

# Capillary NMR Instructions

1. Check logbook and solvent sheet (by injector) to determine solvent currently in use. If the solvent listed is the same as your solvent, you can either assume the last person has flushed the probe with 2 X 50uL of this solvent, or you can give it this flushing to be sure (Injection procedure is on wall). You can now skip to step 2. If you want to use a different solvent, perform the following steps:
  - i. Flush the line with air (see procedure on wall).
  - ii. Flush with 2 X 50uL of acetone-d6 (see basic injection procedure on wall).
  - iii. Flush line with air to expel acetone-d6.
  - iv. Prime line with 50uL of your chosen solvent.
  - v. Go to step 2.
2. Inject 10uL of your sample. Follow this with 9uL of your solvent to push your sample into the flow-cell. NOTE: If you have 19uL of sample, you can avoid the extra injection step by injecting 19uL of your sample (not recommended for mass limited samples).
3. If you are running an experiment that lasts longer than 20 minutes, it is recommended that you plug the evacuation line (see procedure on wall).
4. See instructions by workstation for changes in experiment setup.

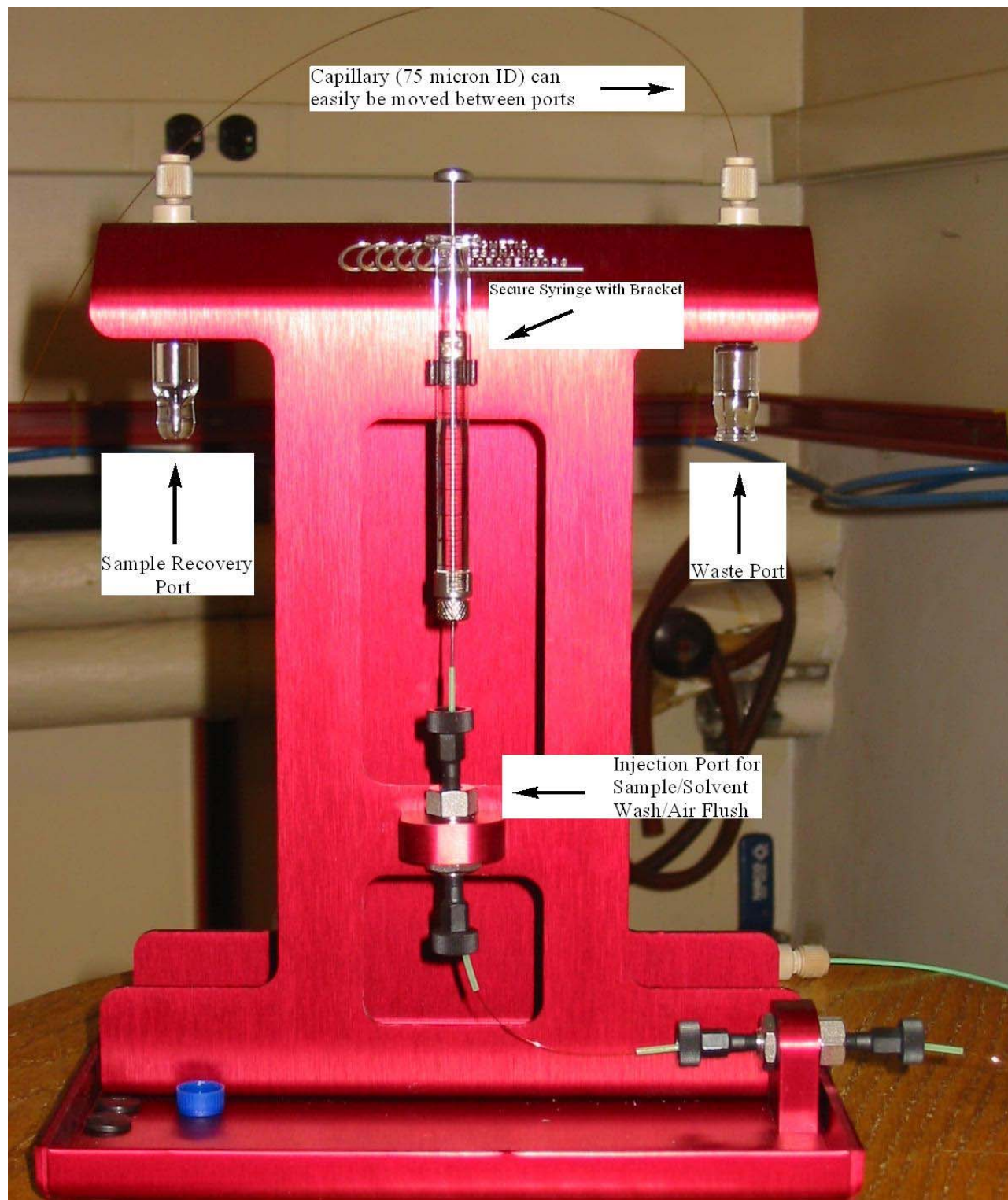
5. After the experiment, you can recollect your sample a number of ways (BE SURE TO UNPLUG THE LINE IF YOU PLUGGED IT FOR YOUR EXPERIMENT):

- i. If you are using a volatile solvent, you can push your sample out with air (you will get ~28uL), followed by 2 X 50uL rinses (yielding ~ an additional 72uL...28uL will remain in line).
- ii. If you are using a non-volatile solvent. Inject another 9uL of your solvent letting this go to waste, followed by 10uL more that you will collect, and finally perform a rinse with 2 X 50uL of your solvent to clean the line.

6. Be sure to designate the solvent you used both in the logbook and on the sheet by the manual injector.

NOTE: If you have any questions, please call me at X33069, or page me at 401-235-8306.

# Manual Injector

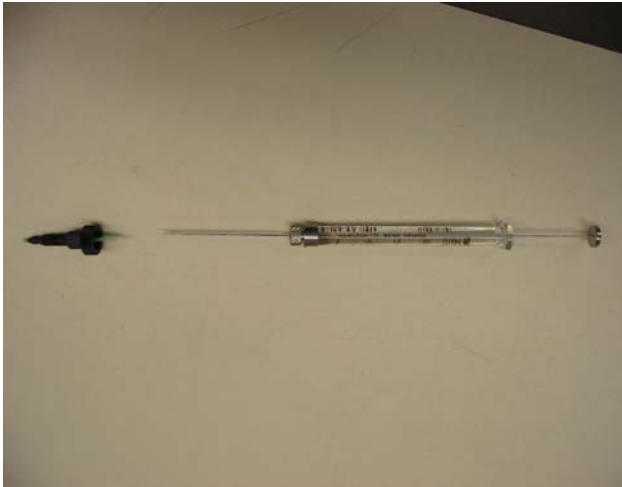


# Manual Injection Procedure

1. Remove the 50uL syringe from the manual injection port by unscrewing the black nut, releasing the syringe from the bracket, and pulling the syringe with the nut, ferrel and sleeve out of the injection port.



2. If you are using the same solvent specified in the logbooks by the previous user, you can either slide the black nut/ferrel/sleeve assembly over the needle of the 25uL syringe containing your sample or you can perform another 50uL rinse with your solvent to ensure a well rinsed capillary and flow-cell.



3. Place the assembly into the injection port and snap the syringe into the bracket. The syringe needle tip should make contact with the bottom of the injection port. Tighten the nut while ensuring the needle is seated against the injection port stop (easily ensured by rotating the needle while tightening).
4. Inject the sample at a flow rate no greater than 1uL/sec. You should observe drops falling into the waste port on the injection stand. NOTE: Higher viscosity solvents will exhibit greater resistance during injection, do not attempt to inject too quickly.

## Procedure for Sealing Off Waste Line

1. Follow the capillary line back from the waste port on the manual injector to the first union. Unscrew the connector from the side closest to the manual injector and screw in the plug which should be located in the base of the manual injector. You are finished. Remember to remove this plug and screw back in the connector of the line going to the waste before trying to recover/or send your sample to waste.

To probe



To Waste



