

NUCLEAR AND CHEMICAL SCIENCE CORE FACILITY
RESEARCH INSTRUMENTATION STANDARD PROCEDURES

**RESEARCH INSTRUMENTATION STANDARD PROCEDURE-1 (RISP-1) FOR
RADIOACTIVE SAMPLES AND EXPERIMENTS USING NMR AT NUCS FULMER**

REVISION 1.3, 5/17/2022

TABLE OF CONTENTS

	Page
1 General	3
2 Spills, Contamination, & Exposure	3
2.1 Upon Accidental Exposure:.....	3
2.2 Upon Accidental Release:.....	4
3 Records.....	5
4 Sample Preparation.....	6
5 NUCS Fulmer Facility and Sample Analysis	7
6 Backup Instrument for NMR Analysis of Radioactive Samples	10
7 Post Measurement	15
8 Training	17

1 GENERAL

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

All researchers and Principle 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 NMR Lab. 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 NMR machines.

- 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 NMR 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 work areas.
- Waste disposal is the responsibility of the PI unless otherwise indicated.

A copy of this procedure is available at the Varian 500 MHz NMR spectrometer.

2 SPILLS, CONTAMINATION, & EXPOSURE

2.1 Accidental Exposure

For any radioactive spills in the NUCS Fulmer lab, immediate notification of the NUCS Core Facility 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 NMR 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.

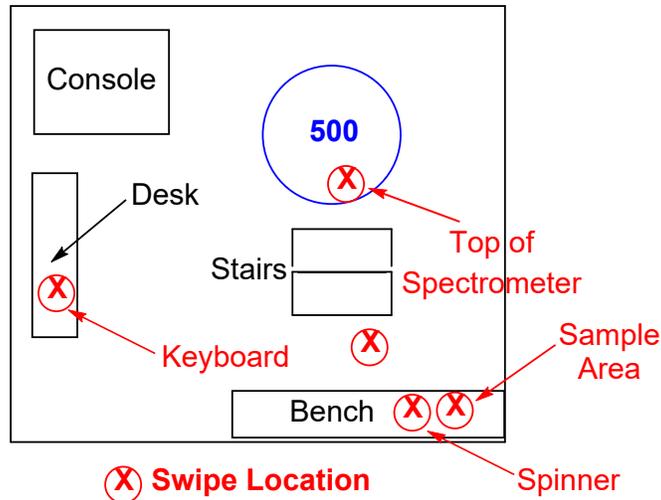
Absorb with an inert dry material, or if the released substance is a solid, use appropriate tools to collect it, and place in an appropriate waste disposal container (resealable bag, etc.) and dispose of as hazardous waste (see above WASTE DISPOSAL PROCEDURES).

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.

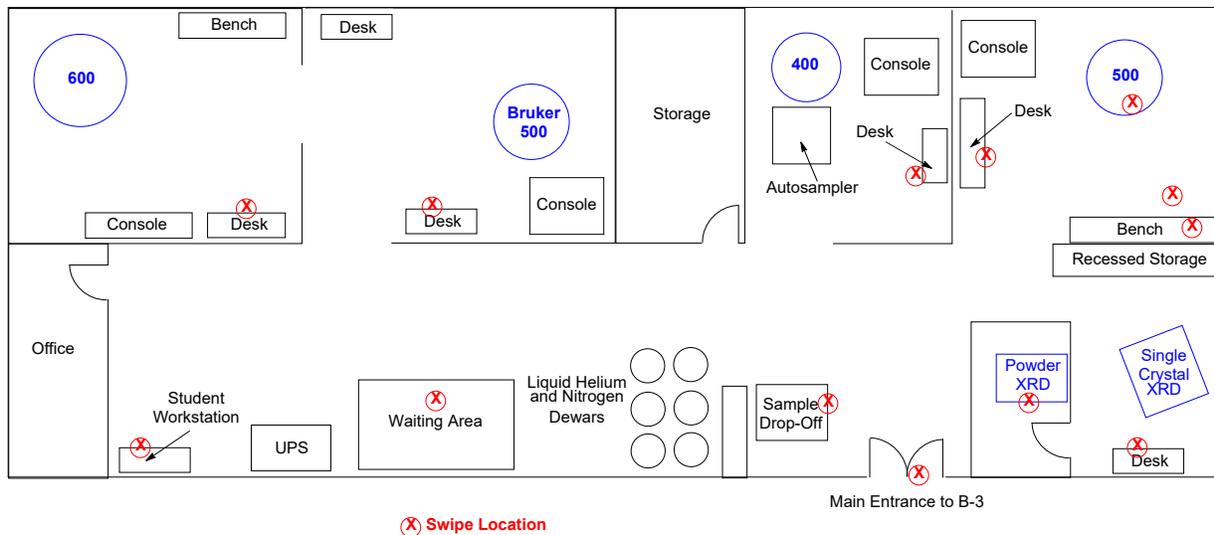
3 RECORDS

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 Core 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.

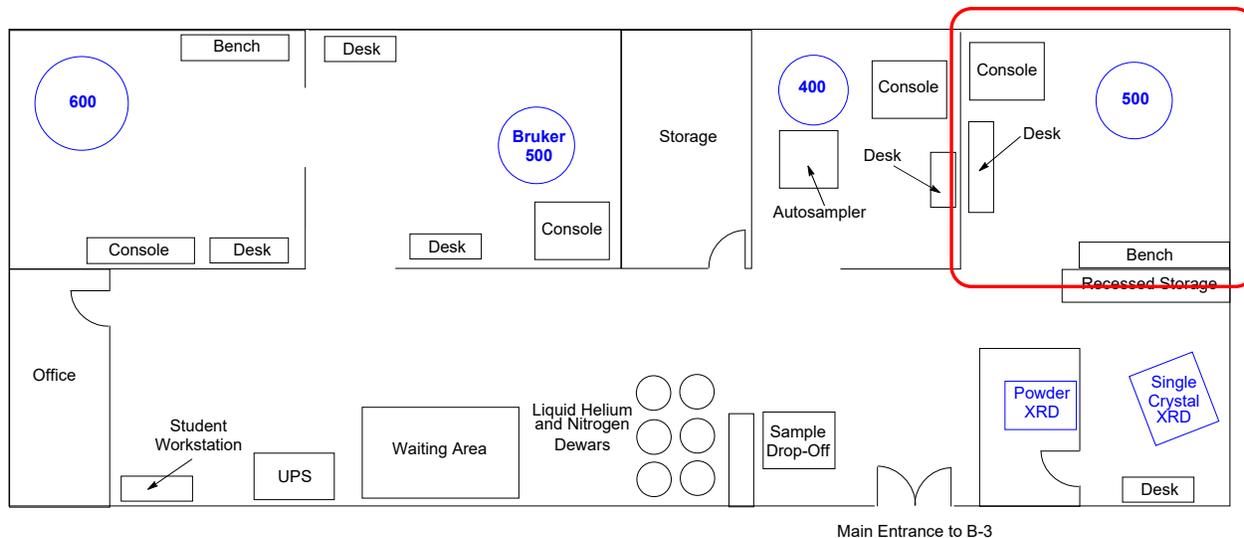
4 SAMPLE PREPARATION

NMR 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 NUCS Core Facility and the Radiation Safety Office.

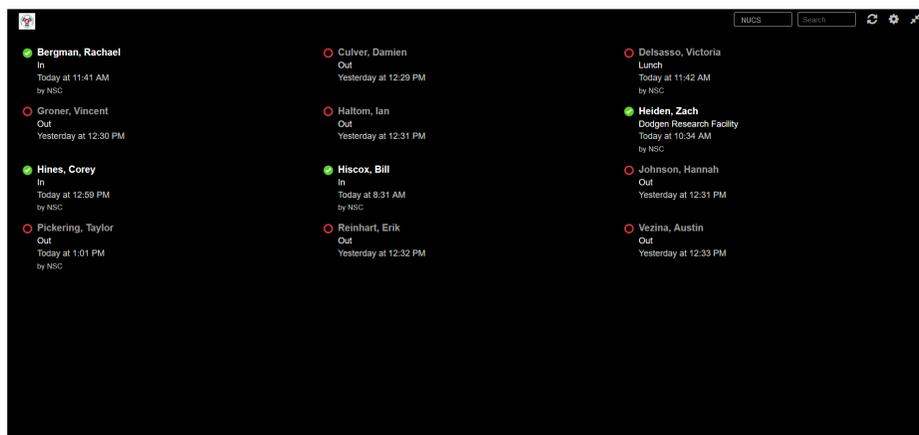
- 4.1. Prepare the solution NMR sample in a polytetrafluoroethylene (PTFE) NMR tube liner from Wilmad (Item #6005), seal with the associated PTFE plug, slip the liner into a 5 mm NMR tube, and seal the NMR tube with either a PTFE valve or a standard NMR cap wrapped with Teflon tape.
- 4.2. Remove the NMR tube from the RCA using standard techniques (refer to the relevant radiological work SOPs) and decontaminate the outside of the NMR tube with a Kim wipe wet with Radiac or another approved decontamination solution. Wipe the NMR tube clean and dry with a Kim wipe.
- 4.3. Swipe the NMR tube 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.
- 4.4. Seal the clean NMR tube within a clean plastic bag with a radioactive label on it (this label may be tape or a sticker with a trefoil symbol).
- 4.5. Remove your radiological work specific PPE following previously established procedures and now you may remove the sample from the buffer area ONLY.
- 4.6. Record the sample identity, researcher, analytical method, location of analysis and check out time in the sample logbook stored in the RBA of the Principal Investigator's Research Lab.
- 4.7. Prior to leaving the RBA of the Principal Investigator's Research Lab, place sample in a plastic carrier for transportation and tertiary containment.

5 NUCS FULMER FACILITY AND SAMPLE ANALYSIS

The Varian 500 MHz NMR spectrometer (highlighted below in red) is the only instrument approved for the analysis of radioactive samples.

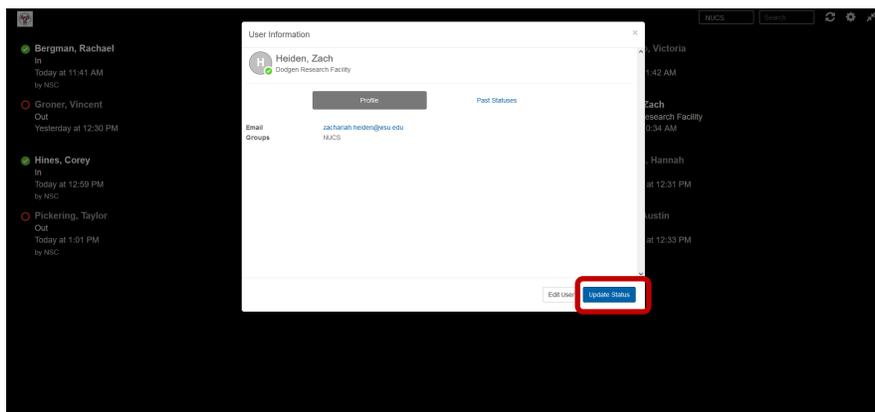


- 5.1. Sign up for instrument time on the 500 MHz Varian NMR spectrometer using the RADIOACTIVE SAMPLES option on iLab. This reservation must be made at least one hour in advance and the instrument time must run until the post use swipe/surveys have been completed and the area has been deemed usable 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.

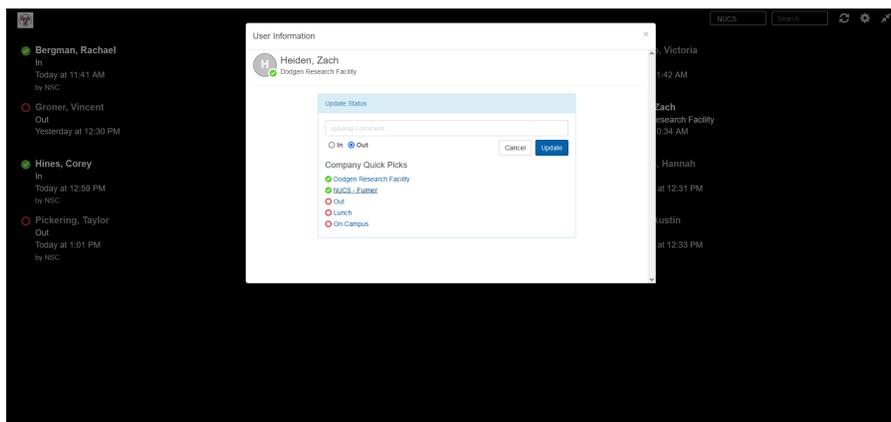


- 5.3. If the screen is black or requires a login, please restart the computer and it will autologin and open a browser with Simple In/Out.
- 5.4. To login your sample click on your name. If your name is not present, please inform the NUCS Core Facility Supervisor to be added.

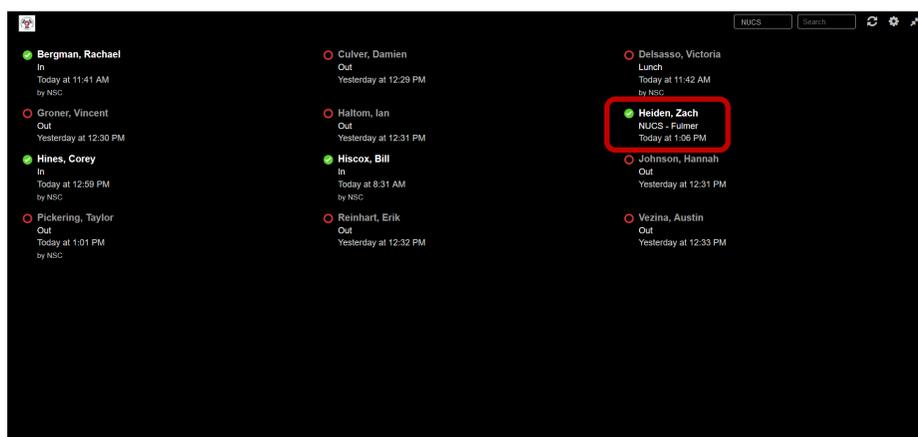
- 5.5. A window with your name should appear. Click on the Update Status button (highlighted in red).



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

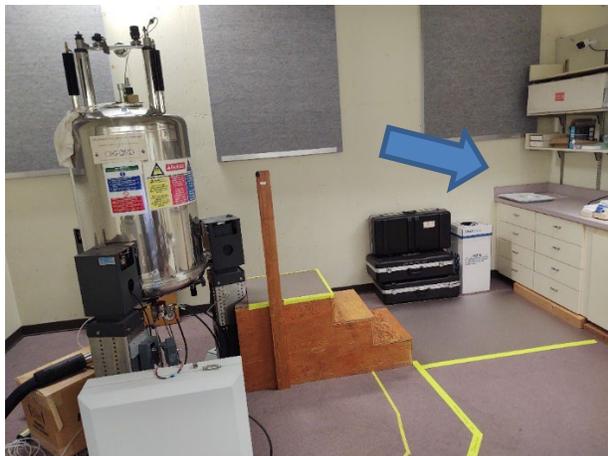


- 5.7. Your name should have a green checkmark next to it, and the status should read: NUCS – Fulmer, indicating that you have checked in.



- 5.8. At the instrument, cover the empty tray, located on the desk next to the NMR 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 or radioactive material stickers

available next to the NMR spectrometer, the lab paper and radioactive material stickers found in the radioactive materials area for the 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.



- 5.3 Place the container containing radioactive material on the sample preparation bench (in the designated tray located in the designated radioactive sample area) across from the magnet and to the right of the entryway (not the computer table).



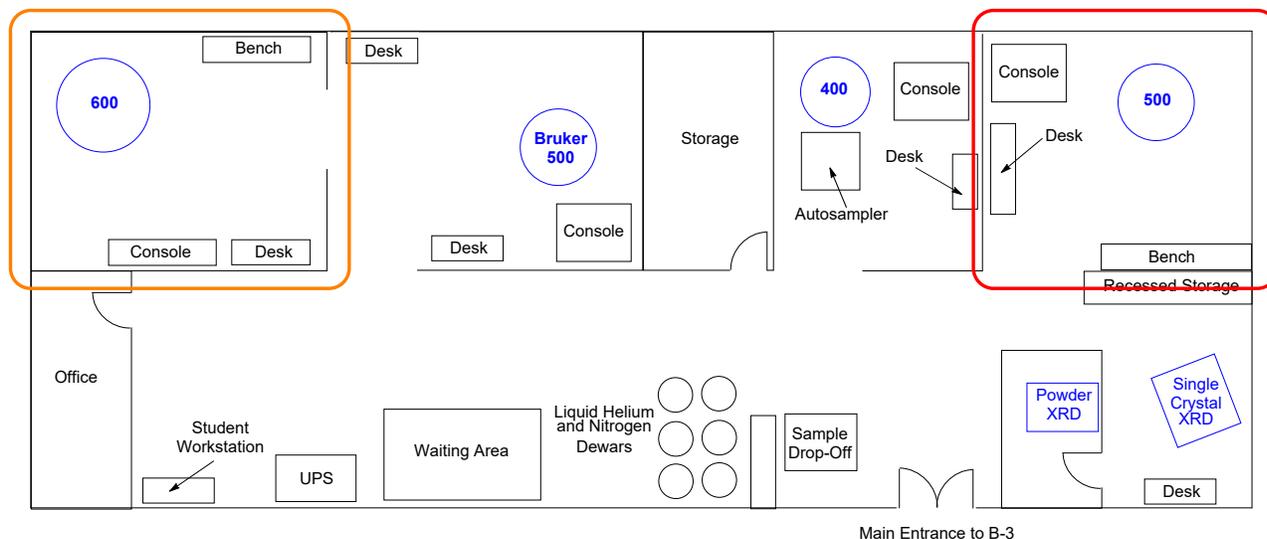
- 5.4 Samples are to be only placed in the designated area (in the tray) for radioactive samples.
- 5.5 Put up the appropriate signage and/or chain at the entrance to the room containing the instrument indicating the presence of radioactive material is present in the area.



5.6 Record the NMR spectra following standard practices using the spinner and depth gauge that is designated for radiological samples. Radiological NMR samples must not be spun on the instrument, spinning NMR samples is no longer necessary on modern NMR instruments due to gradient shimming. Radiological samples are not currently approved to be analyzed by solid-state NMR spectroscopy at WSU.

6 BACKUP INSTRUMENT FOR RADIOACTIVE SAMPLES

In an event that the Varian 500 MHz NMR spectrometer (highlighted below in red) is not available, the Varian 600 MHz NMR spectrometer (highlighted below in orange) is approved for the analysis of radioactive samples **only** when the Varian 500 MHz NMR spectrometer is not available due to maintenance/repair.



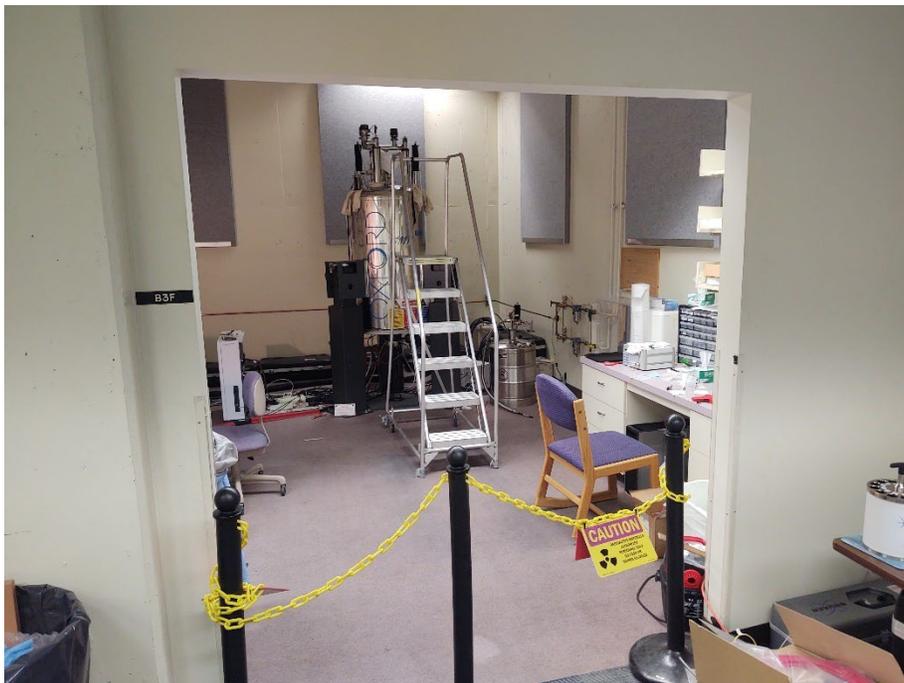
- 6.1 Sign up for instrument time on the 600 MHz Varian NMR spectrometer using the RADIOACTIVE SAMPLES option on iLab. This reservation must be made at least one hour in advance and the instrument time must run until the post use swipe/surveys have been completed and the area has been deemed usable for the next user.
- 6.2 At the instrument, cover the empty tray, located on the desk next to the NMR 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 or radioactive material stickers available next to the 600 MHz NMR spectrometer, the lab paper and radioactive material stickers found in the radioactive materials area for the X-ray diffractometer or the 500 MHz NMR spectrometer can be used. Please inform the staff of the NUCS Core Facility if more lab paper and/or radioactive material stickers are needed.



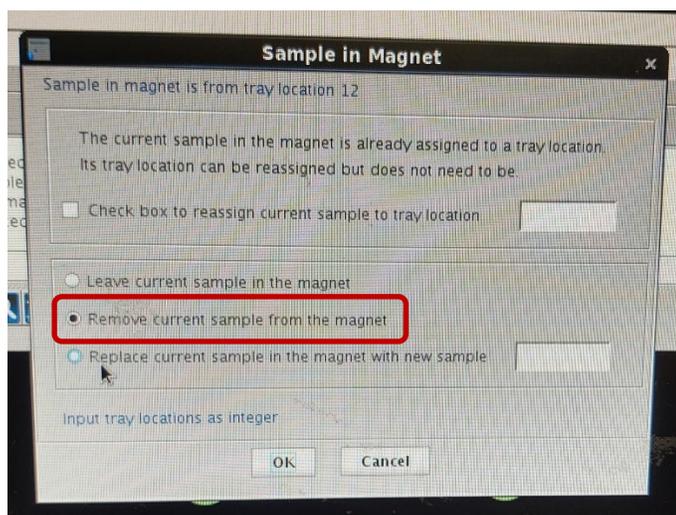
- 6.3 Place the container containing radioactive material on the sample preparation bench (in the designated tray located in the designated radioactive sample area) next to the magnet and to the right of the entryway (not the computer table).



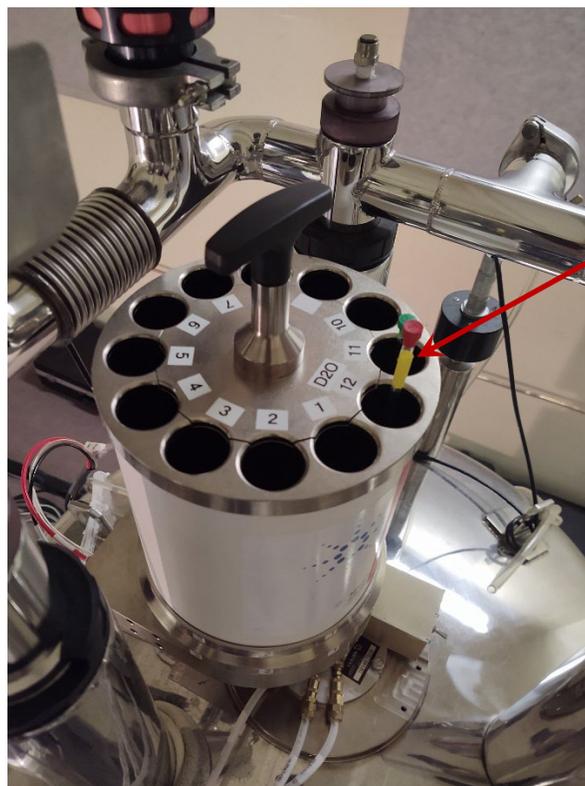
- 6.4 Samples are to be only placed in the designated area (in the tray) for radioactive samples.
- 6.5 Put up the appropriate signage and/or chain at the entrance to the room containing the instrument indicating the presence of radioactive material is present in the area.



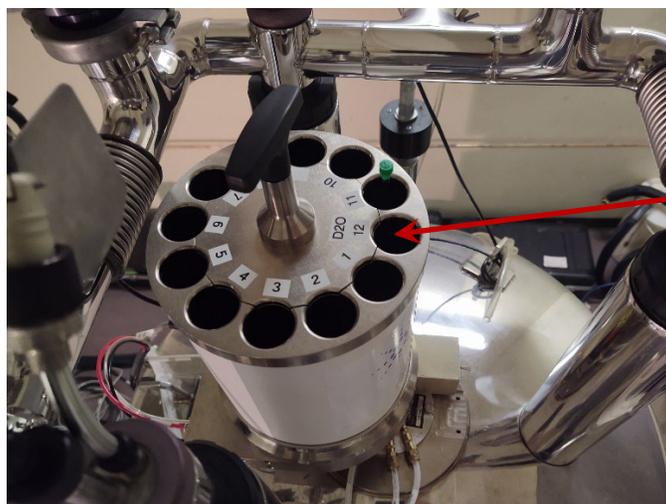
- 6.6 Record the NMR spectra following standard practices using the spinner and depth gauge that is designated for radiological samples. The spinner and depth gauge can be found at the Varian 500 MHz NMR spectrometer, if it is not already present at the 600 MHz NMR spectrometer. Radiological NMR samples must not be spun on the instrument, spinning NMR samples is no longer necessary on modern NMR instruments due to gradient shimming. The autosampler must not be used to prevent the samples from the low chance the NMR tube is crushed when the autosampler rotates. Samples must be manual inject/ejected from the “Sample 12” slot, which contains the D₂O standard.
- 6.7 To eject the sample in the “Sample 12” slot, in the VNMRJ software, click on Tools -> Sample in Magnet. This will open a window. Select the Remove current sample from magnet option and click OK.



- 6.8 The sample that is in the magnet will be ejected from the magnet and it will sit in the autosampler.



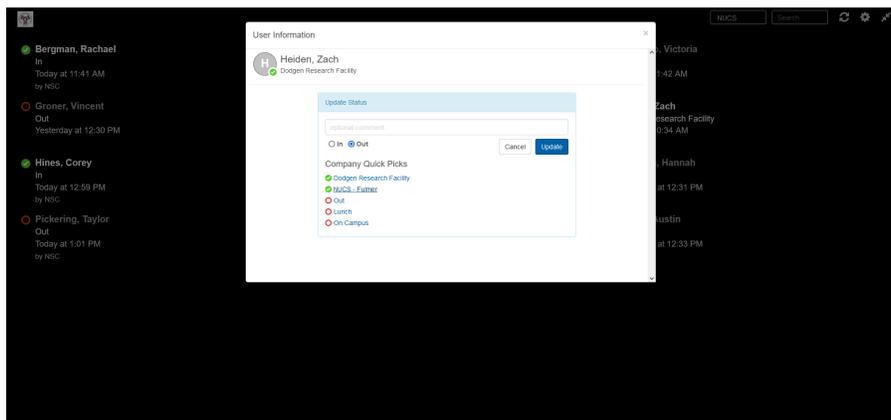
- 6.9 Remove the sample in position 12 and place the radioactive sample in the spinner in position 12.
- 6.10 Go back to the VNMRJ software and select Tools -> Sample in Magnet which will open up a window. Enter the position of the radioactive sample (should be position 12) and click OK. Do not enter a number other than the position of the radioactive sample or the autosampler will rotate. Upon clicking OK, the sample will be inserted into the magnet.



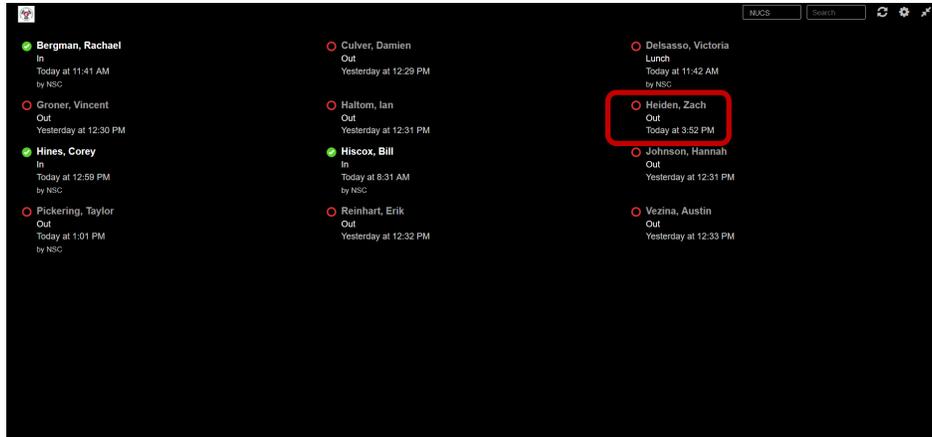
- 6.11 Collect NMR spectroscopic data as normal.
- 6.12 When finished with data collection, repeat steps 6.7-6.10, but insert the D₂O standard and see Section 7.
- 6.13 Radiological samples are not currently approved to be analyzed by solid-state NMR spectroscopy at WSU.

7 POST MEASUREMENT

- 7.1 After the measurements are complete, return the NMR sample to the plastic bag and the carrier.
- 7.2 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.
- 7.3 To logout your sample click on your name.
- 7.4 A window with your name should appear. Click on the Update Status button.
- 7.5 A list of options will be present, click on Out to indicate that radioactive samples have been checked out of the NUCS Fulmer Facility.



- 7.6 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.



- 7.7 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.
- 7.8 Don the appropriate radiological work PPE and return the NMR sample to the appropriate RCA
- 7.9 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.
- 7.10 If the sample(s) is/are free from contamination, then return to the NMR lab, deface the radioactive material stickers, dispose of the paper covering the radioactive material 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.



- 7.11 If the sample(s) is/are found to be contaminated, contact the Principal Investigator, and the NMR Laboratory Supervisor (NSC Emergency Line if after hours). In the rare event that the plastic bag, NMR tube, and PTFE liner all fail, 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 Varian 500 MHz NMR spectrometer (or Varian 600 MHz NMR spectrometer), as there is no cell reception in the NMR lab. To dial off campus, dial 7+1+area code+number.

8 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 NMR 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