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OPTIMIZATION AND PRACTICAL IMPLEMENTATION OF ULTRAFAST 2D NMR EXPERIMENTS

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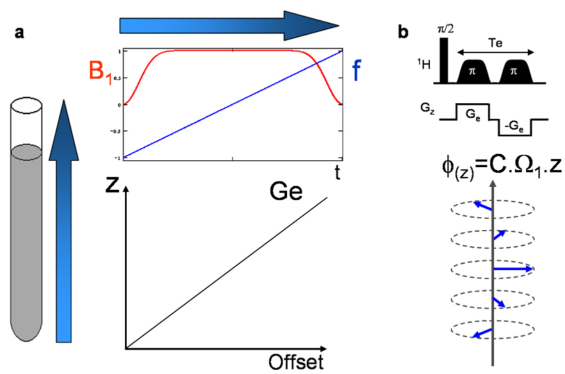

Figure 1S. *(a) Graphical representation of continuous spatial encoding, in which a chirp pulse is applied concomitantly to a gradient pulse (G_e) along z axis. (b) The scheme proposed by Pelupessy[20] comprises the application of a 90º hard pulse followed by two π chirp pulses applied together with a bipolar pair of gradients*

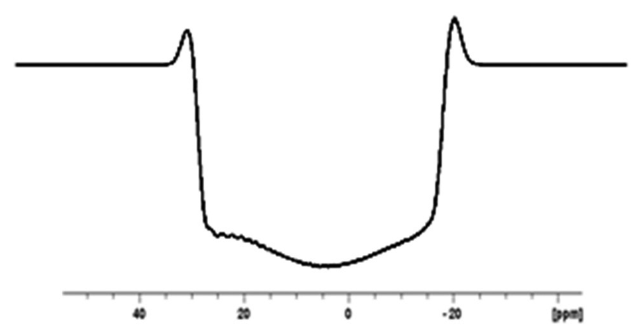

Figure 3S. *Image of the excitation profile obtained from the pulse sequence in Figure 1 (with the extra block) and after phase correction, allowing for the chirp pulse power calibration before performing ultrafast experiments. The acquisition and processing parameters are the same mentioned for the Figure 2b, unless the chirp pulse power that was 0.32 W*

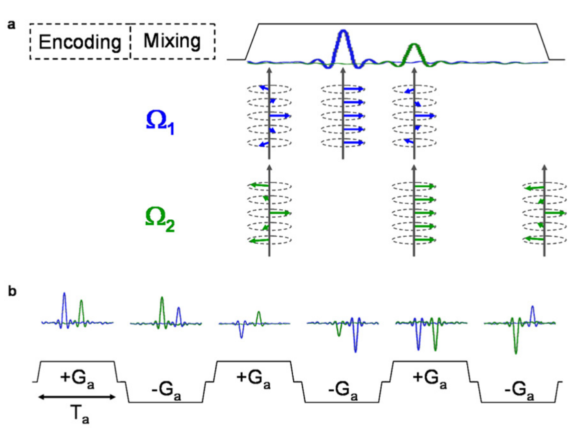

Figure 2S. *(a) Representation of the ultrafast dimension acquisition by the application of a gradient pulse, in order to remove the dephasing created during the spatial encoding step. As result, the echo peaks are formed as the dephasing is being refocused. (b) Representation of the conventional dimension acquisition by the use of a bipolar pair of gradient pulses, which results in the monitoring of conventional parameters evolution as a series of sub-spectra are being collected*

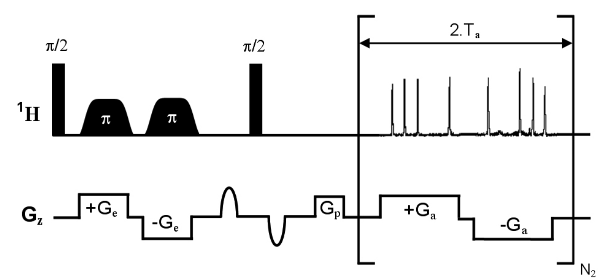

Figure 4S. *Ultrafast COSY pulse sequence based on the constant-time spatial encoding scheme proposed by Pelupessy.[20] To perform this experiment, the coherence selection gradients strength was 45 G/cm during 1000 µs, and the purge gradient strength was -10 G/cm with 400 µs of duration. The acquisition times for direct and indirect dimensions were 65.59 and 0.12 ms, respectively. For the acquisition 35 and -35.008 G/cm (to compensate for shearing effects) gradients were applied for 236 µs each, with a gradient rise time of 20 µs (recovery delay in pulse sequence)*

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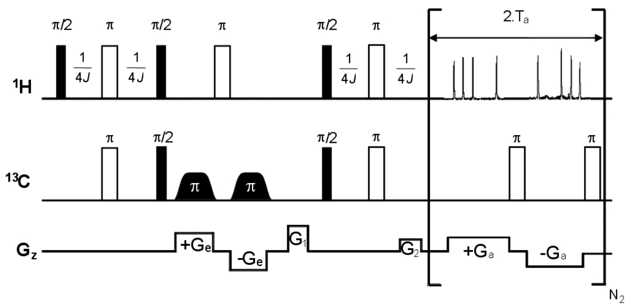

Figure 5S. *Ultrafast HSQC pulse sequence based on the constant-time spatial encoding scheme proposed by Pelupessy.[20] An asymmetric phase cycle (x,-x,x,x) was used within each single shot acquisition on the decoupling pulse to avoid the formation of decoupling artifacts. It was also used a real phase cycle (x,-x), requiring several scans, on the last ¹³C 90º pulse and the receiver. To perform this experiment, identical chirp pulses than those for UF-COSY were used, but applied in the presence of ± 20 G/cm encoding gradients to account for the larger ¹³C frequency range. The acquisition times for direct and indirect dimensions were 65.59 and 0.02 ms, respectively. Acquisition gradients were identical to those employed for UF-COSY. The INEPT delay was set to 1.72 ms. Eight scans were recorded for sensitivity and phase-cycling purposes*

PULSE SEQUENCE FOR UF-COSY

```
;ufcosy
;avance-version

;$CLASS=HighRes
;$DIM=1D
;$TYPE=
;$SUBTYPE=
;$COMMENT=

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

1 ze
  100u UNBLKGRAD
2 d1 pl1:f1
  p1 ph1                 → 90º pulse
  10u gron0              ┐
  p11:sp1:f1 ph2         │
  10u groff              │
  10u gron1              ├ spatial encoding block
  p11:sp1:f1 ph4         │
  10u groff              ┘
  10u pl1:f1
  10u
  p23:gp23               → coherence selection gradient
  10u
  p1 ph1                 → mixing period
  10u
  p26:gp26               → coherence selection gradient
  d25 gron25             → purge gradient
  10u groff
  10u
  ACQ_START(ph30,ph31)   ┐ starting acquisition
  1u DWELL_GEN:f1        ┘
3 d20 gron2              → positive acquisition gradient
  d6 groff               → gradient recovery delay
  d20 gron3              → negative acquisition gradient
  d6 groff               → gradient recovery delay
 lo to 3 times l3        → loop for acquisition
 rcyc=2
 100u BLKGRAD
 30m mc #0 to 2 F1QF(ip10, id0)
exit

ph1=0
ph2=0
ph4=2
ph30=0
ph31=0
```

PULSE SEQUENCE FOR UF-HSQC

```
;ufhsqc
;avance-version

;$CLASS=HighRes
;$DIM=2D
;$TYPE=
;$SUBTYPE=
;$COMMENT=

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

"p2=p1*2"
"p4=p3*2"
"d4=1s/(cnst2*4)"
"d6=d4-d15"
"d10=p20"
"d11=p21"
"p15=(td*dw)/(2*l3)-2*d17-p4"
1 ze
  100u UNBLKGRAD
2  30m pl2:f2
  d1 pl1:f1
  p1 ph0                            ┐
  d4                                │
  (center (p2 ph1) (p4 ph4):f2 )    ├ INEPT block
  d4                                │
  p1 ph2                            │
  (p3 ph3):f2                       ┘
  d11
  10u gron0                         ┐
  p7:sp1:f2 ph1                     │
  10u groff                         │
  d10                               │
  10u                               ├ spatial encoding block
  p2 ph1                            │   → 180º pulse
  10u                               │
  10u gron1                         │
  p7:sp1:f2 ph1                     │
  10u groff                         ┘
  d14 gron4 pl2:f2                  → coherence selection and folding gradient
  10u groff
```

```
 (p3 ph5):f2                  }
 (p1 ph1)                     } retro-INEPT block
 d4                           }
 (center (p2 ph1) (p4 ph4):f2 )}
 d6
 d15 gron5        ---->  coherence selection and folding gradient
 10u groff
 10u
 d15 gron7        ---->  purge gradient
 10u groff
 ACQ_START(ph30,ph31) } starting acquisition
 1u DWELL_GEN:f1      }
3 p15:gp15        ---->  positive acquisition gradient
 d17              ---->  recovery delay
 (p4 ph6):f2      ---->  decoupling  pulse
 d17              ---->  recovery delay
 p15:gp16         ---->  negative acquisition gradient
 d17              ---->  recovery delay
 (p4 ph6):f2      ---->  decoupling  pulse
 d17 ipp6         ---->  recovery delay / phase (ph6) increment
 lo to 3 times l3 ---->  loop for acquisition
 rcyc=2
 100u BLKGRAD
 30m mc #0 to 2 F1QF(id2)
exit

ph0=0
ph1=0
ph2=1
ph3=0
ph4=0
ph5=0 2
ph6=0 2 0 0       ---->  asymmetric phase cycle on the
                         decoupling pulse to avoid the formation
                         of decoupling artifacts
ph7=1
ph8=3
ph30=0
ph31=0 2
```