KJM 5250 and KJM 9250

One Dimensional SELHSQC and SELHMBC Experiments on the AVneo400 Spectrometer. ^{1}H detected with selective ^{13}C excitation.

Version 3.1
Topspin 4.3

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March 2024

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March 2024

AVneo400 SELHSQC + SELHMBC Experiments

1.1 Introduction

Prior to running **SELHSQC** and **SELHMBCCT** experiments determine the frequency of the ^{13}C signal to be selectively excited in Hz and enter this value as **O2 (Hz).**

SELHSQC and **SELHMBCCT** experiments use a selective (soft) 180 degree refocusing **Q3 type** pulse.

The use of ^{13}C decoupling during FID acquisition in **SELHSQC** experiments on the Neo-400 results in a modest degree of probe warming with some loss of shim and resolution drift unless the FID acquisition time is short (ca 0.5 sec). Accordingly the **awselhsqc** experiment is set up as a 15.6 ppm ^{1}H window expt with 6400 data points (AQ = 0.512 sec) which when processed can be zero filled to 16-32K points (your choice) with optional linear prediction to 12-16K data points.

Decoupling related probe heating does not occur when running **SELHSQCND** experiments (ND = not decoupled).

The **SELHMBCCT** experiment (which does not use ^{13}C decoupling) **_only_** *affords correlations ex quaternary (s) carbons.*

1.2 Signals with short T$_2$'s

^{13}C signals with short T_2's may give no result in a **SELHSQC** or **SELHMBCCT** experiments run with a **180º inversion p36 = 40000 usec Q3 pulse** in which case (*after* using the getprosol command) one can try *halving* the **p36** pulse time to **20000 usec** and *subtracting* **6 db** from its **sp26(db)** *power level.*

1.3 SELHSQC and SELHMBCCT Experiments

The following 1D-Selective **HSQC** and **HMBCCT** experiments have been set up on the **Ne0-400** spectrometer.

2.1	SELHSQC	with ^{13}C decoupling during AQ
2.2	SELHSQCND	without ^{13}C decoupling
2.3	SELHMBCCT	quaternary carbons only

2.1 SELHSQC Spectrum

Parameter set: **awselhsqc** (+ getprosol)
Pulse programme: **awselhsqcgpsisp**

 Prior to running a **SELHSQC** experiment run a standard **^{13}C** or **DEPT** experiment and determine the **O1** frequency of the **^{13}C signal** in **Hz** to be selectively excited. Enter this value as **O2 (Hz***)   (Important)*

TD = 6400, **SI** = 16 or 32 K.
Check **AQ** is ~ 0.5 sec (or less*) (Important)*
SW = 15.6 ppm, **O1P** = 6 ppm.
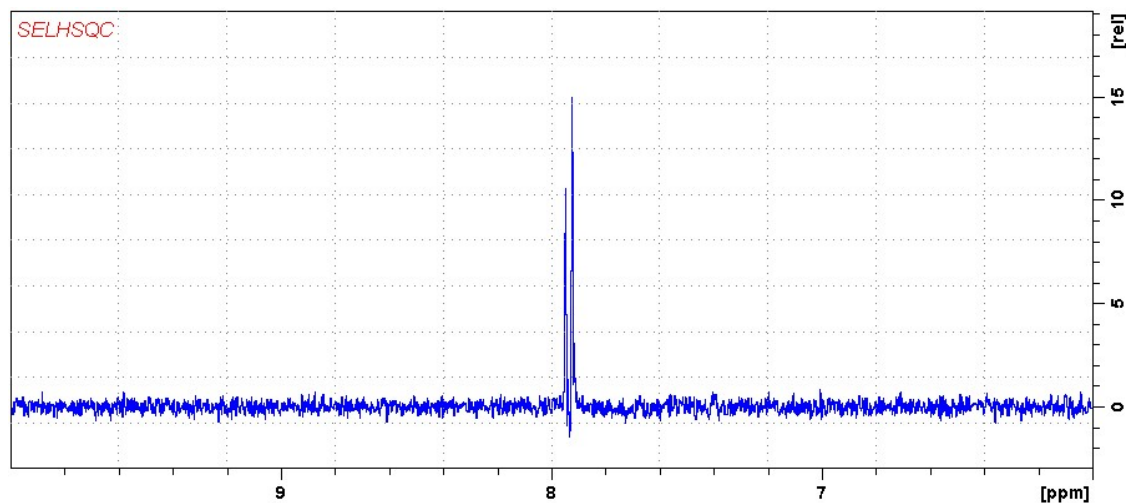**NS** = multiple of 8 or 16, **DS** = 4 or 8.

D1 = 1.5-2 sec or other value of your choice.
D24 is automatically calculated from **CNST2** ($^1J_{C-H}$).
CNST2 = $^1J_{C-H}$; typically 125 to 160 Hz for sp^3-sp^2 carbons. Furan or pyrrole ring carbons adjacent to hetero atoms will have ^1J = 200-220 Hz.

Shaped pulse types and powers are read in by the **getprosol** command.

Process with **EFP** (applies **LB,** typically use 0.3- 0.5 Hz).

Neo-400 **SELHSQC** spectrum of quinine in D_6-DMSO (6-10 ppm region) with selective excitation of the ^{13}C signal at 131.6 ppm (**O2 = 13244.1 Hz**). The ^1J correlated proton signal of this carbon occurs at 7.95 ppm.

2.2 SELHSQCND Spectrum

Parameter set: **awselhsqcnd** (+ getprosol)
Pulse programme: **awselhsqcndgpsisp**

 Prior to running a **SELHSQCND** experiment run a standard ^{13}C or **DEPT** experiment and determine the **O1** frequency of the ^{13}C **signal** in **Hz** to be selectively excited. Enter this value as **O2 (Hz***)   (Important).*

TD = **SI** = 16 or 32 K.
SW = 15.6 ppm, **O1P** = 6 ppm.
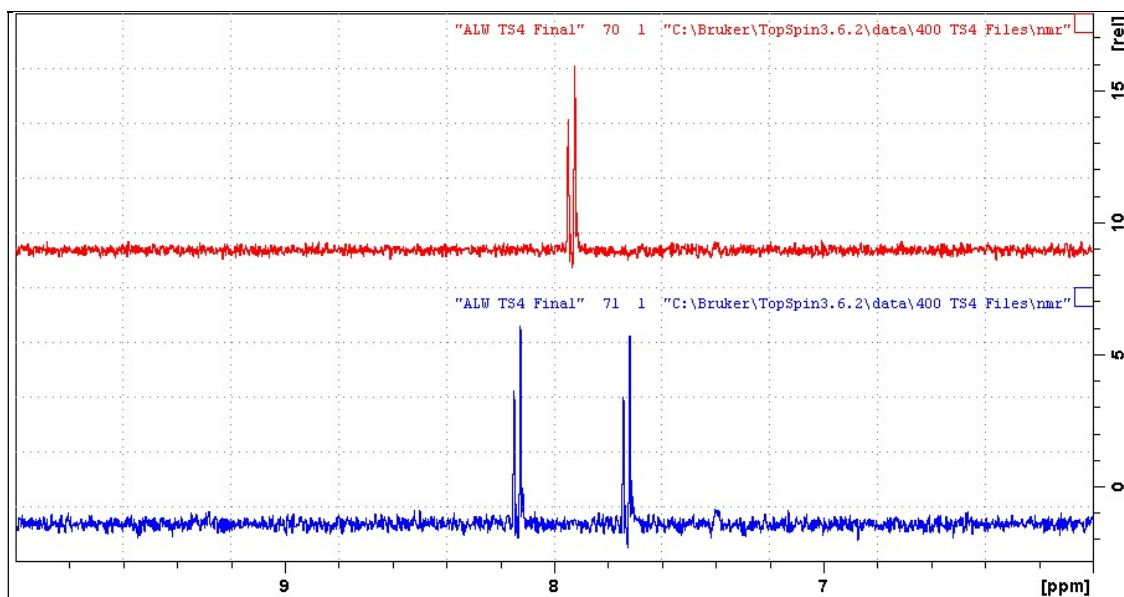**NS** = multiple of 8 or 16, **DS** = 4 or 8.

D1 = 1.5-2 sec or other value of your choice.
D24 is automatically calculated from **CNST2** ($^1J_{C-H}$).
CNST2 = $^1J_{C-H}$; typically 125 to 160 Hz for sp^3-sp^2 carbons. Furan or pyrrole ring carbons adjacent to hetero atoms will have ^1J = 200-220 Hz.

Shaped pulse types and powers are read in by the **getprosol** command.

Process with **EFP** (applies **LB**, typically use 0.3- 0.5 Hz).

Lower: Neo400 **SELHSQCND** spectrum of quinine in D$_6$-DMSO (6-10 ppm region) with selective excitation of the ^{13}C signal at 131.6 ppm (**O2 = 13244.1 Hz**).
Upper: ^{13}C decoupled **SELHSQC** spectrum of quinine

2.3 SELHSQCCT Spectrum

Parameter set: **awselhmbcct** (+ getprosol)
Pulse programme: **awselhmbcct**

 Prior to running a **SELHMCCT** experiment run a standard ^{13}C or **DEPTQ** experiment and determine the **O1** frequency of the ^{13}C **signal** in **Hz** to be selectively excited.
Enter this value as **O2 (Hz***)   (Important)*

TD = **SI** = 16 or 32 K.
SW = 15.6 ppm, **O1P** = 6 ppm.
NS = multiple of 8 or 16, **DS** = 4 or 8.

D1 = repetition delay = **1.5 sec** or other time of your choice.
**CNST6** = min ^1J coupling constant = **120 Hz** or other value of your choice.
CNST7 = max ^1J coupling constant = **170 Hz** or other value of your choice.
CNST13 = ^nJ selection filter = **8 Hz** or other value of your choice.
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Shaped pulse types and powers are read in by the **getprosol** command.

Process with **EFP** *and* **MC** (applies **LB,** typically use 0.3- 0.5 Hz).

awSELHMBCCT NB: Only use for quarternary (s) carbon correlations
Process with EFP and MC
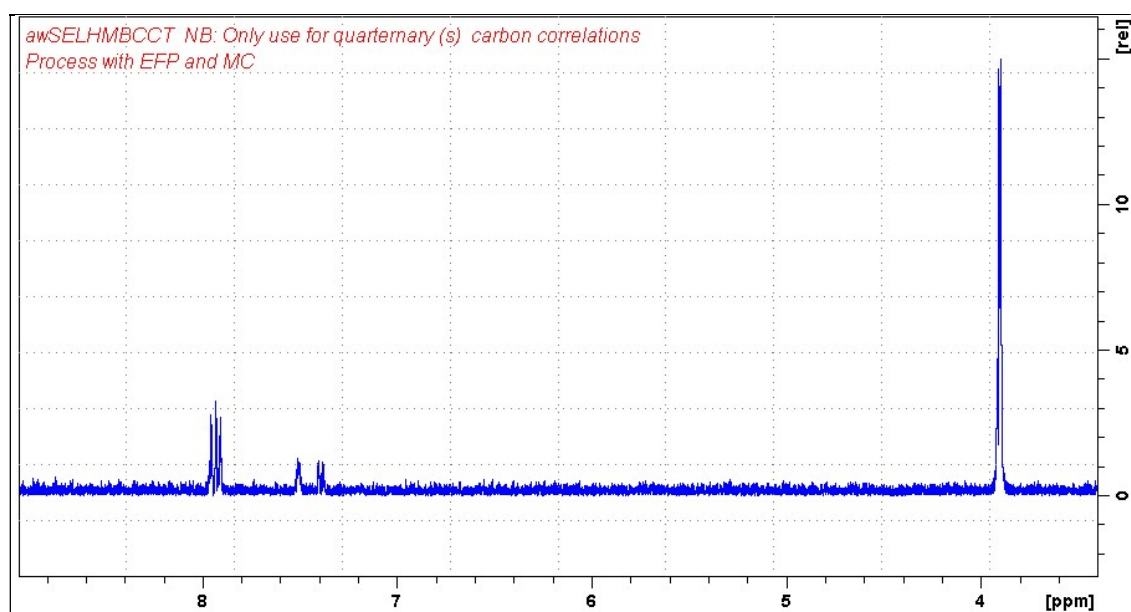
Neo400 **SELHMBCCT** spectrum of quinine in D$_6$-DMSO (6-10 ppm region) with selective excitation of the ^{13}C signal at 157.2 ppm.