KJM 9250

SHSQC, SHMBC spectra on the AVI600 Spectrometer

Version 7.3

Topspin 1.3 Windows XP AVI600

January 2020

AVI-600 SHSQC and SHMBC Experiments

1.0 Introduction

aw coded **AVI-600 SHSQC** and **SHMB**C parameter sets are set up with 2K acquired ^{1}H points and 128 ^{13}C increments. ^{1}H and ^{13}C spectral windows and their mid points must be determined in a standard ^{13}C or HSQC/HMBC experiment before setting up an S**HSQC** or **SHMBC** experiments.

NB: Some of the shaped pulse powers that are used in AVI-600/TS2.1 SHSQC and SHMBC experiments *are not prosol Table linked so they are saved in parameter sets*.

Processing

SHSQC45 and SHSQC135 experiments are phase sensitive experiments. These spectra should be phased **before** using the **abs1** and **abs2** commands. Low level ^{2}J correlations may be observed in **SHSQC** spectra. **SHMBCQ5** spectra are processed with **xfb** *and* **xf2m.**

The ^{13}C axis resolution of **SHSQC** and **SHMBC** spectra acquired using 128 increments across a 30 ppm window is typically 4-5 times greater than that of standard full window **HSQC** and **HMBC** spectra acquired with 160-256 or more increments.

2.0 SHSQC, SHMBC Experiments

2.1 SHMBC45 spectrum

2.2 SHSQC135 spectrum

2.3 SHMBCQ5 spectrum

2.1 SHSQC45 Spectrum

Parameter set: **awshsqc45 (+ getprosol)**
Pulse programme: **awshsqcetgpsisp2.2-45**
d24 is automatically calculated from **cnst2**

SW(^{13}C) = **30 ppm,** excitation band width is ca **20 ppm**
TD(F2) = 2K, **TD(F1)** = 128 points or other value of your choice (64-256 points).

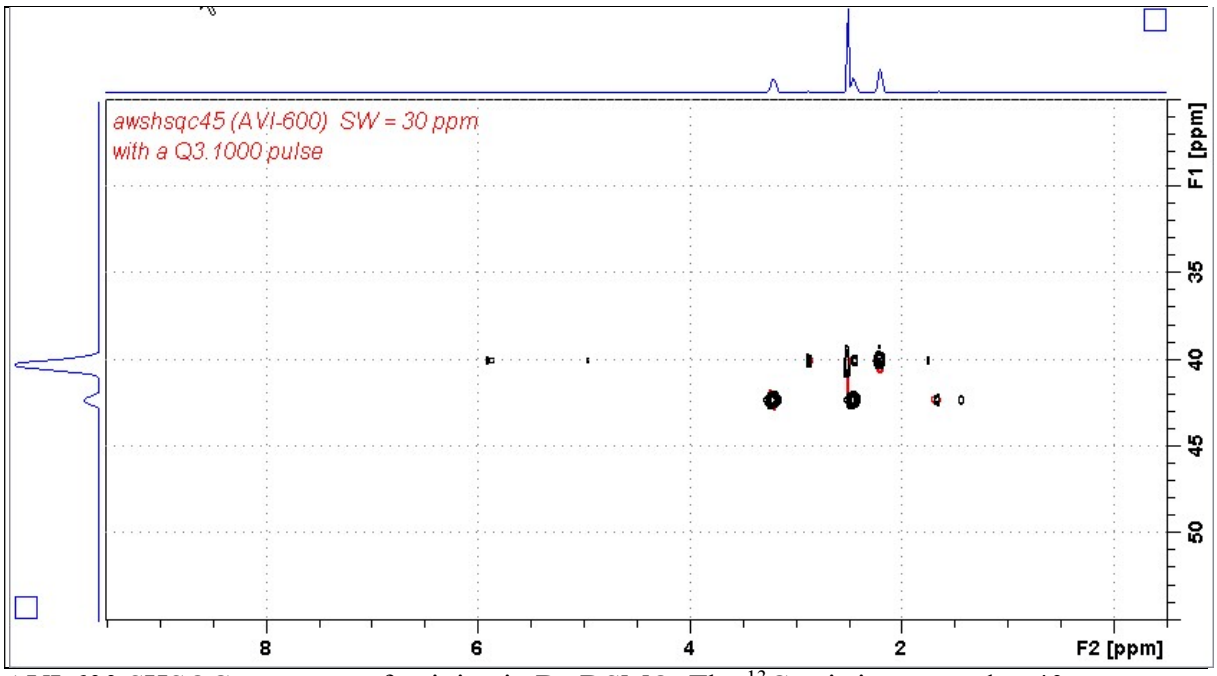
Type **eda** (enter) and enter **SW(^{1}H)** and **O1P = ^{1}H** spectral window midpoint in ppm.
Enter **O2P = ^{13}C** spectral window midpoint in ppm.

NS = multiple of 4, 8 or 16, **DS** = 8 or 16.
D1 = repetition delay = **1.5 sec** or other time of your choice.
CNST2 = ^{1}J coupling constant = **145 Hz** or other value of your choice (eg: 125-160 Hz).

Type **ased** (enter) and review parameters used in the job.
Check that gradients and shaped pulses are OK, including a **2000 usec p33:sp28 Q3.1000** pulse with a parameter set saved **PL28** power level of ~ **20 db.**

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

Process with: **SI(F2) = 2K, SI(F1) = 512 or 1024 points**
WDW(F1) = WDW(F2) = QSINE
SSB(F2) = SSB(F1) = 2
xfb, abs1 and abs2



AVI-600 SHSQC spectrum of quinine in D_6-DSMO. The ^{13}C axis is centered at 40 ppm.

2.2 SHSQC135

Parameter set: **awshsqc135 (+ getprosol)**
Pulse programme: **awshsqcedetgpsisp2.3-135**
d21 and **d24** are automatically calculated from **cnst2**

SW(^{13}C) = **30 ppm,** excitation band width is ca **20 ppm**
TD(F2) = 2K, **TD(F1)** = 128 points or other value of your choice (64-256 points).

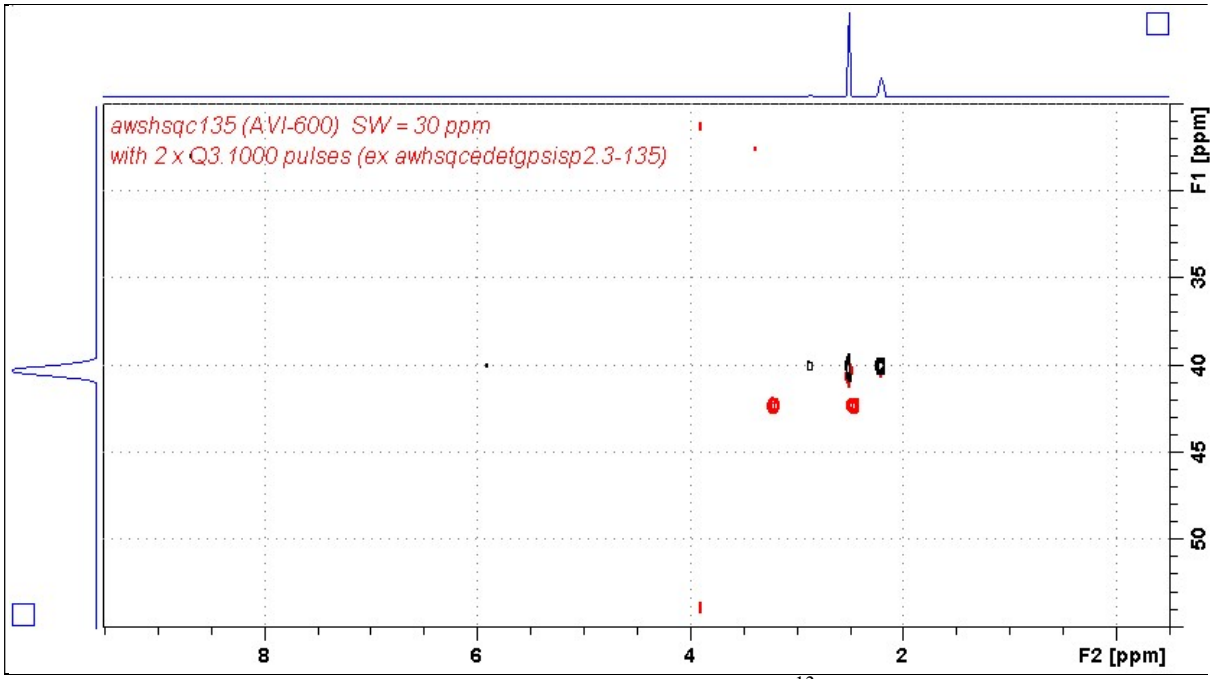
Type **eda** (enter) and enter **SW(^{1}H)** and **O1P = ^{1}H** spectral window midpoint in pp
Enter **O2P = ^{13}C** spectral window midpoint in ppm.

NS = multiple of 4, 8 or 16, **DS** = 8 or 16.
D1 = repetition delay = **1.5 sec** or other time of your choice.
CNST2 = ^{1}J coupling constant = **145 Hz** or other value of your choice (eg: 125-160 Hz).

Type **ased** (enter) and review parameters used in the job.
Check that gradients and shaped pulses are OK, including a **2000 usec p33:sp28 Q3.1000** pulse with a parameter set saved **PL28** power level of ~ **20 db.**

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

Process with: **SI(F2) = 2K, SI(F1) = 512 or 1024 points**
WDW(F1) = WDW(F2) = QSINE
SSB(F2) = SSB(F1) = 2
xfb, abs1 and abs2



AVI-600 SHSQC135 spectrum of quinine in D_6-DSMO. The ^{13}C axis is centered at 40 ppm.

2.3 SHMBC

Parameter set: **awshmbcq5 (+ getprosol)**
Pulse programme: **awshmbcq5**

SW(^{13}C) = **30 ppm,** excitation band width is ca **15-18 ppm.**
TD(F2) = 2K, **TD(F1)** = 128 points or other value of your choice (64-256 points).

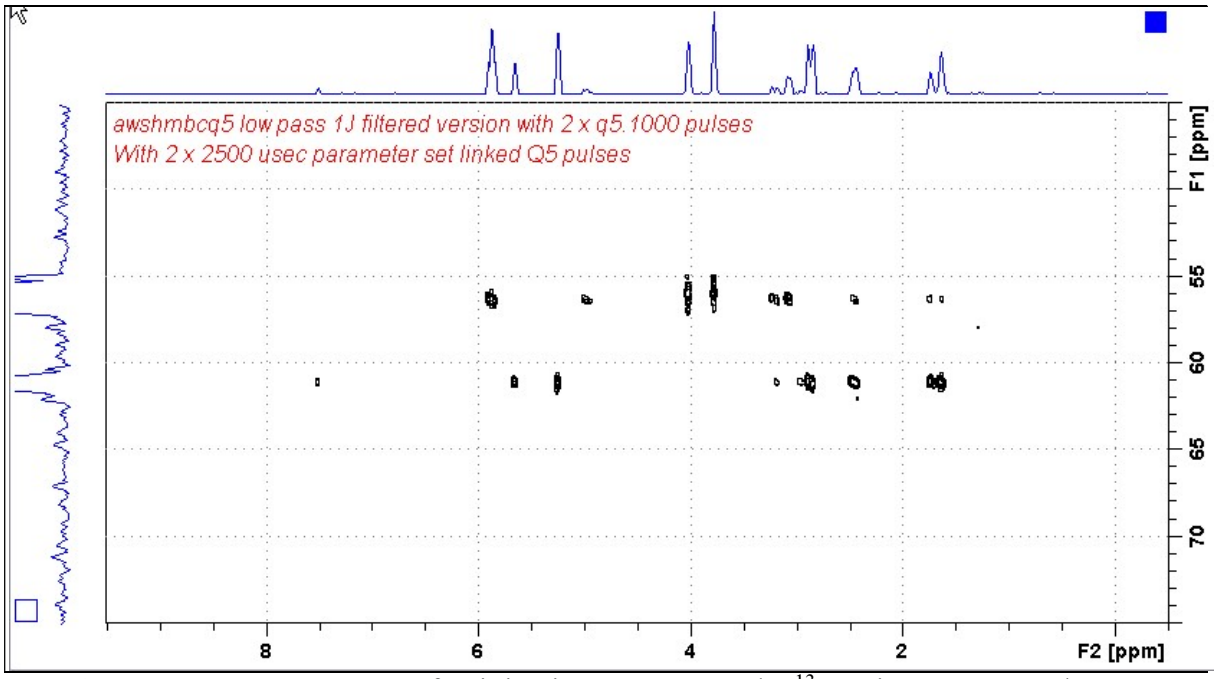
Type **eda** (enter) and enter **SW(^{1}H)** and **O1P = ^{1}H** spectral window midpoint in ppm.
Enter **O2P = ^{13}C** spectral window midpoint in ppm.

NS = multiple of 4, 8 or 16, **DS** = 8 or 16.
D1 = repetition delay =**1.5 sec** or other time of your choice.
CNST2 = ^{1}J coupling constant **= 145 Hz** or other value of your choice (eg 125-220 Hz).
CNST13 = ^{n}J selection filter = **8 Hz** or other value of your choice (eg: 6-14 Hz).

Type **ased** (enter) and review parameters used in the job.
Check that gradients and shaped pulses are OK, including a **2500 usec p17:sp30 Q5.1000** pulse with a parameter set saved **PL30** power level of ~ **19 db.**

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

Process with: **SI(F2) = 2K, SI(F1) = 512 or 1024 points**
WDW(F1) = WDW(F2) = QSINE
SSB(F2) = SSB(F1) = 2
xfb, abs1 and abs2



AVI-600 SHMBCQ5 spectrum of quinine in D_6-DMSO. The ^{13}C axis was centered at 60 ppm.