Basic NMR Spectrometer Operation (Diels 400 MHz)

The following guidelines provide an overview to operation of the 400 MHz Bruker NMR spectrometer called "Diels" and are to be used as a memory aid for new users. Before using NMR spectrometers for the first time, users **must** attend a training session. Steve Huhn should be consulted immediately if any problems are encountered when using the instruments.

1. Arrival at NMR

- log-in and run Topspin software (single click on icon)
- press "lift" button to engage sample air lift
- after cleaning with tissue paper, place NMR sample tube in spinner and adjust to correct depth with gauge
- gently place NMR tube/spinner on air cushion in center of magnet
- turn-off air lift (press "lift" button again), sample will sink into the magnet

2. Data acquisition **[new → zz → gpro & rsh → lock & shim → rga → zg]**

- **`new <CR>`** set-up new file for NMR experiments connected to this sample

  filename: use your lab book ref. plus an appropriate extension letter to indicate origin of sample, e.g. "**`PRB502A`**"

  exp. no.: distinguishes between different NMR experiments run on the same sample; it is recommended that you standardize how this index number is used, e.g. 1 for ^{1}H NMR, 2 for ^{13}C NMR, 3 for DEPT135, 4 for COSY

  proc. no.: 1

  solvent: choose solvent from drop-down list as appropriate

  title: use the following standard format: sample ref., approx. amount, # of scans, solvent, NMR field strength, date, e.g.

  "**`PRB502A, 5 mg, 16 sc, CDCl3, 300 MHz, 2/2/2016`**"

- **`zz <CR>`** brings up a list of common NMR experiments; choose as appropriate
- **`ns <CR>`** (optional) set number of scans (default is 32); for standard ^{1}H NMR's and samples of 5 mg, 16 scans is ample (ns should be a multiple of 4)
- **`gpro <CR>`** read probe information
- **`rsh <CR>`** read current optimized shim set
- **`lock <CR>`** automatically sets deuterium lock; choose appropriate solvent from list; machine will take ca. 5 seconds to lock, indicates when finished

manual shimming: while viewing lock display window, adjust "z" and "z^2" (on axis) to optimize shim set (i.e. make lock line as high as possible); do z first, then z^2, then go back to z etc. (nb. activate "fine" control as needed); if

line goes off screen reduce "lock gain" (can use this function to raise and lower line as desired); when happy with shim, press "std. by" to prevent accidental change of parameters

[use **topshim** command to automatically shim if on Robinson]

- **wobb <CR>** (optional) tune and match the probe; maximize and center the signal using the T (tuning) and M (match) adjustor screws at bottom of NMR

  [use **atma** command to automatically tune/match if on Robinson]

- **rga <CR>** automatically set receiver gain; machine will pulse sample with rf and monitor strength of NMR signal; procedure takes ca. 5-10 seconds

- **zg <CR>** "zero go"; command starts data acquisition and will accumulate FID for the desired number of scans

  to stop data acquisition early, type "**halt <CR>**", this stops scans *and* saves FID data; *nb. the alternative stop command,* "stop <CR>" *does not save collected data to the hard disk*

3. Data processing

[summary: ft → phase → baseline → calibrate → integrate → pick peak]

- **ft <CR>** to perform Fourier transform on collected FID data; zero-filling can be conducted prior to FT by typing "**si <CR>**" and then doubling number (e.g. from 32 K to 64 K), this operation is optional and it improves the cosmetic appearance of peaks making them smoother

- *phase correction* enter phase correction by clicking on icon (or access from processing menu) [or use command **apk <CR>** for **a**utomatic **p**hase **k**orrection]

  zero-order: hold mouse pointer over "0" icon and with left mouse button depressed, move mouse up/down to change zero-order phase angle; adjust until line shape of largest peak in spectrum is nicely symmetrical about its base

  first-order: hold mouse pointer over "1" icon and with left mouse button depressed, move mouse up/down to change first-order phase angle; adjust so that a peak distant from the largest one has good symmetrical shape about its base

- **abs n <CR>** performs automatic baseline correction without auto-integration

- *calibration:* zoom in on desired ref. peak, click the calibrate icon, move calibration line onto the peak and click left mouse button; enter the desired ppm value (see BRG website for values; nb. $\delta_{H\ (CHCl3)} = 7.26$ ppm)

- *integration:* click integration icon; integrate by dragging integrals across desired regions; click save and exit icon when happy with integrals

- *pick-peak:* pick-peak by dragging box across all signals of interest; click save and exit icon when happy with peak-picks

- **plot <CR>** enter NMR plot software and call-up a BRG standard plot template