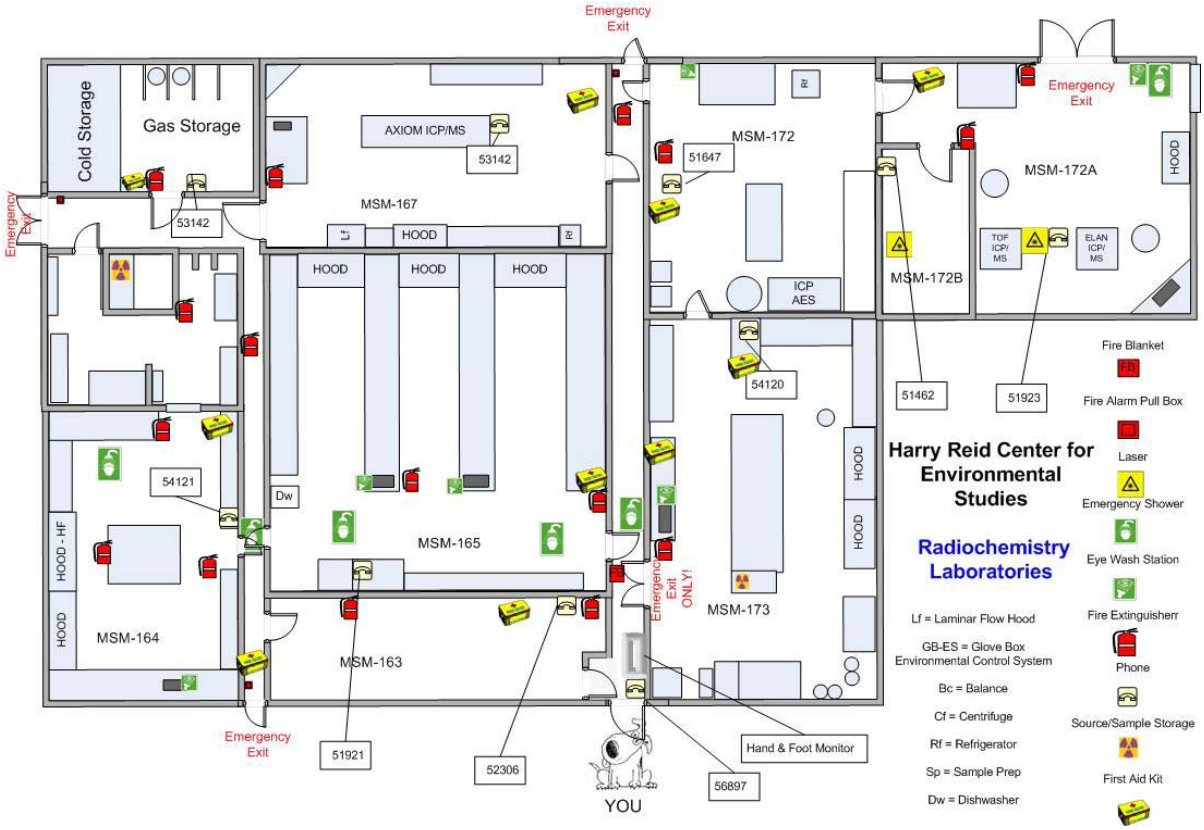
University of Nevada, Las Vegas
Facilities and Other Resources/Laboratories

The Radiochemistry Laboratories at UNLV-Harry Reid Center (HRC) provide the structure for the primary laboratories of the UNLV Radiochemistry program. The infrastructure of the laboratories is maintained by UNLV Facility Maintenance and a small staff of laboratory management personnel. Some of the services provided by the structure are: Pure water systems supplied by a large RO system. Argon gas provided to the labs and supplied by a bulk gas system. Gasses used for other purposes are stored away from the labs. Each lab is individually secured; high level labs are alarm protected. Stock solutions of radioactive material are provided multiple levels of protection. Radioactive waste is collected from the labs and transferred to the UNLV RSO.



The Radiochemistry Laboratories at the UNLV-HRC

Equipment

Canon on Solid-Phase Analytical Equipment

The instrumentation canon to accomplish a proposed work scope is described in the following. Each of the equipment on solid phase analysis is suited to allow the analysis of fresh transmutation fuel including uranium, plutonium and the minor actinides (americium, neptunium, and curium).

Metallographic Microscope: Leica IRM
Metallographic microscopy will be applied to examine phase evolution and phase distributions of calcined and sintered specimen also in regard to homogeneity and microstructure (grain boundary, pore space, density). The Leica IRM has a superior optical performance and is well equipped to perform analysis to highest standards from magnification of 40 to 1,000 times.

X-Ray Powder Diffraction: PANalytical X'Pert Pro and Bruker-AXS D8 Advance Vario
X-ray powder diffraction is the most powerful tool to determine crystalline phases, lattice parameter and the progress of solid solution formation. UNLV is in the unique situation to allow the combination of X-ray diffraction data collected with highest diffracted intensities (PANalytical X'Pert Pro) with those collected to highest lattice-space resolutions possible today (Bruker-AXS D8 Advance Vario). Qualitative phase analysis will be performed based on the latest ICDD-PDF2 database for X-ray diffraction powder data and qualitative data analysis based on the ICSD database in combination with Rietveld analysis.

Rietveld Analysis: Bruker-AXS TOPAS 3.0
Rietveld analysis is the 21st century analytical tool to quantify diffracted intensities of X-ray powder diffraction data and to advance in the determination of the structure-amplitude (yield of structure factor). The Rietveld algorithm allows to fit and to calculate the diffracted peak intensities based on the structure factor equation and allows the quantitative determination of the crystalline phases present. Therefore input data from the Inorganic Structure Database (ICSD) will be used and refined. Least square lattice parameter refinement and the use of an internal profile standard (SRM 640c, SRM 660a) will provide crystallographic data of highest precision and lattice parameter will be refined to accuracies of typical ± 50 fm (± 0.0005 Å).

Scanning Electron Microscopy (SEM): JEOL JSM-5610
The JSM-5610 SEM will be used to determine microstructure in the magnification range of 40 to 5000 times, and is optimized for imaging of submicron scale topographies of the ceramics. The JSM-5610 is equipped with detectors to image secondary and backscattered electrons and an analytical Oxford ISIS EDS system. This energy dispersive spectroscopy system is capable of qualitative and semi-quantitative elemental analyses, and also features X-ray mapping. The system also includes a most recent Oxfors EBSD (electron backscatter diffraction) attachment to determine and quantify grain orientations and texture. The JSM-5610 SEM is used as essential tools for microstructural analysis and for materials development. It serves as a reliable and easy to maintain workhorse to UNLV researchers.

The features of the JEOL JSM-5610 SEM:

- Electron gun: Tungsten (W) filament
- Resolution: 3.5 nm
- Accelerating voltage: 0.5 to 30 kV
- Electron detectors: SEI, BEI (COMPO, TOPO, Shadow)
- EBSD (electron backscatter diffraction) detector
- Qualitative analytical function: Oxford ISIS EDS system
- Magnification: x18 to 300,000
- Detectable element for qualitative analysis: boron to uranium
- Image display: 640 x 480 pixels

Electron Probe Micro-Analyzer (EPMA): JEOL JXA-8900 Superprobe

Electron probe microanalysis (EPMA) is an analytical technique that will be used to quantitatively determine chemical compositions of the individual solid phases present in the specimens with spatial resolutions of several micrometers. The EPMA is the decisive instrument to quantify isomorphic substitution in the fuel phases and will be used to analyze precipitations, zonings, and new phase formations with highest spatial resolution and accuracy possible. In general the EPMA bombards specimen with a beam of accelerated electrons which are focused on the surface of a specimen using a series of electromagnetic lenses, and these energetic electrons produce characteristic X-rays within a small volume (typically between one and nine cubic microns) of the specimen. The detection limits differ for each element and are affected by the overall composition of a sample and the analytical conditions. For most elements, the detection limits for the wavelength dispersive spectrometers (WDS) is between 50 and 500 parts per million (ppm). As a result, EPMA is well-suited to the analysis of heterogeneous specimens. The JXA-8900 EPMA is equipped with backscattered electron, secondary electron, and cathodoluminescence detectors capable of producing "real time" images, or automated images in tandem with X-ray mapping to further characterize the area of interest. The EPMA will be upgraded by a third-party software (John J. Donovan, Pres. Probe Software) to allow the analysis of all actinide metals.

The features of the JEOL JXA-8900 Superprobe Micro-Analyzer:

- Qualitative phase analysis to accuracies of ± 0.1 wt.-% per element
- Detectable element range: beryllium to uranium (0.087 to 9.3 nm)
- Pu and minor actinides can be added through third-party software (e.g. Donavan, UO)
- Detection limits: 50-500 ppm.
- Spatial resolution: one-nine cube-micrometer (beam interaction volume)
- Spectrometer: 4 WDS (wavelength dispersive), 1 EDS (energy dispersive)
- Detectors: SEI (secondary electrons), BEI (backscattered electrons), Cathodoluminescence
- Image magnification: x40 to 300,000
- Accelerating voltage: 0.2 to 40kV (100V steps)
- Probe current range: 10^{-12} to 10^{-5}A
- Probe current stability: 1 x 10^{-3}/h
- Standards: geological oxide standards from beryllium to uranium
- Actinide standards: to be prepared and added

Analytical Transmission Electron Microscopy (TEM): FEI Tecnai G^2 F30 S-Twin

The Transmission Electron Microscopy (TEM) User Facility at the Harry Reid Center (HRC) provides researchers at UNLV with the ability to characterize ceramics, metals, polymers, and biological materials at atomic-scale resolution (point-to-point resolution = 2 Ångström = 0.2 nm). The Tecnai G^2 F30 Super-Twin TEM is a perfect high-end analytical laboratory tool with excellent and versatile capabilities for high-resolution imaging as well as extremely good analytical performance. In this context, this analytical TEM will be used to perform nano-probing on prospective contaminant-metal bearing host to spatial resolution of several nanometers. We will determine chemical gradients within fuel phases, in precipitation layers and in new phase formations by energy-dispersive X-ray spectroscopy (EDX). Using the TEM as electron probe nano-analyzer will provide us with immensely important data to assess the behavior of the actinide metals in fresh fuel with unmatched spatial resolution. The Tecnai G^2 F30 S-Twin TEM at the HRC possesses a 300 kV field emission gun (Schottky mode) to provide the highest possible resolution and contrast and is the only analytical

research equipment at UNLV to truly suit the purpose of nanotechnology, surface technology, metallurgy, materials science, mineralogy, and radiochemistry and to characterize structural features (e.g., dislocations, defects, and grain boundaries and interfaces) at magnifications of 1,000,000 times in standard TEM mode and 1, 350,000 times in scanning TEM mode. In addition to high-quality imaging at atomic resolution, the Tecnai F30 will serve as a nano-probe, allowing qualitative chemical analysis with spatial resolution of 10 nm using energy dispersive X-ray spectroscopy (EDX) and parallel energy loss spectroscopy (PEELS). Energy-filtered electron microscopy (EFTEM) and PEELS are possible through the installation of a Gatan GIF 2000 CCD camera.

The features of the Tecnai G^2 F30 S-Twin TEM:

- 300 kV Schottky Field Emission Gun
- Point resolution: 0.20 nm
- Line resolution: 0.10 nm
- Information limit: 0.14 nm
- HR STEM resolution: 0.17 nm
- EFTEM magnification: 50 - 1.35 million
- Detectors: CCD camera, Energy filter
- Gatan Imaging Filter: HAADF, PEELS
- Qualitative analytical function: EDAX EDX system
- Spatial analytical resolution: 10 nm

X-Ray Fluorescence (XRF), PANalytical Axios Sequential Wavelength-dispersive X-ray Fluorescence Spectrometer

X-ray fluorescence (XRF) spectrometry is based on the emission of characteristic secondary or fluorescent X-rays from a material that has been excited by bombarding with high-energy X-rays and is commonly used for elemental chemical analysis particularly in the investigation of geological materials, metals, glass, and ceramics. The PANalytical Axios is an advanced sequential wavelength-dispersive X-ray fluorescence spectrometer and is perfectly suited to quantitatively perform major and trace elemental analysis, especially for silicates-based rocks and minerals. The sequential XRF spectrometer enables any number and combination of elements from beryllium to uranium and beyond to be measured one after another. The software Pro-Trace enables users to perform accurate and reliable trace element analysis on a broad range of sample types and pushing the limits of quantification down to the theoretical detection limits at sub-ppm level. Therefore, sample preparation is essential for the acquisition of quality data, and we will fuse the powdered samples into beads, which is widely seen as an excellent way of overcoming problems of particle size variation as well as surface and mineralogy effects. Hereby, the powdered sample is mixed with a flux, heated in a crucible to between 900-1300 °C, and cast in a dish to produce a homogeneous glass-like bead.

The features of the PANalytical Axios X-ray Fluorescence Spectrometer:

- Quantitative elemental analysis to accuracies of ±0.1 wt.-% using standards
- Standard-less semi-qualitative analysis to accuracies of ±1 wt.-%
- Quantitative elemental analysis of mayor and trace elements from Be to U and beyond
- Analysis of trace elements with sub-ppm detection limits.
- Analysis of up to 168 samples overnight