



KJM 9250
AVIIIHD-800 Homonuclear Decoupling Experiments

Version 1.0

Topspin 3.5
Windows 7



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

AVIIIHD-800 Homonuclear Decoupling Experiments

1.0 Introduction

Homonuclear decoupling experiments can be performed using Bruker's `zghd` or `zghd.2` pulse programmes which incorporate homonuclear decoupling during FD acquisition, or during both FID acquisition and the interpulse delay period (**d1**), respectively.

Variants of Bruker's `zghd` pulse programme which incorporate excitation sculpturing (ES), continuous wave (CW) presaturation, or combined ES and CW presaturation on F1 have been created.

The `hd` power level can be *increased* (raised) by subtracting 3-12 db or decreased (attenuated) by adding 3-12 db *respectively* to the prosol Table linked **PL24** power level which is applied via F2.

The application of a decoupling radio frequency slightly increases the frequency of nearby signals. This effect is known as the Bloch-Seigert effect.

2.0 Homonuclear Decoupling Experiments

The following **aw** coded **homonuclear decoupled** parameter sets are installed on the AVIIIHD-800. Identical **uio** coded versions of these parameter sets have also been saved

- | | | |
|------------|-----------------------|--|
| 2.1 | awprotonhd | <i>with hd during FID acquisition</i> |
| 2.2 | awprotonhd.2 | <i>with hd during D1 + FID acquisition</i> |
| 2.3 | awprotonhdpr | <i>with pr during D1 + hd during FID acquisition</i> |
| 2.4 | awprotoneshd | <i>with ES peak suppression + hd during FID acquisition</i> |
| 2.5 | awprotoneshdpr | <i>with combined ES and pr + hd during FID acquisition</i> |
| 2.6 | awprotoneshdpr | <i>with two peaks suppressed + hd during FID acquisition</i> |

2.1 awprotonhd with homonuclear decoupling at O2 during FID acquisition

parameter set: **awprotonhd** (+ **getprosol**)

pulse programme: **zghd**

Prior to setting up a **homonuclear decoupling** experiment determine the frequency in **Hz** of the signal to be decoupled in a standard ^1H NMR spectrum.

This experiment runs with **DIGMOD = digital**

Type **O2** (enter) and enter the frequency in Hz of the signal to be decoupled.

Type **eda** (enter) and adjust acquisition parameters as required.

SW = 16 ppm, **O1** = 6 ppm, **TD** = 64 K points or other values of your choice.

NS = multiple of 2, 4 or 8, **DS** = 2, 4 or 8.

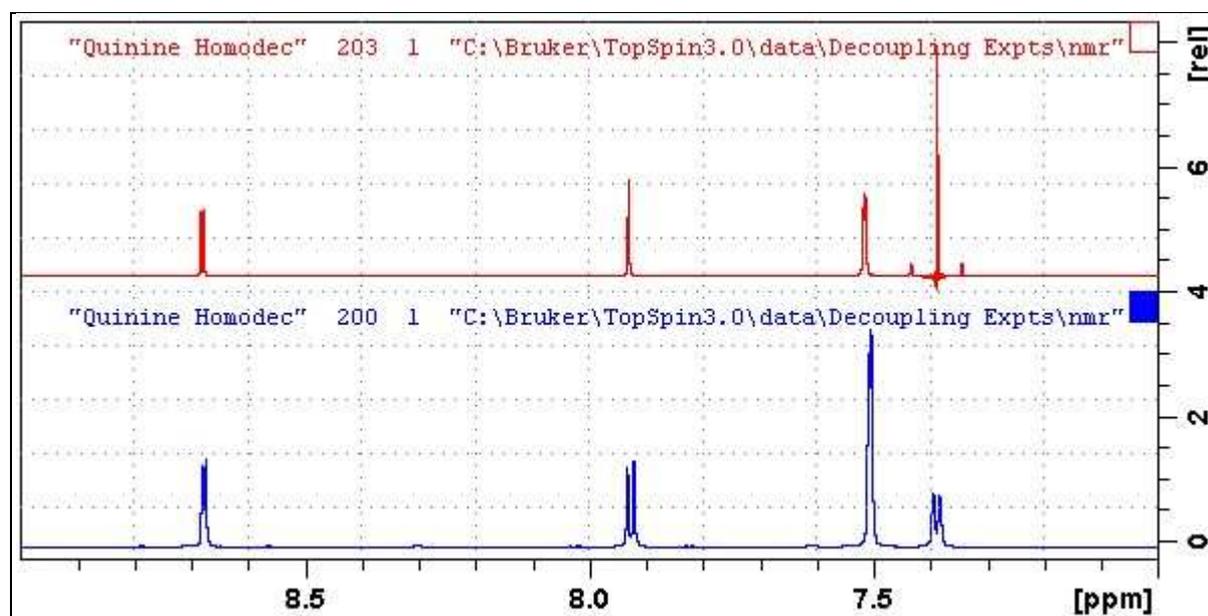
D1 = repetition delay = 2.0, 3.0 sec or other time of your choice.

Type **ased** (enter) and review other parameters including the **PL24** power level.

Do not adjust the **PL1** or **PL2** power levels.

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

Process with: **EF** or **EFP** (applies **LB** = 0.1, 0.3 Hz or other values of your choice)
or **FT** (no line broadening factor applied).



Lower: Expansion of the 7-9 ppm region of the AVIIIHD-800 ^1H NMR spectrum of quinine in $\text{D}_6\text{-DMSO}$.

Upper: Homonuclear decoupling during FID acquisition of the signal at 7.49 ppm. The signal at 7.93 ppm is collapsed to a singlet.

2.2 awprotonhd.2 with homonuclear decoupling during d1 and FID acquisition

parameter set: **awprotonhd.2** (+ **getprosol**)

pulse programme: **zghd.2**

Prior to setting up a **homonuclear decoupling** experiment determine the frequency in **Hz** of the signal to be decoupled in a standard ^1H NMR spectrum.

This experiment runs with **DIGMOD = digital**

Type **O2** (enter) and enter the frequency in Hz of the signal to be decoupled.

Type **eda** (enter) and adjust acquisition parameters as required.

SW = 16 ppm, **O1** = 6 ppm, **TD** = 64 K points or other values of your choice.

NS = multiple of 2, 4 or 8, **DS** = 2, 4 or 8.

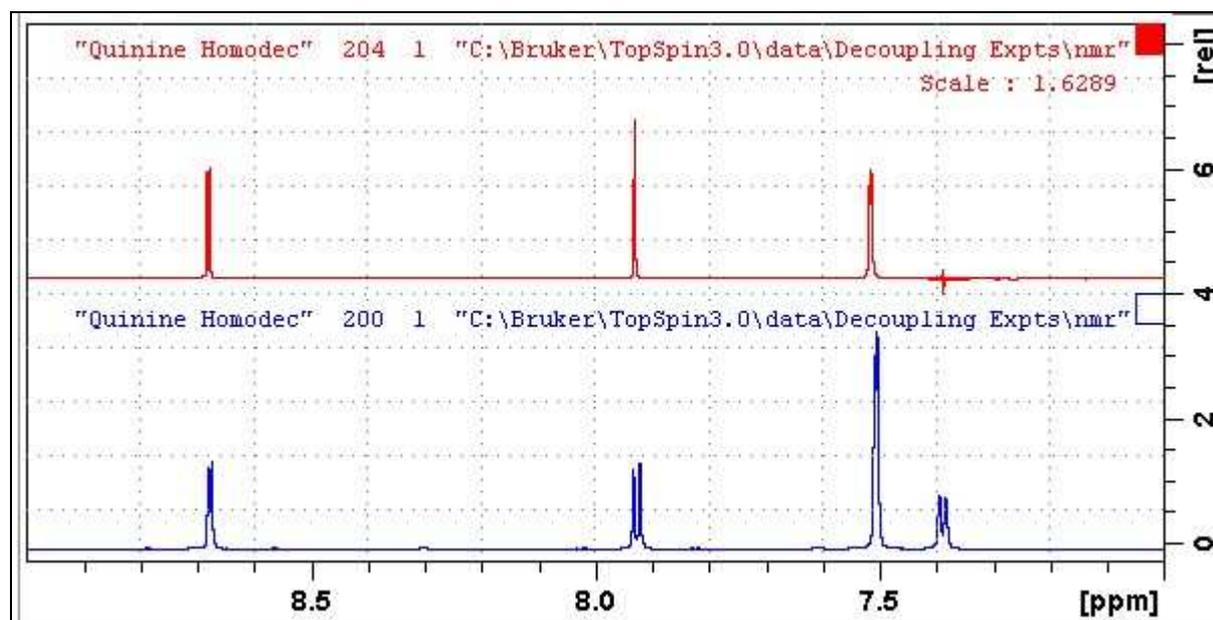
D1 = repetition delay = 2.0, 3.0 sec or other time of your choice.

Type **ased** (enter) and review other parameters including the **PL24** power level.

Do not adjust the **PL1** or **PL2** power levels.

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

Process with: **EF** or **EFP** (applies **LB** = 0.1, 0.3 Hz or other values of your choice)
or **FT** (no line broadening factor applied).



Lower: Expansion of the 7-9 ppm region of the AVIIIHD-800 ^1H NMR spectrum of quinine in D_6 -DMSO.

Upper: Homonuclear decoupling during **D1** and FID acquisition of the signal at 7.49 ppm. The signal at 7.93 ppm is collapsed to a singlet.

2.3 awprotonhdpr with CW presaturation and homonuclear decoupling during FID acquisition

parameter set: **awprotonhdpr (+ getprosol)**
pulse programme: **awzghdpr**

Prior to setting up a **homonuclear decoupling** experiment determine the frequencies in **Hz** of the signals to be presaturated (**O1**) and decoupled (**O2**) respectively in a standard ^1H NMR spectrum.

This experiment runs with **DIGMOD = digital**.

Type **O1** (enter) and enter the frequency in Hz of the signal to be presaturated.

O1 will be set as the spectrum's midpoint.

Type **O2** (enter) and enter the frequency in Hz of the signal to be decoupled.

Type **eda** (enter) and adjust acquisition parameters as required.

SW = 16 ppm, **TD** = 64 K points or other values of your choice.

NS = multiple of 2, 4 or 8, **DS** = 2, 4 or 8.

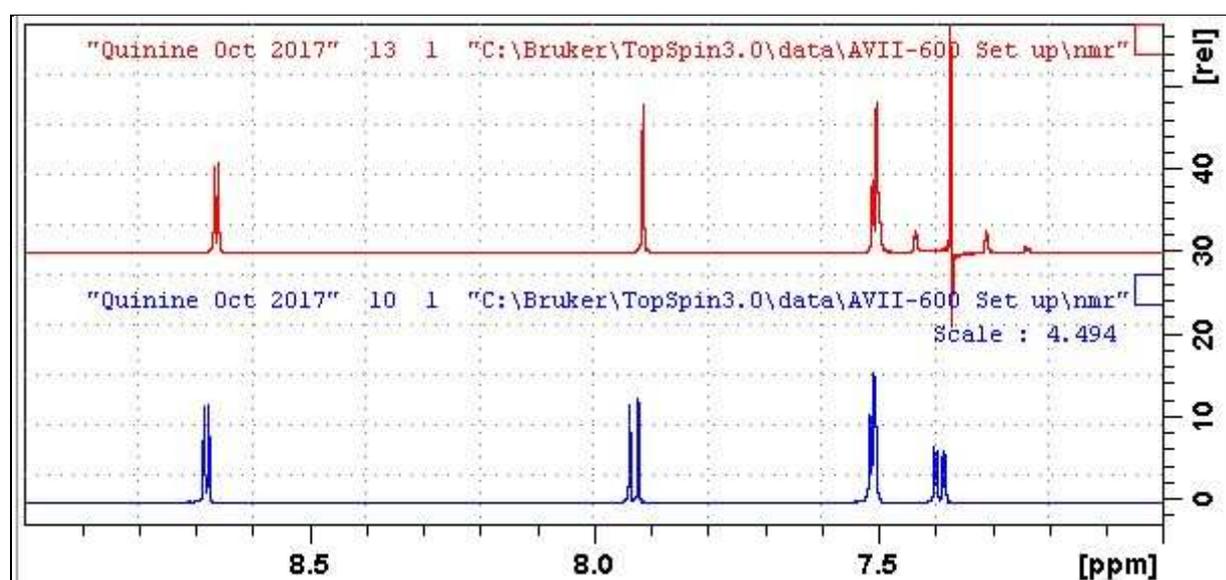
D1 = repetition delay = 2.0, 3.0 sec or other time of your choice.

PL9 = presaturation power level, applied during **D1**.

Type **ased** (return) and review other parameters including the **PL24** hd and **PL9** presaturation power levels

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

Process with: **EF** or **EFP** (applies **LB** = 0.1, 0.3 Hz or other values of your choice)
or **FT** (no line broadening factor applied)



Lower: Expansion of the 7-9 ppm region of the AVIIHD-800 ^1H NMR spectrum of quinine in D_6 -DMSO.

Upper: Homonuclear decoupling during FID acquisition of the signal at 7.49 ppm. CW presaturation was applied during **d1** to the HOD signal at 3.38 ppm. The signal at 7.93 ppm is collapsed to a singlet.

2.4 awprotoneshd with ES peak suppression and homonuclear decoupling during FID acquisition

parameter set: **awprotoneshd (+ getprosol)**
pulse programme: **awzgeshd**

Prior to setting up a **homonuclear decoupling** experiment determine the frequencies in **Hz** of the signals to be **ES** suppressed (**O1**) and decoupled (**O2**) respectively in a standard ^1H NMR spectrum.

This experiment runs with **DIGMOD = digital**.

Type **O1** (enter) and enter the frequency in Hz of the signal to be presaturated.

O1 will be set as the spectrum's midpoint.

Type **O2** (enter) and enter the frequency in Hz of the signal to be decoupled.

Type **eda** (enter) and adjust acquisition parameters as required..

SW = 16 ppm, **TD** = 64 K points or other values of your choice.

NS = multiple of 2, 4 or 8, **DS** = 2, 4 or 8

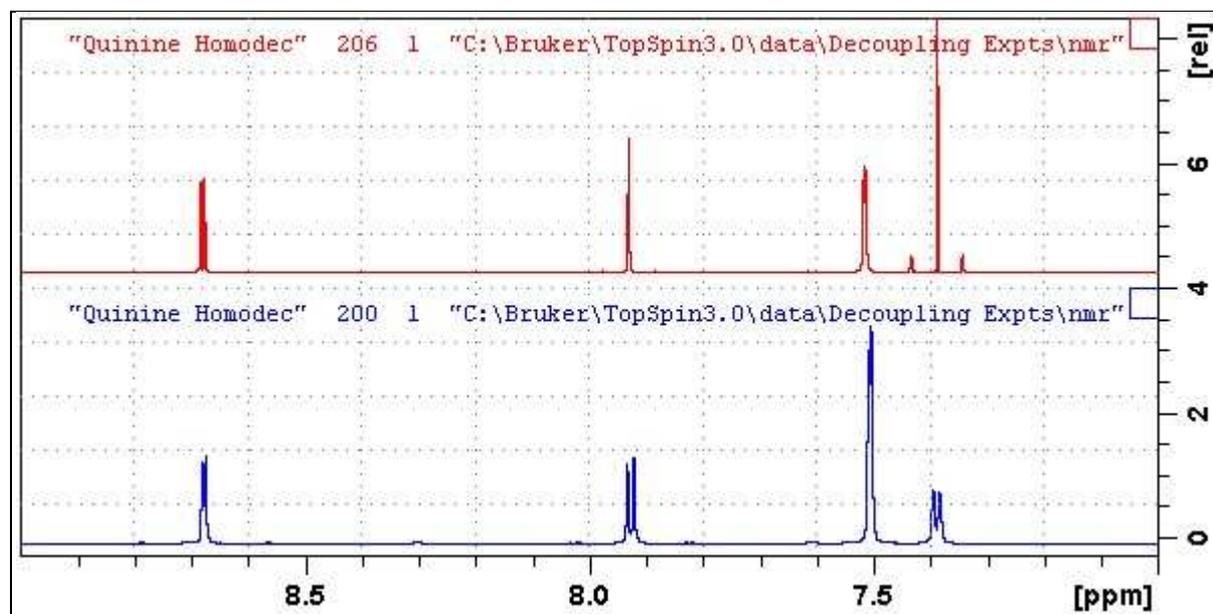
D1 = repetition delay = 2.0, 3.0 sec or other time of your choice.

Type **ased** (enter) and review other parameters including the **PL24** hd power level.

Check **SMSQ10.100** gradients are set to 31% (GPZ1) and 11% (GPZ2).

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

Process with: **EF** or **EFP** (applies **LB** = 0.1, 0.3 Hz or other values of your choice) or **FT** (no line broadening factor applied).



Lower: Expansion of the 7-9 ppm region of the AVIIIHD-800 ^1H NMR spectrum of quinine in D_6 -DMSO.

Upper: Homonuclear decoupling during FID acquisition of the signal at 7.49 ppm. ES was applied to the HOD signal at 3.38 ppm. The signal at 7.93 ppm is collapsed to a singlet.

2.5 awprotoneshdpr with combined ES + CW presaturation and homonuclear decoupling during FID acquisition

parameter set: **awprotoneshdpr (+ getprosol)**
pulse programme: **awzgeshdpr**

Prior to setting up a **homonuclear decoupling** experiment determine the frequencies in **Hz** of the signals to be combined **ES** and **CW** suppressed (**O1**) and decoupled (**O2**) respectively in a standard ^1H NMR spectrum.

This experiment runs with **DIGMOD = digital**

Type **O1** (enter) and enter the frequency in Hz of the signal to be presaturated.

O1 will be set as the spectrum's midpoint.

Type **O2** (enter) and enter the frequency in Hz of the signal to be decoupled.

Type **eda** (enter) and adjust acquisition parameters as required.

SW = 16 ppm, **TD** = 64 K points or other values of your choice.

NS = multiple of 2, 4 or 8, **DS** = 2, 4 or 8.

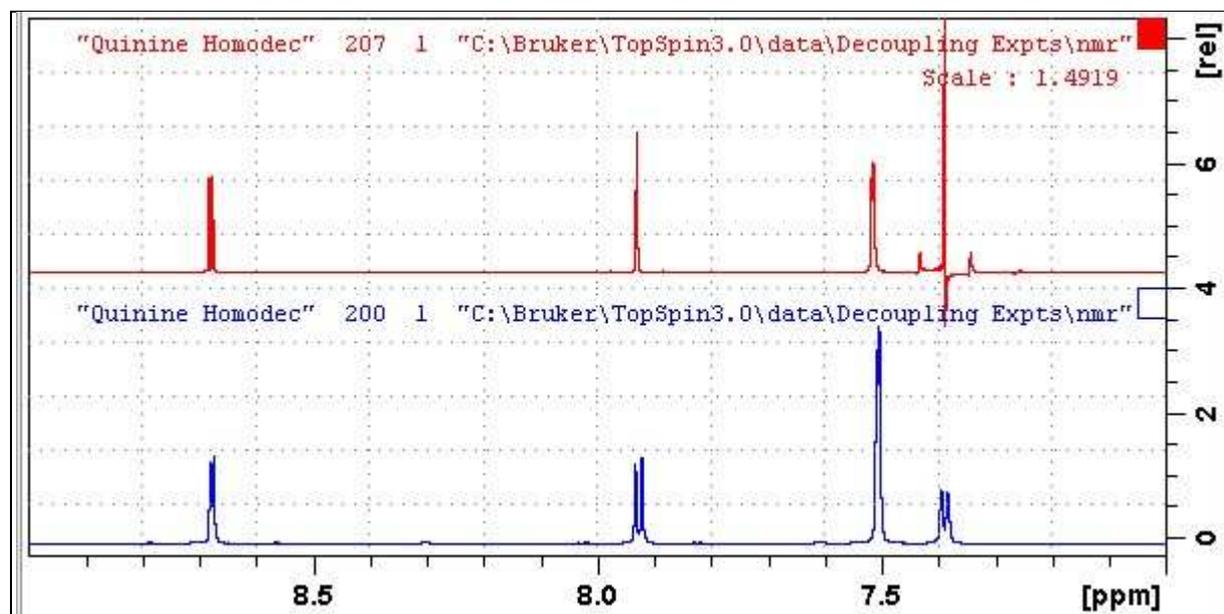
D1 = repetition delay = 2.0, 3.0 sec or other time of your choice.

PL 9 = CW presaturation power level applied during **D1**.

Type **ased** (enter) and review parameters including the **PL24** hd and **PL9** presaturation power levels. Check **SMSQ10.100** gradients are set to 31% (GPZ1) and 11% (GPZ2).

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

Process with: **EF** or **EFP** (applies **LB** = 0.1, 0.3 Hz or other values of your choice)
or **FT** (no line broadening factor applied).



Lower: Expansion of the 7-9 ppm region of the AVIIIHD-800 ^1H NMR spectrum of quinine in D_6 -DMSO.

Upper: Homonuclear decoupling during FID acquisition of the signal at 7.49 ppm. Combined ES + CW presaturation was applied to the HOD signal at 3.38 ppm. The signal at 7.93 ppm is collapsed to a singlet.

2.6 awprotoneshdpr with two peak suppression and homonuclear decoupling during FID acquisition

parameter set: awprotoneshdpr (+ getprosol)
pulse programme: awzgeshdpr

Prior to setting up a two peak suppressed **homonuclear decoupling** experiment determine the frequencies in **Hz** of the signals to CW presaturated (**O1**), ES suppressed (**O1***) and decoupled (**O2**) respectively in a standard ^1H NMR spectrum.

This experiment runs with **DIGMOD = digital**

Type **O1** (enter) and enter the frequency in Hz of the signal to be CW presaturated during d1.
O1 will be set as the spectrum's midpoint.

Type **SPOFFS1** (enter) and enter the frequency in Hz of the signal to be ES suppressed.
as **O1*-O1**. This offset value may be a positive or negative value.

Type **O2** (enter) and enter the frequency in Hz of the signal to be decoupled.

Type **eda** (enter) and adjust acquisition parameters as required.

SW = 16 ppm, **TD** = 64 K points or other values of your choice.

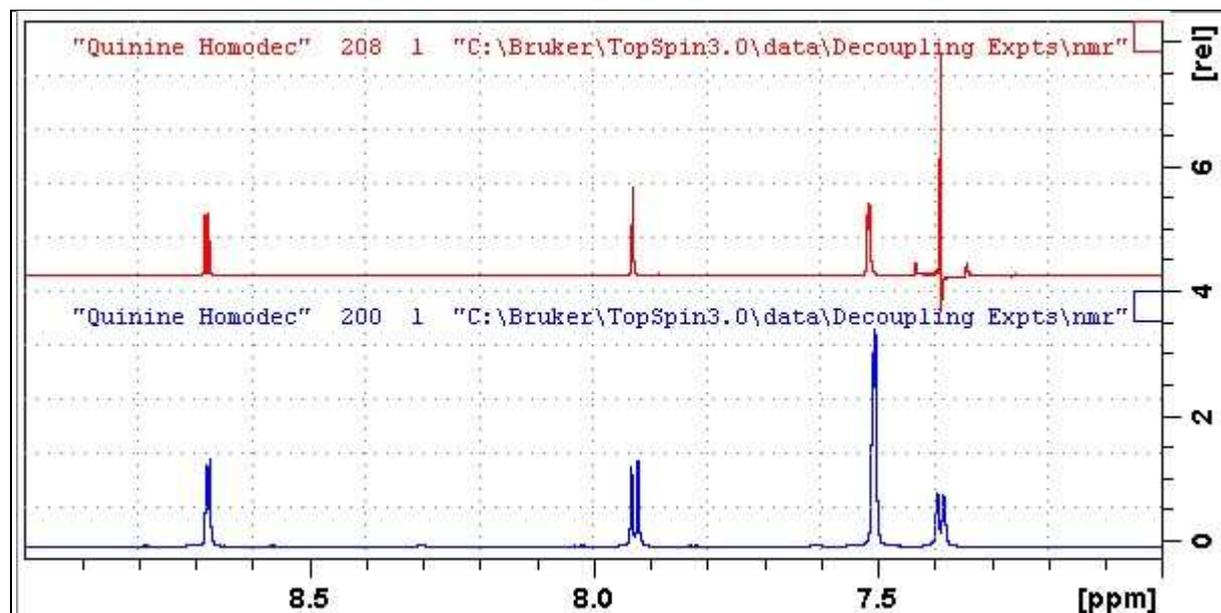
NS = multiple of 2, 4 or 8, **DS** = 2, 4 or 8.

D1 = repetition delay = 2.0, 3.0 sec or other time of your choice.

PL 9 = CW presaturation power level applied during **D1**.

Type **ased** (enter) and review other parameters including **SPOFFS1** and the **PL24** hd and **PL9** presaturation power levels. Check **SMSQ10.100** gradients are set to 31% (GPZ1) and 11% (GPZ2). Set **receiver gain** using **RGA** (*important!*).

Process with: **EF** or **EFP** (applies **LB** = 0.1, 0.3 Hz or other values of your choice)
or **FT** (no line broadening factor applied).



Lower: Expansion of the 7-9 ppm region of the AVIIIHD-800 ^1H NMR spectrum of quinine in D_6 -DMSO.

Upper: Homonuclear decoupling during FID acquisition of the signal at 7.49 ppm. ES was applied to the quinine OCH_3 signal at 3.90 ppm. CW presaturation was applied to the HOD signal at 3.38 ppm. The signal at 7.93 ppm is collapsed to a singlet.