Supporting Material

Visualizing unresolved scalar couplings by real-time J-upscaled NMR

*Simon Glanzer and Klaus Zangger**

Institute of Chemistry / Organic and Bioorganic Chemistry,

University of Graz, Austria

*to whom correspondence should be addressed at:

Institute of Chemistry / Organic and Bioorganic Chemistry, University of Graz, Heinrichstrasse 28, A-8010 Graz, Austria

Email: klaus.zangger@uni-graz.at

Tel : ++43 316 380-8673

Fax : ++43 316 380 9840

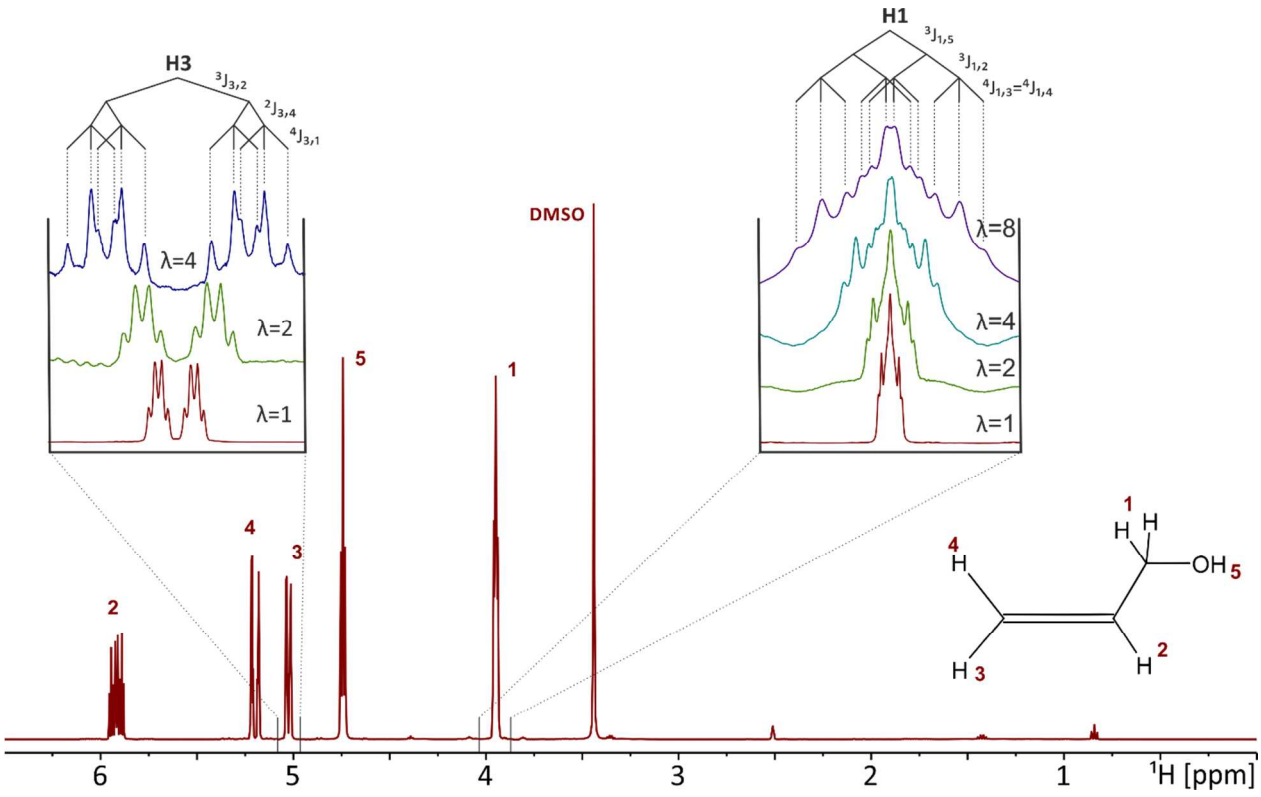

Fig. S1: A regular 1D ^{1}H spectrum of 2-propenol in DMSO-d_6 in red. Close up views of the regular and upscaled spectra of the H3 and H1 peak respectively are shown in the insets. For all spectra 32k data points were recorded. The number of loops was between 84 and 140 and the chunking times were between 15 and 24 ms.

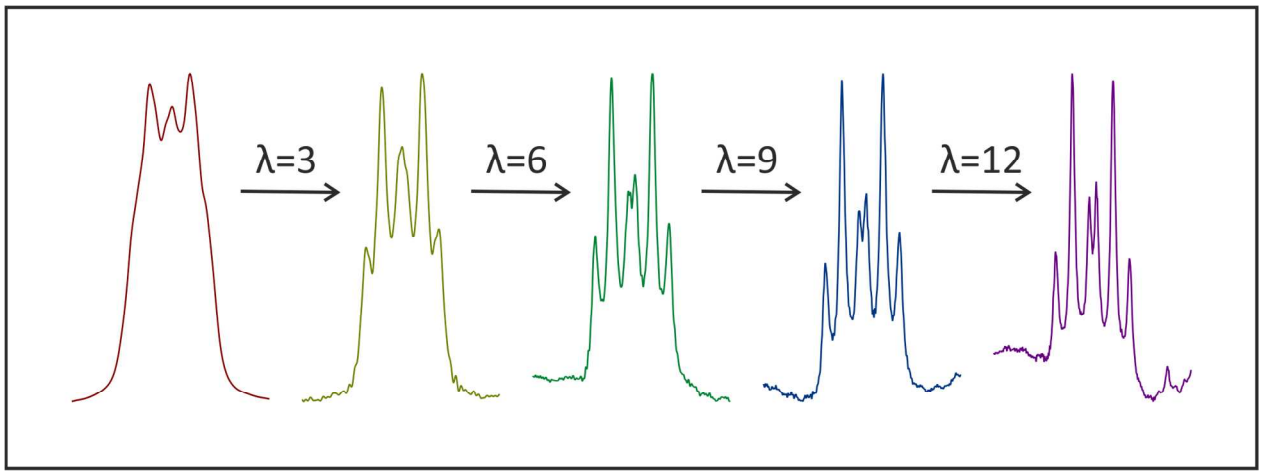

Fig. S2: The H2-signal of nicotinic acid extracted from figure 3 of the main text. The signals are compressed by the scaling factors (i.e. all signals are displayed with the same width) to demonstrate the actual resolution enhancement. The signal extracted from a regular ^{1}H spectrum is displayed in red.

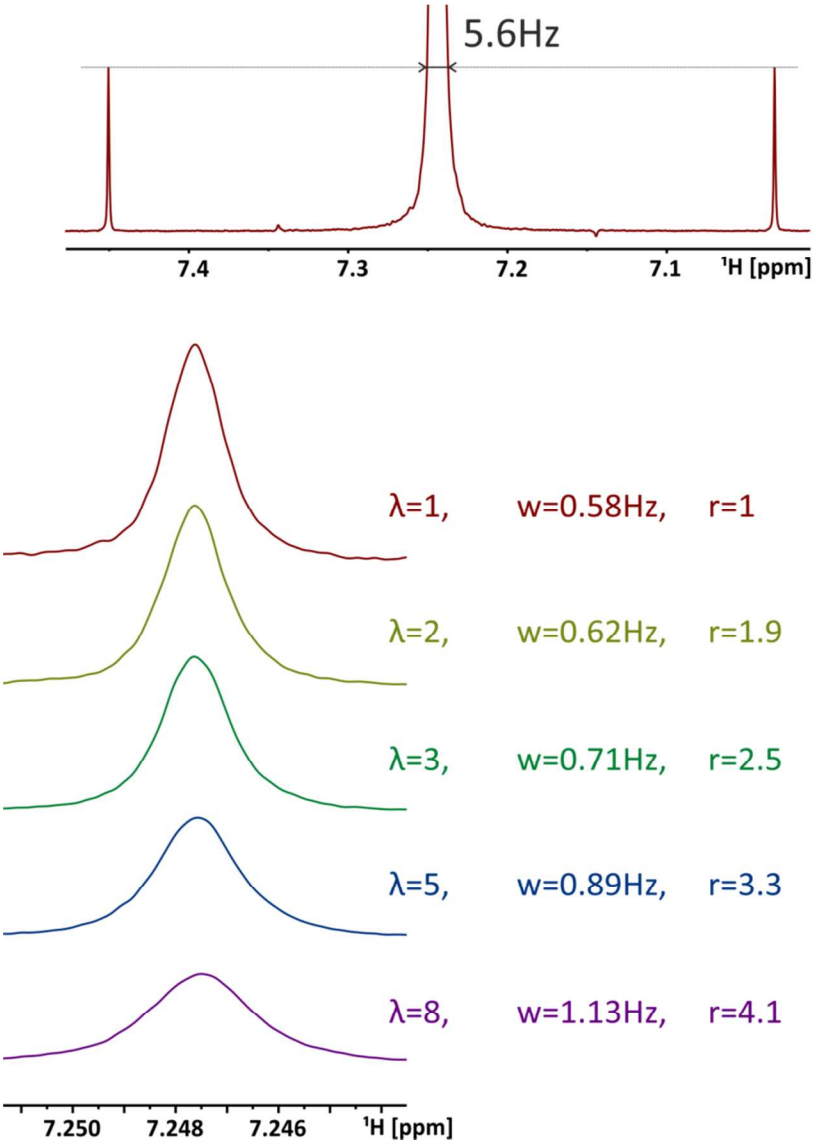

Fig. S3: To investigate the resolution enhancement and also signal intensity reduction upon J-upscaling, without the influence of any potentially unresolved long-range couplings, the ^{1}H linewidth of $CHCl_3$ (1% in $CDCl_3$) was monitored as a function of the J-upscaling factor. After extended gradient and manual shimming, a linewidth at the height of the ^{13}C satellites of 5.6 Hz (see above) was obtained, corresponding to a signal width at half height (w) of 0.58 Hz. Upon J-upscaling the signal width increased by the indicated numbers. J-upscaling by e.g. λ=8 only doubles the linewidth, while any coupling constants would be expanded 8 fold, resulting in an effective resolution enhancement (r) by a factor of ~4. For the same scaling factor, the intensity is reduced down to 31 % as a result of the increased linewidth. For the regular experiment 128k data points were recorded. For all other spectra 32k data points were recorded. The number of loops was between 200 and 360 and the chunking times were between 18-33 ms.

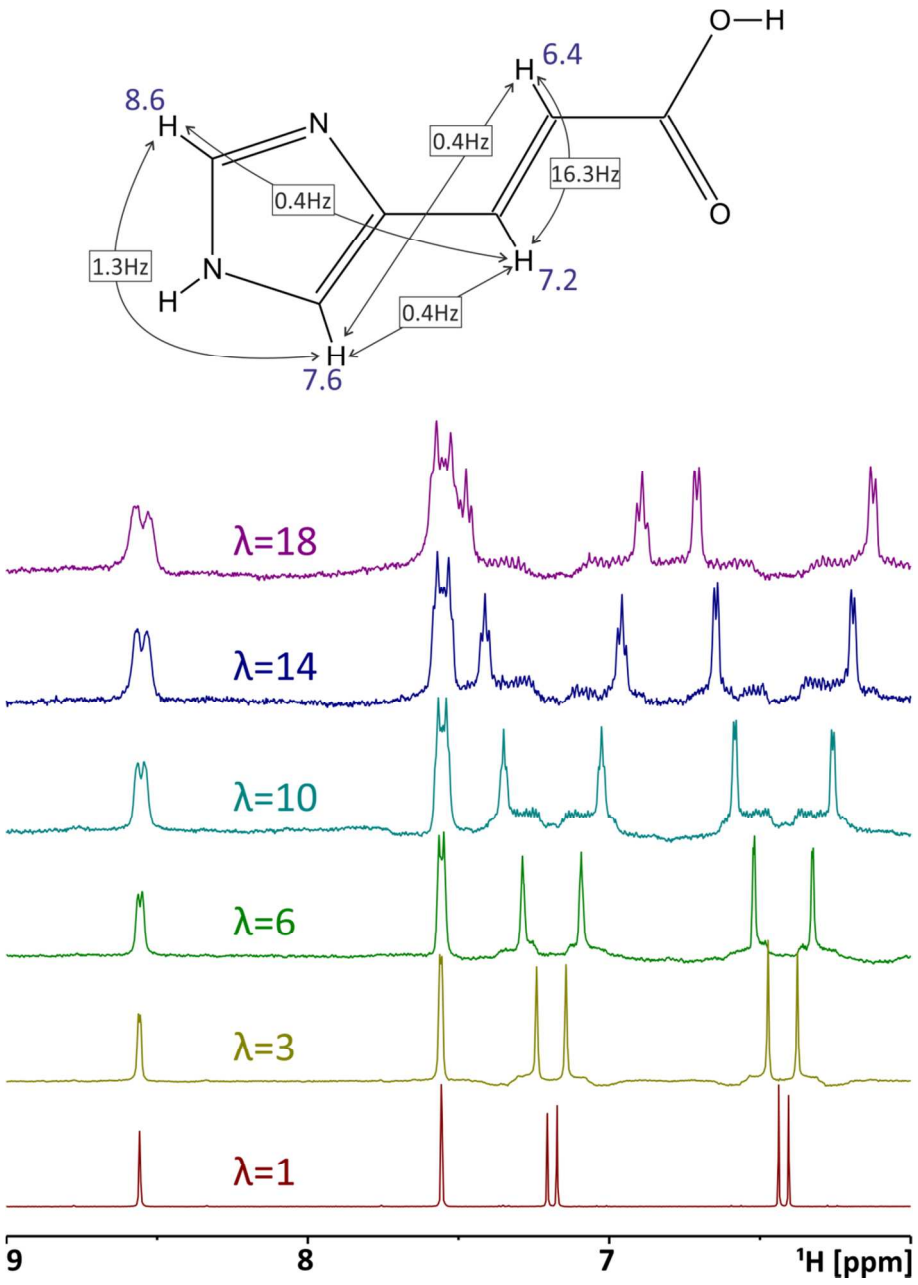

Fig. S4: To demonstrate the effect of extreme J-upscaling on weakly coupled signals, regular and J-upscaled spectra of urocanic acid in DMSO-d_6 are compared. While the two doublets of the CH=CH moiety (at 6.4 and 7.2 ppm) almost overlap in the eightfold upscaled spectrum, no enhanced roof effect is visible. During detection of the individual FID chunks, the system is still weakly coupled. For the regular experiment 64k data points were recorded. For λ=3 and λ=6 32k, for λ=10 and λ=14 16k and for λ=18 8k data points were used. Experiments for λ=3 used 150-270 loops with data chunks 12-22 ms, λ=6 used 250-450 loops and chunks of 7-13 ms, λ=10 used 170-306 loops and chunks of 6-10 ms, λ=14 used 240-432 loops and data chunks of 4-7 ms and λ=18 used 160-288 loops and data chunks of 3-5 ms.

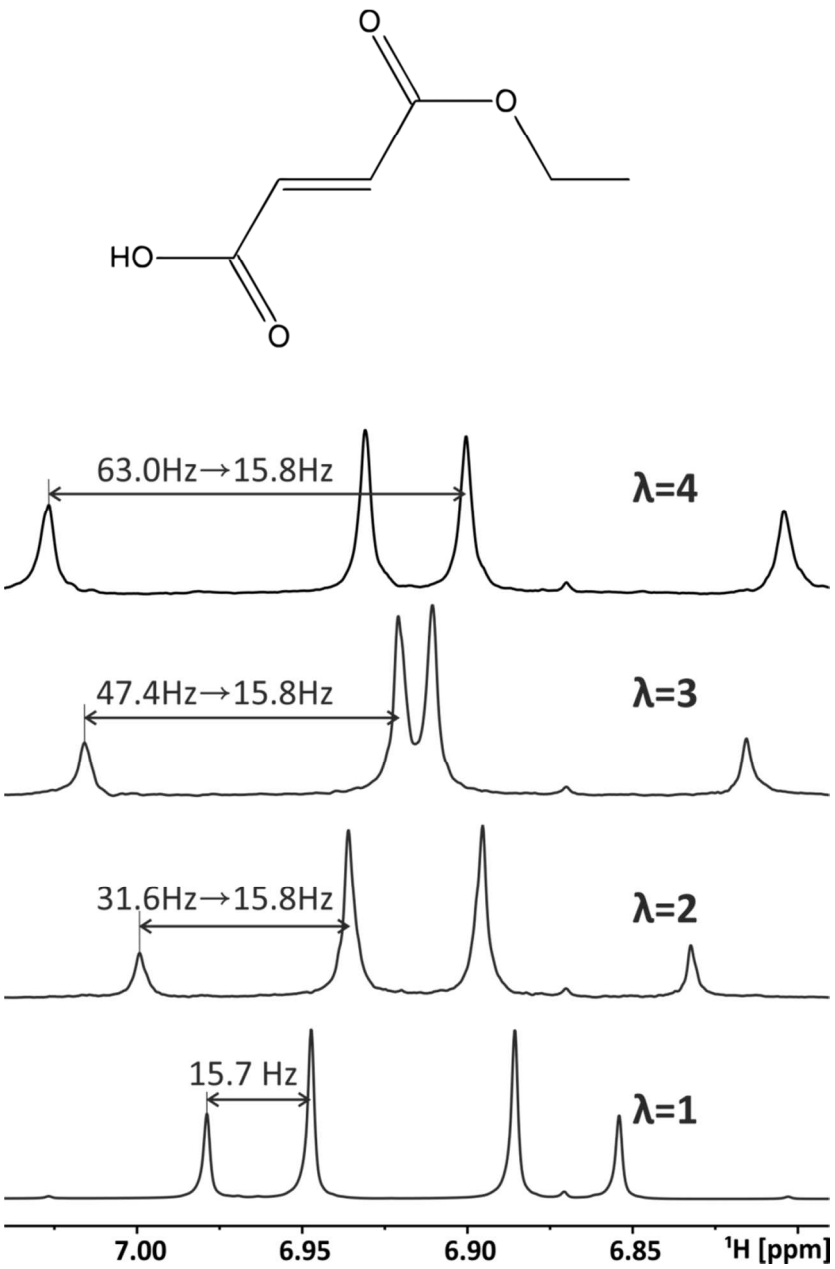

Fig. S5: To demonstrate the effect of J-upscaling on strongly coupled signals, regular and J-upscaled spectra of fumaric acid monoethyl ester in DMSO-d_6 are compared. While the two doublets of the CH=CH moiety (at 6.87 and 6.96 ppm) overlap in the fourfold upscaled spectrum, the roof effect is not enhanced. The moderately strong coupling does not get stronger during the detection of the individual data chunks. For all spectra 32k data points were recorded. The number of loops was 250 and the chunking time was 8.2 ms.

Pulse-sequence for a 1D J-upscaling experiment in Bruker format

```
;zgadc2
;avance-version (06/01/20)
;1D sequence with explicit programming of acquisition
;
;$CLASS=HighRes
;$DIM=1D
;$TYPE=
;$SUBTYPE=
;$COMMENT=


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

dwellmode explicit

define list<delay> dlist = { 1 1.1 1.2 1.3 1.4 1.5 1.6 1.7 1.8 }

"d12=6.5u"
"d2=aq/(l0)"
"d3=d2/2"
"l1=l0-1"
"p2=2*p1"
"d10=(d2*l9-d2)/2"


1 ze

3 3m
  "d22=aq/(l0*dlist)"
  "d20=(d22*l9-d22)/2"
  "d23=d22/2"
  "l5=l0*dlist"
  4u BLKGRAD
  d1 rpp2
  50u UNBLKGRAD

  d12 pl1:f1
  p1 ph1
  d12

  ACQ_START(ph30,ph31)

   0.05u DWL_CLK_ON
   0.1u REC_UNBLK
   d23:r
   0.1u REC_BLK
   0.05u DWL_CLK_OFF

   d20
   p2 ph2
   d12

   10u
   d20
```

```
   p2 ph3
   d12

4  0.05u DWL_CLK_ON
   0.1u REC_UNBLK
   d22:r
   0.1u REC_BLK
   0.05u DWL_CLK_OFF

   d20
   p2 ph2
   d12

   10u
   d20
   p2 ph3
   d12

lo to 4 times l5

  0.05u DWL_CLK_ON
   0.1u REC_UNBLK
   d23:r
   25m
   0.1u REC_BLK
   0.05u DWL_CLK_OFF

dlist.inc
rcyc=3


wr #0


exit

;ph1  = 0
;ph2  = 0
;ph31 = 0
;ph30 = 0
ph1 =0 2 2 0 1 3 3 1
ph2 = 0 2
ph3 = 2 0
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
;d1 : relaxation delay; 1-5 * T1
;NS: 1 * n, total number of scans: NS * TD0
;l20:
;l9 : J-scaling factor (lamda)
;l5 : loop counter * dlist
;d10 : J-scaling delay (tau)
;d2 : duration one acquisition block


;$Id: zgadc,v 1.12 2009/07/02 16:40:47 ber Exp $
```