Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information.

Quick Re-introduction of Selective Scalar Interactions in Pure-Shift NMR Spectrum

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Contents

1. Sample Preparation

All the samples were used as purchased without any further purification. The chemicals used for investigation are propylene carbonate, strychnine and *L*-menthol. The solutions were prepared by dissolving approximately 5mg/5µl of the sample in 500 µl of $CDCl_3$

2. Expansion of spin echo loop: Discussion for time matching on both sides of spin echo.

The expansion of spin echo loops of the pulse sequence is given below. In this QG-SERF 1d sequence there are many spin echo blocks. Except for the first and last spin echo all other spin echo blocks shares approximately equal times on both side of hard 180^0 pulse even in the presence of a selective pulse, which is discussed in the figure given below. As a consequence of equal times on both sides of selective pulse, the error in measurement is minimized.

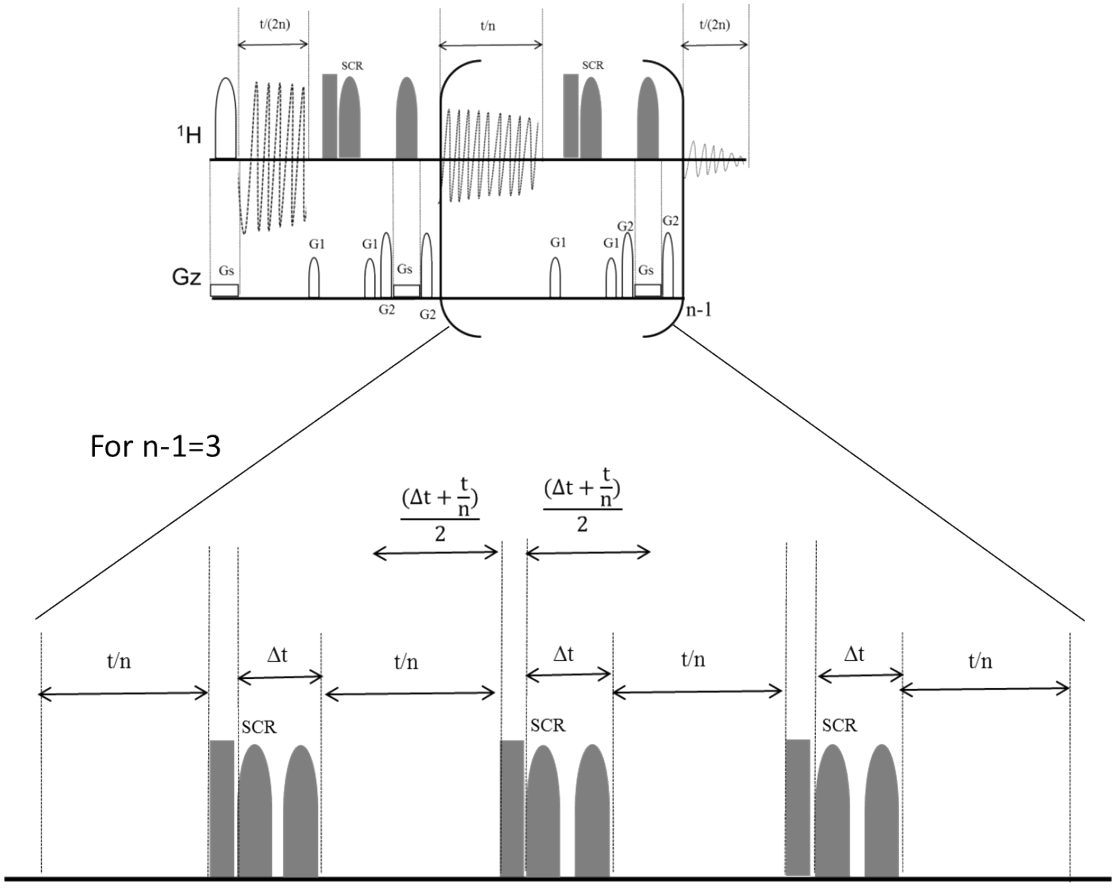


Figure S1: Block diagram of expanded spin echo loop

3. QG-SERF pulse program code for Bruker NMR spectrometer

For Avance –III version, topspin 2.1 and 3.1

```
#include <Avance.incl>
#include <Grad.incl>
#include <Delay.incl>
#include <De.incl>

dwellmode explicit


"d12=6.5u"
"d2=aq/l0"
"d3=d2/2"
"l1=l0-1"
"p2=2*p1"


1 ze
2 3m
 d1
 50u UNBLKGRAD
 10u pl0:f1
 300u gron4            ; Slice selection gradient On
 (p11:sp1 ph1):f1      ; 90 (selective)
 100u groff            ; Slice selection gradient Off


 ACQ_START(ph30,ph31)

 0.05u setrtp1|0
 0.1u setrtp1|5
 d3:r
 0.1u setrtp1^5
 0.05u setrtp1^0

 p16:gp2
 d16 pl1:f1
 p2 ph2
 2u pl0:f1
(p14:sp4 ph3):f1      ; selective couplings re-introductory 180^0 pulse (SCR)
```

```
  p16:gp2
  d16

  p16:gp3
  d16
  300u gron4            ; Slice selection gradient On
 (p12:sp2 ph3):f1       ; 180 (selective)
  100u groff            ; Slice selection gradient Off
  p16:gp3
  d16

3  0.05u setrtp1|0
  0.1u setrtp1|5
  d2:r
  0.1u setrtp1^5
  0.05u setrtp1^0

  p16:gp2
  d16 pl1:f1
  p2 ph2
  2u pl0:f1
 (p14:sp4 ph3):f1       ; selective couplings re-introductory 180^0 pulse (SCR)
  p16:gp2
  d16

  p16:gp3
  d16
  300u gron4            ; Slice selection gradient On
 (p12:sp2 ph3):f1       ; 180 (selective)
  100u groff            ; Slice selection gradient Off
  p16:gp3
  d16

lo to 3 times l1

  0.05u setrtp1|0
  0.1u setrtp1|5
  d3
  30m
  0.1u setrtp1^5
  0.05u setrtp1^0

rcyc=2
```

wr #0

exit

ph1=0 2 2 0 1 3 3 1
ph2=0 2
ph3=0 2
ph30=0
ph31=0 2 2 0 1 3 3 1

;pl1 : f1 channel - power level for pulse (default)
;p1 : f1 channel - high power pulse
;p12 : f1 channel - 180 degree selective pulse [ms]
;p14: f1 channel - selective couplings re-introductory 180^0 pulse (SCR)
;p16: homospoil/gradient pulse [500 us]
;sp1: f1 channel - shaped pulse power level for selective excitation
;spnam1: shaped pulse for selective excitation [EBurp1]
;sp2: f1 channel - shaped pulse power level for selective inversion
;spnam2: shaped pulse for selective inversion [Gauss]
;sp4: f1 channel - shaped pulse power level for selective refocusing
;spnam2: shaped pulse for selective refocusing [Gauss]
;d1 : relaxation delay
;d16: delay for homospoil/gradient recovery
;NS: 1 * n
;DS: 0
;l1: number of concatenated loops

4. Experimental Details.

All the experiments were carried out on a Bruker 800 MHz NMR spectrometer equipped with a cryo probe. The maximum gradient strength available is 53.5 Gcm^{-1}.

There is 3-4 times gain in sensitivity by the cryoprobe, compared to normal probes. Thus a comparison of the sensitivity gain and the save in time between QG-SERF and G-SERF experiments is given below for strychnine.

G-SERF

The G-SERF spectrum was obtained on 600 MHz NMR spectrometer with a normal probe. The concentration of the solution was 35 mg of strychnine in approximately 500 µl of $CDCl_3$.

QG-SERF

The QG-SERF spectrum was obtained on 800 MHz NMR spectrometer with a cryoprobe. In order to compensate the sensitivity gained due to cryoprobe and higher magnetic field, we have reduced the concentration of the solution. The sample concentration used is 5mg of strychnine in approximately 500 µl of $CDCl_3$ (7 times lesser concentration than in G-SERF).

(A) Quick G-SERF on propylene carbonate

The Hard 180^0 pulse of 16 µs duration was used. For 90^0 selective pulse, 40 ms duration (band width of 105 Hz) EBurp shaped pulse was used. The 180^0 selective pulse was of 10 ms duration (band width of 90 Hz). Gaussian shaped pulse was used. Number of loops "n" used was 40 for the acquisition time of 1 sec. The gradient strengths used were G1=21.7%, G2=33.7% and Gs =0.8% of maximum gradient strength available. G1 and G2 are applied for duration of 500 µs (p16) with gradient recovery time of 20 µs (d16).

(B) Quick G-SERF on Strychnine

The Hard 180^0 pulse of 16 µs duration was used. For 90^0 selective pulse, 40 ms duration (band width of 105 Hz) EBurp shaped pulse was used. The 180^0 selective pulse was of 5 ms duration (band width of 160 Hz). Gaussian shaped pulse was used. Number of loops "n" used was 40 during acquisition time of 1 sec. The gradient strengths used were G1=21.7%, G2=33.7% and Gs =0.9% of maximum gradient strength available. G1 and G2 are applied for duration of 500 µs (p16) with gradient recovery time of 20 µs (d16).

5. Quick G-SERF: Experimental details and spectra of *L*-menthol

The Hard 180^0 pulse of 16 µs duration was used. For 90^0 selective pulse, 40 ms duration (band width of 105 Hz) EBurp shaped pulse was used. The 180^0 selective pulse was of 10 ms duration (band width of 90 Hz). Gaussian shaped pulse was used. Number of loops "n" used was 40 for the acquisition time of 1 sec. The gradient strengths used were G1=21.7%, G2=33.7% and Gs =1.2 % of maximum gradient strength available. G1 and G2 are applied for duration of 500 µs (p16) with gradient recovery time of 20 µs (d16).

For measurement of all the inter-proton spin couplings of proton labeled as 6 in *L*-menthol.

The chosen region of conventional 1D spectrum of *L*-menthol is given in Fig. A. The proton 6 shows scalar interactions with protons 5, 5' and 1. From this 1D spectrum, it is difficult to extract accurately and unambiguously all these couplings. On the other hand the recorded QG-SERF spectrum given in Fig. C, exhibits enhanced resolution due to broadband homo-decoupling, while retaining the all scalar interaction of proton 6 with its coupled partners. The measured coupling values are reported on the top of each peak. The FID was acquired for 80 transients in 5 mins, which otherwise would have taken 1.5 hours from G-SERF to gather identical information.

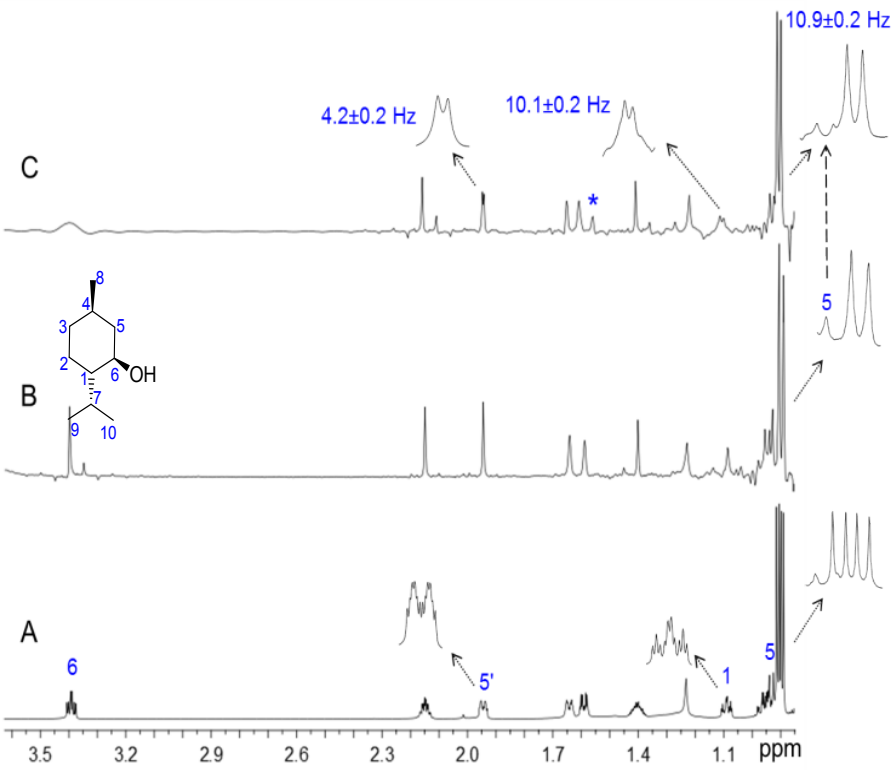


Figure S2. Labeled chemical structure, conventional ^{1}H 1D spectrum (A), 1D pure-shift ZS-spectrum (B) and 1D QG-SERF spectrum (C) of *L*-menthol. Spectrum C gives only proton-proton couplings of proton 6. Artifact peak is marked with *.

6. 400 MHz spectra of Strychnine and *L*-menthol

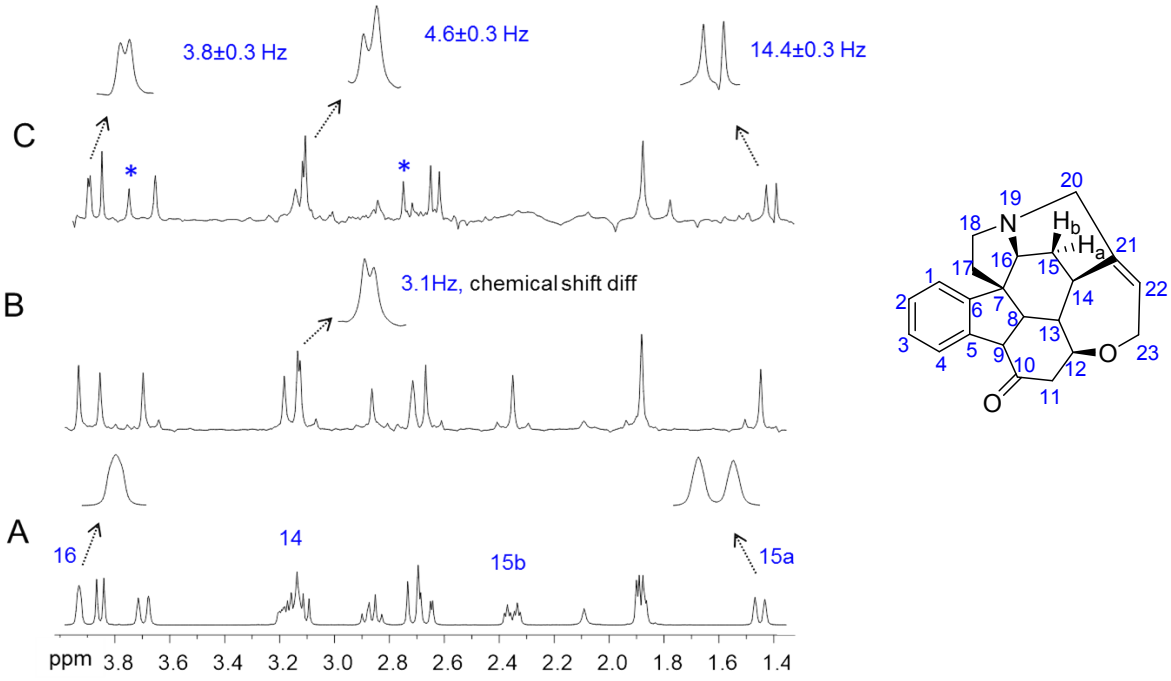


Fig S3: 400 MHz spectra of strychnine; (A) conventional one dimensional ^{1}H spectrum, (B) instant ZS pure-shift and (C) QG-SERF. Splitting for proton peak of 14 is overlapped with neighboring one in C due to less chemical shift dispersion (compared to 800 MHz spectrum). The selective pulse of 15 ms was used. Gz =1.4 %

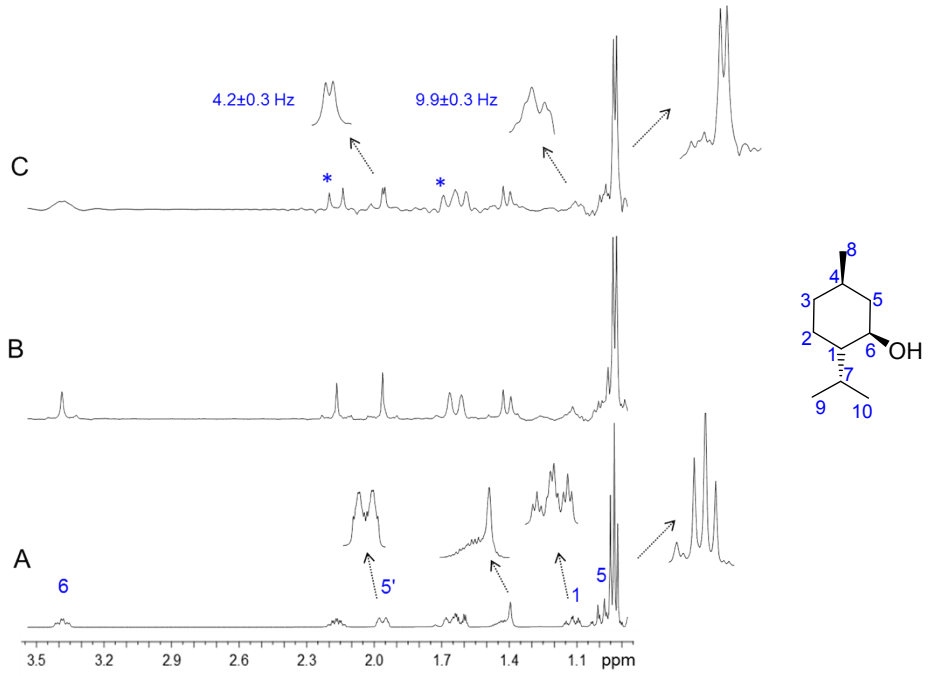
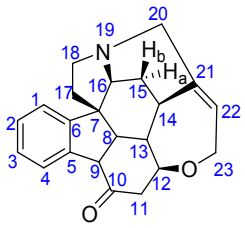


Fig S3: 400 MHz spectra of menthol; (A) conventional one dimensional ^{1}H spectrum, (B) instant ZS pure-shift and (C) QG-SERF. An additional artifact appeared in the spectrum than compared to 800 MHz spectrum. The proton peak 5 and its splitting (C) is unidentified due to severe overlap with other closely resonating strong intensity peaks (due to reduced chemical shift dispersion at 400 MHz). The selective pulse of 20 ms was used. Gz =1.4 %

7. Comparison of couplings measured by different techniques

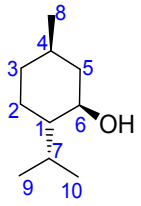
(a) Strychnine



Experimental Method	J_{HH} for different pairs of protons in Hz			Reference
	H_{15b}-H_{14}	H_{15b}-H_{16}	H_{15b}-H_{15a}	
QG-SERF	4.8±0.2	3.9±0.2	14.5±0.2	Present work

G-SERF	5.2	4.1	14.4	Nicolas Giraud *et al., Angew. Chem. Int. Ed. 2010,* **49**, 3481
J doubling method	4.76	3.89	14.32	F. D Río-Portilla *J Magn Reson series A., 1994,* **111,** 132,
1D 1H method	4.58	4.11	14.37	J. C Carter et al., *J Magn Reson. 1974,* **15,** 122

(b) Menthol



Experimental Method	**J_{HH} for different pairs of protons in Hz**			**Reference**
	H_6-$H_{5'}$	**H_6-H_1**	**H_6-H_5**	
QG-SERF	4.2±0.2	10.1±0.2	10.9±0.2	Present work
G-SERF	4.1	9.9	10.7	Nicolas Giraud *et al., Angew. Chem. Int. Ed. 2010,* **49,** 3481