KJM 9250

SELHSQC and SELHMBC Experiments on the AVI600 Spectrometer

Version 7.3

Topspin 1.3 Windows XP AVI600

April 2020

AVI-600 SELHSQC and SELHMBC Experiments

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.

Some of the shaped pulse powers that are used in AVI-600/TS2.1 1D-SEL ^{13}C-^{1}H experiments *are not prosol Table linked so they are saved in the parameter sets.*

Processing.

SELHSQC spectra should be processed as standard 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)**.

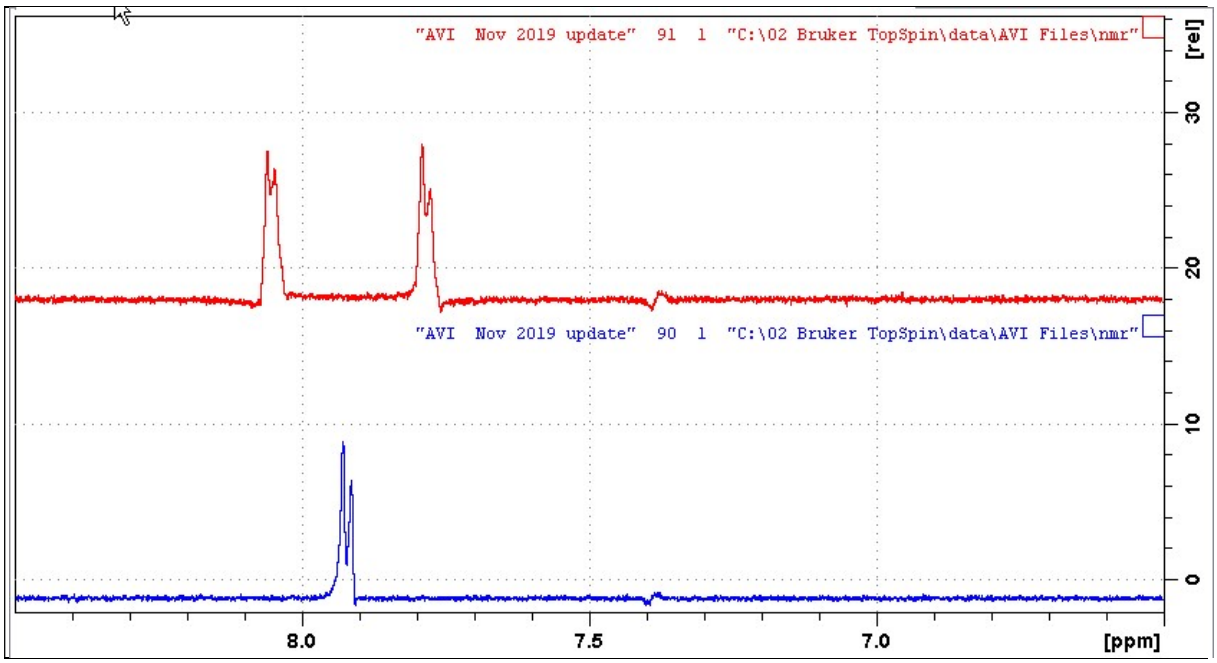
TD = **SI** = 32 K.
SW = 12 ppm, **O1P** = 5 ppm. Adjust **SW** and **O1P** as required.
O2: frequency of the **^{13}C** signal in **Hz** to be selectively excited.

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.
P36 = 40000 usec, **SPNAM26** = Q3.1000 selective pulse.

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

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



Expansion of the 6.5-8.5 ppm region of the **AVI-600 SELHSQC** ***(lower)*** and **SELHSQCND** ***(upper)*** spectra determined for quinine in D_6-DMSO with selective excitation of the ^{13}C signal at 131.6 ppm (O2 = 19857 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)**.

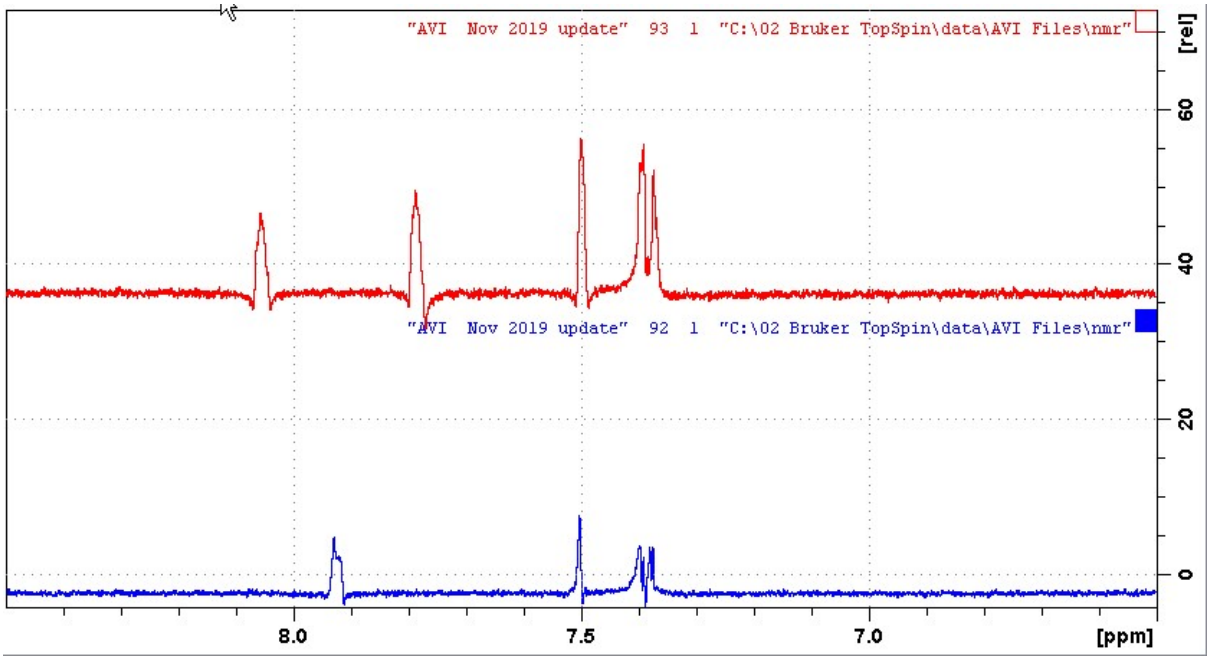
TD = **SI** =32 K.
SW = 12 ppm, **O1P** = 5 ppm. Adjust **SW** and **O1P** as required.
O2: frequency of the **^{13}C** signal in **Hz** to be selectively excited.

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.
P36 = 40000 usec, **SPNAM26** = Q3.1000 selective pulse.

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

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



Expansion of the 6.5-8.5 ppm region of the **AVI-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 131.6 ppm (O2 = 19857 Hz). The ^{1}J correlated proton signal occurs at 7.95 ppm.

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

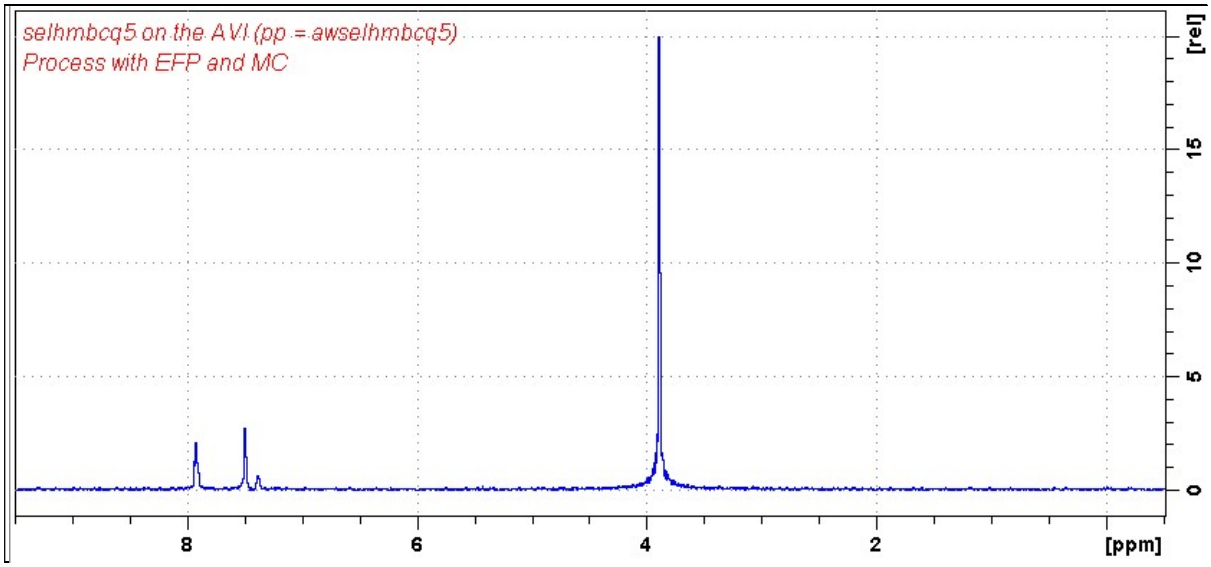
Parameter sets: **awselhmbcq5 (+ getprosol)**
Pulse programmes: **awselhmbcq5**

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 = 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.
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**
P11 = **40000 usec**, **SPNAM28** = **Q5.1000** selective pulse.

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



AVI-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. This experiment has a somewhat wider excitation window than that of the **SELHMBCQ5** which uses **a 40000 usec Q5.1000** pulse.