Radiochemistry and Nuclear Chemistry Laboratory at Washington University

1. Background:
The Department of Chemistry at Washington University for many decades offers a strong program of graduate and undergraduate education in Nuclear Chemistry, Radiochemistry and Nuclear Medicine.

The Radiochemistry laboratory is an independent course and includes a one-hour lecture and a five-hour laboratory. The students and the instructor select experiments covering a period of 12 working weeks from a collection of 18 well-structured experiments (see Section 3 below for a list of abstracts). Professors A.C. Wahl, D.G. Sarantites, L.G. Sobotka, and C. Anderson have contributed to the development of these 18 experiments over the years.

During the semester access to the instructor, teaching assistant and the analysis equipment is provided during each working week. A laboratory report with the analysis and interpretation of the data provides the basis for evaluating the student performance. A fully structured laboratory manual has been prepared and is offered to the students. It contains the detailed write up of each experiment as well as the instructor’s lecture notes in detail.

2. Recently Added Experiments to the Radiochemistry Laboratory course:

(1) Preparation and characterization of a radiopharmaceutical compound. (2 weeks) (C. Anderson)
The four parts of this assignment include (a) Preparation of $^{99m}$Tc-PnAO via direct reaction of $^{99m}$TcO$_4^-$ with PnAO in a reducing environment. (b) Preparation of $^{99m}$Tc-PnAO using an exchange ligand. (c) Characterization by paper chromatography. (d) Characterization by solvent extraction.

(2) Multi-elemental analysis by X-ray fluorescence with a Si-PIN diode. (1 week).
Abstract: The advent of inexpensive high-resolution X-ray spectrometers based on thermoelectrically cooled Si-PIN diodes has opened the possibility of many innovative experiments in analytical chemistry with many applications in art and archeology, environmental monitoring, nuclear medicine, toxic dump site monitoring and many others. Elemental analysis of known and unknown samples is done with environmentally interesting samples. Analysis for Pb poisoning in simulated bone material is easily done in this experiment. In these experiments $^{109}$Cd or $^{55}$Fe sources, and/or a pyro-electric X-Ray generator are used for activation of the samples.

(3) Mossbauer back-scattering Spectroscopy (2 weeks).
The Mossbauer effect is one of the most elegant spectroscopic probes of the local magnetic and electric fields. Just as important, in the educational setting, understanding the phenomenon requires the melding of knowledge from several fields. For example, beta (electron capture) and gamma decay, momentum transport and balance, intrinsic and Doppler broadened line widths, material science, and quantum mechanics in general are required for a proper explanation of the Mossbauer effect.

The standard mode for employing the Mossbauer effect is in the absorption mode. The material under study is placed between a moving source and a fixed detector. When the
velocity brings the gamma-ray photon from the radioactive decay within the intrinsic line width, the absorption is enhanced. The set of velocities with enhanced absorption map out the possible set of transitions between levels split by the local magnetic field or an inhomogeneous electric field.

On the other hand, a backscattering geometry is also possible. This geometry is attractive in that it allows for "remote sensing" and thus has been selected for an analytic probe for an upcoming mission to Mars. In this geometry, the moving source is on the same side (at about 160°) from the detector. Essentially elastic backscattering occurs (rather than a Compton process) when the source velocity allows resonance. This is a truly beautiful experiment, capturing all the science embodied in the basic spectroscopy along with the potential for remote sensing.

The equipment used are: a) Cooled Si PIN (as used in x-ray fluorescence) b) source drive, c) gated PHA.

3. Complete List of Available Experiments offered presently in the “Laboratory in Nuclear and Radiochemistry”

1: Measurement and absorption of beta and gamma rays. Preparation of samples for beta counting - Efficiency of the beta counter (1 week)

Abstract: Part 1 of this lab period familiarizes the student with the operation of a radiation dosimeter, a radiation survey monitor, and a beta/alpha proportional counter. In part 2 the student learns to prepare samples for β counting, and determine the efficiency of his β- counter for 234Pa β particles as a function of sample weight.

2: α-Particle spectroscopy - The thorium decay series – Recoil Chemistry – Parent daughter relationship (2 weeks)

Abstract: Part 1 of this laboratory familiarizes the student with the radiochemical separation of 212Pb from natural 232Th by a PbS precipitation with lead carrier. In the dissolved PbS, the Pb(Ac)₄⁻² is formed and a carrier free separation of the 60-min 212Bi is achieved by spontaneous electroplating on Ni. The rapid separation of the 3.2 min 208Tl from the 212Bi is achieved using the recoil energy following α-decay of the 212Bi. The decay of 208Tl is followed by β-counting. The β-to-α-branching ratio of the 212Bi decay is measured quantitatively using α-particle spectroscopy with a surface barrier Si detector to obtain a spectrum of the α-particles. Part 2 (second week) of this laboratory teaches the student the determination of the genetic (parent-daughter) relationship of the 212Bi and the 212Tl by successive rapid recoil chemistry (milking of the daughter at successive intervals). The use of a computer based multi-channel analyzer is introduced for the α-spectroscopy.

3: γ-Ray measurements with scintillation and semiconductor Ge detectors (1 week)

Abstract: This experiment teaches the student the quantitative use of the NaI(Tl) scintillation detector both as a spectrometer and a well-type counter. In addition, quantitative use is made of a Ge detector both as a spectrometer and as an analytical tool. The relative and absolute efficiency of a Ge detector is made with several multi-peak radioactive and/or absolutely calibrated radioactive standard sources. Use of a computer based multi-channel analyzer is routinely made and the data are analyzed with the computer.
4: Principles of Liquid-Scintillation counting: Measurement of $^{14}$C radiation (1 week)

**Abstract:** In this experiment the use of $^{14}$C measurement by means of liquid scintillation counting is introduced and compared with the direct $\beta$-counting. Particular emphasis is given in the determination of quenching of the scintillation radiation by various substances. The student measures the quenching via standards and thus understands the operation of automatic blind boxes used routinely in chemistry and biology. The concept of specific activity is introduced and explored.

5: Neutron activation - Hot-atom chemistry - The Cyclotron (1 week)

**Abstract:** In this experiment the student learns about the concept of "hot atom chemistry". Following a nuclear reaction involving an atom bound to a molecule, bonds are broken and active free atoms (radicals) are produced that cause new chemical reactions. Here the $^{127}$I ($n_{\text{th}},\gamma$) is used to break the C-I bonds in CH$_3$CH$_2$-I. The enrichment factor is determined experimentally in a radiochemical procedure. Thermal neutrons are produced at the medical school cyclotron and as a side product the student learns to calculate nuclear reaction cross sections from experimental data.

6: Analysis by Neutron activation and by Isotope dilution (1 week)

**Abstract:** In the first part of this experiment the student learns to do a neutron activation analysis using the ($n_{\text{th}},\gamma$) reaction in a sample containing an unknown metal. Using a half-life measurement and a Ge $\gamma$-ray spectrum the student has to find the identity of the unknown metal. In the second part, $^{12}$C-labeled benzoic acid of measured specific activity is used to analyze a sample contain an unknown fraction of benzoic acid by the method of isotopic dilution. Here $^{14}$C liquid scintillation counting is employed.

7: Instrumentation for nuclear spectroscopy - Construction of a Decay scheme - The Coincidence method (2 weeks)

**Abstract:** In the first part of this experiment the student practices with the electronics setup for a $\gamma$-$\gamma$ coincidence measurement. Use is made of slow and fast (nsec) constant-fraction timing discriminators, single channel analyzers, shaping and delay amplifiers, and the pulse height analyzer. The student measured the timing resolution and the relationship between random and true coincidences as a function of counting rate. The second part of the experiment deals with the quantitative construction of a level scheme following $\beta$-decay of an "unknown" radio-nuclide. The decay scheme is constructed from "singles" $\gamma$-ray energy and intensity measurements with a Ge detector and the $\gamma$-$\gamma$ coincidence relationships in time using two NaI(Tl) detectors. Analysis of the data is done with the acquisition computer after the measurement of the spectra.

8: Angular correlation of the positron annihilation quanta - Angular correlation of gamma rays in Cascade (1 week)

**Abstract:** In the first part of this experiment the angular relationship of the two 511-511 keV annihilation quanta following $\beta^-$-decay is determined. The co-linearity of the two quanta is established despite the limited angular resolution due to the finite solid angle of the $\gamma$-ray detectors. In the second part the $\gamma$-$\gamma$ angular correlation following an M4$\rightarrow$E2$\rightarrow$ cascade from $^{207}$Bi is measured with sufficient accuracy to determine both the $A_2$ and the
A4 coefficients of a Legendre polynomial expansion of the correlation. Two 5.08×7.62 cm NaI(Tl) detectors are used in the correlation measurements and the electronics setup from Assignment 7 is used.

9: Solubility of PbI₂ by use of ¹³¹I tracer (1 week)
Abstract: In this exercise the students use a radioactive tracer, ¹³¹I, to measure the solubility at 25° of the very insoluble salt, PbI₂. Use is made of the limiting law of Debye-Hückel for weak electrolytes and the solubility is measured as a function of ionic strength. By extrapolating to zero ionic strength the thermodynamic value of the solubility equilibrium constant is obtained. The experiment can be easily extended to derive the thermodynamic constant at different equilibrium temperatures.

10: Study of the iodine fission products from neutron irradiated uranium (1 week)
Abstract: In this experiment the student learns to separate radiochemically the iodine fission products from a ²³⁵U sample irradiated thermal neutrons. Use of oxidation/reduction reactions together with solvent extraction procedures are used to purify the ¹³³,¹³²,¹³¹I activities from the fission products. The students determine the activities of these genetically unrelated isotopes at the end of irradiation. This is done by resolving the β-decay curves. The difference in half-life. The concepts of fractional and cumulative independent yields and decontamination factor are introduced and explored.

11: Isotopic Exchange (1 week)
Abstract: In this exercise the students use the radioactive tracer ¹³¹I to measure the rate of isotopic exchange of iodine atoms between ethyl iodide and sodium iodide in an ethanol solution. The reaction rate and the half reaction time are measured and the rate constant for the reaction is calculated. Use and verification of the Arrhenius equation is made.

12: Behavior of a Carrier-Free Tracer. Chemical Properties of ²³⁹Np (1 week)
Abstract: In this experiment the students learn about the very different chemical behavior of substances in carrier free amounts. Substances present at very small (tracer) concentrations (e.g., ~ 10⁻¹⁰ M) may behave differently chemically than they do at ordinary macro-concentrations (e.g., ~ 0.1 M) for several reasons. One reason is that at very small concentrations a large fraction of a substance may be absorbed in a mono-layer (or less) on a surface a few square centimeters in area, such as the walls of the containing vessel, the surface of a precipitate, or the interface between two immiscible liquids. The chemistry of ²³⁹Np is studied in some detail.

13: Passive Radon Monitoring (1 week)
Abstract: Indoor exposures to daughters of ²²²Rn contribute significantly to the radiation dose that the general public receives. The magnitude of this activity in homes in the United States varies from less than 0.1 pCi/liter to as much as 20 pCi/liter. In this assignment the student uses a passive monitor to measure the radon activity in his home. Near the end of the semester the detector will be etched and from a count of the tracks due to natural radon activity the ²²²Rn concentration is deduced.
14: Fission (2 weeks)

Abstract: In this experiment the students study the energy division in the spontaneous fission of $^{252}$Cf. This is manifested by the emission of a different number of neutrons from the light and heavy fragments. The students measure the angular distribution of the emitted neutrons coincident with the light and then the heavy fragments. The observed difference between the angular distributions is mainly due to the kinematic boost of the neutrons from the moving fragments. The neutron multiplicities are included in the analysis of the measured yields. The techniques of pulse-shape discrimination between ν-γ and time of flight are employed in the measurements. Surface barrier detectors are used to detect the fission fragments and to separate the heavy from the light fragments on the basis of their kinetic energy distributions.

15: Positrons and Positronium (2 weeks)

Abstract: In this experiment the precise deviation of the emission of the 511-511 photons following $\beta^+$ annihilation in a number of media (Cu, Al, and graphite) is measured. From this information the Fermi energy of the electrons in the conduction band of the slowing down media (Cu, Al, and graphite) is calculated and differences are discussed.

16: X-ray Fluorescence (1 week)

Abstract: (A) X-ray fluorescence will be used to measure the relative Pb content of plaster-of-Paris (Tibia) phantoms. The K vacancies in the Pb will be created using low energy γ-ray emitters and the X-rays will be detected in a solid-state intrinsic Ge detector. Abstract: (B) Multi-elemental analysis by X-ray fluorescence. (use in Art, Archeology and environmental science)(1 week).

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