**NMR Tube Cleaning**

1. Empty all contents from dirty NMR tubes into appropriate residue container. Remove the labels on the NMR tube by rinsing with ethanol.
2. Fill the container outside the protect bottles with liquid nitrogen and cool it down.
3. Load the tubes into the tube cleaning apparatus (tube washer) with proper solvents as noted below.
4. The operation of the tube washer:
5. With the left valve closed, slowly and briefly opening the T-valve to the vacuum position evacuates the air from the Labconco® vacuum bottle and places it under a slight vacuum without removing the solvent.
6. Turn the T-valve to the vent position opens the vacuum bottle to atmosphere breaking the vacuum and forcing solvent into the NMR tube.
7. Switch the T-valve back to the vacuum position places the NMR tubes back under vacuum and remove the solvent from the NMR tube.
8. Repeat the process two or three times effectively washes the NMR tubes with solvent.
9. Switch the T-valve to the vent position and opening the left valve rapidly removes the solvent from the NMR tube cleaner into the filter flask. The process can be repeated with other solvents.
10. Rinse NMR tubes with nano pure water by filling with water then empty all contents from dirty NMR tubes into appropriate residue container.
11. Rinse NMR tubes with ethanol by filling with ethanol then empty all contents from dirty NMR tubes into appropriate residue container.
12. Rinse NMR tubes with acetone by filling with acetone then empty all contents from dirty NMR tubes into appropriate residue container. The amount of organic solvents should be well-controlled.
13. Fill tubes with nano pure water.
14. Place NMR tubes in a concentrated nitric acid bath and soak 24hrs.
15. Empty contents of NMR tubes into the acid bath.
16. Rinse the NMR tubes under the tap water to remove most of the acid.
17. Load the tubes back to the tube washer.
18. Rinse NMR tubes in baking soda solution to neutralize acid.
19. Rinse NMR tube thoroughly with nano pure water until the solvent waste is neutral.
20. Dry NMR tubes: lay tubes horizontally in the oven.
21. Once dry inspect tubes for any crack, divots or other defect. The tubes with crack on the opening should be collected for further modification and reuse. Other problematic tubes have to be disposed.

