Radioanalytical Chemistry Experiments

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Introduction to the Course

The experiments in this manual were selected to accompany the textbook *Radioanalytical Chemistry*. The manual is intended to acquaint the senior or graduate student with the practices of radioanalytical chemistry and develop some familiarity with the various techniques and methods commonly used in the radioanalytical laboratory and the counting room. The authors believe that only hands-on experience can translate the guidance provided by a textbook to an understanding of the applications that form the basis of this aspect of radiochemistry.

These experiments are based on methods actually applied or developed for instruction by the two authors during their careers in the radiochemistry laboratory. Each experiment has been tested by students in an academic laboratory setting. The experiments were practice-taught for one semester to a group of graduate chemistry students. The students' responses were then used to modify the presentation of the experiments for inclusion in this manual.

The focus of the experiments is on the work in the radioanalytical chemistry laboratory – initial sample processing, radioanalyte purification, and preparation of the sample for counting. Certain aspects of radiation detection, such as counting efficiency and self-absorption for the various radiations, are addressed in these experiments. We expect that the student learns about the principles and applications of radiation detection instruments in a separate radiation detection course, which can be presented before, after, or in parallel with this course.

More experiments were included than can be assigned in the typical 15-week semester, to allow the instructor to select experiments that most appropriately fit the selected instructional program or available resources. Most experiments are intended for two 3-hour laboratories and one 1-hour pre- or post-laboratory session per week. A few more elaborate experiments are divided into several parts so that some parts may be dropped, or additional laboratory time may be assigned to the entire experiment.

Two experiments (#11 and #17) require extensive reading of the radiochemical literature, design of a proposed experiment, discussions with the instructor, and presentation to fellow students before the student begins to perform laboratory work. If selected, these experiments should be assigned near the beginning of the semester and evaluated near the end.

Selection of experiments undoubtedly will be guided by the availability (or rather, unavailability) of specific radiation detection instruments, radionuclide solutions, or chemicals required by certain experiments. Within the limits of what is available, the instructor will wish to select those experiments that provide the best learning experience for the students and are consistent with the overall objectives of the teaching program. From personal experience, the

authors also expect the instructor to modify some experiments and substitute others that have been found instructive and stimulating.

The specific practices and laboratory skills associated with individual experiments are briefly identified below to provide the instructor with an overview and assist in selecting experiments. A discussion of the principles to be presented and emphasized is discussed in the following section of this Introduction. The subsequent section emphasizes the safety precautions to be maintained in the radioanalytical chemistry laboratory.

Practices addressed by individual experiments

- Pipetting and weighing: #1
- Precipitation separation: #7, 8, 10, 13, and 16
- Ion exchange separation: #7, 13, 14, and 15
- Solvent extraction separation: #7 and 16
- Distillation separation: #9
- Electrodeposition: #15
- Mass spectrometry: #19
- Carrier preparation: #5, 8, 10, 13, and 14
- Tracer preparation and use: #6, 8, 15 and 16
- Methods development: #11 and 17
- Sample processing: #12
- Reagent blank use: #8, 9, 14 and 19
- Effect of radiation self-absorption: #2, 3 and 10
- Choice of methods: #7, 11, 13/14, 15/16; 17

Use of specific radionuclides

- Cesium-137: #2
- Cobalt 60: #2
- Iodine-131: #10 and 11
- Potassium-40: #3 and 4
- Plutonium-242 and -239: #6, 15, 16 and 17
- Radium-226 and -228: #8
- Strontium-90: #13 and 14
- Strontium-89: #2
- Thallium-204: #2
- Thorium-234 (from uranium): #7
- Tritium (H-3): #9
- Uranium: #7 and 19
- Mixed gamma-ray standard solution (europium-154 and -155, antimony-125): # 2
- Mixed fission product solution (about 1-week old): #18

Use of specific radiation detectors

- Alpha-particle spectrometer: #6, 7, 15 and 16
- Gas-flow proportional counter (beta and alpha particles): #2, 4, 6, 13 and 14
- Liquid scintillation counter (low-energy beta particles): #9
- Gamma-ray spectrometer: #2, 3, 8 and 18
- Radiation detector calibration: #2, 3, 4, 6, 8, 10, and 18

Before the students begin their laboratory work, the instructor is advised to emphasize the importance of the following considerations:

- Laboratory safety, as discussed below. Consult the Material Data Safety Sheets (MDSS) for information regarding hazards – carcinogens, flammability, corrosiveness, etc. – associated with chemicals used in the experiments.
- Knowledge of pertinent radioanalytical chemistry principles, including the appropriate vocabulary.
- Good laboratory practices, in particular, preparation for performing the experiment. This includes, but is not limited to, reading and comprehending the assigned experiment prior to the laboratory period and having at hand all required equipment and reagents.

Principles of Radioanalytical Chemistry

The primary function of the radioanalytical chemistry laboratory is to prepare samples for radioactivity measurement. The radioactive species are identified by detecting the radiation that they emit. The goal of the analysis is to produce a sample for counting that has no interference from other radioactive species and to quantify the recovery of the radioactive species in the analysis.

The method selected to prepare a radionuclide for counting depends on the skills and preferences of the analyst, available detectors, and conditions associated with the radioactive analyte, accompanying radionuclides, and the sample matrix. All aspects have to be considered to obtain a measurement that meets reliability and sensitivity specifications for radionuclide identification and detection. In some cases, the analyst has many options; in others, choice is restricted by circumstances such as small samples, low radionuclide concentration, half-life considerations, or unavailability of certain detector types.

The more the analyst knows concerning the numerous nuclear and radiochemical properties and characteristics of the analyzed radionuclide, the easier it is to select the most appropriate analysis and to resolve problems in ascertaining the quality and validity of the results. Especially for non-routine sample analysis, the nuclear properties of the radionuclide of interest must guide selection of the method of analysis and detection. The appropriate passages of the accompanying text *Radioanalytical Chemistry* are referenced when more detailed discussions are needed.

Overall, radiochemical analysis for the measurement of a radioactive species requires three basic steps:

1) preparation of the radionuclide in the sample by chemical separation and purification,
2) detection of the emitted radiation by an instrumental method, and
3) collection, analysis and treatment of data.

The execution of each step requires a range of activities, each of which are covered thoroughly in the appropriate chapters of the *Radioanalytical Chemistry* textbook. Sample separation and purification are of particular concern, in the sense that no reasonable counting data can be obtained and

analyzed from improperly prepared samples. In brief, the following chemical preparation steps are part of any radiochemical analysis and should be performed with care and attention to detail to obtain good laboratory results.

Chemical Preparation of the Sample

Initial Sample Preparation. When received, the sample must be handled according to the proper protocol to maintain its chemical and legal integrity. Some pretreatment to preserve the sample usually is performed at collection time and should be properly described in "chain of custody" documentation that accompanies the sample, as described in the *Radioanalytical Chemistry* text.

In the laboratory, a solid sample must be dissolved or leached to release the analytes. The analysis will be conducted with the resultant solution if chemical purification is needed.

Carrier or Tracer Addition. To quantify the purified final sample that will be measured by a radiation detection instrument (as compared to a mass spectrometer), a carrier or tracer is added to the sample. The carrier usually is the same element as the radioanalyte ("isotopic carrier") and is standardized, typically at 5–20 mg/mL concentration. The carrier serves two purposes: to provide macro quantities so that certain chemical steps (such as precipitation) may be performed on the sample, and to determine the chemical yield, usually by weight. A tracer serves only to determine the chemical yield of the process; its nanogram quantities or less, comparable to the radioanalyte in the sample, prevent use as carrier. The tracer is measured by its characteristic radiation at the same time as the radioanalyte. An advantage in alpha-particle spectral analysis is that the activity of the analyte can be calculated from the activity of the tracer without knowledge of the detector counting efficiency, as discussed below.

Isotopic Exchange/Equilibrium. Chemical steps are required at the outset of the procedure to insure isotopic exchange between the radionuclide to be analyzed (the radioanalyte) and the tracer or carrier that has been added. The carrier or tracer and the radioanalyte must be in the same oxidation state and chemical species in solution. This effort is not required for radionuclides that exist in only a single form, such as Group 1A (Li, Na, K, Rb) elements that are consistently in their +1 state in solution. Other elements (such as I or Ru) that have multiple oxidation states, and also can form stable complexes, will require steps to insure that the added carrier or tracer and the radioanalyte exchange before the analysis is started.

To illustrate this concept, assume that a sample is to be analyzed for radio-lanthanum. The lanthanum in the sample is complexed with some organic ligand (L^-) that forms an anionic complex, LaL_5^{2-}. Lanthanum carrier as La^{+3} is added to the sample but no exchange steps are performed. If the first step for the separation and purification of lanthanum is to adsorb it on a cation-exchange resin, the carrier lanthanum cation may absorb on the column while the radio-lanthanum as a complex anion will pass through the column. When the analysis is completed, an adequate carrier yield but no lanthanum radionuclide may be observed in the sample to give an erroneously low radioanalytical result. This problem could have been avoided if the complex had been destroyed in the first step or sufficient reagent had been added to

complex the carrier and allow the radio-lanthanum and carrier lanthanum to be in the same state.

The case is different if, for instance, radio-sodium is being analyzed in a sample. The sodium ion does not form complexes, nor does it have multiple oxidation states. When mixed, the carrier ions and the radio-sodium ions are immediately rendered indistinguishable in terms of the chemical separation and purification steps. In this case, only mixing and no exchange step is required.

Chemical Separation Steps. The radioanalytical or radiochemical procedure is a series of chemical steps performed on the sample to insure that the suitably exchanged radioanalyte plus carrier are separated from substances, both radioactive and non-radioactive, that will interfere with the analysis. The final sample must be free of radionuclides that could be mistaken as the radioanalyte when counted. It also must be free of non-radioactive elements that would falsely elevate the chemical yield (recovery), excessively attenuate the emitted radiation, or otherwise interfere in the identification and quantification of the radioanalyte.

The first step after exchange often concentrates the radionuclide and carrier to perform subsequent steps more easily in a smaller volume. Further steps by precipitation, solvent extraction cycles, ion exchange, or distillation improve sample purification. In these processes, the analyte is separated from various known impurities. One type of separation step is adjusting the oxidation states of analyte or impurity. A special step may be inserted to enhance separation of an impurity that is difficult to remove.

As an example of concentration from a large volume, a silver radioanalyte can be precipitated as AgCl in an early step. This process combines purification with concentration because only palladium follows silver under specified conditions.

An example of removing multiple interfering elements is strontium purification in the presence of fission products. Ferric ion is added as a "holdback" carrier for the rare earths (and other radionuclides) and then precipitated as $Fe(OH)_3$, the "scavenger" that carries these radioactive impurities. This or any other step can be repeated for enhanced removal of impurities.

Preparation of the Purified Sample for Counting. The last chemical step is preparing a source for measuring the radiation emitted by the radioanalyte. Traditionally, the sample is prepared as a stoichiometric compound that is easily and reliably weighed to determine yield. The counting forms may be metals such as silver or ruthenium, complex salt such as Cs_2IrCl_6 for measuring iridium, or – more commonly – a non-hydrated insoluble simple salt or oxide. An unstable chemical form, such as one that is hygroscopic or light sensitive, would not be chosen for yield determination and counting.

When the analyte and added tracer both emit alpha particles, the samples typically are electrodeposited. Analyte and tracer are counted simultaneously, and the analyte activity is determined from the tracer activity and the ratio of the net count rate for the analyte relative to the tracer.

The sample in the final counting form must be handled with care and skill. When radionuclides that emit alpha particles are poorly electrodeposited, the quality of the counting data – the alpha-particle spectrum – may be degraded due to extraneous material deposited over the active surface. The energy peak

and the count rate beneath the peak may not be as well-defined as they should be, and one peak may interfere with another.

For samples that emit beta particles, the sample must be evenly distributed, with defined and uniform thickness. Quantifying geometry and self-absorption of beta particles is unreliable for an unevenly deposited source.

Characteristics of Successful Sample Preparation

The following characteristics should be examined to indicate whether a radioanalytical separation is successful:

- *Good Chemical Yield*. If the yield is too low, the loss suggests a sample processing problem, and measurement reliability is decreased. Many radioanalytical laboratories impose a cutoff at a minimum chemical yield value such as 50% for an analysis to be acceptable.
- *Reliable Sample for Radiation Detection*. The sample must be prepared for counting by an effective method. Many such methods have been published, each appropriate for its element. For example, strontium may be prepared for counting as strontium oxalate or carbonate, and iodine as silver or palladium iodide. Samples submitted for mass spectrometric analysis must be in a form that is suitably volatilized and processed by the instrument.
- *Low Count Rates in Blanks*. Radioactive contamination in a "blank" should be zero, or constant and extremely small. A procedure blank is a deionized water sample that is processed through the complete analysis. Carrier is added, every step is performed to the end of the analysis, and the final form is counted. Blanks are processed as part of each sample batch to check the quality of the analysis with regard to laboratory contamination for this batch.

  The blank shows whether the reagents, glassware, and work environment contribute any radioactivity to samples. A blank will have a zero net count rate – that is, the measured or gross count rate minus the detector background count rate – if no radioactivity is observed. If the net count rate is above zero, it should be low compared to the net count rate of the analyte. Efforts should be made to find and reduce the source of contamination.
- *Radiochemical Purity*. A sample is radiochemically pure at the time of counting if no other radionuclide is detected in it. As a general rule, the radiochemical procedure is chosen to separate from the radioanalyte all other radionuclides that are in the sample. Purification steps must be added to the usual procedure if the level of contaminant radionuclides is very high relative to the concentration of the radioanalyte.

  Re-analysis with better purification should be considered if the count rate is unexpectedly high or the observed half life and radiation energies are not those of the radioanalyte. Contaminant radionuclides may be tolerated if they do not interfere with counting the radioanalyte, or can be subtracted from the count rate with only a minor increase in detection uncertainty. In spectral analysis of alpha particles and gamma rays, for example, contaminant radionuclides are tolerated in the sample if they do not interfere with counting the characteristic spectral peaks of the analyte.

- *Chemical Yield versus Radiochemical Purity*. The conditions for acceptance or rejection on the basis of chemical yield may have to be revised when a sample contains major contaminants The needed additional purification steps invariably decrease the chemical yield, hence a chemical yield below 50 percent may be acceptable. A skilled radiochemist is capable of minimizing those losses while producing a high-purity sample. This ability to balance purity and yield comes with practice, knowledge of radioanalytical techniques, and familiarity with the chemistry of the elements involved.

Additional Considerations: Radionuclide Source and Category

Because more than 1,000 radionuclides have been observed, laboratories generally specialize by categories such as source, half-life, type of emitted radiation, sampling location, and amount. Overlap in categories is inevitable. For example, the products of fission may be accompanied by activation products; they may be short-lived at high levels in the process stream and long-lived at low levels in the environment.

Analyte half-lives need to be considered to arrange for rapid collection, transfer to the laboratory, and radioanalytical chemistry processing before they decay to poorly-detectable low amounts. Types of emitted radiation control the detector that must be purchased, calibrated, and operated. Radiochemists and radiation-detector operators who commonly handle a specific category of radionuclides become skilled in purifying and counting these radionuclides.

Among common radionuclide sources are the natural environment, fallout from nuclear weapon tests, effluents from nuclear research laboratories, the nuclear power fuel cycle, radiopharmaceutical development, manufacturing, and various application, teaching and research uses. Decontamination and decommissioning activities at former nuclear facilities and the potential of terrorist radionuclide uses are current topics of interest for radioanalytical chemistry laboratories. Simplified information on the numerous radionuclides is conveniently found in Charts of the Nuclides such as Nuclides and Isotopes (revised by J. R. Parrington, H. D. Knox, S. L. Breneman, E. M. Baum, and F. Feiner, 15th Edition, 1996, distributed by GE Nuclear Energy).

The sources of the radionuclide samples sent to the laboratory include the environment, nuclear facilities (for process control and bioassay monitoring), and nuclear research laboratories. Each contributes its own set of radionuclides. In environmental samples, for example, one may find the naturally-occurring radionuclides $^{226+228}$Ra and isotopes of U and Th, and man-made ^{3}H (tritium), ^{90}Sr, ^{99}Tc, $^{131+129}$I, ^{137}Cs, isotopes of Pu, and ^{241}Am. Appendix 1 has a list of commonly analyzed radionuclides in environmental samples, and Appendices 2, 3, and 4 show the natural decay series for ^{238}U, ^{235}U, and ^{232}Th, respectively.

Laboratories specialize in analyzing radionuclides at either high or low levels. Personnel-protection installations and practices are required for high levels of radiation from high levels of radionuclides. Low-level radioanalytical chemistry requires separation from high levels because results are easily undermined by contamination.

Working with Radioactivity: Rules, Practices and Safety Precautions

Any chemistry laboratory is a place that has many sources of hazards, including explosive, toxic or flammable chemicals, noxious vapors, broken glass, and hot liquids and solids. In addition to the rules given below, good practice requires that the instructor and student review each experiment for all potential hazards and discuss steps to avoid or mitigate such hazards. The review should include considerations of dangerous chemicals and conditions in each experiment.

Radioanalytical chemistry laboratories generally are divided into three separate areas: (1) a sample-receiving facility, (2) a wet laboratory for initial sample processing, radionuclide separations, and counting-source preparations, and (3) counting rooms where radiation detection and measurement equipment is housed. The counting room is separated to maintain the detectors and their electronic systems in a clean and stable environment. The student is required to follow all procedures, rules, and protocols established for the laboratory and counting room. Failure to do so may result in radioactive contamination and the considerable expense and effort to restore the working facilities to their original utility and cleanliness. The institution where this course is being taught undoubtedly has training requirements that must be met before the student embarks on any work with radioactive materials. The instructor will inform the students of these requirements.

Safe handling of chemicals and radionuclides in these experiments requires practice and the exercise of attention and care. This is true for all levels of radioactivity, including levels in the becquerel range.

For the purposes of these experiments, the following practices are strongly encouraged. The instructor may insist on additional practices and recommend reading a designated safety manual. In addition, Chapter 14 of the textbook *Radioanalytical Chemistry* covers the topic of laboratory safety. The student's responsibility is to follow all rules presented here, by the instructor, and in the safety manual.

- Wear personal protective equipment when chemical and radioactive materials are handled. This includes eye protection gear, a laboratory coat, and gloves (rubber or plastic).
- Always treat laboratory equipment in a radioanalytical chemistry laboratory as if it contained or had contained radioactive material. Glassware that has been used with radioactive material should be segregated and properly labeled.
- Always handle radioactive solids, solutions, and gases in a work area that can be easily cleaned and checked for radioactivity.
- Never pipette by mouth.
- Do not perform any laboratory work in the counting room area.
- Do not eat, drink, smoke or chew in a radiological area.
- Perform experiments with volatile materials or radioactive gases in a well-ventilated fume hood rated for that class of work. Good practice for safety purposes is to conduct most work with radioactive materials in a fume hood.
- After a radioactive spill or other contamination of persons or environment, immediately notify the laboratory instructor so that he/she can inform the

Radiological Safety staff and instruct you about the proper method and protocol for cleaning up the spill, not spreading the contaminant, and monitoring persons and areas.

- At the conclusion of the laboratory period or an experiment, and when leaving the laboratory area for any reason, monitor hands, feet, head, body, and clothing with a hand-held radiation counter before exiting, as specified by your instructor. Your laboratory instructor will demonstrate the proper technique and show you the proper instruments for the type of radiation you that you will use.
- Dispose of all wastes in accord with instructions.
- Return all radioactive and chemical materials to their place of storage. This may simply mean returning the radioactive materials to the laboratory instructor and the chemicals to their storage cabinets.
- Keep all laboratory equipment and materials in the radiological work areas. They should not be transported to non-radiological work areas unless given permission by the laboratory instructor.
- Maintain good housekeeping in the laboratory. Keep hoods, bench tops, and floor unobstructed. When carrying over an experiment from one period to the next, store materials that you are using neatly and place them so they do not interfere with activities by others or are in danger of being spilled or overturned. At the conclusion of an experiment, clean up so no residue of the work remains.
- Ask the instructor if you are uncertain regarding any practice or step. In case of doubt, do not proceed on your own.

Experiment 1

Practice in Pipetting and Weighing Samples for Radioactivity Counting

Objective

To practice pipetting small volumes and weighing small quantities that are encountered in a radioanalytical chemistry laboratory.

Introduction

The radioanalytical chemist must be skilled in handling samples for making radioactivity measurements. While weighing and pipetting are two skills that chemists should already have mastered, it is possible that the small quantities of material normally required for radioanalytical work might present some difficulty. This experiment is designed to hone the student's ability to perform small-scale mass and volume measurements. These exercises focus on using an analytical balance that is capable of measuring mass to within 0.01 mg, and pipettes in the range of 10 lambda (l λ = 0.001 mL) to 1.0 mL in capacity.

Any functional set of pipettes can be used. However, the student should be familiar with the two main types of pipettes: glass lambda pipettes and automatic pipettes.

As a reminder, pipettes are classified either TD (to deliver) or TC (to contain). The TD pipettes are the more common of the two. With a TC-type pipette, the residual contents in the pipette must be extracted by repeated washing. Always read the label of a glass pipette to determine if it is TD or TC.

Automatic plastic pipettes have become a common feature in the laboratory, and are gradually replacing glass lambda pipettes in everyday use. These pipettes are controlled by a specified-volume button or dial-volume controls commonly, and are used to deliver small volumes with accuracy.

In this experiment, three or four types of pipettes are selected to deliver water to a clean container. By weighing the delivered volumes, the accuracy of the different delivery methods is compared. The experiment also allows for the precision of pipette delivery to be observed.

Safety Reminders

- Follow the usual safety procedures when working in a radiological laboratory.
- Caution should be exercised when preparing and working with corrosive mineral acids.
- All liquids/solids are to be properly disposed of according to laboratory rules and protocol.

Equipment and Supplies

If your laboratory no longer employs glass pipettes, use only the automatic plastic type in this experiment.

- 1-mL volumetric pipette designated TD (to deliver)
- 1-mL pipette, graduated in 0.1 mL units (Mohr type)
- 0.01 mL (10 λ) automatic pipette designated TD (to deliver)
- 0.1 mL (100 λ) automatic pipette designated TD (to deliver)
- 0.1 mL (100 λ) glass micro-pipette designated TC (to contain) (optional)
- planchets with 0.5-cm-high lip (sides), stainless steel (or other suitable container)
- analytical balance capable of weighing to 0.1 mg
- forceps

Reagents

- Deionized water

Procedure

Step 1. Mark and weigh six clean, dry planchets. Record weights to the nearest 0.1 mg. *Use forceps. Do not handle the planchets with your hands because residue from fingers will compromise weight*.

Step 2. Deliver the following quantities of water to the planchet and immediately weigh after each delivery. *If the liquid is not weighed immediately*, *sample loss by evaporation can cause error*. Perform each measurement twice.

A	1 mL of water with the 1-mL volumetric pipette designated **TD**
B	1 mL with the 1-mL pipette, graduated in 0.1 mL units
C	0.1 mL with the 1-mL pipette, graduated in 0.1 mL units
D	0.1 mL with the 100-lambda glass micro-pipette designated **TC**
E	0.1 mL with the 100 lambda automatic pipette designated **TD**
F	0.01 mL with the 10 lambda automatic pipette designated **TD**

Record these measurements in Data Table 1.1.