

# Radiochemical Processing Laboratory



**Pacific Northwest**  
NATIONAL LABORATORY

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The RPL is being transformed into the cornerstone of Pacific Northwest National Laboratory's Capability Replacement Laboratory to facilitate cutting-edge science with radioactive materials using state-of-the-art equipment and instrumentation.



# RPL:

## Radiochemical Processing Laboratory

Organizations needing innovative processes and technical solutions for environmental cleanup, nuclear energy, nonproliferation, homeland security and the beneficial use of radioactive materials can look to the Radiochemical Processing Laboratory at the Pacific Northwest National Laboratory.

At the RPL, we are committed to developing solutions to clients' most difficult problems by providing highly qualified staff, safe and efficient operations, state-of-the-art facilities and an organization dedicated to teamwork.

As a Hazard Category 2 non-reactor nuclear facility, the RPL houses specialized facilities for work with microgram-to-kilogram quantities of fissionable materials and megacurie activities of other radionuclides. These provide a platform for radiochemical process development, chemical and physical separations, radiomaterial characterization, radioisotope research, reactor dosimetry and radioactive and hazardous waste management.

Housed within the Department of Energy's Hanford Site in southcentral Washington State, the RPL is being transformed into the cornerstone of Pacific Northwest National Laboratory's Capability Replacement Laboratory to facilitate cutting-edge science with radioactive materials using state-of-the-art equipment and instrumentation.



## Radiochemistry and Processing

### Developing Radiochemical Processes at All Scales

Among the key features of the RPL are extensive specialized facilities and instrumentation to identify and quantify chemical species and radioactive isotopes in simple and complex media. Our staff can work with highly radioactive material, highly dispersible isotopes, trace levels of radionuclides and anything in between.

Our capabilities in radiochemical process engineering can be applied to:

- ▶ Develop process flowsheets
- ▶ Design, install and test radiochemical process systems
- ▶ Develop engineered systems for toxic and highly radioactive systems.

Our primary expertise lies in development, scale-up and deployment of first-of-a-kind processes to solve environmental problems. This includes extensive experience with Department of Energy tank waste and actinide process streams.

Our glovebox and shielded facility capabilities allow us to design, install and test small-scale radiochemical processes to support the development of large-scale counterpart facilities.

At the RPL, actual waste samples have been analyzed to determine the fluid-flow properties of the complex high-salt, solids-laden fluid. Non-radiological waste simulants that possess similar fluid-flow properties have been developed at the RPL to support large-scale mixing and transport tests.



Researchers design, build and operate small-scale radiochemical process test equipment in gloveboxes.

### Expert Chemical and Physical Separations

The RPL's unique facilities and multidisciplinary staff enable separations research that spans from the molecular level to testing of flowsheets for industrial applications. Our expertise in the fundamental chemistry of

Development of innovative and improved radiochemical separations methods at the RPL can lead to dramatic increases in productivity in radioanalytical laboratories.

Here, a researcher works with a rheometer to study the fluid-flow properties of complex non-Newtonian media such as solid-liquid suspensions.



radionuclides and our knowledge of radiochemical separations technologies enable us to develop innovative solutions to meet our clients' radiological separation and processing needs.

The focus of separations research in the RPL is testing and experimentation with the actual radioactive materials requiring processing. Existing testing capabilities include ion exchange, solvent extraction and cross-flow ultrafiltration separation techniques. Specialized capabilities for flowsheet testing of these techniques include 2-cm centrifugal contactors for solvent extraction and a cell-unit filter for radioactive and non-radioactive cross-flow ultrafiltration. In addition, we employ spectroscopic techniques, calorimetric methods

and other equipment to determine chemical (e.g., thermodynamics, actinide speciation) and physical parameters (e.g., rheology, thermal conductivity, particle-size distribution) of the radioactive materials to be processed. This information provides us with the insight necessary to design better separations or to refine chemical separation processes.

Separations research in the RPL also encompasses developing new methods for radioanalytical applications that can lead to dramatic increases in productivity in radioanalytical laboratories. Examples include development of new separations media and design and testing of automated radiochemical separations.

## Plutonium and Tritium Processing Capabilities

The RPL has capabilities for testing plutonium and tritium processing flowsheets and providing direct design input or solutions for full-scale facilities. Staff at the RPL perform laboratory-scale to bench-top processes for the Tritium Extraction Facility and Pit Disassembly and Conversion Facility at Savannah River Site including:

- ▶ Determining processing parameters needed to meet facility functional design criteria
- ▶ Determining input for process flowsheets
- ▶ Providing input on equipment design or specification
- ▶ Providing input for process model development.

The laboratories at the RPL have high-temperature (1100°C) vacuum furnaces for tritium extraction in a shielded area and equipment for handling plutonium and tritium. The furnaces can process irradiated materials such as stainless steel and zircaloy with sample sizes from a single gram to kilograms and containing up to thousands of curies of tritium. The collected gas can be analyzed by a high-resolution mass spectrometer.



One of the unique capabilities of the RPL is actinide research. Here, a researcher works with a plutonium sample using one of several gloveboxes in the RPL.

Plutonium purification and separation processes can be demonstrated in lab-scale furnace and dissolution equipment. For the Pit Disassembly and Conversion Facility, we demonstrated gram-size plutonium metal separation from uranium and other metals and reaction with oxygen mixtures. The products from the process can be analyzed directly or by methods described in the Nuclear Materials Examination section. These capabilities complement the RPL's expert chemical and physical separations capability.



A researcher works with a fluid-handling robot (at right) and flow injection and sequential injection-based apparatus designed in the RPL. These instruments serve as valuable research tools to study radiochemical processes, develop novel separation methods and perform highly reproducible automated radioanalytical measurements.

## Automated Process Monitoring and Radiochemical Separations

Staff at the RPL have developed automated process monitors and radiochemical separation processes. These include radiochemical analyzers that perform a suite of complex wet chemical functions within closed fluidic systems. Our systems are custom tailored to analyze specifically targeted radionuclides in a wide variety of sample matrices—ranging from high-activity tank waste supernatant to trace-level groundwater. Automated instrumentation has been designed to perform in-line sample matrix modification, spike addition, column separations and flow-through radiometric detection.

Ongoing projects include automated sample preparation processes for determination of actinides from environmental samples, design of a medical isotope generator system for yttrium-90 production and construction of a field-deployable radioanalytical system for remote groundwater analysis.

We have developed a Raman process monitor that combines a Coriolis meter and conductivity probe to quickly generate real-time data on process streams. This system has applications not only for high-activity radioactive waste systems but for industry. The system can be tailored to specific analyte detection in a variety of complex mixtures in chemically harsh environments, such as pulp and paper processing liquids, electroplating solutions and radioactive tank wastes.

We have developed an automated radiochemical sensor that can detect and quantify beta-emitting radionuclides, such as technetium-99 or strontium-90, in groundwater. The sensor requires no reagents to function and therefore can be used for in situ measurements of groundwater contamination plumes. This novel system permits continuous monitoring of groundwater contamination levels.

## Providing Radiological NMR Spectroscopy

Highly radioactive samples, including those containing fissile isotopes, can be examined in the RPL's Radiological Nuclear Magnetic Resonance Laboratory. Instruments include a three-channel spectrometer with a 7.1 Tesla widebore superconducting magnet and a broadband instrument interfaced to a variable-field electromagnet. The facility is equipped with an array of probes providing a broad spectrum of capabilities for investigating solid and liquid radioactive samples. NMR research has been performed on nuclear waste forms, solution-state uranium complexes, Hanford tank wastes, radioisotope extractant materials and technetium solids. The RPL is the only

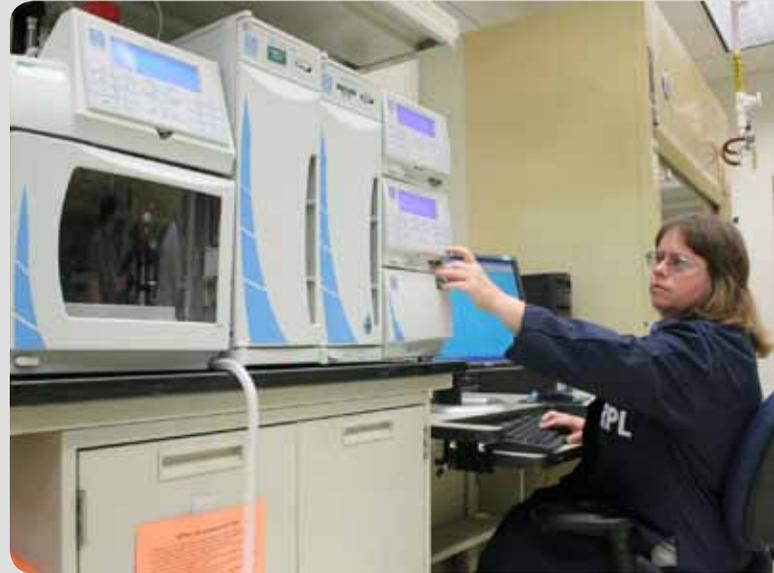
laboratory in the United States that can perform solution-state tritium NMR measurements. The solids-structure and structure-in-solution experiments are fundamental to structural synthesis and characterization. Work performed here has been reported in *Nature*, *Journal of the American Chemical Society*, *Review of Scientific Instruments*, *Radiochimica Acta* and *Journal of Solution Chemistry*, among other publications.

The RPL is the only laboratory in the United States that can perform solution-state tritium NMR measurements.

## Providing Analytical Chemistry—From Hot to Not

The RPL's experienced staff, state-of-the-art facilities and specialized instrumentation provides clients capabilities that support process development and R&D. These include radionuclide quantification in a fully equipped radioanalytical counting room, radionuclide separations and sample preparation laboratories (including hot cells), inorganic analyses methods for determining either high-concentration or trace-level analytes, wet chemistry methods, physical property methods and methods for organic/inorganic carbon content. All of these capabilities support the analysis of radioactive and non-radioactive samples.

We specialize in the analysis of highly radioactive materials and very complex sample matrices. The often-complex nature of the samples analyzed calls for customized and adaptable analyses designed by the scientists to meet client objectives. This work is performed by trained staff using established procedures and a regulatory-compliant quality assurance plan. Examples of sample types analyzed include Hanford tank waste, K-Basin sludges/spent fuel, metals, oils, filters/smears, residues, debris, groundwater and soils.



RPL's ion chromatograph provides analysis of anion and oxyanion chemical species in solution. Here a researcher is performing sample analysis.

### Medical Isotope Production for Cancer Treatment

The development of processes to produce radioisotopes for cancer treatment is an exciting capability offered within the RPL. Radioisotopes are separated from nuclear materials and then purified for use in cancer therapy. These radioisotopes can be linked to targeting molecules such as monoclonal antibodies or peptides that attach to specific sites on the surface of a cancer cell. The RPL helped a small company, IsoRay Medical, Inc., develop a new treatment for prostate cancer using seeds containing cesium-131. This novel technology won an R&D 100 Award and an FLC Excellence in Technology Transfer Award.



The RPL's Radiological NMR Laboratory houses a 300-MHz spectrometer and a variable field spectrometer.

## Nuclear Materials Examination

### Post-Irradiation Examination

The RPL has developed capabilities to perform post-irradiation examination of full-length rods in support of the Tritium Readiness Campaign for the National Nuclear Security Administration. Since 2003, the RPL has received and sectioned full-length Tritium-Producing Burnable Absorber Rods (TPBARs) in our High-Level Radiochemistry Facility. To process the rods, our staff has developed several cutting and handling methods, including longitudinal cutting and disassembly.

Once the rods are disassembled, researchers use a variety of techniques to examine them, including:

- ▶ Microscopy
- ▶ Hydrogen isotope assay (hydrogen, deuterium, tritium)
- ▶  $^3\text{He}$  assay of steel
- ▶ Surface analysis
- ▶ Thin-film thickness by Fourier Transform Infrared Spectroscopy.

These examinations can also be performed on classified materials.



The post-irradiation examination capability at the RPL includes the ability to puncture and then section full-length Tritium-Producing Burnable Absorber Rods. Shown here is the puncture apparatus used on TPBARs to release gases before sectioning.

### Microanalysis and Characterization

The RPL provides integrated examination and characterization capabilities related to radioactive materials. The RPL's 300-keV analytical

transmission electron microscope provides radionuclide materials analysis at the nanometer scale, almost a thousand times higher resolution than other imaging technologies. This microscope provides researchers with information on a sample's composition, structure and morphology through spectroscopy, electron diffraction and imaging.

The RPL's shielded scanning electron microscope with light-element dispersive spectroscopy is used to analyze highly radioactive samples, such as spent nuclear fuel. These capabilities, combined with the RPL's ability to receive and prepare highly active and dispersible materials, enable opportunities for radioactive materials research not normally available elsewhere.

The RPL's 300-keV analytical transmission electron microscope provides unsurpassed insight into actinide and technetium chemistry and can support projects in heavy-element chemistry, environmental remediation and radioactive waste management.

Microanalysis and characterization capabilities, combined with the RPL's ability to receive and prepare highly active and dispersible materials, enable opportunities for radioactive materials research not normally available elsewhere.



## Meeting Spent Nuclear Fuels Challenges

Commercial and defense spent nuclear fuels present challenging cleanup and disposition problems. The RPL's extensive expertise coupled with our materials characterization and testing capabilities support development of processing and disposal pathways for these highly radioactive materials. Our work with these materials supports Department of Energy missions, ranging from development of a geologic repository for disposal of high-level nuclear waste to interim storage strategies for site-specific materials.

## Examining Radioactive Surface Samples

The RPL's Radiological Surface Science Laboratory provides a wide range of instruments for examining surfaces. The RSSL combines these powerful research tools with the ability to examine radiological samples, creating new opportunities for basic through applied research. The RSSL can receive, test and prepare highly active and dispersible samples for surface analysis.

Surface analysis capabilities include scanning Auger Electron Spectroscopy (AES), X-ray Photoelectron Spectroscopy (XPS), Secondary Ion Mass Spectrometry (SIMS) and secondary electron and absorbed-current imaging. Depth profiles, or plots of how the concentrations of element vary with depth, can be collected using each of these techniques.

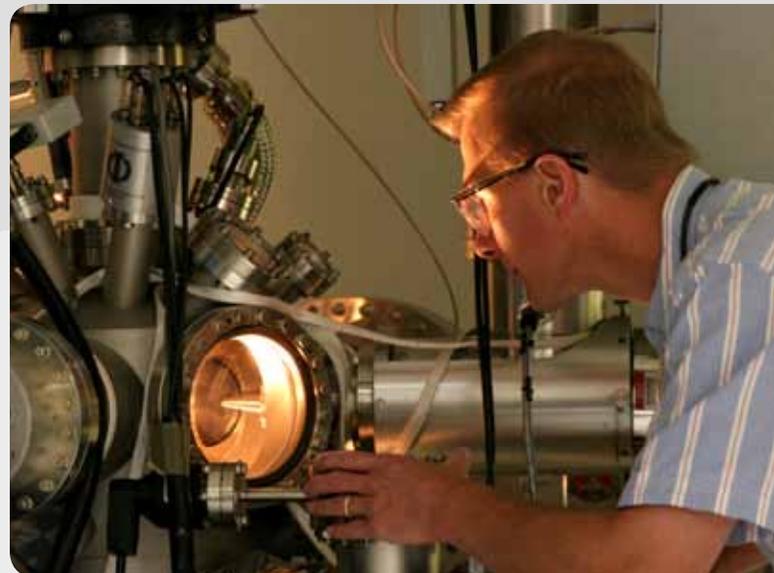
## Comprehensive Dosimetry Services

The RPL's dosimetry services can help characterize complex reactor environments, effectively use material test data, increase reactor safety and plant life extension, develop advanced reactor alloy materials and design advanced fission and fusion reactors.

Our comprehensive services include neutron fluence and spectral measurements, hydrogen and helium gas measurements and extensive computer calculations of radiation damage effects.

We also provide custom neutron flux monitor capsules containing 14 or more materials for the simultaneous measurement of multiple reaction products and for total helium generation. One small monitor capsule can contain both radiometric and helium gas monitors. Combining multiple monitors into one capsule and providing comprehensive reactor services minimizes the need for reactor irradiation time, providing lower cost and faster service to our clients.

The RPL has a comprehensive suite of analytical instrumentation for measuring activation products by gamma spectrometry, alpha or beta counting following radiochemical separations, mass spectrometry on radioactive or stable isotopes and hydrogen and helium gas measurements.



A researcher prepares to examine surface oxidation of a spent nuclear fuel sample using a SPECS XPS spectrometer mounted on a Physical Electronics 545 Scanning Auger System.

## Shielded Facilities

The RPL contains two fully staffed and equipped hot cell complexes for conducting work with highly radioactive materials. These complexes provide unique, complementary capabilities for conducting bench-scale to pilot-scale work with wide varieties and forms of radioactive materials.

The RPL's Shielded Analytical Laboratory hot cells are specially designed to perform a variety of analytical chemistry operations on radioactive samples with dose rates of up to 2000R/h. The High-Level Radiochemistry Facility hot cells are designed to shield radiation levels of up to 10E6 roentgens per hour at 1.2-MeV gamma. Work

performed in these cells includes waste tank characterization and processing verification, pretreatment, advanced analytical methods development, isotope processing, advanced

separations and reactor fuel handling. Work with classified materials and documentation is also conducted in the facility.

Our experience and capabilities include:

- ▶ Radiochemical separation and purification
- ▶ Sectioning of full-length tritium-producing absorber rods for complete post-irradiation examination
- ▶ Irradiated fuel/target sectioning and processing
- ▶ Metallography and ceramography
- ▶ Activated metals physical properties testing
- ▶ Thermal processing
- ▶ Materials physical properties testing (solid/liquid separation, centrifugation, settling behavior)
- ▶ Radioanalytical and preparatory chemistry operations (acid dissolution, aqueous/solvent extraction or leaching, distillation, ion exchange, caustic fusion).



Researchers at the RPL conduct analytical research on nuclear materials using hot cells that shield the researcher from the radiation.

## Subsurface Science

The Environmental Science Laboratory within the RPL provides forefront equipment, instrumentation and laboratory facilities that support Pacific Northwest National Laboratory's subsurface science capability. These capabilities provide methods for investigating the earth's subsurface and support Department of Energy programs and industrial clients. Study areas are divided into three groups: solids, solution composition and water/geologic media interactions.

A researcher holds a sample cup for the x-ray fluorescence analyzer in the RPL's Environmental Science Laboratory. The XRF uses x-rays to produce characteristic photons for individual elements. Both liquid and solid samples can be analyzed. XRF has distinct advantages for elemental analysis of solids because little sample preparation is required.





## Ensuring Safety, Efficiency and Compliance

The RPL's Nuclear Operations organization works in tandem with researchers to ensure that work is performed safely and efficiently. The RPL is compliant with all applicable nuclear safety, quality, environmental safety and health, radiological control and waste management regulations and Laboratory implementing systems and procedures. A state-permitted treatment, storage and disposal facility is located onsite.

Our Integrated Operations software provides tools to conduct safety and health self-assessments; initiate and manage staff and visitors' access; determine training and qualification requirements and facilitate productivity, efficiency and safety. These tools facilitate hazard identification and provide mitigation strategies to manage those hazards.

The RPL also has its own online operations manual that covers all aspects of work performed

### Special Features of the RPL

- ▶ Permitted waste treatment storage and disposal facility
- ▶ Low-level waste compactor
- ▶ Double-shielded, instrumented waste tanks for hot cell use
- ▶ Remote capabilities to inspect dangerous waste tanks
- ▶ Continuous program alarming and monitoring systems to ensure safe operating conditions
- ▶ Exhaust air sampling capabilities for radioactive material sampling
- ▶ Laboratory gas distribution system for P10, methane, UPH methane, acetylene, hydrogen

in a nuclear facility and integrates requirements from the Department of Energy and other federal agencies to bench-level operations.

Our Quality Assurance and self-assessment programs work in concert with Laboratory operations, ensuring delivery of the highest value to our customers, who dictate the level of QA applied to their work. We have experience working to DOE/RW-0333P and ASME NQA-1 QA requirements.

## About Pacific Northwest National Laboratory

The Pacific Northwest National Laboratory, located in southeastern Washington State, is a U.S. Department of Energy Office of Science laboratory that solves complex problems in energy, national security and the environment, and advances scientific frontiers in the chemical, biological, materials, environmental and computational sciences. The Laboratory employs 4,000 staff members, has a \$760 million annual budget, and has been managed by Ohio-based Battelle since 1965.

The Radiochemical Science and Engineering Group Manager of the Energy and Environmental Directorate and the Nuclear Operations Division Manager of the Operational Systems Directorate work collaboratively to deliver world-class research to a diverse customer set.

For more information about the Radiochemical Processing Laboratory, contact:

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