Use of the 600 MHZ NMR SPECTROMETER

Purpose

This SOP covers the use of the 600 MHz NMR spectrometer:

- Setting the temperature properly
- Setting the sample in the spinner to the proper depth
- Tuning the probe
- Staying within pulse length and power limitations
- Call or text Andy LiWang @ 209.777.6341 at any time day or night with questions.

Summary of Significant Health and Physical Hazards

- The magnet is very strong and can affect metal implants, and pacemakers.
- In the event of a quench of the superconducting magnet, oxygen in the room can be reduced to dangerously low levels.

Risk Assessment

The overall health and safety risk for use of this spectrometer in accordance with the procedure and protocol in the following section is considered **LOW** based on:

- The spectrometer does not contain hazardous materials.

Protocol/Procedure

1. Only use the 600 MHz spectrometer if you have been allowed to do so by Andy LiWang. Failure to adhere to any part of this SOP will result in the denial of access to this instrument. If you have any questions regarding this SOP, see Andy LiWang.

2. Login and start Topspin software.

3. If there is an error message at anytime during the setup or running of an experiment, stop and call Andy LiWang immediately at 209.777.6341.

4. Check whether the probe contains a sample. If it does, eject the sample.

5. Enter edte in the Topspin window. Check the following:
   - Airflow = 670 l/h
   - Proportional band = 78.0
   - Integral time = 111.0
   - Derivative time = 18.0

6. In the edte window, set the max power to the lowest possible value that still allows the probe to reach the temperature you desire.

7. In the edte window, change the temperature in steps of 10 °C maximum until you reach the desired temperature. Using steps of 5 °C is highly recommended for probe longevity. Before proceeding to the next temperature step, wait until the probe temperature is within 2 °C of the current step temperature. (Mechanical stress due to rapid temperature changes will shorten the lifetime of the probe, which costs $200,000.) Also, do not go below 0 °C or exceed 60 °C!!
8. Set the sample to the correct depth in the spinner using the depth gauge. **Expensive damage will occur to the probe if the sample is set too deep in the spinner.**

9. Insert the sample into the magnet.

10. Tune the probe using the wobb command. When tuning the probe, keep the following in mind:
    - Do not grab any part of the magnet, lines, or hoses when crawling under or out from under the magnet.
    - When you feel resistance to turning the tuning rods, do not continue to turn them in the same direction, because doing so will strip the threads on the tuning rods and require a costly repair.

11. Shim the sample.

12. Calibrate pulses. Do not exceed the power and length limitations for pulses. **Doing so shortens the lifetime of the probe and can fry the electronics, resulting in costly repairs.** Note that most likely you will only need to calibrate your $^1$H pulses, because most of the $^{13}$C, $^{15}$N, and $^2$H pulses have already been calibrated (see attachment).

    - $^1$H max pulse length @ max power: 12 $\mu$s @ 6 W (i.e., do not use more than 6 W of power ever and the longest pulse at 6 W is 12 $\mu$s.)
    - $^{13}$C max pulse length @ max power: 15 $\mu$s @ 72.5 W (i.e., do not use more than 72.5 W of power ever and the longest pulse at 72.5 W is 15 $\mu$s.)
    - $^{15}$N max pulse length @ max power: 45 $\mu$s @ 75 W (i.e., do not use more than 75 W of power ever and the longest pulse at 75 W is 45 $\mu$s.)
    - $^2$H max pulse length @ max power: 150 $\mu$s @ 6.6 W (i.e., do not use more than 6.6 W of power ever and the longest pulse at 6.6 W is 150 $\mu$s.)
    - Maximum power for decoupling on any channel for AQ > 100 ms is 0.04 W.
    - For AQ < 100 ms, $^{13}$C decoupling: pcpd2 = 60 $\mu$s @ plw12 = 4.75 W (or use CHIRP decoupling as described in the green box of the attached pulse-calibration file.)
    - For AQ < 100 ms, $^{15}$N decoupling: pcpd3 = 240 $\mu$s @ plw16 = 2.63 W
    - For AQ < 100 ms, $^2$H decoupling: pcpd4 = 400 $\mu$s @ plw17 = 0.93 W

13. If you need to use a $^{13}$C, $^{15}$N, or $^2$H pulse that is not in the calibration file (attached), and does not exceed pulse and power limits, you use the following equation:

\[
\frac{plw2}{plw1} = (\frac{p1}{p2})^2
\]
where p1 & p2 are pulse lengths and plw1 & plw2 are the corresponding power levels. If you know p1 and plw1, and want to calculate plw2 for p2, then you can easily get plw2:

\[ \text{plw2} = \text{plw1} \times \left(\frac{p1}{p2}\right)^2 \]

14. Before running your experiment, double check all pulse lengths and power levels and durations of decoupling periods. If you have any questions, ask Andy LiWang before starting your experiment at any hour of the day or night.

In the unlikely event of a magnet quench, there will be a massive and rapid jettison of helium gas from the top of the magnet. It is vital that you evacuate the magnet room and laboratory as quickly as possible and call Environmental Health and Safety at 209.228.4261.
Documentation of Training  (signature of all users is required)

• Prior to conducting any work with the chemicals listed above, designated personnel must provide training to his/her laboratory personnel specific to the hazards involved in working with this substance, work area decontamination, and emergency procedures.

• The Principal Investigator must provide his/her laboratory personnel with a copy of this SOP and a copy of the SDS provided by the manufacturer.

• The Principal Investigator must ensure that his/her laboratory personnel have attended appropriate laboratory safety training or refresher training within the last one year.

I have read and understand the content, requirements, and responsibilities of this SOP:

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