KJM 5250 and KJM 9250

COSY, TOCSY, DIPSI2, NOESY, ROESY and ROESY2 Spectra with Excitation Sculptured (ES) Solvent Peak Suppression NMR spectra on the AVneo400 spectrometer.

Version 3.1

Topspin 4.3

AVneo400 COSY, TOCSY, DIPSI2, NOESY, ROESY and ROESY2 Spectra with Excitation Sculptured (ES) Peak Suppression

1.1 Spectral Window Set up

The spectral window width (in ppm), centered around the peak to be **ES** suppressed should be determined in a standard ^{1}H NMR spectrum _before_ setting up **a COSYES, TOCSYES,** etc experiment. There should be no signals within 0.5 ppm of the upper or lower limits of the spectral window. The frequency of the peak to be **ES** suppressed should be determined **in Hz**

The **getprosol** command will read in **prosol Table** saved pulse time, powers and types into an experiment. After using the **getprosol** command the **pulsecal** command can be used to adjust the ^{1}H degree pulse times and **prosol Table** linked pulse powers to take account of solvent and/or buffer matrix effects which influence a samples 90 degree pulse time.

1.2 ES set up

ES is applied at **O1 Hz** = the spectral window midpoint, or it can _optionally be offset_ at **O1* Hz** where **SPOFFS1** (or **SPOFFS10** in some ES pp's) = **O1*-O1 Hz.** _The use of a 2000 usec Sinc1.1000 ES pulse suppresses signals ~ ± 0.7 ppm (~ 280 Hz) either side of its frequency. This band width is 2-3 times greater than that of PR presaturation._

1.3 Processing

COSYES experiments are absolute value experiments– no phasing is required.

TOCSYES, DIPSI2ES, NOESYES, ROESYES and ROESY2ES experiments are phase sensitive experiments which should be phased **before** using the **abs1** and **abs2** (and optional **syma**) commands.

2.0 Experiments and Parameter Sets

2.1 COSYES with Excitation Sculpting

  parameter set: **awcosyes (+ getprosol)**
  pulse programme: **awcosyes**

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 in Hz** of the signal to be presaturated.
        O1 = spectral window midpoint.
Type **O2** (return) and enter the **O1** frequency in Hz as the **O2** frequency.

TD(F2) = 1K or 2K, **TD(F1) =** 128-256 (your choice).
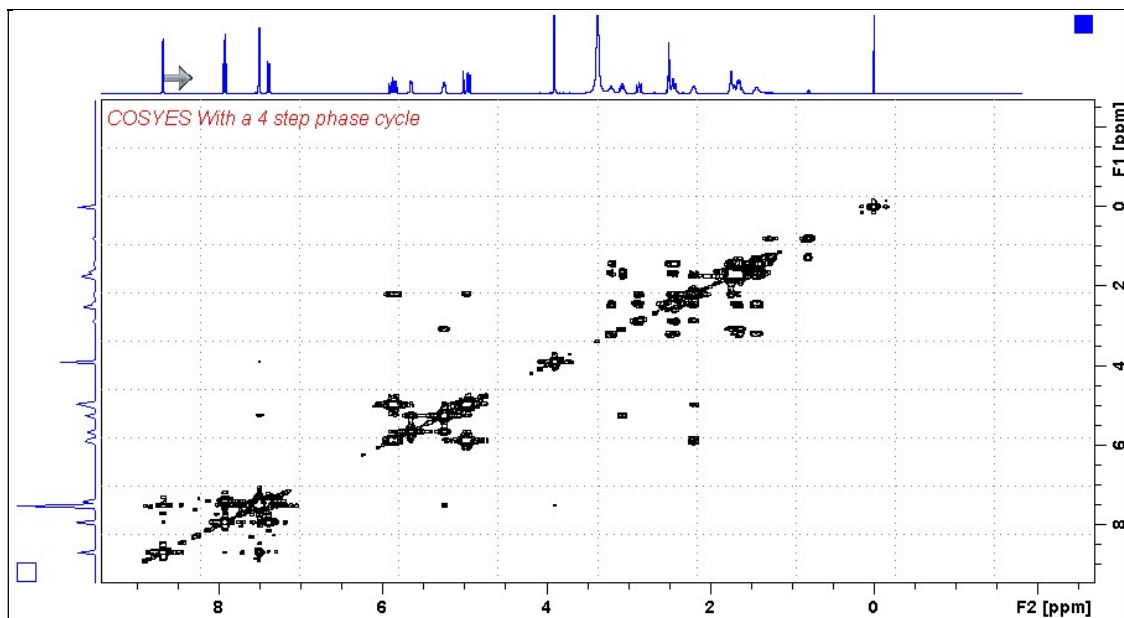**NS** = multiple of 2, 4 or 8, **DS** = 2, 4 or 8.
D1 = presaturation time = **2 sec** or other time of your choice.

Type **ased** (enter) and review parameters used in the job.
Verify **gradients** are OK and that **a 2000 usec sinc1.1000** shaped pulse is used.
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**

Neo400 DIPSI2ES spectrum of quinine in D_6-DMSO with the HOD line at 3.37 ppm **ES** suppressed. The spectrum is centered at the O1 frequency of the HOD line.

2.2 TOCSYES with Excitation Sculpting

   parameter set: **awtocsyes (+ getprosol)**
   pulse programme: **mlevesgpph**

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 in Hz** of the signal to be presaturated.
        O1 = spectral window midpoint.
Type **O2** (return) and enter the **O1** frequency in Hz as the **O2** frequency.

TD(F2) = 1K or 2K, **TD(F1)** = 128-256 (your choice).
NS = multiple of 2, 4 or 8, **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.
Verify **ES gradients** are OK and that a **2000 usec s sinc1.1000** shaped pulse is used.
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**
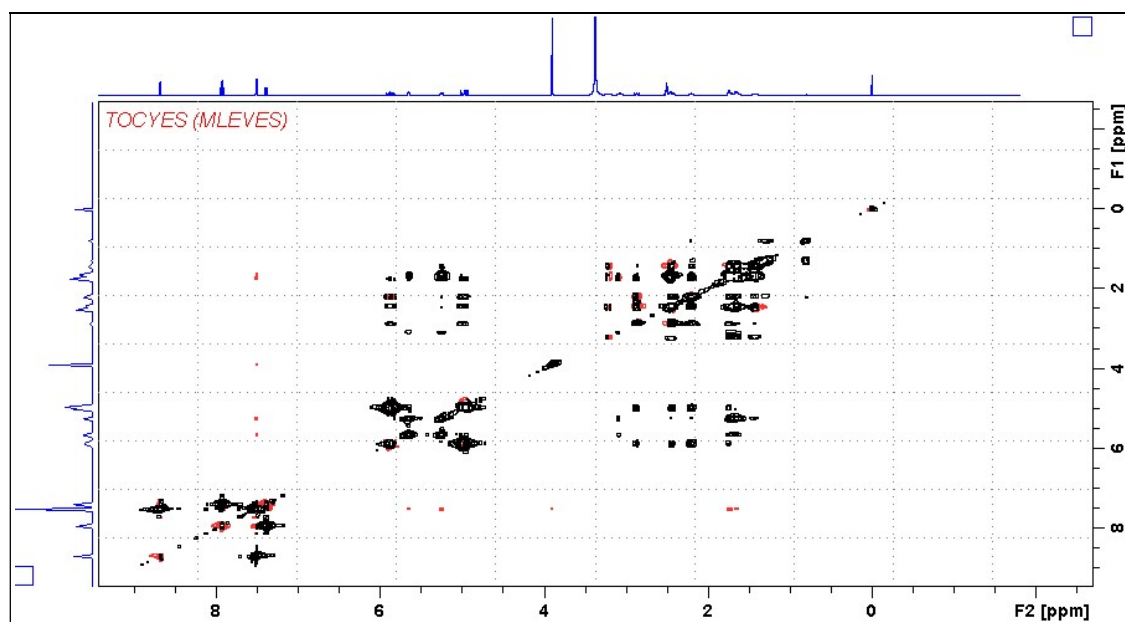
Neo400 TOCSYES spectrum of quinine in D_6-DMSO with the HOD line at 3.37 ppm **ES** suppressed. The spectrum is centered at the O1 frequency of the HOD line.

2.3 DIPSI2ES with Excitation Sculpting

  parameter set: **awdipsi2es (+ getprosol)**
  pulse programme: **awdipsi2es**

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 in Hz** of the signal to be presaturated.
       O1 = spectral window midpoint.
Type **O2** (return) and enter the **O1** frequency in Hz as the **O2** frequency.

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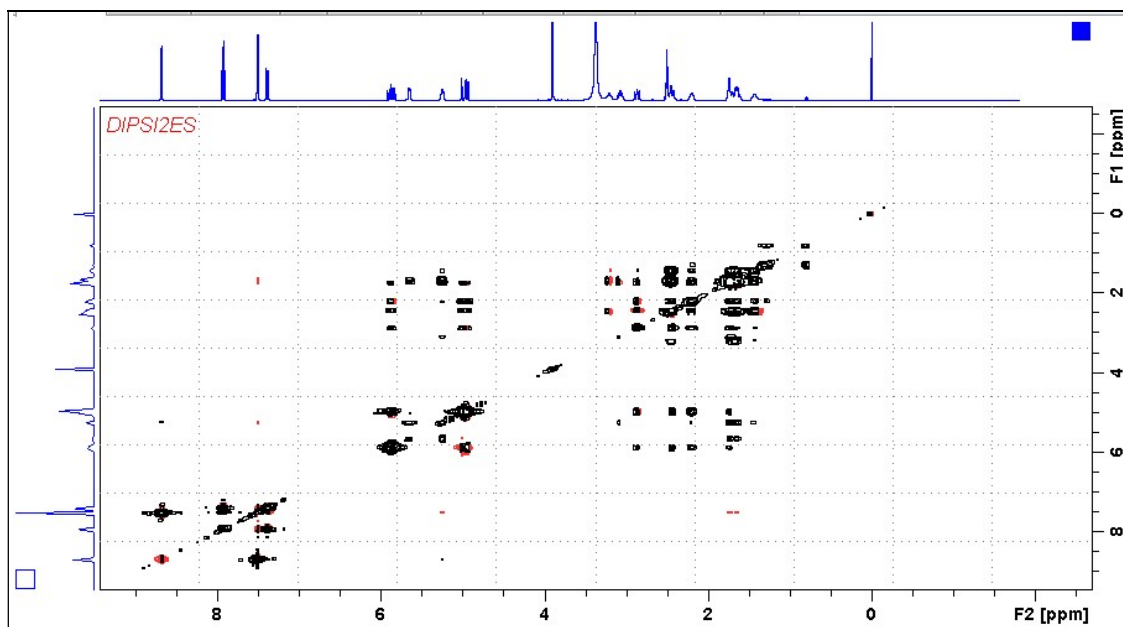**D1** = repetition time = **1.5 sec** or other time of your choice.
D9 = correlation time = **80 msec** or other time of your choice (6-240 msec).

Type **ased** (enter) and review parameters used in the job.
Verify **ES gradients** are OK and that a **2000 usec sinc1.1000** shaped pulse is used.
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**

Neo400 DIPSI2ES spectrum of quinine in D$_6$-DMSO with the HOD line at 3.37 ppm **ES** suppressed. The spectrum is centered at the O1 frequency of the HOD line.

2.4 NOESYES with Excitation Sculpting

  parameter set: **awnoesyes (+ getprosol)**
  pulse programme: **noesyesgpph**

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 in Hz** of the signal to be presaturated.
        O1 = spectral window midpoint.
Type **O2** (return) and enter the **O1** frequency in Hz as the **O2** frequency.

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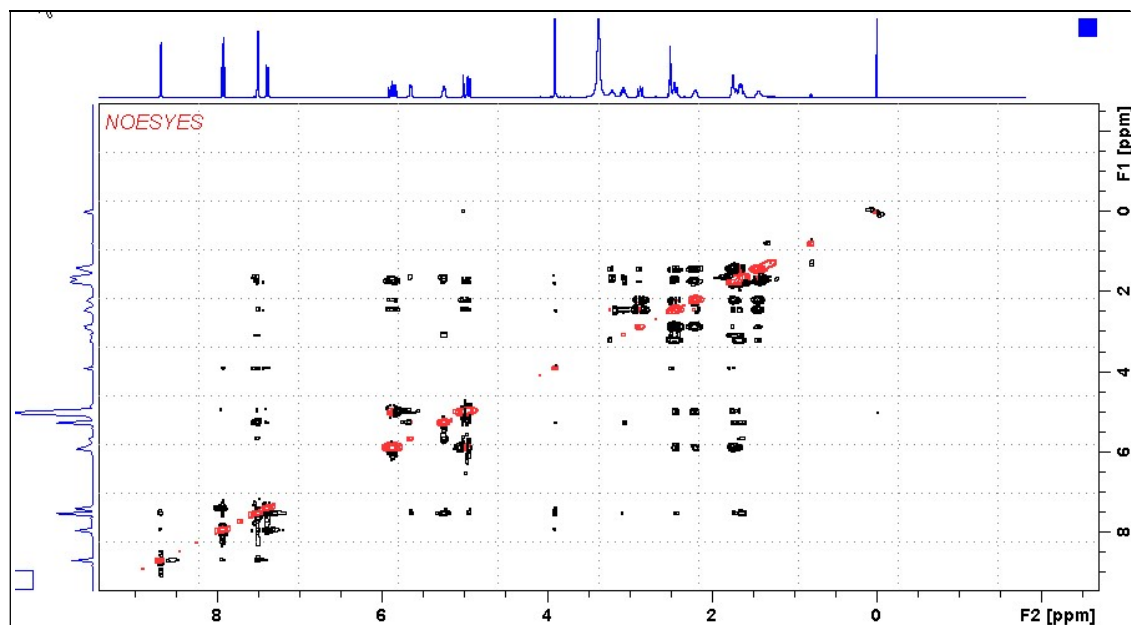**D1** = repetition time = **1.5 sec** or other time of your choice.

Type **ased** (enter) and review parameters used in the job and check gradients are OK.
Verify that a **2000 usec squa100.1000** shaped pulse is used.
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**

Neo400 NOESYES spectrum of quinine in D_6-DMSO with the HOD line at 3.37 ppm **ES**
suppressed. The spectrum is centered at the O1 frequency of the HOD line.

2.5 ROESYES with Excitation Sculpting

   parameter set: **awroesyes (+ getprosol)**
   pulse programme: **roesyesgpph** (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 in Hz** of the signal to be presaturated.
        O1 = spectral window midpoint.
Type **O2** (return) and enter the **O1** frequency in Hz as the **O2** frequency.

TD(F2) = 1K or 2K, **TD(F1)** = 128-256 (your choice).
NS = multiple of 4 or 8**, 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 and check gradients are OK.
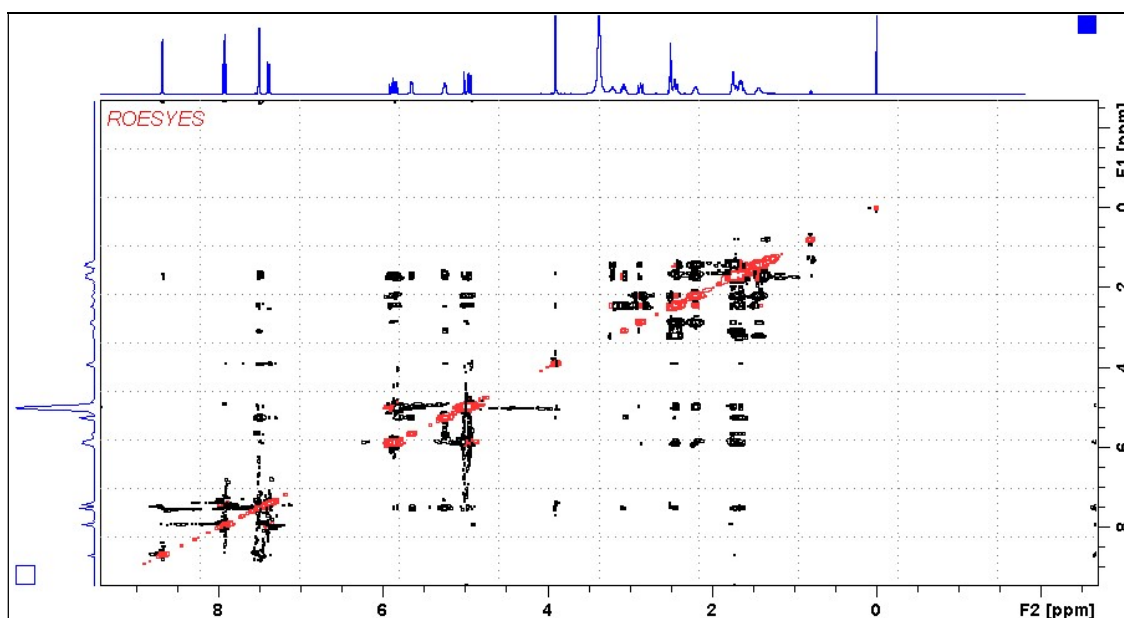Verify that a **2000 usec sinc1.1000** shaped pulse is used.
Set **receiver gain** using **RGA** *(important!)*.

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**

Neo400 ROESYES spectrum of quinine in D$_6$-DMSO with the HOD line at 3.37 ppm **ES** suppressed. The spectrum is centered at the O1 frequency of the HOD line.

2.6 ROESY2ES with Excitation Sculpting

  parameter set: **awroesy2es (+ getprosol)**
  pulse programme: **roesyesgpph.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 in Hz** of the signal to be presaturated.
       O1 = spectral window midpoint.
Type **O2** (return) and enter the **O1** frequency in Hz as the **O2** frequency.

TD(F2) = 1K or 2K, **TD(F1)** = 128-256 (your choice).
NS = multiple of 4 or 8, **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 and check gradients are OK.
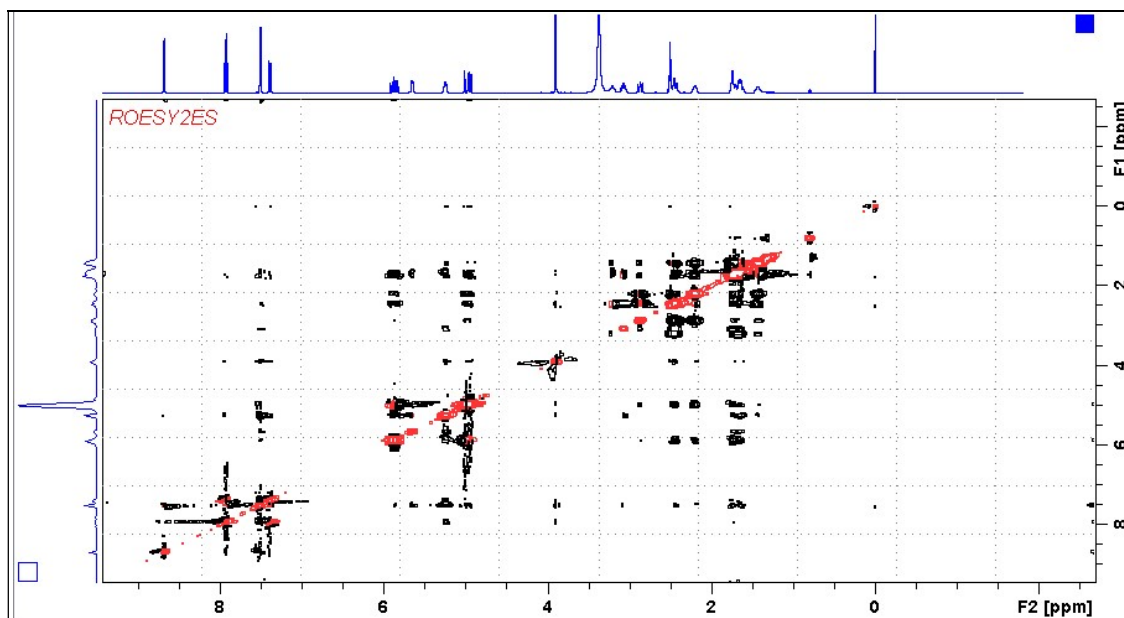Verify that a **2000 usec sinc1.1000** shaped pulse is used.
Set **receiver gain** using **RGA** *(important!)*.

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**

Neo400 ROESY2ES spectrum of quinine in D_6-DMSO with the HOD line at 3.37 ppm **ES** suppressed. The spectrum is centered at the O1 frequency of the HOD line.