KJM 9250

AVII-600 SHSQC and SHMBC Experiments

Version 7.3

Topspin 3.2 Windows 7 AVII600

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January 2020

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1.0 Introduction

aw coded **AVII-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 should be determined before setting up S**HSQC** or **SHMBC** experiments.

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

A greater range of **SHSQC** and **SHMBC** experiments is available on the **AVII-600** than is the case for the case for the **AVI-600**

1.2 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**. **SHMBCCT** spectra are acquired in echo-antiecho mode and transformed with **xfb** *and* **xf2m.**

2.0 AVII-600 Experiments and Parameter Sets

2.1	**SHSQC45**	semi-selective hsqc45
2.2	**SHSQC45.m**	medium-selective hsqc45
2.3	**SHSQC135**	semi-selective hsqc135
2.4	**SHSQC135.m**	medium-selective hsqc135
2.5	**SHMBCQ5**	semi-selective shmbc experiment
2.6	**SHMBCCT**	constant time mode shmbc experiment

2.1 SHSQC45

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

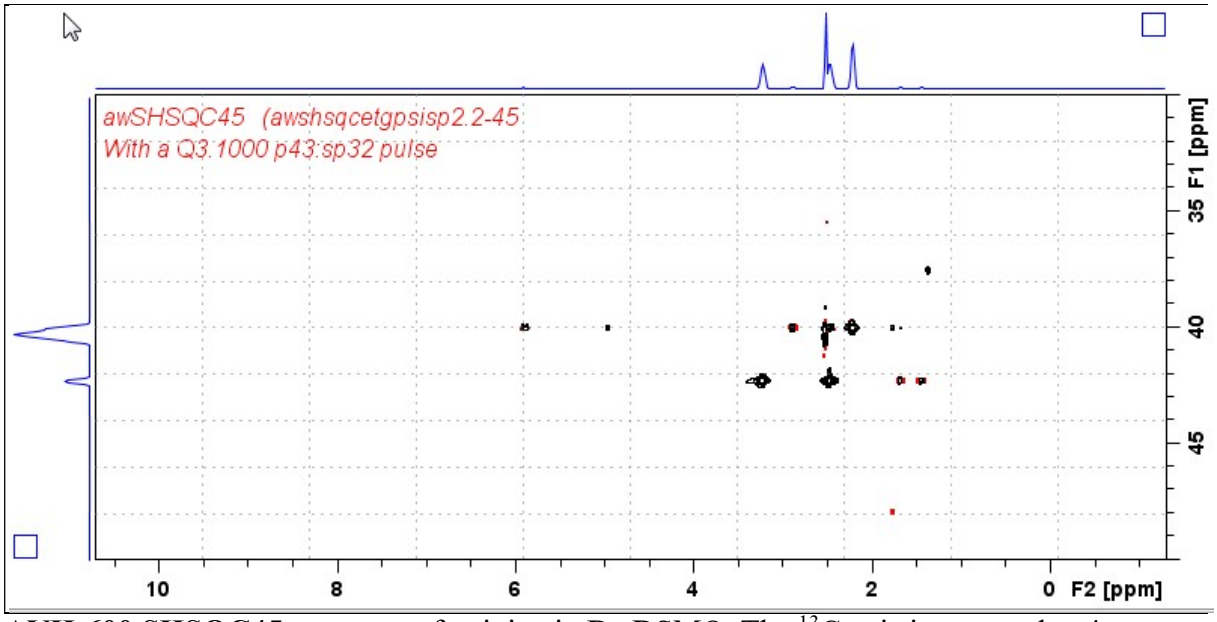
Type **eda** (enter) and enter **SW(^{1}H)** and **O1P = ^{1}H** spectral window midpoint in ppm.
Enter **O2P = ^{13}C** spectral window midpoint in ppm.
SW(^{13}C) = **20 ppm,** excitation band width is ca **10-12 ppm.**
TD(F2) = 2K, **TD(F1)** = 128 points.

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 **3000 usec p43:sp32** band selective **Q3.1000** pulse.

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



AVII-600 SHSQC45 spectrum of quinine in D_6-DSMO. The ^{13}C axis is centered at 4 ppm.

2.2 SHSQC45.m

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

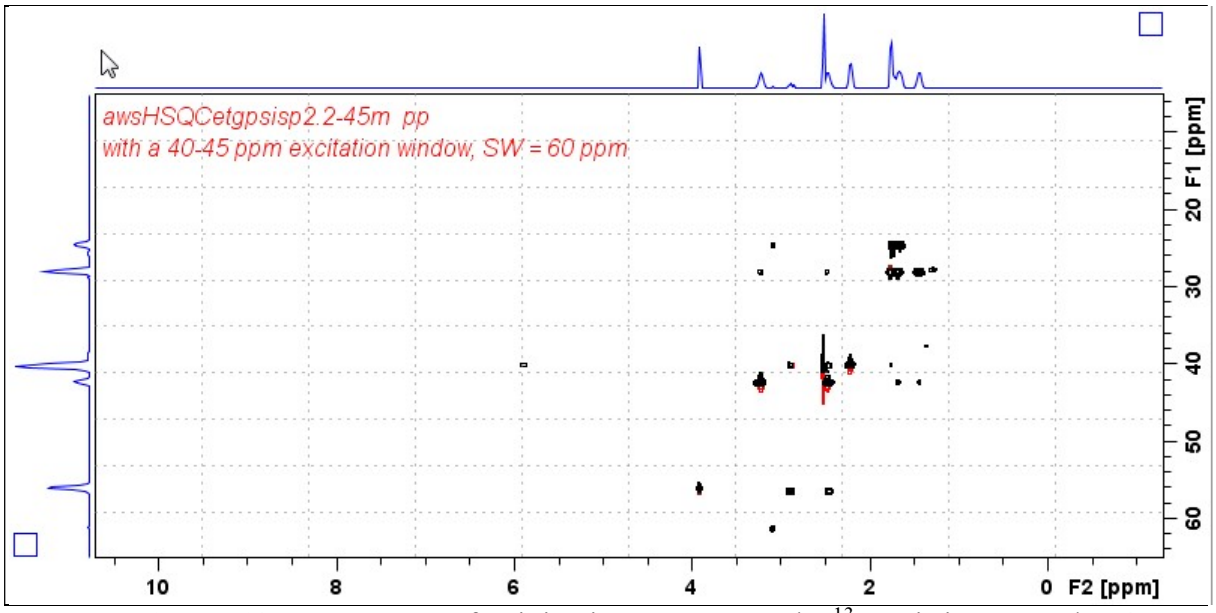
Type **eda** (enter) and enter **SW(^{1}H)** and **O1P = ^{1}H** spectral window midpoint in ppm.
Enter **O2P = ^{13}C** spectral window midpoint in ppm.
SW(^{13}C) = 60 ppm, excitation band width is ca **40-45 ppm.**
TD(F2) = 2K, **TD(F1)** = 128 points.

NS = multiple of 4, 8 or 16, **DS** = 8 or 16.
D1 = repetition delay = **1.5 sec** or other time of your choice.
CNST2 = ^1J 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 **700 usec p33:sp23** band selective **Q3.1000** pulse.

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



AVII-600 SHSQC45.m spectrum of quinine in D_6-DSMO. The ^{13}C axis is centered at 40 ppm.

2.3 SHSQC135

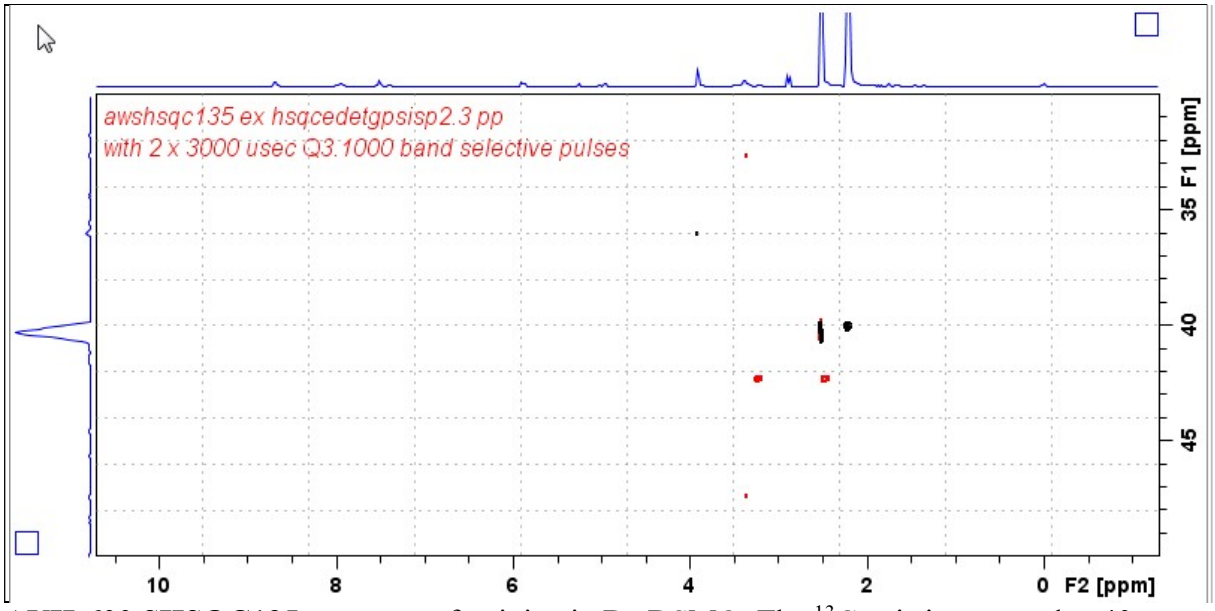
Parameter set: **awshsqc135 (+ getprosol)**
Pulse programme: **awshsqcedetgpsisp2.3-135**
d21 and **d24** are automatically calculated from **cnst2**

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.
SW(^{13}C) = **20 ppm,** excitation band width is ca **10-12 ppm**
.
TD(F2) = 2K, **TD(F1)** = 64-128 points.
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 **3000 usec p24:sp32** band selecive **Q3.1000** pulse.

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



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

2.4 SHSQC135.m

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

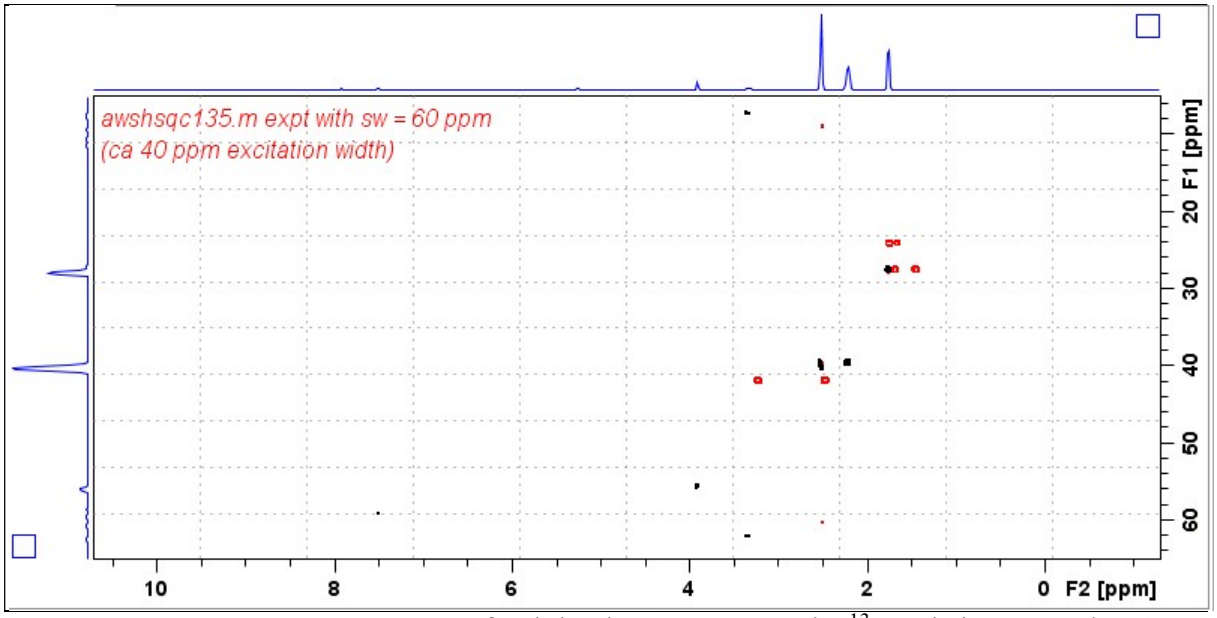
Type **eda** (enter) and enter **SW(^{1}H)** and **O1P = ^{1}H** spectral window midpoint in ppm.
Enter **O2P = ^{13}C** spectral window midpoint in ppm.
SW(^{13}C) = **60 ppm,** excitation band width is ca **40-45 ppm**
TD(F2) = 2K, **TD(F1)** = 64-128 points.

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 **768 usec p30:sp23** band selecive **Q3.1000** pulse.

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



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

2.5 SHMBCQ5

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

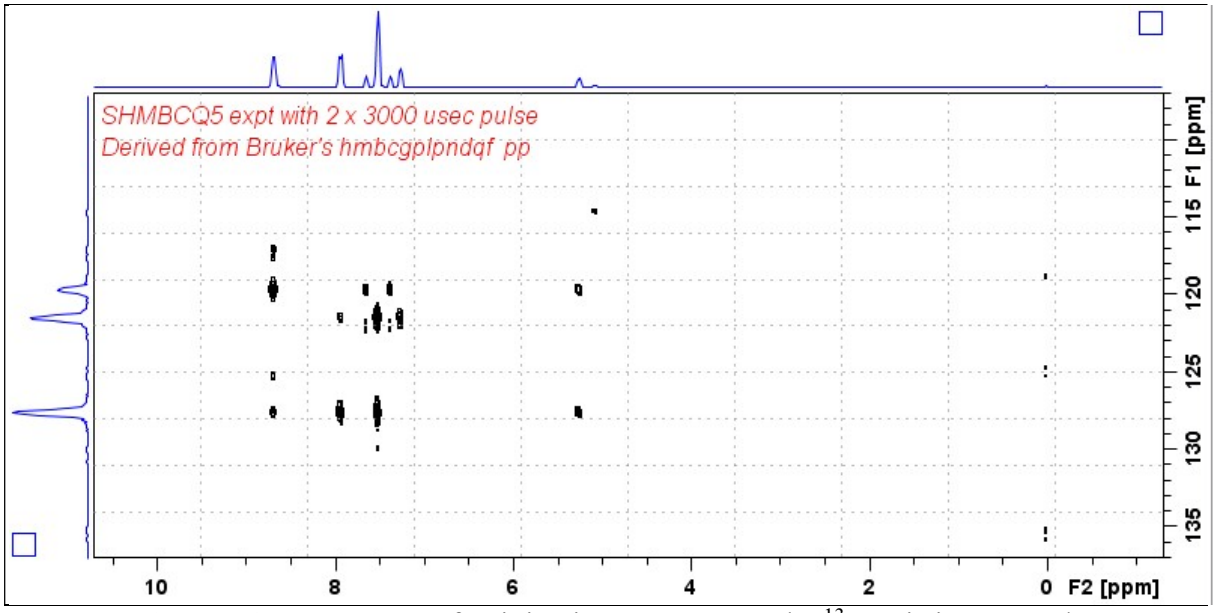
Type **eda** (enter) and enter **SW(^{1}H)** and **O1P = ^{1}H** spectral window midpoint in ppm.
Enter **O2P = ^{13}C** spectral window midpoint in ppm.
SW(^{13}C) = **20 ppm,** excitation band width is ca **12 ppm**
TD(F2) = 2K, **TD(F1)** = 64-128 points

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 **3000 usec p35:sp27** band selective **Q5.1000** pulse.

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



AVII-600 SHMBCQ5 spectrum of quinine in D_6-DMSO. The ^{13}C axis is centered at 122 ppm**.**

2.6 SHMBCCT (Constant time mode SHMBC experiment)

Parameter set: **awshmbcct (+ getprosol)**
Pulse programme: **awshmbcctetgpl2nd**

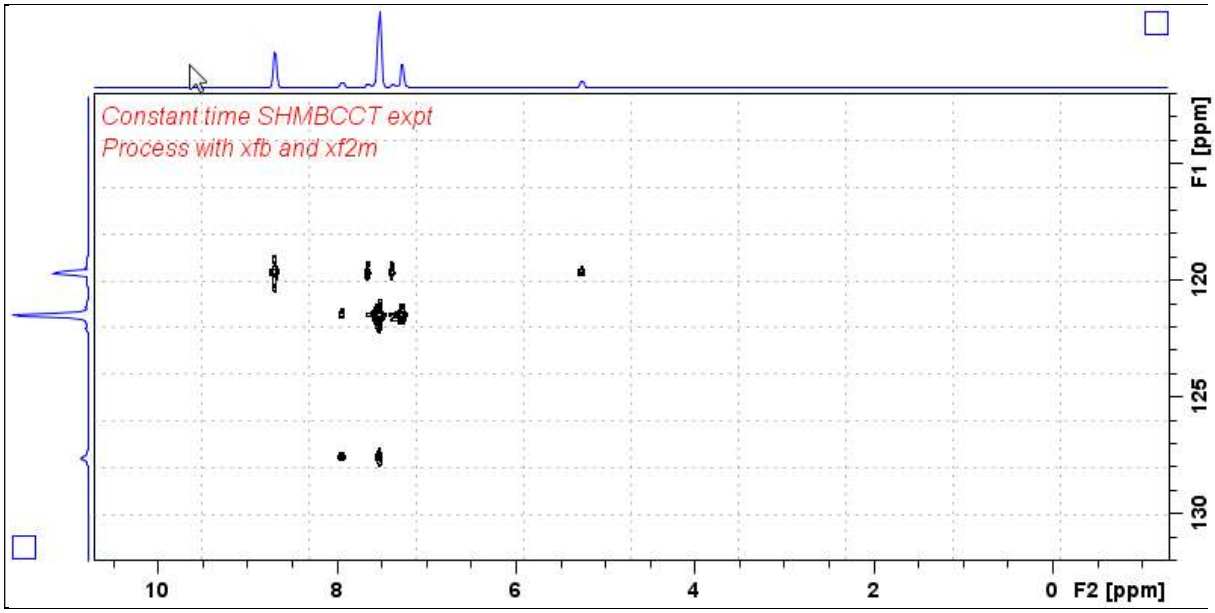
Type **eda** (enter) and enter **SW(^{1}H)** and **O1P = ^{1}H** spectral window midpoint in ppm.
Enter **O2P = ^{13}C** spectral window midpoint in ppm.
SW(^{13}C) = **20 ppm,** excitation band width is ca **10-12 ppm.**
TD(F2) = 2K, **TD(F1)** = 64-128 points

NS = multiple of 4, 8 or 16, **DS** = 8 or 16.
D1 = repetition delay =**1.5 sec** or other time of your choice.
CNST6 = **120 Hz**, **CNST7 = 170 Hz** = min/max ^1J coupling constants.
CNST13 = ^nJ 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 **3000 usec p43:sp32** band selective **Q3.1000** pulse.

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 *and* **xf2m , abs1 and abs2**



AVII-600 SHMBCCT spectrum of quinine in D_6-DMSO. The ^{13}C axis is centered at 122 ppm.