RESEARCH INSTRUMENTATION STANDARD PROCEDURE-5 (RISP-5) FOR RADIOACTIVE SAMPLES AND EXPERIMENTS USING X-RAY SCATTERING AT NUCS DODGEN

REVISION 1.0, 3/13/2024

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1 GENERAL

This standard Operating Procedure should be used for the preparation, transportation, and analysis of samples containing radioactive material in the Principal Investigator's Research Lab and Dodgen 114 using X-ray scattering (SAXS).

All researchers and Principal Investigators must comply with the Nuclear Science Center's (NSC) Accident Prevention Program and the Laboratory Safety Manual available on the Nuclear Science Center user site or in the NUCS Dodgen Facility (https://nsc.wsu.edu/safety/). Contact the Nuclear & Chemical Science (NUCS) Core Facility staff for access to the NSC user site.

The following numbered items are basic requirements for the analysis of radioactive samples with the Xenocs Xeuss 3.0:

- Dodgen 114 is a designated radioactive material lab. All users must survey their hands and feet with a GM meter, prior to leaving the room.
- Analysis of radioactive samples at the NUCS Core Facility must be approved by a student's Principal Investigator and the NUCS Core Facility staff prior to analyzing radioactive samples. To request approval, a student must fill out the radioactive sample analysis request form found on the NUCS Core Facility website: https://nsc.wsu.edu/radioactive-sample-analysis/
- PIs are responsible for radioactive material sample preparation, transportation, and safety in their respective labs prior to entry into the NUCS labs.
- PPE are the responsibility of the PI. Extra PPE will be made available in the NUCS Dodgen Facility work areas.
- Waste disposal is the responsibility of the PI unless otherwise indicated.

A copy of this procedure is available at the Xenocs Xeuss 3.0 X-ray spectrometer.

2 SPILLS, CONTAMINATION, & EXPOSURE

2.1 Accidental Exposure

For any accidental radioactive exposure in the NUCS Dodgen Facility, immediate notification of the NUCS Dodgen Lab Supervisor (509-335-0936) or the NSC Emergency Line (509-335-0004) is required.

In all cases immediately contact WSU Radiation Safety Office at (509) 335-7183 and the Principal Investigator.

In case of **eye contact**, flush eyes with copious amounts of water at an emergency eyewash station for at least 15 minutes and seek medical attention.

In case of **skin contact**, flush skin with copious amounts of water for 15 minutes and seek medical attention. For exposure over a large portion of the body, remove clothing and shoes and rinse thoroughly in an emergency shower for at least 15 minutes. Seek medical attention.

In case of **inhalation**, move person to fresh air and immediately seek medical attention.

In case of **ingestion**, immediately seek medical attention and follow instructions on SDS. Do not induce vomiting.

2.2 Accidental Release

For any radioactive spills in the NUCS Dodgen Facility, the NUCS Core Facility Supervisor (509-335-0936) or the NSC Emergency Line (509-335-0004) must be immediately notified. A spill kit for radioactive samples is available in area designated for radioactive samples in the NUCS Dodgen 114.

Small Spill: If a small amount of a radioactive sample is spilled **within the radioactive sample prep area (RSPA) only** (it can be cleaned up in 10 minutes) and you have been appropriately trained to clean it up, you may do so. Trained personnel should wear at the minimum dual layer of latex gloves, chemical safety goggles, and a fully-buttoned lab coat.

Small spill outside of the RSPA requires immediate contact of the NSC Emergency Line (509-335-0004) and the Principal Investigator. Cordon off the area and follow all instructions from Radiation Safety Officer and the Principal Investigator. Do not leave the spill unattended.

Additional PPE such as respirators may be necessary depending upon material and concentration released. (Note: You must be medically cleared, fit tested and enrolled in WSU's respiratory protection program to wear a respirator). If it is necessary to use a respirator, and personnel are not cleared to wear a respirator, and not trained to appropriately clean up the spill, the employee should immediately evacuate, secure the area, and call 911 to contact EH&S and the Radiation Safety Office.

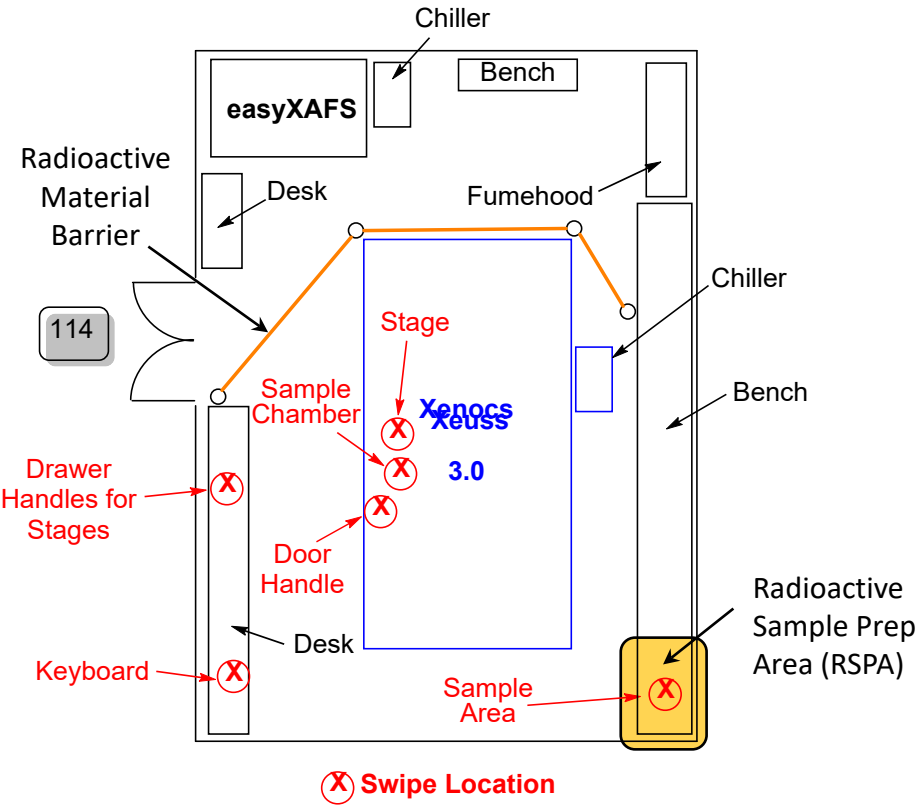
Absorb with an inert dry material, or if the released substance is a solid, use appropriate tools to collect it, and place in an appropriate waste disposal container (resealable bag, etc.) and dispose of as hazardous waste (see above WASTE DISPOSAL PROCEDURES).

As with all accidents, report any exposure as soon as possible to your Principal Investigator or Supervisor. Additional health and safety information can be obtained by referring to the SDS or by calling the EH&S Office (335-3041). All accidents shall be reported in accordance with WSU policies and procedures.

3 RECORDS

All swipe records shall be kept in accordance with Radiation Safety Office policies and procedures and shall be readily accessible for inspection by the NUCS Dodgen Facility Supervisor, the Nuclear Science Center (authorized user for the NUCS Core Facility) or the Radiation Safety Office.

Research groups undertaking the analysis of radioactive samples are required to undertake post use swipes of the locations indicated on the map below.



The Nuclear Science Center will undertake weekly swipes of the NUCS Dodgen Facility at the locations indicated in the areas designated on the map. The records will be kept according to the procedures of the Nuclear Science Center and are available for inspection when requested.

The swipes and surveys undertaken by the NSC are not substitutes to the swipes to be taken by the research groups that analyze radioactive samples.

4 SAMPLE PREPARATION

X-ray scattering samples containing radioactive material must be prepared within an appropriate radiological material lab within the radiological buffer area (RBA) in the Principal Investigator's Research Lab.

4.1 Preparation of radioactive samples (U-238, U-nat, Th-232 or isotopes included in the AU's license) will be done exclusively in a radiological material lab of the Principal Investigator's research lab.

4.2 The NUCS Core Facility associate director needs to be informed via email (zachariah.heiden@wsu.edu) of any previously unmeasured isotopes prior to measurement.

4.3 Samples can be prepared in different ways, whether they be solid, liquid, thin film, etc.

4.4 For capillaries, samples will be loaded into glass/quartz capillaries and sealed with epoxy, wax, or flame sealed. Samples may either be packed solids or liquid.

4.5 Solid samples will be placed into a Kapton or mylar bag.

4.6 Samples may additionally be placed in Kapton/mylar bags or approved contained sample holders to account for air sensitivity and provide added containment. Such containment will be considered superfluous. Additional layers of containment are required for the analysis of samples containing isotopes other than U-238, U-nat, and Th-232.

4.7 The outer layer of the capillary or Kapton/mylar (or other containment as described in b) will be swipe surveyed with a Geiger meter and an alpha meter or an alpha/beta counter or LSC to verify that the outside is free of removable contamination.

4.8 Sample will be placed in a secondary clean plastic bag marked with a radioactive label.

4.9 Radiological work specific PPE will then be removed following previously established procedures and now the sample may be removed to the RBA.

4.10 Sample identity will be recorded, researcher, analytical method, location of analysis, and check out time in the sample logbook stored in the RBA of the PI's Research Lab.

4.11 Prior to leaving the RBA of the PI's research Lab, sample will be placed in a plastic container for transport and tertiary containment.

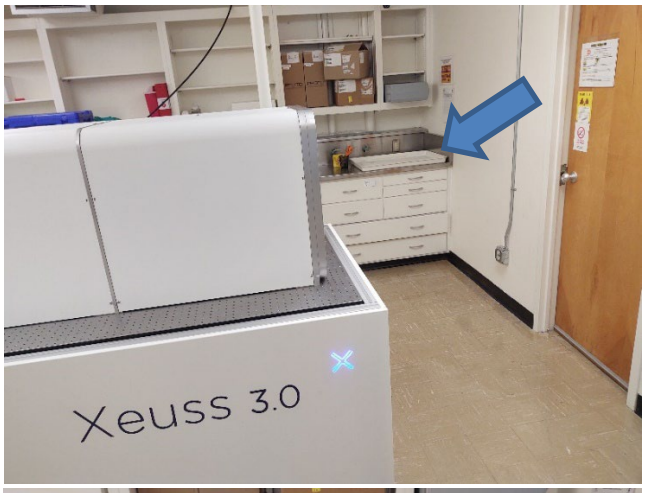
4.12 If the radioactive material will reside in Dodgen 114 or the Dodgen Research Facility for more than 24 hours, a radioactive material transfer (https://rso.wsu.edu/transfers/) will need to be filled out when the material enters and leaves the Dodgen Research Facility.

5 NUCS DODGEN FACILITY AND SAMPLE ANALYSIS

Being that the Xenocs Xeuss 3.0 X-ray scattering spectrometer is located in a designated radioactive material laboratory, it is approved for the analysis of radioactive X-ray scattering samples.

5.1 Sign up for instrument time on the Xenocs Xeuss 3.0 X-ray spectrometer using the RADIOACTIVE SAMPLES option on iLab. The comments section of the reservation must indicate the radioactive isotopes present in the sample(s) to be analyzed. This reservation must be made at least one hour in advance and the instrument time must run until the post use swipe/surveys have been completed and the area has been deemed usable for the next user.

5.2 At the instrument, cover the empty tray, located on the south bench behind the Xenocs Xeuss 3.0 X-ray spectrometer, with lab paper (provided) and adhere 1-2 of the radioactive materials stickers (provided) to the lab paper on the tray. If there are no lab paper (in a drawer below the tray, shown in picture) or radioactive material stickers available next to the X-ray spectrometer please inform the staff of the NUCS Core Facility.



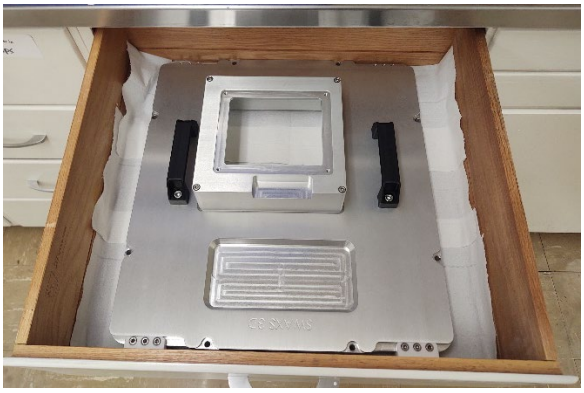
5.3 Place the container holding the radioactive sample in the designated tray located in the designated radioactive sample area.

5.4 Samples are to only be placed in the designated area (in the tray) for radioactive samples.

5.5 Since Dodgen 114 is a designated radioactive materials lab, no further signage is needed, but it is highly recommended to place barriers around the SAXS to prevent other users from entering the area during the analysis of radioactive samples.

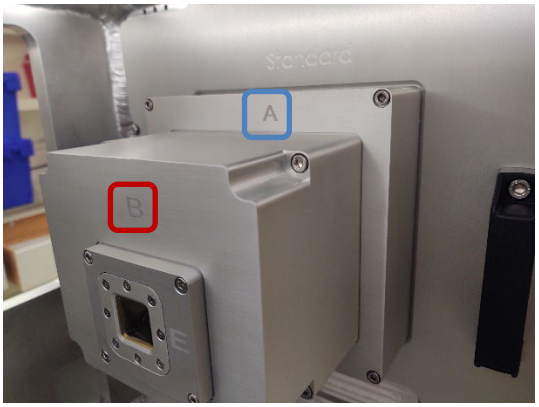
5.6 Radioactive samples are not allowed to be analyzed under vacuum. The default instrument configuration has the atmospheric caps installed, however in the event that they are not in place, complete steps 5.7 to 5.11 to install them before instrument use. If the atmospheric caps are already installed, skip to step 5.13.

5.7 Atmospheric caps are found in the top two drawers (highlighted in red) directly behind the sample chamber on the south bench. If the imaging plate (labeled Front Atmo) is not already installed in the instrument, it can be found in a drawer directly below the drawers containing the atmospheric caps.



5.8 Install the main atmospheric cap (labeled piece A) with the screws (labeled ATMO CAP SCREWS) found in the Atmosphere Caps drawer. Allen wrenches are found in a drawer labeled allen wrenches and screws near the computer. If the screws do not go in easily, they are not going in correctly and the threads are being cross threaded. The screws only need to be moderately snug. Do not overtighten the screws, the atmospheric caps will pull tight under vacuum.

5.9 Connect piece B (highlighted in red) to piece A (highlighted in blue) using the screws found in the atmospheric caps drawer in piece B. Piece E or C, containing a Kapton window, can be attached to piece B before attaching piece B to piece A. Piece C is the preferred piece for use with the standard (#1) or capillary (#4) stage. If piece E is used, make sure the E is upside down or the atmospheric cap will block the X-ray beam.



5.10 Connect the snorkel with a Kapton window (highlighted in red) to the imaging plate (labeled Front Atmo). There are two size options, pick the snorkel that sits closest to your sample stage but does not interfere with it. The longer snorkel is preferred for the standard (#1) or capillary (#4) stage. Only tighten by hand to avoid overtightening.

5.11 Correct installation of the atmospheric caps for the analysis of capillary samples is shown below.

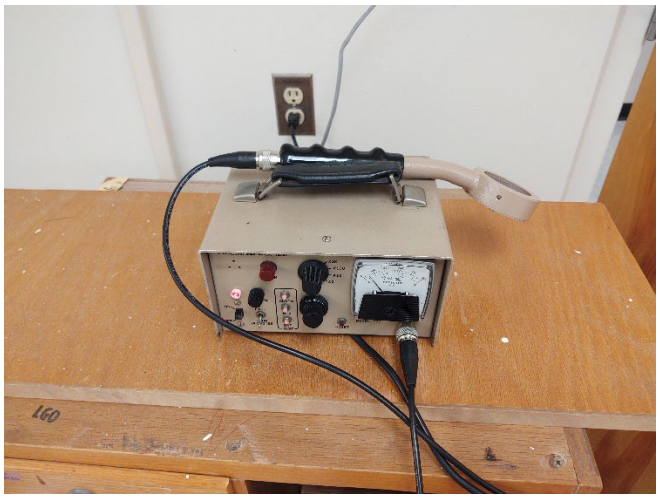
5.12 Start to evacuate the system with the door open. If the instrument struggles to pump down below 800 mbar (takes more than 5 minutes to get below 800 mbar), then there is a leak in the seals of the atmospheric caps and the screws need to be tightened. The instrument may already be pumped down if the atmospheric caps are already installed.

5.13 Record the X-ray scattering data following standard practices. Note: the instrument door must be closed and latched to open an X-ray tube shutter.

6 POST MEASUREMENT

6.1 After the measurements are complete, return the X-ray scattering sample to the plastic bag and the carrier.

6.2 Survey your hands and feet with the Geiger counter located near the door to Dodgen 114 prior to exiting the room.



6.3 Return the sample to the radiological material lab in the Principal Investigator's Research Lab and log the sample as returned on the log sheet.

6.4 Don the appropriate radiological work PPE and return the X-ray scattering sample to the appropriate radioactive material control area.

6.5 Swipe the sample for contamination with Kim wipe and count the Kim wipe with a Geiger meter and an alpha meter or an alpha/beta counter or LSC to verify that the outside is free of removable radioactive contamination.

6.6 Dispose of radioactive waste according to radioactive material handling procedures for the respective Principal Investigator's laboratory.

6.7 If the sample(s) is/are free from contamination, then return to the Dodgen 114, deface the radioactive material stickers, dispose of the lab paper covering the radioactive material tray (leaving an empty tray) indicating the presence of radioactive material in the area has been removed and the area is clean for the next user.

6.8 Remove barrier to indicate that the instrument is available for the next user.

6.9 If the sample(s) is/are found to be contaminated, contact the Principal Investigator, and the NUCS Core Facility Supervisor (NSC Emergency Line if after hours). In the rare event that the plastic bag, X-ray scattering sample, and radioactive material is released outside of a radioactive material control area call the NSC Emergency Line immediately. Guard the spill and do not leave the area unless instructed otherwise.

7 TRAINING

Training is the responsibility of the PI. Training documentation will be provided to the NUCS Core Facility prior to experimentation with radioactive materials. The following PIs are trainers for WSU students performing radioactive X-ray scattering experiments:

Liane Moreau, Troy 224 & Dodgen 217, 509-335-6073, liane.moreau@wsu.edu

Zach Heiden, Dodgen 250C, NUCS Core Facility Associate Director, 509-335-0936, zachariah.heiden@wsu.edu

Nuclear Science Center Emergency Line: 509-335-0004