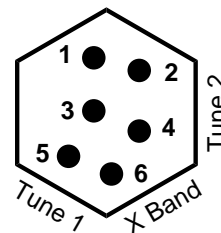


Daytona Basics!

Setting Up An Experiment

1. Before entering the NMR facility
 - Sign-up for Daytona time online using Daytona WebCal
 - If using N₂ on the probe then additionally sign-up using shared Siena/Daytona N₂ WebCal
 - NMR tubes should be clean
2. At instrument
 - Set up probe, shims, and probe channels prior to tuning
 - Click on Probe tab, load standard shims
 - Carefully center the liquid in the tube around the center line marked on the sample depth gauge
 - Load parameters for desired experiment and type su to set them up in the hardware
 - Tuning the probe
 - Use the minimum amount of force possible on tuning wands and cable connectors
 - Tune to 1/10 of original readout for good signal to noise; time spent reducing the value further will not help you
 - Tune box should be returned to channel 0 before leaving the pit

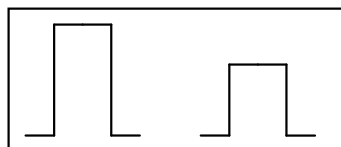
AutoX Probe Diagram



- 1 = ¹H Match
- 2 = ¹H Tune
- 3 = X Band
- 4 = X Tune 2
- 5 = X Tune 1
- 6 = X Match

Selecting a Quarter Wavelength Cable		
Short Cable	120 – 220 MHz	³¹ P, ¹¹ B, etc.
Medium Cable	60 – 130 MHz	¹³ C, ²⁹ Si, etc.
Long Cable	45 – 65 MHz	¹⁵ N, etc.

3. At the computer
 - Find Z₀ is automated using the button or under the acquisition drop down menu
 - Shimming
 - For best gradient shimming results use the “Set Up Gradient Shimming” tab
 - Load tab via Tools\Standard Calibration Experiments\Set Up Gradient Shimming
 - To determine whether gradient shimming will be successful, run “Acquire Trial Spectra”



Jagged? Temperature gradient!
 Flat profile? Inhomogeneous sample!
 or Z₀ is very off!

- 1/3 to 2/3 original height similar shape
- Run “Gradient Autoshim on Z”
 - Running gradient autoshim should lead to flat red and blue values along the x axis (rms err <1.00)
 - Must quit gradient autoshim prior to proceeding further
- Manual adjustments
 - Z₀ can be checked using the lock scan feature under Acquisition
 - Without spinning maximize lock by adjusting X₁, Y₁, XZ, and YZ
 - Lock phase adjustments may be necessary if doing variable temperature NMR

Running Experiments

nt=#	set number of transients	bs=#	set block sizes
sw=#	change spectra width	d1=#	change relaxation delay
ga or go	get acquisition, ga allows stoppage and resumption of acquisition	sa or aa	stop acquisition (allows you to restart acquisition), abort all acquisition
jexp#	jump to another experiment window	ra	resume acquisition
wft	weighted Fourier transform	f	displays full spectrum
aph	autophase	bc	baseline correct between integrated regions
lb=#	line broadening	vsadj	maximizes height of peaks in window
text('###')	annotates spectrum with text	svf('###')	saves spectrum and annotation
e	ejects sample	i	inserts sample

Variable Temperature

- Must stay with the instrument if adjusting the temperature
- Use Pyrex NMR tubes such as a Wilmad 535PP (sold in the chemistry stockroom). Most cheap NMR tubes are not Pyrex, they are soft borosilicate glass, and their manufacturers say they are not to be used for VT experiments.
- Use N₂ on probe whenever liquid nitrogen is used for cooling, or over 100 °C
 - N₂ is shared between Daytona and Siena and can be booked using the Nitrogen WebCal
 - High pressure N₂ cylinders are located by the lab door. Plan ahead as it generally takes 1 day of lead time to get replacements.
 - Probe should be under flowing N₂ for more than 5 minutes before starting heating or putting LN₂ on coils
- To start temperature regulation
 - Use the spin/temperature menu on the start tab
 - Type desired temperature in °C on the Temperature section and hit enter.
 - This should move the toggle, however, the temperature can also be adjusted using the toggle
 - Click “Regulate Temperature” button to start temperature changes
 - Can monitor current temperature using the panel and the “Current Temperature” readout
 - Walk to desired temperature
- Acquisition over variable temperature
 - Tune, lock, phase, and shims will all change with changing temperature
 - Changes, particularly in tunes, may be very dramatic
- Instrument must be brought to 25 °C at the end of the experiment
 - Don't drop LN₂ from the coils with a sample in the instrument