

Development and Implementation of Nuclear Chemistry Experiments at the Undergraduate Level

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ABSTRACT: There currently exists a significant deficiency in the nuclear chemistry workforce within the United States, despite its importance in areas of medicine, national security, and energy. Poor coverage of this topic across many chemistry departments at the undergraduate level likely contributes to this shortage. Increasing interest and hands-on experience through the availability of laboratory experiments may help alleviate this burden. Herein, we describe a set of nuclear chemistry experiments designed for undergraduate students that can be readily implemented in chemistry departments without specialized programs in this area. These experiments emphasize several concepts related to safe work practices with radioactive materials, such as the effects of distance and shielding. Additionally, the use of a commercially available radionuclide generator was implemented to have students identify an "unknown" radionuclide based on its measured half-life and gamma (γ) spectrum. This laboratory experiment was carried out by two chemistry courses at Cornell University. Student feedback obtained from these sections showed that this hands-on experiment enhanced student understanding of several key concepts of nuclear



chemistry and also successfully stimulated interest in this topic. Therefore, this study demonstrates that simple nuclear chemistry laboratory experiments can be implemented in a diverse range of chemistry departments and are effective at fostering student understanding and interest in this topic.

KEYWORDS: First-Year Undergraduate/General, Second-Year Undergraduate, Upper-Division Undergraduate, Laboratory Instruction, Interdisciplinary/Multidisciplinary, Analytical Chemistry, Hands-On Learning/Manipulatives, Nuclear/Radiochemistry

INTRODUCTION

The United States currently faces the challenge of maintaining a workforce skilled in the fields of nuclear chemistry and radiochemistry, areas critical for securing national expertise in nuclear medicine, energy, and security.^{1–5} This challenge can be addressed by bolstering the educational pipelines for these topics. However, nuclear chemistry and radioactivity can be difficult subjects to teach due to the existing misconceptions about these topics and the lack of available hands-on activities for aiding in their instruction.^{6–11} The development of these types of activities or laboratory experiments that are amenable to the K–12 and undergraduate levels is challenging due to the general concern at both the institutional and societal levels regarding handling radioactive materials. Despite these challenges, there have been significant efforts in designing new K–12 activities^{12–14} and undergraduate-level laboratory experiments^{15–20} that enhance student understanding and dispel misconceptions regarding nuclear chemistry.

There are currently few chemistry departments in the United States that have vibrant nuclear chemistry education and research programs. These programs have the necessary infrastructure, support, and faculty expertise to provide a rich undergraduate experience in this topic with relevant laboratory work. By contrast, for most chemistry departments in the U.S., this subject is sparsely covered and contains few or no laboratory components. The availability of simple nuclear chemistry laboratory experiments that can be carried out at the undergraduate level in these departments would broaden the understanding of this topic at the national level. Here, we detail our efforts to implement a previously described interactive nuclear chemistry laboratory^{18–22} experiment in two undergraduate courses in the Department of Chemistry and Chemical Biology at Cornell University. This experiment is designed to use only readily available and New York State regulation-exempt quantities of radioactive materials. Our evaluation of the student learning outcomes indicates that this laboratory is an effective tool for conveying a number of important nuclear chemistry principles, thus suggesting that widespread use of this experiment in other chemistry

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departments may be a valuable means for overcoming the skill gap in this field.

CONCEPT AND EDUCATIONAL OBJECTIVES

The activities described in this report are adapted from several well-known experiments in nuclear chemistry^{21,22} and cover several key concepts with an emphasis on how they apply to the safety principles of "As Low as Reasonably Achievable" (ALARA). More specifically, students investigate the role of distance and shielding on the measured counts, or exposure, from a radioactive point source. Furthermore, students measure the half-life and γ spectrum of an "unknown" radionuclide obtained from the widely used ¹³⁷Cs/^{137m}Ba generator^{21,22} to characterize and determine its identity. The whole experiment is designed for a 3 h laboratory section. We have demonstrated the feasibility of implementing this set of experiments in the Cornell University chemistry course CHEM 2900: Introductory Physical Chemistry Laboratory, composed primarily of second-year chemical engineering and upper division chemistry students, as well as CHEM 3030: Honors Experimental Chemistry III, composed of junior and senior chemistry majors. This laboratory experiment was initially designed for lower-division chemistry courses (courses designed for primarily freshman and sophomore chemistry students), but the experiments were adapted for upper division chemistry classes (courses designed for primarily junior and senior chemistry students) to create a more advanced understanding of these concepts. The concepts and execution of this laboratory experiment may possibly be implemented in general chemistry courses as well.

This set of laboratory experiments aims to address the following educational goals:

- Investigate the mathematical relationship between the measured counts of a radioactive point source and its distance from the detector. This specific experiment relates to how maintaining distance is an effective means to minimize exposure to radiation.
- Probe the effects of different shielding materials on the attenuation of counts measured by a detector. Three different shielding materials are investigated to determine their linear attenuation coefficients (μ 's). This part of the experiment relates to the use of different materials to block and minimize radiation exposure.
- Observe how shielding thicknesses affect the measured counts and μ . This experiment illustrates how thicker shields are more useful for blocking radiation.
- Understand the concept of half-life and γ energy in a practical manner using a radionuclide eluted from a radionuclide generator. This aspect of the laboratory allows students to understand that radionuclides, like chemical compounds, can be conclusively identified on the basis of their properties and spectroscopic characterization.

Although we noted above that these experiments could be implemented in general chemistry courses, several adaptions would be required. Splitting this lab into separate experiments or only using parts of it would allow general chemistry students, with less advanced laboratory skills and background knowledge, to have more time to carefully execute them and to attend more lectures on these topics. Performing parts A and B in one section, followed by part C in the next section, would be a reasonable way to split this lab up and to make it more accessible for general chemistry students.

OVERVIEW OF LABORATORY EXPERIMENT

The laboratory experiment consists of three parts: (A) determining the effect of distance between a sealed radioactive point source and the detector on measured counts, (B) determining the effect of shielding thickness and type of material on measured counts, and (C) the identification of an unknown radionuclide eluted from a radionuclide generator. This laboratory is designed such that each part can be completed in any order, allowing the students to make effective use of their time and available equipment. A list of materials used for this experiment and their associated vendors for purchase are listed in Table S1 (Supporting Information, SI). These experiments were performed in the classes CHEM 2900 in the spring semester of 2020 and CHEM 3030 in the spring semester of 2021. These courses were taught by different instructors, and both courses were supplemented with teaching assistants (TAs) that administered the laboratory exercises, three of whom are authors of this paper. The laboratory sessions were all preceded by a single, 50 min lecture period. In CHEM 2900, the experiments were divided into two lecture periods, each with six laboratory sections. The students worked in groups of 3-4, and the number of groups per laboratory section ranged from 2 to 4. This experiment was performed by undergraduate students in a total of nine different laboratory sections. The laboratory was carried out initially by the first lecture period (lecture 1), which had six laboratory sections. On the basis of feedback from the students and TAs from lecture 1, we modified the protocol for the experiment described in part B, which is further discussed in the description for part B. After modifying the procedure, students from lecture 2 carried out the experiment. Although lecture 2 also consisted of six laboratory sections, the COVID-19 pandemic led to a suspension of in-person undergraduate teaching, only allowing the first three laboratory sections from lecture 2 to conduct the experiments in-person. The last three sections from lecture 2 carried out data analysis using the information supplied to them. A maximum of 3 h was provided for students, but the average length required for completing this laboratory was 2-2.5 h. In CHEM 3030, the experiments were carried out in four different laboratory sections with only one lecture period. The students worked in groups of 2-3, with the number of groups per section ranging from 2 to 4. There was no significant interference by COVID-19 on the completion of these experiments in this course, as social distancing, mask wearing, and other procedures had been implemented to allow for students to participate in-person. The data analysis and completion time were identical to those for CHEM 2900.

SAFETY HAZARDS AND CONSIDERATIONS

In collaboration with Environment, Health and Safety (EH&S) at Cornell University, we developed a laboratory protocol that is safe for both instructors and students and is compliant with New York State regulations governing the use of radioactive materials. This set of experiments requires sealed point sources of ⁶⁰Co (1 μ Ci) and ¹³⁷Cs (5 and 10 μ Ci) and eluted samples of ^{137m}Ba ($\leq 10 \mu$ Ci) from a ¹³⁷Cs/^{137m}Ba generator, all of which are exempt quantities under New York State and Nuclear Regulatory Commission (NRC) regulations.²³ In-



Figure 1. (A) Cross section of the spherically isotropic emission of radiation is shown, demonstrating that the density of radioactive particles, and therefore exposure, is proportional to the inverse square of the distance. (B) Representation of how the spherical isotropic emission of radiation can impact exposure when close to the source and (C) far from the source. This image demonstrates that exposure is decreased with increased distance from the radiation source.

stitutions working with exempt sources of radioactive material should be fully aware of their purchase, intended use, and disposal. Institutions with a radioactive material license are expected to dispose of these materials as required by their license. Institutions without a radioactive material license are required to follow disposal instructions provided by the manufacturer. The dose rate from the 10 μ Ci 137 Cs/ 137m Ba generator is approximately 0.036 mrem/h at 30 cm, and 32 mrem/h at 1 cm. Personal dosimeters are not required for these experiments with exempt sources. Regulations require monitoring for anyone likely to receive more than 10% of the occupational dose limit in a year, or for anyone entering a high or very high radiation area. The exposures associated with these short lab activities with NRC-exempt sources are far below the thresholds requiring dosimetry. Students were trained to use good safety practices, emphasizing typical concerns associated with working with radioactive material. In addition, all students and staff received radiation safety training as part of the lab instruction. Students were instructed to follow ALARA principles of time, distance, and shielding to reduce their radiation exposure. The only unsealed radioactive source that could present a contamination hazard in this experiment is the ^{137m}Ba eluted from the generator, which was handled over a plastic-backed absorbent bench pad. Per standard safety protocols in our department, students were instructed to wear gloves, laboratory aprons, and safety goggles. Although contamination is generally not a concern due to the 2.55 min half-life of 137m Ba, the 137m Ba generator eluent was also surveyed before disposal to check for ¹³⁷Cs breakthrough. Although we encountered no issues during this work, we recommend that only institutes with an experienced radiation safety program use this generator. It is also important to stress that the instructions given by the manufacturer of the generator were followed exactly to maintain radiation safety of an exempt quantity of radioactive material. Students conducted postuse contamination surveys of work benches and equipment, including laboratory notebooks, pens, computer mice, laptops, and gloves to teach the best practices for laboratory work with radioactive materials. A procedure for cleaning up any radioactive spills is given in the SI (Table S2).

THEORY AND BACKGROUND

Factors That Affect Radiation Exposure

There are several factors that contribute to radiation exposure, and understanding these variables is important for working

safely with radioactive materials. Demonstrating strategies to limit and reduce exposure is the main purpose for this set of laboratory experiments. In part A of our experiment, we discuss one method for reducing exposure to radiation by increasing the distance between the individual or detector from the radioactive source.^{24,25} The emission of radiation is spherically isotropic, meaning that it emits in all directions from the point source. When one is closer to the source, the density of radioactive particles is greater than when an individual is farther away. Shown in Figure 1 is a 2D section of the 3D isotropic emission of radioactive particles, represented as arrows, from a radioactive source. Using Figure 1 as an example, at a distance r from the source, there are x radioactive particles passing through a spherical surface of $4\pi r^2$. If one moves twice as far from the source to a distance 2r, the same xparticles are now distributed over a larger surface area of $16\pi r^2$. Thus, upon moving twice as far from a source, the radiation density that one is exposed to is decreased 4-fold. This phenomenon, which is observed for many physical processes like the magnitudes of gravitational and electrostatic forces, is known as the inverse square law, or the distance law in radiation safety. Mathematically, this relationship can be expressed by eq 1

$$I \propto r^{-2}$$
 (1)

where I is the intensity of the radiation and r is the distance from the source. This relationship is important for operating under the principles of ALARA because it provides guidance on how one expects worker exposure to change as they move away from a radioactive source.

The efficacy of radiation at penetrating through matter is dependent on multiple factors, which must be considered to maintain acceptable exposure levels.^{24–26} Both α and β particles are relatively easy to shield in comparison to γ rays, which are massless and chargeless photons of electromagnetic radiation. The penetration of γ rays through matter depends on several factors. First, the energy of the γ ray will dictate its ability to penetrate matter. Different γ -emitting radionuclides release these particles with distinct characteristic energies, a property that is discussed within part B of this lab. Higherenergy γ rays are more effective at penetrating matter. Second, both the composition and thickness of the matter that is used to shield the γ rays will also dictate their penetration efficiency. Intuitively, thicker shielding materials will attenuate γ rays better than thinner materials of the same composition.

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Additionally, matter composed of elements with larger atomic numbers will shield γ radiation more effectively than those of lower atomic number elements. For example, Pb (Z = 82) is a highly effective absorber of radiation and is therefore commonly used as a shield when handling radioactive materials. Mathematically, the attenuation of γ rays through a shielding material can be expressed by eq 2

$$I = I_0 e^{-\mu d} \tag{2}$$

where I_0 is the incident γ ray intensity, I is the transmitted γ ray intensity, d is the thickness of the material, and μ is the linear attenuation coefficient. As this relationship implies, the incident radiation intensity is diminished in an exponential manner as it passes through materials of increasing thickness. The rate of this exponential decrease is dictated by μ , which has units of inverse distance. This empirically determined constant, μ , varies for both different shielding material and γ photon energy. As discussed above, μ is larger for materials with high atomic numbers and lower-energy γ photons. If μ is known, one can predict the required thickness of shielding materials to attenuate the radiation intensity to acceptable levels.

Radionuclide Generators and Characteristic Properties of Radionuclides

In addition to exploring the role of distance and shielding on exposure to radioactivity, this experiment investigates aspects of nuclear chemistry that are useful in modern medicine. Specifically, in this laboratory the students use a radionuclide generator to obtain an "unknown" radionuclide, which they must characterize and identify. Radionuclide generators contain a relatively long-lived radionuclide that decays into a shorter-lived nuclide. The parent radionuclide is adsorbed onto a solid support, and the daughter can be selectively eluted with an appropriate solution. An advantage of these radionuclide generator systems is that one can obtain multiple batches of the daughter nuclide as the parent decays, thereby enabling them to be shipped across the country for clinical use. In the context of nuclear medicine, the ⁹⁹Mo/^{99m}Tc generator is the most widely used. In this experiment, the students will use a ¹³⁷Cs/^{137m}Ba generator. ¹³⁷Cs decays via β^- emission to form the metastable isomer ^{137m}Ba. This generator has been widely used for educational purposes,^{21,22,27-29} in part because the half-life of ^{137m}Ba is short (2.55 min) and the half-life of ¹³⁷Cs is long (30.1 years). An example of the elution process from this generator is shown in Figure 2.

In this experiment, the ¹³⁷Cs/^{137m}Ba generator is used without disclosing its composition to the students, who are given the eluted ^{137m}Ba radionuclide and tasked with identifying it. The students receive the eluted source on a planchet placed in a plastic weigh boat as secondary containment. Prior to its use for secondary containment, the plastic weigh boat was cut to provide an easily used handle, which we found practical to allow for manipulation of the sample while minimizing the risk of contamination. Other institutions may choose to use shorter and smaller weigh boats that meet their needs. Different radionuclides vary in their modes of decay, half-lives, and energies of their γ ray emissions. Thus, by determining these features of a given nuclide, one can conclusively identify it. The radionuclide half-life can be assessed by monitoring its radioactive decay as a function of time, and its γ emission energies can be measured using a γ



Figure 2. Depiction of the ¹³⁷Cs/^{137m}Ba radionuclide generator, which is a semiclosed, exempt quantity radiation source. The long-lived parent, ¹³⁷Cs ($t_{1/2} = 30.17$ y), is adsorbed onto an ion exchange medium. Its radioactive decay continuously produces short-lived ^{137m}Ba ($t_{1/2} = 2.55$ min), while keeping the parent ¹³⁷Cs nuclide adsorbed on the solid support to generate more daughter nuclides for the next elution. The eluting solution used is 0.9% NaCl and 0.04 M HCl, as provided by the manufacturer of the generator.³⁰

spectrometer. Radioactive decay proceeds with first order kinetics, following eq 3

$$A = A_0 e^{-\lambda t} \tag{3}$$

where A is the activity over time t, A_0 is the initial activity, and λ is the decay constant. The decay constant λ describes the rate of radioactive decay and has a characteristic value for specific radionuclides. The half-life $(t_{1/2})$ of the radionuclide, based on first order kinetics, can then be expressed as eq 4

$$t_{1/2} = \frac{\ln 2}{\lambda} \tag{4}$$

By measuring activity over time, the resulting data can be fit to eq 3, allowing the decay constant, λ , and therefore the $t_{1/2}$, to be determined.

The emitted γ photon energies are also characteristic for specific radionuclides. The emission of γ rays occurs as a nucleus relaxes from a nuclear excited state to a state of lower energy. The energies of these nuclear excited states and γ rays are dictated by the configuration of neutrons and protons in nucleon orbitals. Each nuclear state has nuclear spin, I, which is a consequence of the overall angular momentum of the particular proton and neutron configuration. The decay scheme in Figure 3 shows that the metastable excited state of ¹³⁷Ba (I = -11/2) is 662 keV higher in energy than the ground state (I = +3/2). Thus, a γ photon of this precise energy is emitted upon relaxation. The characteristic γ energies can be measured using γ spectroscopy. The spectra are acquired using semiconductor detectors or inorganic scintillators. In this experiment, an affordable Tl-doped NaI inorganic scintillator is used to resolve the γ energies of the unknown



Figure 3. Decay scheme of ¹³⁷Cs, including the nuclear spin state (*I*) of each nuclide and the branching ratios for the decay modes. The nuclear spin is a quantum mechanical property of the nucleus that can be used to differentiate excited metastable states and ground states, as well as the quantum mechanical allowedness based on selection rules of a particular decay mode and pathway. The branching ratios depicted indicate the extent to which the parent nucleus, ¹³⁷Cs, decays into the daughters ^{137m}Ba and ¹³⁷Ba, as dictated by these quantum mechanical selection rules. As depicted, there is a 92% percent probability that the ¹³⁷Cs nuclide will undergo β^- decay to metastable ^{137m}Ba nuclide and an 8% percent probability that it will decay directly to the ground state ¹³⁷Ba nuclide. Although these concepts of nuclear spin and branching ratios are beyond the scope of the lab experiment, they are included in this decay scheme for completeness.

^{137m}Ba radionuclide. With both the $t_{1/2}$ and γ emission profile known, depositories of nuclear data, such as several online versions of the Chart of the Nuclides,^{31,32} can be consulted to identify radionuclides with these characteristic properties.

RESULTS AND DISCUSSION

Part A: Relationship of Distance with Measured Counts

In this portion of the laboratory experiment, students determined and evaluated the relationship of radioactive exposure to distance. The students collected the background counts over the course of 30 s and measured counts from a sealed 5 or 10 μ Ci ¹³⁷Cs point source at 5 different distances over the same time period using a Geiger–Müller detector. For these measurements, counts were determined with a 2 s sampling period. Representative student data is shown in Table 1 and Figure 4.

Table 1. Sample Data for Part A

Distance (cm)	Corrected Distance (Distance + 0.5) $(cm)^{a}$	Gross Average Counts ^{b,c}	Net Counts = Gross Counts - Background Counts ^{c, de}
0.5	1.0	1246	1245
1.5	2.0	351	350
2.5	3.0	178	177
3.0	3.5	124	123
6.0	6.5	35	34

^{*a*}The corrected distance accounts for the 0.5 cm from the bottom of the radiation monitor to the actual detector. ^{*b*}Average of three replicates. ^{*c*}Acquired for sampling times of 2 s per measurement. ^{*d*}Average background counts = 1.

After plotting the respective data (distance versus net counts), the students were asked to interpret their data in relation to eq 5

$$A = Xd^n \tag{5}$$

where A is the measured activity, X is the respective coefficient, and d is the distance from the detector. The objective was to determine the value of n, rounded to the nearest integer.



Figure 4. Sample data demonstrating the relationship between distance and measured counts from a 137 Cs point source (5 μ Ci). Sampling time was 2 s per measurement.

Theoretically, this value should be equal to -2, as dictated by the inverse square law (eq 1). Figure 4 shows that the representative data in Table 2 are able to capture the inverse square law reasonably well. Students were then asked to rationalize this relationship by considering how the surface area of the sphere changes with increasing radius. The net counts are related to the number of radioactive emissions that reach the detector. The spherical isotropic nature of these emissions means that, as the detector is moved farther away from the source, the radioactive particle per area decreases and fewer particles interact with the detector.

Part B: Effects of Shielding Material and Thickness on Exposure

In this portion of the experiment, students explored the effect of shield thickness and material on the measured counts of the two different sealed point sources, 137 Cs (5 μ Ci) and 60 Co (1 μ Ci). The goals of this experiment were to illustrate that (1) thicker shields yield better attenuation, (2) higher-energy γ rays are more difficult to block, and (3) shielding materials comprising heavier elements are more effective. The radiation from a ¹³⁷Cs point source was analyzed with and without aluminum shielding to determine the effectiveness of attenuating the measured counts. The same procedure was repeated for 60Co with aluminum shields, and then, a second shielding material was used for the ¹³⁷Cs point source. The use of both ¹³⁷Cs and ⁶⁰Co allows students to compare the effects of the γ photon energy. The sole γ photon resulting from ¹³⁷Cs decay has 662 keV of energy, whereas ⁶⁰Co has two significantly higher-energy principal γ emissions at 1.2 and 1.3 MeV. For CHEM 2900, lead shields were used as the second shield type for lecture 1, and lecture 2 used plastic shields made of high-density polyethylene (HDPE) as the second shielding material.

In lecture 1 of CHEM 2900, students conducted the experiment to obtain three data sets: ¹³⁷Cs with aluminum shields, ⁶⁰Co with aluminum shields, and ¹³⁷Cs with lead shields. Representative student data for each point source with different shields are shown in Tables S2–S7 (SI). Poor-quality exponential fits were obtained during lecture 1, which arose as a consequence of missing key data points within the curvature of the exponential decay of activity versus shielding thickness plots. Representative plots for lecture 1 are shown in Figure S1 (SI). In lecture 2, we aimed to optimize the procedure so that students could obtain quantitative data in the form of the linear attenuation coefficients, μ . The same sources as those used in

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Table 2. Comparison of Literature (Lit.) and Experimental (Exptl) Attenuation Coefficients Determined in CHEM 3030^a

Source	γ Energies (keV)	μ Pb (Lit., cm ⁻¹)	μ Pb (Exptl, cm ⁻¹)	% Error	μ Al (Lit, cm ⁻¹)	μ Al (Exptl, cm ⁻¹)	% Error
¹³⁷ Cs	660	1.05 ^b	1.014	3.43%	0.198 ^c	0.14	29.3%
⁶⁰ Co	1173, 1332	0.625 ^b	0.597	4.48%			

^{*a*}The higher percent error observed in the aluminum shielding data is most likely a consequence of the incomplete shielding (\approx 50%) observed using even the thickest shielding material, resulting in poorer exponential fits. ^{*b*}Reference 33. ^{*c*}Reference 34.



Figure 5. Sample data for CHEM 3030 demonstrating the relationship between shielding thickness and net counts for the (A) 137 Cs point source with lead shields, (B) 60 Co with lead shields, and (C) 137 Cs with aluminum shields. Sampling time was 20 s per measurement.

lecture 1, ¹³⁷Cs and ⁶⁰Co, were used, but the aluminum shields were chosen to have smaller thicknesses to fill in these missing data points. Additionally, because the ¹³⁷Cs activity was too efficiently attenuated by lead, we replaced them with plastic (HDPE) shields. Representative data with these modifications are shown in Figure S2 (SI). As illustrated in these figures, both data sets from ¹³⁷Cs can be fit satisfactorily to a monoexponential decay ($R^2 > 0.99$). These fits yield μ values of 43 and 15 cm⁻¹ for aluminum and plastic, respectively (Figure S2a,c, SI). Thus, as expected, the higher-Z material (aluminum) is more effective at absorbing and shielding radiation. For the ⁶⁰Co source (Figure S2b, SI), the aluminum does not effectively shield its higher-energy γ radiation, preventing a quantitative determination of μ . However, a comparison of these μ values to those reported in the literature^{33,34} reveals that they are significantly larger than expected, indicating that a systematic error was present during the implementation of this experiment in CHEM 2900.

In Spring 2021, these experiments were executed for the course CHEM 3030, implementing modifications to arrive at μ values that are consistent with literature precedence. In this procedure, the same sealed point sources of ¹³⁷Cs (5 or 10 μ Ci) and ⁶⁰Co (1 μ Ci) were used, but only with aluminum and lead shielding materials. Because the attenuation values obtained in CHEM 2900 were several orders of magnitude larger than those expected, we reasoned that the shielding effects that we were seeing were predominantly due to blockage of the β particle emissions, rather than the γ rays, of the ⁶⁰Co and ¹³⁷Cs point sources. Notably, as purchased, these disk point sources present two distinct orientations: face-up, where the yellow label of radionuclide and activity are displayed, and face-down, where the source is fully exposed (Figure S3, SI). In CHEM 2900, we used the point sources in a face-down orientation when measuring activity and shielding. In this configuration, all types of radiation, that is, both the γ and β particles, will easily reach the detector. To exclusively look at the effects of lead and aluminum shielding on the γ emissions, we used the point sources in a face-up orientation in CHEM 3030. In this orientation, we found that the plastic label was sufficient to attenuate the β particle emissions of these radionuclides, allowing only γ photons to penetrate and interact with our shielding materials. Using the point sources in this configuration, students in all sections obtained three data sets: ¹³⁷Cs with lead shields, ⁶⁰Co with lead shields, and ¹³⁷Cs with aluminum shields. Representative student data for each point source with the different shielding materials is shown in Tables S9-S11 (SI). Plots of the thicknesses of the shields versus the net counts, which are background-subtracted count measurements over 20 s sampling times, collected with each shield, are shown in Figure 5. The students analyzed these data by fitting them to an exponential function, as shown in eq 2. Compared to the results obtained in CHEM 2900 the prior year (Figures S1 and S2, SI), the data obtained by students show better exponential fits, as reflected by improved R^2 values, and more importantly give rise to linear attenuation coefficients that are significantly closer to those reported in the literature (Table 2). From these data, the students were able to conclude that lead is a more effective shielding material than aluminum for γ radiation and that the high-energy γ emissions of 60 Co are less effectively attenuated than the low-energy γ rays of ¹³⁷Cs.

Part C: Determination of an Unknown Radionuclide Eluted from a Generator

The objectives for part C include the determination of the halflife and γ energy of an unknown radionuclide (^{137m}Ba). This information can then be used with tabulated data found within various nuclide property databases to identify the unknown radionuclide. A website that we found particularly useful for this laboratory was the Lund/LBNL Nuclear Data Search site (http://nucleardata.nuclear.lu.se/toi/index.asp). After obtaining the radionuclide eluted from the ¹³⁷Cs/^{137m}Ba generator, the students measured its activity for at least 30 min. Representative raw data is shown in Figure 6. These unprocessed data show the background collection that precedes the addition of the radionuclide at t = 240 s. To fit the data to an exponential curve, only data in the region of curvature should be used. On the basis of eqs 3 and 4, the decay constant of this exponential fit can be used to calculate the $t_{1/2}$ of the radionuclide. From the representative data shown in Figure 6, a $t_{1/2}$ of 2.65 min is obtained. This value is within 5% deviation of the expected $t_{1/2}$ of 2.55 min. The γ



Figure 6. Raw data for the unknown daughter radionuclide decay. The exponential fit is shown in red. Sampling time was 20 s per measurement.

spectrum was acquired using a small commercially available Tldoped NaI scintillation detector, which was cross-calibrated with both ⁶⁰Co and ¹³⁷Cs γ emission lines prior to each laboratory period. A representative spectrum is shown in Figure 7. This spectrum displays the prominent 662 keV



Figure 7. γ spectrum of the unknown daughter radionuclide eluted from the ${}^{137}\text{Cs}/{}^{137m}\text{Ba}$ generator. The 662 keV peak arises from the ${}^{137m}\text{Ba}$ radionuclide, and the lower-energy features arise from Compton scattering.

photopeak attributed to the relaxation of the ^{137m}Ba excited state, along with lower-energy features that can be attributed to Compton scattering (the inelastic scattering of light by a free electron at a wavelength different than that of the incident radiation).³⁵ Inputting these data into the nuclear data website yields a list of potential radionuclides. A step-by-step procedure for inputting these data into the website is available in the laboratory manual provided in the SI. Students were instructed to find a radionuclide with their measured half-life and γ energy.

Feedback from Students

As discussed above, students in CHEM 2900 in Spring 2020 attended a single, 50 min lecture prior to performing the experiments. An in-depth introduction in the laboratory manual (SI) helped supplement this lecture on nuclear and radiochemistry. In this lecture, students were taught how distance and shielding affect radiation dosage, the concept of a radioactive generator, and how to practice ALARA. Therefore,

in both the pre- and postlaboratory questionnaire, we probed the students' attentiveness to the lecture, the laboratory manual, and the introduction prior to starting the experiments. Despite providing the necessary materials for the students to grasp these concepts, the prelaboratory survey questions indicate that most students did not have a good understanding of the mathematical relationship between distance and measured counts (38% in agreement with question 1, Figure S4, SI). However, a moderate number of students expressed confidence in their knowledge of how different types of shielding (53%) and shield thicknesses (60%) can be used to reduce exposure. Only 43% of students believed they understood how radioactive generators operate and how they are used in medicine, but the majority (93%) of students understood how to use the principles of ALARA for working with radioactive materials safely. Overall, the students in lecture 2 had similar responses to these questions, in which 83% of the students had a good understanding of ALARA, but only 25% had a good understanding of the concepts of shielding addressed in question 2 (Figure S5, SI).

After performing the experiments, we saw a remarkable increase in the number of students in lecture 1 that understood the mathematical relationship between distance and measured count rate (81%) as well as the effects of shielding materials (86%) and shield thicknesses (91%). Students also seemed to better understand how radioactive generators work (76%) and the concept of ALARA (98%) (Figure S6, SI). These results confirm that hands-on activities, rather than lectures alone, help students understand concepts more effectively.^{36,3} Students in lecture 2 had similar responses with 83% understanding the mathematical relationship of distance and counts, 92% understanding the effects of shielding material, and 92% having a good understanding of radionuclide generators (Figure S7, SI). In terms of the experimental procedures, most students in lecture 1 agreed (98%) that they had enough time to complete the three experiments in a 3 h laboratory period and that the experiments illustrated the concepts of nuclear chemistry and radioactivity well (91%). The combination of lecture and the Introduction section of the laboratory manual (SI) were sufficient for preparing students for the laboratory experiments (90%), and most students believe that the manual can be adapted for a freshman general chemistry course (88%). Lecture 2 students also felt that these materials prepared them for the experiments (92%) and that this manual could be adapted for a lower-level chemistry course (83%). Many students commented that the laboratory was "very interesting" and both "well-organized" and "wellstructured". One student even remarked, "...this experiment is one of the best experiments I've ever done at Cornell."

In CHEM 3030 of Spring 2021, this experiment was completed in four laboratory sections ranging from 4 to 8 students per section. These students were given the same survey questions as those asked of the CHEM 2900 students. The prelaboratory survey responses are tabulated in Figure S8 (SI). The students received a lecture before performing the experiments, similar to the one given in CHEM 2900. Similar trends were observed in this course. The majority of students felt that they did not have a strong grasp on the relationship between distance and count rate (only 35% in agreement). A moderate number of students felt that they understood how different types of shielding (70%) and thicknesses (75%) reduce radiation exposure. In this course, a very small amount (20%) of students felt that they understood how radioactive

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generators are used and implemented in nuclear medicine, but a large majority (90%) of the students felt that they understood the practices of ALARA.

After performing the experiments, the students in CHEM 3030 were asked the same postlaboratory survey questions as given to the students in CHEM 2900. These results were tabulated in Figure S9 (SI). We observed similar increases in understanding from the prelaboratory to the postlaboratory survey questions. The majority of students understood the relationship between distance and measured count rate (80%), as well as how types of shields (65%) and thicknesses (95%) attenuate γ radiation. Students still felt as though they did not understand how radioactive generators work and are used in medicinal applications (45%), but this was improved from the prelaboratory responses. Nearly all students (95%) agreed that they understood ALARA. Most students (70%) felt as though they could complete the experiment in the 3 h laboratory period provided, and an even greater majority (85%) felt that this experiment appropriately demonstrated the concepts of nuclear chemistry and radioactivity. A slight majority of students (55%) felt as though the lecture and introduction in the laboratory manual were helpful. A moderate number (60%) of students felt that this experiment was appropriate for a freshman chemistry course. In comparison to students in CHEM 2900, however, there were fewer students in CHEM 3030 who "strongly agreed" with an enhanced learning of the topics of nuclear chemistry. We note that students in CHEM 3030 were in the full midst of the COVID-19 pandemic, which limited access to in-person lecture content and led to smaller classroom and laboratory sizes, features that could have negatively affected their learning capabilities.

Despite the success of this lab, some students were concerned that the Introduction section in the laboratory manual (SI) is too long and can be "hard to follow". They suggested having the Introduction "more clearly divided into the different parts of the experiment". The lecture portion could also be modified to cover more material that was included in the laboratory introduction to eliminate the students' confusion. Overall, the feedback for this laboratory experiment is extremely positive and demonstrates how it can be a useful tool for teaching nuclear chemistry at the undergraduate level even in departments that do not have a precedence in covering this topic.

Evaluation of Learning Success

Having conducted this set of experiments in two separate chemistry courses over a 2 year span, we have obtained ample data for evaluating the learning success from our CHEM 2900 and CHEM 3030 students. The learning success of the laboratory was evaluated on the basis of the student laboratory reports. The TAs graded these reports and analyzed the student understanding of the effects of (Q1) distance on measured count rates, (Q2) shielding materials, (Q3) varying γ energies from different radionuclides, and if students were able to (Q4) identify the correct unknown daughter nuclide eluted from the generator (Figure 8). Our results indicate that most students grasped these concepts fairly well. We also note that 100% of students in CHEM 2900 lecture 2 who performed this experiment in-person displayed an understanding of all four questions in their laboratory reports. Thus, the incorporation of the feedback and modification of the protocols after CHEM 2900 lecture 1 most likely contributed to the enhanced learning success of the lecture 2 students. These results, along



Figure 8. Assessment of achieving educational objectives based on laboratory report responses from students enrolled in both courses for CHEM 2900 lecture 1 (red, n = 44), CHEM 2900 lecture 2 (blue, n = 12), and CHEM 3030 (green, n = 22). TAs were asked to look for certain criteria in the laboratory reports. These binary data represent students who were able to understand and successfully describe the particular concepts.

with the student feedback discussed above, illustrate that this set of experiments is appropriate for teaching nuclear chemistry and radioactivity at the undergraduate level. On the basis of both forms of evaluation, we believe that these experiments have met our learning objectives, as the students understood the effects and importance of distance, shielding, and radionuclide decay.

CONCLUSIONS

In this report, we have modified pre-existing experiments and incorporated them into two of Cornell University's undergraduate physical chemistry laboratory courses. These experiments were adapted to provide students with hands-on experience and gain familiarity with basic concepts of nuclear chemistry at Cornell University, an institution that has lacked chemistry courses covering radioactivity in-depth within recent years. Therefore, through the incorporation of nuclear chemistry laboratory experiments in CHEM 2900 and CHEM 3030, two physical chemistry courses, we aimed to provide the students with a basic understanding of the concepts of distance, shielding, and decay through a set of simple experiments. By using easily attainable and safe radioactive materials, such as contained point sources and a radionuclide generator containing small quantities of radioactivity, we were able to achieve these educational objectives. Our results indicate that students found this laboratory experiment both entertaining and educational. Analysis of laboratory reports revealed that our educational objectives were met, illustrating how these experiments facilitate the learning and understanding of nuclear chemistry. We envision that implementing these experiments at universities across the country will inspire students to pursue careers in nuclear and radiochemistry.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available at https://pubs.acs.org/doi/10.1021/acs.jchemed.1c00626.

Tables and figures, including procedures, sample data, and questions and responses (PDF) Laboratory manual (PDF)

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Notes

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REFERENCES

(1) Trager, R. Dire Warnings that the Pipeline for New Radiochemists is Running Dry. https://www.chemistryworld.com/ news/warnings-that-radiochemistry-is-dying/3007779.article (accessed Mar 11, 2019).

(2) Wogman, N. A.; Bond, L. J.; Waltar, A. E.; Leber, R. E. The Nuclear Education and Staffing Challenge: Rebuilding Critical Skills in Nuclear Science and Technology. *J. Radioanal. Nucl. Chem.* **2005**, 263 (1), 137–143.

(3) Shafer, J. State of Decay: Is Radiochemistry in Danger of Collapse?. https://www.chemistryworld.com/opinion/is-radiochemistry-in-danger-of-collapse/3007931.article (accessed Mar 11, 2019).

(4) Hricak, H.; Dauer, L. T. Radiation Brain Drain? The Impact of Demographic Change on U.S. Radiation Protection. *Health Phys.* **2017**, *112* (2), 126–130.

(5) National Research Council. Assuring a Future U.S.-Based Nuclear and Radiochemistry Expertise; National Academies Press: Washington, DC, 2012.

(6) Tsaparlis, G.; Hartzavalos, S.; Nakiboğlu, C. Students' Knowledge of Nuclear Science and Its Connection with Civic Scientific Literacy in Two European Contexts: The Case of Newspaper Articles. *Sci. Educ.* **2013**, *22* (8), 1963–1991.

(7) Zephrinus, C.; Njoku; Phoebe, M. I.; Eze-odurukwe. Resolving Nigerian Secondary School Students' Learning Difficulties in Nuclear Chemistry Using Computer Animation Solutions. *Procedia - Soc. Behav. Sci.* **2015**, *176*, 1034–1040.

(8) Nakiboglu, C.; Tekin, B. B. Identifying Students' Misconceptions about Nuclear Chemistry A Study of Turkish High School Students. *J. Chem. Educ.* **2006**, *83* (11), 1712–1718.

(9) Norman, J. C. Potassium in Apricots-A Nuclear Chemistry Experiment. J. Chem. Educ. 1985, 62 (5), 439.

(10) Christian, M. C. An Exercise to Teach Concepts of Half-Life without Using Radioactive Isotopes. *J. Chem. Educ.* **1988**, 65 (1), 48–49.

(11) Perry, J. T.; Sankey, M. A. Cloud Chamber Activities for the High School Classroom. J. Chem. Educ. 1995, 72 (4), 339–343.

(12) Olbris, D. J.; Herzfeld, J. Nucleogenesis! A Game with Natural Rules for Teaching Nuclear Synthesis and Decay. *J. Chem. Educ.* **1999**, 76 (3), 349–352.

(13) Liguori, L.; Adamsen, T. C. H. Learning Nuclear Chemistry through Practice: A High School Student Project Using PET in a Clinical Setting. J. Chem. Educ. 2013, 90 (7), 877–882.

(14) Marker, S. C.; Konkankit, C. C.; Walsh, M. C.; Lorey, D. R., II; Wilson, J. J. Radioactive World: An Outreach Activity for Nuclear Chemistry. J. Chem. Educ. 2019, 96 (10), 2238–2246.

(15) Ling, A. C. Nuclear Science in the Undergraduate Curriculum the New Nuclear Science Facility at San Jose State University. J. Chem. Educ. 1979, 56 (9), 595–596.

(16) Leenson, I. A. Ernest Rutherford, Avogadro's Number, and Chemical Kinetics. J. Chem. Educ. **1998**, 75 (8), 998–1003.

(17) LeFevre, J. W. Determination of the Enantiomeric Purity of Commercial L- $[U^{-14}C]$ Valine: An Experiment Utilizing Reversed-Phase Thin-Layer Chromatography and Liquid Scintillation Counting. J. Chem. Educ. **1998**, 75 (10), 1287–1290.

(18) University of Colorado, Colorado Springs. Virtual General Chemistry Laboratories: Nuclear Chemistry. https://vgcl.uccs.edu (accessed May 29, 2020).

(19) Hoeling, B.; Reed, D.; Siegel, P. B. Going Bananas in the Radiation Laboratory. *Am. J. Phys.* **1999**, 67 (5), 440–442.

(20) Nuclear I: Gamma Spectroscopy. http://galileo.phys.virginia. edu/classes/318.gbh.spring05/nuclear/ (accessed May 29, 2020).

(21) Katz, D. A. Experiments for nuclear chemistry. http://www. chymist.com/Nuclearchem.pdf (accessed May 29, 2020).

(22) United States Naval Academy Experiment 13N. https://www. usna.edu/ChemDept/_files/documents/112pdf/2021_Documents/ Week_15_-Exp_13N_Radioactivity_SP21.pdf (accessed May 29, 2020).

(23) United States Nuclear Regulatory Commission. https://www. nrc.gov/reading-rm/doc-collections/cfr/part030/part030-0071.html (accessed August 17, 2021).

(24) Burchfield, L. A. Radiation Safety: Protection and Management for Homeland Security and Emergency Response; John Wiley & Sons, Inc.: Hoboken, NJ, 2009.

(25) Grupen, C. Introduction to Radiation Protection: Practical Knowledge for Handling Radioactive Sources; Rhodes, W. T., Stanley, H. E., Eds.; Springer: Berlin, Germany, 2010.

(26) Loveland, W. D.; Morrissey, D. J.; Seaborg, G. T. Modern Nuclear Chemistry, 2nd ed.; John Wiley & Sons, Inc.: Hoboken, NJ, 2017.

(27) Pinajian, J. J. A Cesium-137 - Barium-137m Isotope Generator. J. Chem. Educ. 1967, 44 (3), 212–213.

(28) Choppin, G. R.; Nealy, C. L. Demonstration of a Parent-Daughter Radioactive Equilibrium Using ¹³⁷Cs-^{137m}Ba. J. Chem. Educ. **1964**, 41 (11), 598-600.

(29) Ruddy, F. H. A Simple ^{137m}Ba-¹³⁷Cs Milking System for Secular Equilibrium Studies. *J. Chem. Educ.* **1974**, *51* (10), 689–690.

(30) Spectrum Techniques. Cs-137/Ba-137m Isotope Generator Operating Instructions.

(31) Chu, S. Y. F.; Ekström, L. P.; Firestone, R. B. The Lund/LBNL Nuclear Data Search http://nucleardata.nuclear.lu.se/toi/index.asp (accessed May 29, 2020).

(32) NuDat 2.8; https://www.nndc.bnl.gov/nudat2/ (accessed May 29, 2020).

(33) Buyuk, B.; Tugrul, A. B. Comparison of Lead and WC-Co Materials against Gamma Irradiation. *Acta Phys. Pol., A* **2014**, *125* (2), 423–425.

(34) Yildirim, S.; Tugrul, A. B.; Buyuk, B.; Demir, E. Gamma Attenuation Properties of Some Aluminum Alloys. *Acta Phys. Pol., A* **2016**, *129* (4), 813–815.

(35) Compton, A. H. A Quantum Theory of the Scattering of X-Rays by Light Elements. *Phys. Rev.* **1923**, *21* (5), 483–502.

(36) Holstermann, N.; Grube, D.; Bögeholz, S. Hands-on Activities and Their Influence on Students' Interest. *Res. Sci. Educ.* 2010, 40, 743-757.

(37) Bergin, D. A. Influences on Classroom Interest. *Educ. Psychol.* **1999**, 34 (2), 87–98.