KJM 5250 and KJM 9250

^{1}H NMR spectra on the AVneo400 spectrometer.

Version 3.1

Topspin 4.3

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AVneo400 ^{1}H NMR Spectra. Topspin 4.3.

1.0 Introduction

A series of aw coded **^{1}H NMR** parameter sets have been created on the **NEO-400** spectrometer. **NEO400** spectra can be processed using **Topspin 3.5/3.6 or TS4**.

FT and **PK** (or **APK**) 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 to 0.3 and 0 respectively) after **GFP** processing.

1.1 Presaturation Experiments

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

CW presaturation (PR) 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 program to acquire presaturated **^{1}H NMR** spectra and **QNMR** spectra.

The **ES** shaped pulse's excitation window can be decreased (softened) 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.

1.2 Homonuclear Decoupling

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. 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 ^{1}H NMR experiments

2.1 ^{1}H spectrum with a 30 or 90 degree pulse

2.2 ^{1}H spectrum with CW presaturation

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

2.4 ^{1}H spectrum with ES peak suppression

2.5 ^{1}H NOESYPR1D spectrum

2.6 ^{1}H spectrum with homonuclear decoupling during FID acquisition

or during D1 + FID acquisition

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

Parameter sets: **awproton30b or awproton90 (+ getprosol)**
Pulse programmes**: zg30 or zg respectively**

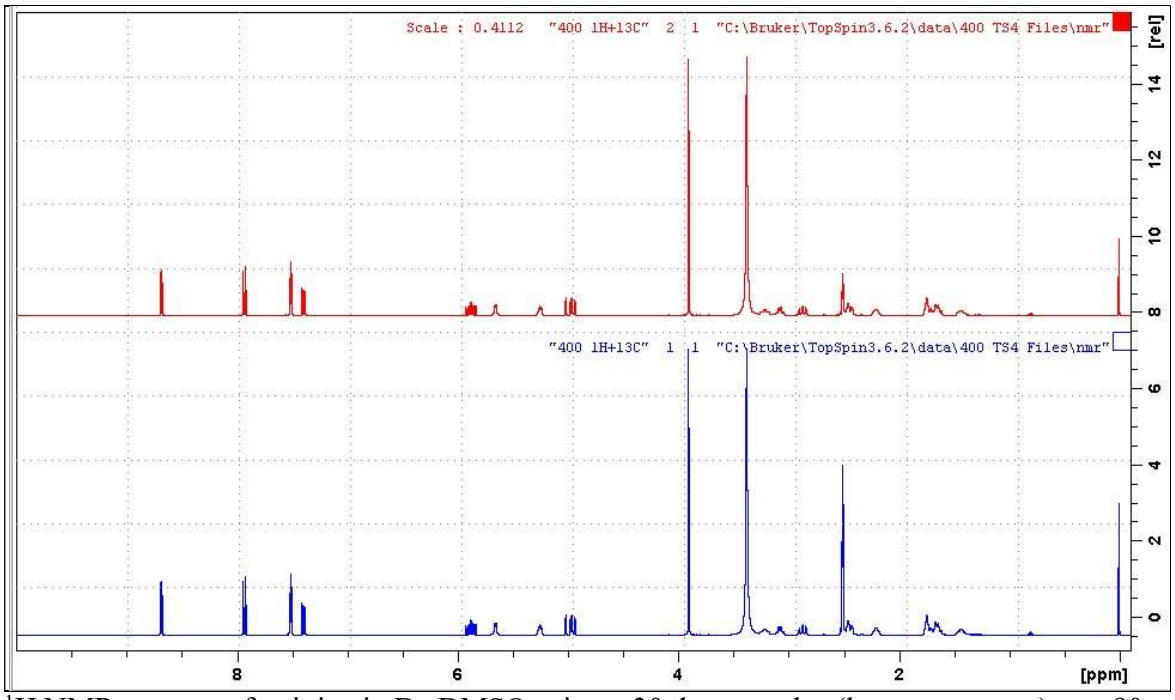
TD = 32 K, **SI** =32 K.
SW = 16 ppm, **O1P** = 6 ppm

D1 = 2 - 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**).



^{1}H NMR spectra of quinine in D_6-DMSO using a 30 degree pulse (lower spectrum) or a 90 degree pulse (upper spectrum)..

2.2 ^{1}H NMR with CW presaturation

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

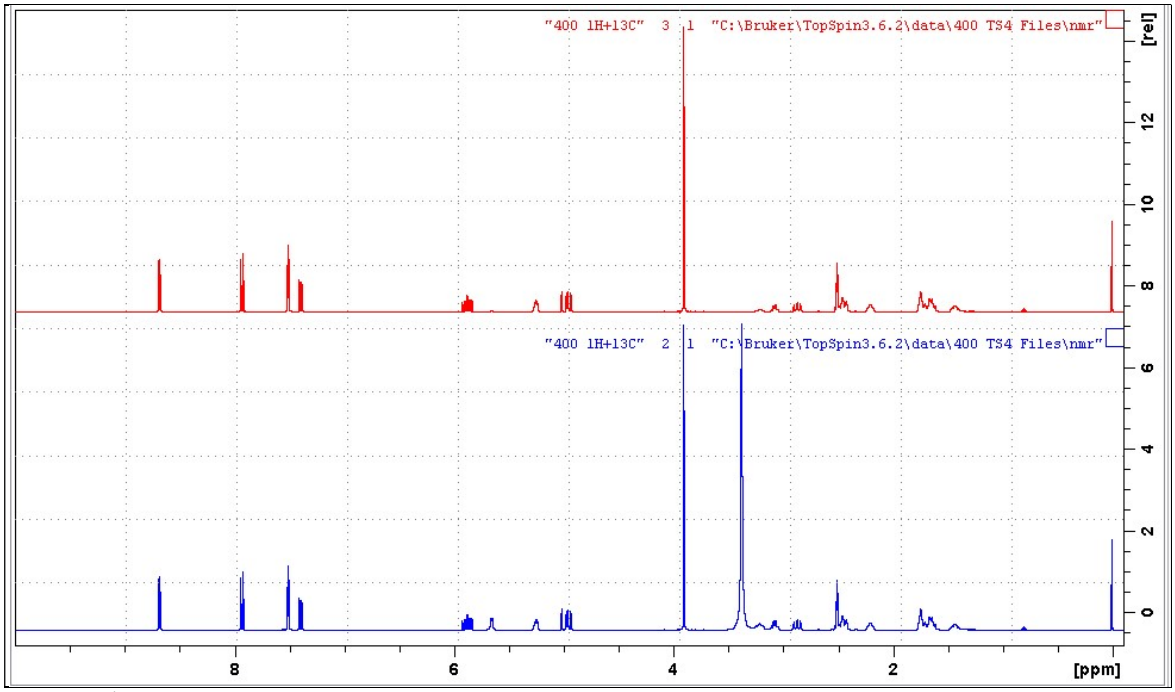
TD = 32 K, **SI** =32 K.
SW = 16 ppm, **O1P** = 6 ppm

O1 = frequency in Hz of the F1 signal to be presaturated
= spectral window mid-point. Check **SW** is wide enough.
PLW9(db) = **F1** presaturation power applied during **D1**.
D1 = 2-5 sec or other time of your choice.

Type **ased** (enter) and review parameters used in the job.
Add (or subtract) 3-12 db to **PLW9** 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 CW presaturation of the HOD line at 3.37 ppm.

2.3 ^{1}H NMR with dual CW presaturation

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

TD = 32 K, **SI** =32 K.
SW = 16 ppm, **O1P** = 6 ppm

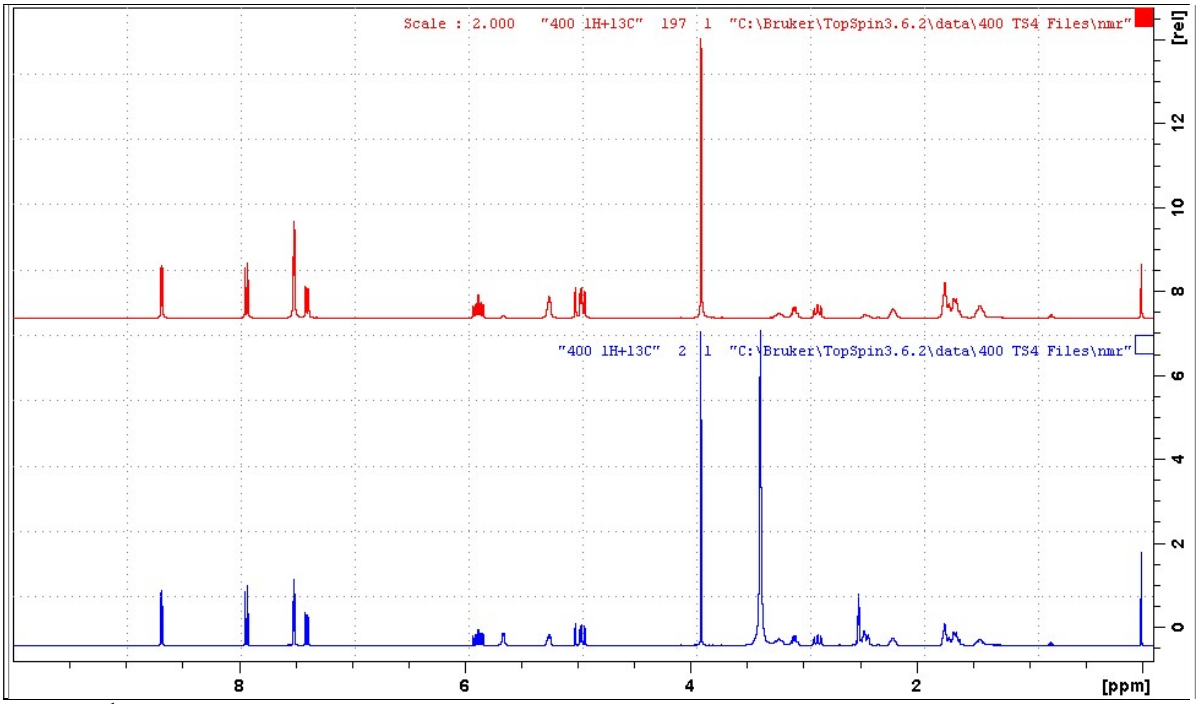
O1 = frequency in Hz of the F1 signal to be presaturated
= spectral window mid-point. Check **SW** is wide enough.
O2 = frequency in Hz of the F2 signal to be presaturated.

PL9 = F1 presaturation power applied during **D1**.
PL21 = F2 presaturation power applied during **D1**.
D1 = 2-5 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 **PL21** 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 CW presaturation of the HOD (3.37 ppm) and DMSO (2.5 ppm) lines.

2.4 ^{1}H NMR with ES peak suppression

Parameter sets: **awprotones (+ getprosol)**
Pulse programmes: **zgesgp**

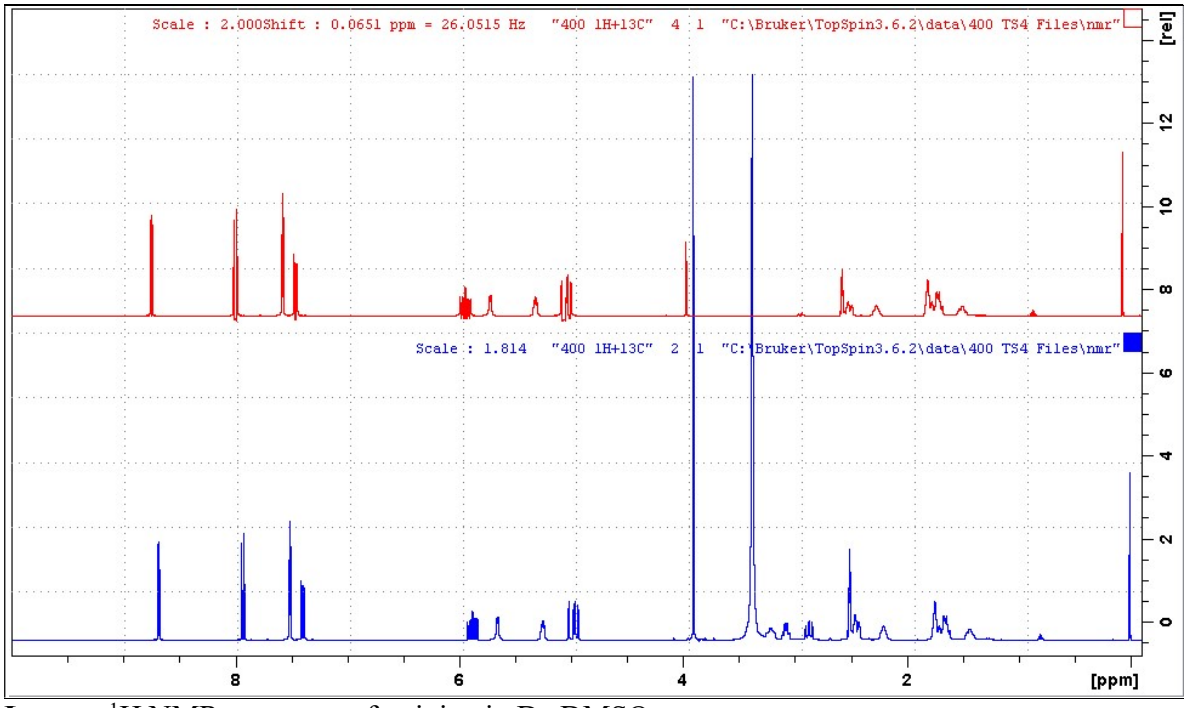
TD = 32 K, **SI** =32 K.
SW = 16 ppm, **O1P** = 6 ppm

O1 = frequency in Hz of the F1 signal to be ES suppressed
= spectral window mid-point. 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: ^{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. The ES spectrum is slightly offset relative to the 1H NMR spectrum.

400 MHz ES suppression of the 3.37 ppm HOD line significantly or completely reduces the peak areas of quinine signals located 0.5-0.8 ppm either side of the HOD line.

2.5 NOESYPR1D Spectrum with CW Presaturation

Parameter set: **awnoesypr1d (+ getprosol)**
Pulse programme: **noesypr1d**

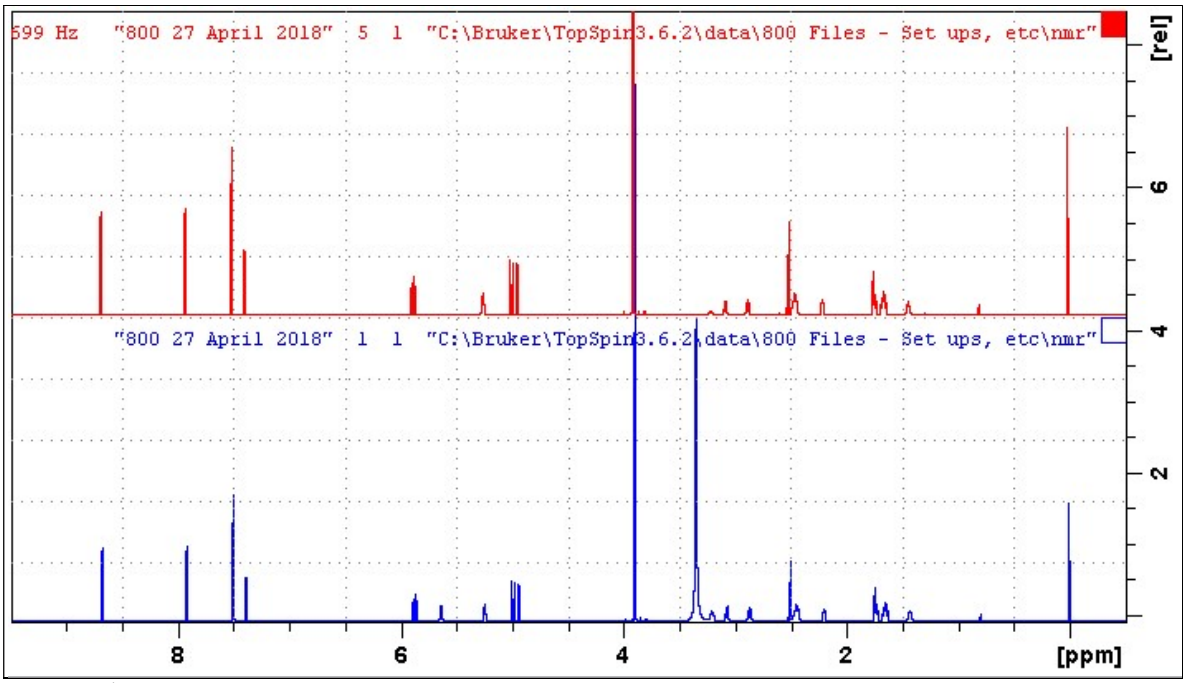
TD = 32 K, **SI** =32 K.
SW = 16 ppm, **O1P** = 6 ppm

O1 = frequency in Hz of the F1 signal to be presaturated
= spectral window mid-point. Check **SW** is wide enough.
PLW9 = prosol Table linked presaturation power applied during **D1**.
D8 = 0.05 sec or other time of your choice
D1 = 2-5 sec or other time of your choice.

Type **ased** (enter) and review parameters used in the job.
Add (or subtract) 3-12 db to **PLW9(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: NOESYPR1D spectrum with CW presaturation of the HOD line at 3.37 ppm

2.6 ^{1}H spectrum with homonuclear decoupling during FID acquisition *or* during D1 + FID acquisition

Parameter set: **awprotonhd or awprotonhd.2 (+ getprosol)**
Pulse programme: **zghd or zghd.2**

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

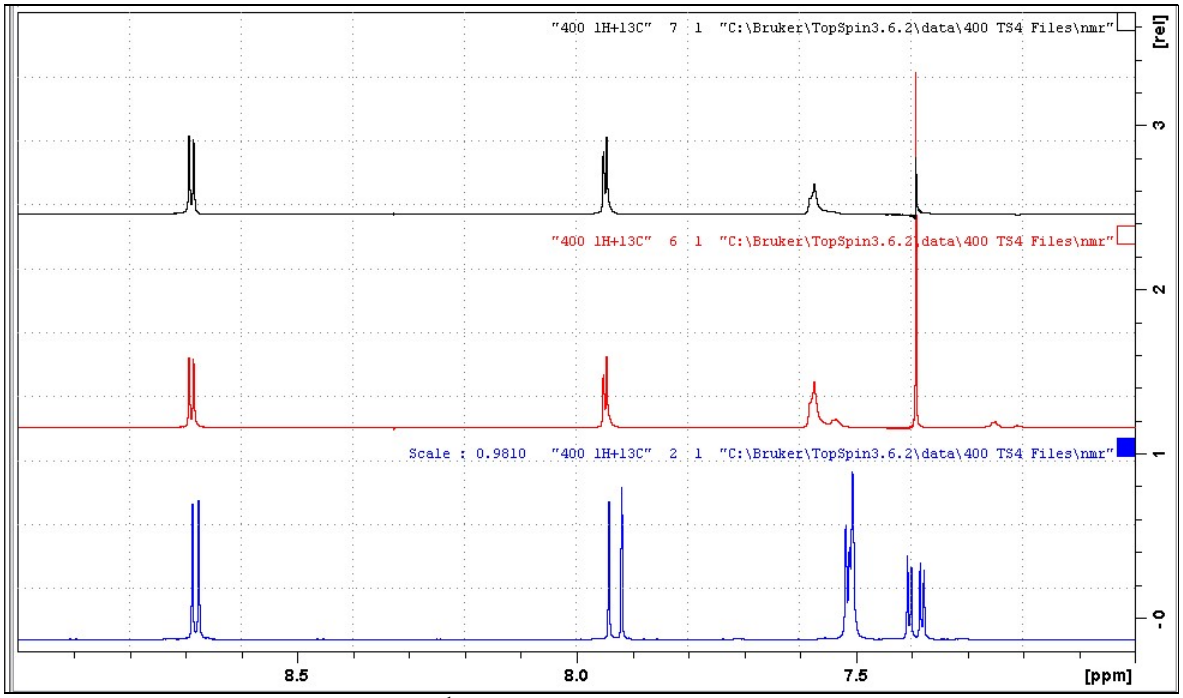
Type **O2** (enter) and enter the frequency in Hz of the signal to be decoupled.

Type **eda** (enter) and adjust acquisition parameters as required.
SW = 16 ppm, **O1P** = 6 ppm, **TD** = 32 K points or other values of your choice.
NS = multiple of 2, 4 or 8, **DS** = 2, 4 or 8.
D1 = repetition delay = 2-3 sec or other time of your choice.

Type **ased** (enter) and review other parameters including the **PL24** power level.
Do not adjust the **PL1or 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: The 7-9 ppm region of the ^{1}H NMR spectrum of quinine in D_6-DMSO.
Center: 7-9 ppm region with homonuclear decoupling at 7.39 ppm during FID acquisition.
Upper: 7-9 ppm region with homonuclear decoupling at 7.39 ppm during D1 + FID acquisition.