posting, placarding, and contents of packages.

Only the UNLV RSO can authorize transport of radioactive materials from UNLV on public roads. Do not transport radioactive materials off campus without specific permission of the UNLV RSO.

Persons, who transport sources off campus must be trained in DOT requirements, understand the source control measures needed for transportation of their specific source; know how to use the transportation checklist for radioactive materials, and how to provide the DOT required documentation for their shipment.

What about transporting radioactive material between laboratories?

Whenever transporting radioactive materials between laboratories in the same building or in different buildings on campus, you should be primarily concerned with preventing a spill of the material. The radiation safety manual provides guidance as follows: Always use rubber or plastic gloves when handling radioactive material. Laboratory coats shall be worn in the laboratory and left in the laboratory. Do not wear gloves or laboratory coats out of the laboratory unless PPE is required when transporting radioactive material (ask the RSO for guidance).

Some Other Transportation Considerations

Always work over trays or work surfaces lined with absorbent material. Keep and transport radioactive materials doubly contained.

When moving a radioactive solution to another approved location, place the solution in a covered secondary container to prevent a spill.

When moving radioactive material between non-connecting rooms, fluids must be in closed containers to prevent spills and solids must be completely enclosed. The exterior container must be free of contamination. The transfer shall be made directly, such that radioactive material is not carried about more than is necessary and is never left unattended. Hard beta and gamma sources must be adequately shielded.

There are many other logical control measures that should be taken when transporting radioactive materials, consider the following:

Bring protective clothing on your transportation journey (a minimum of gloves) just in case you have a spill. Use absorbent in the transport container and bring some extra absorbent material to recover from a spill.

Carry a cell phone or have another person with you to ensure that you can contact help if you need to. In case you are transporting a sensitive source or sample and need assistance, contact the RSO for assistance. Newsletter 8 – Using Radiation Detectors

The only way to identify the immediate presence of ionizing radiation is through the use of a radiation detector. There are many different types of radiation detectors depending on the type and quantity of radiation you must measure.

What kind of instrument do I need?

The type of instrument required for your application must take into account the type of radiation emitted from the material or machine that you are using, the energy of that radiation, and how much radiation is emitted.

If you are using ³H or ⁶³Ni, the energy of the beta radiation emissions from these radionuclides is very low and a portable instrument is not very useful to detect them. Control should be by the use of smears counted on a liquid scintillation counter.

If you are using ⁹⁹Tc, ³³P, ³²P, ³⁵S, or ¹⁴C, a pancake GM is adequate for contamination control because the energy of the beta emissions can be detected with this type of instrument.

If you are using ²⁴¹Am, ²³⁹Pu, or other alpha emitting radionuclide, the most appropriate instrument may be an alpha scintillation detector for contamination control.

If you are working with high intensity x-ray machines (analytical), the most appropriate instrument is a thin wall ionization chamber. This type detector will adequately alert you to a high dose rate hazard associated with scatter from this type of machine.

If you have a new application or use for radioactive materials or radiation producing machines, please contact me. I will evaluate your situation and recommend the most appropriate instrument.

What is the basic procedure to use an instrument?

Before you use any radiation detector to measure radiation you must:

- 1. Look at the instrument for damage,
- 2. Look at the calibration sticker to see if it is in calibration (the calibration due date should be in the future),
- 3. Turn the instrument on and check the battery (there may be a battery check position on the dial, or you may have to push a battery check button),
- 4. Turn the instrument to the scale that you want to use and place the detector over the check source. You should get a response as indicated on the calibration sticker,
- 5. Write down the reading on the survey form for your laboratory.

What if the battery check indicates a less than satisfactory reading?

In this case, don't use the instrument until you change the batteries. The readings will only be valid with a satisfactory battery check.

You can replace the batteries just as in a flashlight or TV remote control. Find the compartment and identify the battery orientation. Remove the old batteries and place the new batteries in the compartment – close the battery compartment cover and once again check the batteries.

When do I have to take measurements?

You should take measurements each time that you use radioactive materials or use a different scattering configuration for the radiation producing machine. These measurements may be for dose rate or surface contamination. The instrument will give you an idea if the magnitude of dose rate or contamination where the measurement is taken. If measurements are different than expected, contact the RSO.

What is background and why must I measure/record it?

Background radiation is the radiation emitted from the materials that make up our earth and from materials and machines in our laboratories. Any response from your instrument that is not what you want to measure can be considered to be from background emissions. These measurements should be subtracted from your readings of the things that you do want to measure. The net result is the measurement that you are interested in and all measurements must be recorded.

What is the difference between dose rate and count rate?

Dose is the deposition of ionizing radiation energy in a material like our bodies. Dose rate measurements from an instrument indicate how much energy would be deposited in our bodies in a time period. The units of measurements on dose rate meters are usually millirem/hour or microrem/hour.

Count rate is typically associated with the response of an instrument designed to measure surface contamination. The "count rate" is due to the number of radiation interactions in the detector per unit of time. The unit of measurement for count rate meters is typically counts per minute (CPM).

What do the readings mean?

If you have a dose rate meter, the reading on the meter is the dose rate in the units for that meter setting (millirem/hour or rem/hour). Don't forget to use the scale multiplier. Take a reading outside of your laboratory – this is the background dose rate. If the readings in your laboratory are similar, you are only exposed to background radiations. The background dose rate is usually less than 20 micro-rem/hour in our buildings and outside. If you have dose rates in excess of 1 millirem/hour and the RSO is not aware of your operation, please contact him or her. You may have a condition that warrants further control measures.

If you have a contamination measurement instrument such as a pancake GM detector, the reading indicates CPM or counts per minute. Take a reading on a surface outside of your laboratory; this is background (typically between 20 to 200 CPM for a beta/gamma instrument and 0 to 20 CPM for an alpha instrument.

How do I convert CPM to surface activity concentration?

The conversion of count rate to surface activity is dependent upon the type and energy of the emissions from the radionuclides that you use in your laboratory. The detector used will have different detection efficiencies for the emissions from different radionuclides. This efficiency is indicated in percent or in counts per disintegration.

Consider that the count rate from a smear is 2000 CPM, the radionuclide

is ¹⁴C, background is 100 CPM and the detector is a Ludlum 44-9 probe. The detection efficiency for this detector, for this radionuclide's emission is approximately 10% so the surface activity "seen" by the detector is approximately:

A = (2000 CPM - 100 CPM) / 0.1 C/D

A = 19000 DPM.

19,000 DPM /60 Bq/DPM

A = 317 Bq

Newsletter 9 – Laboratory Access

Laboratory access to the radiochemistry laboratories is strictly controlled via proximity cards and keys to provide security for our instruments, radioactive materials, and experiments.

Who has access to the radiochemistry laboratories?

General access to the laboratories is authorized by the Directors and Primary Investigators of the HRC. People allowed access include faculty and staff of the Nuclear Science and Technology Division, students in the radiochemistry program, safety personnel who may provide an emergency function, and visitors from other universities or national laboratories working with the radiochemistry program.

CAUTION

Do not allow anyone that you do not know to use your proximity card. Each card provides information to the proximity card tracking system that identifies the card used to open any door. If you loan your card to someone else and they are not trustworthy – you are responsible for any loss of material, loss of instrument function, or damage to the laboratory that was entered.

How are keys and proximity cards controlled?

The Radioactive Materials Laboratory Manager is responsible for proximity card access to the laboratories through coordination with the lock shop. The Facility Director controls key access to any part of the Center. Laboratory access using keys is only allowed for emergency purposes or special access to rooms that do not have proximity access.

What if I do not have a proximity card and need one?

Contact your supervisor and have them submit a key or proximity card request. If they need assistance, then have them contact the RSO, or Laboratory Manager. If your request is granted, a card will be delivered to you within a week. If it is not, then you may contact the Laboratory Manager to find out why.

What if I need access to another room that was not on my original authorization?

Similar to the authorization for a proximity card, contact the Laboratory Manager and indicate your need. The need will be discussed with your supervisor and you will be notified of the decision regarding your access to the room.

What if someone else needs access and I have it? Can I let them in?

If someone that has access to the laboratories needs something from a laboratory that they do not have access to, then consider the need and provide access if the request is reasonable. However, if you allow someone into a laboratory, ensure that you accompany them for the whole time that they stay – otherwise don't let them in. All authorized people should be very cautious to only allow access to people that they know and trust.

What if someone is outside of the laboratories and doesn't have a proximity card?

If the person does not have a proximity card and you don't know them as a part of the program, then have them contact the Authorized User for the laboratory or the Laboratory Manager. Do not let anyone into the laboratory area unless you absolutely know that they are allowed to be there.

What if a maintenance technician needs access to fix something in the laboratories?

In the case of an emergency, let them in and stay with them so that they can check themselves out of the area. Be sure that our contamination control measures are always followed. Contact the Laboratory Manager immediately.

If it is not an emergency, then do not allow them to enter, contact one of the Authorized Users so that the person can be escorted into the area and assist them if equipment needs to be moved or turned off and so that the can check themselves out of the area. The Laboratory Manager is the primary contact for work in the laboratories.

What if I find a door to one of the laboratories open or unlocked?

If a door is open and should be closed, close it. If a door is unlocked that should be locked, then call the laboratory manager and let him/her know about the door.

What if I see someone in the laboratory area that I have never seen before?

In this case, introduce yourself and find out who they are. If they don't belong in the laboratories, then either ask them to leave or contact the Laboratory Manager to help them find their way out of there.

Some Final Words:

Every person who does work in the Radioactive Materials laboratory area is allowed to work there based on the fact that they received training and understand that work in the laboratory is governed by a radioactive materials license that defines controls. Compliance with the rules is not an option, it is a necessity!

The risk associated with allowing unauthorized people to enter laboratories without controls cannot be understated. The ability to do the type of research that is allowed here is based on the ability to meet the conditions of the radioactive materials license. It is up to each person authorized to work in the laboratories to ensure license compliance so that research can continue. Newsletter 10 – Personnel Contamination

Personnel decontamination methods should be of interest to all personnel using radioactive materials. If you are an "Authorized or Assistant User", the people that you supervise are under your direct care and as such may need your assistance for decontamination. If there is any instance of personnel or clothing contamination ensure that there is appropriate documentation of the event.

The most important response will ensure that the contaminated person is not injured by the material either as a result of its chemical or radiological properties. In most of our laboratories the chemical properties of the materials handled will present the most important hazard. If personnel are contaminated with chemicals, ensure that there is immediate response to any event that may cause injury.

In documentation identify the people involved, the radionuclide(s), chemical form, activity, body location, skin area, and other information pertinent to estimation of skin dose as a result of this occurrence. If you have a camera, get a picture.

Please review this newsletter to identify the proper technique for decontamination and your responsibilities should you, your assistants, visitors, or students, experience personnel contamination.

Skin Contamination: Your Own or Someone Else's.

Upon finding contamination on the skin, use the proper survey instrument for the radionuclide present to determine the highest contact response rate and the highest response rate at 10 cm from the contaminated body location. Use a Geiger counter, gas proportional detector or scintillation detector, as appropriate and note the meter readings (measured in counts per minute CPM) and the estimated size of area that is contaminated.

Do not use abrasive soaps or brushes to remove the contamination. This may remove the protective layer of the skin, opening up the body for intake.

Wash the contaminated area with soap and **warm water**. Cold water will constrict the pores of the skin making it more difficult to remove the contamination. However, hot water may expand the pores of your skin that may allow the contamination to further penetrate the skin surface.

Notify the Radiation Laboratory Director or the RSO as soon as possible.

After the first washing, re-survey the contaminated area, noting the response and determine if the decontamination has caused a reduction in the response of the meter.

If there has been no reduction, **<u>do</u> not** proceed with further decontamination. Wait for the Authorized User or the RSO to assess the contamination and prescribe any further actions.

If the contamination has been removed, record the information required on form RSO-D1. Notify the Authorized User and the RSO of the event and ensure that you have documentation of the individual's name, date, the model number of the instrument used, the maximum response of the instrument used, duration of activity on the skin, location where the event occurred, and reason for the contamination.

If the first decontamination attempt was somewhat successful, conduct another wash (do not exceed two washes without the Authorized User or the RSO present). After the second wash re-survey the area with the appropriate contamination survey instrument and record the instrument response and information similar to the previous paragraph.

After the contamination has been removed, the information will be sent to the RSO who will document a dose assessment. Knowing the activity of the material that was on the skin and the time it was there will greatly assist in RSO's efforts to accurately estimate a skin dose.

Remember, contamination of the skin can be avoided with good laboratory practices as well as using the appropriate personal protective equipment such as laboratory coat, gloves, etc.

Clothing Contamination

Remove contaminated items immediately. Monitor the skin under the contaminated clothing. If skin is contaminated, use the skin decontamination procedure. Place all clothing items that are contaminated in a bag and label with the individual's name, date, radionuclide used, the type of meter used (model and serial number), and the count rate observed.

Notify the Radiation Laboratory Director or the UNLV RSO as soon as possible.

Contamination of the Eyes

NOTE: Chemical contamination of the eyes is the greatest threat to loss of sight and must be of primary concern.

Immediately flush eye(s) with water. Evaluate the results of the flushing periodically to determine its effectiveness. Do not flush more than twice without one of the HRC Users or the RSO present. Each flushing should not exceed 2 minutes.

If there is apparent damage and sight is not immediately restored, notify a physician! Call 911 for emergency assistance. Newsletter 11 – Protective Clothing

Protective clothing is worn when working with radioactive materials to prevent contamination of the skin and minimize the spread of contamination from the work area.

When working with radioactive materials in the radiochemistry laboratories, always wear your laboratory coat, gloves, and safety glasses as the minimum PPE (Personal Protective Equipment). Consult your User for additional requirements.

The UNLV Radiation Safety Manual provides the following guidance for protective clothing use (page 21):

"F. PROTECTIVE CLOTHING POLICY

- 1. Personnel working in areas designated on laboratory maps where radioactive materials are in use or stored must wear protective garments. Open toed shoes and sandals are not permitted. The usual laboratory coat and disposable gloves are considered minimum fulfillment of this requirement. Persons working with greater than 1 mCi of an unsealed source of radioactivity must cover their legs with pants or a long skirt to protect them against absorption of activity in the case of a spill. Additional protective garments may be required by the User or the UNLV RSO.
- 2. Maintenance/custodial personnel shall receive access clearance and protective clothing requirement instructions prior to entering and working in designated radioactive material storage and use areas."

Protective clothing is the responsibility of the Authorized User of radioactive materials. If there is ever a question about the need for more than the minimum requirement the appropriate User for the material that you are using should be contacted.

Every effort must be made to use engineering controls to ensure that personnel are not exposed to unnecessary levels of airborne radioactive materials. In the unusual case where engineering controls are not available or cannot provide adequate control, respiratory protection may be required. If respirators are required, you must be medically evaluated to ensure that the respirator will not damage your respiratory system, you must be fit tested, and you must be trained to use the specific respirator that you will wear.

Check Your Protective Clothing Before Wearing It!

Any time that you will be wearing protective clothing; take the time to ensure that it will provide the proper protection.

For gloves, check to ensure that they do not have holes and are not degraded to where they will develop holes or tears when you wear them.

For laboratory coats, leg covers, or coveralls, ensure that they do not have holes or rips in the fabric. If you are using liquids, wear a fabric that will not allow transmission of the liquid to you or your clothing. Plastic clothing or Tyvek[®] will usually provide this protection.

For face masks, ensure that they do not obscure your vision as a result of scratches in the mask. Also, ensure that the head straps are adequate to hold the mask firmly in position over your face.

For respirators, check to ensure that the mask is clear, the straps are not worn or abraded, the inlet and outlet valve work properly, and you get a tight fit each time that you wear one. Newsletter 12 – Ventilation of Radiological Laboratories at HRC

Laboratories where radioactive materials are used or stored have ventilation requirements to minimize or prevent inhalation or ingestion of radioactive materials. This ventilation may be provided by a chemical fume hood approved for the radionuclides and/or chemicals used in the laboratory.

The following are details of the UNLV Chemical Fume Hood Guide:

Facilities Maintenance Personnel shall contact the person responsible for the laboratory to schedule service, and shall not enter a laboratory or area posted for radiation safety unless accompanied by the authorized user or Radiological Safety Office personnel. Written Radiological Safety Officer (RSO) approval shall be posted on a radiological hood prior to servicing.

Any person working on or assisting with fume hood repairs in radioactive materials laboratories must be under the direct supervision of the responsible laboratory director or a member of the Radiation Safety Office staff. The only exception is work that has the specific written approval of the RSO.

The RSO shall routinely monitor fume hoods during inspections, routine surveys, at the request of the authorized user, or prior to scheduled repair or maintenance.

The authorized user of radioactive materials shall control radioactive materials used in hoods as follows:

Radioactive materials shall be secured against unauthorized removal, and all surfaces decontaminated and surveyed to assure that no contamination remains when unattended. This is to ensure that no radiation hazard is present during routine non-scheduled maintenance activities. The authorized user or his assistants shall promptly notify the RSO of any spill, accident, or any operation which may have contaminated the hood or released any contamination through the hood to ductwork or air in an uncontrolled area. THIS IS IMPORTANT!

The ventilation systems in the radioactive materials laboratories discharge through High Efficiency Particulate Air (HEPA) systems.

Surveys

If you do work with radioactive materials in a hood you should recognize that some materials may easily spread from the work location to other parts of the hood because of turbulence and air movement in the hood.

OBJECTS SHOULD NOT BE PLACED AT THE REAR OF THE HOOD WHERE THEY MIGHT IMPEDE AIR FLOW.

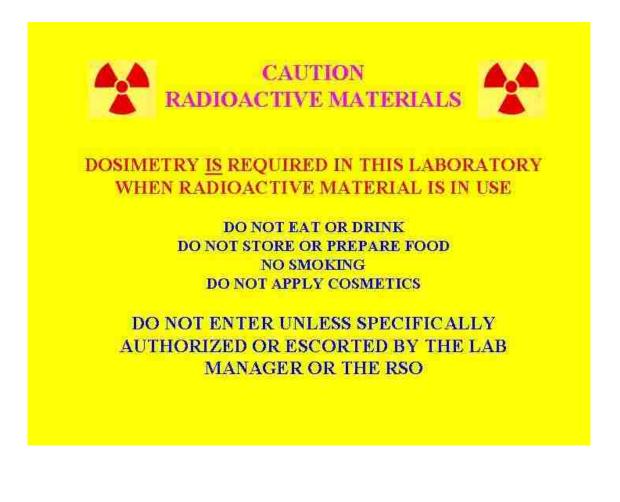
Smear surveys should be done in the hood right after any use of radioactive materials especially if work is done with other materials that are not radioactive.

A hood is not necessarily a shield for your material and high activity gamma or neutron emitting sources may cause high dose rates **on the other side of the wall** that the hood is mounted on.

REMEMBER THAT ANY TIME YOU CAN SMELL A CHEMICAL OR SOLVENT, YOU ARE BEING EXPOSED TO THAT SUBSTANCE. NOTIFY YOUR SUPERVISOR IMMEDIATELY IF YOU THINK THAT THERE COULD BE A PROBLEM WITH A LABORATORY HOOD! Newsletter 13 - Postings for Radiation Safety

This month's newsletter discusses how areas and materials are labeled or posted to provide awareness of potential hazards. Signs are used to identify the presence of radioactive materials or radiation producing devices in rooms or buildings on campus. Labels are used to identify the radionuclides, activity, or hazard class of radioactive materials or the emission location for x-rays from a machine.

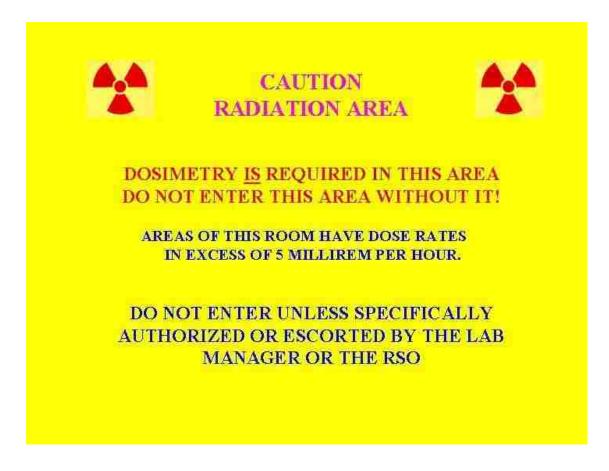
Rooms or buildings where radioactive materials are used or stored are posted with a "Caution Radioactive Materials" sign. This type of sign may have many different phrases. A typical sign on campus where radioactive materials are used may be as follows: A common feature of radiation safety signs is the trefoil, the international symbol for radiation or radioactive materials. The trefoil is usually magenta on a yellow background but may be red on yellow or black on white or silver.



Areas where the dose rate is greater than 5 mrem/hour are posted "Caution - Radiation Area". If the dose rate is greater than 100 mrem/hour, the area is posted "Caution - High Radiation Area". We do not have any High Radiation Areas or Very High Radiation Areas in the Radiochemistry Laboratories. The posting for a radiation area may be as follows:

When you are in a laboratory where a radiation area exists from a source or a radiation producing machine, be aware of the location of the highest dose rates and minimize the time that you might be close to the source or to the location where radiation is emitted from a machine.

Use time, distance, and shielding to minimize your dose when in these areas!



There may be any number of ways a source of radioactive material is labeled. A typical label indicates the radionuclide, activity, and dose rate at a specific distance from the material.

CAUTION	1
RADIOACTIVE MA	VIERIALS.
Radionuclide	
Radionuclide	
Radionuclide Activity (mCi)	
Activity (mCi)	mr/hr
	mr/hr hr
Activity (mCi) Dose Rate (contact)	
Activity (mCi) Dose Rate (contact) Dose Rate (1 meter)	

X-ray machines are required to be posted with specific wording to alert the patient and the operator of a potential radiation hazard.



Other signs and labels may be used on campus to identify radioactive materials or the existence of radiation at levels higher than normal background radiation. Be aware of the postings and labels used, the location of sources of radiation, and how to minimize your dose. If you have questions, please call an Authorized User or the RSO! Newsletter 14 – Radioactive Materials Regulations

All people using radioactive materials at UNLV are required to ensure that the regulations of the State of Nevada and our radioactive materials license are complied with. This newsletter provides you with some simple reasons why these regulations are important and the ways that compliance is typically achieved.

Why is radioactive material licensed?

Most governments require the control of radioactive materials or radiation producing devices to ensure that they are used safely and their use does not cause harm to other people or to the environment.

Radioactive material licenses require the submission of an application that describes what material is licensed, why the material is required, how the material will be controlled to prevent unnecessary exposure of people, safety procedures, emergency procedures, worker's training and experience, facility diagrams, personnel and facility monitoring information, and environmental protection.

What regulations govern work with radioactive materials at UNLV?

The State Radiological Health Section in accordance with the provisions of NAC 459.010 through 459.794 regulates radiation sources at UNLV, and has specified its own rules and regulations for the control of radioactive material and radiation producing devices (RPDs). These regulations are available at the RSO offices and on the internet at <u>http://www.leg.state.nv.us/NAC/NAC-459.html</u>.

While regulations may be subject to interpretation, they are interpreted by the regulators in the strictest possible way for UNLV because \underline{we} are expected to be working with these materials correctly and by the rules. In the words of a regulator, we set the example for our students and thus we are "held to a higher standard".

The State of Nevada issued UNLV our radioactive materials licenses. It is required that we not only abide by the regulations, but that we also conduct our work with radioactive materials in accordance with our license and license conditions.

What is a radioactive materials license?

This is a privilege bestowed upon the University to enable the use of radioactive materials for research. UNLV has two radioactive materials licenses to enable use and storage of radioactive materials and also to provide radioactive material analysis services to customers outside of the University.

The license indicates how much of each radionuclide we are allowed to have in our possession, who the Radiation Safety Officer is and what his/her qualifications are, how we will work with radioactive materials, and where we will work with these materials.

How does UNLV comply with these regulations?

UNLV has established a Radiation Safety Office to be headed by a qualified Radiation Safety Officer (RSO) who is responsible for developing and implementing policies and procedures to ensure compliance with the regulations (with approval from the State Radiological Health Section). The RSO is directly responsible to the State of Nevada in all matters of radiation safety.

The RSO is specifically indicated by name on the Radioactive Materials License as the supervisor of all radioactive materials use and radiation producing machine operation on the campus. Any operations that are considered out of compliance by the RSO may be paused or terminated by the RSO to ensure that we will achieve compliance.

The Radiation Safety Office ensures that training is provided to all UNLV personnel that use radioactive materials or radiation producing machines (X-ray machines, lasers, particle accelerators, etc.); maintains inventories of radioactive materials, evaluates areas where radioactive materials and machines that produce radiation are used, collects radioactive waste for proper disposal, provides personnel dosimetry, and maintains records of personnel radiation exposure.

What is the RSO's job?

According to the Nevada Administrative Code, the Radiation Safety Officer shall:

(a) Investigate overexposures, accidents, spills, thefts, unauthorized receipts, uses, transfers, disposals, mis-administrations and other deviations from approved radiation safety practices and implement corrective actions as necessary.

(b) Establish and implement written policies and procedures for:

- (1) Authorizing the procurement of radioactive material;
- (2) Receiving and opening packages of radioactive material;
- (3) Storing radioactive material;
- (4) Keeping inventory of radioactive material;
- (5) Safely using radioactive material;
- (6) Taking action if radioactive material is lost;
- (7) Performing surveys of radiation periodically;
- (8) Performing checks of instruments for surveying;
- (9) Performing checks of other safety equipment;
- (10) Training personnel who work in restricted areas or are otherwise occupationally exposed to radiation; and

(c) Brief management at least once per year on the usage of radioactive material at the facility;

(d) Establish levels of exposure for personnel which, when exceeded, will be investigated by the radiation safety officer to determine the cause of the exposure and methods that can be used to prevent recurrence of the exposure; and

(e) If the licensee has a committee on radiation safety, assist the committee in the performance of its duties.

What are the consequences of non-compliance?

If anyone at UNLV fails to provide compliance with regulations for the safe use of radioactive materials or radiation producing machines, we (UNLV) could lose our authorization to use these materials or machines. Any research that requires use of these materials or devices would be required to stop and perhaps continue at another University.

Usually before a regulator will take an action such as this, the offending licensee would be allowed to show reason why they should be allowed to continue working with radiation.

How do we prove that we have complied with regulations?

Compliance with regulations is demonstrated by maintaining records that show that we have: properly evaluated all radiation hazards, taken precautions to protect our personnel, taken precautions to protect the environment, and have trained all personnel to protect themselves from unnecessary radiation exposure.

If you are working with radioactive materials, the surveys that **you** do of **your** work area and laboratory are very important to demonstrate that we provide compliance with the regulations. Please ensure that these are done and documented regularly.

All people working with radioactive materials or that provided services to laboratories where radioactive materials are used must ensure that their training and experience provide for the safe use of these materials. The intent of our radioactive material licenses are to provide for the protection of ourselves and the people that we work with, always keep these thoughts in mind when working with radiation producing devices or materials. Newsletter 15 – X-ray Producing Devices

Two new x-ray producing machines were obtained by the Radiochemistry Program in 2007 to provide new sample analysis capability for the nuclear group.

These machines are located in the radiation laboratories and provide for:

X-ray diffraction analysis of powders, and X-ray crystallography analysis of single crystals.

X-Ray Diffraction (XRD) is a non-destructive technique for analyzing a wide range of materials, including fluids, metals, minerals, polymers, catalysts, plastics, pharmaceuticals, thin-film coatings, ceramics and semiconductors. Throughout industry and research institutions, XRD has become a useful method for materials investigation, characterization and quality control. Example areas of application include qualitative and quantitative phase analysis, crystallography, structure and relaxation determination, texture and residual stress investigations, controlled sample environment, micro-diffraction, nano-materials, laboratory, and process automation, and high-throughput polymorph screening.



Figure 1 - Powder XRD

Chemical Crystallography

Chemical Crystallography provides accurate and precise measurements of molecular dimensions in a way that no other science can begin to approach. Historically, single crystal X-ray diffraction was used to determine the structure of what was thought of as "small molecules". Twenty years ago, it was possible to solve structures with an average of only 100 non-hydrogen atoms. However, thanks to developments in hardware and software, the upper limit has risen to about 500 and solved. recently. even а 1000-atom structure was Chemical crystallographers study compounds which are both of chemical and biological interest - new synthetic chemicals, catalysts, pharmaceuticals, natural products, minerals... and more.



Figure 2 - Single Crystal XRD

What are the radiation hazards associated with these machines?

X-ray diffraction machines generate very large numbers of very low energy x-rays. The x-rays interact with the material under investigation to provide information about the structure of the material, so as you can imagine, the primary beam dose rates are very high and represent the most important hazard in the machine. This hazard is only within the cabinet of the machine and the x-ray beam is "turned off" when the cabinet is opened due to an interlock on the doors. Operators should always ensure that the x-ray beam did turn off when the cabinet was opened and never attempt to defeat the door interlocks. A survey was done when we first received the machines in order to assess the radiation levels outside of the machines during operation. The readings all indicated only a background response and no indication of x-rays penetrating the cabinet walls or doors of the machines.

Are there other potential hazards associated with this room or the XRD instruments?

Yes, some of the sample preparation equipment and some of the operation support equipment may present a hazard to researchers or people who enter an XRD laboratory.

During sample preparation for single crystal analysis, the preparer will heat up a glass rod and pull it to a very fine point for mounting the sample. The heating uses an open flame and is done on a desk inside the XRD laboratory. The sample preparer must be careful to abide by fire safety regulations and the glass sample holder is very sharp and represents a potential "stick" hazard.

General Safety in the XRD Laboratories

Everyone should immediately recognize that the XRD laboratories are very small and more than 3 people in the laboratories may present an overcrowding situation where things may get bumped or spilled. In order to minimize the probability of a spill with powders or crystals, you should minimize the amount of material brought into the room and ensure that you are working with only what you need.

Gasses are used to provide cooling to the single crystal detector (liquid nitrogen), and provide fuel for the torch (oxygen and propane). Be sure that any gas tank is properly secured so that it will not fall over and that all connections are appropriate.

Radioactive Materials in XRD Samples

If the samples contain radioactive materials, then there is a concern that a spread of contamination is possible. All areas where the samples have been prepared or have resided, should be evaluated for the presence of radioactive contamination after each use. Since the samples that are being analyzed have only small amounts of activity, the contamination will not present much of a hazard to personnel, but we do have to keep track of all material and control is necessary for license compliance. Newsletter 16 - Instrument Calibration and Periodic Checks

Portable Instruments are used in all radioactive material laboratories for control of radioactive contamination.

What is Instrument Calibration?

Calibration is simply a verification of proper operation and adjustment to ensure that the value of a response is the same as the expected response from the same energy emission. Calibration of our portable contamination control instruments is done by the UNLV Radiation Safety Office and calibration of dose measuring instruments is done at a facility certified to provide that function.

When are Portable Instruments Required?

Instruments are required in laboratories that have emissions that are detectable by portable instruments. For example, laboratories that use normal form radionuclides such as ⁹⁹Tc, ²³³U, ²³⁸U, ²⁴¹Am, ²³⁹Pu, must have an instrument in their laboratory to aid in control of radioactive contamination.

How Often is Calibration Required?

Portable instruments are required to be calibrated annually or when significant changes are made in the detector assembly or when repairs are made. Calibration is not required if only the batteries are changed or the same type GM tube is used to replace a broken GM tube.

How do I know when an instrument has been calibrated last?

Each instrument has a sticker attached to the side of the instrument case that indicates, who calibrated it, when it was calibrated, when it is due for calibration, and who the instrument belongs to.

How Often are Periodic Checks Required?

Each time an instrument is used a check must be made to ensure that it responds to radiation as it did when it was calibrated. This is a requirement in the Nevada Administrative Code. In order to demonstrate this requirement, a record of each check should be documented.

A radiation detection instrument is the only way to identify the presence of radiation or measure its intensity. You should always:

- 1. Check the instrument that you are about to use for damage.
- 2. Check the battery to ensure it is OK to use.
- 3. Check the response to ensure that it responds to radiation.
- 4. Place it in a safe and ready position for the next person.

What if the Instrument is Contaminated?

If the response of the instrument is above 100 cpm, then it may be possible that the instrument screen or detector face are contaminated. **DO NOT ATTEMPT TO CLEAN IT YOURSELF!** Please contact me so that I can clean the detector.

What kind of Documentation is Required for Instrument Checks?

Whenever an instrument is checked, that check must be documented. Remember, if you did not write it down, you did not do it. Checks of portable instruments are documented on form RSO-IC. Newsletter 17 – Background Radiation

Many of the natural elements on the earth have unstable isotopes that emit radiation during radioactive decay. The radiation emitted may be alpha particles, beta particles, or x-rays or gamma rays (photons). Because we live on the earth, our bodies are always exposed to radiation. The largest source of natural background radiation exposure to humans is terrestrial radon, a colorless, odorless, chemically inert gas that causes approximately 55% of our average non-occupational exposure.

There is another source of background radiation that we are exposed to, and it originates in outer space. Cosmic radiation interacts with our atmosphere and causes particles and rays that penetrate to the surface of the earth.

"Everything on this planet, including every living thing, is bathed in a sea of radiation from these sources. This is commonly referred to as 'natural background', 'background radiation', or more simply, 'background'" -NUREG 1501.

Wherever you are on the earth, on the average, thousands of photons impinge on your body every second, and some interact and deposit energy. In addition, we breathe, drink, and eat radioactive atoms because of natural activity in the soil, food, water, and air. Our background radiation exposure in the course of a year is approximately 360 millirem.

In addition to natural sources of radiation, people are exposed to manmade sources of ionizing radiation such as x-rays in dental and medical facilities. Manmade sources provide only a fraction of the dose that non-radiation workers typically receive from natural sources. Depending on your job in radiation work you may receive up to 5000 millirem in a year. Under <u>very</u> unusual circumstances, limits may be even higher.

Total of all Sources - Natural + Man-made = 360 millirem

This information is from Regulatory Guide 8.29 by the Nuclear Regulatory Commission - February 1996. The NCRP reported in report 160 that medical exposure increased to the point where it causes an average of 50% of the annual exposure of the people of the United States (2006). From this information, the total annual dose from all sources is 720 millirem.

What effect does background radiation have on my job?

The primary effect that background has on our ability to control radioactive materials is the nuisance that it provides when we monitor an area or a person. Background radiation causes a response on our instruments that is not related to the activity we are trying to control. If this response is minimized, we can more accurately measure any low activity material that may be present in the radioactive materials work area.

For example, consider that you are working in an area with concentrations of radioactive material that are very low. The bench that you are working on is made of a mineral that contains low levels of natural activity and the walls and floor of the building are made of concrete (that also contains low levels of natural activity).

When we place your dosimetry on a rack near your work area, the dosimetry is exposed to natural emissions from radioactive material in the floor and walls of the structure. We also place control badges that monitor all emissions that the rack is exposed to, so that we can subtract the background exposure from the badge you are wearing to get your occupational exposure (the dose that the badge accumulated while on your body). If the control is lost or not turned in with the badges that we wear, the dose on the control badge cannot be subtracted so the resultant dose on our badges is higher.

So you see, background emissions do affect our ability to monitor emissions from the materials we work with. The typical control badge on a dosimetry rack at UNLV accumulates approximately 15 to 45 millirem during a monitoring period (2 months).

How can I minimize the response due to background when I am surveying my work area?

The best way is to ensure that usable radioactive materials are not in the work area when you survey. This will reduce the number of emissions that may cause a response in your detector. Another way is to shield your detector from the work area. Let's say you are using the portable instrument to count your smears. If the detector is shielded from the work area, the response from source emissions in the area will be lower and your ability to detect low level activity on the smears will be enhanced.

You use the same methods to minimize background exposure to your equipment as you use to protect yourself from external radiation exposure; time, distance, and shielding.

Is Background Radiation Harmful?

Background radiations, alpha, beta, gamma, x-ray, and subatomic particles, are the same as radiations emitted from man-made radioactive materials or radiation producing machines. Since humans have always been exposed to radiation, it may be an important part of our existence to be exposed to low level radiation.

To say background radiation is "harmful" would be difficult since it is an effect associated with a phenomena that may actually sustain life or even make biological (human) systems stronger. However, for radiation protection purposes, we consider that any radiation exposure may be harmful and that all exposure to radiation should be justified.



Many of the radionuclides used in research at HRC mav require analysis ('counting") using а Liquid Scintillation Counter (LSC), and use of the LSC is very common in surveys and contamination control. Sample preparation may be simple or complicated, but operation of the instrument is usually straightforward. The activity that can be detected is very low, and samples with very little activity vield can excellent data.

How does a liquid scintillation counter work?

A liquid scintillation counter (LSC) is a simple device that measures the amount and the energy of light pulses given off by chemicals after excitation by energetic particles (radiation). The sample is dissolved, suspended, or otherwise thoroughly mixed with a solvent and a "fluor"— from "fluoresce" (another organic compound), and other additives such as surfactants and emulsifiers. These chemicals may or may not be regulated as hazardous.

The radiation imparts energy to the solvent, which in turn imparts energy to the fluor. The fluor can return to a stable state by emitting light, and it is these tiny light emissions that are detected by photomultiplier tubes in the counter. The amount of light emitted by the fluor is proportional to the amount of activity in the sample, and the energy of the light produced is proportional to the energy of the radiation. Thus both qualitative and quantitative analyses are possible. The photomultiplier tubes (PM tubes) which detect the tiny light emissions are very heavily shielded, as is the sample compartment, to reduce the instrument's response to background radiation. The instruments are thus very heavy. Great care should be taken in moving these instruments.

All work at this time in the Radiochemistry Laboratories use an environmentally friendly cocktail from Perkin Elmer called Ultima Gold. This cocktail has a much higher flash point, is non-volatile (has a very low vapor pressure), has low toxicity, is biodegradable, and is classified as non-flammable. This cocktail is recommended for all future work at the radiochemistry laboratories. If a different cocktail is desired, it must be evaluated by the Authorized User or the UNLV Radiation Safety Office.

Where can I prepare samples for the LSC?

In the Harry Reid Center, samples containing radioactive liquids may only be prepared in the radiochemistry laboratories where work with normal form radioactive a material (liquids, powders, or gases) is allowed. Samples for liquid scintillation counting should never be prepared in the LSC laboratory as a spill may affect not only your results but the results of others as well.

How sensitive are they?

Liquid scintillation counters have very good sensitivity to directly ionizing radiation (alpha and beta particles). They have moderate sensitivity to indirectly ionizing radiation such as gamma and x-ray radiation.

The background response is typically very low (less than 50 counts per minute) and the detection efficiency very high (more than 50%). As a result these detectors can detect as low as a few picocuries in a 10 minute count.

Are there safety hazards associated with their operation?

If operated properly, there are no safety hazards associated with LSC's. Some instruments have an internal calibration source, usually Cs-137, on the order of 20 to 30 μ Ci. These are well shielded and manipulated mechanically. Never try to remove an LSC source, and notify the radioactive material user or the RSO if background counts increase suddenly. The calibration source may be leaking or another source in the area may be causing this response.

Sample conveyor apparatus can move suddenly, but mechanical hazards are few. Never take the LSC apart as high voltages (~2000 volts) are required to run the photomultiplier tubes. Samples with very high dose rates should never be run in an LSC (not only for personal protection) because the photomultiplier tubes would be damaged.

Chemicals used may be toxic, flammable, corrosive, or all three, and must be handled carefully. Know your radionuclides and your chemicals, and treat both with appropriate respect. Always use secondary containers to contain spills, and always clean up any spills as soon as practical. Double check the screw caps of the vials for tightness, but do not over tighten to the point of stripping threads. Wear gloves and appropriate personal protective equipment.

What type of vials should I use?

There are a few different options here. Glass vials or plastic vials and 20 ml or 7 ml. Since we work in an analysis laboratory (MSM-242) where sample preparation is not allowed and every precaution should be taken to prevent spills, plastic vials are strongly encouraged. Also, since we would always like to minimize consumption of LSC cocktail and also minimize the creation of radioactive waste, the smaller vials are encouraged.

However, everyone has special needs for their research and that consideration is the most important. The sensitivity of the LSC for your particular needs may require the use of the larger size glass vials. It is necessary that you evaluate your needs and consider the issues of waste creation and accident avoidance.

Are there regulatory issues associated with LSC media?

There <u>may</u> be. Recall that some chemicals in the cocktails may be regulated as hazardous materials. The solvents used in some of the common LSC cocktails have a low flash point (are flammable) and are not biodegradable. These become hazardous wastes. When radionuclides are present, these materials become **mixed waste**. The pickup of these wastes starts a regulatory clock. They cannot be stored indefinitely, they cannot be treated, and they cannot be rendered non-hazardous. Disposal of mixed waste is <u>very</u> expensive.

Short-lived radionuclides can be allowed to decay, but long-lived nuclides may present a problem with the creation of mixed waste. This is not to say that these regulated chemicals cannot be used in LSC cocktails, when in the judgment of the researcher no satisfactory substitute can be found. In such a case, provision for the disposal of mixed waste should be made PRIOR to its generation, and sufficient funds be made available for disposal. There is no typical provision in the Radiological Safety Office budget for the disposal of mixed waste.

What do I do with the samples after the analysis?

WAIT!! Don't just throw your samples into a bag! Don't throw samples into the clean waste or the radioactive waste! Please place the samples in the box that the vials came in from the manufacturer.

There are some important considerations here as well. One is "can you recycle the material in the samples?" If recycling is possible, it may save many thousands of dollars of your research funds. Even 10 mg of ²³⁹Pu is \$5,000.00.

If recycling is not possible, place a waste information log on the box indicating the radionuclide(s), the activity, and your initials. **Place the vials into their cardboard racks**, and place them in the liquid waste disposal area in MSM-163. The RSO will pick up the vials and the liquid waste and the attendant paperwork.

There are many issues associated with the use of an LSC for sample analysis. However, the information collected from this incredible analysis tool can be tremendous. Newsletter 19 – Transportation of Radioactive Materials

Radioactive material has been shipped in the U. S. for more than 50 years with no occurrences of death or serious injury from exposure to the contents of these shipments.

As you can see from this table, there are thousands of shipments of hazardous material in the US each day. All radioactive shipments in the United States are regulated by the **Department of Transportation** (DOT) and the **Nuclear Regulatory Commission (NRC)**.

Hazardous Material Shipments for 1 Year		
Internationally	300 million	
United States	3 million	
DOE	<1% or 5,000 (out of 3 million)	
	[U.S. DOE NTP, 1999, Transporting Radioactive Materials]	

Since transport accidents cannot be prevented, the regulations are primarily designed to:

• Ensure safety in routine handling situations for minimally hazardous material

• Ensure package integrity under all circumstances for highly dangerous materials.

These goals are accomplished by focusing on the packaging and its ability to:

- Contain the material (prevent leaks)
- Prevent unusual occurrences (such as criticality)
- Reduce external radiation to safe levels (provide shielding)

How is radioactive material packaged for transport on highways?

There are many different types of packaging used for radioactive materials, some are simple packages such as boxes or cans, more robust packaging such as tested drums or even steel canisters are used for very high levels of radioactive material.



Excepted Packaging is designed to survive normal conditions of transport.

Excepted packaging is used for transportation of materials that are either Low Specific Activity (LSA) or Surface Contaminated Objects (SCO) and that are limited quantity shipments, instruments or articles, articles manufactured from natural or depleted uranium or natural thorium; empty packaging is also excepted (49CFR 173.421-428).

Excepted packaging can be almost any packaging that meets the basic

requirements, with any of the above contents. They are excepted from several labeling and documentation requirements. Most of the material that we ship to other locations are shipped in excepted packages.

Industrial Packaging (IP) is designed to survive normal conditions of transport (IP-1) and at least the drop test and stacking test for Type A packaging (IP-2 and IP3).

Industrial packaging (IP) is used for transportation of materials with very small amounts of radioactivity <u>(Low Specific Activity [LSA]</u> or <u>Surface</u> <u>Contaminated Objects [SCO]</u>). Industrial packaging (IP) is usually metal boxes or drums.

Type A Packaging is designed to survive normal transportation,

handling, and minor accidents. They are used for the transportation of quantities of radioactive material (RAM) that would not result in significant health effects if they were released. Type A packaging may be cardboard boxes, wooden crates, or drums. The shipper and carrier must have documentation of the certification of the packages being transported.

Type B packaging must be able to survive severe accidents. They are used for the transportation of large quantities of radioactive material. A Type B packaging may be a metal drum or a huge, massive shielded transport container. Type B packaging must meet severe accident performance standards that are considerably more rigorous than those required for Type A packages.

What about labeling?

Labeling radioactive materials is as important as packaging for the prevention of problems. Markings on the package detail the proper shipping name, an emergency response identification number, the shipper's name and address and any other relevant information.

Labels are placed on opposite sides of a package to identify the contents and activity level. The label is determined by the type of material shipped and radiation levels of the package's contents. Labels also provide a hazard index to ensure correct handling. Shippers use one of three labels; Radioactive White, Radioactive Yellow II or Radioactive Yellow III. Shipments with extremely low levels of radioactivity may be excluded from labeling requirements.

In some cases, there is also a requirement for the vehicle transporting radioactive materials to have a placard on the front, rear and sides.

How do I ship Radioactive Material?

If you are asking this question, the answer is quite simply, you can't! Any person who ships radioactive material must be trained to do so. Any person who presents radioactive material for shipment or assists in the preparation of packages for shipment must be trained in accordance with 49CFR (Title 49 of the Code of Federal Regulations).

The way that you ship radioactive material is to provide your material to a trained shipper. The shipper will get all important information from you and prepare the package and its labeling in accordance with 49CFR. The shipper will then take the package to a transportation company (such as FedEx) and put the material on the road.

What are the Consequences of Not Shipping IAW 49CFR?

Shipping radioactive material correctly is extremely important. Proper packaging and labeling have prevented many problems from occurring and are responsible for the excellent safety record of radioactive material shipping over the years. Consider the following sign from a FedEx office:

Shipment of Dangerous Goods Statement WARNING

Federal Regulations Require Shippers of Dangerous Goods To Properly Classify, Identify, Package, Mark, Label, and Document These Items Before Offering Them To An Air Carrier.

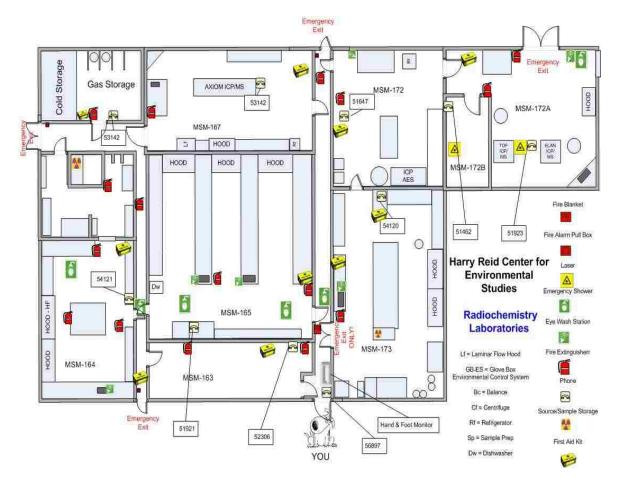


Failure To Follow The Regulations Affecting The Offering And Transport of Dangerous Goods Can Result In Severe Monetary Penalties And Imprisonment As Follows:

A VIOLATION CAN RESULT IN FIVE YEARS IMPRISONMENT AND PENALTIES OF \$250,000 OR MORE (49 U.S.C. 5124)

Offered in accordance with Federal Hazardous Materials Regulation (49 CFR parts 171 - 180) FedEx Express Corporate Safety (October 2001) Newsletter 20 - Emergency Equipment in the HRC Radioactive Material Laboratory Areas

As you work in the radiochemistry laboratories, you focus on your projects and only think of emergency equipment when you need it. However, you should be aware of what is in the laboratories, how to use it, and how to ensure that it is always available when you need it. During some recent inspections of the laboratories we found emergency equipment that was made unavailable because of actions taken by someone that demonstrated a failure to understand the need for that equipment.



The emergency equipment map is placed on the entry door to the laboratories and is located at each phone throughout the laboratories. This should serve as a continuous reminder to be aware of the location of all emergency equipment and ensure that you do not prevent the use of this equipment by obstructing its location or depleting its resources. If any of the emergency resources that we have are not available, please contact Gary, Ken, Trevor, or me so that we can get it back.

Emergency Showers – There are six emergency showers in the laboratory area, two in MSM-165, one in MSM-164, one in MSM-167, and two in the hallways on either side of MSM-165. No one should ever block access to any of the showers. These showers are fed by a dedicated water line and are tested every week to ensure that they are available.

Eye Wash Stations – There are four permanent eye wash stations and several smaller eye wash bottles in the laboratory area. No one should ever block access to these stations by putting things in front of the handle that operates the water flow or preventing immediate access to the water.

Fire Extinguishers – There are 17 fire extinguishers placed throughout the laboratories and hallways in the first floor laboratory area. Fire extinguishers are checked weekly. If you use a fire extinguisher, please make sure that it is put aside for recharging and not put back in its "ready" position. Never change the location of a fire extinguisher.

Fire Pull Boxes – There are three (3) fire pull boxes located in the hallways between the laboratories at the location of the emergency exits.

Fire Blankets – There are three (3) large fire blankets. Large fire blankets, for use in laboratory and industrial situations, are often made of wool (sometimes treated with a flame retardant fluid). These blankets are mounted in vertical quick-release cabinets so that they can be easily pulled out and wrapped round a person whose clothes are on fire. Alternatively, they could be used to smother a small fire. The fire blankets are located at the control point adjacent to the PCM, outside of MSM-164, and in MSM-168 near the source storage room.

First Aid Kits – There are ten (10) first aid kits in the laboratory area, nine in the laboratories and one in the hallway outside of MSM-164. The contents of each kit are checked monthly. If you use the contents of a kit, please let Trevor know that it needs replenishment.

Telephones – Each laboratory has a telephone. The numbers are as follows:

This section lists the phone number for each laboratory.

Emergency Exits

There are four (4) emergency exits from the laboratory area and one designated emergency exit from MSM-173 – near the PCM. These exits are for emergency exit only. They must not be used for routine access and egress from the laboratories in order to ensure that contamination control of the facility is maintained.

Evacuation

If there is an emergency situation in the laboratory area or in case of a fire in the main building, put your work in a safe configuration and leave the area. As you exit the building, warn others of the situation and the need to leave the area. Take a contamination control detector with you. Gather outside of the laboratories near the entrance to MSM-162 if possible. Avoid contact with others before you check yourself for contamination. At this location, you should survey yourself and allow others to do the same so that you can all verify that you are free of radioactive contamination.

Emergency Phone Numbers

Name: Here you should list the name and contact information for all personnel crucial to the response and recovery from the accident.

Medical or Fire

Radiation Safety Officer Occupational Safety Officer Biological Safety Officer Laboratory Safety Officer Fire and Life Safety Officer

In case of an injury in the laboratories, contact <u>the laboratory</u> <u>manager and the laboratory director</u> immediately. In case of a contaminated and injured person (life threatening injuries) <u>do not</u> <u>delay medical treatment</u> for radiological contamination control. If hospital care is required, be sure that a radiation safety trained individual accompanies or meets the contaminated patient to the hospital (with radiation detection equipment) and the RSO is notified immediately.

Newsletter 21 – Access to Laboratories Containing Radioactive Materials

Laboratories containing radioactive materials are "controlled to ensure the security of radioactive materials and to prevent unnecessary exposure of personnel to radiation".

While this concept is very simple, in practice, preventing unauthorized access to rooms containing radioactive materials is sometimes difficult. Everyone must be aware of the rules in order to ensure that the meaning of the term **unauthorized access** is understood by all.

So what is unauthorized access?

If you are not a member of the radiation safety staff or you are not a member of a professor's laboratory staff or if you are a member of one of these groups but have not received your annual radiation safety training, you are not authorized to enter a laboratory where radioactive material is in use.

What about laboratory visitors?

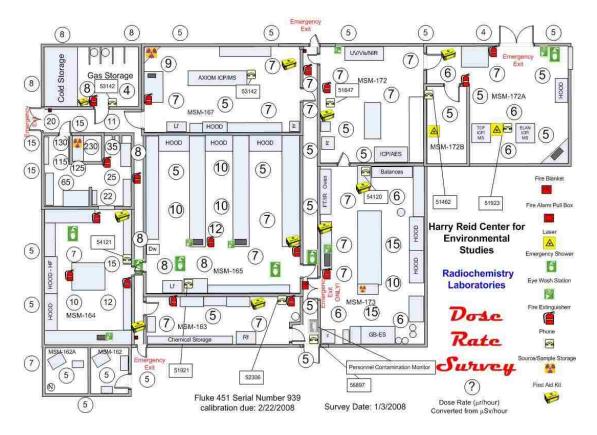
Visitors to the radiochemistry laboratories must be escorted at all times. The escort must be a trained member of the Radiochemistry Program staff. An Authorized User or the Laboratory Manager must be aware of any tours.

Visitors may become radiation workers for collaborative work with HRC scientists. All work with radioactive materials must be done under the authorization of a UNLV Authorized Radioactive Material User.

What are the dose rates in the radiochemistry laboratory areas?

The dose rate in the laboratory areas are 10 to 20 times background in areas where the largest amounts of radioactive material are used or stored. There are no dangerous levels of external dose rate in any areas of the laboratories. This map shows a display of the common dose rates in the laboratory areas in units of micro-rem/hour:

You can see from this map that the dose rates in the hallways and in many of the laboratory areas are similar to the normal background dose rate for Las Vegas which ranges from approximately 5 micro-rem/hour to 15 micro-rem/hour.



What are the rules regarding security of radioactive material?

A policy approved by the Radiation Safety Advisory Committee established the requirements for security of radioactive materials at UNLV.

It shall be the policy of the University of Nevada Las Vegas to properly control radioactive materials in accordance with the requirements of the UNLV Radiation Safety Manual and the Nevada Administrative Code.

The following issues are addressed in this policy:

- 1. Security of Radioactive Materials in Storage Areas.
- 2. Security of Radioactive Materials in Laboratories.

All areas where radioactive materials are used shall be posted with a "Caution Radioactive Materials" sign. Radioactive materials shall be used <u>only</u> by trained personnel. All personnel working in a laboratory where radioactive materials are in use shall be aware of the presence of the material in the laboratory. All radioactive materials not in use shall be in a locked container. These four statements form the basis of security of radioactive materials at the University.

It is most desirable to have two levels of security for all radioactive materials at the University. However, it is recognized that this is inefficient for some situations and may cause more radiation exposure than if the sources were not handled as much. In this regard, at least one level of security shall be in effect to prevent the removal of sources from all laboratories and storage areas.

IN NO CIRCUMSTANCES IS IT ACCEPTABLE FOR UNTRAINED PERSONNEL TO BE LEFT ALONE IN POSTED, UNCLEARED AREAS. "TRAINING," FOR THESE PURPOSES, MEANS RADIOLOGICAL SAFETY TRAINING PERFORMED BY, OR ACCEPTED BY, RADIATION SAFETY PERSONNEL. Newsletter 22 – New Laboratories in HRC Completed

We now have two new laboratories for use of radioactive materials in the Harry Reid Center, MSM-234 and MSM-236. These laboratories offer three new fume hoods with HEPA filtered ventilation, a new perchloric acid hood, and two new glove boxes as well as more than 40 linear feet of laboratory bench space, two new water purification systems, and two sinks.

Our new laboratories will be controlled similar to the way that we control our other laboratories where radioactive materials are used. However, experiments with flowing water are restricted from these laboratories because they are located above the Marjorie Barrick Museum.

Many features of these new laboratories are discussed in this newsletter.

Fume Hoods

There are two four foot and one six foot fume hood available for work with radioactive materials. Flow into the hoods is greater than 100 ft/minute with the sash at 18 inches. Each hood has a flow rate alarm to immediately identify when a hood is not ventilated. The vent pipes that provide flow from each hood are stainless steel and are welded at each seam. Each hood flows independently to its own HEPA filtered discharge.

Perchloric Acid Hood

A fourth hood in the MSM-234 laboratory is designed for work with perchloric acid. The ventilation lines for this hood have a wash-down system with restricted access. The wash-down system will spray the inside of the vent pipes with water that will collect in the hood and transfer to a five gallon tank under the hood. The volume of water used in the wash-down must be closely monitored to prevent overflow of the collection tank. In order to prevent accidental overflow of the tank, this operation will be restricted to specifically trained people. The valves that provide water to the spray lines will be locked out when the wash-down system is not in use. The perchloric acid hood does not have a filtered discharge.

Air Conditioning and Heating

Temperature control is provided by two large AC systems, a 13 ton system providing support for MSM-234 and a 10 ton system providing support for MSM-236. Thermostats are on the front walls of the laboratories near the exit doors. Thermostats may be adjusted to provide a comfortable temperature in the laboratories.

Bench Tops

The bench tops are standard laboratory tops with acid and heat resistance and similar to laboratory benches in other areas, they must be covered with bench paper when radioactive material will be used on them.

Chemical Storage

Cabinets have been installed for chemical storage, however, the primary location for chemical storage will continue to be MSM-163 in the main laboratory area on the first floor.

Water Systems

There are two Pall Cascada DI water systems in these laboratories. They are supplied from our new 1000 gallon per day reverse osmosis system. As a reminder, do not use water cooling systems in this room. A leak or uncontrolled flow of water could be disastrous to the museum.

Sinks

Two sinks are provided for personal washing, and equipment cleaning. These sinks must not be used for release of chemicals or radioactive materials. All radioactive liquids are collected for disposal by the UNLV Radiation Safety Office. Hazardous materials are collected by the UNLV Hazardous Materials Technicians. Hazardous and radioactive liquids are stored in MSM-163 prior to transfer to RMS personnel for disposal.

Glove Boxes

Two new Labconco glove boxes are in MSM-236. These boxes will be initially under argon atmosphere and have regeneration capability. We anticipate that these boxes will be available for use by mid-November. Parts are on order to allow setup of the boxes.

Electrical Supply

There is a variety of electrical supply including (2) 30 amp 208V outlets, (2) 20 amp 110V outlets, and several 110V service outlets on each bench.

Emergency Equipment

Eye-wash stations are located at each sink, the emergency shower is on the south wall of MSM-234. Fire extinguishers are located at the emergency exit door in MSM-236 and inside MSM-234 at the doorway between MSM-234 and MSM-236. First aid kits are located at each door to the hallway.

Hand & Foot Monitor

The Thermo HFM-11 hand and foot monitor is at the exit from MSM-234 to provide a simple method to prevent radioactive contamination from leaving the room. This monitor is a set of gas flow proportional detectors, operated on P-10 gas with a microprocessor controlled system that monitors system operation and provides an alarm upon detection of activity above background. Upon identification of an alarm, check yourself for contamination using a frisker, or if alpha contamination is detected, use an alpha scintillation detector (Ludlum 4393 / 2360) to identify the contaminated location and allow cleanup.

Proximity card access has been allowed for all radiochemistry program personnel.

Newsletter 23 – Radiological Emergency Preparedness

You are working in your laboratory with a solution containing radioactive materials and due to some unforeseen circumstance you drop the vial containing your entire inventory. As you drop the vial, you notice that your laboratory partner, in an attempt to prevent the spill, falls to the ground and is hurt. Are you prepared to respond to this situation? What will you do?

Your response to an <u>uncontrolled</u> spill of radioactive material

Evaluate the scene, if someone is injured, get medical assistance on the way before any other action - call 911 from any campus phone. Ensure that the injured person is in a safe location and take any medical action that you are qualified for, to help the individual. Get Help if you need to.

1. Stop the Spill

If you can stop the spill by up-righting the container or turning off flow of liquid to a drain or preventing the flow of the liquid to a floor drain, do so. If you are dripping, take off your laboratory coat in the area. If possible use it to absorb the spill or stop the flow of the liquid.

2. Warn Others in the Area

Let other people in the area know that you have a problem. Loudly indicate that you have spilled radioactive material and no one should enter the spill area. Do this to minimize the spread of material from the area and minimize exposure to others.

3. Isolate the Area

You don't want other people coming back into the spill area while there is still a problem. Don't leave, prevent others from going into the area, lock the laboratory doors, put up a sign, and rope off the area. If you don't control the area, someone might enter the area, cause further spread of the material, and receive unnecessary exposure.

4. Minimize Exposure

Minimize your exposure and the exposure of others by using time, distance, and shielding. Move away from the spill area and use physical structures between you and the spill to lower the dose rate in your area. Ensure others in the laboratory either leave the immediate laboratory area or also minimize their exposure.

5. Notify the RSO

The people in the UNLV Radiation Safety Office can assist you in your efforts to minimize exposure and get the laboratory back into normal operation. Call X54226 for anyone in RMS, X54419 or X54941 for the Radiation Safety Officer or Radiation Safety Technician, or 3404419 for the RSO cell phone. Then call people on the HRC Emergency Call List posted in any of the HRC radiation laboratories. If there is an injured contaminated person, contact the Radiation Laboratory Director immediately.

Your response to a <u>controlled</u> spill of radioactive material

Well, what's the difference? You have taken the precautions to assure that the impact of a spill will be minimized. Consider the following:

You are working with the same liquid as above, in a hood, on a tray with sides to catch a spill. Your actions should be similar but will not be as extensive.

If there is an injury, medical response to injured people always takes priority!

Use the same sequence **S-W-I-M-N** as needed.

The spill is **S**topped by the tray, the need to **W**arn others is minimized to those who may need access to the hood,

you can Isolate the hood by closing it and telling others in the laboratory.

you can Minimize your exposure and the exposure of others by moving yourself

and others away from the hood, then Notify the RSO for help.

Preparing for an emergency involving radioactive materials is really a way to minimize the impact of the event on yourself and others in your laboratory.

Some things that you can do in your laboratory to prevent or provide easy response to spills involving radioactive material:

Always ensure that radioactive materials are used over a secondary container that can contain twice the volume of liquid or solid materials if your container is accidentally knocked over. Always work over absorbent material with a plastic backing.

When you are ready to work with radioactive materials, take one more look at your apparatus and work area to ensure that movement of materials in the area will not cause a problem and items that may fall or be knocked over will spill into the area that you setup to contain the material.

Ensure that the work area is not cluttered and there is space to move around unimpeded. Ensure others in the laboratory are aware of what you are doing and the potential for a spill if your area is crowded. Use radioactive materials in a hood when possible.

If you will be transferring components and material from the work area to another area of the laboratory or another laboratory, ensure that the pathway is open and available before you attempt the transfer. Put all materials in a secondary container to prevent a spill. Call ahead to the destination to ensure that you will not confront a locked door when you get there.

Keep a spill kit in your laboratory to assist you with rapid and efficient containment and cleanup of a spill. Your kit, as a minimum, should contain a box of protective gloves, absorbent material, clean rags, rubber drain covers (if there are floor drains in your laboratory), and a standard first aid kit.

You should know and trust everyone in the laboratory with you. If you see something that someone is doing that may cause harm to themselves or someone else, ask them about it, if there is a problem with what they are doing, don't let them do it! We all have to peacefully coexist in our laboratories; you and I must be comfortable that the laboratories are a safe place to be.

Finally, periodically check the location and condition of emergency equipment. If there is anything missing let the Laboratory Manager know about it.

Your primary goal is obviously successful completion of your research. Your secondary goal (my primary goal) is to ensure that you do so safely and ensure control of your radioactive material.

Newsletter 24 – Portable Instruments – Ludlum Model 3 / 44-9

This issue will begin a series of newsletters with the primary subject area as portable radiation detection instruments. This first issue on that topic will describe the workhorse of our laboratories, the ratemeter and pancake GM detector.

The Model 3/44-9 is a combination rate-meter with a model 44-9 GM detector from Ludlum Measurements Inc.

The Ludlum model 3 rate-meter is a very simple measurement device with a minimum of controls. The controls are as follows:

- 1. A rotary selection switch with positions: OFF, BAT, X100, X10, X1, X0.1.
- 2. An on/off switch for audio indication.
- 3. A switch for fast (4 second)/slow (22 second) response from 10% to 90% of the maximum reading.
- 4. A reset button that takes the meter indication to zero.

The Model 3 rate meter is a tool for identification of the response rate



from a source of ionizing radiation emissions. The range of measurement of the Model 3 is up to a maximum count rate of 500,000 cpm. The response is linear within $\pm 10\%$ of the true response over the entire response range. The speaker provides an output of more than 60 db at 2 feet from the instrument.

Batteries -The battery compartment 2 "D" contains cells. The battery compartment is accessible from the top of the instrument. The battery configuration is indicated on the underside of the battery compartment cover. Alkaline batteries should provide greater than 2000 hours of instrument operation.

The Case – The Model 3 has a cast aluminum body with a steel handle and speaker holes on the side. This provides for a light instrument that is also rugged. Even so, if you drop this instrument, check it for damage and operability – go through all of the instrument checks before you use it for a measurement.

Temperature Operating Range – The normal calibration of this instrument provides acceptable response from $-4^{\circ}F$ ($-20^{\circ}C$) to $122^{\circ}F$ ($50^{\circ}C$). Instrument operation outside of this range may require different calibration.

The Ludium Model 44-9 Pancake GM Probe – This is the standard probe on GM instruments used in the radiochemistry laboratory and is a very common detector used in many industries.



This probe houses a pancake type halogen quenched GM detector with a typical detection efficiency of 19% for ⁹⁹Tc beta emissions at ¹/₄" from the source. The window area is approximately 15 cm² and the open window area is 12 cm². The GM detector in this probe operates on 900 volts, has a typical dead time of approximately 80 microseconds.

The Detector

The GM detector in this instrument is about 2 inches in diameter and has a mica window with a thickness of 2 mg/cm². The filling gas is neon with a halogen quenching agent (chlorine or bromine). The window has a diameter of 1.75 inches (44.5 mm).

The background response from external gamma radiation will be about 500 CPM in a field of 10 mrem/hour (0.1 mSv/hour).



In the illustration, the detector on the left is intact, while the one on the right has the window removed and you can see the anode inside the detector. A broken window cannot be repaired on these detectors. A replacement detector costs approximately \$100.00.

What it Detects

Ionization in the detection gas caused by alpha, beta, or gamma radiations will cause a pulse to be transmitted to the meter. However, because the detector is not in a consistent geometry, alpha particles may or may not penetrate the window to cause ionization of the gas, so a consistent alpha detection efficiency cannot be accurately defined. Gamma radiation has a low probability of interaction in the gas so "measurement" of dose rate is not recommended from this type of detector, even when the meter has a dose rate scale. Beta radiation with energy greater than 100 keV is detectable and "measurable" with this type of detector.

Direct Measurements

One of the more useful purposes for the pancake GM detector and ratemeter is basic contamination control of beta emitting radionuclides. In order to have consistent detection efficiency while "scanning" a surface for beta emitting contamination, the speed of movement of the detector and the surface to detector distance must be controlled. The speed of movement is important so that there is enough residence time over a contaminated area to provide adequate response. The surface to detector distance will cause a direct change to the detection efficiency.

If you move the detector over a surface at a speed of 2 inches per second, and have a 10% efficiency for the radionuclide that you are interested in, you will have a certain probability of detection as follows:

Let's consider a stationary beta GM pancake detector, the probe area is 15 cm^2 , the background count rate is assumed to be 40 cpm, the detection efficiency is assumed to be 20%. From table 6.4 in MARSSIM, the critical level is 15 counts, the detection limit is 32 counts, and the MDC is 1800 Bq/m² (1080 dpm/100 cm²). This might be typical of ⁹⁹Tc on a smooth flat surface.

Scanning is quite different than simply holding a detector stationary, there are some additional considerations, you are moving a detector over a surface and at a specified distance from the surface to keep the detection efficiency constant. You actually have a certain efficiency to do that. The count time is related to how fast you move the detector over a surface – consider that a spot being surveyed would pass under the detector for a duration that begins as the leading edge of the detector goes over the spot and ends as the back edge passes over the spot. So, the count time for a 1.75 in diameter detector passing over a spot at 2 inches per second would have a count time on that spot of: t = 1.75 in/2 in/s = 0.875 seconds.

So, you can see that the probability of detecting activity on that spot would become lower as you move the probe faster. You should also recognize that moving the probe at a constant speed and holding it a constant distance from the surface are going to be difficult tasks and your efficiency as a surveyor will continue to decrease as you get more tired.

Taking this information a few steps further following MARSSIM, the "scan MDC" for a pancake probe GM detector as we described is approximately $3770 \text{ dpm}/100 \text{ cm}^2$. This value is more than 3 times the activity that you could detect with the stationary instrument. Imagine if you moved the probe much faster.

Preparing for Operation

- 1. Always make sure that your instrument is in good condition. No major dents in the case, it looks like it should work (no wires hanging out), etc.
- 2. Make sure that the instrument has been calibrated in the past year. Check the calibration sticker for the date that calibration is due.
- 3. Turn the instrument to the BAT position and make sure that the meter reading is beyond the battery OK position in the meter.
- 4. Turn the knob to the lowest scale and check for a normal background response. For a pancake GM probe, the normal response should be between 20 and 100 counts per minute in a low background area.

Precautions

As with any instrument, there are some precautions that you can take to ensure that it provides you with the information that you need and that it continues to work. These are:

- 1. Never put the instrument in water it will never work again.
- 2. When surveying contaminated areas try not to touch the probe to the surface you are measuring this will minimize the probability that you will contaminate the probe.
- 3. Never survey sharp objects; wire, wire brushes, brooms, things that can make a hole in things. This action will cause the window to be broken and the instrument to be nonresponsive.
- 4. Never use the probe as a hammer. There is a floating anode inside the detector that can easily be shaken out of position maybe even short out anyway use a hammer for hammering.

- 5. Try not to drop the meter or detector dropping the meter could break a circuit board inside the case dropping the probe may break the window or short out the anode.
- 6. If there is an accident and you contaminate the detector, please remove the detector from normal use and let me or Trevor know that the instrument is OOS. We can usually decontaminate or repair the instrument in less than an hour.

Surveying

Whenever you check your work area after working with radioactive materials, you have several actions to take and decisions to make;

- 1. Check your instrument is it calibrated? are the batteries OK? is the background response OK? does it respond to ionizing radiation?
- 2. If you are working with beta emitting radionuclides that emit beta particles sufficiently energetic to be detected by a pancake GM detector, then the Ludlum Model 3/44-9 may be the instrument that you could use for your survey.
- 3. Turn on the audible response. This is your most sensitive indicator, each "click" is caused by an ionizing event in the detector, if it clicked it detected something!
- 4. I recommend always using the "slow" response mode for low activity measurements or clearance surveys. "Fast" response is OK for higher activity measurements. You should not use "fast" response for surveys where you are verifying that the area is free of contamination.
- 5. As you check the work area, pay particular attention to areas immediately adjacent to the area where your radioactive material was used.
- 6. Your 44-9 Pancake GM probe should be held at a distance of ¹/₄" to ¹/₂" from the surface that you are surveying with the window facing the area that you are surveying.
- 7. Move the probe over the area slowly. You can survey a large area by moving the probe at 1" to 2" per second. Don't expect to detect anything if you wave the probe like a magic wand.
- 8. If you identify a response continue to survey wait until you characterize the entire work area before you start your cleanup. This will help you to decide on the best method to do cleanup and provide information for the decision about what PPE to wear.
- 9. If you exceed the response capability of your instrument, be sure to control the area, identify the location where the emissions causing that response are coming from. This may be an area where material was spilled or maybe your stock material was not removed from the work area. If you recognize this as a potential problem, then take action to prevent spread of the contamination.

~ CAUTION ~

Radiation detectors allow you to detect and measure radiations emitted by the materials that you are working with. It is important that you take care of them so that they continue to provide their function and allow you to protect yourself and others from ionizing radiation. Newsletter 25 – Portable Instruments – Ludlum Model 2360 / 43-93

This issue will continue the series of newsletters with the primary subject area as portable radiation detection instruments. This second issue on that topic will describe a very important instrument for some of our laboratories, the rate-meter / scaler and alpha-beta scintillation detector.

The Model 2360/43-93 is a combination rate-meter and scaler with a model 43-93 Alpha-Beta scintillation detector from Ludlum Measurements Inc.

The Ludlum model 2360 rate-meter/scaler is a simple measurement device with a few more controls than the model 3. The controls are as follows:

- 1. A rotary switch with positions: OFF, BAT, X1000, X100, X10, and X1.
- 2. A volume switch and volume control for audio indication.
- 3. A switch for selection of signals from the alpha scintillators or the beta scintillators or both.



1. A reset switch which also allows reading of the high voltage.

2. A rotary switch that allows selection of the count time for the scaler, and a start button that allows starting the count integration mode.

The Model 2360 rate meter is a tool for identification of the response rate or integrated counts over a specified time from a source of ionizing radiation emissions. The range of

measurement of the Model 2360 is up to a maximum count rate of 500,000 cpm. The response is linear within $\pm 10\%$ of the true response over the entire response range. The speaker provides an output of more than 60 db at 2 feet from the instrument and the volume is adjustable.

Batteries -The battery compartment contains 2 "D" cells. The battery compartment is accessible from the top of the instrument. The battery configuration is indicated on the underside of the battery compartment cover. Alkaline batteries should provide greater than 2000 hours of instrument operation.

The Case – The Model 2360 has a cast aluminum body with a steel handle and speaker holes on the side. The start button for integrated counts is located in the handle. This instrument is light and also rugged. Even so, if you drop this instrument, check it for damage and operability – go through all of the instrument checks before you use it for a measurement, just like any other instrument. The probe contains a glass photomultiplier tube which could be broken from shock.

Temperature Operating Range – The normal calibration of this instrument provides acceptable response from $-4^{\circ}F$ ($-20^{\circ}C$) to $122^{\circ}F$ ($50^{\circ}C$). Instrument operation outside of this range may require different calibration.

The Ludlum Model 43-93 Alpha Beta Scintillation Probe – This is the probe on the Ludlum 2360 instruments used in the radiochemistry



laboratory and is a very common detector used in many industries that handle small quantities of alpha and beta emitting radionuclides.

This probe houses a photomultiplier tube that observes the light output of a dual scintillator. This detector

has a typical detection efficiency of 15% for 99 Tc beta emissions at $^{1}\!4$ " from the source. The window area is approximately 100 cm² and the open window area is 89 cm².

The detector in this probe operates on 1650 volts, has a typical response time of approximately 2 to 10 microseconds for 90% of the maximum response.

The Detector

The detector in this instrument is about 3.5 inches by 5.75 inches and has a mylar window with a thickness of 0.5 mg/cm². The detector is a plastic plate with a coating of zinc sulfide. The background response from external gamma radiation will be about 5000 CPM in a field of 10 mrem/hour (0.1 mSv/hour).

In the illustration, the detector is the solid white rectangle in the middle. At the top middle is the mylar window, on the left is the cover plate which holds the detector in place and on the right is a metal grid that fits over the window to provide some protection for the window. A broken window can be repaired on these detectors. A replacement window costs approximately \$30.00.



What it Detects

Interactions of alpha radiation in the zinc sulfide, or beta, or gamma radiations in the plastic scintillator will cause a pulse to be transmitted to the meter. The voltage of the pulse will depend on whether the interaction was in the ZnS or the plastic. The 2360 ratemeter/scaler will separate the signals to allow determination of alpha or beta response.

Gamma radiation has a low probability of interaction in the gas so "measurement" of gamma dose rate is not recommended from this type of detector, even when the meter has a dose rate scale. Beta radiation with energy greater than 100 keV is detectable and "measurable" with this type of detector.

Direct Measurements

One of the more useful purposes for this scintillation detector and ratemeter is basic contamination control of alpha or beta emitting radionuclides. In order to have consistent detection efficiency while "scanning" a surface for alpha or beta emitting radionuclides in contamination, the speed of movement of the detector and the surface to detector distance must be controlled. The speed of movement is important so that there is enough residence time over a contaminated area to provide adequate response. The surface to detector distance will cause a direct change to the detection efficiency.

If you move the detector over a surface at a speed of 2 inches per second, and have a 15% efficiency for the alpha emitting radionuclide that you are interested in, you will have a certain probability of detection as follows:

Let's consider a stationary 43-93 detector, the probe width is 100 cm², the background count rate is assumed to be 1 cpm, the detection efficiency is assumed to be 15%. From table 6.4 in MARSSIM, the critical level is 2 counts, the detection limit is 7 counts, and the MDC is 150 Bq/m² (90 dpm/100 cm²). This might be typical of ²³⁹Pu on a smooth flat surface.

Scanning is quite different than simply holding a detector stationary, there are some additional considerations, you are moving a detector over a surface and at a specified distance from the surface to keep the detection efficiency constant. <u>You</u> actually have a certain efficiency to do that. The count time is related to how fast you move the detector over a surface – consider that a spot being surveyed would pass under the detector for a duration that begins as the leading edge of the detector goes over the spot and ends as the back edge passes over the spot. So, the count time for a detector that is 7 cm wide passing over a spot at 5 cm per second would have a count time on that spot of: t = 7 cm/5 cm/s = 1.4 seconds.

So, you can see that the probability of detecting activity on that spot would become lower as you move the probe faster. You should also recognize that moving the probe at a constant speed and holding it a constant distance from the surface are going to be difficult tasks and your efficiency as a surveyor will continue to decrease as you get more tired.

Taking this information a few steps further following MARSSIM, the "scan MDC" for a scintillation detector probe for alpha contamination as we described is approximately 85 dpm/100 cm². This value is more than 4 times the activity that alpha emitting contamination should be controlled to (22 dpm/100 cm²). Imagine if you moved the probe much faster.

Preparing for Operation

- 1. Always make sure that your instrument is in good condition. No major dents in the case, it looks like it should work (no wires hanging out), etc.
- 2. Make sure that the instrument has been calibrated in the past year. Check the calibration sticker for the date that calibration is due.
- 3. Turn the instrument to the BAT position and make sure that the meter reading is beyond the battery OK position in the meter.
- 4. Turn the knob to the lowest scale and check for a normal background response. For a 43-93 scintillation probe, the normal response should be between 0 and 10 counts per minute for alpha and 100 to 300 counts per minute for beta in a low background area.

Precautions

As with any instrument, there are some precautions that you can take to ensure that it provides you with the information that you need and that it continues to work. These are:

- 1. Never put the instrument in water it may never work again.
- 2. When surveying contaminated areas try not to touch the probe to the surface you are measuring this will minimize the probability that you will contaminate the probe.
- 3. Never survey sharp objects; wire, wire brushes, brooms, things that can make a hole in things. This action will cause the window to be broken and the instrument to respond to ambient light (remember a scintillation detector works by detection of photons from the scintillator).
- 4. Never use the probe as a hammer. There is a glass photomultiplier tube in the probe that will break if subjected to shock anyway use a hammer for hammering.

- 5. Try not to drop the meter or detector dropping the meter could break a circuit board inside the case dropping the probe may break the window or the photomultiplier tube.
- 6. If there is an accident and you contaminate the detector, please remove the detector from normal use and let me or Trevor know that the instrument is OOS. We can usually decontaminate or repair the instrument in less than an hour.

Surveying

Whenever you check your work area after working with radioactive materials, you have several actions to take and decisions to make;

- 1. Check your instrument is it calibrated? are the batteries OK? is the background response OK? does it respond to ionizing radiation?
- 2. If you are working with alpha emitting radionuclides that emit alpha particles sufficiently energetic to be detected by the 43-93 detector, then the Ludlum Model 2360/43-93 may be the instrument that you could use for your survey.
- Turn on the audible response. This is your most sensitive indicator, each "click" is caused by an ionizing event in the detector, if it clicked
 it detected something! Notice that there are two different click frequencies one for alpha particles detected, and one for beta particles detected.
- 4. As you check the work area, pay particular attention to areas immediately adjacent to the area where your radioactive material was used.
- 1. Your 43-93 probe should be held at a distance of ¹/₄" to ¹/₂" from the surface that you are surveying with the window facing the area that you are surveying.
- 2. Move the probe over the area slowly. You can survey a large area by moving the probe at 1" to 2" per second. Don't expect to detect anything if you wave the probe like a magic wand.
- 3. If you identify a response continue to survey wait until you characterize the entire work area before you start your cleanup. This will help you to decide on the best method to do cleanup and provide information for the decision about what PPE to wear.
- 4. If you exceed the response capability of your instrument, be sure to control the area, identify the location where the emissions causing that response are coming from. This may be an area where material was spilled or maybe your stock material was not removed from the

work area. If you recognize this as a potential problem, then take action to prevent spread of the contamination.

~ CAUTION ~

Radiation detectors allow you to detect and measure radiations emitted by the materials that you are working with. It is important that you take care of them so that they continue to provide their function and allow you to protect yourself and others from ionizing radiation.

Newsletter 26 – Laboratory Inspection

This issue will explain what a laboratory inspection is for and provide you with guidance that you can use to ensure that your area meets reasonable criteria for safety.

One very important aspect of our radiation safety program is evaluation of laboratories to ensure that we are in compliance with our radioactive materials license, x-ray machine registrations, and State of Nevada regulations.

We do inspections of every laboratory containing radioactive materials – every day; the Radiation Safety Office checks our laboratories during a monthly visit to ensure that we meet their criteria for a safe laboratory as well. The inspection is NOT an in depth evaluation of compliance, but we do check at least the following:

POSTINGS AND LABELING

- Proper labeling of the room for radioactive materials.
- "No Eating, Drinking or Smoking" sign still posted.
- No evidence of food or drink in the laboratory.
- Proper labeling of the storage area and work areas.
- Proper labeling of containers.
- Emergency phone numbers posted near the phone.
- Notice to Employees and NRC-1 posted.
- Laboratory map posted on the door. RADIOACTIVE MATERIAL

ACCOUNTABILITY

- Radioactive materials use log.
- Source storage containers are secured at all times.
- Samples containing radioactive materials are appropriately labeled.
- Samples containing radioactive material are appropriately stored.
- Sealed and unsealed stock sources are appropriately stored.

CONTAMINATION CONTROL

• Contamination surveys of the room maintained.

• Work areas do not present a safety hazard to other laboratory inhabitants.

- Work areas are properly identified when contaminated.
- Researchers check their areas for contamination before leaving.

WASTE ITEMS

- Solid waste is appropriately disposed of in appropriate containers.
- Liquid waste is placed in the appropriate disposal area.
- Aqueous and organic waste streams are separate.
- Organic waste streams are minimized.
- Mixed waste is not created on a large scale.
- LSC Waste is maintained in LSC vials and a waste form is attached.
- There are no radioactive materials or labels in the clean trash.

BENCHTOPS

- Bench tops should be free of clutter.
- There should be adequate room for work when no experiments are taking place.
- Equipment that generates a lot of heat should not be on flammable bench coverings.
- Samples, equipment, and stock solutions should be toward the center of the bench to prevent them from being knocked off.
- Radioactive stock solutions should be stored in the safe when they are not immediately in use.
- Open containers of liquids must be in a secondary containment.

GLOVE BOXES

- Glove boxes should be maintained relatively clean inside.
- The gloveless box should be maintained clean at all times when work is not in progress in that box.
- The port covers should always be on the gloveless box when it is not in immediate use.
- The gloves and box should be checked for integrity every day.

Purpose

The purpose of our inspection is not to pick on anyone or slow down research. The purpose is to ensure that we do maintain compliance with our Radioactive Materials License and general safety requirements.

Notification

We do not send out a warning that we will be inspecting laboratories because we must verify that compliance is the norm, not just something that is present when we know an inspector will visit our laboratories. When the State of Nevada regulators visit our facility, they will not call to tell us when they are coming to inspect us. We must be ready for their inspection of our laboratories at all times.

If there are experiments in progress that may be adversely affected by persons entering the laboratory, then please send a message to the group to identify these situations.

Inspectors

Our inspections may be done by any one of the persons on our staff. Typically, they are done by the laboratory manager or an Authorized User However; anyone can identify potential problems and should identify the problems to the laboratory managers as soon as possible.

Findings

When we find a condition that is in conflict with a regulation or requirement, the Laboratory Director or Laboratory Manager is notified and he/she will make contact with the person responsible for the laboratory area to provide simple means of ensuring compliance. We will work with the researcher to achieve compliance for any violation or item of concern with as little impact on the research in that laboratory as possible.

Consider the inspection process as a good way to ensure that our radiation safety program meets license conditions and State regulations.

Newsletter 27 – Radioactive Material Inventory

As a Radioactive Material licensee in the State of Nevada, we have limitation on the total activity of radioactive materials that we may have at any one time. These limits are a maximum amount of activity for each different radionuclide.

In order to establish compliance we determine the fraction of the allowed activity for each radionuclide, then we then 'sum the fractions' and the sum must be less than 1 for the determination of compliance.

For example, let's say that we have 20 mCi of ²⁴¹Am and 50 mCi of ¹³⁷Cs. If our license limit for ²⁴¹Am is 30 mCi and our license limit for ¹³⁷Cs is 150 mCi, then we have reached:

Sum of Fractions = 20/30 + 50/150 = 100% of our license limit.

User Limits

Each user has a radioactive materials allowance in order to maintain the total possession of all users less than our license limits. The activity assigned as a User limit is determined by the RSO. The items that are important in determination of the User limits are:

The limits of our radioactive materials license and ensuring that other users are not deprived of experimentation because one person restricts our ability to maintain license compliance by having an unusual amount of a certain radionuclide with a low license limit. Some radionuclides have much lower license limits than others and may be more or less restrictive.

For example, our limit for ${}^{32}P$ is 1 Curie and our limit for ${}^{3}H$ is 100 Curies. If these were the only radionuclides that we had, life would be simple.

The license limit for some radionuclides is based on the mass of the nuclide.

For example, the license limit for ²³⁹Pu is included in our 'special nuclear material limit of 200 grams. Whereas our limit for ²³⁸U is included in our 'source material' limit of 100 pounds.

And there are some radionuclides that have extremely low limits such as ²²⁸Th that has a limit of 0.00005 Ci (50 microcuries).

Periodic Inventory Checks

YOU SHOULD ALWAYS BE AWARE OF YOUR RADIOACTIVE MATERIALS. IF YOU DISCOVER THAT A SOURCE IS MISSING FROM YOUR STORAGE OR USE AREAS, PLEASE CONDUCT A SEARCH FOR THE MATERIAL AND CONTACT ONE OF THE USERS IMMEDIATELY!

The Radiation Safety Office must be aware of the location of all radioactive materials at UNLV. To ensure this, they conduct a complete inventory check every 6 months. Just because a source is 'exempt', this does not mean that it is not held under our license.

ALL RADIOACTIVE MATERIALS THAT ARE NOT NATURALLY OCCURRING RADIOACTIVE MATERIALS (NORM) ARE CONTROLLED UNDER OUR RADIOACTIVE MATERIALS LICENSE.

Consider the inventory process as another good way to ensure that our radiation safety program meets license conditions and State regulations.

Laboratory Control of Sources

Control of sources of radioactive material in our laboratories is of course not just to ensure license compliance, it's for our protection and the protection of others in the laboratories and it is for the protection of our radiochemistry program. We must always ensure that our radioactive material is within the boundaries described by our license and no one will get hurt by our material.

There are sign out sheets located at the primary source storage area in MSM-168. If you are a user, be sure to sign out any materials that are provided to others for work – and make sure that you retrieve the material from them when they are done taking the amount that they need. The amount removed should be recorded on the form.

When you get a stock solution or compound from a user, make sure that you return the remaining material to the user so that it can be properly stored in one of our primary storage areas. If a source or solution is completely used, and the container is disposed, then annotate that on the sign out form.

If you know the location of a source with an HRC-XXX label on it (where XXX is a number), you should make sure that this source is returned to the safe in MSM-168 as soon as practical.

Newsletter 28 – Survey Documentation

Surveys are done to ensure compliance with Federal and State regulations and our Radioactive Materials License conditions. When an inspector from the State of Nevada comes to the campus to review our program, he/she reviews documentation to ensure that we have complied with regulations. Nevada Administrative Code (NAC) section 459.337 is the requirement that applies to surveys.

NAC 459.337 Surveys and monitoring. (NRS 459.030)

1. Each licensee and registrant shall make, or cause to be made, surveys that:

(a) Are necessary for the licensee or registrant to comply with <u>NAC</u> 459.010 to 459.950, inclusive; and

- (b) Are necessary under the circumstances to evaluate:
- (1) The magnitude and extent of radiation levels;
- (2) Concentrations or quantities of radioactive material; and
- (3) The potential radiological hazards.

2. The licensee or registrant shall ensure that instruments and equipment used for quantitative radiation measurements are calibrated for the radiation measured at intervals not to exceed 12 months.

One important concept to understand regarding surveys: all information must be written and the survey must be signed by the person doing the survey and the survey reviewer, and signatures must be dated.

The following information is necessary for that documentation:

The location surveyed (be as specific as possible).

The date of the survey.

The model and serial number of the portable instruments used for the survey.

The model and serial number of the laboratory instruments used for smear or sample analysis.

The date that the next calibration of this instrument is due.

The location of any smears or dose rate readings taken in the laboratory.

The results of smears analyzed (an attached analysis sheet is fine).

The dose rates in the area at the location of measurement.

A note about actions taken when the survey identifies an abnormal situation (such as contamination outside the labeled work area).

How often are surveys required?

The required frequency of radiation and contamination surveys in laboratories differs depending on the amount of activity used and the potential for contamination spread to personnel traffic routes in the buildings. However, every time we are in the laboratory working with radioactive materials, we need to ensure (by survey) that we did not spread contamination and that all radioactive materials are secure before we leave the laboratory.

How long should it take to do and document a survey?

A basic contamination control survey of a complete laboratory should not take more than an hour. A routine survey of a small work area should only take minutes. This depends on the size of the laboratory and whether or not contamination was identified that must be cleaned up. Some laboratories may also need a periodic evaluation of the dose rate in the laboratory; this should take no more than half an hour.

What forms should be used to document the surveys?

Forms to document surveys of the laboratories are maintained in the main corridor of the first floor laboratory s. The laboratory managers can help you find what you need to document your surveys. When completed, all survey forms are kept in the binders that are maintained in the hallways (a very good place to identify the results of past surveys and the current radiological profile of the laboratories).

Basic Survey Information

Always wear gloves to protect your skin from radioactive or chemical contamination that may be on surfaces.

Smear surveys are done to identify the activity of removable contamination from a surface.

Smears are small 2" disks of paper or cloth that are rubbed on a surface to evaluate the amount of radioactive material that could be removed from a surface by normal touching of that surface.

The standard area of a smear is 100 cm^2 , 100 cm^2 is about 4 inches by 4 inches.

Use even pressure when rubbing the smear on a surface.

Don't press too hard or you will destroy the smear.

Wear gloves when doing a contamination survey, you are looking for removable contamination from surfaces - you might find some.

When measuring the dose rate in an area, hold the meter at the location of measurement for at least 30 seconds. Record each measurement when it is taken. The typical height above the floor at which to take measurements is approximately 1 meter.

Evaluation of Smears

All measurements that are documented for purposes of radiation protection must be made with equipment that has calibration traceable to the National Institute of Standards and Technology. We have several analysis systems that meet this qualification. All Liquid Scintillation Counters, the Tennelec Gas Proportional Counter in MSM-145, and the Berthold Gas Proportional Counter in MSM-167 have traceable calibration.

Special Situations

In case of spills, cleanup should be done as soon as practical and the area affected should be controlled to prevent the spread of contamination. Most importantly, after the spill is cleaned up, the survey that demonstrates the area to be free of contamination must be made with equipment that has calibration traceable to the National Institute of Standards and Technology.

Newsletter 29 – Fume Hoods in the HRC Radiochemistry Program

Chemical fume hoods in the radiochemistry laboratories provide us with a great deal of protection during our work. There are currently 7 fume hoods with HEPA filtered ventilation and 6 without filtered ventilation in our laboratories. It is important to know the limitations of the fume hoods that we are working in so that we do not have a release of radioactive materials from the laboratories and so that we do not cause damage to the systems or a contamination control problem in the hoods or in the laboratories.

Laboratory fume hoods are the first defense to minimize chemical exposure to research workers. They are considered the primary means of protection from inhalation of hazardous vapors. It is, therefore, important that all potentially harmful chemical work be conducted inside a properly functioning fume hood. To ensure safety, all fume hoods are evaluated for flow rate by the safety organization annually. This newsletter is intended to help identify fume hood types and outline exposure control practices in relation to the hood.

All fume hoods currently at the HRC are <u>conventional</u> hoods. This term is used to describe a constant air volume (CAV) hood, an older, traditionally less elaborate hood design used for general protection of the worker. Because the amount of exhausted air is constant, the face velocity of a CAV hood is inversely proportional to the sash height. That is, the lower the sash, the higher the face velocity. However, not all hoods in the HRC operate the same way. Some have an exhaust discharge point that is high in the hood body and some exhaust air from the lower back of the hood.

The HF hood in MSM-164 exhausts from the lower back. A drawback to this design is associated with the flow of air around anything that is between the exhaust point and the sash. Turbulence within the hood is undesirable and may cause a flow of air from the apparatus that you want to ventilate. For this hood, keep clutter inside the hood to a minimum and do not block the lower back exhaust port.

Using a Fume Hood

There are several types of protection that fume hoods provide us, so let's look at some of those.

- 1. A fume hood provides a sash that can be pulled down to a level where it provides shielding from splashes of caustics, acids, or low energy emissions such as beta particles and very low energy photons.
- 2. A fume hood has a ventilation system that pulls air into the hood and away from us. This prevents vapors and airborne particulates that may have been created by your samples from coming into your air space preventing airborne exposure to aromatic compounds and airborne chemicals or radioactive materials.
- 3. A fume hood provides some degree of splash protection for uncovered parts of the body. This protection may also be appropriate should there be an energetic reaction with your material that may instantaneously over pressurize the hood.
- 4. A fume hood provides services that may be required for your experiments such as electrical outlets, gas supply, water supply, etc.
- 5. Flammable and corrosive cabinets typically comprise the bottom supporting structure of the fume hood. They are vented or non-vented enclosures used primarily for storage of flammable or corrosive materials. If vented, the flammable storage cabinet is connected to the hood exhaust.

Fume Hoods in MSM-164

There are two fume hoods in MSM-164, as you face them; the one on the left is a normal HEPA filtered hood that is useful for general radiochemistry and the one on your right is a HEPA filtered hood designed for use with Hydro-Fluoric acid. This is the only HF certified hood that we have and if you want to use HF, you must have HF training and you must have another HF trained person with you when you do the work.

Fume Hoods in MSM-165

There are three <u>unfiltered</u> fume hoods in laboratory MSM-165 all of them are for use of radioactive materials. Because these have unfiltered exhaust, they can only be used with low level radioactive materials.

As you face the three hoods, the hood on the far right is designed for use of perchloric acid. All of these hoods are very useful for chemistry experiments that require only small amounts of radioactive material. The sample activity that you work with in these hoods should typically be less than 10 micro-Curies and be in solution or in a solid mass that will not likely become airborne.

All unfiltered hoods represent a possible release point for radioactive materials or chemicals that are not contained. Because of the ease with which activity could be released from these hoods, it is necessary to ensure that radioactive materials will not be removed from your experiment and go up the stack.

Fume Hood in MSM-167

There is one unfiltered hood in MSM-167, it is approved for use of radioactive materials and is a smaller hood than is in MSM-165. Since this room houses analysis equipment and the hood is unfiltered, it is desirable to use only very low level radioactive materials in this room to minimize the impact on any instrumentation and prevent a release to the outside.

Fume Hood in MSM-172A

There is one unfiltered hood in MSM-172A, it is approved for use of radioactive materials and is a smaller hood than is in MSM-165. Since this room houses analysis equipment, it is desirable to use only very low level radioactive materials in this room to minimize the impact on any instrumentation.

Fume Hoods in MSM-173

The fume hoods in the technetium laboratory (MSM-173) are HEPA filtered and the most desirable hoods to use higher levels of activity. The ventilation from these hoods goes through a double HEPA filter train before being released.

Fume Hoods in MSM-234

There are two fume hoods in laboratory MSM-234, one is a perchloric acid hood and the other is a HEPA filtered general chemistry hood. As in any unfiltered hood, the activity in the perchloric acid hood must be limited to prevent an uncontrolled release of activity from the laboratory. The HEPA filtered general chemistry hood will of course support higher levels of activity.

One more item about the perchloric acid hood in MSM-234; DO NOT OPERATE THE WATER FAUCET – THERE IS NO DRAIN IN THIS SYSTEM. This also applies to the wash-down system on this hood. Without the ability to collect the water, it is necessary that a collection system be provided when it is necessary to do a wash-down.

Fume Hoods in MSM-236

There are two fume hoods in laboratory MSM-236; both are HEPA filtered general chemistry hoods.

TIPS FOR FUME HOOD SAFETY

1. Conduct all operations that may generate air contaminants at or above the appropriate Threshold Limit Value TLV inside a filtered hood. The TLV for a chemical may be found in its MSDS. Radionuclides that may become airborne in excess of the DAC value must be used in a HEPA filtered fume hood.

2. Keep all apparatus at least 6 inches back from the face of the hood. A stripe on the bench surface is a good reminder.

3. Users should always keep their faces outside the plane of the hood sash.

4. Hood sash openings should be kept to a minimum. Hoods are tested (and should be used) with a hood sash opening of 15 inches.

5. Do not use the hood as a waste disposal mechanism except for small quantities (< 10 ml) of volatile materials.

6. Do not store chemicals or apparatus in the hood. Store chemicals in an approved safety storage cabinet.

7. Keep the slots in the hood baffle free of obstruction by apparatus or containers.

8. Minimize foot traffic past the face of the hood to prevent disruptions in air flow.

9. Keep laboratory doors closed when working in the hood.

10. Do not place electrical receptacles or other spark sources inside the hood when flammable liquids or gases are present. No permanent electrical receptacles are permitted in the hood.

11. Use an appropriate barricade (e.g. a blast shield) if there is a chance of explosion or implosion.

12. Do not remove hood sash or panels except when necessary for apparatus set-up; replace the sash or panels before operating.

Newsletter 30 – Risks associated with Radiation Exposure at HRC

More than one hundred years have passed since Roentgen discovered Xrays in 1895. Since then, radiation has become widely used for medical and industrial purposes and is a byproduct of energy generation, communication, and electronic components. We are exposed to manmade radiation through its medical uses, by radiation-emitting products, employment in industries using radiation or radioactive materials, and nuclear weapons.

Sources of Radiation Exposure

Most of the dose received by members of the general population comes from natural and not man-made sources. These natural sources include cosmic rays, terrestrial radiation, and internally deposited radionuclides. Radon, a decay product of uranium-238, is the largest contributor to population dose. Radon decays to several short lived daughters that are inhaled and internally irradiate the lung. Estimates of total radiation exposure for the United States show that radon contributes over half (50%) of the estimated effective dose, and man-made sources contribute less than 20%.

Health Effects Research

Research into the health effects of radiation exposure has been conducted since the early 1900's. Radiation burns and radiation sickness were recognized in the operators of early X-ray machines. Radiation-caused skin cancers were also observed. By the mid 1900's, the potential for external irradiation and internally deposited radionuclides to cause cancer at other sites was documented. The most famous episodes involved the radium dial painters in the United States, and the survivors of the Hiroshima and Nagasaki atomic bomb blasts in Japan.

The effects observed as a result of these high exposures indicate that even low radiation exposures may cause damage to our bodies. Limits for exposure to radiation set by the federal government are set at a small fraction of the dose that effects have been observed. The occupational limit for whole body exposure to ionizing radiation is 5000 mrem/year.

Studies of people exposed to low levels of radiation have been in progress for many years involving hundreds of thousands of workers. Even though there is no indication that low doses of radiation cause us harm, the mechanisms for damage to our bodies cannot be quantitatively defined in the presence of other mechanisms that damage our bodies. Some of the many other damage mechanisms are smoking, drinking, intense sports, driving, and even walking across the street.

What happens if a human is exposed to a high dose of radiation to the whole body all at once?

• For less than 25,000 millirem, there are no directly observable effects. There are changes in some human cells that can be observed with a microscope at exposures above 10,000 mrem.

• 25,000 to 50,000 millirem, there will be no symptoms, but there might be some changes in the chemistry of the individual's blood.

• 100,000 to 300,000 millirem, some physical changes (such as skin reddening and temporary hair loss) are seen, particularly at the high end of the range.

• 300,000 to 1,000,000 millirem, vomiting is the first symptom, and the human loses his/her ability to produce blood. At the upper end of this range, bone marrow transplants are generally needed and, if medical care is not available, the condition can be fatal within one month of exposure.

• 1,000,000 to 5,000,000 millirem, there will be vomiting, loss of blood production, and failure of the gastrointestinal system. In general, an acute dose of this magnitude is fatal within two weeks.

• Greater than 5,000,000 millirem, central nervous system failure is likely, and death will occur within a period of days.

How much dose is received by occupational radiation workers and other people working at HRC?

Monitoring of people on campus has shown that in all areas of research and at locations where radiation producing machines are used, the average annual dose to anyone (including members of the RSO staff) is less than 5 millirem/yr.

The highest dose received by any individual, monitored for occupational exposure at the Harry Reid Center for Environmental Studies during 2008 was 25 mrem.

Risks From Low Doses

Generally speaking, no observable risks or effects are seen from either acute or chronic doses of less than 25,000 millirem. Statistical methods are used to predict the likelihood of long-term effects, such as cancer, for large populations exposed to low doses. Unfortunately, there are complications, such as natural incidence of cancer and cancer caused by other agents such as smoking, that make these evaluations difficult. The radiation protection industry, for a number of years, has conservatively assumed that there is some risk associated with any radiation dose, no matter how small. We talk about this in training as ALARA, we maintain our dose As Low As Reasonably Achievable. The risk is assumed to increase linearly with dose, meaning the higher the dose, the greater the risk. Based upon these very conservative assumptions, the risk of dying from cancer as a result of a 1,000 millirem radiation dose is 5 in 10,000 or 0.0005.

Is a risk of 0.0005 a large risk?

When compared to the types of risks people incur every day of their lives, that risk is actually quite small. For example, the U. S. Department of Labor gives the following lifetime probabilities of death:

- Cancer 0.35 Highway vehicles 0.25
- Heart attacks 0.11 Falls 0.11
- Electrocutions 0.10 Explosions 0.04
- Airline Crash 0.03 Fires 0.01

Most of us face more significant risks associated with our normal life style, like driving to and from UNLV, than we do on our job working with radioactive materials or radiation producing machines. However, one of the fundamental principles of radiation protection is that no radiation dose is acceptable unless there is a corresponding benefit associated with that dose that is at least as large as the risk.

The National Safety Council maintains a web site indicating statistics associated with fatal injuries (both accidental and intentional). If you have more interest in risk, take a look at the site: <u>http://www.nsc.org/lrs/statinfo/odds.htm</u>

Like any job where there are hazards, work in a field where you are exposed to radiation involves some degree of risk, is a personal choice. If you feel that the risks are too high, then maybe you should not be working with radiation. However, ensure that you properly evaluate the risks associated with any other job. Many people who are exposed to radiation at power plants, in hospitals, in the military, or in universities have decided that the risks of working around radiation are acceptable. Newsletter 31 – Labeling Radioactive Material

Consider the following situation: (You are authorized to use radioactive materials.)

You are in your laboratory cleaning up after an experiment and you see a container that has eluded you for a few years, a residual package from a previous researcher. The package has no markings on it and appears to be intact. You pick up the package and open it. A puff of dust permeates the room and you notice that the inner package has a small trefoil symbol on it. You use a contamination survey meter and identify that the area is contaminated, the room is contaminated, you are contaminated, and you most likely have sustained an intake of radioactive material.

How do I know what I was exposed to?

The simple answer is - you don't! The label didn't tell you that there was radioactive material in the container, it did not tell you how much, or what radionuclide you were exposed to, and sometimes more important; what chemical compound were you exposed to? An evaluation will have to be made of the material, the work area, (possibly outside the work area), and your body (to determine the magnitude of the intake and the associated dose that you received).

What do I do now?!

Call the RSO! This may be a serious exposure with long-term consequences, not the least of which may be a call to State regulators. In any case, the material will have to be checked and inventoried by the RSO.

How can I prevent this type of accident from happening?

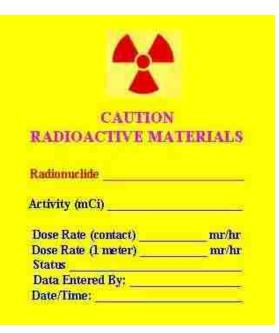
A simple label on the outside of the package could have prevented exposing you to the radioactive dust or a chemical hazard.

If you find such a container or package in your laboratory or work area, you really have no need to open it. If after discussion with the laboratory manager and the RSO, you decide to open it, then do so under controlled conditions. Put on gloves! Put it in a filtered hood or other protective enclosure! Use a frisker to check for contamination, or the presence of a gamma or energetic beta source within the package. An operating glove box or filtered hood should provide adequate control for opening a package containing an unknown. Ensure that you are appropriately protected and other persons in the area are also well protected. Note: the RSO must be notified immediately any time that radioactive material is found. Finally, label the package, container, and/or contents with all the information you have regarding it.

What should the label read?

A label for radioactive material should be similar to the following:

Indicate the radionuclide or radionuclides, the activity in the package, and the dose rate on contact and at 1 meter, indicate the status of the package (If you don't want someone to open it, write "**DO NOT OPEN**" in bold letters on the package). Indicate who you are and the date and time you created the label.



What materials should be labeled?

Certainly, all radioactive materials should be labeled with the information indicated above (NAC 459.355-357, inclusive). And you must label all hazardous chemicals with the name of the chemical and the principle hazard. Small vials should be placed in larger, labeled

containers or racks, and all dilutions, fractions, etc. should bear a reference number which will allow identification if YOU are not around. This is especially important if you would not want someone else to open the package!

Some Other Thoughts.

Whenever you leave a material unattended, it should be labeled. If you will be away from the material for any length of time, ensure that the material is stored properly. Provide detailed instructions of what is in the package, how to open the package, how to handle the material, and how to dispose of the material.

Experienced Authorized Users may open "unknowns," although the RSO must always be notified when radioactive materials that we did not know about are found. Assistants and students must contact the AU for the laboratory and should not under any circumstances open an unknown package. Human nature (and scientific curiosity) being what they are, however, surprises will happen. What we can do therefore is ensure that all radioactive materials that we know about are properly labeled.

Newsletter 32 - Safety and Security in the HRC Laboratories

The radiochemistry laboratories in the Harry Reid Center for Environmental Studies (HRC) allow work with radioactive materials by trained personnel who are trusted to protect themselves and others from harm.

Safety

Before you first started to do work with radioactive materials at the HRC you receive training in basic aspects of radiation safety. Let me remind you of some of your responsibilities for safety while you are working in the laboratories.

- 1. Good housekeeping is required where radioactive materials are used. Work areas must be clearly defined and uncluttered.
- 2. Work surfaces shall be covered to facilitate easy decontamination. Bench coverings shall be changed frequently, i.e., weekly, or whenever the covering is noticeably soiled, torn, or contaminated.
- 3. Locate work areas away from heavy traffic or doorways.
- 4. When moving radioactive solutions between approved locations, place the material within covered secondary containers that contain sufficient absorbing material to absorb twice the quantity of liquid.
- 5. You must wear protective clothing when you or others in a laboratory are working with chemicals or radioactive materials. As a minimum, this is a laboratory coat, safety glasses, long pants, and close-toed shoes.
- 6. Radioactive materials shall be stored so as to prevent unauthorized access or removal from their place of storage. The storage shall not create a "Radiation Area" and must be shielded or sealed to keep exposures ALARA. Radionuclides shall not be left unsecured in unoccupied laboratories.
- 7. Containers with radioactive materials for storage, processing, or use, shall be individually and conspicuously labeled. The label must specify the identity of the radionuclide, the estimated activity (amount), the initials of the contact person for that material, and the date. Containers of radioactive material may be placed in properly labeled secondary containers for storage.

- 8. Empty and decontaminated containers must have the label removed or defaced.
- 9. You have a continuous monitoring device that you wear during all work in the laboratories. This device is very sensitive to external radiations and is specifically assigned to you. Do not loan your dosimeter to anyone else, and do not wear anyone else's dosimeter.
- 10. Your experiences in the laboratories may cause you to have a need for training to use Hydrofluoric Acid (HF) or other potentially dangerous compounds. You should always find out about the elements and compounds that you are working with, look in the MSDS for each compound make absolutely sure that you protect yourself.
- 11. Be sure that the gloves that you wear are appropriate for protection from the chemicals you are using.
- 12. If you need to use HF, you must have HF training, you must have a buddy (who is also trained) with you, the buddy must have a tube of calcium gluconate ready to apply should you spill any on you.

Security

- 1. There are several ways that we ensure security of our laboratories and the radioactive materials that we use. On October 1, 2009 we will institute more controls to protect the laboratories – the back hallway will be locked at all times and the main entry (near the parking lot) will be guarded against unauthorized entry. Your Marlock cards will provide you with access. DO NOT – UNDER ANY CIRCUMSTANCES LOAN ANYONE ELSE YOUR MARLOCK CARD.
- 2. In addition to the Marlock permissions, you may have a proximity card that provides you with access to the primary radioactive material research laboratories. Your Marlock card is assigned to you and is ONLY for YOUR use. DO NOT UNDER ANY CIRCUMSTANCES LOAN ANYONE ELSE YOUR PROXIMITY CARD.
- 3. If at any time, you cannot find your Marlock or Proximity card, please call the Radiation Laboratory Director immediately so that your card can be disabled. If you don't, you are potentially jeopardizing our laboratories and radioactive materials.
- 4. Our primary supply of radioactive materials is maintained under the control of the primary Authorized Radioactive Material Users. Access to these materials requires a key and a combination. This area must never be left open and unattended. If you see this area open, call one

of the primary users immediately!

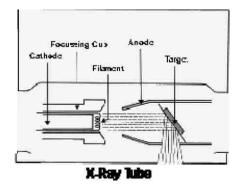
Our work with radioactive materials and potentially dangerous substances is very important. Our trust in each other is essential to the proper protection of ourselves and those who work with us. Our protection of the laboratories and protection of the materials that we work with is essential to ensure that we can continue to do what we do. Newsletter 33 - X-ray Machines

There are many types of X-ray machines used at UNLV, Dental X-ray, Diagnostic X-ray, X-ray Fluorescence, and Crystallography machines.

What are X-rays?

X-rays are electromagnetic radiation similar to light but with a higher energy. They are produced in the electron energy shells of atoms when electrons or other charged particles excite the atoms of a dense material.

How does an X-ray machine make X-rays?



X-ray machines have an evacuated glass (X-ray) tube where a high voltage is put across two electrodes, the negative cathode, and the positive anode. The voltage ranges from several thousand volts to several hundred thousand volts. This diagram shows the typical components of an x-ray tube.

What do these different machines do?

Dental x-ray machines make pictures of the internals of our teeth. They are low energy and low dose rate machines where the distance from the source to film is small and the resulting picture is small.

Medical diagnostic x-ray machines may be used to evaluate our internal bone structures or other structures in our bodies. They are typically higher in energy than dental machines because they have to penetrate a larger depth of the body (and bigger bones) than dental x-rays.

X-ray Fluorescence machines are low energy machines that are used to identify the elements in a sample. The source may be electronic or a source of low energy x-ray emitting radioactive material. The x-rays may be specialized to look for one element (such as lead) or output to a spectrometer to identify several elements in an item. The output is usually higher because the response depends on radiation scattered from the item being analyzed. Fluorescence is a spectro-chemical method of analysis where the molecules of the analyte are excited by irradiation at a certain wavelength and emit radiation of a different wavelength. The emission spectrum provides information for both qualitative and quantitative analysis.

X-ray Crystallography machines are very low energy at extremely high intensity. These machines are designed to observe the structure of molecules through diffraction of the x-ray beam. In order to "see" molecules it is necessary to use a form of electromagnetic radiation with a wavelength on the order of bond lengths, such as X-rays. X-ray crystallography is an experimental technique that exploits the fact that X-rays are diffracted by crystals. It is not an imaging technique.

There are variations of each type of machine in order to enhance certain analyses or reduce equipment costs for a specific purpose. The degree of hazard associated with each machine depends on the radiation safety techniques used to protect the operator. Some machines use key switches and interlocks to prevent inadvertent exposure, and on some machines the exposure time and resulting dose at the operator's position are very low. Training is usually the key to ensuring a low dose to x-ray machine operators.

What are the dose rates from various x-ray machines?

The following table lists the dose rate in Sievert/minute from the different machine types. One Sievert is 100 rads. In this table contact means as close to the source as you can reasonably get. The dose rate at 1 meter is estimated as the 'unshielded' dose rate.

Dose Rate (Sv/minute)

Machine Type	Dental	Medical	Fluorescence	Crystallography
Contact with	0.01	0.1	10	1000
tube				
At 1 meter	0.00001	0.0001	0.1	10

Also, different medical procedures deliver different doses to patients as shown in the following table:

Diagnostic Procedure	Typical Effective Dose (mSv) ¹	Number of Chest X rays (PA film) for Equivalent Effective Dose ²	Time Period for Equivalent Effective Dose from Natural Background Radiation ³
Chest x ray (PA film)	0.02	1	2.4 days
Skull x ray	0.07	4	8.5 days
Lumbar spine	1.3	65	158 days
I.V. urogram	2.5	125	304 days
Upper G.I. exam	3.0	150	1.0 year
Barium enema	7.0	350	2.3 years
CT head	2.0	100	243 days
CT abdomen	10.0	500	3.3 years

What dose do machine operators receive?

If operators of X-ray machines are cautious and use time, distance, and shielding appropriately, they will receive no measurable radiation dose from most X-ray procedures.

The highest dose received by UNLV employees and students is from fluoroscopic diagnostic X-ray machines and is incurred when they are assigned off campus to clinical work in hospitals. Fluoroscopy machines are essentially an X-ray movie where the beam is on continuously to allow a physician to observe movement of body components or materials in the body. Since the patient may have to be moved around on the X-ray table during the 'filming', the technicians moving the patient are closer to the X-ray beam and receive higher doses. There are ways to reduce the exposure even with fluoroscopy, such as reducing the beam 'on time'. For example, if a machine puts out half the number of X-rays in the same 'on time', the dose to everyone (including the patient) is cut in half.

For low energy machines such as dental X-ray, fluorescence, or diffraction machines the dose is primarily to the skin (shallow dose). If directly exposed to the beam from a florescence or crystallography machine for even a small time period may cause burns. The information gained by the use of X-rays is extremely valuable but care must be taken to get that information. The highest dose is received when you must repeat a procedure many times to get the information that you need. Try to do it right the first time, take your time, think it out and prevent unnecessary exposure. Newsletter 34 – Some Good Work Practices

I was walking through the laboratories on my standard waste pickup on Wednesday and took some photos of conditions in the laboratories that should not happen. This led me to the topic for this month, some good work practices.

In a discussion of good radiological work practices, highlighted undesirable situations should help to understand what might be better.

Some pictures

This was the first area that caught my eye. This picture shows items piled into a yellow tray with a "Radioactive Material" pad. This pad represents a contaminated area. The items piled into that area may not have been contaminated, but now they must be considered contaminated.



This picture represents a similar situation, clean items in a potentially contaminated area, but also shows some potentially contaminated pads flowing off of the area. This could lead to dripping of radioactive liquids from the pads and an uncontrolled spill.



When you set up a contaminated area, keep things that are not contaminated from getting contaminated, and keep the number of things that could get contaminated to a minimum. Vial covers in the "disposable centrifuge tube" box should not be inside the potentially contaminated area.



Tube trays and holders should be outside the contaminated area unless they are holding tubes with contaminated liquid in them, or they are contaminated from previous experiments. In this case, they are just taking up room in the contaminated area. In this picture, radioactive solutions are in a tube holder, but there is no absorbent in the tray and nothing to indicate that the tray is a potentially contaminated area (no blue pad).



Any materials brought into one of the higher activity laboratories should be taken out of packaging in a clean area, and the packaging disposed of as clean waste. In this case, in MSM-173, packing materials on some items had to be disposed as radioactive waste.



Your reference materials and notebooks should be maintained free of contamination at all times, if possible. There is never a reason to put a document in a contaminated area. The pen, sharpie, and tape should also be maintained as non-contaminated.





We have always said that blue pads represent a contaminated area and that green represents a clean area. In this picture a green pad is used for radioactive liquids and the tray to the right with radioactive solutions, has no liner.

There seems to be a lot of confusion about what to use as a tray liner, so here is the desired plan.

- 1. Use blue pads with the words "Radioactive Materials" to provide an absorbent surface for radioactive liquids should they spill.
- 2. Use green pads for non-radioactive liquids
- 3. Don't use absorbent pads if you do not have liquids in the tray. A spill of a powder onto the absorbent pads could make the spill worse.

As always, be considerate to others in the laboratory, be a part of the friendly environment that lets us complete our research with minimal interference as a result of poor forethought on the part of someone else.

Keep your work areas clean and non-contaminated, use time distance, and shielding to reduce your external dose, minimize your intake of radioactive materials via inhalation and ingestion by preventing radioactive materials from entering your breathing zone.

- 1. You must survey your work area each time that you do work there.
- 2. Always prepare your samples in a radiologically clean area.
- 3. You are part of your work area; always check yourself for contamination before leaving the work area.
- 4. Prevent contamination from leaving the laboratory area by verifying that you are not contaminated using the PCM.
- 5. Prevent contamination from leaving the laboratories by surveying all items before you take them out of a laboratory.

Newsletter 35 – Wearing Dosimetry

One requirement that is most important to a radiation safety program is monitoring the radiation dose that any radiation worker receives. From a regulatory standpoint we monitor dose to ensure that people don't receive dose in excess of limits and that they maintain their dose As Low As Reasonably Achievable (ALARA). In addition, the radiation safety office will periodically take bioassay samples to ensure that we minimize intake of radioactive materials.

Who wears dosimetry at the HRC?

Any person working with radioactive materials or radiation producing machines at the HRC or in the radio-chemistry laboratories at the SEB who may receive a dose in excess of 500 millirem in a year is required to wear dosimetry. However, we typically provide dosimetry to many more than is required to demonstrate that doses are low in our laboratories.

How do I get dosimetry for a new radiation worker?

You can contact the Radiation Safety Office at 5-4226 or visit their web site to download a form <u>Form # 09 - Radiation Dosimetry Request</u>. Just fill out the form and return it to the RSO. They will request your new dosimeter and if necessary provide you with a badge immediately.

How often are dosimeters exchanged?

Every two months the radiation safety office collects our old dosimetry badge and exchanges it for a new one. The badges are sent to Landauer (the dosimetry company that UNLV uses) for processing. The results are typically received in 15 days or so after Landauer receives the badges. Remember that these results are for the previous 2 month monitoring period. It is possible to request an "emergency" reading of a dosimetry badge, for which Landauer charges an additional \$50.00. This is done only if a very high dose is expected and has never been done at UNLV.

What if my badge is lost?

When you lose your dosimeter, the RSO will need to provide you another dosimeter – but in order to ensure that they have a reasonable record of your dose (from the time that the lost badge was worn); they will need to have a Lost Badge Report filed and evaluated by the RSO.

What if I am exposed to radiation at more than one job?

Some people are exposed to radiation at UNLV and at another place where they work. Those people wear the UNLV dosimeter for UNLV (or student) functions and their other dosimeter at the other work function. Only one dosimeter is worn for each job.

How is my dose controlled if I am exposed at two locations?

The only way that dose can be controlled when you work at two locations is an effort by YOU to ensure that the RSO at both locations knows that you wear dosimetry at another location. Then the RSO's can communicate information to ensure that you do not exceed dose limits. If the locations are both on campus, then the same badge issued by the UNLV RSO should be worn in both places unless different instruction is received from the UNLV RSO.

What if I have declared my pregnancy?

This is one of the few times that the RSO may provide someone with two dosimetry badges. The badge monitoring the mother is worn on the part of the body expected to receive the highest dose to the mother and the other is worn on the front surface of the baby's location under any protective equipment (example: a lead apron). Any specific concerns about exposure of an unborn child should be discussed with the RSO. Newsletter 36 - Surveying Your Workspace

A responsibility that we all have when working with radioactive materials is to ensure that our radioactive materials do not affect others. We do this by preventing the spread of radioactive contamination from our work areas. The only way that we can accomplish this is by using some method to identify the presence of the activity in and around our work areas each time we use radioactive materials.

What is a survey for?

A contamination survey simply identifies the location of radioactive material in the area surveyed.

What are the main methods of finding out where contamination is?

This depends on the radionuclide emissions that we desire to evaluate, what form the radionuclide(s) are in, and what methods we have to measure the emissions.

Radionuclides in our laboratories could be in solutions, in powders, or in solids. The emissions from these materials could be electrons, photons, or alpha particles. Also, we have a few different detectors that may or may not be appropriate for the measurement that we may need.

What are direct measurements?

Direct measurements of contamination are made using portable instruments that can be moved over a work surface and the rate of detection for particles can be evaluated by a meter, or by sound. The 'click' that is made by the speaker on portable detectors is a direct indication of an interaction in the detector.

Direct measurements are useful for immediate indication of high concentrations of activity on item, bench tops, floors, or work areas. There are two portable detectors that are currently used by our program to locate emissions from radionuclides:

To do a direct measurement survey, check the instrument for operability (see the next section), measure emissions from the work area, adjacent areas, the bench in front of you, the floor under where you were standing, and your clothing. To measure the emissions, hold the probe at 1 cm from the area you want to check, move the probe slowly, 3 to 6 cm per second is a reasonable speed. While the probe is in motion, pay attention to the sound of the meter. If you hear an increase in the frequency of the 'clicks', move the probe back over that area and hold the probe there for about 30 seconds to assess the magnitude of the increase.

If the increase is three times the background count rate, then consider that area to be contaminated, wipe the area and throw the wipe in the solid radioactive waste.

Ludlum Model 3

The Ludlum Model 3 rate-meter with a 44-9 Geiger probe is currently used in our program for evaluation of emissions from alpha and/or beta emissions from most of the radionuclides that we use. It is useful for detection and measurement of beta radiation and detection of gamma radiation or alpha radiation. This detector has a small sensitive area; the window has a diameter of 4.75 cm, an overall area of 17.7 cm^2 .

The detection efficiency for ⁹⁹Tc beta emissions for the model 3 is approximately 10% (0.1 counts/transformation), so a measurement of a surface activity concentration of 1000 dpm/probe area would be approximately 100 counts per minute. With a background count rate of 100 counts per minute, this is the lowest activity that could be measured by the Model 3 with some small degree of confidence.

This instrument is not appropriate for measurement of alpha emissions because they are not consistently detected due to variations in the window thickness of the detector. It is also not appropriate for measurement of high energy photon emissions because of the small sensitive volume of the detector.

Some more specific information about the Ludlum Model 3 Geiger counter is provided in Newsletter HRC-24.

Ludlum Model 2360

The Ludlum Model 2360 rate-meter/scalar with a 43-93 alpha/beta scintillation probe is useful for measurement of both alpha and beta emissions from most of the radionuclides that we use. It is not useful for measurement of gamma emissions although it will detect them. The 43-93 detector has a 100 cm² probe with a very thin mylar window.

The detection efficiency for 99 Tc beta emissions for the model 2360 is approximately 15% (0.15 counts/transformation), so a measurement of a surface activity concentration of 1000 dpm/100 cm² would be approximately 150 counts per minute. With a background count rate of 150 counts per minute, this is the lowest activity that could be measured by the Model 2360 with some small degree of confidence.

This instrument is not appropriate for measurement of high energy photon emissions because of the small sensitive volume of the detector.

Some more specific information about the Ludlum Model 2360 ratemeter with the 43-93 probe is provided in Newsletter HRC-25.

Preparing to Operate an Instrument

1. Always make sure that your instrument is in good condition. No major dents in the case, it looks like it should work (no wires hanging out), etc.

2. Make sure that the instrument has been calibrated in the past year. Check the calibration sticker for the date that calibration is due.

3. Turn the instrument to the BAT position and make sure that the meter reading is beyond the battery OK position in the meter.

4. Turn the knob to the lowest scale and check for a normal background response. For a pancake GM probe, the normal response should be between 20 and 100 counts per minute in a low background area. For an alpha/beta scintillation probe, the normal background response is between 100 to 200 counts per minute.

Portable Instrument Precautions

Radiation detectors are delicate; they are the only way that you can tell if there is ionizing radiation in your area, try to prevent them from damage.

As with any instrument, there are some precautions that you can take to ensure that it provides you with the information that you need and that it continues to work. These are:

1. Never put the instrument in water – it will never work again.

2. When surveying contaminated areas – try not to touch the probe to the surface you are measuring – this will minimize the probability that you will contaminate the probe.

3. Never survey sharp objects; wire, wire brushes, brooms, things that can make a hole in things. This action will cause the window to be broken and the instrument to be non-responsive.

4. Never use the probe as a hammer. There is a floating anode inside the 44-9 Geiger detector that can easily be shaken out of position – maybe even short out. There is a photomultiplier tube in a 43-93 that can shatter – anyway use a hammer for hammering.

5. Try not to drop the meter or detector – dropping the meter could break a circuit board inside the case – dropping the probe may break the window or short out the anode on the 44-9 or break the photomultiplier tube in the 43-93.

6. If there is an accident and you contaminate the detector, remove the detector from normal use and notify the laboratory manager that the instrument is Out Of Service. <u>Usually</u> the instrument can be decontaminated or repaired in less than one hour.

Are there other than direct measurement surveys?

Yes, direct measurement surveys are appropriate to measure the total activity on a surface. However, as you can surmise from the previous discussion, direct measurements with our portable instruments are not adequate to ensure that we are in compliance with the 100 dpm/100 cm², level of concern for removable beta emitting radionuclides and the 20 dpm/100 cm² limit for removable alpha emitting radionuclides.

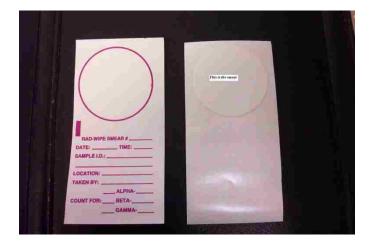
Also, direct measurements are not useful for contamination control if the background response rate is high. A smear survey is capable of providing

a more sensitive evaluation of removable activity on a surface – even in a high background area.

What is a smear?

The smears that we use are small disks of paper with an adhesive back. Each smear comes on a small rectangle of paper with an area to record a smear identification number and some information about the location, date, time, surveyor, and to indicate the analysis method.

The smear is made of paper so if you rub it on rough surfaces like rough concrete or metal, it will tear or shred. It should be used on smooth dry surfaces. Making the smear wet will decrease your detection efficiency for alpha and beta emissions. If you smear a wet area, let the smear dry prior to analysis.



What are smear surveys?

In smear surveys, a smear is rubbed over an area of 100 cm^2 to collect a sample of the removable radioactive material on the surface. One hundred square centimeters is used in order to provide a standard area.

The smear location is indicated on a survey map, usually by writing the smear number on the map at the location where the smear was rubbed.

When you have your smears ready for analysis, take the time to evaluate them with a portable instrument to make sure that they do not have too much activity on them. If they do, they could cause contamination of the low background detector (then it won't be low background). The smear is then analyzed for activity on a detector appropriate for the emissions from the radionuclides expected to be present. In our case, the smear can be analyzed on a low background alpha/beta counter or a low background alpha/beta scintillation detector.

Newsletter 37 – Weekly Plans for Radiochemistry

In the past several weeks we have implemented a program for planning all work with radioactive materials in the radiochemistry laboratories at the HRC and in other areas where work is done for the radiochemistry program. This program was implemented because an authorized Radioactive Material User (User) must be responsible for all work conducted under the authority of the UNLV Radioactive materials license. In previous years we worked under a few different systems that caused confusion among the Users and prevented a strong User knowledge of the work. This newsletter is to more adequately define the planning process and answer some questions that have come up.

What is a radioactive material User?

A User is a person who has been authorized by the UNLV Radiation Safety Officer (RSO) to work independently with radioactive materials. A Radioactive Materials User is a permanent faculty or staff member who has submitted an application to the RSO and has been approved, based on education, experience, and trust to work with licensed radioactive materials at the UNLV main campus, the UNLV Shadow Lane campus, or at a specific (approved) site remote from the main campus. A User must be aware of work that is conducted under their authority and they must be present on campus when that work is in progress.

Who are the Radioactive Material Users that work with the Radiochemistry Program?

There are several radioactive material Users in the Radiochemistry program as follows:

- 1. Dr. Ken Czerwinski,
- 2. Dr. Gary Cerefice,
- 3. Dr. Ralf Sudowe,
- 4. Dr. Thomas Hartmann, and
- 5. Dr. Vern Hodge.

What is in a plan for work with radioactive materials?

The basic form for submitting a plan is simple, the questions to answer are:

What are the dates of your work and who are you?

What radionuclide(s) will you be working with and what do you want to do with them?

How much activity of those radionuclides will be used and where will you use it?

Then check all of the things that you would like to do with the activity and turn the form in to a User.

How is a plan evaluated?

The work plan is reviewed by an authorized User, and if the work looks reasonable without additional controls, then the User checks the "box" at the bottom of the form that indicates, the submission has been "Compared to RMS guidelines and is accepted without additional controls" and signs the form, and puts the information on the board indicating that work may proceed.

A User signature is REQUIRED and your project must be on the board before work may proceed. If you have submitted a plan and your project is not listed on the board, then assume it has not been approved.

What is the planning board? And what is its purpose?

Work within the radiochemistry laboratories requires the knowledge and presence of a radioactive materials User. The white board at the laboratory bay entry is a simple way of identifying who could be working in the laboratories and what they are working on.

Why do I have to submit a new plan each week?

A new plan is required each week so that each User knows that the plan is current and the work described in the plan is accurate. We previously had a board that rarely changed and was not a very good indicator of the work going on in the laboratories. Documentation ensures information is available for the UNLV Radiation Safety Office to evaluate our work.

What if I don't submit a plan and must do work with radioactive materials?

A User MUST be aware of any use of radioactive materials and must approve that use for any time that they are responsible. A User may approve use of radioactive materials as needed.

However, if a User has not approved your plan and you are working with radioactive materials, you are in violation of the UNLV radioactive materials license and will be excused from the laboratories and your reentry will be restricted until the UNLV Radiation Safety Officer, the HRC Laboratory Director, and your supervisor approve your authorization to continue your work.

What if my plan is not approved?

If you have submitted a plan that involves evolutions that may be hazardous to you, others in the laboratory, or may result in release of materials from the laboratory areas, the User evaluation may cause the work to be held up until the work is discussed with you and reasonable controls are put in place to ensure the safety of all. If the work involves significant amounts of activity, a work plan may be required and approval by the RSO may be required.

Remember, the Radiation Safety Officer at UNLV has TOTAL authority to stop work by anyone using radioactive materials on campus at any time. Our license is a broad scope type B license with the State of Nevada and the RSO is the supervisor of all radioactive material use at UNLV.

What about surveys?

If you work with radioactive materials in the radiochemistry laboratories, you have a responsibility to yourself and others in the laboratory bay to prevent the spread of radioactive contamination from your work area. As such, you must do and document surveys to provide compliance with the UNLV Radiation Safety Manual and thus the UNLV Radioactive Materials License(s). If you did not document a survey of your work area(s), then you did not do a survey of the work area! Documentation is required.

How can I learn more about controls for my project?

There are many ways to become familiar with controls that may be applicable to your work as follows:

1. Review the UNLV Radiation Safety Manual for requirements. The UNLV Radiation safety Manual is now specifically indicated as a part of our radioactive materials license.

2. Discuss your project with your supervisor and ensure that you have considered all hazards associated with the materials that you will use.

3. Review the Material Safety Data Sheets for the chemicals that you use in your research; be sure to control the chemical hazards as well as the radiological hazards.

4. Discuss your project with the UNLV Radiation Safety Officer and/or the HRC Radiation Laboratory Director.

A general rule that I have about work with any hazardous materials is: "Make sure that you are comfortable with the controls before you start your project.

Always ask yourself - am I ready?"

Location Number	Decon Factor for Removable Activity	Fraction of Removable Activity	Decontamination Factor for Total Surface Activity	Fraction of Fixed Activity Removed
1	55	0.028	126	0.992
2	259	0.053	63	0.984
3	7078	0.337	71	0.986
4	582	0.234	94394	1.000
5	4565	0.148	107622	1.000
6	1019	0.241	95	0.989
7	207	0.102	45	0.978
8	148	0.075	14	0.927
9	23	0.025	37	0.973
10	92	0.012	25	0.960
11	139	0.085	19	0.948
12	175	0.050	15	0.935
13	80	0.015	31	0.968
14	126	0.022	31	0.968
15	78	0.057	35	0.971
16	57	0.021	5	0.801
17	151	0.015	11	0.909
18	259	0.054	33	0.970
19	291	0.061	13	0.923
20	316	0.121	97	0.990
21	123	0.172	18	0.943
22	4308	0.240	19	0.946
23	1220	0.128	19	0.947
24	538	0.134	12	0.917
25	3996	0.060	32	0.969
26	368	0.035	31	0.968
27	156	0.035	13	0.923
28	352	0.018	18	0.945
29	4250	0.085	13	0.924
30	14	0.161	36756	1.000
31	12	0.055	46	0.978
32	13	0.074	6803	1.000
33	23	0.209	6331	1.000
34	43	0.175	6898	1.000
35	52	0.122	5	0.788
36	179	0.139	2	0.596
37	8	0.120	9	0.885

 Table 3-2. Fraction of Total Activity on the Surface Removed by the Gel

Location Number	Decon Factor for Removable Activity	Fraction of Removable Activity	Decontamination Factor for Total Surface Activity	Fraction of Fixed Activity Removed
38	20	0.079	11433	1.000
39	38	0.253	8598	1.000
40	41	0.144	78	0.987
41	105	0.130	5	0.802
42	84	0.125	4	0.759
43	18	0.098	4	0.760
44	51	0.187	2	0.516
45	83	0.168	4	0.728
46	320	0.147	3	0.707
47	88	0.263	17	0.941
48	65	0.416	14	0.927
49	85	0.296	16913	1.000
50	27	0.128	11433	1.000
51	79	0.137	12094	1.000
52	84	0.241	13323	1.000
53	154	0.297	9	0.892
54	60	0.392	6	0.831
55	113	0.212	5	0.793
56	64	0.285	3	0.675
57	367	0.245	5	0.811
58	139	0.238	3	0.652
59	140	0.282	2	0.593
60	98	0.627	6	0.841
61	70	0.213	7	0.856
62	96	0.221	7	0.864
63	65	0.228	10	0.902
64	149	0.442	15	0.935
65	689	0.424	16252	1.000
66	18	0.271	27780	1.000
68	36	0.136	9638	1.000
69	31	0.139	30	0.967
70	59	0.128	32220	1.000
71	153	0.190	90	0.989
72	425	0.061	37	0.973
73	54	0.208	17575	1.000
74	10	0.010	22583	1.000
75	81	0.275	22772	1.000
76	89	0.013	97	0.990
77	7	0.118	259	0.996
78	106	0.143	45	0.978
79	29	0.394	13	0.920
80	27	0.358	28	0.964
81	193	0.423	74	0.987
82	279	0.558	24	0.958
83	51	0.012	33	0.970

Location Number	Decon Factor for Removable Activity	Fraction of Removable Activity	Decontamination Factor for Total Surface Activity	Fraction of Fixed Activity Removed
84	327	0.208	13	0.924
85	248	0.149	14	0.930
86	321	0.074	18	0.943
87	1727	0.195	13	0.921
89	96	0.033	4	0.762
90	57	0.012	5	0.801
91	49	0.008	11	0.909
92	20	0.002	18	0.943
93	675	0.110	25	0.960
94	2266	0.098	21	0.953
95	236	0.033	19	0.946
97	47	0.042	18	0.944
98	81	0.053	14	0.928
100	283	0.071	15	0.934
101	1145	0.160	9	0.887
102	126	0.101	20	0.951
104	140	0.039	12	0.918
105	476	0.174	7	0.857
106	330	0.127	9	0.884
108	107	0.079	17	0.942
109	401	0.150	5	0.814
110	180	0.166	7	0.851
111	375	0.219	11	0.912
112	241	0.170	48094	1.000
113	694	0.077	17	0.942
114	710	0.446	12756	1.000
115	488	0.310	26740	1.000
116	56	0.031	48	0.979
117	132	0.033	63	0.984
118	1008	0.314	9	0.882
119	1090	0.258	179	0.994
120	1155	0.021	107622	1.000
121	100	0.013	94394	1.000
122	351	0.032	65102	1.000
123	446	0.002	71	0.986
124	201	0.019	126	0.992

Appendix D - A Model Standard Procedure for Radionuclide Recovery

INTRODUCTION

This procedure is a model to identify the specific aspects of a chemical procedure for reuse of radioactive materials that are important to the safety of personnel involved in the operation and those that may be in the area of this work. Each of the measures that should be taken for control of hazardous or radioactive chemicals are common and based on documents cited throughout this model procedure. This is not intended to be an extensive guide to chemical safety; all research facilities have Health and Safety or Risk Management groups with experienced personnel to provide guidance in experimentation with radiochemicals. Personnel who work in radiochemistry laboratories should experience radiation safety training, chemical hygiene training, and specific laboratory training for the research that they will do.

PURPOSE

The purpose of this procedure is to provide guidance for safety considerations appropriate for removal of radionuclides from chemical compounds or mixtures. This guidance is to minimize the hazards experienced or protect personnel from those hazards. The goal is to recover radionuclides from so that they are in more easily stored and used again without presenting a hazard to the laboratory.

RADIOACTIVE MATERIALS

Radioactive materials emit radiation; the degree of hazard associated with their emissions depends on the type of radiation emitted and the energy of that radiation. Those radionuclides that emit gamma radiation or high energy beta radiation may provide more of an external radiation hazard, one in which the emissions have sufficient energy and penetrability to cause an important degree of risk to researchers even though the material is outside of the body. This external radiation risk is controlled by minimizing the time of exposure to high dose rates, maximizing the distance from the source to minimize the dose rate, and using shielding to minimize the dose rate.

Some other materials may have emissions that are not important as an external radiation hazard, but are important if inside of the body. For

example, radionuclides that emit alpha or low energy beta radiation might be important if inhaled, absorbed through the skin or a wound, or ingested. There may also be radionuclides that emit both penetrating and non-penetrating radiation, for example gamma and alpha or alpha emitters in a compound with a light element that may emit neutrons when hit with an alpha particle.

The emissions and their energy are well documented for radionuclides [148]. The appropriate measures to be taken will vary by radionuclide and when working with materials that contain activity in excess of 1 Annual Limit on Intake [149] for the most restrictive intake pathway, inhalation or Ingestion, the Radiation Safety Officer or Health Physicist should be consulted to ensure appropriate controls are used. A control guideline was established for use in the radiochemistry laboratories at UNLV [150].

Once established, methods of proper use will ensure:

Control of radioactive contamination to minimize inhalation or ingestion.

Proper security to prevent theft of radioactive materials.

Exposure reduction by external exposure control methods.

CHEMICALS

Chemistry safety involves active measures to prevent contact with the skin, inhalation, and ingestion. The specific hazards of chemicals are identified in Material Safety Data Sheets published by the manufacturer of the chemicals [151]. The MSDS is required by OSHA guidelines [152]. A second source of information regarding the hazards associated with chemicals is the NIOSH Pocket Guide to Chemical Hazards [153]. With these sources of information a researcher should be able to identify the potential effects of exposure to specific chemicals, the levels which have shown to be safe to work with, and the mechanisms that are reasonable for protection.

The most common hazards associated with chemicals are those hazards that are considered 'characteristic' such as ignitable, corrosive, reactive, or toxic. These classifications are generally applied to hazardous waste [154], but are important for laboratory safety. The measures to protect personnel from these hazards are well known. Training to assist people in protection methods and accident response is common for personnel working in the hazardous waste industry and is required by OSHA as HAZWOPER training [155].

BASIC SAFETY

Know the hazard

Any time chemicals and/or radioactive materials are used, the hazard associated with their use must be known. Review the known safety information about the materials known to be in the residue. For example; the Material Safety Data Sheets (MSDS) or other manufacturer's information [156]. If there is no applicable MSDS, then consider the compounds that will be created by the processes used to return the radioactive residue to a reusable material, the compounds used in the research to make the material, and base controls on all of those.

Throughout the work to recover the radioactive elements or return the material to a more desirable form for reuse, a number of important safety considerations must be made. These may involve controls associated with:

Adding other reactive chemicals and

- Heating
- Cooling
- Mixing
- Freeze drying
- Bubbling
- Evaporating

Each of these methods has its own requirements for safety that may add a degree of complexity to the determination of the total hazard associated with the recovery. If there is a degree of concern for strong energetic release from a mixture, then consult a senior member of the research group or do a test with a small mass of material before using a larger mass.

PLANNING

An evaluation of the material to be recovered should be completed to ensure that adequate control measures have been implemented to provide for the safety of personnel and the facility. Radiation, chemical, and basic safety must all be considered as part of your plan to recover the material. The following safety levels are as defined in the UNLV Risk Assessment and Control Guideline for Radioactive Materials.

The degree of authorization should be commensurate with the importance of the resulting damage should something deviate from the desired outcome. Conditions for including the authorization to proceed might be as described in table B-1.

Table B-27 - Controls for Recovery of Radionuclides from Residues

LEVEL I – User Authorization

The dose equivalent rate at working distance from the material is less than 0.01 mSv/hour, and the limiting activity fraction for level 1 is less than 1.

This level is appropriate for general laboratory conditions where the risk to all personnel is low and typical of most work in the laboratories where the safety of personnel is trusted to the researcher.

LEVEL II - User Authorization

The dose equivalent rate at working distance from the material is less than 0.1 mSv/hour, and the limiting activity fraction for level 2 is less than 1.

This level would be appropriate for laboratory conditions where the risk to all personnel is low and typical of most hazardous work in the laboratories where the safety of personnel is trusted to the Authorized User.

LEVEL III - Radiation Safety Officer Authorization

The dose equivalent rate at working distance from the material is less than 1 mSv/hour, and the limiting activity fraction for level 3 is less than 1.

This level would be appropriate for laboratory conditions where the risk to all personnel is elevated and not typical of work in the laboratories. In these situations, which are rare, participation of the radiation safety staff is important in planning the experiment and the attendance by a member of the Radiation Safety Office may be considered necessary. In this situation it is considered appropriate to minimize the number of personnel working in the lab who may be affected by the operation.

LEVEL IV - Radiation Safety Committee Authorization

The dose equivalent rate at working distance from the material is less than 1 mSv/hour, and the limiting activity fraction for level 4 is less than 1.

This level would be appropriate for laboratory conditions where the risk to all personnel is elevated and not typical of work in the laboratories. In these situations, which not anticipated, participation of the radiation safety committee is important in planning the experiment and attendance by a member of the Radiation Safety Office is necessary. In this type of situation, the laboratory is restricted to only personnel who are involved in the experiment, and that number should be limited.

DOSE RATE CONTROLS

For beta and gamma emitting radionuclides, there may be a consideration for the shallow or deep dose equivalent rates. If the consideration is primarily for beta radiation, then a plexiglass shield between the researcher and the material will probably be adequate. This type of shield is also appropriate for protection from splattering, splashing, or flying debris and may protect a researcher from caustic or thermal burns.

For gamma emitters, an important consideration is the energy of the gamma emissions, a simple method to determine the thickness of shielding required to lower the dose rate is the use of a chart similar to Table B-1. If the dose rate can be minimized by the use of shielding, that action should be taken without reducing the controls which would minimize inhalation or ingestion intake of the material.

CONTROL LEVEL DETERMINATION

Using the guidance of the UNLV Radiation Safety Office, the limiting activity fraction for each level would be as follows. If the Limiting ALI Fraction is not less than 1, then elevate to the next level:

LFi(M) = Limiting ALI Fraction for radionuclide mixture M for Rad Level i*:

$$LFi(M) = SF + \sum_{n=1}^{N} \left(\frac{An}{ALi}\right)$$

EQUATION A-1

* The limiting ALI for radionuclides of interest for each Rad Level at UNLV is shown on Tables B-3, B-4, and B-5: Risk Assessment and Control Guides for Radiochemistry Radionuclides.

n = the index number for each radionuclide considered.

An = the activity of radionuclide(n) that is in the residue.

ALi = the limiting activity of radionuclide(n) for Rad Level i.

SF = A safety factor that may be based on minimizing the activity that will undergo heating, or bubbling. This factor would be a fraction of 1.

SAFETY FACTOR

The safety factor indicated in equation A-1 is added to the ALI fraction as a means of establishing a more limiting set of controls. At this time, while this concept is in its early stages, the values for safety factor (SF) presented in Table B-2.

The establishment of a safety factor for any process that may cause the development of a hazard as a result of the radioactive material or another constituent of a solution or solid is simply a means to allow a stronger control to be applied. The assignment of such a factor may be made by a researcher, an Authorized User, or by Radiation Safety or Hazardous Material Safety Personnel based on their knowledge and experience in dealing with a specific material.

As can be identified from the information in Table B-2, it is recognized that a temperature, degree of mixing or any process at which adverse reactions in unknown solutions will occur is of course unknown. Thus inappropriately establishing a "safety" factor based on limited knowledge is undesirable. The researcher and their supervision should strive to apply a safety factor to prevent situations that will be hazardous to any person in a laboratory. They should use the information that they have about what went into a solution and what may have been created from environmental influences on the solution.

Table B-2 establishes a minimum safety factor based on the simple fact that some tasks have an inherent hazard that cannot be avoided. When items are heated, or stirred, or bubbled, the possibility of contamination spread is higher than processes that will not cause a physical change to the solution. One process that could be considered benign to the solution might be storage unless other influences of heat or cold could affect the material or its container.

Other considerations appropriate to the development of safety practices should also consider the extreme case where explosion or a significant release of radioactive material to the public could occur. In these cases, every effort should be taken to more accurately characterize the degree of hazard before processing the material. Processing a small quantity may be an appropriate practice to evaluate how the material will behave.

Process	Hazard Discussion	Safety Factor
Heating	In heating, the concern is associated with causing the radioactive material to become airborne, splashing out of its container or causing emission of hazardous gas from the solution. Consider multiple factors for heating depending on the fraction of an important temperature (melting point, boiling point) for the material heated. If the possible outcome is simply a warming of the solution then the factor is lower. If the outcome could be volatilization or splashing, then the factor is higher.	0.1 to 1.0
Mixing, stirring, or shaking	In mixing, the aggressiveness of mixing can be associated with improving the probability of a spill of liquids. The safety factor associated with slow mixing with a stir bar could be 0 and the safety factor associated with aggressive mixing in a blender or centrifuge could cause a serious spill of radioactive material if there is equipment failure.	0.1 to 1.0
Freeze Drying	Freeze drying removes water and volatile liquids from a solution. Improper setup and use can cause sprays and spills of process materials or internal contamination of the freeze drier. If a positive outcome of freeze drying is known for a substance, then the safety factor of 0 is appropriate. If there is the possibility that it may not go well consider a higher safety factor.	0.1 to 1.0

Process	Hazard Discussion	Safety Factor
Bubbling	In bubbling a gas through a solution to get a reaction to occur, the bubbling will cause droplets of the solution to splash out of the container unless precautions are taken to avoid this. Bubbling without protection against splashing should not be done, however, if it cannot be avoided consider a higher safety factor.	0.1 to 1.0
Other processes	This list considers only some of the processes that are used in the laboratory for separation of liquids and solids from liquids. Consider the hazard, if there is a likelihood of creating airborne activity or causing a spread of radioactive contamination, apply a safety factor appropriate to abate the hazard.	0 to 1.0

SAMPLE RECOVERY

Consider use of 10 MBq of Am-241 and 15 MBq of Pu-239 for a recovery,

Consider a safety factor of 0.1 because the material will be heated.

A review of the Control Guidelines indicates that this material must be done in a glove box.

Am-241 has a limiting activity at Level 4 of 29.6 MBq (Considering no airborne activity). Am-243 has the same limiting activity.

Applying equation A-1, LFi(M) = 0.1 + 10/29.6 + 15/29.6 = 0.945

Since LFi(M) is less than 1, the material can be recovered at RAD LEVEL IV, the dose equivalent rate at one meter from the material is:

H = 10/29.6 • 2.51x10⁻³ mSv/h + 15/29.6 • 1.465x10⁻³ mSv/h = 1.59 μ Sv/h

At RAD LEVEL IV, the recovery should be reviewed by the Authorized User with the RSO to ensure that adequate controls are established during the recovery.

Other items that must be considered are training of personnel involved, documentation of the recovery, disposition of the waste produced, cleanup and contamination control surveys.

METHODS

This section provides some specific safety considerations that would depend on the specific methods that might be considered for a recovery. While these items are appropriate for safety associated with the selected process, they may not be the only safety measure that should be taken. Protective clothing for example, will only provide appropriate protection when it is intact. The researcher must make continuous observation of the process so that unexpected situations are readily identified and actions can be taken to provide protection.

Analysis

In analysis of materials, it is possibly more important to provide protection to the instrument so as to avoid damage. It is also important to recognize that many of these instruments operate on high voltage and may have very hot or cold surfaces. These three hazards are some of the most important to be aware of. Knowing the presence of the hazard is important, knowing how to protect yourself from it is more important. Review some of the methods that are used in the laboratory and the hazards associated with them. Consider the methods and protection in Table B-2.

	Gamma Constant	RAD LE	VEL 1	RAD LE	VEL 2
Nuclide	mSv/hr/MBq/m ²	Less Than (MBq)	Maximum Dose Equivalent Rate (mSv/hour)	Not Airborne & Less Than (MBq)	Maximum Dose Equivalent Rate (mSv/hour)
Am-241	8.479E-05	4.921	4.173E-04	0.00037	3.137E-08
Am-242m	4.950E-05	4.921	2.436E-04	0.00037	1.832E-08
Am-243	8.456E-05	4.921	4.161E-04	0.00037	3.129E-08
Ba-133	1.231E-04	0.10582	1.303E-05	25.9	3.188E-03
Cd-109	4.983E-05	0.2775	1.383E-05	1.48	7.375E-05
Cm-244	1.741E-05	3.7	6.442E-05	0.00037	6.442E-09
Cm-248	1.227E-05	3.7	4.540E-05	0.000074	9.080E-10
Co-57	4.087E-05	0.21127	8.635E-06	25.9	1.059E-03
Co-60	3.703E-04	0.24679	9.139E-05	1.11	4.110E-04
Cs-137	1.032E-04	0.0185	1.909E-06	3.7	3.818E-04
Eu-152	2.012E-04	1.48	2.978E-04	0.74	1.489E-04
Eu-154	2.042E-04	0.925	1.889E-04	0.74	1.511E-04
Eu-155	1.804E-05	1.628	2.937E-05	3.33	6.007E-05
Hf-175	6.443E-05	0.12321	7.938E-06	33.3	2.146E-03
I-125	7.432E-05	0.02479	1.842E-06	1.48	1.100E-04
Mn-54	1.382E-04	0.0925	1.278E-05	29.6	4.091E-03
Na-22	3.620E-04	0.02479	8.974E-06	14.8	5.358E-03
Np-237	1.251E-04	4.625	5.786E-04	0.000148	1.851E-08
Pb-210	6.801E-05	0.00185	1.258E-07	0.037	2.516E-06
Po-210	1.424E-09	0.185	2.634E-10	0.0222	3.161E-11
Pu-236	2.405E-05	3.7	8.899E-05	0.00074	1.780E-08
Pu-238	2.135E-05	4.773	1.019E-04	0.00037	7.900E-09
Pu-239	8.145E-06	4.921	4.008E-05	0.00037	3.014E-09
Pu-240	2.030E-05	4.921	9.990E-05	0.00037	7.511E-09
Pu-241	NA	4.921		0.0111	
Pu-242	1.684E-05	4.218	7.103E-05	0.00037	6.231E-09
Ra-226	3.274E-06	0.12321	4.034E-07	0.0222	7.268E-08
Sb-125	1.028E-04	0.148	1.521E-05	18.5	1.902E-03
Sr-85	2.052E-04	0.0555	1.139E-05	74	1.518E-02
Sr-90	NA	0.2775		0.148	
Tc-99	1.242E-10	0.21127	2.624E-11	25.9	3.217E-09
Tc-99m	3.317E-05	0.0148	4.909E-07	2960	9.818E-02
Th-229	1.989E-04	24.679	4.909E-03	0.000037	7.359E-09
Th-230	1.861E-05	24.679	4.593E-04	0.00037	6.886E-09
Th-232	1.848E-05	25.9	4.786E-04	0.000037	6.838E-10
U-232	2.403E-05	9.25	2.223E-04	0.00037	8.891E-09
U-233	7.866E-06	9.25	7.276E-05	0.00148	1.164E-08
U-235	9.159E-05	9.25	8.472E-04	0.00148	1.356E-07
U-238	1.763E-05	9.25	1.631E-04	0.00148	2.609E-08

Table B-9 – Risk Assessment Guide for Radiochemistry – Level 1 & 2

	Gamma Constant	RAD LEVEL 3			
Nuclide	mSv/hr/MBq/m ²	If Not Airborne Less Than (MBq)	Maximum Dose Equivalent Rate (mSv/hour)	lf Airborne Less Than (MBq)	Maximum Dose Equivalent Rate (mSv/hour)
Am-241	8.479E-05	0.00000222	1.882E-10	0.0296	2.510E-06
Am-242m	4.950E-05	0.00000222	1.099E-10	0.0296	1.465E-06
Am-243	8.456E-05	0.00000222	1.877E-10	0.0296	2.503E-06
Ba-133	1.231E-04	0.259	3.188E-05	74	9.109E-03
Cd-109	4.983E-05	0.0148	7.375E-07	11.1	5.531E-04
Cm-244	1.741E-05	0.0000037	6.442E-11	0.037	6.442E-07
Cm-248	1.227E-05	0.00000074	9.080E-12	0.0074	9.080E-08
Co-57	4.087E-05	0.259	1.059E-05	148	6.049E-03
Co-60	3.703E-04	0.0111	4.110E-06	7.4	2.740E-03
Cs-137	1.032E-04	0.037	3.818E-06	3.7	3.818E-04
Eu-152	2.012E-04	0.0074	1.489E-06	29.6	5.956E-03
Eu-154	2.042E-04	0.0074	1.511E-06	18.5	3.778E-03
Eu-155	1.804E-05	0.0333	6.007E-07	148	2.670E-03
Hf-175	6.443E-05	0.333	2.146E-05	111	7.152E-03
I-125	7.432E-05	0.0148	1.100E-06	1.48	1.100E-04
Mn-54	1.382E-04	0.296	4.091E-05	74	1.023E-02
Na-22	3.620E-04	0.148	5.358E-05	14.8	5.358E-03
Np-237	1.251E-04	0.00000148	1.851E-10	0.0185	2.314E-06
Pb-210	6.801E-05	0.00037	2.516E-08	0.037	2.516E-06
Po-210	1.424E-09	0.000222	3.161E-13	0.111	1.581E-10
Pu-236	2.405E-05	0.0000074	1.780E-10	0.074	1.780E-06
Pu-238	2.135E-05	0.00000259	5.530E-11	0.0333	7.110E-07
Pu-239	8.145E-06	0.00000222	1.808E-11	0.0296	2.411E-07
Pu-240	2.030E-05	0.00000222	4.507E-11	0.0296	6.009E-07
Pu-241	NA	0.000111		1.48	
Pu-242	1.684E-05	0.00000259	4.362E-11	0.0296	4.985E-07
Ra-226	3.274E-06	0.000222	7.268E-10	0.074	2.423E-07
Sb-125	1.028E-04	0.185	1.902E-05	74	7.607E-03
Sr-85	2.052E-04	0.74	1.518E-04	111	2.278E-02
Sr-90	NA	0.00148		1.11	
Тс-99	1.242E-10	0.259	3.217E-11	148	1.838E-08
Tc-99m	3.317E-05	1.85	6.136E-05	185	6.136E-03
Th-229	1.989E-04	0.00000333	6.623E-11	0.0222	4.416E-06
Th-230	1.861E-05	0.00000222	4.131E-11	0.148	2.754E-06
Th-232	1.848E-05	0.0000037	6.838E-12	0.0259	4.786E-07
U-232	2.403E-05	0.00000296	7.113E-11	0.074	1.778E-06
U-233	7.866E-06	0.0000148	1.164E-10	0.37	2.910E-06
U-235	9.159E-05	0.0000148	1.356E-09	0.37	3.389E-05
U-238	1.763E-05	0.0000148	2.609E-10	0.37	6.523E-06

Table B-10 - Risk Assessment Guide for Radiochemistry – Level 3

	Gamma Constant	RAD LEVEL 4			
	Sumu Sonotant		Maximum		Maximum
			Dose		Dose
Nuclide		If Not	Equivalent	If Airborne	Equivalent
	mSv/hr/MBq/m ²	Airborne Less	Rate (mSv/hour)	Less Than	Rate (mSv/hour)
Am-241	8.479E-05	Than (MBq) 1.48	1.255E-04	(MBq) 0.0111	· · · · · · · · ·
Am-242m	4.950E-05	1.40	7.326E-04	0.0111	9.412E-07
Am-242m Am-243	4.950E-05 8.456E-05	1.40	1.251E-05	0.0111	5.495E-07
Ba-133	1.231E-04	1.40	2.277E-04	1295	9.386E-07 1.594E-01
Cd-109	4.983E-05	1850	9.219E-01	74	3.687E-03
Cm-244		1.85		0.0185	3.221E-07
Cm-244 Cm-248	<u>1.741E-05</u> 1.227E-05	0.37	3.221E-05	0.0037	
			4.540E-06		4.540E-08
Co-57	4.087E-05	1850	7.561E-02	1295	5.293E-02
Co-60 Cs-137	3.703E-04 1.032E-04	1850 1850	6.851E-01 1.909E-01	<u> </u>	2.055E-02
					1.909E-02
Eu-152 Eu-154	2.012E-04 2.042E-04	1850 1850	3.722E-01 3.778E-01	<u> </u>	7.444E-03 7.555E-03
Eu-154 Eu-155	1.804E-05	1850	3.337E-02	166.5	3.004E-03
Hf-175	6.443E-05	1850	1.192E-01	1665	1.073E-01
I-175					
Mn-54	7.432E-05 1.382E-04	74 1850	5.500E-03 2.557E-01	<u>74</u> 1480	5.500E-03 2.045E-01
Na-22	3.620E-04	1850	6.697E-01	740	2.679E-01
Ng-22 Np-237	1.251E-04	0.925	1.157E-04	0.0074	9.257E-07
Pb-210	6.801E-04	1.85	1.157E-04 1.258E-04	1.85	1.258E-04
Po-210	1.424E-09	5.55	7.903E-04	1.11	1.581E-09
Pu-236	2.405E-05	3.7	8.899E-05	0.037	8.899E-07
Pu-238	2.135E-05	1.665	3.555E-05	0.01295	2.765E-07
Pu-239	8.145E-06	1.48	1.205E-05	0.01233	9.041E-08
Pu-240	2.030E-05	1.48	3.004E-05	0.0111	2.253E-07
Pu-241	NA	74	0.004⊏-00	0.555	2.2000-01
Pu-242	1.684E-05	1.48	2.492E-05	0.01295	2.181E-07
Ra-226	3.274E-06	3.7	1.211E-05	1.11	3.634E-06
Sb-125	1.028E-04	1850	1.902E-01	925	9.509E-02
Sr-85	2.052E-04	1850	3.796E-01	1850	3.796E-01
Sr-90	NA	55.5	0	7.4	
Tc-99	1.242E-10	1850	2.298E-07	1295	1.608E-07
Tc-99m	3.317E-05	1850	6.136E-02	1850	6.136E-02
Th-229	1.989E-04	1.11	2.208E-04	0.00185	3.680E-07
Th-230	1.861E-05	7.4	1.377E-04	0.0111	2.066E-07
Th-232	1.848E-05	1.295	2.393E-05	0.00185	3.419E-08
U-232	2.403E-05	3.7	8.891E-05	0.0148	3.556E-07
U-233	7.866E-06	18.5	1.455E-04	0.074	5.821E-07
U-235	9.159E-05	18.5	1.694E-03	0.074	6.778E-06
U-238	1.763E-05	18.5	3.262E-04	0.074	1.305E-06

Table B-11 - Risk Assessment Guide for Radiochemistry – Level 4

Method	Equipment	Hazard
Analysis	Atomic Emission Spectroscopy	High voltage
		Hot surfaces
		Gasses – Asphyxiates - Ar
		Gasses - Toxic
		Laser reflections
		Sample handling - liquids
	Mass Spectroscopy	High voltage
		Hot surfaces
		Gasses – Asphyxiates - Ar
		Gasses - Toxic
		Laser
		Sample handling - liquids
		High Vacuum
	Gamma Spectroscopy	High voltage
		Cold surfaces - LN
		Sample handling - liquids
		Gasses – Asphyxiates
	Alpha Spectroscopy	Vacuum
		Airborne materials*
	Laser Spectroscopy	Airborne materials*
		Laser reflections*
	UV Vis Photospectroscopy	UV Exposure*
		Sample handling - liquids
		Gasses – Asphyxiates
	FT/iR Photospectroscopy	IR Exposure*
		Sample handling - liquids
		Gasses – Asphyxiates
	Liquid Scintillation Counting	Hazardous cocktails*
		Sample handling - liquids
		Contamination control
	Alpha/Beta Counting	High Voltage
		Flammable gasses – CH ₄
		Gasses – Asphyxiates - Ar
		Contamination control
	Transmission Electron Microscope	High Voltage
		X-ray emissions
		High Vacuum
		Contamination control
	Scanning Electron Microscope	High Voltage
		X-ray emissions
		Contamination control
	X-Ray Diffraction Spectroscopy	High Voltage
	A-Kay Dimacuon Specifoscopy	High dose rates
	Y Pow Fluorescence	
	X-Ray Fluorescence	High Voltage Contamination control
		Contamination Control

Table B-12 – A Sample of Possible Equipment Hazards in Laboratories

Method	Equipment	Hazard
Sample Prepa	aration	
Heating	Furnace	High voltage
		Hot surfaces
		Gasses – Asphyxiates, Toxic
	Hot Plate	High voltage
		Hot surfaces
	Oven	High voltage
		Hot surfaces
	Arc Furnace	High voltage
		Hot surfaces
		IR Exposure*
		Airborne materials*
	Reactor	High voltage
		Hot surfaces
		High Pressures
Cooling	Chilling in Dry Ice	Cold Surfaces
cooming		Gasses – Asphyxiates – CO ₂
	Refrigeration and Chiller Operations	Cold Surfaces
		Liquid under pressure
	Gas Flow	Gasses – Asphyxiates
		Gas under pressure
	Flowing Liquid Coolant	Cold Surfaces
		Liquid under pressure
		Electrical hazard
Other	Freeze Drying	Cold Surfaces
other		Contamination Control
		Vacuum
	Dissolution	Caustic liquids
		Contamination Control
	Dilution	Contamination Control
	Filtration	Dose Rate Increase
		Liquids under pressure
	Stirring	Contamination Control
		Spill Potential
	Ball Mill	Contamination Control
		Heavy moving parts
	Caustic Bath	Contamination Control
		Serious Spill Danger
	Pellet Pressing	Contamination Control
		Airborne Activity Hazard
	Polishing	Contamination Control
		Airborne Activity Hazard
	Grinding	Contamination Control
	Grinding	Airborne Activity Hazard

Appendix E - Data from Experiments for Radionuclide Recovery

This appendix provides the data for experiments done to recover ⁹⁹Tc from residue in solutions that were no longer useful to the research for which they were created. The data for each experiment was collected as it was determined whether the activity in each solution was substantial enough to provide a useful recovery and as the process continued once the decision was made that it would be valuable. The data is primarily in a form that was devised to provide for consistent data collection. Important information about each residue was collected in as simple a way as possible to minimize costs. The result of this evaluation is a mechanism that is applicable to residues from laboratory experiments and provides a low cost method to determine desirability of recovery for radionuclides in the solution.

Residue Numbers 12-27-10-1 and 12-27-10-2

Residues #12-27-10-1 and #12-27-10-2 have a relatively low technetium concentration and limit the mass of ⁹⁹Tc available for recovery. With less than 1 mg of ⁹⁹Tc each, these samples are low volume and can be solidified for waste. Recovery of ⁹⁹Tc from these samples is noted as not recommended. Samples were absorbed into contamination control pads, dried, and disposed as solid waste.

UNLV In-Process Experimental Products Form

Residue Number: #12-27-10-1 & #12-27-10-2

Date of Storage: Unknown

Has a value analysis been completed? (Yes \boxtimes , No \Box)

Is recovery of radioactive material recommended? (Yes \Box , No \boxtimes)

Volume of solution: 100 + 200 mL Solution pH: unknown

Radionuclide (1): ⁹⁹Tc; Mass (1) = 7.28E-1 mg; Act. (1) = 0.5 MBq

Radionuclide (2): ⁹⁹Tc; Mass (2) = 1.72E-1 mg; Act. (2) = 0.1 MBq

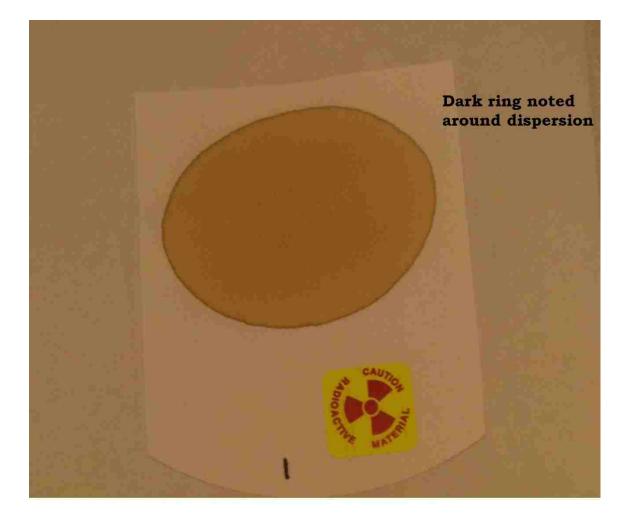
Chemical compound(s): 1 – TBP-TcO4

Chemical compound(s): 2 – Dark liquid/No label

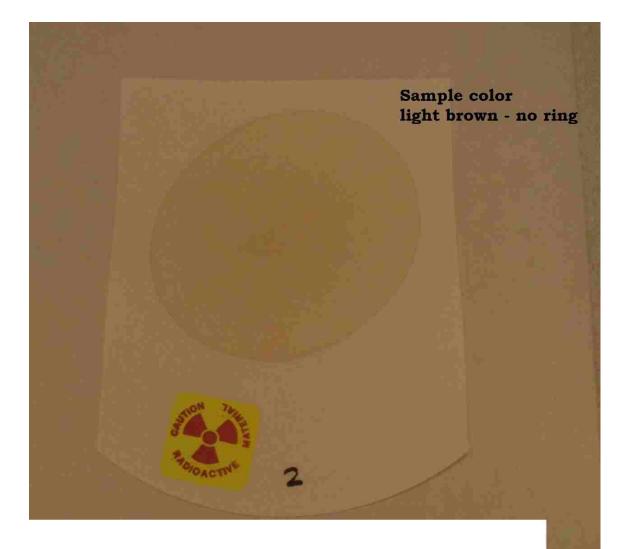
Hazardous Component: Flammable \Box Corrosive \Box Oxidizer \Box Toxic \Box Unknown \boxtimes

If recovery of radioactive material in this product is not desired, is it possible to remove the hazardous component to allow disposal as radioactive waste? (Yes \boxtimes , No \square , N/A \square)

Notify the UNLV Radiation Safety Office? (Yes \Box , No \boxtimes)



Sample 12-27-10-1 100 microliters on Whatman #1 Filter Disk Spot expanded to 5.5 cm by 7 cm GM Net Response at Center of spot = 100 CPM Scintillator Net Response = 3145 CPM - beta No net alpha response

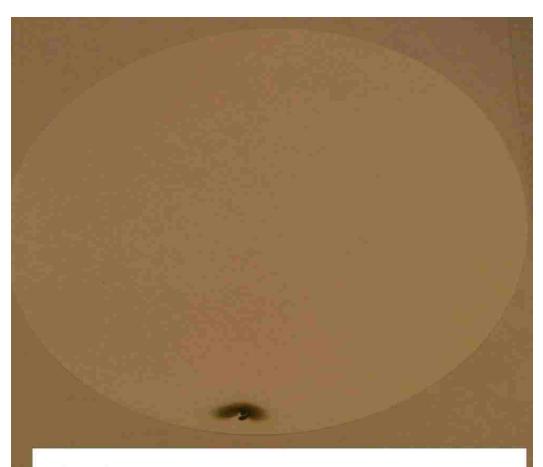


Sample 12-27-10-2 100 microliters on Whatman #1 Filter Spot expanded to 6 cm by 7 cm GM Net Response at Center of spot = 30 CPM Scintillator Net Response = 229 CPM - beta No net alpha response

Residue #12-27-10-3 was labeled "Organic liquids containing Pu & U", and the matrix was indicated as n-dodecane. The activity appeared low from the LSC analyses, but this may be a "desirable recovery effort" since there were no other solutions containing these radionuclides. The residue was a light brown liquid and had no film on the inside of the 125 mL polyethylene container.

UNLV In-Process Experimental Products Form
Residue Number: #12-27-10-3
Date of Storage: Unknown
Has a value analysis been completed? (Yes \boxtimes , No \square)
Is recovery of radioactive material recommended? (Yes \Box , No \boxtimes)
Volume of solution: 30 mL Solution pH: 5
Radionuclide (1): ²³⁹ Pu; Mass (1) = 1.49E-2 mg; Act. (1) <0.1 MBq
Chemical compound(s): Contained both Pu & U in n-dodecane
Chemical compound(s): CAS 112-40-3
Hazardous Component: Flammable \Box Corrosive \Box Oxidizer \Box Toxic \Box Unknown \boxtimes
If recovery of radioactive material in this product is not desired, is it possible to remove the hazardous component to allow disposal as radioactive waste? (Yes \Box , No \Box , N/A \boxtimes)
Notify the UNLV Radiation Safety Office? (Yes \Box , No \boxtimes)

The sample had no color on the filter. The dispersion continued to the extent of the filter and there was no ring around the dispersion. This indicated that the material was not very volatile, not very viscous, and did not have many visible particles in the mixture. The portable instrument response from this residue was similar to background and a decision was made to solidify and dispose of this material as radioactive waste.



Sample 12-27-10-3 100 microliters on Whatman #1 Filter No color on the filter Dispersal through the entire filter paper 15 cm by 5 cm No ring around dispersion GM Net Response at Center of spot = 0 CPM Scintillation detector response = 196 CPM - beta No net alpha response

Residue #12-27-10-4 initially contained 800 mL of an unidentified solution. LSC analysis indicated an estimated 2.1 grams of ⁹⁹Tc.

UNLV In-Process Experimental Products Form

Residue Number: # 12-27-10-4

Date of Storage: Unknown

Has a value analysis been completed? (Yes \boxtimes , No \square)

Is recovery of radioactive material recommended? (Yes \boxtimes , No \square)

Volume of solution: 800 mL Solution pH: 3

Radionuclide (1): ⁹⁹Tc; Mass (1) = 2.15E+03 mg; Act. (1) =1350 MBq

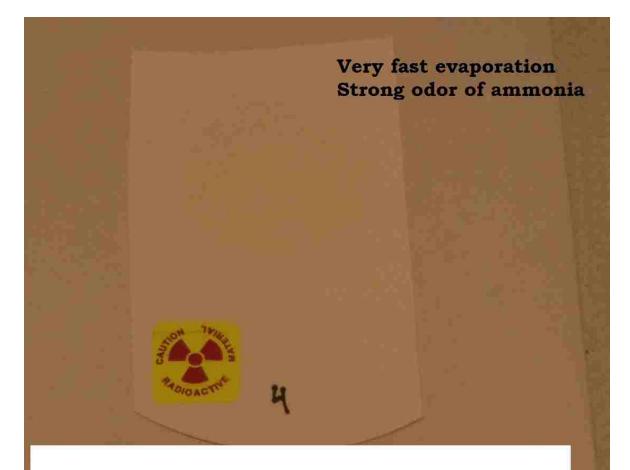
Chemical compound(s): Organic Acid, light brown in color

Hazardous Component: Flammable \Box Corrosive \Box Oxidizer \Box Toxic \Box Unknown \boxtimes

If recovery of radioactive material in this product is not desired, is it possible to remove the hazardous component to allow disposal as radioactive waste? (Yes \Box , No \Box , N/A \boxtimes)

Notify the UNLV Radiation Safety Office? (Yes \Box , No \boxtimes)

If the material was homogeneous in this solution, then it would have a concentration of approximately 2.7 mg/mL. This sample had great recovery potential and could be the source used for collection of data for several recovery techniques for ⁹⁹Tc. There was no film on the inside of the container and a strong odor of ammonia emanated from the container. The sample was colorless on the filter. The dispersion continued to an ellipse of 4.5 cm by 4 cm, without a ring around the ellipse perimeter, and it evaporated relatively quickly. This information indicated that the material was more volatile, not very viscous, and did not have many visible particles in the mixture.



Sample 12-27-10-4 100 microliters on Whatman #1 Filter Very light yellow color on the filter No ring around the dispersion GM Net Response at Center of spot = 10,500 CPM Scintillation detector response = 154,395 CPM - beta No alpha response

Residue #12-27-10-5 was 300 mL of organic solvent containing an estimated 1.75 grams of ⁹⁹Tc in solution. The solution appears to be homogeneous, and has a concentration of approximately 5.8 mg of ⁹⁹Tc/mL. This sample had great recovery potential and could also be a source used for collection of data for several recovery techniques. There was no film on the inside of the container.

UNLV In-Process Experimental Products Form

Residue Number: #12-27-10-5

Date of Storage: Unknown

Has a value analysis been completed? (Yes \boxtimes , No \square)

Is recovery of radioactive material recommended? (Yes \boxtimes , No \square)

Volume of solution: 300 mL Solution pH: 3

Radionuclide (1): ⁹⁹Tc; Mass (1) = 1.75E+3 mg; Act. (1) = 1100 MBq

Chemical compound(s): Organic, dark brown colored liquid

Hazardous Component: Flammable \Box Corrosive \Box Oxidizer \Box Toxic \Box Unknown \boxtimes

If recovery of radioactive material in this product is not desired, is it possible to remove the hazardous component to allow disposal as radioactive waste? (Yes \Box , No \Box , N/A \boxtimes)

Notify the UNLV Radiation Safety Office? (Yes \Box , No \boxtimes)

The sample color on the filter paper was light brown. The dispersion continued to an ellipse of 4.8 cm by 6 cm with no visible ring. This information indicated that the material was volatile, not very viscous, and did not have many visible particles in the mixture.



Sample 12-27-10-5 100 microliters on Whatman #1 Filter Light brown color dispersion with green tint Light green ring around the dispersion GM Net Response at Center of spot = 44,000 CPM - beta Scintillation detector response = 789,244 CPM - beta Scintillation detector response = 1 CPM - alpha

Residue Number 12-27-10-6

Residue #12-27-10-6 was labeled as an unspecified organic acid. Because of its small volume, 30 mL, and small black particles in the liquid, it was decided to filter the residue to see if this separation could be simple. The residue was filtered through a paper filter into a 250 mL beaker, which was then washed with 100 mL of a 0.1 M potassium chloride solution and allowed to settle for 24 hours. This was a simple attempt to observe the effect of a salt on the solution. No other salts were tested in this fashion.

After this treatment, several black specks were observed to be on the bottom of the beaker with several white crystals, when counted separately from each other, the highest response on a Geiger counter was from the black specs, possibly a compound containing ⁹⁹Tc. A 50 mL sample of 3% H₂O₂ was added to the beaker to assist oxidation of any other ⁹⁹Tc in solution. The KCl salt dissolved and the black particles containing the majority of the ⁹⁹Tc stayed at the bottom of the beaker, they did not dissolve. The mixture was transferred into two centrifuge tubes and a small volume of the mixture containing the ⁹⁹Tc residue was removed by transfer pipette into a conical bottom 5 mL glass tube. This recovery caused collection of approximately 10 mg of the ⁹⁹Tc compound. With an estimated 47 mg available from LSC analysis, the recovery was 21.3% and considered successful. While this is not a drastic quantity of ⁹⁹Tc, the technique was simple, the cost was only the time required to filter the solution and wash the filter, and the cost of chemicals was less

than \$1.00. The dispersion information indicated that the material was not very volatile or viscous and had few visible particles in the mixture.

UNLV In-Process Experimental Products Form
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Residue Number: #12-27-10-6

Date of Storage: Unknown

Has a value analysis been completed? (Yes \boxtimes , No \square)

Is recovery of radioactive material recommended? (Yes \boxtimes , No \Box)

Volume of solution: 25 mL Solution pH: unknown

Radionuclide (1): ⁹⁹Tc; Mass (1) = 4.70E+1 mg; Act. (1) = 29.5 MBq

Chemical compound(s): Organic acid, black suspended particles, light brown liquid

Hazardous Component: Flammable \Box Corrosive \Box Oxidizer \Box Toxic \Box Unknown \boxtimes

If recovery of radioactive material in this product is not desired, is it possible to remove the hazardous component to allow disposal as radioactive waste? (Yes \Box , No \Box , N/A \boxtimes)

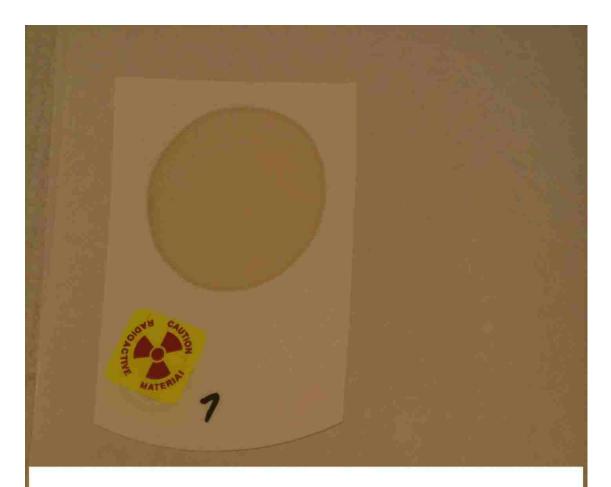
Notify the UNLV Radiation Safety Office? (Yes \Box , No \boxtimes)

Residue #12-27-10-7 was labeled organic waste. The activity identified by LSC analysis indicated an approximate ⁹⁹Tc mass of 1.58 grams available for recovery.

UNLV In-Process Experimental Products Form Residue Number: #12-27-10-7 Date of Storage: Unknown Has a value analysis been completed? (Yes \boxtimes , No \square) Is recovery of radioactive material recommended? (Yes \boxtimes , No \square) Volume of solution: 125 mL Solution pH: unknown Radionuclide (1): ⁹⁹Tc; Mass (1) = 1.58E+3 mg; Act. (1) = 992 MBq Chemical compound(s): Organic waste, light brown liquid Hazardous Component: Flammable \square Corrosive \square Oxidizer \square Toxic \square Unknown \boxtimes If recovery of radioactive material in this product is not desired, is it possible to remove the hazardous component to allow disposal as radioactive waste? (Yes \square , No \square , N/A \boxtimes) Notify the UNLV Radiation Safety Office? (Yes \square , No \boxtimes)

The dispersion continued to a 5 cm by 5.4 cm ellipse with a slightly darker ring around the dispersion. This experiment indicated that the material was volatile and did not have many visible particles in the mixture. The emissions measured from the filter by portable instrument indicated an approximate activity of 5×10^7 Bq/mL or a total activity in the container of 8×10^8 Bq in the container. This indicates an

approximate mass of ⁹⁹Tc of 1.27 grams and is reasonably consistent with the estimate by LSC analysis.



Sample 12-27-10-7 100 microliters on Whatman #1 filter Light brown dispersion on the filter Darker brown ring around the dispersion GM Net Response at the center of the filter = 45,000 CPM - beta Scintillation detector response = 846,860 CPM - beta Scintillation detector response = 2 CPM - alpha

Residue # 12-27-10-8, by discussion with the researcher [116], was found to be a 99 Tc₂S₇ compound. While the results from samples in this study may not be similar in ease or cost, each can be concluded with a degree of certainty that recovery may be possible with the exception of this one.

UNLV In-Process Experimental Products Form

Residue Number: #12-27-10-8

Date of Storage: Unknown

Has a value analysis been completed? (Yes \boxtimes , No \square)

Is recovery of radioactive material recommended? (Yes \Box , No \boxtimes)

Volume of solution: 100 mL Solution pH: unknown

Radionuclide(1): ⁹⁹Tc; Mass (1) = 1.11E+1 mg; Act. (1) = 6.9 MBq

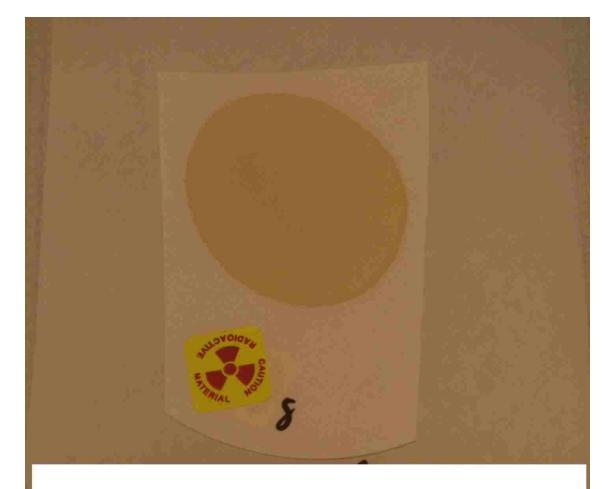
Chemical compound(s): Black viscous solid by design

Hazardous Component: Flammable \Box Corrosive \Box Oxidizer \Box Toxic \Box Unknown \boxtimes

If recovery of radioactive material in this product is not desired, is it possible to remove the hazardous component to allow disposal as radioactive waste? (Yes \Box , No \boxtimes , N/A \Box)

Notify the UNLV Radiation Safety Office? (Yes \Box , No \boxtimes)

With regard to this particular sample, the residue was found to be a compound mixture, which created a black viscous mass having the property of easily drying to a solid and being available for disposal as a solid waste. In the previously referenced discussion with the researcher, he indicated this to be a product that is not easy to break down. Although this product (⁹⁹Tc₂S₇) is easily made, it presents very low recovery potential and therefore is an undesirable step in a recovery process. It may be a useful waste form for technetium isotopes [19]. The recovery potential for this material was considered to be low because of the particulate matter that was present in the waste. The compound became a tar-like solid mass in the container. This material was disposed as solid radioactive waste.

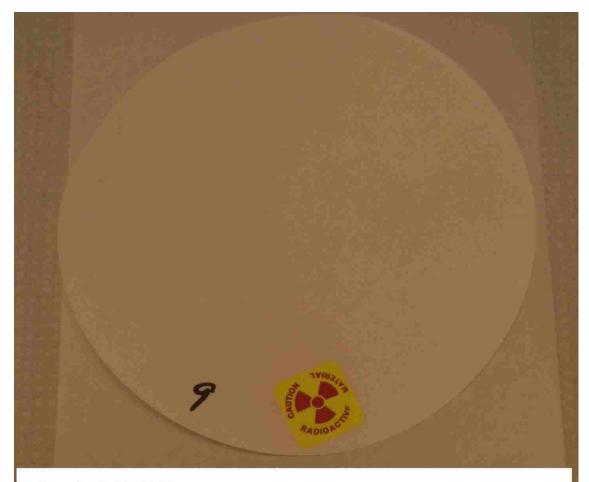


Sample 12-27-10-8 100 microliters on Whatman #1 Filter Orange colored dispersion on the filter Slightly darker around the periphery of the dispersion GM Net Response at Center of the spot = 250 CPM - beta Scintillation detector response = 4,545 CPM - beta Scintillation detector response = 2 CPM - alpha

Residue #12-27-10-9 had a mass concentration of 99 Tc similar to #2-27-10-8, however, the matrix was noted as H₂O.

UNLV In-Process Experimental Products Form
Residue Number: #12-27-10-9
Date of Storage: Unknown
Has a value analysis been completed? (Yes \boxtimes , No \Box)
Is recovery of radioactive material recommended? (Yes \Box , No $oxtimes$)
Volume of solution: 400 mL Solution pH: unknown
Radionuclide(1): ⁹⁹ Tc; Mass (1) = 5.12E+1 mg; Act. (1) = 32.1MBq
Chemical compound(s): H ₂ O matrix
Hazardous Component: Flammable \Box Corrosive \Box Oxidizer \Box Toxic \Box Unknown \boxtimes
If recovery of radioactive material in this product is not desired, is it possible to remove the hazardous component to allow disposal as radioactive waste? (Yes \Box , No \boxtimes , N/A \Box)
Notify the UNLV Radiation Safety Office? (Yes \Box , No \boxtimes)

This sample also did not represent an opportunity to regain a significant quantity of material and was therefore excluded from the recovery experiments. The material was absorbed into a contamination control pad, allowed to dry, and disposed of as solid radioactive waste.

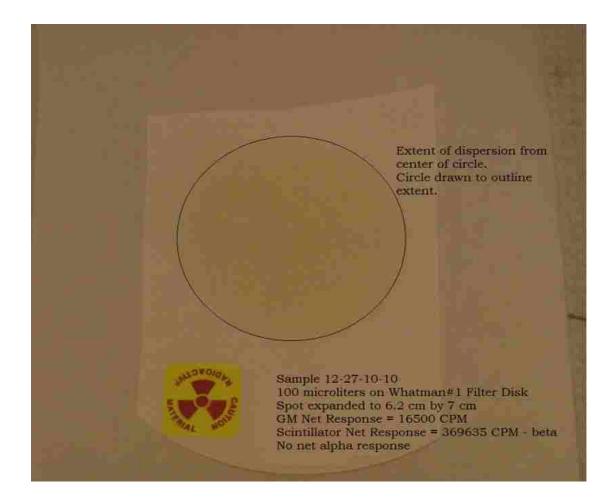


Sample 12-27-10-9 100 microliters on Whatman #1 Filter Very light colorless stain on the filter Slightly darker stain at outline GM Net Response at Center of the spot = 450 CPM - beta Scintillation detector response = 12,068 CPM - beta Scintillation detector response = 12 CPM - Alpha

Residue #12-27-10-10 was labeled, "FP waste 5/5/06". With a mass concentration of approximately 4 mg/mL, this could be a valuable reclamation.

UNLV In-Process Experimental Products Form Residue Number: #12-27-10-10 Date of Storage: Unknown Has a value analysis been completed? (Yes \boxtimes , No \square) Is recovery of radioactive material recommended? (Yes \boxtimes , No \square) Volume of solution: 200 mL Solution pH: 0 Radionuclide(1): ⁹⁹Tc; Mass (1) = 7.92E+2 mg; Act. (1) = 497 MBq Chemical compound(s): Dark brown colored liquid Hazardous Component: Flammable \square Corrosive \square Oxidizer \square Toxic \square Unknown \boxtimes If recovery of radioactive material in this product is not desired, is it possible to remove the hazardous component to allow disposal as radioactive waste? (Yes \square , No \boxtimes , N/A \square) Notify the UNLV Radiation Safety Office? (Yes \square , No \boxtimes)

There was no film on the inside of the container. The sample color on the filter paper was pale green. The dispersion continued to an ellipse of 6.2 cm by 7 cm and with no ring. Figure 3-3 provides a photograph of the dispersion experiment for this sample. Based on the filter dispersion, the mixture was likely to contain a volatile fluid. As shown in Figure 3-3, the response rate from the activity on the filter indicates a rough activity of 1.2×10^6 dpm (2×10^4 Bq). This relates to an activity concentration of 2×10^5 Bq/mL or for the 800 mL volume, a total activity of 16.4 MBq, approximately 3% of the activity determined from LSC analysis. In the determination of the activity on the filter, a detection efficiency of 30% was assumed. This appears to be much higher than appropriate for measurement of emissions from the filter.



Residue #12-27-10-11 did not represent an opportunity to regain a significant quantity of material, so recovery of ⁹⁹Tc from this sample is not feasible. The residue was neutralized using NaOH to a pH of 9 and solidified using Aquaset II a commercial solidification agent **Invalid source specified.Invalid source specified.** to make an immobile product, then disposed of as solid waste.

UNLV In-Process Experimental Products Form

Residue Number: # 12-27-10-11

Date of Storage: Unknown

Has a value analysis been completed? (Yes \boxtimes , No \square)

Is recovery of radioactive material recommended? (Yes \Box , No \boxtimes)

Volume of solution: 300 mL Solution pH: unknown

Radionuclide (1): ⁹⁹Tc; Mass (1) = 5.85E+0 mg; Act. (1) = 3.7 MBq

Chemical compound(s): None

Hazardous Component: Flammable \Box Corrosive \Box Oxidizer \Box Toxic \Box Unknown \boxtimes

If recovery of radioactive material in this product is not desired, is it possible to remove the hazardous component to allow disposal as radioactive waste? (Yes \Box , No \Box , N/A \boxtimes)



Sample 12-27-10-11 100 microliters on Whatman #1 Filter Light uncolored stain on the filter No ring around the dispersion GM Net Response at Center of spot = 220 CPM - beta Scintillation Detector Response = 1378 CPM - beta No Alpha Response (2 CPM)

Residue #12-27-10-12, an unlabeled sample, with a mass concentration of approximately 1.4 mg/mL was potentially a good reclamation. There was a thick orange film on the inside of the container. On the filter paper the sample color was light green. The dispersion continued to an ellipse of 6.2 cm by 7 cm and with no discernible dispersion ring. The material in this residue appears similar to residue #12-27-10-10.

UNLV In-Process Experimental Products Form

Residue Number: #12-27-10-12

Date of Storage: Unknown

Has a value analysis been completed? (Yes \boxtimes , No \Box)

Is recovery of radioactive material recommended? (Yes \boxtimes , No \square)

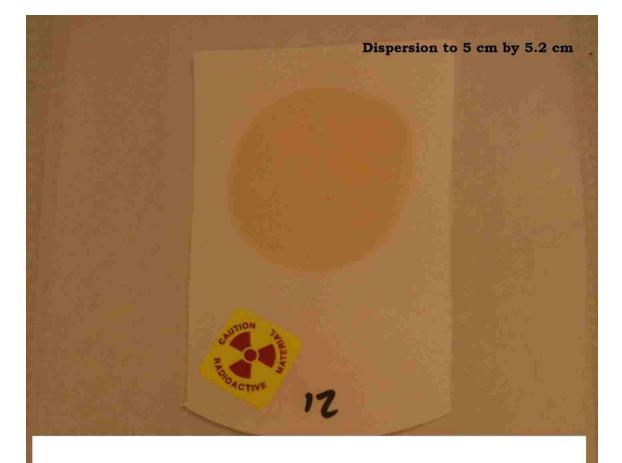
Volume of solution: 800 mL Solution pH: 0

Radionuclide (1): ⁹⁹Tc; Mass (1) = 1.12E+3 mg; Act. (1) = 704 MBq

Chemical compound(s): Orange colored liquid

Hazardous Component: Flammable \Box Corrosive \Box Oxidizer \Box Toxic \Box Unknown \boxtimes

If recovery of radioactive material in this product is not desired, is it possible to remove the hazardous component to allow disposal as radioactive waste? (Yes \Box , No \Box , N/A \boxtimes)



Sample 12-27-10-12 100 microliters on Whatman #1 Filter Orange color dispersion on filter Light green ring around the dispersion GM Net Response at Center of spot = 6500 CPM - beta Scintillation Detector Response = 107,624 - beta No Alpha Response

Residue #12-27-10-13, a labeled sample ("Tc waste organic liquids") with a mass concentration of approximately 1.2 mg/mL, had an estimated recovery quantity at almost 600 mg and presented an excellent opportunity for reclamation. There was no dispersion data for this sample and all subsequent samples because it was felt that the data already collected was sufficient to allow a decision for recovery. There was no film on the inside of the container.

UNLV In-Process Experimental Products Form

Residue Number: # 12-27-10-13

Date of Storage: Unknown

Has a value analysis been completed? (Yes \boxtimes , No \square)

Is recovery of radioactive material recommended? (Yes \boxtimes , No \square)

Volume of solution: 500 mL Solution pH: unknown

Radionuclide (1): ⁹⁹Tc; Mass (1) = 5.91E+2 mg; Act. (1) = 370 MBq

Chemical compound(s): Tc waste organic liquids, dark brown colored liquid

Hazardous Component: Flammable \Box Corrosive \Box Oxidizer \Box Toxic \Box Unknown \boxtimes

If recovery of radioactive material in this product is not desired, is it possible to remove the hazardous component to allow disposal as radioactive waste? (Yes \Box , No \Box , N/A \boxtimes)

Residue #12-27-10-14 was also a labeled ("Tc waste in H₂O, NOx 11/15/2010 NF") sample with a mass concentration of approximately 1.3 mg/mL, this sample was determined to be a good candidate for reclamation. There was no film on the inside of the container.

UNLV In-Process Experimental Products Form

Residue Number: # 12-27-10-14

Date of Storage: Unknown

Has a value analysis been completed? (Yes \boxtimes , No \Box)

Is recovery of radioactive material recommended? (Yes \boxtimes , No \square)

Volume of solution: 200 mL Solution pH: unknown

Radionuclide (1): ⁹⁹Tc; Mass (1) = 2.62E-2 mg; Act. (1) = 164 MBq

Chemical compound(s): Tc waste in H_2O , NOx 11/15/2010 NF, dark brown liquid

Hazardous Component: Flammable \Box Corrosive \Box Oxidizer \Box Toxic \Box Unknown \boxtimes

If recovery of radioactive material in this product is not desired, is it possible to remove the hazardous component to allow disposal as radioactive waste? (Yes \Box , No \Box , N/A \boxtimes)

Residue #12-27-10-15 was also a labeled ("0.5 g of ⁹⁹Tc in organic acid") sample with a mass concentration of approximately 4.4 mg/mL; this sample was determined to be a good candidate for reclamation. There was no film on the inside of the container.

UNLV In-Process Experimental Products Form

Residue Number: # 12-27-10-15

Date of Storage: Unknown

Has a value analysis been completed? (Yes \boxtimes , No \square)

Is recovery of radioactive material recommended? (Yes \boxtimes , No \square)

Volume of solution: 500 mL Solution pH: unknown

Radionuclide (1): ⁹⁹Tc; Mass (1) = 2.18E+3 mg; Act. (1) =1370 MBq

Chemical compound(s): 0.5g of Tc in organic acid, dark brown liquid

Hazardous Component: Flammable \Box Corrosive \Box Oxidizer \Box Toxic \Box Unknown \boxtimes

If recovery of radioactive material in this product is not desired, is it possible to remove the hazardous component to allow disposal as radioactive waste? (Yes \Box , No \Box , N/A \boxtimes)

Residue Numbers 12-27-10-16 and 12-27-10-17

Residues #12-27-10-16 and #12-27-10-17 were considered reasonable for recovery based on their concentrations, 0.48 mg/ml and 0.473 mg/ml respectively. Both samples were listed on the same form as another means of cutting down the administrative burden from this process.

UNLV In-Process Experimental Products Form

Residue Number: #12-27-10-16 & #12-27-10-17

Date of Storage: Unknown

Has a value analysis been completed? (Yes \boxtimes , No \square)

Is recovery of radioactive material recommended? (Yes \boxtimes , No \square)

Volume of solution: 50 + 450 mL Solution pH: unknown

Radionuclide (1): ⁹⁹Tc; Mass (1) = 2.38E+1 mg; Act. (1) = 14 MBq

Radionuclide (2): ⁹⁹Tc; Mass (2) = 2.13E+2 mg; Act. (2) = 133 MBq

Chemical compound(s): 1 - No notation

Chemical compound(s): 2 - No notation

Hazardous Component: Flammable \Box Corrosive \Box Oxidizer \Box Toxic \Box Unknown \boxtimes

If recovery of radioactive material in this product is not desired, is it possible to remove the hazardous component to allow disposal as radioactive waste? (Yes \Box , No \Box , N/A \boxtimes)

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