KJM 5250 and KJM 9250

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

Version 3.1

Topspin 4.3

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AVneo400 1D-SELECTIVE Proton NMR Experiments

1.1 Introduction

aw coded **1D-SELECTIVE** experiments are set up with soft a default prosol Table linked pulse time of 80000 usec. Pulse powers are read in using the **getprosol** command.

The effective excitation window of an 80000 usec 180^{o} soft pulse experiment at 400 MHz is ~ 30-35 Hz. For a wider or narrower excitation window use one of the power and time combinations below. The greater the pulse power attenuation (larger the db value) the narrower the excitation window. Adding 6 db halves the pulse power.

Selective pulse power + time combinations
Default: P12 = 80000 usec at the PL2W(db) level read in by the **getprosol** command
Harder: P12 = 40000 usec and subtract 6 db from the PL2W(db) power level
Softer: P12 = 160000 usec and add 6 db to the PL2W(db) power level

1.2 1D-SELECTIVE NMR Experiments

The following 1D-Selective experiments have been set up on the Neo400 spectrometer.

2.1 SELCOSY spectrum

2.2 SELTOCSY spectrum

2.3 SELDIPSI2 spectra

2.4 Phasing SELNOESY and SELROESY spectra

2.5 SELNOESY spectrum

2.6 SELROESY spectrum

2.7 SELROESY.2 spectrum

2.1 SELCOSY Spectrum

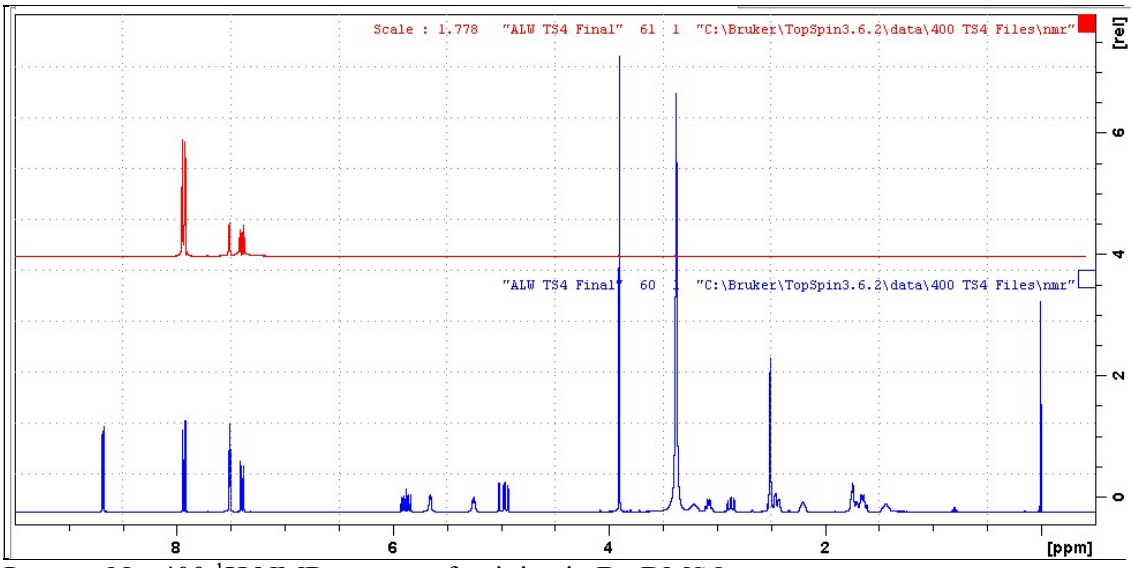
Parameter set: **awselcosy (+ getprosol)**
Pulse programme: **selcogp**

TD = 32 K, **SI** = 32 K, **SW** = 16 ppm.
O1 = Frequency in Hz of signal to be selectively correlated. **O1** Excitation is applied *on resonance* at the middle of SW. Check SW is wide enough.

NS = multiple of 4 or 8, **DS** = 4 or 8.
D1 = 2 sec or other time of your choice.
CNST 1 = 7 Hz or other J_{H-H} coupling of your choice
D4 = $1/4J_{H-H}$ (typical 30-40 msec) is auto-calculated from **CNST1**.

P12 = 80000 usec, **SP2(db)** = shaped pulse power

Process with **EFP and MC** (applies **LB**, typically use 0.1 - 0.3 Hz).



Lower: Neo400 ^{1}H NMR spectra of quinine in D_6-DMSO.
Upper: SELCOSY spectrum ex the signal at 7.39 ppm

Note: *The 12/01/11 TS3.6.2 copy of the SELCOGP pp uses D14 as the 1/4J related delay in the pp whereas the 21/0/15 TS4.2 version of the pp uses D4.*

2.2 SELTOCSY Spectrum

Parameter set: **awseltocsy (+ getprosol)**
Pulse programme: **selmlgp**

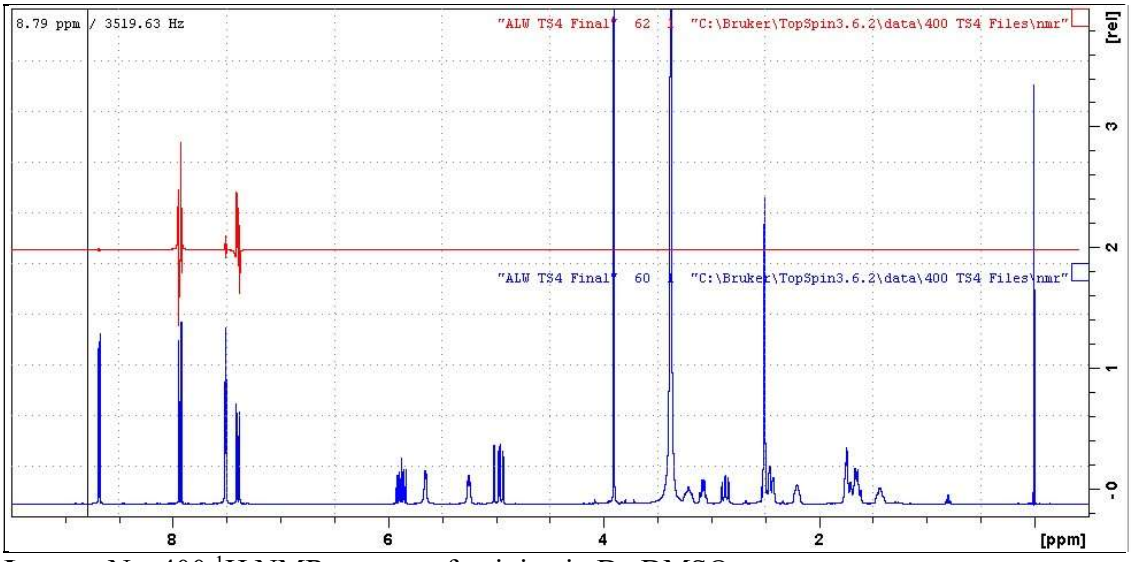
TD = 32 K, **SI** = 32 K, **SW** = 16 ppm.
O1 = Frequency in Hz of signal to be selectively correlated. **O1** Excitation is applied *on resonance* at the middle of SW. Check SW is wide enough.

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

D1 = 2 sec or other time of your choice.
D9 = 80 msec for medium range correlations or other time of your choice.
160 msec for long range correlations, 6-15 msec for short range correlations.

P12 = 80000 usec, **SP2** = shaped pulse.
PLW10 = TOCSY spin lock power level read in using the getprosol command.

Process with **EFP** (applies **LB,** typically use 0.1-0.3 Hz); all peaks should be positive. Strongly coupled peaks may exhibit some negative artifact lines which tend to decrease as **D9** is increased.



Lower: Neo400 ^{1}H NMR spectra of quinine in D_6-DMSO.
Upper: D9 = 80 msec **SELTOCSY** spectrum ex the signal at 7.39 ppm.

2.3 SELDIPSI2 Spectrum

Parameter set: **awseldipsi2 (+ getprosol)**
Pulse programme: **seldigp**

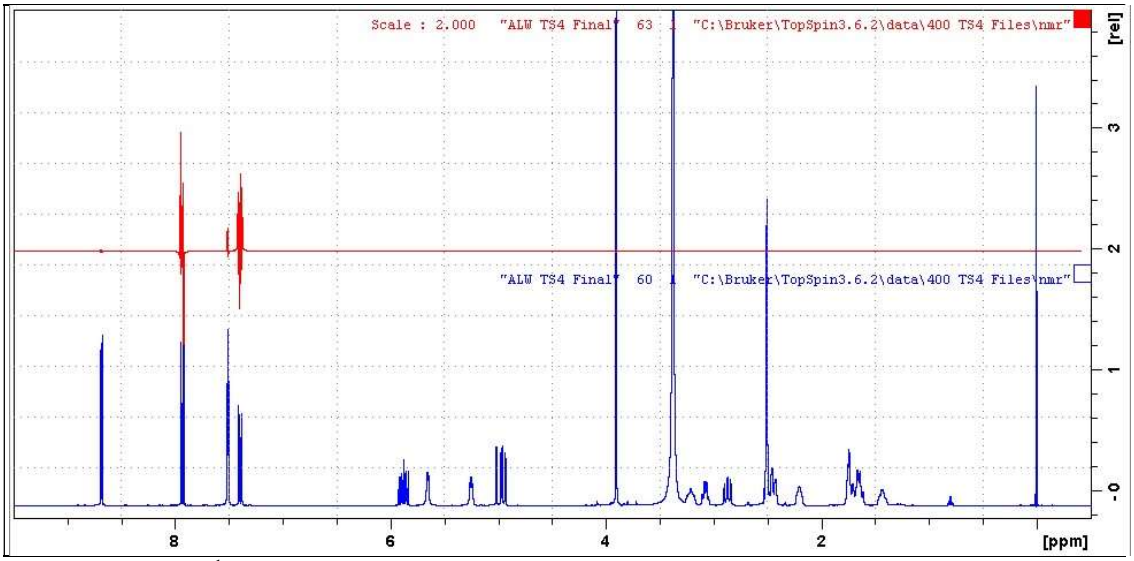
TD = 32 K, **SI** = 32 K, **SW** = 16 ppm.
O1 = Frequency in Hz of signal to be selectively correlated. **O1** Excitation is applied *on resonance* at the middle of SW. Check SW is wide enough.

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

D1 = 1 sec or other time of your choice.
D9 = 80 msec for medium range correlations or other time of your choice.
160 msec for long range correlations, 6-15 msec for short range correlations.

P12 = 80000 usec, **SPW2** = shaped pulse power read in using the getprosol command.
PLW10 = spin lock power level read in using the getprosol command.

Process with **EFP** (applies **LB,** typically use 0.1 - 0.3 Hz); all peaks should be positive. Strongly coupled peaks may exhibit some negative artifact lines which tend to decrease as **D9** is increased.

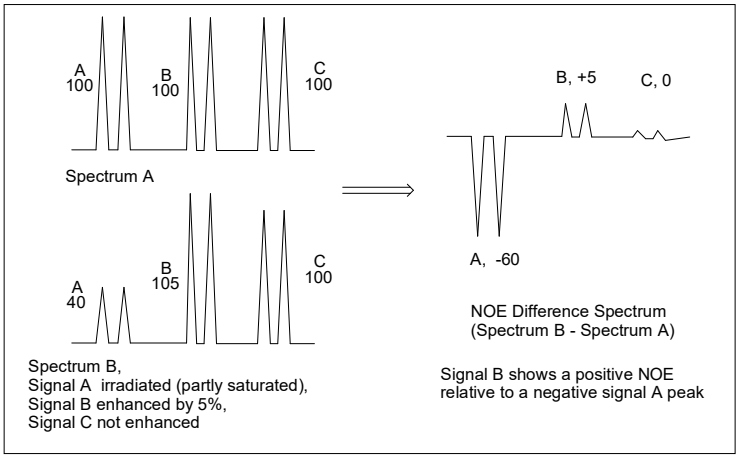


Lower: Neo400 ^{1}H NMR spectra of quinine in D_6-DMSO.
Upper: D9 = 80 msec **SELDIPSI**2 spectrum ex the signal at 7.39 ppm

2.4 Phasing of 1D-SELNOESY and SELROESY spectra

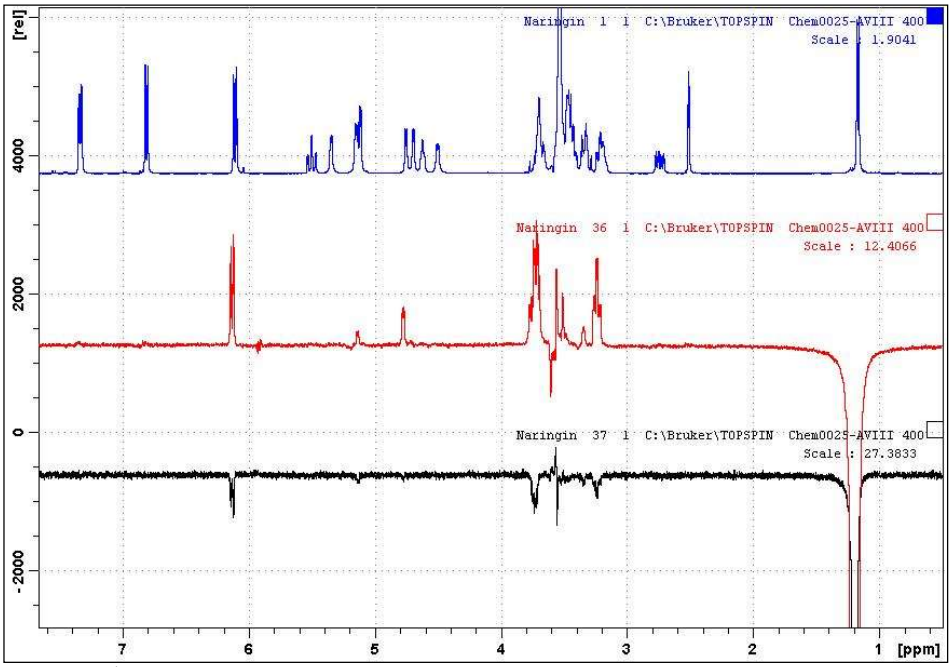
NOESY and SELNOESY peaks may be positive, zero or negative depending on correlation times whereas *ROESY and SELROESY peaks are always positive.*

In a **classic NOE-difference experiment** a positive NOE is one which affords a positive enhanced signal relative to the residual negative irradiated signal observed when a reference spectrum is subtracted from the irradiated spectrum.



A positive NOE is one in which a positive enhanced peak is observed relative to a ***negative irradiated peak*** *in a 1D-SELNOESY experiment , or a* ***negative diagonal peak*** *in a 2D-NOESY experiment.*

An example of a signal showing a positive ROESY response and a negative NOESY response is shown below.



Upper: ^{1}H NMR spectrum of naringin (0.5-8 ppm region plotted).
Center: SELROESY spectrum ex the methyl signal at 1.2 ppm.
Lower: SELNOESY spectrum ex the methyl signal at 1.2 ppm.

2.5 SELNOESY Spectrum

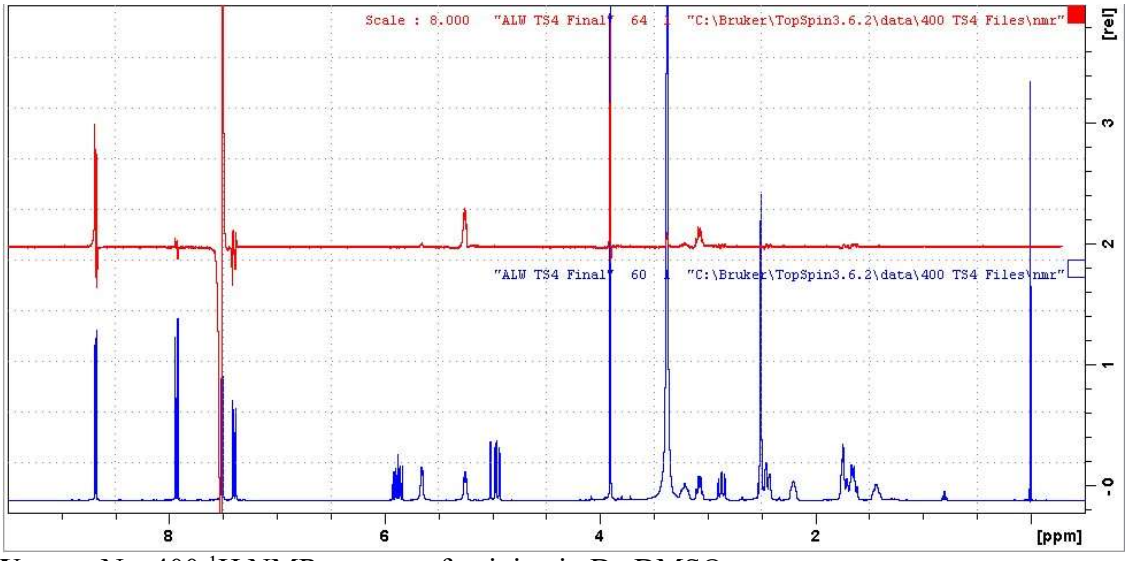
Parameter set: **awselnoesy (+ getprosol)**
Pulse programme: **selnogp**

TD = 32 K, **SI** = 32 K, **SW** = 16 ppm.
O1 = Frequency in Hz of signal to be selectively correlated. **O1** Excitation is applied *on resonance* at the middle of SW. Check SW is wide enough.

D1 = 1 sec or other time of your choice.
D6 = 0.5 to 0.8 sec (NOESY correlation time).

P12 = 80000 usec, **SPW2** = shaped pulse power read in using the getprosol command

Process with **EFP** (applies **LB,** typically use 0.1 - 0.3 Hz).
Phase the spectrum to afford a **negative** excited signal. NOESY peaks may be *positive, zero or negative* depending on correlation times. COSY artifact peaks ex strongly coupled signals may be present in NOESY spectra.



Upper: Neo400 ^{1}H NMR spectra of quinine in D_6-DMSO.
Lower: SELNOESY spectrum ex the signal at 7.51 ppm.

2.6 SELROESY (CW spin locked) Spectrum

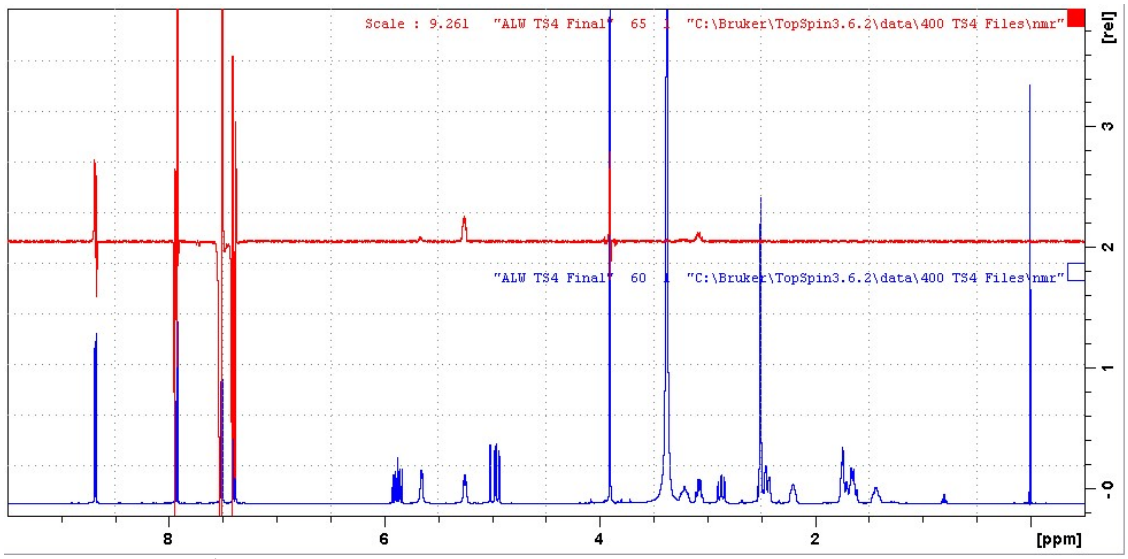
Parameter set: **awselroesy (+ getprosol)**
Pulse programme: **selrogp**

TD = 64 K, **SI** = 64 K, **SW** = 20 ppm.
O1 = Frequency in Hz of signal to be selectively correlated. **O1** Excitation is applied *on resonance* at the middle of SW. Check SW is wide enough.

D1 = 2 sec or other time of your choice
P15 = 200000 or 250000 usec (ROESY spinlock time)

SPW2 = Shaped pulse power level (read in by the getprosol command)
PL11 = ROESY CW spin lock power (read in by the getprosol command)

Process with **EFP** (applies **LB,** typically use 0.1 - 0.3 Hz). *Phase the spectrum to afford a **negative** excited signal and positive ROESY peaks. Negative or antiphase TOCSY artifact peaks may be present in ROESY spectra.*



Upper: Neo400 ^{1}H NMR spectra of quinine in D_6-DMSO.
Center: SELROESY spectrum ex the signal at 7.51 ppm

In this example strong negative TOCSY artifact peaks are seen at 7.39 and 7.94 ppm, TOCSY artifacts are generally much less significant in pulsed spin locked SELROESY.2 spectra.

2.7 SELROESY2 (pulsed spin locked) Spectrum

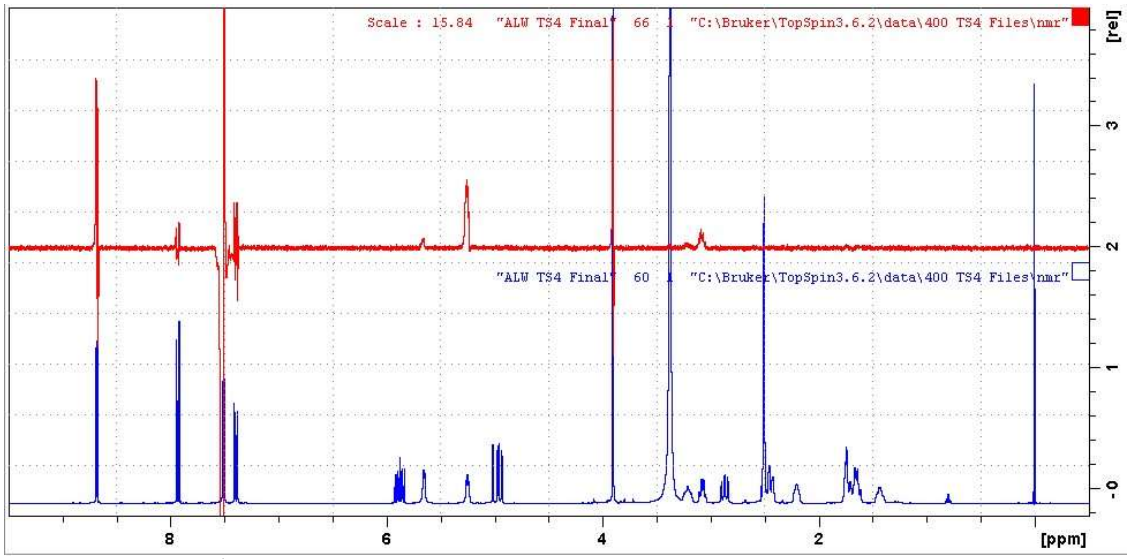
Parameter set: **awselroesy2 (+ getprosol)**
Pulse programme: **selrogp.2**

TD = 32 K, **SI** = 32 K, **SW** = 16 ppm.
O1 = Frequency in Hz of signal to be selectively correlated. **O1** Excitation is applied *on resonance* at the middle of SW. Check SW is wide enough.

D1 = 2 sec or other time of your choice.
P15 = 200000 or 250000 usec (ROESY spinlock time).

SPW2 = Shaped pulse power level (read in by the getprosol command).
PLW27 = ROESY pulsed ROESY spinlock power (read in by the getprosol command).

Process with **EFP** (applies **LB,** typically use 0.1-0.3 Hz). *Phase the spectrum to afford a* ***negative*** *excited signal and positive ROESY peaks.*



Upper: Neo400 ^{1}H NMR spectra of quinine in D_6-DMSO.
Lower: **SELROESY2** spectrum ex the signal at 7.51 ppm.

Antiphase (mixed positive/negative) TOCSY artifact peaks may occasionally be present in SELROESY.2 spectra. TOCSY artifact peaks are generally much less significant in pulsed spin locked SELROESY.2 spectra than is the case for CW spin locked SELROESY spectra.