KJM 9250

^{1}H NMR spectra on the AVII-600 spectrometer.

Version 7.3

Topspin 3.2 Windows 7 AVII600

^{1}H NMR spectra on the AVII-600

1.0 Introduction

aw coded ^{1}H NMR parameter files generally use a 90^o pulse for maximum ^{1}H signal.

Best ^{1}H resolution is obtained using **FT** and **PK** (or **APK**) processing. **FT** processing does not apply a line broadening factor. **EF** or **EFP** processing applies a line broadening factor (**LB**).

Resolution enhancement uses negative **LB** values. Try **LB** = -1.0 to -2.5 Hz with **GB** = 0.33, and **GFP** processing. Remember to reset **LB** and **GB** to their normal values (0.1 and 0 respectively) after **GFP** processing.

1.1 Presaturation Experiments

Continuous wave or excitation sculptured (ES) can be used to presaturate ^{1}H NMR signals. The simplest of these techniques is continuous wave presaturation.

CW presaturation power levels (db settings) can be increased or decreased by subtracting or adding 3-12 db respectively. 6 db = a factor of 2. Bruker sometimes uses the **NOESYPR1D** pulse programme with an appropriate **d8** time to acquire **QNMR** spectra.

AVII-600 **ES** pulses are defined **as 2000 usec p12:sp1** or **p40:sp10 squal100.1000** pulses depending on which **prosol relations** option is used in a pulse program.

The **ES** shaped pulse's excitation window can be decreased by doubling its shaped pulse time from 2000 usec to 4000 usec and halving its power by adding 6 db to that read in using the **getprosol** command.

PS presaturation experiments use prosol Table linked a **100 msec** F1 channel **p18:sp6** pulse and a non-prosol Table linked **100 msec** F2 channel **p18:sp56 squal100.1000** pulse

2.0 ^{1}H NMR experiments

2.1 ^{1}H NMR with a 30, 45 or 90 degree pulse

2.2 ^{1}H NMR F1 CW presaturation

2.3 ^{1}H NMR with F1 + F2 CW presaturation

2.4 ^{1}H NMR with ES peak suppression

2.5 ^{1}H NMR with combined ES + CW presaturation on F1

2.6 ^{1}H NMR with combined ES + CW presaturation on F1 and CW presaturation on F2

2.7 ^{1}H NMR with three peak ES + dual CW presaturation

2.8 ^{1}H NOESYPR1D

2.9 ^{1}H NMR with F1 PS presaturation

2.10 ^{1}H NMR with F1 + F2 PS presaturation

2.1 ^{1}H NMR spectra with a 30, 45 or 90 degree pulse

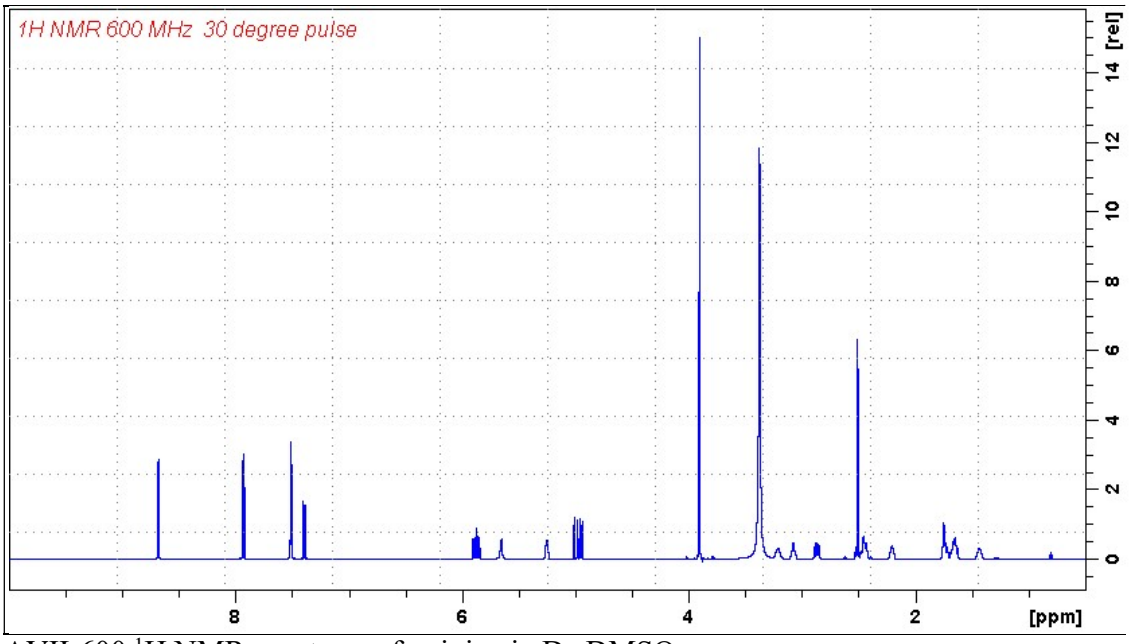
Parameter sets: **awproton30, awproton45, awproton90(+ getprosol)**
Pulse programmes**: zg30, awzg45 or zg respectively**

TD = 64 K, **SI** = 64 K.
SW = 16 ppm, **O1P** = 7.0 ppm.
D1 = 1.5 sec or other time of your choice.
NS = any number, **DS =** 2, 4 or 8.

Type **ased** (enter) and review parameters used in the job.

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

Process with **FT** (no line broadening) or **EFP** (applies **LB**).



AVII-600 ^{1}H NMR spectrum of quinine in D_6-DMSO.

2.2 ^{1}H NMR spectrum with CW presaturation

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

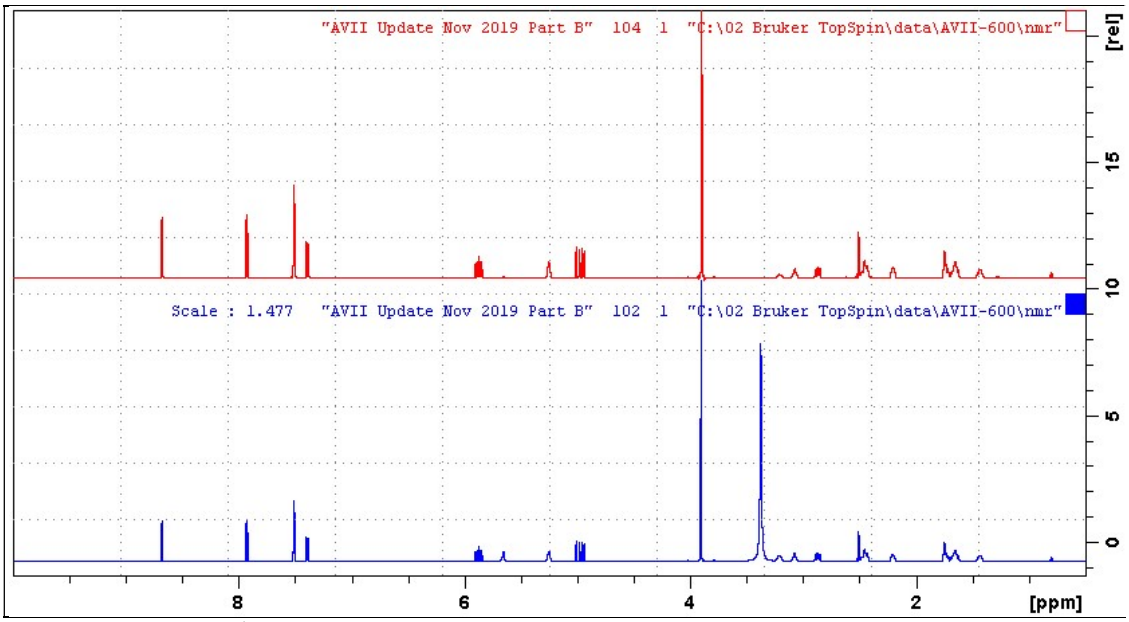
TD = 64K, **SI** = 64 K.
SW = 18 ppm.

O1 = frequency in Hz of the F1 signal to be presaturated.
= spectral window midpoint. Check **SW** is wide enough.
PL9 = **F1** presaturation power applied during **D1**.
D1 = 2 sec or other time of your choice.

Type **ased** (enter) and review parameters used in the job.
Add (or subtract) 3-12 db to **PL9** to decrease (or increase) the presaturation power.
6 db = a factor of 2. A larger attenuation setting decreases the power level.

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

Process with **EFP** (applies **LB**).



Lower: AVII-600 ^{1}H NMR spectrum of quinine in D_6-DMSO.
Upper: ^{1}H NMR spectrum with CW presaturation of the HOD line at 3.37 ppm.

2.3 ^{1}H NMR spectrum with dual CW presaturation

Parameter set: **awprotonprf1prf2 (+ getprosol)**
Pulse programme: **awprotonprf1prf2**

TD = 64 K, **SI** = 64 K.
SW = 18 ppm.

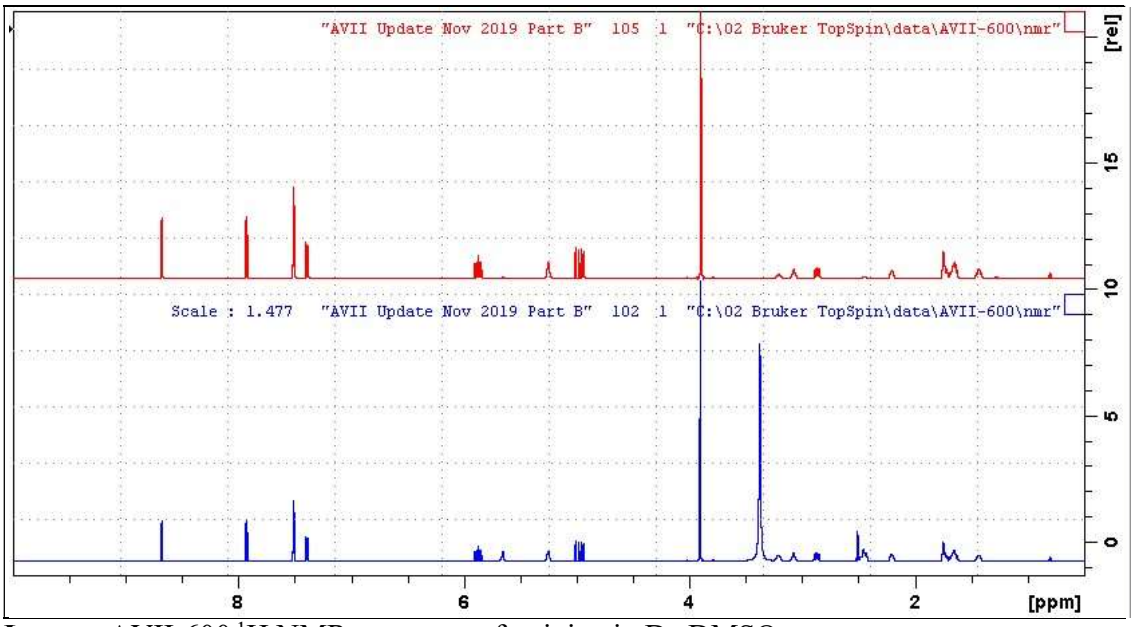
O1 = frequency in Hz of the F1 signal to be presaturated.
= spectral window midpoint. Check **SW** is wide enough.
O2 = frequency in Hz of the F2 signal to be presaturated.

PL9 = F1 presaturation power applied during D1.
PL21 = F2 presaturation power applied during D1.
D1 = 2 sec or other time of your choice.

Type **ased** (enter) and review parameters used in the job.
Add (or subtract) 3-12 db to **PL9** and/or **PL2**1 to decrease (or increase) the presaturation power. 6 db = a factor of 2. A larger attenuation setting decreases the power level.

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

Process with **EFP** (applies **LB**).



Lower: AVII-600 ^{1}H NMR spectrum of quinine in D_6-DMSO.
Upper: ^{1}H NMR spectrum with CW presaturation of the HOD (3.37 ppm) and DMSO (2.5 ppm) lines.

2.4 ^{1}H NMR spectrum with ES peak suppression

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

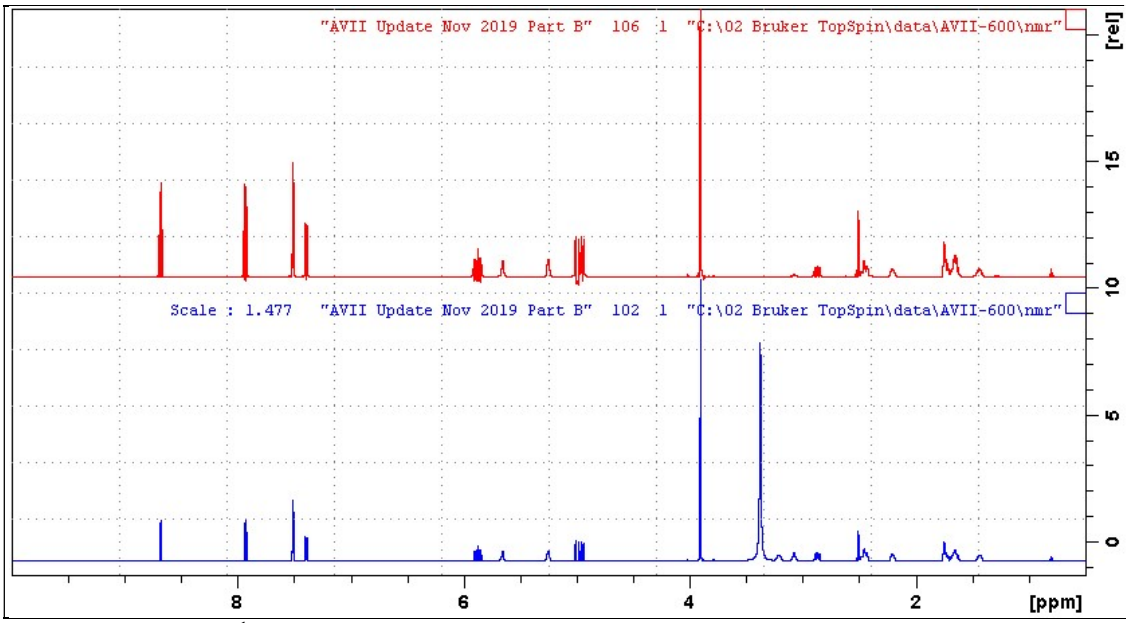
TD = 64 K, **SI** = 64 K.
SW = 18 ppm.

O1 = frequency in Hz of the F1 signal to be ES suppressed.
= spectral window midpoint. Check. **SW** is wide enough.
D1 = 1.5 sec or other time of your choice.

Type **ased** (enter) and review parameters used in the job. Verify gradients are OK.
Check **P12** = 2000 usec, **SPNAM1** = **squa100.1000**

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

Process with **EFP** (applies **LB**).



Lower: AVII-600 ^{1}H NMR spectrum of quinine in D_6-DMSO.
Upper: ^{1}H NMR spectrum with ES suppression of the HOD line at 3.37 ppm.

2.5 ^{1}H NMR with combined ES and CW presaturation on F1

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

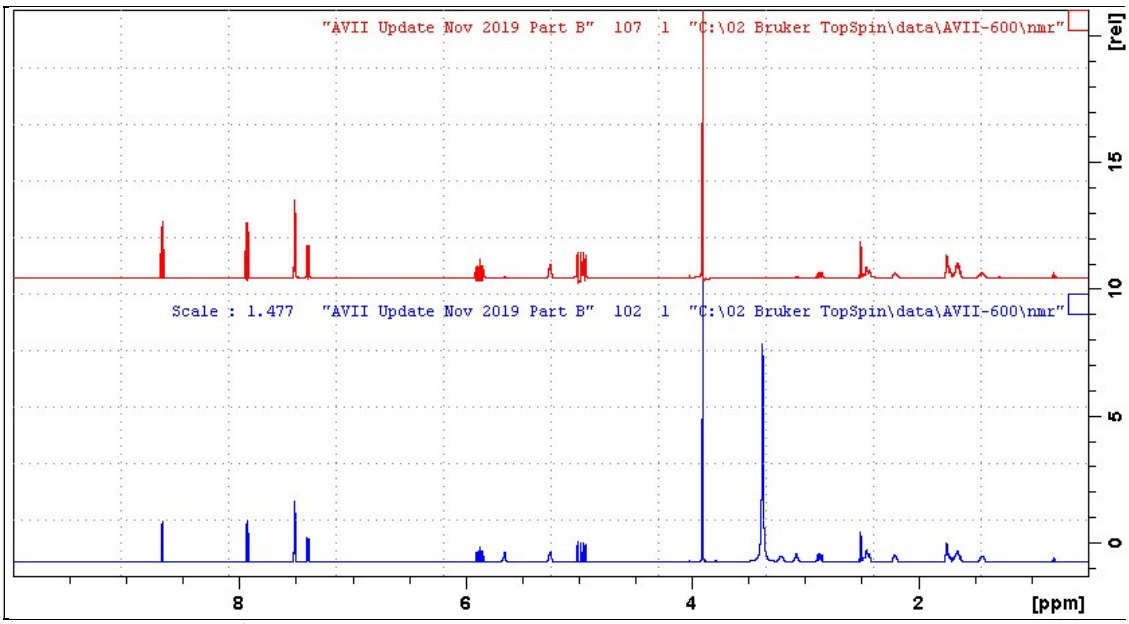
TD = 64 K, **SI** = 64 K.
SW = 18 ppm.

O1 = frequency in Hz of the F1 signal to be ES suppressed.
= spectral window midpoint. Check SW is wide enough.
PL9 = F1 presaturation power applied during D1.
D1 = 2 **sec** or other time of your choice.

Type **ased** (enter) and review parameters used in the job. Verify gradients are OK.
Check **P12** = **2000 usec**, **SPNAM1** = **squa100.1000**.

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

Process with **EFP** (applies **LB**).



Lower: AVII-600 ^{1}H NMR spectrum of quinine in D_6-DMSO.
Upper: ^{1}H NMR spectrum with combined ES and CW presaturation of the HOD line at 3.37 ppm.

2.6 ^{1}H NMR spectrum with combined ES+CW presaturation on F1 and CW presaturation on F2

Parameter set: **awprotonesprf1prf2 (+ getprosol)**
Pulse programme: **awprotonesprf1prf2**

TD =64 K, **SI** = 64 K.
SW = 18 ppm.

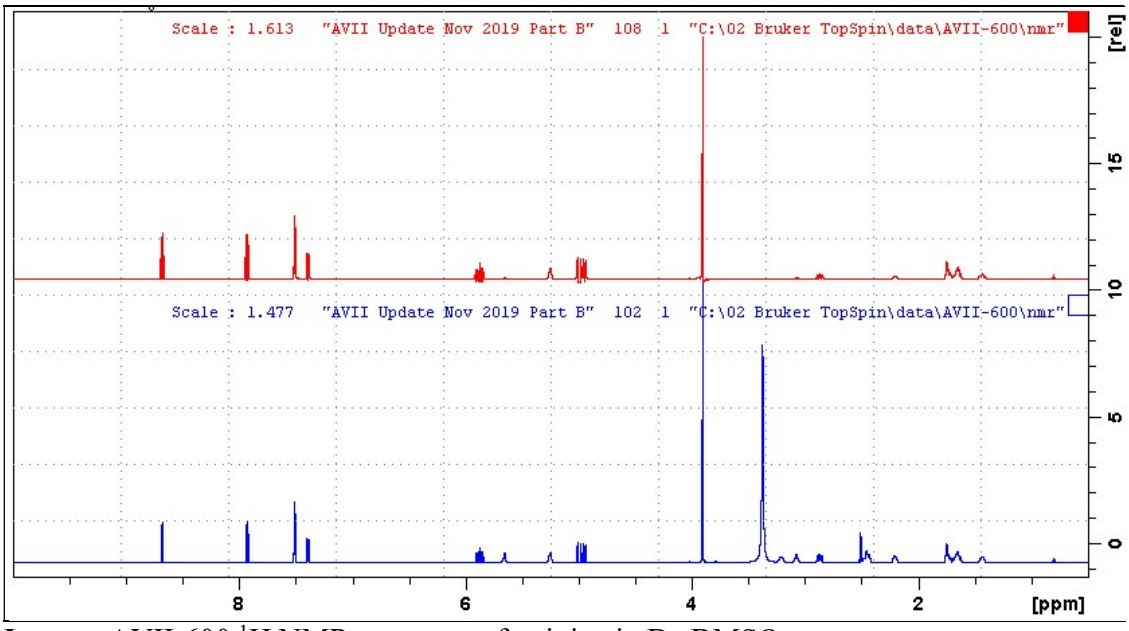
O1 = frequency in Hz of the F1 signal to be combined ES + CW suppressed.
= spectral window mid-point. Check SW is wide enough.
O2 = frequency in Hz of the F2 signal to be CW presaturated.

PL9 = F1 presaturation power applied during **D1**.
PL21 = F2 presaturation power applied during **D1**.
D1 = 2 sec or other time of your choice.

Type **ased** (enter) and review parameters used in the job. Verify gradients are OK.
Check **P40 = 2000 usec**, **SPNAM10** = **squa100.1000**.

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

Process with **EFP** (applies **LB**).



Lower: AVII-600 ^{1}H NMR spectrum of quinine in D_6-DMSO.
Upper: ^{1}H NMR with combined ES + CW presaturation of the HOD line (3.37 ppm) on F1 and the DMSO line (2.5 ppm) on F2.

2.7 ^{1}H NMR spectrum with three peak ES+ dual CW presaturation

Parameter set: **awprotonesprf1prf2 (+ getprosol)**
Pulse programme **awprotonesprf1prf2**

TD = 64 K, **SI** = 64 K.
SW = 20 ppm.

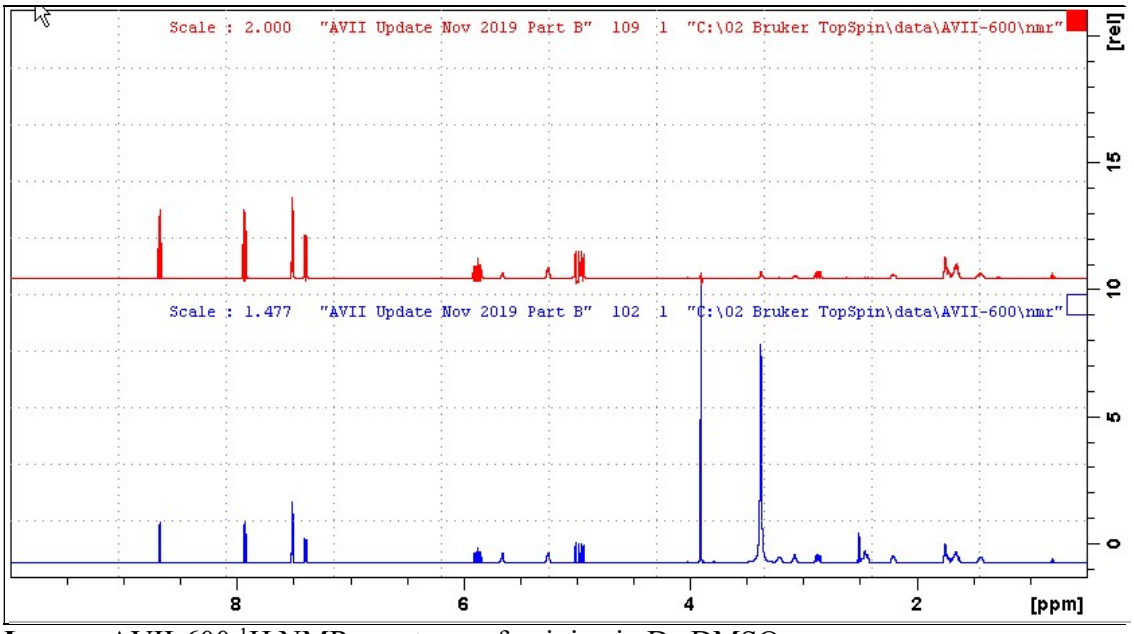
O1 = frequency in Hz of the F1 signal to be CW suppressed.
= spectral window midpoint. Check SW is wide enough.
O1* = frequency in Hz of the F1 signal to be ES suppressed
SPOFFS10 = **(O1*-O1)** Hz (may be a positive of negative value).
O2 = frequency in Hz of the F2 signal to be CW presaturated.

PL9 = F1 presaturation power applied during **D1**.
PL21 = F2 presaturation power applied during **D1.**
D1 = 2 sec or other time of your choice.

Type **ased** (enter) and review parameters used in the job. Verify gradients are OK.
Check **P40** = **2000 usec**, **SPNAM10** = **squa100.1000**.

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

Process with **EFP** (applies **LB**).



Lower: AVII-600 ^{1}H NMR spectrum of quinine in D_6-DMSO.
Upper: ^{1}H NMR with CW presaturation on F1 of quinine's OCH_3 signal (3.89 ppm), offset ES suppression of the HOD line (3.37 ppm) and CW presaturation on F2 of the DMSO signal (2.5 ppm).

2.8 NOESYPR1D with CW presaturation

Parameter set: **awnoesypr1d (+ getprosol)**
Pulse programme: **awnoesypr1d**

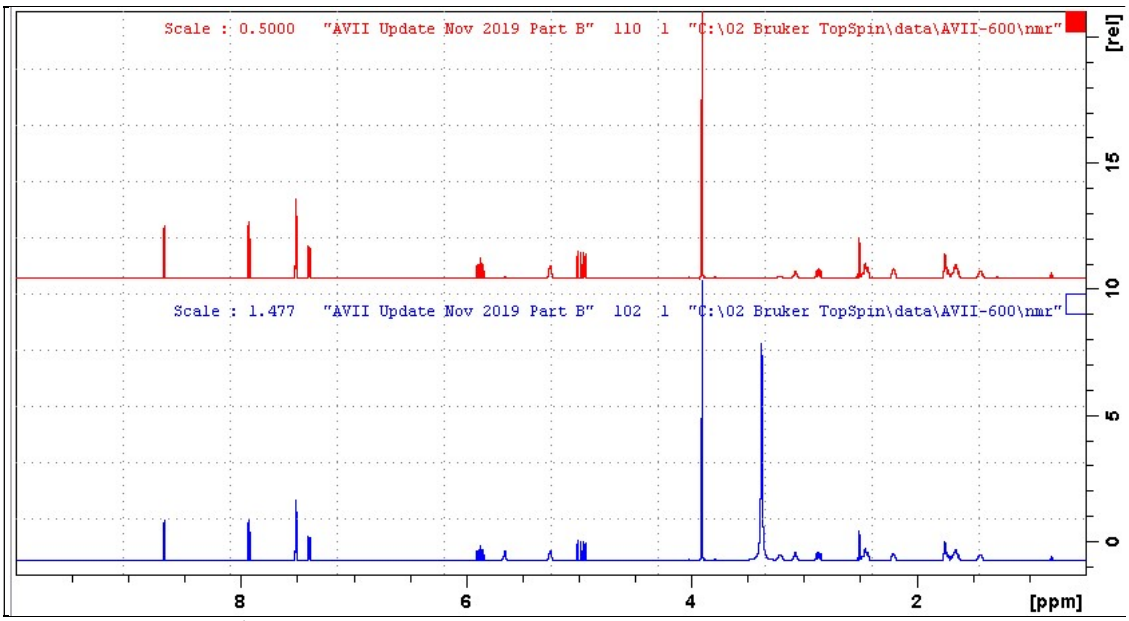
TD = 32 K or 64K , **SI** = 32 K or 64 K.
SW = 18 ppm.

O1 = frequency in Hz of the F1 signal to be presaturated.
= spectral window midpoint. Check **SW** is wide enough.
PL9 = **F1**presaturation power applied during **D1**.
D1 = 2 sec or other time of your choice.
D8 = 0.05 sec (NOESY delay) or other time of your choice.

Type **ased** (enter) and review parameters used in the job.
Add (or subtract) 3-12 db to **PL9** to decrease (or increase) the presaturation power.
6 db = a factor of 2. A larger attenuation setting decreases the power level.

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

Process with **EFP** (applies **LB**).



Lower: AVII-600 ^{1}H NMR spectrum of quinine in D_6-DMSO.
Upper: ^{1}H NOESYPR1D spectrum with CW presaturation of the HOD line at 3.37 ppm.

2.9 ^{1}H NMR spectrum with PS presaturation

Parameter set: **awprotonps (+ getprosol)**
Pulse programme: **awprotonps**
TD = 64K, **SI** = 64 K.
SW = 18 ppm.

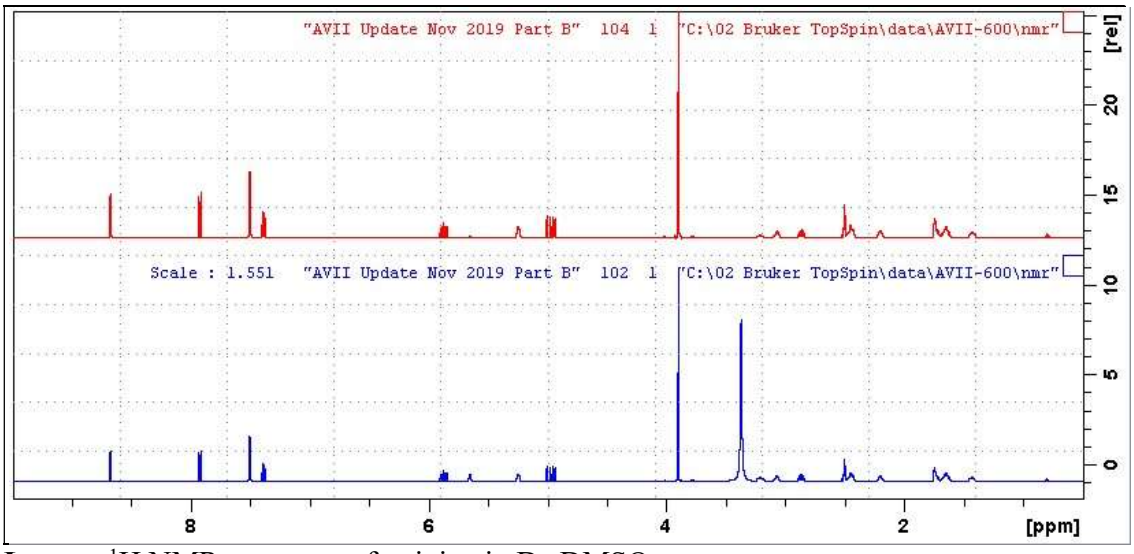
O1 = frequency in Hz of the F1 signal to be presaturated.
= spectral window midpoint. Check **SW** is wide enough.

D1 = **2 sec** or other time of your choice.
P18 = 100 msec Squa100.1000 pulse for presaturation.
L6 = number of **P18** pulse loops is auto-calculated from **D1**.
SP6(db) = F1 prosol linked presaturation power applied during **D1** (typically ca 48 db).
Add (or subtract) 3-12 db to **SP6(db)** to decrease (or increase) the presaturation power.
6 db = a factor of 2. A larger attenuation setting decreases the power level.

Type **ased** (enter) and review parameters used in the job.
Add (or subtract) 3-12 db to **SP6(db)** to decrease (or increase) the presaturation power.
6 db = a factor of 2. A larger attenuation setting decreases the power level.

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

Process with **EFP** (applies **LB**).



Lower: ^{1}H NMR spectrum of quinine in D_6-DMSO.
Upper: ^{1}H NMR spectrum with pulsed presaturation of the HOD line at 3.37 ppm.

2.10 ^{1}H NMR spectrum with dual PS presaturation

Parameter set: **awprotonpsf1psf2 (+ getprosol)**
Pulse programme: **awprotonpsf1psf2**

TD = 64 K, **SI** = 64 K.
SW = 18 ppm.

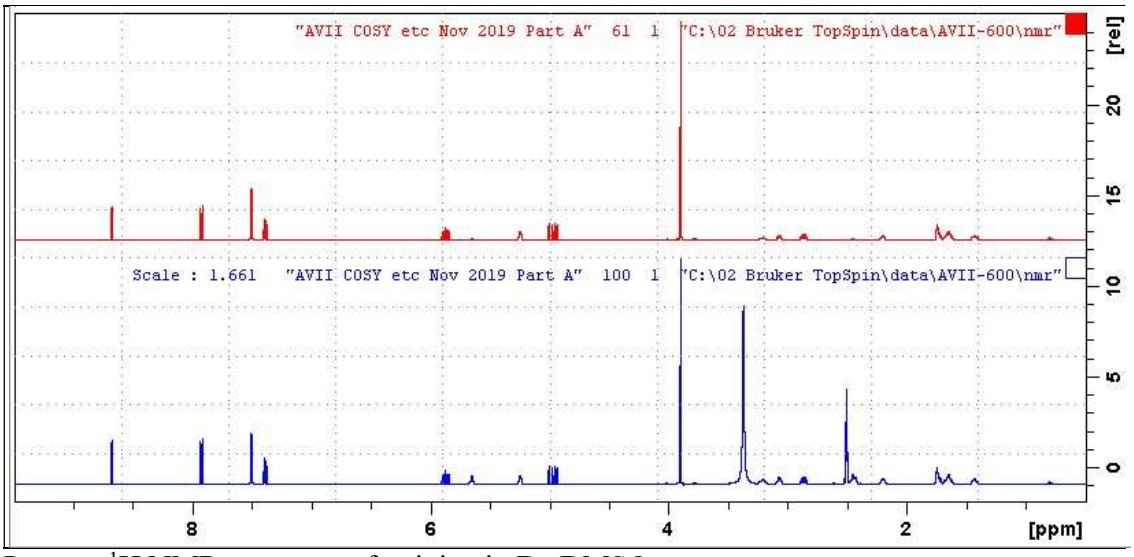
O1 = frequency in Hz of the F1 signal to be presaturated.
= spectral window midpoint. Check **SW** is wide enough.
O2 = frequency in Hz of the F2 signal to be presaturated.

D1 = 2 sec or other time of your choice.
P18 = 100 msec Squa100.1000 pulse for presaturation.
L6 = number of P18 pulse loops is auto-calculated from **D1**.
SP6(db) = F1 prosol linked presaturation power applied during **D1** (typically ca 48 db).
SP56(db) = F2 presaturation power applied during **D1** *is not prosol Table linked* **.**
SP56(db) is typically set to the same power level as that for **SP6(db)**
Add (or subtract) 3-12 db to **SP6(db) and/or SP56(db)** to decrease (or increase) the presaturation power. 6 db = a factor of 2. A larger attenuation setting decreases the power level.

Type **ased** (enter) and review parameters used in the job.

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

Process with **EFP** (applies **LB**).



Lower: ^{1}H NMR spectrum of quinine in D_6-DMSO.
Upper: ^{1}H NMR spectrum with pulsed presaturation of the HOD (3.37 ppm) and DMSO (2.5 ppm) lines.