8. $^{13}$C NMR on AM-500/ACs
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I. Sample In
• After putting the sample into the probe, turn the air valve to VT reserve (air - routine heteronuclear; N$_2$ - low temperature NMR) and cap the probe. Adjust AIR FLOW to ~5 if necessary (this cools the decoupler).

**AIR FLOW**: 0-15 range (instead of 0-5 on WP-270).
• Lock as usual before probe tuning (shim after tuning the probe).

II. Probe Tuning
(not needed on AC spectrometers)
• This is an electrical adjustment of the probe, which can affect the shims but is not part of the shimming process.
• Make certain that there is a 2dB attenuator placed on the POWER OUT channel! This is important in creating pulse sequences with correct pulse widths.

**RJ C13.SET; II; ZG** (not GS) to create the rapid frequency pulse (~200 pulses/sec) for the probe tuning.

**BB Preamp**: 2, D and 3 for $^{13}$C (125MHz); see plate on preamp for appropriate setting.

**Initial Setting**: tuning slide (z) at 7995.5; matching slide (z2) at 992 for $^{13}$C; see card on probe for correct settings.
• Minimize the value in **LOAD MATCHING METER** on the side of the console by match-tuning the two slides under the probe. When match-tuning, adjust only the last digit on the slides by 0.5 unit at a time. Note that the change in the tuning slide acts in opposite direction to the change in the matching slide.
• For $^{13}$C, the final **LOAD MATCHING METER** reading should be approximately 0.02mA. For other sensitive nuclei, it should be <0.05mA.
• **<ctrl> H** to halt acquisition and to go on to decoupler tuning.

III. Decoupler Tuning
(not needed on AC spectrometers)
(The principle is same as in probe tuning; however, the decoupler operates on the $^1$H at 500MHz. The objective of this tuning is to minimize the reflected power.)

• Set the decoupler power (**DP**) approximately 10 times higher than the power actually used for decoupling. The **DP** value represents an attenuation; thus if you want to run with **DP**=16, set **DP**=6 for the next step. Note that **DP**=0 is the decoupler at full power, which can damage the probe!

**RJ C13ACET.1DJ** (for acetone-d$_6$; **CPD; II; DP** = 10H; then, go under the probe and find two screws M (matching) and T (tuning) for the decoupler. Carefully adjust T (longer screw) first and, if necessary, M until the intensity of **DEC REFLECTED** light on the switch panel decreases and finally goes off.)
• The switch panel has three positions: (1) DEC FORWARD - used to verify the decoupler output; (2) DEC REFLECTED - initially this red light is on, but want it "off" ultimately; and (3) OBS REFLECTED - verifies output on the X transmitter (e.g., $^{13}$C output).

IV. Shimming

(Exactly same procedure as in $^1$H NMR, but with only one difference: somewhat higher lock power is required for the heteronuclear case because two independent coils—heteronuclear observe coil and decoupler $^1$H coil—are used instead of one.)

• DP = 20H
• Match-tune $z$, $z^2$, and $z^3$. Do not change $z^4$. Do not use Autolock or Autoshim.
  
  Autolock: usually lock power is increased (used for long runs).
  Autoshim: used for overnight runs. (hit $z$, then, Autoshim to activate.)

• <STD BY> when shimming is done.

V. Taking $^{13}$C NMR Spectrum

• RJ C13ACET.1DJ; SW = 28900; O1 = 13350 (-10 to 220 ppm); O2 = 7500 (for CDCl$_3$; O2, not O1, varies from solvent to solvent!); CPD (composite pulse decoupling sequence); NS = -1 (infinite number of scans, or set to number if known to give sufficient sensitivity); II; ZG.

• From time to time, transfer the acquired FIDs to another Job and process it to see if enough FIDs have been collected. This can be done in the following manner:
  
  • TR = 3 (if FIDs are being collected in Job 1 or Job 2); LB = 2; EF; EP.
  • Repeat this until satisfactory sensitivity is obtained. Then, <ctrl> H; WR <filename>.

VI. DEPT

• While the normal $^{13}$C spectrum is being acquired in Job 1, switch to Job 2 for setting up the DEPT experiment.

• RJ DEPTACET.1DJ; SW = 28900; O1 = 13350 (-10 to 220 ppm); O2 = 7500 (for CDCl$_3$; O2, not O1, varies from solvent to solvent!); do not II if the data are being accumulated in another Job

• AS DEPT.AU – parameters for DEPT.AU microprogram:

  D1 = 3 (sec)
  S1 = 0H (max. decoupler power)
  P1 = 12.5 (90° pulse in µsec in decoupler channel; see pulselength sheet for newest calibration)
  D2 = 3.3 to 3.6M (1/2J)
  P2 = 25 (2×P1)
  P3 = 9 (90° pulse in µsec in observe channel; see pulselength sheet for newest calibration)
  P0 = 18.7 for DEPT-135: 1.5×P1 = 135° pulse; CH and CH$_3$’s are positive, CH$_2$’s are negative.
  12.5 for DEPT-90; CH positive, CH$_2$ is zero (tuning experiment), CH$_3$ close to zero.

  P4 = 18 (2×P3)
  S2 = 20H (decoupler power)
  RD = PW = 0
  DE (set automatically by the instrument)
  NS = -1
DS = 0
P9 = 110.0 (see pulsewidth sheet for newest calibration of 90° at DP power)

• Then, II; ZE; AU to run the DEPT microprogram.
• Check the acquisition from time to time as in the normal case as shown above. Then, <ctrl> H; WR <filename>.

Caution: Before you leave, make certain that you have turned OFF the decoupler! If you have not, use PO to turn it off.

VII. Data Processing

• Process the FIDs of the normal spectrum in the following manner: Set LB = 2 (Hz); EF; DPO (digital plotter options; to set the plot parameters); CX (the width of the spectrum in cm); CY (the height of the tallest peak in the spectrum in cm; 0 autoscales); EP (phase the spectrum, put it in memory and plot); <ret>; MI (minimum intensity in cm; try 1 cm first); activate the printer; PP (peak pick; prints the peak positions on the line printer and/or on the display); SR (spectral reference: the numeric value of O1 for 0 ppm).

[CX = 20; CY = 13 for a thesis-size spectrum.]
• Process the DEPT FIDs in the same manner as above, i.e., set LB = 2; EF; DPO; CX; CY; SR (set this equal to the previous numerical value to align both spectra); EP (phase, save, and plot using S to put the DEPT spectrum just above the previous normal spectrum).
• To improve the apparent S/N ratio, one can use PS (power spectrum) command. This will square every data point in the spectrum so that all peaks (and noise!) will be positive. Also note that there may be some broadening at the base of peaks if a dilute sample is used.