KJM 9250

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

Version 1.0

Topspin 3.5 Windows 7
Topspin 1.3 Windows XP

© Professor Emeritus Alistair Lawrence Wilkins,
University of Waikato, New Zealand.
January 2018

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

1.0 Introduction

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

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.

2.0 ^{1}H NMR experiments

> **2.1 ^{1}H NMR with a 30, 45 or 90 degree pulse**
>
> **2.2 ^{1}H NMR with CW presaturation**
>
> **2.3 ^{1}H NMR with dual 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**

2.1 ¹H 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
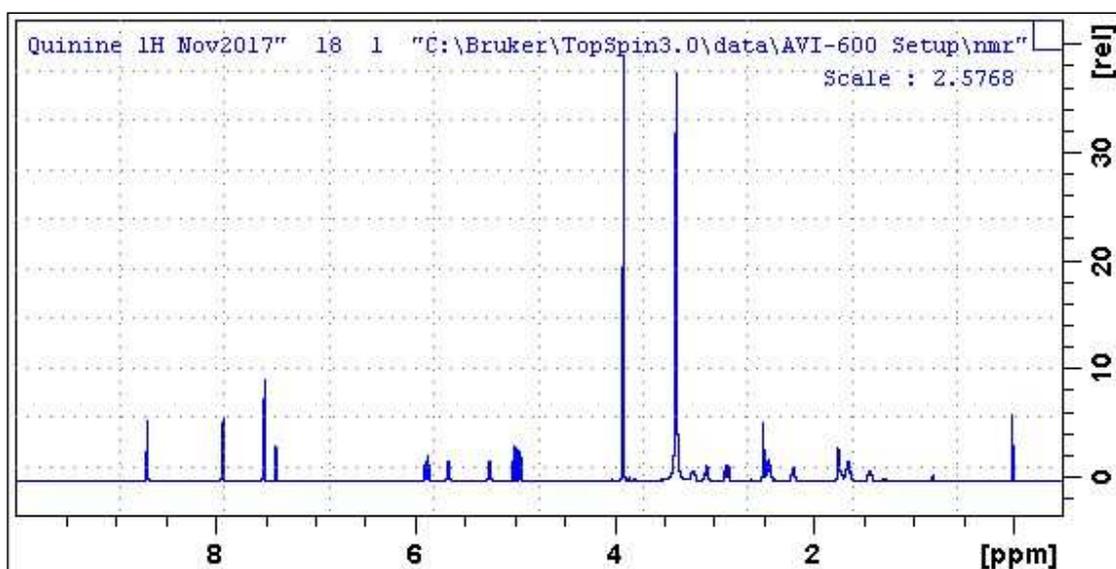**SW** = 16 ppm, **O1P** = 7.0 ppm.
D1 = 1.5 sec or other time of your choice.
NS = any number, **DS** = 2, 4, 8.

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**).

600 MHz ¹H NMR spectrum of quinine in D₆-DMSO.

2.2 ^{1}H NMR spectrum with CW presaturation

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

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**.
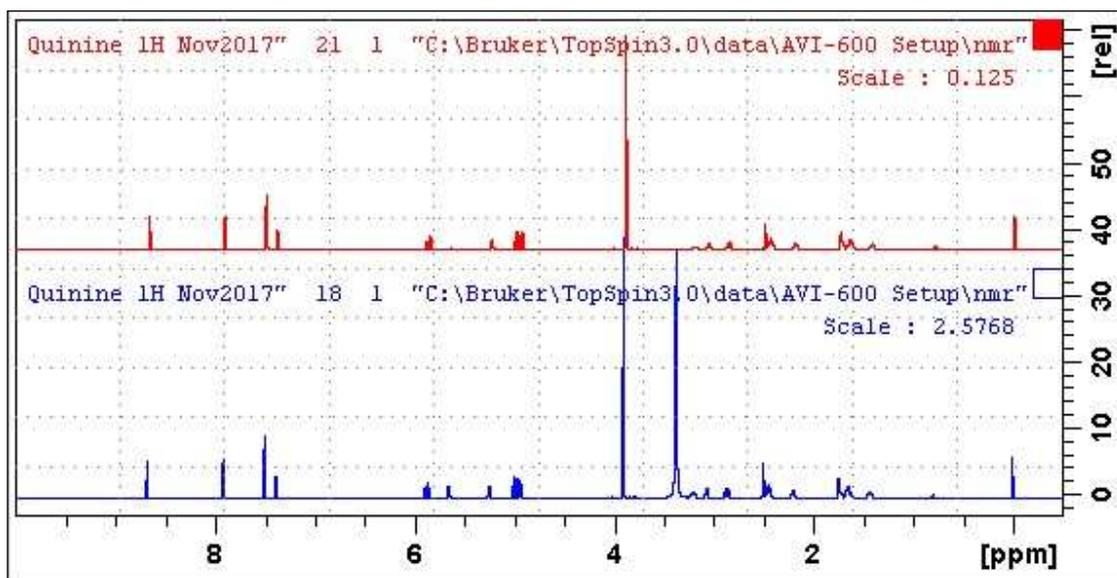**D1** = 2 sec or other time of your choice.

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 <u>larger</u> attenuation setting <u>decreases</u> the power level.

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

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

Lower: 600 MHz ^{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.
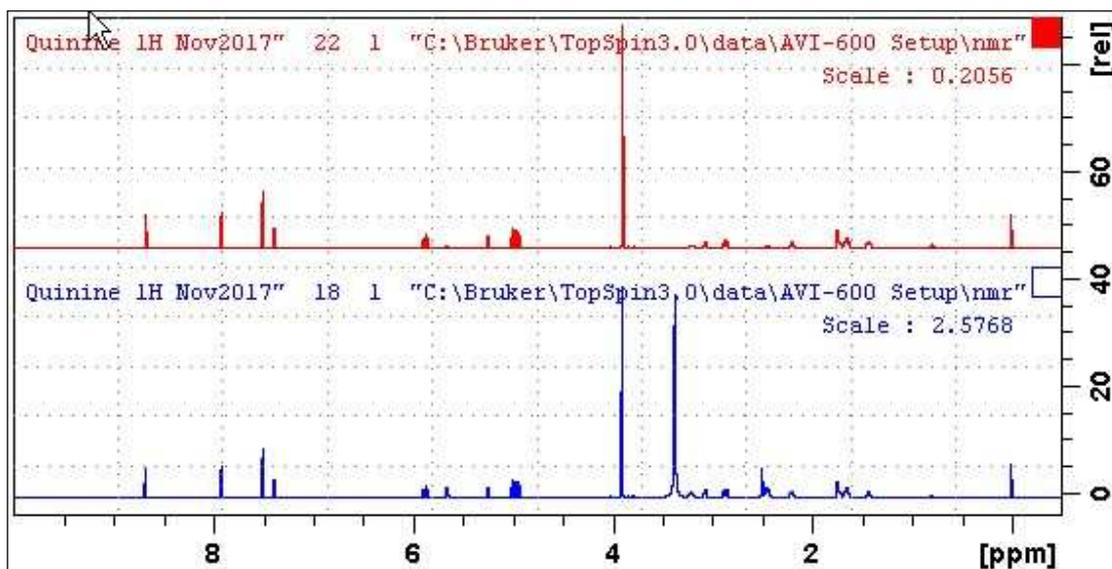**PL21** = F2 presaturation power applied during D1.
D1 = 2 sec or other time of your choice.

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 <u>larger</u> attenuation setting <u>decreases</u> the power level.

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

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

Lower: 600 MHz ^{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 sets: **awprotones (+ getprosol)**
Pulse programmes: **zgesgp**

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

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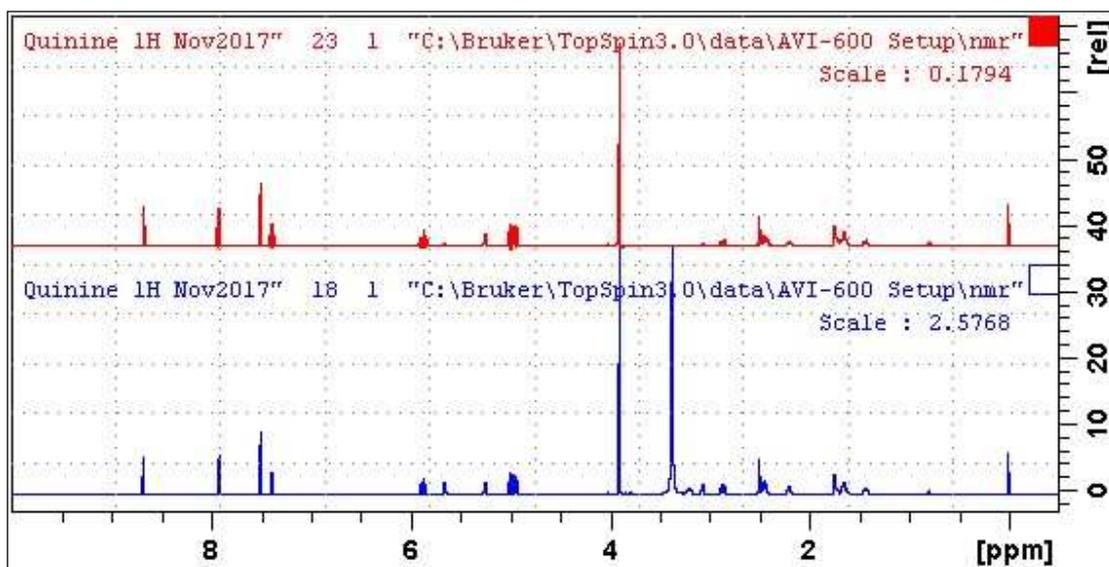   = spectral window midpoint Check. **SW** is wide enough.
D1 = 2 sec or other time of your choice.

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: 600 MHz ^{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 programmes: **awprotonespr**

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

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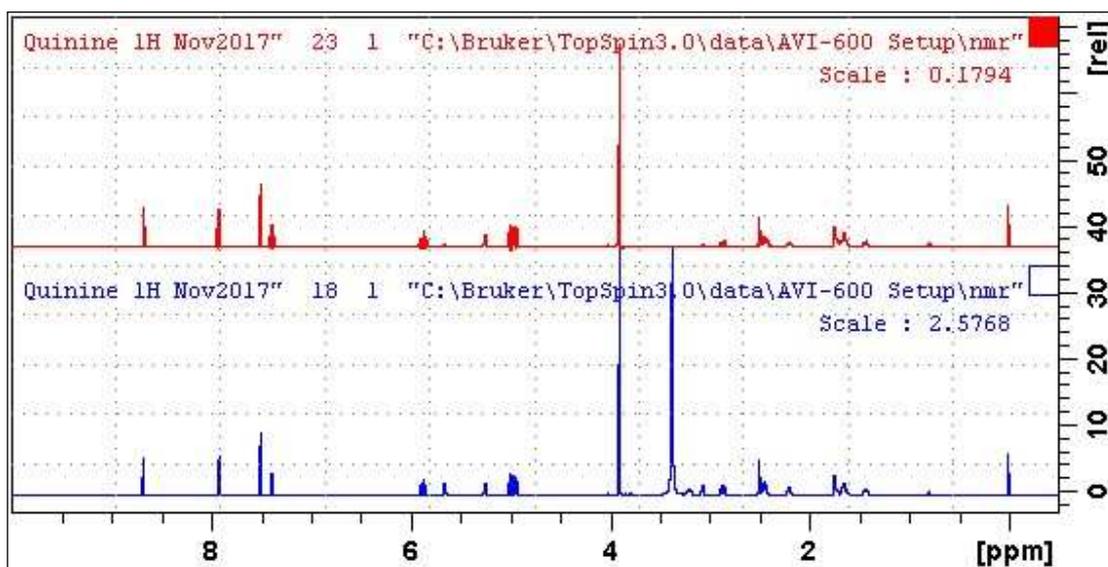   = spectral window midpoint. Check SW is wide enough.
PL9 = F1 presaturation power applied during D1.
D1 = 2 **sec** or other time of your choice.

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: 600 MHz ^{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 ¹H NMR spectrum with combined ES+CW presaturation on F1 and CW presaturation on F2

Parameter set: **awprotonesprf1prf2 (+ getprosol)**
Pulse programmes: **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**.
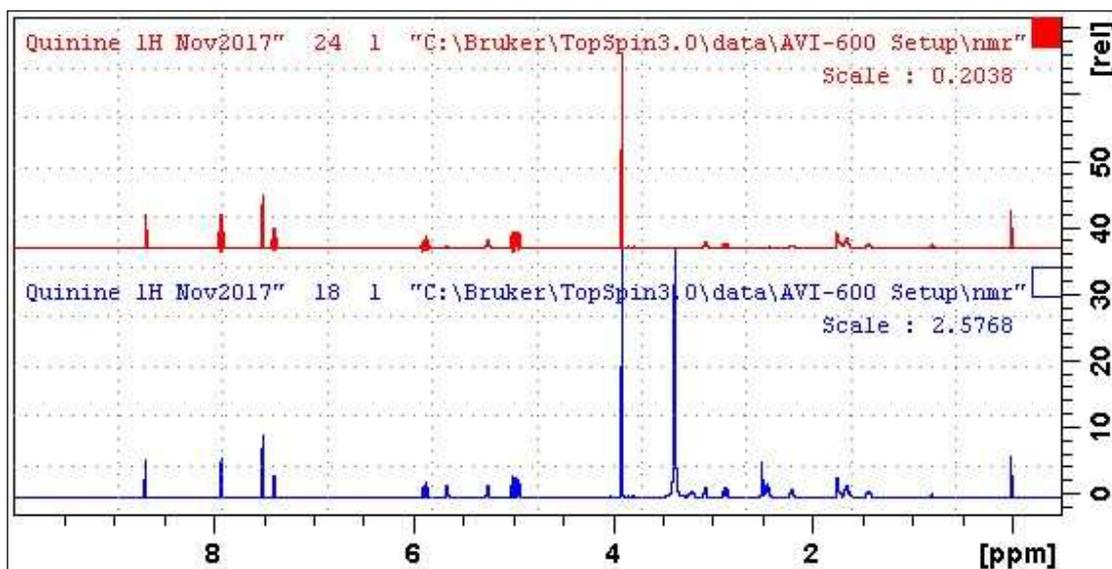**PL21** = F2 presaturation power applied during **D1**.
D1 = 2 sec or other time of your choice.

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: 600 MHz ¹H NMR spectrum of quinine in D_6-DMSO.
Upper: ¹H 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 ¹H NMR spectrum with three peak ES + dual CW presaturation

Parameter set: **awprotonesprf1prf2 (+ getprosol)**
Pulse programmes: **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**.
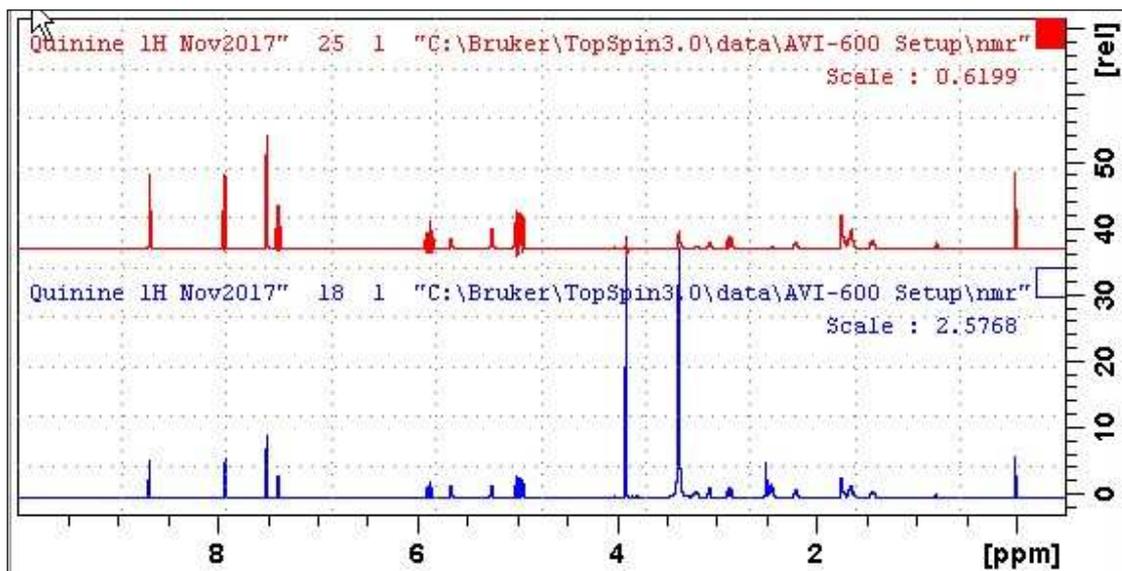**PL21** = F2 presaturation power applied during **D1.**
D1 = 2 sec or other time of your choice.

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: 600 MHz ¹H NMR spectrum of quinine in D_6-DMSO.
Upper: ¹H 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).