Obtaining A "One-Click" Spectrum Using The JEOL ECX-300 NMR Spectrometer

Caution: The NMR magnet produces strong magnetic fields. If you depend on a medical device that is sensitive to magnetic fields DO NOT ENTER THE NMR ROOM. Do not approach within 5 feet of the magnet if you contain or have attached or are carrying iron or steel objects. Also, keep magnetic media, including credit cards, at least 5 feet from the magnet.

Sample Preparation & NMR Operation –

1. Place between 0.25 and 0.5 milliliters of the NMR solvent you are going to use in a small test tube. Duterochloroform, $CDCl_3$, is the most commonly used solvent; use it unless your sample is not soluble in it. Add ~50 - 100 milligrams of your solid or 3 - 4 drops of your liquid to the NMR solvent. Stir to dissolve your sample in the solvent. Using a Pasteur pipet transfer a little more than 0.25 ml of the solution to an NMR tube. The tube should be filled to a depth of between 4 and 5 centimeters. Cap the NMR tube.

2. Push the NMR tube into the spinner (Fig. 1). Position the NMR tube in the spinner using the depth gauge, pushing the NMR tube into the spinner until it bottoms out in the depth gauge, as shown to the right.

3. Place the NMR tube and spinner into a slot in the autosampler which is located on top of the NMR magnet. You may need to use the step-stool. Note the number of the slot into which you put your sample. (If the spectrometer is in use, see step #8 on page #3.)

Figure 1

4. At the computer console click on the Delta (Δ) symbol on the bar at the bottom of the screen. This will bring up the "Delta" window, shown to the right (Fig. 2). [Note: The operating system on this computer is Linux so the computer does not have exactly the same features as MS Windows. However, the KDE graphical interface is similar to Windows. The JEOL NMR Delta software runs the same way on Windows, Linux and Mac boxes; it is platform independent.]
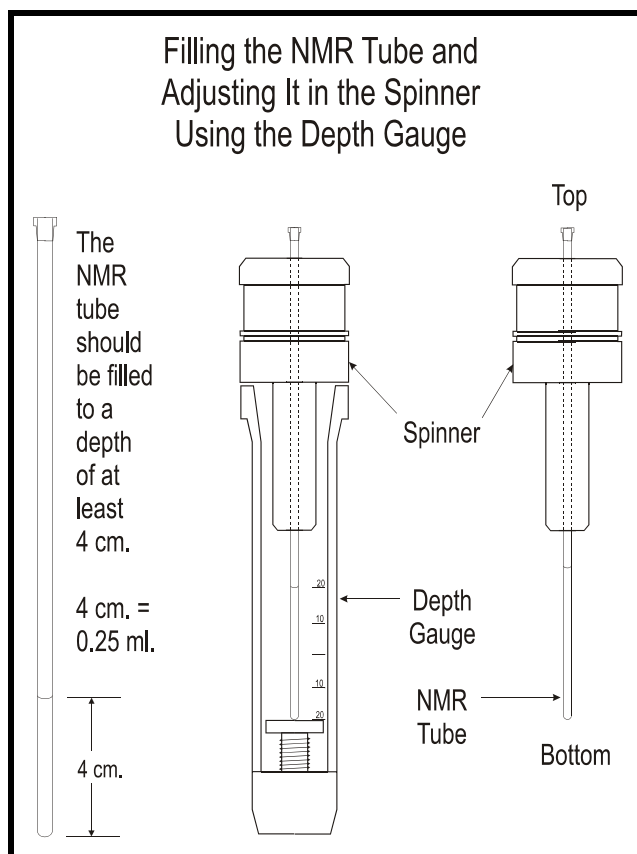

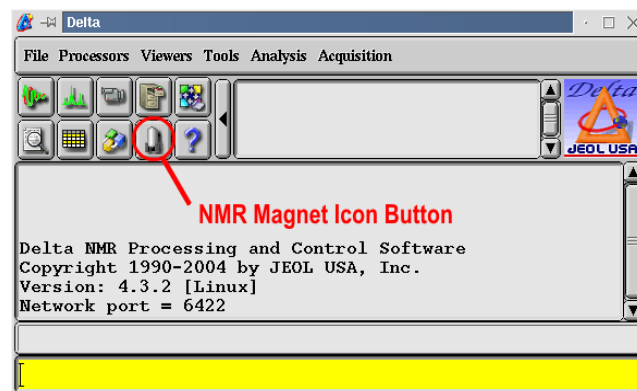Figure 2 - The Delta Window

5. Click on the NMR Magnet Icon Button. This brings up the "Spectrometer Control" window, shown to the right (Fig. 3). In this "one-click" mode a built-in script causes the spectrometer to be selected and placed under the control of the Delta software automatically.

6. Next, click on the "Auto" button at the bottom of the "Spectrometer Control" window. The "Automation" window (Figure 4) will appear.

7. Place the cursor over the white area next to where you see "Filename:" and enter a filename. (For example, you might use your name followed by a number. You will then record in your lab notebook what sample corresponds to this filename.) [To enter text into a text box in the JEOL Delta software running on Linux it is necessary for the mouse cursor to be over the text box.] Enter the number of the autosampler slot that contains your sample and select the solvent that you are using. If you wish to have the free induction decay (FID) data e-mailed to yourself, enter your e-mail address into the "Export to:" box. [At some future date the Delta software will be installed on other computers in the Physical Science building. If you import the FID file into the software you will be able to process it as if you were sitting at the computer that controls the NMR.]

8. To obtain the spectra you want, press the corresponding buttons in the khaki colored area of the automation window. Each button you press will add that experiment to the queue to be executed in turn. Suggestion: If others are waiting to run spectra, limit yourself to the "Proton", "Carbon and DEPT135", and "Edited DEPT triad or to *one* of the other buttons. The computer will cause the autosampler to insert your sample into the spectrometer and will perform a gradient autoshim of the spectrometer before obtaining the spectra.
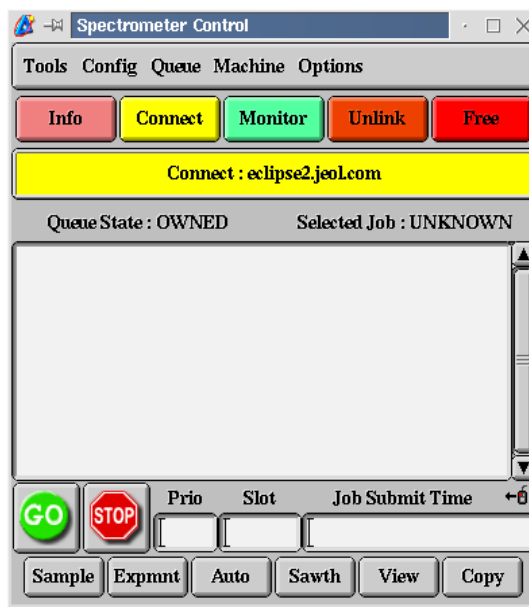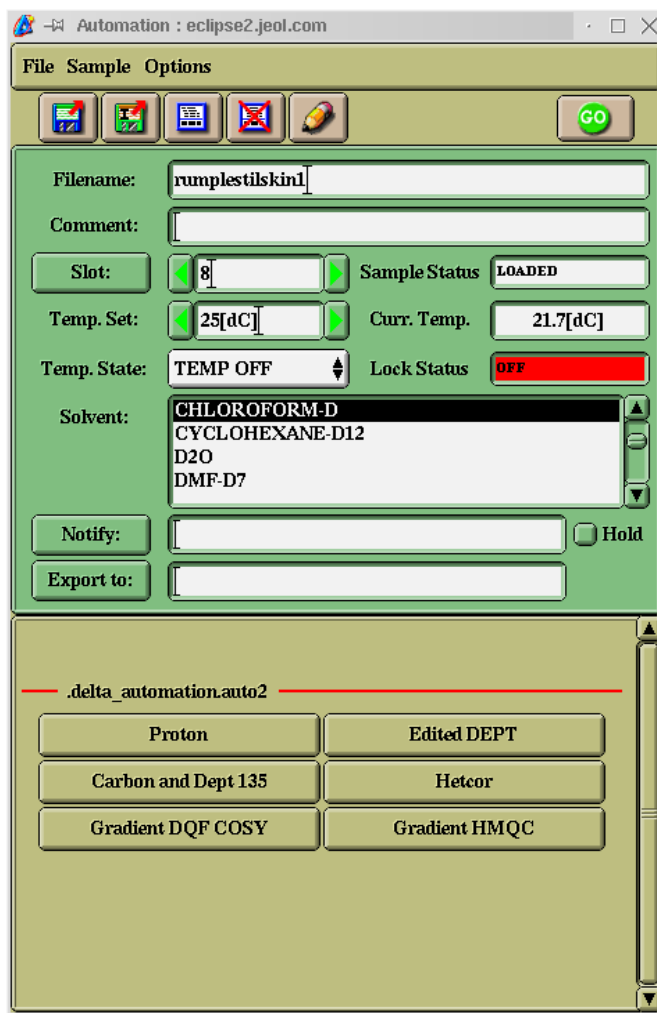
Figure 3 - Spectrometer Control

Figure 4 - Automation Window

If spectra are being run when you get to the console, insert your sample into an empty slot in the autosampler, remember its number, and start at step 7. One or more experiments are already in the queue and by entering your information, you are submitting your job(s) to the queue and they will be run in order.

When your job(s) have run, remove your sample(s) from the autosampler and place the spinner in the spinner holder. Remove the sample from the NMR tube, rinse the tube with ordinary acetone, and place it upside down in a beaker to dry.

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