KJM 9250

2D-COSY, TOCSY, CLEAN-TOCSY, DIPSI2, NOESY, ROESY and ROESY2 Experiments on the AVIIIHD-800 Spectrometer

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Topspin 3.5

Windows 7

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1.0 Introduction

1.1 Spectral Window Set up

The spectral window width and midpoint should be determined in a standard ^{1}H NMR spectrum before setting up a 2D-COSY, TOCSY, etc experiment. The smaller the spectral window the higher the resolution of the resulting spectrum. There should be no signals within 0.5 ppm of the upper or lower ^{1}H shift limits.

1.2 The Clean-Tocsy Experiment

The aw coded variant of Bruker's **clmlevp**r experiment is prosol compatible and includes:

(i) **auto-calculation of d20** from the prosol table linked **p6** pulse time

(ii) **auto-calculation of L1** rounded off to the nearest whole number from a requested **d9** spin lock time input as per a standard TOCSY experiment

(iii) the set **d9** time is displayed as **d10** in the experiment's ***ased*** display immediately below the requested **d9** time.

Bruker's **clmlevpr** pp notes incorrectly include 2 x p17 pulses in their manual spin lock time calculation formula. While p17 appears twice in Bruker's **TOCSY** pp's it appears only once in their **clmlev** pp's.

1.3 Processing

The COSY experiment is an absolute value experiment – no phasing is required.

TOCSY, CLEAN-TOCSY, DIPSI2, NOESY, ROESY and ROESY2 experiments are phase sensitive experiments. These spectra should be phased **before** using the **abs1** and **abs2** commands.

2.0 COSY, TOCSY, etc Experiments and Parameter Sets

The following **aw** coded COSY, TOCSY, etc parameter sets have been set up on the AVIIIHD-800 spectrometer:

2.1 awcosy
2.2 awtocsy
2.3 awcleantocsy
2.4 awdipsi2
2.5 awnoesy
2.6 awroesy
2.7 awroesy2

2.1 COSY with a P0 excitation pulse

parameter set: **awcosy (+ getprosol)**
pulse programme: **cosygpqf**

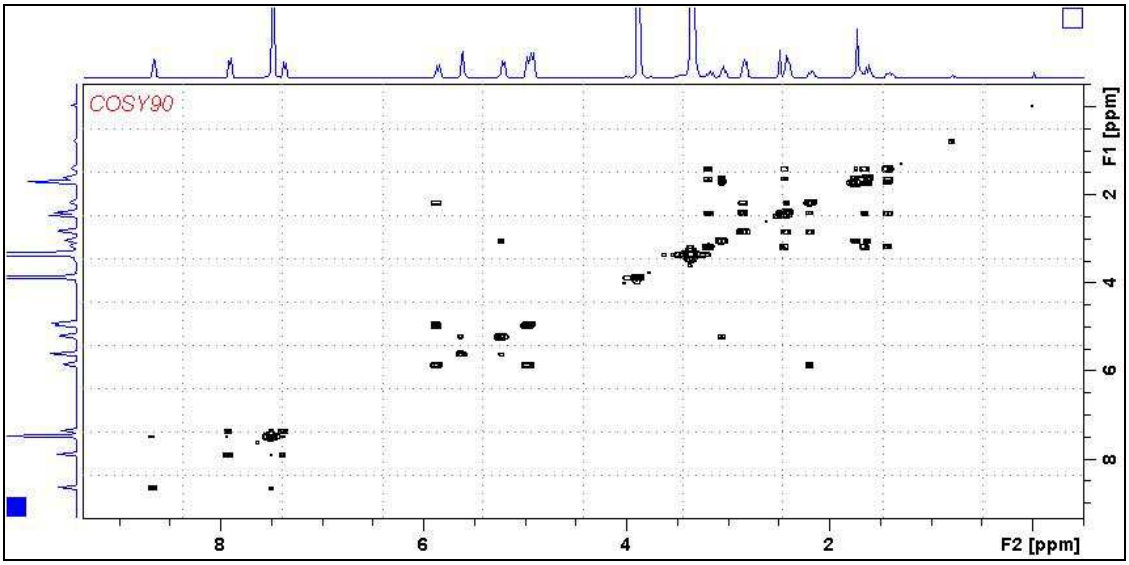
Type **eda** (enter) and enter **SW(F2) in ppm**, note the spectral window in **Hz** that appears in the **SWH(F2)** box and copy and paste this value into the **SWH(F1)** box. Check **SWH(F2)** = **SWH(F1)** in Hz including all dp's.

Enter **O1 =** spectral window midpoint in Hz or ppm.
Type **O1** (enter), note the **O1** value in Hz that appears and enter it as **O2** (Hz).

P0 = θ degree excitation pulse time, typically use a 45° or 90° pulse.
TD(F2) = 1K or 2K, **TD(F1)** = 128 - 256 (your choice).
NS = 2, 4, 8 (any number is OK), **DS** = 2, 4 or 8.
D1 = repetition delay = **1.5 sec** or other time of your choice.

Type **ased** (enter) and review parameters used in the job. Check **gradients** are OK.
Set **receiver gain** using **RGA** *(Important!).*

Process with: **SI(F2) = SI(F1) = 1K** or **2K**
WDW(F1) = WDW(F2) = SINE
SSB(F2) = SSB(F1) = 0
xfb, abs1, abs2 and (optionally) **sym**



COSY spectrum of quinine in D_6-DMSO. The spectrum is centered at 4.5 ppm.

2.2 TOCSY

parameter set: **awtocsy (+ getprosol)**
pulse programme: **mlevph**

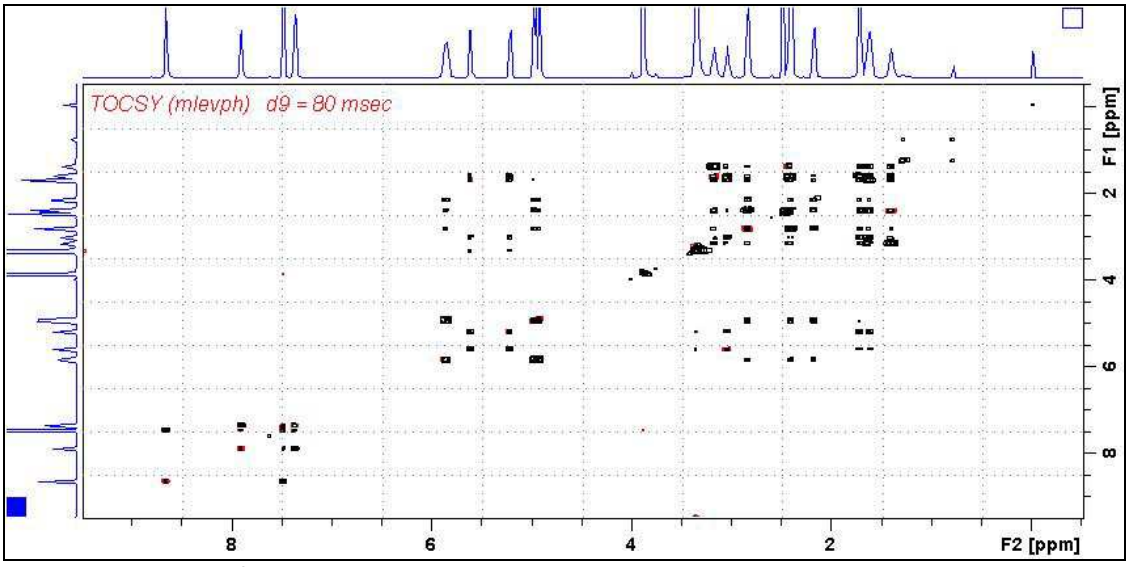
Type **eda** (enter) and enter **SW(F2) in ppm**, note the spectral window in **Hz** that appears in the **SWH(F2)** box and copy and paste this value into the **SWH(F1)** box. Check **SWH(F2)** = **SWH(F1)** in Hz including all dp's.

Enter **O1 =** spectral window midpoint in Hz or ppm.
Type **O1** (enter), note the **O1** value in Hz that appears and enter it as **O2** (Hz).

TD(F2) = 1K or 2K**, TD(F1)** = 128 - 256 (your choice).
NS = 2, 4, 8 (multiple of 4 or 8 recommended), **DS** = 4 or 8.
D1 = repletion time = **1.5 sec** or other time of your choice.
D9 = correlation time **= 80 msec** or other time of your choice (6-240 msec).

Type **ased** (enter) and review parameters used in the job.
Set **receiver gain** using **RGA** *(Important!).*

Process with: **SI(F2) = SI(F1) = 1K** or **2K**
WDW(F1) = WDW(F2) = QSINE
SSB(F2) = SSB(F1) = 2
xfb, abs1, abs2 and optionally **syma**



TOCSY spectrum of quinine in D_6-DMSO. The spectrum is centered at 4.5 ppm.

2.3 CLEAN-TOCSY

parameter set: **awcleantocsy (+ getprosol)**
pulse programme: **awclmlev**

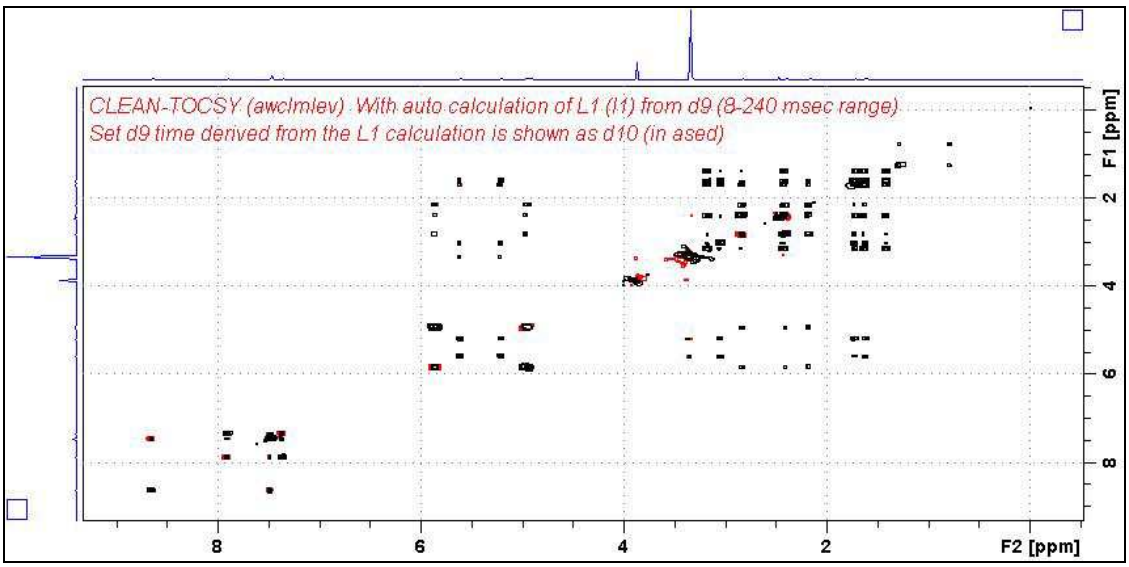
Type **eda** (enter) and enter **SW(F2) in ppm**, note the spectral window in **Hz** that appears in the **SWH(F2)** box and copy and paste this value into the **SWH(F1)** box. Check **SWH(F2)** = **SWH(F1)** in Hz including all dp's.

Enter **O1 =** spectral window midpoint in Hz or ppm.
Type **O1** (enter), note the **O1** value in Hz that appears and enter it as **O2** (Hz).

TD(F2) = 1K or 2K**, TD(F1)** = 128 - 256 (your choice).
NS = 2, 4, 8 (multiple of 4 or 8 recommended), **DS** = 4 or 8.
D1 = repetition time = **1.5 sec** or other time of your choice.
D9 = correlation time = **80 msec** or other time of your choice (6-240 msec).

Type **ased** (enter) and review parameters used in the job.
Check the **D10** time derived from the requested **D9** time is OK.
Set **receiver gain** using **RGA** *(Important!).*

Process with: **SI(F2) = SI(F1) = 1K** or **2K**
WDW(F1) = WDW(F2) = QSINE
SSB(F2) = SSB(F1) = 2
xfb, abs1, abs2 and optionally **syma**



CLEAN-TOCSY spectrum of quinine in D_6-DMSO. The spectrum is centered at 4.5 ppm.

2.4 DIPSI2

parameter set: **awdipsi2 (+ getprosol)**
pulse programme: **dipsi2ph**

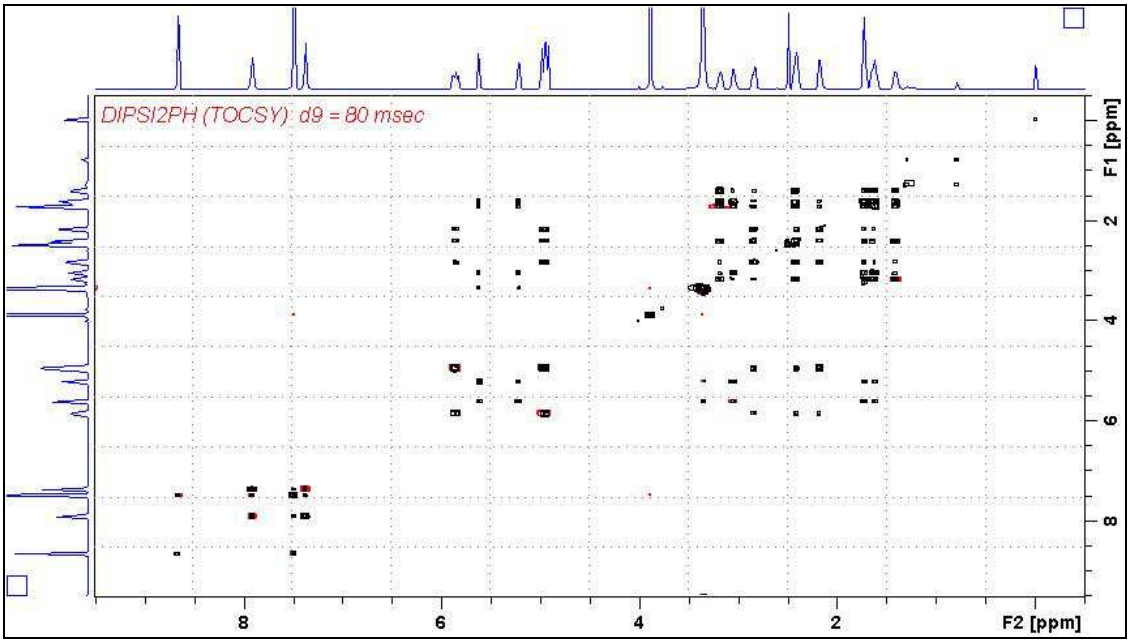
Type **eda** (enter) and enter **SW(F2) in ppm**, note the spectral window in **Hz** that appears in the **SWH(F2)** box and copy and paste this value into the **SWH(F1)** box. Check **SWH(F2)** = **SWH(F1)** in Hz including all dp's.

Enter **O1 =** spectral window midpoint in Hz or ppm.
Type **O1** (enter), note the **O1** value in Hz that appears and enter it as **O2** (Hz).

TD(F2) = 1K or 2K**, TD(F1)** = 128 - 256 (your choice).
NS = 2, 4, 8 (any number is OK), **DS** = 2, 4 or 8.
D1 = repletion time = **1.5 sec** or other time of your choice.
D9 = correlation time = **80 msec** or other time of your choice (6-240 msec).

Type **ased** (enter) and review parameters used in the job.
Set **receiver gain** using **RGA** *(Important!).*

Process with: **SI(F2) = SI(F1) = 1K** or **2K**
WDW(F1) = WDW(F2) = QSINE
SSB(F2) = SSB(F1) = 2
xfb, abs1, abs2 and optionally **syma**



DIPSI2 spectrum of quinine in D_6-DMSO. The spectrum is centered at 4.5 ppm.

2.5 NOESY

parameter set: **awnoesy (+ getprosol)**
pulse programme: **noesygpph**

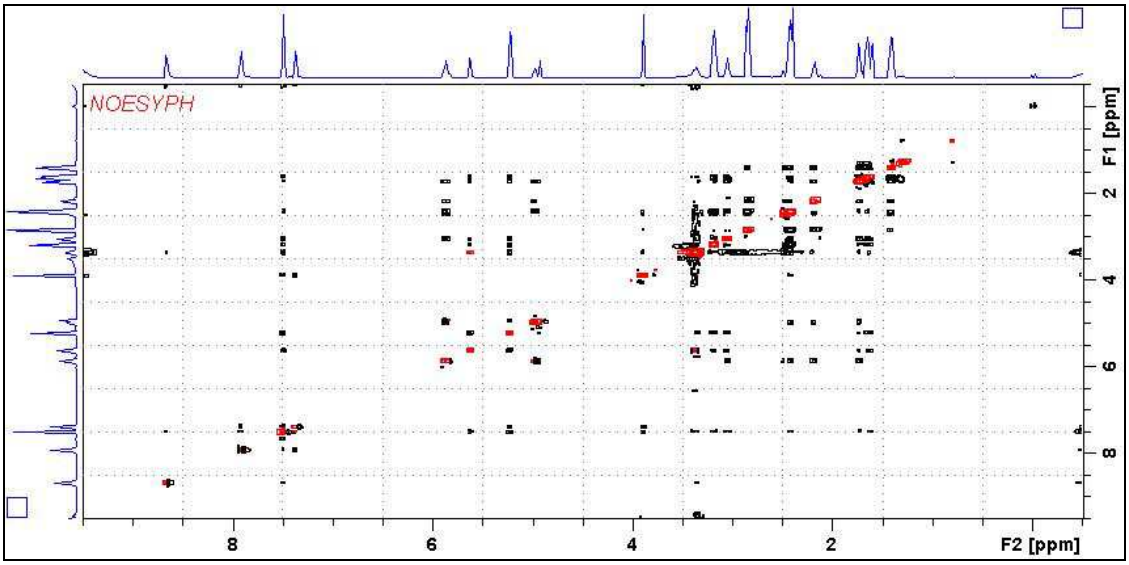
Type **eda** (enter) and enter **SW(F2) in ppm**, note the spectral window in **Hz** that appears in the **SWH(F2)** box and copy and paste this value into the **SWH(F1)** box. Check **SWH(F2)** = **SWH(F1)** in Hz including all dp's.

Enter **O1 =** spectral window midpoint in Hz or ppm.
Type **O1** (enter), note the **O1** value in Hz that appears and enter it as **O2** (Hz).

TD(F2) = 1K or 2K, **TD(F1)** = 128 - 256 (your choice).
NS = 4, 8 (multiple of 4 or 8 recommended), **DS** = 4 or 8.
D1 = repletion time = **1.5 sec** or other time of your choice.
D8 = NOE mixing time = **0.5 sec** or other time of your choice.

Type **ased** (enter) and review parameters used in the job and check gradients are OK.
Set **receiver gain** using **RGA** *(Important!).*

Process with: **SI(F2) = SI(F1) = 1K** or **2K**
WDW(F1) = WDW(F2) = QSINE
SSB(F2) = SSB(F1) = 2
xfb, abs1, abs2 and optionally **syma**



NOESY spectrum of quinine in D_6-DMSO. The spectrum is centered at 4.5 ppm.

2.6 ROESY

parameter set: **awroesy (+ getprosol)**
pulse programme: **roesyph** (with CW spin lock)

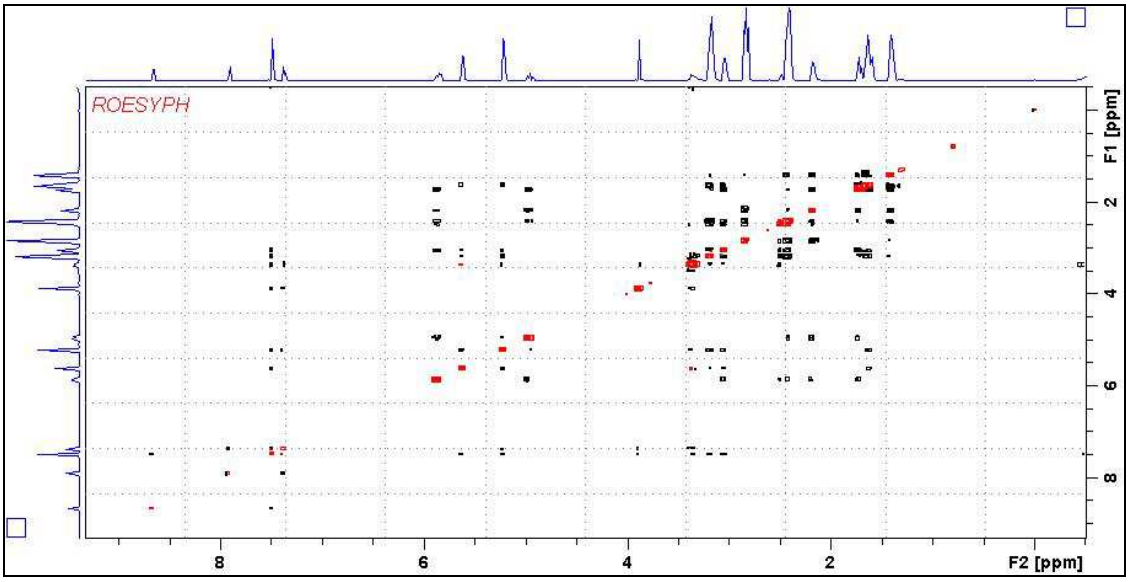
Type **eda** (enter) and enter **SW(F2) in ppm**, note the spectral window in **Hz** that appears in the **SWH(F2)** box and copy and paste this value into the **SWH(F1)** box. Check **SWH(F2)** = **SWH(F1)** in Hz including all dp's.

Enter **O1 =** spectral window midpoint in Hz or ppm.
Type **O1** (enter), note the **O1** value in Hz that appears and enter it as **O2** (Hz).

TD(F2) = 1K or 2K**, TD(F1)** = 128 - 256 (your choice).
NS = 4, 8 (multiple of 4 or 8 recommended), **DS** = 4 or 8.
D1 = repetition time = **1.5 sec** or other time of your choice.
P15 = spin lock time = **200000** or **250000 usec** (200 or 250 msec).

Type **ased** (enter) and review parameters used in the job.
Set **receiver gain** using **RGA** *(Important!).*

Process with: **SI(F2) = SI(F1) = 1K** or **2K**
WDW(F1) = WDW(F2) = QSINE
SSB(F2) = SSB(F1) = 2
xfb, abs1, abs2 and optionally **syma**



ROESY spectrum of quinine in D_6-DMSO. The spectrum is centered at 4.5 ppm.

2.7 ROESY2

parameter set: **awroesy2 (+ getprosol)**
pulse programme: **roesyph.2** (with pulsed spin lock)

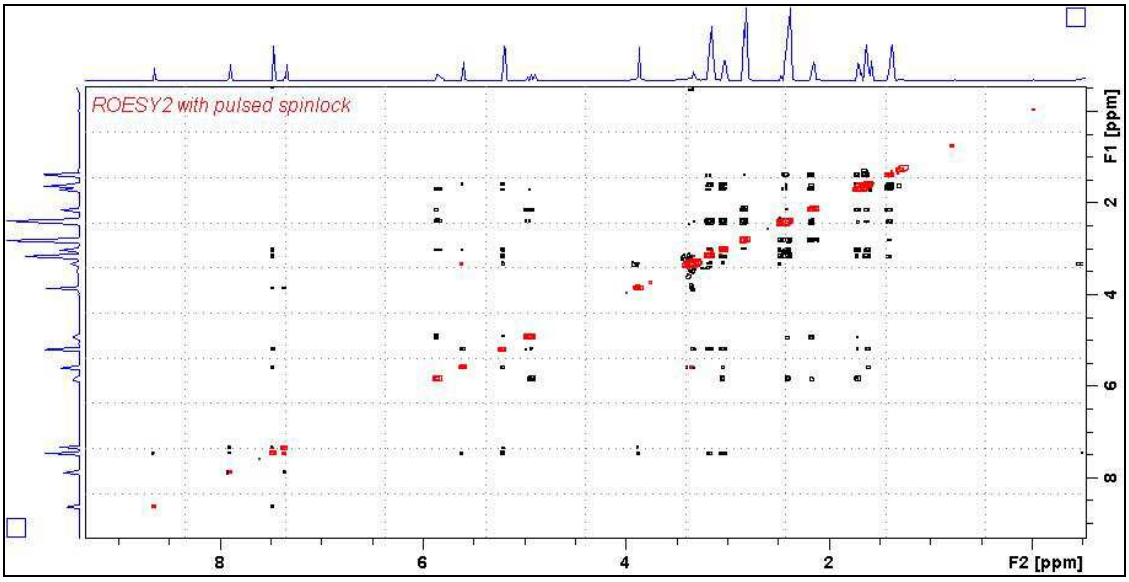
Type **eda** (enter) and enter **SW(F2) in ppm**, note the spectral window in **Hz** that appears in the **SWH(F2)** box and copy and paste this value into the **SWH(F1)** box. Check **SWH(F2)** = **SWH(F1)** in Hz including all dp's.

Enter **O1 =** spectral window mid point in Hz or ppm.
Type **O1** (enter), note the **O1** value in Hz that appears and enter it as **O2** (Hz).

TD(F2) = 1K or 2K, **TD(F1)** = 128 - 256 (your choice).
NS = 4, 8 (multiple of 4 or 8 recommended), **DS** = 4 or 8.
D1 = repetition time = **1.5 sec** or other time of your choice.
P15 = spin lock time = **200000** or **250000 usec** (200 or 250 msec).

Type **ased** (enter) and review parameters used in the job.
Set **receiver gain** using **RGA** *(Important!).*

Process with: **SI(F2) = SI(F1) = 1K** or **2K**
WDW(F1) = WDW(F2) = QSINE
SSB(F2) = SSB(F1) = 2
xfb, abs1, abs2 and optionally **syma**



ROESY2 spectrum of quinine in D_6-DMSO. The spectrum is centered at 4.5 ppm.