Technical Bulletin L0410
Direct Carbon ^{13}C Detection using the CapNMR™ Probe on Varian Spectrometers
01 Feb 2005, AGW, MRM

Some of our users have demonstrated direct carbon observation using our CapNMR probes. While not optimized for direct detection of carbon, the CapNMR probe is capable of observing ^{13}C. This application note details the methods these users have reported.

Prerequisite
The spectrometer must be configured for ^{13}C detection. This may be verified using a conventional probe. Many systems have a wide-band (X) channel that can be tuned to allow ^{13}C detection.

Sample
A popular model compound for ^{13}C examination is 60% Benzene-d_6 40% Dioxane (protonated). Note that 60% Benzene-d_6 40% Acetonitrile(protonated) provides similar information and is typically more readily available. Either sample will allow calibration of proton decoupling as well as provide a reference S/N from the Benzene-d_6 triplet. These samples have long T1 relaxation times (4-5 minutes, typ.), so shorter pulse widths (e.g. 45^o or 30^o) may provide acceptable results with shorter times for excitation recovery.

Spectrometer setup
Provided that the spectrometer of interest is configured for ^{13}C detection, there is no need for re-cabling for use of the CapNMR probe. The following steps should be followed:

1) Start by creating a standard s2pul single proton pulse sequence.
2) Enter rtp('/vnmr/test/C13sn') to load the C13sn direct carbon parameters. This should load the parameters from your probe file, but this step must be checked manually. Pay special attention to check that following transmitter powers are obtained:
    a. tpwr < 45 dB,
    b. pwxlvl < 45 dB (may not exist on some experiments)
    c. dpwr < 35 dB
3) Set the receiver gain to maximum. If a receiver overflow is encountered, step down the gain setting by 6 dB until the overflow message stops.
4) Recalibrate the X channel pulse widths using the power settings (pwxlvl and dpwr) of step (2). **Do not assume the value of X-channel pulse width obtained during probe installation(indirect detection) is accurate for a direct detect experiment.** Use standard approaches to array the pulse width parameters to find the appropriate pw90's.

At this point you should be able to take direct carbon data. Note that the sensitivity of natural abundance carbon is 4 orders-of-magnitude less than the sensitivity of proton, and that S/N varies with the number of scans squared. Consequently, the number of scans required for ^{13}C detection may be significantly greater than that required for ^{1}H detection. Below is an example of a direct carbon spectrum taken by one of our users. (See Figure 1 below)

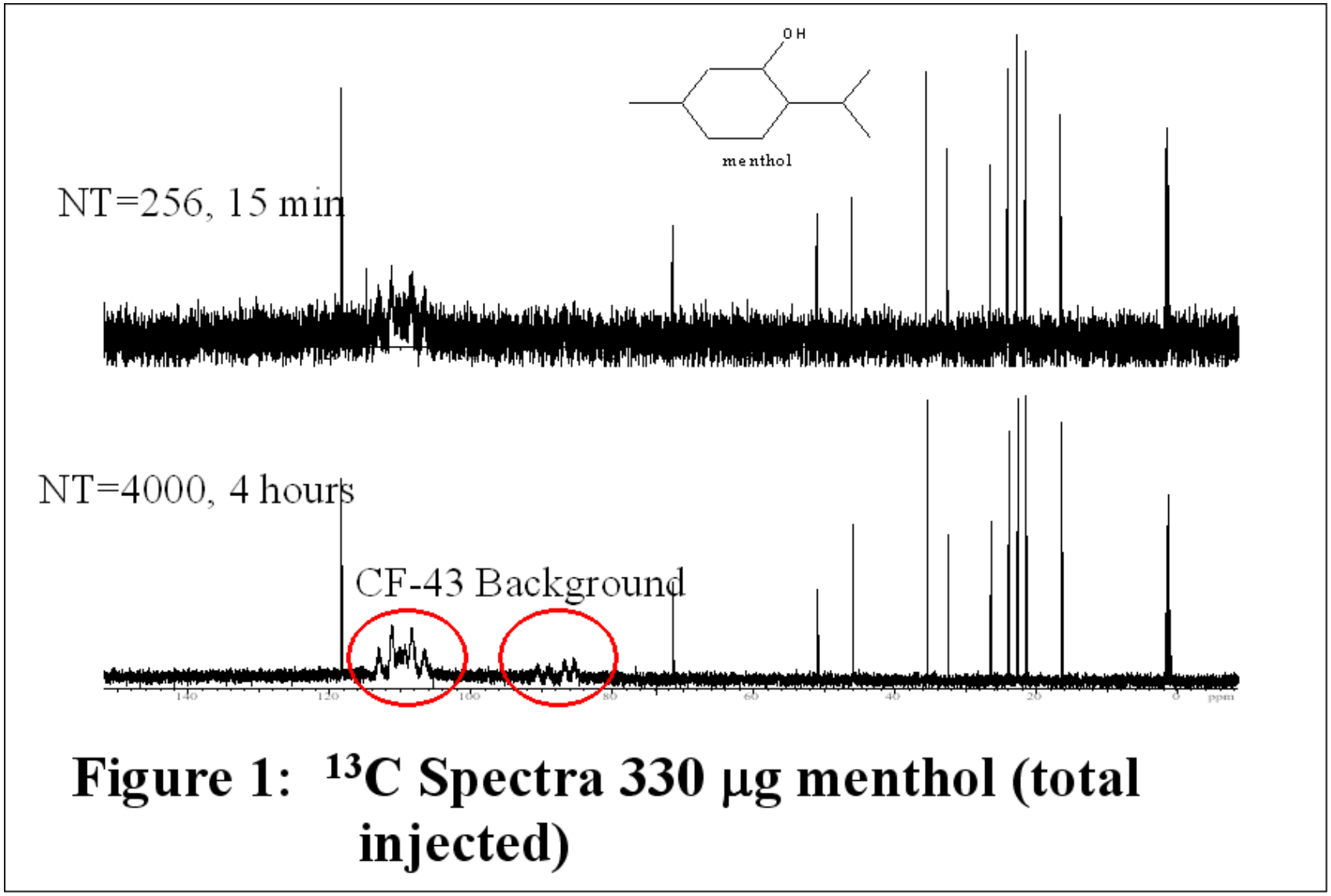

Figure 1: ^{13}C Spectra 330 µg menthol (total injected)

Data Courtesy of Andreas Kaerner, Eli Lilly