Supplementry material for Speeding up direct ^{15}N detection: hCaN 2D NMR experiment

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Bruker pulseprogram: hCaN 2D NMR experiment

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;3D sequence with
;   15N detected experiment
;   F2(Ca, t1) -> F1(N,t2)
;on/off resonance 13C pulses using shaped pulses
;phase sensitive STATES-TPPI(t1)
;f1:N, f2:H, f3:C, f4:D
;prosol relations=<triple_c>


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

"p2=p1*2"
"p4=p3*2"

"d11=30m"
"d12=20u"

"d21=11.3m"
"d22=14.3m"
"d23=11.3m"

;definition for constant time for t1
"d31=d22/2-p12/2" ;delay 1 for Ca constant time
"d30=d22-d21-p1" ;delay 2 for Ca constant time
"d29=d21-d22/2-p12/2-p1" ;delay 3 for Ca constant time
"d28=d22/2-p12/2" ;delay 4 for Ca constant time


;definition for nitrogen transverse delays
"d27=d23/2-p12/2"
"d26=d27-d12-4u"
"d14 = 1/(4*cnst4)";
" d13 = d14 - 0.5*p12"
"d25 = d28 - d13"

"in31=in0/4"
"in30=0"
"in29=in0/4"
;"in28=in0/4"
"in25=in0/4"

;cnst21: CO chemical shift (offset, in ppm)
;cnst22: Calpha chemical shift (offset, in ppm)

"d4=1/(4*cnst4)"
"d2=d4-d3";0.529*p6";10u";d4-0.5*p7-1000u";;0.53*p6";-0.5*p7

"spoff23=0"
"spoff24=0"
"spoff25=0"
"spoff26=bf3*((cnst21-cnst22)/1000000)"
"spoff15=bf3*((cnst21-cnst22)/2000000)"
```

```
1 ze
d11 pl16:f3
d11 LOCKDEC_ON
d11 pl17:f4
2 d11 do:f2 do:f3 do:f4
3 d11 H2_LOCK
9m LOCKH_OFF
d1 pl1:f1 pl2:f2 pl0:f3
50u UNBLKGRAD
d12 H2_PULSE
5u
d12 fq1:f4  ;D-alpha
d12 ;cpd4:f4 ;cpd2:f2
5u

(p1 ph1):f1
(p12:sp24 ph1):f3
p16:gp1
50m

  ;***** 1H--Ca INEPT****
(p3 ph4):f2
d4
(center (p4 ph1):f2 (p12:sp24 ph1):f3 );
d4
  (p3 ph2):f2
 ;*****HzCz****

  ;**** Ca --> N detection******;
(p11:sp23 ph3:r):f3 ; a90 Q5     ;Ca indirect_____________
d31
(center (p12:sp26 ph1):f3 (p4 ph1):f2);
d31
(p12:sp24 ph1):f3 ; Ca180 Q3
d30
(p2 ph1):f1 ; N180
d29
(p12:sp26 ph8):f3
d13;
(p4 ph1):f2
d25 ;= d28 - d13
(p11:sp25 ph8):f3 ; a90tr Q5tr   ;_____________________

4u do:f4 do:f2
5u
p16:gp1
d16 fq1:f4  ;D-N
20u pl12:f2
20u cpd2:f2 cpd4:f4
5u

(p1 ph1):f1                ;Ny i->i+/-1 transfer____
d27
(p12:sp26 ph1):f3
d27
(center (p2 ph1):f1 (p12:sp24 ph1):f3)
d27
```

```
(p12:sp26 ph1):f3
d26
d12 pl16:f3 ;pl12:f2
4u BLKGRAMP
go=2 ph31 cpd3:f3

d11 do:f2 do:f3 do:f4 mc #0 to 2
F1PH(ip3, dd31 & id30 & id29 & id25)

  d11 do:f2 do:f3 do:f4
  d11 H2_LOCK
  d11 LOCKH_OFF
  d11 LOCKDEC_OFF


exit

ph4=0 2
ph1=0
ph2=1
ph3=0
ph5=0
ph31=0 2 ;ph31 follow ph3
ph8 = 0

;pl1 : f1 channel - power level for pulse (default)
;pl3 : f3 channel - power level for pulse (default)
;pl12: f2 channel - power level for CPD/BB decoupling
;pl16: f3 channel - power level for CPD/BB decoupling
;sp3: f2 channel - shaped pulse 180 degree (adiabatic)
;sp23: f1 channel - shaped pulse  90 degree  (on resonance)
;sp24: f1 channel - shaped pulse 180 degree  (on resonance)
;sp25: f1 channel - shaped pulse  90 degree  (on resonance)
;                   for time reversed pulse
;sp26: f1 channel - shaped pulse 180 degree  (C=O off
resonance)
;sp27: f1 channel - shaped pulse 180 degree  (Ca off
resonance)
;sp28: f1 channel - shaped pulse 180 degree  (Ca on
resonance)
;p11: f1 channel -  90 degree shaped pulse
;p12: f1 channel - 180 degree shaped pulse
;p14: f2 channel - 180 degree shaped pulse for inversion
(adiabatic)
;d0 : incremented delay (F1 in 2D)                    [3
usec]
;d1 : relaxation delay; 1-5 * T1
;d11: delay for disk I/O                              [30
msec]
;d12: delay for power switching                       [20
usec]
;d21: 11.3m 1/8JaN  delay for Ca evolution can be determined
by paropt
;d22: 14.3m 1/(2*JCAB)
;d23: 11.3m 1/8JaN  delay for N evolution can be determined
by paropt
;o1p: Calpha chemical shift (cnst22)
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```
;in0: 1/(1 * SW(Ca)) =  DW(Ca)
;nd0: 1
;td1: number of experiments in F1
;FnMODE: States-TPPI (or TPPI) in F1
;cpd2: decoupling according to sequence defined by cpdprg2
;cpd3: decoupling according to sequence defined by cpdprg3
;pcpd2: f2 channel - 90 degree pulse for decoupling sequence
;pcpd3: f3 channel - 90 degree pulse for decoupling sequence
;cnst4: Ha-13Ca J coupling
;cnst21: CO chemical shift (offset, in ppm)
;cnst22: Calpha chemical shift (offset, in ppm)
;$Id: c_can_mq.2,v 1.1.4.1 2004/11/23 15:08:14 ber Exp $
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