Protocol for the Use of the Gemini-2000 NMR Spectrometer

1. Collect spectrum of menthol in $CDCL_3$ using standard organic chemistry laboratory automated procedures.
    - autolock, autoshim and autogain will all active.
    - Execute "ga" to perform any automated operations, acquire the data, perform the post-acquisition Fourier transformation and phasing and display the resulting spectrum.
    - Check baseline.
    - Check chemical shift of $CDCl_3$ signal.
    - Check line shape and resolution of ~3.2 ppm multiplet.
    - Compare acquired spectrum to printed reference spectrum.
    - If OK, proceed. If not OK, fix it, otherwise contact teaching staff.

2. Replace menthol sample with user sample.
    - Confirm start of sample spinning by observation of the flashing lamp and meter indication between 10 and 20 RPS (revolutions per second).

3. Reconfigure spectrometer.
    - Select appropriate "nucleus" and "solvent" and execute "su".
    - Confirm proper lock operation.
    - Either manually adjust Z^1 and Z^2 shims now or use automatic shimming later.
    - Disable autogain and set gain to minimum value.
    - Disable autolock and autoshim for maximum throughput or enable either, as needed by experimental protocol.

4. Check for receiver overload.
    - Execute "nt=1" to acquire one transient (not "scan").
    - Execute "go" to collect the one transient without any prior automatic setting of parameters or operations and without any post-acquisition processing.
    - Execute "df" to display the acquired transient ("display FID").
    - Examine the acquired transient and look for evidence of ADC (analog-to-digital converter) "clipping" where the signal is outside of the ADC's operating range.
    - If clipping occurs, reduce the excitation RF pulse width. The parameter "pw" is typically set to 6.4 μs to obtain maximum signal. Reduce it (for example, by one-microsecond steps) until clipping is eliminated.

5. Acquire a trial spectrum.
    - Execute "nt=16" to acquire sixteen transients.
    - Execute "ga" to perform any automated operations, acquire the data, perform the post-acquisition Fourier transformation and phasing and display the resulting spectrum.

6. Check spectrum quality.
    - Adjust phase, if necessary.
    - Look for evidence of receiver overload appearing as many undulations of the baseline.
    - Confirm that an adequate signal-to-noise ratio (S/N) has been obtained and that no additional transients are required.
    - Confirm that adequate line shape and width have been obtained and that no additional shimming is required.

- Save the spectrum to the NMR host computer's disk: execute "svf('xxx_yyy_mmdd_ii')" where xxx is the course number, yyy is the user's initials, mm is the current month, dd is the current day and ii is an index number used to identify the user's samples. Use only lower case characters throughout.

7. Perform all necessary measurements, changing sample as required and making any necessary adjustments such as shimming.

8. Restore standard organic chemistry laboratory configuration.
    - Retrieve appropriate parameter set: execute "rts('ochem_std')" then execute "su".
    - Confirm the following parameter values:
        - pw=6.4
        - nt=16
        - alock='y'
        - gain='y'
        - wshim='e'

9. Collect spectrum of menthol in $CDCL_3$ using standard organic chemistry laboratory automated procedures.
    - Use same protocols found in section #1 of these instructions.
    - Execute "ga"; observe acquisition status at all stages.
    - Compare resulting spectrum to reference spectrum posted near spectrometer. If OK, plot data, scale and parameters, annotate hardcopy plot with user's last name, date and time of acquisition and post with other reference spectra. If not OK, contact teaching staff.

List of useful VNMR commands and parameters:

- go - acquire time domain data (FID)
- ga - perform pre-acquisition automated procedures, acquire time domain data, perform post-acquisition Fourier transformation and phasing and display resulting spectrum
- df - display FID
- aph - perform zeroth and first order spectrum phasing automatically
- aph0 - perform only zeroth order spectrum phasing automatically
- svf('file name') - save NMR data to NMR host computer disk
- rts('file name') - save spectrometer parameters to NMR host computer disk
- su - transfer current set of parameters to spectrometer hardware
- wshim='e' - execute computer-controlled automatic shim procedure prior to acquisition of time domain data
- wshim='n' - do not execute automatic shim procedure
- alock='y' - execute computer-controlled automatic lock search and capture procedure prior to acquisition of time domain data
- alock='n' - do not execute automatic lock procedure
- gain='n' - execute computer-controlled automatic receiver gain setting procedure prior to acquisition of time domain data
- gain=0 - do not execute automatic receiver gain setting procedure and manually set receiver gain to minimal value

Typical lock parameter values used in standard organic chemistry laboratory procedures:

- Z_0 - 463
- lock power - 17
- lock gain - 30
- lock phase - 308