KJM 9250

1D-SELECTIVE NMR Experiments on the AVI-600 Spectrometer.

Version 7.3

Topspin 1.3 Windows XP AVI600

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January 2020

1D-SELECTIVE NMR Experiments on the AVI-600

1.1 Introduction

aw coded 1D-Selective experiments use soft 180° refocusing pulses with 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° soft pulse experiment is ca 25 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: 80000 usec at the PLW level read in by the **getprosol** command
Other: 40000 usec, subtract 6 db to the PLW level read in by the **getprosol** command
120000 usec, add 3 db to the PLW level read in by the **getprosol** command
160000 usec, add 6 db to the PLW level read in by the **getprosol** command
240000 usec, add 9 db to the PLW level read in by the **getprosol** command

1.2 NS x TD0 option

1D-SELECTIVE experiments can be run using the ZG command in which case a *multiple of 4 or 8 scans should be acquied***,** or the ***NS** x **TD0*** option where **NS** is a multiple of 8, 16, 32, 64 (etc) and **TD0** is any number >1.

Do NOT use the **TR** command as a **NS x TD0** experiment proceeds. Multiples of **NS** scans will be automatically saved and can be processed using the FT or EFP commands the experiment proceeds.

A **NS x TD0** run can be terminated at any time using the **STOP** (*NB not the* **HALT** command). This will ensure a multiple of 8 (or 16) scans is saved as is required by some of the selective excitation experiments.

1.3 1D-SELECTIVE Experiments

The following 1D-Selective experiments have been set up on the AVI-600 MHz spectrometer

2.1 SELCOSY spectra

2.2 SELTOCSY spectra

2.3 SELDIPSI2 spectra

2.4 Phasing SELNOESY and SELROESY spectra

2.5 SELNOESY spectra

2.6 SELROESY spectra

2.7 SELROESY.2 spectra

2.1 SELCOSY spectra

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

TD = 32 K, **SI** = 32 K
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.

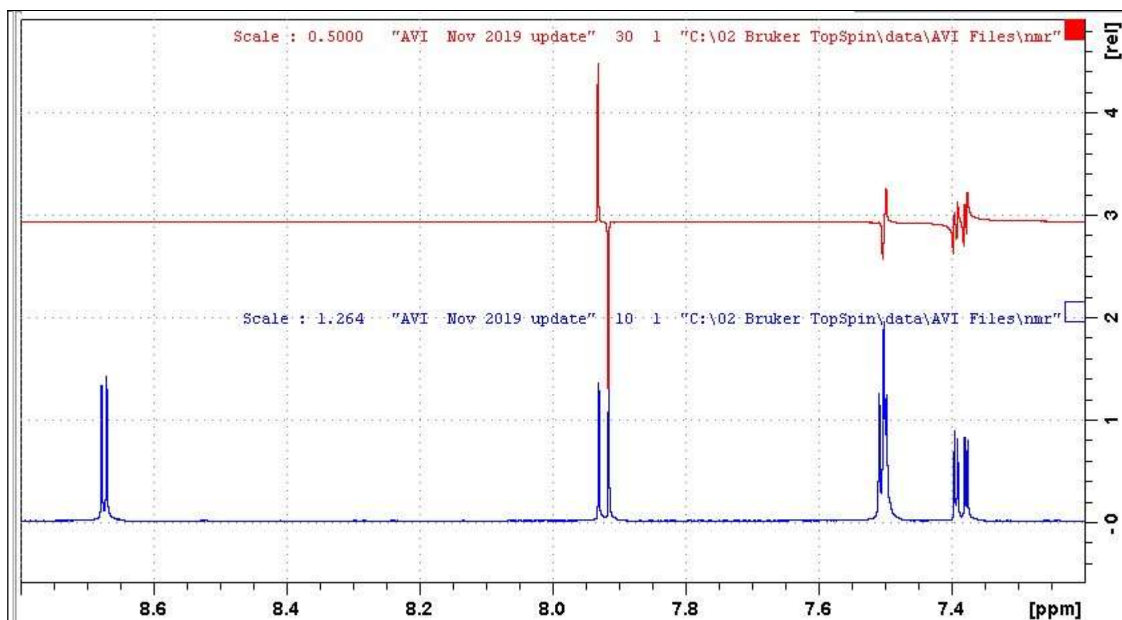
NS = multiple of 8 or 16, **DS** = 4 or 8.
or **NS x TD0** scans where **TD0** = any positive number.

D1 = 1 sec or other time of your choice
D14 = $1/4J_{H\text{--}H}$. A value in the range 30-40 msec (default 35 msec) generally works well irrespective of $J_{H\text{—}H}$.

P12 = 80000 usec, **SPW2** = **Gaus1_180r.100** shaped pulse power (read in using the getprosol command).

Process with **EFP** (applies **LB** = 0.1 Hz).
Phase the spectrum to afford ***antiphase positive and negative signal intensities.***

Alternatively process the spectrum in **MC** (**magnitude)** mode to afford ***positive correlation peaks*** (see the next page).



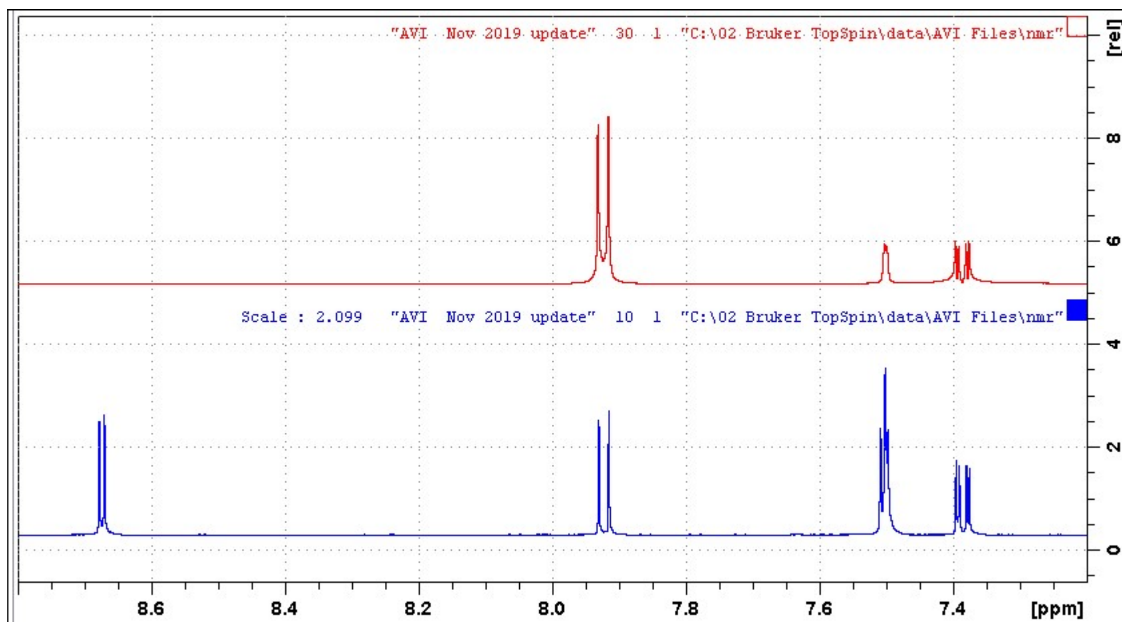
Lower: 7-8 ppm region of the AVI-600 NMR spectrum of quinine in D_6-DMSO.
Upper: SELCOSY spectrum ex the signal at 7.39 ppm showing it is coupled to the signal at 7.93 ppm and one of the signals at 7.5 ppm.

Magnitude mode (PS) processing of a SELCOSY spectrum

To convert an antiphase SELCOSY spectrum generated by normal **FT** or **EF** processing to a **Magnitude spectrum** type:

MC (return), and (if required)

abs (return) to position the spectrum at its normal baseline position.



Lower: 7-9 ppm region of the AVI-600 ^{1}H NMR spectrum of quinine in D_6-DMSO.
Upper: Magnitude (MC) mode processed SELCOSY spectrum ex the signal at 7.39 ppm.

2.2 SELTOCSY spectra

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

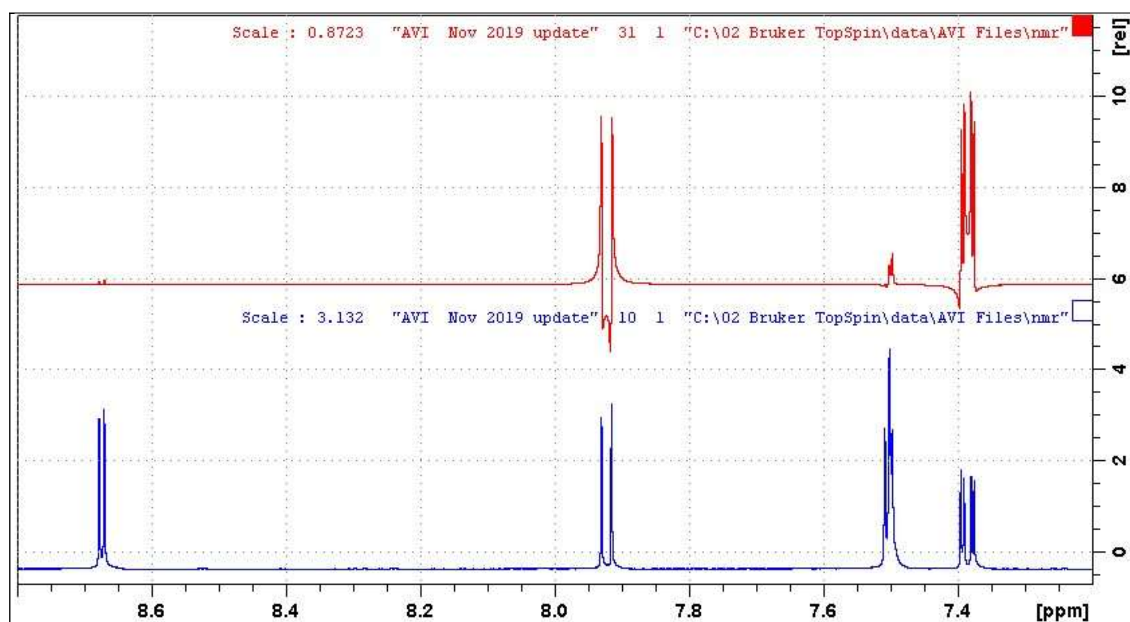
TD = **SI** = 32 K or 64K
O1 = Frequency in Hz of signal to be selectively correlated. **O1** Excitation is applied *on resonance* at the middle of SW. Check SW is wide enough.

NS = multiple of 8 or 16, **DS** = 4 or 8.
or **NS x TD0** scans where **TD0** = any positive number.

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** = **Gaus1_180r.100** shaped pulse.
PL10 = 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: 7-9 ppm region of the AVI-600 ^{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 spectra

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

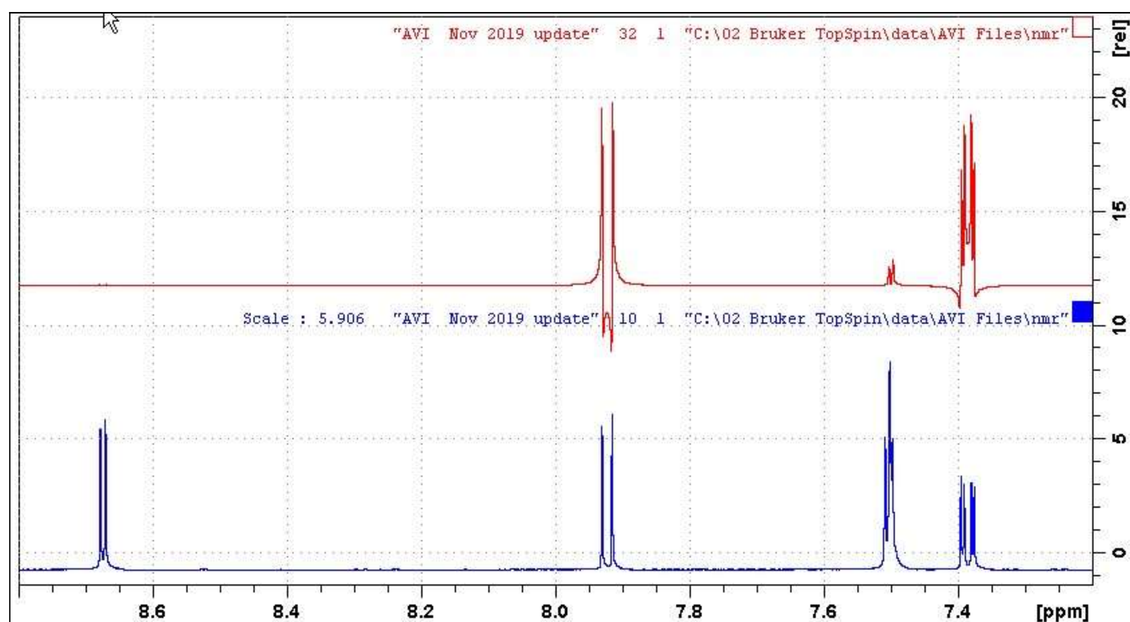
TD = 64 K, **SI** = 64 K, **SW** = 16 ppm.
O1 = Frequency in Hz of signal to be selectively correlated. **O1** Excitation is applied *on resonance* at the middle of SW. Check SW is wide enough.

NS = multiple of 8 or 16, **DS** = 4 or 8.
or **NS x TD0** scans where **TD0** = any positive number.

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** = **Gaus1_180r.100** shaped pulse power .
PL10 = 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.

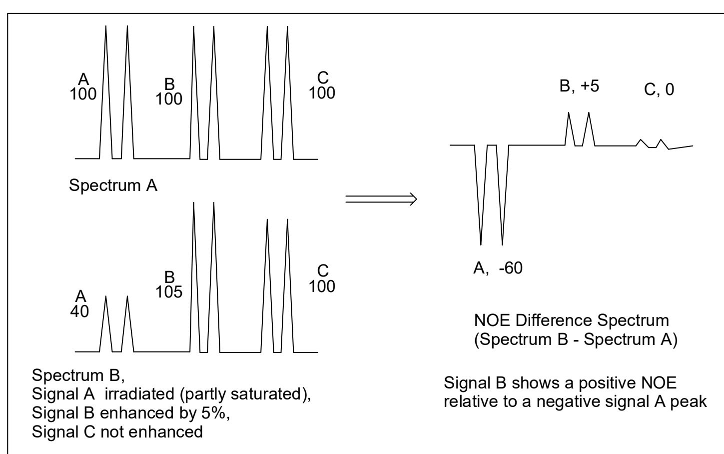


Lower: 7-9 ppm region of the AVI-600 ^{1}H NMR spectra of quinine in D_6-DMSO.
Upper: D9 = 80 msec SELDIPSI2 spectrum ex the signal at 7.39 ppm.

2.4 Phasing of 1D-SELNOESY and SELROESY spectra

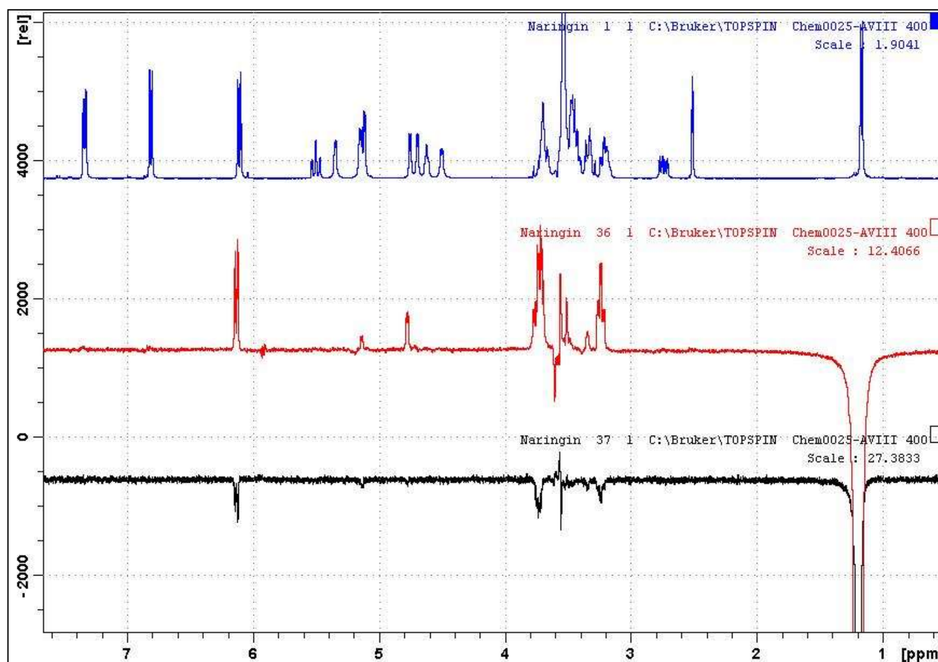
NOESY and SELNOESY peaks may be positive, zero or negative depending on correlation times whereas *ROESY and SELROESY peaks are always positive.*

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 spectra

Parameter set: **awselnoesy (+ getprosol)**
Pulse programme: **selnogp4**

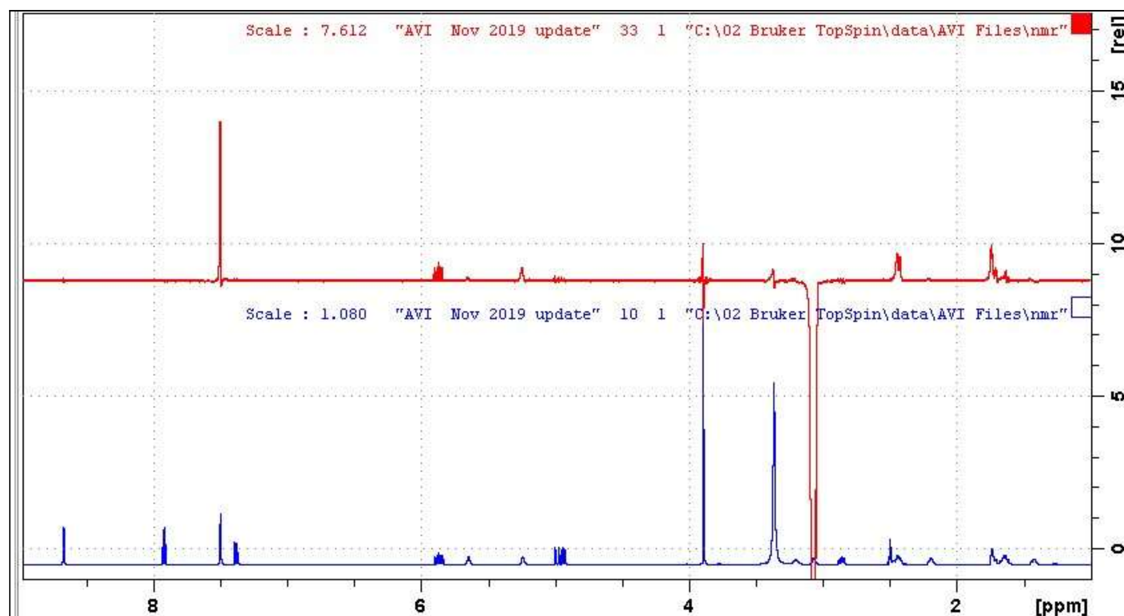
TD = 32 K, **SI** =32 K, **SW** = 16 ppm.
O1 = Frequency in Hz of signal to be selectively correlated. **O1** Excitation is applied *on resonance* at the middle of SW. Check SW is wide enough.

NS = multiple of 4, 8 or 16, **DS** = 4 or 8.
or **NS x TD0** scans where **TD0** = any positive number.

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

P12 = **80000 usec**, **SPW2** = **Gaus1_180r.100** shaped pulse power read.
PLW10 = TOCSY spin lock power level (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 occasionally be present in NOESY spectra.



Lower: 1-9 ppm region of the AVI-600 ^{1}H NMR spectra of quinine in D_6-DMSO.
Upper: SELNOSY spectrum ex the signal at 3.07 ppm.

2.6 SELROESY (CW spin locked) spectra

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

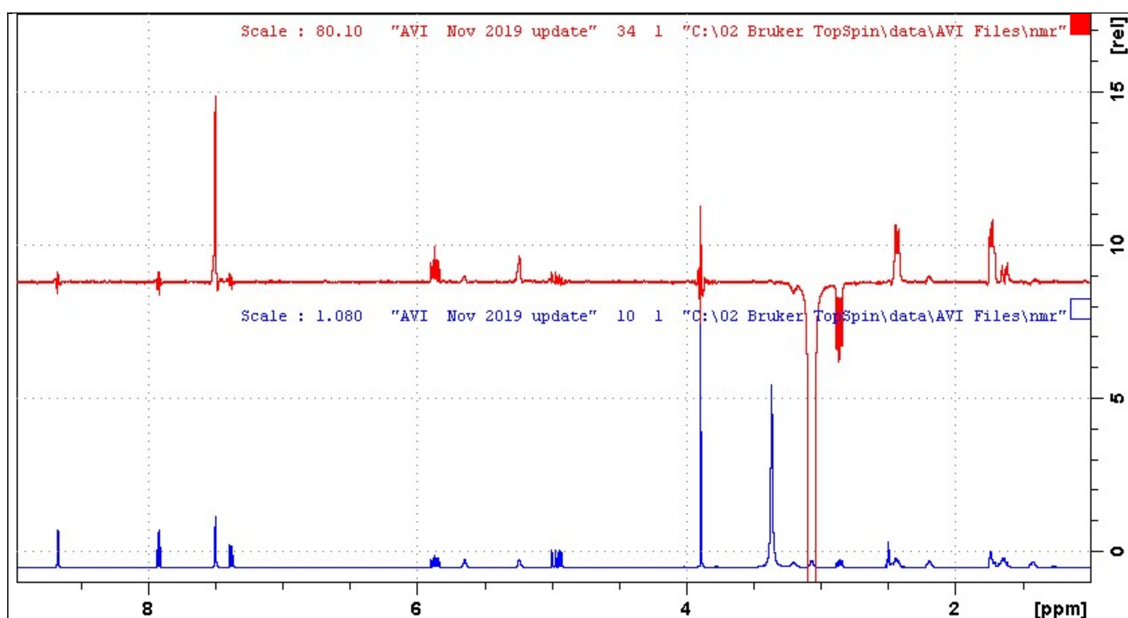
TD = 32 K, **SI** = 32 K, **SW** = 20 ppm.
O1 = Frequency in Hz of signal to be selectively correlated. **O1** Excitation is applied *on resonance* at the middle of SW. Check SW is wide enough.

NS = multiple of 4, 8 or 16, **DS** = 4 or 8.
or **NS x TD0** scans where **TD0** = any positive number.

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

P12 = 80000 usec, **SPW2 = Gaus1_180r.100** shaped pulse power.
PLW11 = 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: 1-9 ppm region of the AVI-600H NMR spectra of quinine in D_6-DMSO.
Lower: SELROESY spectrum ex the sig nal at 3.07 ppm. A negative TOCSY artifact signal ex a strongly coupled signal appears at 2.88 ppm

TOCSY artifact peaks are generally less significant in pulsed spin locked SELROESY.2 spectra \ (see Section 2.7).

2.7 SELROESY2 (pulsed spin locked) spectra

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

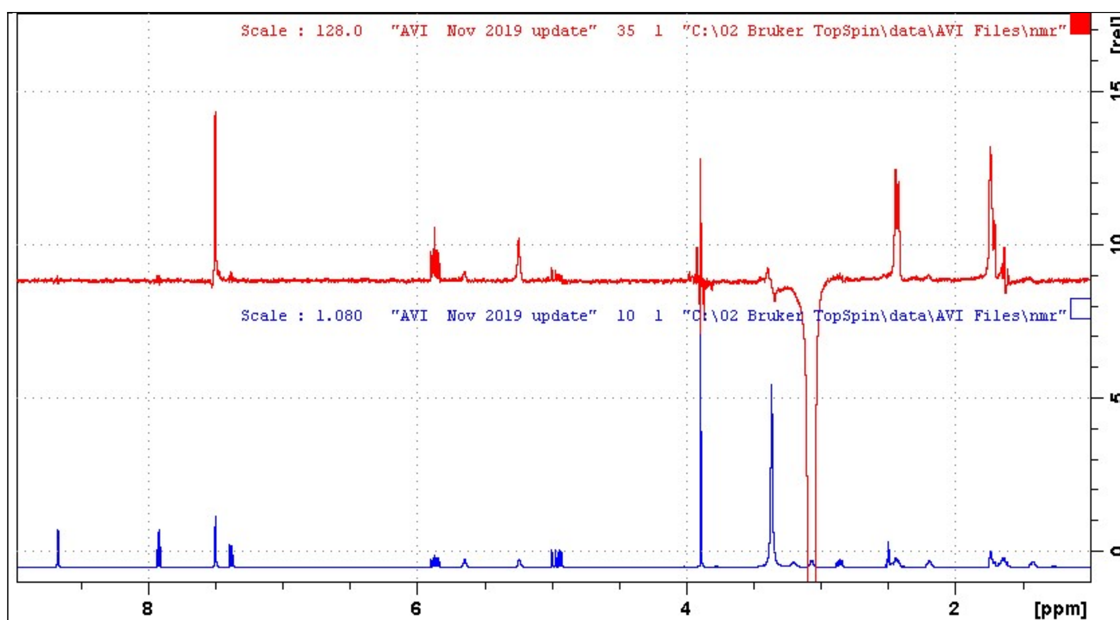
TD = 32 K, **SI** = 324 K, **SW** = 16 ppm.
O1 = Frequency in Hz of signal to be selectively correlated. **O1** Excitation is applied *on resonance* at the middle of SW. Check SW is wide enough.

NS = multiple of 4, 8 or 16, **DS** = 4 or 8.
or **NS x TD0** scans where **TD0** = any positive number.

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

SP2 = shaped pulse power level (read in by the getprosol command).
PLW27 = 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: 7-9 ppm region of the AVI-600 ^{1}H NMR spectra of quinine in D_6-DMSO.
Lower: SELROESY2 spectrum ex the signal at 3.07 ppm.

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