# Enantioselective Synthesis of Highly Functionalized Octahydro-6-oxo-1-phenylnaphthalene-2-carbaldehydes via Organocatalytic Domino Reactions. 

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## SUPPORTING INFORMATION:

Contents: (1) ORTEP plots for X-ray crystal structures of $\mathbf{5 a}$ and $\mathbf{5 j}$.
(2) Spectra copies for compounds 3a-8.
(3) Ee analysis by HPLC with chiral column, in Table 1-2.


Figure S1. ORTEP plots for X-ray crystal structures of 5a.
CCDC 716016 contains the supplementary crystallographic data for $\mathbf{5 a}$. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif. Crystallographic data for 5a: $\mathrm{C}_{17} \mathrm{H}_{18} \mathrm{O}_{2}, \mathrm{M}=254.31$, monoclinic, space group P 21, $\mathrm{T}=294(2) \mathrm{K}, a=6.1387(7), b=11.0076(13), c=10.7760(13) \AA, \beta=$ $103.653(2)^{\circ}, V=707.58(14) \AA^{3}, Z=2, D=1.194 \mathrm{~g} / \mathrm{cm}^{3}, \lambda\left(\mathrm{Mo}-K_{\alpha}\right)=0.71073 \AA$, 7023 reflections collected, 2596 unique reflections, 173 parameters refined on $F^{2}, R=0.0435, w R 2\left[F^{2}\right]=0.1224$ [2392 data with $F^{2}>2 \sigma\left(F^{2}\right)$ ].


Figure S2. ORTEP plots for X-ray crystal structures of $\mathbf{5 j}$.
CCDC 716017 contains the supplementary crystallographic data for $\mathbf{5 j}$. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif. Crystallographic data for $5 \mathbf{j j}: \mathrm{C}_{18} \mathrm{H}_{19} \mathrm{BrO}_{2}, \mathrm{M}=347.24$, monoclinic, space group P 21, $\mathrm{T}=292(2) \mathrm{K}, a=7.063(3), b=10.733(5), c=10.436(5) \AA, \beta=$ $91.248(8)^{\circ}, V=791.0(6) \AA^{3}, Z=2, D=1.458 \mathrm{~g} / \mathrm{cm}^{3}, \lambda\left(\mathrm{Mo}-K_{\alpha}\right)=0.71073 \AA, 6951$ reflections collected, 3652 unique reflections, 191 parameters refined on $F^{2}, R=0.0415, w R 2\left[F^{2}\right]=0.0976$ [2717 data with $F^{2}>2 \sigma\left(F^{2}\right)$ ].

Fig S3. 1H NMR of 3a (500 MHz, CDCl3).


| RYN-2-110-F1 |  |  | $\underset{\sim}{\infty} \underset{\sim}{\infty} \underset{\sim}{\circ}$ |
| :---: | :---: | :---: | :---: |
| exp 7 | s2pul |  | $\dot{\sim} \dot{\sim}$ |
|  | s2pur |  |  |
|  | SAMPLE | DEC. s VT |  |
| date | Sep 242008 | dfrq | 499.836 |
| solvent | $t$ cdcl3 | dn | H1 |
| file | exp | dpwr | 39 |
| açuisition |  | dof | 0 |
| sfrq | 125.698 | dm | yyy |
| tn | C13 | dmm |  |
| at | 1.000 | dmf | 11905 |
| np | 62894 | dseq |  |
| sw | 31446.5 | dres | 1.0 |
| $f$ b | 17000 | homo |  |
| bs | 16 | PRocessing |  |
| ss | 2 | 1 b | 1.00 |
| tpwr | 54 | wtfile |  |
| pw | 4.0 | proc | $f t$ |
| d1 | 1.000 | fn | not used |
| tof | 2512.2 | math | f |
| nt | 10000 |  |  |
| ct | 10000 | werr | react |
| alock | $y$ | wexp | procplot |
| gain FL | not used | wbs wnt | testsn |
| 11 | n |  |  |
| in | n |  |  |
| dp | $y$ |  |  |
| hs | nn |  |  |
| DISPLAY |  |  |  |
| sp | -1256.9 |  |  |
| wp | 28906.3 |  |  |
| vs | 50 |  |  |
| sc | 0 |  |  |
| we | 210 |  |  |
| hzmm | 137.65 |  |  |
| is | 500.00 |  |  |
| rff | 10981.5 |  |  |
| rfp | 9677.6 |  |  |
| th | 3 |  |  |
| ins | 100.000 |  |  |
| $\mathrm{nm} \quad \mathrm{ph}$ | ph |  |  |



Fig S4. 13C NMR of 3 a ( $500 \mathrm{MHz}, \mathrm{CDCl} 3$ ).

Fig S5. DEPT of 3a (CDCI3).
TYN-2-110-F1
exp9 DEPT


SAMPLE FLAGS date Sep 242008
solvent solvent edclu sspul $\begin{array}{cc}\text { sample undefined hsglvi } \\ \text { ACOUISITION } & \text { SPECIAL } \\ 4498, ~ t e m p ~ n o t ~\end{array}$ sw ACQUISITION $\quad 4498.4$ temp SPECIAL

EXP4 GHMOC
Fig S7. HMQC of 3 a (CDCI3).

date Sep 242008
solvent $\quad$ cdc 13
sample undefined
sspul
$\begin{array}{cc}\text { sample undefined PFGfig } \\ \text { ACQUISITION } & \text { hsglvil }\end{array}$

$\begin{array}{lrlr} & \text { ACQUISITION } & \text { hsglvi } & 1026 \\ \text { sw } & 4498.4 & \text { SPECIAL } & \\ \text { at } & 0.228 & \text { temp } & \text { not } \\ \text { no } & 2048 & \text { gain } & \\ & 28\end{array}$


| sw | 4498.4 | SPECIAL |  |
| :--- | ---: | :--- | ---: |
| at | 0.228 | temp | not |
| $n \mathrm{n}$ |  | 2048 | gain |
| fb | 3000 | spin | 28 |
| ss | 32 |  | GRADIENTS |



| ss | 32 | GRADIENTS |  |
| :--- | ---: | :--- | ---: |
| di | 1.000 | gzlvil | 1026 |
| nt | 16 | gti | 0.001000 |
| 2D ACQUISITION | gzlvi3 | 516 |  |
| swl | 21367.5 | $\mathrm{gt3}$ | 0.001000 |


| ss | 32 | GRADIENTS |  |
| :--- | ---: | :--- | :--- |
| di | 1.000 | gzlvil | 1026 |
| nt | 16 | gt1 | 0.001000 |
| 2d ACQUISITION | gzlvil | 0.00516 |  |
| Swl | 21367.5 | gt3 | 0.001000 |


$\begin{array}{lllr}\text { 2D ACQUISITION } & \text { gzlvi3 } & 516 \\ \text { sw1 } & 21367.5 & \text { gt3 } & 0.001000 \\ \text { ni } & 128 & \text { gstab } & 0.000500 \\ \text { phase } & \text { arrayed } & \text { F2 PROCESSING }\end{array}$
phase arrayed
TRANSMITTER
$\begin{array}{lr}\text { TRANSMITTER } & \text { arrayed } \\ \text { tn } & \text { H1 } \\ \text { sfrq } & 499.836 \\ \text { tof } & 249.8\end{array}$
$\begin{array}{lrlr} & 49.836 & \text { fn } & \text { not } \\ \text { tof } & 2048 \\ \text { tpwr } & 249.8 & \text { F1 } & \text { PROCESSING } \\ & 56 & \text { gf1 } & 0.006\end{array}$
$\begin{array}{lrlr}\text { tpwr } & \text { pw } & \text { 12.006 } & \text { gf1 } \\ \text { pfs } & \text { not used }\end{array}$
pw DECOUPLER ${ }^{12.000}$
$\begin{array}{lr}\text { dn } & \text { CECOUPLER } \\ \text { dof } & -2515.1 \\ \text { dm } & \text { nny }\end{array}$
$d m$
$d m m$
$d m f$
$d p w r$
$p w x$
dpwr
pwxlvi
${ }_{\text {pwx }}^{\text {pw }}$
pwx HMQC
35
51
14.700
$\begin{array}{lr}\text { j1×h } & \text { HMQC } \\ \text { nullf1g } & 140.0 \\ y\end{array}$
$\begin{array}{cc}\text { F2 } & \text { PROCESSING } \\ \text { gfs } & \text { not used }\end{array}$
026
ACQUISITION ARRAYS
$\begin{array}{llr} & \begin{array}{l}\text { ACQUISITION } \\ \text { n } \\ \text { array }\end{array} & \text { phase } \\ y & \text { arraydim } & 256\end{array}$
julfig $\quad 140$
$8 \quad 2$
arraydim
phase
256
phase

```
6
$
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phase
$\frac{1}{2}$
$\frac{1}{2}$

## RYN-2-110-F1

date SAMPLE 2008 hs fags date
solvent solvent cdcl3


498.4 hsglv/ SPECIAL
$\square$
4498.4
0.228 $\begin{array}{ll}.228 & \text { temp not } \\ 2048 & \text { gain } \\ 3000 & \text { npin }\end{array}$ 3000
32 Spin
F2 PROCESSING

$$
\begin{aligned}
& d 1 \\
& \text { nt } \\
& \text { swi } 2 \mathrm{DCQUISIT}
\end{aligned}
$$ .000

$$
\operatorname{tn}_{\text {sfra }}
$$

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\begin{aligned}
& \text { tn } \\
& \text { sfrqq } \\
& \text { tof }
\end{aligned}
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\begin{aligned}
& \text { tot } \\
& \text { tpwr }
\end{aligned}
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\begin{aligned}
& \text { tpwr } \\
& \text { pw } \\
& \text { noesy }
\end{aligned}
$$ nnnn

0
0
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C13
nnn 50 p
2048

$$
\pi \mathrm{PRESATURATION}^{0}
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\begin{aligned}
& \text { PRESAT } \\
& \text { satmode } \\
& \text { satowr }
\end{aligned}
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\begin{aligned}
& \text { satmoaes } \\
& \text { satpwr } \\
& \text { ctat }
\end{aligned}
$$

satdly
$d n$

$d m$ | C 13 |
| :---: |
| $\mathrm{n} \pi$ | 504.2

4494.0
504 504.2
594.0
504.2 400

$$
\text { atfrq } \quad \text { DECOUPLER }
$$ PLOT



| hs |
| :---: |
|  |  |
|  |
| hsglv |
| temp |
| gain |
| sp F 2 |
| gf |
| gfs |
| F1 |
| gf 1 |
|  |  |
|  |
| fn1 |
| sp |
| wp |
| spl |
| wp 1 |
| $r f 1$ |
| rfp ${ }_{\text {rfl }}$ |
|  |  |
|  |
| wc |
| sc |
| Wc2 |
| sc2 |
| vs |
| th |
| ai |

ph

Fig S9. 1H NMR of 4 a ( $500 \mathrm{MHz}, \mathrm{CDCl} 3$ ).





## C13 spectrum of

Fig S12. 13 NMR of 4 a ( $100 \mathrm{MHz}, \mathrm{CDCl} 3,42$ centidegree).


Fig S13. DEPT of $4 \mathrm{a}(100 \mathrm{MHz}, \mathrm{CDCl} 3,42$ centidegree $)$.


Current Data Parameters
NAME
PROCNO

| F2-Acquisition Parameters |  |
| :---: | :---: |
| Date_ | 20090122 |
| Time | 18.14 |
| INSTRUM | spect |
| PROEHD | 5 mm ONP 1H |
| pulprog | zgpg30 |
| TD | 65536 |
| SOLVENT | COC13 |
| NS | 3956 |
| OS | 4 |
| SWH | 25125.629 Hz |
| FIDRES | 0.383387 Hz |
| A 0 | 1.3042164 sec |
| HG | 5792.6 |
| OW | 19.900 usec |
| DE | 6.50 usec |
| TE | 300.0 K |
| D1 | 2.00000000 sec |
| d11 | 0.03000000 sec |
| d12 | 0.00002000 sec |
| ======= | CHANNEL $\mathrm{f} 1 \mathrm{=}=$ |
| NUC1 | 13C |
| P1 | 10.80 usec |
| PL1 | 0.00 dB |
| SFO1 | 100.6237959 MHz |

============ CHANNEL $f 2$ ============

| CPOPAG2 | Walt 216 |
| :--- | ---: |
| NUC2 | 1 H |
| PCPD2 | 90.00 use |
| PL2 | -3.00 dB |
| PL12 | 15.70 dB |
| PL13 | 18.70 dB |
| SF02 | 400.1326008 MHz |

F2 - Processing parameters
SI 32768

| SF | 100.6127646 MHz |
| :--- | :---: |
| WDW | EM |
| SSB | 0 |
| LB | 0.30 Hz |
| GB | 0 |
| PC | 1.40 |

10 NMA plot parameters

| CX | 20.00 cm |
| :--- | ---: |
| F1P | 220.000 ppm |
| F1 | 22134.81 Hz |
| F2P | -10.000 ppm |
| F2 | -1006.13 Hz |
| PPMCM | $11.50000 \mathrm{ppm} / \mathrm{cm}$ |
| HZCM | $1157.04700 \mathrm{~Hz} / \mathrm{cm}$ |

exp25 gHMQC

$\operatorname{exp10} \mathrm{gCOSY}$

| SAMPLE | flags |  |
| :---: | :---: | :---: |
| date Jan 162009 | hs | nn |
| solvent caclu | sspul | n |
| sample undefined | hsglvl | 1026 |
| ACQUISITION |  | SPECIAL |
| sw 4498.4 | temp | not used |
| at 0.228 | gain | 30 |
| np 2048 | spin | 0 |
| fb 3000 | F2 PR | PROCESSING |
| ss 16 | sb | -0.114 |
| d1 1.000 | sbs | not used |
| nt 16 | f | 2048 |
| 2 ACQUISITION | F1 PR | PROCESSING |
| Swl 4498.4 | sb1 | -0.028 |
| TRANSMITTER | sbs 1 | not used |
|  | procl |  |
| $t \mathrm{n}$ Hl | fn1 | 2048 |
| sfrq 499.836 |  | OISPLAY |
| tof 249.8 | sp | 495.7 |
| tpwr 57 | wp | 4494.0 |
| pw 13.000 | spl | 496.7 |
| GRADIENTS | wp 1 | 4494.0 |
| gzlvl1 1026 | rfi | 1386.6 |
| gt1 0.001000 | rfp | 1877.9 |
| gstab 0.000500 | rfil | 1385.5 |
| DECOUPLER | rfpi | 1877.9 |
| dn C13 |  | PLOT |
| dm nnn | wc | 155.0 |
|  | sc | 10.0 |
|  | wc2 | 155.0 |
|  | Sc2 | 0 |
|  | vs | 1814 |
|  | th | 9 |
|  | ai cde | dc av |



Fig S16. NOESY of 4a (CDCl3).


Fig S17. 1H NMR of 5 a ( $500 \mathrm{MHz}, \mathrm{CDCl} 3$ ).

## RYN-2-115


 6
6
695
613
00
0
0
$n n$
$c$
200
1.0
$n$





Fig S18. 13C NMR of 5 a ( $125 \mathrm{MHz}, \mathrm{CDCl} 3$ ).

Fig S19. DEPT of 5a (CDCI3).
RYN-2-115
exp23 DEPT


RYN-2-115
exp24 gCos $Y$



Fig S21. HMQC of 5a (CDCl3).
RYN-2-115
exp25 gHMQC



RYN-2-115
expzo noesy
date SAMPLE $\begin{aligned} & \text { Oct } 162008 \text { hs FLAGS }\end{aligned}$
solvent 162008
sample


$$
\begin{array}{lr}
2 \mathrm{D} & \text { ACQUISITION } \\
\mathrm{SWI} & 4298.5 \\
\mathrm{ni} & 200
\end{array}
$$

ni TRANSMITTER
tn TRANSMITTER
$\begin{array}{lr}\text { sfrq } & 499.836 \\ \text { tof } & 349.8 \\ \text { pwr } & 57\end{array}$

ix NOESY
$\stackrel{0}{\text { PRESATURATION }} \underset{ }{0.4010}$ satmode
satpwr
satdiy
DECOUPLER
$d n$
$d m$
${ }^{0.400}$
nnnn
0
0
0
$C 13$
$n n n$
nnn
ph


$$
\begin{aligned}
& \begin{array}{lr}
\text { gf1 } & 0.043 \\
\text { gfs } 1 & \text { not } u s e d
\end{array} \\
& \text { dISplay } \\
& \text { pp DISPLAY } \\
& \text { Plot } \\
& \text { PLOT } \\
& \begin{array}{ll}
\text { Sc } & \\
\text { wc2 } & \\
\text { sc2 } & \\
\text { vs } & \\
\text { th } & \\
\text { ai } & \text { ph }
\end{array}
\end{aligned}
$$

Fig S23. 1H NMR of 5 b ( $500 \mathrm{MHz}, \mathrm{CDCl} 3$ ).







Fig S24. 13C NMR of 5b (125 MHz, CDCl3).

RYN-2-122
exp34 DEPT


RYN-2-122
exp31 gCos
date SAMPLE 0 Ot 222008 hs FLAG date
solvent $\quad \begin{array}{lll}22008 & \text { hs } \\ \text { cdcla } & \text { sspul }\end{array}$ solvent cdclu sspul
sample undefined hsglvi Samp
sw 4498.4 temp not
$\begin{array}{lrlr}\text { sw } & 4498.4 & \text { temp } & \text { not us } \\ \mathrm{at} & 0.228 & \text { gain } & \\ \mathrm{np} & 2048 & \text { spin } \\ \mathrm{fb} & 3000 & \mathrm{~F} 2 & \text { Processing } \\ \text { ss } & 16 & \mathrm{sb} & -0.1\end{array}$
$\begin{array}{lr}\text { ss } & 3000 \\ \text { d1 } & 16 \\ \mathrm{nt} & 1.000 \\ 20 & 16 \\ \text { 2D ACQUISITION }\end{array}$

$\mathrm{sw1}$
ni
4498
20
 $\begin{array}{rr}\text { frq } & 499.836 \\ \text { tof } & 249.8 \\ \text { tpwr } & 57\end{array}$ towr
GRADIENTS
$\begin{array}{lr}\text { gzlvil } & 1026 \\ \text { t1 } & 0.001000\end{array}$
gstab 0.0000000
dn DECOUPLER C13
C13
nnn

$d n$
$d m$


Fig S27. HMQC of 5b (CDCl3).
exp32 gHMQC

$\begin{array}{lrl}\text { ss } & 32 & \text { GRADIENTS } \\ \text { d1 } & 1.000 & \text { gzlvil }\end{array}$
1026
$\begin{array}{llr}\mathrm{nt} \\ 2 \mathrm{D} \text { ACQUISITION } & { }^{16} & \mathrm{gt1} \\ \mathrm{gzlv} \\ \mathrm{gzl3} & 0.001000 \\ 516\end{array}$
$\begin{array}{llll}\mathrm{swl} & 28912.2 & \mathrm{gt3} & 0.001000\end{array}$ $\begin{array}{lrl}\text { ni } & 128 & \text { gstab } 0.00050 \\ \text { phase } & \text { arrayed } & \text { F2 PROCESSING }\end{array}$
tn TRANSMITTER

$\begin{array}{lr}\text { tn } & \text { H1 } \\ \text { sfrq } & 499.836 \\ \text { tof } & 249.8\end{array}$ | gf |  |
| :--- | ---: |
| gfs | not used |
| fn | 0.105 |

$\begin{array}{lrrr}\text { tof } & 249.8 & \text { F1 PROCESSING }\end{array}$

DECOUPLER
dof
$d m$
$d m m$
$d m \mathrm{mf}$
dpw
dpwr
pwxlvi
pwx
j1xh HMQC
jullfig

proc1 display

$\xrightarrow{\longrightarrow}(\mathrm{ppm})$ sp
wp
sp1
$w p 1$
rff
rfp
rf7
rfp
$w e$


RYN-2-122
 sample undefined $\begin{gathered}\text { cocla } \\ \text { PFGflo }\end{gathered}$

ACQUISITION hsglv
sw ACQUISITION 4498.
sw
$a t$
$n p$
fb
ss
$d i$
np
fb
$s s$
$d 1$
$n t$ $\begin{array}{lrr} & 3000 & \text { spin } \\ \text { d1 } & 32 & \mathrm{~F} 2 \\ \mathrm{nt} & 1.000 & \mathrm{gf}\end{array}$ 2 2DACQUSITION $^{8} \underset{\mathrm{gfs}}{\mathrm{gn}}$

$$
\begin{array}{llll} 
& 0.041 \\
\text { TRANSMITTER } & \mathrm{gfi} & \text { not } \mathrm{gfs} 1
\end{array}
$$

$$
\begin{array}{lrl}
\text { sfrq } & 499.836 & f n 1 \\
\text { tof } & 249.8 & \\
\text { tnwer } & 57 & \mathrm{cn}
\end{array}
$$

$$
\begin{array}{lrrr}
\text { tof } & 249.8 & & \text { DISPLAY } \\
\text { tpwr } & 57 & \text { sp } & \\
\text { pw } & 13.000 & \text { wp } & 4
\end{array}
$$

pw noesy ${ }^{1}$
$\underset{\text { PRESATURATION }}{0} \stackrel{0}{0}$
satmode
satpwr
satdly
satfrq
DECOUPLER
$d n$
$d m$
nnnn
0
0
0
C13
nnn
ph

Fig S29. 1H NMR of 5c (500 MHz, CDCl3).

| RYN-2-127 |  |  |  |
| :---: | :---: | :---: | :---: |
| exp22 s2pul m. |  |  |  |
| SAMPLE DEC. \& VT |  |  |  |
| date | Oct 302008 | dfrq | 125.695 |
| solvent | $t \mathrm{cdcl3}$ | dn | C13 |
| file | exp | dpwr | 30 |
| ACQUISITION |  | dof | 0 |
| sfrq | 499.836 | dm | nnn |
| tn | H1 | dmm | c |
| at | 3.000 | dimf | 200 |
| np | 48000 | dseq |  |
| sw | 8000.0 | dres | 1.0 |
| $f \mathrm{~b}$ | 4000 | homo | n |
| bs | 4 |  | SSING |
| tpwr | 57 | wtfi |  |
| pw | 4.8 | proc | $f t$ |
| d1 | 1.000 | fn | not used |
| tof | 499.7 | math | f |
| nt | 4 |  |  |
| ct | 4 | wer r | react |
|  | y | wexp | procplot |
| alock <br> gain <br> F | not used FLAGS | wbs wnt | wft |
| i] | $n$ |  |  |
| in | n |  |  |
| dp | y |  |  |
| hs | nn |  |  |
|  | IISPLAY |  |  |
| $s p$ | -250.1 |  |  |
| wp | 5498.0 |  |  |
| vs | 85 |  |  |
| sc | 0 |  |  |
| wc | 210 |  |  |
| hzmm | 26.18 |  |  |
| is | 147.35 |  |  |
| rfi | 4632.6 |  |  |
| rfp | 3618.8 |  |  |
| th | 4 |  |  |
| ins | 100.000 |  |  |
| nm ph | ph |  |  |





Fig S30. 13C NMR of 5c (125 MHz, CDCl3).

Fig S31. DEPT of 5 c (CDCI3).
exp24 DEPT


Fig S32. COSY of 5c (CDCI3).


Fig S33. HMQC of 5 c (CDCI3).

ai cde ph $\begin{array}{ll}\text { solvent cdcla } & \text { sspul } \\ \text { sample undefined } \\ \text { PFGfig }\end{array}$
sw ACQUISITION 4498.4 hsgivl SPECIAL 1026
sw ACQUISITION 4498 .
np
fb
ss
d 1
$n t$
$\begin{array}{lr}\text { d1 } & 1.000 \\ \text { nt } & 16 \\ \text { 20 } & \text { ACQUISITION } \\ \text { swl } & 21367.5\end{array}$
$\begin{array}{lr}\text { Swl } & 21367.5 \\ \mathrm{ni} & 128 \\ \text { phase } & \text { arrayed }\end{array}$ tn TRANSMITTER $\begin{array}{lr}\text { tn } & \text { H1 } \\ \text { sfrq } & 499.836 \\ \text { tof } & 249.8 \\ \text { tpwr } & 57 \\ & 13.000\end{array}$

$$
\begin{array}{lrlr}
\text { sfrq } & 499.836 & \text { fn } & 204 \\
\text { tof } & 249.8 & \text { F1 } & \text { PROCESSING } \\
\text { tpwr } & 53.5 & \text { gf1 } & 0.00 \\
\text { ow } & 13.000 & \text { ofs1 } & \text { not use }
\end{array}
$$

$$
\begin{array}{lrlr}
\text { tpwr } & 57 & \text { gf1 } & 0.006 \\
\text { pw } & \text { 13.000 } & \text { gfs1 } & \text { not used } \\
\text { DECOUPLER } & \text { proc1 } & \text { fos }
\end{array}
$$

$$
\begin{array}{lr}
\text { dn DECOUPLER } \\
\text { dof } & -2515.1
\end{array}
$$

$$
\begin{array}{lr}
\text { dof } & -2515.1 \\
\text { dm } & \text { nny } \\
\text { dmm } & \text { ccp } \\
\text { dmf } & 32258
\end{array}
$$

$$
\begin{array}{lr}
\text { umm } & \text { ccp } \\
\text { dmf } & 32258 \\
\text { dpwr } & 35
\end{array}
$$

$$
p w \times l v 1
$$

$$
\begin{aligned}
& \text { pwx|vi } \\
& \text { pwx }
\end{aligned}
$$

$$
\begin{aligned}
& \text { pwx } \\
& \text { j1xh }
\end{aligned} \quad \text { HMQC } \begin{aligned}
14.700 \\
140.0
\end{aligned}
$$



ACQUISITION ARRAYS
ACQUISITION ARRAYS
array array
a
$\begin{array}{lr}i & \text { phase } \\ 1 & 1\end{array}$


Fig S34. NOESY of 5c (CDCl3).


Fig S35. 1H NMR of 5 d ( $500 \mathrm{MHz}, \mathrm{CDCl} 3$ ).





Fig S36. 13C NMR of 5 d ( $125 \mathrm{MHz}, \mathrm{CDCl} 3$ ).

Fig S37. DEPT of 5d (CDCl3).
exp31 DEPT

exp32 gcosy


exp33 gHMQC
 ai cdc ph

ACQUISITION ARRAYS array phase arraydim

Fig S39. HMQC of 5d (CDCl3).

exp35 NOESY

| SAMPLE | flags |  |
| :---: | :---: | :---: |
| date Nov 32008 | hs | ก |
| solvent cdcl3 | sspul | $y$ |
| sample undefined | PFGflg | $g \quad y$ |
| ACQUISITION | hsglvi | 1026 |
| SW 4498.4 |  | SPECIAL |
| at 0.228 | temp | not used |
| np 2048 | gain | 28 |
| fb 3000 | spin | 0 |
| ss 32 | F2 P | PROCESSING |
| d1 1.000 | gf | 0.105 |
| nt 8 | gfs | not used |
| 2 A ACQUISITION | $f \mathrm{n}$ | 2048 |
| SW1 4498.4 | F1 P | PROCESSING |
| ni 200 | gf1 | 0.041 |
| TRANSMITTER | gfs 1 | not used |
| $t \mathrm{n}$ H1 | proc1 |  |
| sfrq 499.836 | fn1 | 2048 |
| tof 249.8 |  | DISPLAY |
| tpwr 57 | sp | 485.9 |
| pw 13.000 | wp | 4494.0 |
| NOESY | spl | 487.5 |
| mix 0.400 | wpl | 4494.0 |
| PRESATURATION | rfi | 2452.5 |
| satmode nnnn | rfp | 2934.0 |
| satpwr | rfil | 2450.9 |
| satdly | rfp1 | 2934.0 |
| satfra |  | PLOT |
| DECOUPLER | wc | 155.0 |
| dn C13 | Sc | 10.0 |
| dm nnn | wc2 | 155.0 |
|  | Sc2 | 0 |
|  | vs | 28 |
|  | th | ph 3 |



Fig S41. 1H NMR of $5 \mathrm{e}(500 \mathrm{MHz}, \mathrm{CDCl} 3)$.

| RYN-2-130 |  |  |  |
| :---: | :---: | :---: | :---: |
| exp11 s2pul m |  |  |  |
| SAMPLE DEC. \& VT |  |  |  |
| date | Nov 32008 | dfrq | 125.6915 |
| solvent | $t \quad \mathrm{cdcl3}$ | dn | C13 |
| file | UISITION $\exp$ | dpwr | 30 |
|  | UISITION | dof | 0 |
| sfrq | 499.836 | $d m$ | nnn |
| t | H1 | dmm | c |
| at | 3.000 | dmf | 200 |
| np | 48000 | dseq |  |
| SW | 8000.0 | dres | 1.0 |
| $f b$ | 4000 | homo | n |
| bs | 4 |  | SSING |
| tpwr | 57 | wtfil |  |
| pw | 4.8 | proc | $f t$ |
| d1 | 1.000 | fn | not used |
| tof | 499.7 | math | f |
| nt | 4 |  |  |
| ct | 4 | werr | react |
| alock | $y$ | wexp | procplot |
| gain | not used |  | wft |
| $\dagger 1$ | $n$ |  |  |
| in | $n$ |  |  |
| dp | $y$ |  |  |
| DISPLAY |  |  |  |
| sp | -250.1 |  |  |
| wp | 5498.0 |  |  |
| vs | 90 |  |  |
| sc | 0 |  |  |
| wc | 210 |  |  |
| hzmm | 26.18 |  |  |
| is | 119.62 |  |  |
| rfi | 4632.1 |  |  |
| rfp | 3618.8 |  |  |
| th | 4 |  |  |
| ins | 100.000 |  |  |
| nm cdc ph |  |  |  |


$\dot{\sim} \dot{\sim} \sim \dot{\sim} \dot{\sim}$



Fig S42. 13C NMR of 5 e ( $125 \mathrm{MHz}, \mathrm{CDCl} 3$ ).

Fig S43. DEPT of $5 \mathrm{e}(\mathrm{CDCl} 3)$.
expl3 DEPT


Fig S44. COSY of 5 e (CDCl3).
expl4 gCosy
SAMPLE 2008 FL date Nov 32008
solvent hs
sdci3
sspul sample undefined hsgivi
ACQUISITION




Fig S45. HMQC of 5 e (CDCI3).


Fig S46. NOESY of 5e (CDCl3).


FLAGS
date Nov 32008 hs
olvent
sample undefined sspul sw ACQUISITION

$$
\begin{aligned}
& \text { PFGflg } \\
& \text { hsglvi }
\end{aligned}
$$


${ }_{20} 1$
$\begin{array}{lll}\text { SW1 } & 4308.5 & \text { fn } \\ \text { Fi PROCESSING }\end{array}$
TRANSMITTER $\begin{array}{lll}200 & \text { gfi } 1 & 0.043 \\ \text { gfs } 1 & \text { not used }\end{array}$

$\begin{array}{lrl}\text { of } & 344.8 & \text { sp } \\ \text { tpwr } & 535 P L A Y\end{array}$
pw noesy
mix
PRESATURATION
0. atmode ion satmode
satpwr
satfr
$d n$
$d m$

$$
\begin{array}{r}
\mathrm{N} \\
308.5 \\
0.238 \\
2048 \\
2000 \\
32 \\
1.000 \\
10 \mathrm{~B} \\
308.5 \\
200 \\
\mathrm{R} \\
\mathrm{H} 1 \\
9.836 \\
344.8 \\
57 \\
3.000 \\
0.600 \\
0 N \\
n n n n \\
0 \\
0
\end{array}
$$

lvi
not
not temp gain not used 20 2048 gain
2000 spin
32 F2 PROCESSING 32
1.000
gf 2
0.110 0
10
80会 0 DECOUPLER
ph

2048
680.6
4304.3
677.0
4304.3
2247.7
2924.0
2251.2
2924.0
155.0
10.0
155.0
0
113
2
whaturn , H

$$
\begin{aligned}
& \text { C13 } \\
& \text { nnn }
\end{aligned}
$$

Fig S47. 1H NMR of 5 f ( $500 \mathrm{MHz}, \mathrm{CDCl} 3$ ).

| RYN-2-133 |  |  |  |
| :---: | :---: | :---: | :---: |
| exp3 s | s2pul |  | $\stackrel{¢}{0}$ |
|  | SAMPLE | DEC. \& | \& VT |
| date | Jan 62009 | dfrq | 125.695 |
| solvent | nt cdci3 | dn | C13 |
| file ${ }_{\text {ACQUI }}$ | utsition exp | dpwr | 30 |
|  | QUISITION |  | 0 |
| sfrq | 499.836 | dm | nnn |
| tr | H1 | dmm | c |
| at | 3.000 | dmf | 200 |
| np | 48000 | dseq |  |
| sw | 8000.0 | dres | 1.0 |
| fb | 4000 | homo | n |
| bs | 4 | PROCES | SSING |
| tpwr | 57 | wtfile |  |
| pw | 4.8 | proc | $f t$ |
| d1 | 1.000 | f | not used |
| tof | 499.7 | math | $f$ |
| nt | 4 |  |  |
| ct | 4 | wer r | react |
| alock | $y$ | wexp | procplot |
| gain FL | not used flags | wbs wnt | wft |
| il | n |  |  |
| in | n |  |  |
| dp | $y$ |  |  |
| hs | nn |  |  |
| DISPLAY |  |  |  |
| sp | -250.1 |  |  |
| wp | 5498.0 |  |  |
| vs | 34 |  |  |
| sc | 0 |  |  |
| wc | 210 |  |  |
| hzmm | 26.18 |  |  |
| is | 110.59 |  |  |
| rfi | 4631.3 |  |  |
| rfp | 3618.8 |  |  |
| th | 8 |  |  |
| ins | 100.000 |  |  |
| nm ph | ph |  |  |







Fig S48. 13C NMR of 5 f ( $125 \mathrm{MHz}, \mathrm{CDCl} 3$ ).

Fig S49. DEPT of 5 f (CDCI3).
exp32 DEPT


Fig S50. COSY of $5 f(\mathrm{CDCl} 3)$.
exp33 gCos $\gamma$

exp34 gHMQC


Fig S52. NOESY of 5 f (CDCl3).
RYN-2-133
exp35 NOESY



Fig S53. 1H NMR of 5 g ( $500 \mathrm{MHz}, \mathrm{CDCl} 3$ ).

| N-2-138 |  |
| :---: | :---: |
| exp21 s2pul | - |
| SAMPLE | DEC. \& VT |
| date NOV 152008 | dfrq 125.695 |
| solvent cdcl3 | dn C13 |
| file exp | dpwr 30 |
| ACQUISITION | dof 0 |
| sfrq 499.836 | dm nnn |
| tn H1 | dmm |
| at 3.000 | dmf 200 |
| $\mathrm{np} \quad 48000$ | dseq |
| sw 8000.0 | dres 1.0 |
| fb 4000 | homo |
| bs 4 | PROCESSING |
| tpwr 57 | wtfile |
| pw 4.8 | proc ft |
| d1 1.000 | fn not used |
| tof 499.7 | math |
| nt 4 |  |
| ct 4 | werr react |
| alock y | wexp procplot |
| gain flags ${ }^{\text {not used }}$ | whs <br> wnt |
| il n |  |
| in $n$ |  |
| dp y |  |
| hs min |  |
| DISPLAY |  |
| sp $\quad-250.1$ |  |
| wp 5498.0 |  |
| vs 28 |  |
| Sc 0 |  |
| wc 210 |  |
| hzmm 26.18 |  |
| is 119.13 |  |
| rfl 4631.8 |  |
| rfp 3618.8 |  |
| $t \mathrm{th}$ |  |
| ins 100.000 |  |
| nm ph |  |




Fig S54. 13C NMR of 5 g ( $125 \mathrm{MHz}, \mathrm{CDCl} 3$ ).

Fig S55. DEPT of 5 g (CDCI3).
exp23 DEPT


| 27 gcosy |  |  |
| :---: | :---: | :---: |
| SAMPLE | flags |  |
| date Nov 172008 | hs | nn |
| solvent cdcl3 | sspul | n |
| sample undefined | hsgivi | 1026 |
| ACQUISITION |  | SPECIAL |
| sw 4498.4 | temp | not used |
| at 0.228 | gain | 30 |
| np 2048 | spin | 0 |
| fb 3000 | F2 Pr | ROCESSING |
| ss 16 | sb | -0.114 |
| d1 1.000 | sbs | not used |
| nt 16 | fn | 2048 |
| 2 D ACQUISITION | F1 Pr | ROCESSING |
| SW1 4498.4 | sbl | -0.028 |
| ni 128 | sbs 1 | not used |
| TRANSMITTER | proc1 | 1p |
| tn H1 | fni | 2048 |
| sfrq 499.836 |  | ISPLAY |
| tof 249.8 | sp | 486.0 |
| tpwr 57 | wp | 4494.0 |
| pw 13.000 | spl | 489.2 |
| GRADIENTS | wp 1 | 4494.0 |
| gzlv11 1026 | rf1 | 2437.4 |
| gt1 0.001000 | rfp | 2919.0 |
| gstab 0.000500 | rfil | 2434.2 |
| DECOUPLER | rfpl | 2919.0 |
| dn C13 |  | PLOT |
| dm nnn | wc | 155.0 |
|  | sc | 10.0 |
|  | wc2 | 155.0 |
|  | sc2 | 0 |
|  | vs | 18 |
|  | thic th | c av 8 |



Fig S57. HMQC of 5 g (CDCl3).
exp26 gHMQC


Fig S58. NOESY of 5 g (CDCI3).
exp24 NOESY

| SAMPLE | flags |  |
| :---: | :---: | :---: |
| date Nov 152008 | hs | n |
| solvent cdcl3 | sspul | y |
| sample undefined | PFGfig | $g$ y |
| ACQUISITION | hsglv? | 1026 |
| SW 4498.4 |  | SPECIAL |
| at 0.228 | temp | not used |
| np 2048 | gain | 50 |
| fb 3000 | spin | 0 |
| ss 32 | F2 P | PROCESSING |
| di 1.000 | gf | 0.105 |
| nt 8 | gfs | not used |
| 2D ACQUISITION | fn | 2048 |
| SW1 4498.4 | F1 P | PROCESSING |
| ni 200 | gf1 | 0.041 |
| TRANSMITTER | gfs 1 | not used |
| $t \begin{aligned} & \text { tr }\end{aligned}$ | proc1 | $1 p$ |
| sfrg 499.836 | fn1 | 2048 |
| tof 249.8 |  | dISPLAY |
| tpwr 57 | sp | 489.7 |
| pw 13.000 | wp | 4494.0 |
| Noesy | spl | 504.2 |
| mix 0.200 | wp1 | 4494.0 |
| PRESATURATION | rff | 2433.7 |
| satmode nnnn | rfp | 2919.0 |
| satpwr | rfil | -499.8 |
| satdly 0 | rfpl | 0 |
| satfrq 0 |  | PLOT |
| DECOUPLER | wc | 155.0 |
| dn C13 | sc | 10.0 |
| dm nnn | wc2 | 155.0 |
|  | sc2 | 0 |
|  | vs | 1814 |
|  | th | ph 4 |
|  | ai | ph |



Fig S59. 1H NMR of 5 h ( $500 \mathrm{MHz}, \mathrm{CDCl} 3$ ).



Fig S60. 13C NMR of 5 h ( $125 \mathrm{MHz}, \mathrm{CDCl} 3$ ).


Fig S62. COSY of 5h (CDCI3).

## RYN-2-140

exp14 gcosy
 $\begin{array}{lrr}\text { solvent } & \text { cdclis } & \text { sspul } \\ \text { sample } & \text { undefined } & \text { hsglvi } \\ & 1026\end{array}$
 $\begin{array}{lrlr}\text { sw } & 4498.4 & \text { temp } & \text { not used } \\ \text { at } & 0.228 & \text { gain } & 22 \\ \text { np } & 2048 & \text { spin } & 0 \\ \text { fb } & 3000 & \text { F2 } & \text { PROCESSING } \\ \text { ss } & 16 & \text { sb } & -0.114\end{array}$ $\begin{array}{lrlr} & 16 & \text { Sb } & -0.114 \\ \text { d1 } & 1.000 & \text { sbs } & \text { not used } \\ \text { it } & 16 & \text { fn } & 2048\end{array}$ ${ }_{\text {2D }}$ D ACQUISITION ${ }_{4}{ }^{16}$ F1 PROCESSING $\begin{array}{lrlr}\text { SW1 } & 4498.4 & \text { sb1 } & \text { not } \\ \text { ni } & 128 & \text { sbs1 } & \text { not } \\ \text { TRANSMITTER }\end{array}$

tn
sfra
tof tof

pw | 249.8 | sp | 482 |
| ---: | ---: | ---: |
| 57 | wp | 4494. |
| GRADIENTS | 1300 | sp1 | $\begin{array}{llr} & & 485.3 \\ \text { gzlvil } & \text { spidIENTS } & \text { wp1 }\end{array}$ $\begin{array}{llll}\text { gt1 } & 0.001000 & \text { rfp } & 2595 . \\ & 3074 .\end{array}$

 $\begin{array}{lll}\text { nnn } & \text { WC } & 155.0 \\ & \text { SC } & 10.0 \\ & \text { WC2 } & 155.0 \\ & \text { SC2 } & \end{array}$



Fig S63. HMQC of 5h (CDCl3).


Fig S64. NOESY of 5h (CDCl3).
RYN-2-140
expl6 NOES
date SAMPLE NOV 152008 hs FLAGS solvent cdci3 $\begin{array}{ll}\text { hs } \\ \text { spul }\end{array}$ $\begin{array}{lrll}\text { sample } & \text { undefined } & \text { pFGfig } & \\ \text { ACQUISITION } & \text { hsglvl } & \\ \text { sw } & 4498.4 & \text { temp SPECIAL } & \\ \text { at } & 0.228 & \text { not } \\ \text { np } & 2048 & \text { gain } & \end{array}$
$\begin{array}{lrlr}\text { at } & 2048 & \text { gain } & \\ \text { np } & 3000 & \text { spin } & \\ \text { fb } & 32 & \text { F2 } & \text { PROCESSING } \\ \text { ss } & 1.000 & \text { gf } & 0 .\end{array}$
$\begin{array}{ll}\text { di } \\ \text { nt } \\ 20 & 1 . \\ \end{array}$


$\begin{array}{lllll}\text { tn } & & \text { H1 } & \text { gfs1 } & \text { proci }\end{array} \quad$ not us
$\begin{array}{lrll}\text { sfrq } & 499.836 & \text { fni } & \\ \text { tof } & 249.8 & \text { DISPLAY } \\ \text { tpwr } & 57 & \text { sp } & \end{array}$
tpwr
pw NOESY
mix NOESY
satmode
satpwr
satdly
satfrq DECOUPLER
dn
dm



Fig S65. 1H NMR of 5 i ( $500 \mathrm{MHz}, \mathrm{CDCl} 3$ ).




Fig S66. 13C NMR of 5 i ( $125 \mathrm{MHz}, \mathrm{CDCl} 3$ ).

Fig S67. DEPT of 5i (CDCl3).
expl3 DEPT


Fig S68. COSY of 5i (CDCl3).

## $\operatorname{expl} 14$ gCos $Y$


$\begin{array}{lll}\text { date Nov } 172008 & \text { hs } \\ \text { solvent } & \text { cdcl3 } & \text { sspul }\end{array}$
$\begin{array}{lrr}\text { solvent } & \text { cdcis } & \text { sspul } \\ \text { sample } & \text { undefined } & \text { hsglvil } \\ \text { ACQUISITION } & 1026\end{array}$

| sw | and |
| :--- | :--- |
| at | 4998. |

$\begin{array}{lrrr}\text { at } & 0.205 & \text { gain } & \text { not used } \\ \text { np } & 2048 & \text { spin } & 30 \\ \text { fb } & 3000 & \text { F2 } & \text { PROCESSING } \\ \text { ss } & 16 & \text { sb } & -0.102\end{array}$
$\begin{array}{lrl}\mathrm{fb} & 3000 & \text { F2 PROCESSING } \\ \mathrm{ss} & 16 & \mathrm{sb} \\ \mathrm{d} & -0.102\end{array}$
$\begin{array}{ll}d 1 & 1.000 \\ \text { nt } \\ 20 \\ \text { 20 ACQUISITION }\end{array}$

| SW1 |  |
| :--- | ---: |
| ni | 4998. |
| 12 |  |

$\begin{array}{lr}\operatorname{tn} & H 1 \\ \text { sfra } & 499.836 \\ \text { tof } & -0.1 \\ \text { tpwr } & 57\end{array}$
tpwr
pw GRADIENTS
$\begin{array}{ll}27 v 11 & 0.001026 \\ & 0.0050\end{array}$ gt1
gstab Stab O.0
DECOUPLER C13 C 13
nnn
 $\begin{array}{cc} & \text { not used } \\ \text { fn } & 2048 \\ \text { F1 } & \text { PROCESSING } \\ \text { sb1 } & -0.026 \\ \text { bsi } & \text { not used }\end{array}$ dm

DISPLAY


PLOT
 i cdc av

都


RYN-2-141
expl5 gHMQC

flags
ags
$\begin{array}{lrl}\text { ds } & 32 & \text { GRADIENTS } \\ \text { d1 } & 1.000 & \text { gzlvil }\end{array}$
 $\begin{array}{lrlr}\text { ni } & 128 & \text { gt3 } & 0.001000 \\ \text { phase } & \text { arrayed } & \text { F2 } & 0.000500\end{array}$


Fig S69. HMQC of 5 i (CDCI3).


Fig S70. NOESY of 5 i (CDCl3).
RYN-2-141
exple noesy
SAMPLE



Fig S71. 1H NMR of 5j ( $500 \mathrm{MHz}, \mathrm{CDCl} 3$ ).

| RYN-2-145 |  |  |  |
| :---: | :---: | :---: | :---: |
| exp21 s2pul mm |  |  |  |
|  | SAMPLE 2008 | $\mathrm{fra}^{\text {D }}$ | \& VT |
| date | Nov 282008 | dfrq | 125.695 |
| solvent | nt cdcl3 | dn | C13 |
| file | exp | dpwr | 30 |
| ACQUISITION |  | dof | 0 |
| sfrq | 499.836 | dm | nnn |
| tnat | H1 | dmm | c |
|  | 3.000 | dmf | 200 |
| np | 48000 | dseq |  |
| sw | 8000.0 | dres | 1.0 |
| $f b$ | 4000 | homo | n |
| bs | 4 | P | SSING |
| tpwr | 57 | wtfil |  |
| pw | 4.8 | proc | $f t$ |
| d1 | 1.000 | fn | not used |
| tof | 499.7 | math | $f$ |
| nt | 4 |  |  |
| ct | 4 | werr | react |
| alock <br> gain <br> F | y | wexp | procplot |
|  | not used FLAGS | wbs wnt | wft |
| i1 | n |  |  |
| in | $n$ |  |  |
| dp | $y$ |  |  |
| hs | nn |  |  |
| display |  |  |  |
| sp | -250.1 |  |  |
| wp | 5498.0 |  |  |
| vs | 77 |  |  |
| sc | 0 |  |  |
| wc | 210 |  |  |
| hzmm | 26.18 |  |  |
| isrif | 131.36 |  |  |
|  | 4626.2 |  |  |
| $r f p$ | 3618.8 |  |  |
| th | . 7 |  |  |
| ins | 100.000 |  |  |
| $\mathrm{nm} \quad \mathrm{ph}$ | ph |  |  |








$084.52 \square$
$100 \cdot 1 \varepsilon$


Fig S72. 13C NMR of 5 j ( $125 \mathrm{MHz}, \mathrm{CDCl} 3$ ).

Fig S73. DEPT of 5 j (CDCl3).
exp22 DEPT



Fig S75. HMQC of 5 j (CDCI3).


ACQUISITION ARRAYS array $\begin{array}{r}\text { arraydim } \\ \end{array} \quad 256$ i phase phase
1
2


Fig S76. NOESY of 5 j (CDCl3).


Fig S77. 1H NMR of compound 6 ( $500 \mathrm{MHz}, \mathrm{CDCl} 3$ ).

RYN-2-148
exp24 s2pul





Fig S78. 13C NMR of compound 6 ( $125 \mathrm{MHz}, \mathrm{CDCl} 3$ ).


RYN-2-148
Fig S80. COSY of compound 6 (CDCI3).
exp27 gCOSY
SAMPLE FLAGS
date
solvent cdci3 sspul nn sample undefined hsglvi 1026 sample undefine ACQUISITION
$\begin{array}{lll}\text { SW } & \text { at } & \text { SPECIAL } \\ \text { at } & \text { not use }\end{array}$ $\begin{array}{lll}\text { at } & 0.138 & \text { gain } \\ \text { np } & 1024 & \text { spin } \\ \text { fb } & 2000 & \text { F2 PROCESSING } \\ \text { ss } & 10 & \text { sb }\end{array}$ $\begin{array}{llll}\text { ss } & 1.0 & \text { sb } & -0.069 \\ \text { d } 1 & 1.000 & \text { sbs } & \text { not used }\end{array}$ $\mathrm{nt}_{20}$ ACQUISITION ${ }^{16}$ SW1 $\quad 3698.7$ SbI PROCESSING ni

s
t
t $\begin{array}{llll}\text { tof } & -350.0 & \mathrm{Sp} & \text { DISLAY } \\ \text { tpwr } & 301.1\end{array}$ $\begin{array}{lll}\text { towr } & 57 \mathrm{wp} & 3691.5 \Longrightarrow\end{array}$
GRADIENTS 13.000 $g z l v 11$
$g t 1$ 026 $\begin{array}{ll}\text { gtt } & 0.00100 \\ \text { gstab } & 0.00050\end{array}$ DECOUPLER C13 nnn $\begin{gathered}\text { wc } \\ \\ \\ \\ \\ w c\end{gathered}$

PLOT
$\begin{array}{lll}\text { wc } & \\ \text { sc } & \\ \text { wc2 } & \\ \text { sc2 } & \\ v s & \\ \text { th } & \\ \text { ai cde av }\end{array}$

Fig S81. HMQC of compound 6 (CDCl3).
exp28 gHMQC


RYN-2-148
exp29 NOESY
SAMPLE
 $\begin{array}{lll}\text { solvent } & \text { cdclu } & \text { sspul } \\ \text { sample } & \text { undefined } & \text { PFGflg }\end{array}$ $\begin{array}{ccr}\text { sample } & \text { undefined } & \text { PFGflg } \\ \text { SW ACQUISITION } & \text { hsglvil } & \text { y } \\ 3698.7 & \text { SPECIAL } & \end{array}$
sw
at
n $\begin{array}{lll}\text { np } & 0.138 & \text { temp not used }\end{array}$
$\begin{array}{lrll}\text { fb } & 2000 & \text { gpin } & \\ \text { ss } & 32 & \text { F2 PROCESSING } \\ \text { d } & 1.000 & \text { gf } & \end{array}$
$\begin{array}{lrlr}\text { d1 } & 1.000 & \mathrm{gf} & 0.064 \\ \mathrm{nt} & 8 & \mathrm{gfs} & \text { not used }\end{array}$
20 ACQUISITION ${ }^{8} \mathrm{gn}^{2}$ RROCESSINO 1024
SW1 ACQUISITION $\quad 3698.7 \quad$ F1 PROCESSING
$\begin{array}{lrrr}\text { SW1 } & 3698.7 & \text { F1 } & \text { PROCESSING } \\ \text { ni } & 200 & \text { gfi } & 0.050\end{array}$

$\begin{array}{lrll}\text { sfrq } & 499.835 & \text { fn1 } & \\ \text { tof } & -350.0 & & \text { display } \\ \text { tpwr } & 57 & \text { sp } & \\ \text { pw } & 13.000 & \text { wp } & \end{array}$
pW NOESY
mix
PRESATURATION
13.000
0.200
satmode
satpwr nnnn
satpwr
satdly
satfrq
dn DECOUPLER
dn
dm
.200
$n n n n$
$\begin{array}{ll} \\ & \\ & \mathrm{C13} \\ \mathrm{nnn}\end{array}$


Fig S83. 1H NMR of compound 7 ( $500 \mathrm{MHz}, \mathrm{CDCl} 3$ ).

RYN-2-153
$\begin{aligned} & \text { exp2 } \text { s2pul } \\ & \text { SAMPLE }\end{aligned}$


Dec 312008
DEC. \& VT $\begin{array}{lrrr}\text { date } & \text { Dec } 31 \begin{array}{rlr}2008 & d f \\ \text { solvent } & \text { cdcl3 } & d n \\ \text { file } & & \text { exp }\end{array} \quad d p\end{array}$ ACQUISITION exp dpwr sfrq
tn
tn
at
np
$\begin{array}{lrlr}\text { np } & \mathbf{4 8 0 0 0} & \text { dseq } & \\ \text { sw } & 8000.0 & \text { dres } & 1.0 \\ \text { fb } & 4000 & \text { homo } & \text { PROCESSING } \\ \text { bs } & 4 & \\ \text { tpwr } & 57 & \text { wtfile } & \\ \text { pw } & 4.8 & \text { proc } & \text { ft }\end{array}$

$\begin{array}{lr}\text { tof } & 499.7 \\ n t & 4 \\ c t & 4\end{array}$
alock
gain
il
in
in
dp
$h s$
hs Display nn
sp DISPLAY
$\begin{array}{lr}\text { sp } & -250.1 \\ w p & 4998.3 \\ \text { vs } & 48 \\ \text { sc } & 0 .\end{array}$
wC
hzmem
is
rf
rfp
rfp
th
ins
ins
$n \mathrm{~m}$
$\mathrm{cdc} \mathrm{ph}{ }^{1}$
react
cplot
flags not used wbs $\begin{aligned} & \text { wht } \\ & \text { wht }\end{aligned}$ 210
23.80 67.22 4630.6 3618.8
 695
013
30
0
$n n$
$c$

RYN-2-153
exp3 s2pul




Fig S84. 13C NMR of compound 7 ( $125 \mathrm{MHz}, \mathrm{CDCl} 3$ ).

Fig S85. DEPT of compound 7 (CDCl3).
$\begin{array}{ll}\text { exp4 } & \text { DEPT } \\ & \text { SAMPLE }\end{array}$

$\begin{array}{ll}\text { bs } & \\ \text { ss } & \\ \text { d } 1 & 1 \\ n t & \\ c t & \end{array}$
TRANSMITTER 1800
tn TRANGMITER
tof C13 1p 77.
$\begin{array}{lrl}\text { tof } & 2512.2 & \text { ai } \\ \text { tode } & \text { cde ph } \\ \text { REFERENCE }\end{array}$

dn
n OECOUPLER
dof
dpwr
dpwr
dm
$d m m$
$\underset{d m m}{d m}$
pplv7
pp

$\begin{array}{cc}\mathrm{ccW} & \mathrm{hzmm} \\ 11905 & \text { th }\end{array}$
vs
hzmm
th
11905
49
29.400


Fig S86. COSY of compound 7 (CDCl3).
RYN-2-153
$\exp 5 \mathrm{gCOS} Y$

| SAMPLE |  | flags |  |
| :---: | :---: | :---: | :---: |
| date | c 312008 | hs | nn |
| solvent | cdcl3 | sspul |  |
| sample | undefined | hsgiv | 1026 |
| ACQUISITION |  | SPECIAL |  |
| Sw | 3998.6 | temp | not used |
| at | 0.128 | gain | 34 |
| $n \mathrm{p}$ | 1024 | spin | 0 |
| fb | 2000 | F2 | PROCESSING |
| ss | 16 | sb | -0.064 |
| d1 | 1.000 | sbs | not used |
| $n \mathrm{t}$ | 32 | fn | 1024 |
| 2D AC | ISITION | $F 1$ | PROCESSING |
| sw1 | 3998.6 | sbl | -0.032 |
| ni | 128 | sbs 1 | not used |
| TRANSMITTER |  | proc1 |  |
| tn | ${ }^{\mathrm{H} 1}$ | fnl | 1024 |
| sfrq | 493.835 |  | DISPLAY |
| tof | -499.9 | sp | -5.6 |
| tpwr | 57 | wp | 3990.8 |
| pw | 13.000 | sp1 | 7.9 |
| GRADIENTS |  | wp1 | 3990.8 |
| gzlvl1 | 1026 | rfi | 2348.7 |
| gt1 | 0.001000 | rfp | 2335.2 |
| gstab | 0.000500 | rfil | -0.1 |
| DECOUPLER |  | rfp1 | 0 |
| $\begin{aligned} & \mathrm{dn} \\ & \mathrm{dm} \end{aligned}$ | C13 |  | PLOT |
|  | nnn | wc | 155.0 |
|  |  | sc | 10.0 |
|  |  | wc2 | 155.0 |
|  |  | Sc2 | 0 |
|  |  | vs | 57 |
|  |  | th | 8 |



RYN-2-153
Fig S87. HMQC of compound 7 (CDCl3).
EXp6 gHMQC


Fig S88. NOESY of compound 7 (CDCl3).
RYN-2-153
exp16 NOESY



Fig S89. 1H NMR of compound 8 (CDCl3).
RYN-2-166

|  |  |  |  |
| :---: | :---: | :---: | :---: |
| exp2 | s2pul |  | ¢゙心 |
|  | SAMPLE | DEC. \& VT |  |
| date | Feb 262009 | dfrq | 125.695 |
| solvent | t cdcl3 | dn | C13 |
| file | exp | dpwr | 30 |
| ACQUISITION |  | dof | 0 |
| sfrq | 499.836 | dm | nnh |
| tn | H1 | dmm | - |
| at | 3.000 | dmf | 200 |
| np | 48000 | dseq |  |
| sw | 8000.0 | dres | 1.0 |
| fb | not used | homo |  |
| bs | 4 |  | SSING |
| tpwr | 57 | wtfi |  |
| pw | 4.8 | proc | $f t$ |
| d1 | 1.000 | fn | not used |
| tof | 499.7 | math |  |
| nt | 4 |  |  |
| ct | 4 | wer r | react |
| alock |  | wexp | procplot |
| gain | not used <br> FLAGS | wbs wnt | wft |
| il | n |  |  |
| in | n |  |  |
| dp | y |  |  |
| hs | DISPLAY |  |  |
|  |  |  |  |
| sp | -250.1 |  |  |
| wp | 5498.0 |  |  |
| vs | 19 |  |  |
| sc | 0 |  |  |
| wc | 210 |  |  |
| hzmm | 26.18 |  |  |
| is | 193.87 |  |  |
| rff | 4630.6 |  |  |
| rfp | 3618.8 |  |  |
| th | 3 |  |  |
| ins | 100.000 |  |  |
| nm cdc | c ph |  |  |






Fig S91. DEPT of compound 8 (CDCl3).
RYN-2-166


Fig S92. DEPT of compound 8 (CDCI3), Expanded.
RYN-2-166
expl3 DEPT


Fig S93. HMQC of compound 8 (CDCI3).
RYN-2-166
exp14 gHMQC


Fig S94. HMQC of compound 8 (CDCl3), expanded.
RYN-2-166
exp14 gHMQC
date $\quad \begin{gathered}\text { SAMPLE } \\ \text { Feb } 26 \\ 2009\end{gathered}$ hs FLAG date
solvent
sample solvent
sample cdcl3 hs spul ACQUISITION
$\begin{array}{rr}\text { ACQUISITION } & \text { ARRAYS } \\ \text { array } \\ \text { arraydim } & \text { phase } \\ \text { i } & 256 \\ 1 & \text { phase } \\ 2 & 1 \\ 2 & 2\end{array}$ i $\begin{array}{ccc}4490.3 & \text { SpeCIAL } \\ 0.228 & \text { temp } & \text { not used }\end{array}$ 2048 gain
not used spin 32 gRADIENT
$\begin{array}{lrlr}\text { d1 } & 1.000 & \text { gzlvil } & \text { GRADIENTS } \\ \text { nt } & 1006 \\ \text { 2D ACQUISITION } & \text { gt1 } & \text { gzlvis } & 0.001000 \\ & 506\end{array}$
 $\begin{array}{lllr}\text { swl } & 21367.5 & \text { gts } & 0.001000 \\ \text { ni } & 128 & \text { gstab } & 0.000500 \\ \text { phase } & \text { arrayed } & \text { f2 } & \end{array}$ $\begin{array}{cc}\text { phase arrayed } \\ \text { TRANSMITTER F2 } \\ \mathrm{gf} & \text { FROCESSING } \\ 0.105\end{array}$
 $\begin{array}{llll}\text { tof } & 249.8 & \text { F1 PROCESSING } \\ \text { tow } & 57 & \mathrm{gf1} & 0.006\end{array}$ $\begin{array}{lrlr}\text { tpw } & 57 & \text { gf1 } & 0.006\end{array}$

DECOUPLER

cdc ph


Fig S95. COSY of compound 8 (CDCl3).
RYN-2-166



Fig S96. NOESY of compound 8 (CDCl3).
RYN-2-166
exp15 noesy
date SAMPLE FLAGS



Fig S97．HPLC analysis of the racemic 5a（Table 1，entry 19）．
（For comparison）
ryn－2－117 racemate col IA 20\％EA／Hex

Report produced on 2008／10／18 at 下午 02：21：29 by Put your name here


2008／10／18 aUaĖ 12：01：06 Flow set to 1.00 at 0.01 minutes
2008／10／18 aUaĖ 12：49：52 Run stopped by operator

PEAK REPORT

| \＃ | begin | end | area | percent | maximum | time | begins as | name |
| :--- | ---: | :---: | ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 19.46 | 20.90 | 601 | 49.2 | 69.47 | 19.92 | Baseline |  |
| 2 | 21.69 | 23.36 | 620 | 50.8 | 70.80 | 22.28 | Baseline |  |



Chromatogram Report
First line of organization＇s address Second line of organization＇s address ryn－2－121－col IA 20\％EA／Hex

Report produced on 2008／11／16 at 上午 12：22：48 by Put your name here


2008／10／18 aUaÈ 03：45：29 Flow set to 1.00 at 0.00 minutes
2008／10／18 aUםĖ 04：23：23 Run stopped by operator

## PEAK REPORT

| \＃ | begin | end | area | percent | maximum | time | begins as name |
| ---: | ---: | ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 20.29 | 21.80 | 2429 | 100.0 | 104.17 | 20.66 | Baseline |

Fig S99．HPLC analysis of 5a obtained（Table 1，entry 10 and Table 2，entry 1）．


Chromatogram Report
First line of organization＇s address Second line of organization＇s address ryn－2－115 chiral col IA 20\％EA／Hex

Report produced on 2008／10／18 at 下午 02：24：56 by Put your name here


2008／10／18 aUaÈ 12：52：19 Flow set to 1.00 at 0.00 minutes
2008／10／18 aUםÈ 01：39：23 Run stopped by operator

PEAK REPORT

| \＃ | begin | end | area | percent | maximum | time | begins as name |
| ---: | ---: | ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 20.98 | 22.87 | 2565 | 100.0 | 123.54 | 21.43 | Baseline |

Fig S100．HPLC analysis of the mixture of racemic and chiral 5a obtained

## Chromatogram Report

ryn－2－115－co－col IA 20\％EA／Hex
Report produced on 2008／10／18 at 下午 02：26：07 by Put your name here


2008／10／18 aUaÈ 01：42：02 Flow set to 1.00 at 0.01 minutes
2008／10／18 aUםĖ 02：18：07 Run stopped by operator

PEAK REPORT

| \＃ | begin | end | area | percent | maximum | time | begins as name |  |
| :--- | ---: | :---: | ---: | ---: | ---: | ---: | ---: | :--- |
| 1 | 18.72 | 19.84 | 215 | 9.5 | 66.08 | 19.15 | Baseline |  |
| 2 | 20.64 | 22.49 | 2045 | 90.5 | 112.50 | 21.11 | Baseline |  |



## Chromatogram Report

ryn－2－119－col IA 20\％EA／Hex

Report produced on 2008／11／16 at 上午 12：21：30 by Put your name here


2008／10／18 aUaÉ 03：07：12 Flow set to 1.00 at 0.00 minutes
2008／10／18 aUםĖ 03：42：57 Run stopped by operator

PEAK REPORT

| \＃ | begin | end | area | percent | maximum | time | begins as name |
| :--- | ---: | ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 20.29 | 21.72 | 1975 | 100.0 | 94.23 | 20.67 | Baseline |

Fig S102．HPLC analysis of the racemic 5a（Table 1，entry 18）．

## Chromatogram Report

ryn－2－117 racemate 3 col－IA 22\％EAI Hex blank
Report produced on 2008／11／16 at 上午 12：00：38 by Put your name here


2008／11／15 aUaÈ 02：37：55 Flow set to 1.00 at 0.00 minutes
2008／11／15 aUםÈ 03：03：12 Run stopped by operator

PEAK REPORT

| \＃ | begin | end | area | percent | maximum | time | begins as name |
| :--- | ---: | ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 12.80 | 13.73 | 718 | 48.7 | 75.66 | 13.16 | Baseline |
| 2 | 13.93 | 14.90 | 756 | 51.3 | 75.87 | 14.31 | Baseline |



Chromatogram Report
First line of organization＇s address Second line of organization＇s address ryn－2－125－D chiral col－IA 22\％EA／Hex blank

Report produced on 2008／11／16 at 上午 12：02：16 by Put your name here


2008／11／15 aUaÈ 03：05：19 Flow set to 1.00 at 0.03 minutes
2008／11／15 םUםÈ 03：29：12 Run stopped by operator

PEAK REPORT

| \＃ | begin | end | area | percent | maximum | time | begins as | name |
| :--- | ---: | :---: | ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 13.22 | 14.39 | 1534 | 77.0 | 112.60 | 13.57 | Baseline |  |
| 2 | 14.55 | 15.37 | 458 | 23.0 | 64.82 | 14.83 | Baseline |  |

Fig S104．HPLC analysis of the mixture of racemic and chiral 5a obtained（Table 1，entry 18）．

Report produced on 2008／11／16 at 上午 12：04：15 by Put your name here


2008／11／15 aUaÈ 03：31：13 Flow set to 1.00 at 0.00 minutes
2008／11／15 aUםÈ 03：54：13 Run stopped by operator

PEAK REPORT

| \＃ | begin | end | area | percent | maximum | time | begins as | name |
| :--- | ---: | ---: | ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 13.57 | 14.67 | 1123 | 68.2 | 92.99 | 13.93 | Baseline |  |
| 2 | 14.86 | 15.78 | 523 | 31.8 | 67.52 | 15.21 | Baseline |  |

Fig S105．HPLC analysis of the racemic 5b（Table 2，entry 2）．


## Chromatogram Report

ryn－126 racemate IA 20\％ea／hex 1ml／min
Report produced on 2009／1／22 at 下午 04：22：36 by Put your name here


2009／1／22 aUxÈ 01：37：08 Flow set to 1.00 at 0.00 minutes
2009／1／22 aUaÈ 02：53：53 Run stopped by operator

PEAK REPORT

| $\#$ | begin | end | area | percent | maximum | time | begins as name |  |
| :--- | ---: | :---: | ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 49.38 | 52.89 | 6875 | 49.2 | 151.90 | 50.55 | Baseline |  |
| 2 | 56.96 | 60.42 | 7098 | 50.8 | 145.08 | 58.19 | Baseline |  |



## Chromatogram Report

ryn－122 chiral IA 20\％ea／hex 1ml／min
Report produced on 2009／1／22 at 下午 04：24：18 by Put your name here


2009／1／22 aUxÈ 02：56：22 Flow set to 1.00 at 0.01 minutes
2009／1／22 aUaÈ 04：19：49 Run stopped by operator

## PEAK REPORT

| \＃ | begin | end | area | percent | maximum | time | begins as |
| ---: | ---: | ---: | ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 59.98 | 64.03 | 10203 | 100.0 | 158.08 | 61.28 | Baseline |

Fig S107．HPLC analysis of the mixture of racemic and chiral 5b obtained（Table 2，entry 2） （For comparison）


## Chromatogram Report

ryn－2－122＋ryn－2－126 co inj col－IA 20\％EAI Hex
Report produced on 2008／11／16 at 上午 12：14：56 by Put your name here


2008／11／15 aUaÈ 07：17：54 Flow set to 1.00 at 0.01 minutes
2008／11／15 םUםĖ 08：57：04 Run stopped by operator

PEAK REPORT

| \＃ | begin | end | area | percent | maximum | time | begins as name |
| :--- | ---: | ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 51.02 | 53.42 | 3863 | 15.5 | 102.75 | 51.99 | Baseline |
| 2 | 58.11 | 61.95 | 21082 | 84.5 | 257.49 | 59.19 | Baseline |

Fig S108．HPLC analysis of the racemic 5c（Table 2，entry 3）．
ryn－2－128 racemate col－IA 20\％EA／Hex

Report produced on 2008／11／16 at 上午 12：16：03 by Put your name here


2008／11／15 aUaÈ 08：59：15 Flow set to 1.00 at 0.00 minutes


PEAK REPORT

| \＃ | begin | end | area | percent | maximum | time | begins as name |  |
| :--- | ---: | ---: | ---: | ---: | ---: | ---: | ---: | :--- |
| 1 | 25.46 | 26.87 | 2266 | 48.2 | 93.31 | 25.92 | Baseline |  |
| 2 | 27.04 | 28.46 | 2436 | 51.8 | 101.15 | 27.46 | Baseline |  |



## Chromatogram Report

## ryn－2－127 chiral col－IA 20\％EAI Hex

Report produced on 2008／11／16 at 上午 12：17：23 by Put your name here


2008／11／15 aUaÈ 09：59：46 Flow set to 1.00 at 0.01 minutes
2008／11／15 aUםÈ 10：40：57 Run stopped by operator

PEAK REPORT

| \＃ | begin | end | area | percent | maximum | time | begins as name |
| ---: | ---: | ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 27.17 | 29.10 | 7089 | 100.0 | 180.17 | 27.61 | Baseline |

Fig S110．HPLC analysis of the mixture of racemic and chiral 5c obtained（Table 2，entry 3）
ryn－2－127＋ryn－2－128 co inj col－IA 20\％EAI Hex
Report produced on 2008／11／16 at 上午 12：18：31 by Put your name here


2008／11／15 aUaÈ 10：43：29 Flow set to 1.00 at 0.03 minutes
2008／11／15 aUaÈ 11：49：51 Run stopped by operator

PEAK REPORT

| \＃ | begin | end | area | percent | maximum | time | begins as name |
| :--- | ---: | ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 27.14 | 28.19 | 710 | 12.9 | 55.31 | 27.58 | Baseline |
| 2 | 28.35 | 30.11 | 4777 | 87.1 | 138.56 | 28.76 | Baseline |

Fig S111．HPLC analysis of the racemic 5d（Table 2，entry 4）．

## Chromatogram Report

RYN－2－131 racemate col IA 20\％Ea／Hex
Report produced on 2008／11／17 at 下午 11：34：12 by Put your name here


2008／11／17 aUaÈ 12：10：18 Flow set to 1.00 at 0.00 minutes
2008／11／17 םUםEE 12：49：18 Run stopped by operator

## PEAK REPORT

| \＃ | begin | end | area | percent | maximum | time | begins as | name |
| ---: | ---: | ---: | ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 17.49 | 18.60 | 657 | 49.7 | 85.06 | 17.93 | Baseline |  |
| 2 | 18.77 | 19.88 | 666 | 50.3 | 86.75 | 19.15 | Baseline |  |

Fig S112．HPLC analysis of 5d obtained（Table 2，entry 4）．


Chromatogram Report
First line of organization＇s address Second line of organization＇s address

RYN－2－129 chiral col IA 20\％Ea／Hex

Report produced on 2008／11／17 at 下午 11：35：58 by Put your name here


2008／11／17 aUaÈ 12：51：08 Flow set to 1.00 at 0.00 minutes
2008／11／17 םUםÈ 01：57：19 Run stopped by operator

PEAK REPORT

| \＃ | begin | end | area | percent | maximum | time | begins as name |
| ---: | ---: | ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 19.75 | 21.10 | 1316 | 100.0 | 109.57 | 20.25 | Baseline |

Fig S113．HPLC analysis of the mixture of racemic and chiral 5d obtained（Table 2，entry 4） （For comparison）

RYN－2－129＋RYN－2－131 co inj col IA 20\％Ea／Hex
Report produced on 2008／11／17 at 下午 11：37：35 by Put your name here


2008／11／17 aUaÈ 02：00：51 Flow set to 1.00 at 0.00 minutes
2008／11／17 םUםÈ 02：40：20 Run stopped by operator

PEAK REPORT

| \＃ | begin | end | area | percent | maximum | time | begins as | name |
| :--- | ---: | ---: | ---: | ---: | ---: | ---: | ---: | :--- |
| 1 | 20.36 | 21.11 | 138 | 16.3 | 80.64 | 20.80 | Baseline |  |
| 2 | 21.28 | 22.35 | 708 | 83.7 | 103.68 | 21.56 | Baseline |  |

Fig S114．HPLC analysis of the racemic 5 e （Table 2，entry 5）．


## Chromatogram Report

## First line of organization＇s address

 Second line of organization＇s address
## RYN－2－132 racemate col IA 10\％Ea／Hex

Report produced on 2008／11／20 at 下午 02：09：27 by Put your name here


2008／11／19 aUaÈ 04：14：53 Flow set to 1.00 at 0.01 minutes
2008／11／19 aUםÈ 05：12：27 Run stopped by operator

## PEAK REPORT

| \＃ | begin | end | area | percent | maximum | time | begins as |
| :--- | ---: | :---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 32.93 | 34.88 | 221 | 52.6 | 39.89 | 33.98 | Baseline |
| 2 | 35.04 | 37.18 | 198 | 47.2 | 38.95 | 35.63 | Baseline |

Fig S115．HPLC analysis of the chiral 5e obtained（Table 2，entry 5）．


## Chromatogram Report

## RYN－2－130 chiral col IA 10\％Ea／Hex

Report produced on 2008／11／20 at 下午 02：10：37 by Put your name here


2008／11／19 aUaÈ 05：14：50 Flow set to 1.00 at 0.01 minutes
2008／11／19 aUםĖ 06：18：47 Run stopped by operator

PEAK REPORT

| \＃ | begin | end | area | percent | maximum | time | begins as name |
| :--- | ---: | ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 33.24 | 37.26 | 2035 | 100.0 | 64.69 | 34.55 | Baseline |

Fig S116．HPLC analysis of the mixture of racemic and chiral 5e obtained，（Table 2，entry 5） （For comparison）


## Chromatogram Report

RYN－2－130＋ 132 co injection col IA 10\％Ea／Hex
Report produced on 2008／11／20 at 下午 02：12：44 by Put your name here


2008／11／19 aUaÈ 06：20：31 Flow set to 1.00 at 0.00 minutes
2008／11／19 aUםĖ 07：29：38 Run stopped by operator

PEAK REPORT

| \＃ | begin | end | area | percent | maximum | time | begins as | name |
| :--- | ---: | :---: | ---: | ---: | ---: | ---: | ---: | :--- |
| 1 | 35.11 | 38.62 | 766 | 87.0 | 48.78 | 36.78 | Baseline |  |
| 2 | 39.19 | 41.13 | 114 | 13.0 | 38.94 | 40.05 | Baseline |  |

RYN－2－136 racemate 2 col IA 20\％Ea／Hex
Report produced on 2008／11／17 at 下午 11：45：00 by Put your name here


2008／11／17 aUaÈ 04：43：12 Flow set to 1.00 at 0.00 minutes
2008／11／17 aUםĖ 06：05：21 Run stopped by operator

## PEAK REPORT

| \＃ | begin | end | area | percent | maximum | time | begins as | name |
| :--- | ---: | :---: | :---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 33.53 | 36.70 | 3423 | 53.1 | 133.20 | 34.47 | Baseline |  |
| 2 | 40.75 | 52.80 | 3026 | 46.9 | 96.82 | 49.55 | Baseline |  |

Fig S118．HPLC analysis of chiral 5f obtained（Table 2，entry 6）


## Chromatogram Report

RYN－2－133 chiral 2 col IA 20\％Ea／Hex

Report produced on 2008／11／17 at 下午 11：46：39 by Put your name here


2008／11／17 aUaÈ 06：08：00 Flow set to 1.00 at 0.01 minutes
2008／11／17 aUםĖ 07：14：40 Run stopped by operator

PEAK REPORT

| \＃ | begin | end | area | percent | maximum | time | begins as name |
| :--- | ---: | ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 34.85 | 38.69 | 8587 | 100.0 | 198.94 | 35.93 | Baseline |

Fig S119．HPLC analysis of the mixture of racemic and chiral 5f obtained（Table 2，entry 6）

## Chromatogram Report

RYN－2－133＋RYN－2－136 co inj col IA 20\％Ea／Hex
Report produced on 2008／11／17 at 下午 11：50：27 by Put your name here


2008／11／17 aUaÈ 07：16：45 Flow set to 1.00 at 0.01 minutes
2008／11／17 aUaÈ 08：46：06 Run stopped by operator
PEAK REPORT

| \＃ | begin | end | area | percent | maximum | time | begins as | name |
| :--- | ---: | :---: | :---: | ---: | ---: | ---: | ---: | :--- |
| 1 | 34.76 | 38.44 | 6831 | 84.7 | 182.09 | 35.67 | Baseline |  |
| 2 | 51.45 | 55.17 | 1238 | 15.3 | 88.84 | 52.78 | Baseline |  |

Fig S120．HPLC analysis of racemic 5 g （Table 2，entry 7） （For comparison）


## Chromatogram Report

RYN－2－139 racemate col IA $20 \% \mathrm{Ea} / \mathrm{Hex}$
Report produced on 2008／11／20 at 下午 02：49：39 by Put your name here


2008／11／17 aUaÈ 08：48：14 Flow set to 1.00 at 0.00 minutes
2008／11／17 aUaÈ 09：47：36 Run stopped by operator

PEAK REPORT

| \＃ | begin | end | area | percent | maximum | time | begins as name |
| :--- | ---: | ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 22.25 | 24.56 | 4155 | 50.3 | 170.42 | 22.94 | Baseline |
| 2 | 24.99 | 27.92 | 4111 | 49.7 | 173.18 | 26.38 | Baseline |



## Chromatogram Report

RYN－2－138 chiral col IA 20\％Ea／Hex
Report produced on 2008／11／17 at 下午 10：35：07 by Put your name here


2008／11／17 aUaÈ 09：50：39 Flow set to 1.00 at 0.00 minutes
2008／11／17 םUםĖ 10：33：58 Run stopped by operator

PEAK REPORT

| \＃ | begin | end | area | percent | maximum | time | begins as name |
| :--- | ---: | ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 25.63 | 28.09 | 8798 | 100.0 | 269.31 | 26.40 | Baseline |

Fig S122．HPLC analysis of the mixture of racemic and chiral 5 g obtained（Table 2，entry 7） （For comparison）


## Chromatogram Report

RYN－2－138＋RYN－2－139 co inj col IA 20\％Ea／Hex
Report produced on 2008／11／17 at 下午 11：25：31 by Put your name here


2008／11／17 aUaĖ 10：40：01 Flow set to 1.00 at 0.00 minutes
2008／11／17 aUaÈ 11：24：03 Run stopped by operator

## PEAK REPORT

| \＃ | begin | end | area | percent | maximum | time | begins as | name |
| ---: | ---: | ---: | ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 23.03 | 25.20 | 1699 | 19.5 | 110.35 | 23.74 | Baseline |  |
| 2 | 26.15 | 29.12 | 7006 | 80.5 | 222.86 | 26.97 | Baseline |  |

Fig S123．HPLC analysis of racemic 5h（Table 2，entry 8）

## Chromatogram Report

RYN－2－143 racemate col IA 10\％Ea／Hex
Report produced on 2008／11／20 at 下午 02：01：44 by Put your name here


2008／11／19 aUaÈ 02：29：54 Flow set to 1.00 at 0.00 minutes
2008／11／19 aUםÈ 03：03：42 Run stopped by operator

PEAK REPORT

| \＃ | begin | end | area | percent | maximum | time | begins as name |  |
| :--- | ---: | :---: | ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 14.37 | 15.91 | 2761 | 53.9 | 150.47 | 15.11 | Baseline |  |
| 2 | 16.08 | 17.64 | 2362 | 46.1 | 129.75 | 16.39 | Baseline |  |



Chromatogram Report
First line of organization＇s address Second line of organization＇s address

RYN－2－140 chiral col IA 10\％Ea／Hex
Report produced on 2008／11／20 at 下午 02：03：47 by Put your name here


2008／11／19 aUaÈ 03：05：33 Flow set to 1.00 at 0.03 minutes
2008／11／19 aUםÈ 03：38：06 Run stopped by operator

PEAK REPORT

| \＃ | begin | end | area | percent | maximum | time | begins as name |
| :--- | ---: | ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 15.21 | 17.03 | 4515 | 99.5 | 198.15 | 15.80 | Baseline |

Fig S125．HPLC analysis of the mixture of racemic and chiral 5h obtained（Table 2，entry 8） （For comparison）

## Chromatogram Report

RYN－2－140＋RYN－2－143 co inj col IA 10\％Ea／Hex
Report produced on 2008／11／20 at 下午 02：05：01 by Put your name here


2008／11／19 aUaÈ 03：40：05 Flow set to 1.00 at 0.00 minutes
2008／11／19 aUםÈ 04：12：38 Run stopped by operator

PEAK REPORT

| \＃ | begin | end | area | percent | maximum | time | begins as | name |
| :--- | ---: | :---: | :---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 15.72 | 17.57 | 3768 | 77.9 | 169.61 | 16.42 | Baseline |  |
| 2 | 17.74 | 19.24 | 1068 | 22.1 | 73.93 | 18.15 | Baseline |  |

Fig S126．HPLC analysis of racemic 5i（Table 2，entry 9）

## RYN－2－144 racemate Col IA 15\％Ea／Hex

Report produced on 2008／11／24 at 下午 05：56：34 by Put your name here


2008／11／24 aWaÈ 11：29：28 Flow set to 1.00 at 0.01 minutes
2008／11／24 aUםÈ 12：45：49 Run stopped by operator

## PEAK REPORT

| \＃ | begin | end | area | percent | maximum | time | begins as name |  |
| :--- | ---: | ---: | ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 49.99 | 54.81 | 8855 | 49.4 | 141.88 | 51.32 | Baseline |  |
| 2 | 56.49 | 61.39 | 9061 | 50.6 | 137.44 | 58.00 | Baseline |  |



## Chromatogram Report

## RYN－2－141 chiral Col IA 15\％Ea／Hex

Report produced on 2008／11／24 at 下午 05：59：34 by Put your name here


2008／11／24 aUaÉ 12：47：01 Flow set to 1.00 at 0.00 minutes
2008／11／24 aUaÈ 02：10：39 Run stopped by operator

PEAK REPORT

| \＃ | begin | end | area | percent | maximum | time |  |
| :--- | ---: | ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 57.59 | 64.76 | 19950 | 100.0 | 205.82 | 60.18 | Basegins as |

Fig S128．HPLC analysis of the mixture of racemic and chiral 5i obtained（Table 2，entry 9） （For comparison）


## Chromatogram Report

Report produced on 2008／11／24 at 下午 06：00：40 by Put your name here


2008／11／24 aUaÈ 02：18：50 Flow set to 1.00 at 0.03 minutes
2008／11／24 aUaÈ 03：47：36 Run stopped by operator

PEAK REPORT

| \＃ | begin | end | area | percent | maximum | time | begins as name |  |
| :--- | ---: | :---: | ---: | ---: | ---: | ---: | ---: | :---: |
| 1 | 47.48 | 52.14 | 4336 | 20.4 | 123.18 | 49.91 | Baseline |  |
| 2 | 52.30 | 57.41 | 16920 | 79.6 | 236.47 | 53.35 | Baseline |  |



## Chromatogram Report

RYN－2－146 Racemate col IA 10\％ea／hex

Report produced on 2008／11／28 at 下午 04：06：12 by Put your name here


2008／11／28 aUaÈ 12：23：44 Flow set to 1.00 at 0.00 minutes
2008／11／28 aUםĖ 02：09：07 Run stopped by operator

PEAK REPORT

| \＃ | begin | end | area | percent | maximum | time | begins as name |  |
| :--- | ---: | ---: | ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 33.75 | 36.82 | 2042 | 49.7 | 69.63 | 34.57 | Baseline |  |
| 2 | 39.41 | 44.61 | 2069 | 50.3 | 59.61 | 41.75 | Baseline |  |



## Chromatogram Report

RYN－2－145 chiral col IA 10\％ea／hex

Report produced on 2008／11／28 at 下午 04：07：24 by Put your name here


2008／11／28 aUaÈ 02：11：15 Flow set to 1.00 at 0.01 minutes
2008／11／28 aUםĖ 03：03：54 Run stopped by operator

PEAK REPORT

| \＃ | begin | end | area | percent | maximum | time | begins as name |
| ---: | ---: | ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 34.06 | 38.16 | 6861 | 100.0 | 119.79 | 34.81 | Baseline |

Fig S131．HPLC analysis of the mixture of racemic and chiral 5 j obtained（Table 2，entry 10）

## Chromatogram Report

RYN－2－145＋RYN－2－146 co inj 2 col IA 10\％ea／hex
Report produced on 2008／11／28 at 下午 05：39：50 by Put your name here


2008／11／28 aUaÈ 04：10：25 Flow set to 1.00 at 0.00 minutes
2008／11／28 aUםĖ 05：16：03 Run stopped by operator

PEAK REPORT

| \＃ | begin | end | area | percent | maximum | time | begins as name |  |
| :--- | ---: | :---: | ---: | ---: | ---: | ---: | ---: | ---: |
| 1 | 35.40 | 38.98 | 3792 | 73.5 | 85.15 | 36.14 | Baseline |  |
| 2 | 43.84 | 47.69 | 1364 | 26.5 | 51.16 | 44.94 | Baseline |  |

