

## Solid-state NMR routine (2019/10/18)

**These instructions are only valid within the McGill Chemistry Department MC<sup>2</sup> Facility. Improper usage at McGill or elsewhere can severely damage equipment.**

The routine listed here only gives vague guidelines! You must understand what you are doing and ask questions if you are unsure. Any instructions you have been given by Robin that deviate from these instructions must be followed.

### *Preliminary setup*

1. pack rotor (if not already packed)
2. record rotor in rotor log book (unless your group owns the rotor) – write your name, your chemical's name and log book number, and any safety notes (e.g. contains mercury)
3. check that probe is set up for the nuclei you are interested in (ask Robin if you are unsure); insert tuning rod / cap if needed

### *Standard sample*

1. load standard sample (e.g. glycine, adamantane, DSS, NH<sub>4</sub>PO<sub>4</sub>)
2. start spinning (go to 5 kHz)
3. spin sample to desired spin rate, in steps of 5 kHz or less – if there is trouble, go back down in steps of 5 kHz or less
4. tune sample – **do this every time** – type *trtune* to enter tune mode, then *ff* to ensure display is correct
5. load previous experiment for standard sample
6. acquire (*au*), process (*wft*), phase (*apk* – use if needed), check that signal-to-noise looks reasonable (ask Robin if you are unsure), reference signal (put cursor near line, type *nl*, type *rl*(*reference chemical shift*[p]) (example: *rl*(176.5p) for glycine), set reference for future spectra (type *setBaseref*(*reference chemical shift*[p]) (example: *setBaseref*(176.5p) for glycine), use File / Autosave to save (say OK if it asks about overwriting)
7. spin sample down in steps of 5 kHz or less to 5 kHz, then press Stop

### *Actual sample*

1. insert actual sample
2. start spinning (go to 5 kHz)
3. spin sample to desired spin rate, in steps of 5 kHz or less – if there is trouble, go back down in steps of 5 kHz or less
4. tune sample – **do this every time** – type *trtune* to enter tune mode, then *ff* to ensure display is correct
5. load previous experiment for standard sample
6. click Clear in Start / Sample info and type sample name
7. acquire (*au*), process (*wft*), phase (*apk* – use if needed), check that signal-to-noise looks reasonable
8. spin sample down in steps of 5 kHz or less to 5 kHz, then press Stop
9. return probe to standard state (<sup>13</sup>C), if needed AND you know how

10. fill out log book with your name, the time you used, the nucleus you acquired, the spin rate you used, and any problems you encountered (also tell Robin by email or phone, 514-550-8751 if serious or you don't know if it's serious)

***Minimum information you should know for an experimental writeup***

To find what a parameter is in VnmrJ, type the name of the parameter followed by a question mark. For example, to know how many scans you will run, type *nt?* To find out how many scans you actually ran, type *ct?*

Parameter	Value
Spectrometer model	Varian (now Agilent, Santa Clara, CA, USA) VNMRS with wide-bore 9.4 T magnet
Resonance frequency of nuclei you are using	see Acquire / Channels
Probe	usually Varian T3 double-resonance 4 mm probe
Spin rate	see MAS unit
Experiment you are running	see top of window: usually cross-polarization magic angle spinning (tancpx), CPMAS with echo for baseline correction (tancpxecho), quantitative CP using Johnson and Schmidt-Rohr's method – see <b>and cite</b> their paper! (tanquantcpxecho or tanquantcpx), CPNQS (tancpxidref), CP with editing (tancpxedit), or single pulse (onepul)
Number of scans	ct
Recycle delay	d1 (in seconds)
Total time	usually nt * d1, though this does not work for quantitative CP
Acquisition time	at
CP contact time	tHX
Number of CP periods, for quantitative CP	nCP
Time between CP periods, for quantitative CP	t1ZF
Dipolar dephasing period, for NQS experiments	t1Xidref
Length of second CP period, for edited CP experiments	t1HXedit

***Example experimental writeup*** (NOT VALID GENERALLY – YOU MUST USE THE CORRECT VALUES FOR YOUR SAMPLE AND EXPERIMENT!)

MAS spectra and the static <sup>113</sup>Cd powder pattern were acquired on a Varian VNMRS (now Agilent,

Santa Clara, CA, USA) spectrometer operating at 399.8 MHz for <sup>1</sup>H, 100.5 MHz for <sup>13</sup>C, 89.5 MHz for <sup>199</sup>Hg, 88.7 MHz for <sup>113</sup>Cd, and 40.5 MHz for <sup>15</sup>N using a 4 mm double-resonance Varian Chemagnetics T3 probe (now Agilent, Santa Clara, CA, USA). Approximately 70 mg of sample were center-packed into rotors using either Teflon or boron nitride inserts, and spun at between 8 and 15 kHz for MAS. The static <sup>199</sup>Hg spectrum of *sql*-Hg(Im)<sub>2</sub> was acquired on a Varian VNMRs spectrometer operating at 499.9 MHz for <sup>1</sup>H and 89.5 MHz for <sup>199</sup>Hg using a 6 mm double-resonance Varian Chemagnetics T3 probe.

The <sup>13</sup>C CPMAS spectrum was acquired in 512 scans under spinning at 8 kHz, using a recycle delay of 10 s with a contact time of 4 ms at a <sup>13</sup>C rf field of approximately 60 kHz. SPINAL-64 decoupling at an rf field of 90 kHz was applied during acquisition. Spectra were referenced using the carbonyl carbon signal in glycine at 176.4 ppm with respect to TMS.<sup>6</sup>

## References

6. R. E. Taylor, *Conc. Magn. Reson. A.* 2004, 22A, 79.