

NEO 500 INSTRUCTIONS – SOP MANUAL MODE

SAMPLE PREPARATION

- Dissolve your sample (5-20 mg) in 700 μ l of deuterated solvent and transfer it to a 5mm NMR tube
- Write your name/sample code on the top part of the NMR tube
- Clean your NMR tube using a kimwipe.
- Insert the NMR tube in a blue spinner, adjusting its position using the sample depth gauge available
- Place the spinner(s) in the carousel of the autosampler, noting its position(s).

DO NOT:

- Use broken NMR tubes
- If the NMR tube slides too easily in the spinner, change the spinner

For NMR tubes longer than 8", such as pressure tubes, J-young etc..., contact NMR staff. They shouldn't be placed in the carousel.

COMPUTER PREPARATION

- Login in as "nmr"
- Click on computer icon to "NMR data" and login using your MUN credentials
- Open TopSpin 4.4.0
- Open the data browser, and find your name: NEO_500 /your_name
- Open the "test" file in the nmr folder by dragging it to the main window/double click
- Type **sxd** in the command line to open sample list. Add you sample(s). Do not check lock and topspin

EXPERIMENT SETUP

- Click on: **Create dataset** Enter the name of your folder, the experiment number (start with 1), the solvent, the experiment protocol (for user, see available options below), title.
- Load you sample: in the sample list window, double click on the sample or click on insert icon. Wait for the light to be yellow.
- Click on **Lock**. Choose the solvent
- Click on **Tune**. Wait until its completed
- Click on **Shim**. Wait until successfully completed.
- Click on **Prosol**.
- Click on **Gain**.
- Type **xaua** in the command line to start the experiment.

EXPERIMENTS AVAILABLE

The user recommended parameters include (not exclusively):

- PROTON: ^1H NMR
- WATER: ^1H with water suppression
- C13CPD: ^{13}C experiment, ns=1k, 235ppm
- C13CPDSN: ^{13}C experiment with sino. Must define signal and noise ranges and sino value.
- DOSY_DC: Fully automated DOSY for small molecules with Dynamic Center processing
- P31: ^{31}P no decoupling
- P31CPD: ^{31}P with ^1H decoupling
- PROP31CPD: ^1H with ^{31}P decoupling
- F19CPD: ^{19}F with ^1H decoupling
- SI29IG: ^{29}Si with inverse gated decoupling
- AL27ND: ^{27}Al NMR
- B11
- H2: ^2H using lock channel.
- 13C{19F} CPD: ^{13}C with ^{19}F decoupling

This list is not exhaustive. Other experiments can be run upon request.

PROCESSING

If processing on TS 4.4, type **xaup**.

Otherwise process with Mnova as usual.

VARIABLE TEMPERATURE

Talk with C-CART staff before running any VT experiment. The probe can go from -150C to +150C and the BCU from -80C to +150C. LN2 will be needed from -80C to -150C.

TO END

- Eject your sample and remove it from autosampler
- Close Topspin
- Sign out from nmr