

Supporting Information

Synthesis and Application of P,Olefin Type Axially Chiral Ligands with *sec*-Alkyl Groups

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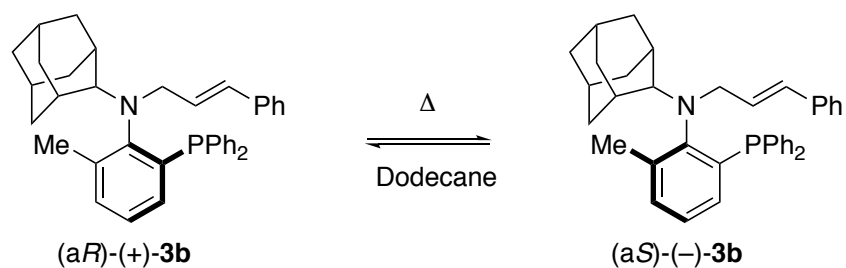
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Table of Contents

1. Data of thermal racemization of **3b-d**.
2. Copies of ¹H, ¹³C and ³¹P NMR spectra of **5**, **6**, **2**, **7**, and **8**.
3. Copies of ¹H, ¹³C and ³¹P NMR spectra and HPLC charts of **3**.
4. Copies of ¹H and ¹³C NMR and HPLC charts of **9**.

Table S1. Time Dependence of Ee Value of (aR)-(+)-**3b** at Various Temperatures



Time (sec)	Ee (%) ^a at 30 °C	Ee (%) ^a at 40 °C	Ee (%) ^a at 50 °C	Ee (%) ^a at 60 °C
0	96.4	96.4	95.6	97.2
1800	94.7	93.7	84.2	65.6
3600	94.4	90.7	73.1	48.5
7200	92.7	84.7	55.9	28.0
14400	90.0	75.2	38.6	7.8
28800	84.5	56.9	14.5	-
86400	67.5	18.0	-	-

^a Ee was determined by chiral HPLC analysis.

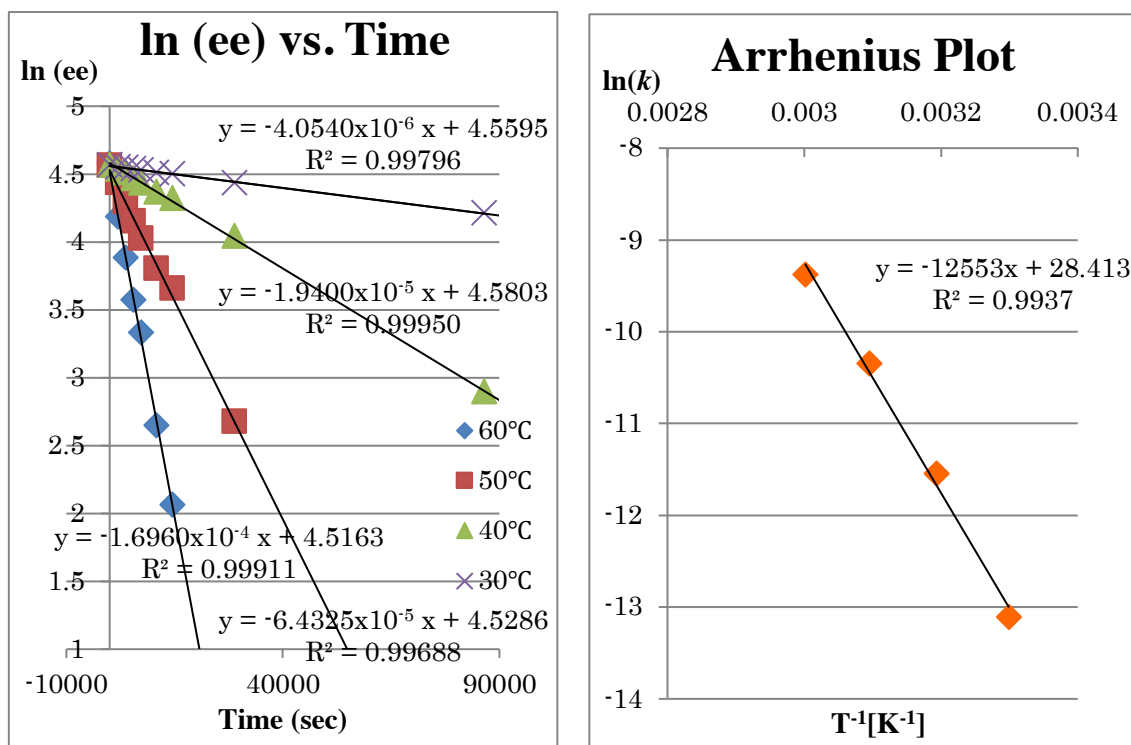
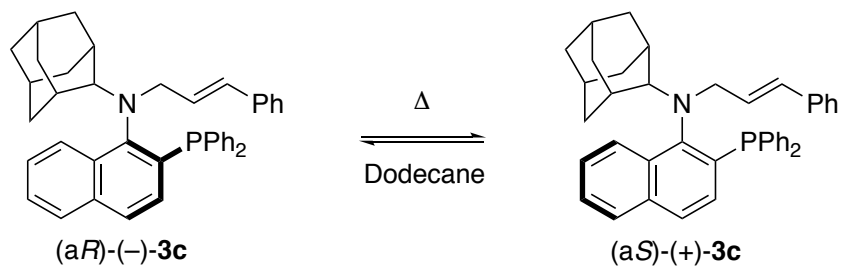


Figure S1. Data for the Racemization and Arrhenius Plot of (aR)-(+)-**3b**

Table S2. Time Dependence of Ee Value of (aR)-(-)-**3c** at Various Temperatures

Time (sec)	Ee (%) ^a at 30 °C	Ee (%) ^a at 40 °C	Ee (%) ^a at 50 °C	Ee (%) ^a at 60 °C
0	91.9	91.9	92.3	92.1
1800	90.6	85.5	72.6	45.6
3600	89.1	78.8	57.0	23.9
7200	86.2	67.5	35.8	7.0
14400	81.2	49.5	13.4	-
28800	71.2	26.2	2.4	-
86400	42.5	-	-	-

^a Ee was determined by chiral HPLC analysis.

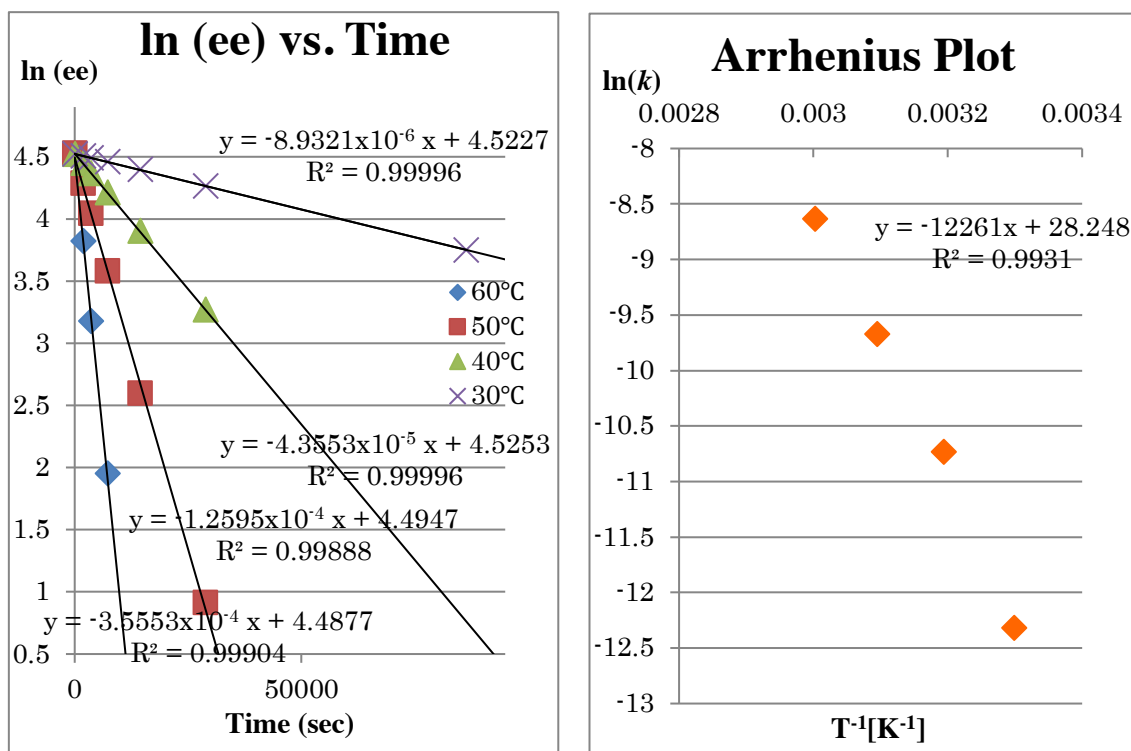
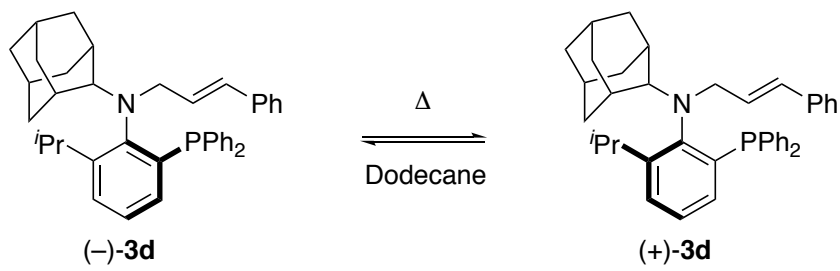
**Figure S2.** Data for the Racemization and Arrhenius Plot of (aR)-(-)-**3c**

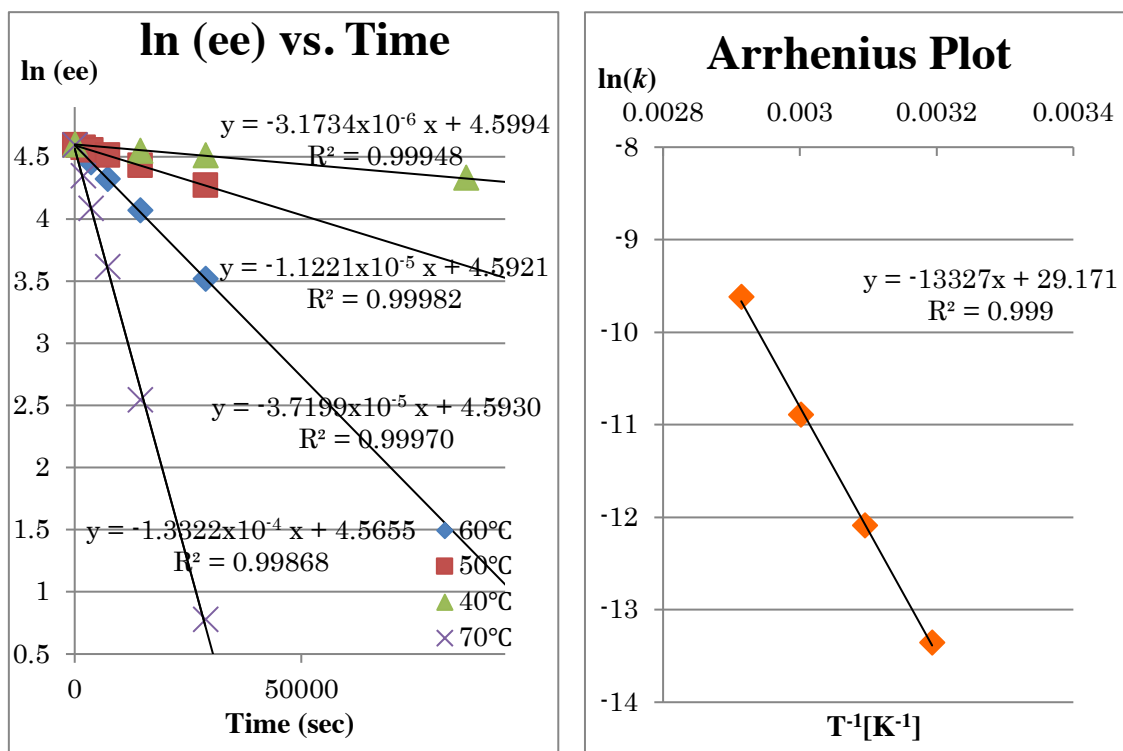
Table S3. Time Dependence of Ee Value of (-)-**3d** at Various Temperatures



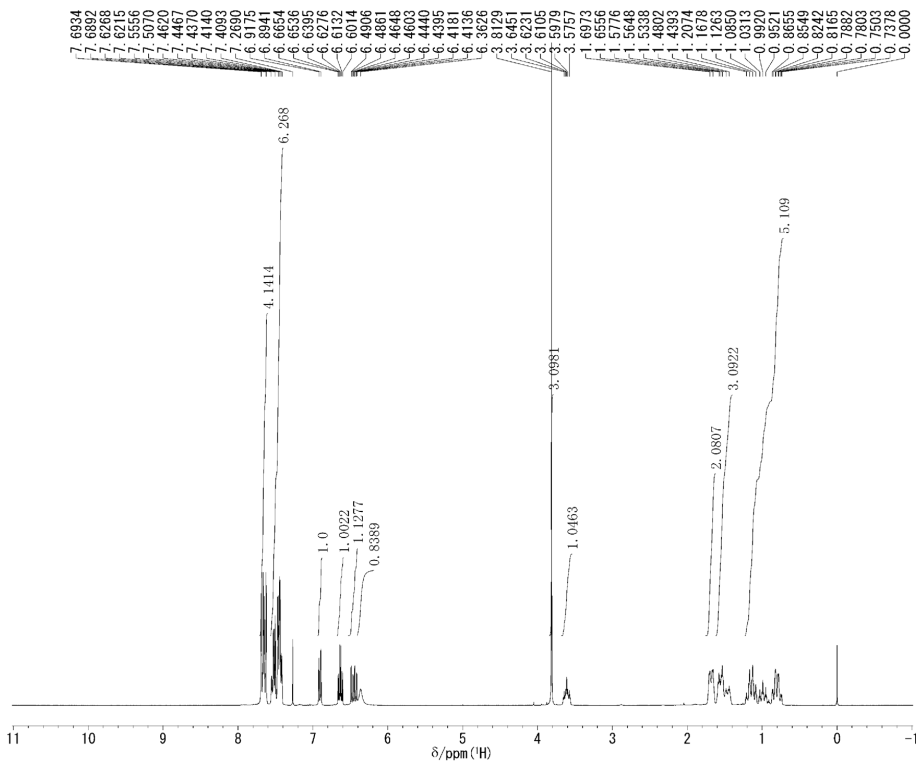
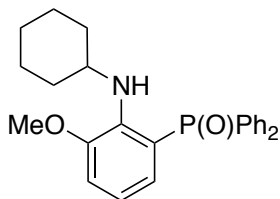
(-)-**3d** $\xrightleftharpoons[\text{Dodecane}]{\Delta}$ (+)-**3d**

Time (sec)	Ee (%) ^a at 40 °C	Ee (%) ^a at 50 °C	Ee (%) ^a at 60 °C	Ee (%) ^a at 70 °C
0	99.6	98.8	98.7	98.7
1800	-	96.8	92.6	77.3
3600	-	94.7	85.9	59.3
7200	-	90.9	75.1	37.0
14400	94.2	83.7	58.5	12.7
28800	91.0	71.5	33.6	2.1
86400	75.9	-	-	-

^a Ee was determined by chiral HPLC analysis.

**Figure S3.** Data for the Racemization and Arrhenius Plot of (-)-**3d**

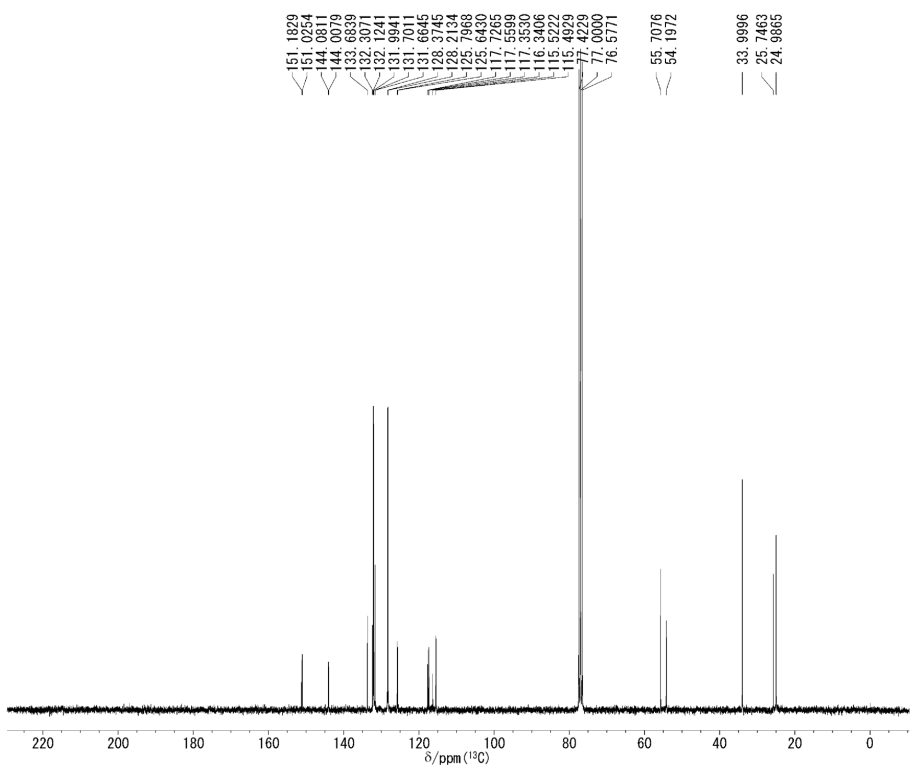
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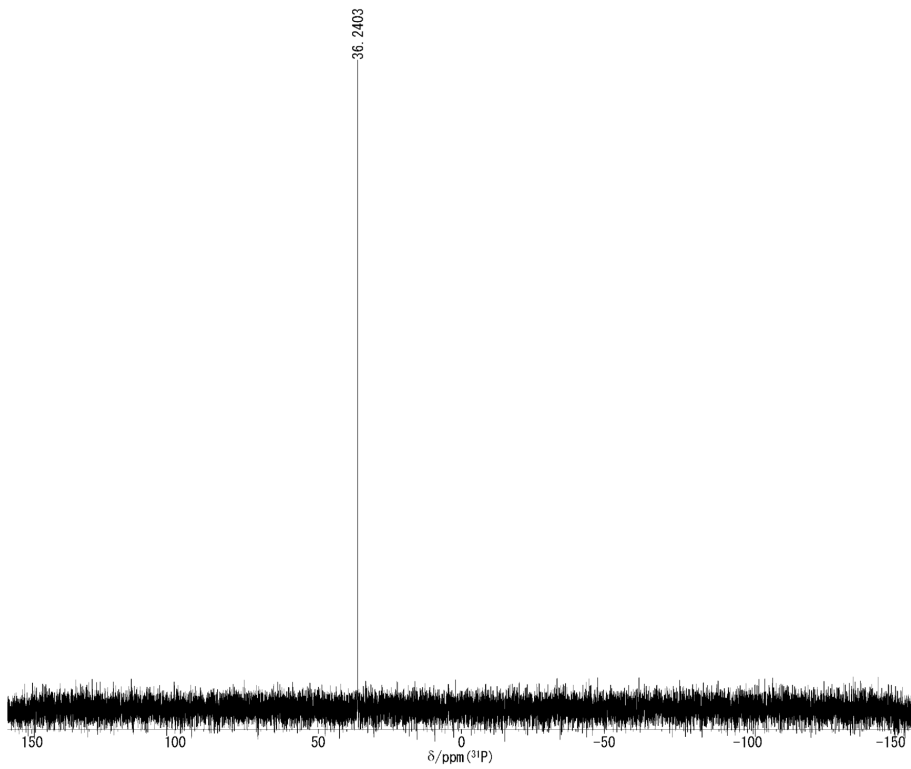
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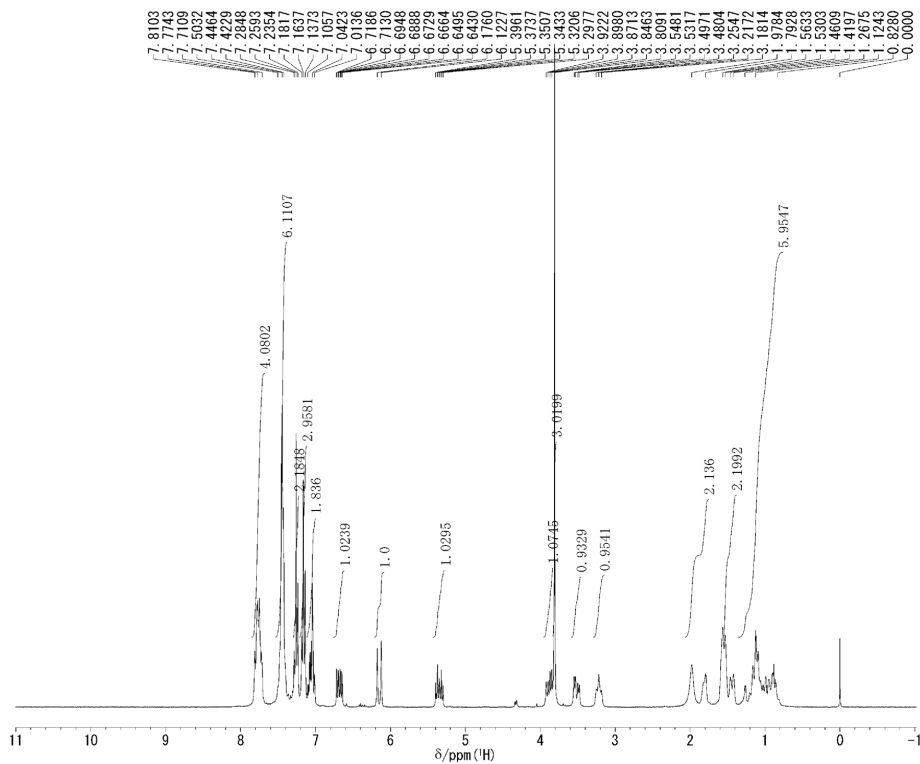
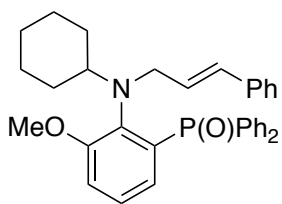


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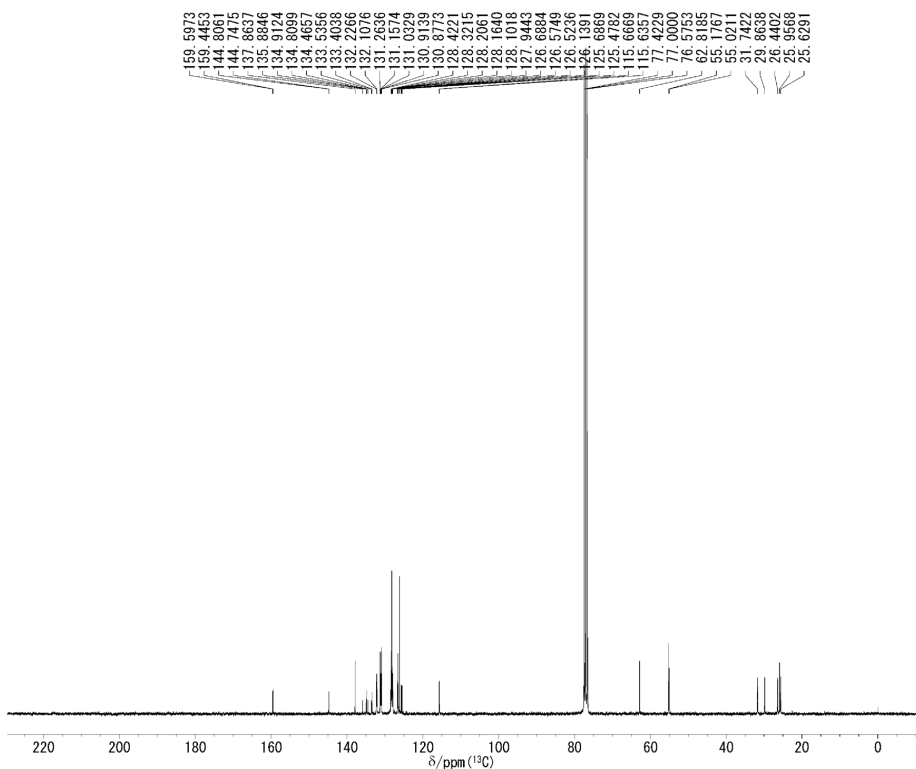
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¹H, ¹³C and ³¹P NMR of 6



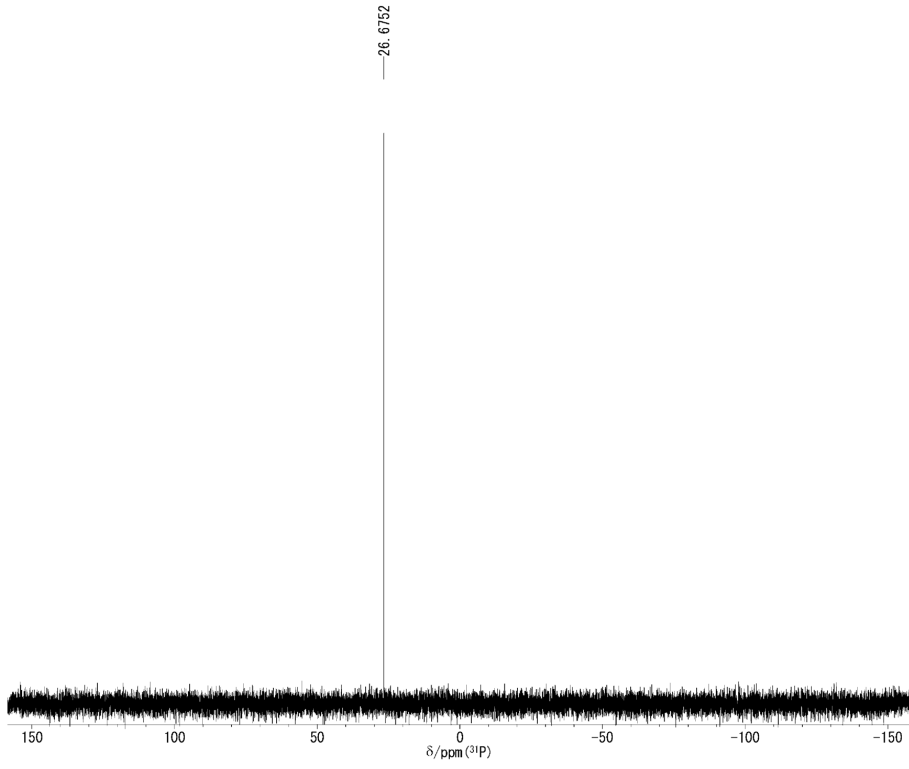
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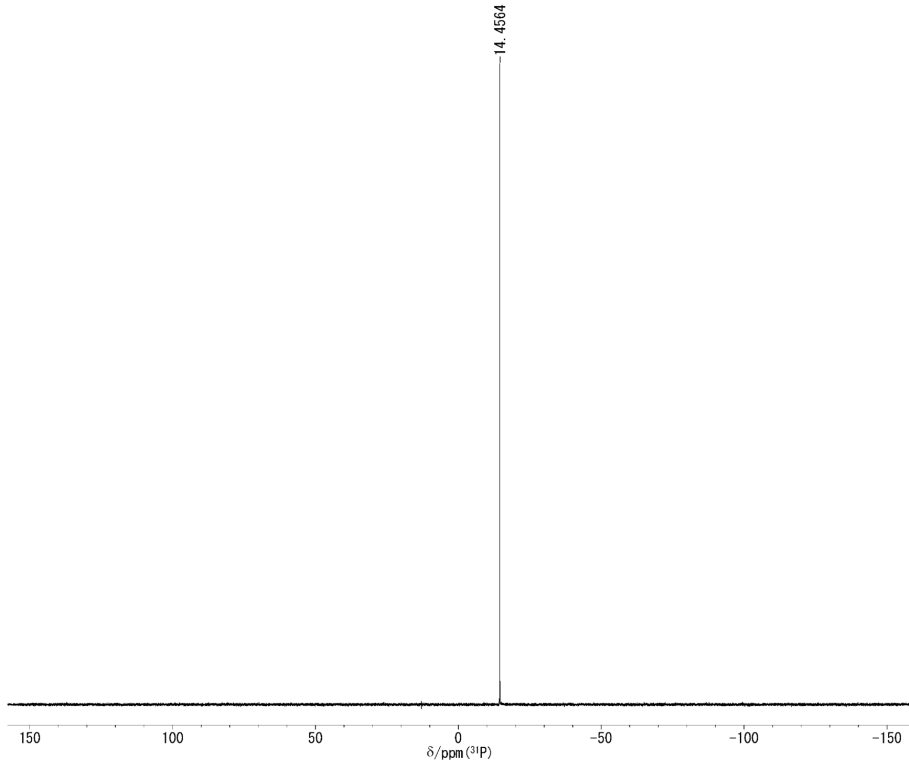
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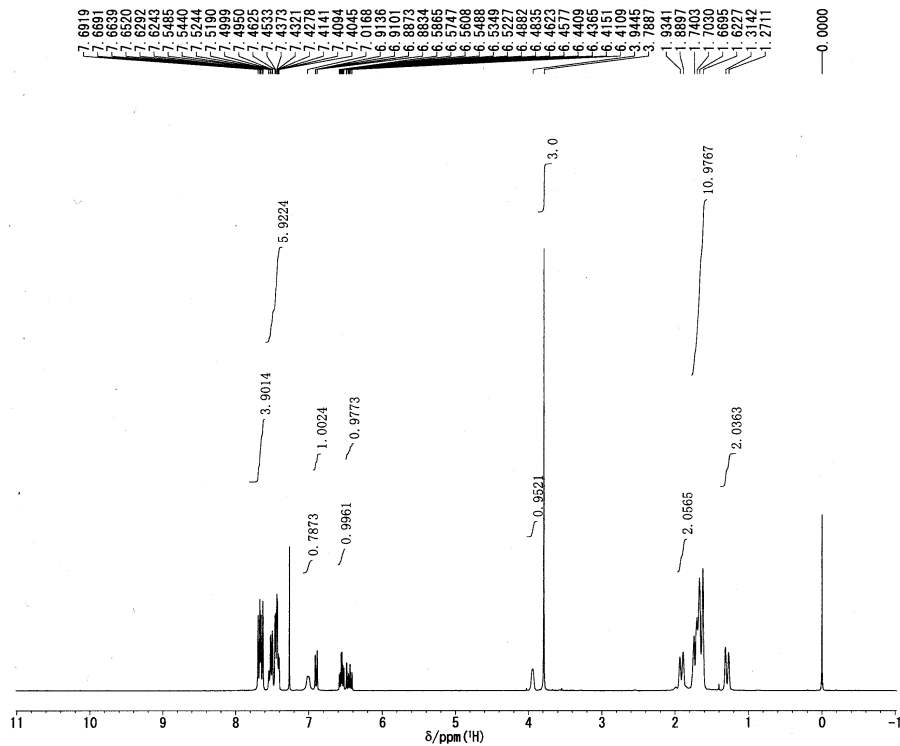
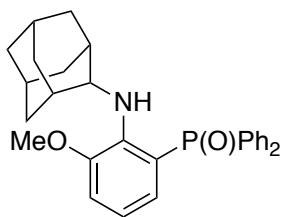
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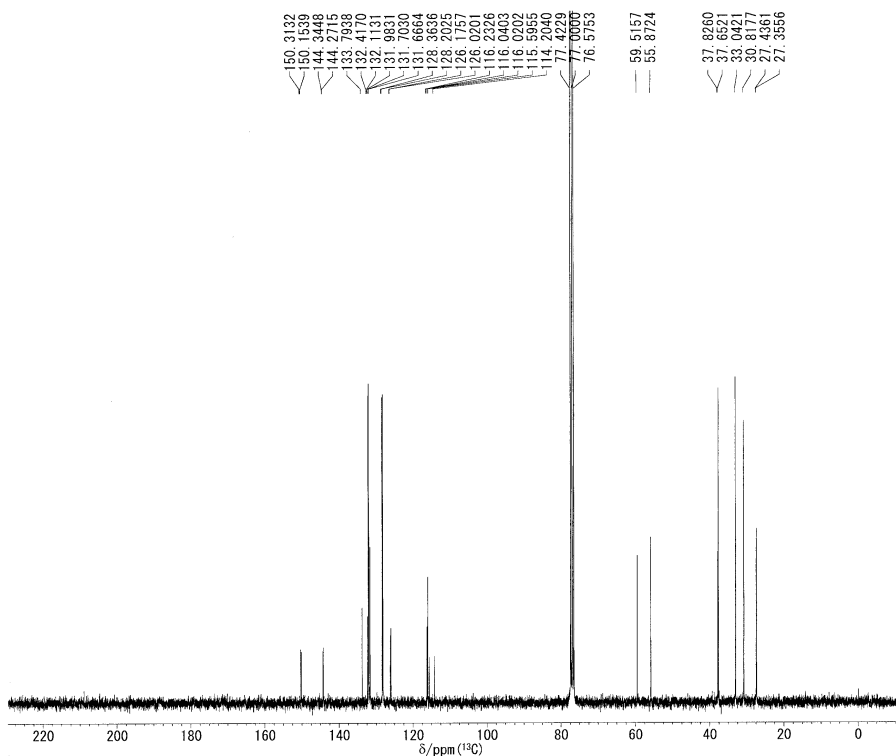
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¹H, ¹³C and ³¹P NMR of **7a**



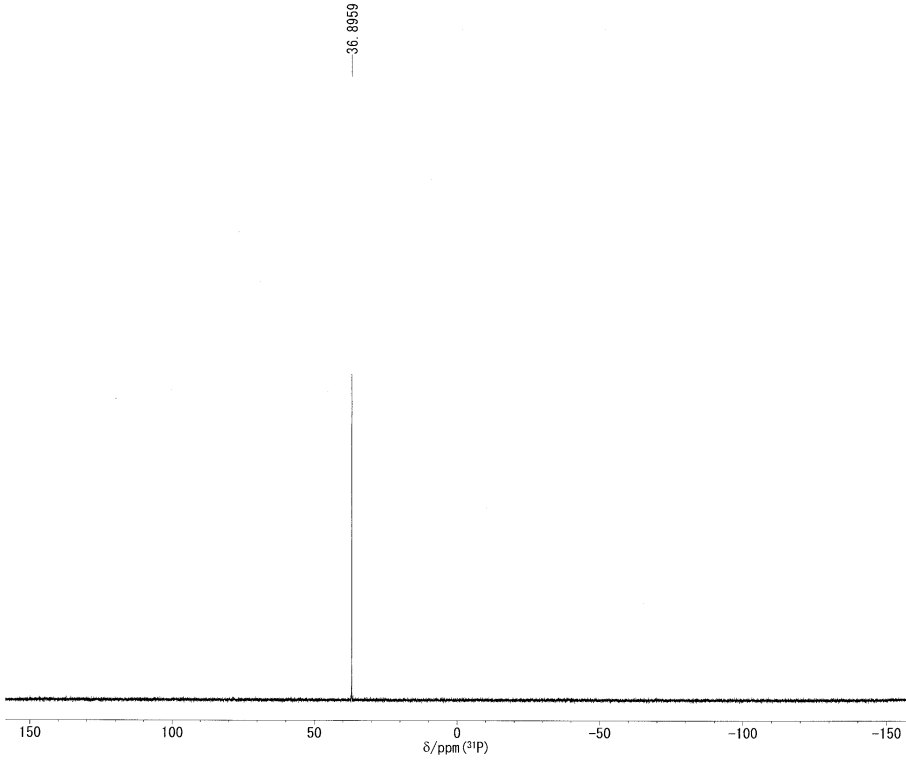
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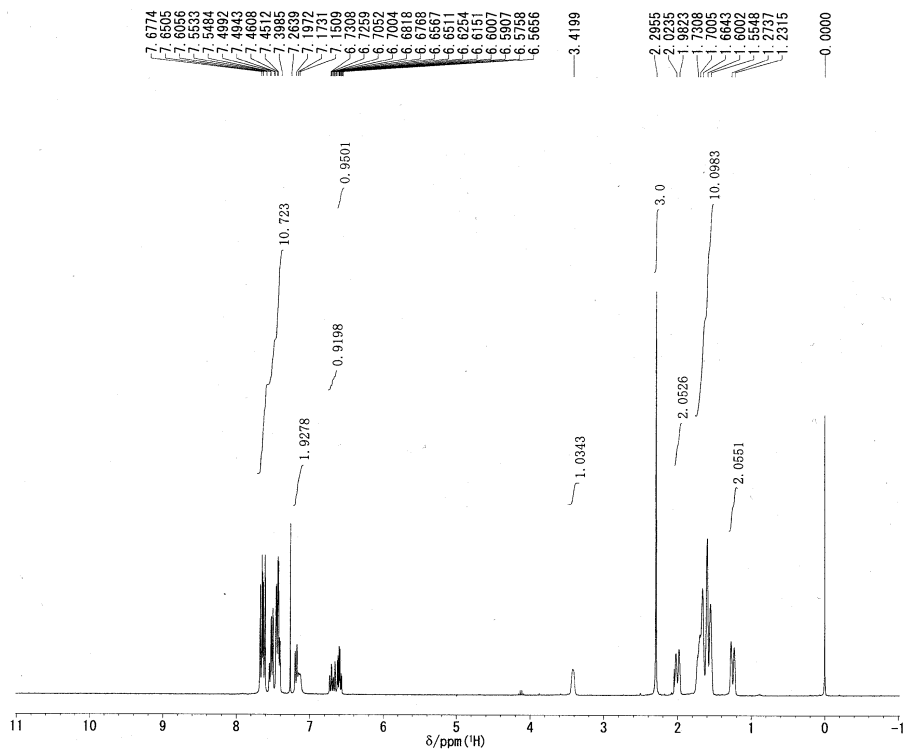
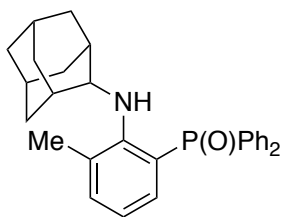


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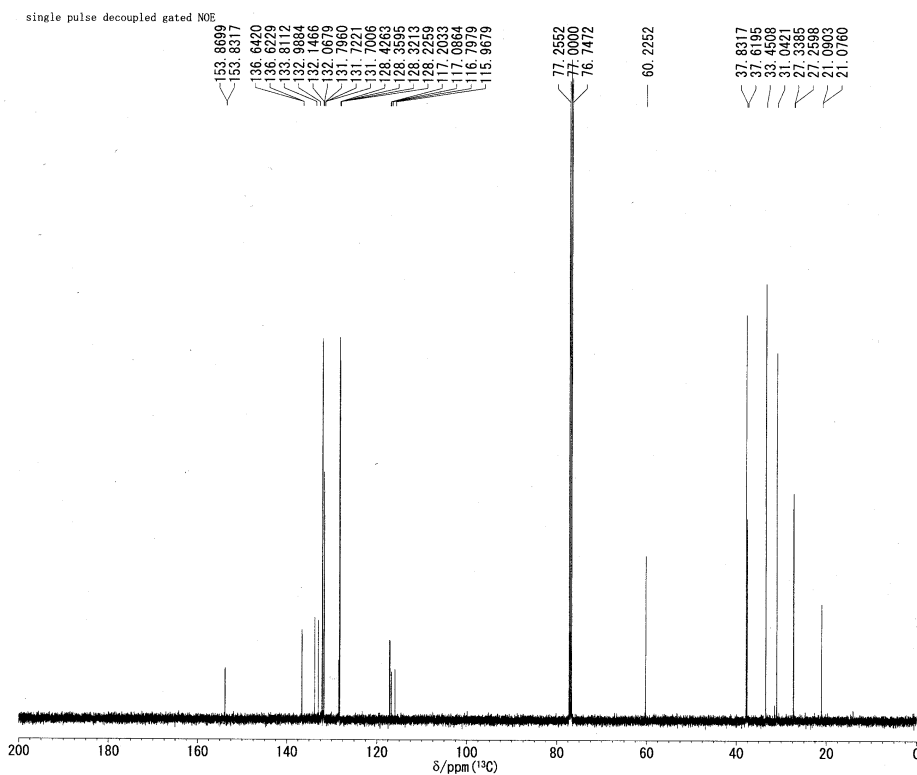
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¹H, ¹³C and ³¹P NMR of **7b**



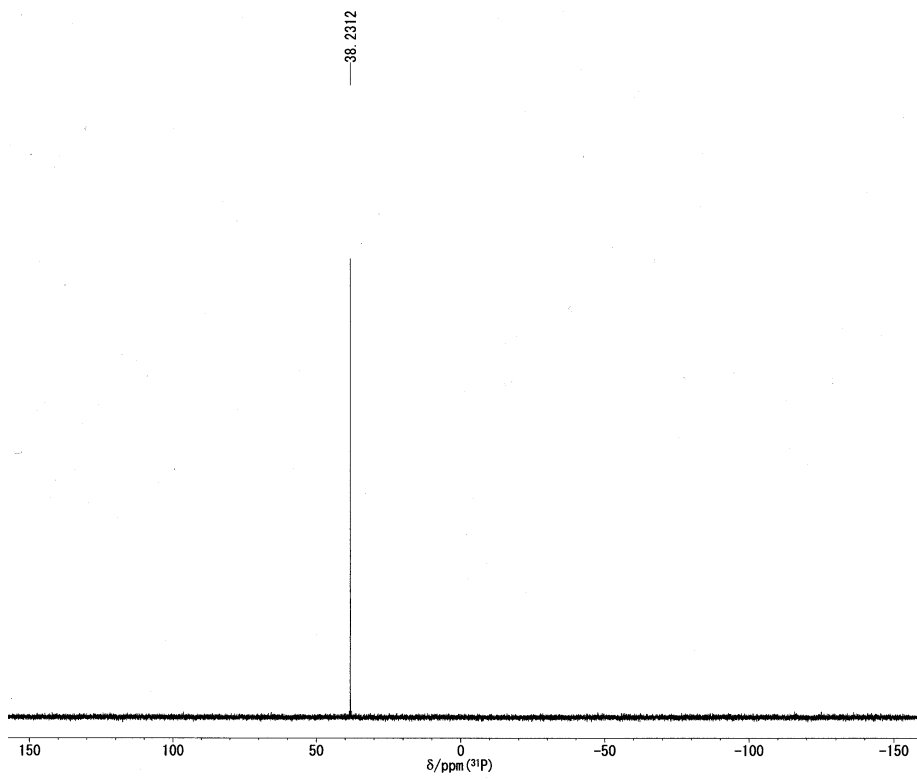
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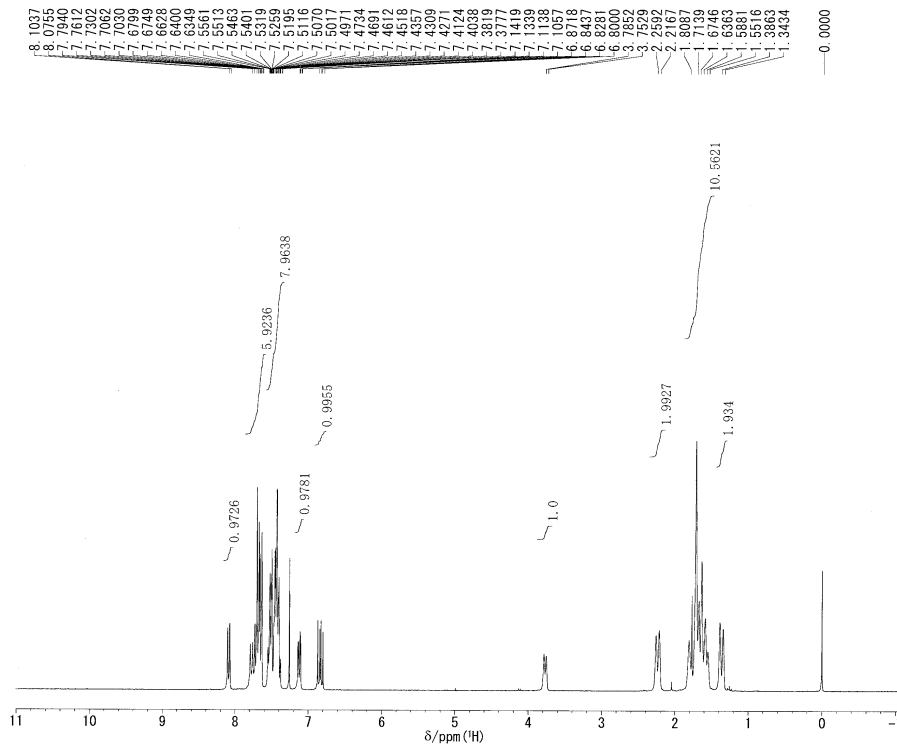
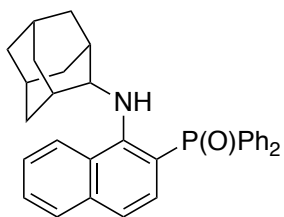
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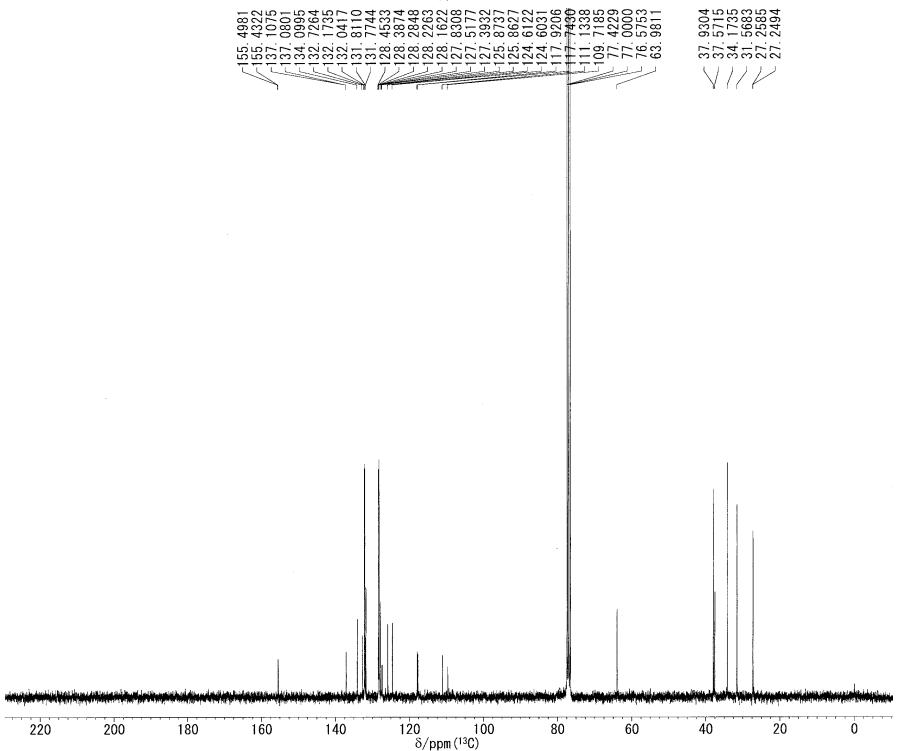
¹H, ¹³C and ³¹P NMR of 7c



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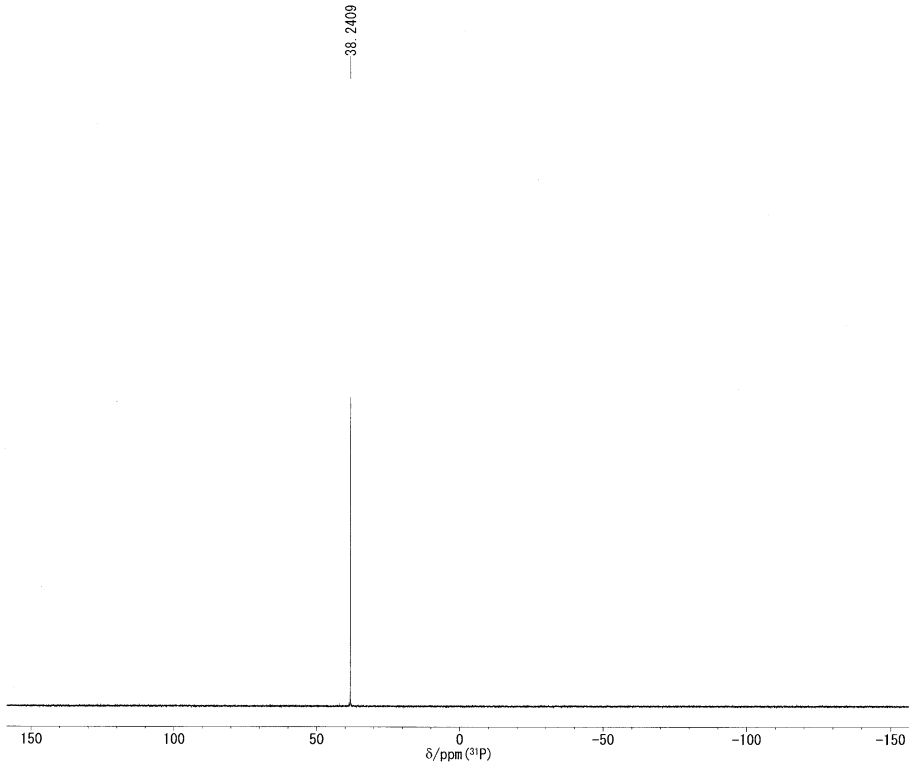
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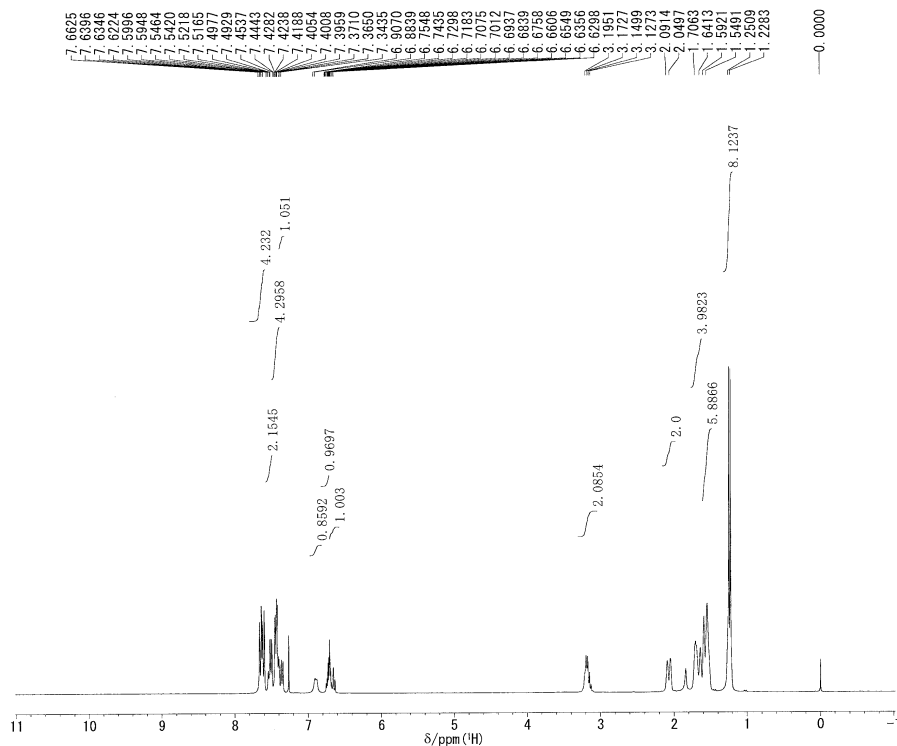
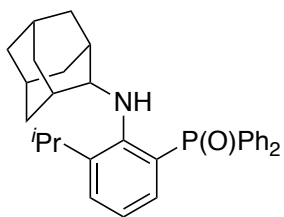


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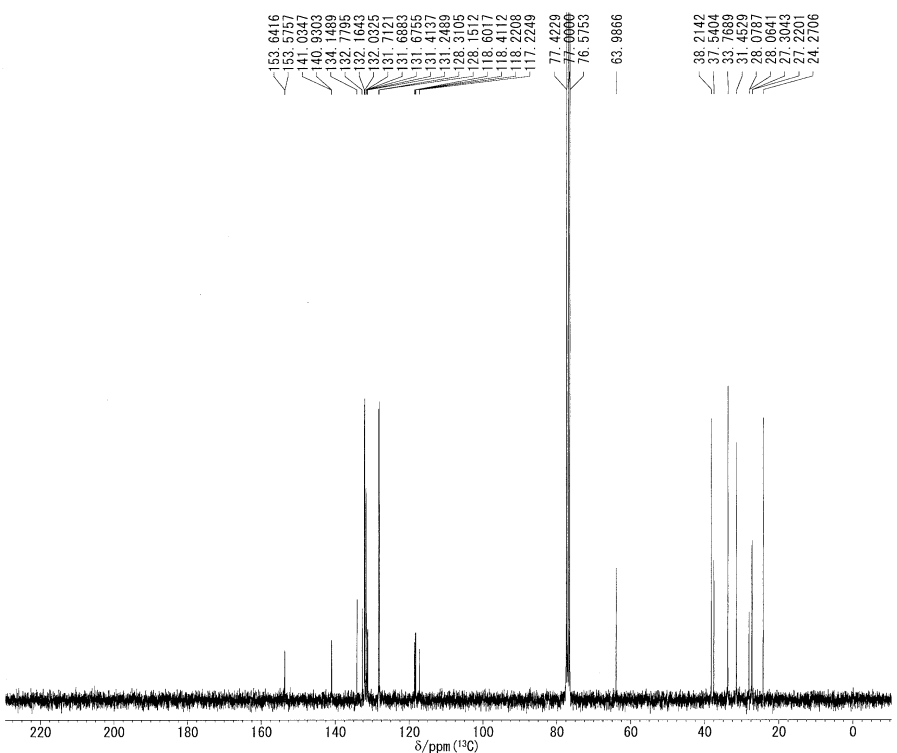
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¹H, ¹³C and ³¹P NMR of 7d



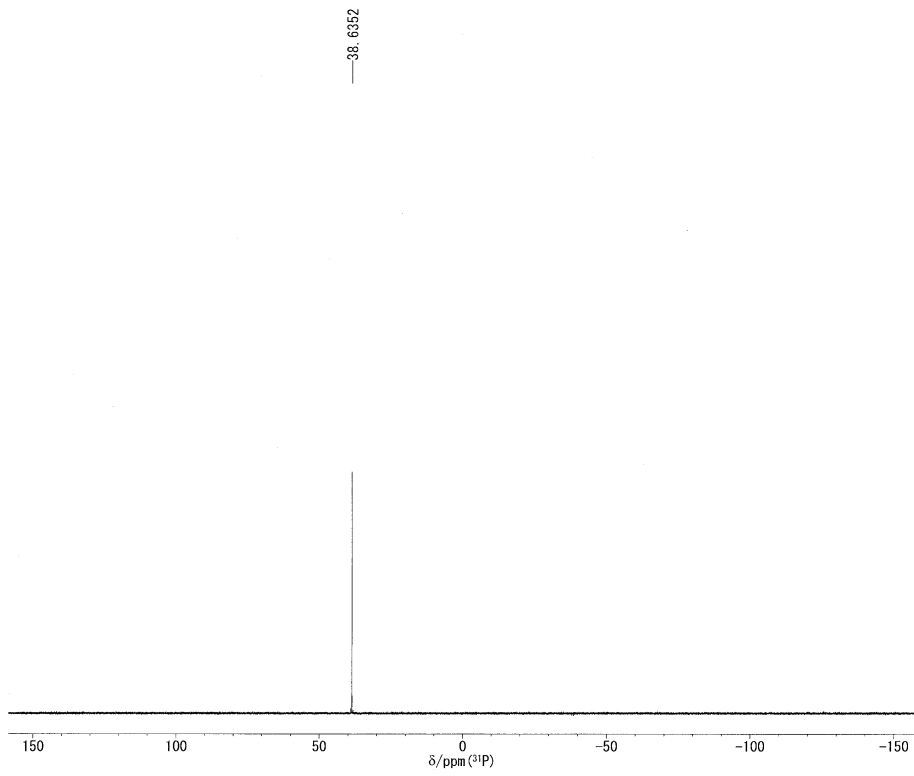
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DATEM 31/Oct/2016 20:55:46
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EXM0D ZGPG30
OBFRQ 75.49 MH
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OBFIN 10003.06 Hz
POINT 32768
FREQU 18115.94 Hz
SCANS 1024
ACQTM 1.8088
=====
PD 2.0 s
PW1 0.0 μs
IRNUC OFF
PROBHD 5 MM BBO BB-1H-D Z-GR
D Z8284/01
=====
INSTRUM SPECT
PULSPRG ZGPG30
GRDPRG
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SLVNT CDCl3
EXREF 77.0 ppm
BF 0.25 Hz
WINDOW Exponential
RGAIN 3251
Operator _____
    
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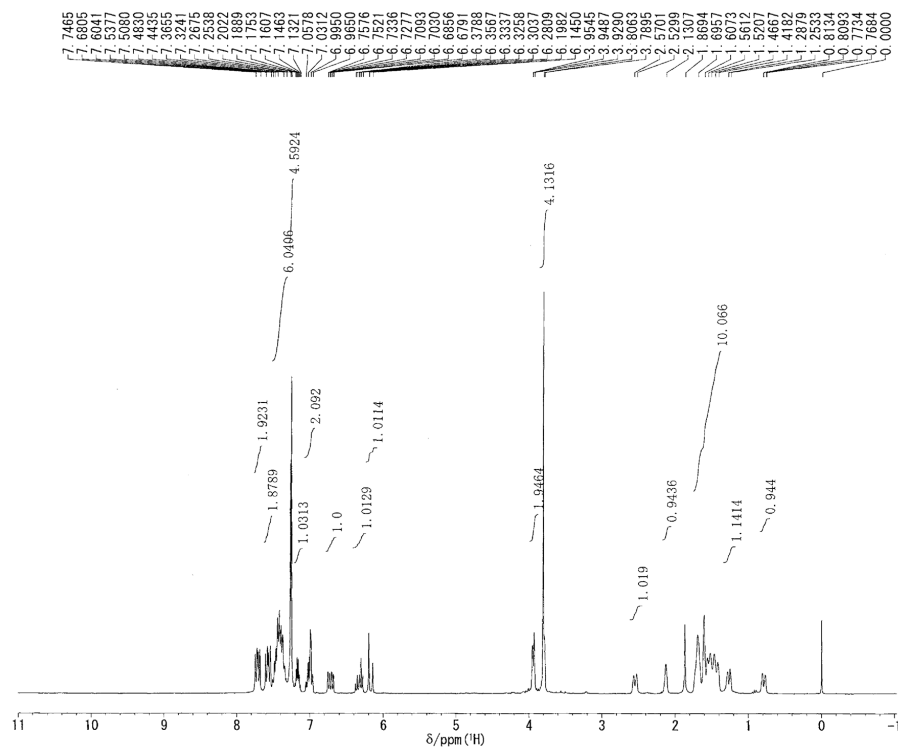
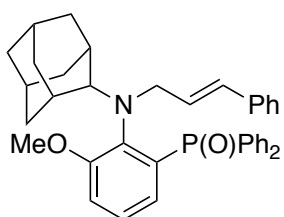


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PREQU 38535.64 Hz
SCANS 8
ACQTM 0.8503
s
PD 8.0 s
PWI 0.0  $\mu$ s
TRNUC 1H
PROBHD 5 MM BBO BB-1H-D Z-GR
D Z8284/01
INSTRUM SPECT
PULSPRG ZPG30
GRDPRG
CTEMP 26.85  $^{\circ}$ C
SLVNT CDCl3
EXREF 158.537 p
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BF 0.25 Hz
WINDOW Exponential
RGAIN 5793
Operator _____

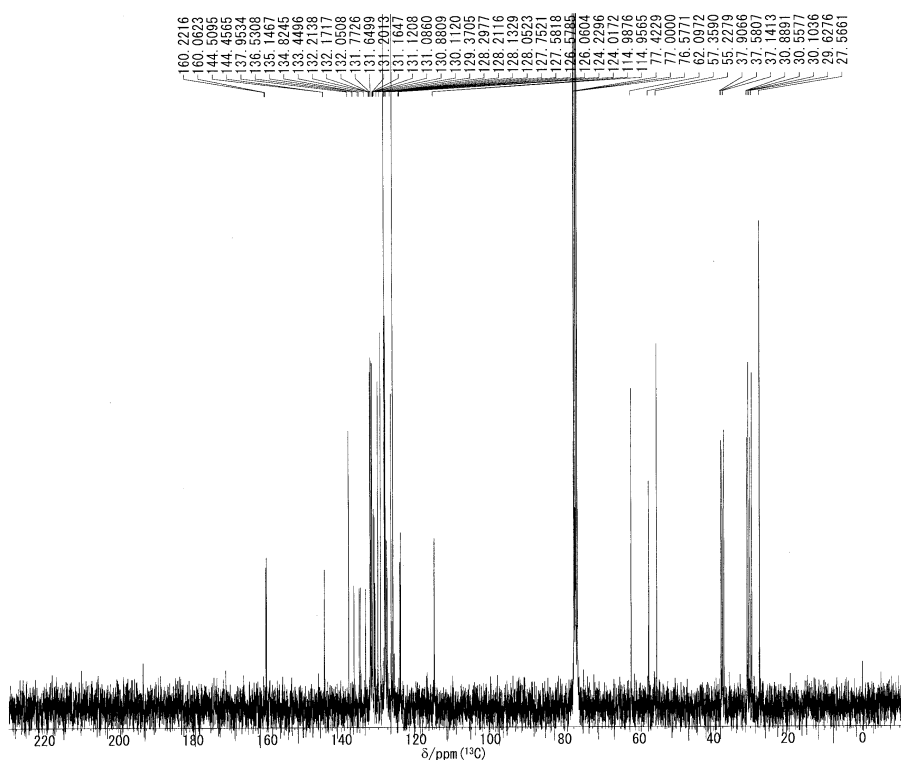
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¹H, ¹³C and ³¹P NMR of **8a**



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DATIM 16/Jul/2016 13:35:29
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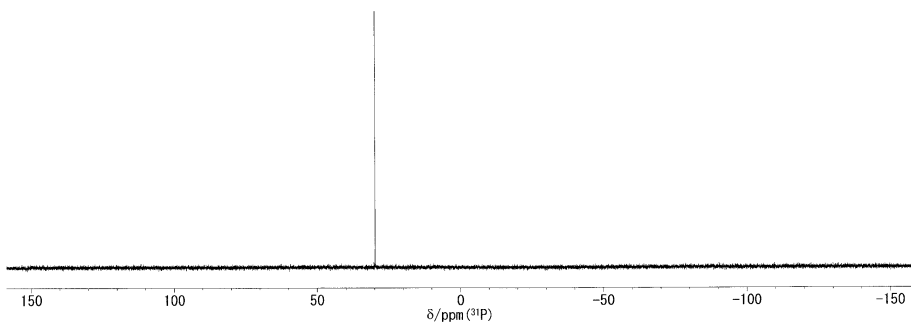
OBNUC ¹H
EXMOD ZG30
OBFRQ 300.23 MH
z
OBSET 0.0 kHz
OBFIN 10004.57 Hz
POINT 16384
FREQU 3612.717 H
z
SCANS 8
ACQTM 4.5351
s
PD 1.0 s
PWI 9.0 μs
FNUC OFF
PROBHD 5 MM BBO BB-1H-D Z-GR
D Z8284/01
INSTRUM SPECT
PULSPRG ZG30
GRDPRG
CTEMP 20.01 °C
SLNT CDCl₃
EXREF 0.0 ppm
BF 0.25 Hz
WINDOW Exponential
RGAIN 181
Operator



DFILE C:\YKHOUSE16\NMR\06MIN020
16\21161CY1\PDATAY111
DATIM 16/Jul/2016 14:42:29
COMNT

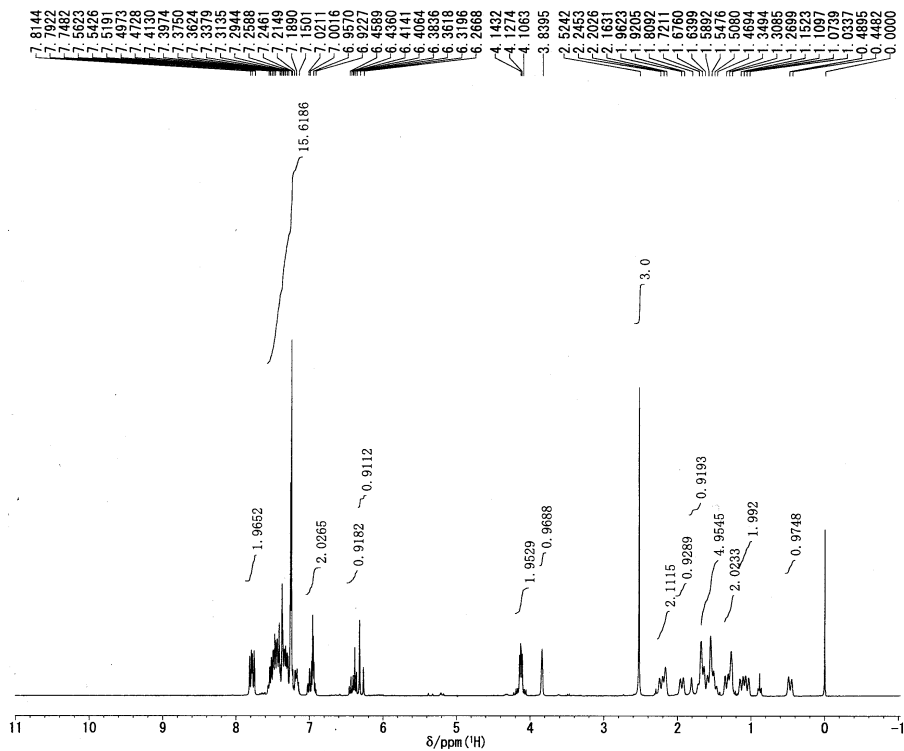
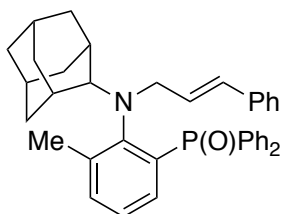
OBNUC ¹³C
EXMOD ZGPG30
OBFRQ 75.49 MH
z
OBSET 0.0 kHz
OBFIN 10003.06 Hz
POINT 32788
FREQU 18115.94 Hz
SCANS 1024
ACQTM 1.8088
s
PD 2.0 s
PWI 0.0 μs
FNUC OFF
PROBHD 5 MM BBO BB-1H-D Z-GR
D Z8284/01
INSTRUM SPECT
PULSPRG ZGPG30
GRDPRG
CTEMP 20.01 °C
SLNT CDCl₃
EXREF 77.0 ppm
BF 0.25 Hz
WINDOW Exponential
RGAIN 2048
Operator

—29.8128



DFILE C:\KYOUSE16\MRV06\MIN020
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DATIM 02/Oct/2015 18:03:17
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EXMOD ZGPG30
OBFRQ 121.54 MH
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OBSET 0.0 kHz
ORFIN 10005.0 Hz
POINT 32768
FREQU 38535.64 Hz
SCANS 8
ACQTM 0.8503
S
PD 8.0 s
PFI 0.0 μ s
IRNUC OFF
PROBHD 5 MM BBO BB-1H-D Z-GR
D Z8284/01
INSTRUM SPECT
PULSPRG ZGPG30
GRDPRG
CTEMP 26.85 $^{\circ}$ C
SLVNT CDCL₂
EXREF 158.537 p
PM
BF 0.25 Hz
WINDOW Exponential
RGAIN 13004
Operator _____

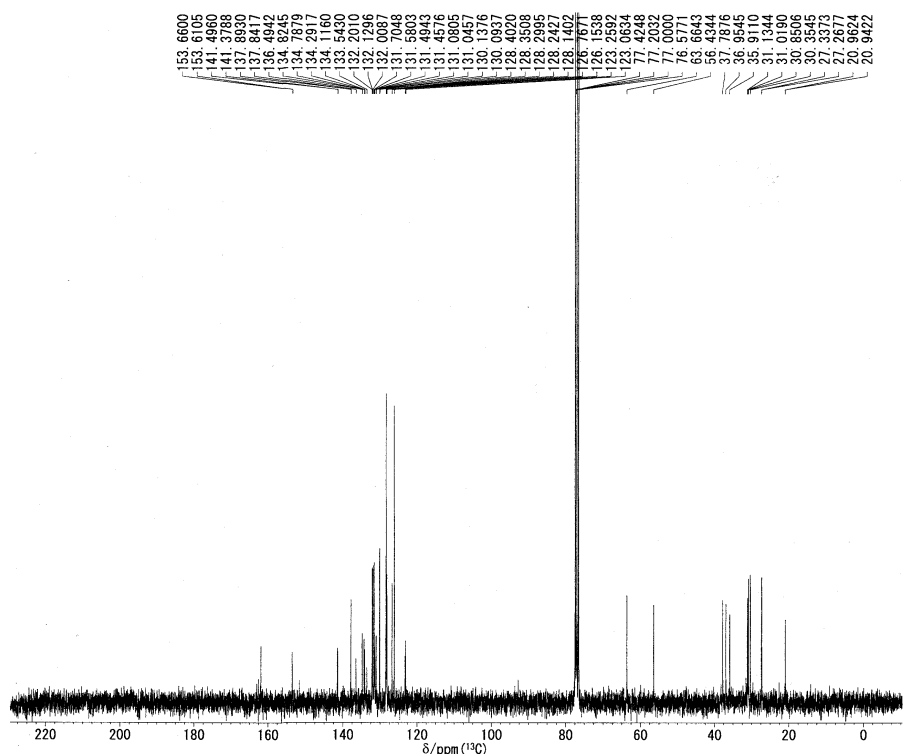
¹H, ¹³C and ³¹P NMR of **8b**



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DATIM 17/Feb/2015 17:58:33
COMT

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OBFRQ 300.23 MH
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OBSET 0.0 kHz
OBFIN 10004.57 Hz
POINT 16384
FREQU 3612.717 H
Z
SCANS 8
ACQTM 4.5351
S
PD 1.0 s
PFI 9.0 μs
IRNUC OFF
PROBH 5 MM BBO BB-1H-D Z-GR
D 28284/01
INSTRUM SPECT
PULSPRG ZG30
GRDPRG
CTEMP 26.85 °C
SLVNT CDCl₃
EXREF 0.0 ppm
BF 0.25 Hz
WINDOW Exponential
RGAIN 256

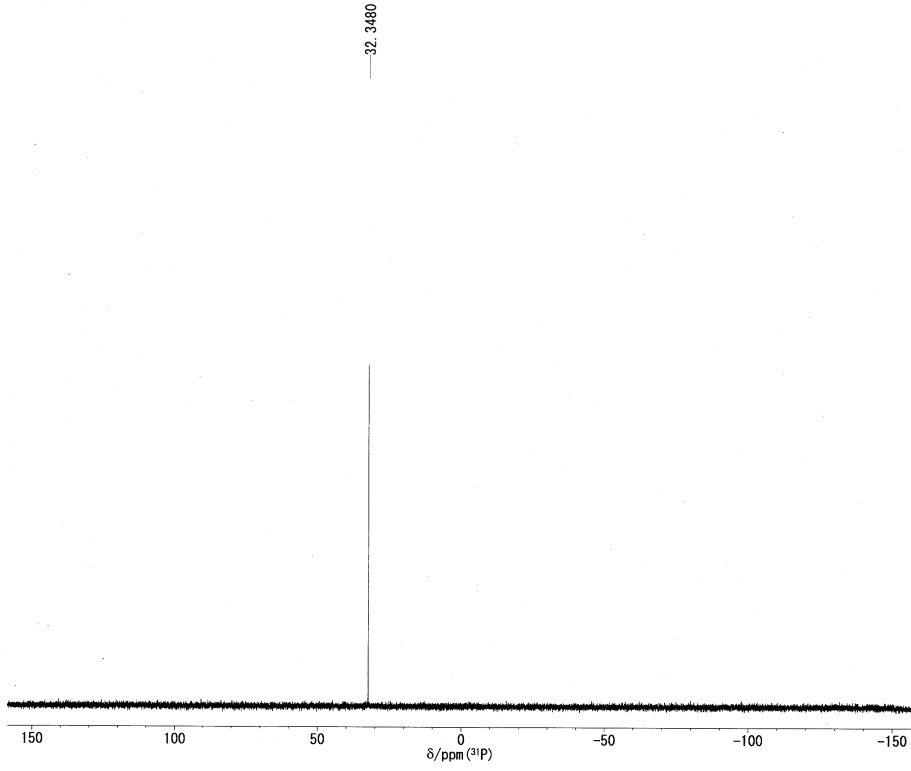
Operator _____



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COMT

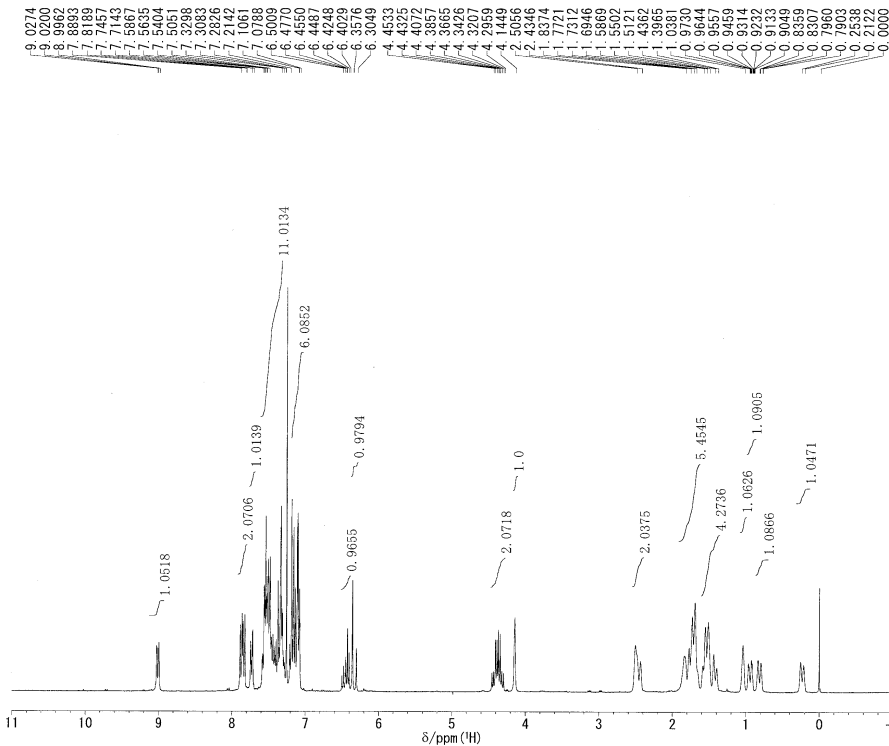
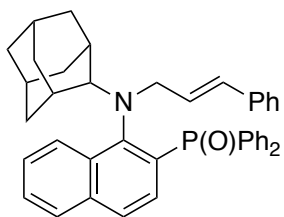
OBNUC ¹³C
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OBFRQ 75.49 MH
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OBSET 0.0 kHz
OBFIN 10003.96 Hz
POINT 32768
FREQU 18115.94 Hz
SCANS 512
ACQTM 1.8088
S
PD 2.0 s
PFI 0.0 μs
IRNUC OFF
PROBH 5 MM BBO BB-1H-D Z-GR
D 28284/01
INSTRUM SPECT
PULSPRG ZGPC30
GRDPRG
CTEMP 26.85 °C
SLVNT CDCl₃
EXREF 77.0 ppm
BF 0.25 Hz
WINDOW Exponential
RGAIN 2048

Operator _____



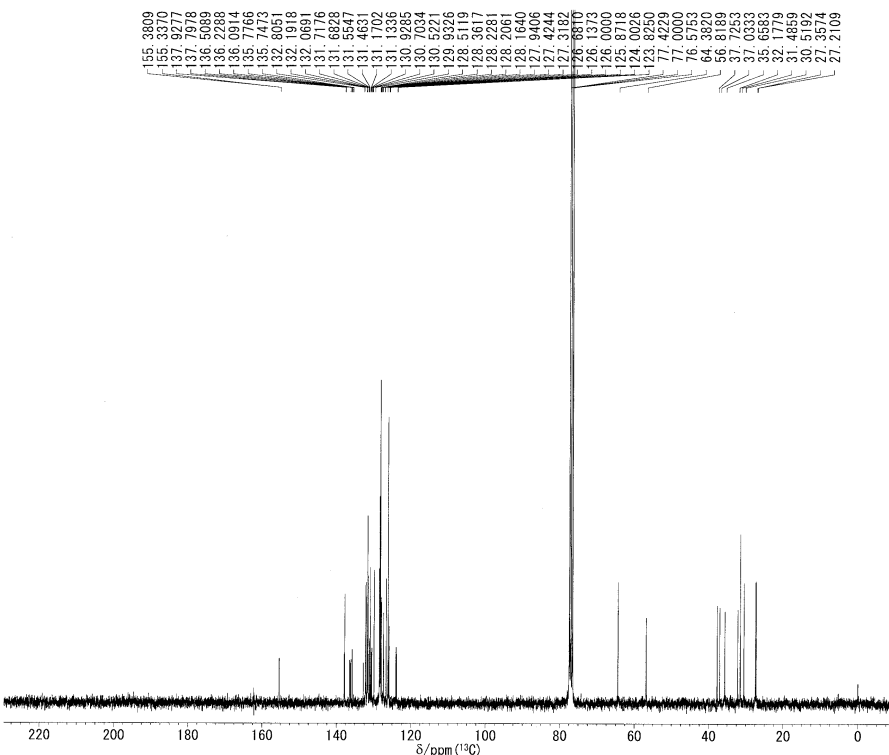
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 EXMOD ZFG30
 OBFRQ 121.54 MH
 Z
 OBSET 0.0 kHz
 OBFIN 10005.0 Hz
 POINT 32768
 FREQU 38535.64 Hz
 SCANS 8
 ACQTM 0.8503
 s
 PD 8.0 s
 PW1 0.0 μs
 TRNUC OFF
 PROHD 5 MM BBO BB-1H-D Z-CR
 D Z8284/01
 INSTRUM SPECT
 PULSPRG ZFG30
 GRDPRG
 CTEMP 26.85 °C
 SLVNT CDCL₄
 EXREF 158.537 p
 pm
 BF 0.25 Hz
 WINDOW Exponential
 RGAIN 11985
 Operator _____

¹H, ¹³C and ³¹P NMR of **8c**



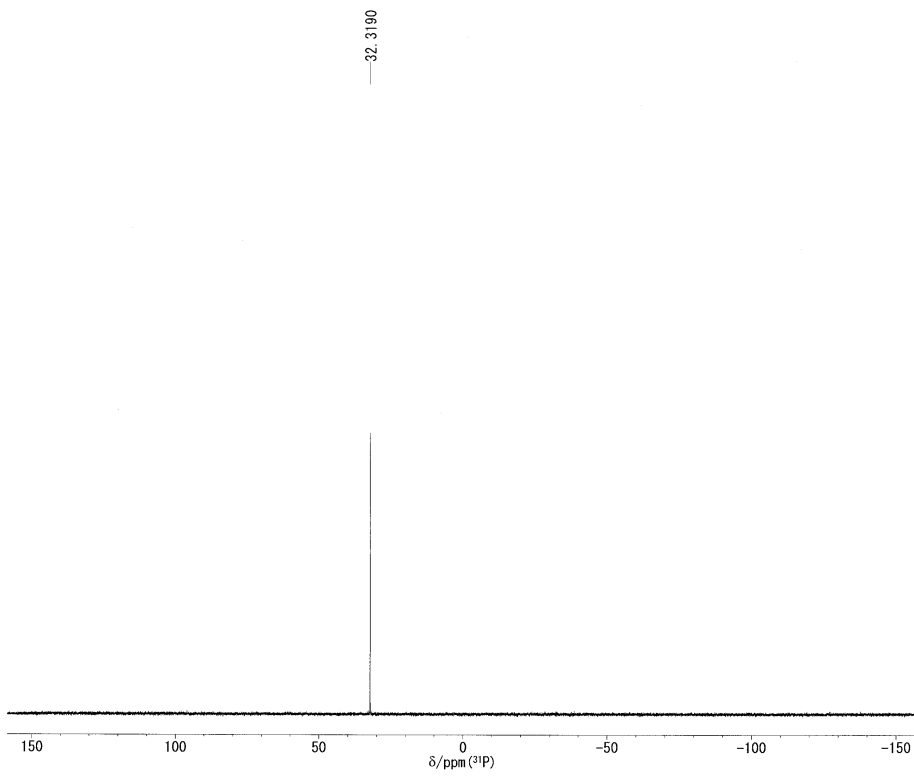
DFILE C:\KYOUSEI6\NMR\06MINO20
15V20284H\1VPDATA\1V11
DATIM 19/Jun/2015 18:10:51
COMNT

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EXMUD ZG30
OBFREQ 300.23 MH
z
OBSFZ 0.0 kHz
OBFIN 10004.57 Hz
POINT 16384
FREQQU 3612.717 H
z
SCANS 8
ACQTM 4.5351
s
PD 1.0 s
PWI 9.0 μs
IRNUC OFF
PROBHD 5 MM BBO BB-1H-D Z-GR
D 28284/01
INSTRUM SPECT
PULSPRG ZG30
GRDPRG
CTEMP 26.85 °C
SLVNT CDCl₃
EXREF 0.0 ppm
BF 0.25 Hz
WINDOW Exponential
RGAIN 228
Operator



DFILE C:\KYOUSEI6\NMR\06MINO20
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DATIM 22/Sep/2016 18:40:48
COMNT

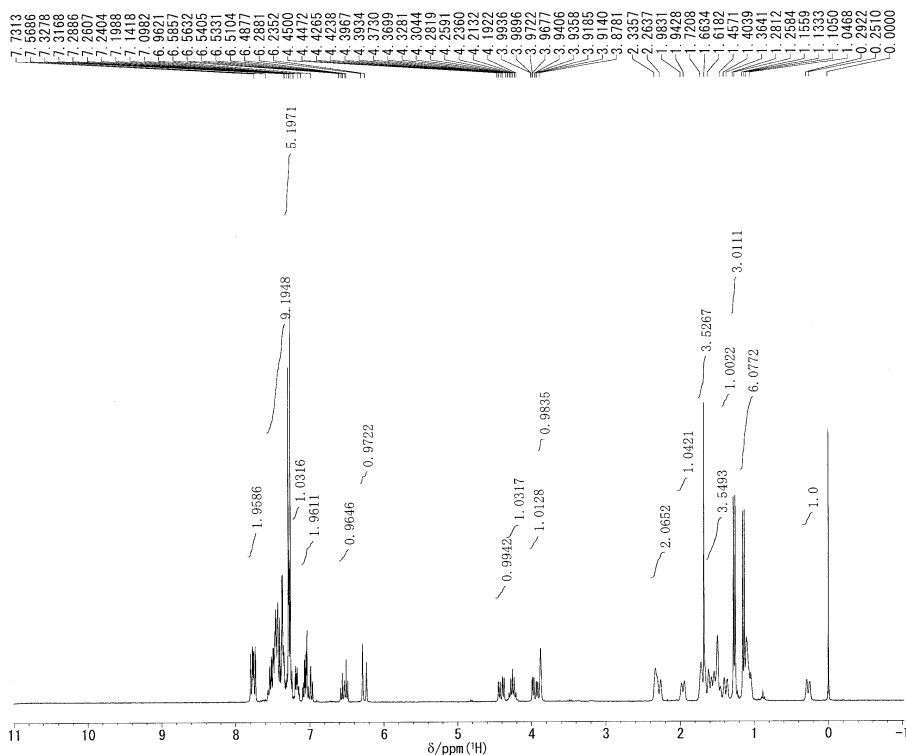
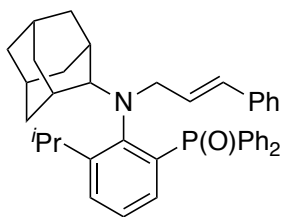
ORNUC ¹³C
EXMUD ZGPG30
OBFREQ 75.49 MH
z
OBSFZ 0.0 kHz
OBFIN 10003.06 Hz
POINT 32768
FREQQU 18115.94 Hz
SCANS 2048
ACQTM 1.8088
s
PD 2.0 s
PWI 0.0 μs
IRNUC OFF
PROBHD 5 MM BBO BB-1H-D Z-GR
D 28284/01
INSTRUM SPECT
PULSPRG ZGPG30
GRDPRG
CTEMP 19.01 °C
SLVNT CDCl₃
EXREF 77.0 ppm
BF 0.25 Hz
WINDOW Exponential
RGAIN 813
Operator



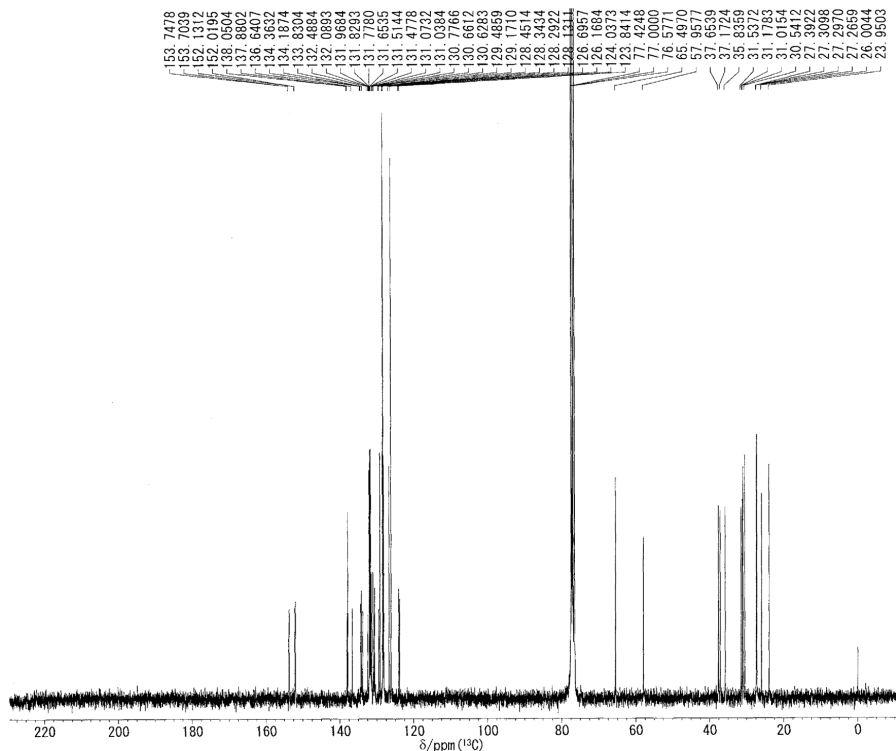
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DATIN 12/Jun/2015 20:32:19
COMNT

OBNUC ³¹P
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OBFRQ 121.54 MH
Z
OBSET 0.0 kHz
OBFIN 10005.0 Hz
POINT 32768
FREQU 38535.64 Hz
SCANS 8
ACQTM 0.8503
PD s 8.0 s
PW1 0.0 μs
IRNUC OFF
PROBHD 5 MM BBO BB-1H-D Z-GR
D ZS284/01
INSTRUM SPECT
PULSPRG ZPGP30
GRDPRG
CTEMP 26.85 °C
SOLVENT CDCl₃
XREF 158.537 p
BF pm 0.25 Hz
WINDOW Exponential
RGAIN 4096
Operator _____

¹H, ¹³C and ³¹P NMR of **8d**

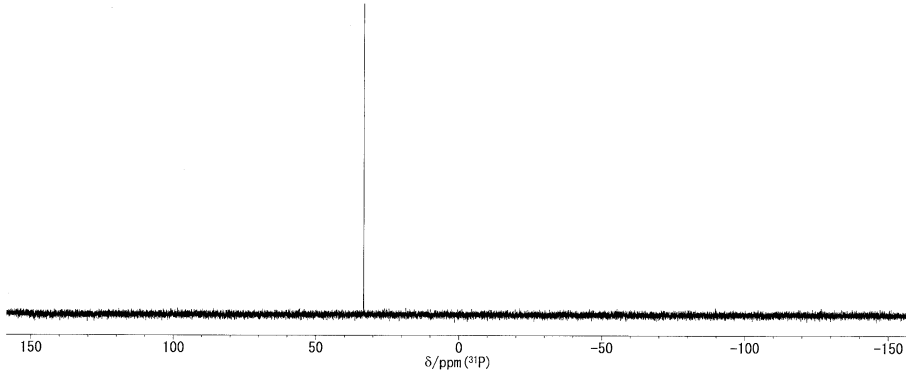


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 16V21490H2VPDATAV111
 DATIM 27/Oct/2016 19:13:28
 COMNT
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 EXMOD ZG30
 OBFREQ 300.23 MHz
 z
 OBSET 0.0 kHz
 OBFIN 10004.57 Hz
 POINT 16384
 FREQU 3612.717 H
 z
 SCANS 16
 ACQTM 4.5351
 s
 PD 1.0 s
 PW1 9.0 μs
 IRNUC OFF
 PROBHD 5 MM BBO BB-1H-D Z-GR
 D Z8284/01
 INSTRUM SPECT
 PULSPRG ZG30
 GRDPRG
 CTEMP 26.85 °C
 SLVNT CDCL₃
 EXREF 0.0 ppm
 BF 0.25 Hz
 WINDOW Exponential
 RGAIN 362
 Operator _____



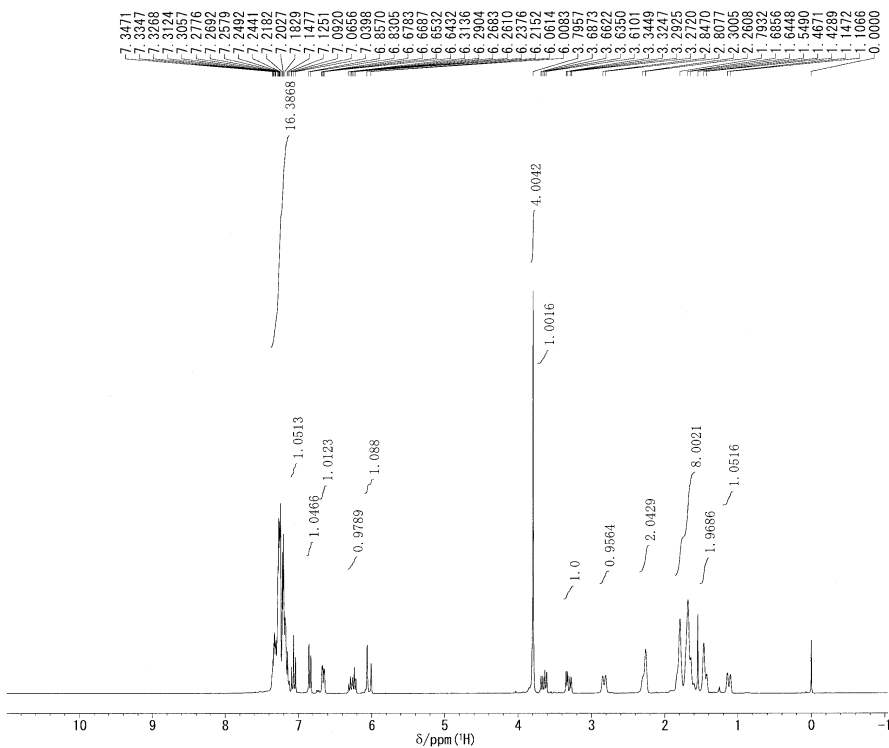
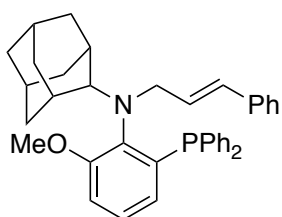
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 V2VPDATAV111
 DATIM 09/Nov/2016 09:44:00
 COMNT
 OBNUC ¹³C
 EXMOD ZGPC30
 OBFREQ 75.49 MHz
 z
 OBSET 0.0 kHz
 OBFIN 10003.06 Hz
 POINT 32768
 FREQU 18115.94 Hz
 SCANS 9539
 ACQTM 1.8088
 s
 PD 2.0 s
 PW1 0.0 μs
 IRNUC OFF
 PROBHD 5 MM BBO BB-1H-D Z-GR
 D Z8284/01
 INSTRUM SPECT
 PULSPRG ZGPC30
 GRDPRG
 CTEMP 26.85 °C
 SLVNT CDCL₃
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 BF 0.25 Hz
 WINDOW Exponential
 RGAIN 2580
 Operator _____

33.3253



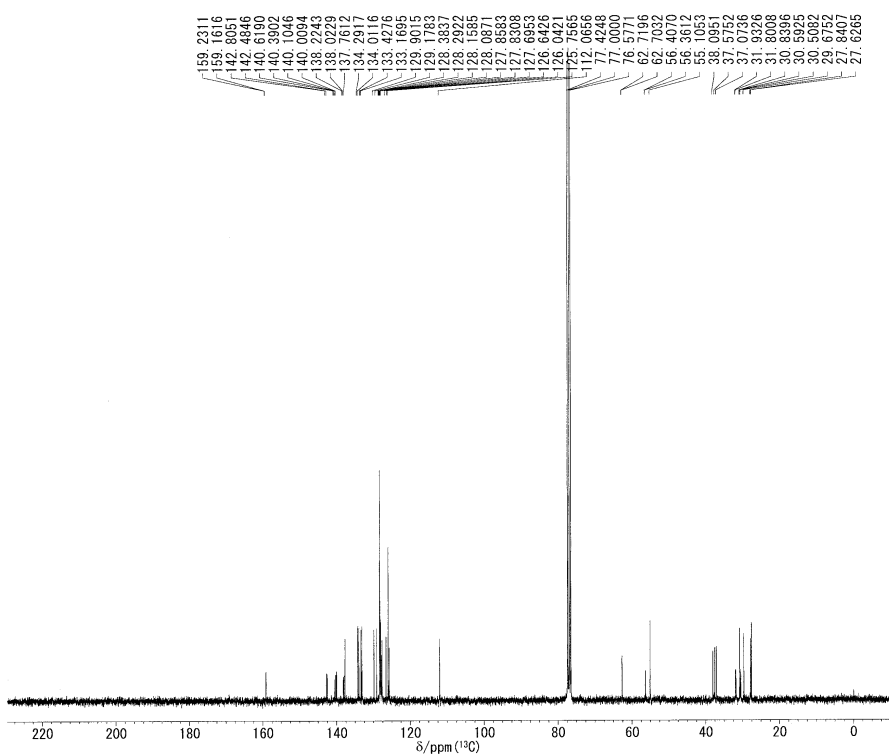
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OBFRQ 121.54 MH
z
OBSET 0.0 kHz
OBFIN 10005.0 Hz
POINT 32768
PRGQU 38535.64 Hz
SCANS 8
ACQTM 0.8503
s
PD 8.0 s
PW 0.0  $\mu$ s
IRNUC OFF
PROBHD 5 MM BBO BB-HH-D Z-CR
D Z8284/01
INSTRUM SPECT
PULSPRG ZGPG30
GRDPRG
CTEMP 26.85  $^{\circ}$ C
SLVNT CDCL3
EXREF 158.537 p
pm
BF 0.25 Hz
WINDOW Exponential
RGAIN 13004
Operator _____
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¹H, ¹³C and ³¹P NMR, and chiral phase HPLC chart of (±)-**3a**



DFILE C:\KYOUSEI\6NMR\Y06M1N020
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DATEM 18/Jul/2016 10:13:16
COMNT

ORNIC ¹H
EXMOD ZG30
OBFRQ 300.23 MH
Z
OBSET 0.0 kHz
OBFIN 10004.57 Hz
POINT 16384
FREQU 3612.717 H
SCANS 8
ACQTM 4.5351
PD 1.0 s
PWI 9.0 μs
IRNUC OFF
PROHD 5 MM BBO BB-1H-D Z-GR
D 28284/01
INSTRUM SPECT
PULSPRG ZG30
GRDPRG
CTEMP 21.01 °C
SLVNT CDCl₃
EXREF 0.0 ppm
BF 0.25 Hz
WINDOW Exponential
RGAIN 181
Operator

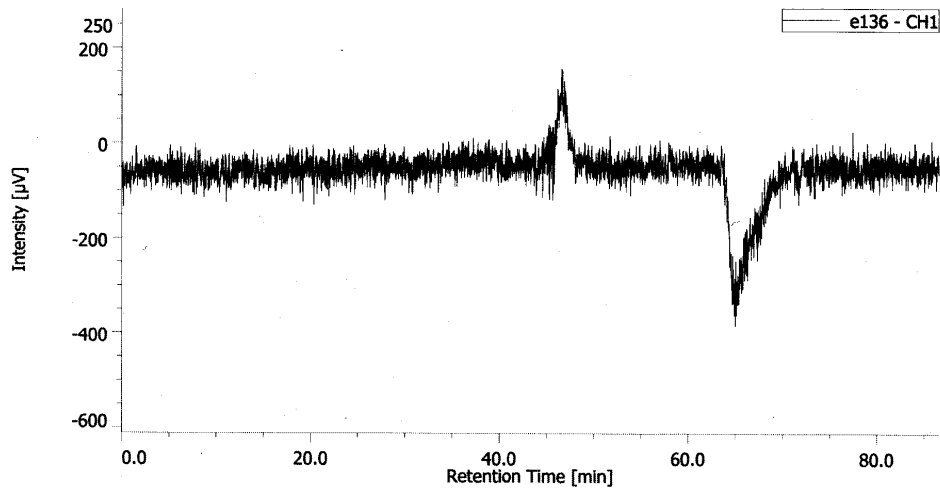
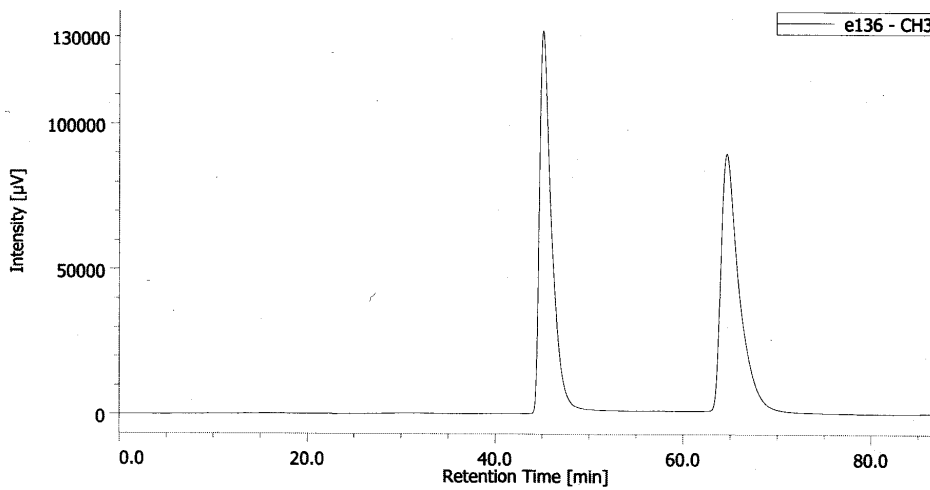
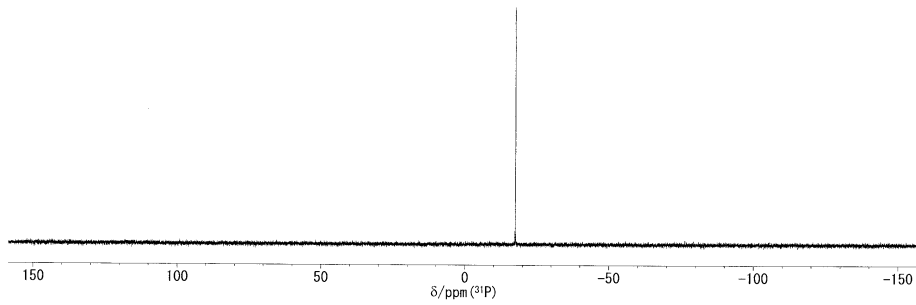


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COMNT

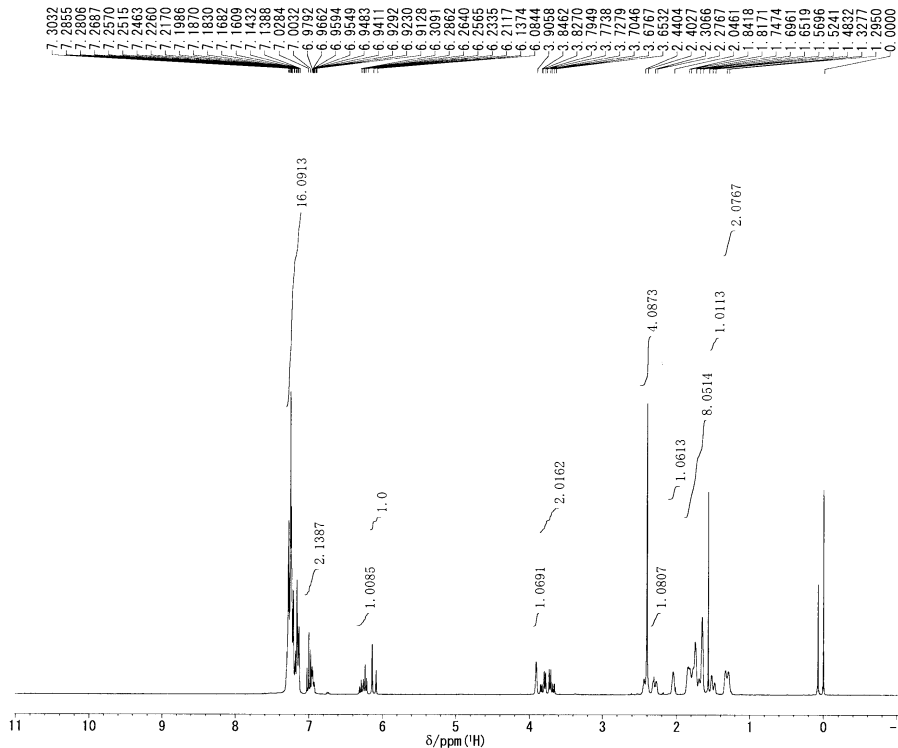
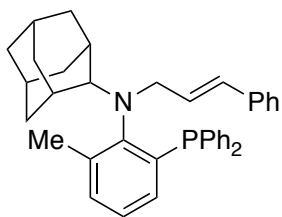
ORNIC ¹³C
EXMOD ZGPC30
OBFRQ 75.49 MH
Z
OBSET 0.0 kHz
OBFIN 10003.06 Hz
POINT 32768
FREQU 18115.94 Hz
SCANS 1024
ACQTM 1.8088
PD 2.0 s
PWI 0.0 μs
IRNUC OFF
PROHD 5 MM BBO BB-1H-D Z-GR
D 28284/01
INSTRUM SPECT
PULSPRG ZGPC30
GRDPRG
CTEMP 21.01 °C
SLVNT CDCl₃
EXREF 77.0 ppm
BF 0.25 Hz
WINDOW Exponential
RGAIN 2048
Operator

17.5504

DFILE C:\KYOUSEI\6NMR\06M1\020
15V20484P3Y3PDATA\1Y11
DATIM 14/Oct/2015 08:57:45
COMNT
OBNUC ³¹P
EXMOD ZPG30
OBFRQ 121.54 MH
Z
ORSET 0.0 kHz
ORPIN 10005.0 Hz
PGINT 32768
FREQU 38535.64 Hz
SCANS 8
ACQTM 0.8503
S
PD 8.0 s
PW1 0.0 μs
TRNUC OFF
PROBHD 5 MM BBO BB-1H-D Z-GR
D Z8284/01
INSTRUM SPECT
PULPROG ZPG30
GRDPROG
CTEMP 26.85 °C
SOLVT CDCl₃
EXREF 158.537 p
pm
BF 0.25 Hz
WINDOW Exponential
RGAIN 5161
Operator _____

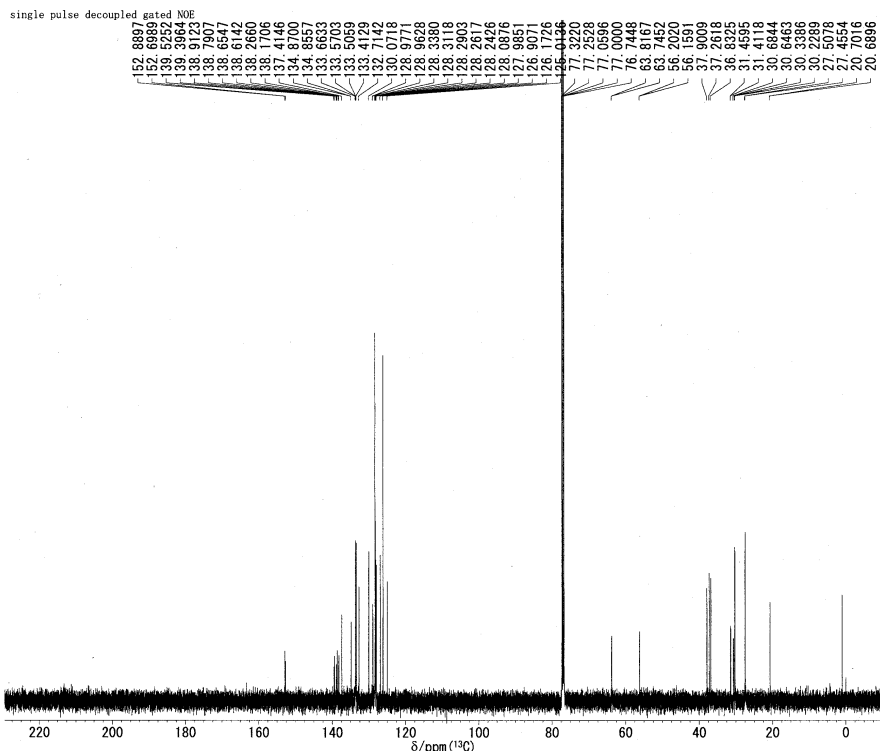


¹H, ¹³C and ³¹P NMR, and chiral phase HPLC chart of (±)-**3b**



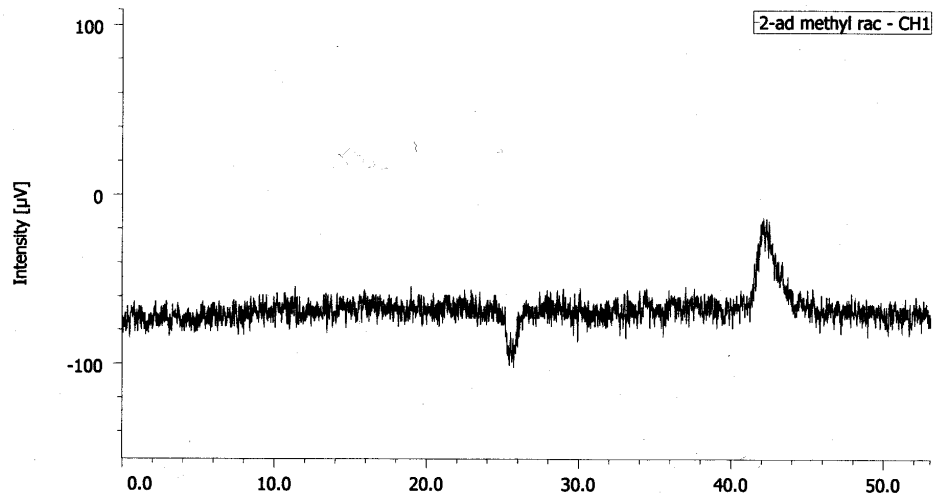
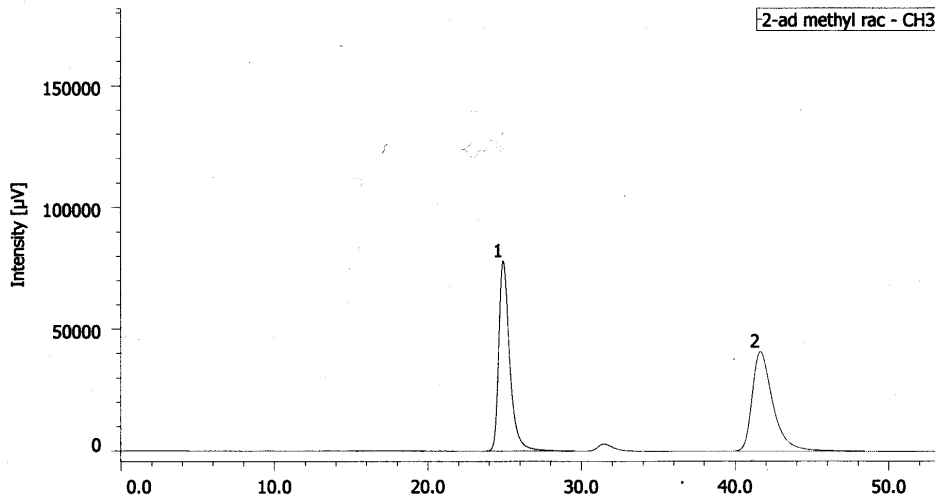
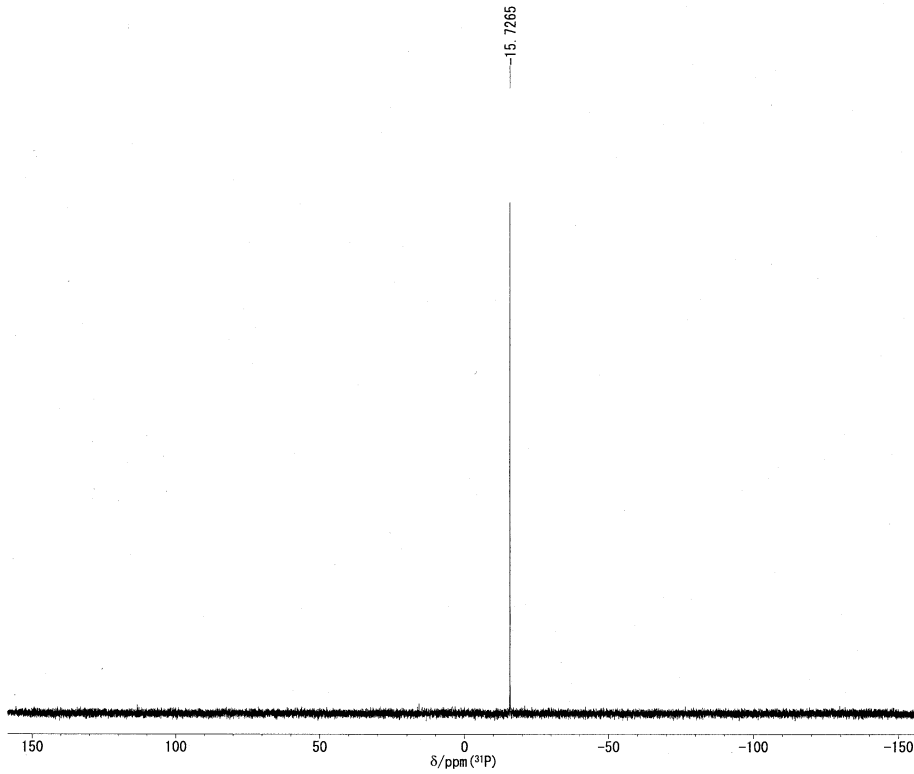
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DATIM 05/Jan/2015 14:09:13
COMNT

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EXMOD ZG30
OBFRQ 300.23 MHz
ORSET 0.0 kHz
OBFIN 10004.57 Hz
POINT 16384
FREQU 3612.717 Hz
SCANS 8
ACQTM 4.5351 s
PD 1.0 s
PWI 9.0 ps
IRNUC OFF
PROBHD 5 MM BBO BB-1H-D Z-GRD
28294/01
INSTRUM SPECT
PULSPRG ZG30
GRDPRG
CTEMP 26.85 °C
SLVNT CDCl₃
EXREF 11.0049 p
BF pm
WINDOW 0.25 Hz
RGAIN Exponential
362
Operator

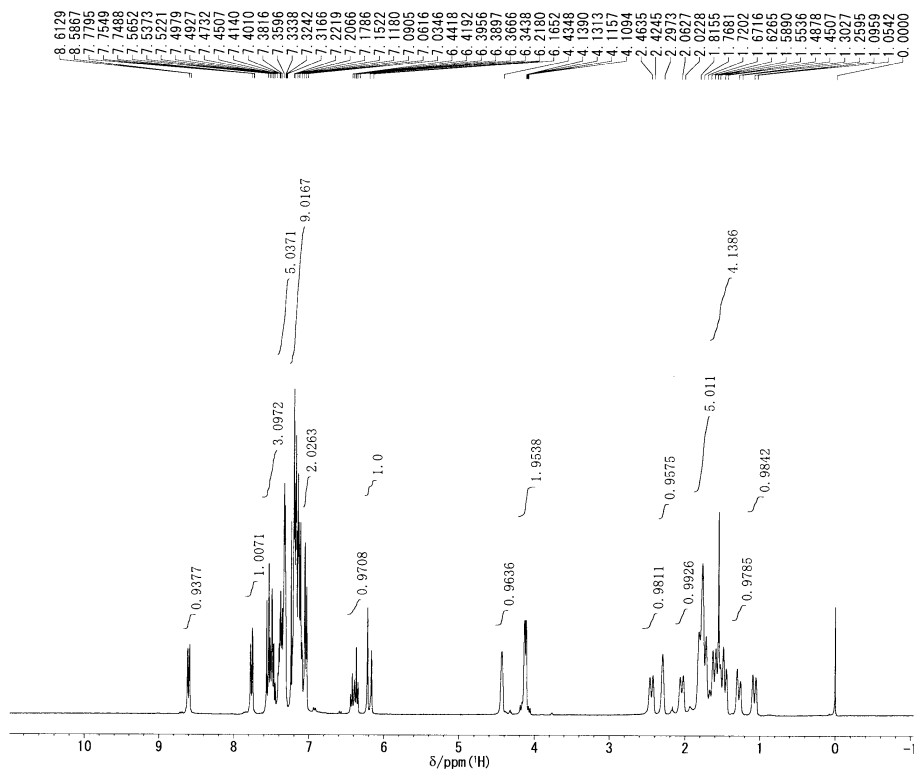
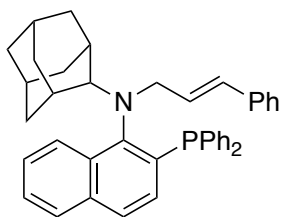


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esktop\20150226_ebi_exp62_
Carbon-1-1.jaf
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COMNT single pulse decoupled gat
ed NOE
ORNUC ¹³C
EXMOD carbon_1jaf
OBFRQ 125.77 MH
ORSET -5.0 kHz
OBFIN 301.0403
Hz
POINT 131072 (ZeroF
111::x4)
FREQU 39308.18 Hz
SCANS 256
ACQTM 0.8336
s
PD 2.0 s
PWI 3.5333
ms
IRNUC ¹H
PROBHD JNM-ECA500
INSTRUM
PULSPRG
GRDPRG
CTEMP 20.4 °C
SLVNT CHLOROFORM-d
EXREF 77.0 ppm
BF 0.25 Hz
WINDOW Exponential
RGAIN 50
Operator

DFILE C:\KYOUSEI\6MRV06MINO20
 14\19920PY1\YDATA\Y111
 DATIM 05/Jan/2015 14:14:32
 COMNT
 OBNUC ³¹P
 EXMOD ZGPC30
 OBFRQ 121.54 MH
 Z
 OBSET 0.0 kHz
 OBFIN 10005.0 Hz
 POINT 32768
 FREQU 38535.64 Hz
 SCANS 8
 ACQTM 0.8503
 S
 PD 8.0 s
 PW1 0.0 μ s
 TRNUC OFF
 PROBHD 5 MM BBO BB-1H-D Z-CR
 D Z8284/01
 INSTRUM SPECT
 PULSPRG ZGPC30
 GRDPROG
 CTEMP 26.85 $^{\circ}$ C
 SLVNT CDCL₃
 EXREF 158.537 p
 pm
 BF 0.25 Hz
 WINDOW Exponential
 RGAIN 13004
 Operator _____

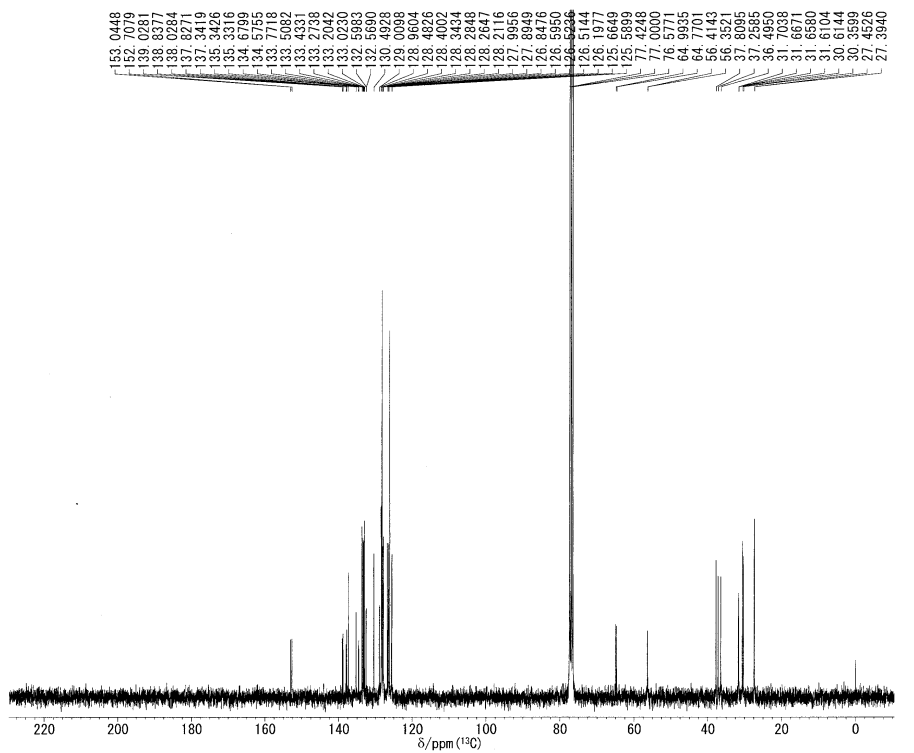


¹H, ¹³C and ³¹P NMR, and chiral phase HPLC chart of (±)-3c



DFILE: C:\KYOUSEI\6NMR\6MINO201
 5V20281H1VPDATA\1V11
 DATIM: 18/Jun/2015 17:59:43
 COMNT:

OBNUC: ¹H
 EXMOD: ZG30
 OBFREQ: 300.23 MHz
 OBSSET: 0.0 kHz
 OBSFIN: 10004.57 Hz
 POINT: 16384
 FREQU: 3612.717 Hz
 SCANS: 8
 ACQTM: 4.5351 s
 PD: 1.0 s
 PFI: 9.0 μs
 IRNUC: OFF
 PROBD: 5 MM BBO BB-1H-D Z-GR
 D 28284/01
 INSTRUM: SPECT
 PULSPRG: ZG30
 GRDPRG: SPECT
 CTEMP: 26.85 °C
 SLVNT: CDCl₃
 EXREF: 0.0 ppm
 BF: 0.25 Hz
 WINDOW: Exponential
 RGAIN: 228
 Operator: _____



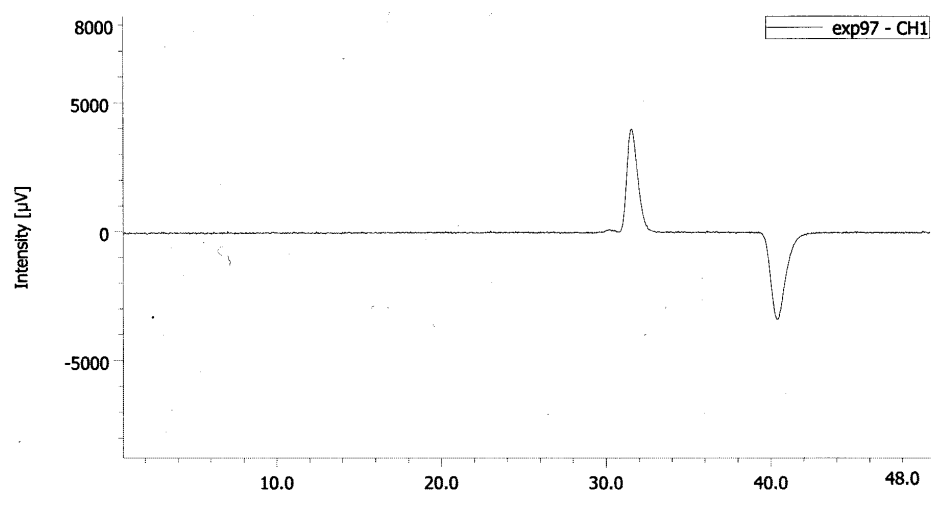
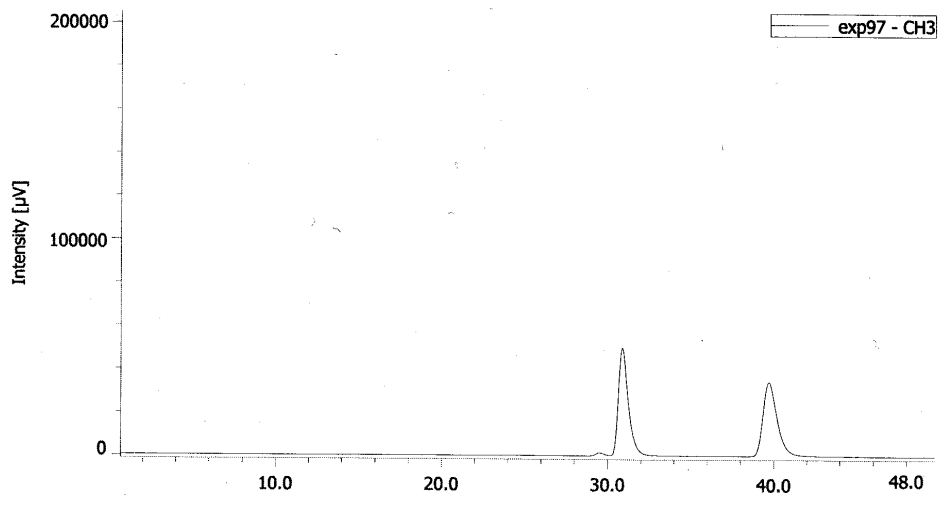
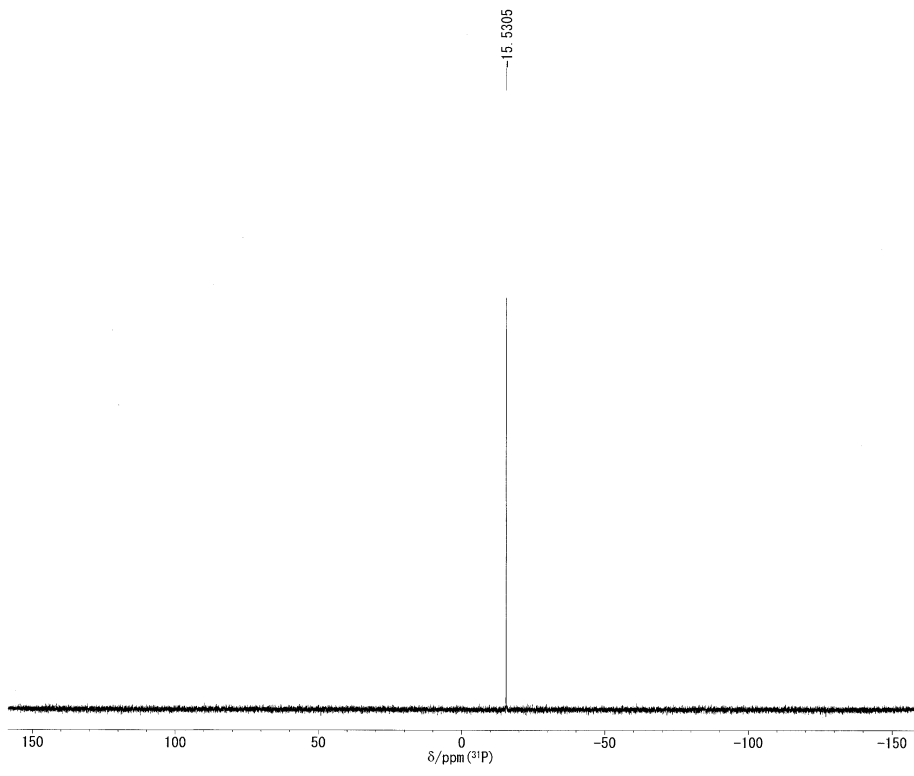
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 DATIM: 22/Sep/2016 20:58:43
 COMNT:

OBNUC: ¹³C
 EXMOD: ZGPG30
 OBFREQ: 75.49 MH
 OBSSET: 0.0 kHz
 OBSFIN: 10003.06 Hz
 POINT: 32768
 FREQU: 18115.94 Hz
 SCANS: 2048
 ACQTM: 1.8088 s
 PD: 2.0 s
 PFI: 0.0 μs
 IRNUC: OFF
 PROBD: 5 MM BBO BB-1H-D Z-GR
 D 28284/01
 INSTRUM: SPECT
 PULSPRG: ZGPG30
 GRDPRG: SPECT
 CTEMP: 20.01 °C
 SLVNT: CDCl₃
 EXREF: 77.0 ppm
 BF: 0.25 Hz
 WINDOW: Exponential
 RGAIN: 1626
 Operator: _____

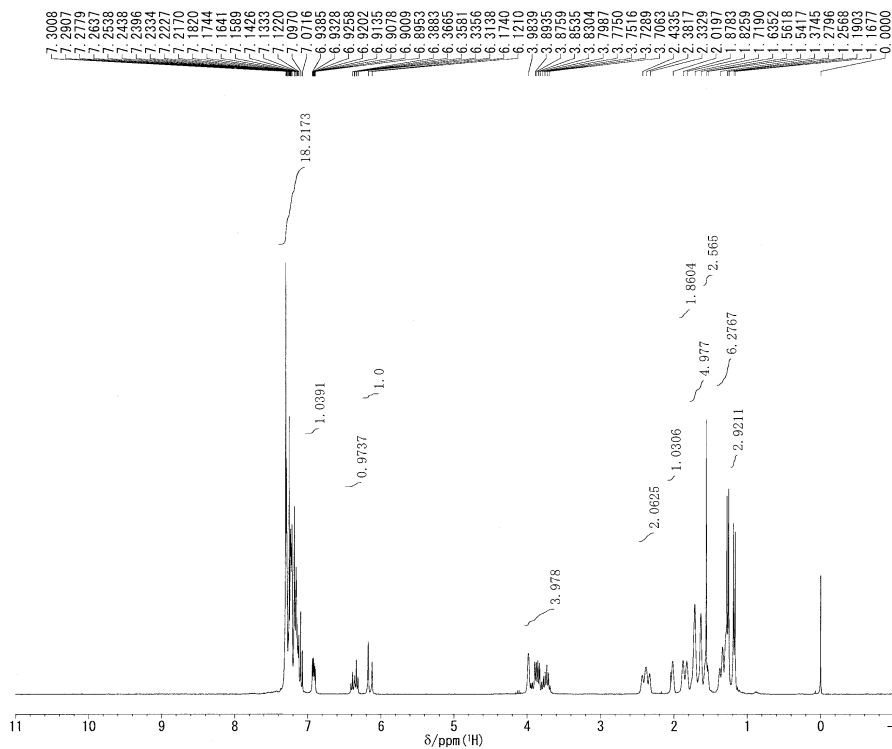
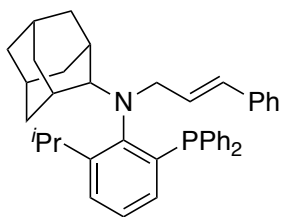
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15V20266PV1\PDAT\TAYIY11
DATIM 12/Jan/2015 20:14:15
COMNT
ORNUC 31P
EXMOD ZGPG30
ORFRQ 121.54 MH
z
ORSET 0.0 kHz
OBFIN 10005.0 Hz
POINT 32768
FREQU 38535.64 Hz
SCANS 8
ACQTM 0.8503
s
PD 8.0 s
PW 0.0  $\mu$ s
FMUC OFF
PROBHD 5 MM BBO BB-1H-D Z-GR
D 28284/01
INSTRM SPECT
PULSFRG ZGPG30
GRDPROG
CTEMP 26.85  $^{\circ}$ C
SLVNT CDCL3
EXREF 158.537 p
pm
RF 0.25 Hz
WINDOW Exponential
RGAIN 13004
Operator _____

```



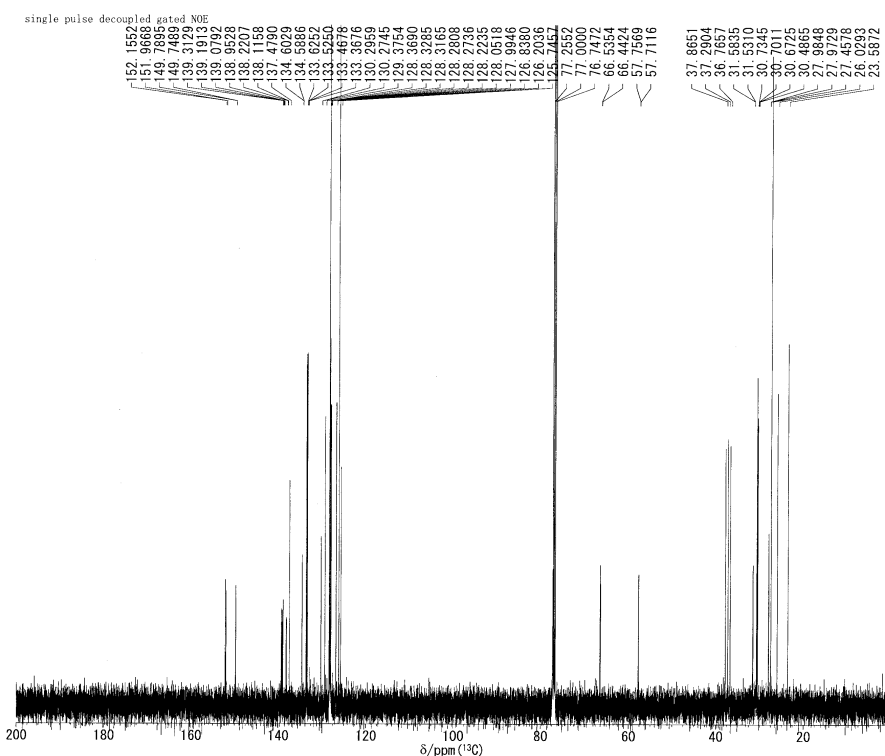
¹H, ¹³C and ³¹P NMR, and chiral phase HPLC chart of (±)-**3d**



```

DFILE C:\YKHOUSE\16NMR\06MIN020
DATE 16V21500HZ29PPATAY1V11
DATIM 31/Oct/2016 18:59:40
COMNT

OBNUC 1H
EXMOD ZG30
OBFRQ 300.23 MH
z
OBSET 0.0 kHz
OBFIN 10004.57 Hz
POINT 16384
FREQU 3612.717 H
z
SCANS 16
ACQTM 4.5351
s
PD 1.0 s
PW1 9.0 us
IRNUC OFF
PROBHD 5 MM BBO BB-1H-D Z-GR
D 28284/01
INSTRUM SPECT
PULSPRG ZG30
GRDPRG
CTEMP 26.85 °C
SLVNT CDCl3
EXREF 0.0 ppm
BF 0.25 Hz
WINDOW Exponential
RGAIN 362
Operator
    
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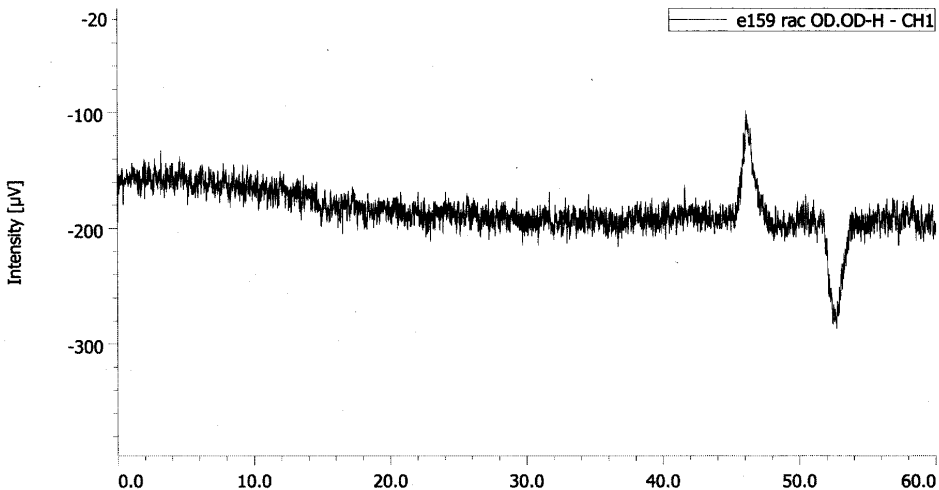
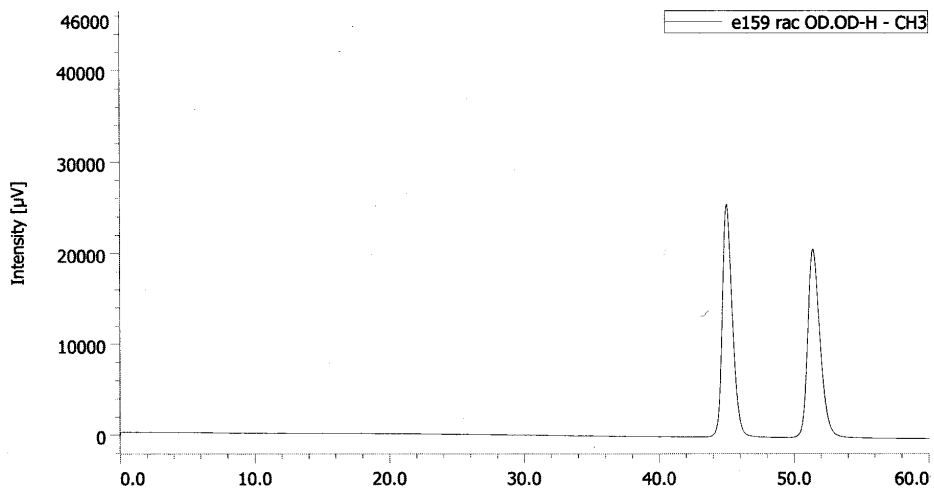
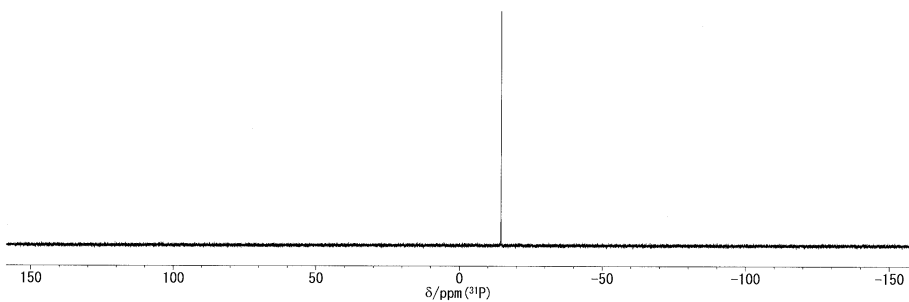


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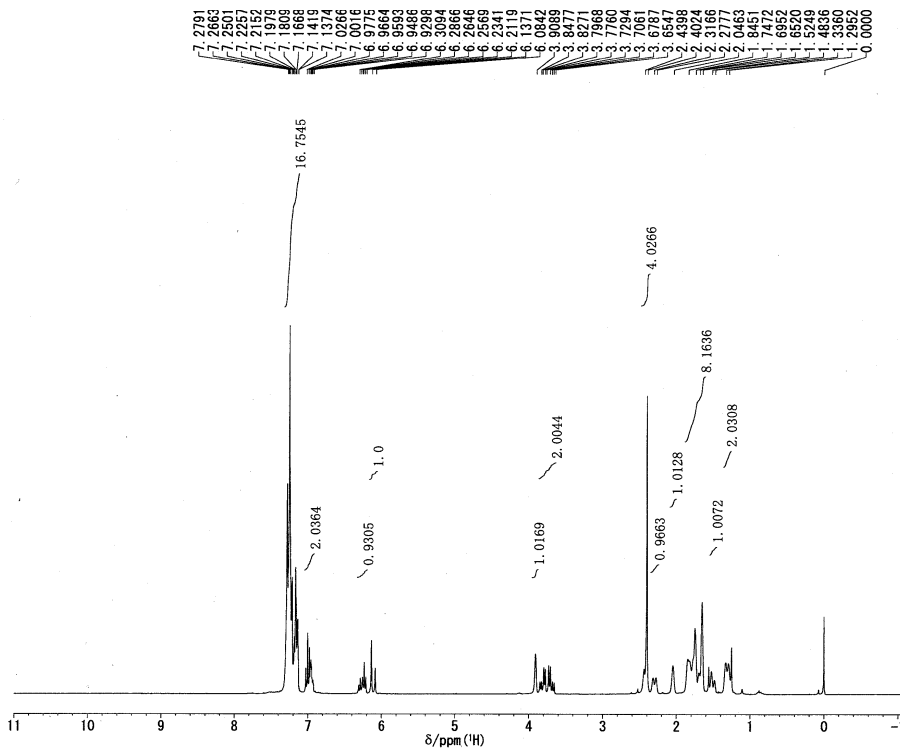
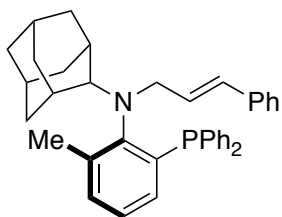
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esktop\20161111\ebisawa e1
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COMNT single pulse decoupled gat
ed NOE
OBNUC 13C
EXMOD carbon_jxp
OBFRQ 125.77 MH
z
OBSET -5.0 kHz
OBFIN 301.0503
Hz
POINT 131072 (ZeroF
i11:x4)
FREQU 39308.18 Hz
SCANS 1024
ACQTM 0.8336
s
PD 2.0 s
PW1 3.7833
ms
IRNUC 1H
PROBHD JNM-ECA500
INSTRUM
PULSPRG
GRDPRG
CTEMP 20.6 °C
SLVNT CHLOROFORM-D
EXREF 77.0 ppm
BF 0.25 Hz
WINDOW Exponential
RGAIN 50
Operator
    
```

14.5242

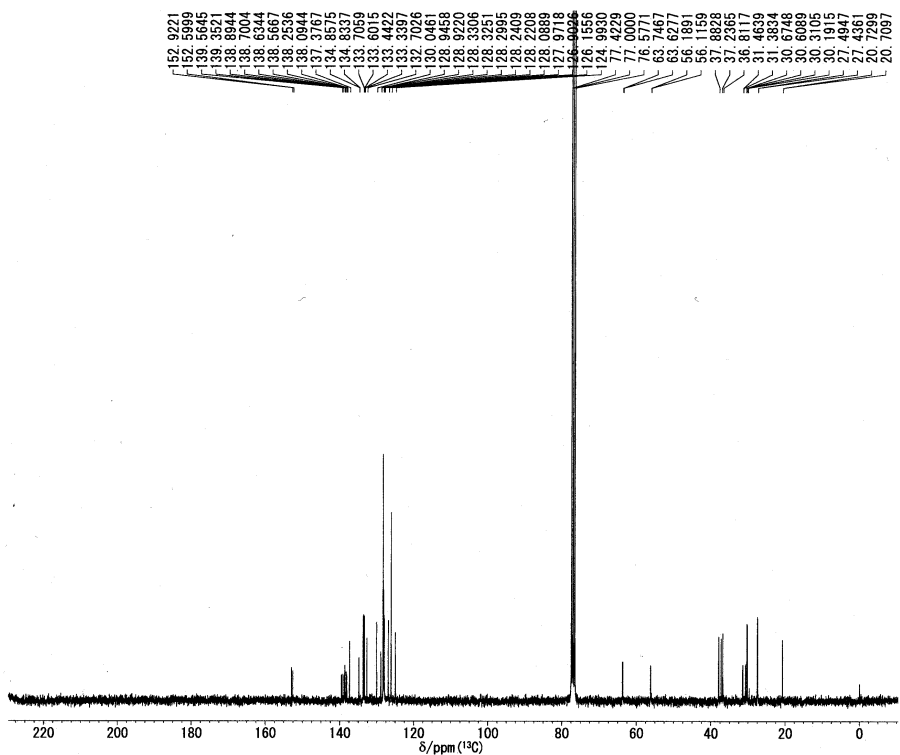
D:\FILE C:\KYOU\SE16\NMR\VOGMINO20
16Y21500PY2Y\DATA\Y111
DATE\TIME 31/Oct/2016 19:01:45
COMENT
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EXMOD ZFGP30
OBFRQ 121.54 MH
Z
OBSET 0.0 kHz
OBFIN 10000.0 Hz
POINT 32768
FREQU 38538.64 Hz
SCANS 8
ACQTM 0.8503
s
PD 8.0 s
PW1 0.0 μs
IRNUC OFF
PROBHD 5 MM BBO BB-1H-D Z-GR
D 28284/01
INSTRUM SPECT
PULSPRG ZFGP30
GRDPRG
CTEMP 26.85 °C
SLVNT CDCl₃
EXREF 158.537 p
pm
BF 0.25 Hz
WINDOW Exponential
RGAIN 9195
Operator _____



¹H, ¹³C and ³¹P NMR, and chiral phase HPLC chart of (*S*)-(-)-**3b**



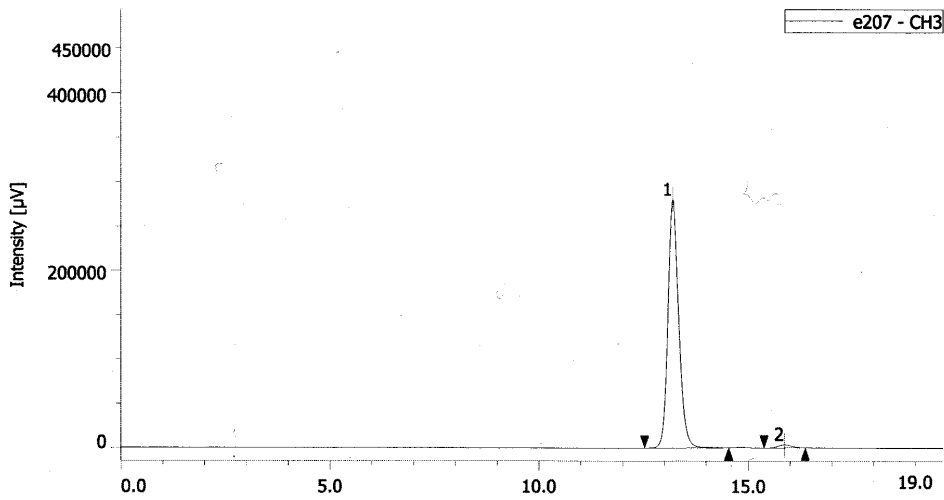
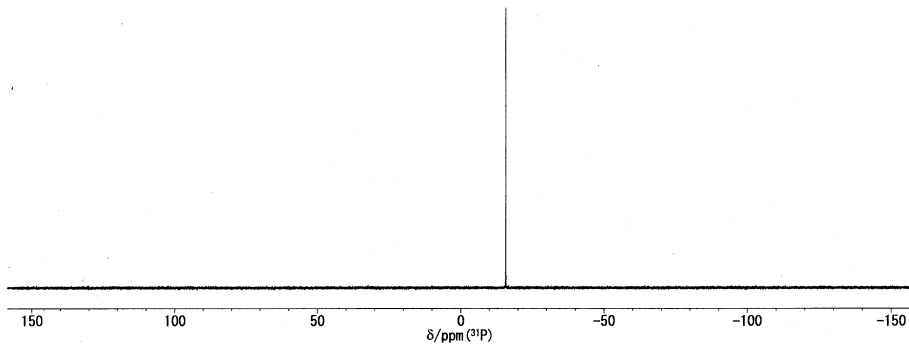
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16V21718V3PDATA\1V11
DATIM 17/Jan/2017 22:58:07
COMNT
OBRUC ¹H
EXMOD ZG30
OBFREQ 300.23 MH
z
OBSET 0.0 kHz
OBFIN 10004.57 Hz
POINT 16384
FREQU 3612.717 H
z
SCANS 16
ACQTM 4.5351
s
PD 1.0 s
PWI 9.0 μs
IRNUC OFF
PROBHD 5 MM BBO BB-1H-D Z-GR
D 28284/01
INSTRUM SPECT
PULSPRG ZG30
GRDPRG
CTEMP 26.85 °C
SLVNT CDCl₃
EXREF 0.0 ppm
BF 0.25 Hz
WINDOW Exponential
RGAIN 256
Operator _____



DFILE C:\KYOUSEI6\NMR\VO6MINO20
16V21718V3PDATA\1V11
DATIM 18/Jan/2017 01:11:40
COMNT
OBRUC ¹³C
EXMOD ZGPG30
OBFREQ 75.49 MH
z
OBSET 0.0 kHz
OBFIN 10003.06 Hz
POINT 32768
FREQU 18115.94 Hz
SCANS 2048
ACQTM 1.8088
s
PD 2.0 s
PWI 0.0 μs
IRNUC OFF
PROBHD 5 MM BBO BB-1H-D Z-GR
D 28284/01
INSTRUM SPECT
PULSPRG ZGPG30
GRDPRG
CTEMP 26.85 °C
SLVNT CDCl₃
EXREF 77.0 ppm
BF 0.25 Hz
WINDOW Exponential
RGAIN 4598
Operator _____

15.6926

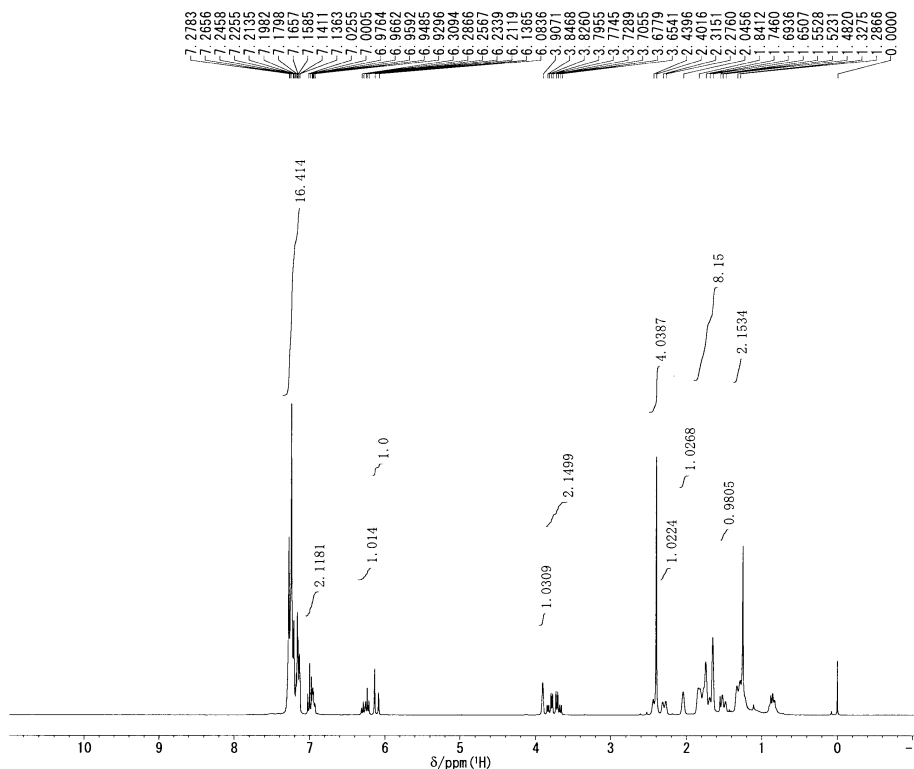
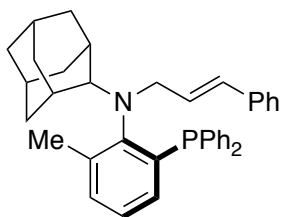
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16\21718PW1\PDATAV1\111
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COMNT
ORNUC ³¹P
EXMOD ZGPC30
ORPRO 121.54 MH
Z
OBSET 0.0 kHz
OBFIN 10005.0 Hz
POINT 32768
FREQU 38535.64 Hz
SCANS 8
ACQTH 0.8503
S
PD 8.0 s
PWI 0.0 μ s
TRNUC OFF
PROBHD 5 MM BBO BB-1H-D Z-GR
D 28284/01
INSTRUM SPECT
PULSPRG ZGPC30
GRDPROG
CTEMP 26.85 $^{\circ}$ C
SUNVT CDCl₃
EKREF 158.537 p
ppm
BF 0.25 Hz
WINDOW Exponential
RGAIN 9195
Operator _____



peak name area area%

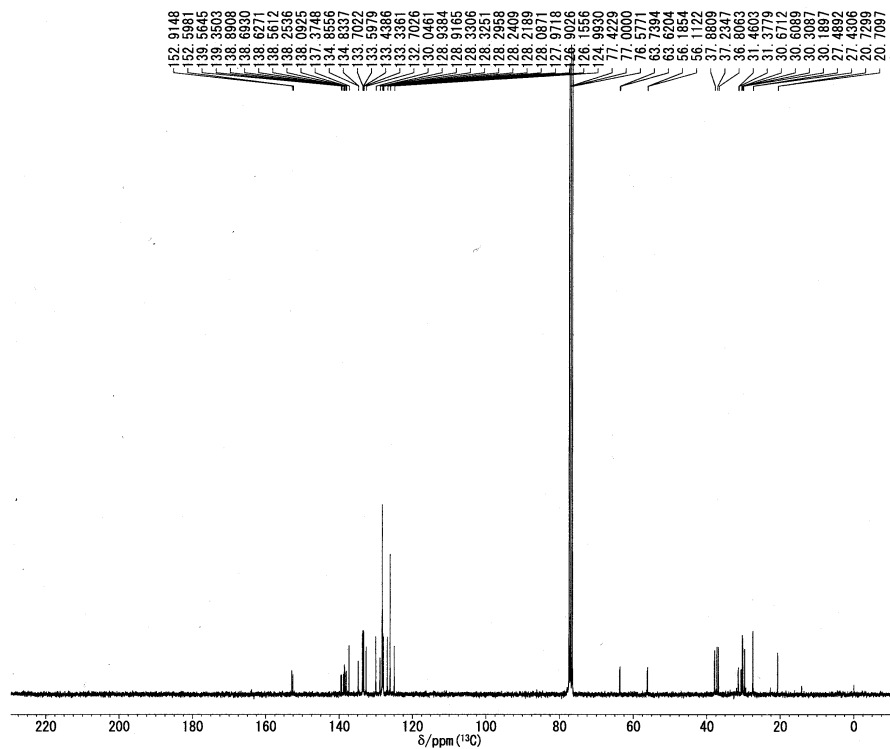
peak name	area	area%
Unknown 3	13.200	5084478 98.514
Unknown 3	15.867	76710 1.486

^1H , ^{13}C and ^{31}P NMR, and chiral phase HPLC chart of (*R*)-(+)-**3b**



DFILE C:\KYOUSEI6\NMR\06MIN0201
 6V21718H4VPDATA1V111
 DATIM 18/Jan/2017 01:25:43
 COMNT

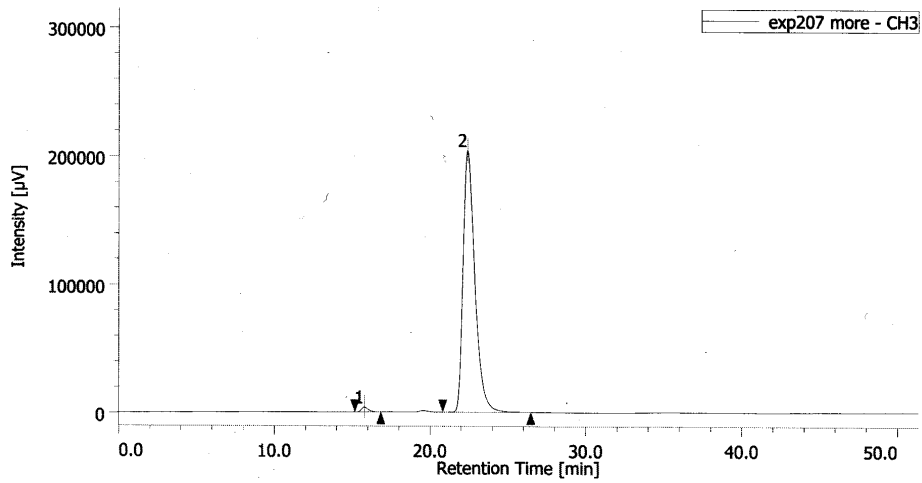
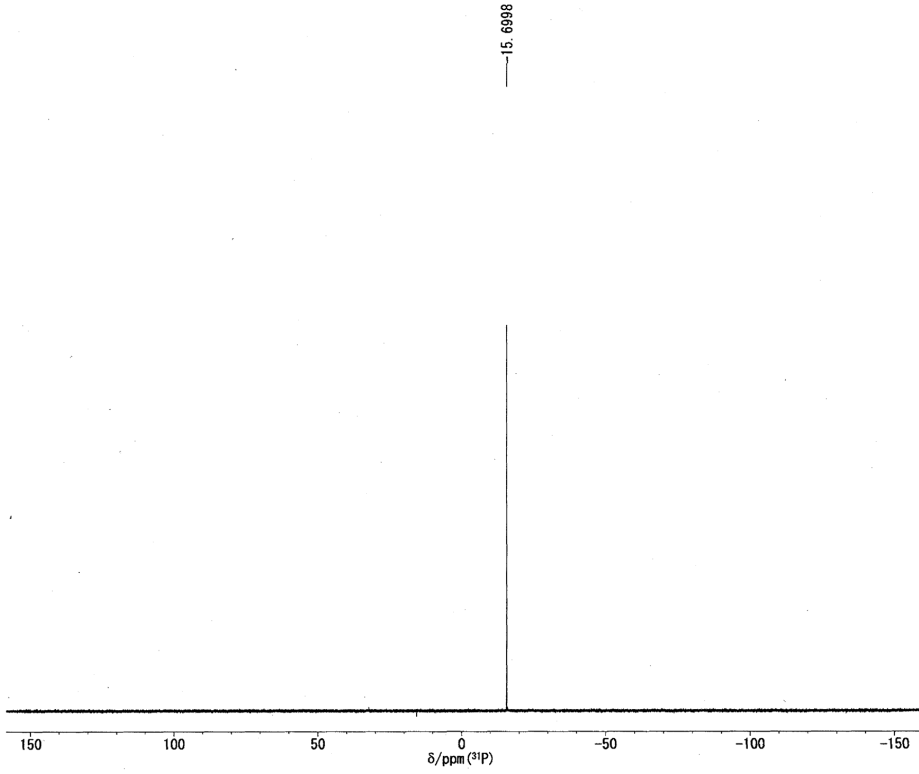
OBNUC ^1H
 EXMOD ZG30
 OBFRQ 300.23 MHz
 OBSET 0.0 kHz
 OBFIN 10004.57 Hz
 POINT 16384
 FREQU 3612.717 Hz
 SCANS 16
 ACQTM 4.5351 s
 PD 1.0 s
 FW 9.0 μs
 IRNUC OFF
 PROBHD 5 MM BBO BB-1H-D Z-GRD
 78284/01
 INSTRUM SPECT
 PULSPRG ZG30
 GRDPRG
 CTEMP 26.85 $^{\circ}\text{C}$
 SLVNT CDCl₃
 EXREF 0.0 ppm
 BF 0.25 Hz
 WINDOW Exponential
 RGAIN 256
 Operator



DFILE C:\KYOUSEI6\NMR\06MIN020
 16V21718C4VPDATA1V111
 DATIM 18/Jan/2017 03:38:44
 COMNT

OBNUC ^{13}C
 EXMOD ZPG30
 OBFRQ 75.49 MH
 OBSET 0.0 kHz
 OBFIN 10003.06 Hz
 POINT 32768
 FREQU 18115.94 Hz
 SCANS 2048
 ACQTM 1.8088 s
 PD 2.0 s
 FW 0.0 μs
 IRNUC OFF
 PROBHD 5 MM BBO BB-1H-D Z-GRD
 78284/01
 INSTRUM SPECT
 PULSPRG ZPG30
 GRDPRG
 CTEMP 26.85 $^{\circ}\text{C}$
 SLVNT CDCl₃
 EXREF 77.0 ppm
 BF 0.25 Hz
 WINDOW Exponential
 RGAIN 4598
 Operator

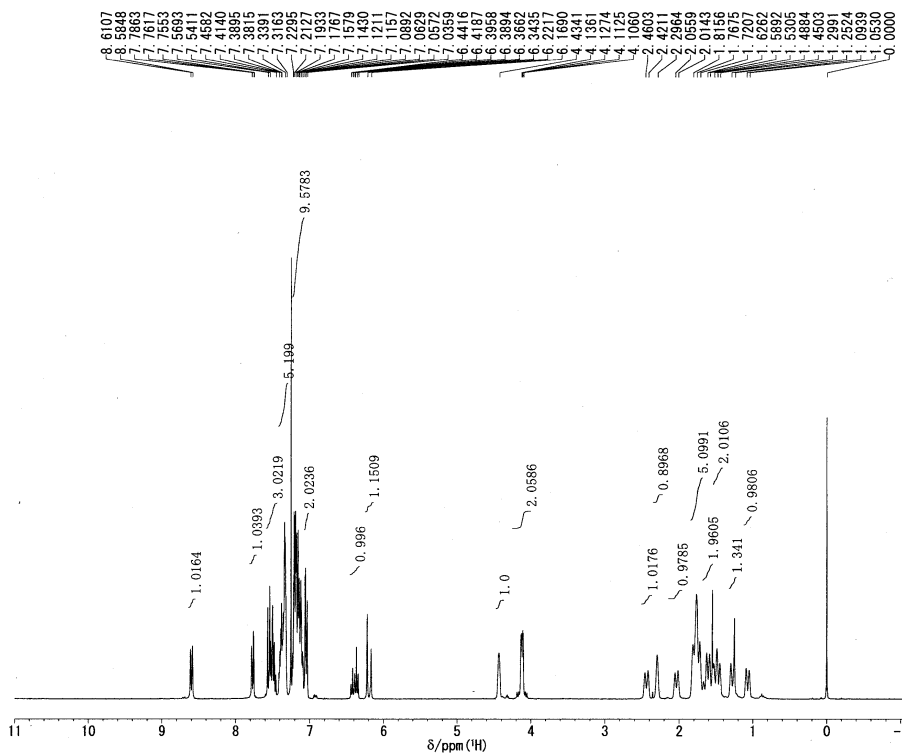
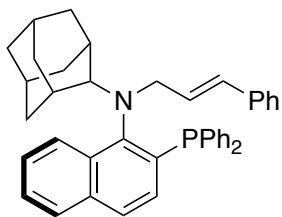
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 16\21718\F2\DATA\1911
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 COMNT
 OBNUC ¹H
 EXMOD ZGPC30
 OBFRQ 121.54 MH
 Z
 OBSET 0.0 kHz
 OBFIN 10005.0 Hz
 POINT 32768
 FREQU 38535.64 Hz
 SCANS 8
 ACQTM 0.8503
 S
 PD 8.0 s
 PFI 0.0 μs
 TRNUC OFF
 PROHD 5 MM BBO BB-IH-D Z-GR
 D Z8284/01
 INSTRW SPECT
 PULSPRG ZGPC30
 GRDPRG
 CTEMP 26.85 °C
 SLWT CDCL₄
 EXREF 158.537 p
 BF ^{pm} 0.25 Hz
 WINDOW Exponential
 RGAIN 9195
 Operator _____



peak name area area%

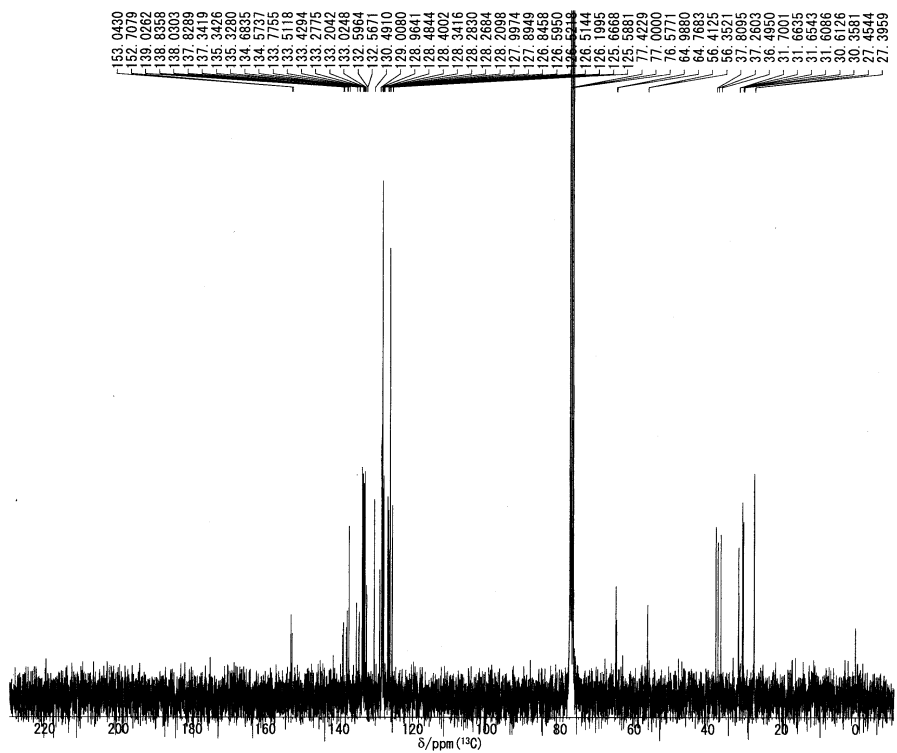
peak name	area	area%
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Unknown 3	22.392	10589311 98.957

¹H, ¹³C and ³¹P NMR, and chiral phase HPLC chart of (S)-(+)-3c



DFILE C:\KYOUSEI6\NMRY06\MINO20
16V21640CV1VPDATA\1V11
DATIM 11/Dec/2016 21:32:38
COMNT

OBNUC ¹H
EXMOD ZG30
OBRFQ 300.23 MH
Z
ORSET 0.0 kHz
OBFIN 10004.57 Hz
POINT 16384
FREQU 3612.717 H
Z
SCANS 16
ACQTM 4.5351
S
PD 1.0 s
PWI 9.0 μs
IRNUC OFF
PROBHD 5 MM BBO BB-1H-D Z-GR
D Z8284/01
INSTRUM SPECT
PULSPRG ZG30
GRDPRG
CTEMP 26.85 °C
SLVNT CDCL₃
EXREF 0.0 ppm
BF 0.25 Hz
WINDOW Exponential
RGAIN 486
Operator _____

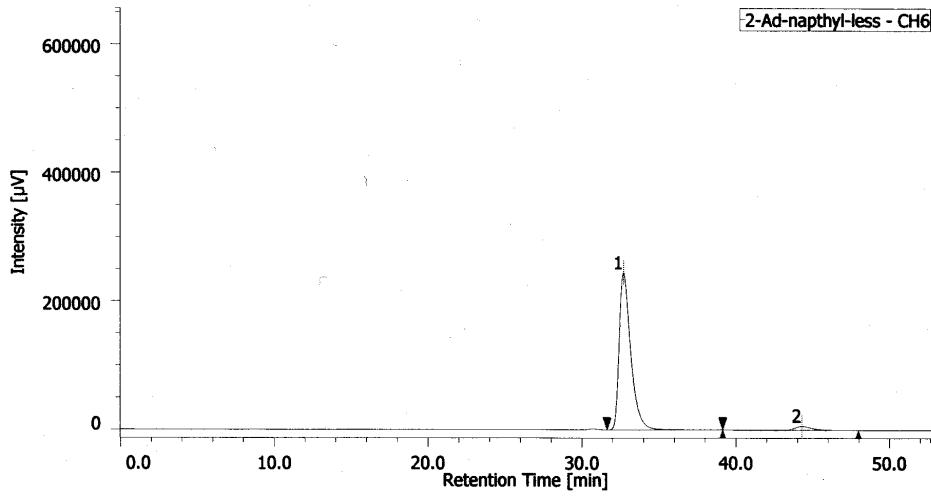
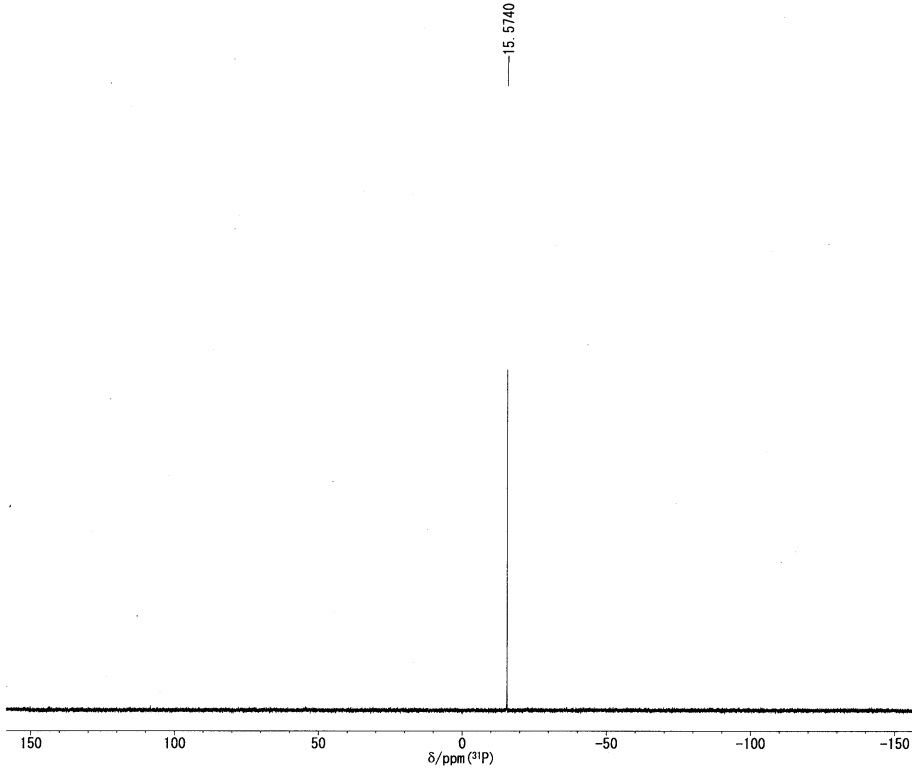


DFILE C:\KYOUSEI6\NMRY06\MINO20
16V21640CV1VPDATA\1V11
DATIM 11/Dec/2016 22:39:38
COMNT

OBNUC ¹³C
EXMOD ZGPC30
OBRFQ 75.49 MH
Z
ORSET 0.0 kHz
OBFIN 10003.06 Hz
POINT 32768
FREQU 18115.94 Hz
SCANS 1024
ACQTM 1.8088
S
PD 2.0 s
PWI 0.0 μs
IRNUC OFF
PROBHD 5 MM BBO BB-1H-D Z-GR
D Z8284/01
INSTRUM SPECT
PULSPRG ZGPC30
GRDPRG
CTEMP 26.85 °C
SLVNT CDCL₃
EXREF 77.0 ppm
BF 0.25 Hz
WINDOW Exponential
RGAIN 2299
Operator _____

D:\FILE C:\KYOUSEI6\NMR\06MINO20
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 DATIM 11/Dec/2016 22:46:47
 COMNT

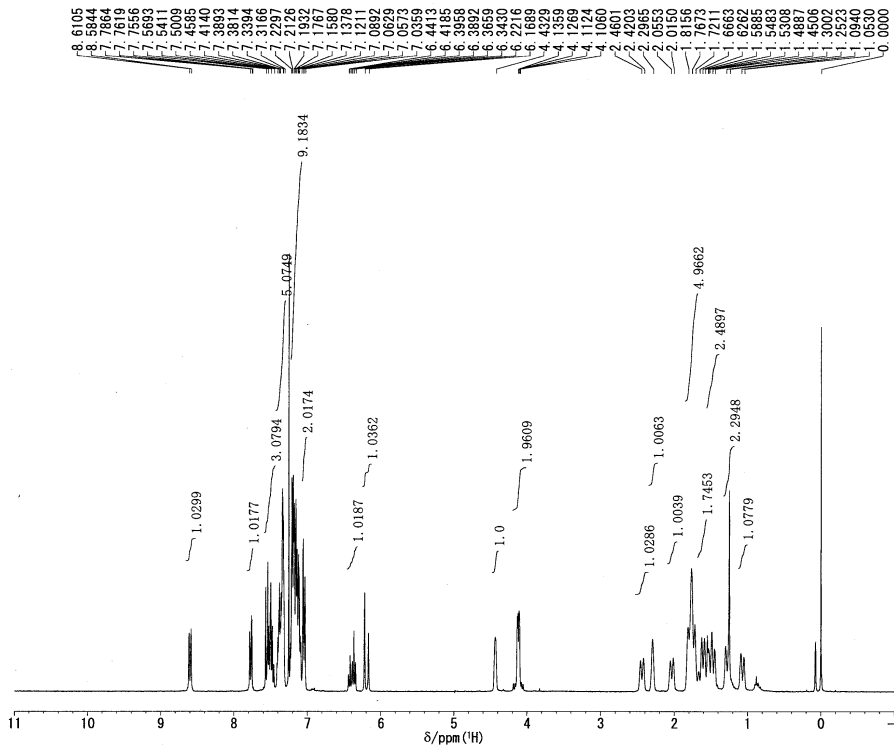
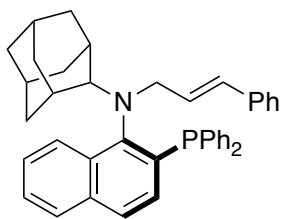
ORNUC ³¹P
 EXMOD ZGPC30
 OFPRQ 121.54 MH
 Z
 ORSET 0.0 kHz
 OFPIN 10005.0 Hz
 POINT 32768
 FREQU 38535.64 Hz
 SCANS 16
 ACQTM 0.8503
 S
 PD 8.0 s
 FWI 0.0 μs
 IRNUC OFF
 PROBHD 5 MM BBO BB-1H-D Z-GR
 D Z8284/01
 INSTRUM SPECT
 PULSPRG ZGPC30
 GRDPRG
 CTEMP 26.85 °C
 SLOWT CDCL
 EXREF 158.537 p
 BP pm
 WINDOW Exponential
 RGAIN 9195
 Operator _____



peak name area height area%

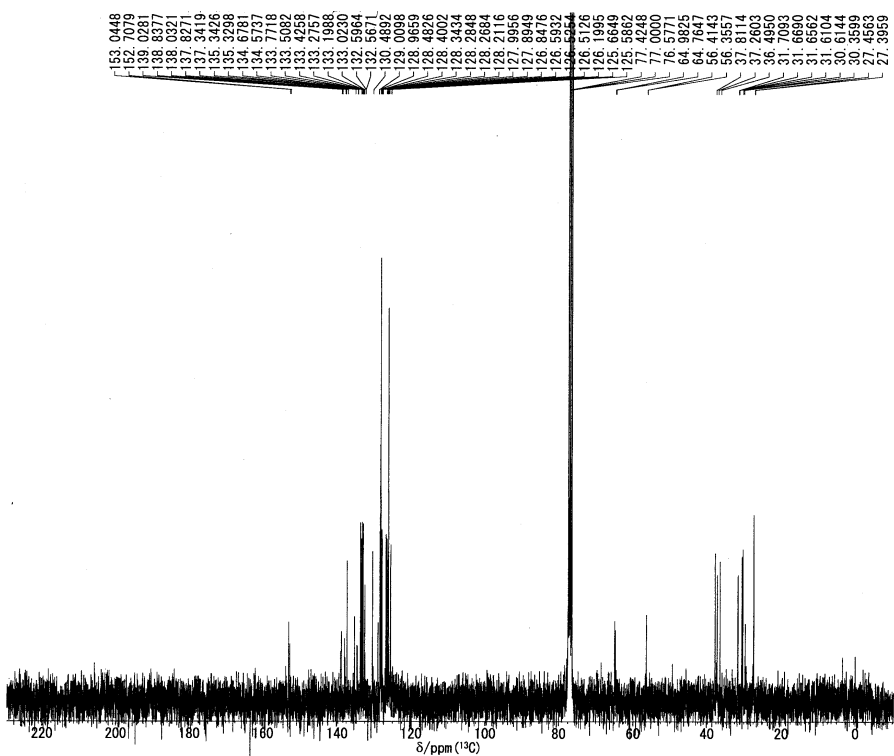
#	Retention Time [min]	Area	Height	Area%
1	32.693	12848301	244544	96.359
2	44.267	485470	6014	3.641

^1H , ^{13}C and ^{31}P NMR, and chiral phase HPLC chart of (*R*)-(-)-**3c**



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16V21640HY2VPDATA\AV1V11
DATIM 11/Dec/2016 23:46:41
COMNT

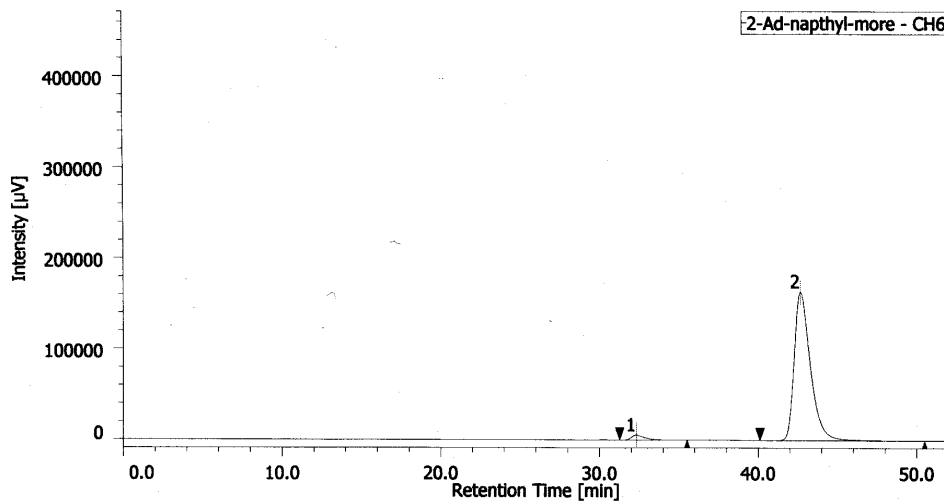
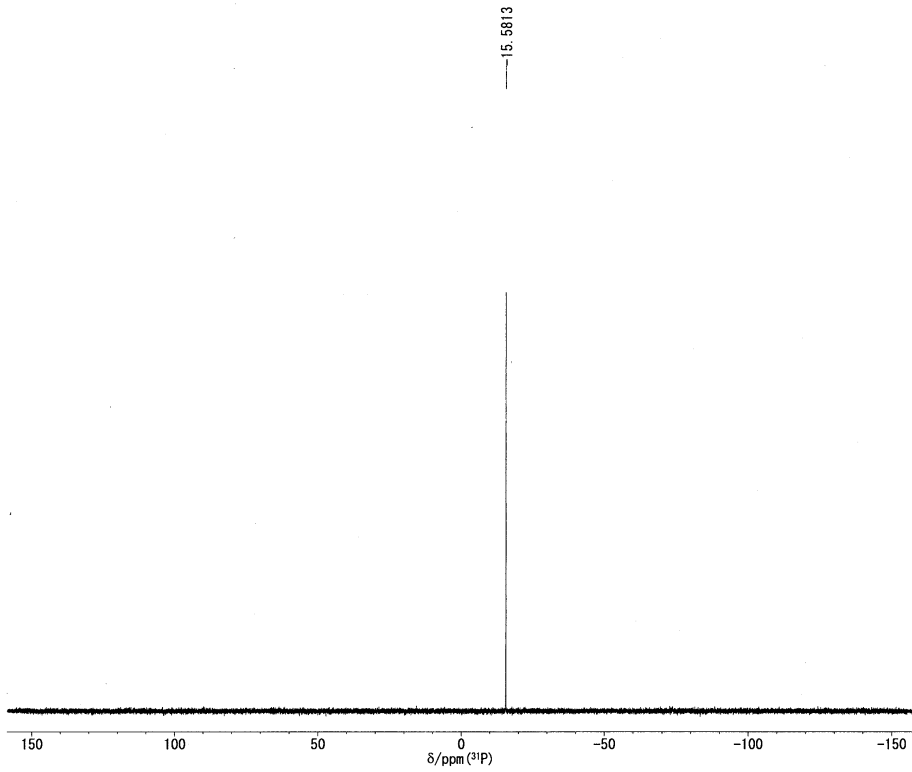
OBNUC ^1H
EXMOD ZG30
OBFREQ 300.23 MH
z
OBSET 0.0 kHz
OBFIN 10004.57 Hz
POINT 16384
FREQ 3612.717 H
z
SCANS 16
ACQTM 4.5351
s
PD 1.0 s
PFI 9.0 μs
IRNUC OFF
PROBHD 5 MM BBO BB-1H-D Z-GR
D 28284/01
INSTRM SPECT
PULSPRG ZG30
GRDPRG
CTMP 26.85 $^{\circ}\text{C}$
SLVNT CDCl_3
EXREF 0.0 ppm
BF 0.25 Hz
WINDOW Exponential
RGAIN 575
Operator _____



D:FILE C:\KYOUSEI\6NMRV06\MINO20
16V21640CY2VPDATA\AV1V11
DATIM 12/Dec/2016 00:59:31
COMNT

OBNUC ^{13}C
EXMOD ZGPC30
OBFREQ 75.49 MH
z
OBSET 0.0 kHz
OBFIN 10003.06 Hz
POINT 32768
FREQ 18115.94 Hz
SCANS 1024
ACQTM 1.8088
s
PD 2.0 s
PFI 0.0 μs
IRNUC OFF
PROBHD 5 MM BBO BB-1H-D Z-GR
D 28284/01
INSTRM SPECT
PULSPRG ZGPC30
GRDPRG
CTMP 26.85 $^{\circ}\text{C}$
SLVNT CDCl_3
EXREF 77.0 ppm
BF 0.25 Hz
WINDOW Exponential
RGAIN 5793
Operator _____

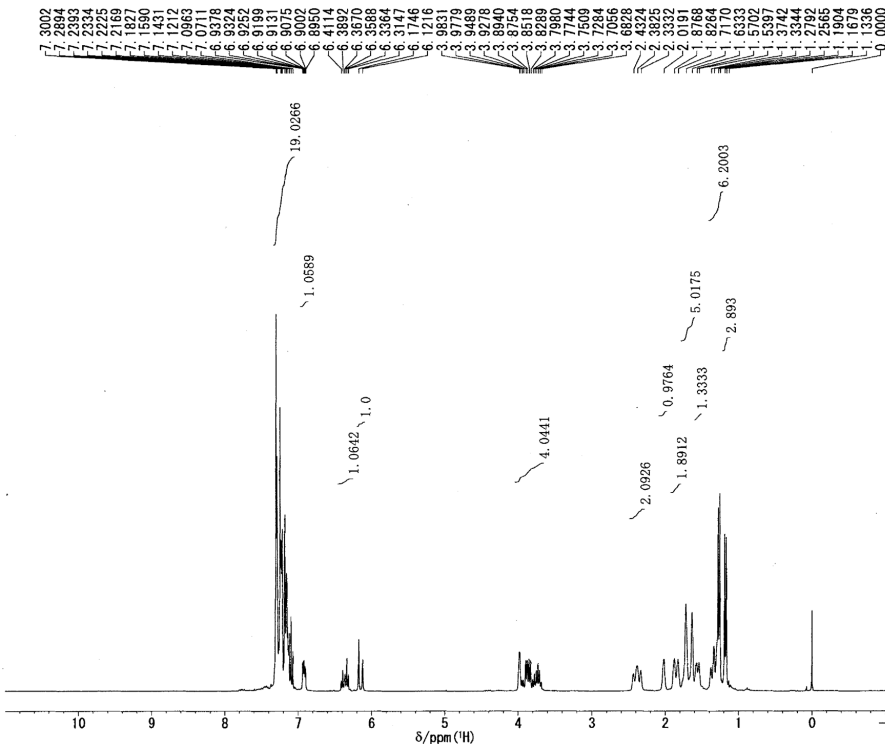
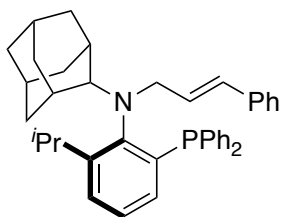
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 DATIM 11/Dec/2016 23:50:50
 COMNT
 ORNUC ³¹P
 EXMOD ZGPC30
 ORFRQ 121.54 MH
 Z
 OBSET 0.0 kHz
 OBFTM 10005.0 Hz
 POINT 32768
 PREQU 38535.64 Hz
 SCANS 16
 ACQTM 0.8503
 S
 PD 8.0 s
 PW1 0.0 μs
 TRNUC OFP
 PROBHD 5 MM BBO BB-1H-D Z-GR
 D Z8284/01
 INSTRUM SPECT
 PULSPRG ZGPC30
 GRDPROG
 CTEMP 26.85 °C
 SLVNT CDCL₃
 EXREF 158.537 p
 pm
 BF 0.25 Hz
 WINDOW Exponential
 RGAIN 11585
 Operator _____



peak name area height area%

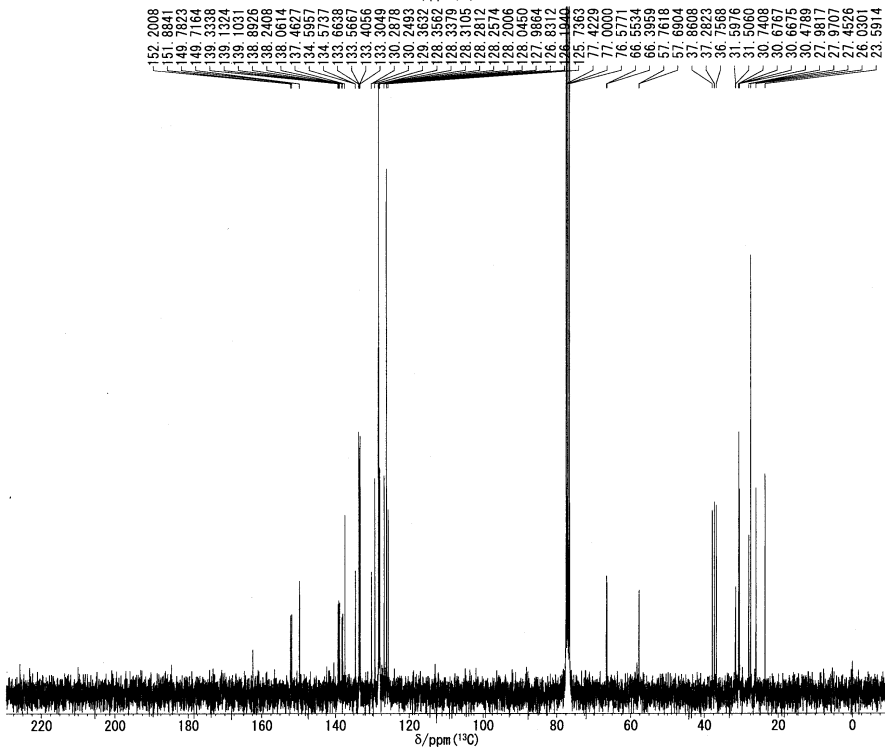
peak	name	area	height	area%		
1	Unknown	6	32.307	329660	5611	2.750
2	Unknown	6	42.653	11656865	163694	97.250

¹H, ¹³C and ³¹P NMR, and chiral phase HPLC chart of (+)-**3d**



DFILE C:\KYOUSEI6NMR\VO6MINO20
16V21636HV1PDATA\V111
DATIM 10/Dec/2016 03:03:41
COMNT

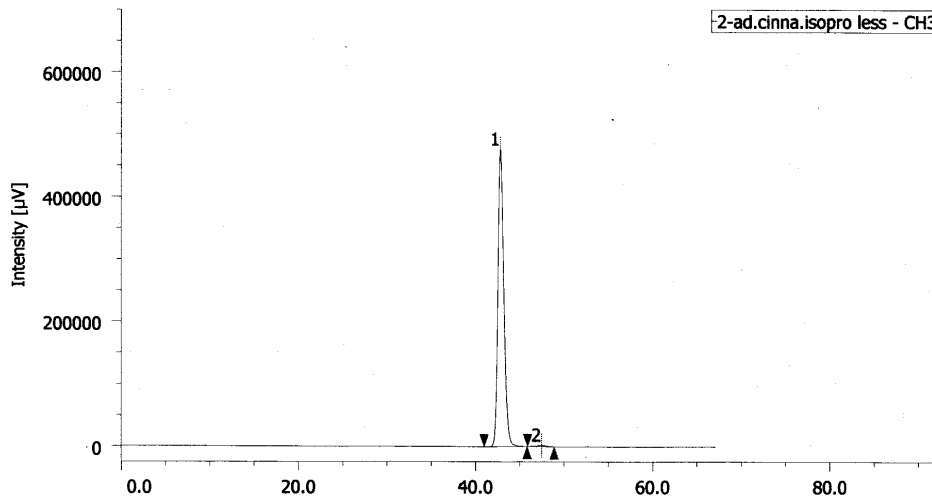
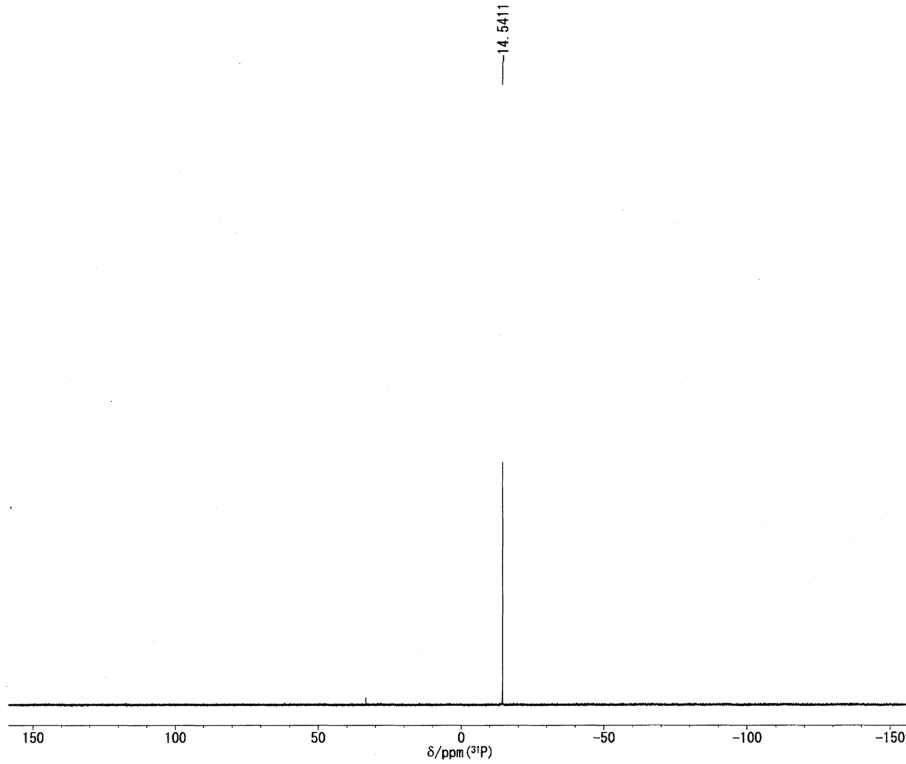
OBNUC ¹H
EXMOD ZG30
OBFRQ 300.23 MH
z
OBSET 0.0 kHz
OBFIN 10004.57 Hz
POINT 16384
FREQU 3612.717 H
SCANS 16
ACQTM 4.5351
s
PD 1.0 s
PWI 9.0 μs
IRNUC OFF
PROBHD 5 MM BBO BB-1H-D Z-GR
D 28284/01
INSTRUM SPECT
PULSPRG ZG30
GRDPRG
CTEMP 26.85 °C
SLVNT CDCL₃
EXREF 0.0 ppm
BS 0.25 Hz
WINDOW Exponential
RGAIN 256
Operator _____



DFILE C:\KYOUSEI6NMR\VO6MINO20
16V21636CY1PDATA\V111
DATIM 10/Dec/2016 04:10:42
COMNT

OBNUC ¹³C
EXMOD ZGPC30
OBFRQ 75.49 MH
z
OBSET 0.0 kHz
OBFIN 10003.06 Hz
POINT 32768
FREQU 18115.94 Hz
SCANS 1024
ACQTM 1.8088
s
PD 2.0 s
PWI 0.0 μs
IRNUC OFF
PROBHD 5 MM BBO BB-1H-D Z-GR
D 28284/01
INSTRUM SPECT
PULSPRG ZGPC30
GRDPRG
CTEMP 26.85 °C
SLVNT CDCL₃
EXREF 77.0 ppm
BS 0.25 Hz
WINDOW Exponential
RGAIN 2299
Operator _____

DFILE C:\YKYOUSEI6MMRY06MIN020
 16Y21636PW1WPDATAV1V11
 DATIM 10/Dec/2016 04:50:27
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 OBNUC ³¹P
 EXMOD ZGPG30
 OBFRQ 121.54 MH
 z
 OBSET 0.0 kHz
 OBFTM 10005.0 Hz
 POINT 32768
 FREQU 38535.64 Hz
 SCANS 16
 ACQTM 0.8503 s
 PD 8.0 s
 PW 0.0 μs
 TRNUC OFF
 PROBHD 5 MM BBO BB-1H-D Z-GR
 D Z8284/01
 INSTRUM SPECT
 PULSPRG ZGPG30
 GRDPRG
 CTEMP 26.85 °C
 SLVNT CDCL₃
 EXREF 158.537 p
 pm
 BF 0.25 Hz
 WINDOW Exponential
 RGAIN 2896
 Operator _____



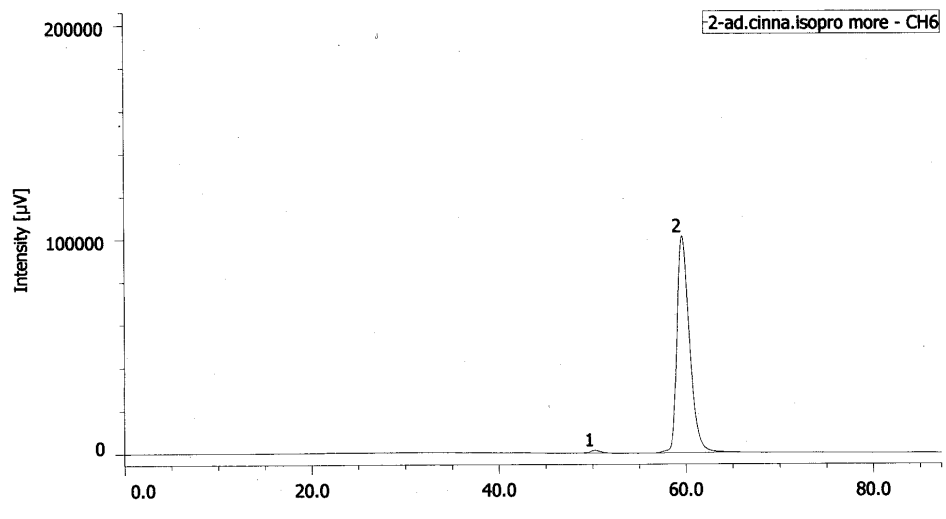
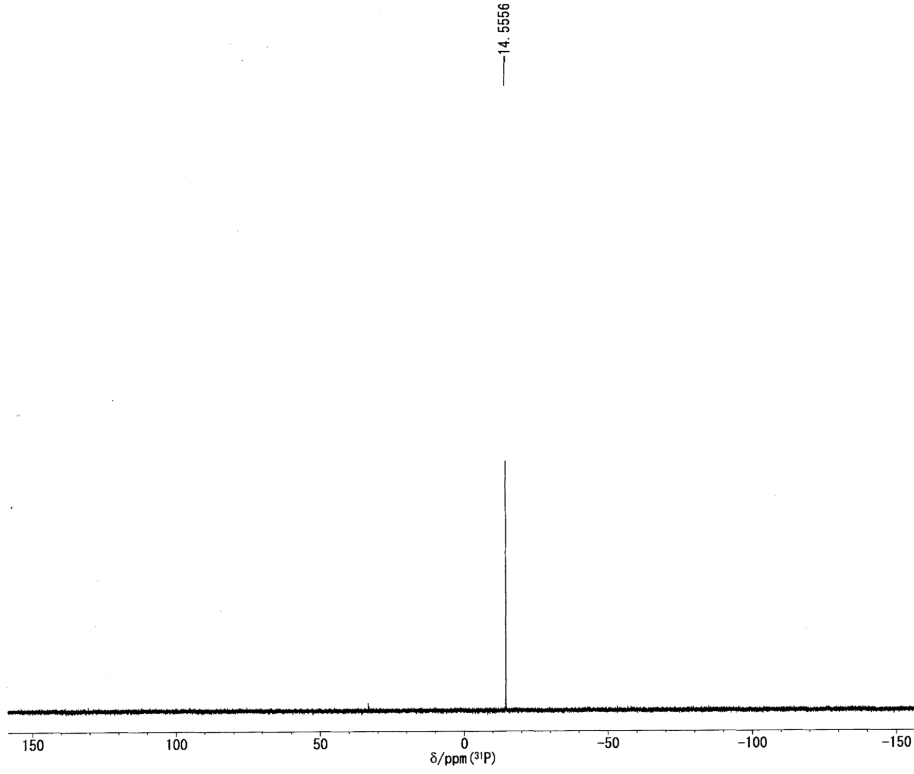
peak name area height area%

#	CH	IR [cm ⁻¹]	Area [μV·s]	Height [μV]	Area%	
1	Unknown	3	42.792	21993084	476599	99.475
2	Unknown	3	47.442	116044	1883	0.525


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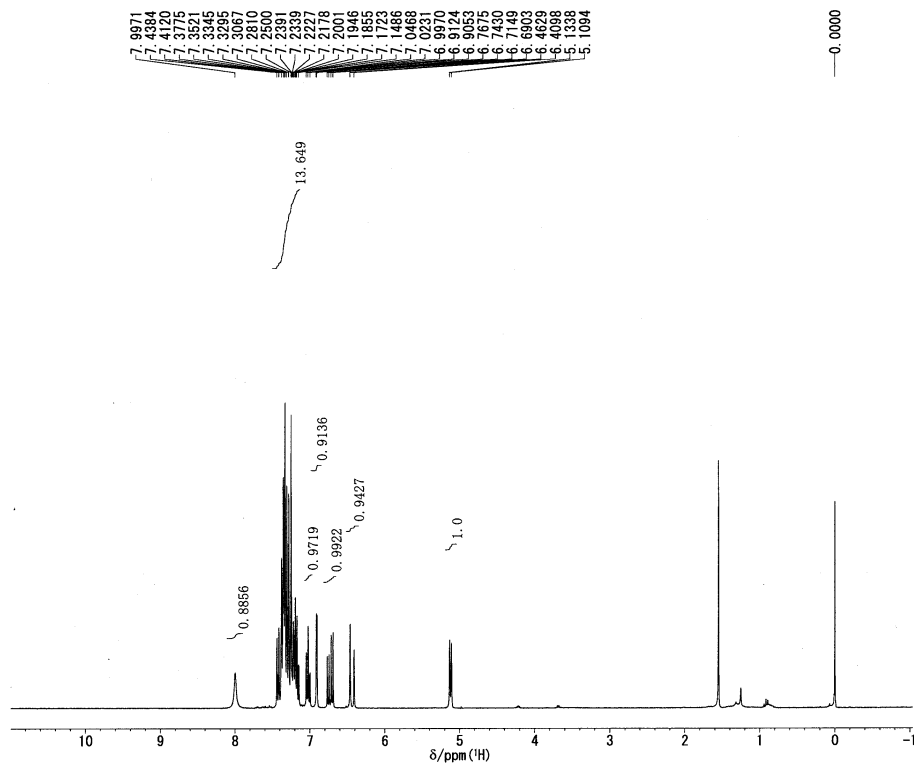
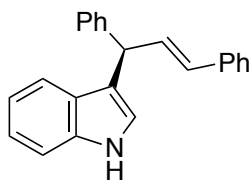
D:\FILE C:\KYOUSEI6\NRV06M\IN020
18921636P42\PDAT\11111
DATIM 10/Dec/2016 09:01:26
COMNT
OBNUC 31P
EXMOD ZFGP30
OBFRQ 121.54 MH
Z
OSSET 0.0 kHz
OBFIN 10005.0 Hz
POINT 32768
FREQU 38538.64 Hz
SCANS 16
ACQTM 0.8503
S
PD 8.0 s
PW1 0.0 μs
IRNUC OFF
PROBHD 5 MM BBO BB-1H-D Z-GR
D Z8284/01
INSTRUM SPECT
PULSPRG ZFGP30
GROPRG
CTEMP 26.85 °C
SLWT CDCL3
EXREF 158.537 p
PM
BF 0.25 Hz
WINDOW Exponential
RGAIN 2896
Operator _____

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peak name	area	height	area%
1 Unknown	105070	1329	1.102
2 Unknown	9429093	101038	98.898

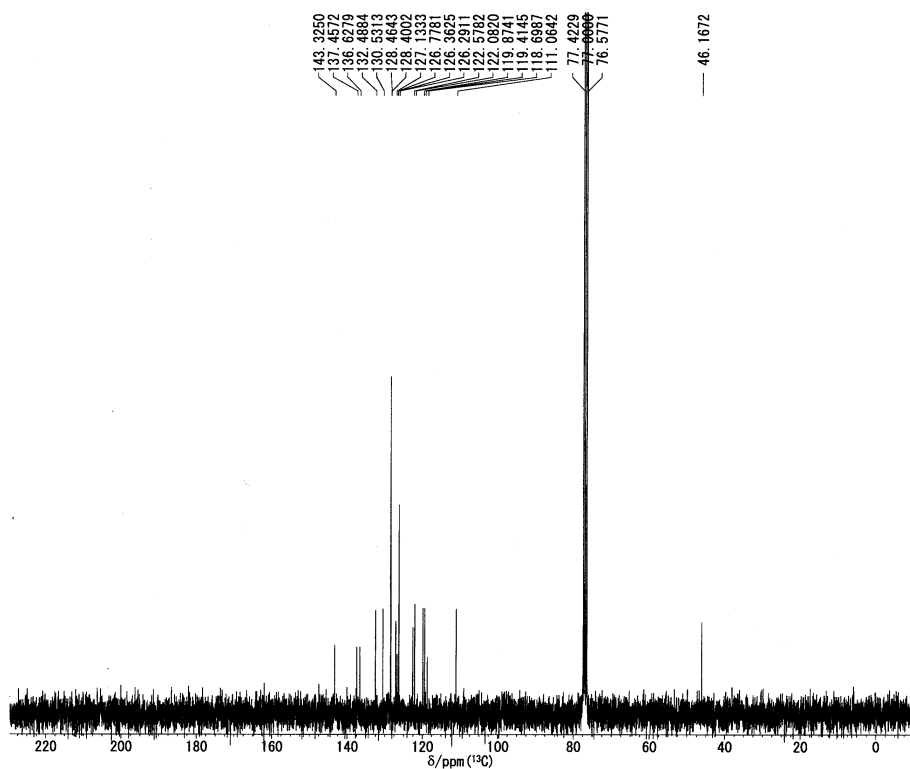
¹H and ¹³C NMR, and chiral phase HPLC chart of (*R*)-**9a** (Table 3, entry 1)



DFILE C:\KYOUSEI6\NMR\VO6MIN020
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DATIM 28/Oct/2016 10:59:13
COMNT

OBNUC ¹H
EXMOD ZG30
OBFREQ 300.23 MH
Z
OBSET 0.0 kHz
OBFIN 10004.57 Hz
POINT 16384
FREQU 3612.717 H
SCANS 16
ACQTM 4.5351
S
PD 1.0 s
PW1 9.0 μs
IRNUC OFF
PROBHD 5 MM BBO BB-1H-D Z-GR
D Z8284/01
INSTRUM SPECT
PULSPRG ZG30
GRDPRG
CTEMP 26.85 °C
SLVNT CDCl₃
EXREF 0.0 ppm
BF 0.25 Hz
WINDOW Exponential
RGAIN 575

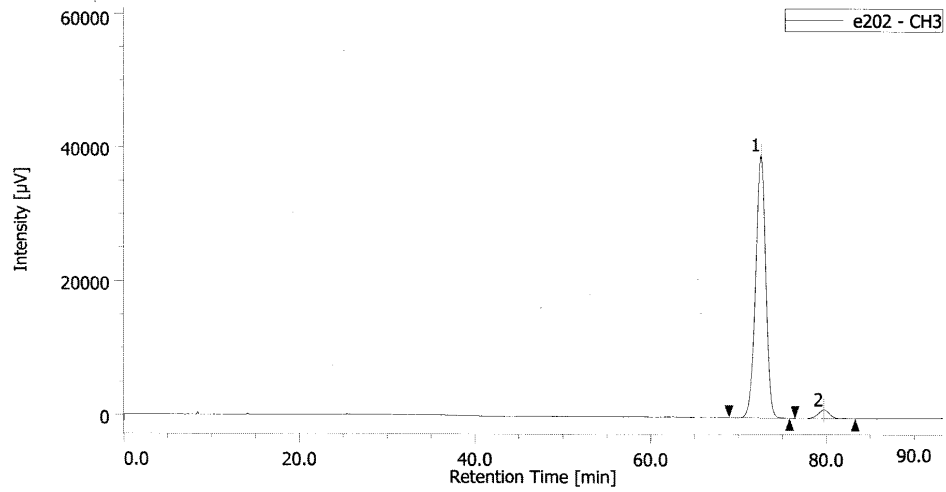
Operator _____



DFILE C:\KYOUSEI6\NMR\VO6MIN020
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DATIM 28/Oct/2016 18:43:37
COMNT

OBNUC ¹³C
EXMOD ZPG30
OBFREQ 75.49 MH
Z
OBSET 0.0 kHz
OBFIN 10003.06 Hz
POINT 32768
FREQU 18115.94 Hz
SCANS 512
ACQTM 1.8088
S
PD 2.0 s
PW1 0.0 μs
IRNUC OFF
PROBHD 5 MM BBO BB-1H-D Z-GR
D Z8284/01
INSTRUM SPECT
PULSPRG ZPG30
GRDPRG
CTEMP 26.85 °C
SLVNT CDCl₃
EXREF 77.0 ppm
BF 0.25 Hz
WINDOW Exponential
RGAIN 2580

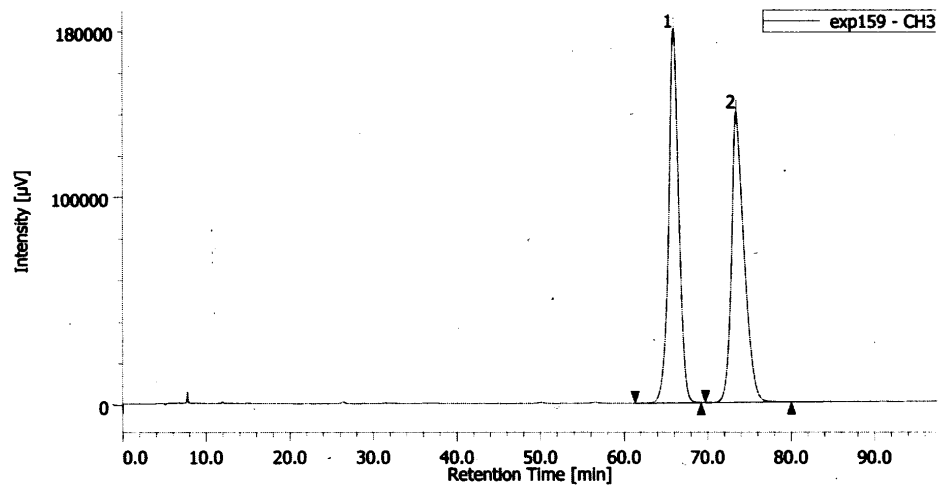
Operator _____



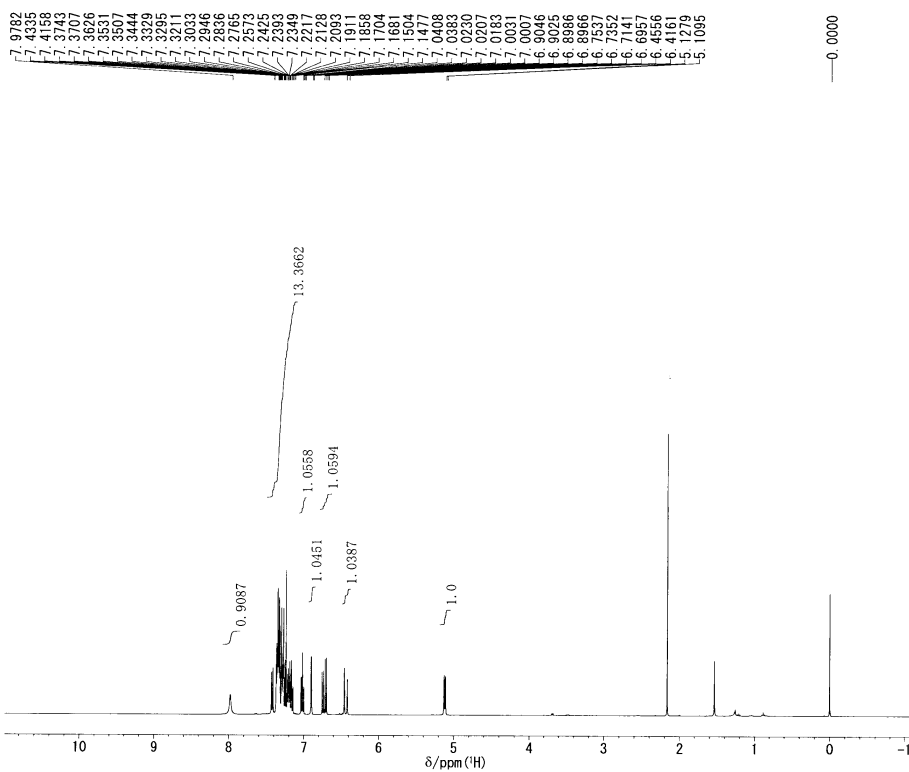
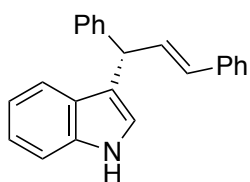
peak name

#	ピーク名	CH	tR [min]	area [$\mu\text{V}\cdot\text{sec}$]	area%
1	Unknown	3	72.500	3127948	96.604
2	Unknown	3	79.683	109944	3.396

(\pm)-**9a**



¹H and ¹³C NMR, and chiral phase HPLC chart of (*S*)-**9a** (Table 2, entry 11)

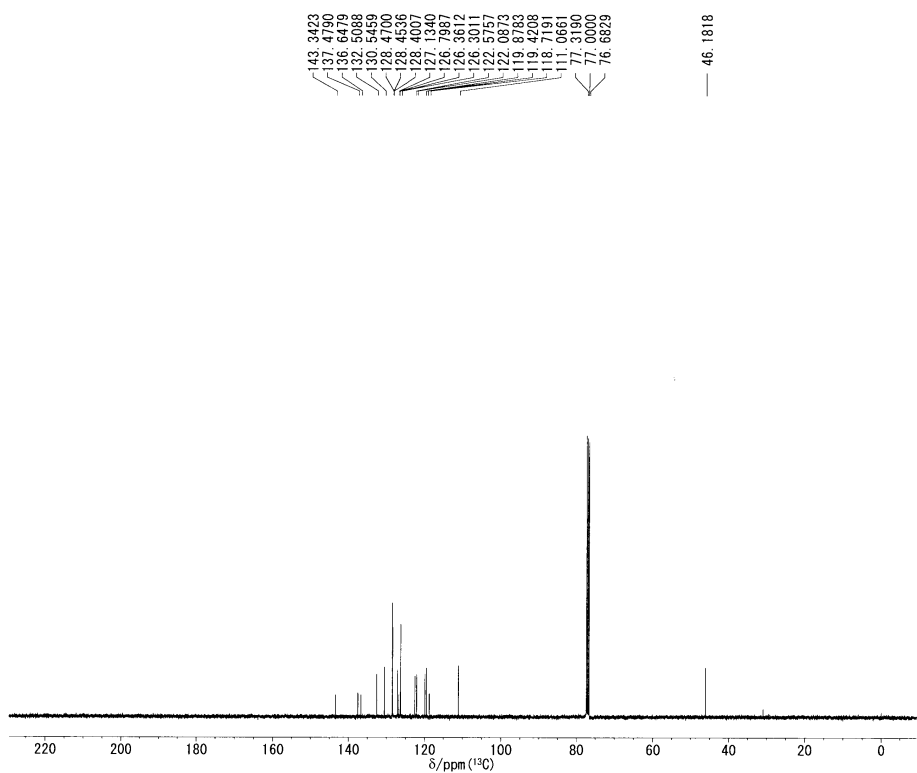


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DFILE C:\KYOUSEI6\NMR\06M\N0201
      8Y4-00315H\1P\DATA\1Y11
DATIM 19/Dec/2018 16:21:15
COMNT

ORNUC 1H
EXMOD ZG30
OBFREQ 400.18 MHz
OBSETE 0.0 kHz
OBFIN 10003.83 Hz
POINT 45536
FREQU 4826.255 Hz
SCANS 19
ACQTM 13.5791 s
PD 1.0 s
PWI 14.0 μs
IRNUC OFF
PROBHD Z108618-0111 (PA BBO 4
      OOS1 BBF-H-D-05 Z PLUS)
INSTRUM SPECT
PULSPRG ZG30
GRDPRG
CTEMP 24.21 °C
SLVNT CDCl3
EXREF 10.989 ppm
      =
BF 0.25 Hz
WINDOW Exponential
RGAIN 203

Operator
    
```

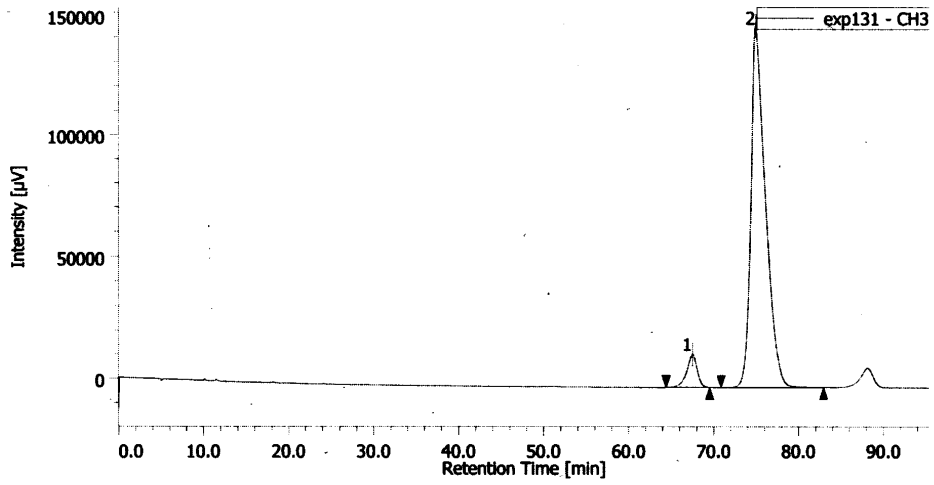


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DFILE C:\KYOUSEI6\NMR\06M\N0201
      8Y4-00315CY\1P\DATA\1Y11
DATIM 19/Dec/2018 16:37:53
COMNT

ORNUC 13C
EXMOD ZGPC30
OBFREQ 100.63 MHz
OBSETE 0.0 kHz
OBFIN 9998.39 Hz
POINT 32768
FREQU 24038.46 Hz
SCANS 273
ACQTM 1.3631 s
PD 2.0 s
PWI 10.0 μs
IRNUC OFF
PROBHD Z108618-0111 (PA BBO 4
      OOS1 BBF-H-D-05 Z PLUS)
INSTRUM SPECT
PULSPRG ZGPC30
GRDPRG
CTEMP 25.01 °C
SLVNT CDCl3
EXREF 77.0 ppm
BF 0.25 Hz
WINDOW Exponential
RGAIN 203

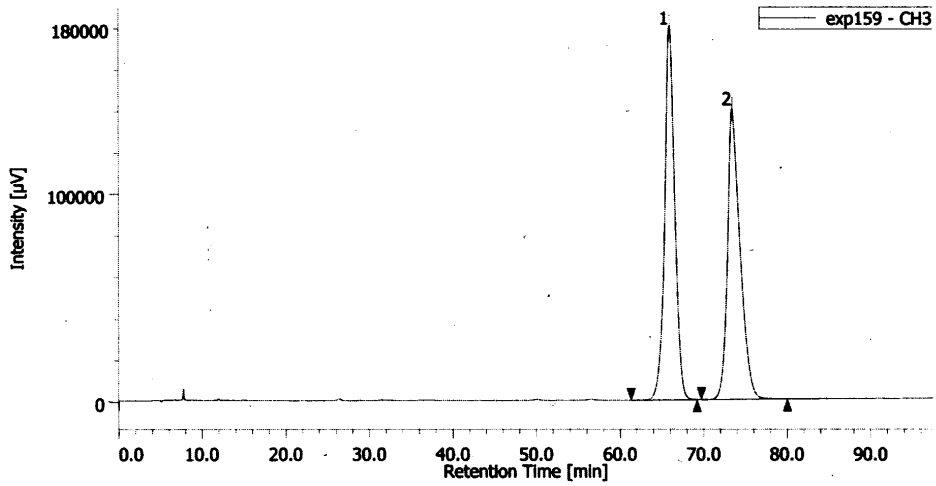
Operator
    
```



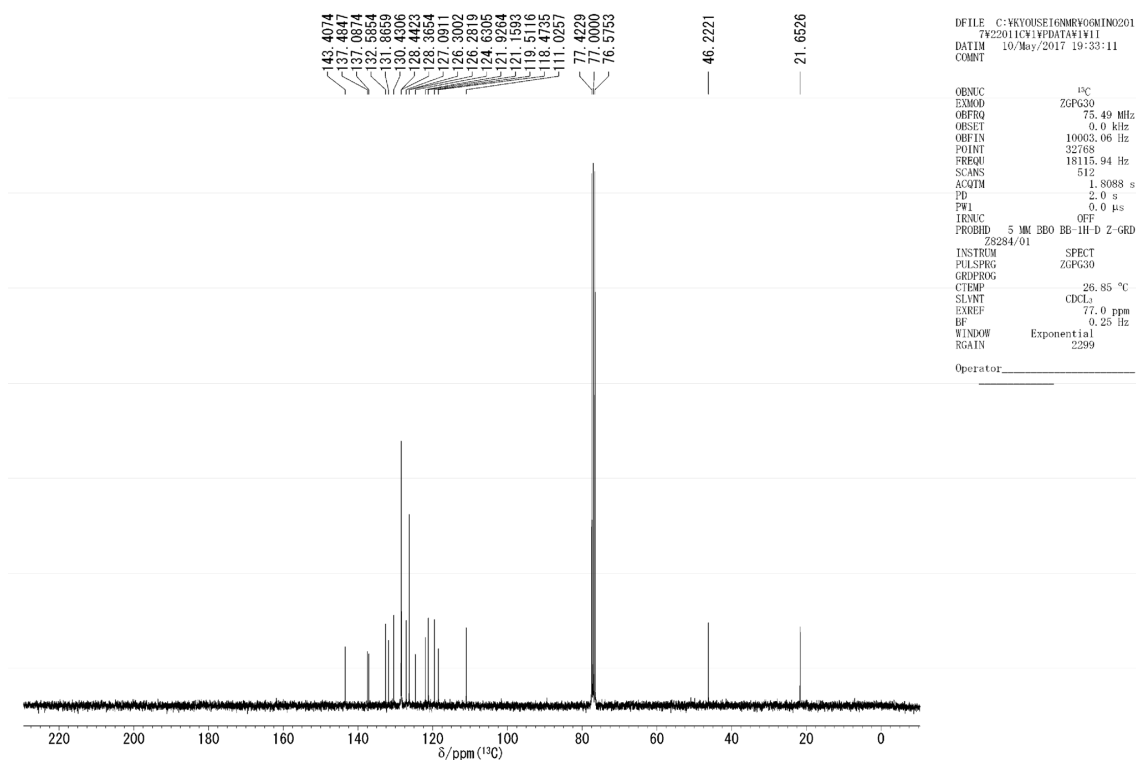
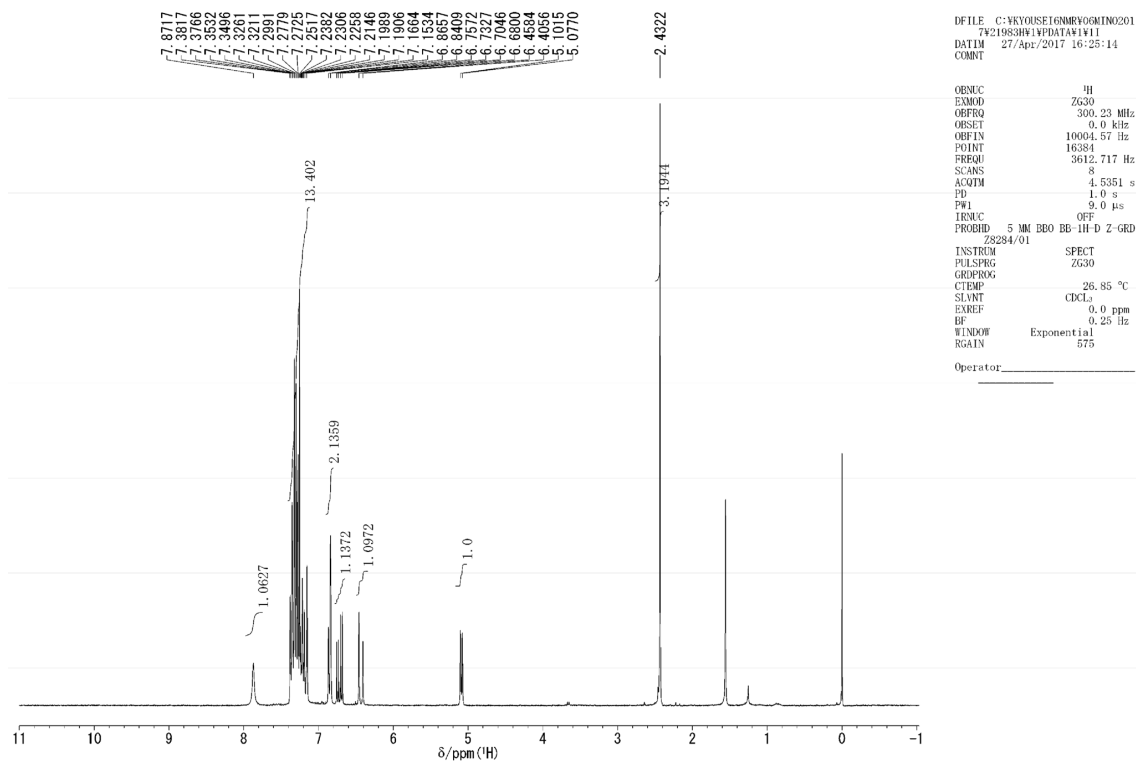
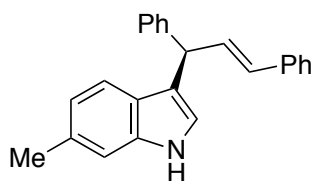
peak name area height area%

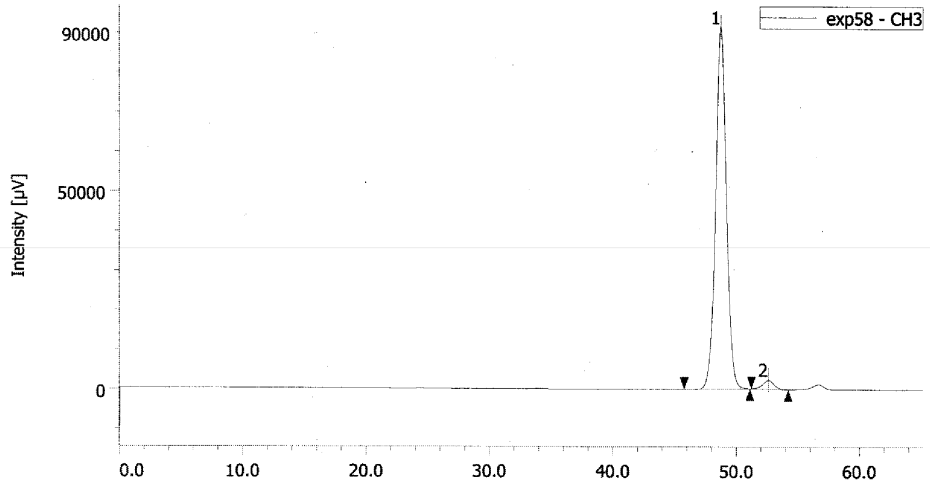
#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%
1	Unknown	3	67.492	1163406	13523	6.217
2	Unknown	3	74.967	17549119	147977	93.783

(±)-9a



¹H and ¹³C NMR, and chiral phase HPLC chart of (*R*)-**9b** (Table 3, entry 2)

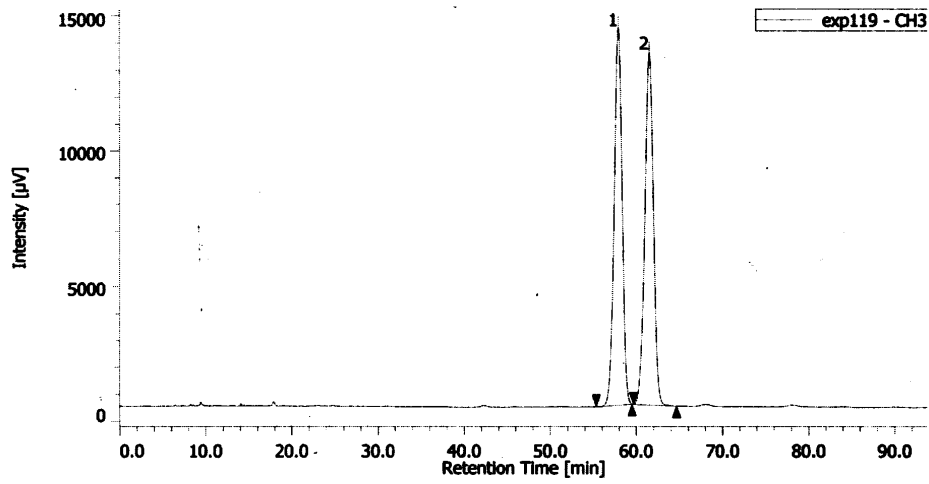




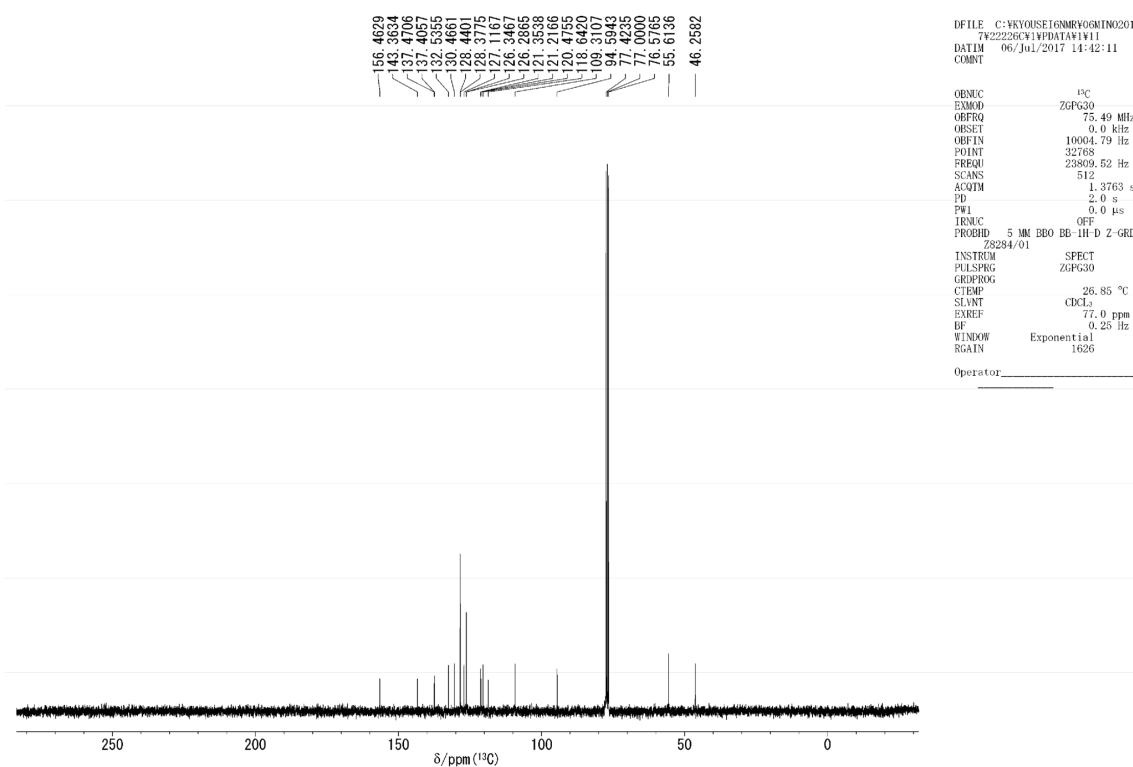
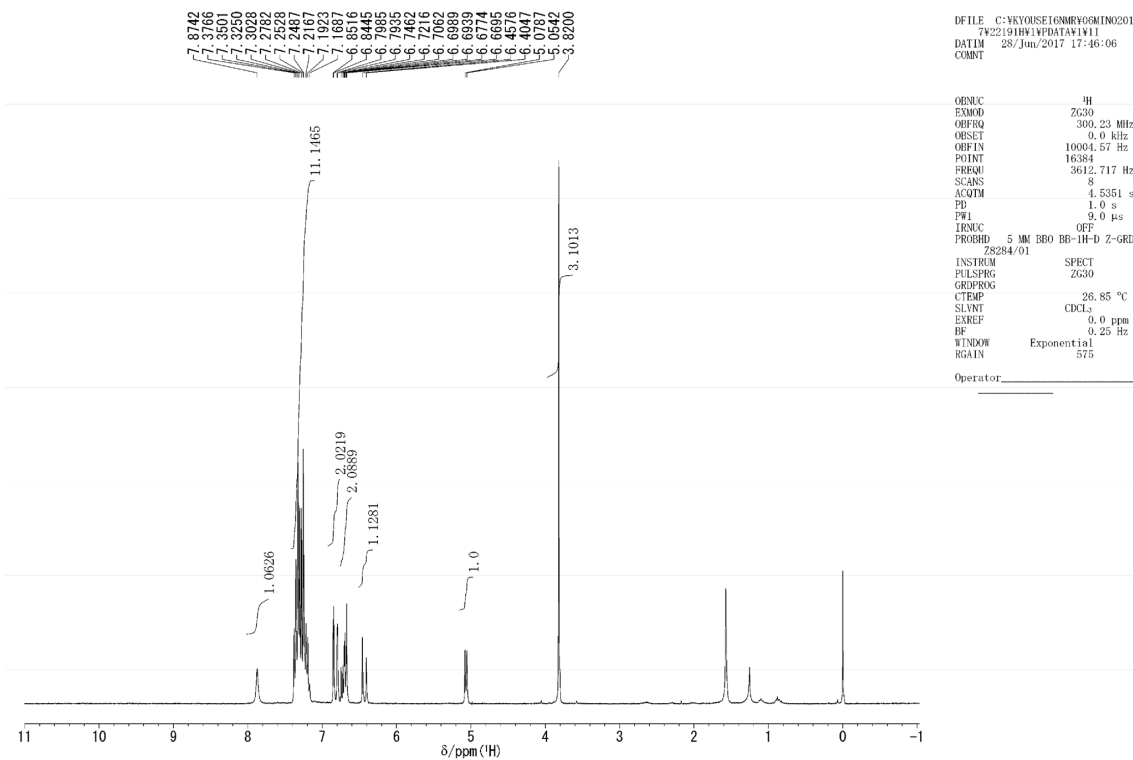
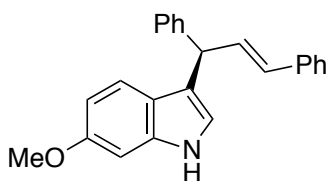
peak name

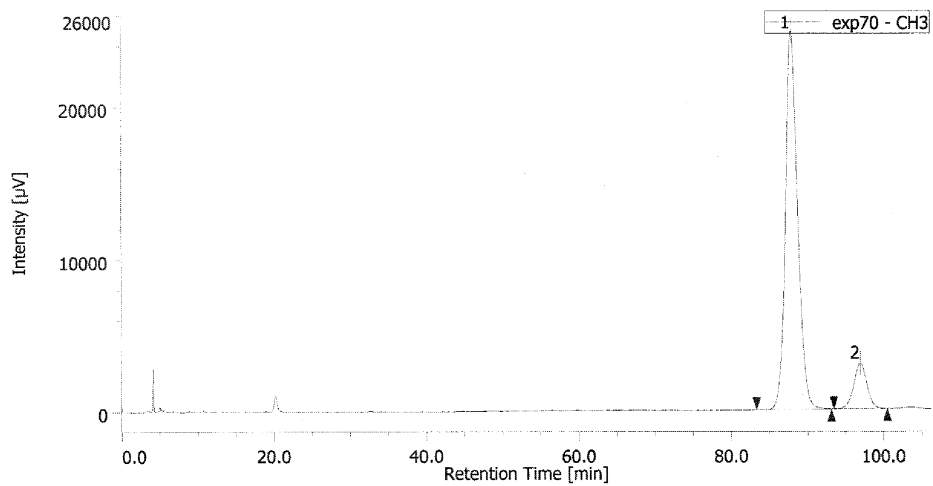
#	ピーク名	CH	tR [min]	area [$\mu\text{V}\cdot\text{sec}$]	area%
1	Unknown	3	48.7	5491026	97.547
2	Unknown	3	52.6	138061	2.453

(\pm)-9b



¹H and ¹³C NMR, and chiral phase HPLC chart of (*R*)-**9c** (Table 3, entry 3)

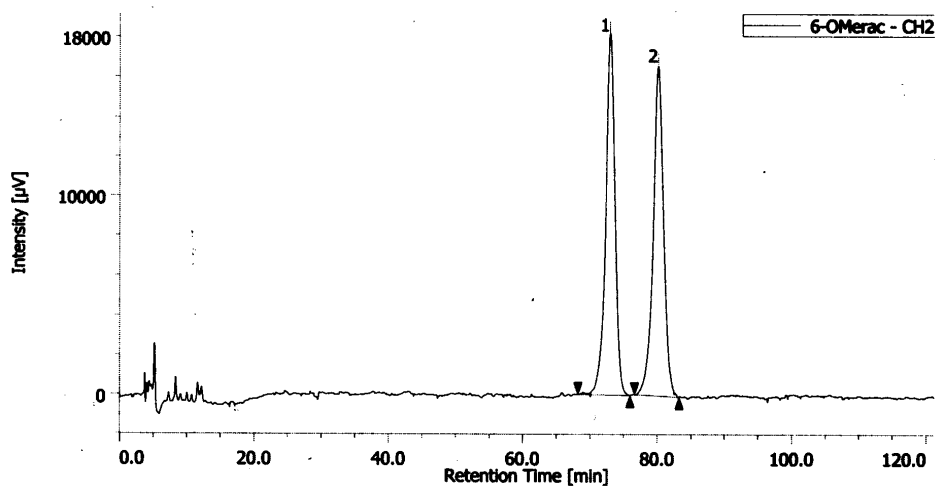




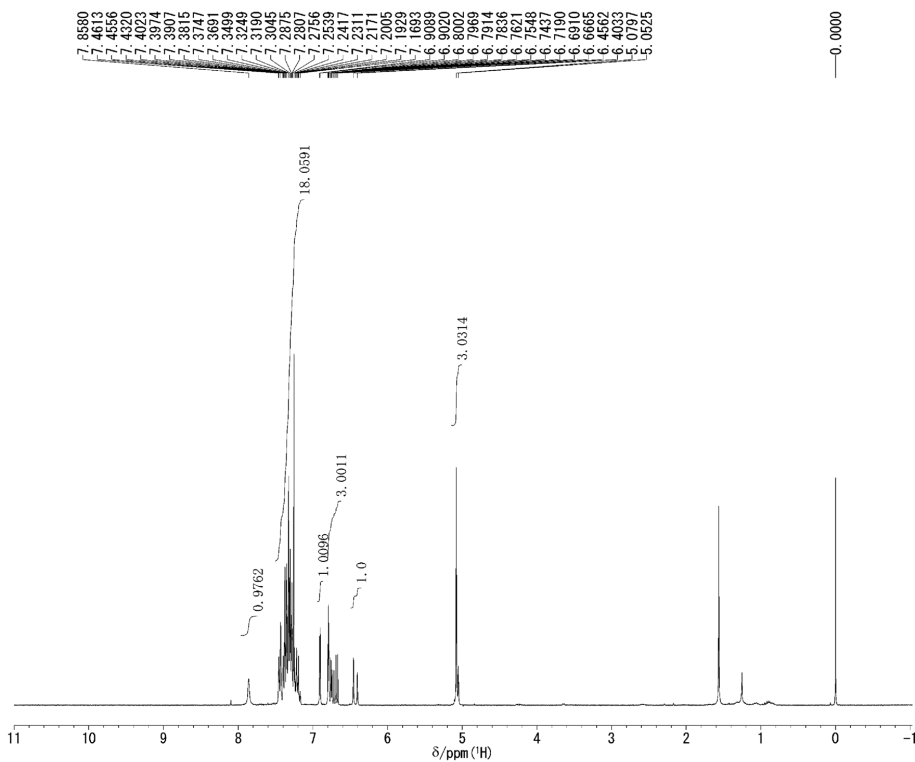
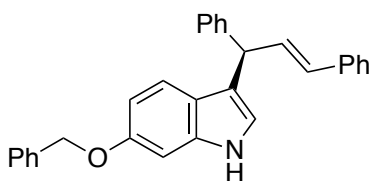
peak name

#	ピーク名	CH	tR [min]	area [μV·sec]	area%
1	Unknown	3	87.867	2733858	88.415
2	Unknown	3	96.950	358203	11.585

(±)-9c

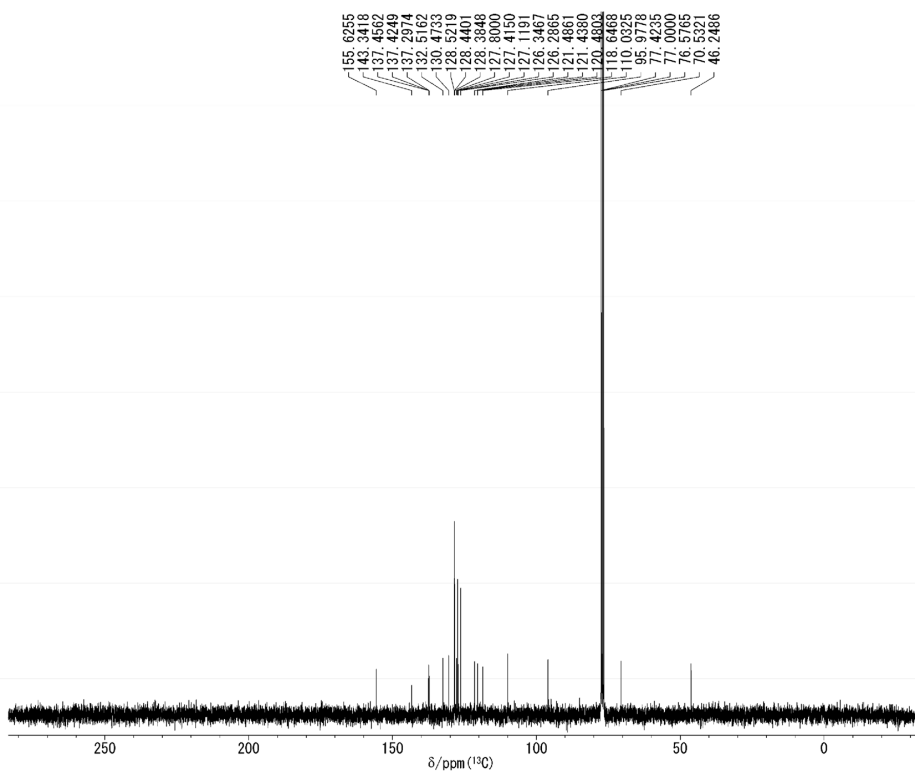


¹H and ¹³C NMR, and chiral phase HPLC chart of (*R*)-**9d** (Table 3, entry 4)



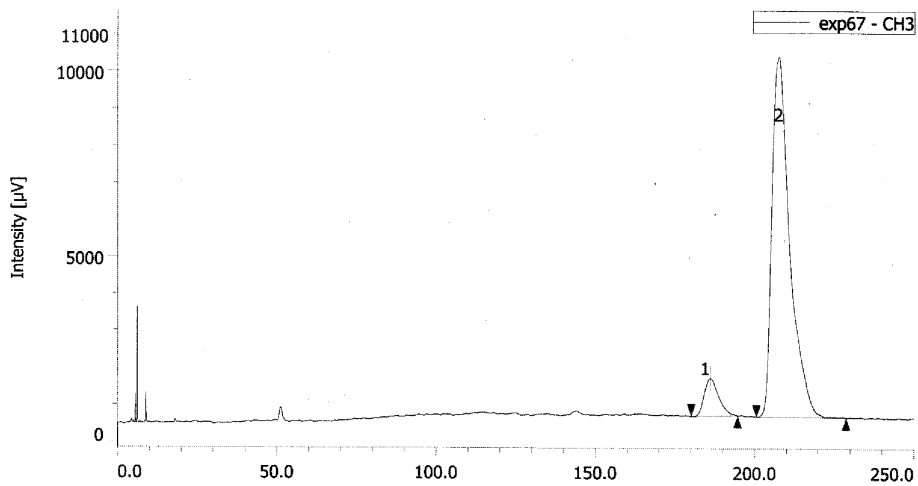
D\FILE C:\KYOUSEI\6NMR\6MIN0201
7\22112PH1\PDAT\1\111
DATEM 09/Jun/2017 15:26:59
COMNT

ORNUC ¹H
EXMOD ZG30
OBFRQ 300.23 MHz
ORSET 0.0 kHz
OBFIN 10004.57 Hz
POINT 16384
FREQC 3612.717 Hz
SCANS 8
AQTM 4.5351 s
PD 1.0 s
PFI 9.0 μs
IRNUC OFF
PROBHD 5 MM BBO BB-1H-D Z-GRD
28284/01
INSTRM SPECT
PULSPRG ZG30
GRDPRG
CTEMP 26.85 °C
SLVNT CDCl₃
EXREF 0.0 ppm
BF 0.25 Hz
WINDOW Exponential
RGAIN 575
Operator _____



D\FILE C:\KYOUSEI\6NMR\6MIN0201
7\22112PH1\PDAT\1\111
DATEM 16/Jun/2017 16:36:59
COMNT

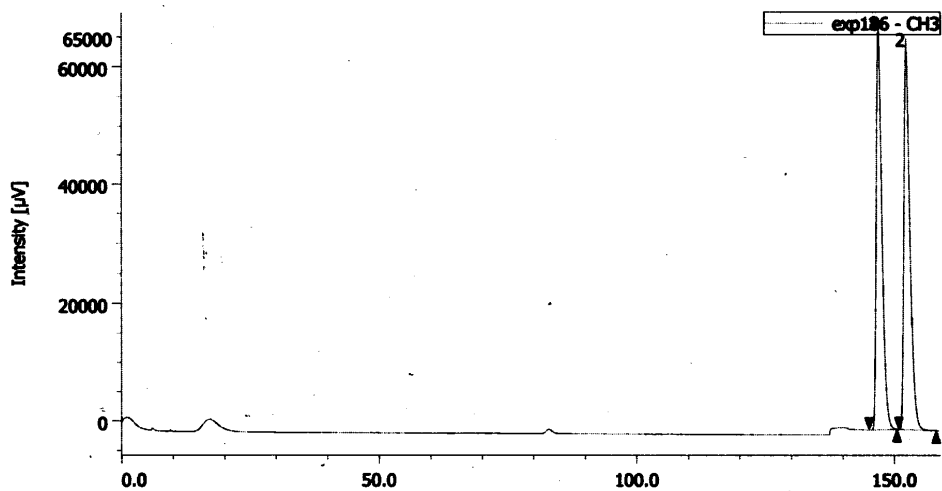
ORNUC ¹³C
EXMOD ZPG30
OBFRQ 75.49 MHz
ORSET 0.0 kHz
OBFIN 10004.79 Hz
POINT 32768
FREQC 23809.52 Hz
SCANS 512
AQTM 1.3763 s
PD 2.0 s
PFI 0.0 μs
IRNUC OFF
PROBHD 5 MM BBO BB-1H-D Z-GRD
28284/01
INSTRM SPECT
PULSPRG ZPG30
GRDPRG
CTEMP 26.85 °C
SLVNT CDCl₃
EXREF 77.0 ppm
BF 0.25 Hz
WINDOW Exponential
RGAIN 4598
Operator _____



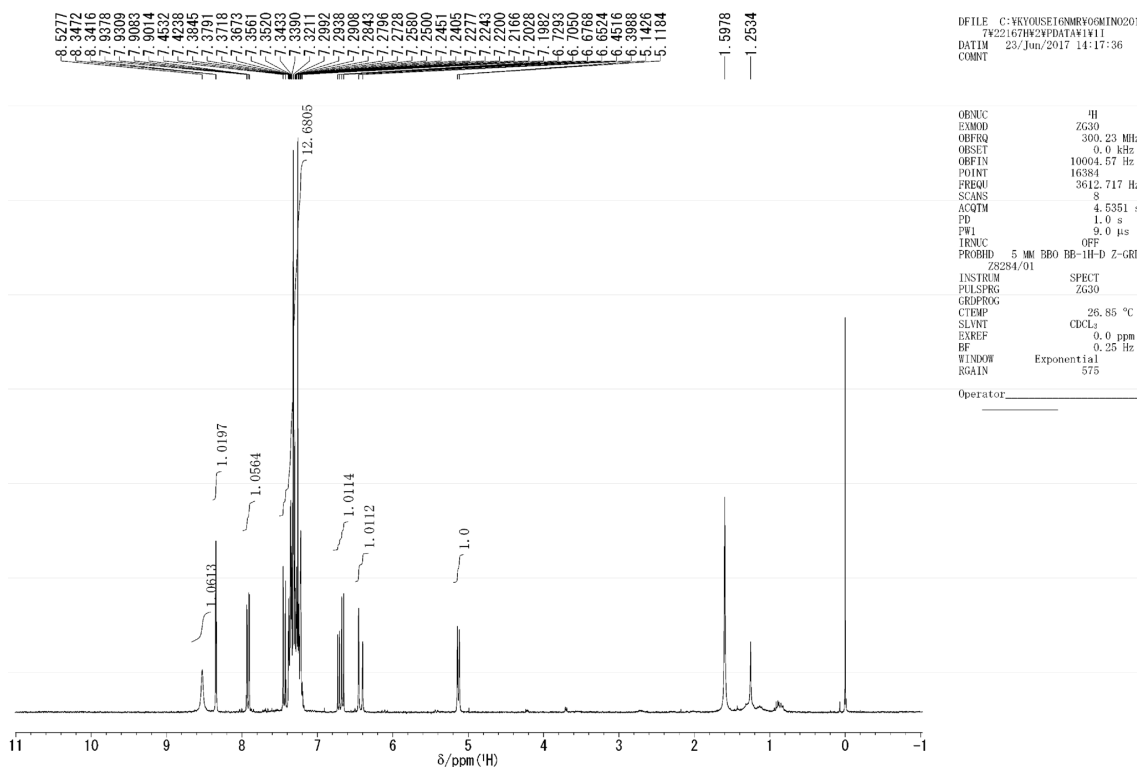
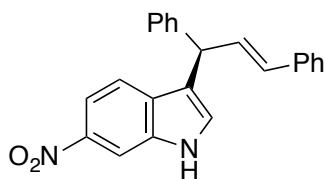
peak name

#	ピーク名	CH	TR [min]	area [μV·sec]	area%
1	Unknown	3	186	315738	7.711
2	Unknown	3	209	3778805	92.289

(±)-9d

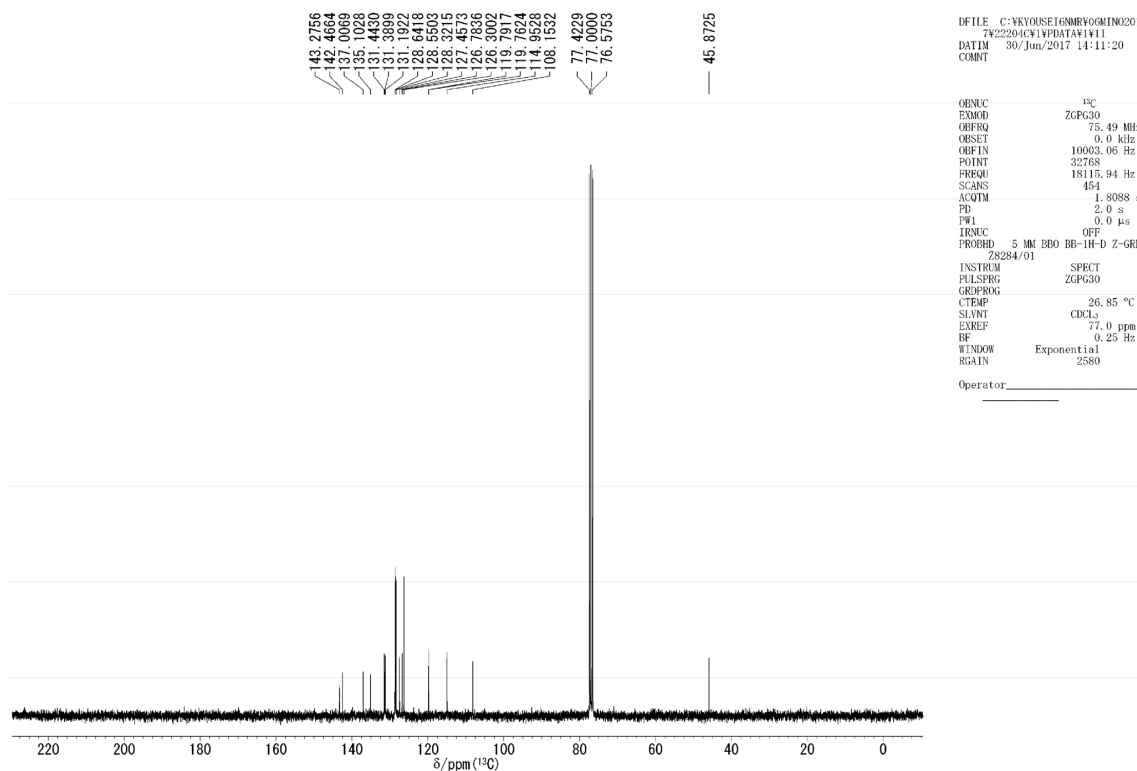


¹H and ¹³C NMR, and chiral phase HPLC chart of (*R*)-**9e** (Table 3, entry 5)



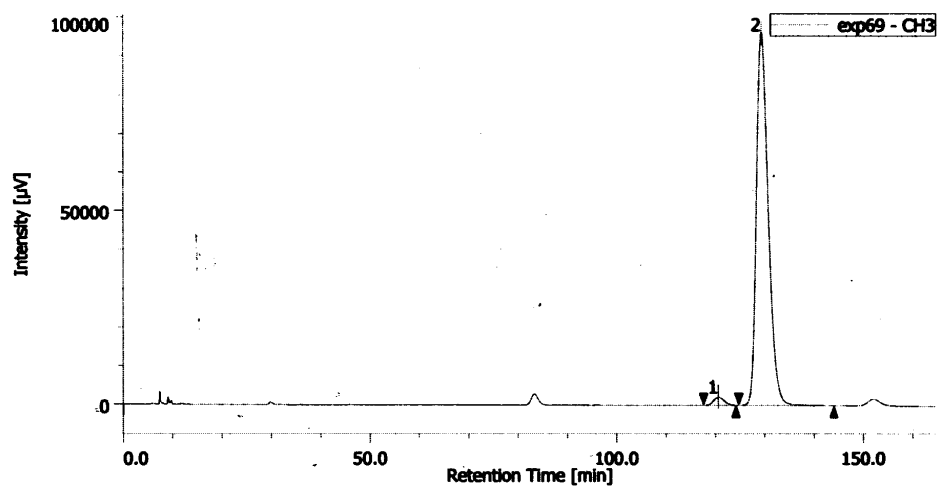
DFILE C:\KYOUSETI\NMR\YOG\INO201
7922167HW\FDATA\F1W1
DATIM 23/ Jun/2017 14:17:36
COMBT

ORNUC ¹H
EXMOD ZG30
OBFREQ 300.23 MHz
ORSET 0.0 kHz
OBFIN 10004.57 Hz
POINT 16384
FREQU 3612.717 Hz
SCANS 8
ACQIM 4.5351 s
PD 1.0 s
PWI 9.0 μs
IRNUC OFF
PROBHD 5 MM BBO BB-1H-D Z-GRD
ZS284/01
INSTRUM SPECT
PULSPRG ZG30
GRPPROG
CTEMP 26.85 °C
SLVNT CDCl₃
EXREF 0.0 ppm
BF 0.25 Hz
WINDOW Exponential
RGAIN 575
Operator



DFILE C:\KYOUSETI\NMR\YOG\INO201
7922203CW\FDATA\F1W1
DATIM 30/ Jun/2017 14:11:20
COMBT

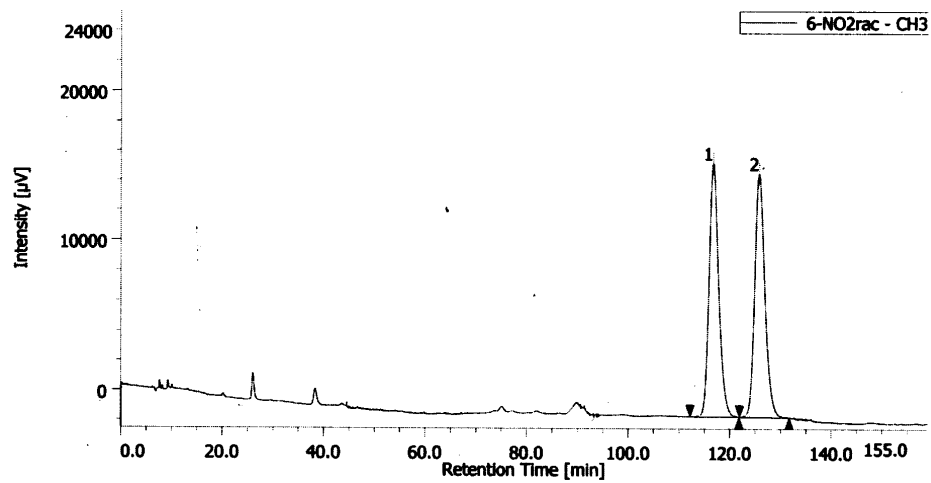
ORNUC ¹³C
EXMOD ZPG30
OBFREQ 75.49 MHz
ORSET 0.0 kHz
OBFIN 10063.06 Hz
POINT 32768
FREQU 18115.94 Hz
SCANS 454
ACQIM 1.8088 s
PD 2.0 s
PWI 0.0 μs
IRNUC OFF
PROBHD 5 MM BBO BB-1H-D Z-GRD
ZS284/01
INSTRUM SPECT
PULSPRG ZPG30
GRPPROG
CTEMP 26.85 °C
SLVNT CDCl₃
EXREF 77.0 ppm
BF 0.25 Hz
WINDOW Exponential
RGAIN 2580
Operator



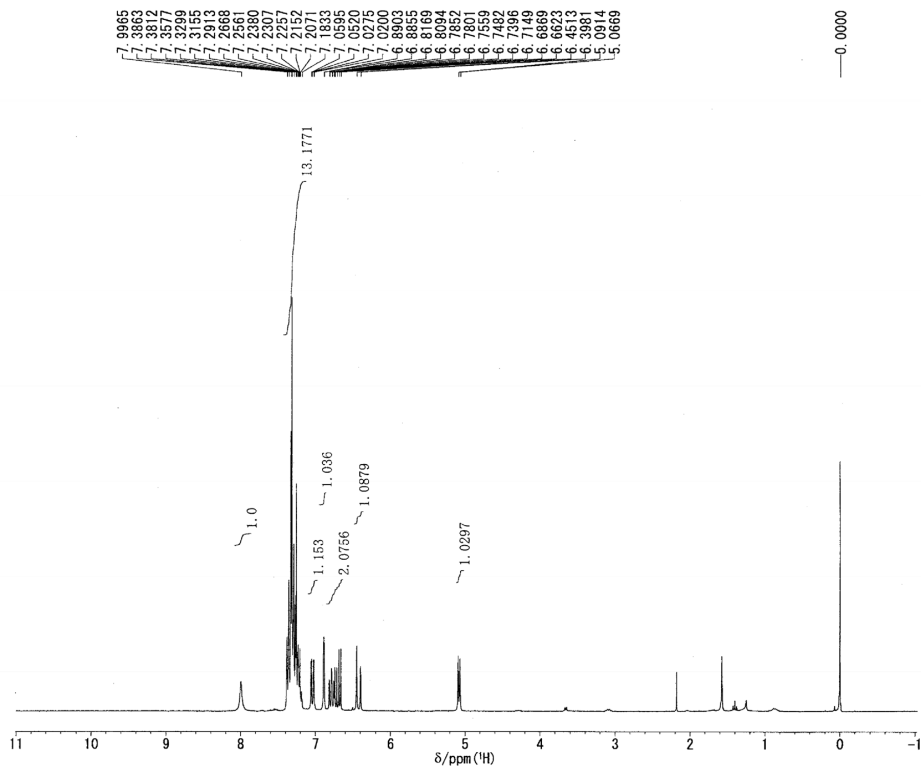
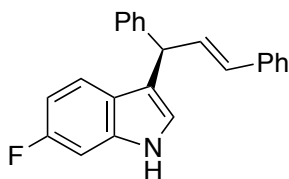
peak name

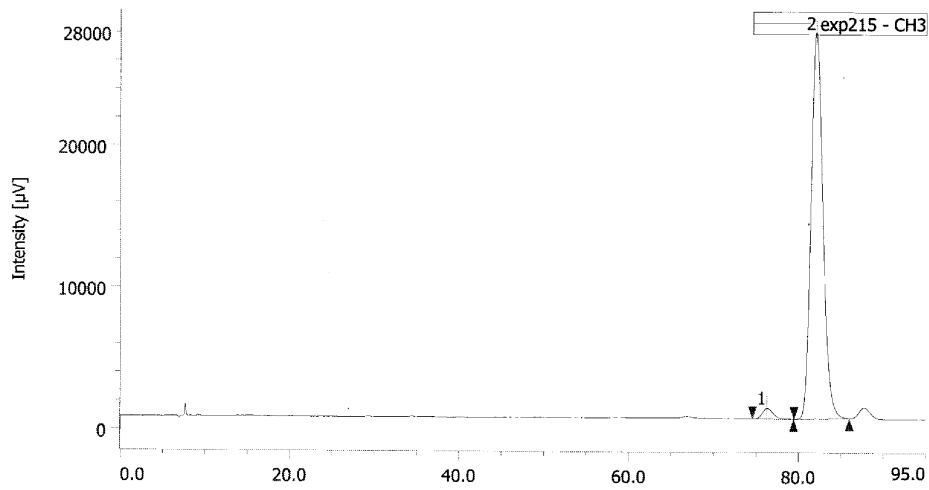
#	ピーク名	CH	tR [min]	area [μV·sec]	area%
1	Unknown	3	120.533	316346	1.934
2	Unknown	3	129.375	16037220	98.066

(±)-9e



¹H and ¹³C NMR, and chiral phase HPLC chart of (R)-9f (Table 3, entry 6)

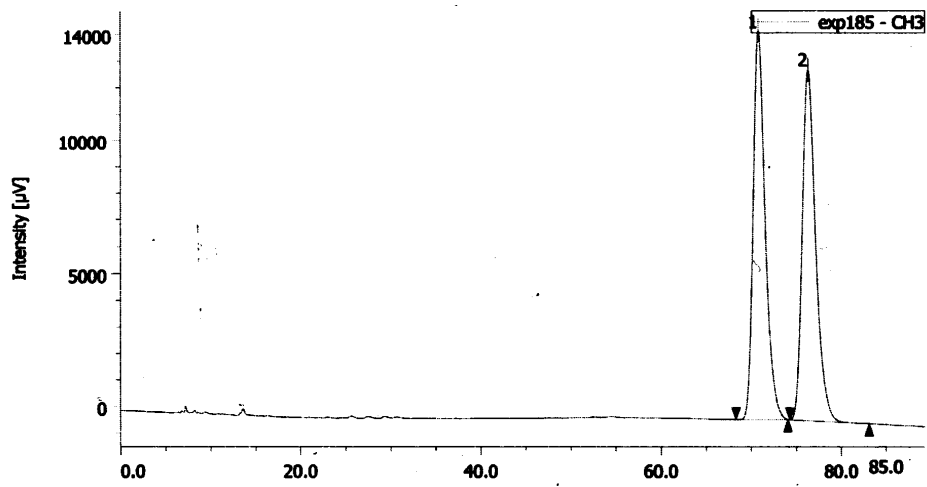


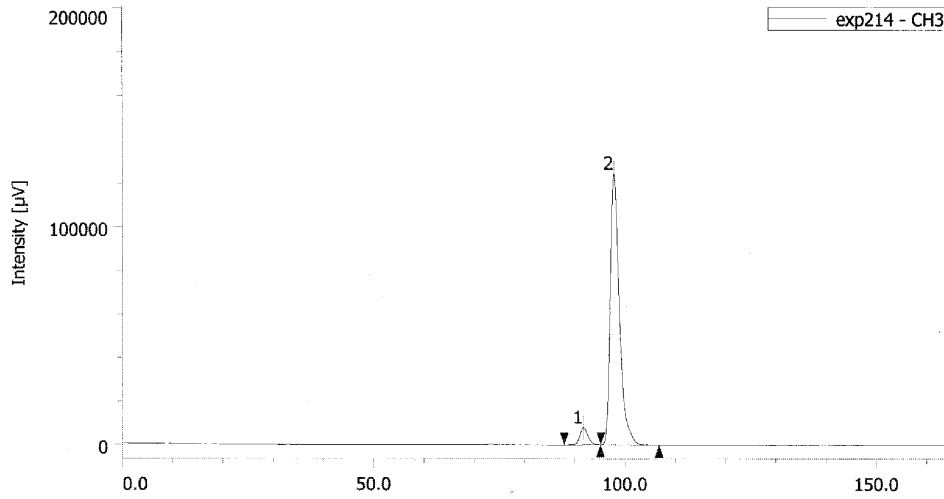


peak name

#	ピーク名	CH	tR [min]	area [μV·sec]	area%
1	Unknown	3	76.3	66210	2.405
2	Unknown	3	82.0	2687301	97.595

(±)-9f

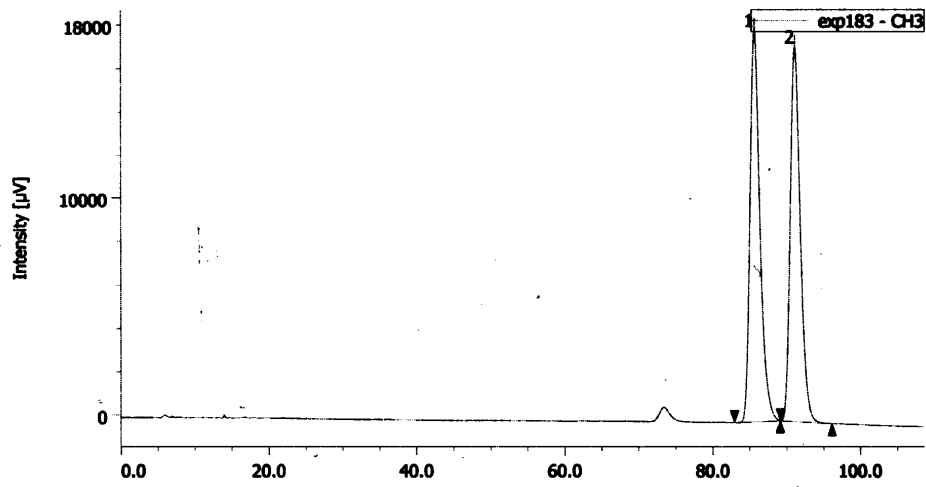




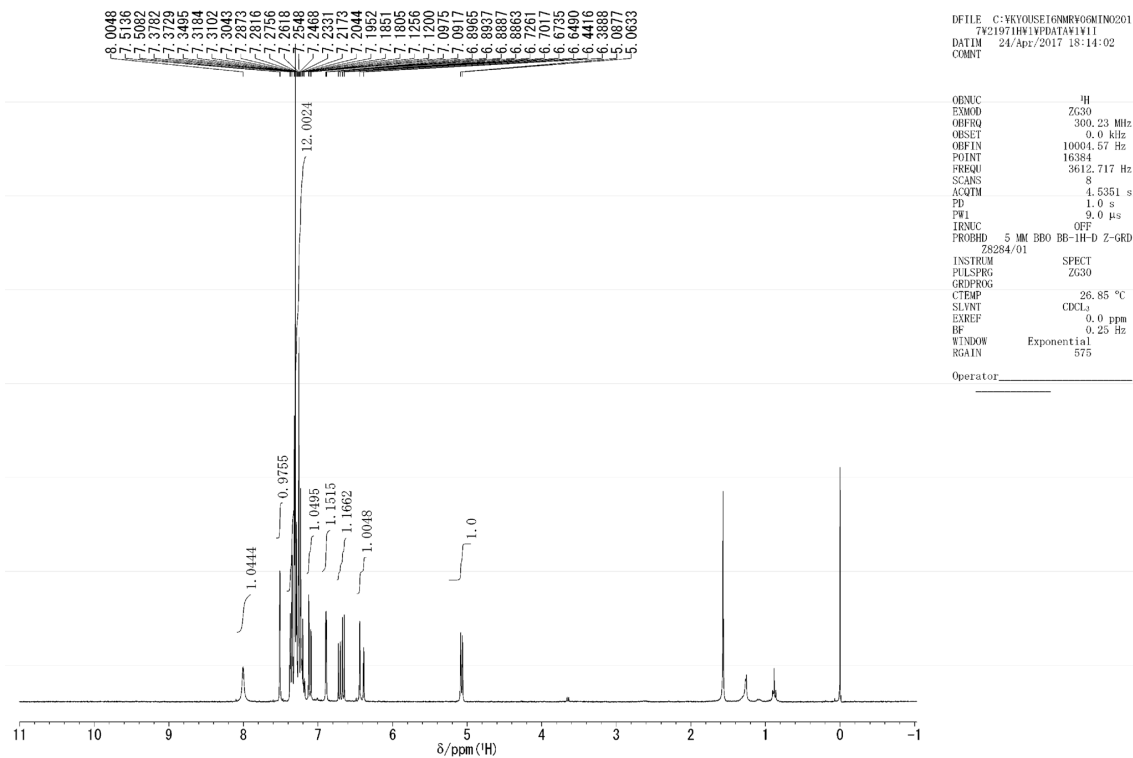
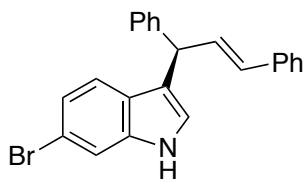
peak name

#	ピーク名	CH	TR [min]	area [μV·sec]	area%
1	Unknown	3	91.7	898727	5.406
2	Unknown	3	97.8	15726310	94.594

(±)-9g



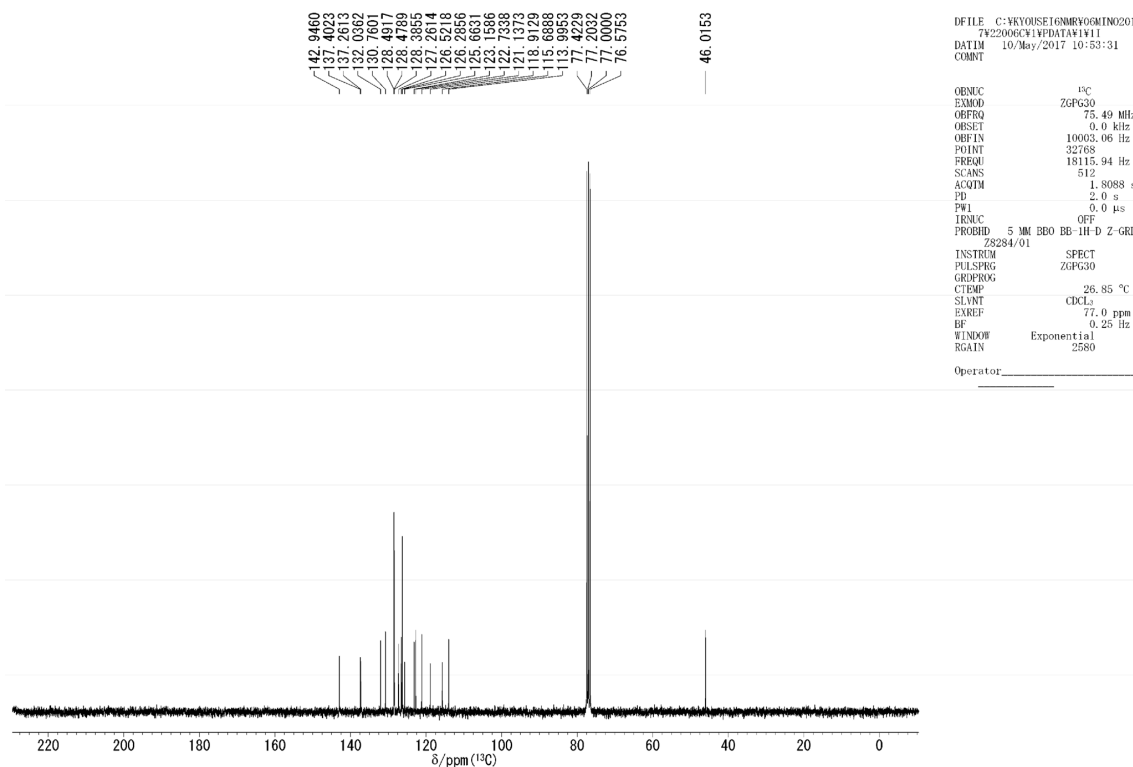
¹H and ¹³C NMR, and chiral phase HPLC chart of (*R*)-**9h** (Table 3, entry 8)



DFILE C:\KYOUSETI6\NMR\9G\IN0201
 7\21971\H1\VPDATA\H1\11
 DATIM 24/Apr/2017 18:14:02
 COMBT

OBNUC ¹H
 EXMOD ZG30
 OFFRQ 300.23 MHz
 ORSET 0.0 kHz
 OFFIN 10004.57 Hz
 POINT 16384
 FREQH 3612.717 Hz
 SCANS 8
 ACQTM 4.5351 s
 PD 1.0 s
 PW 9.0 μs
 IRNUC OFF
 PROBH 5 MM BBO BB-1H-D Z-GRD
 ZG284/01
 INSTRUM SPECT
 PULSPRG ZG30
 GROPRG
 CTMP 26.85 °C
 SLVNT CDCl₃
 EXREF 0.0 ppm
 BF 0.25 Hz
 WINDOW Exponential
 RGAIN 575

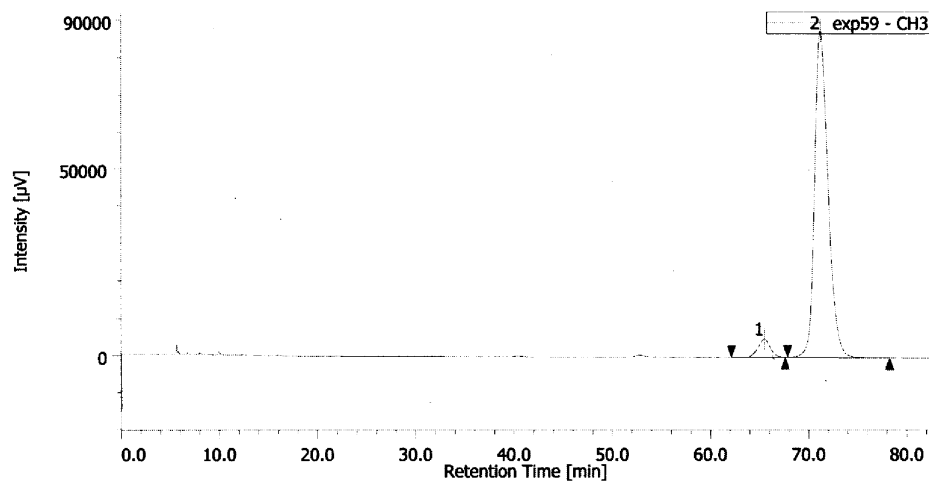
Operator _____



DFILE C:\KYOUSETI6\NMR\9G\IN0201
 7\2200CF\VPDATA\H1\11
 DATIM 10/May/2017 10:53:31
 COMBT

OBNUC ¹³C
 EXMOD ZGPG30
 OFFRQ 75.49 MHz
 ORSET 0.0 kHz
 OFFIN 10003.06 Hz
 POINT 32768
 FREQH 18115.94 Hz
 SCANS 512
 ACQTM 1.8088 s
 PD 2.0 s
 PW 0.0 μs
 IRNUC OFF
 PROBH 5 MM BBO BB-1H-D Z-GRD
 ZG284/01
 INSTRUM SPECT
 PULSPRG ZGPG30
 GROPRG
 CTMP 26.85 °C
 SLVNT CDCl₃
 EXREF 77.0 ppm
 BF 0.25 Hz
 WINDOW Exponential
 RGAIN 2580

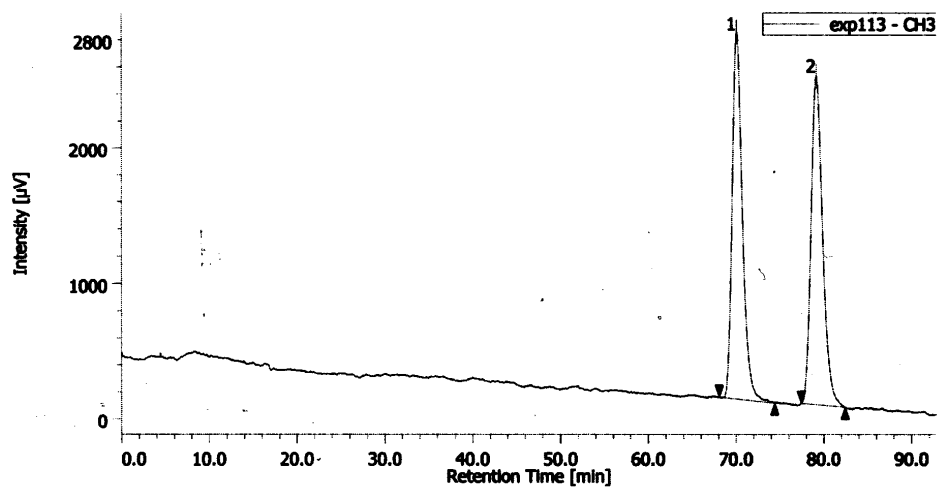
Operator _____



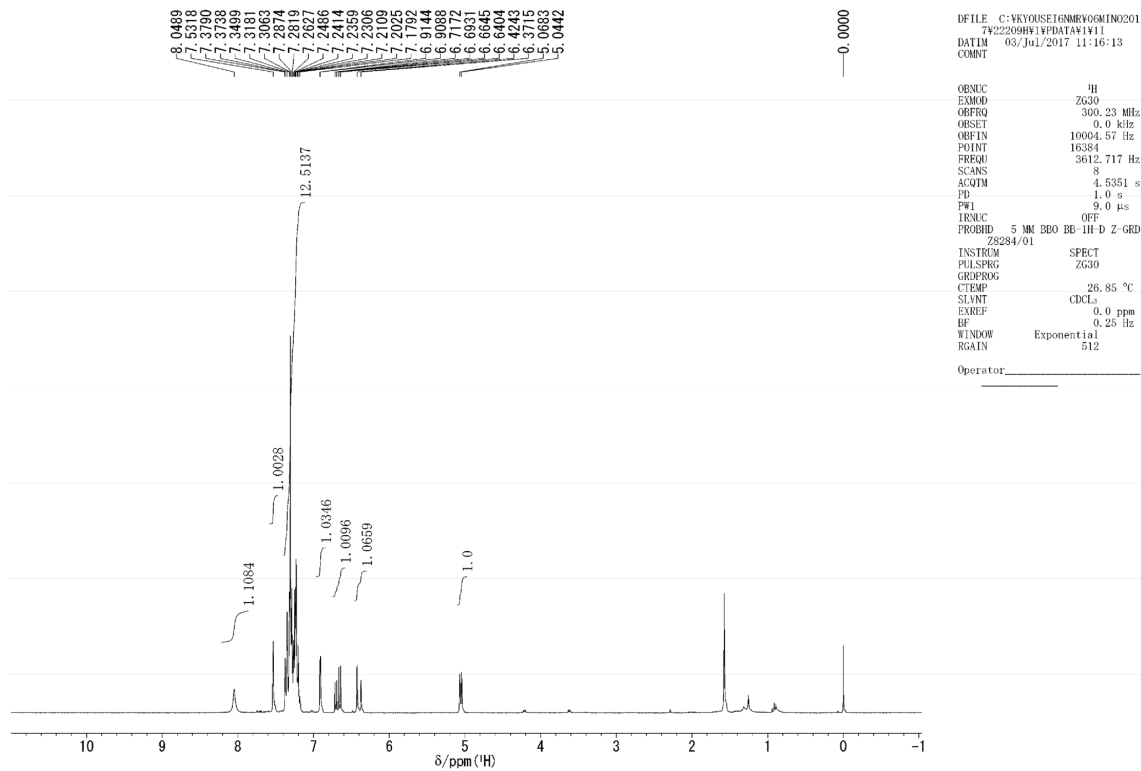
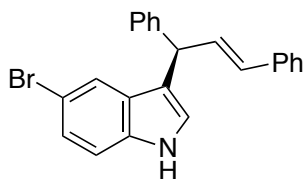
peak name area height area%

Peak	Retention Time [min]	Area	Height	Area%
1	65.500	437396	5042	4.937
2	71.208	8421365	87567	95.063

(±)-9h



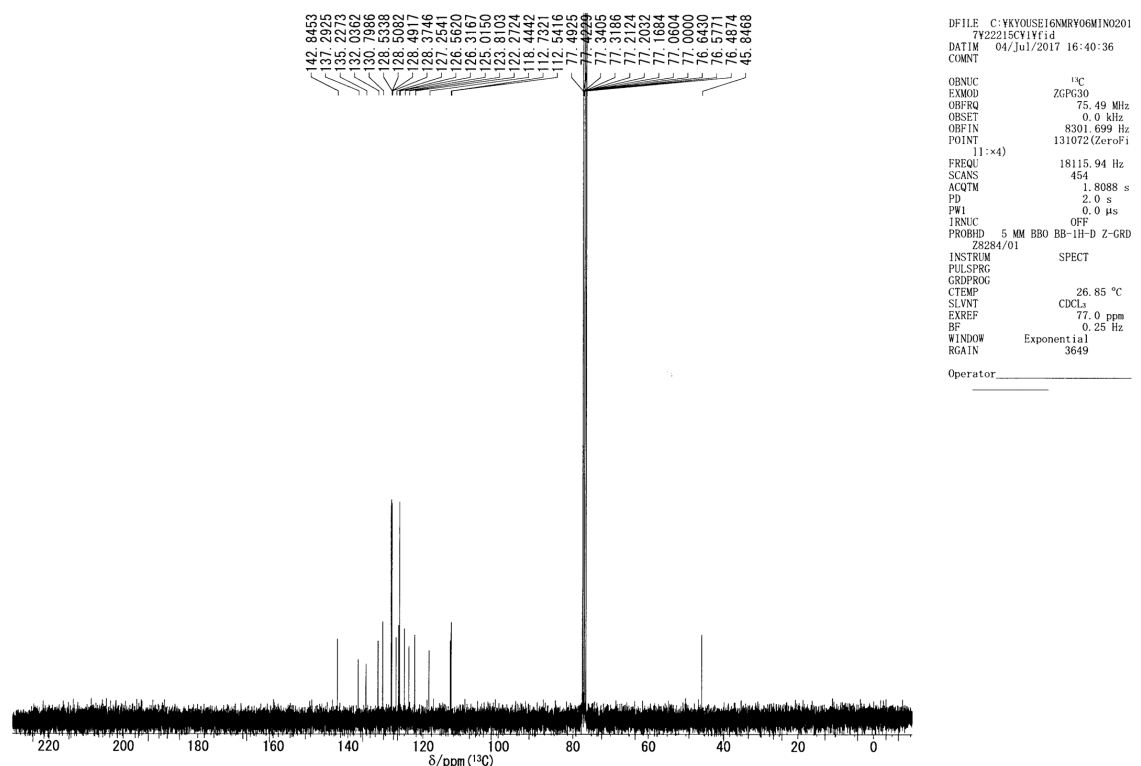
¹H and ¹³C NMR, and chiral phase HPLC chart of (*R*)-**9i** (Table 3, entry 9)



DFILE C:\YK\OUSE16\NMR\YOGMIN0201
7422209HW\FDATA\14111
DATIM 03/Jul/2017 11:16:13
COMNT

ORNUC ¹H
EXMOD ZG30
OBFREQ 300.23 MHz
OBSET 0.0 kHz
OBFIN 10004.57 Hz
POINT 16384
FREQU 3612.717 Hz
SCANS 8
ACQTM 4.5351 s
PD 1.0 s
PWL 9.0 μ s
IRNUC OFF
PROBHD 5 MM BBO BB-1H-D Z-GRD
Z8284/01
INSTRM SPECT
PULSPRG ZG30
GRDPRG
CTEMP 26.85 $^{\circ}$ C
SLVNT CDCl₃
EXREF 0.0 ppm
BF 0.25 Hz
WINDOW Exponential
RGAIN 512

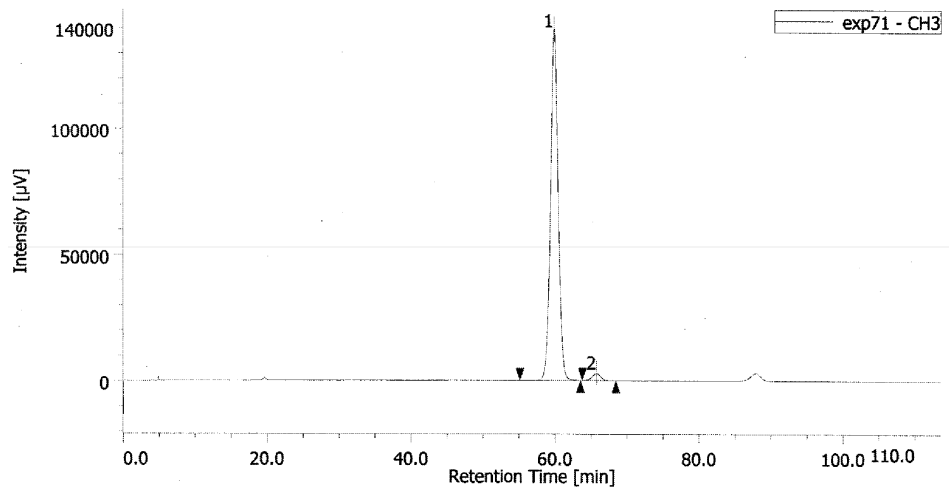
Operator _____



DFILE C:\YK\OUSE16\NMR\YOGMIN0201
7422213CYIYFid
DATIM 04/Jul/2017 16:40:36
COMNT

ORNUC ¹³C
EXMOD ZGPC30
OBFREQ 75.49 MHz
OBSET 0.0 kHz
OBFIN 8301.699 Hz
POINT 131072(ZeroPi
11:~4)
FREQU 18115.94 Hz
SCANS 454
ACQTM 1.8088 s
PD 2.0 s
PWL 0.0 μ s
IRNUC OFF
PROBHD 5 MM BBO BB-1H-D Z-GRD
Z8284/01
INSTRM SPECT
PULSPRG
GRDPRG
CTEMP 26.85 $^{\circ}$ C
SLVNT CDCl₃
EXREF 77.0 ppm
BF 0.25 Hz
WINDOW Exponential
RGAIN 3649

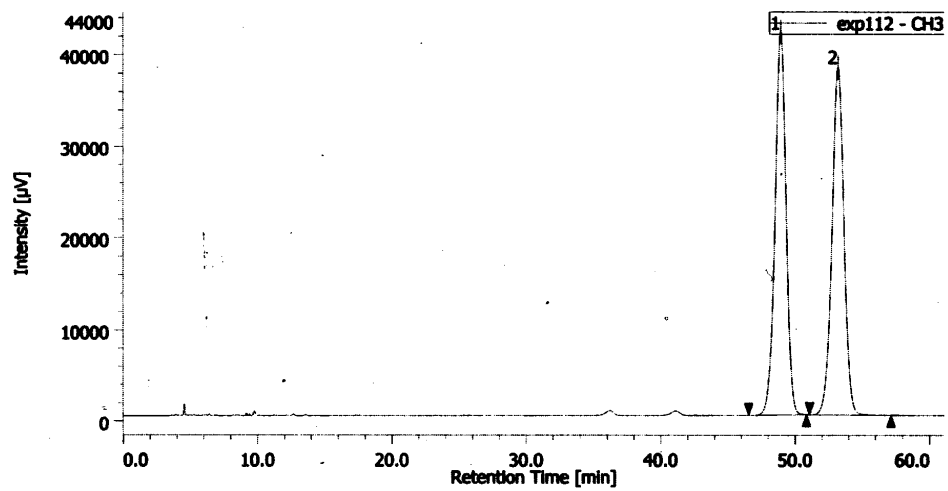
Operator _____



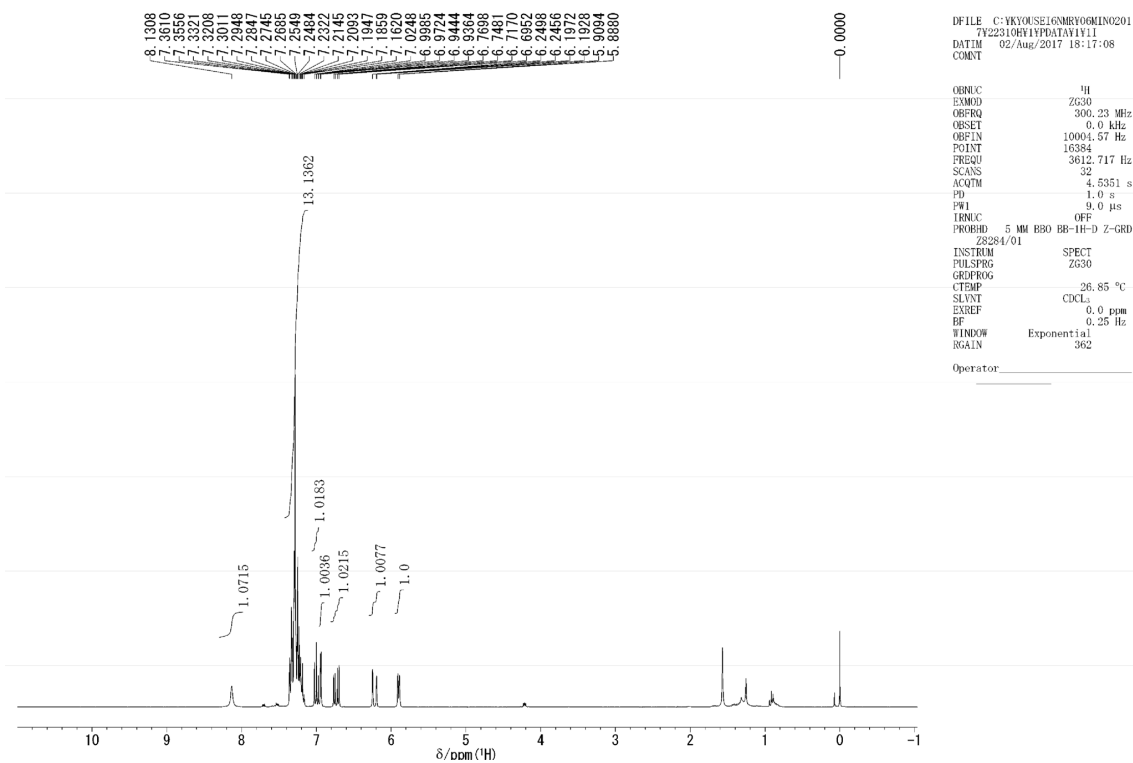
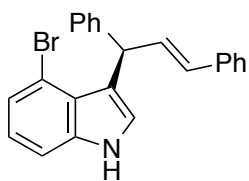
peak name

#	ピーク名	CH	TR [min]	area [$\mu\text{V}\cdot\text{sec}$]	area%
1	Unknown	3	59.850	10094480	97.833
2	Unknown	3	65.742	223582	2.167

(\pm)-9i

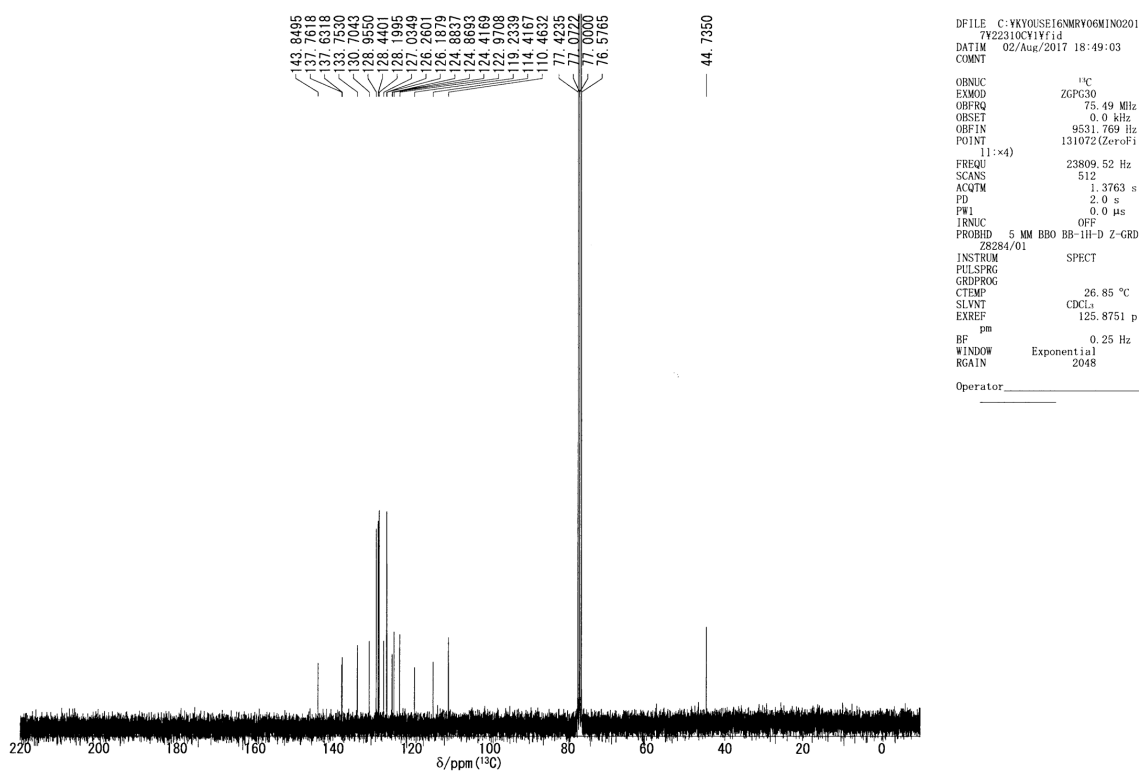


¹H and ¹³C NMR, and chiral phase HPLC chart of (*R*)-**9j** (Table 3, entry 10)



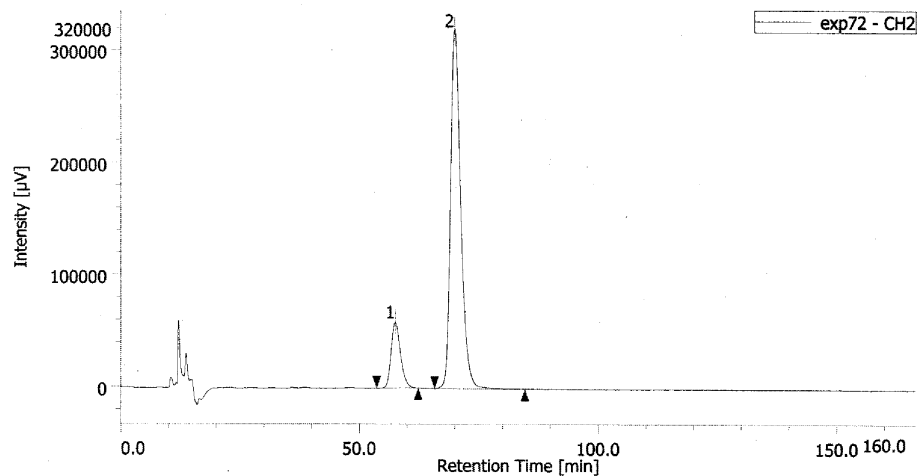
DFILE C:\KYOUSEI\6NMR\VO6M\N0201
7Y22310HV1PDATA\V1Y11
DATIM 02/Aug/2017 18:17:08
COMNT

OBNUC ¹H
EXMOD ZG30
OBFRQ 300.23 MHz
OBSET 0.0 kHz
OBFIN 10004.57 Hz
POINT 16384
FREQU 3612.717 Hz
SCANS 32
ACQTM 4.5351 s
PD 1.0 s
PWI 9.0 μs
IRNUC OFF
PROBHD 5 MM BBO BB-HH-D Z-GRD
28284/01
INSTRUM SPECT
PULSPRG ZG30
GRDPRG
CTEMP 26.85 °C
SLVNT CDCL₃
EXREF 0.0 ppm
BF 0.25 Hz
WINDOW Exponential
RGAIN 362
Operator



DFILE C:\KYOUSEI\6NMR\VO6M\N0201
7Y22310CV1Y11d
DATIM 02/Aug/2017 18:49:03
COMNT

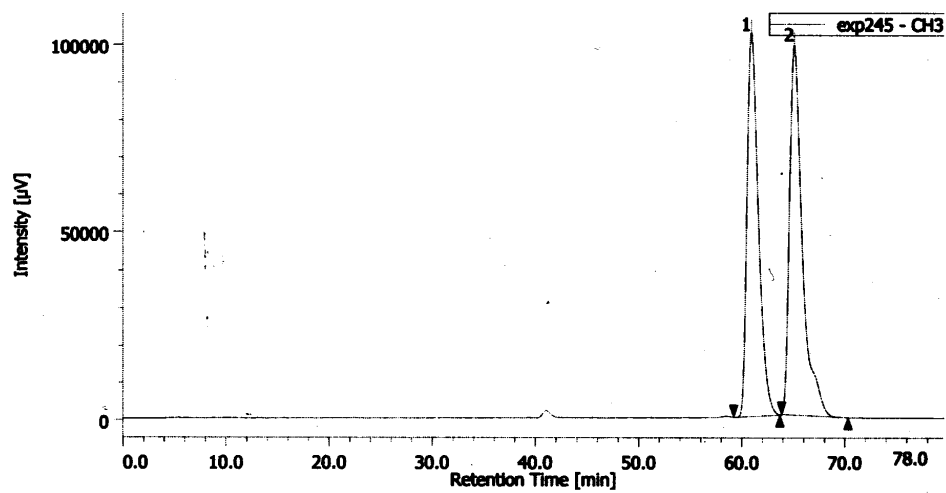
OBNUC ¹³C
EXMOD ZGPG30
OBFRQ 75.49 MHz
OBSET 0.0 kHz
OBFIN 9531.769 Hz
POINT 131072 (ZeroFi)
11: x4
FREQU 23809.52 Hz
SCANS 512
ACQTM 1.3763 s
PD 2.0 s
PWI 0.0 μs
IRNUC OFF
PROBHD 5 MM BBO BB-HH-D Z-GRD
28284/01
INSTRUM SPECT
PULSPRG
GRDPRG
CTEMP 26.85 °C
SLVNT CDCL₃
EXREF 125.8751 p
pm
BF 0.25 Hz
WINDOW Exponential
RGAIN 2048
Operator



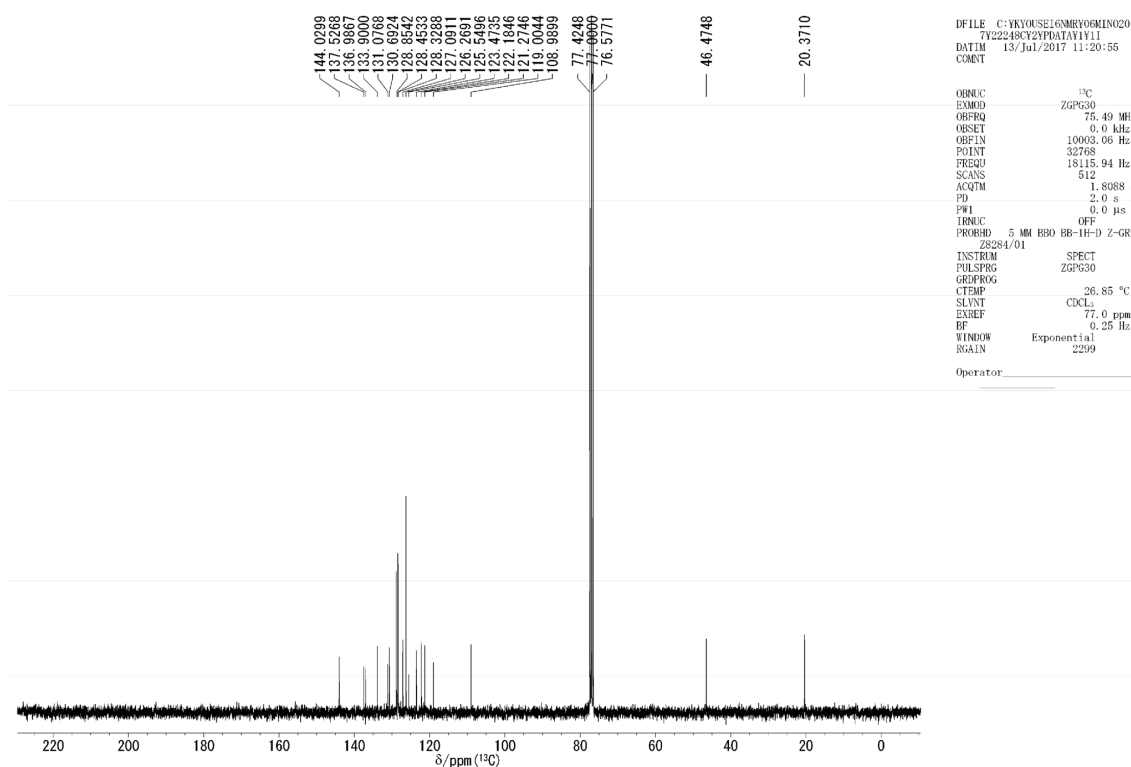
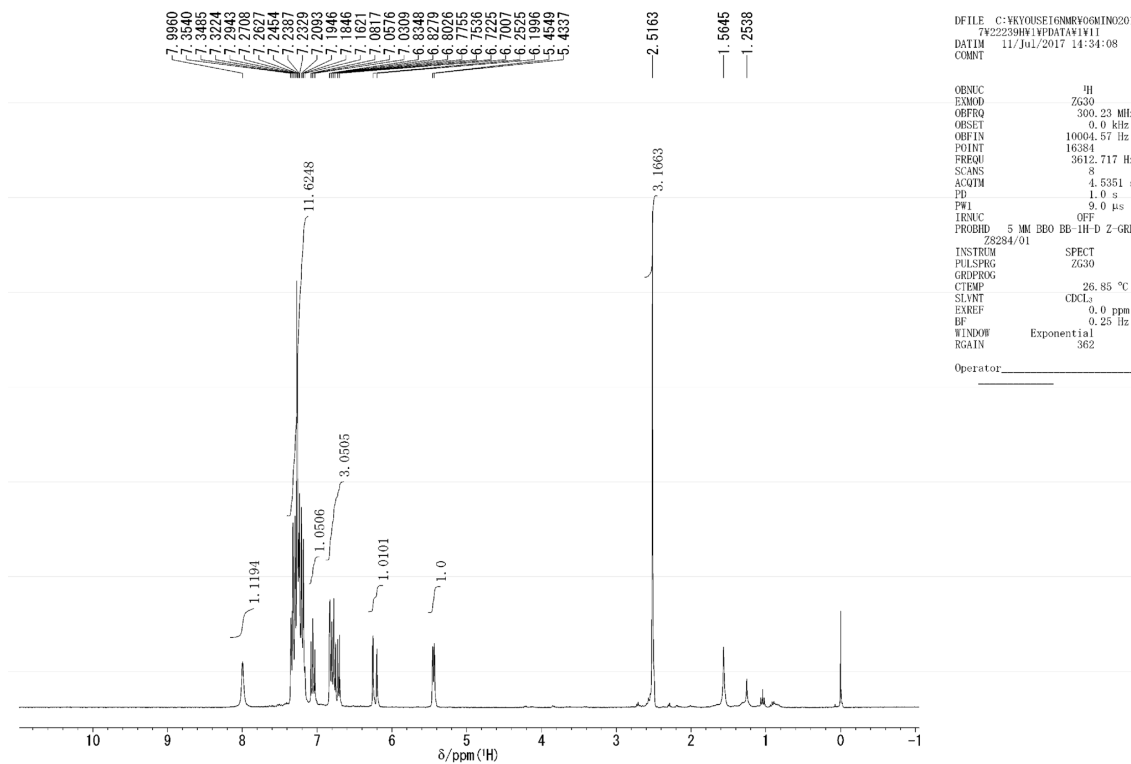
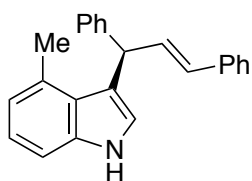
peak name

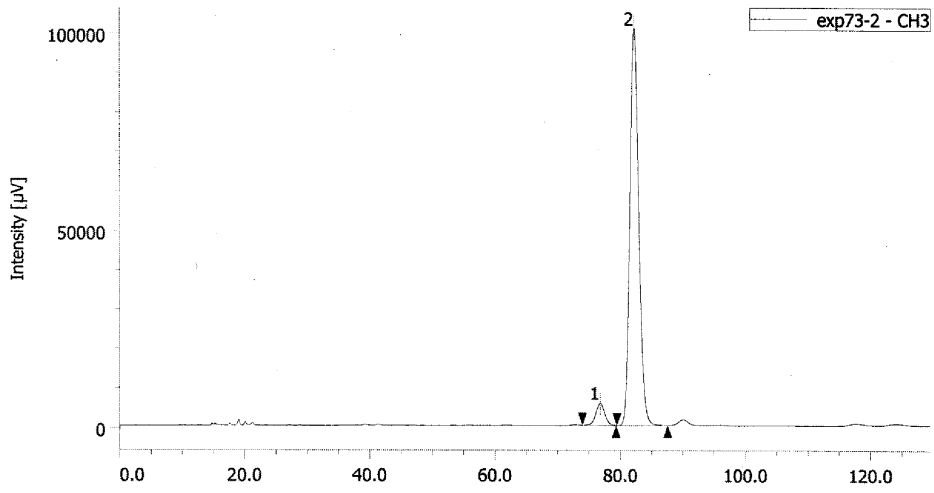
#	CH	CH	tR [min]	area [µV·sec]	area%
1	Unknown	2	57.525	8016504	14.436
2	Unknown	2	69.950	47514564	85.564

(±)-9j



¹H and ¹³C NMR, and chiral phase HPLC chart of (*R*)-**9k** (Table 3, entry 11)

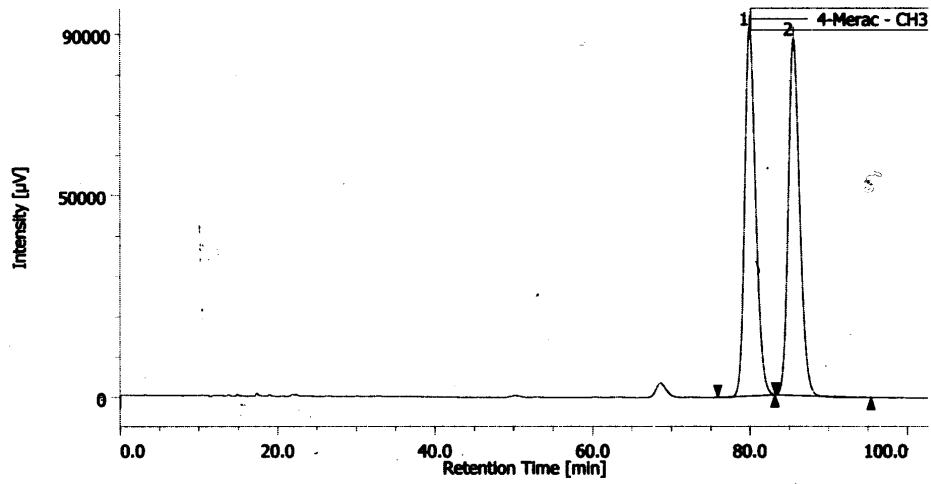




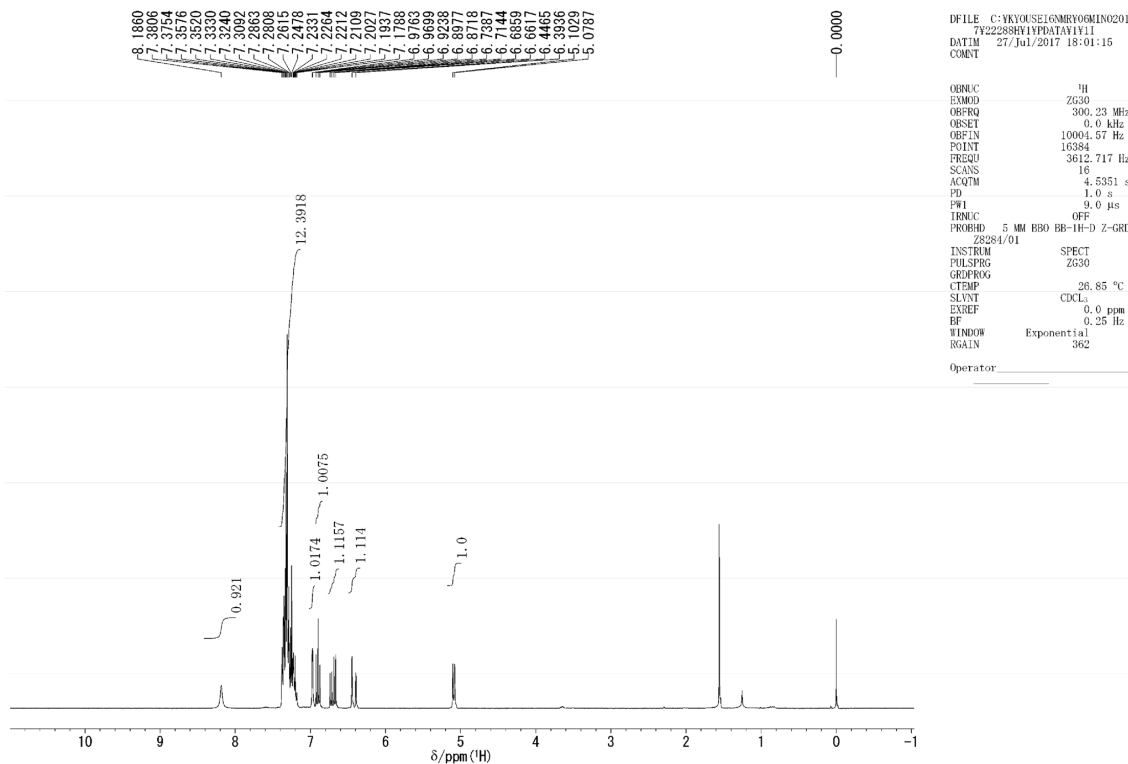
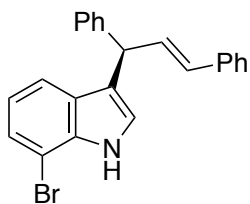
peak name

#	ピーク名	CH	TR [min]	area [μV·sec]	area%
1	Unknown	3	76.8	539268	5.155
2	Unknown	3	82.1	9922279	94.845

(±)-9k

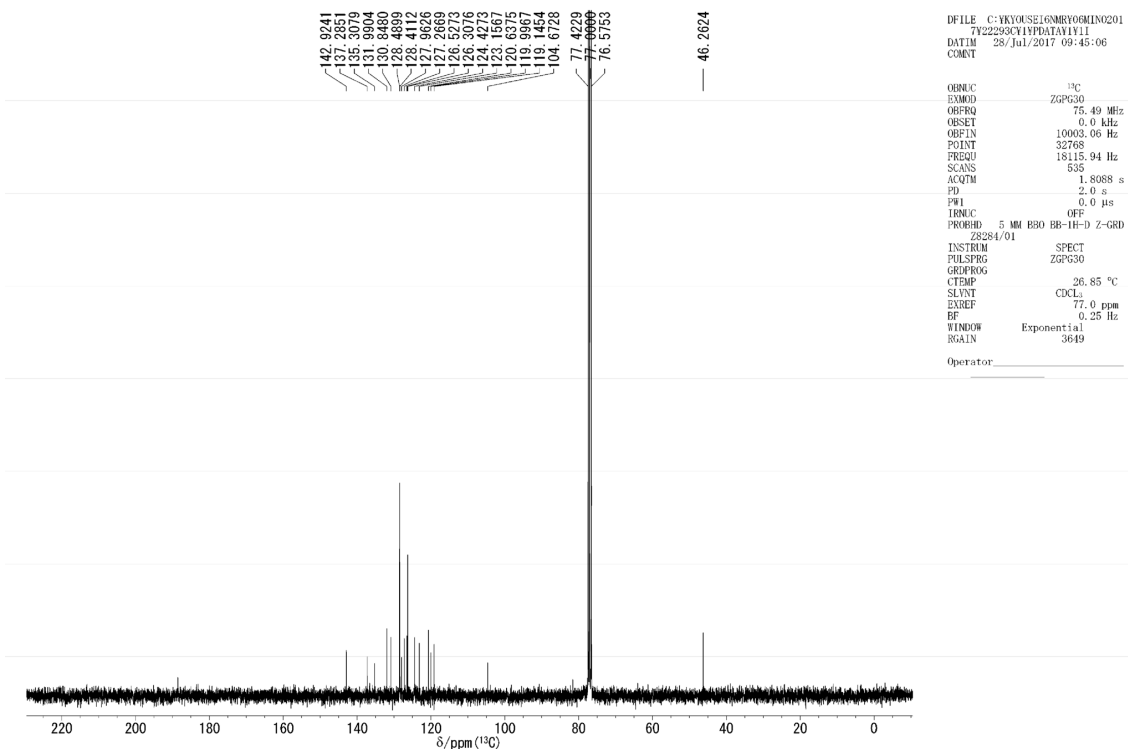


¹H and ¹³C NMR, and chiral phase HPLC chart of (*R*)-**9I** (Table 3, entry 12)



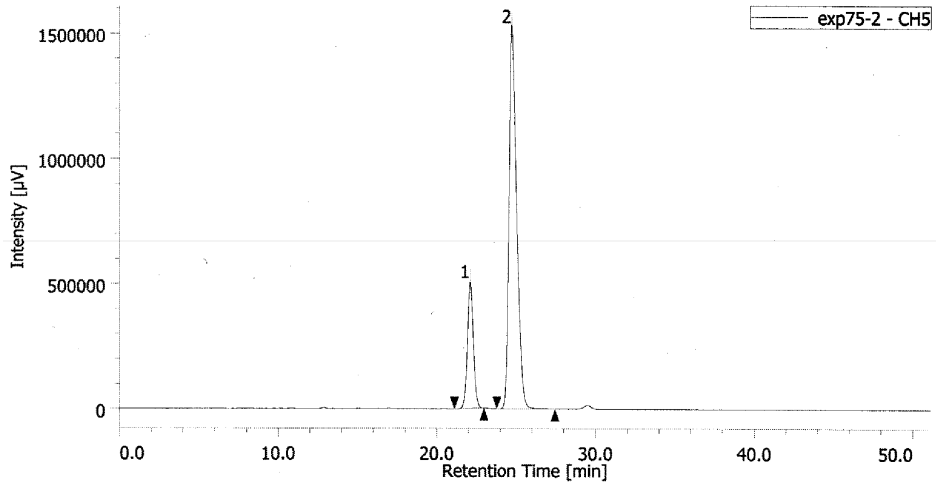
DFILE C:\KYOUSEI\6MR\Y06M10\201
7Y22288Y1YPRATAY11
DATIM 27/Jul/2017 18:01:15
COMT

ORNUC ¹H
EXMOD ZG30
ORFRQ 300.23 MHz
ORSET 0.0 kHz
ORFIN 10004.57 Hz
POINT 16384
FREQU 3612.717 Hz
SCANS 16
ACQTM 4.5351 s
PD 1.0 s
PWI 9.0 μs
IRNUC OFF
PROBHD 5 MM BBO BB-HH-D Z-GRD
ZS284/01
INSTRUM SPECT
PULSPRG ZG30
GRDPRG
CTEMP 26.85 °C
SUNVT CDCL3
EXREF 0.0 ppm
BF 0.25 Hz
WINDOW Exponential
RGAIN 362
Operator



DFILE C:\KYOUSEI\6MR\Y06M10\201
7Y22288Y1YPRATAY11
DATIM 28/Jul/2017 09:45:06
COMT

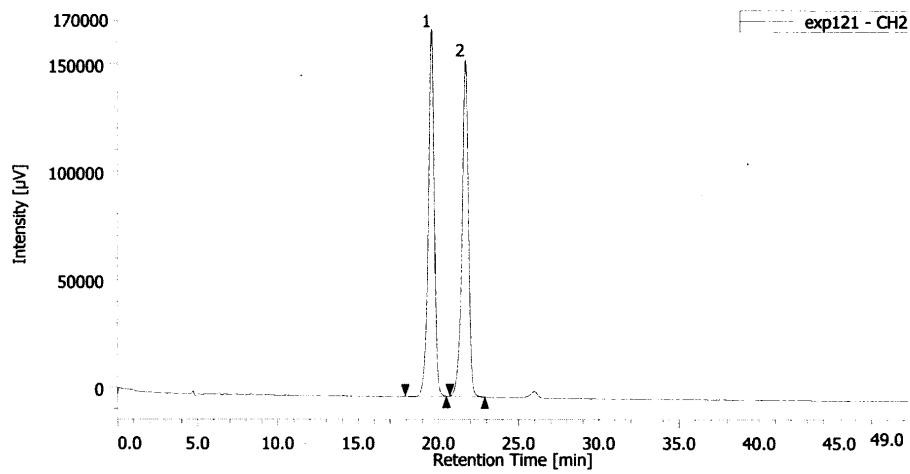
ORNUC ¹³C
EXMOD ZGPG30
ORFRQ 75.49 MHz
ORSET 0.0 kHz
ORFIN 10003.06 Hz
POINT 32768
FREQU 18115.94 Hz
SCANS 535
ACQTM 1.8098 s
PD 2.0 s
PWI 0.0 μs
IRNUC OFF
PROBHD 5 MM BBO BB-HH-D Z-GRD
ZS284/01
INSTRUM SPECT
PULSPRG ZGPG30
GRDPRG
CTEMP 26.85 °C
SUNVT CDCL3
EXREF 77.0 ppm
BF 0.25 Hz
WINDOW Exponential
RGAIN 3649
Operator



peak name

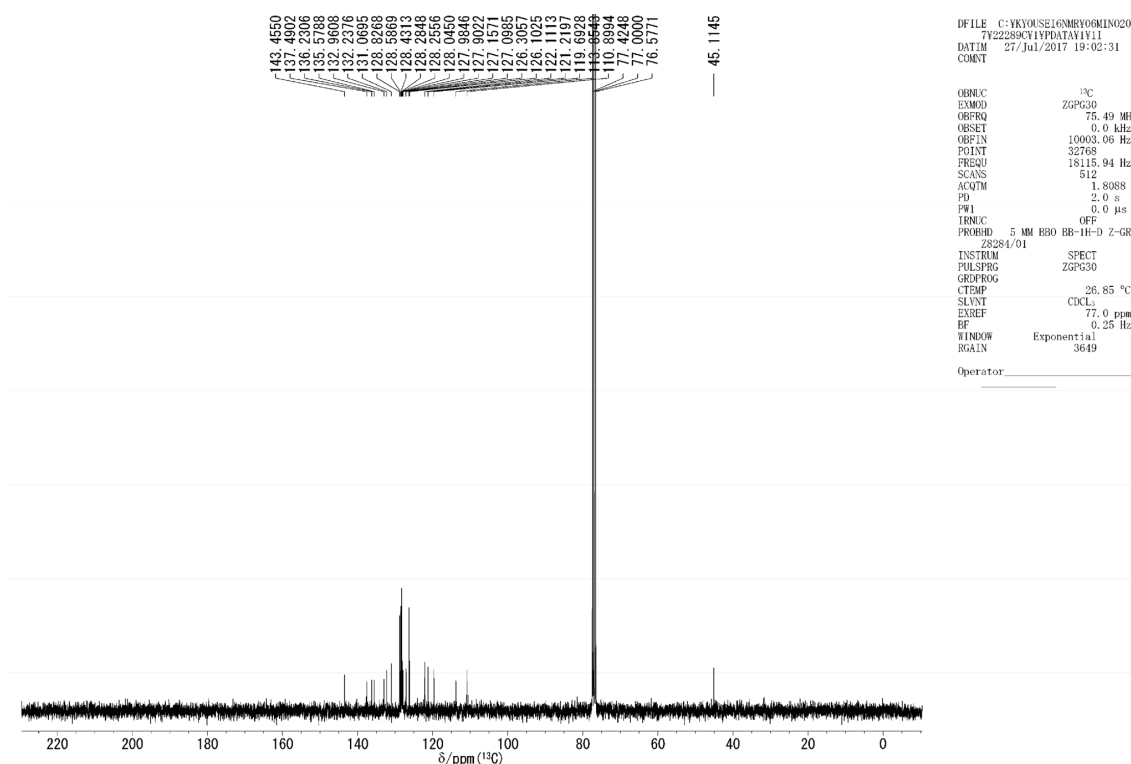
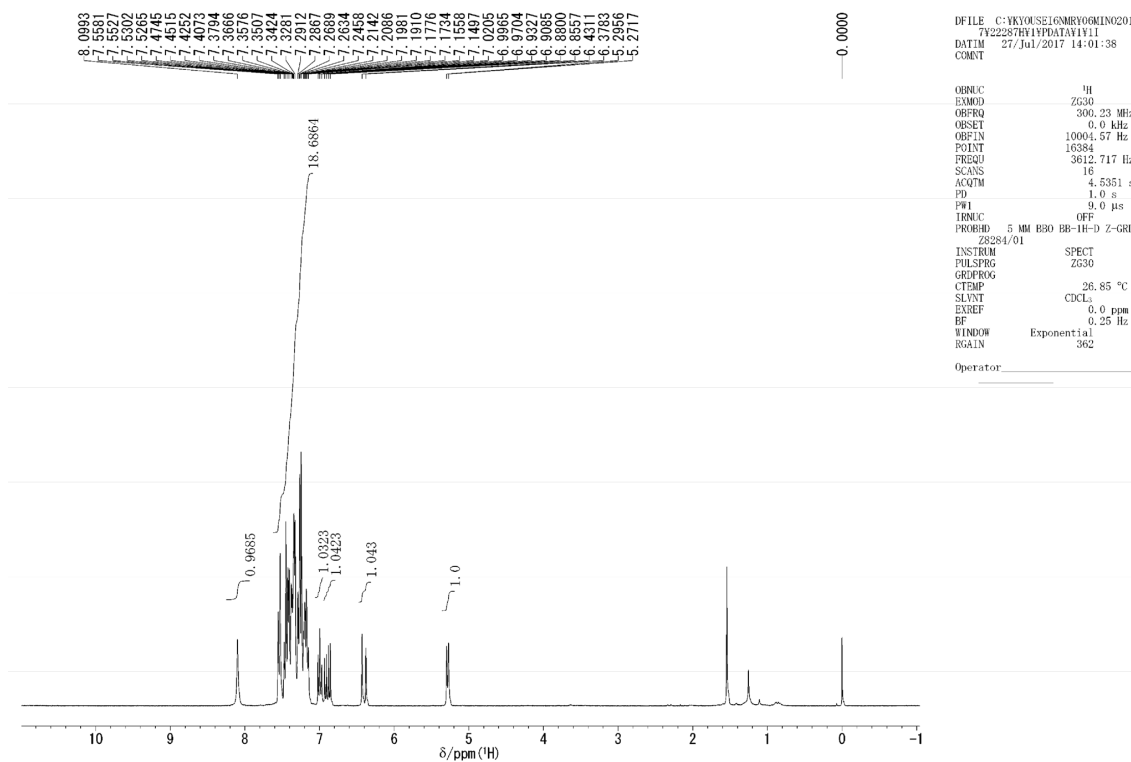
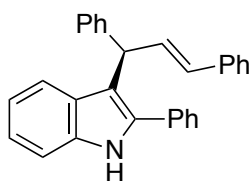
#	CH	TR [min]	area [$\mu\text{V}\cdot\text{sec}$]	area%	
1	Unknown	5	22.100	13035572	20.386
2	Unknown	5	24.710	50907963	79.614

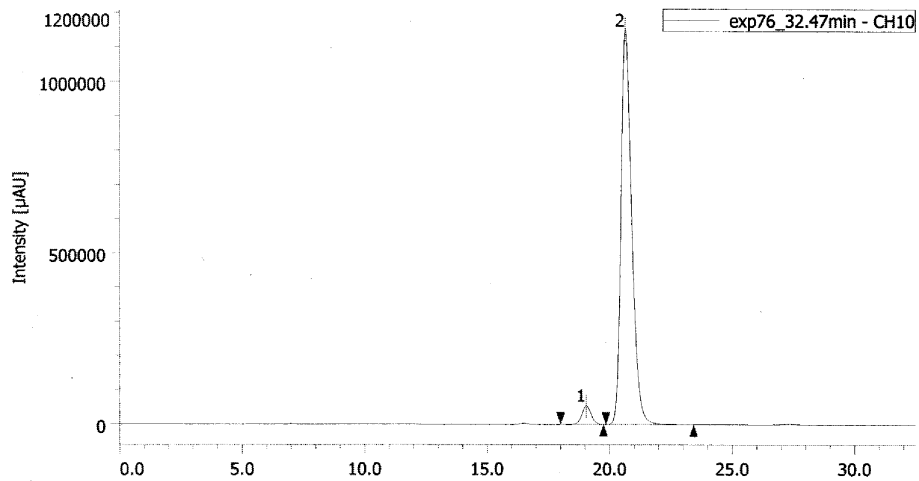
(±)-91



peak name

¹H and ¹³C NMR, and chiral phase HPLC chart of (R)-9m (Table 2, entry 13)





peak name

#	ピーク名	CH	tR [min]	area [μV.sec]	area%
1	Unknown	10	19.0	1394440	3.830
2	Unknown	10	20.6	35014510	96.170

(±)-9m

