

JEOL Instrument Management

NMR Director: Ted Burkey (tburkey@memphis.edu) x2634

NMR Manager/Varian Supervisor: Truc Chi Pham (tcpham@memphis.edu) x4865

JEOL Student Supervisor: Brandon Gindt (bpgindt@memphis.edu) x4428

JEOL NMR student supervisor responsibilities.

The student supervisor will notify Dr. Burkey or Dr. Pham of any malfunctions or irregularities.

Do not use white dewars or any that are not stainless steel, can quench magnet) See picture.

Fill Day: every 6th day except following Monday's fill on the next Friday

(1) Check log book for any malfunctions. Arrange for maintenance as needed.

Take new dewars to 317a and weigh them. Make sure they are low pressure dewars (< 30 psi).

(2) Check LN2 level in JEOL (remove LN2 cap and dip with black composite stick to the bottom of dewar for a few seconds and then check the length of the frosty section) Check Oxford note book for conversion to % level and liters. Record in notebook along with He gas flowlevel.

(3) Transfer LN2 to JEOL: Bring dewar no closer than 4 feet. Do not bring dewar dolly or anything magnetic closer than 4 feet. lightly tighten compression fitting of transfer line (brass fitting/copper tube/latex hose) to liquid dewar port and open liquid port. When liquid comes out remove LN2 port cap and connect rubber hose to LN2 port.

(4) Transfer LN2 to department dewar using all metal line. Open department dewar gas port to relieve pressure. Connect line to rental dewar and open valve. When liquid comes out connect to department dewar liquid port. Open the port as soon and compression nut is tight and fitting is sealed. Transfer until LN2 exits gas port of department dewar or rental dewar is empty.

(temperature of transfer line drops rapidly, need to use thermal couple detector).

(5) record weight of department dewar.

(6) leave transfer line on rental dewar if not empty.

(7) quick dust mop 217a. Hang on tight to dust mop when near magnet.

(8) keep notebook schedule filled 2 weeks in advance.

(9) make sure tissue paper and printer paper is available.

Two days before Fill: Check department dewar and rental dewar level. Tell Adrienne how many dewars are need. If department dewar is less than half full order two dewars. If department dewar is 1/2 to full order 1 dewar. If department dewar is full do not order a dewar. Make sure empty dewars in 317a a returned to SM003 for pick-up

Student supervisor training



The student supervisor will know how to shim, lock, load/eject sample, obtain routine proton/carbon spectra, integrate and ppm lable spectra, transfer data to external computer, and load paper in printer.

Manager and Director repsonsibilities.

1. Supervise and train student supervisor
2. Order liquid helium and fill dewar
3. Annually have training class for routine experiments.
4. Assist in training for non-routing expts to the extent of manager and directors training.
5. periodically replace air filters
6. Set up FACES and JEOL accounts
7. Troubleshoot and fix problems consulting with instrument documentation and vendor engineer
8. Advise Department Chair and Facilities Committee on the necessity of instrument replacement or repair by vendor

User Training on Instrument

- (1) Since many groups use the NMR most users should obtain training for routine use while observving members of their group.
- (2) Otherwise routine training will be administered once a year during the summer.
- (3) Dr. Pham or Dr. Burkey will assist in training for special experiments (VTNMR, broadband applications, etc).

User Guidelines and Scheduling

- (1) Priority for use of the JEOL spectrometer is established by "reserving time" on FACES **prior to use**. If all or part of a reservation cannot be used then users must cancel unused reserved time so that others may use the instrument.
- (2) Sign in the log book **before** you begin using the NMR. If you are late more than 5 minutes for **your reservation, it will be canceled**. If there is not enough room, make a note on the opposite page.
- (3) Between 7 AM and 5 PM. A single user may reserve, in advance, up to **one-half hour**. An additional 1/2 hour can be reserved at the end of a reservation or any other time if no other user has made a reservation by that time. Users requiring larger blocks of time should use the JEOL on weekends or evenings. See T. Burkey for special arrangements.
- (4) If you are using the nmr and have not signed the log book, anyone can sign in, stop your experiment and log you out.
- (5) Do not allow others to sign-up and use the NMR under your account. You will be held accountable for their actions.
- (6) **Do not attempt to reboot any part of the NMR if the nmr does not appear to be operating correctly**. Get assistance from the student supervisor, Dr. Ted Burkey or Dr. Truc Chi Pham.
- (7) All users must have an account or be currently enrolled in a class using the NMR.

- (8) See your advisor or instructor for instructions on routine proton and carbon NMR.
- (9) Make sure the lock (yellow LED on top of spectrometer console is one) is on before you use the NMR. Make a note in the log book if you observe anything unusual or have difficulties.
- (10) Remove the dust cover from probe inlet. Turn on both valves for the air supply before spinning. **Turn off the valves if at any time the traps begin to fill with water.**
- (11) After you are finished, leave the 0.1% ethylbenzene/deuteriochloroform sample in probe, lock and shim the sample to obtain a stable lock signal, stop spinning, turn off the air, and return the dust cover to top of probe inlet.
- (12) Instructional courses have priority even if a user has signed-in. However, course instructors must sign-in and notify any user who loses a reservation 24 hours in advance.
- (13) Repeated failure to conform to these procedure will result in loss of user privileges.

Maintenance and repairs

See **Manager and Director Responsibilities.**