KJM 9250

AVIIIHD-800 MHz SELHSQC, SELHSQC-DIPSI2 and SELHMBC Experiments

Version 1.0

Topspin 3.5

Windows 7

SELHSQC + SELHMBC Experiments on the AVIIIHD-800

1.1 Introduction

aw coded **1D-SELHSQC, SELHSQC-DIPSI2** and **SELHMBC** experiments and parameter sets have been set up on the **AVIIIHD-800** spectrometer.

1.2 NS x TD0 option

SELHSQC and **SELHMBC** experiments can be run using the **NS** x **TD0** option where **NS** is a multiple of 8, 16, 32, 64 (etc) and **TD0** is any number >1.

Do NOT use the **TR** command as a **NS x TD0** experiment proceeds. Multiples of **NS** scans will be automatically saved and can be processed using the FT or EFP commands the experiment proceeds. A run can be terminated at any time using the **STOP** (*NB not the* **HALT**) command. This will ensure a multiple of 4 or 8 scans is saved as is required by some of the selective excitation experiments.

1.3 Signals with short T_2's

^{13}C signals with short T_2's may give no result in **SELHMBCCT** or **SELHSQC** experiments run with a 180 degree **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.*

SELHMBCQ5 experiments are run with a parameter set saved power level for its **20000 usec** 90 degree **Q5** pulses which is *not* over written or updated by the getprosol command.

1.4 SELHSQC and SELHMBC Experiments

The following 1D-Selective experiments have been set up on the **AVIIIHD-800** spectrometer.

2.1 SELHSQC and SELHSQCND spectra
2.2 SELHSQC-DIPSI2 and SELHSQCND-DIPSI2 spectra

2.3 SELHMBCCT spectra

2.4 SELHMBCQ5 spectra

2.1 SELHSQC and SELHSQCND Spectra

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

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).**

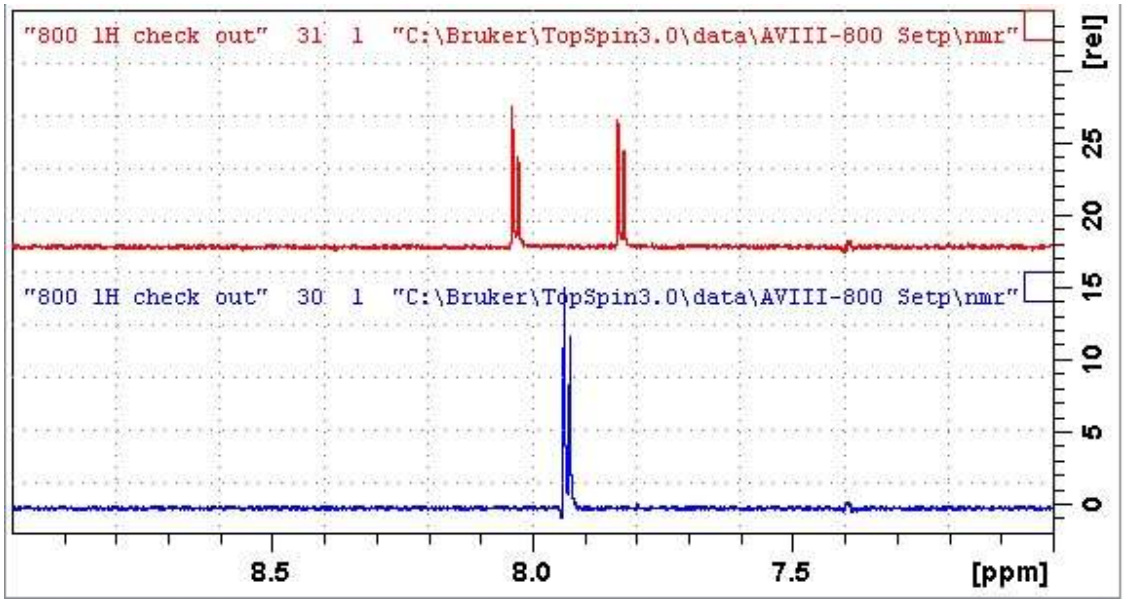
TD = **SI** = 64 K.
SW = 14 ppm, **O1P** = 6 ppm. Adjust **SW** and **O1P** as required.
O2 = frequency of the **^{13}C** signal in **Hz** to be selectively excited.

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

D1 = 1 sec or other value of your choice.
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.

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

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



7-9 ppm region of 800 MHz **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 = 26475 Hz). The ^{1}J correlated proton signal occurs at 7.95 ppm.

2.2 SELHSQC-DIPSI2 and SELHSQCND-DIPSI2 Spectra

Parameter set: **awselhsqc-dipsi2** or **awselhsqcnd-dipsi2 (+ getprosol)**
Pulse programme: **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).**

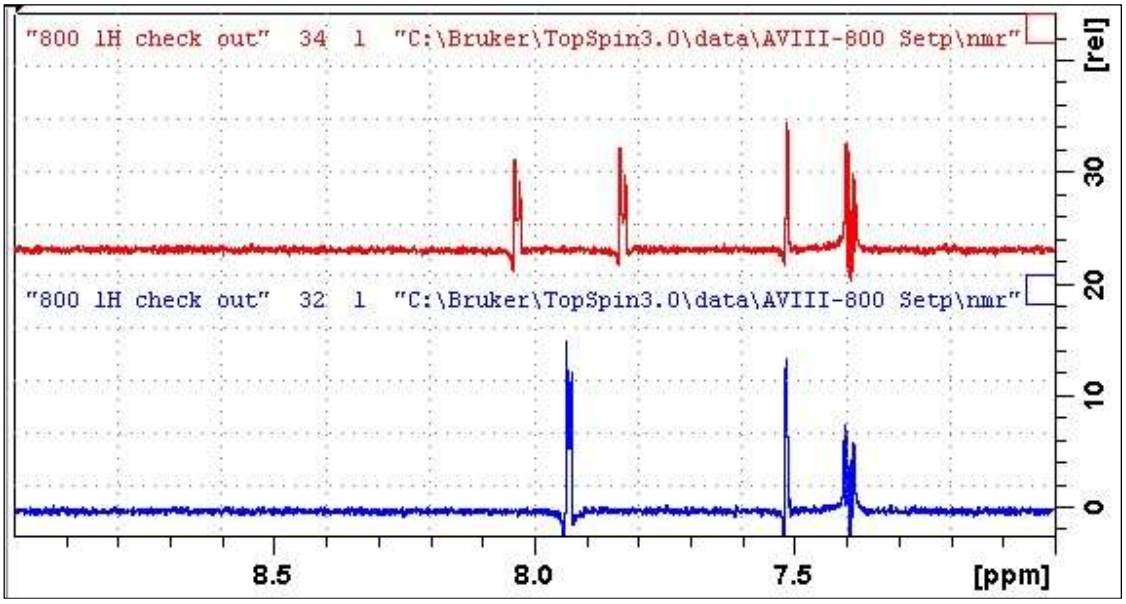
TD = **SI** = 64 K.
SW = 14 ppm, **O1P** = 6 ppm. Adjust **SW** and **O1P** as required.
O2 = frequency of the **^{13}C** signal in **Hz** to be selectively excited.

NS = multiple of 8 or 16, **DS** = 4 or 8.
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** ($^1J_{C\text{-}H}$).
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.

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

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



7-9 ppm regions of the **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 = 26475 Hz). The ^1J correlated proton signal occurs at 7.95 ppm.

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

2.4 SELHMBCQ5 Spectra

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

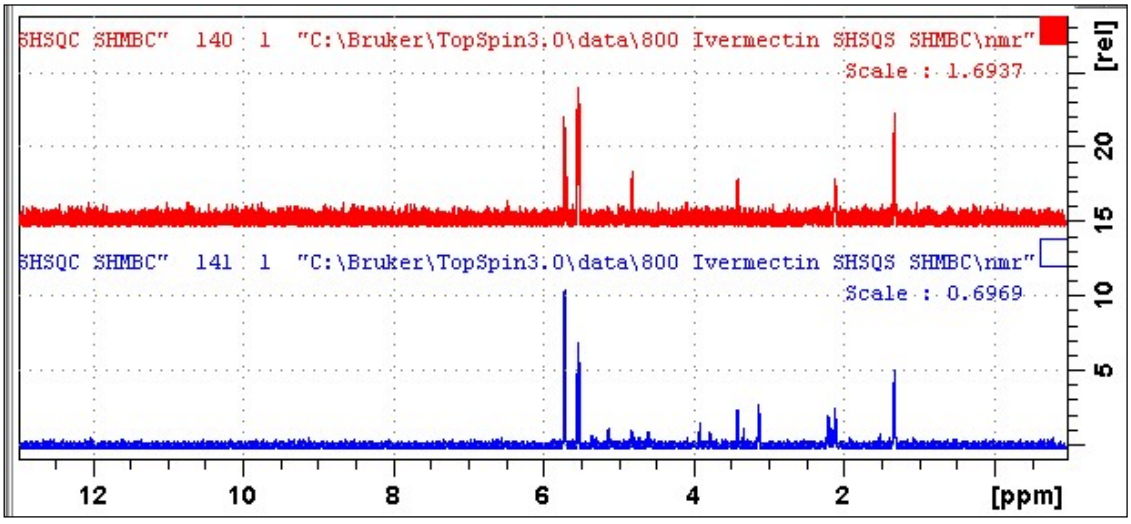
Prior to running a **SELHMMBC** experiment run a standard **^{13}C** experiment and determine the **O1** frequency of the **^{13}C signal** in **Hz** to be selectively excited. Enter this value as **O2 (Hz).**

TD = **SI** = 32 K.
SW = 14 ppm, **O1P** = 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 = 1.5 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 = J_{LR} = 8 Hz or other value of your choice.

Type **ased** (return) and check gradients and other parameters are OK. Check that the experiment is set up to use a **20000 usec/18.66 db p32:sp63 Q5.1000 pulse** which is not prosol Table linked.

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



Lower: SELHMBCSINC spectrum of ivermectin in D_6-DMSO entered at O2 = 19286 Hz (95.9 ppm). **Upper: SELHMBCQ3** centered at O2 = 19286 Hz .

2.4 SELHMBCCT

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

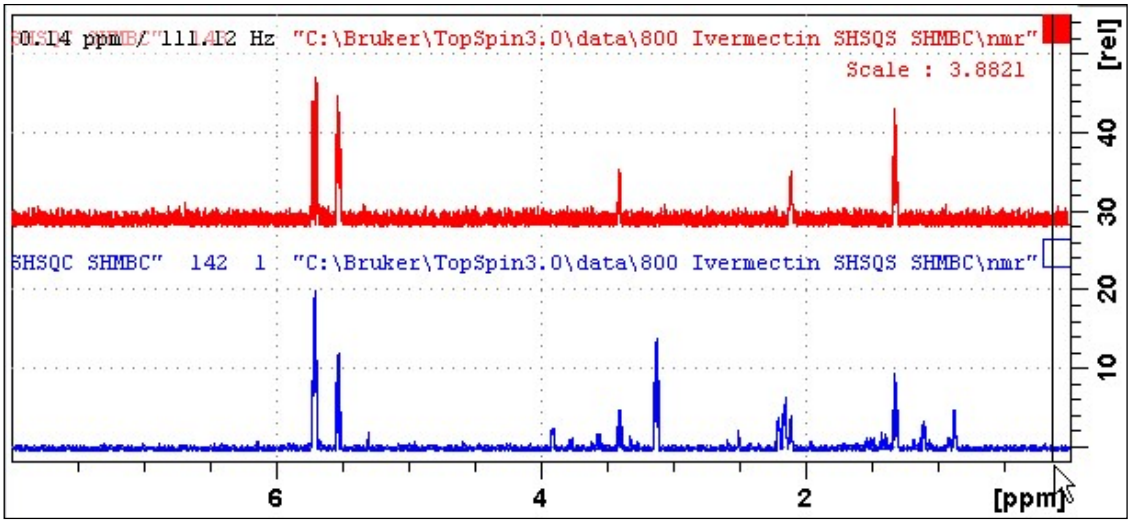
Prior to running a **SELHMMBCCT** experiment run a standard ^{13}**C** experiment and determine the **O1** frequency of the ^{13}**C signal** in **Hz** to be selectively excited. Enter this value as **O2 (Hz).**

TD = **SI** = 32 K.
SW = 14 ppm, **O1P** = 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 = 1.5 sec or other value of your choice.
CNST6 = min. $^{1}J_{C\text{-}H}$ = 120 Hz, **CNST7** = max.$^{1}J_{C\text{-}H}$ = 170 Hz
CNST13 = J_{LR} = 8 Hz or other value of your choice

Type **ased** (return) and check gradients and other parameters are OK including the use of a prosol Table linked **40000 usec p36:sp26 Q3 pulse.**

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



Lower: SELHMBCCT spectrum of ivermectin in D_6-DMSO entered at O2 =

2.5 SELHMBCCT.3 Spectra

Parameter set: **awselhmbcsct.3 (+ getprosol)**
Pulse programme: **awselhmbcct.3**

Prior to running a **SELHMMBCCT.3** experiment run a standard **^{13}C** 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 = 14 ppm, **O1P** = 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 = 1.5 sec or other value of your choice.
CNST6 = min. $^{1}J_{C\text{-}H}$ = 120 Hz, **CNST7** = max.$^{1}J_{C\text{-}H}$ = 170 Hz
CNST13 = J_{LR} = 8 Hz or other value of your choice.

The **Q3_surbop.1** pulse used in the **awselhmbcct.3 pp** is defined as **p32:sp57** where **p32=0.5*p36** [**p32 = 20000 usec, p36 = 40000 usec =** prosol linked value] and the parameter set saved **sp57** power level is **6 db greater** (= attenuated by a factor of 2) than that used for a 40000 usec Q3 pulse.

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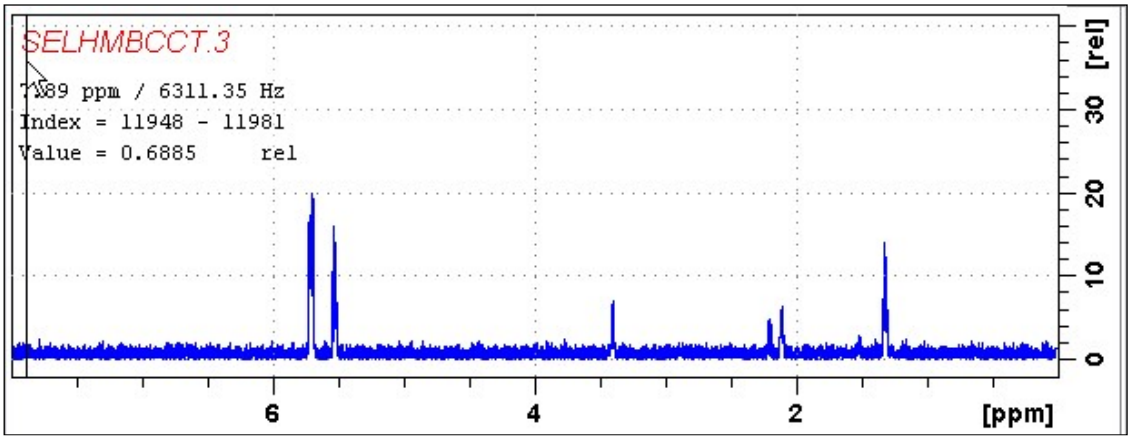
Type **ased** (return) and check gradients and other parameters are OK.

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



SELHMBCCT.3 spectrum of ivermectin in D_6-DMSO entered at O2 = 19286 Hz (95.9 ppm).

SELHMBCCT.3 spectra may include low levels of correlations ex nearby carbon signals.