

NUCLEAR AND CHEMICAL SCIENCE CORE FACILITY  
RESEARCH INSTRUMENTATION STANDARD PROCEDURES

**RESEARCH INSTRUMENTATION STANDARD PROCEDURE-3 (RISP-3) FOR  
RADIOACTIVE SAMPLES AND EXPERIMENTS USING  
POWDER X-RAY DIFFRACTION AT NUCS FULMER**

**REVISION 0.1, 5/17/2022**

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## 1 GENERAL

Standard Operating Procedure for the preparation, transportation and analysis of X-ray diffraction samples containing radioactive material in the Principal Investigator's Research Lab and Fulmer B3.

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 Fulmer Facility. Contact Nuclear & Chemical Science (NUCS) Core Facility staff for access to the NSC user site.

The following numbered items are basic requirements for radioactive samples in the X-ray diffractometers.

- 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 and safety in their respective labs.
- Radioactive X-ray diffraction samples are limited to natural or depleted uranium or natural thorium.
- PPE are the responsibility of the PI. Extra PPE will be made available in the NUCS Fulmer Facility work areas.
- Waste disposal is the responsibility of the PI unless otherwise indicated.

A copy of this procedure is available at the Bruker D2 Phaser powder X-ray diffractometer.

## 2 SPILLS, CONTAMINATION, & EXPOSURE

### 2.1 Accidental Exposure

**For any radioactive spills in the NUCS Fulmer Facility, immediate notification of the NUCS Fulmer Lab Supervisor or the NSC Emergency Line 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 Fulmer Facility, the NUCS Core Facility Supervisor or the NSC Emergency Line must be immediately notified. A spill kit for radioactive samples is available in area designated for radioactive samples in the NUCS Fulmer lab.**

**Small Spill:** If a small amount of a radioactive sample is spilled **within the RBA 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 nitrile gloves, chemical safety goggles, and a fully-buttoned lab coat.

**Small spill outside of the RBA requires immediate contact of 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.

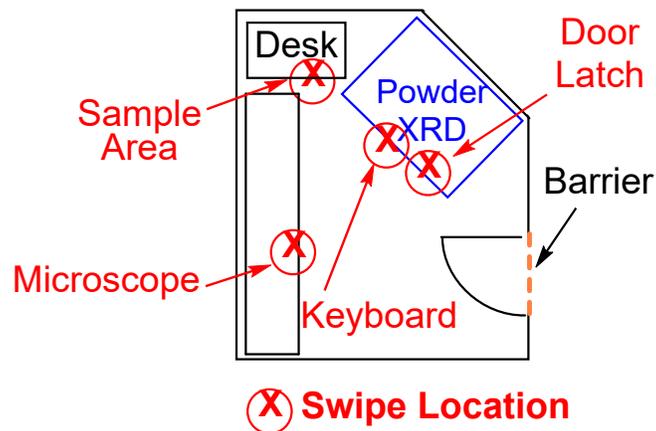
Absorb with an inert dry material, or if the released substance is a solid, use appropriate tools to collect it, and place in an appropriate waste disposal container (resealable bag, etc.) and dispose of as hazardous waste (see above WASTE DISPOSAL PROCEDURES).

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 Fulmer 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 Fulmer 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.**

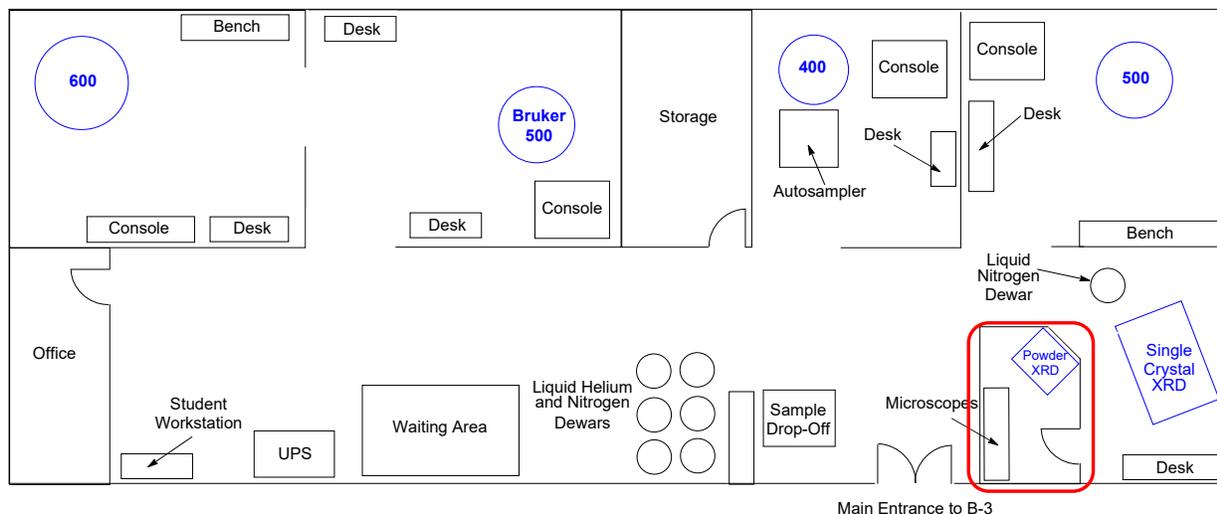
## 4 SAMPLE PREPARATION

X-ray diffraction samples containing natural or depleted uranium or natural thorium must be prepared within an appropriate radiological control area (RCA) within the radiological buffer area (RBA) in the Principal Investigator's Research Lab. Samples involving other radioactive nuclides need prior approval from the WSU NUCS Core Facility and the Radiation Safety Office.

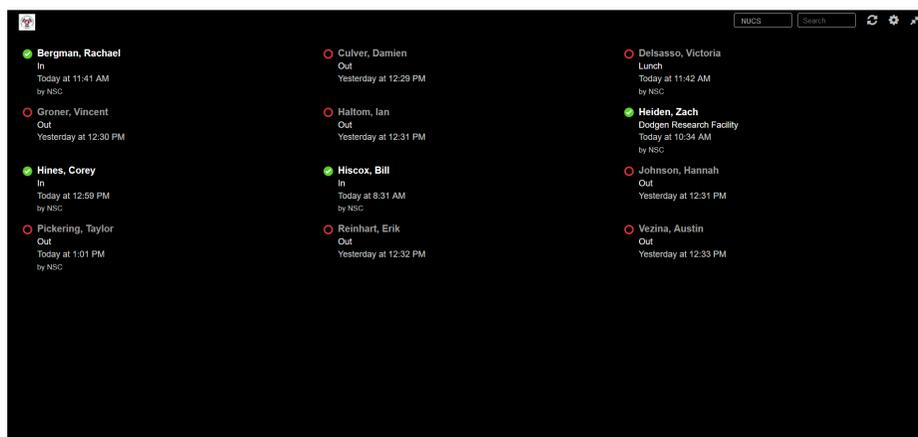
- 4.1 Preparation of radioactive samples (U-238, U-natural, or Th-232) will be done exclusively in an RCA of the Principal Investigator's research lab.
- 4.2 Samples will be prepared differently if in solution or in solid phase.
  - 4.2.1 Material suspended in solution will be drop-cast onto a glass slide, low-background plastic, single crystal silicon or other approved holder, and the solvent allowed to evaporate to dryness. Repeat until desired thickness of sample is reached.
  - 4.2.2 Dried powdered sample will be loaded onto an approved sample holder and sealed with a layer of Kapton tape to prevent dispersion of powders.
  - 4.2.3 Samples may additionally be placed in mylar bags or approved contained sample holders to account for air sensitivity and provide added containment. Such containment will be considered superfluous.
- 4.3 The outer layer of Kapton (or other containment as described in 4.2.3) 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.
- 4.4 Sample will be placed in a secondary clean plastic bag marked with a radioactive label.
- 4.5 Radiological work specific PPE will then be removed following previously established procedures and now the sample may be removed to the RBA.
- 4.6 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.
- 4.7 Prior to leaving the RBA of the PI's research Lab, sample will be placed in a plastic container for transport and tertiary containment.

## 5 NUCS FULMER FACILITY AND SAMPLE ANALYSIS

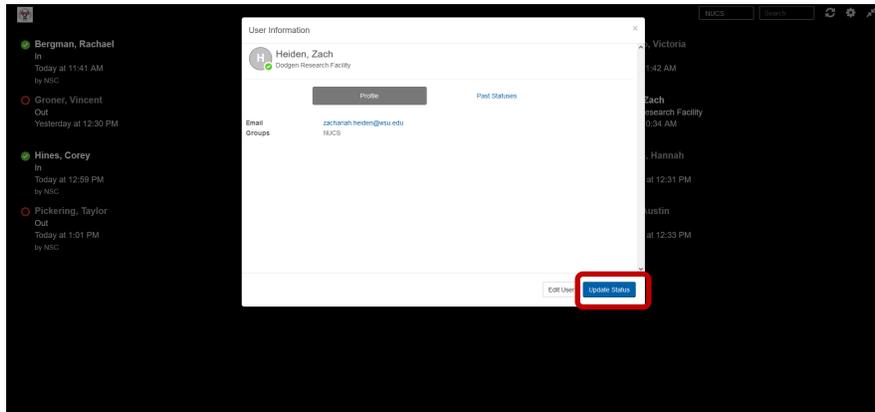
The Bruker D2 Phaser powder X-ray diffractometer (highlighted below in red) is the only instrument approved for the analysis of radioactive powder X-ray diffraction samples.



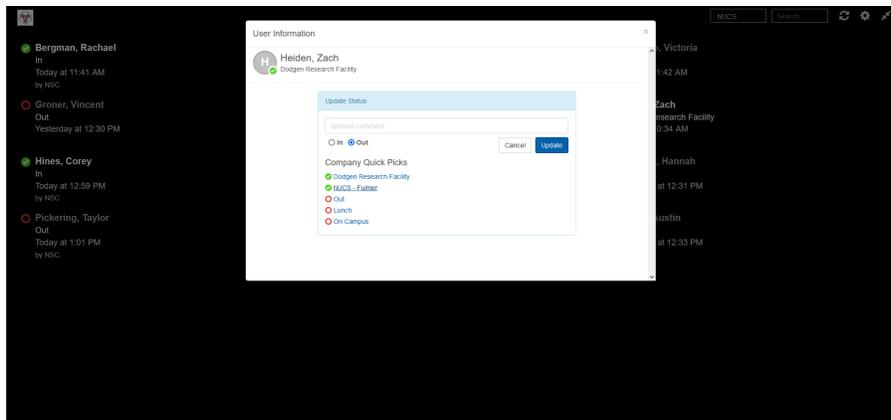
- 5.1 Sign up for instrument time on the Bruker D2 Phaser powder X-ray diffractometer using the RADIOACTIVE SAMPLES option on iLab. This reservation must be made at least one hour in advance and the instrument time must run until the post use swipe/surveys have been completed and the area has been deemed usable for the next user.
- 5.2 Upon entering the NUCS Fulmer Facility, stop by the computer that is located on the Sample Drop-Off table. This computer will house the Simple In/Out software that will be used to check/out radioactive samples. The screen should show the following image.



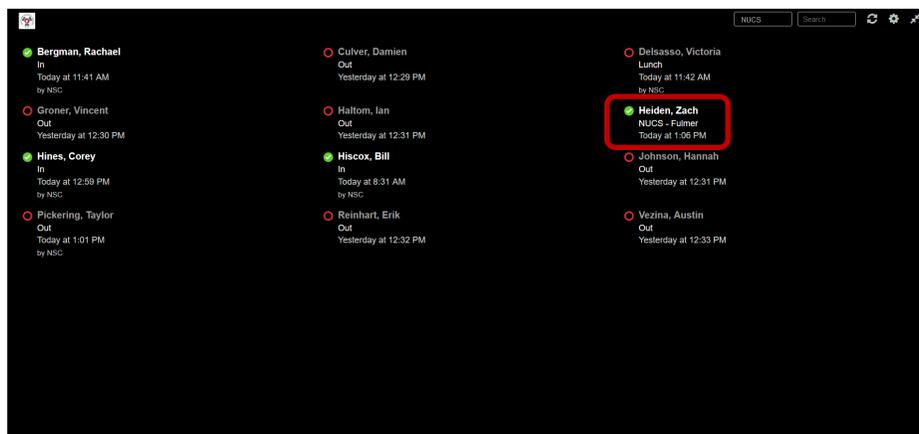
- 5.3 If the screen is black or requires a login, please restart the computer and it will autologin and open a browser with Simple In/Out.
- 5.4 To login your sample click on your name. If your name is not present, please inform the NUCS Core Facility Supervisor to be added.
- 5.5 A window with your name should appear. Click on the Update Status button (highlighted in red).



- 5.6 A list of options will be present, click on NUCS Fulmer to indicate that radioactive samples are checked into the NUCS Fulmer Facility.



- 5.7 Your name should have a green checkmark next to it, and the status should read: NUCS – Fulmer, indicating that you have checked in.



- 5.8 At the instrument, cover the empty tray, located on the desk next to the powder X-ray diffractometer, 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 or radioactive material stickers available next to the X-ray diffractometer, the lab paper and radioactive material stickers found in the radioactive materials area for NMR spectroscopy or the single crystal X-ray diffractometer can be used. Please inform the staff of the NUCS Core Facility if more lab paper and/or radioactive material stickers are needed.



- 5.9 Place the container holding the radioactive sample in the designated tray located in the designated radioactive sample area.
- 5.10 Samples are to only be placed in the designated area (in the tray) for radioactive samples.

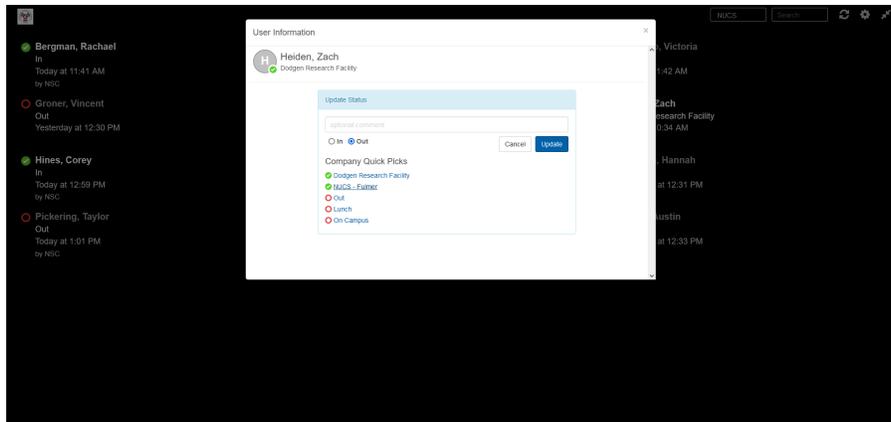
- 5.11 Put up the appropriate signage and/or chain at the entrance to the powder X-ray diffractometer indicating the presence of radioactive material is present in the area.



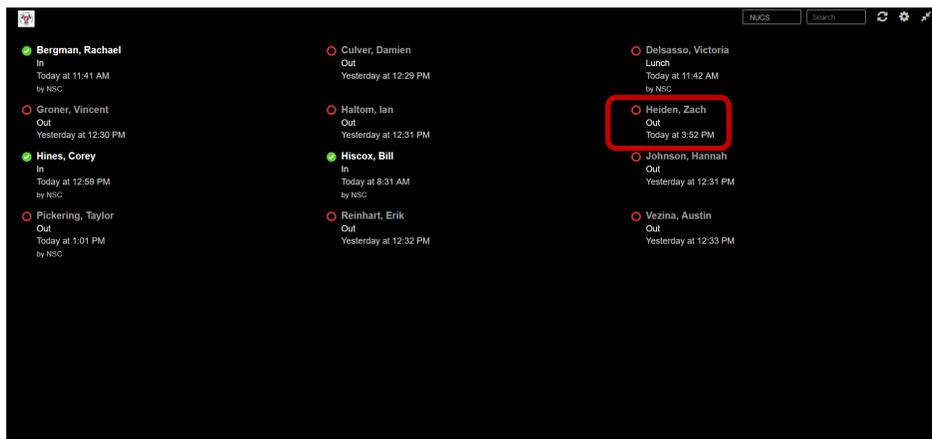
- 5.12 Record the X-ray diffraction data following standard practices.

## 6 POST MEASUREMENT

- 6.1 After the measurements are complete, return the X-ray diffraction sample to the plastic bag and the carrier.
- 6.2 Prior to leaving the NUCS Fulmer Facility, stop by the computer that is located on the Sample Drop-Off table to check out your radioactive sample.
- 6.3 To logout your sample click on your name.
- 6.4 A window with your name should appear. Click on the Update Status button.
- 6.5 A list of options will be present, click on Out to indicate that radioactive samples have been checked out of the NUCS Fulmer Facility.



- 6.6 Your name should have a red circle next to it, and the status should read: Out, indicating that you have checked the sample(s) out.



- 6.7 After checking out the sample, return the sample to the RBA in the Principal Investigator's Research Lab and log the sample as returned on the log sheet.
- 6.8 Don the appropriate radiological work PPE and return the X-ray diffraction sample to the appropriate RCA.
- 6.9 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.
- 6.10 If sample is powdered, first dispose of solids in radioactive solid waste, and then wash sample holder with appropriate solvents and dispose of in corresponding radioactive waste containers. If sample was drop cast, rinse with appropriate solvents and dispose of in corresponding radioactive waste containers.
- 6.11 If the sample(s) is/are free from contamination, then return to the NUCS Fulmer Facility, deface the radioactive material stickers, dispose of the lab paper covering the radioactive material tray (leaving an empty tray), and remove the signage/chain indicating the presence of radioactive material in the area has been removed and the area is clean for the next user.



- 6.12 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 diffraction sample, and radioactive material is released outside of an RCA call the NSC Emergency Line immediately. Guard the spill and do not leave the area unless instructed otherwise. A telephone is available at the Bruker D8 Venture Single Crystal X-ray Diffractometer, as there is no cell reception in the NUCS Fulmer Facility. To dial off campus, dial 7+1+area code+number.

## 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 diffraction experiments:

James Boncella, Fulmer 664

Liane Moreau, Troy 224

Zach Heiden, NMR Lab Director, 509-335-0936

Bill Hiscox, NMR Assistant Director, 509-335-8259

Nuclear Science Center Emergency Line: 509-335-0004