Supplement of Magn. Reson., 2, 447–464, 2021
https://doi.org/10.5194/mr-2-447-2021-supplement


*Supplement of*

# Improved NMR transfer of magnetization from protons to half-integer spin quadrupolar nuclei at moderate and high magic-angle spinning frequencies

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# Supporting Information

**Table S1. Selected R$N_n^\nu$ $|m|$ = 2 SQ hetero-nuclear dipolar recoupling for $\nu_R$ = 20 kHz.**

| $\mathcal{R}$ | R$N_n^\nu$ | ϕ° | $\nu_1/\nu_R$ | $\kappa$ | $\kappa/\left\|\kappa_{\{1,2\}}^{DD_1\times DD_2}\right\|_2$ | $\kappa/\left\|\kappa_{\{1,2\}}^{CSA\times CSA}\right\|_2$ | $\kappa/\left\|\kappa_{\{1,2\}}^{\delta iso\times \delta iso}\right\|_2$ |
|---|---|---|---|---|---|---|---|
| $180_0$ | R$22_2^7$ | 57 | 5.5 | 0.178 | 162 | 7.12 | 17.58 |
| | R$28_3^5$ | 51 | 4.67 | 0.176 | 156 | 5.08 | 18.29 |
| | R$18_2^5$ | 50 | 4.5 | 0.175 | 140 | 7.20 | 18.49 |

**Table S2. Selected R$N_n^\nu$ $|m|$ = 2 SQ hetero-nuclear dipolar recoupling with 45° ≤ ϕ ≤ 135° for $\nu_R$ = 62.5 kHz.**

| $\mathcal{R}$ | R$N_n^\nu$ | ϕ° | $\nu_1/\nu_R$ | $\kappa$ | $\kappa/\left\|\kappa_{\{1,2\}}^{DD_1\times DD_2}\right\|_2$ | $\kappa/\left\|\kappa_{\{1,2\}}^{CSA\times CSA}\right\|_2$ | $\kappa/\left\|\kappa_{\{1,2\}}^{\delta iso\times \delta iso}\right\|_2$ |
|---|---|---|---|---|---|---|---|
| $90_0 240_{90} 90_0$ | R$10_4^3$ | 54 | 2.92 | 0.227 | 39.63 | 2.82 | 12.63 |
| | R$14_6^5$ | 64.3 | 2.72 | 0.232 | 36.33 | 1.87 | 12.39 |
| | R$12_5^4$ | 60 | 2.80 | 0.230 | 36.08 | 2.25 | 12.47 |
| | R$12_7^8$ | 120 | 2.00 | 0.227 | 35.96 | 1.61 | 7.72 |
| $270_0 90_{180}$ | R$16_7^6$ | 67.5 | 2.28 | 0.150 | 17.96 | 1.85 | $3.50\times10^{10}$ |
| | R$16_7^{10}$ | 112.5 | 2.28 | 0.150 | 17.96 | 1.85 | $3.50\times10^{10}$ |
| | R$14_6^5$ | 64.3 | 2.33 | 0.150 | 15.90 | 2.33 | $3.58\times10^{10}$ |
| | R$14_6^9$ | 115.7 | 2.33 | 0.150 | 15.90 | 2.15 | $3.58\times10^{10}$ |
| $90_{-45} 90_{45} 90_{-45}$ | R$10_4^3$ | 54 | 1.88 | 0.186 | 16.70 | 2.97 | 15.07 |
| | R$18_7^5$ | 50 | 1.93 | 0.189 | 15.73 | 1.98 | 25.49 |
| | R$14_6^5$ | 64.3 | 1.75 | 0.177 | 15.55 | 2.09 | 5.49 |
| | R$12_5^4$ | 60 | 1.80 | 0.181 | 15.17 | 2.47 | 8.11 |
| $180_0$ | R$14_6^5$ | 64.3 | 1.16 | 0.085 | 5.35 | 2.26 | 1.34 |
| | R$14_6^9$ | 115.7 | 1.16 | 0.085 | 5.35 | 2.26 | 1.34 |
| | R$16_7^6$ | 67.5 | 1.14 | 0.082 | 4.90 | 1.98 | 1.09 |
| | R$16_7^{10}$ | 112.5 | 1.14 | 0.082 | 4.90 | 1.98 | 1.09 |

**Table S3. Selected R$N_n^\nu$ $|m|$ = 2 SQ hetero-nuclear dipolar recoupling built from single π pulses with 20° ≤ ϕ ≤ 160° and κ ≥ 0.15 for $\nu_R$ = 62.5 kHz.**

| $\mathcal{R}$ | $\mathrm{R}N_n^{\nu}$ | ϕ /° | $\nu_1/\nu_R$ | $\kappa$ | $\kappa/\left\Vert\kappa_{\{1,2\}}^{DD_1\times DD_2}\right\Vert_2$ | $\kappa/\left\Vert\kappa_{\{1,2\}}^{CSA\times CSA}\right\Vert_2$ | $\kappa/\left\Vert\kappa_{\{1,2\}}^{\delta iso\times\delta iso}\right\Vert_2$ |
|---|---|---|---|---|---|---|---|
| $180_0$ | $\mathrm{R}28_5^4$ | 25.7 | 2.75 | 0.163 | 24.42 | 3.34 | 26.42 |
| | $\mathrm{R}22_4^3$ | 24.5 | 2.75 | 0.162 | 22.84 | 4.10 | 27.24 |
| | $\mathrm{R}16_3^2$ | 22.5 | 2.67 | 0.161 | 16.26 | 5.21 | 28.89 |

**Table S4. Selected $\mathrm{R}N_n^{\nu}$ $|m| = 2$ two-spin order hetero-nuclear dipolar recoupling.**

| $\mathcal{R}$ | $\mathrm{R}N_n^{\nu}$ | ϕ° | $\nu_1/\nu_R$ | $\kappa$ | $\kappa/\left\Vert\kappa_{\{1,2\}}^{DD_1\times DD_2}\right\Vert_2$ | $\kappa/\left\Vert\kappa_{\{1,2\}}^{CSA\times CSA}\right\Vert_2$ | $\kappa/\left\Vert\kappa_{\{1,2\}}^{\delta iso\times\delta iso}\right\Vert_2$ |
|---|---|---|---|---|---|---|---|
| $90_0240_{90}90_0$ | $\mathrm{R}16_4^9$ | 101 | 4.66 | 0.131 | 63.17 | 16.48 | 9.31 |
| | $\mathrm{R}20_5^{11}$ | 99 | 4.66 | 0.131 | 60.68 | 16.59 | 14.45 |
| | $\mathrm{R}12_3^7$ | 105 | 4.66 | 0.131 | 51.25 | 16.11 | 9.70 |
| | $\mathrm{R}16_4^7$ | 79 | 4.66 | 0.131 | 45.52 | 15.76 | 13.60 |
| | $\mathrm{R}28_7^{10}$ | 64 | 4.66 | 0.131 | 44.55 | 14.06 | 11.98 |
| | $\mathrm{R}20_5^9$ | 81 | 4.66 | 0.131 | 44.30 | 15.95 | 14.46 |
| | $\mathrm{R}12_3^5$ | 75 | 4.66 | 0.131 | 43.91 | 15.40 | 12.83 |
| | $\mathrm{SR}4_1^2$ | 90 | 4.66 | 0.131 | 42.37 | 22.65 | 10.48 |
| $90_{-45}90_{45}90_{-45}$ | $\mathrm{R}28_7^{11}$ | 71 | 3 | 0.191 | 39.81 | 10.05 | 6.10 |
| | $\mathrm{R}20_5^8$ | 72 | 3 | 0.191 | 39.74 | 10.26 | 5.49 |
| | $\mathrm{R}8_2^3$ | 67.5 | 3 | 0.191 | 39.43 | 9.42 | 7.88 |
| | $\mathrm{R}8_2^{11}$ | 67.5 | 3 | 0.191 | 39.43 | 9.42 | 7.88 |
| | $\mathrm{R}24_6^{10}$ | 75 | 3 | 0.191 | 39.32 | 10.66 | 4.22 |
| | $\mathrm{R}28_7^{10}$ | 64.3 | 3 | 0.191 | 38.82 | 8.65 | 10.13 |
| | $\mathrm{R}12_3^5$ | 75 | 3 | 0.191 | 38.33 | 10.66 | 4.22 |
| | $\mathrm{SR}4_1^2$ | 90 | 3 | 0.191 | 19.95 | 19.48 | 1.33 |
| $270_0 90_{180}$ | $\mathrm{R}24_6^{11}$ | 82.5 | 4 | 0.212 | 33.12 | 25.46 | $8.67\times10^{10}$ |
| | $\mathrm{R}20_5^9$ | 81 | 4 | 0.212 | 31.85 | 25.19 | $8.67\times10^{10}$ |
| | $\mathrm{R}20_5^{11}$ | 99 | 4 | 0.212 | 31.85 | 25.19 | $8.67\times10^{10}$ |
| | $\mathrm{R}16_4^7$ | 78.8 | 4 | 0.212 | 28.56 | 24.69 | $8.67\times10^{10}$ |
| | $\mathrm{R}16_4^9$ | 101.2 | 4 | 0.212 | 28.56 | 24.69 | $8.67\times10^{10}$ |
| | $\mathrm{R}12_3^5$ | 75 | 4 | 0.212 | 20.84 | 23.58 | $8.67\times10^{10}$ |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | $R12_{3}^{7}$ | 105 | 4 | 0.212 | 20.84 | 23.58 | $8.67\times10^{10}$ |
| | $SR4_{1}^{2}$ | 90 | 4 | 0.212 | 35.21 | 149.93 | $8.67\times10^{10}$ |
| | $R16_{4}^{7}$ | 78.8 | 2 | 0.25 | 19.65 | 10.52 | 2.78 |
| | $R16_{4}^{9}$ | 115.7 | 2 | 0.25 | 19.65 | 10.52 | 2.78 |
| $180_0$ | $R12_{3}^{5}$ | 75 | 2 | 0.25 | 18.9 | 9.89 | 3.74 |
| | $R12_{3}^{7}$ | 105 | 2 | 0.25 | 18.9 | 9.89 | 3.74 |
| | $SR4_{1}^{2}$ | 90 | 2 | 0.25 | 13.2 | 22.98 | 1.56 |

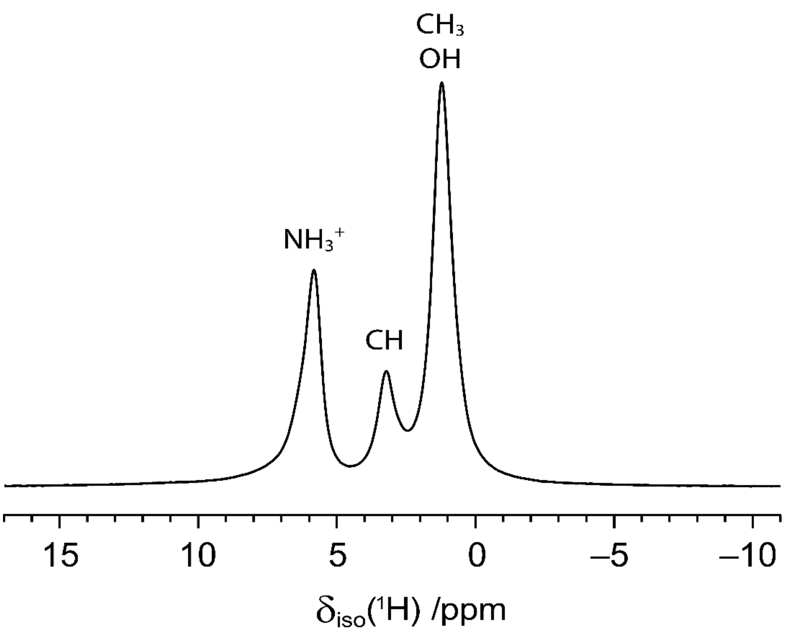


**Figure S1: $^{1}$H MAS spectrum of AlPO$_4$-14 acquired at $B_0$ = 18.8 T and $\nu_R$ = 20 kHz by averaging 16 transients separated by a recycle interval of 1 s, using the DEPTH pulse sequence for probe background suppression, with $\nu_1 \approx$ 208 kHz (Cory and Ritchey, 1988).**

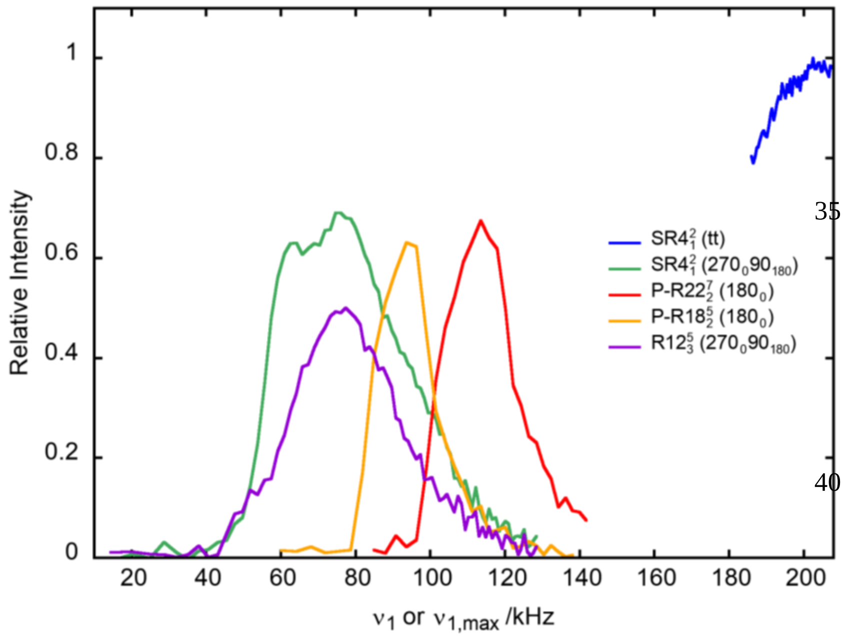


**Figure S2: $^{27}AlO_4$ signal of $AlPO_4$-14 at $\nu_R$ = 20 kHz as function of $\nu_1$ or $\nu_{1,max}$ of the recoupling for PRESTO-R$22_2^7$($180_0$) and -R$18_2^5$($180_0$) as well as RINEPT-CWc-SR$4_1^2$ (tt), -SR$4_1^2$ ($270_0 90_{180}$) and -R$12_3^5$ ($270_0 90_{180}$). For each curve, $\tau$ was fixed to its optimum value given in Table 2.**

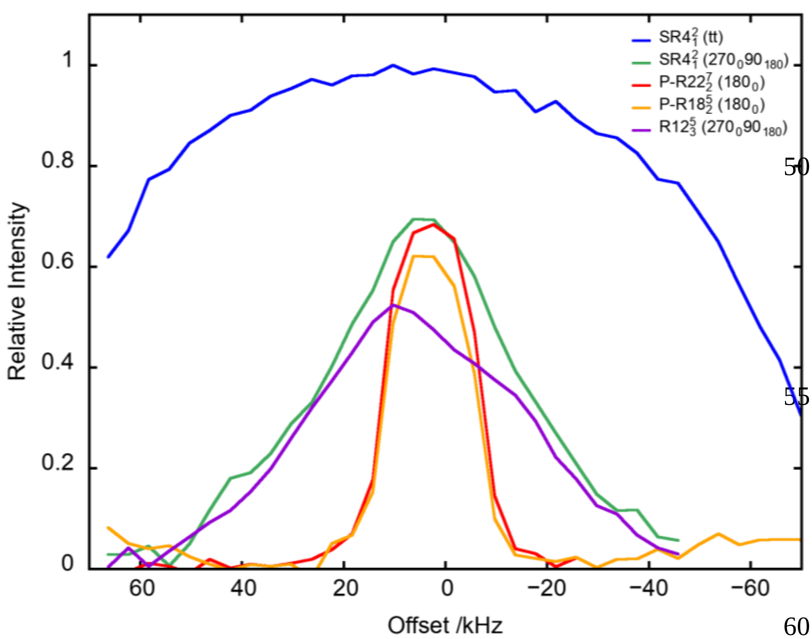


**Figure S3: $^{27}AlO_4$ signal of $AlPO_4$-14 at $\nu_R$ = 20 kHz as function of offset of the recoupling for PRESTO-R$22_2^7$($180_0$) and -R$18_2^5$($180_0$) as well as RINEPT-CWc-SR$4_1^2$ (tt), -SR$4_1^2$ ($270_0 90_{180}$) and -R$12_3^5$ ($270_0 90_{180}$). For each curve, $\tau$ and $\nu_1$ or $\nu_{1,max}$ were fixed to their optimum values given in Table 2.**

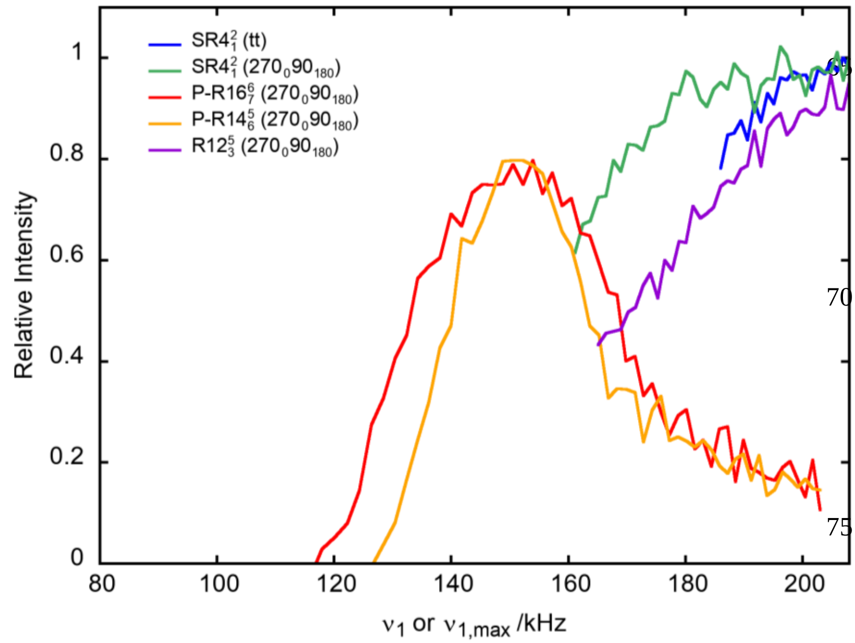


**Figure S4:** **[27]AlO$_4$ signal of AlPO$_4$-14 at $\nu_R$ = 62.5 kHz as function of $\nu_1$ or $\nu_{1,max}$ of the recoupling for PRESTO-R16$_7^6$(270$_0$90$_{180}$) and -R14$_6^5$(270$_0$90$_{180}$) as well as RINEPT-CWc-*S*R4$_1^2$ (tt), -*S*R4$_1^2$ (270$_0$90$_{180}$) and -R12$_3^5$ (270$_0$90$_{180}$). For each curve, $\tau$ was fixed to its optimum value given in Table 4.**

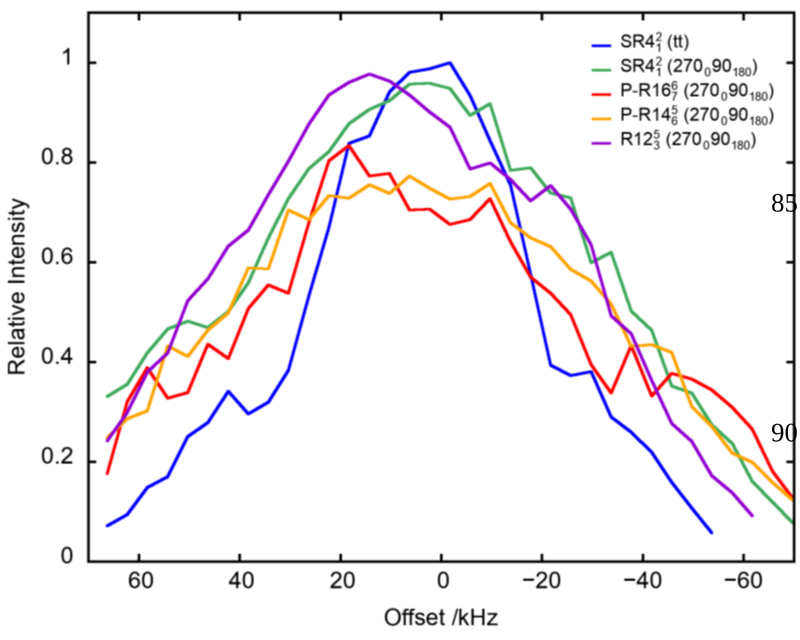


**Figure S5:** **[27]AlO$_4$ signal of AlPO$_4$-14 at $\nu_R$ = 62.5 kHz as function of offset of the recoupling for PRESTO-R16$_7^6$(270$_0$90$_{180}$) and -R14$_6^5$(270$_0$90$_{180}$) as well as RINEPT-CWc-*S*R4$_1^2$ (tt), -*S*R4$_1^2$ (270$_0$90$_{180}$) and -R12$_3^5$ (270$_0$90$_{180}$). For each curve, $\tau$ and $\nu_1$ or $\nu_{1,max}$ were fixed to their optimum values given in Table 4.**

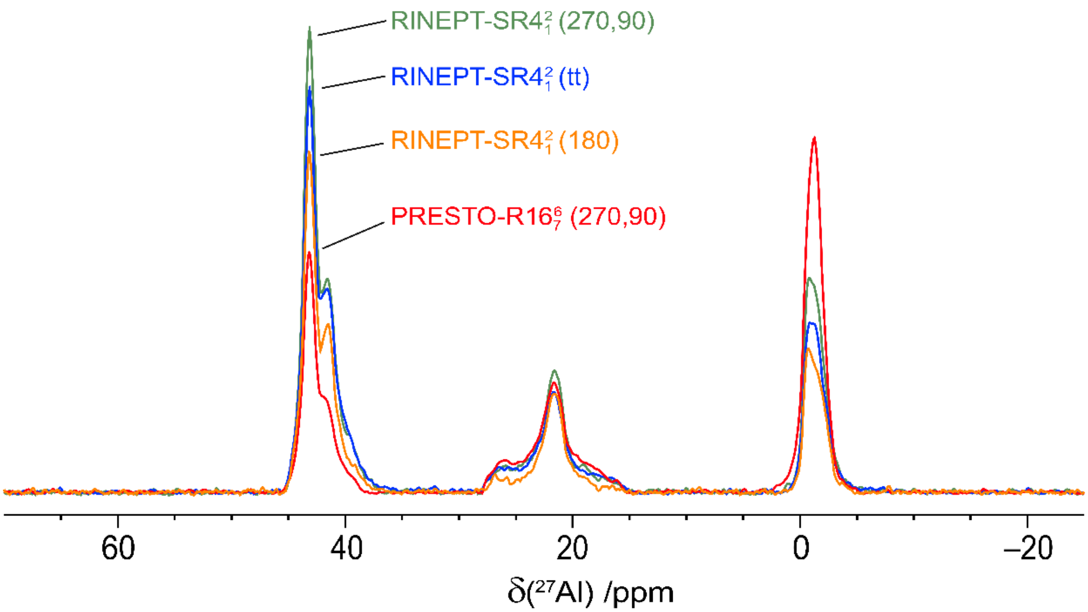


**Figure S6: Skyline projections along $F_2$ of $^1H$-$^{27}Al$ HETCOR 2D spectra of $AlPO_4$-14 recorded with RINEPT-CWc-$SR4_1^2(270_0 90_{180})$, $SR4_1^2$(tt), $SR4_1^2(180_0 90_{180})$ and PRESTO-$R16_7^6$ $(270_0 90_{180})$ transfers. All 2D spectra were acquired using NUS 25% in 72 min.**

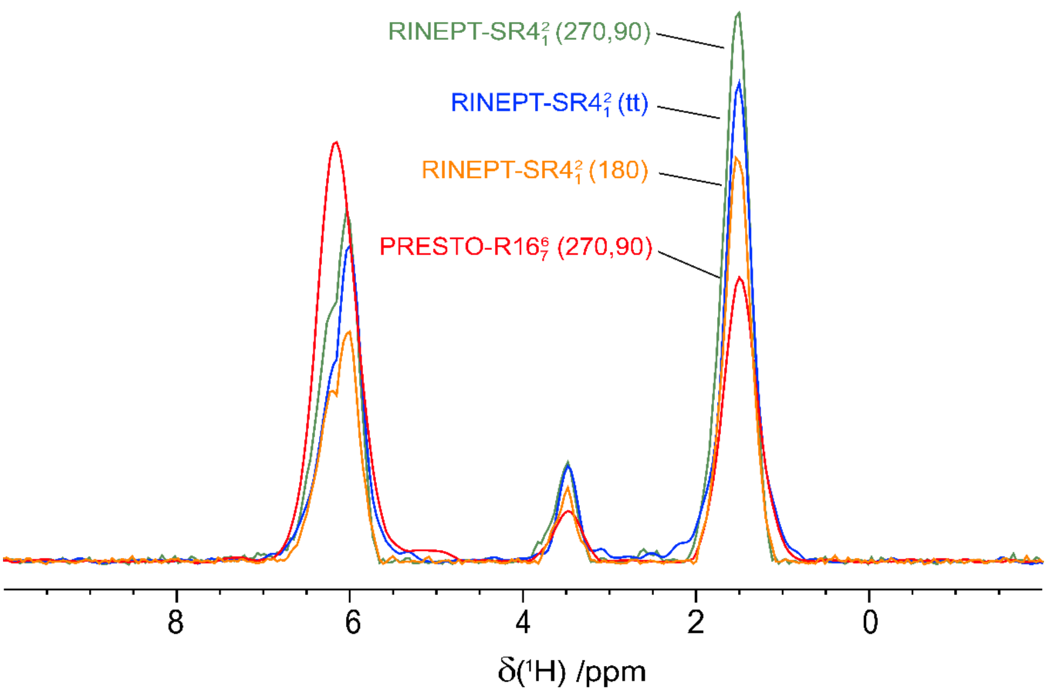


**Figure S7: Skyline projections along $F_1$ of $^1H$-$^{27}Al$ HETCOR 2D spectra of $AlPO_4$-14 recorded with RINEPT-CWc-$SR4_1^2(270_0 90_{180})$, $SR4_1^2$(tt), $SR4_1^2(180_0 90_{180})$ and PRESTO-$R16_7^6$ $(270_0 90_{180})$ transfers. All 2D spectra were acquired using NUS 25% in 72 min.**

**Table S5. Distances between the different hydrogen atoms and their closest Al neighbours in the structure of isopropylamine templated $AlPO_4$-14 determined from X-ray diffraction. (Broach et al., 2003) The H and Al atoms are numbered according to the cif file.**

| H | Al | $r_{HAl}$ /Å |
|---|---|---|
| H1 (OH) | $Al4O_6$ | 2.496 |
| | $Al4O_6$ | 2.499 |
| | $Al1O_5$ | 2.503 |
| | $Al2O_4$ | 4.299 |
| H2 ($NH_3$) | $Al4O_6$ | 3.069 |
| | $Al2O_4$ | 3.779 |
| H3 ($NH_3$) | $Al3O_4$ | 3.778 |
| | $Al4O_6$ | 3.960 |
| H4 ($NH_3$) | $Al2O_4$ | 3.479 |
| | $Al1O_5$ | 3.801 |
| H5 (CH) | $Al2O_4$ | 3.737 |
| | $Al1O_5$ | 4.850 |
| H6 $(CH_3)_1$ | $Al1O_5$ | 3.655 |
| | $Al3O_4$ | 4.594 |
| H7 $(CH_3)_1$ | $Al3O_4$ | 4.082 |
| | $Al1O_5$ | 4.320 |
| H8 $(CH_3)_1$ | $Al2O_4$ | 3.772 |
| | $Al3O_4$ | 4.651 |
| H9 $(CH_3)_2$ | $Al4O_6$ | 3.888 |
| | $Al3O_4$ | 4.124 |
| H10 $(CH_3)_2$ | $Al4O_6$ | 3.509 |
| | $Al3O_4$ | 4.502 |
| H11 $(CH_3)_2$ | $Al4O_6$ | 3.970 |
| | $Al3O_4$ | 4.048 |

Broach, R. W., Wilson, S. T., Kirchner, R. M.: Corrected crystallographic tables and figure for as-synthesized $AlPO_4$-14, Microporous and Mesoporous Materials, 57, 211–214, https://doi.org/10.1016/S1387-1811(02)00563-2, 2003.

Cory, D. G. and Ritchey, W. M.: Suppression of signals from the probe in bloch decay spectra, 80, 128–132, https://doi.org/10.1016/0022-2364(88)90064-9, 1988.

**Pulse sequence for *D*-RINEPT using $SR4_1^2$ ($270_0 90_{180}$) or $R12_3^5$ ($270_0 90_{180}$) recouplings**

```
;INEPT for non-selective polarization transfer
;with decoupling during acquisition
; made of 2 pulses
; different recoupling sequences and composite pulses available
; modified by Julien Trébosc and Jennifer Gómez (2020)
; AVANCE NEO

;d0 initial t1 evolution time (=0)
;d6 probe dead time (should be D6=DE)
;d7 RF offset delay
;d5 Delay after last recoupling for Tr/2
;d8 Delay after last recoupling for Tr/4
;pl1 p1 and p2 power level
;pl12 Heteronuclear dipolar decoupling
;pl19 Presat pulse
;pl2 not used
;pl20 Presat pulse
;pl21 p3 and p4 power level
;pl22 initial spin lock
;pl33 CW23 decoupling
;pl43 CW45 decoupling
;pl44 CW67 decoupling
;pl11 dipolar recoupling power (sr4/sfam)
;spnam5 dipolar recoupling shape pulse
;sp5 power for recoupling shape
;p16 : requested recoupling time
;p17 : actual recoupling time
;l11 sr4/sfam repetition
;cnst30: Tanh/tan offset
;cnst31: spinning speed in Hz
;cnst3: Tanh/tan shape pulse step (ns)
;p1 90 degree pulse for X
;p2 180 degree pulse for X
;p3 90 degree pulse for 1H
;p4 180 degree pulse for 1H
;p6 pulse of the recoupling sequence
;p19 presat pulse for 1H
;p20 presat pulse for X
;p22 initial spin lock for Tr/2
;p23 initial spin lock for Tr/4
;p33 CW45 decoupling for Tr/2
;p34 CW45 decoupling for Tr/4
;p43 CW23 decoupling for Tr/2
;p44 CW67 decoupling for Tr/2
```

```
;p45 CW23 decoupling for Tr/4
;p46 CW67 decoupling for Tr/4

;d1 : relaxation delay; 1-5 * T1
;NS: 16 * n, total number of scans: NS * TD0
;DS: 16
;cpd1: decoupling during R3
;cpdprg1: decoupling during R3
;cpd2: decoupling during AQ and t1
;cpdprg2: decoupling during AQ and t1
;cpd3: decoupling during AQ
;cpdprg3: decoupling during AQ


#include <Avance.incl>

; storeVC option to store VClist used when popting MAS
#ifdef storeVC
#define VCstored vclab, 1u \n lo to vclab times c
#else
#define VCstored
#endif

;-)))))))
#include "presat.incl"
;-)
#ifndef PRESATf2
#undef PRESAT2
#define PRESAT2(f2)
#endif
;-)
#ifndef PRESATf1
#undef PRESAT1
#define PRESAT1(f1)
#endif
;-(((((((
;-----------  DECOUPLING  ----------------
#include "decouple.incl"

#ifdef decF2
#define decF2off do:f2
#define decF2aqon cpds2:f2
#else
#define decF2aqon
#define decF2off
#endif

define delay RF
define delay dummy
```

```
#ifdef _SR4_cp1
;this is SR4 sequence using composite pulse 270(0)-90(180)
#define phaseRN (360) {{{90 270   270 90}*2}^180}^120^240
"p6=0.25s/cnst31"
"p7=p6*3/4.0" ; p270 deg
"p8=p6/4.0"   ; p90 deg
; we have p6 = p7 + p8
;"l11=trunc((p16/p6)/4+0.5)"   ; +0.5 will round to nearest integer
"p17=2*p6*2*l11"
"RF=250e3/p8"
"dummy=RF+p17"
#endif

#ifdef _R1235_cp1
;this is R12_3^5 sequence using composite pulse 270(0)-90(180)
#define phaseRN (360)  75 255   285 105
"p6=0.25s/cnst31"
"p7=p6*3/4.0" ; p270 deg
"p8=p6/4.0"   ; p90 deg
; we have p6 = p7 + p8
;"l11=trunc((p16/p6)/4+0.5)"   ; +0.5 will round to nearest integer
"p17=2*p6*2*l11"
"RF=250e3/p8"
"dummy=RF+p17"
#endif

;............................................
"d24=p3"
"p2=p1*2"
"p4=p3*2"
;"d6=de"
"p22=0.5s/(cnst31)-p3/2.0"
"p23=0.25s/(cnst31)-p3/2.0"
"d5=0.5s/(cnst31)-d6"
"d8=0.25s/(cnst31)-d6"
"p33=0.5s/(cnst31)-p3"
"p34=0.25s/(cnst31)-p3-p4"
"p44=0.5s/(cnst31)-p4/2.0"
"p46=0.25s/(cnst31)-p4/2.0"
"p55=0.5s/(cnst31)-d6"
"p43=0.5s/(cnst31)-p4/2.0-p3"
"p45=0.25s/(cnst31)-p4/2.0"
"d7=0.00000005s"
"plw43=plw33"
"plw44=plw33"

"in0=inf1"

define delay showInAsed
"showInAsed=cnst3+dummy"
```

```
1 ze
VCstored
"showInAsed=cnst3+dummy"

2 30m decF2off
  PRESAT2(f2)
  d1 rpp16 rpp17 rpp14 rpp15 ; not necessary to use different phases and reset but...
  PRESAT1(f1)
  (10u pl21):f2 (10u pl1 ph2):f1
  (p3 ph1):f2


#ifdef _iSL
if "l11 % 2 == 0"
 {
  (p22 pl22 ph27):f2
 }
else
 {
  (p23 pl22 ph27):f2
 }
#endif

  d0

sr4_1, (p7 pl11 ph16^):f2
       (p8 pl11 ph16^):f2
       (p7 pl11 ph16^):f2
       (p8 pl11 ph16^):f2
  lo to sr4_1 times l11

if "l11 % 2 == 0"
 {
  (center (p3 pl21 ph18 p43 pl43 ph21 p4 pl21 ph2 p43 pl43 ph22 p3 pl21 ph18):f2 (p2 ph11):f1 )
 }
else
 {
  (center (p45 pl43 ph18 p4 pl21 ph2 p45 pl43 ph18):f2 (p2 ph11):f1 )
 }

sr4_2, (p7 pl11 ph17^):f2
       (p8 pl11 ph17^):f2
       (p7 pl11 ph17^):f2
       (p8 pl11 ph17^):f2
  lo to sr4_2 times l11

if "l11%2 == 0"
 {
  (center (p3 pl21 ph18 p33 pl33 ph23 p33 pl33 ph24 p3 pl21 ph3):f2 (p1 ph12):f1 )
```

```
 }
else
 {
  (center (p4 pl21 ph5 p3 pl21 ph3 p34 pl33 ph21 p34 pl33 ph22 p4 pl21 ph5 p3 pl21 ph3):f2 (p1 ph12):f1 )
 }

sr4_3, (p7 pl11 ph15^):f2
       (p8 pl11 ph15^):f2
       (p7 pl11 ph15^):f2
       (p8 pl11 ph15^):f2
  lo to sr4_3 times l11

if "l11%2 == 0"
 {
  (center (p44 pl44 ph25 p4 pl21 ph2 p44 pl44 ph26):f2 (p2 ph13):f1 )
 }
else
 {
  (center (p46 pl44 ph25 p4 pl21 ph2 p46 pl44 ph26):f2 (p2 ph13):f1 )
 }

sr4_4, (p7 pl11 ph14^):f2
       (p8 pl11 ph14^):f2
       (p7 pl11 ph14^):f2
       (p8 pl11 ph14^):f2
  lo to sr4_4 times l11

if "l11%2 == 0"
 {
  d5 decF2aqon
 }
else
 {
  d8 decF2aqon
 }
  go=2 ph31
  10u decF2off
  30m mc #0 to 2 F1PH(ip1,id0)

HaltAQ, 1m

exit

ph0=0
ph2=0
ph3=0
ph4=0
ph5= (360) 45
ph6=0
ph7=0
```

```
ph10=0
ph11={{0}*2}^2
ph12={{0}*4}^2
ph13={{0}*8}^2^1^3
ph18=1
ph21=0
ph22=2
ph23=0
ph24=2
ph25=0
ph26=2
ph27=0 2
ph28=0
ph29=3
ph16= phaseRN
ph17= phaseRN
ph15= phaseRN
ph14= phaseRN

#ifdef opt1D
ph1=1 3 0 2
ph31=3 1 2 0
#else
ph1=1 3
ph31={{{1 3}^0}^2}^0^2^2
#endif
presatPH
```

**SIMPSON input file for *D*-RINEPT-CWc-*S*R4$_1^2$(tt)**

```
spinsys {
  channels   1H 13C
  nuclei     1H 13C 1H 1H 1H
# single pair
  shift      1 0 6000 0 0 30 0
  dipole     1 2 -2575 0 0 0
# 2 1H
  shift      3 0 6000 0 0 30 0
  dipole     3 2 0 0 109 0
  dipole     1 3 -7000 0 109 0
# 3 1H
  shift      4 0 6000 0 0 30 0
  dipole     4 2 0 0 109 120
  dipole     1 4 -7000 0 109 120
  dipole     3 4 -7000 0 90 30
# 4 1H
  shift      5 0 6000 0 0 30 0
  dipole     5 2 0 0 109 240
  dipole     1 5 -7000 0 109 240
```

```
  dipole     3 5 -7000 0 90 90
  dipole     4 5 -7000 0 90 330
}

par {
  proton_frequency 400e6
  spin_rate        12500
  sw               spin_rate/2.0
  np               30
  crystal_file     rep66
  gamma_angles     7
  start_operator   I1z
  detect_operator  I2p
  verbose          1101
  variable HRF     100000
  variable DRF     92000
  variable CRF     100000
variable RFmax   spin_rate*11
  variable offmax  2000000
  variable I       1.0/2.0
}

proc gen_tanhtan_shape {pulse_length steps offmax xi K} {
# generate a tanhtan shape with given :
# pulse_length : length of ulse in us
# steps : number of steps defining the shape
# offset : maximum frequency offset of tanhtan sweep
# xi : tanhtan xi parameter
# K : tanhtan kappa parameter
set nhalf [expr $steps/2]
set amp_list [list ]
set phase_list [list ]
for {set i 0} {$i < $steps} {incr i} {
    set x [expr 1.0*$i/(1.0*$steps)]
    if {$i<$nhalf} {
        lappend amp_list [expr tanh(2*$xi*$x)]
      } else {
        lappend amp_list [expr tanh(2*$xi*(1-$x))]
      }
            lappend phase_list [expr -360*$offmax*$pulse_length*(1e-6)*log(abs(cos($K*(1-2*$x))))/(2*tan($K)*$K)]
}
set Tinc [expr 1.0*$pulse_length/$steps]
return [list $amp_list $phase_list $Tinc]
}

proc tanhtan_pulse {shape RF phase } {
# generate simpson pulse following shape argument containing amplitude and phase lists
# shape: as generated by gen_tanhtan_shape procedure
# RF : global maximum RFfield of shape
# phase : global phase of shape
```

```
  set amp_list [lindex $shape 0]
  set phase_list [lindex $shape 1]
  set Tinc [lindex $shape 2]
  foreach amp $amp_list phi $phase_list {
    pulse $Tinc [expr $amp*$RF] [expr $phase+$phi] 0 0
  }
}

proc pulseq {} {
  global par
  maxdt 6.0

  set H90  [expr 0.25e6/$par(HRF)]
  set H180 [expr 0.50e6/$par(HRF)]
  set C90   [expr 0.25e6/$par(CRF)]
  set C180  [expr 0.50e6/$par(CRF)]
  set Taur  [expr 1.0e6/$par(spin_rate)]
   set Td90  [expr 0.5e6/$par(spin_rate)-$H90/2]
   set Td180  [expr 0.5e6/$par(spin_rate)-$H180/2]
  # RN_n^nu parameters
  set N        4.
  set nu    2.
   # set n     1.
    set php  [expr 180*$nu/$N]
    set S90 [expr 0.25e6/$par(RFmax)]
    set S180 [expr 0.50e6/$par(RFmax)]


   set n    100
   set Tp [expr 0.25*$Taur]
   set Tpd [expr 0.25*$Taur]
   # set Q    7.7
   set xi  10.0
   set K    atan(20)
   set pi   [expr atan(1)*4]

   set shape [gen_tanhtan_shape $Tp $n $par(offmax) $xi $K ]

  set ph1 0
  set ph2 120
  set ph3 240

# SR4 using tanhtan inversion
# full block with supercycling
 set superCycling {0 180 120 300 240 60}
 reset
 foreach ph1 $superCycling  {
 reset
 for {set s 0 } {$s<$N/2} {incr s } {
       delay [expr $Tpd/2-$Tp/2]
```

```
   tanhtan_pulse $shape $par(RFmax) [expr $php+$ph1]
      delay [expr $Tpd/2-$Tp/2]
      delay [expr $Tpd/2-$Tp/2]
   tanhtan_pulse  $shape $par(RFmax) [expr -$php+$ph1]
      delay [expr $Tpd/2-$Tp/2]
 }
 store $ph1
 }

 reset [expr -$H90]
 pulse $H90 $par(HRF) 90 0 0
# pulse $Td90 $par(DRF) 0 0 0
 store 19

 reset
 pulse $Td180 $par(DRF) 0 0 0
 pulse $H180 $par(HRF) 0 $par(CRF) 0
 pulse $Td180 $par(DRF) 180 0 0
 store 20

   reset
 pulse $Td90 $par(DRF) 0 0 0
 pulse $H90 $par(HRF) 0 $par(CRF) 0
 pulse $Td90 $par(DRF) 180 0 0
 store 21

   reset
 pulse $Td180 $par(DRF) 0 0 0
 pulse $H180 $par(HRF) 0 $par(CRF) 0
 pulse $Td180 $par(DRF) 180 0 0
 store 22


 reset
 # prop [expr (0%[llength $superCycling])*[lindex $superCycling 1]]
     prop [lindex $superCycling 0]
 store 10

 for {set i 0} {$i < $par(np)} {incr i} {
#reset
 reset [expr -$H90]
  # pulseid $H90 $par(HRF) 90 0 0
 prop 19
 prop 10
 prop 20
 prop 10
 prop 21
 prop 10
 prop 22
 prop 10
```

```
  pulse [expr $Taur/2.0] $par(DRF) 0 0 0
  acq

   reset
   prop 10
#   puts [expr (($i+1)%[llength $superCycling])*[lindex $superCycling 1]]
 #  prop [expr (($i+1)%[llength $superCycling])*[lindex $superCycling 1]]
   prop [lindex $superCycling [expr (($i+1)%6)]]
   store 10

   }
}

proc main {} {
  global par

  set FileRe [open "$par(name)-Re.res" w]
  set FileIm [open "$par(name)-Im.res" w]
  set FileAbs [open "$par(name)-Abs.res" w]

  set f [fsimpson]
  set c 0
  for {set i 1} {$i <= $par(np)} {incr i} {
    incr c
    set Sr [findex $f $c -re]
    set Si [findex $f $c -im]
    puts $FileRe "[expr 1.0e3*$i/$par(sw)] [expr $Sr]"
    puts $FileIm "[expr 1.0e3*$i/$par(sw)] [expr $Si]"
    puts $FileAbs "[expr 1.0e3*$i/$par(sw)] [expr sqrt($Sr**2+$Si**2)]"
   }
  funload $f
  close $FileRe
  close $FileIm
  close $FileAbs
}
```