APT on the U400

PRELIMINARY INFORMATION

1) The APT (Attached Proton Test) experiment gives quaternaries and methylenes up and methines and methyls down. Solvent peaks will appear as positive signals as well. Sensitivity wise, the APT experiment is about the same as the regular ^{13}C experiment. Therefore, an APT spectrum will take about the same amount of time to acquire as a normal ^{13}C spectrum.
2) An alternative to the APT experiment is the DEPT experiment, which will provide the same information, and more. For example, a DEPT-135 will null quaternary carbons (thus identify them on comparison with the normal ^{13}C spectrum) and give methines and methyls up and methylenes down. In addition, if necessary, a DEPT-90 would distinguish methines from methyls, which is not possible with the APT experiment. Moreover, the DEPT experiment is considerably more sensitive than the APT experiment.

1. COLLECT A NORMAL ^{13}C SPECTRUM

† If you have already collected a ^{13}C spectrum previously, load the data into an experiment (*e.g.*, exp1) and process it as usual. Note that when set up this way, the APT spectrum may have to be phased manually.

† If no spectrum has been acquired, acquire one and save and process the data as usual.

2. SET UP THE APT EXPERIMENT — doapt

INTRODUCTION: **doapt** is an interactive macro that will take you through the setup of an APT experiment. In this handout, the dialog will appear **`in this font`**, and the symbol ¶ signifies that a response is required. Most messages will appear in the Status Window, while inputs are entered *via* the Input Window. Window arrangement on the left side of the screen is, from top to bottom, Status, Input, Menu, Graphics, and Text.

2A. Enter the Macro

In the experiment that contains the normal ^{13}C spectrum, enter:

doapt <rtn> macro to set up an APT experiment in exp2

NOTE: If you wish to do the APT in a different experiment, enter **doapt(*exp#*)**, where ***exp#*** is the experiment number for the APT. The **doapt** macro requires that the experiment number be between 1 and 9, excluding 5.

If the APT exp# is the same as the current experiment, the following dialog will appear:

`Do apt in current experiment? (y/n) ¶`

If answered no:

`Enter apt exp#: ¶`

Then the APT experiment will be set up, using the same *nt* and *d1* as the ^{13}C experiment. If you wish to change either *nt* or *d1*, answer the following question accordingly:

`Do you wish to change nt and/or d1? (y/n) ¶`

If answered yes, enter the new values at the following prompts:

`nt= ¶`
`d1= ¶`

The **doapt** macro will terminate at this point. Be sure to check the parameters before starting the experiment.

2B. Start the Experiment

You may start the APT experiment with the **au** or **ga** command:

au — start APT experiment and process the data automatically with the **aptproc** macro when acquisition is complete. NOTE: If the experiment is terminated with **aa** or **sa** (see below), no data processing will occur.
ga — start APT experiment and do a **wft** when acquisition is complete.

NOTE: Once the experiment is started, it may be aborted by clicking the `Abort Acq` button or typing the **aa** command *if no data is to be retained*, such as when you discover that a mistake has been made in the setup of the experiment, or when it has become apparent that the experiment is useless. *If data retention is desired*, such as when sufficient signal-to-noise has been obtained or when you have run out of time, you should use the **sa** command to stop the acquisition. At this point, you should process the data manually because automation will not work when the experiment is terminated before its completion.

When the experiment is finished, save the data and proceed with its processing, displaying, and plotting as described in Part 3 below.

3. PROCESSING, DISPLAYING, AND PLOTTING OF APT DATA

3A. Processing

aptproc <rtn> — macro to process APT data, which does a wft and adjusts *vp* and *vs* as well.

NOTE: If the APT spectrum is not in phase, adjust it manually. *Do not use the **aph** command to phase the APT spectrum!* When phasing, you should use the solvent and any other known signals as the criterion and make sure that the peaks that are supposed to be up are up (*i.e.*, C's, CH_2's, and solvent peaks) and those that are supposed to be down are down (*i.e.*, CH's and CH_3's).

3B. Displaying

aptds <rtn> — macro to display the APT spectrum with reasonable *vp* and *vs* settings

3C. Plotting

aptplot(C13exp#) <rtn> — macro to plot the APT and the normal ^{13}C spectrum on the same page. **C13exp#** is the experiment number that contains the normal ^{13}C spectrum.

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If no argument is provided with the **aptplot** *command, the following dialog will appear:*

`Plot without 13C spectrum? (y/n) ¶`

If answered yes, the macro will terminate here and only the APT spectrum will be plotted (see example in Fig. 1).

If answered no, the dialog will continue:

`Enter exp# containing 13C spectrum: ¶`

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Finally, you are provided with the option to plot the text:

`Plot with text? ¶`

Then the plotting will begin (see example in Fig. 2).

Fig 1. **aptplot:** ***APT without ^{13}C spectrum***

Fig 2. **aptplot:** ***APT with ^{13}C spectrum***