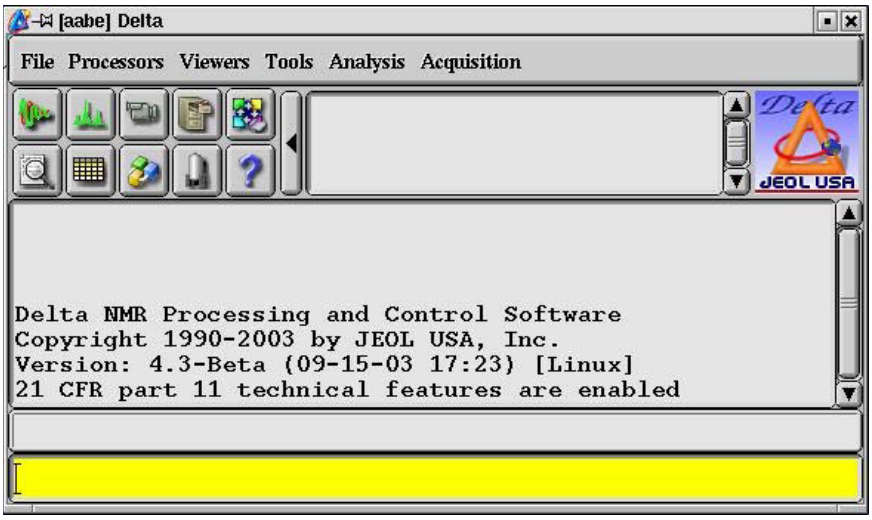
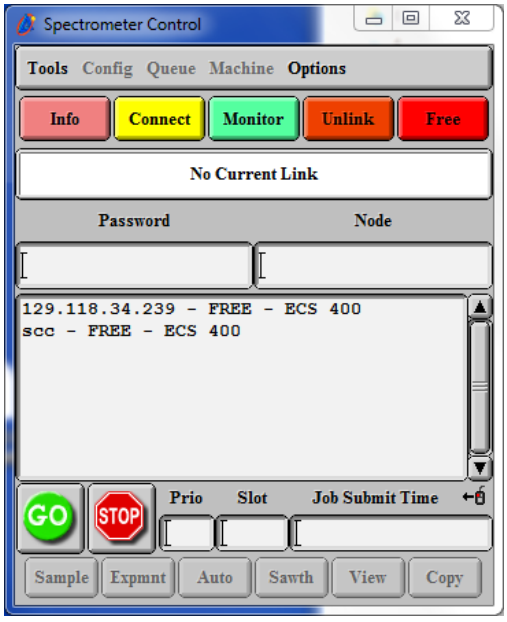
# JEOL ECS 400 - 1D NMR Measurement Set-up and Protocol

1. After logging in to your Windows account, double click the *Delta* icon  . The **Delta Console** window opens:

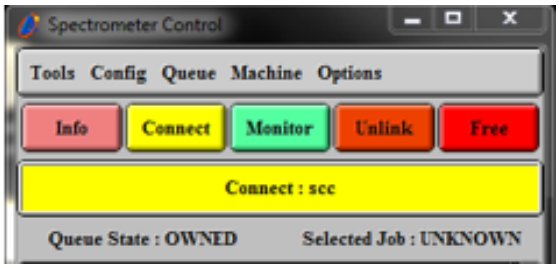



2. Click the  button in the **Delta Console**. The **Spectrometer Control** window opens and a list of the currently available spectrometers is displayed:



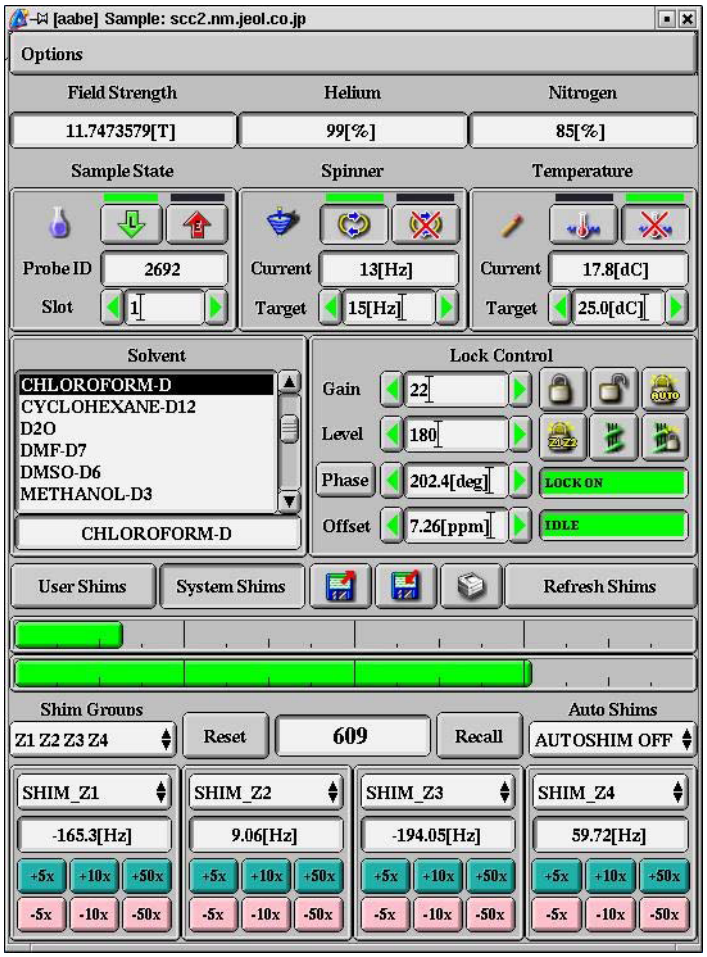

Eclipse 1 displays: **eclipse1 – FREE – ECS 400** while Eclipse 2 displays: **scc – FREE – ECS 400**. Select the spectrometer by clicking that line and then click the **Connect** button. The spectrometer marked as **FREE** is available for connection. The spectrometer marked **OWNED** or **ACTIVE** is currently being used and only the Monitor mode is available. If the console is available

and connection is carried out successfully, the message “Connect….” is displayed in the **Spectrometer Control** window, as shown below:

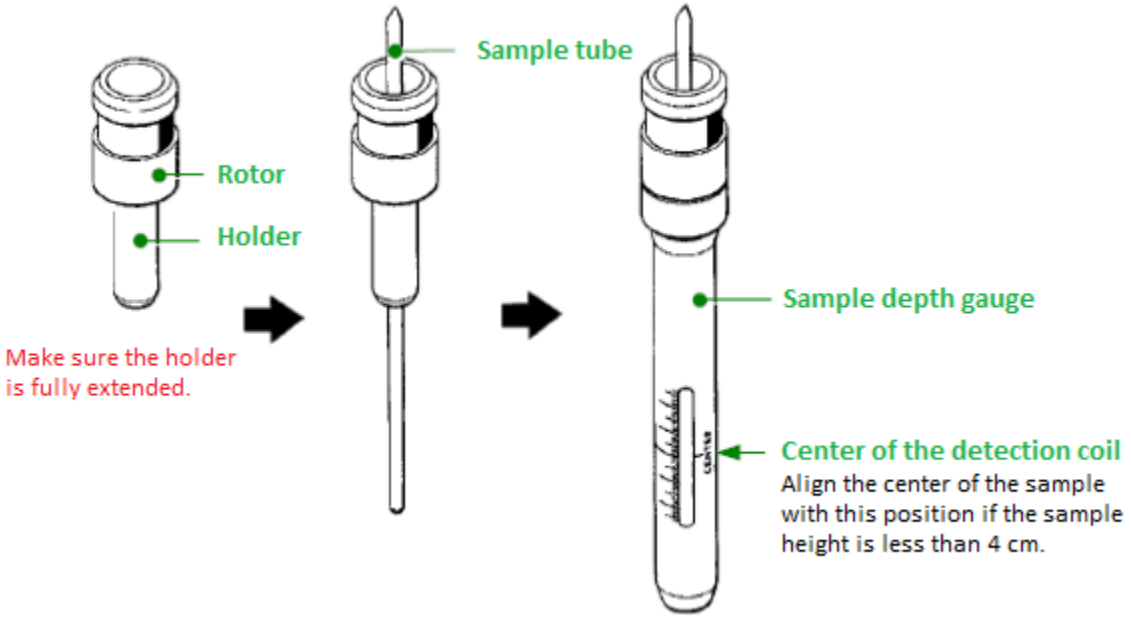



3. Click the **Sample** button in the **Spectrometer Control** window.


The **Sample** window opens:

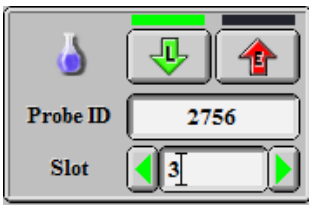


4. Set the sample tube (with the sample in it) in the holder, as shown below. Remember to adjust the depth of the inserted sample tube using the sample depth gauge. Since the resolution varies according to the sample volume (height), it is recommended that you standardize the sample volume. An appropriate sample height is approximately 4 cm (3.8 to 4.2 cm). If you use less solution, the quality of gradient shimming and resolution will be compromised.

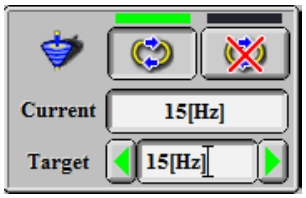



5. The manual method of loading the sample is recommended if you have only one sample. First, you need to click the  button in the **Sample** window which will eject the "standard" (empty tube - green cap). Then, replace it with your sample (i.e. sample tube in the sample holder/rotor) and load it using the  button.

   Alternatively, you can use the auto sample changer. Load the sample (sample tube in the sample holder/rotor) into any available slot of the auto sample changer (in this example, slot # 3). Enter the slot number into the **slot** field of the **Sample State** box as shown below. Remember that the cursor **MUST** be inside the field when you type – this is true for all Delta text boxes entries! Move the cursor out of the **Sample** window and the changer will load your sample from slot # 3 into the magnet.



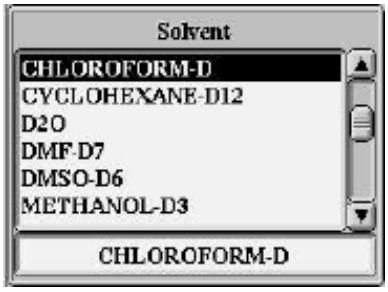

6. After the sample is loaded, it starts to spin. Spin rate should stabilize at 15 Hz – see **Spinner** box below. Wait until it spins at least at 11 Hz before proceeding to the next step.




After the sample load completes, the flask icon in the **Sample State** box becomes full: 

After the sample starts spinning, the toppled top will be upright in the **Spinner** box: 

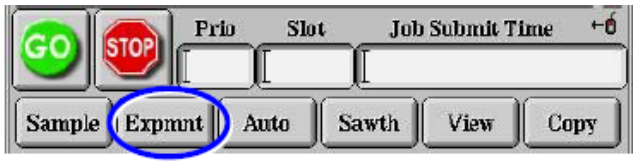
7. Select the solvent to be used for NMR locking from the **Solvent** list box as shown below:



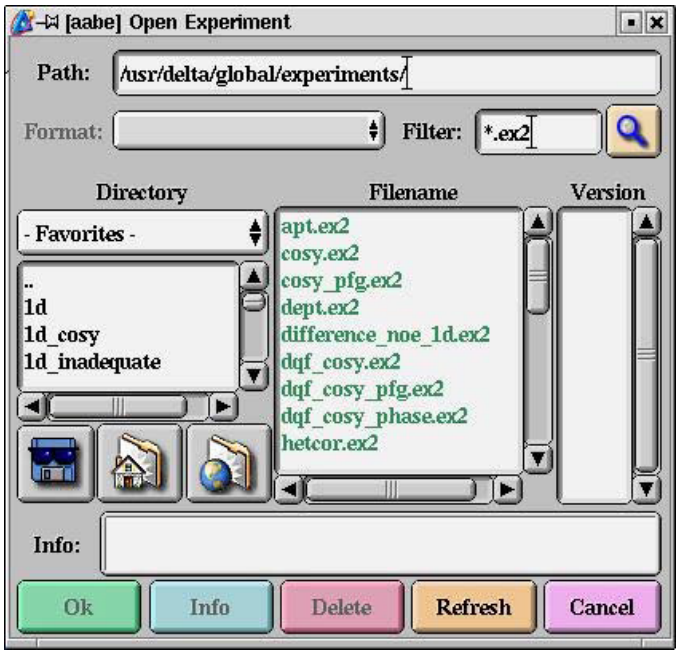

If the solvent you use does not appear in the list box, move the slider at the right side of the box

8. Load system shims by clicking the  button. This loads good starting shim settings for auto-locking and gradient shimming. These are shims optimized for the standard sample. If you skip this step Delta will use shims set by the last user of the system. They can be dramatically misadjusted if he/she used too short sample, "economy" NMR tube etc.
9. If the sample needs gradient shimming you can skip this step and proceed to the next one. Click the  button in order to start auto-lock. When it is done and NMR lock operates, the messages **LOCK ON** and **IDLE** are displayed at the bottom right of the **Lock Control** box:

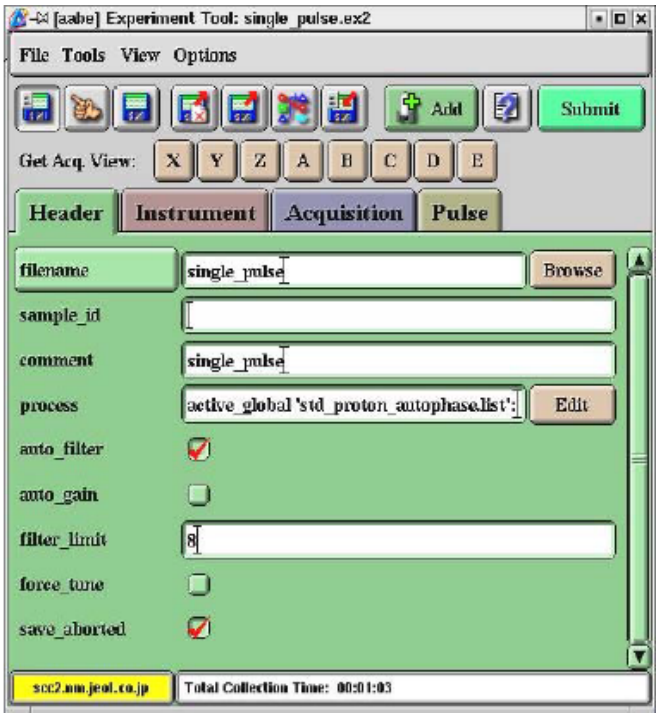
10. Click the  button in order to start gradient shimming, followed by auto-lock. **Spectrometer Control** window will start displaying gradient shimming data acquisition and when it is done, successful auto-lock will be confirmed by **LOCK ON** and **IDLE** in the **Lock Control** box above. **Spectrometer Control** window should be empty at that time, meaning: no data acquisition running.
11. Click the **Expmnt** button in the **Spectrometer Control** window.




The **Open Experiment** window opens:




12. Click the  button in the **Open Experiment** window. A list of the *Experiment* files in the *Global* directory is displayed. For standard proton experiment select the measurement mode **single_pulse.ex2** from the list and click the **OK** button. The **Experiment Tool** window opens:




The Experiment Tool window consists of the four sections (tabs): Header, Instrument. Acquisition, and Pulse. Acquisition parameters can be displayed by clicking on these tabs.

**13. Setting parameters for the $^{1}$H measurement.**

You can use most parameters as default values but you may need to modify the following:

From the **Header** tab:

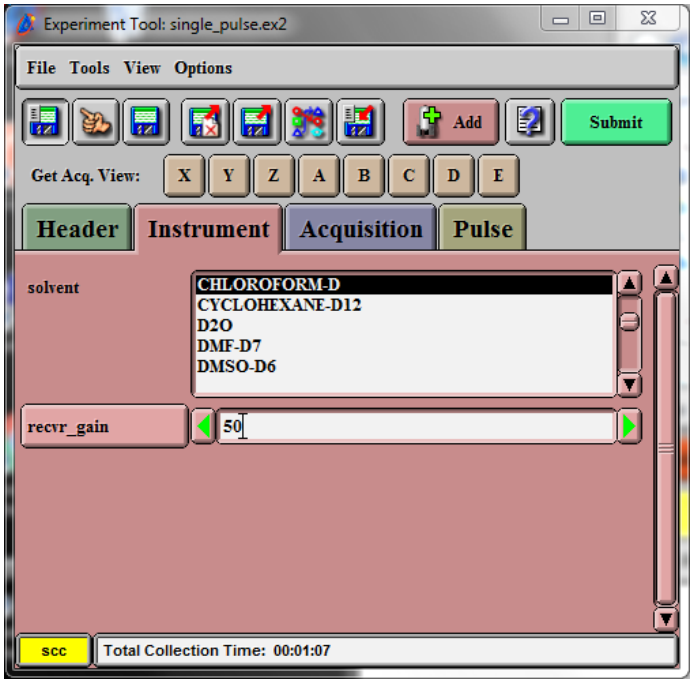
**filename** this name will be used by Delta for auto-saving your data

**comment** you can enter your comments here, it will be saved with NMR data

**auto_gain** for automatic receiver gain adjustment (recommended), click the button to mark it with a check mark. Use it only for proton data acquisition, not for $^{13}$C, $^{31}$P or other low-band nuclei.

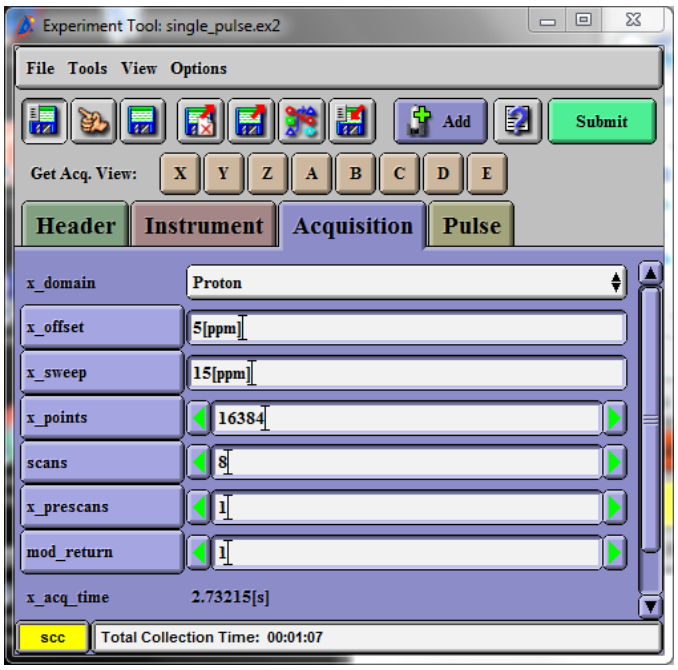
From the **Instrument** tab:

**solvent** select solvent from the list - it may be already correct (setup macro uses your lock- solvent selection)



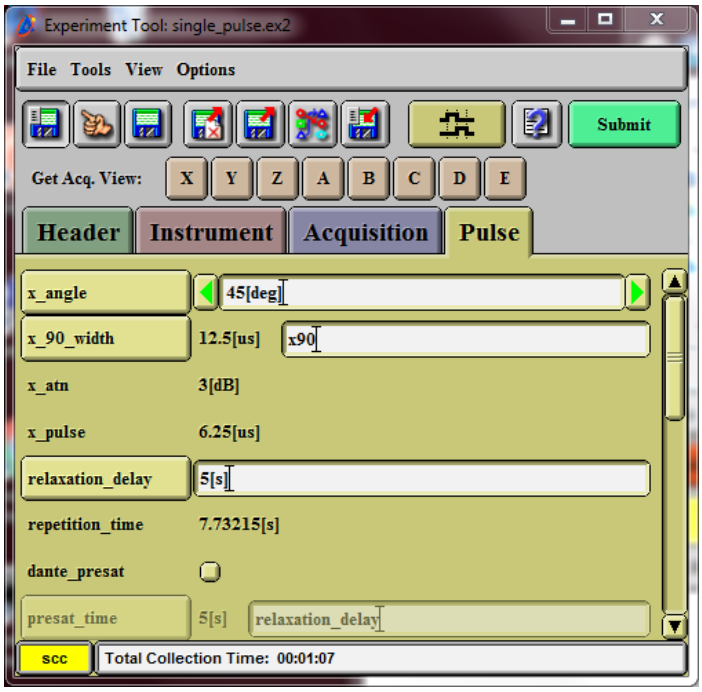

From the **Acquisition** tab:

**scans** leave default value of 8 or change it depending on sample concentration




From the **Pulse** tab:

**relaxation_delay** you can use default of 5 s but longer delay may be needed for accurate integration. Shorter delay can be used for faster data acquisition.




**Total Collection Time:** shows approximate time necessary for this measurement with current acquisition parameters

14. **Setting parameters for the $^{13}C$ measurement.**

Click the **Expmnt** button in the **Spectrometer Control** window. Then, click the  button in the **Open Experiment** window. A list of the *Experiment* files in the *Global* directory will be displayed. For standard $^{13}C$ experiment select the measurement mode **single_pulse_dec.ex2** from the list and click the OK button. The **Experiment Tool** window opens and allows you to adjust acquisition parameters. You can use most of them as default values but may need to modify the following:

From the **Header** tab:

**filename** this name will be used by Delta for auto-saving your data

**comment** you can enter your comments here, it will be saved with NMR data

From the **Instrument** tab:

**solvent** select solvent from the list - it may be already correct (setup macro uses your lock- solvent selection)

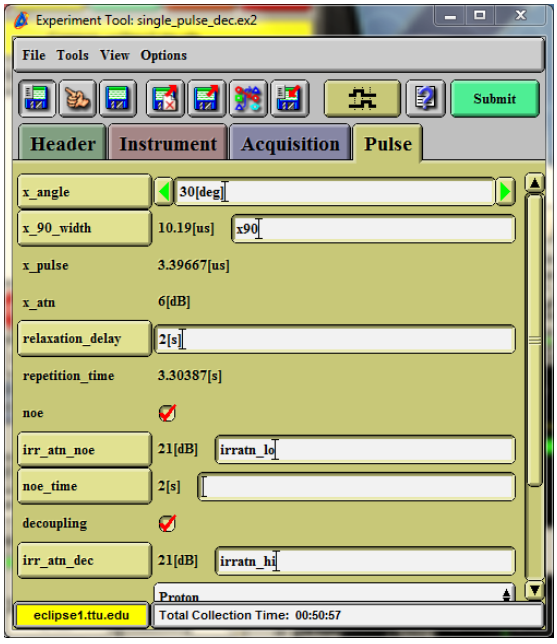
From the **Acquisition** tab:

**scans** $^{13}C$ is dramatically less sensitive NMR nucleus than $^{1}H$. **Scans** setting depends on sample concentration. Remember that signal to noise ratio is proportional to the square root of the number of scans which means that quadrupling **scans** setting only doubles signal to noise (which is not really an impressive improvement).

From the **Pulse** tab:

**relaxation_delay** very important setting if you need to observe $^{13}C$ nuclei with long T1 relaxation time constant (like certain carbonyl or quaternary carbon atoms). Sometimes even tens of seconds relaxation delay may be required. Otherwise, the signal would be missing, regardless of the number of scans. For most of the protonated carbons the default 2 s is sufficient.

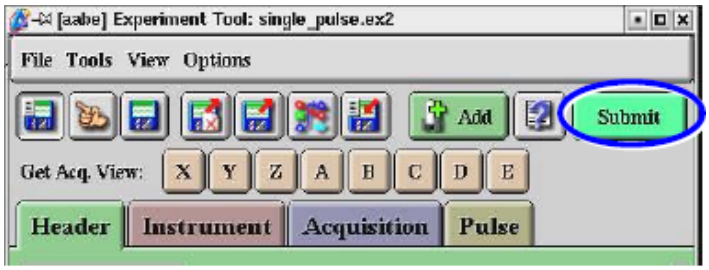
**noe** and **decoupling** these options are selected by default (see Experiment Tool window below) which means that broadband proton decoupling is on during both relaxation delay and acquisition time periods which yields the highest signal to noise ratio and this is what you generally need in the $^{13}C$ NMR spectrum. Deselecting **noe** will turn off decoupling during relaxation delay and suppress nuclear Overhauser effect (NOE). Deselecting **decoupling** will turn off proton decoupling during acquisition time, leaving some NOE $^{13}C$ signal enhancement but resulting in a proton-coupled $^{13}C$ spectrum. Deselecting both **noe** and **decoupling** will result in a proton-coupled $^{13}C$ spectrum with no NOE enhancement (lowest signal to noise).



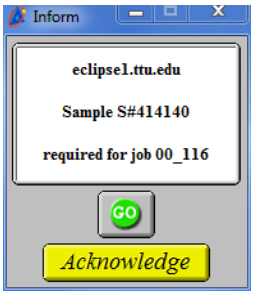

**Total Collection Time:** shows approximate time necessary for this measurement with current acquisition parameters. Always check that value before starting $^{13}C$ acquisition since this can be rather long experiment (many scans and long relaxation delay).

## 15. Starting data acquisition

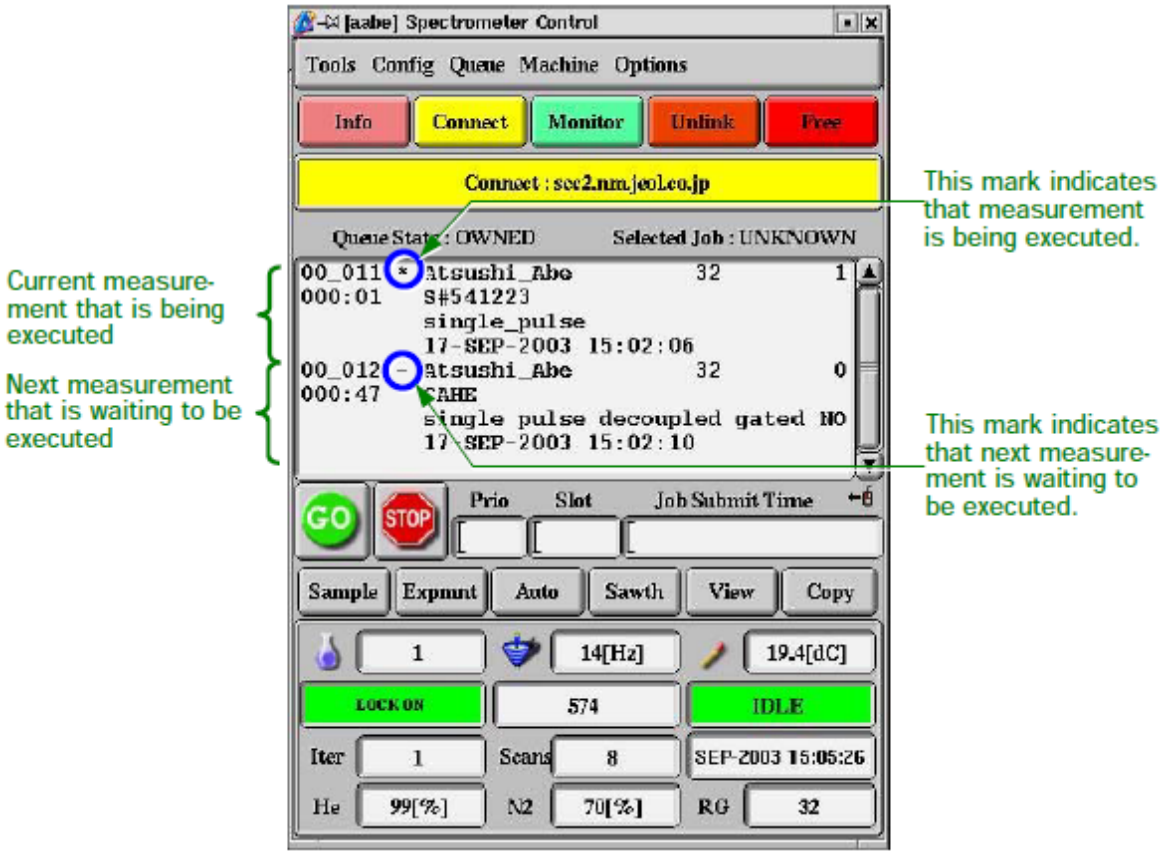
Click the **Submit** button in the **Experiment Tool** window.




Then, acknowledge experiment submission by clicking **Go** in the window shown below:




The experiment is entered into the spectrometer control queue (see below). The spectrometer executes measurement on a first-in, first-out basis. Commands for starting acquisition can be issued repeatedly, even if another measurement is being performed. After one experiment finishes, the data is auto-saved and the next experiment will be started, using the stored acquisition parameters.




16. Data acquisition process can be monitored by selecting corresponding line in the **Spectrometer Control** window and clicking the **View** button. It can display both raw, time-domain data (free induction decay) and frequency-domain spectrum (after Fourier transformation).

    If needed, the measurement can be aborted by clicking the 

button, after selecting the experiment in the **Spectrometer Control** window.
    Normally, when the measurement finishes (i.e. the requested number of scans has been collected), the **1D Processor** window opens and the corresponding line in the **Spectrometer Control** window disappears.

17. Load the "standard" sample into the magnet (it is usually stored in slot #1 of the sample changer). If you used the sample changer, enter number **1** into the **slot** field of the **Sample State** box and move the cursor out of the **Sample** window. This will eject the sample and store it in the sample changer. **Do not leave your sample there!**
    However, if you used the manual method for loading your sample, you need to eject it by clicking  and reloading the "standard" by clicking the  button.

18. When done, click the 

button in the **Spectrometer Control** window. Then, terminate the Delta program by selecting **File-Quit** from the **Delta Console** window and clicking **OK** in the **Confirm** window. You can now **logoff** from the Windows account.