

VII. Equipment

N. NMR

There are 3 NMR instruments typically used by students in our lab: 300 MHz in KNCL 119 and GDYR 103a and 500 MHz in GDYR 103a. *Students must be trained by the Magnetic Resonance Center (MRC) staff for each type of NMR.* Training can be scheduled by emailing Dr. Venkat Dudipala at vrd@uakron.edu

Once your MRC account is set up, you may schedule 2x15min blocks of time per day on the 300 instruments, and 2x1hr blocks on the 500 instrument. *Do not go over your scheduled time.* The 500 instrument requires 8 inch sample tubes. Generally, 5-10 mg sample in 0.7 mL deuterated solvent is good enough for H NMR (use 20-30 mg sample for polymers), while 50-100 mg sample / 0.7 mL solvent is required for C NMR.

More information about the MRC and detailed operating procedures for the 300 instrument may be found at <http://www.uakron.edu/chemistry/magnet/>

The best way to clean NMR tubes is to promptly rinse them with organic solvents. NEVER clean tubes by sticking the tip of an organic solvent wash bottle into the NMR tube. This frequently leads to broken tubes and sadness. If you don't want to shoot organic solvent at close range into an NMR tube, clean tubes with the glass NMR tube cleaner.

1. Attach the NMR tube cleaner to a 24/40 Erlenmeyer flask.
2. Apply vacuum from a water line aspirator.
3. Empty the solvent from your NMR tube into the solvent waste. Place a cap on the bottom of the tube and fit it into the cleaner. The cap should make a nice seal.
4. Add organic solvents to the reservoir. The vacuum will pull the solvents through the NMR tube. Always finish with an acetone rinse.
5. Never dry tubes in the vacuum oven, the high heat can cause the tubes to warp.

Dirtier NMR tubes may require some scrubbing with a pipe cleaner. If you've managed to dirty up a tube so badly that it still remains unclean, aqua regia may be your last resort. Be extremely careful using aqua regia, and do not attempt to clean tubes using this method unless first shown how by another lab member (*see Working with Aqua Regia*). NMR tubes are never cleaned using basic solutions and never go in the base bath; this can etch the tubes.

NMR tube caps may be cleaned by several rinses of solvent (THF is a popular choice) in a 20 mL scintillation vial.