Standard Operating Procedure for Transferring Air-Sensitive Samples into NEXSA Using the Vacuum Transfer Module (VTM)

Contact Manager:
Dr. Cassie Ward  
ward@wayne.edu  
Office room 061 Chemistry  
Lab: (313) 577-2587

Dr. Sameera Perera  
fp4261@wayne.edu  
Office room 125 Chemistry

Location
Lumigen Instrument Center NMR Laboratory  
Department of Chemistry  
Wayne State University  
Rm 40 Chemistry Building  
5101 Cass Avenue  
Detroit, MI 48202

Safety Requirements
Access to the lab will be revoked if you do not follow these safety procedures.

1. While working in the NMR Laboratory, researchers are always required to wear Personal Protective Equipment (PPE). The appropriate PPE include safety glasses, long pants/skirt covering the legs completely, closed-toe shoes, and gloves. Do not wear gloves while using the computer keyboard or mouse. All required PPE needs to be supplied by the user.
2. Food and beverages cannot enter the lab. Never eat or drink inside the lab.
3. Please keep the area around the instruments and prep stations clean. If you use the lab’s sample holders, clean them after use. Properly dispose glass waste in the appropriate container. All samples must be properly disposed of in your research lab.
4. Only approved users can reserve the vacuum transfer module for XPS experiments.
5. Approved users can borrow the vacuum transfer module 24 hrs. before the scheduled experiment time. Users will be charged $10.00/24 hrs.
6. The approved users must fill out a form to provide information such as the user’s name, PI’s name, location of the research lab, etc.
7. Return and damage policy,
   a. The vacuum transfer module must be returned immediately after the experiment. If not returned on time, a $50.00 per day penalty fee will be charged.
   b. Users are obliged to replace a VTM or pay a $10000.00 fine for any damage to the VTM caused by mishandling.
8. Following a spill, please clean it up with a paper towel or broom. If you are unsure how to clean up the spill, contact the lab manager (Dr. Ward or Mr. Anderson) or the LIC Director (Dr. Westrick). If the spill occurs after hours with personal injury, please contact WSU police department (7-2222) and Dr. Westrick, immediately.
9. The Standard Operating Procedures (SOPs) are next to the instrument. The users are to follow what is written in these procedures.
10. You must log the usage time into the appropriate instrument logbook.
11. All researchers working in LIC must complete the EH&S initial course for Laboratory
Safety Training and show proof of completion. Users whose safety training has expired will not be permitted access to the laboratory.

12. In case of an instrument malfunction, turn off the instrument and contact Dr. Ward immediately.

**Additional Safety Requirements for the XPS**

1. In case of an instrument malfunction, turn off the instrument by turning the switch in Figure 1 to the OFF position. Contact Dr. Ward immediately.

![Figure 1: Picture of the NEXSA. The red arrow is pointing to the back left of the instrument, where the emergency power shut down knob is located. Turn the knob to the “off” position.](image)

2. All users must pass the two online Citi training for the EH&S Laboratory Safety Training, and Radiation (X-ray) Generating Machine training (https://about.citiprogram.org/en/homepage/). Print the certificates after completing the quiz and bring them to Dr. Ward. The two safety trainings must be completed every January and the printed certificates must be brought to Dr. Ward.

**LIC Authorization Information**

1. Contact Dr. Ward for training to become a user of the XPS.
2. Before running, you must have an infinity account with an index number from your PI.
3. You can reserve the XPS instrument on the Infinity calendar.
4. Before starting your experiment, write your name and start time into the Logbook next to the computer. Also, include a description of your sample and elemental composition.
Overview

1. The vacuum transfer module (VTM) allows air-sensitive samples to be transferred from inert environments (i.e., glovebox) to the XPS instrument.

Operation

The VTM has three parts: sample cover, sample base, and retaining plate (see Figure 1).

1. Always use gloves and clean tweezers to avoid contamination.
   a. Do not touch the areas you wish to analyze.
   b. You can clean the holders and utensils with isopropyl alcohol only.
2. The parts (i.e., sample cover, base, and the retaining plate) should be loaded into the glovebox separately to prevent the device sealing during the antechamber evacuation cycles.

3. As shown in Figure 2, the sample base comprises two parts: the copper base and the stainless-steel sample mounting plate.
   a. The sample mounting plate has a flat side and a raised side. Depending on the thickness of the sample the height and the position of the sample mounting plate can be adjusted.
   b. For thin samples (i.e., 0–2 mm), keep the sample mounting plate at its default position. You can use the raised and flat sides of the mounting plate for ~1 mm and ~2 mm thick samples, respectively.
   c. If you wish to analyze a thick sample (i.e., 2–6 mm), position of the sample mounting plate needs to be adjusted. Please contact Dr. Ward or Perera if you need to change the position of the sample mounting plate.
Please follow the steps given below to load air-sensitive samples to the vacuum transfer module.

1. Load the three parts of the vacuum transfer module separately into the glovebox. Always use a large Ziplock bag as an envelop to avoid possible contamination.
2. Steps (a–) should perform in the glovebox.
   a. As shown in Figure 3, mount the samples using spring clips. Cu tapes can be used alternatively (e.g., for powders and solid flakes).

![Figure 2](image1.png)

Figure 2. Parts of the sample base. (a) flat side (b) raised side of the sample mounting plate. (c) Copper base. The position of the sample mounting plate can be adjusted to accommodate thick samples.

![Figure 3](image2.png)

Figure 3. Sample mounted on the top plate using a spring clip. Parts of the spring clips should not touch the edges of the copper base (circled in red).
b. After mounting the samples, the unit needs to be sealed. Before sealing the system make sure that the rubber O-ring seal is clean. The sample cover has two pins that engage with two notches on the edges of the Cu sample base. (see Figure 4a and b.) The front side of the sample cover and the base should match (see Figure 4c).

![Figure 4](image)

**Figure 4.** (a) Pins located at the bottom of the sample cover (b) notches located on the edges of the copper sample base engage with the pins. (c) Front sides of the Cu sample base and the sample cover should match.

c. Once the unit is closed, fit the retaining plate as shown in Figures 5a and b. This keeps the unit together while the unit is pumped down. Ensure that the metal clips engage before tightening the thumbscrews. Do not overtight the plastic thumbscrews as they tend to deform under excessive forces.

![Figure 5](image)

**Figure 5.** (a) Bottom view of the VTM unit after fitting the retaining plate. (b) Metal clips of the sample cover should engage with the retaining plate before tightening thumbscrews. (c) Sealed VTM is pumped down in the antechamber.
d. Pump down the clamped unit in the antechamber of the glovebox for at least an hour to make sure that the system is fully evacuated (see Figure 5c).

e. Once evacuated, the unit can hold the vacuum for about 30-45 minutes. It is critically important that you bring the evacuated system to the XPS facility as quickly as possible.

f. In the XPS facility, pump down the system for another ~30 minutes using the rough pump. Contact Drs. Ward or Perera to get access to the rough pump.

g. Connect the rubber hose to the ‘one-way’ value as shown in Figure 6 and turn on the pump.

![Image of vacuum pump](Image)

Figure 6. Evacuating the vacuum transfer module using a rough pump

h. After pumping down the system using the rough pump, remove the retaining plate. The external air pressure will hold the unit together even after removing the retaining plate.

i. Turn the vacuum transfer module upside down to make sure that the unit is fully evacuated (see Figure 8a).

j. As shown in Figure 8b, place the module in the load-lock chamber. Make sure that the front side of the module is facing toward the user and the clips of the sample cover engage correctly with the edges of the load-lock chamber.

k. In the ThermoAvantage software, go to the sample tab and select ‘Pump and park in the entry lock’

l. Wait until the pressure goes down to $5 \times 10^{-7}$ mbar. This may take a while (~30 min.)

m. The sample cover will separate from the base during the pump down. Changes in pressure to release the sample holder from the VTM carrier is subtle. If you watch the pump down through the transfer view camera you would see a little shift down. That is the release of the sample holder from the sample cover.

n. Now the sample holder can be transferred to the analysis chamber. Once transferred, chose an analysis point and register it (Figure 7)
**Unloading the Vacuum Transfer Module**

When the experiment is over, transfer the holder to the load-lock chamber.

1. Open the load-lock chamber (vent the load-lock).
2. To remove the sample cover, press the two buttons circled in red in *Figure 8*. Disengage the sample cover from the load-lock chamber and lift the cover.
3. Then, unload the sample base and close the load-lock chamber.
Special Instructions

- Always use the designated plastic container to transport the VTM from one place to another (e.g., from your lab to the XPS facility).
- After evacuating the unit, do no try to force open the sample cover. Doing so will deform the sample cover and damage the vacuum seal.
- Do not use strong organic solvents (e.g., acetone) to clean the viewport or the rubber O-ring seal. *Use isopropyl alcohol only.*

References