KJM 9250

AVII-600 SELHSQC and SELHMBC Experiments

Version 7.3

Topspin 3.2 Windows 7 AVII600

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1.0 Introduction

The **^{13}C O1 frequency** of a target signal must be determined before running **a SELHSQC** or **SELHMBC** experiment and entered as the **O2 frequency** when setting up these experiments.

SELHSQC spectra should be processed as per standard ^{1}H NMR spectra.

SELHMBCQ5 spectra should be processed with **EFP** *and* **MC.**

2.0 SELHSQC and SELHMBC Experiments

2.1 SELHSQC and SELHSQCND spectra

2.2 SELHSQC-DIPSI2 and SELHSQCND-DIPSI2 spectra

2.3 SELHMBCQ5 spectrum

2.1 SELHSQC and SELHSQCND spectra

Parameter sets: **awselhsqc** o**r awselhsqcnd (+ getprosol)**
Pulse programmes: **awselhsqcgpsisp** or **awselhsqcndgpsisp**

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

TD = **SI** = 32 K.
SW = 12 ppm, **O1P** = 5 ppm. Adjust **SW** and **O1P** as required.
O2: frequency of the **^{13}C** signal in **Hz** to be selectively excited.

NS = multiple of 8 or 16, **DS** = 4 or 8.

D1 = 1 sec or other value of your choice.
D24 is automatically calculated from **CNST2** ($^1J_{C\text{-}H}$).
CNST2 = $^1J_{C\text{-}H}$; 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.

Check that gradients and shaped pulses are OK, including a **40000 usec p36:sp26** band selective **Q3.1000** pulse.

Set **receiver gain** using **RGA** *(Important!).*

Process with: **EFP** (applies **LB** = 0.1-0.3 Hz)

AVII-600 SELHSQC ***(lower)*** and **SELHSQCND** ***(upper)*** spectra determined for quinine in D_6-DMSO with selective excitation of the ^{13}C signal at 102.4 ppm (O2 = 15534 Hz).

2.2 SELHSQC-DIPSI2 and SELHSQCND-DIPSI2 spectra

Parameter sets: **awselhsqc-dipsi2** or **awselhsqcnd-dipsi2 (+ getprosol)**
Pulse programmes: **awselhsqcgpdigpsisp** or **awselhsqcgpdigpndsisp**

Prior to running a **SELHSQC-DIPSI2** 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)**.

TD = **SI** =32 K.
SW = 12 ppm, **O1P** = 5 ppm. Adjust **SW** and **O1P** as required.
O2: frequency of the **^{13}C** signal in **Hz** to be selectively excited.

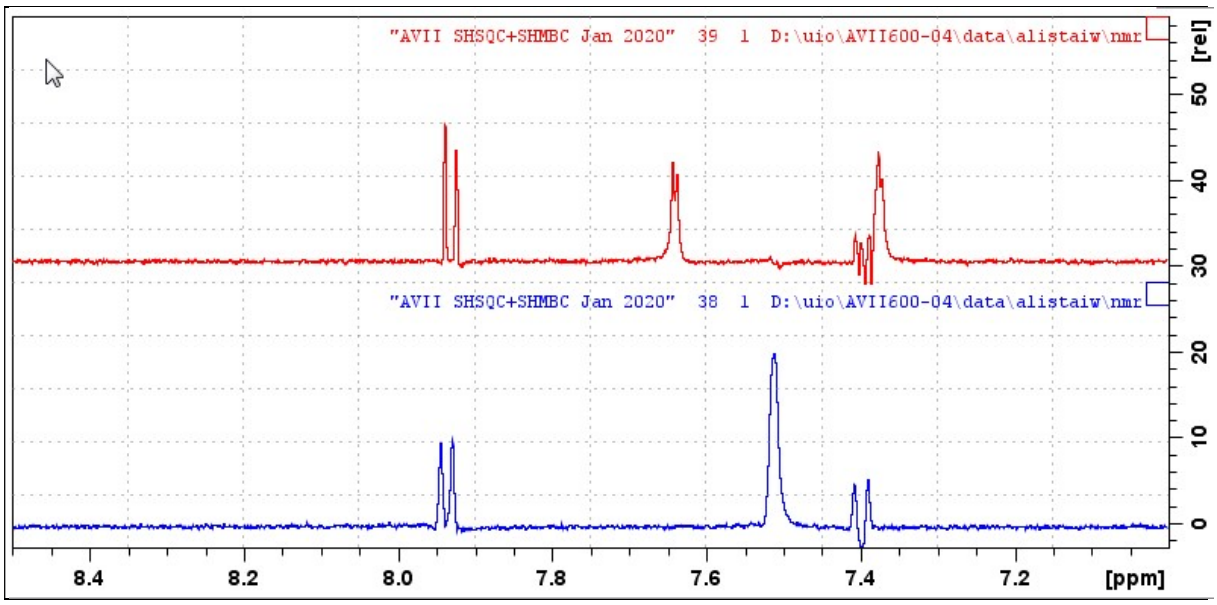
NS = multiple of 8 or 16, **DS** = 4 or 8.
or **NS x TD0** scans where **TD0** = any positive number.

D1 = 1 sec or other value of your choice.
D9 = 80 msec or other time of your choice (6-160 msec).
D24 is automatically calculated from **CNST2** ($^{1}J_{C\text{-}H}$).
CNST2 = $^{1}J_{C\text{-}H}$; typically 125 to 160 Hz for sp^3-sp^2 carbons. Furan or pyrrole ring carbons adjacent to hetero atoms will have ^{1}J = 200-220 Hz.

Check that gradients and shaped pulses are OK, including a **40000 usec p36:sp26** band selective **Q3.1000** pulse.

Set **receiver gain** using **RGA** *(Important!).*

Process with: **EFP** (applies **LB** = 0.1-0.3 Hz)



Expansion of the 7.0-8.5 ppm region of the **AVII-600 SELHSQC-DIPSI2** ***(lower)*** and **SELHSQCND-DIPSI2** ***(upper)*** spectra determined for quinine in D_6-DMSO with selective excitation of the ^{13}C signal at 102.9 ppm (O2 = 15534 Hz).

Correlated ^{1}H NMR signals observed in coupled SELHSQCND-DIPSI2 spectra show ^{1}J, ^{2}J, or ^{n}J ^{13}C-^{1}H couplings depending on the number of bonds between the selectively excited ^{13}C signal and correlated proton signals.

2.3 SELHMBC spectrum

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

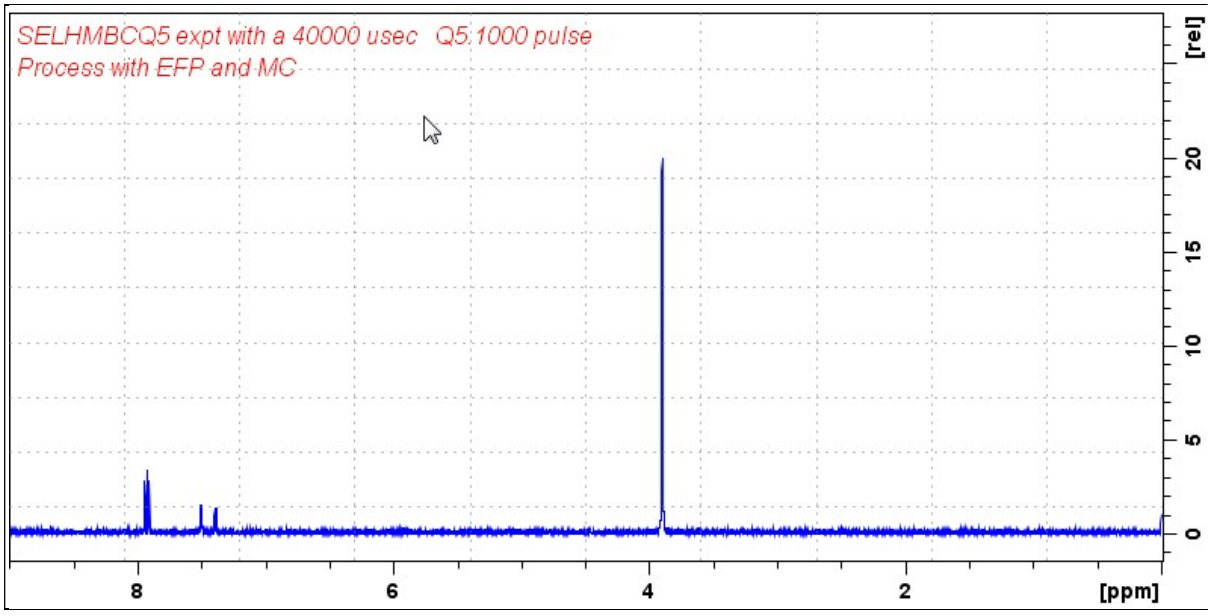
Prior to running a **SELHMBC** experiment run a standard **^{13}C** or **DEPT** experiment and determine the **O1** frequency in Hz of the **^{13}C** signal to be selectively excited. Enter this value as **O2 (Hz)**.

TD = **SI** = 32 K.
SW = 10 or 12 ppm, **O1P** = 5 or 6 ppm. Adjust **SW** and **O1P** as required.
O2: frequency of the **^{13}C** signal in **Hz** to be selectively excited.

NS = multiple of 8 or 16, **DS** = 4 or 8.
D1 = 1 sec or other value of your choice.
CNST2 = $^{1}J_{C\text{-}H}$; typically 125 to 160 Hz for sp^3-sp^2 carbons. Furan or pyrrole ring carbons adjacent to hetero atoms will have ^{1}J = 200-220 Hz.
CNST13 = **8 Hz** or other value of your choice.
D6 is autocalculated from **CNST13**

Check that gradients and shaped pulses are OK, including a **40000 usec p36:sp26** band selective **Q3.1000** pulse.

Process with **EFP** (applies **LB** = 0.3 Hz) *and* **MC.**



AVII-600 **SELHMBCQ5** spectrum determined for the signal which occurs at 156.4 ppm.

If no signal is observed (due to T_2 or other relaxation issues) try the **SELHMBCQ5.2** experiment which uses as **20000 usec Q5.1000** pulse. The **SELHMBCQ5.2** experiment has a somewhat wider excitation window than that of the **SELHMBCQ5** experiment which uses **a 40000 usec Q5.1000** pulse