

Enantioselective Synthesis of *H*-Phosphinic Acids Bearing Natural Amino Acids Residues

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Supporting Information

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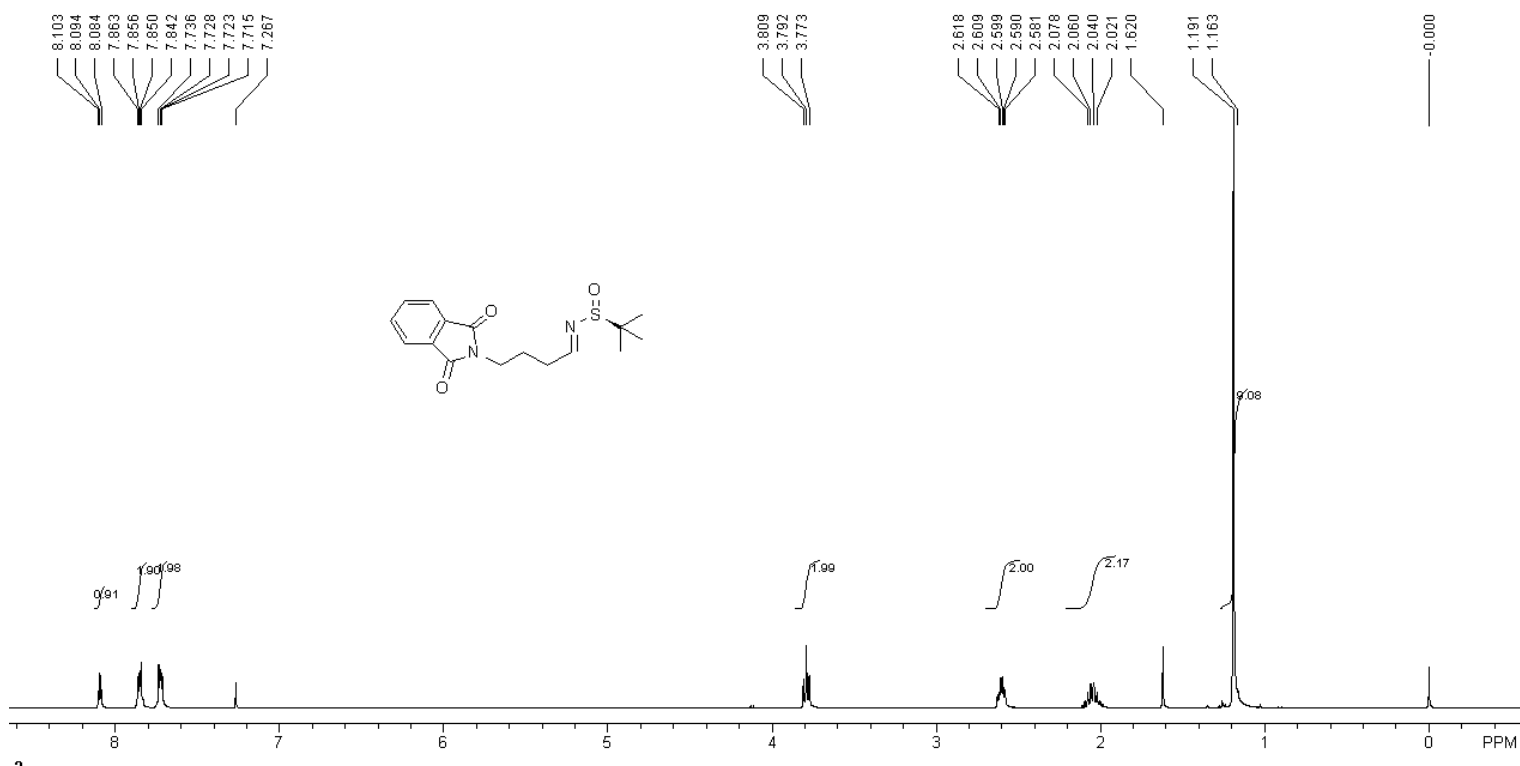
1. NMR Spectra of Compounds 1–4, 9, 10.

General Experimental Procedures.

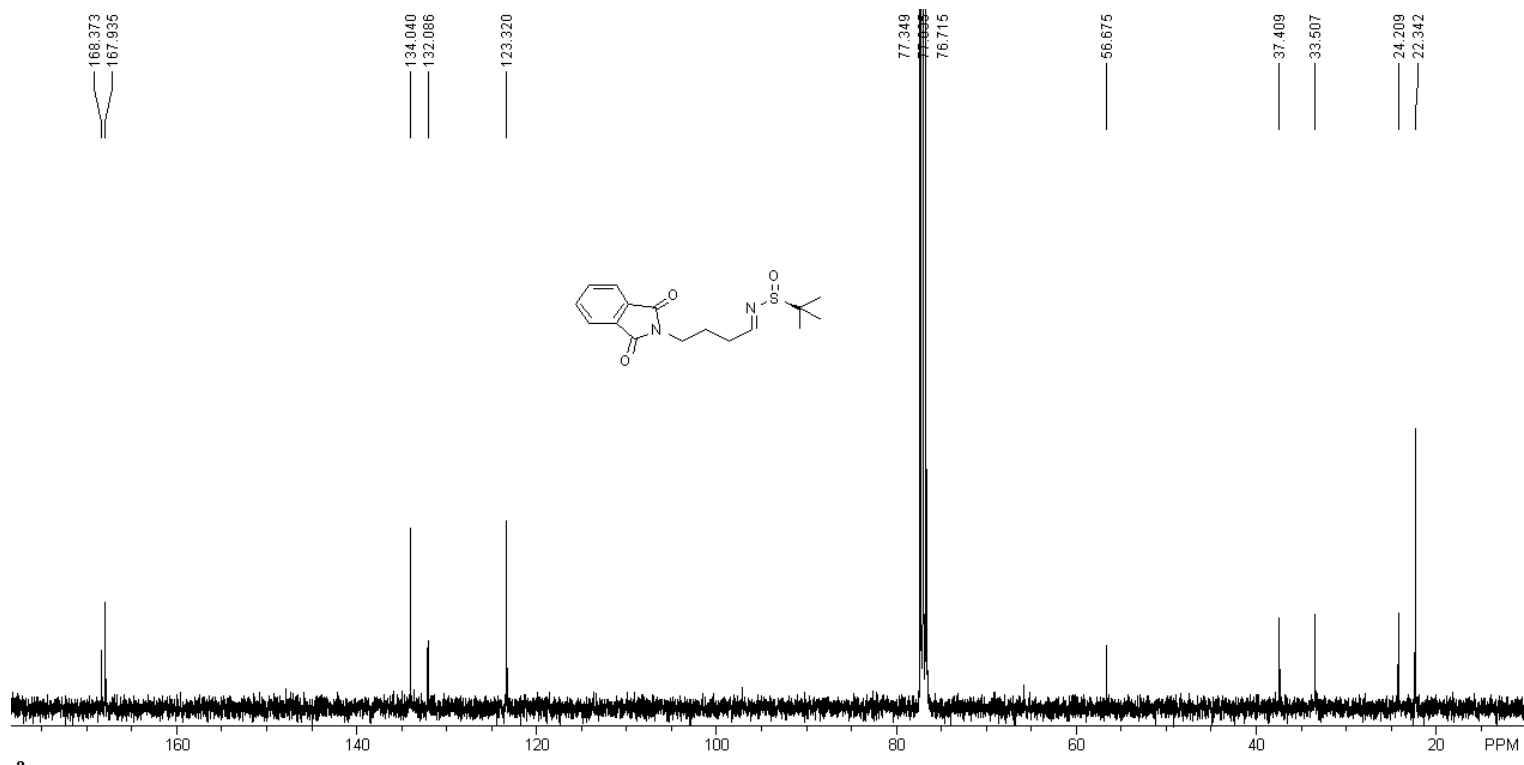
Unless otherwise noted, all the reactions were performed under nitrogen atmosphere with dried solvents. Dichloromethane was distilled from CaH_2 under nitrogen. THF was distilled from sodium under nitrogen. All other commercial reagents were used without further purification. All the reactions were monitored by TLC. ^1H NMR spectra (300 or 400 MHz) were recorded on a spectrometer with CDCl_3 as solvent and trimethylsilane (TMS) as an internal standard. ^{13}C NMR (100 MHz or 75 MHz) spectra were recorded on a spectrometer with CDCl_3 as solvent. ^{31}P NMR (121 or 162 MHz) spectra were recorded on a spectrometer with CDCl_3 as the solvent with 85% H_3PO_4 as external standard. Mass spectra were performed at 2020 eV. HR-MS were detected by FTICR. Elemental analyses were conducted in an apparatus. Optical rotation values were measured on a spectrometer. IR spectra were recorded in CHCl_3 on a spectrometer.

Compound 1b:

^1H NMR:

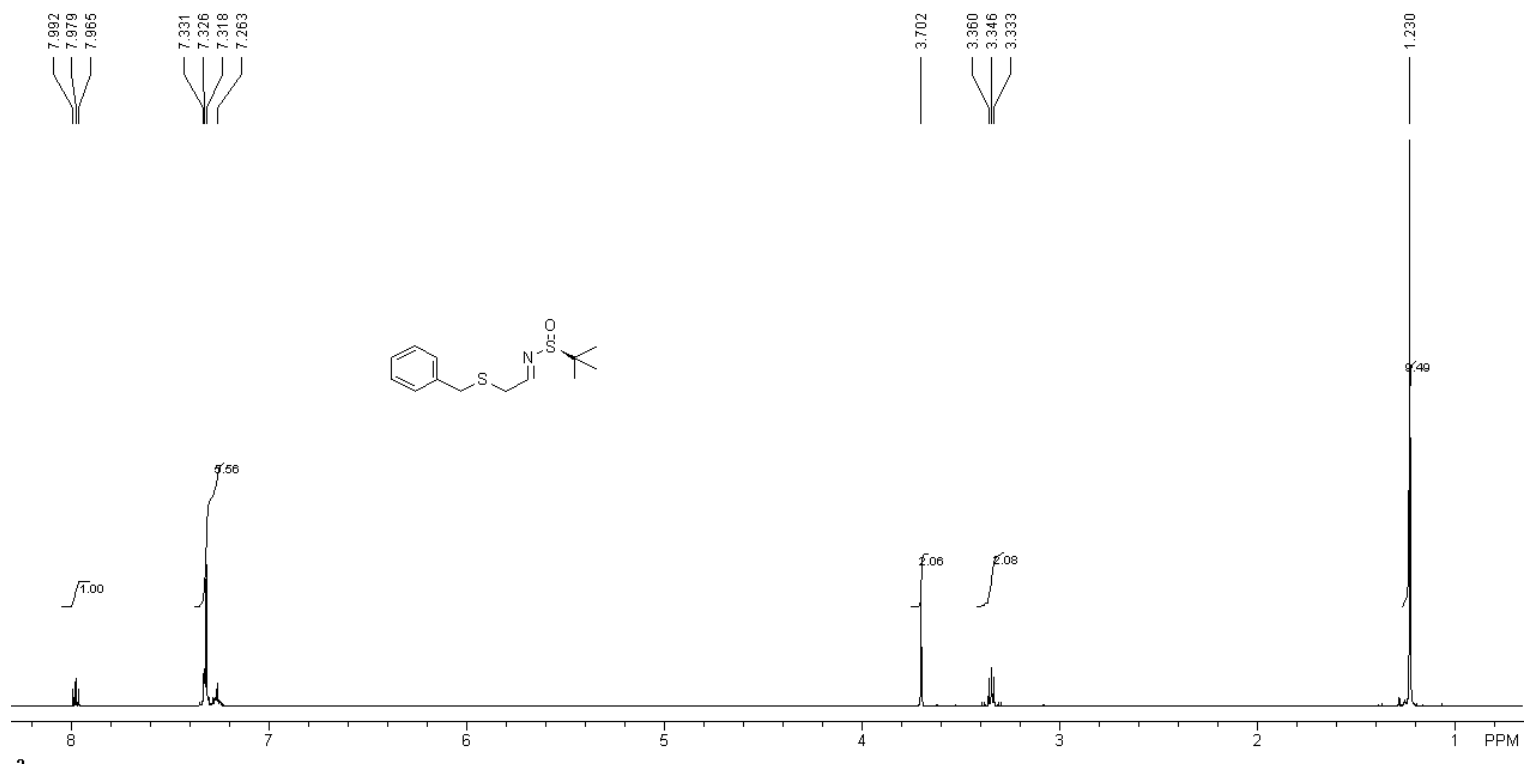


^{13}C NMR:

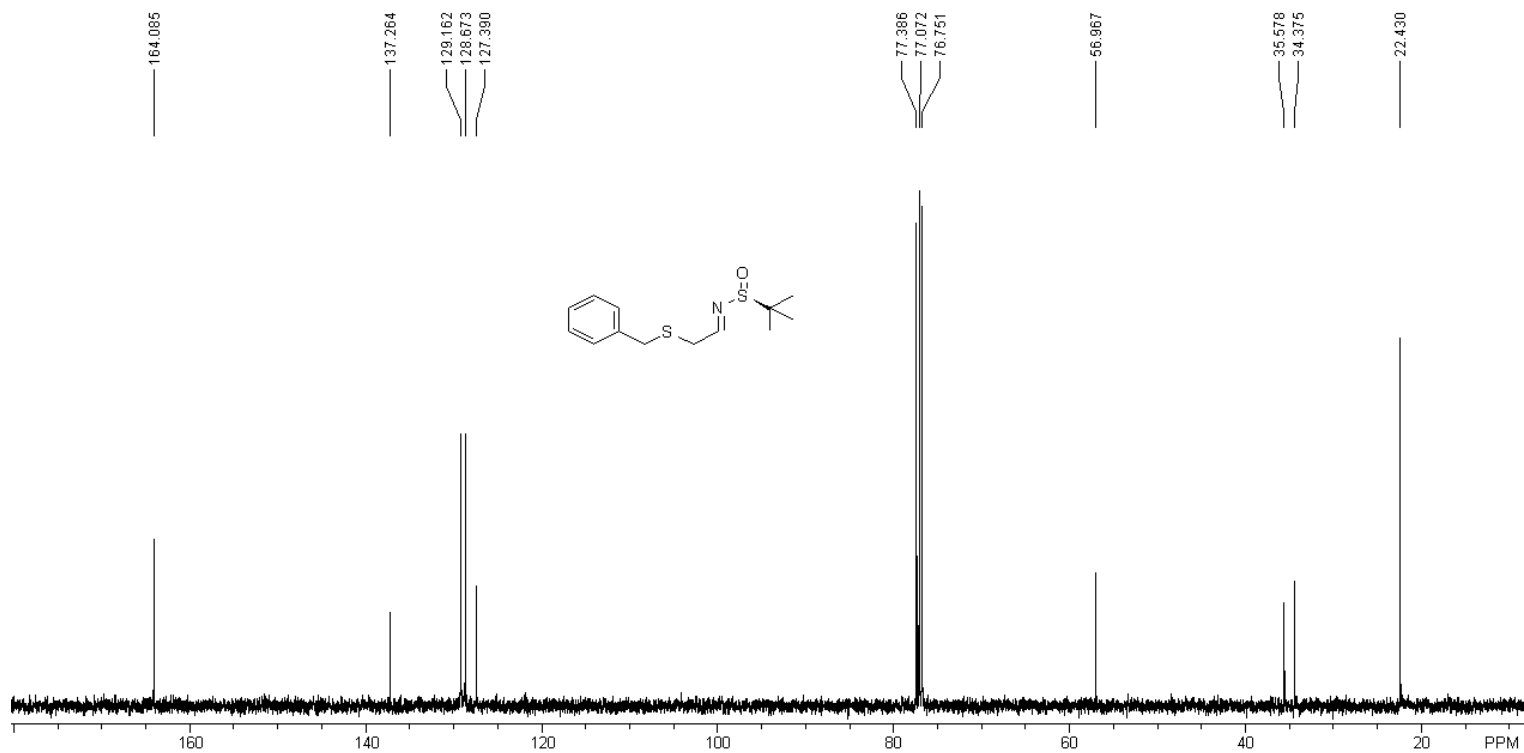


Compound 1c:

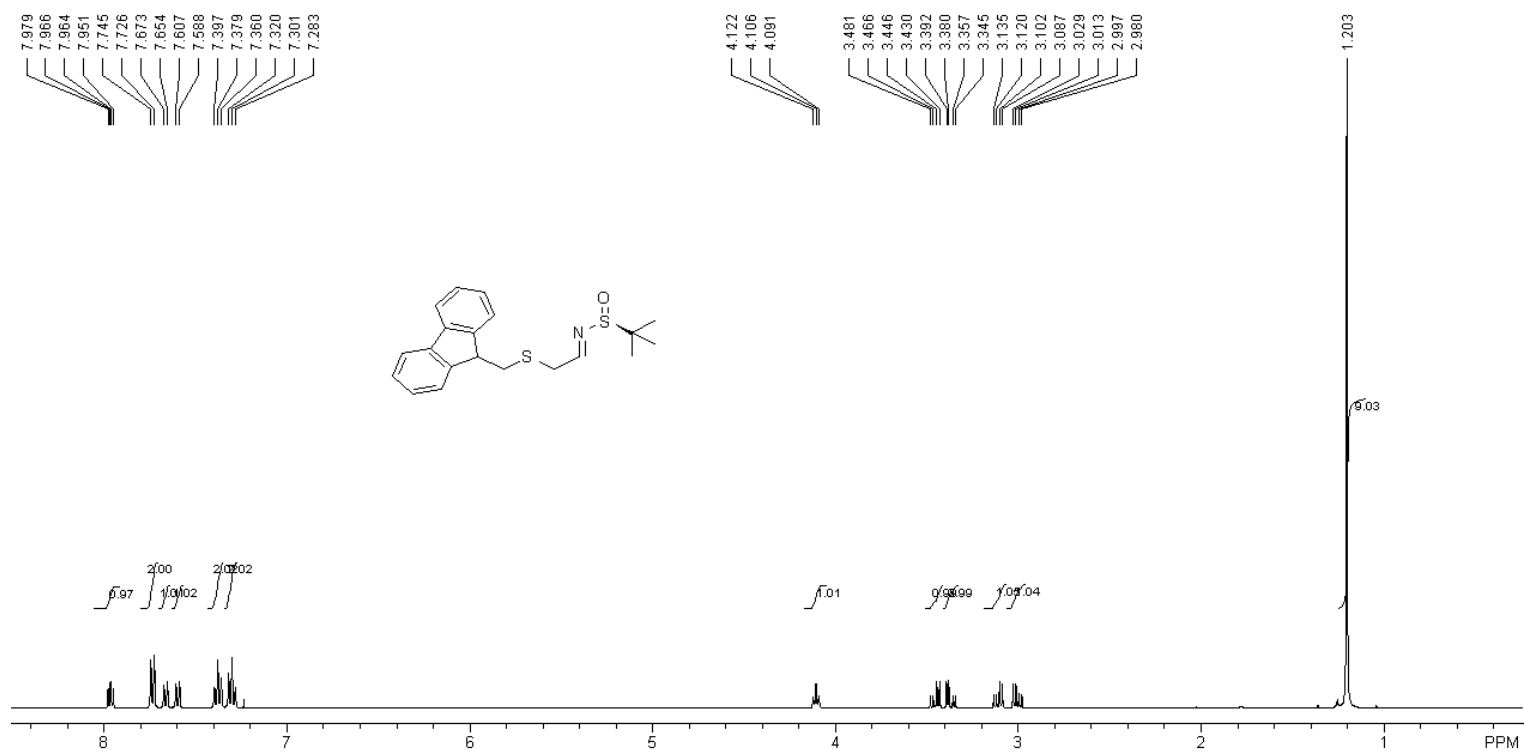
¹H NMR:



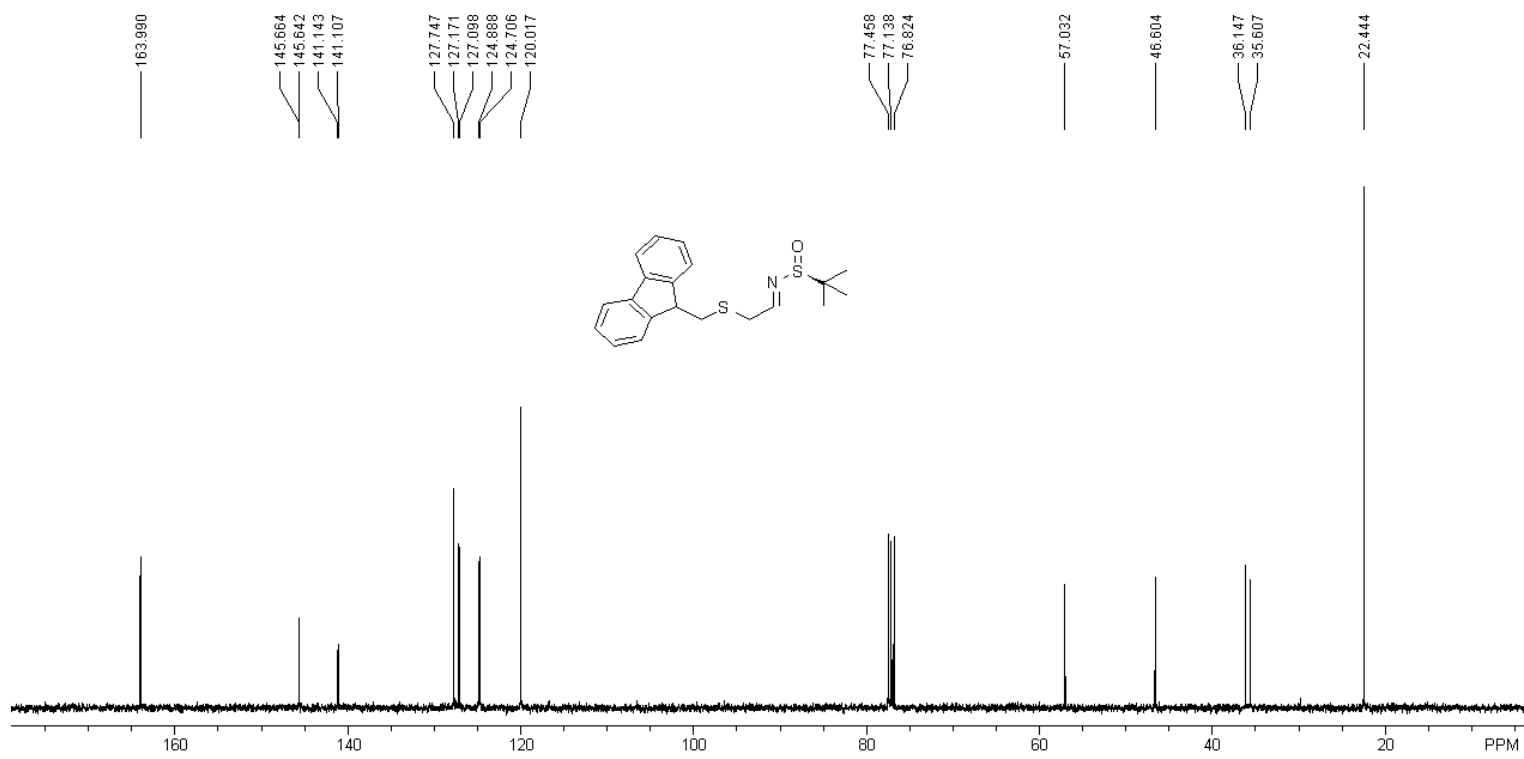
¹³C NMR:



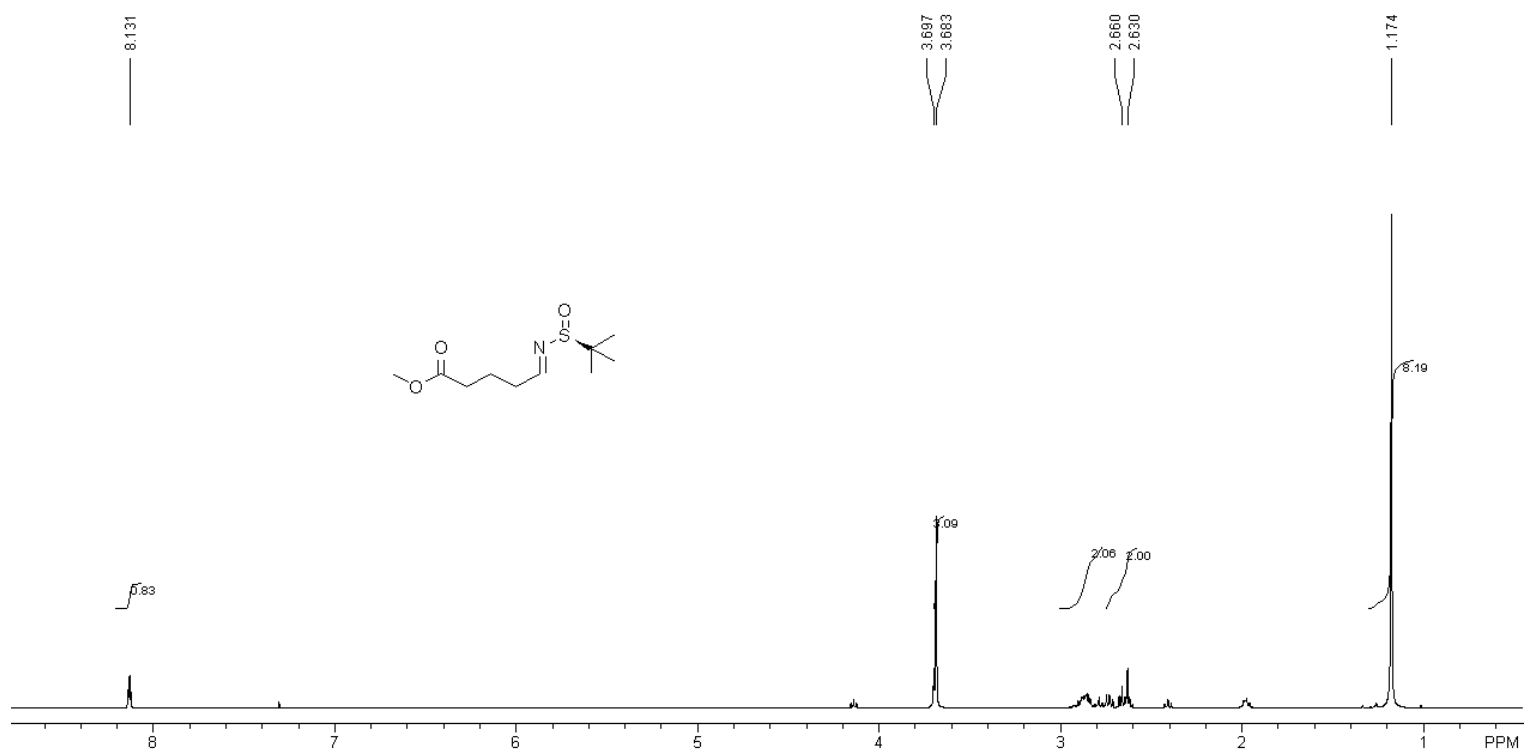
Compound 1d:



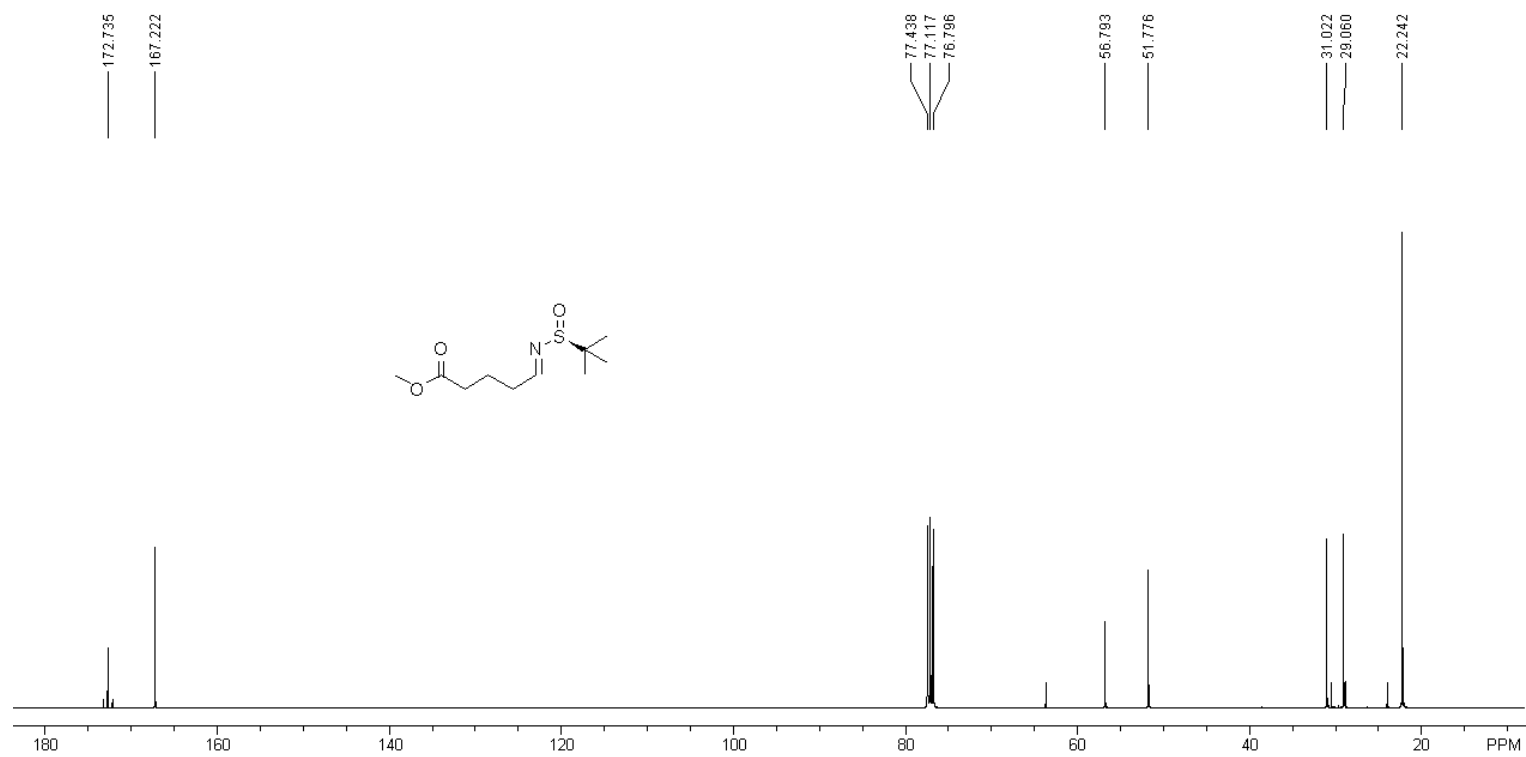
¹³C NMR:



Compound 1e:

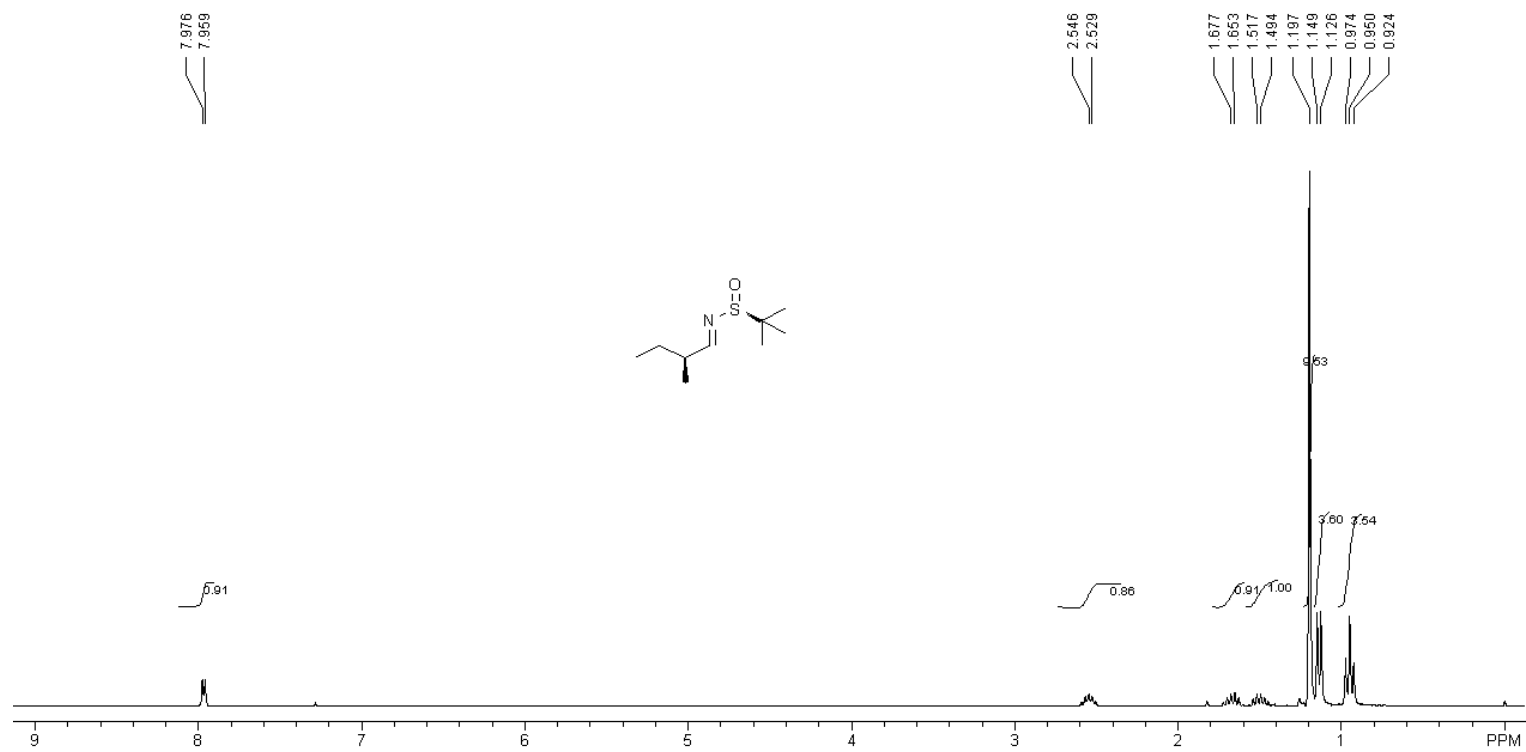


¹³C NMR:

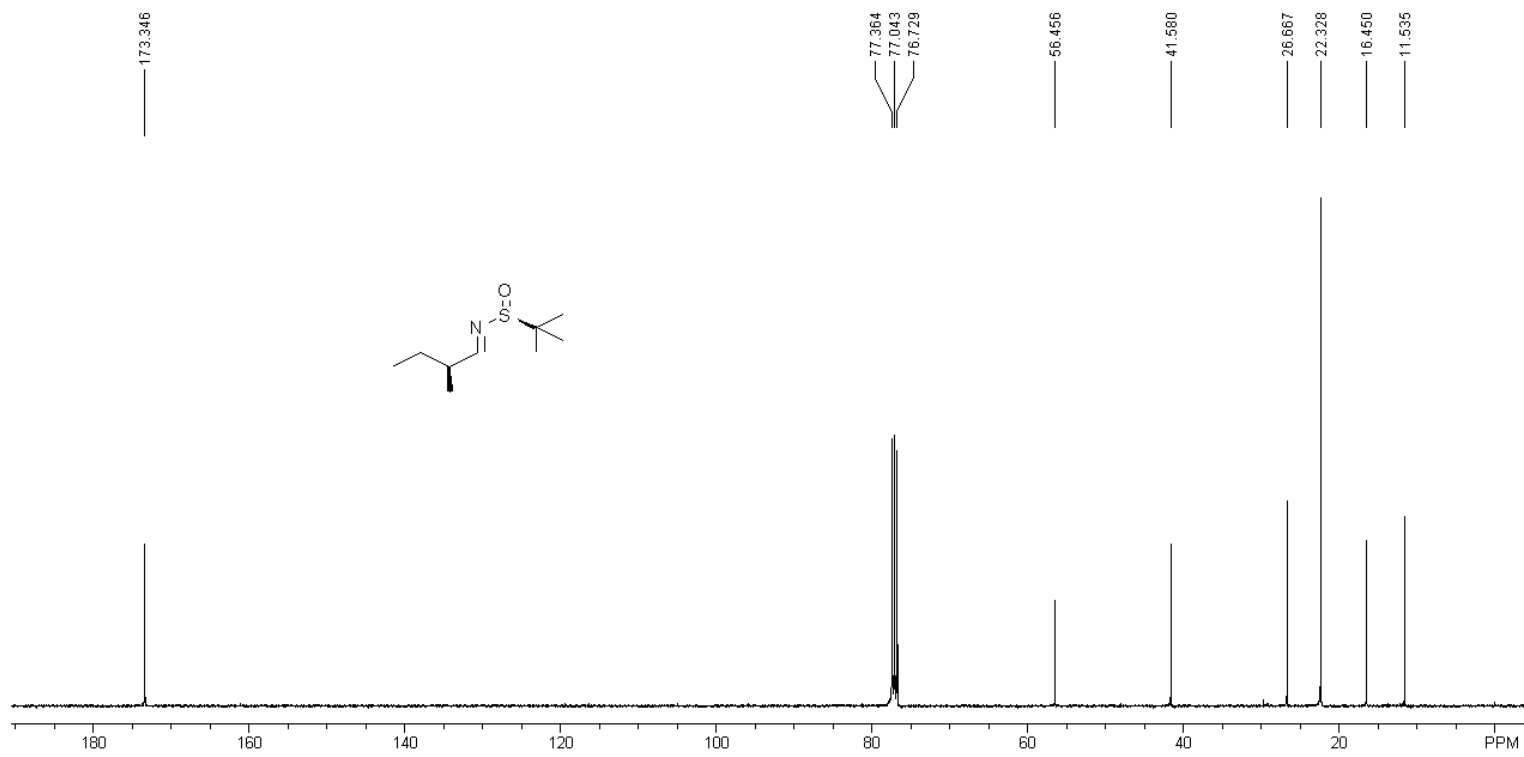


Compound 1f:

¹H NMR:

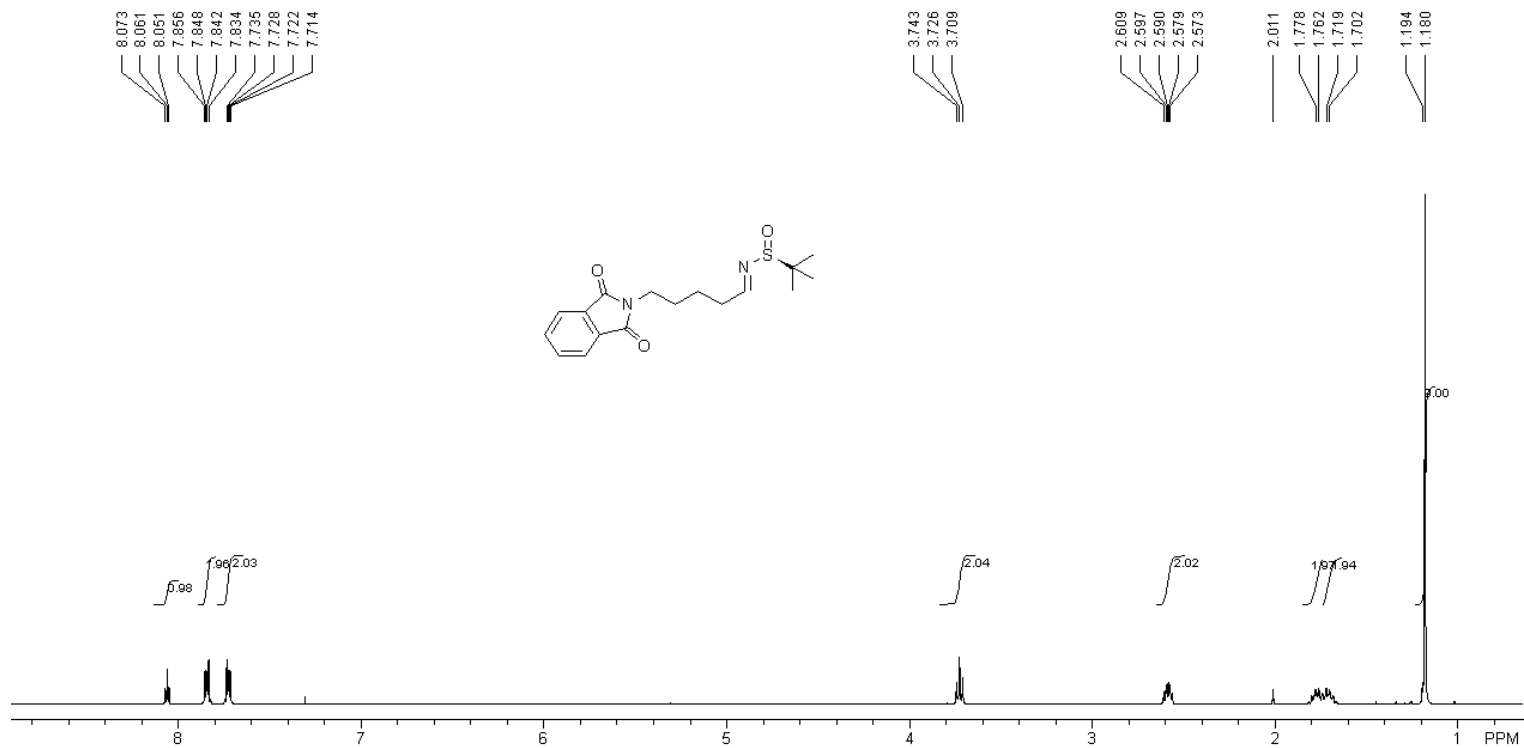


¹³C NMR:

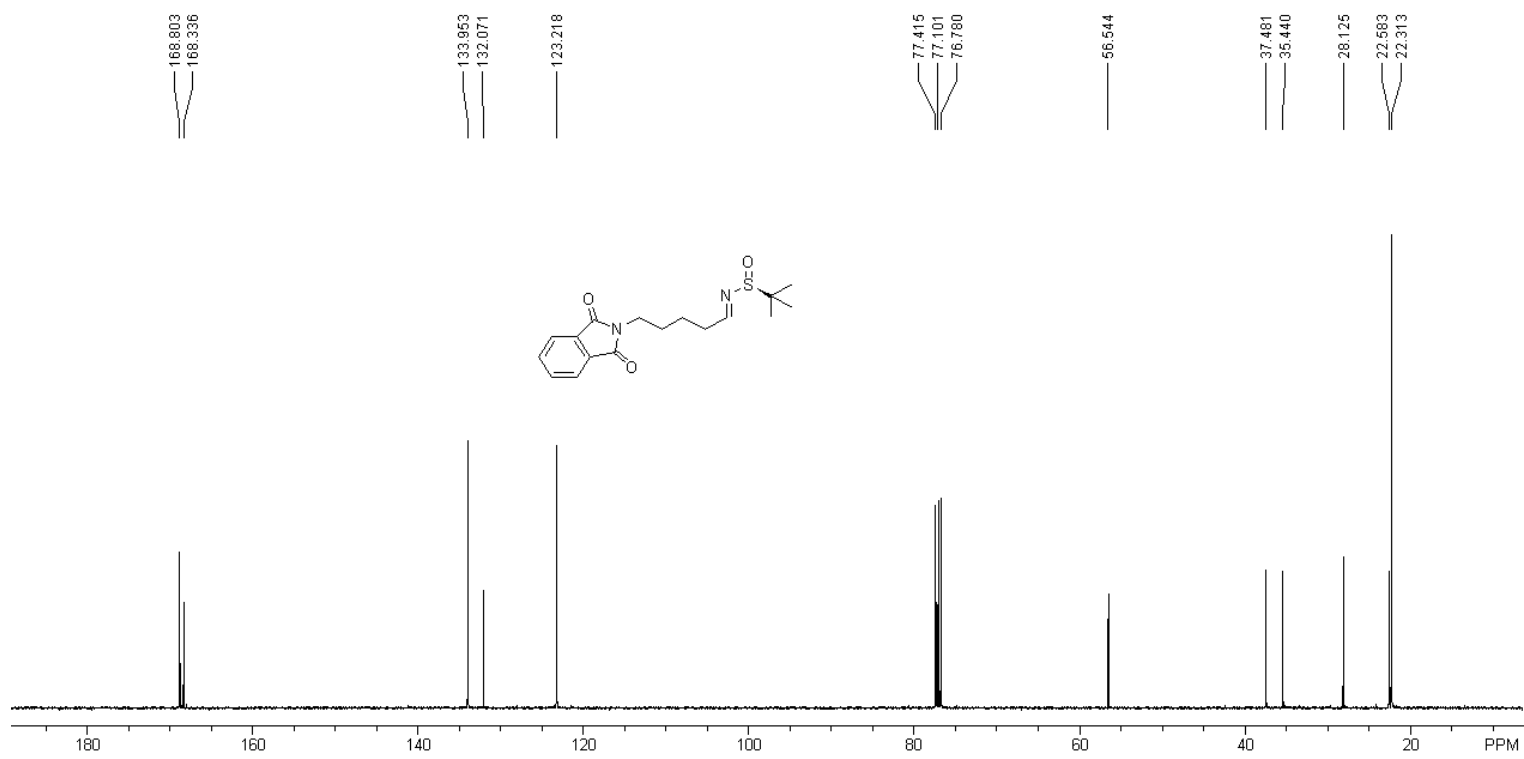


Compound 1h:

¹H NMR:

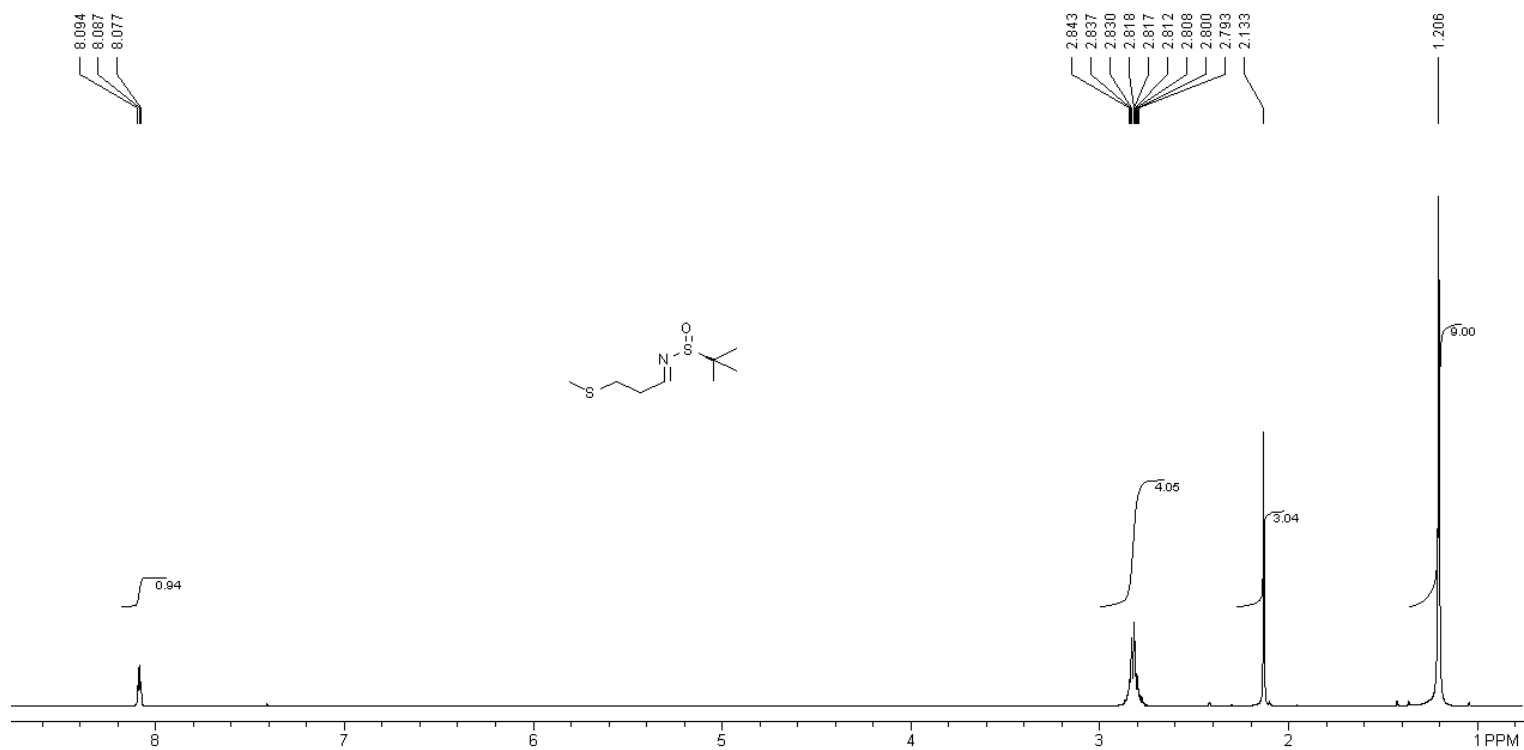


¹³C NMR:

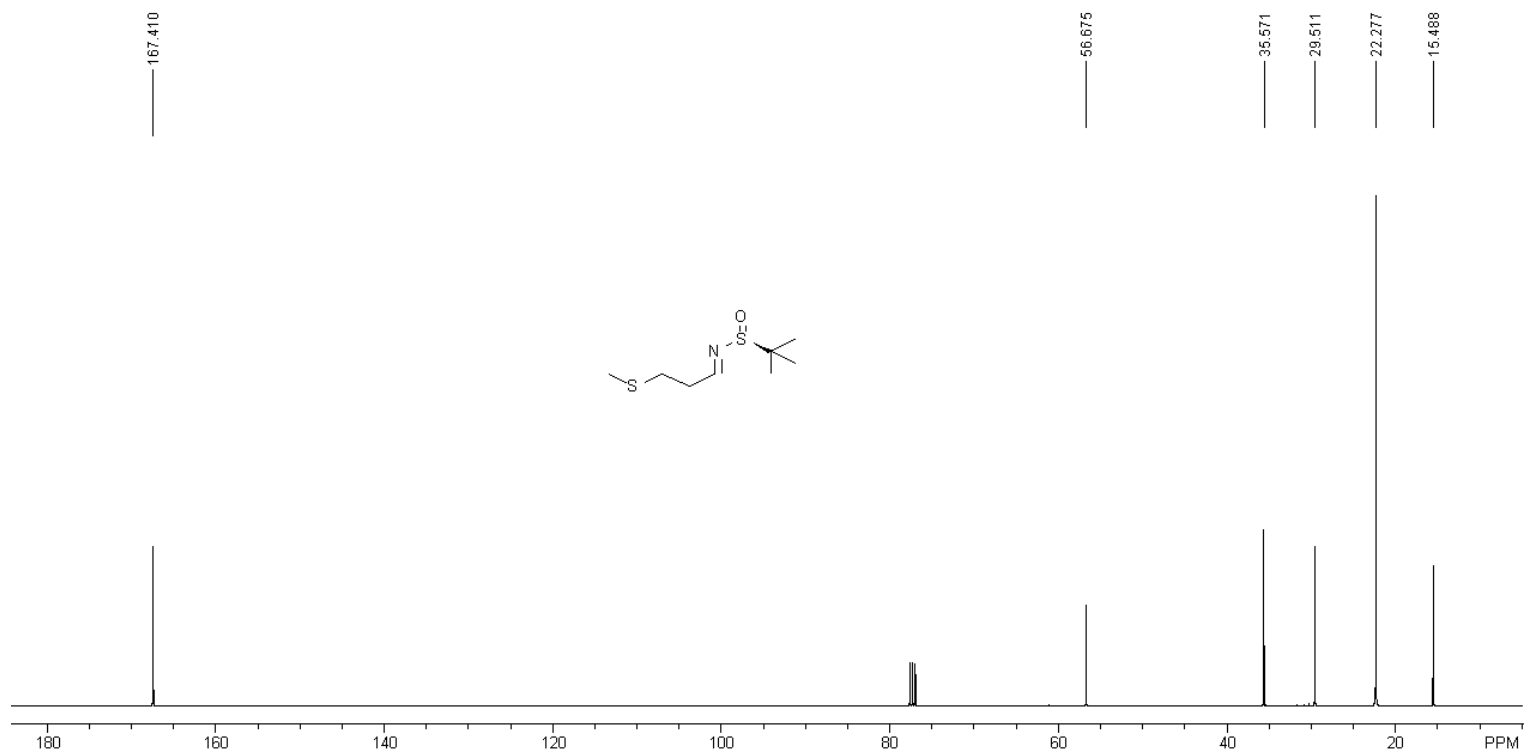


Compound 1i:

¹H NMR:

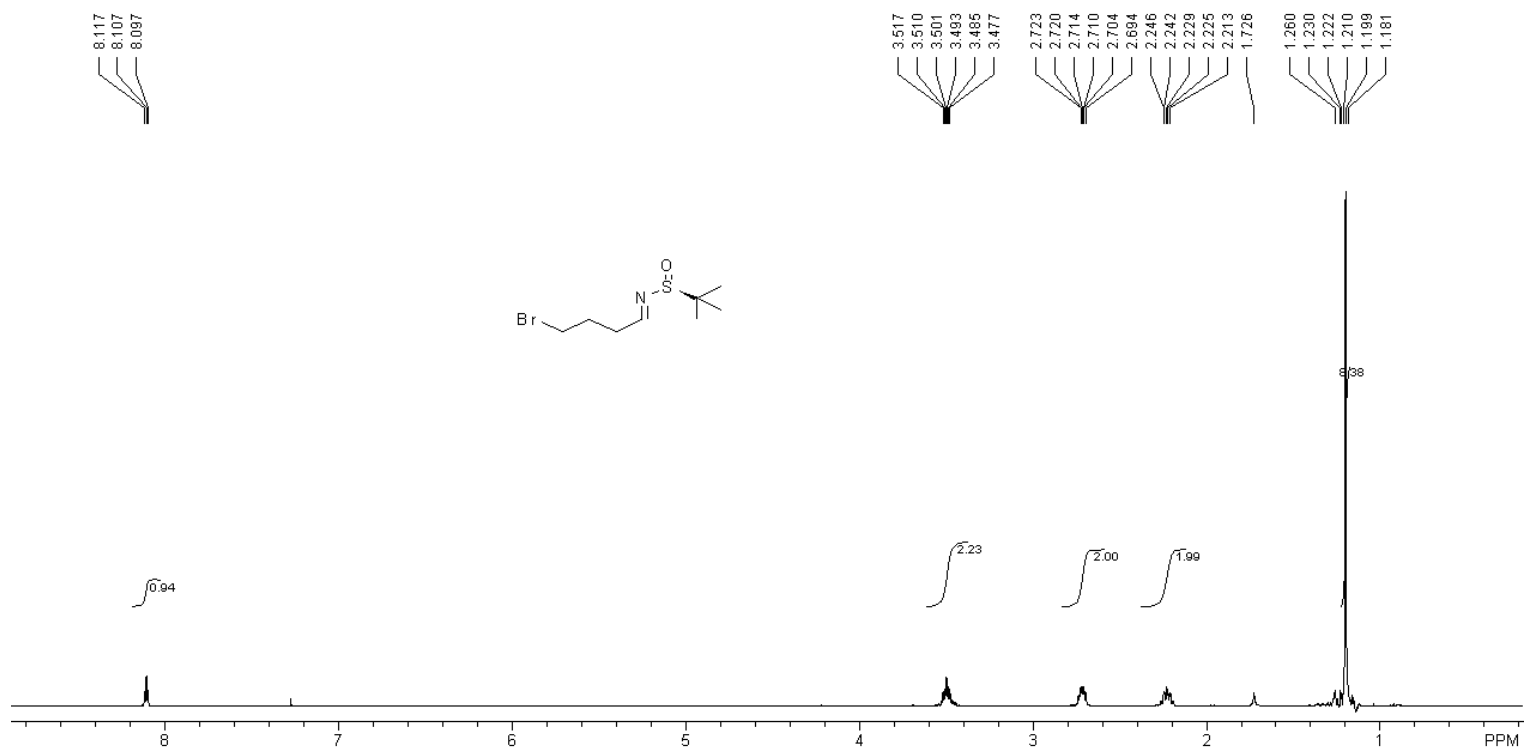


¹³C NMR:

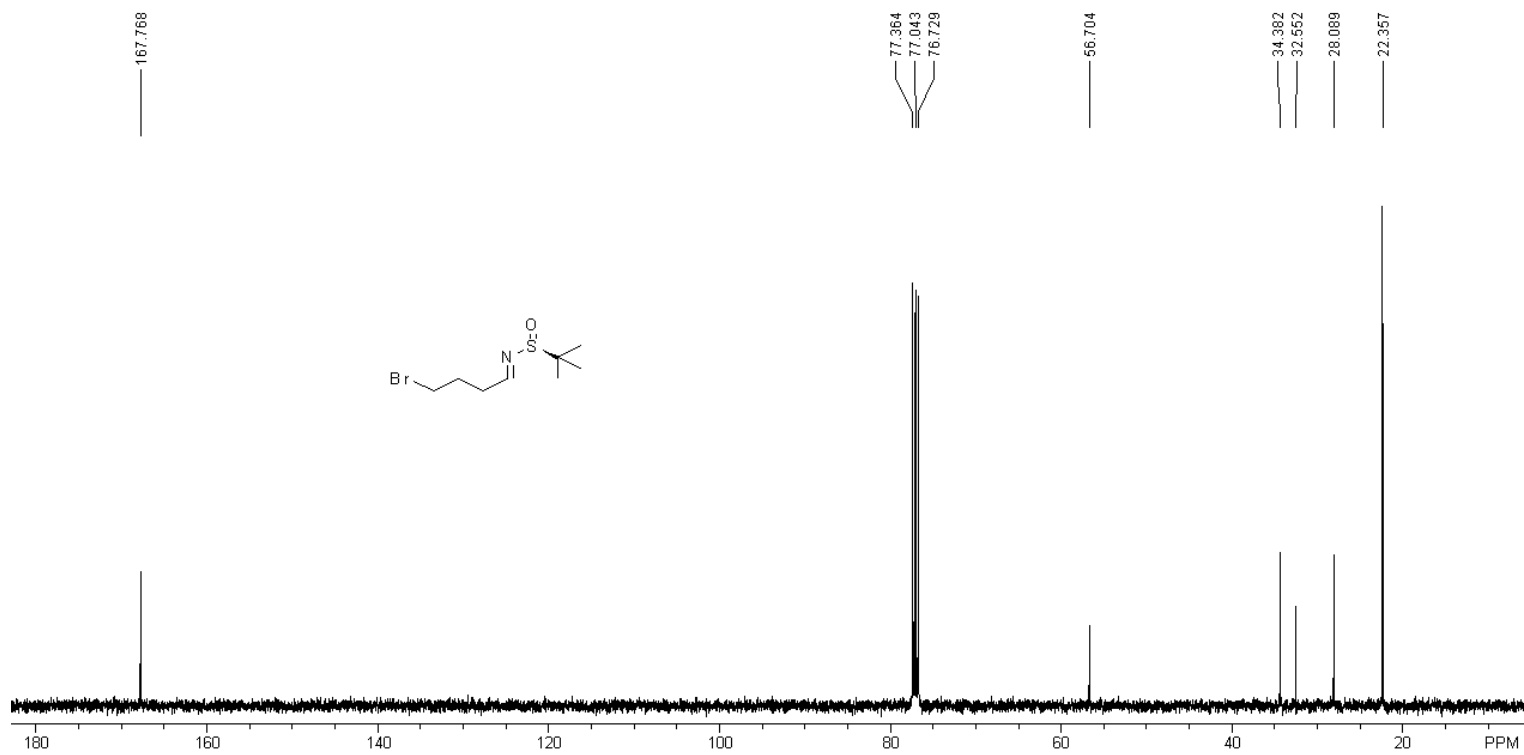


Compound 1k:

¹H NMR:

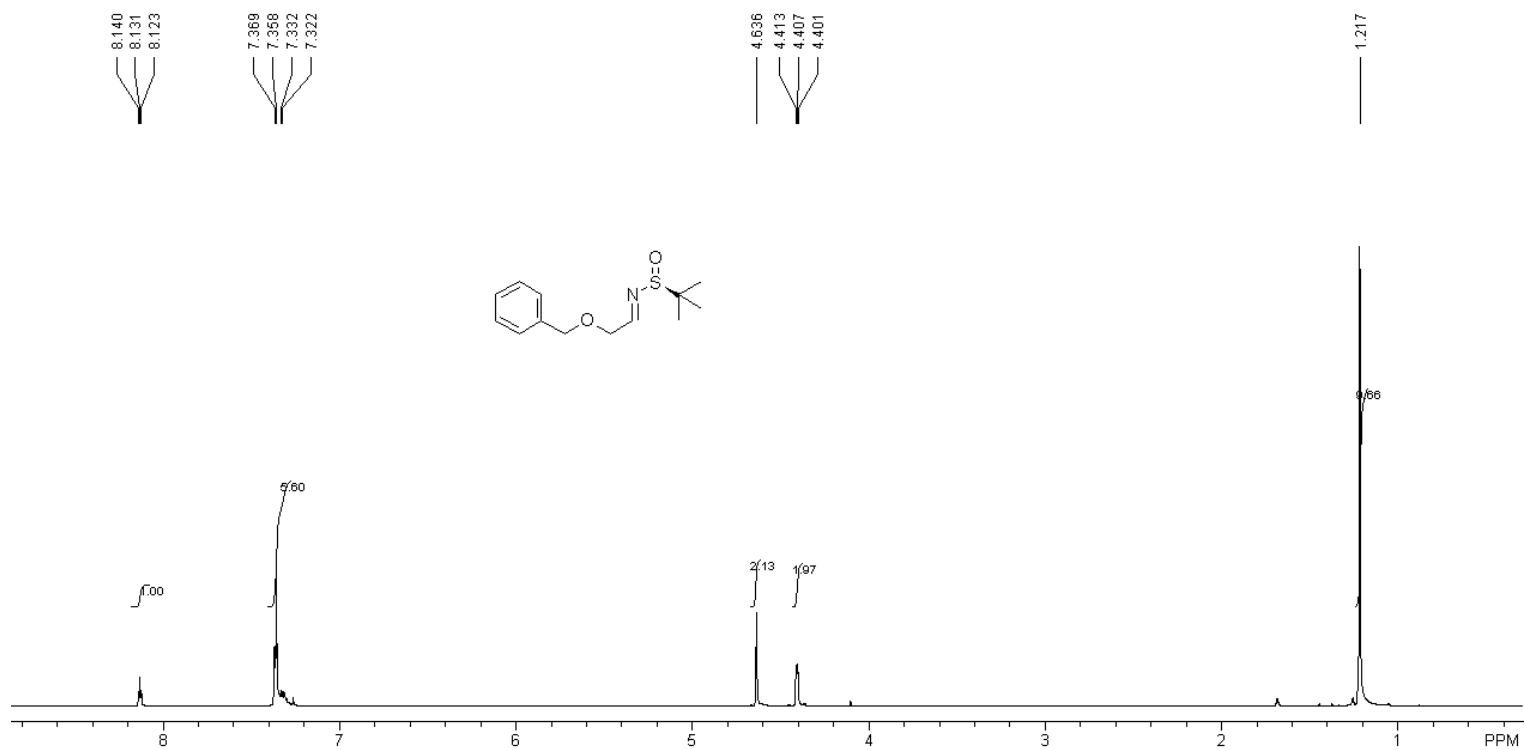


¹³C NMR:

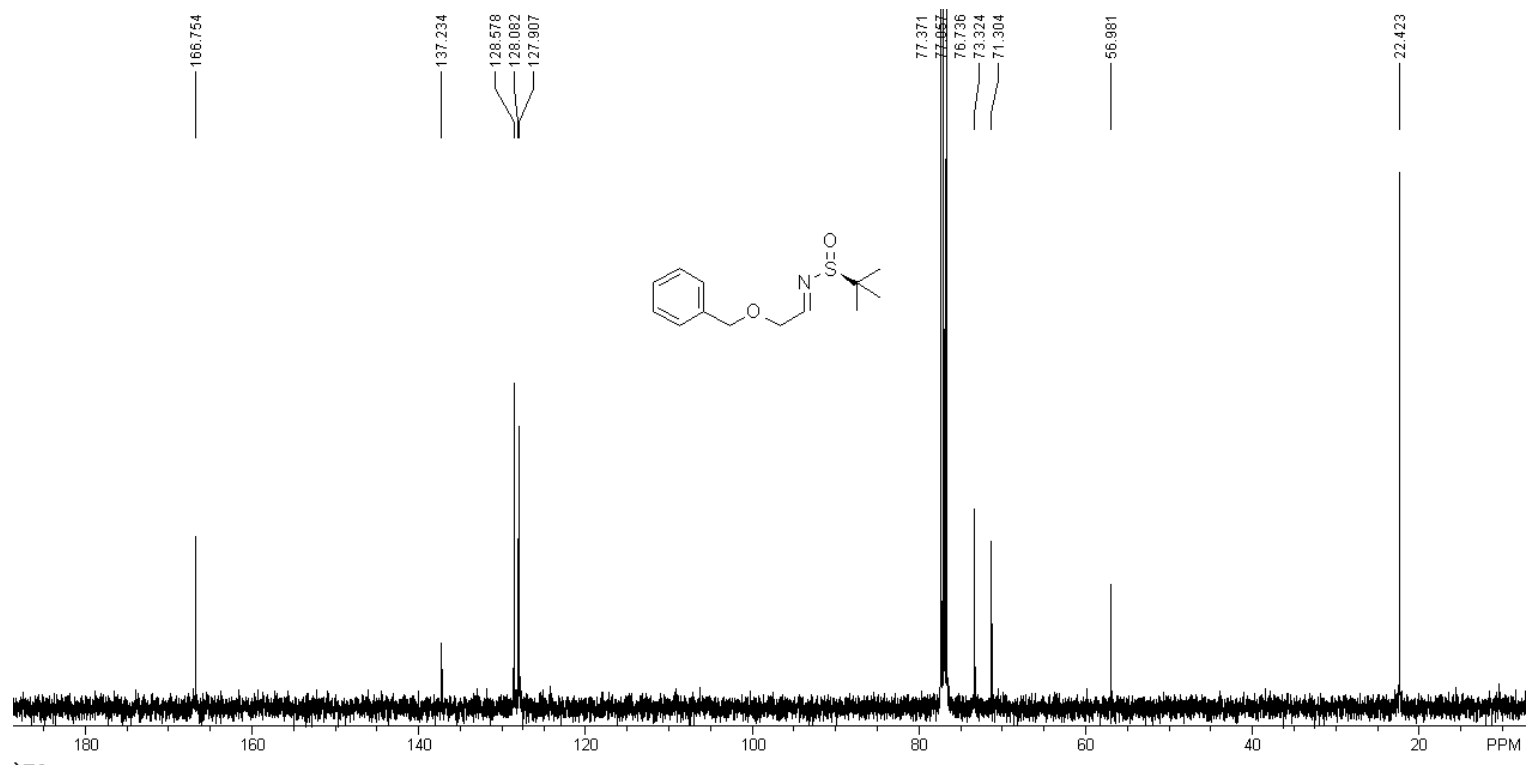


Compound 11:

¹H NMR:

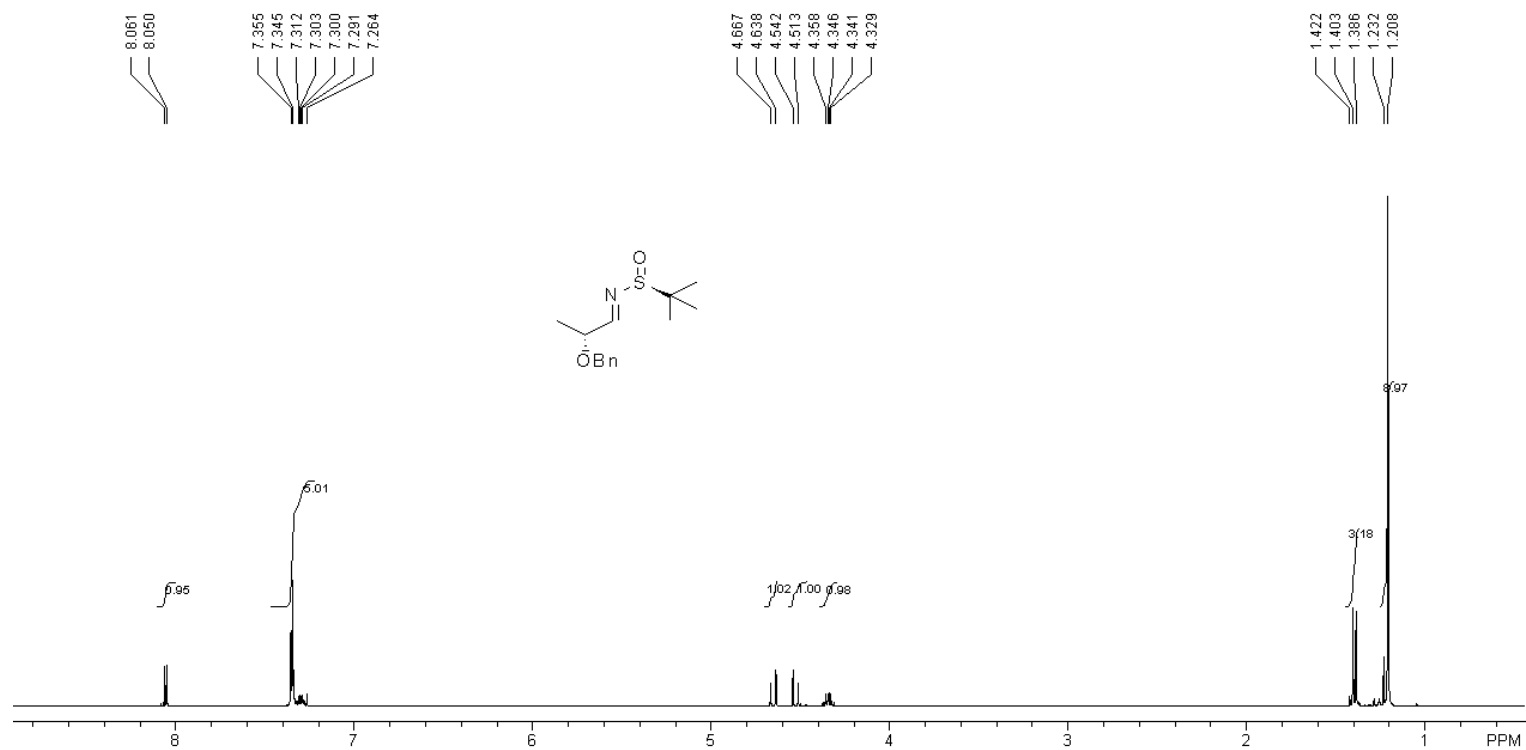


¹³C NMR:

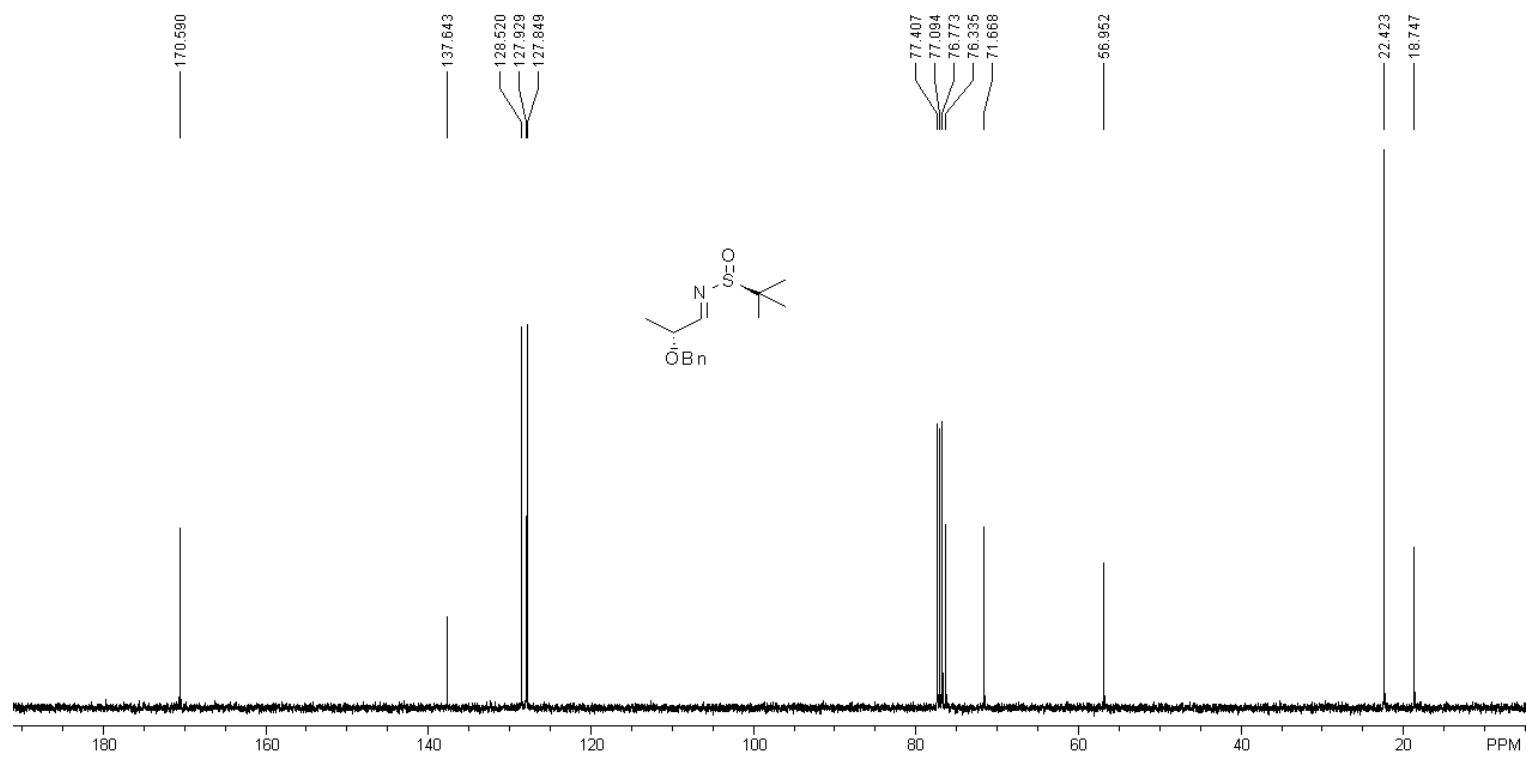


Compound 1m:

¹H NMR:

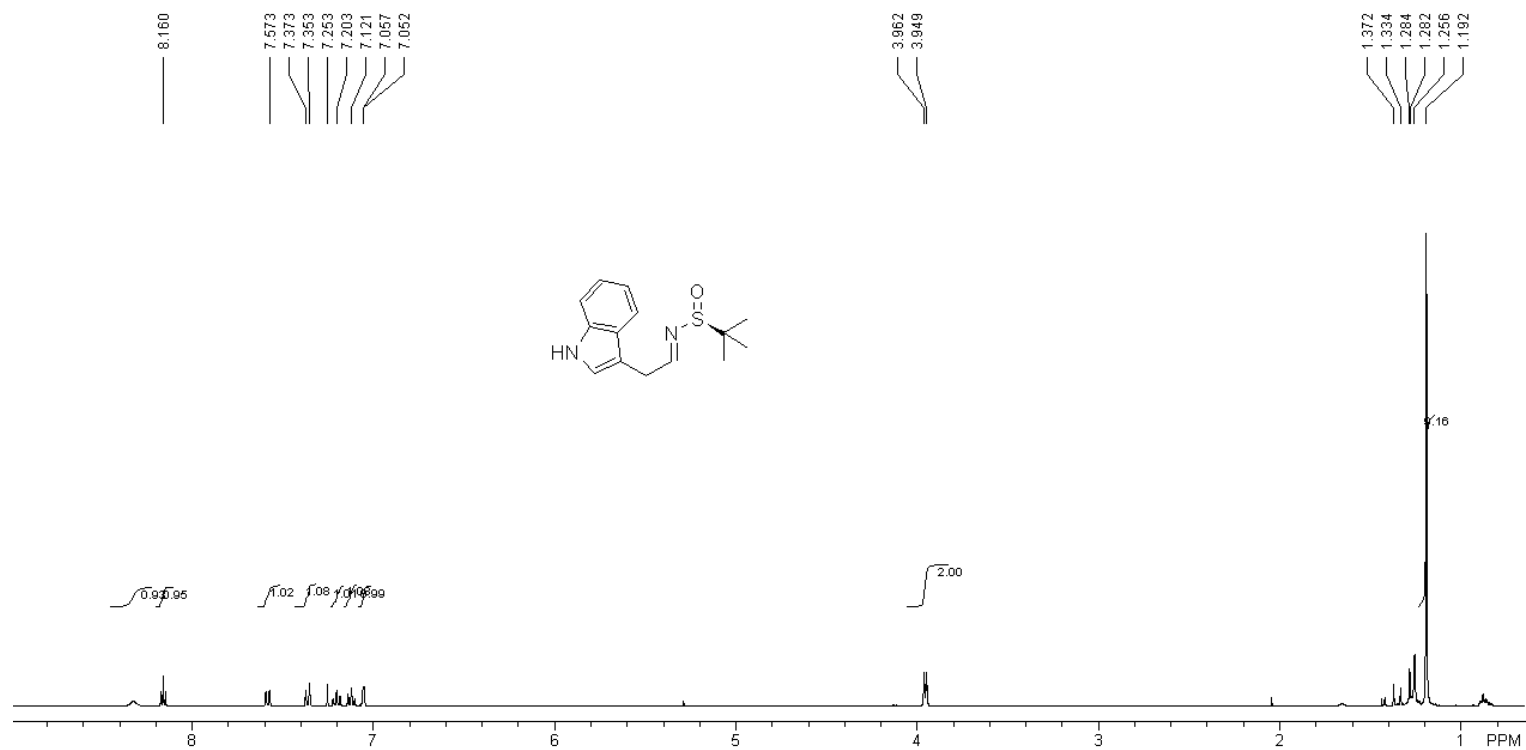


¹³C NMR:

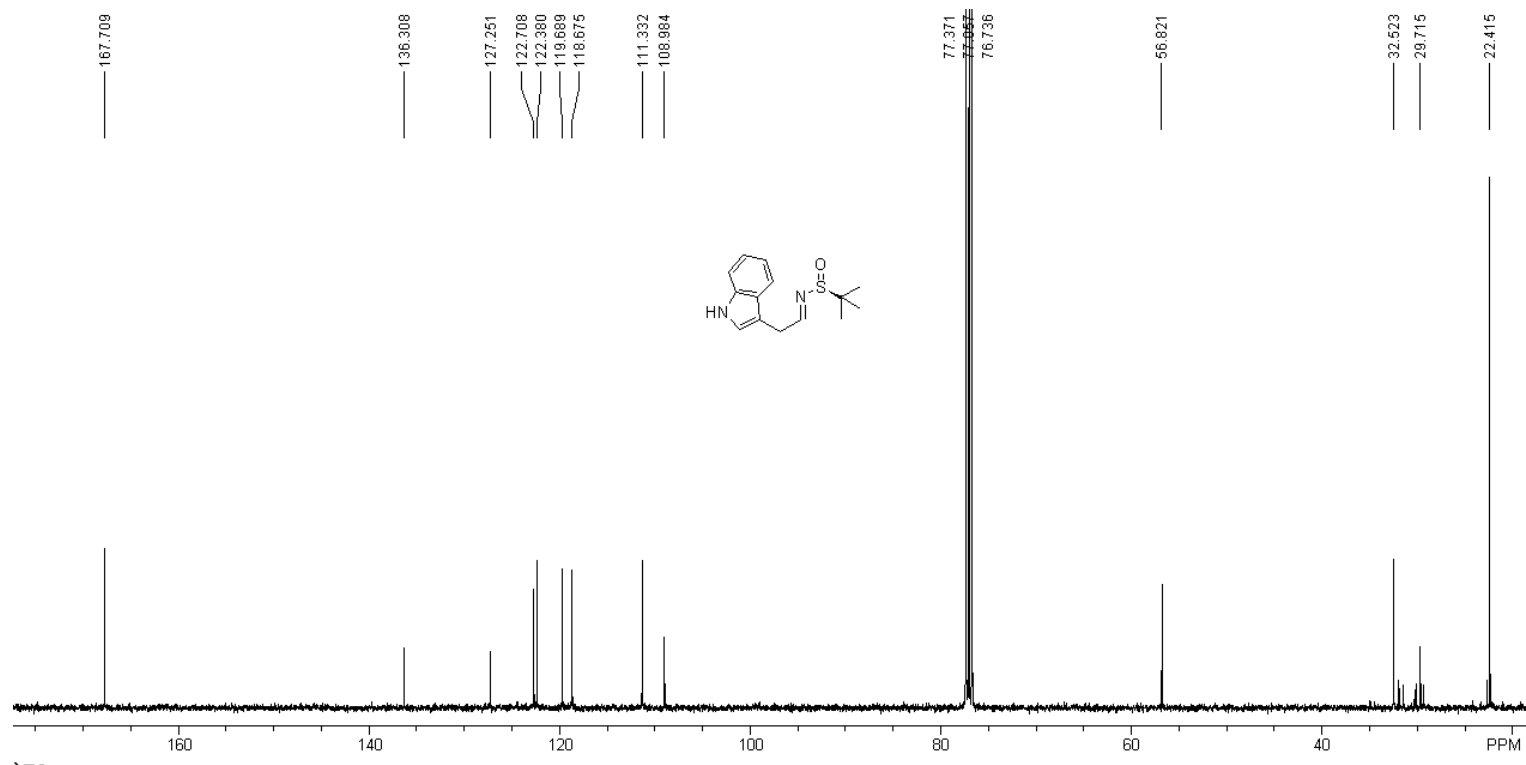


Compound 1n:

¹H NMR:

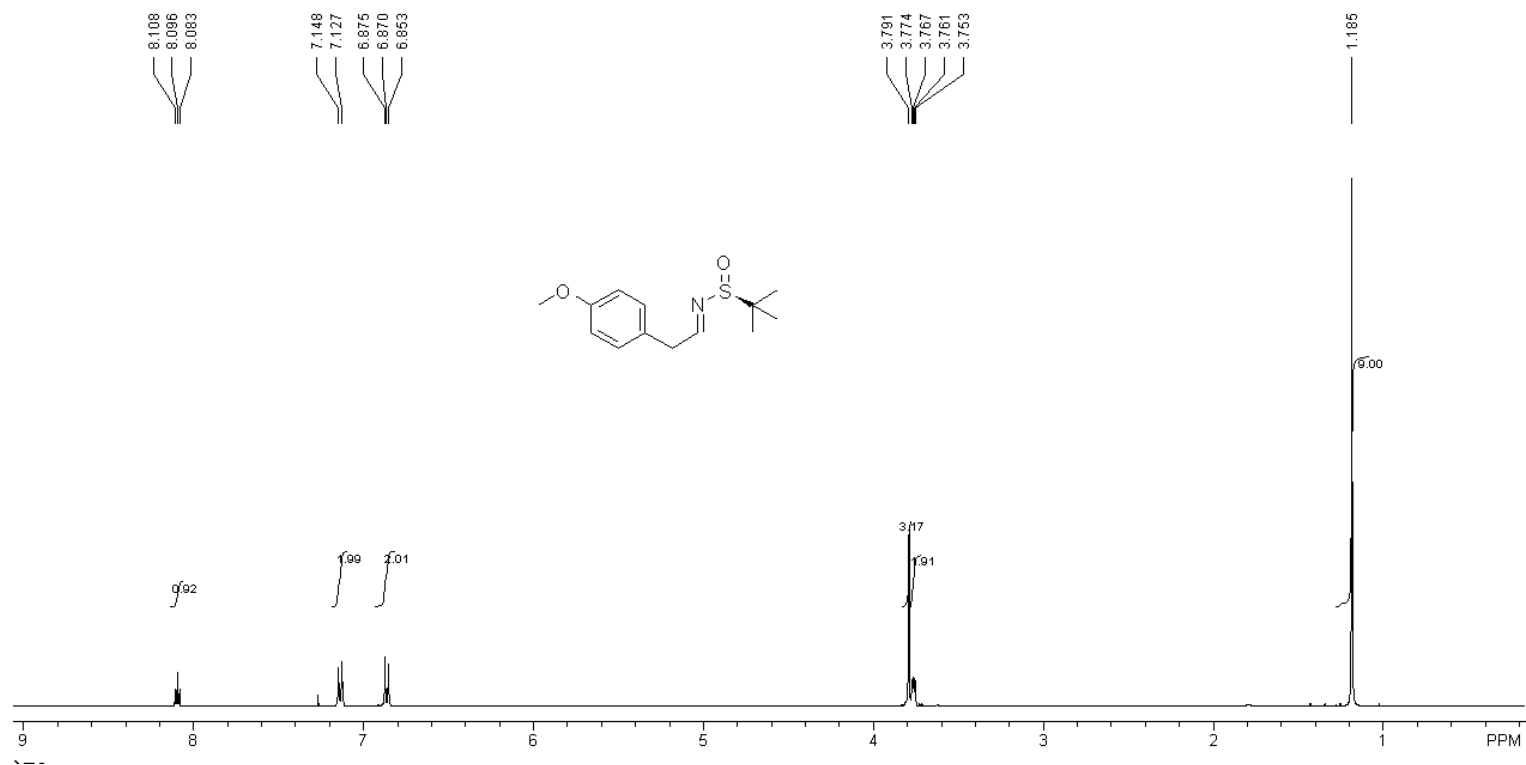


¹³C NMR:

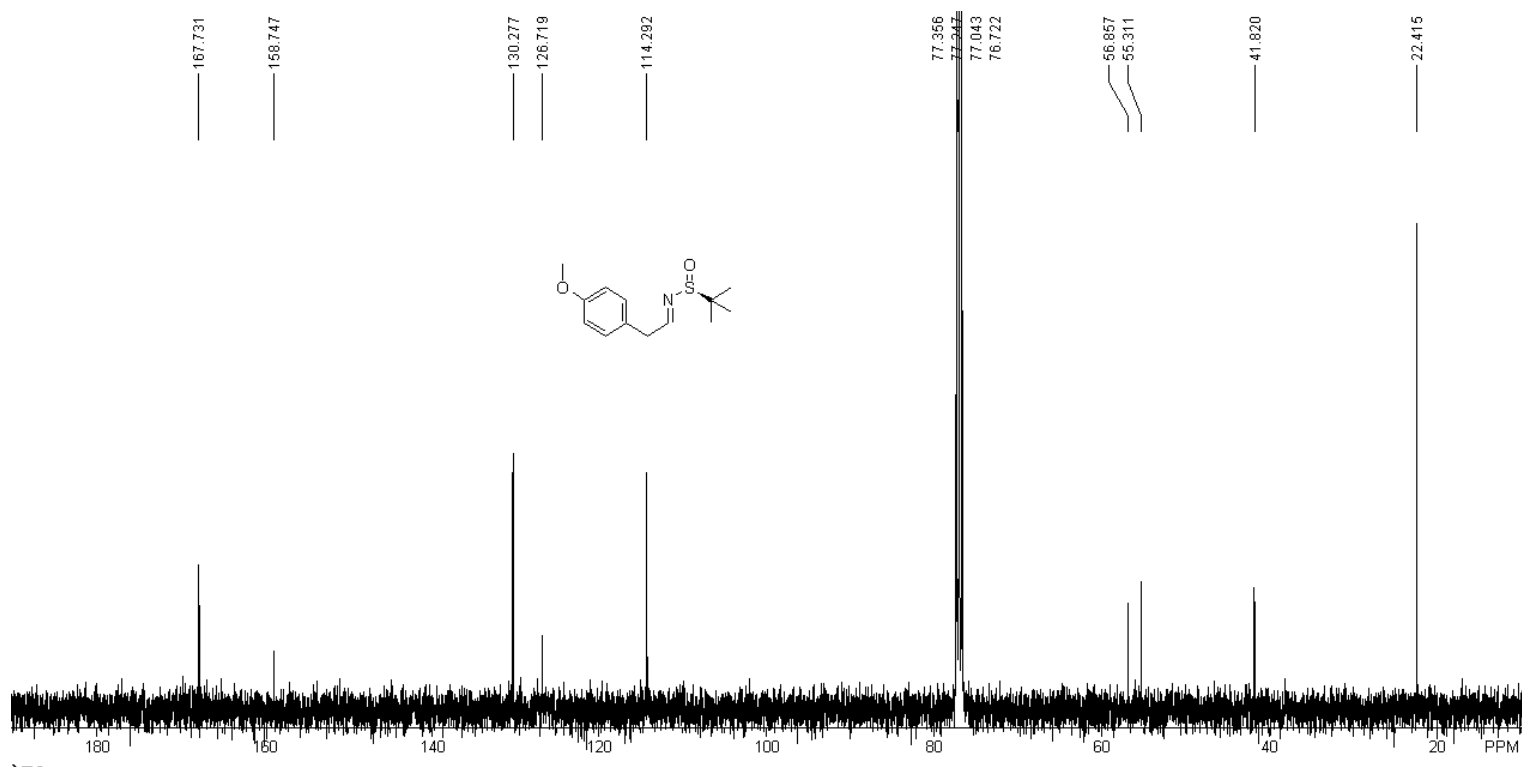


Compound 1o:

¹H NMR:

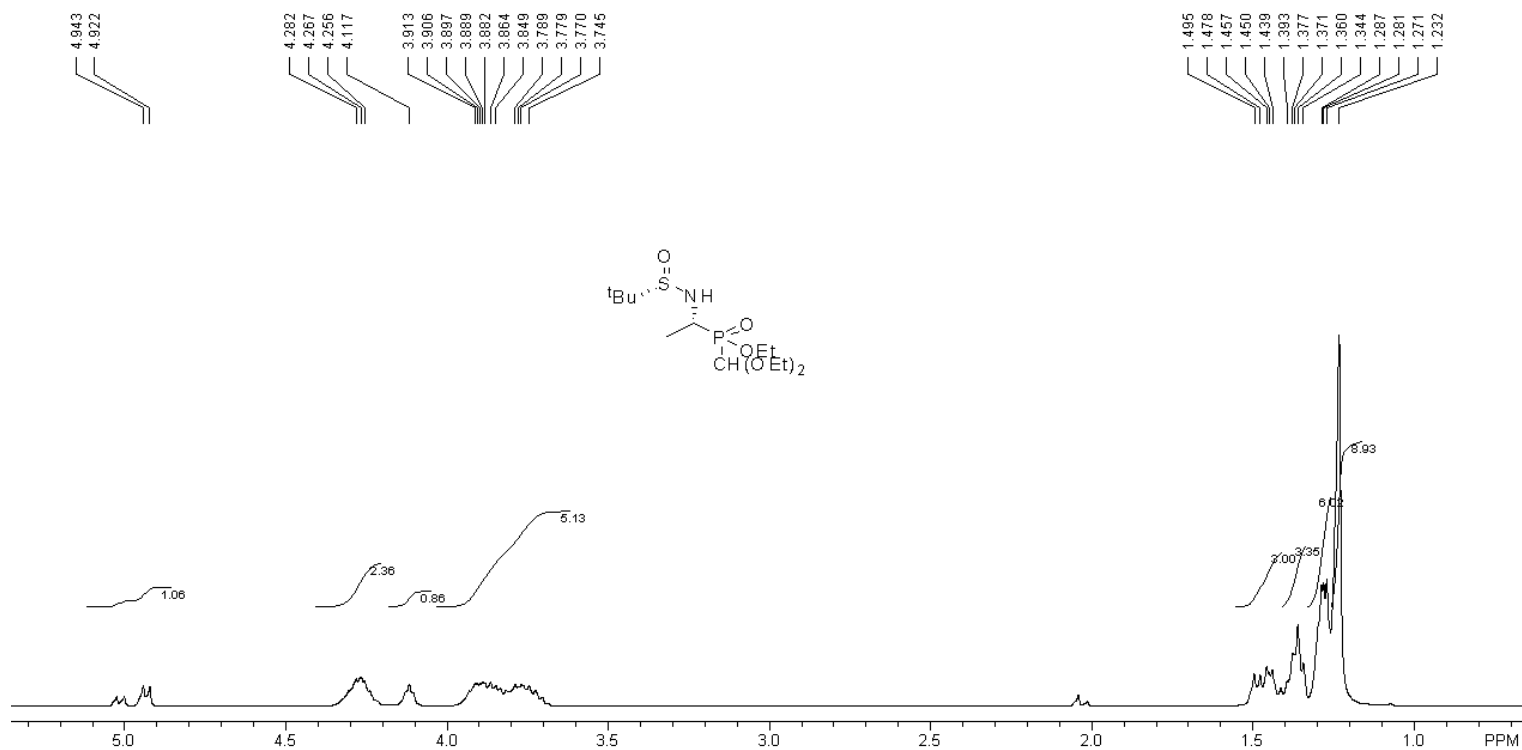


¹³C NMR:

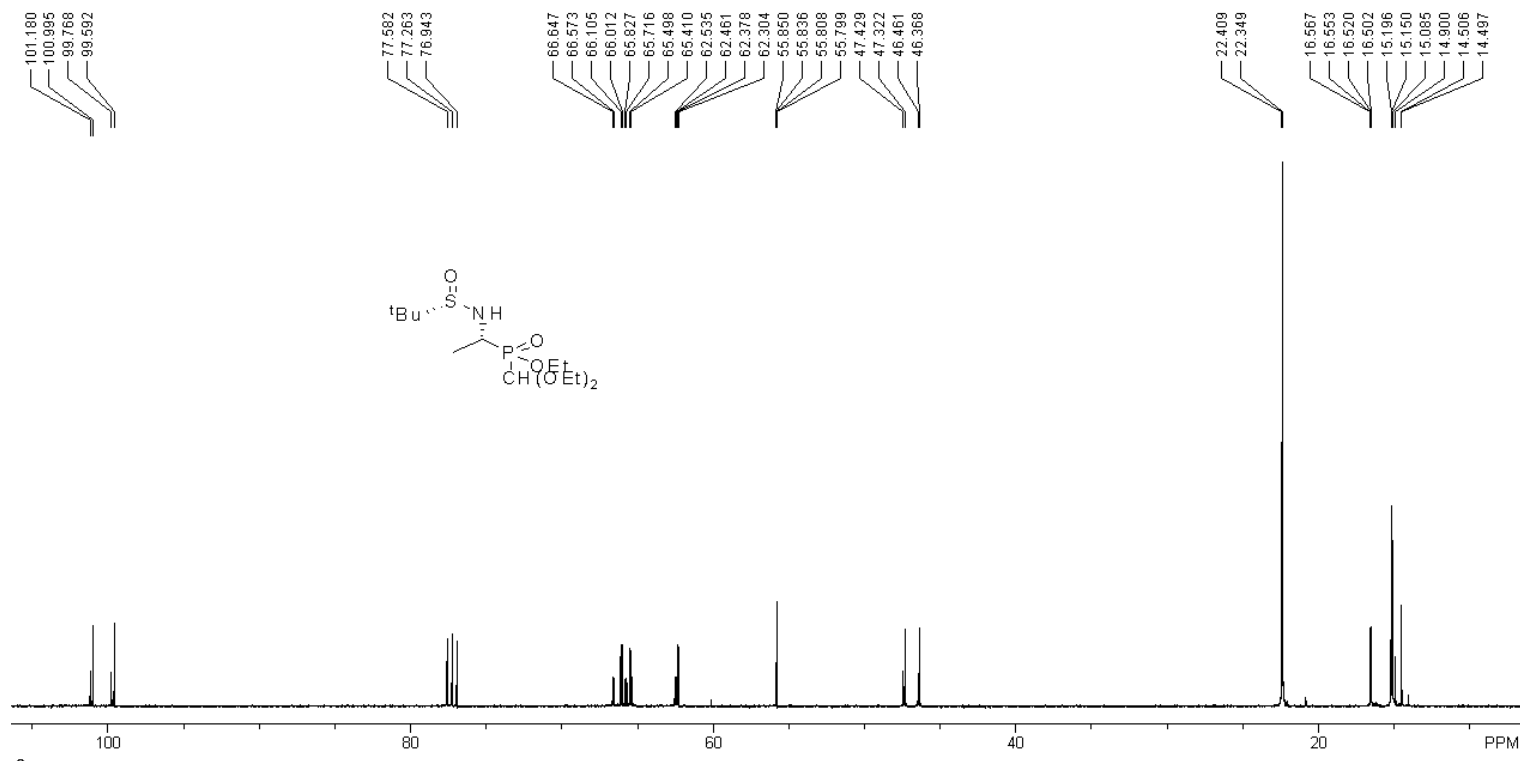


Compound 2a:

¹H NMR:

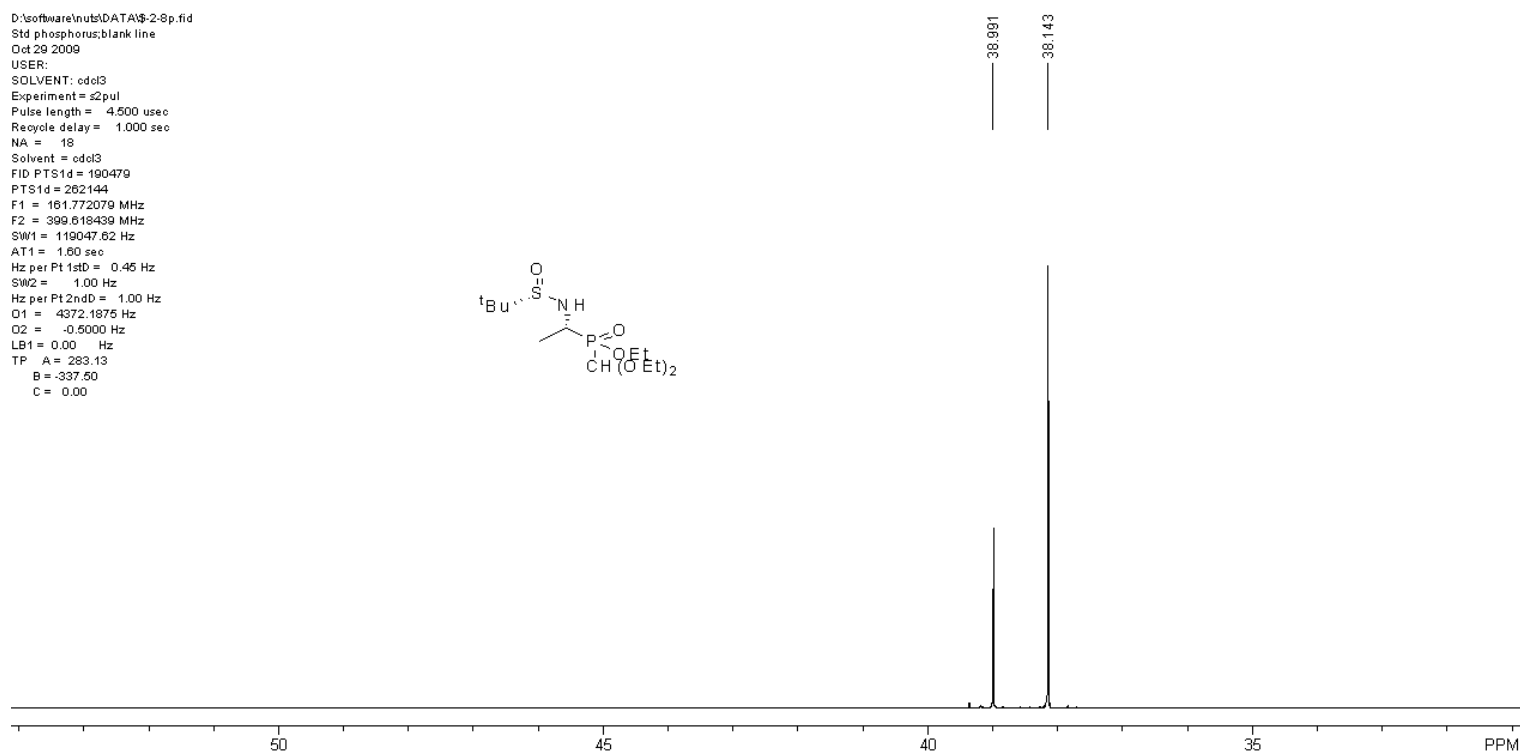


¹³C NMR:



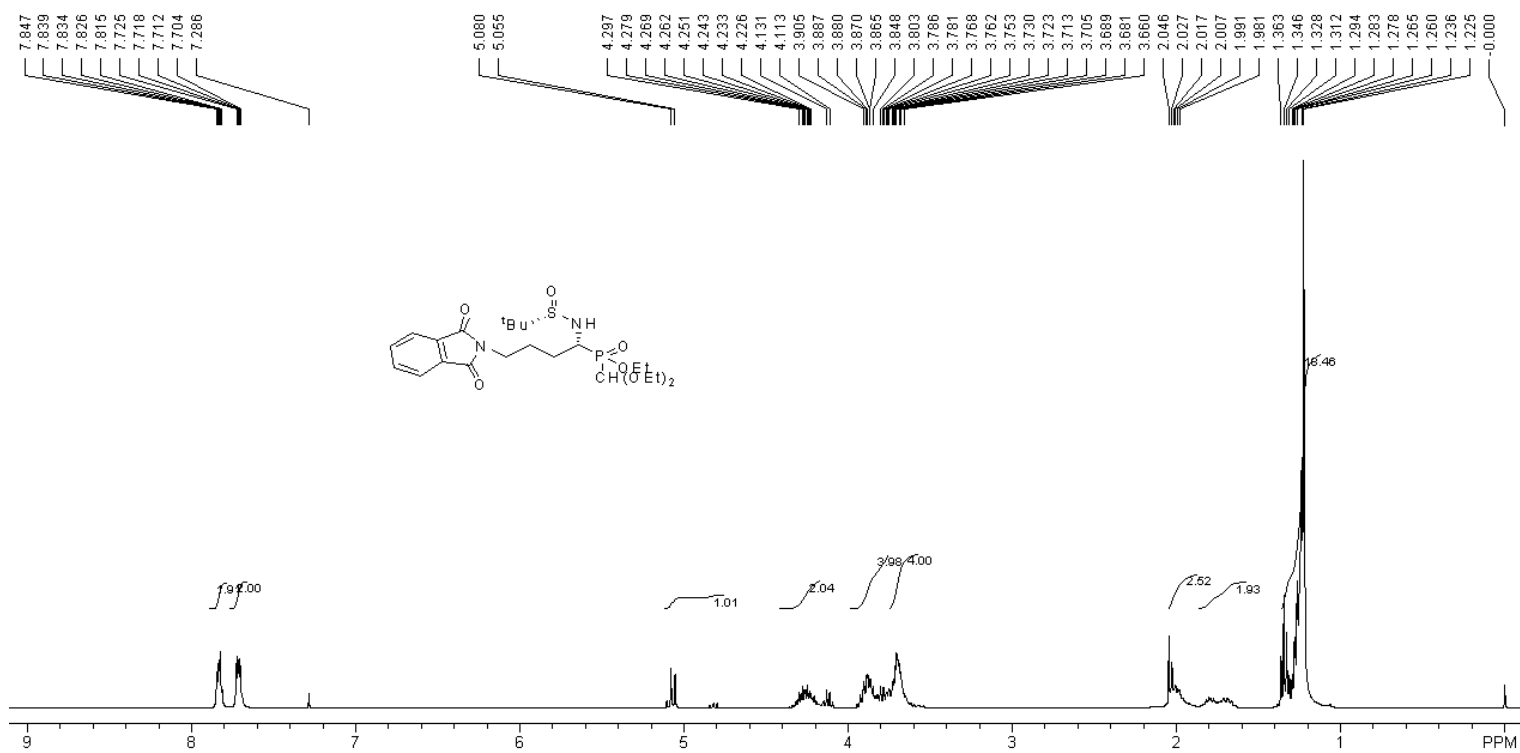
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 Oct 29 2009
 USER:
 SOLVENT: cdcl3
 Experiment = s2pul
 Pulse length = 4.500 usec
 Recycle delay = 1.000 sec
 NA = 18
 Solvent = cdcl3
 FID PTS1d = 190479
 PTS1d = 262144
 F1 = 161.772079 MHz
 F2 = 399.618439 MHz
 SW1 = 119047.62 Hz
 AT1 = 1.60 sec
 Hz per Pt1sD = 0.45 Hz
 SW2 = 1.00 Hz
 Hz per Pt2ndD = 1.00 Hz
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 Q2 = -0.5000 Hz
 LB1 = 0.00 Hz
 TP A = 283.13
 B = -337.60
 C = 0.00

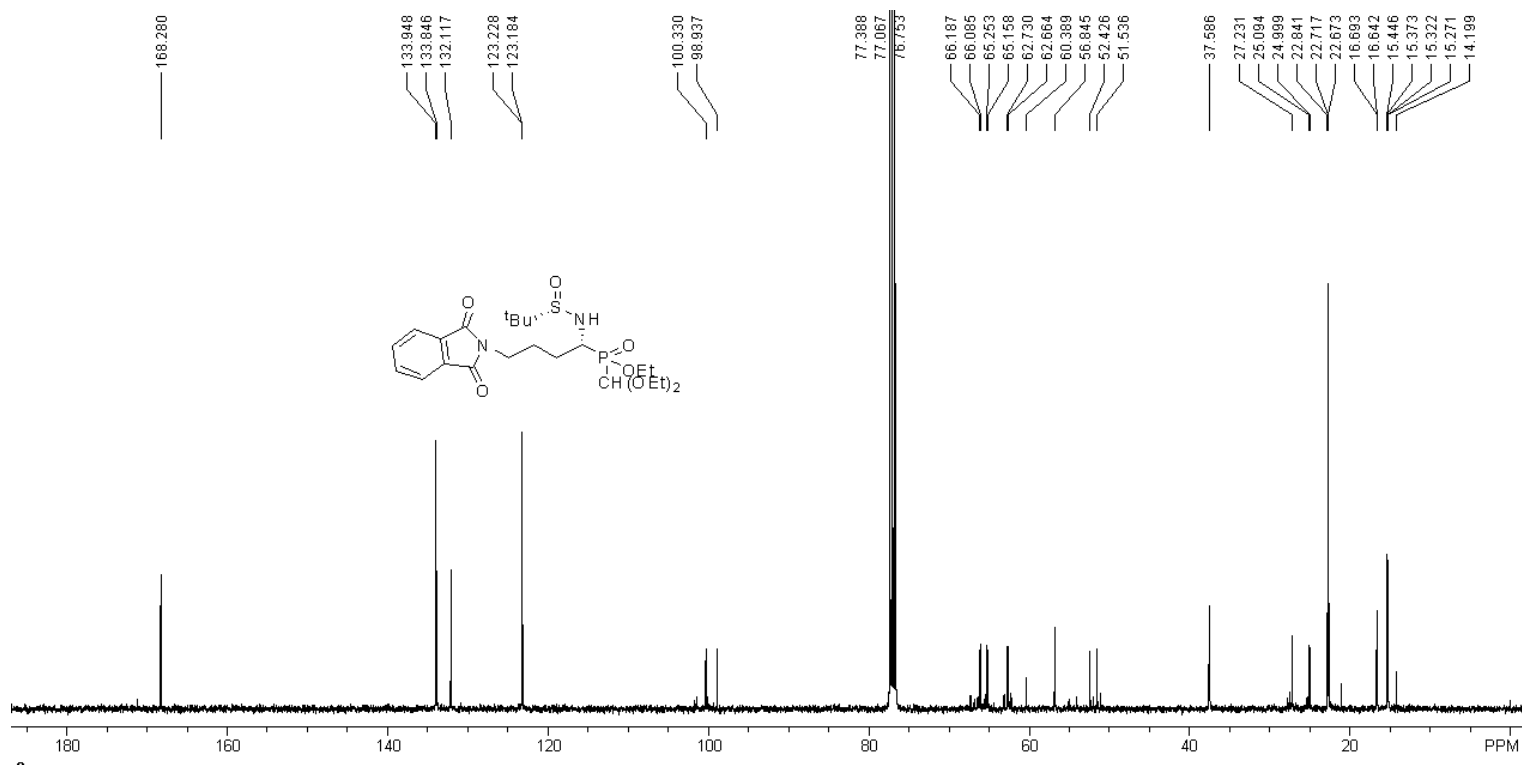


Compound 2b:

¹H NMR:

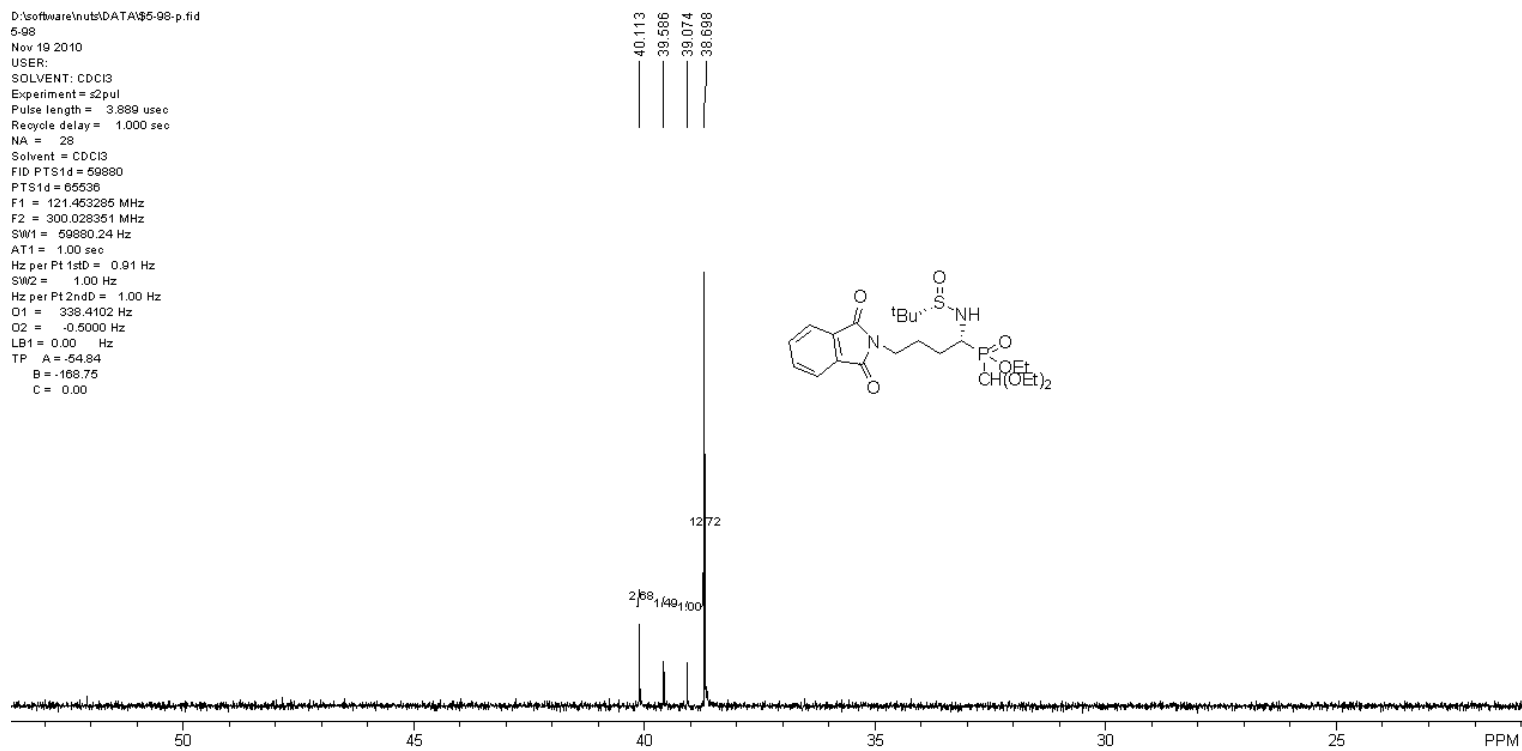


¹³C NMR:



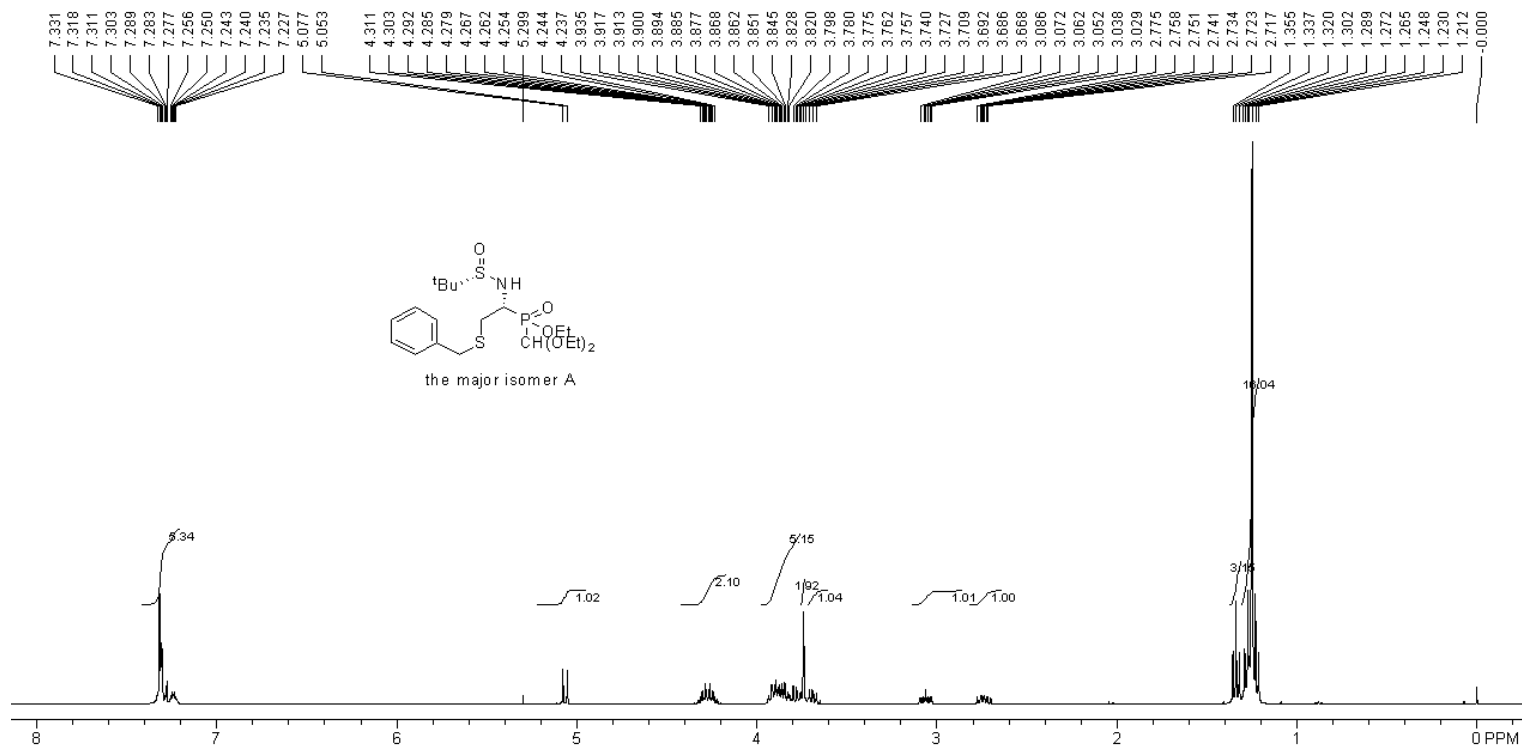
³¹P NMR:

D:\software\nuts\DATA\5-98-p.fid
 5-98
 Nov 19 2010
 USER:
 SOLVENT: CDCl3
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 Pulse length = 3.889 usec
 Recycle delay = 1.000 sec
 NA = 28
 Solvent = CDCl3
 FID PTS1d = 69880
 PTS1d = 65536
 F1 = 121.463285 MHz
 F2 = 300.028351 MHz
 SW1 = 69880.24 Hz
 AT1 = 1.00 sec
 Hz per Pt1st0 = 0.91 Hz
 SW2 = 1.00 Hz
 Hz per Pt2nd0 = 1.00 Hz
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 O2 = -0.6000 Hz
 LB1 = 0.00 Hz
 TP A = -64.94
 B = -168.76
 C = 0.00

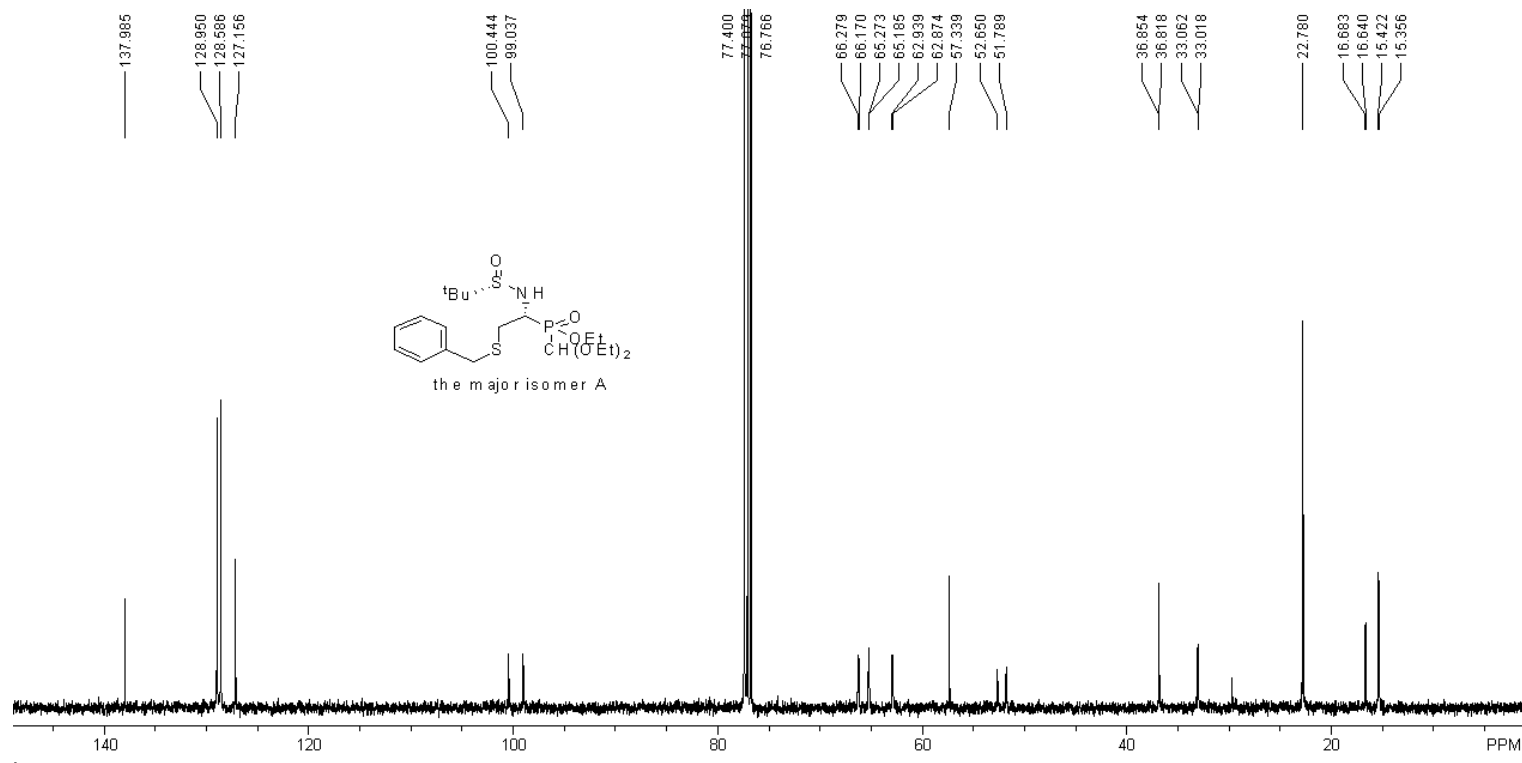


Compound 2c:

¹H NMR:



¹³C NMR:



³¹P NMR:

D:\software\nmr\DATA\2008119-10-11A_11PNMR.1

22:29:46.546 +0800 nmr@EP.ZH107708

USER: nmr

SOLVENT: CDCl3

Experiment = zgpg30

Pulse length = 8.300 usec

Recycle delay = 2.000 sec

NA = 16

Solvent = CDCl3

PTS1d = 32768

F1 = 161.971542 MHz

F2 = 1.000000 MHz

SW1 = 64102.56 Hz

AT1 = 0.51 sec

Hz per Pt1s0 = 1.96 Hz

SW2 = 1.00 Hz

Hz per Pt2nd = 1.00 Hz

Q1 = -8098.6865 Hz

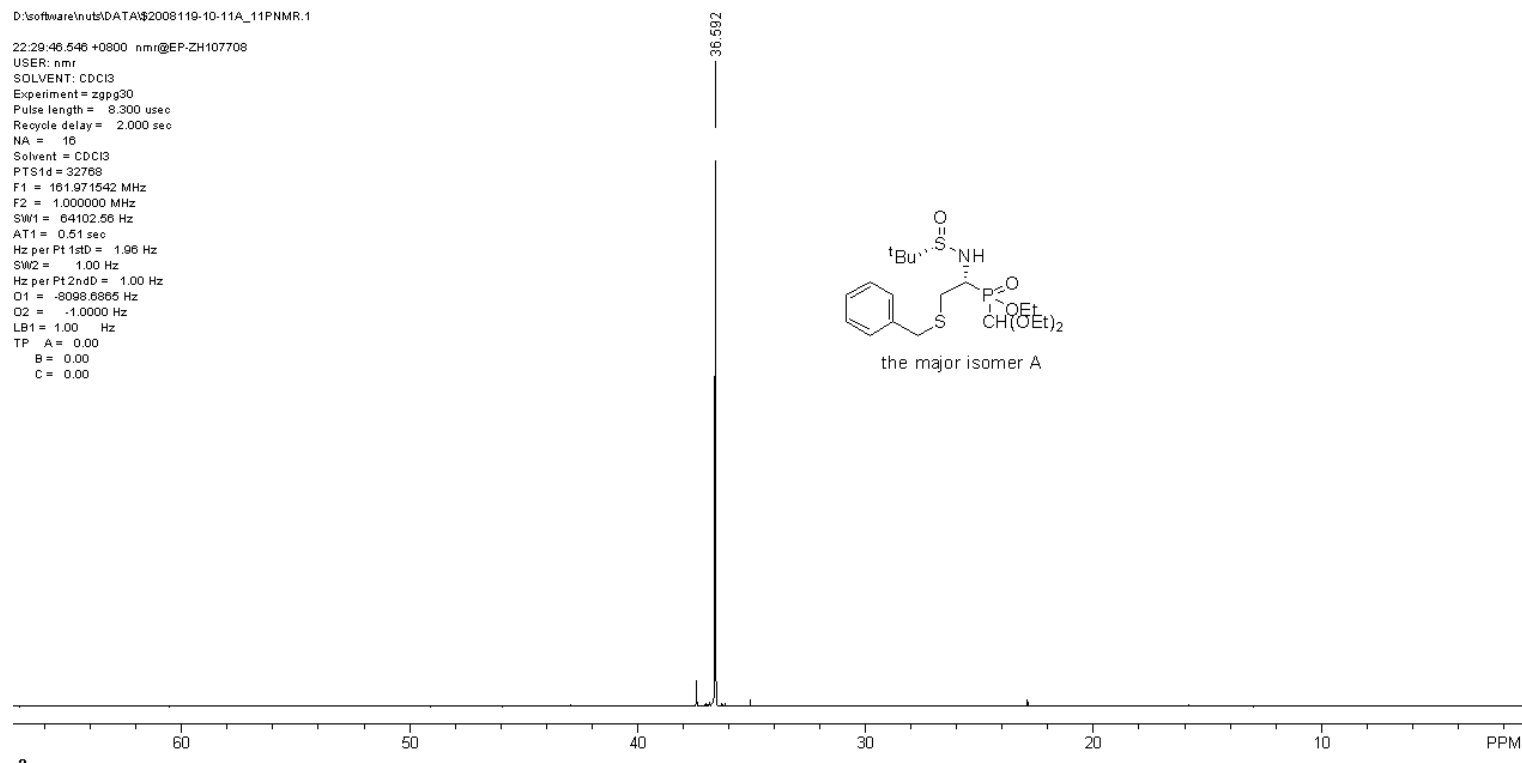
Q2 = -1.0000 Hz

LB1 = 1.00 Hz

TP A = 0.00

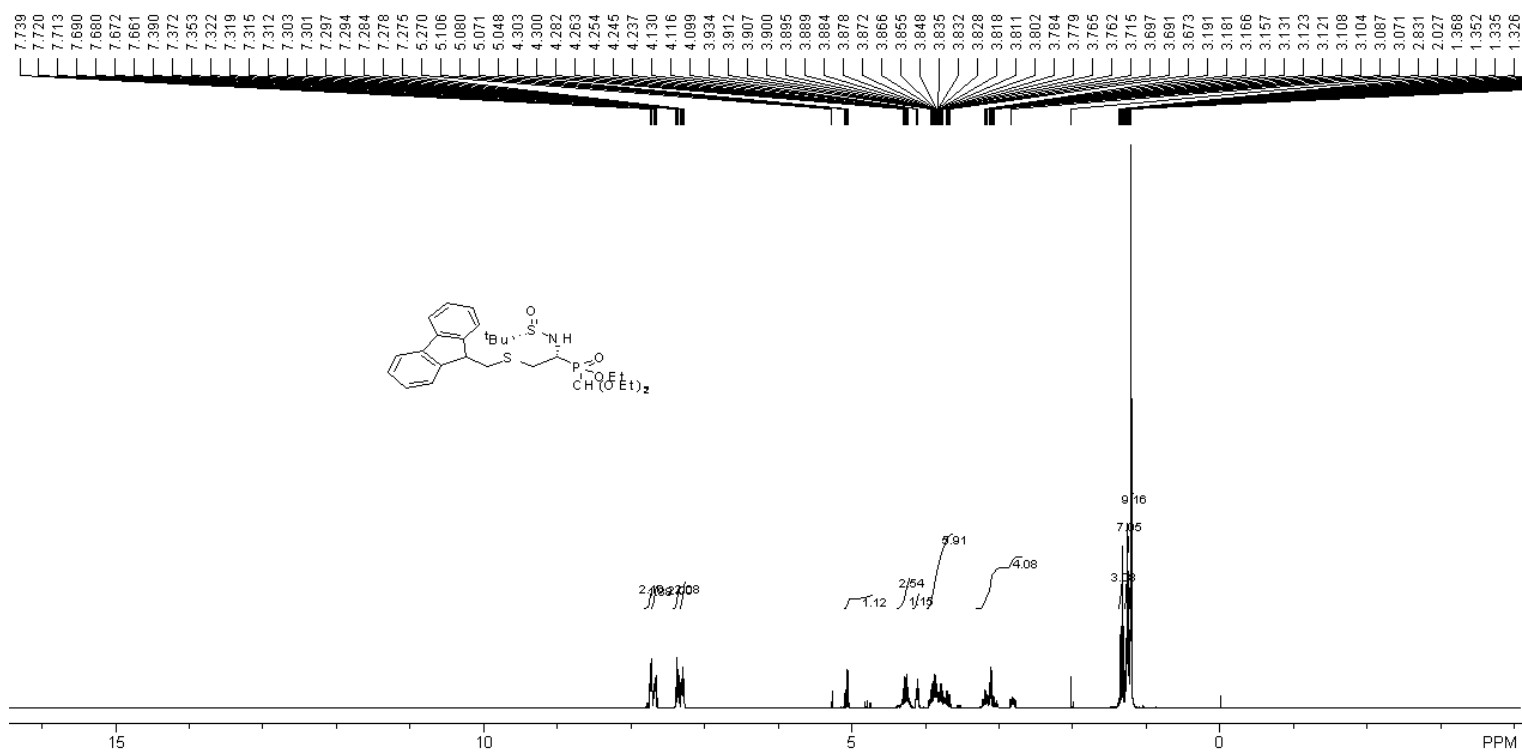
B = 0.00

C = 0.00

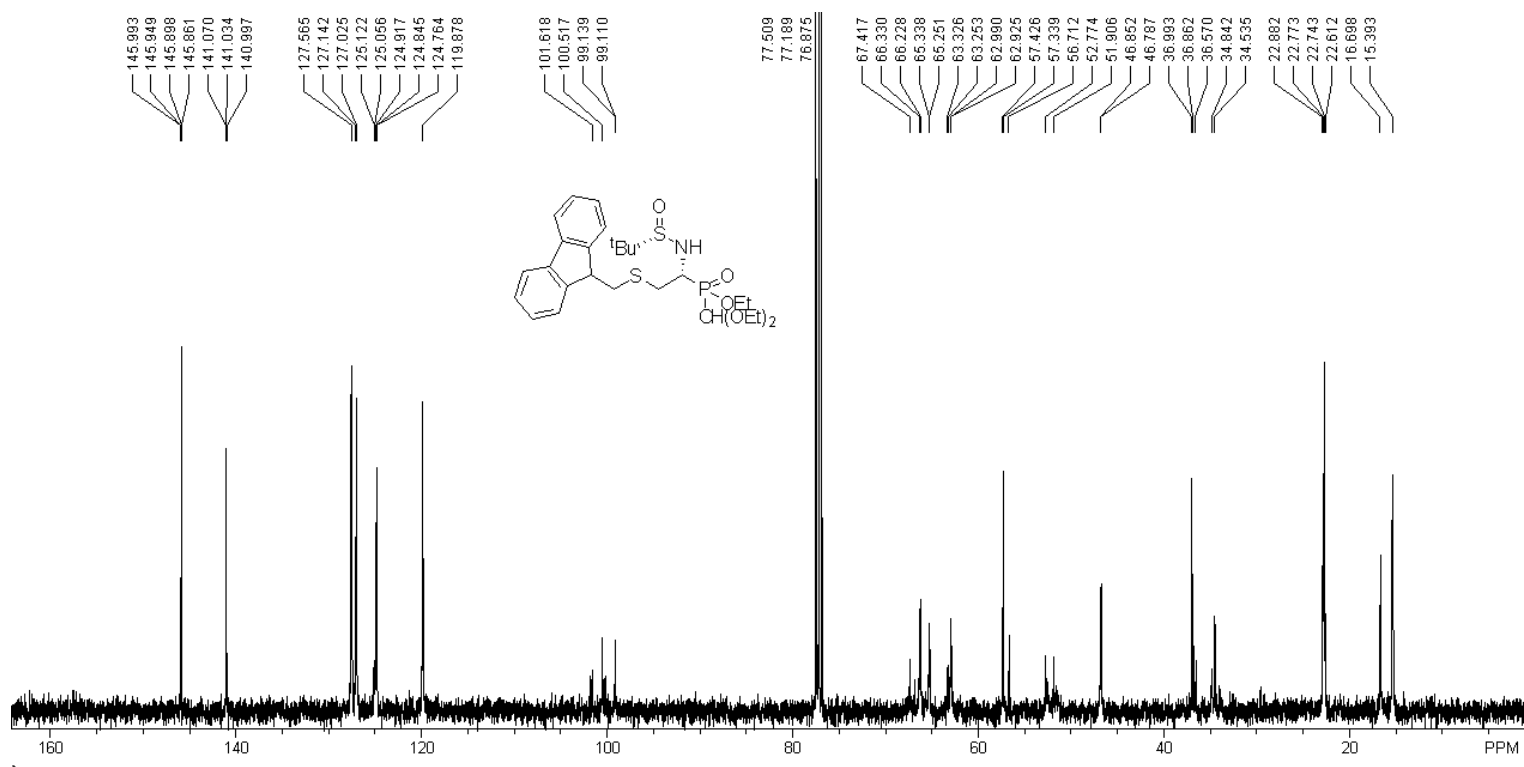


Compound 2d:

¹H NMR:



¹³C NMR:



³¹P NMR:

D:\software\nuts\DATA\2008119-10-93m_12PNMR.1

17:31:51.750 +0800 nmr@EP-ZH107708

USER: nmr

SOLVENT: CDCl3

Experiment = zgpg30

Pulse length = 8.300 usec

Recycle delay = 2.000 sec

NA = 16

Solvent = CDCl3

PTS1d = 32768

F1 = 161.971542 MHz

F2 = 1.000000 MHz

SW1 = 64102.56 Hz

AT1 = 0.51 sec

Hz per Pt1stD = 1.96 Hz

SW2 = 1.00 Hz

Hz per Pt2ndD = 1.00 Hz

O1 = -8098.6865 Hz

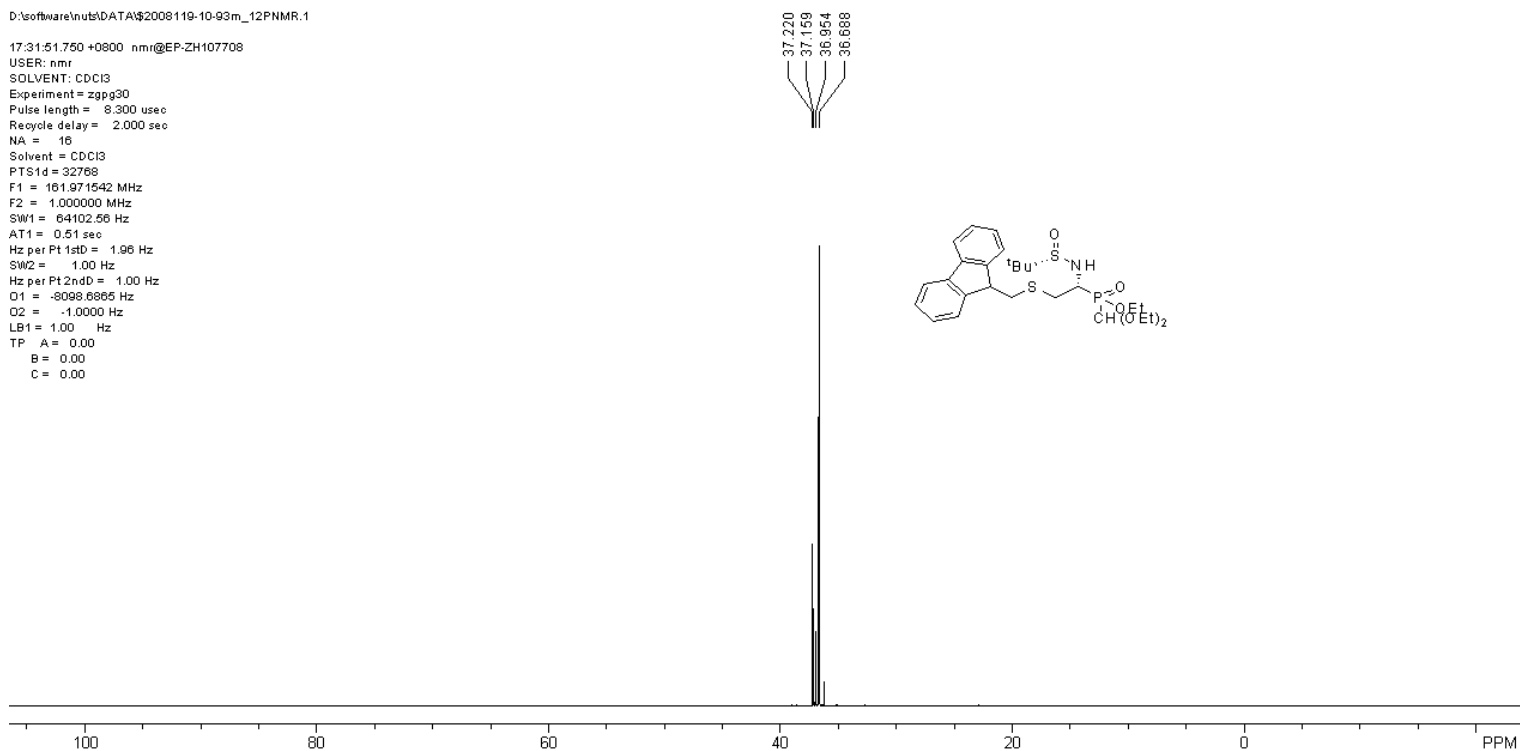
O2 = -1.0000 Hz

LB1 = 1.00 Hz

TP A = 0.00

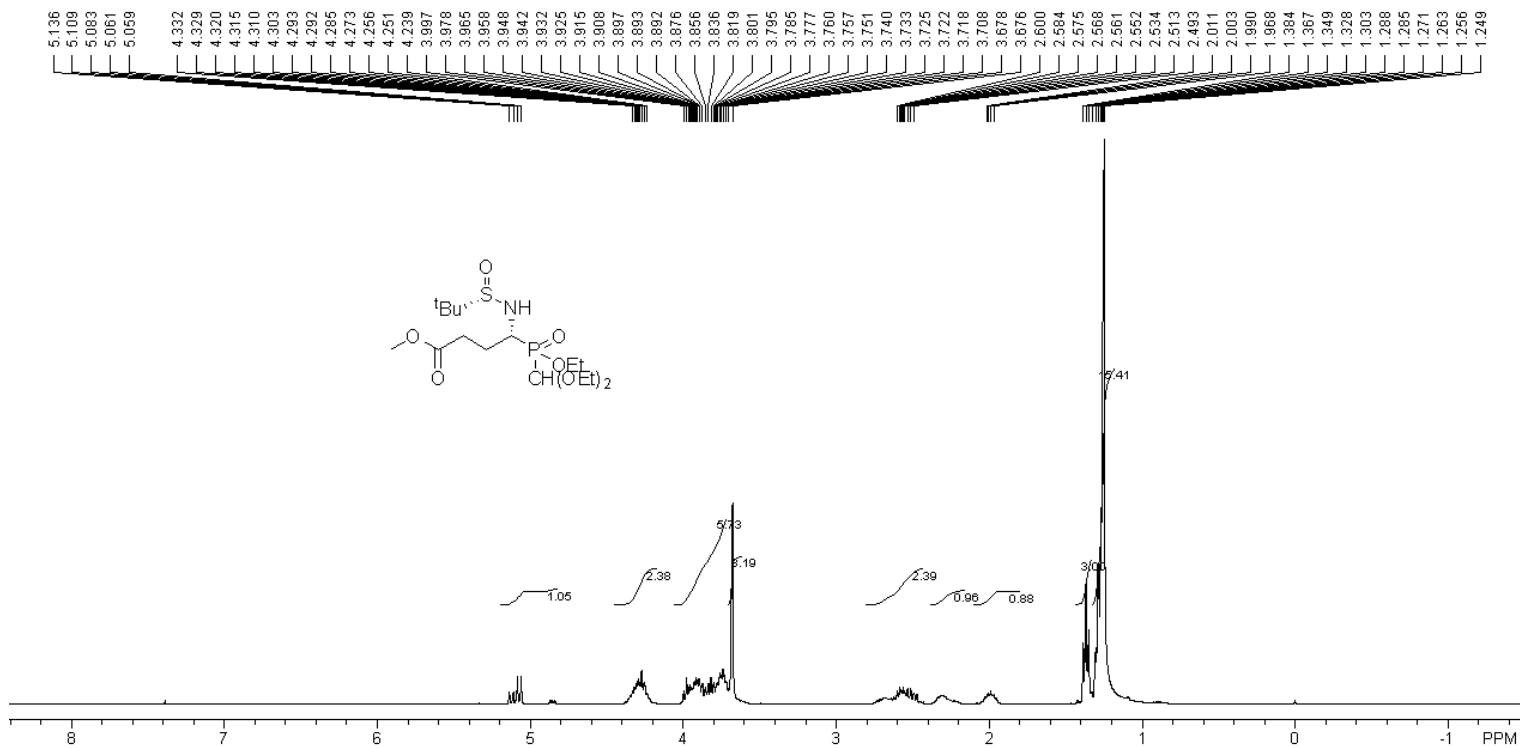
B = 0.00

C = 0.00

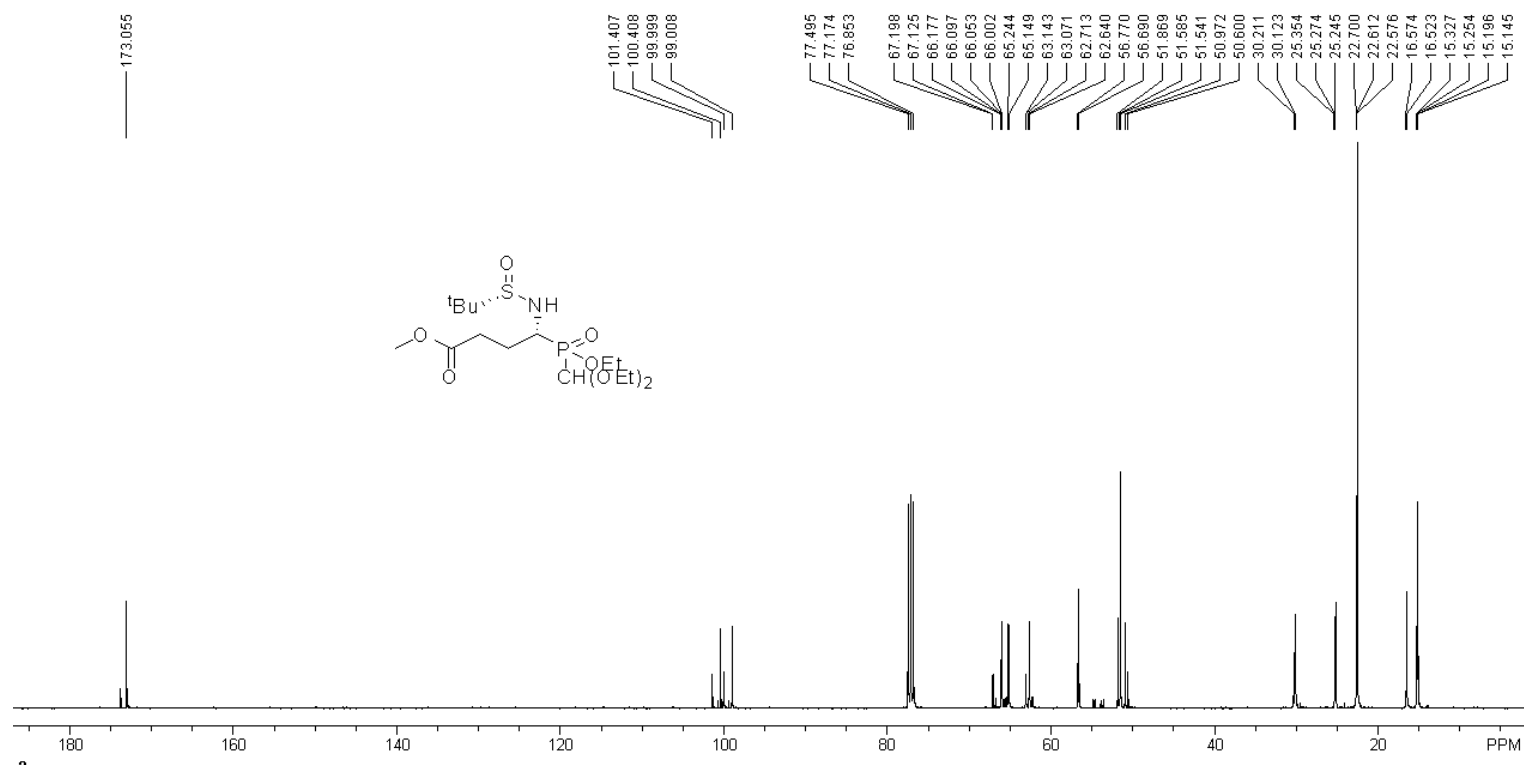


Compound 2e:

¹H NMR:

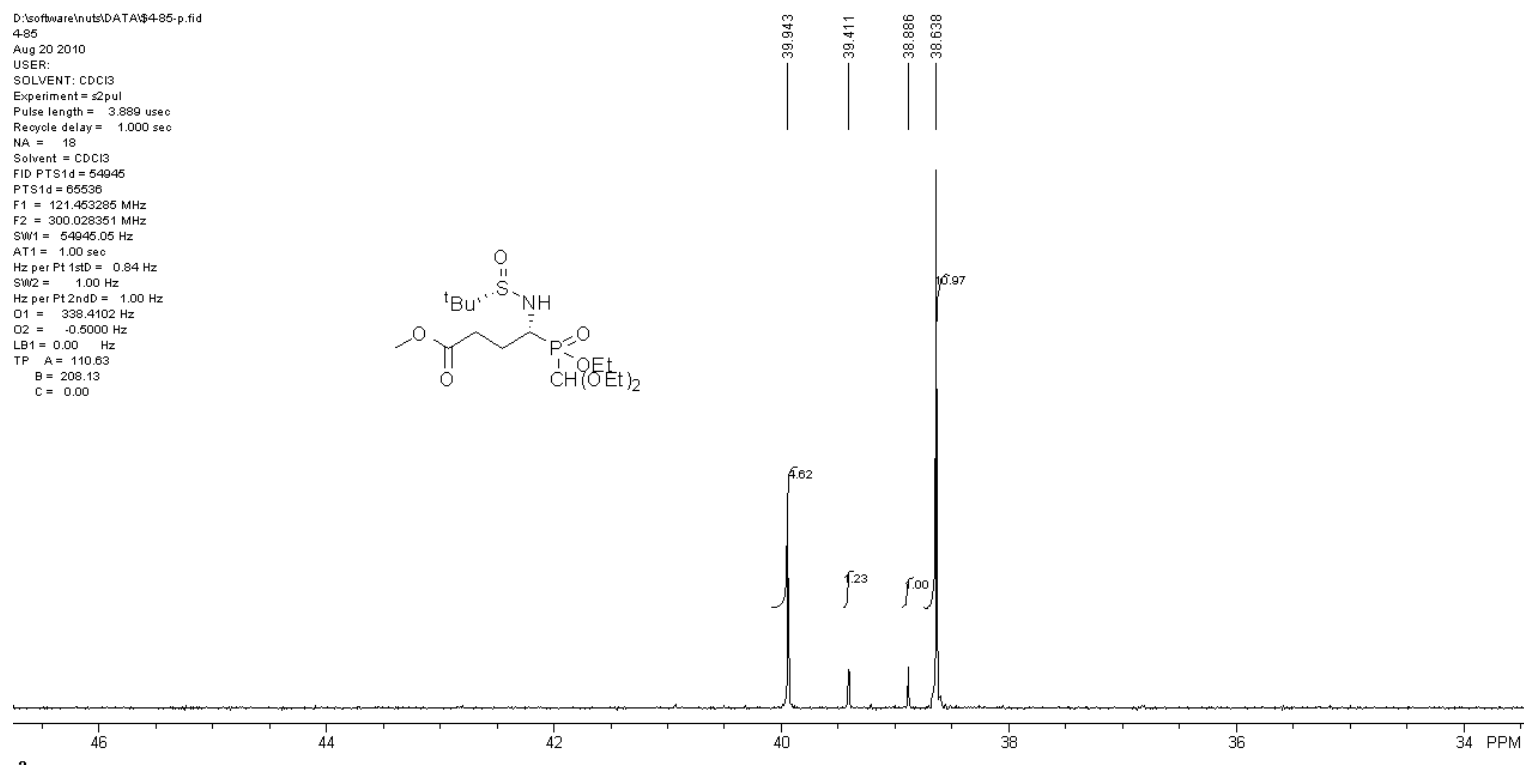


¹³C NMR:



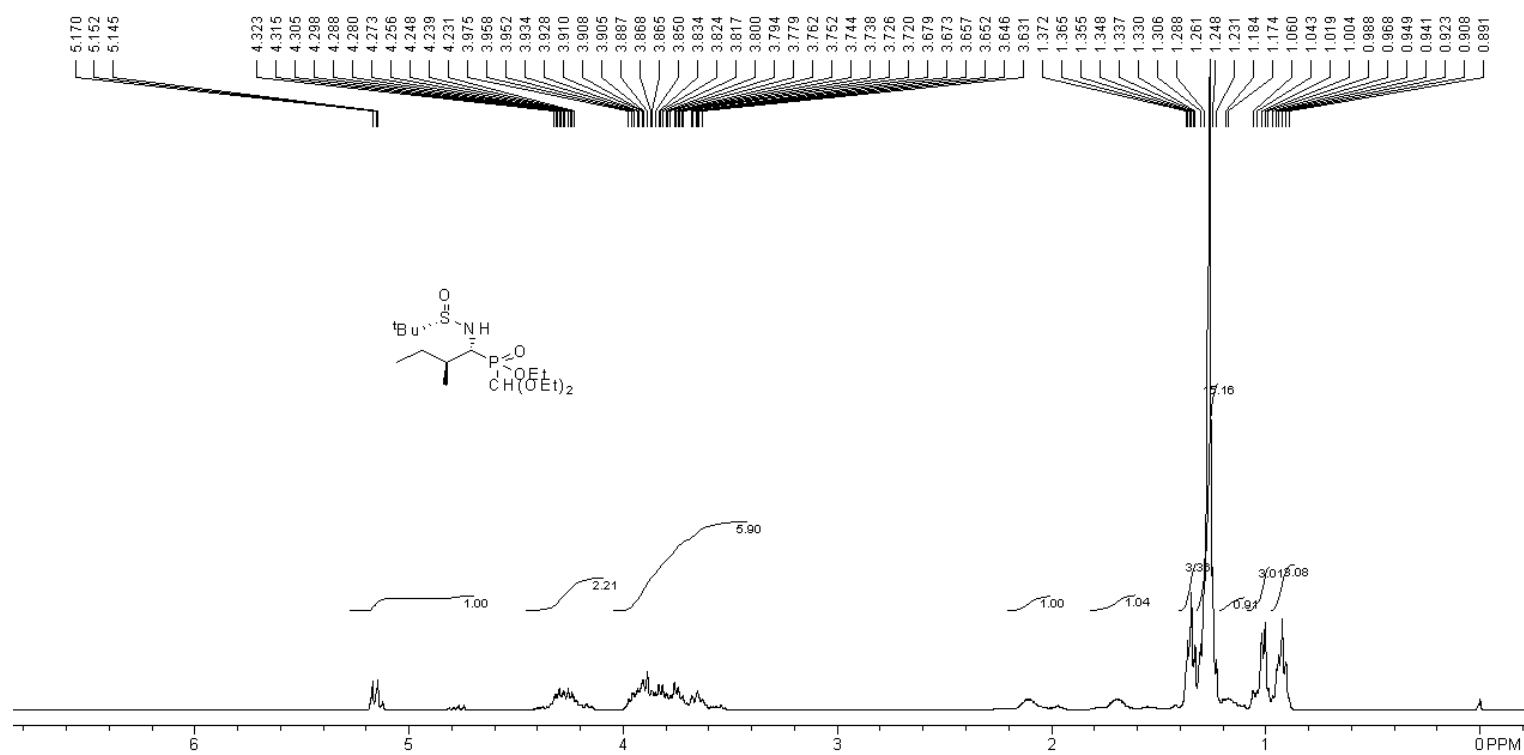
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 4-85
 Aug 20 2010
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 Pulse length = 3.889 usec
 Recycle delay = 1.000 sec
 NA = 18
 Solvent = CDCl3
 FID PTS1d = 54945
 PTS1d = 65536
 F1 = 121.463285 MHz
 F2 = 300.029351 MHz
 SW1 = 54945.05 Hz
 AT1 = 1.00 sec
 Hz per Pt1sD = 0.84 Hz
 SW2 = 1.00 Hz
 Hz per Pt2ndD = 1.00 Hz
 Q1 = 338.4102 Hz
 Q2 = -0.5000 Hz
 LB1 = 0.00 Hz
 TP A = 110.63
 B = 208.13
 C = 0.00

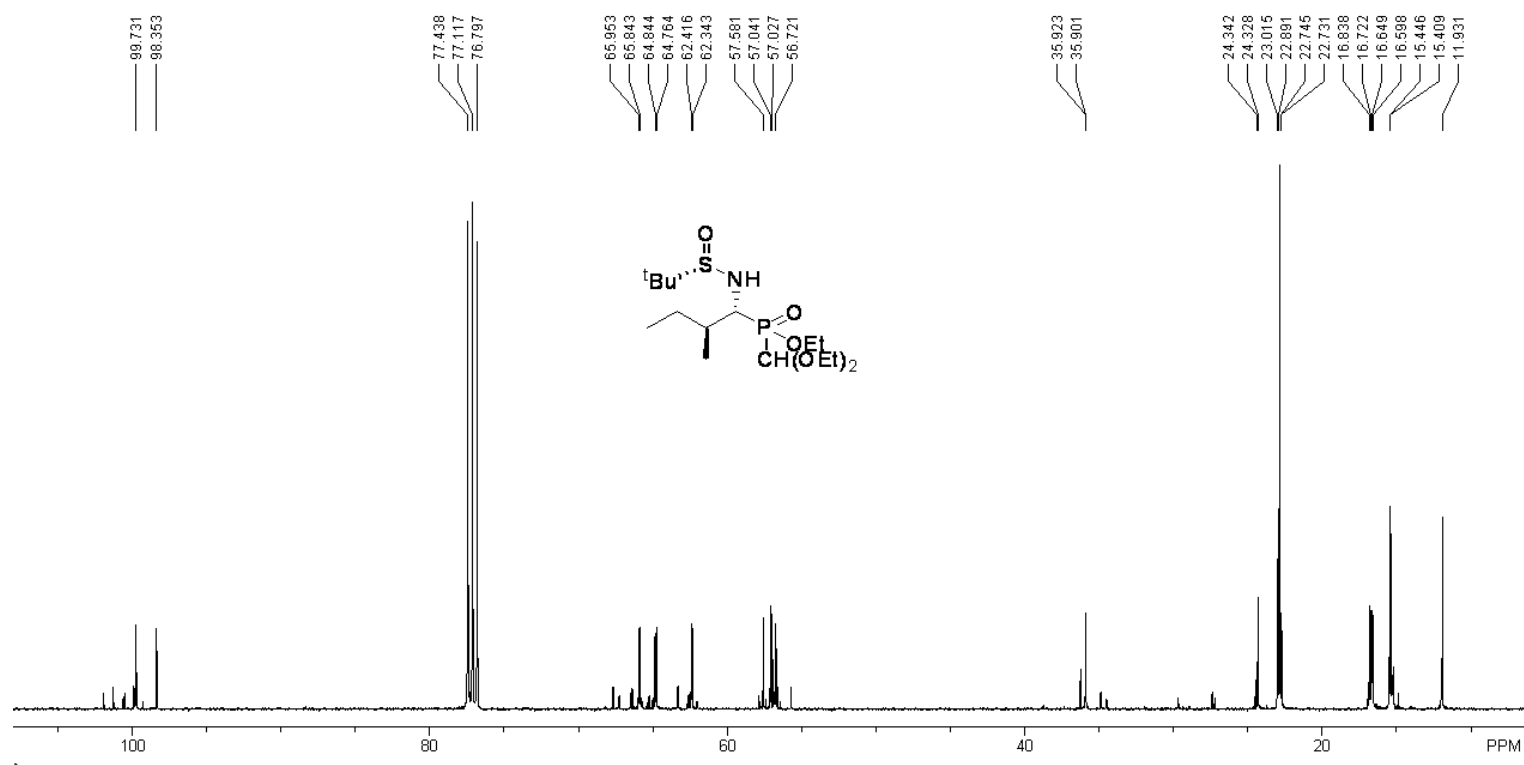


Compound 2f:

¹H NMR:



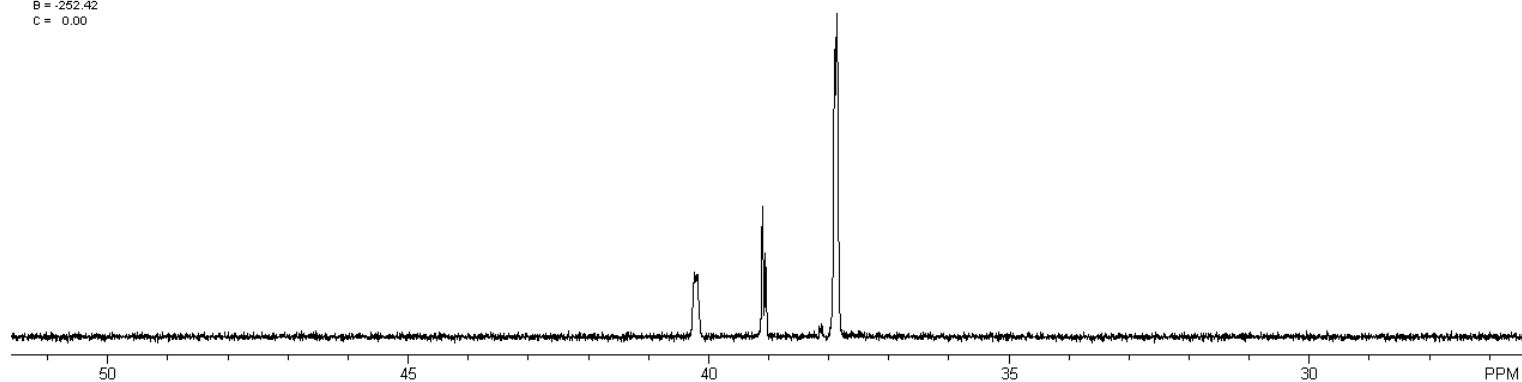
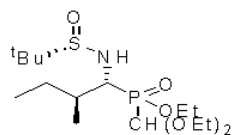
¹³C NMR:



³¹P NMR:

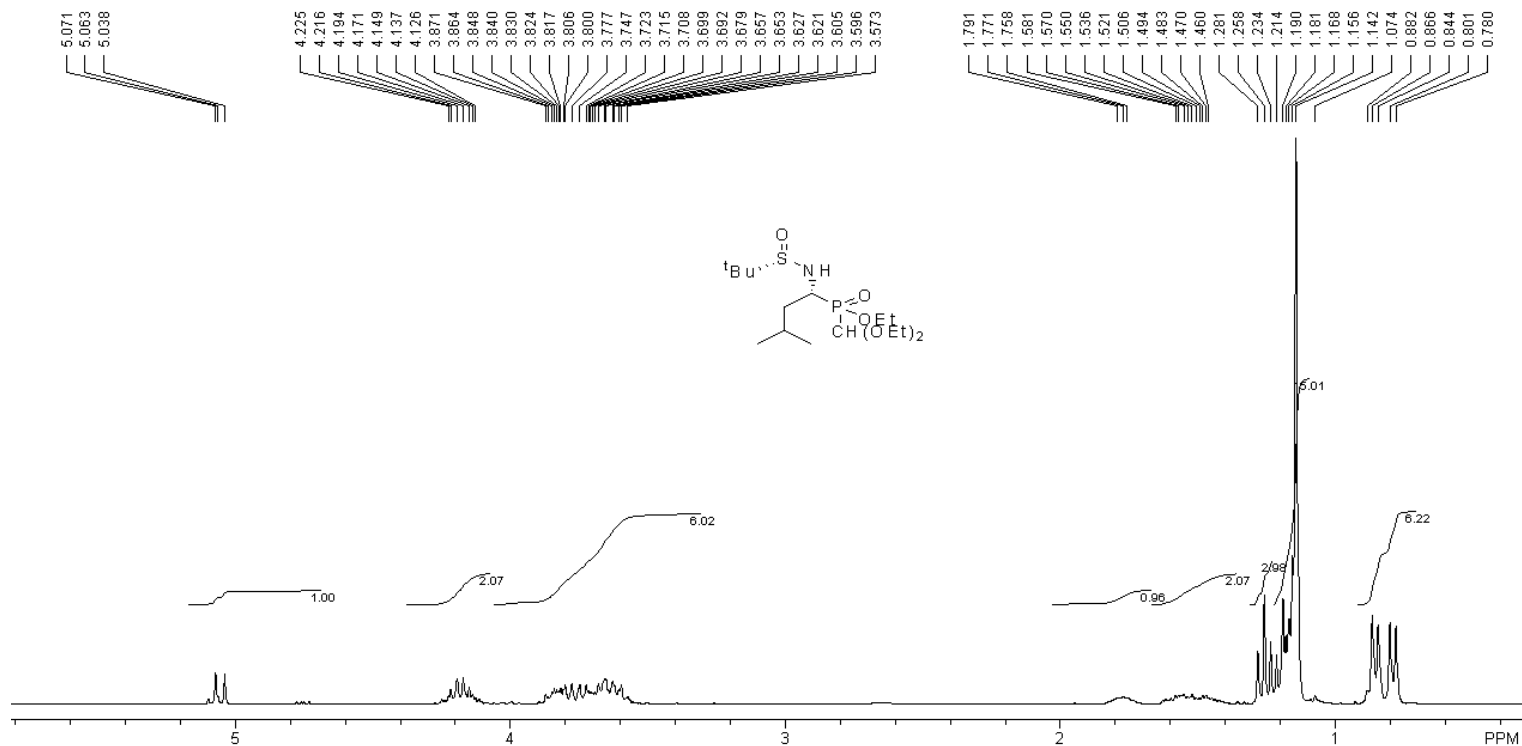
D:\software\nuts\DATA\p-3-92.fid
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 Jun 3 2010
 USER:
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 NA = 12
 Solvent = cdcl3
 FID PTS1d = 64617
 PTS1d = 65536
 F1 = 161.780167 MHz
 F2 = 399.624237 MHz
 SW1 = 40322.58 Hz
 AT1 = 1.60 sec
 Hz per Pt1st0 = 0.62 Hz
 SW2 = 1.00 Hz
 Hz per Pt2nd0 = 1.00 Hz
 O1 = 10110.6566 Hz
 O2 = -0.6000 Hz
 LB1 = 0.00 Hz
 TP A = 320.16
 B = -252.42
 C = 0.00

40.232
 40.206
 40.183
 39.103
 39.057
 37.905
 37.863

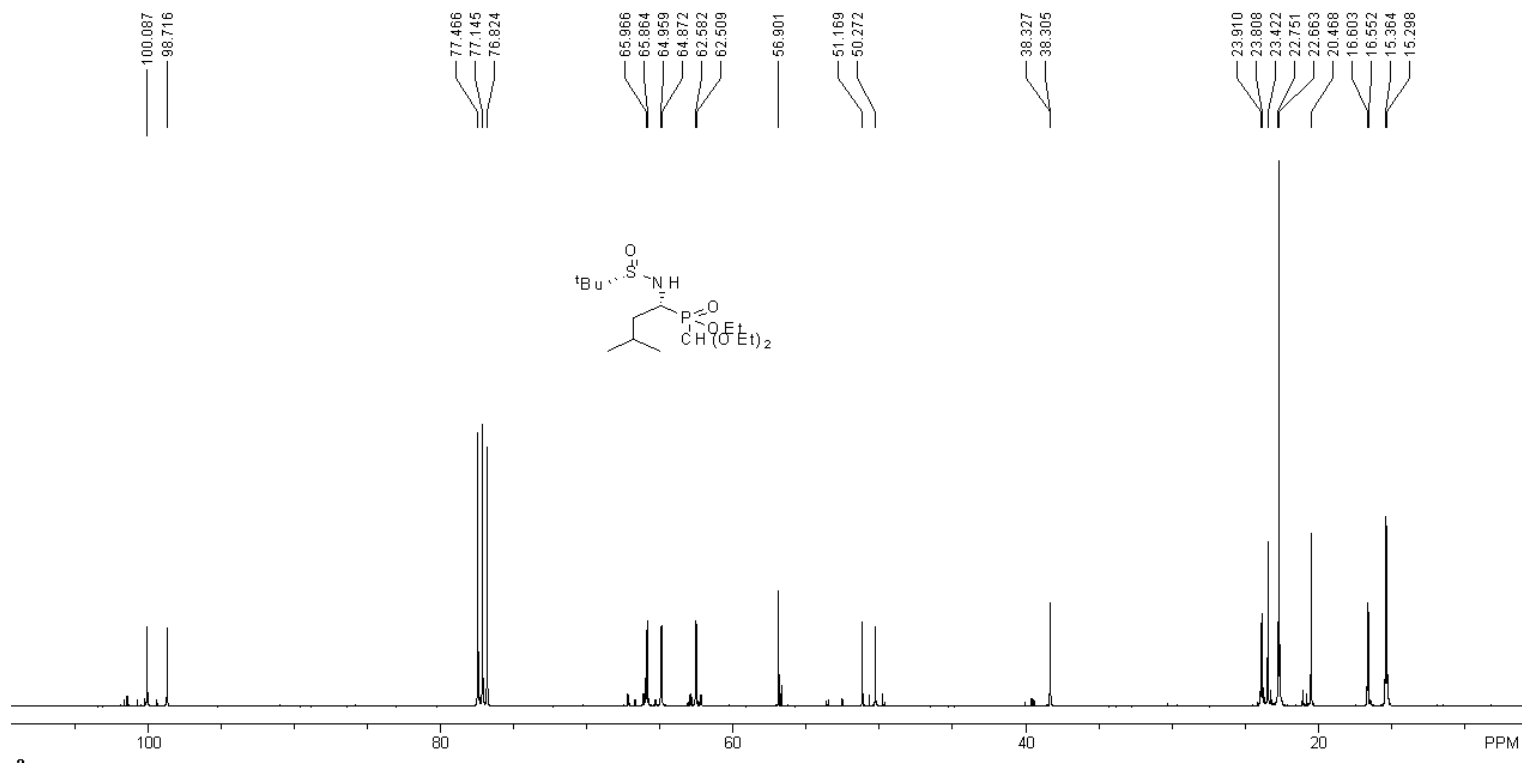


Compound 2g:

¹H NMR:

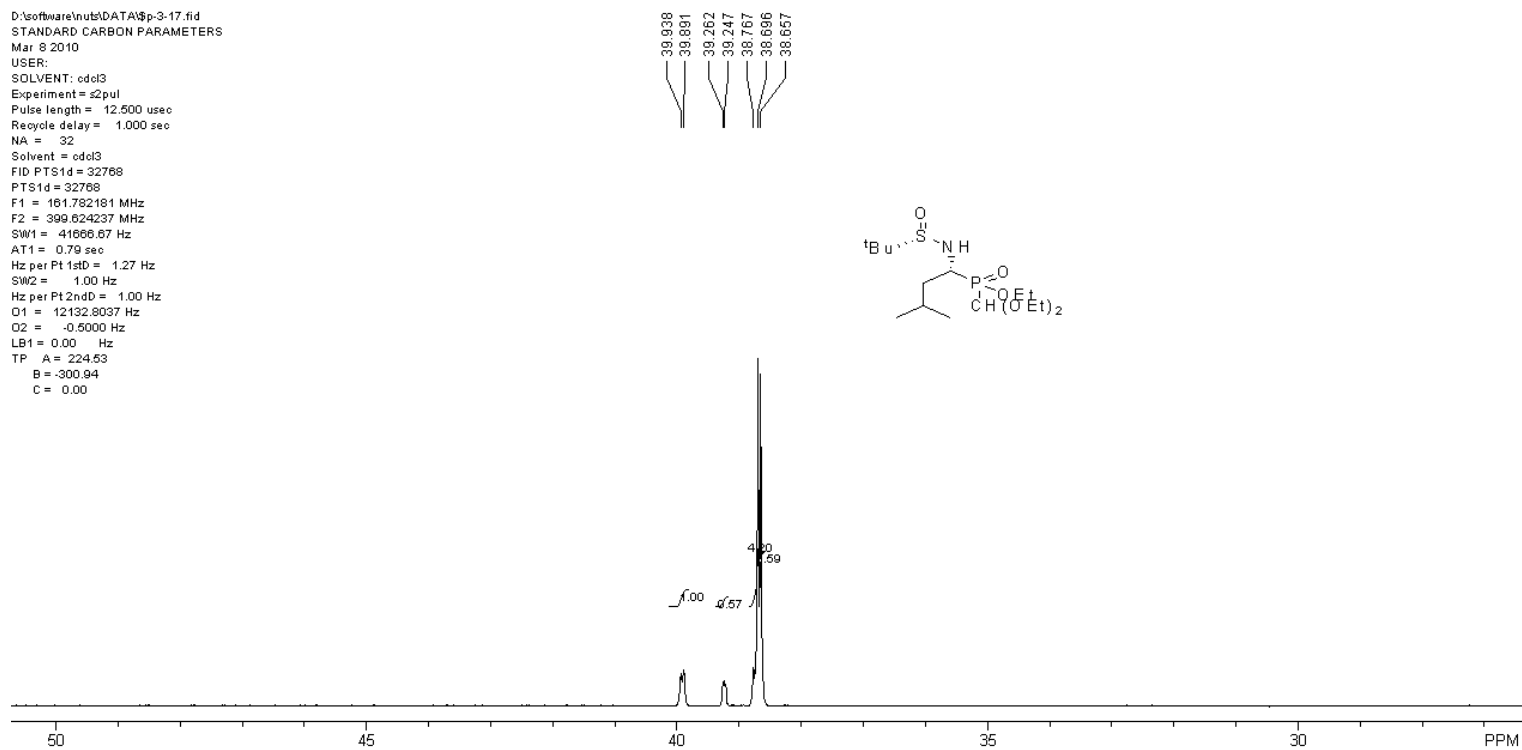


¹³C NMR:



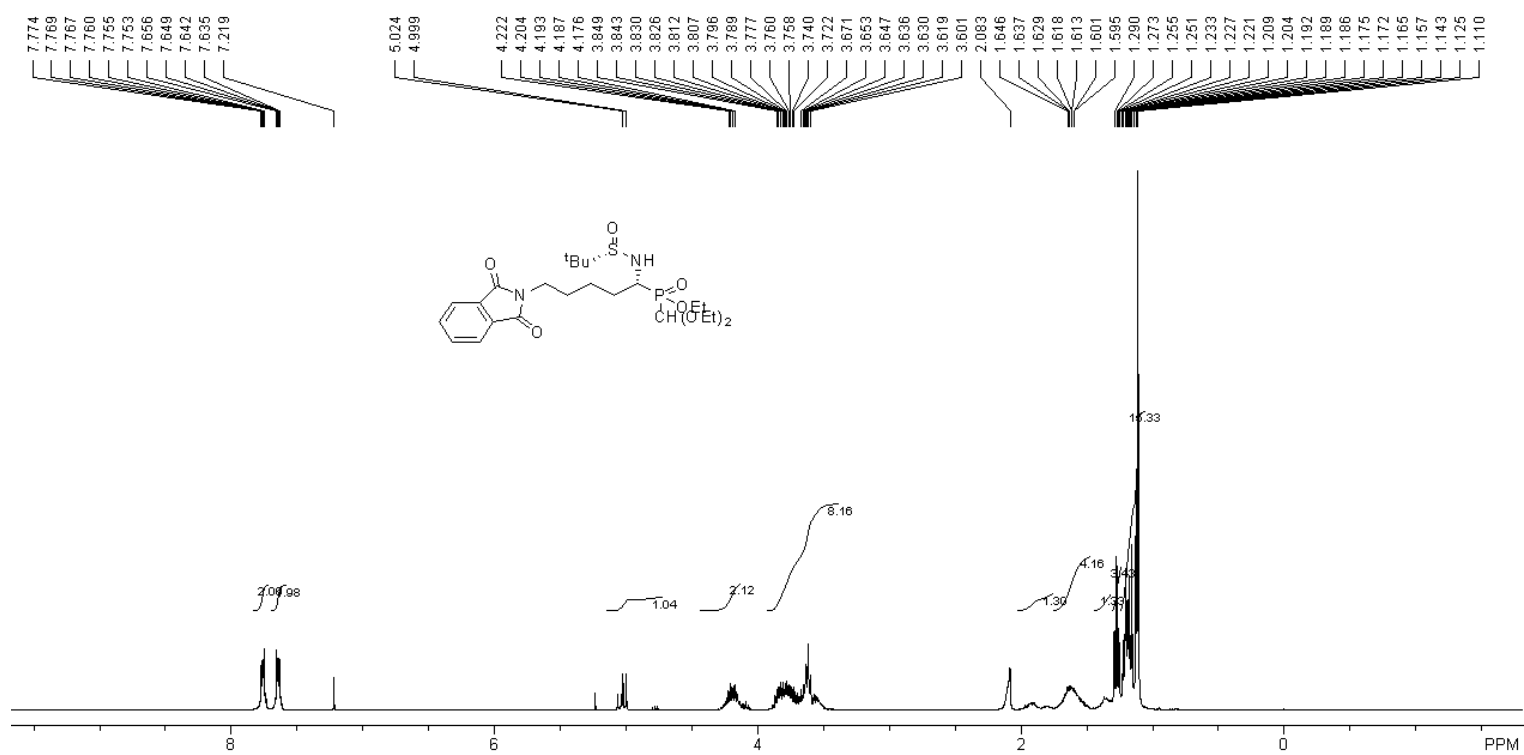
³¹P NMR:

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 STANDARD CARBON PARAMETERS
 Mar 8 2010
 USER:
 SOLVENT: cdcl3
 Experiment = s2pul
 Pulse length = 12.500 usec
 Recycle delay = 1.000 sec
 NA = 32
 Solvent = cdcl3
 FID PTS1d = 32768
 PTS1d = 32768
 F1 = 161.782181 MHz
 F2 = 399.624237 MHz
 SW1 = 41666.67 Hz
 AT1 = 0.79 sec
 Hz per Pt1s0 = 1.27 Hz
 SW2 = 1.00 Hz
 Hz per Pt2nd0 = 1.00 Hz
 O1 = 12132.8037 Hz
 O2 = -0.6000 Hz
 LB1 = 0.00 Hz
 TP A = 224.63
 B = -300.94
 C = 0.00

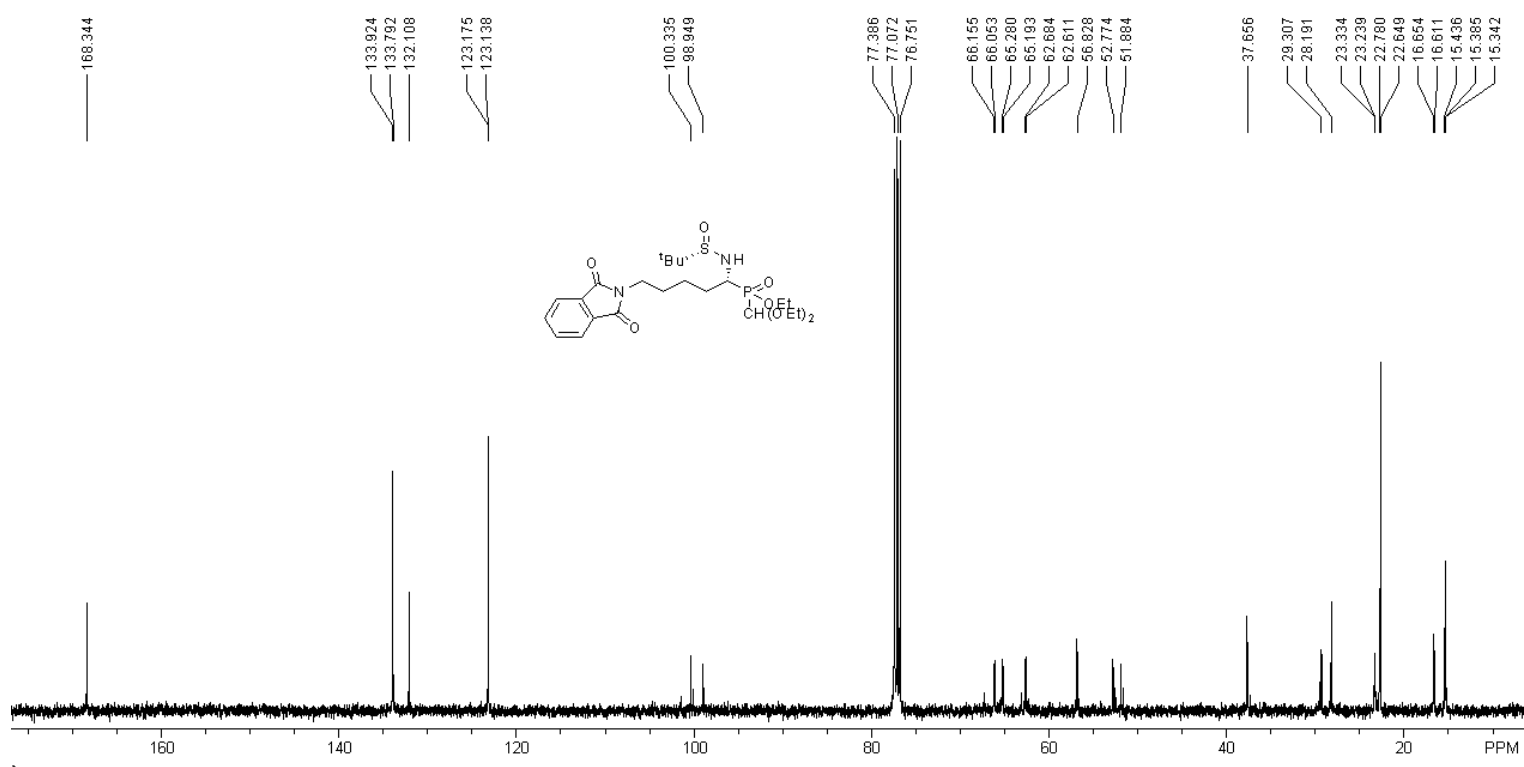


Compound 2h:

¹H NMR:

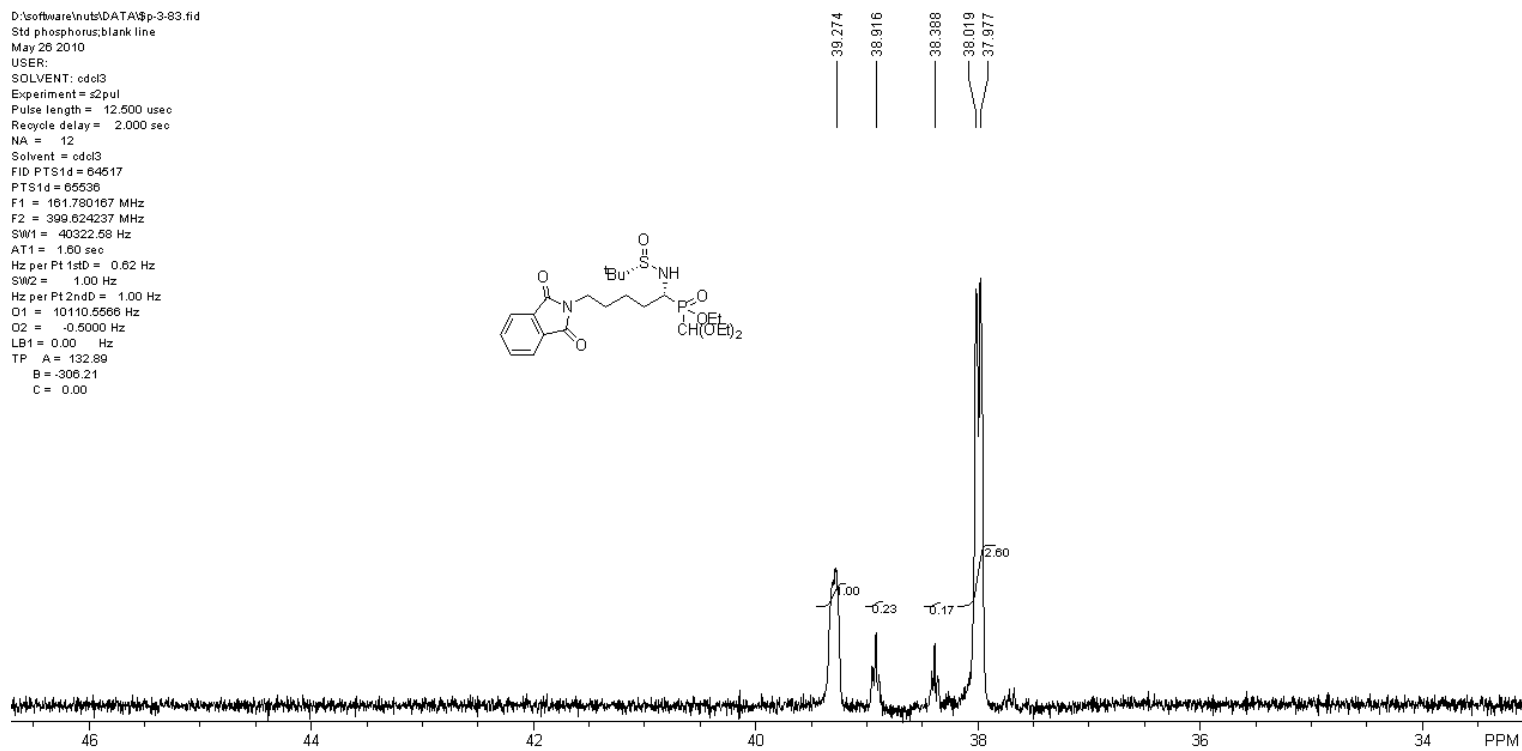
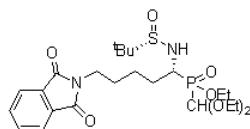


¹³C NMR:



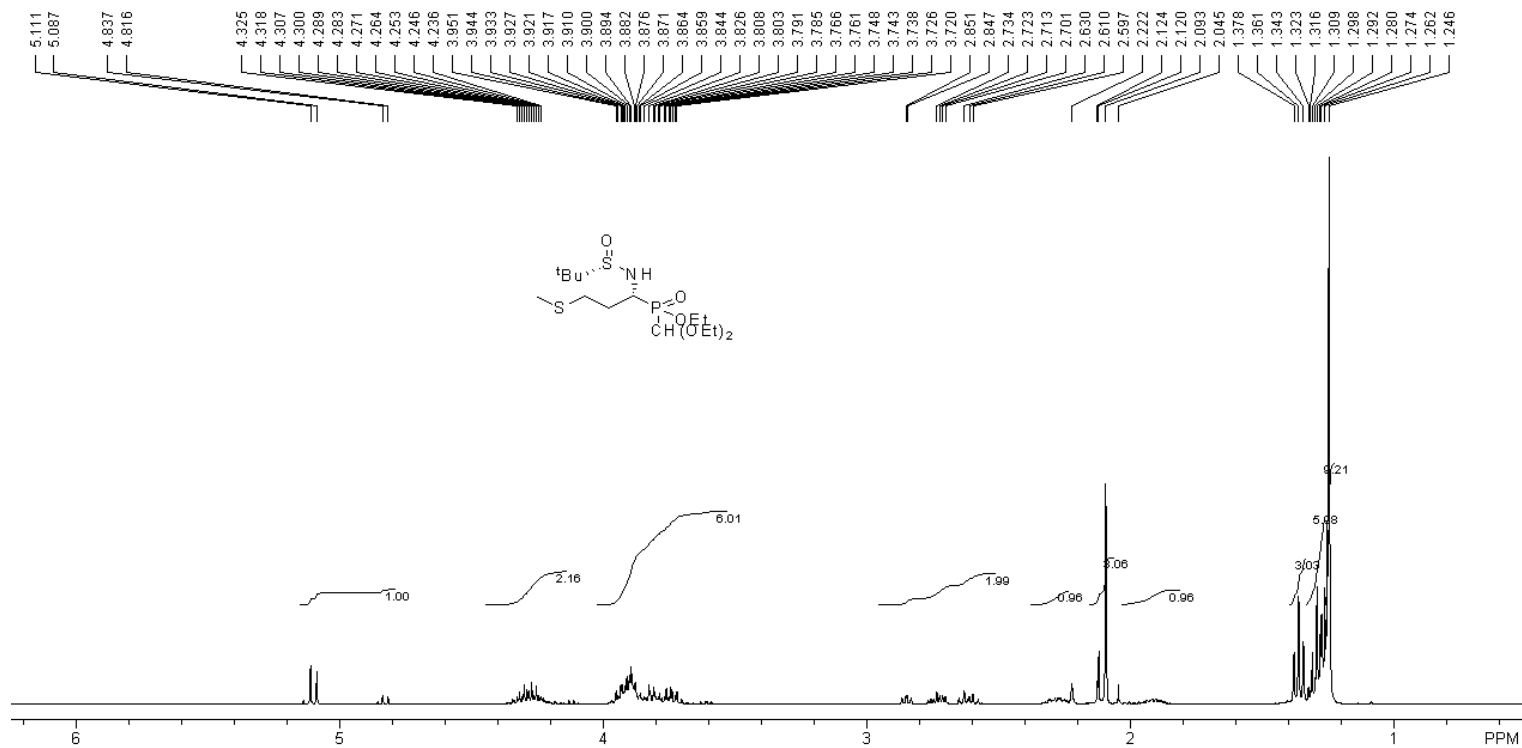
³¹P NMR:

D:\software\nuts\DATA\p-3-83.fid
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 May 26 2010
 USER:
 SOLVENT: cdcl3
 Experiment = s2pul
 Pulse length = 12.500 usec
 Recycle delay = 2.000 sec
 NA = 12
 Solvent = cdcl3
 FID PTS1d = 64617
 PTS1d = 65536
 F1 = 161.780167 MHz
 F2 = 399.624237 MHz
 SW1 = 40322.58 Hz
 AT1 = 1.60 sec
 Hz per Pt1st0 = 0.62 Hz
 SW2 = 1.00 Hz
 Hz per Pt2nd0 = 1.00 Hz
 O1 = 10110.6586 Hz
 O2 = -0.6000 Hz
 LB1 = 0.00 Hz
 TP A = 132.89
 B = -306.21
 C = 0.00

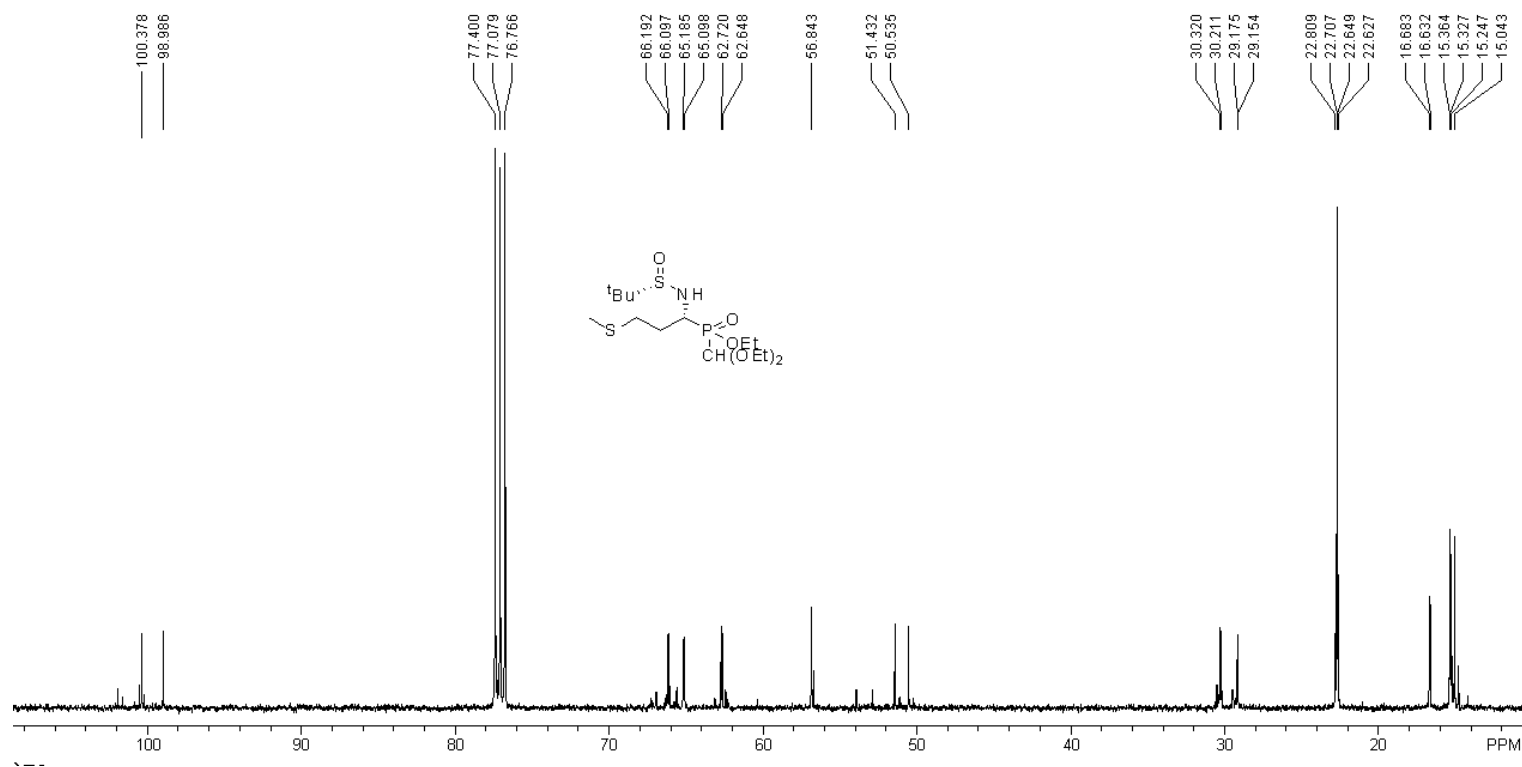


Compound 2i:

¹H NMR:

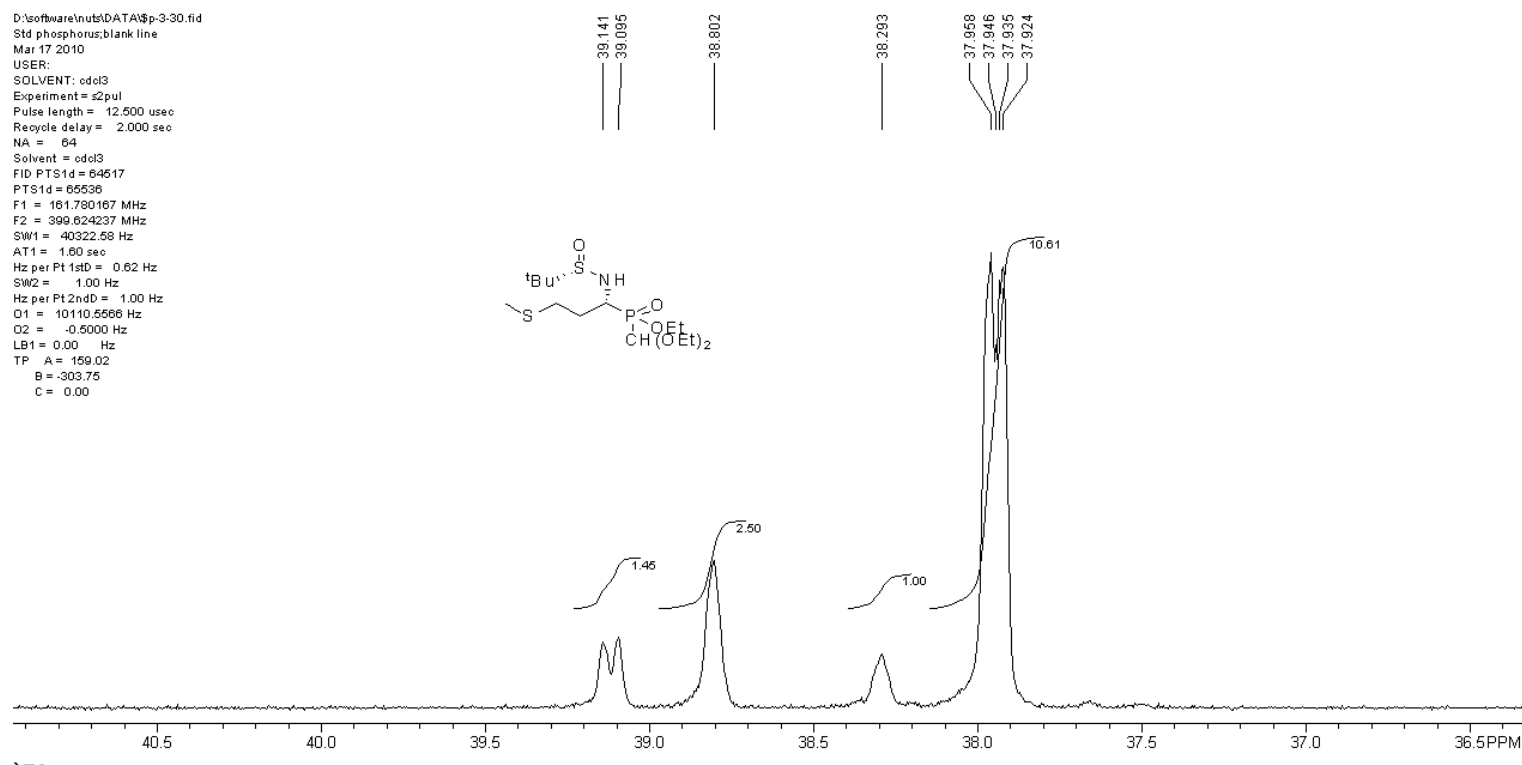


¹³C NMR:



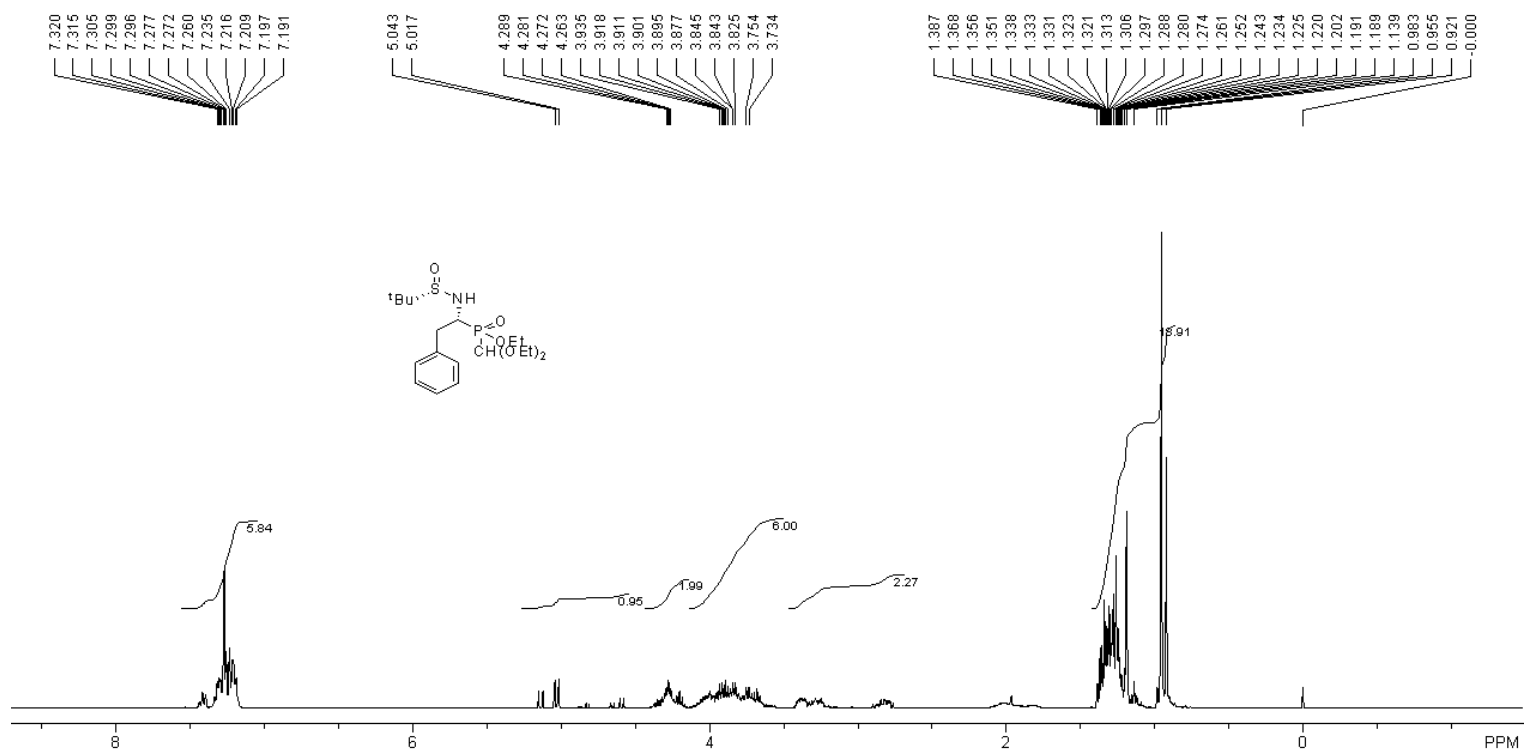
³¹P NMR:

D:\software\nuts\DATA\p-3-30.fid
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 Mar 17 2010
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 Recycle delay = 2.000 sec
 NA = 64
 Solvent = cdcl3
 FID PTS1d = 64617
 PTS1d = 65536
 F1 = 161.780167 MHz
 F2 = 399.624237 MHz
 SW1 = 40322.58 Hz
 AT1 = 1.60 sec
 Hz per Pt1s0 = 0.62 Hz
 SW2 = 1.00 Hz
 Hz per Pt2nd0 = 1.00 Hz
 Q1 = 10110.5566 Hz
 Q2 = -0.5000 Hz
 LB1 = 0.00 Hz
 TP A = 159.02
 B = -303.75
 C = 0.00

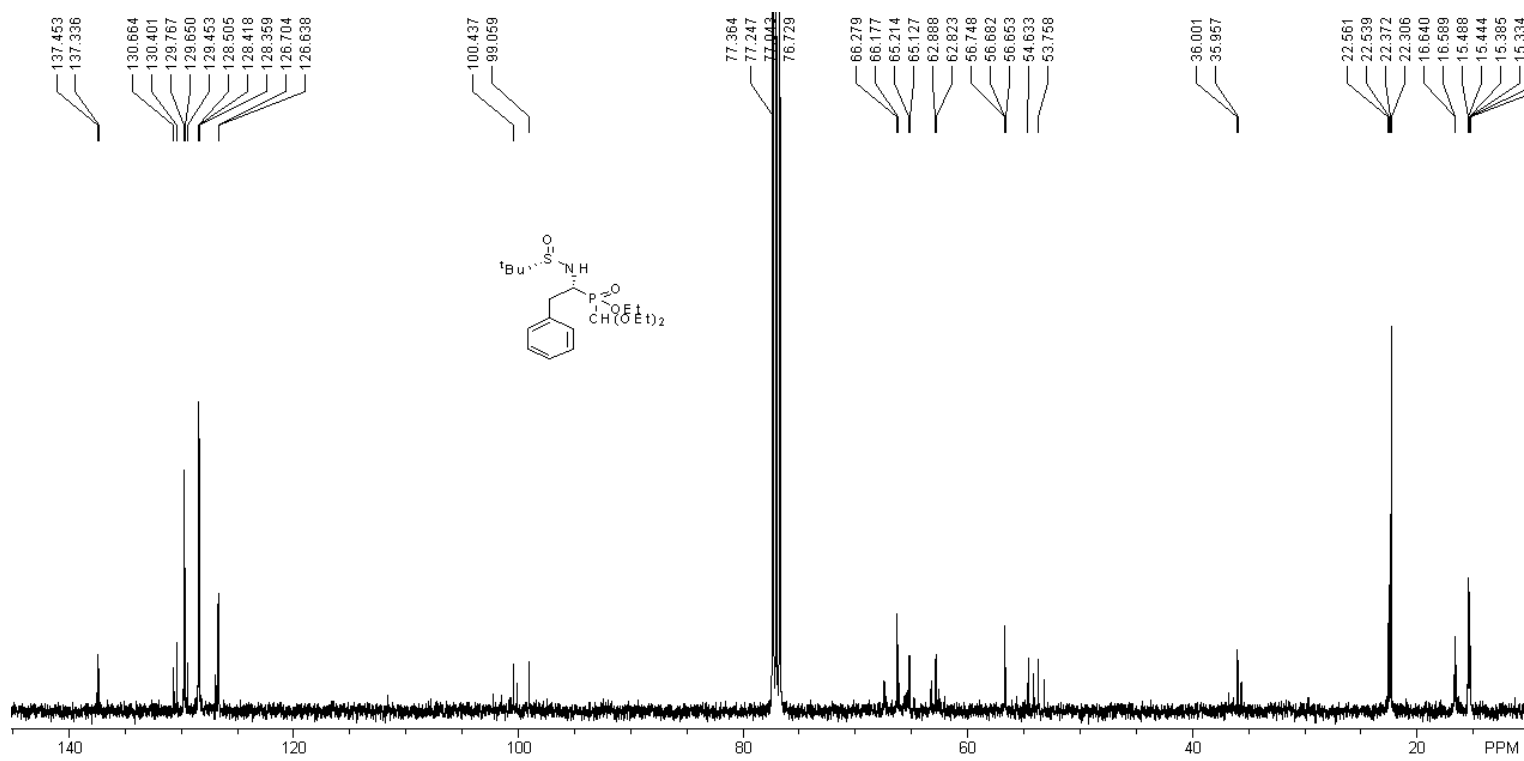


Compound 2j:

¹H NMR:

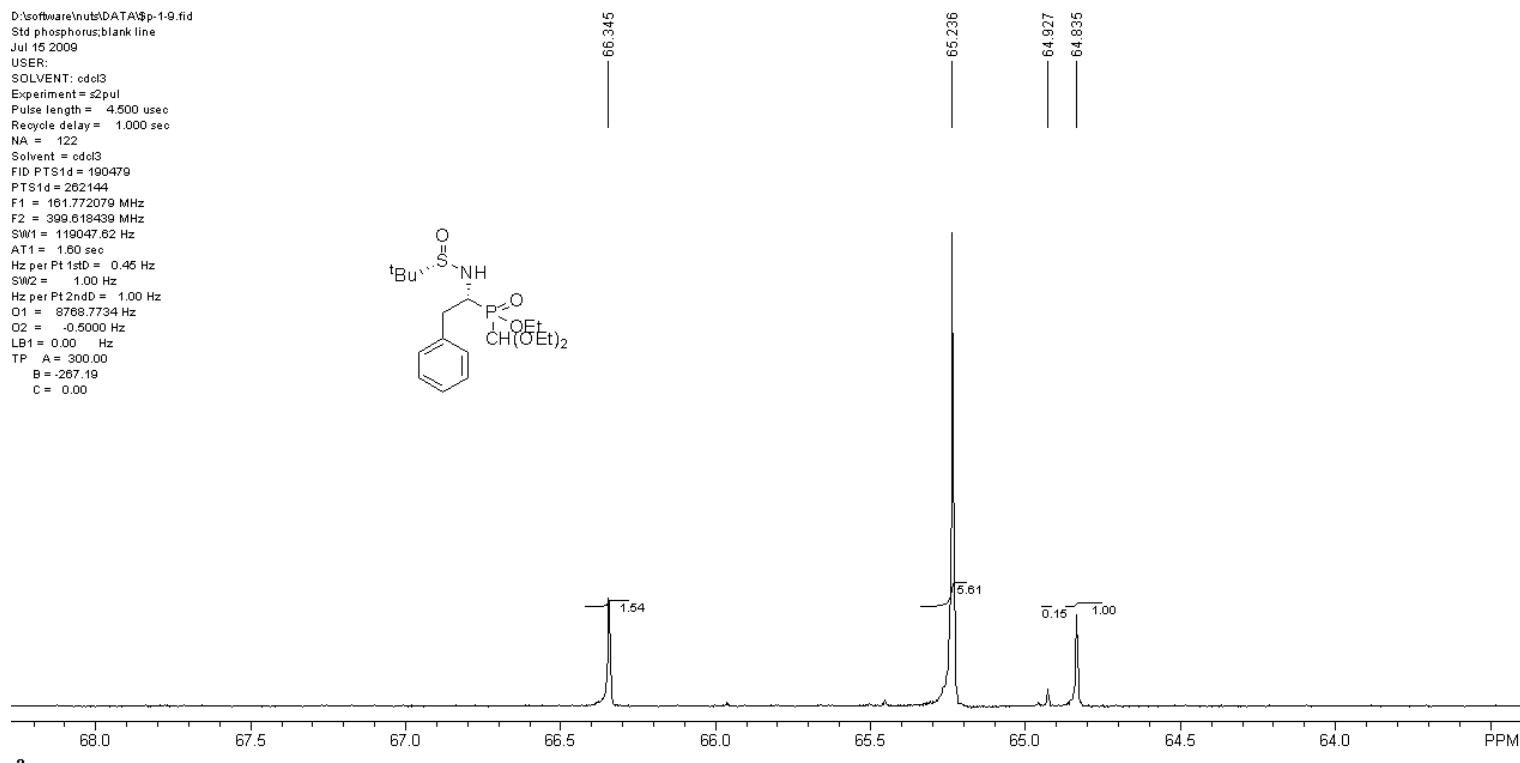
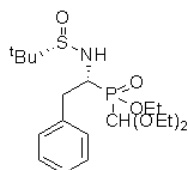


¹³C NMR:



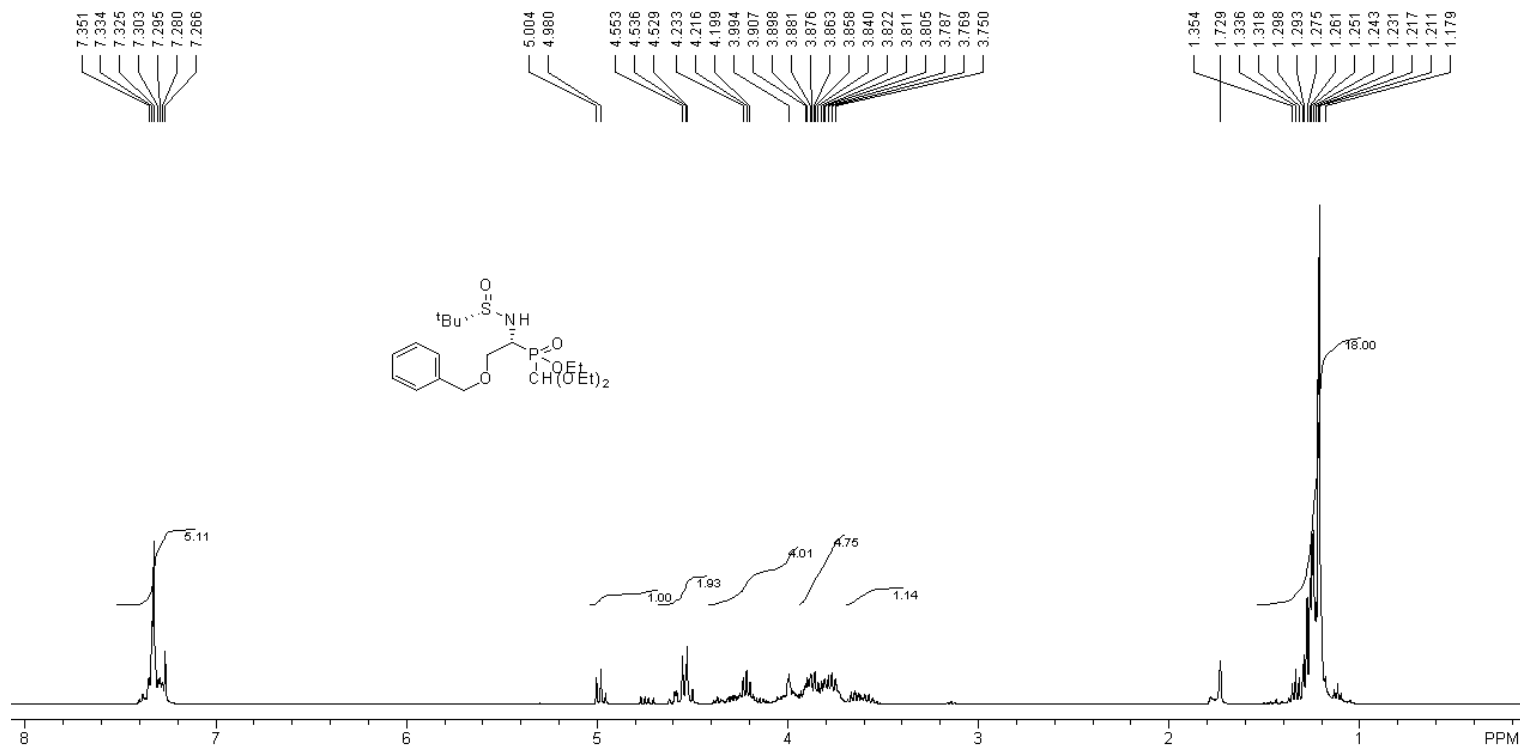
³¹P NMR:

D:\software\nuts\DATA\p-1-9.fid
 Std phosphorus;blank line
 Jul 15 2009
 USER:
 SOLVENT: cdcl3
 Experiment = s2pul
 Pulse length = 4.500 usec
 Recycle delay = 1.000 sec
 NA = 122
 Solvent = cdcl3
 FID PTS1d = 190479
 PTS1d = 262144
 F1 = 161.772079 MHz
 F2 = 399.618439 MHz
 SW1 = 119047.62 Hz
 AT1 = 1.60 sec
 Hz per Pt1stD = 0.45 Hz
 SW2 = 1.00 Hz
 Hz per Pt2ndD = 1.00 Hz
 O1 = 8768.7734 Hz
 O2 = -0.6000 Hz
 LB1 = 0.00 Hz
 TP A = 300.00
 B = -267.19
 C = 0.00

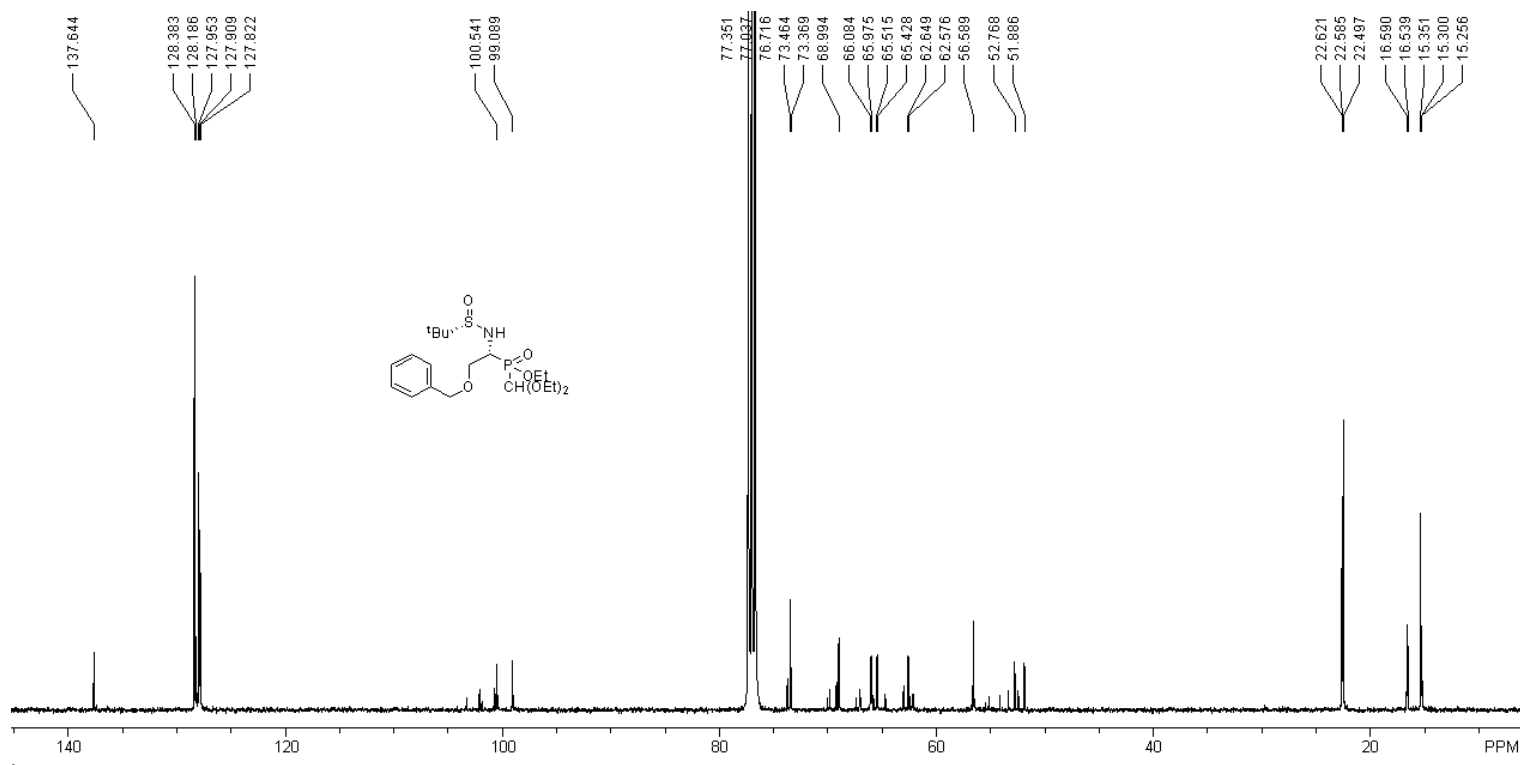


Compound 21:

¹H NMR:

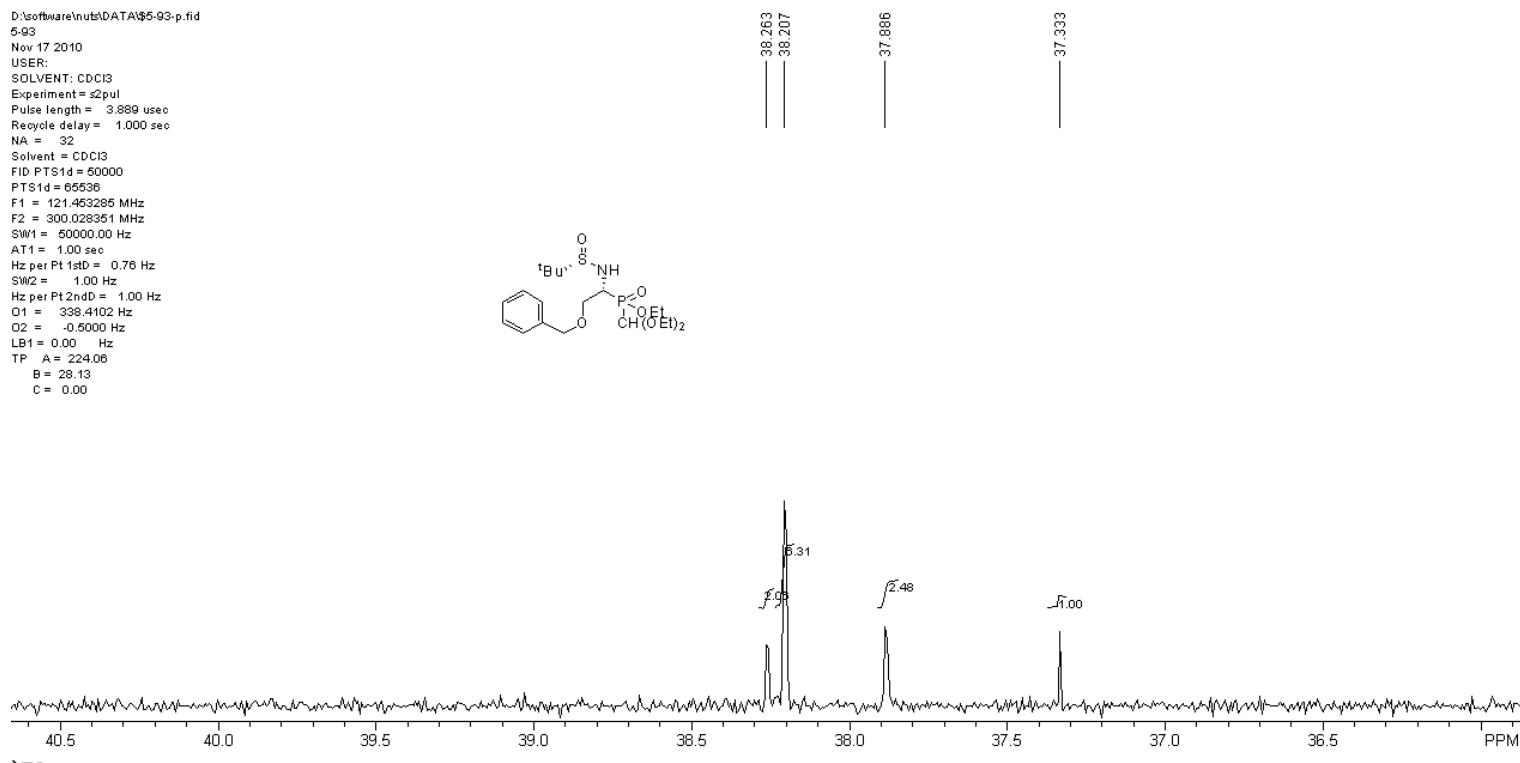


¹³C NMR:



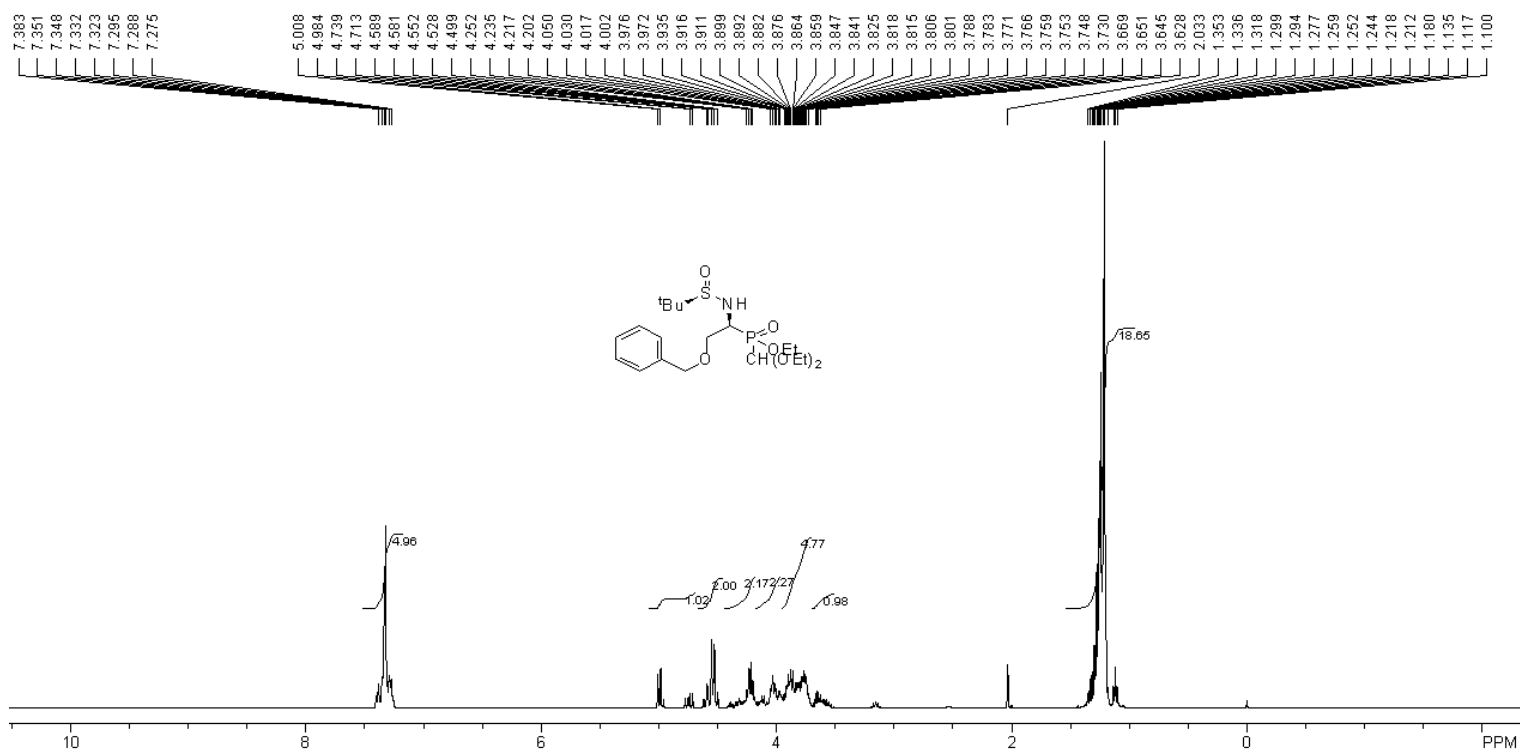
³¹P NMR:

D:\software\nuts\DATA\5-93-p.fid
 5-93
 Nov 17 2010
 USER:
 SOLVENT: CDCl3
 Experiment = s2pul
 Pulse length = 3.889 usec
 Recycle delay = 1.000 sec
 NA = 32
 Solvent = CDCl3
 FID PTS1d = 50000
 PTS1d = 65536
 F1 = 121.463285 MHz
 F2 = 300.028351 MHz
 SW1 = 50000.00 Hz
 AT1 = 1.00 sec
 Hz per Pt1s0 = 0.76 Hz
 SW2 = 1.00 Hz
 Hz per Pt2nd0 = 1.00 Hz
 Q1 = 338.4102 Hz
 Q2 = -0.5000 Hz
 LB1 = 0.00 Hz
 TP A = 224.06
 B = 28.13
 C = 0.00

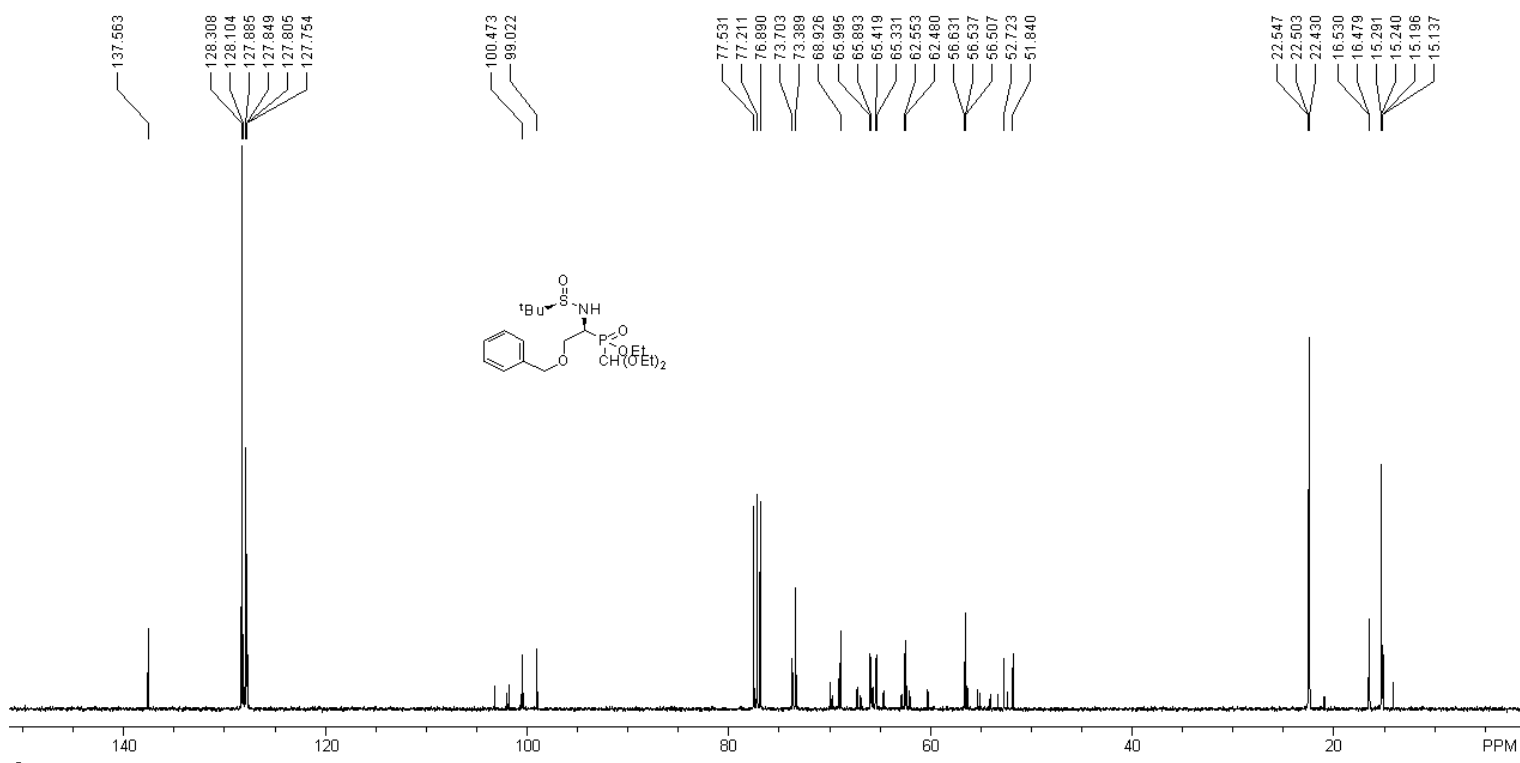


Compound (-)-2l:

¹H NMR:



¹³C NMR:



³¹P NMR:

D:\software\nuts\DATA\2008119-11-17H_11.1

21:07:10.890 +0800 nmr@EP-ZH107708

USER: nmr

SOLVENT: CDCl3

Experiment = zgpg30

Pulse length = 8.300 usec

Recycle delay = 2.000 sec

NA = 16

Solvent = CDCl3

PTS1d = 32768

F1 = 161.971542 MHz

F2 = 1.000000 MHz

SW1 = 64102.56 Hz

AT1 = 0.51 sec

Hz per Pt1stD = 1.96 Hz

SW2 = 1.00 Hz

Hz per Pt2ndD = 1.00 Hz

O1 = -8098.6865 Hz

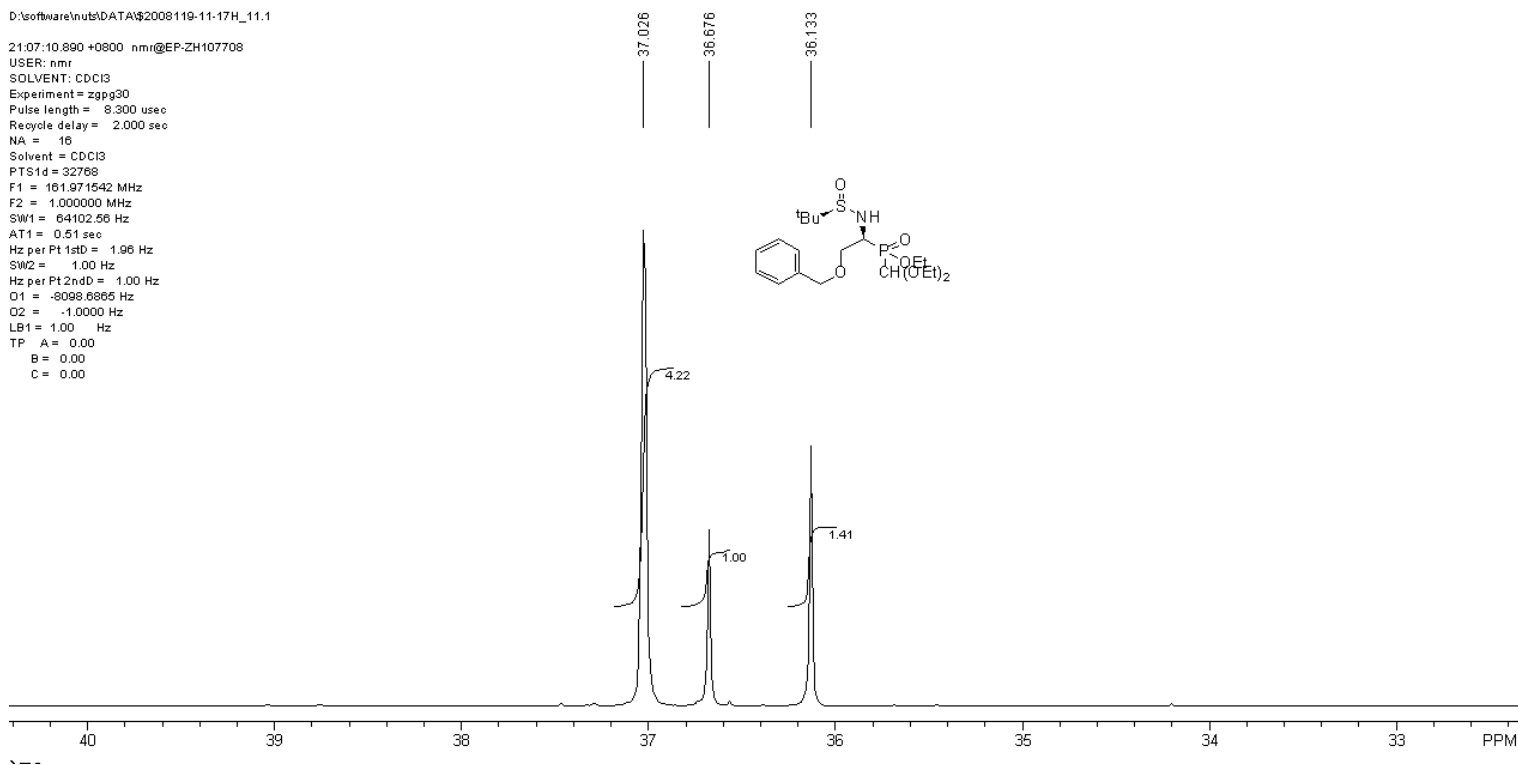
O2 = -1.0000 Hz

LB1 = 1.00 Hz

TP A = 0.00

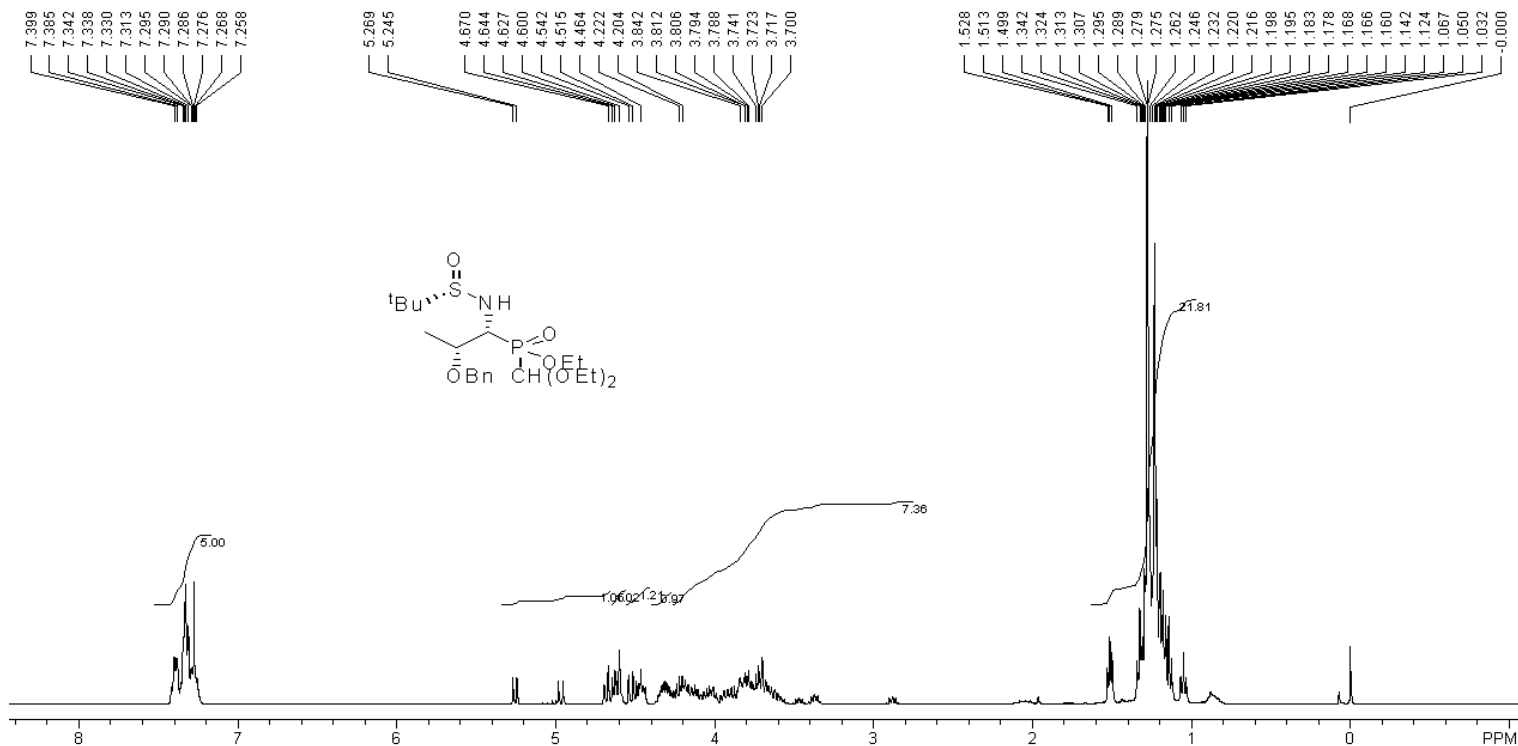
B = 0.00

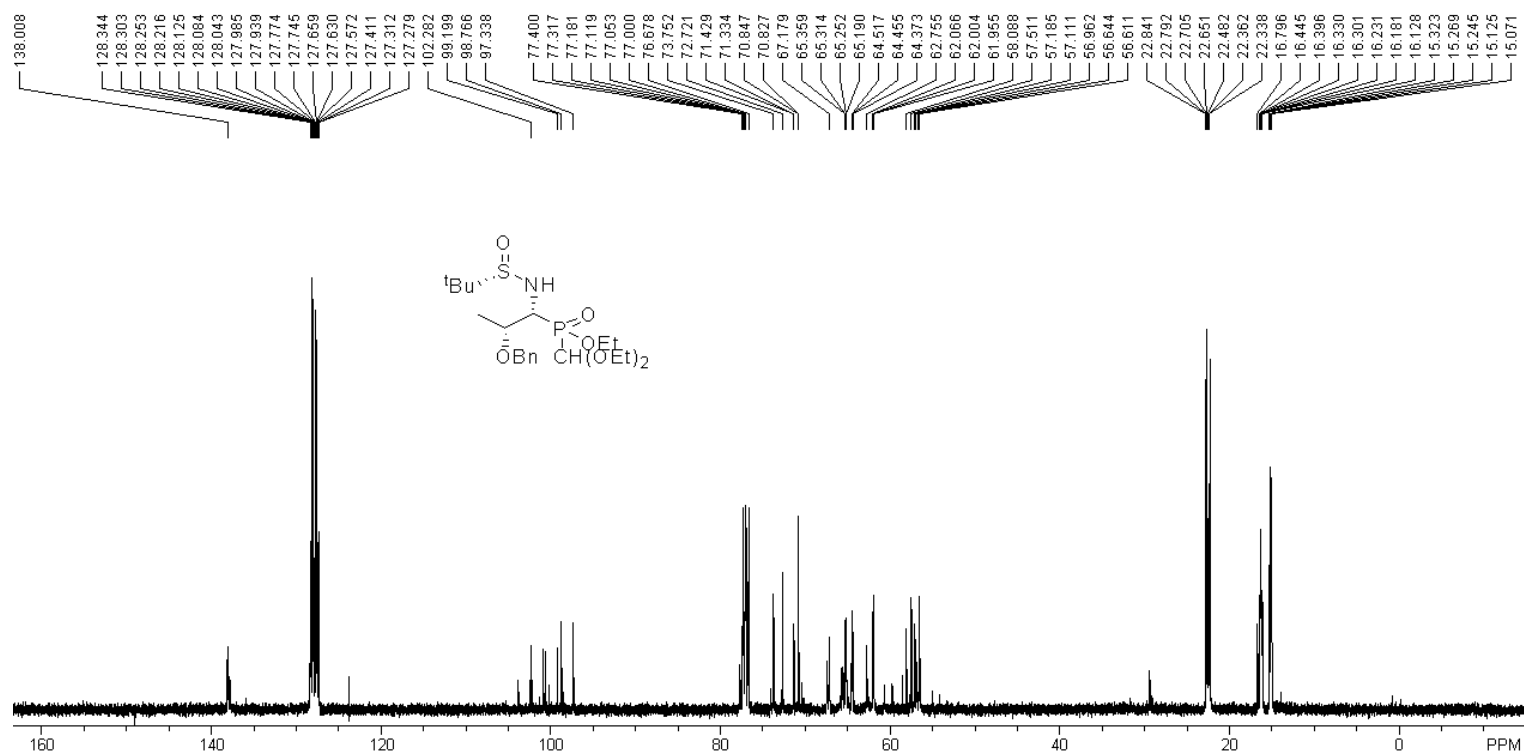
C = 0.00



Compound 2m:

¹H NMR:





³¹P NMR:

D:\software\nuts\DATA\20081109-9-90c_11.1

21:04:31.984+0800 nmri@EP.ZH107708

USER: nmri

SOLVENT: CDCl3

Experim: --

Pulse le: --

Recycle: --

NA: --

Solvent: --

PTS1d: --

F1: 1

F2: --

SW1: --

AT1: --

Hz per f: --

SW2: --

Hz per f: --

O1: --

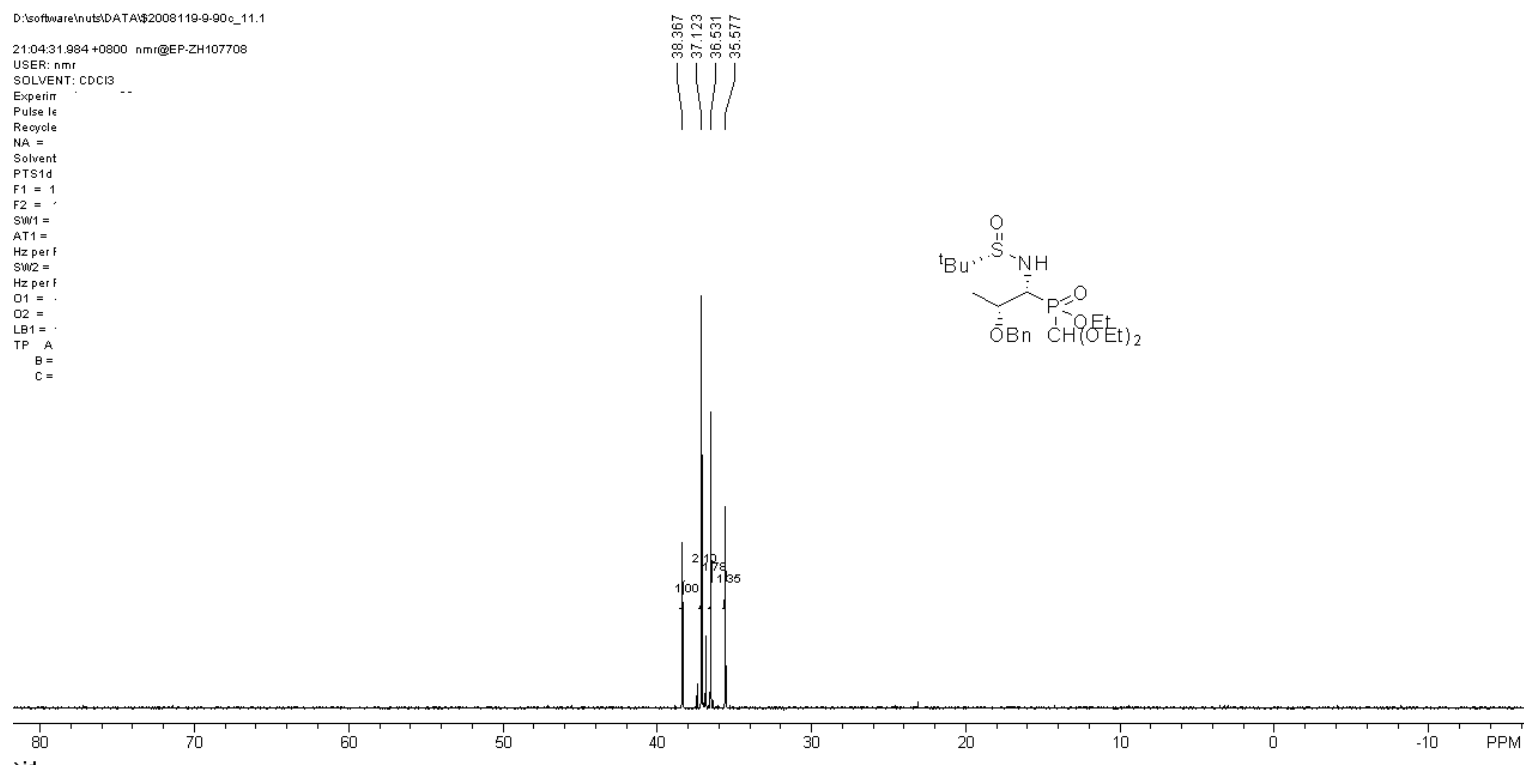
O2: --

LB1: --

TP: A

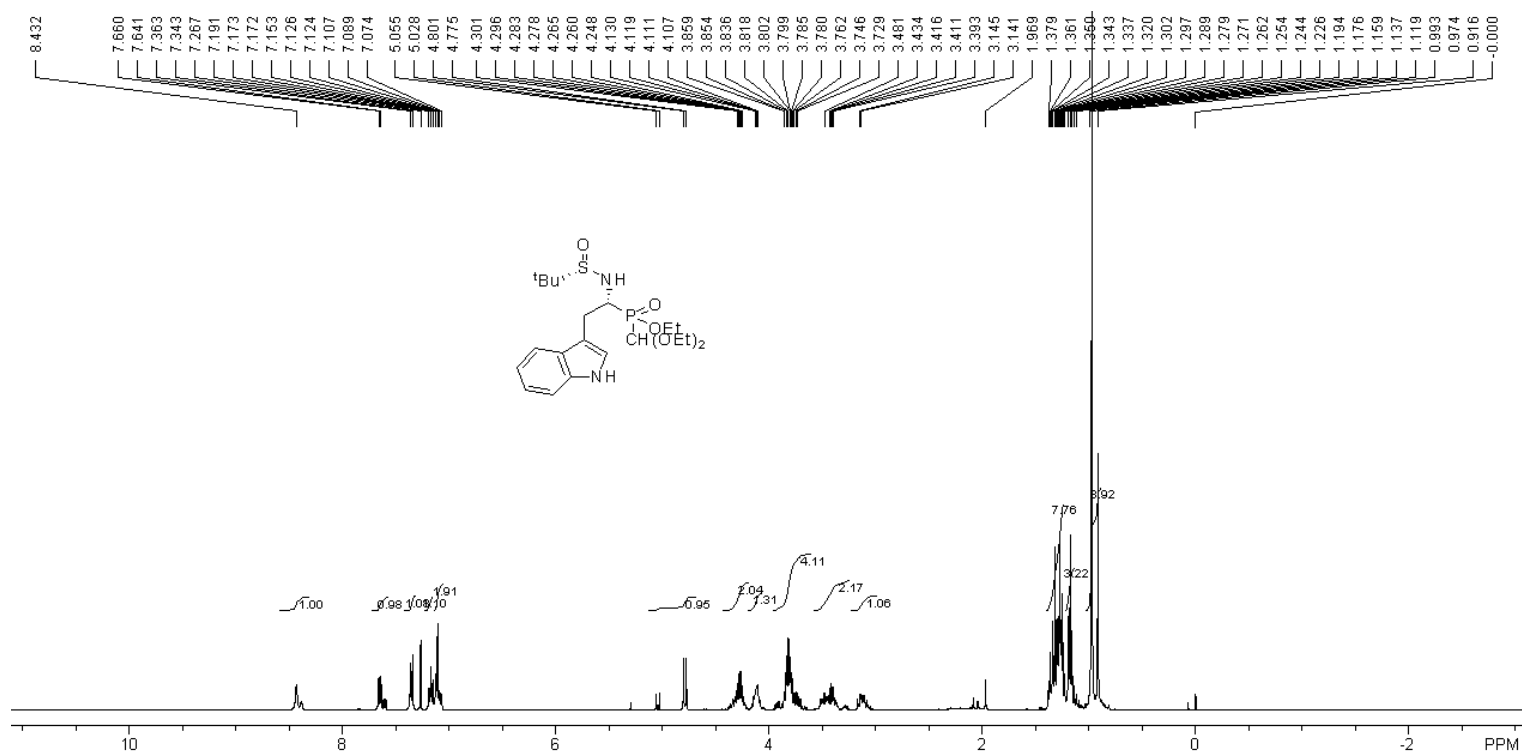
B: --

C: --

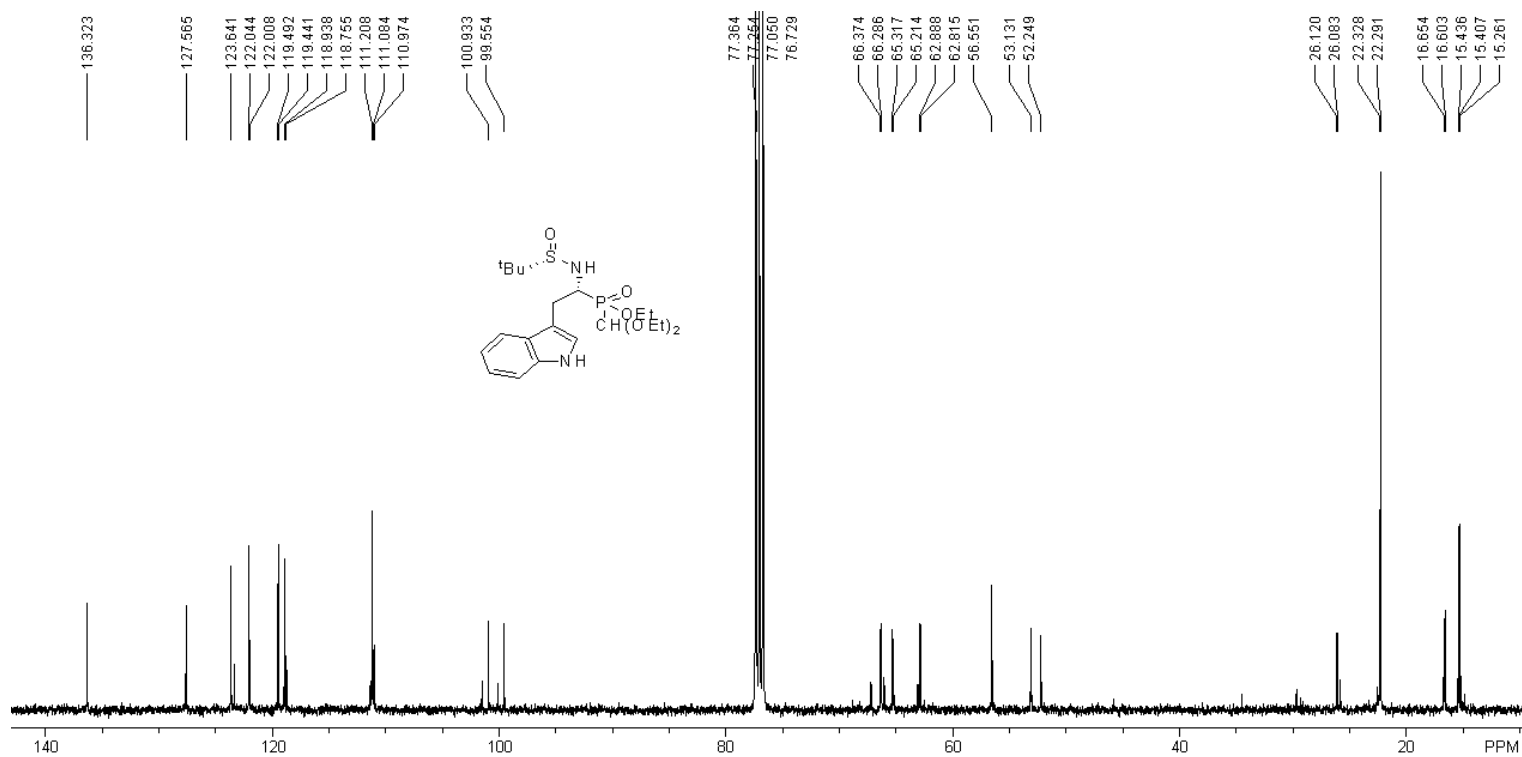


Compound 2n:

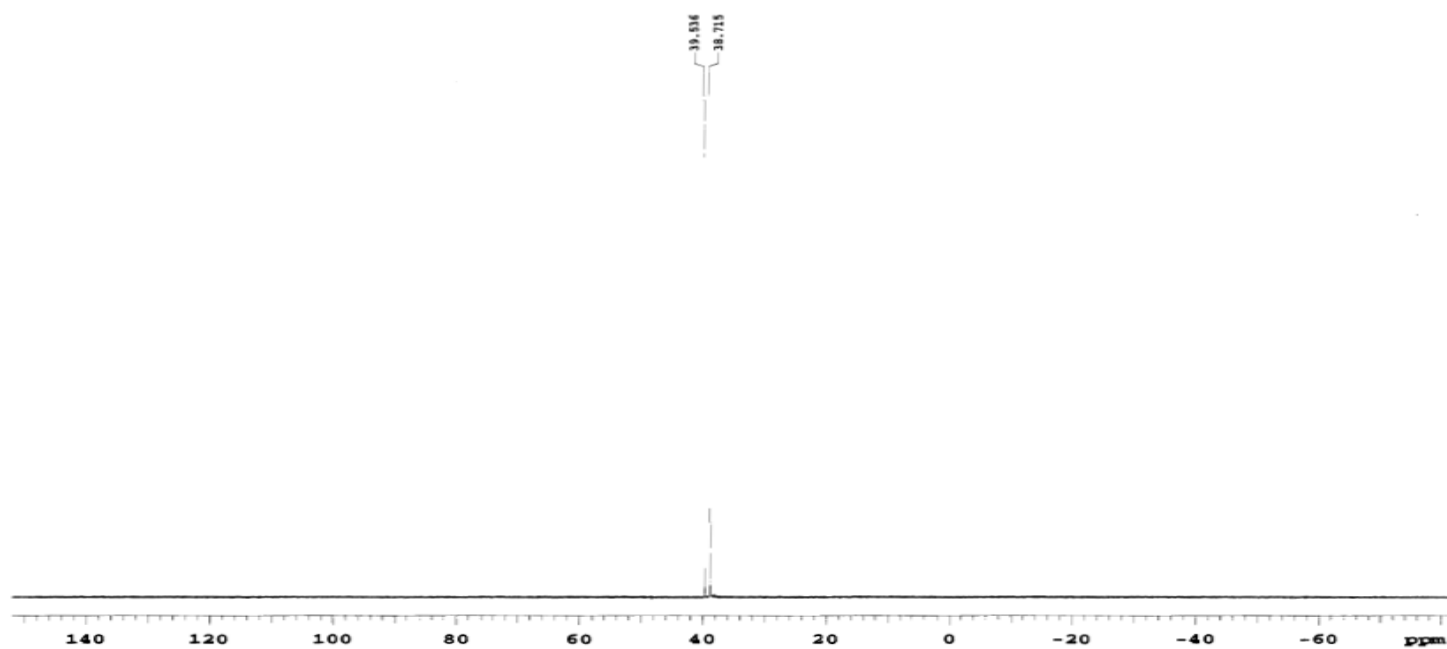
¹H NMR:



¹³C NMR:

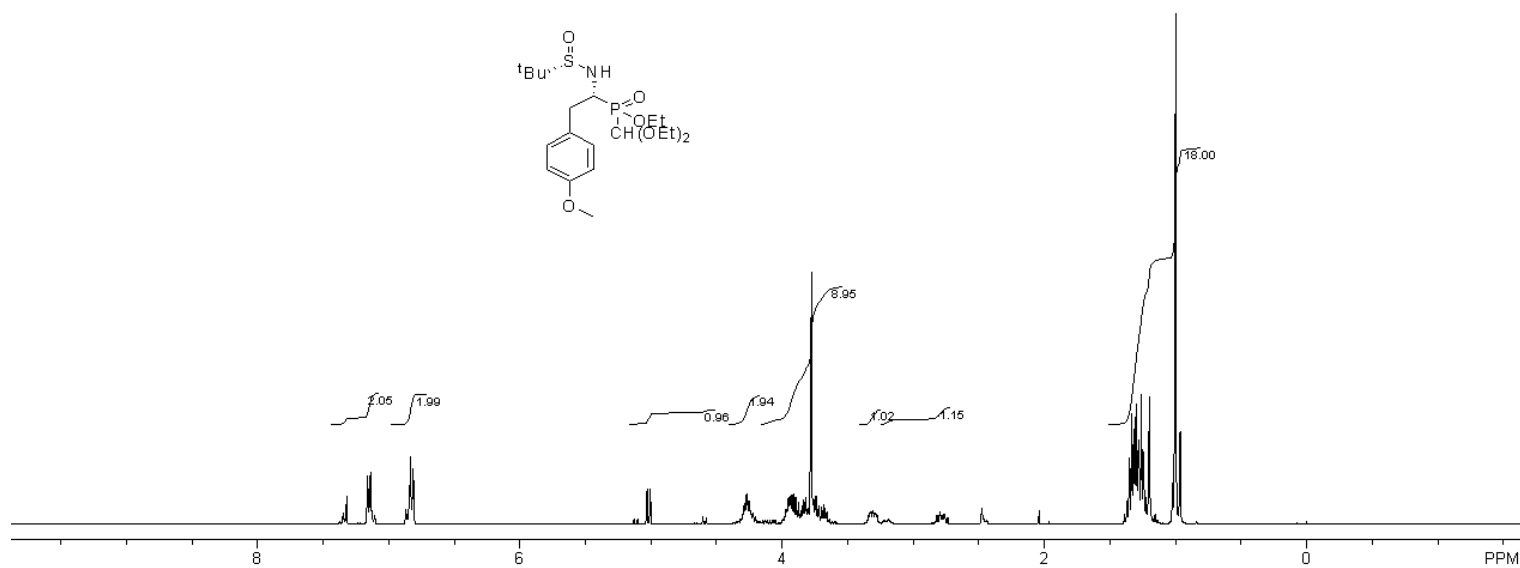
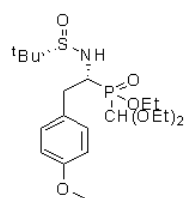
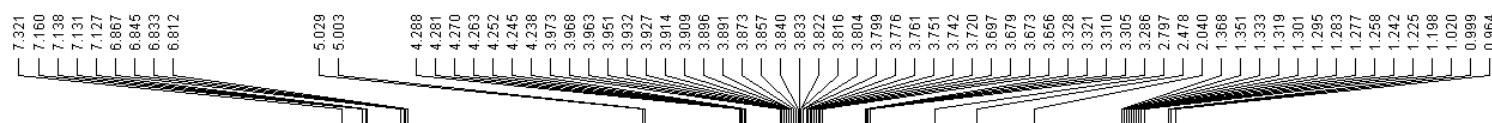


³¹P NMR:

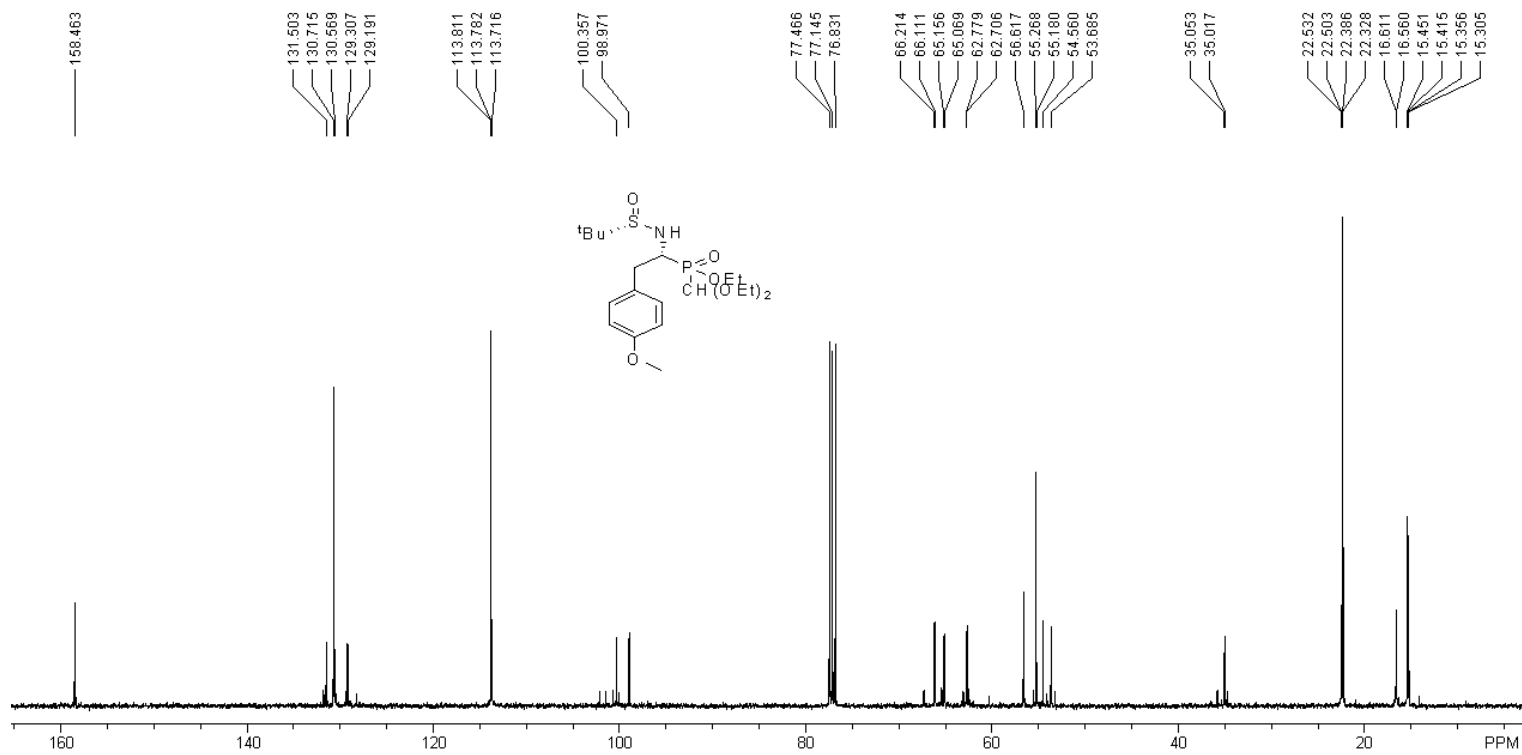


Compound 2o:

¹H NMR:

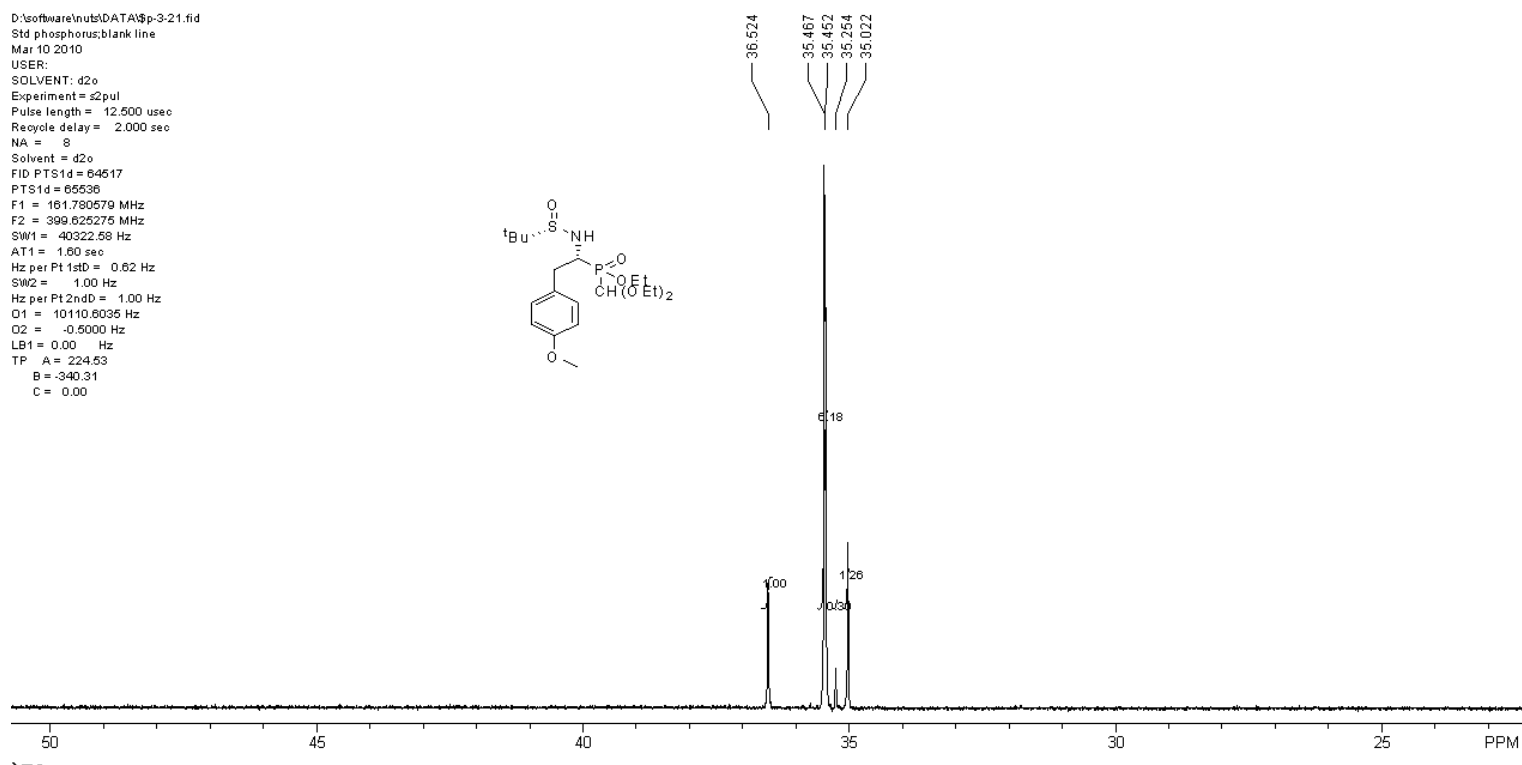


¹³C NMR:



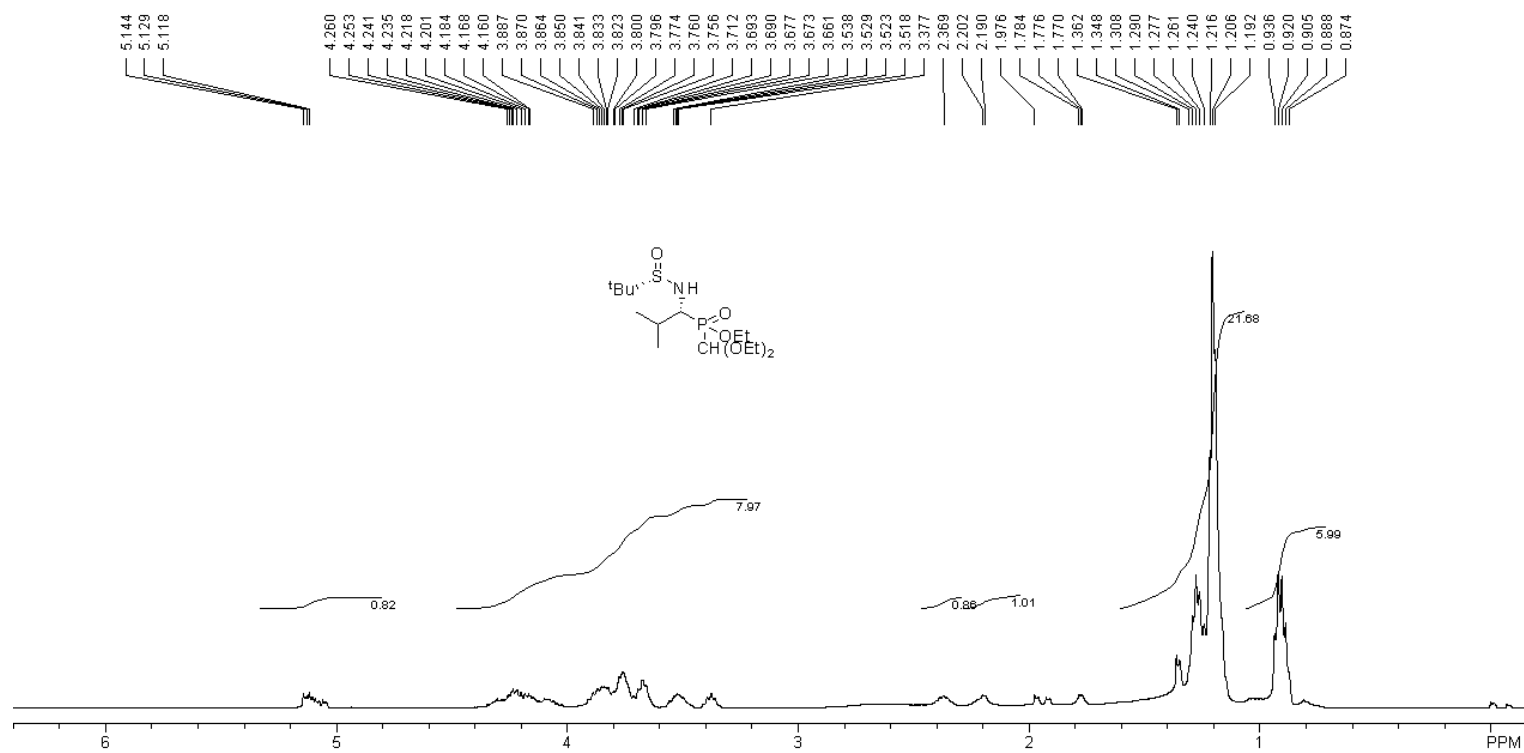
³¹P NMR:

D:\software\nuts\DATA\p-3-21.fid
 Std phosphorus; blank line
 Mar 10 2010
 USER:
 SOLVENT: d2o
 Experiment = s2pul
 Pulse length = 12.500 usec
 Recycle delay = 2.000 sec
 NA = 8
 Solvent = d2o
 FID PTS1d = 64617
 PTS1d = 65536
 F1 = 161.780679 MHz
 F2 = 399.625275 MHz
 SW1 = 40322.58 Hz
 AT1 = 1.60 sec
 Hz per Pt1s0 = 0.62 Hz
 SW2 = 1.00 Hz
 Hz per Pt2nd0 = 1.00 Hz
 Q1 = 10110.8035 Hz
 Q2 = -0.5000 Hz
 LB1 = 0.00 Hz
 TP A = 224.63
 B = -340.31
 C = 0.00

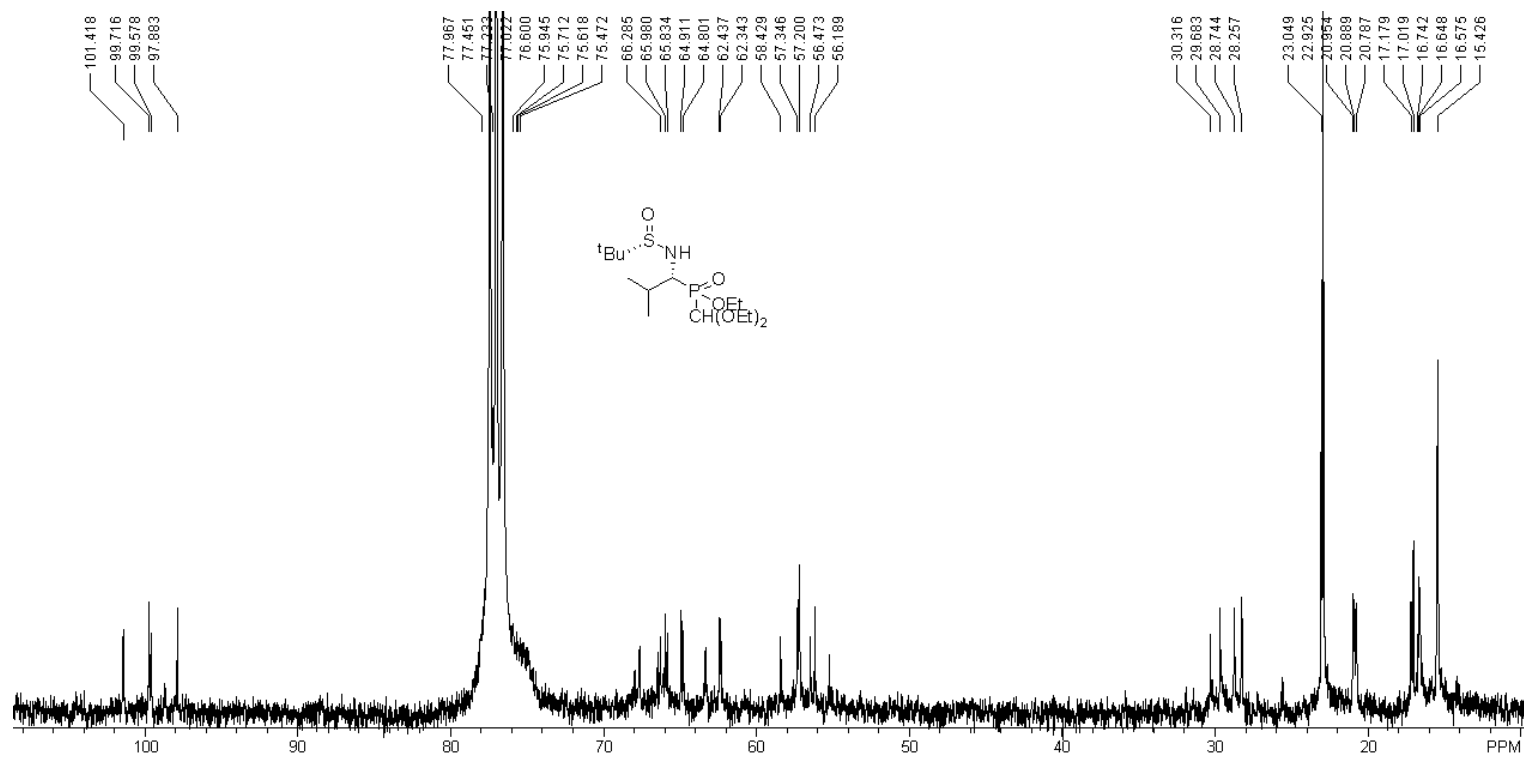


Compound 2p:

¹H NMR:

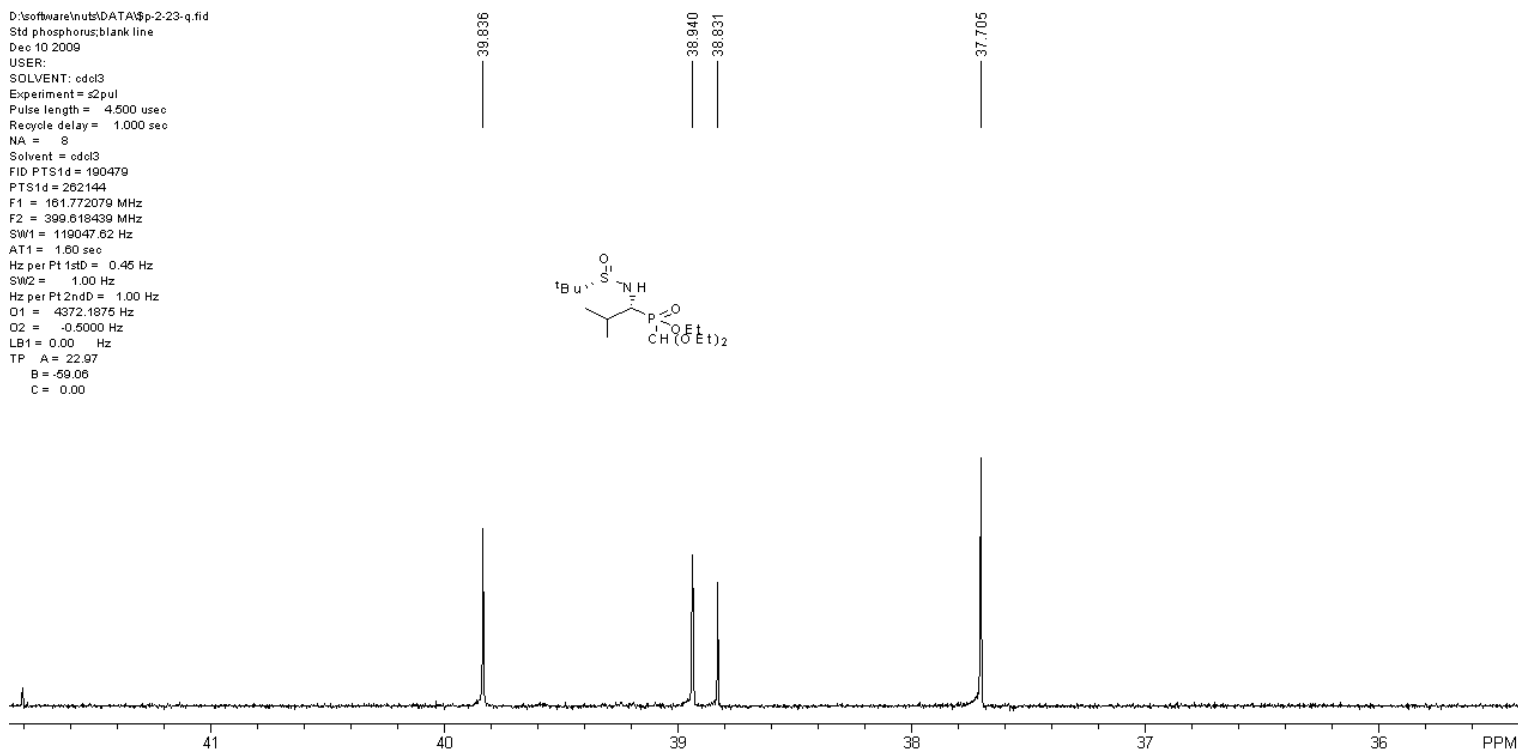
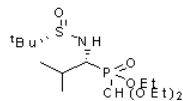


¹³C NMR:



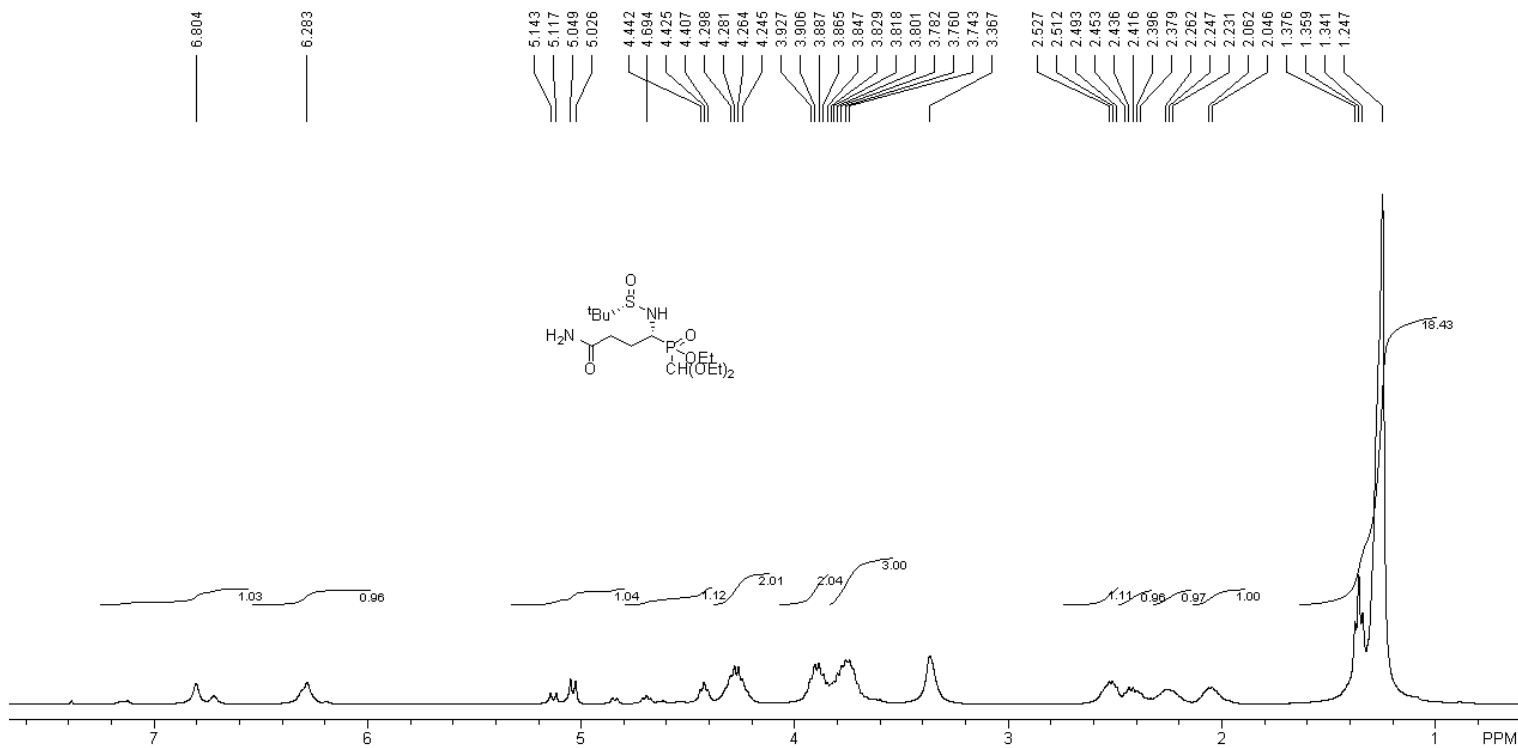
³¹P NMR:

D:\software\nuts\DATA\p-2-23-q.fid
 Std phosphorus;blank line
 Dec 10 2009
 USER:
 SOLVENT: cdcl3
 Experiment = s2pul
 Pulse length = 4.500 usec
 Recycle delay = 1.000 sec
 NA = 8
 Solvent = cdcl3
 FID PTS1d = 190479
 PTS1d = 262144
 F1 = 161.772079 MHz
 F2 = 399.618439 MHz
 SW1 = 119047.62 Hz
 AT1 = 1.60 sec
 Hz per Pt1st0 = 0.45 Hz
 SW2 = 1.00 Hz
 Hz per Pt2nd0 = 1.00 Hz
 O1 = 4372.1875 Hz
 O2 = -0.5000 Hz
 LB1 = 0.00 Hz
 TP A = 22.97
 B = -59.06
 C = 0.00

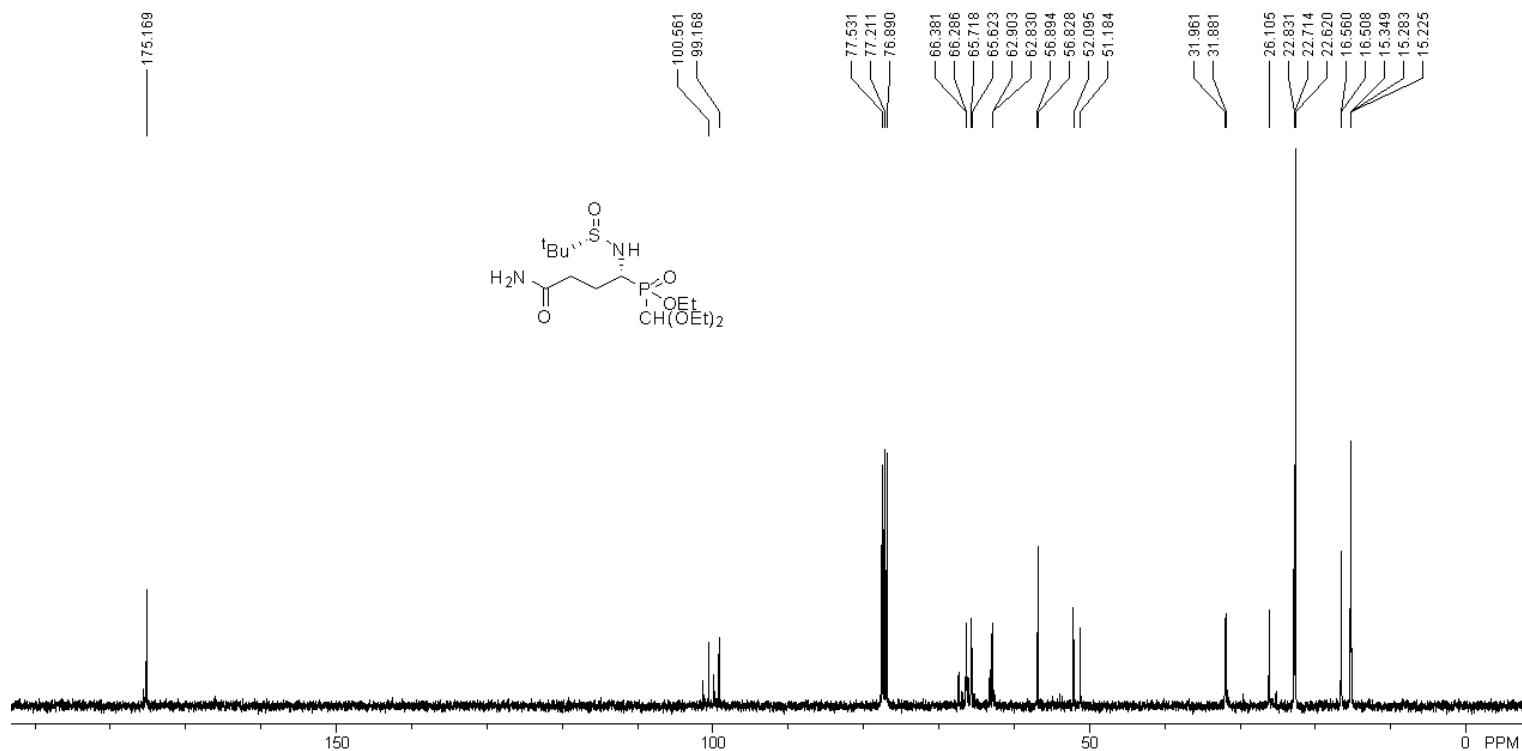


Compound 2q:

¹H NMR:



¹³C NMR:



³¹P NMR:

D:\software\nuts\DATA\补充数据_8-28-P.1

20:08:33.125 +0800 nmri@EP.ZH107708

USER: nmri

SOLVENT: CDCl₃

Experiment = zgpg30

Pulse length = 8.300 usec

Recycle delay = 2.000 sec

NA = 8

Solvent = CDCl₃

PTSD = 32768

F1 = 161.971542 MHz

F2 = 1.000000 MHz

SWH = 64102.56 Hz

AT1 = 0.51 sec

Hz per Pt1sd = 1.96 Hz

SW2 = 1.00 Hz

Hz per Pt2nd = 1.00 Hz

O1 = -8098.6865 Hz

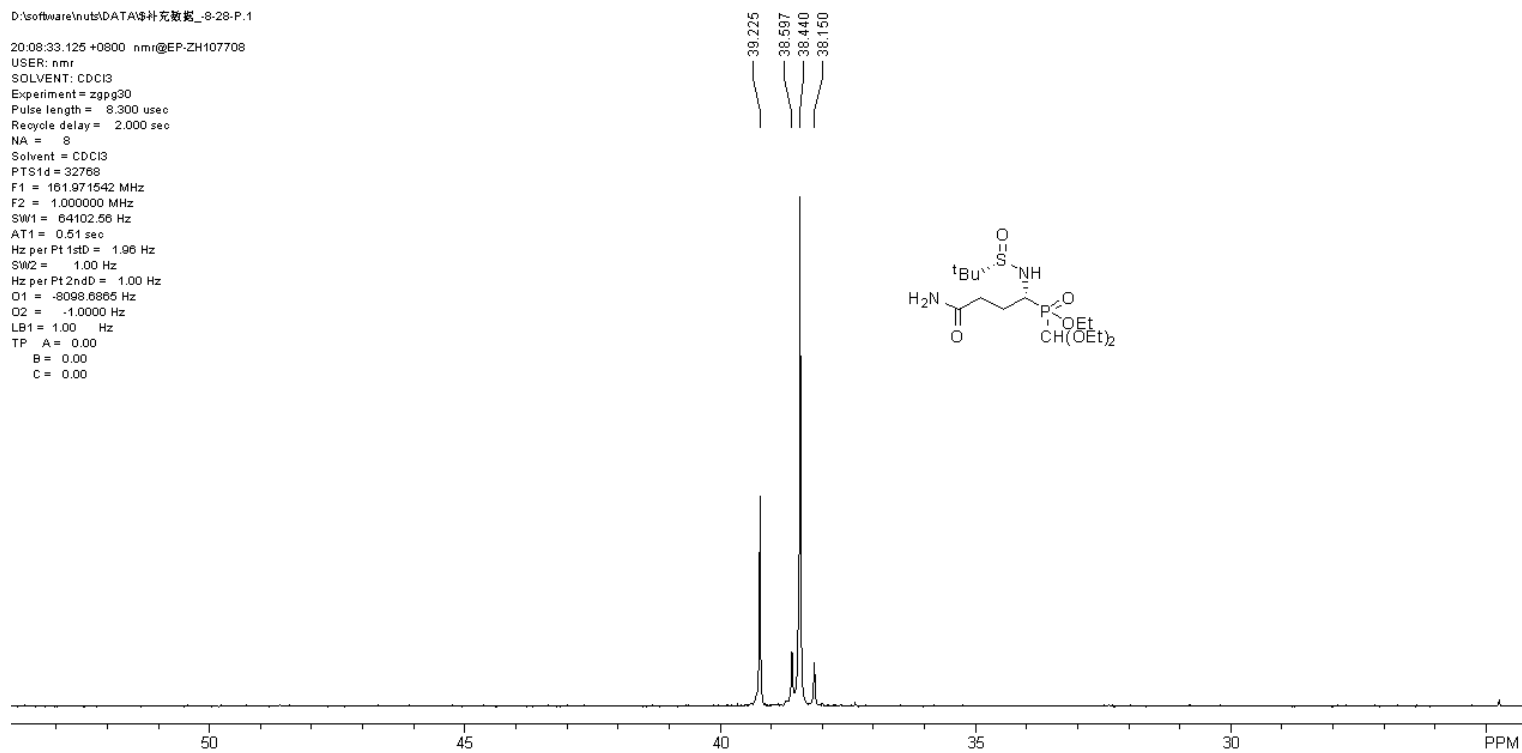
O2 = -1.0000 Hz

LB1 = 1.00 Hz

TP A = 0.00

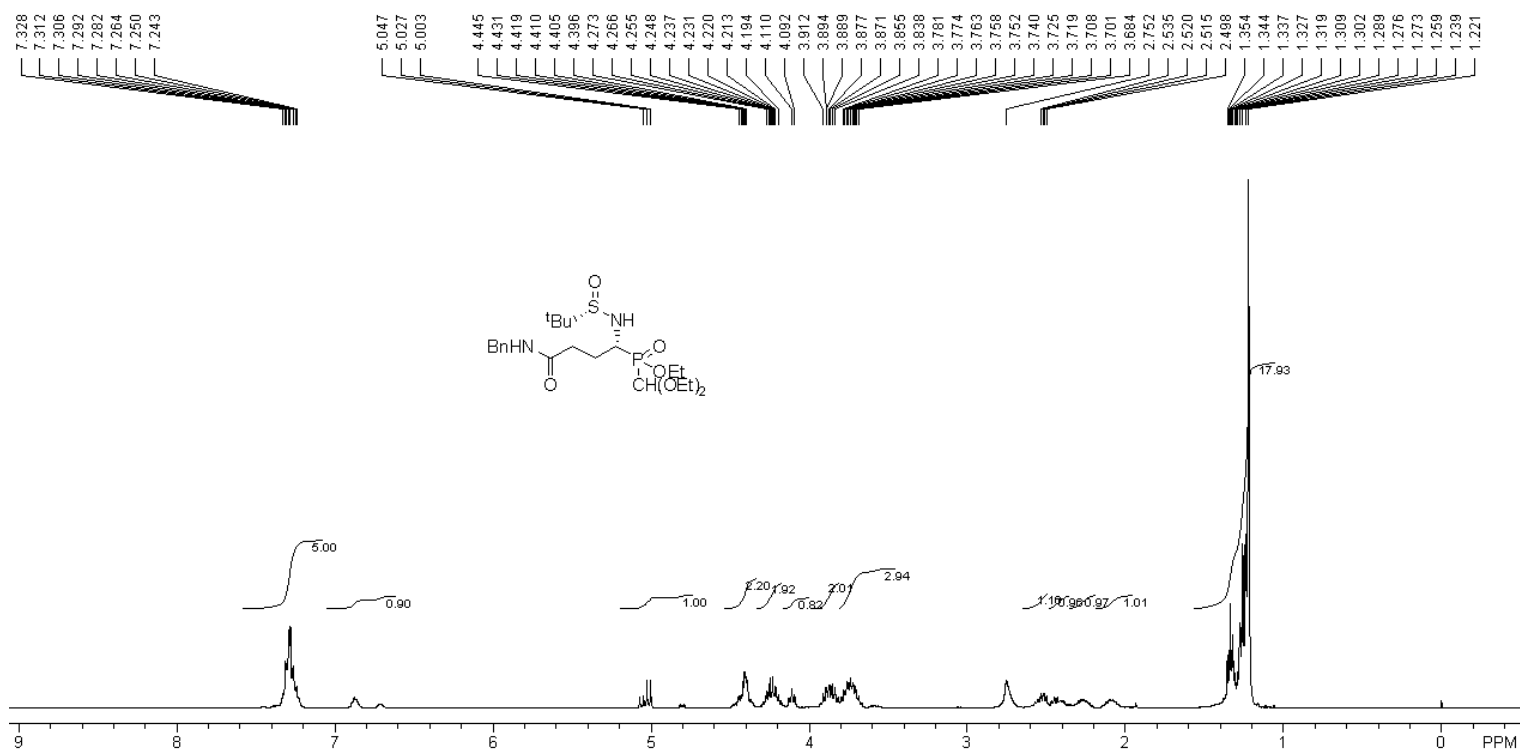
B = 0.00

C = 0.00

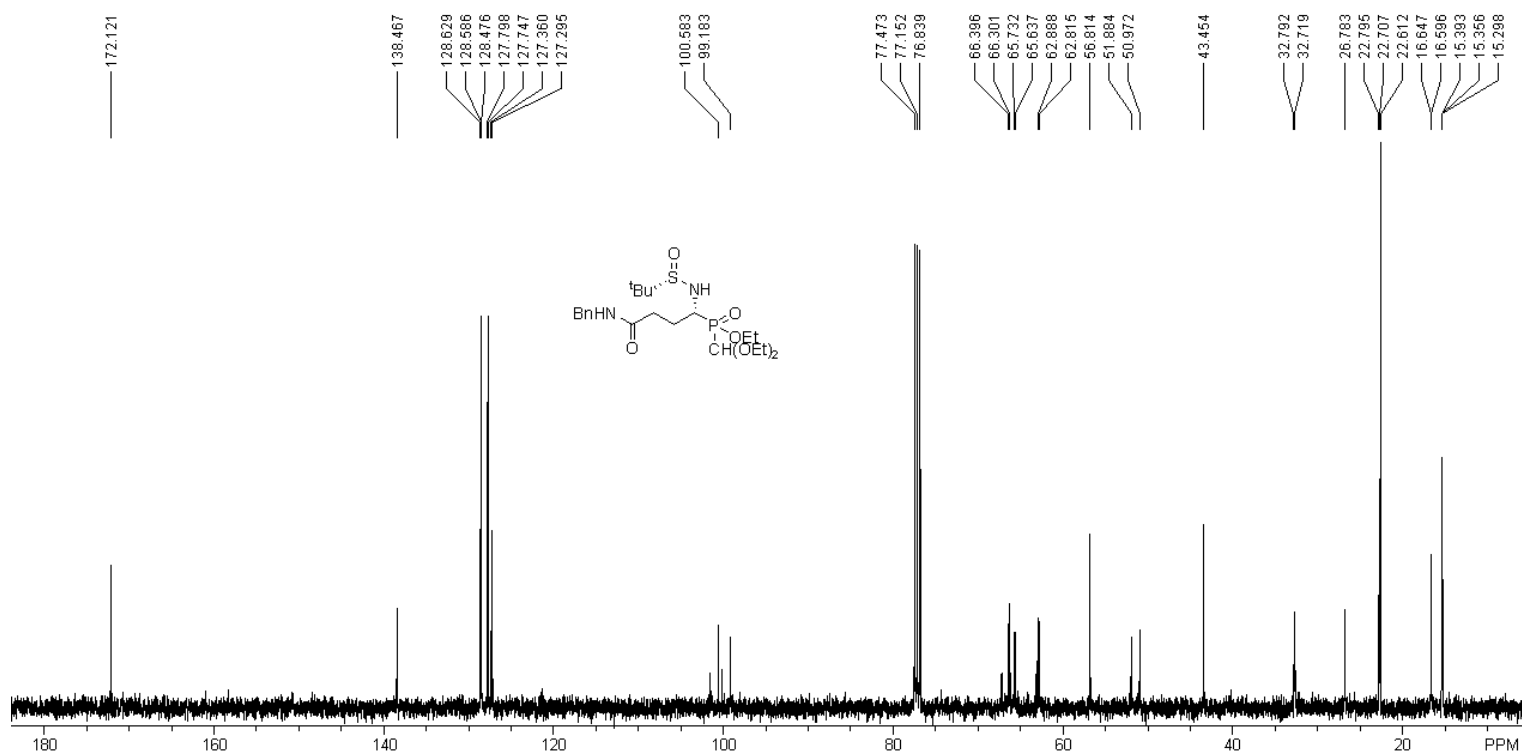


Compound 2r:

¹H NMR:



¹³C NMR:



³¹P NMR:

D:\software\nuts\DATA\峰补充数据_8-22-PNMR.1

20:29:25.875 +0800 nmr@EP-ZH107708

USER: nmr

SOLVENT: CDCl3

Experiment = zgpg30

Pulse length = 8.300 usec

Recycle delay = 2.000 sec

NA = 8

Solvent = CDCl3

PTS1d = 32768

F1 = 161.971542 MHz

F2 = 1.000000 MHz

SW1 = 64102.56 Hz

AT1 = 0.51 sec

Hz per Pt1stD = 1.96 Hz

SW2 = 1.00 Hz

Hz per Pt2ndD = 1.00 Hz

O1 = -8098.6865 Hz

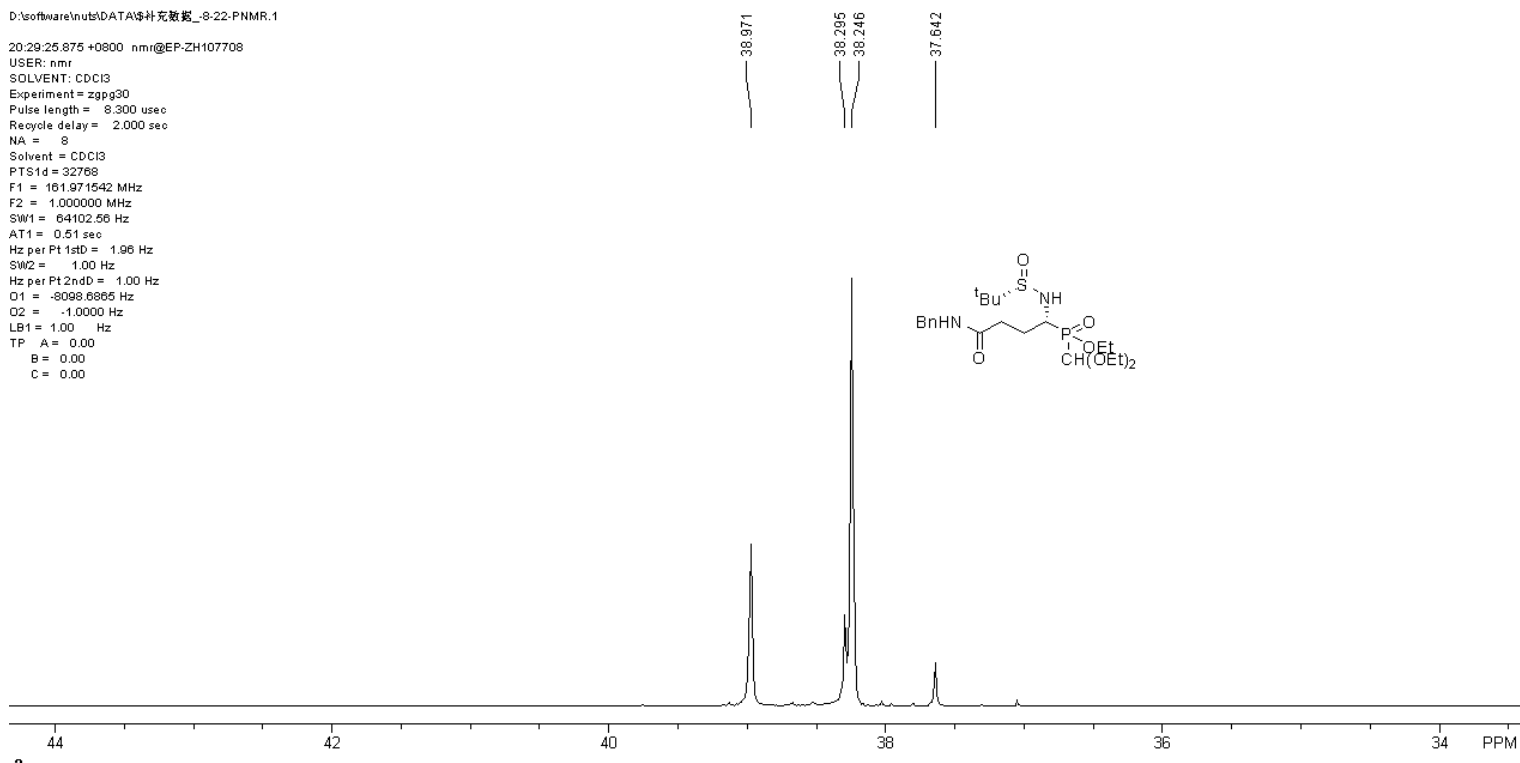
O2 = -1.0000 Hz

LB1 = 1.00 Hz

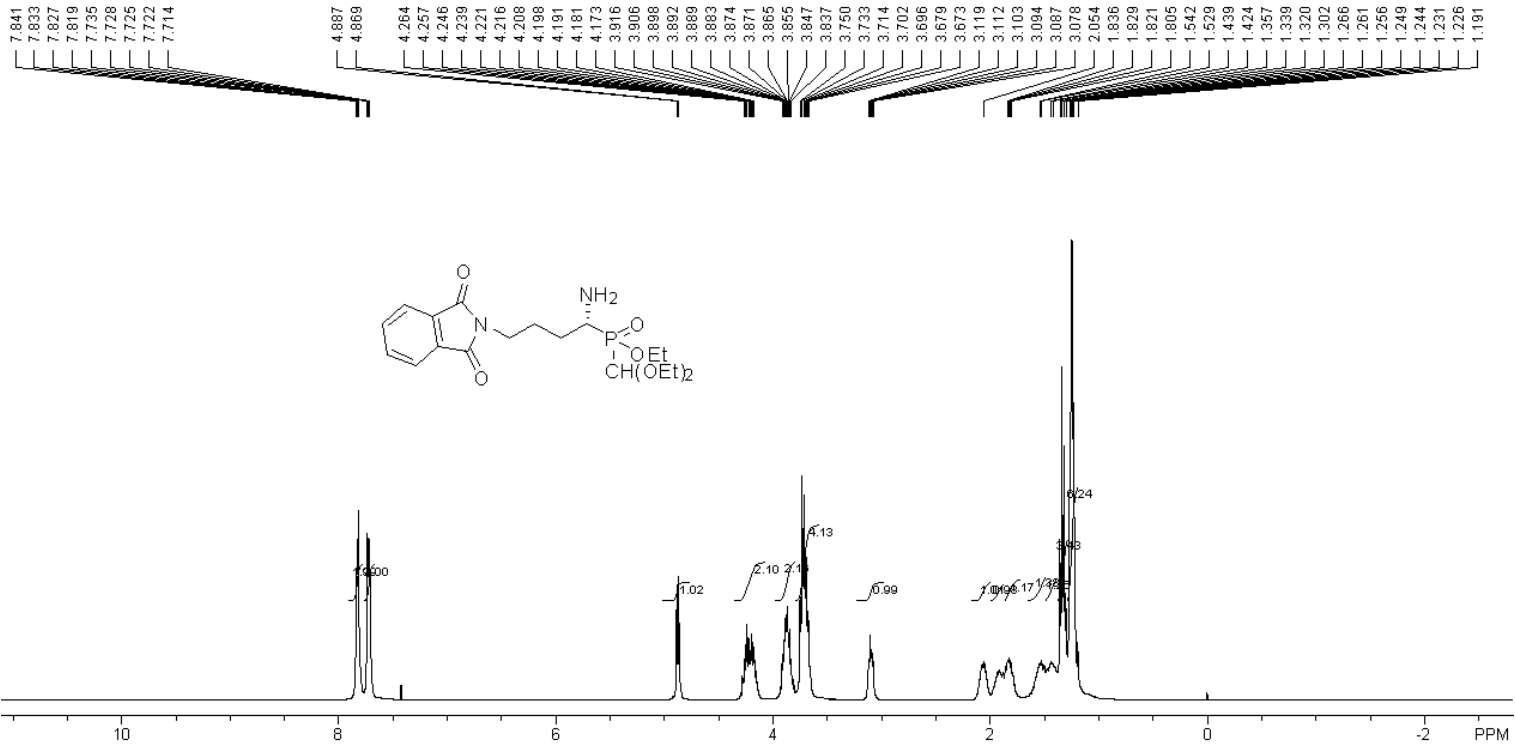
TP A = 0.00

B = 0.00

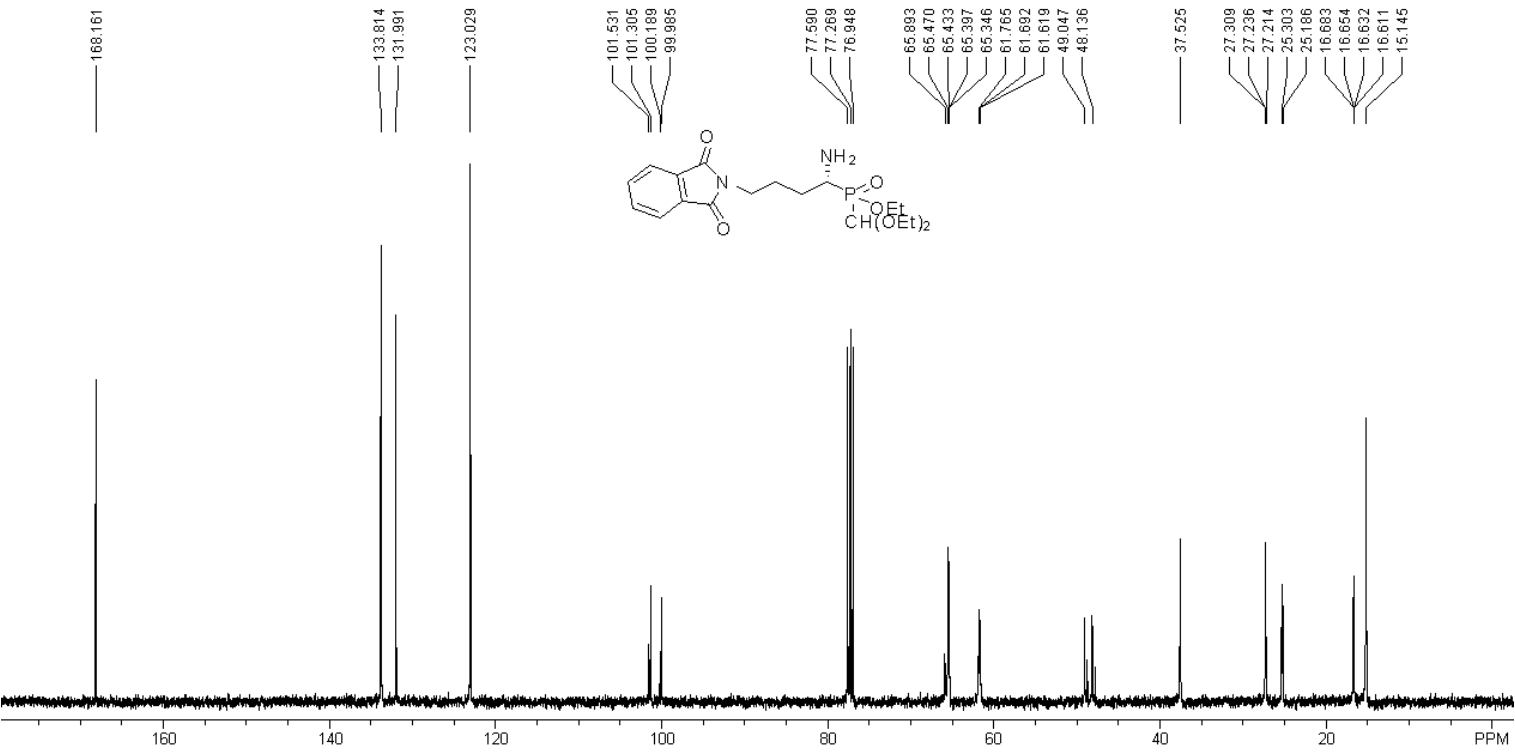
C = 0.00



Compound 3b:
H NMR:



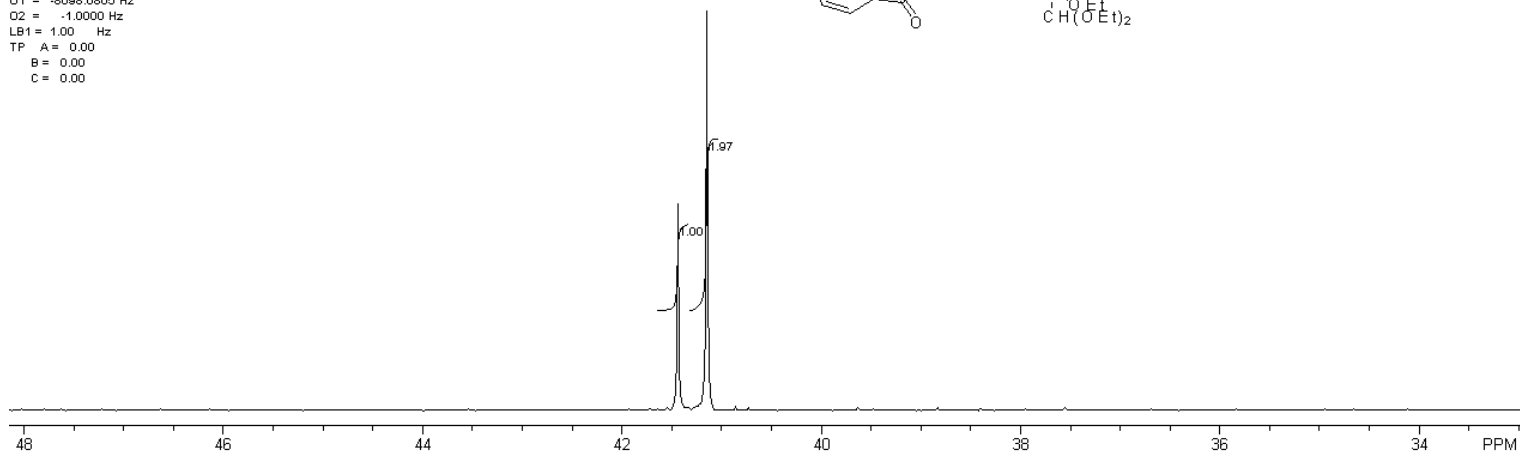
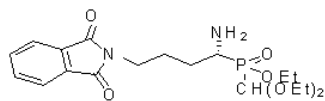
¹³C NMR:



³¹P NMR:

D:\software\inputs\DATA\谱图\7-66-1.1
 15:34:09.250 +0800 nmr@EP-ZH107708
 USER: nmr
 SOLVENT: CDCl3
 Experiment = zgpg30
 Pulse length = 8.300 usec
 Recycle delay = 2.000 sec
 NA = 4
 Solvent = CDCl3
 P1 = 32768
 F1 = 161.971542 MHz
 F2 = 100.000000 MHz
 SW1 = 64102.56 Hz
 AT1 = 0.51 sec
 Hz per Pt1stD = 1.06 Hz
 SW2 = 1.00 Hz
 Hz per Pt2ndD = 1.00 Hz
 O1 = -8098.6865 Hz
 O2 = -1.0000 Hz
 LB1 = 1.00 Hz
 TP A = 0.00
 B = 0.00
 C = 0.00

41.435
 41.145

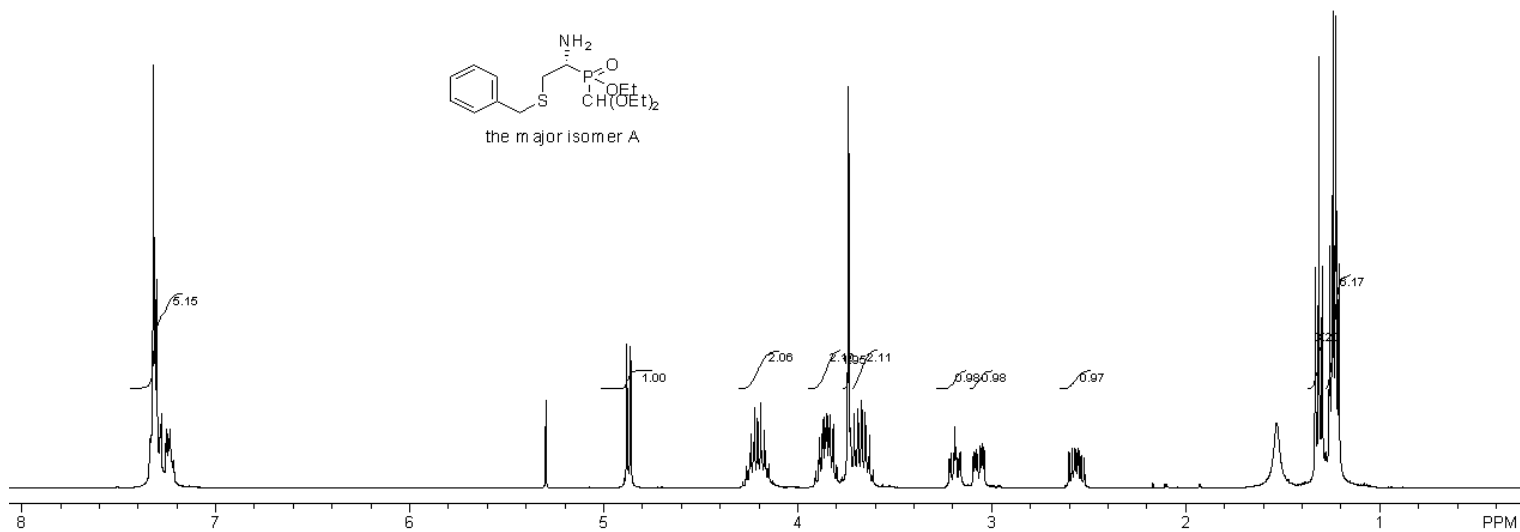
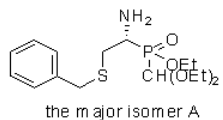


Compound 3c:

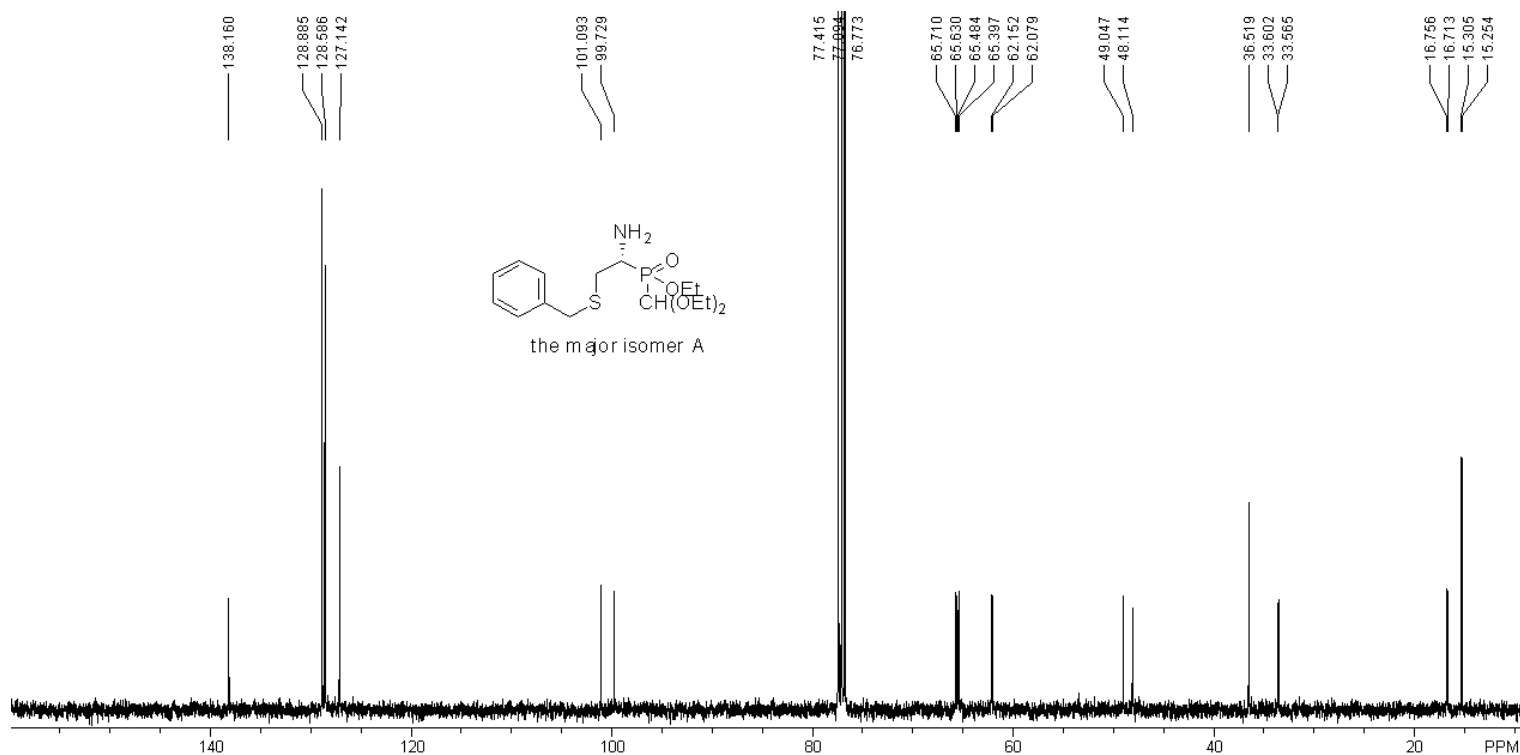
¹H NMR:

7.336
 7.321
 7.317
 7.305
 7.289
 7.285
 7.280
 7.257
 7.251
 7.243
 7.235

4.880
 4.862
 5.298
 4.240
 4.222
 4.208
 4.204
 4.190
 4.173
 4.165
 3.889
 3.872
 3.866
 3.858
 3.852
 3.849
 3.840
 3.835
 3.817
 3.739
 3.727
 3.709
 3.704
 3.691
 3.686
 3.670
 3.653
 3.647
 3.630
 3.189
 3.182
 3.080
 3.060
 3.052
 3.046
 3.038
 2.985
 2.573
 2.566
 2.556
 2.551
 1.530
 1.331
 1.314
 1.296
 1.257
 1.245
 1.240
 1.227
 1.222
 1.210



¹³C NMR:



³¹P NMR:

D:\software\inputs\DATA\补充数据_10-12A-P.1

10-06-22.828 +0800 nmr@EP-ZH107708

USER: nmr

SOLVENT: CDCl3

Experiment = zgpg30

Pulse length = 8.300 usec

Recycle delay = 2.000 sec

NA = 8

Solvent = CDCl3

PTS1d = 32768

F1 = 161.971542 MHz

F2 = 1.000000 MHz

SW1 = 64102.56 Hz

AT1 = 0.51 sec

Hz per Pt1stD = 1.96 Hz

SW2 = 1.00 Hz

Hz per Pt2ndD = 1.00 Hz

O1 = -8098.6865 Hz

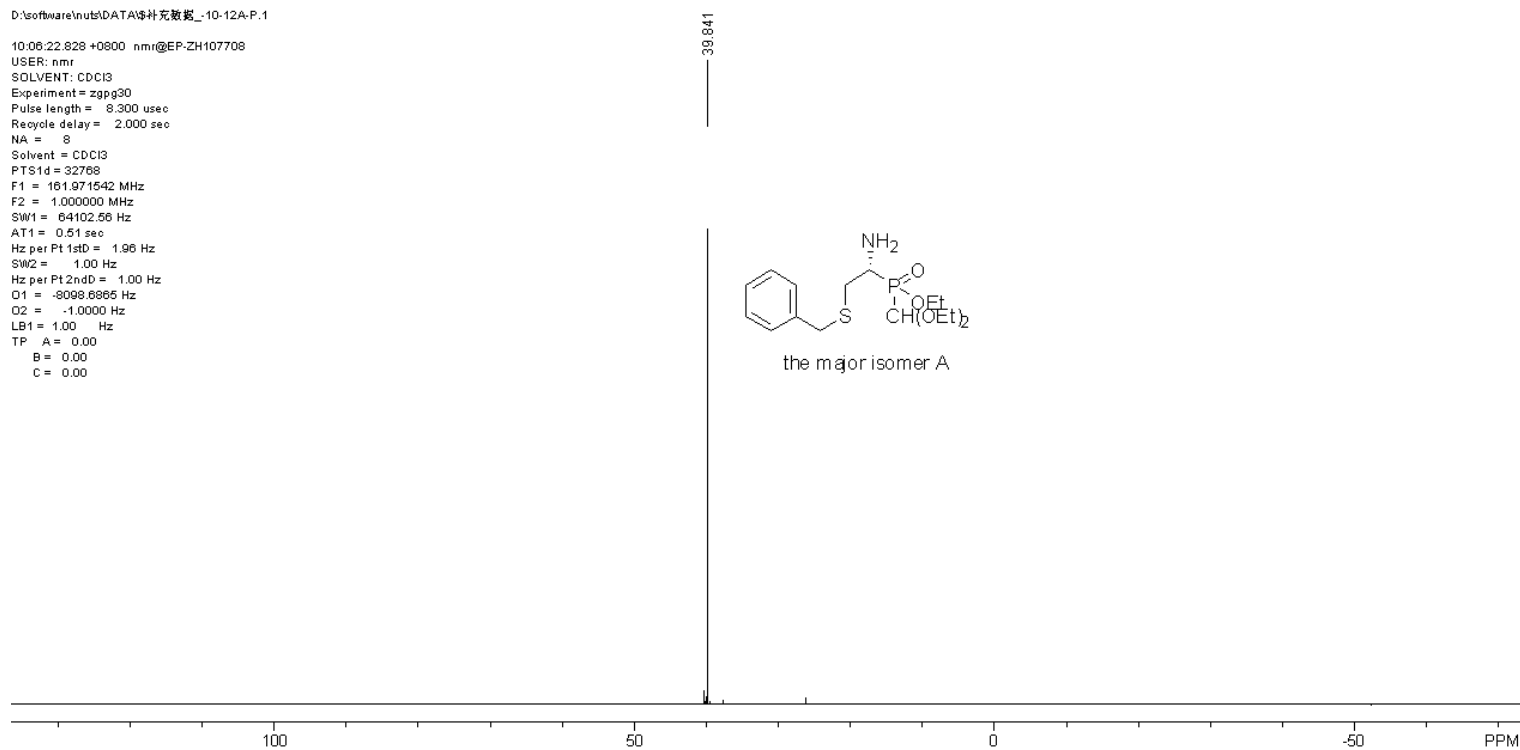
O2 = -1.0000 Hz

LB1 = 1.00 Hz

TP A = 0.00

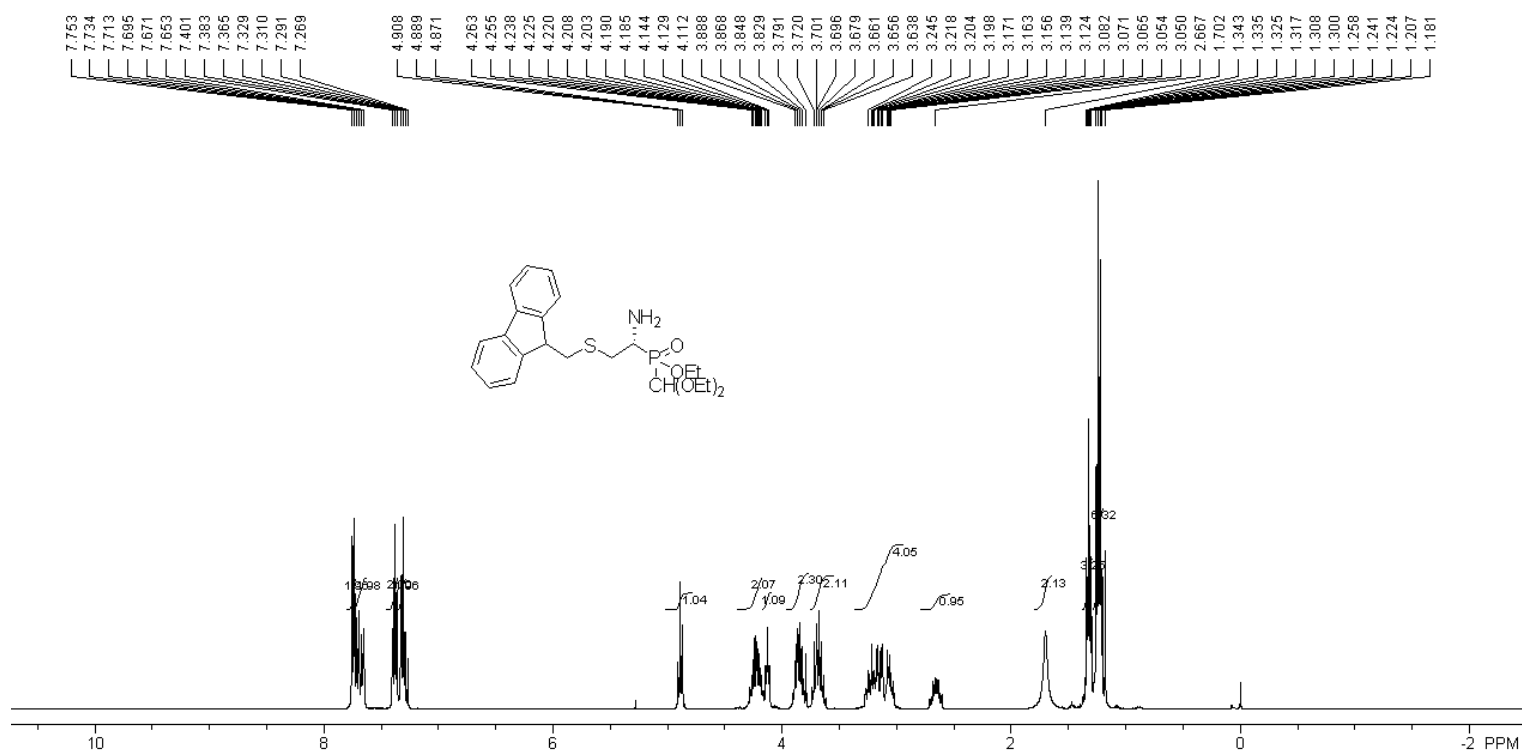
B = 0.00

C = 0.00

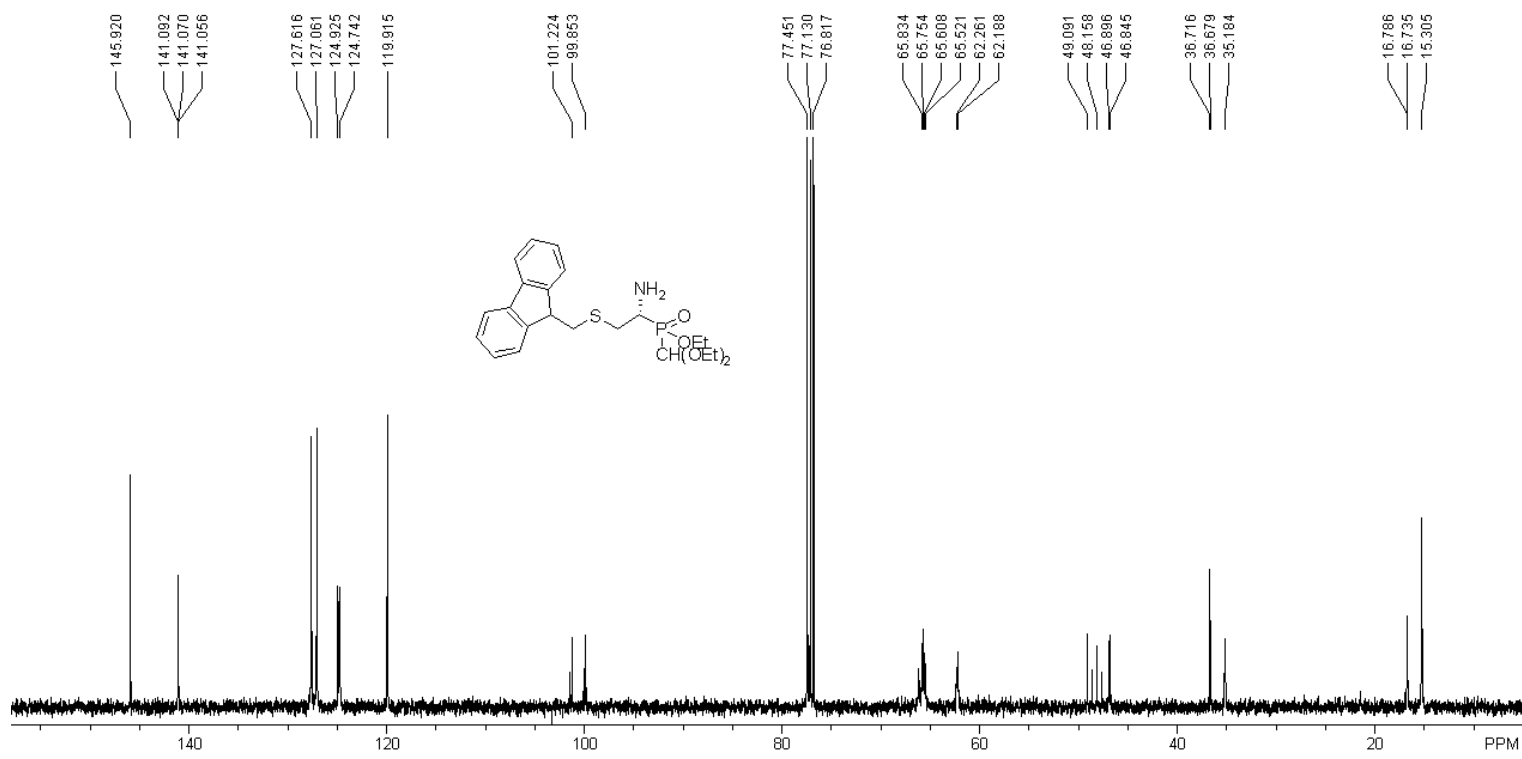


Compound 3d:

¹H NMR:



¹³C NMR:



³¹P NMR:

D:\software\nuts\DATA\2008119-10-96P_10PNMR.1

17:18:15.968 +0800 nmr@EP-ZH107708

USER: nmr

SOLVENT: CDCl3

Experiment: zgpg30

Pulse length = 8.300 usec

Recycle delay = 2.000 sec

NA = 8

Solvent = CDCl3

PTS1d = 32768

F1 = 161.971542 MHz

F2 = 1.000000 MHz

SW1 = 64102.56 Hz

AT1 = 0.51 sec

Hz per Pt1stD = 1.96 Hz

SW2 = 1.00 Hz

Hz per Pt2ndD = 1.00 Hz

O1 = -8098.6865 Hz

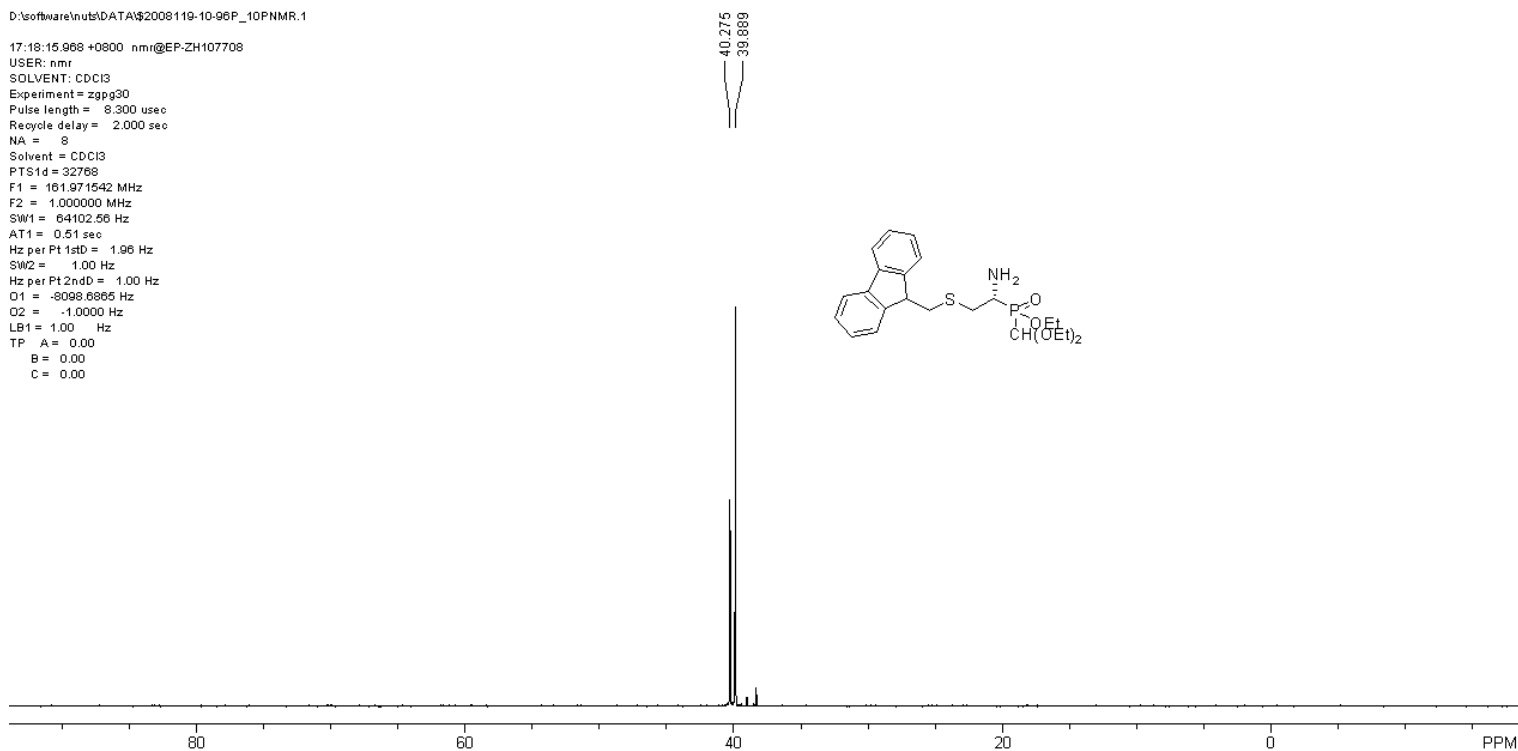
O2 = -1.0000 Hz

LB1 = 1.00 Hz

TP A = 0.00

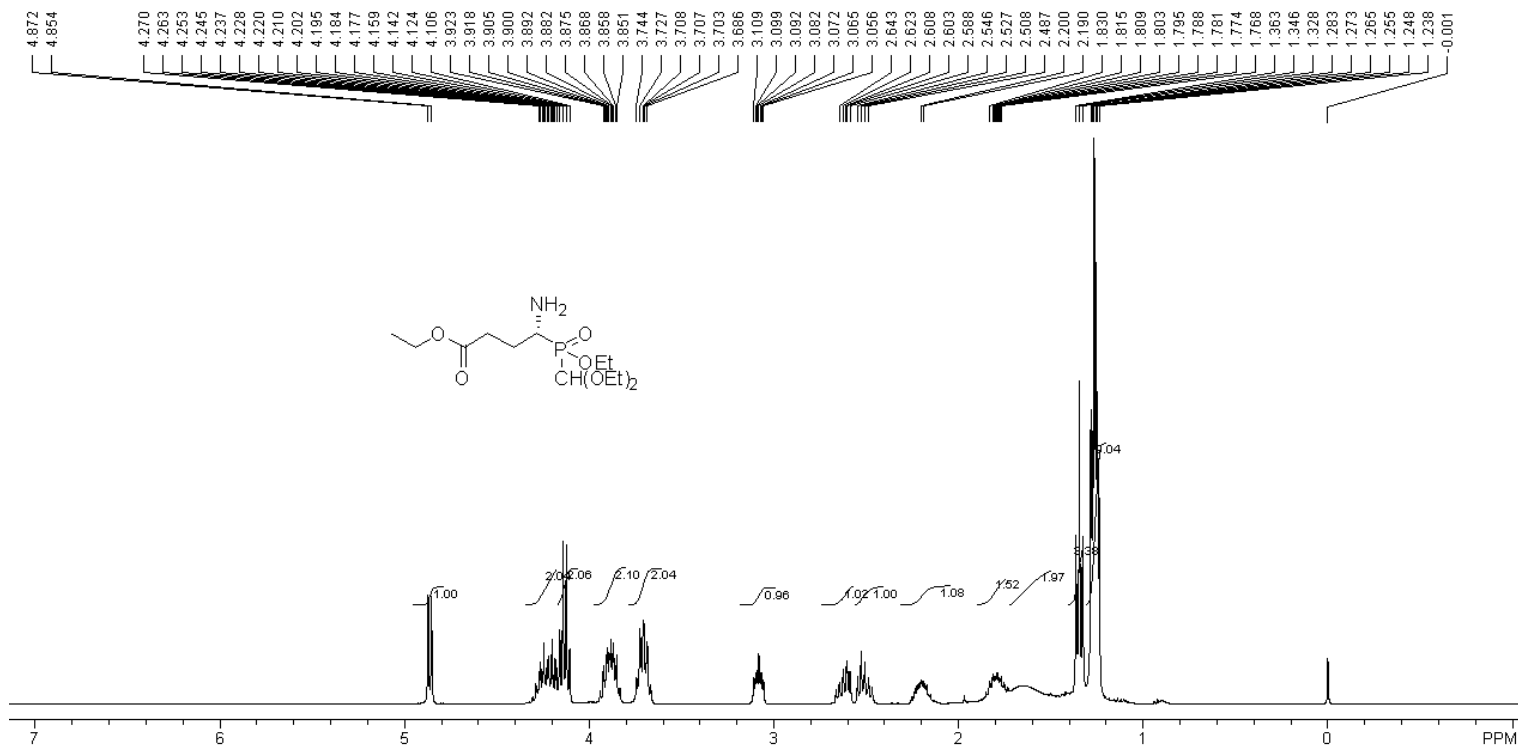
B = 0.00

C = 0.00

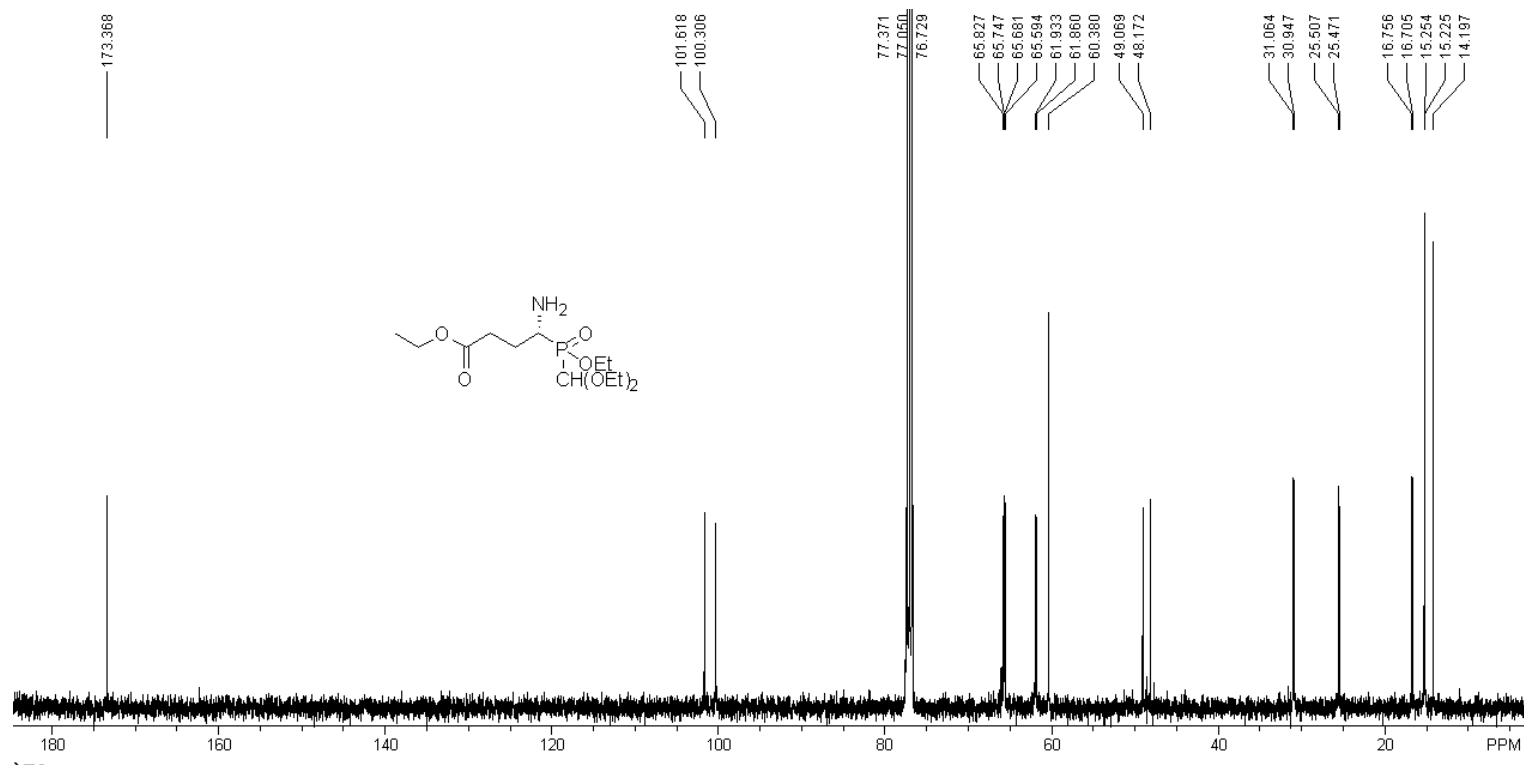


Compound 3e:

¹H NMR:

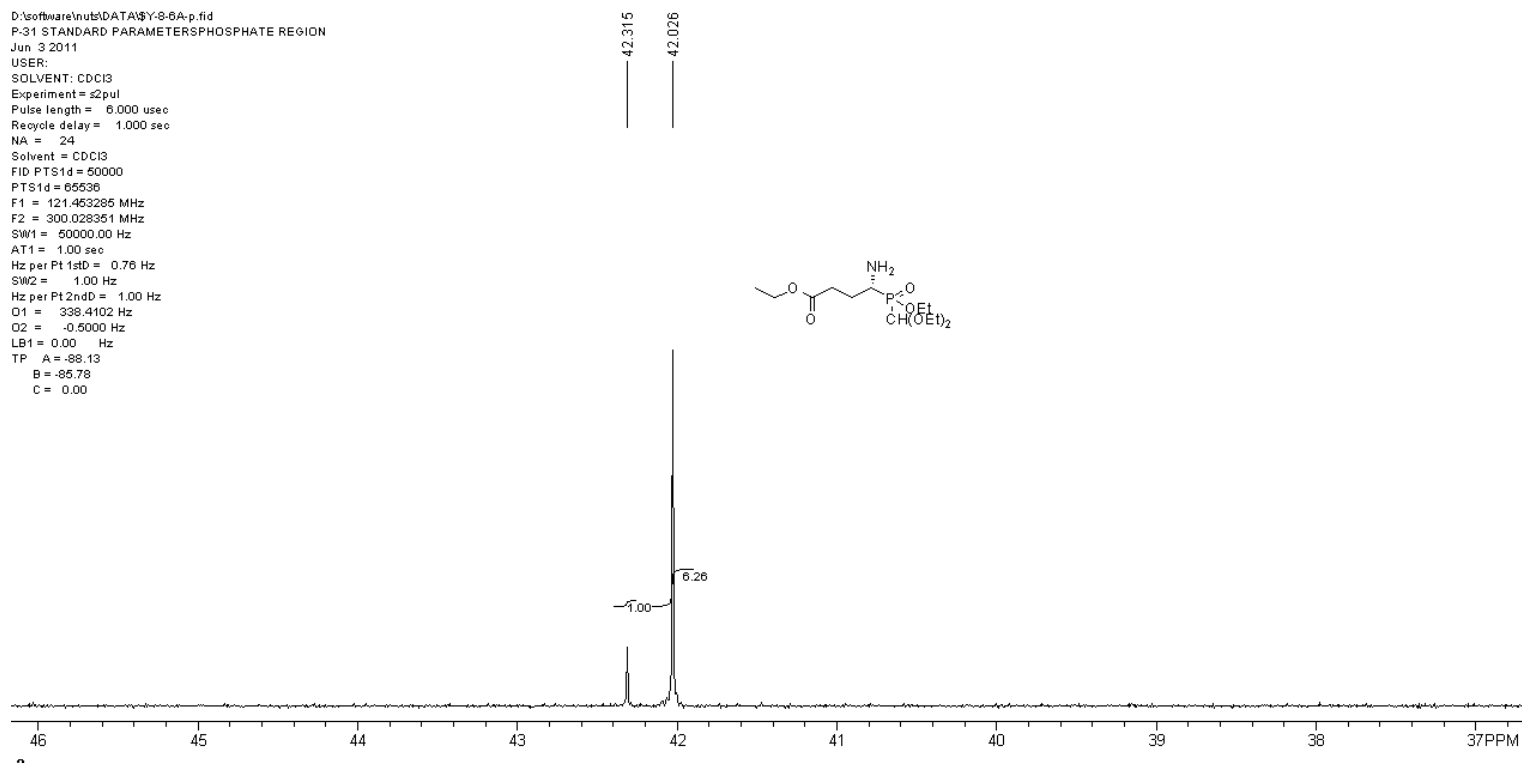


¹³C NMR:



³¹P NMR:

D:\software\nuts\DATA\Y-8-6A-p.fid
P-31 STANDARD PARAMETERSPHOSPHATE REGION
Jun 3 2011
USER:
SOLVENT: CDCl3
Experiment = s2pul
Pulse length = 6.000 usec
Recycle delay = 1.000 sec
NA = 24
Solvent = CDCl3
FID PTS1d = 50000
PTS1d = 65536
F1 = 121.463285 MHz
F2 = 300.028351 MHz
SWH = 50000.00 Hz
AT1 = 1.00 sec
Hz per Pt1s0 = 0.76 Hz
SW2 = 1.00 Hz
Hz per Pt2nd0 = 1.00 Hz
Q1 = 338.4102 Hz
Q2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = -88.13
B = -85.78
C = 0.00



Compound 3f:

¹H NMR:

D:\software\nuts\DATA\\$NMR of amino acid_3-9451.HM801

23:05:55.390 +0800 nmr@EP-ZH107708

USER: nmr

SOLVENT: MeOD

Experiment = zg30

Pulse length = 9.300 usec

Recycle delay = 1.000 sec

NA = 16

Solvent = MeOD

PTS1d = 32768

F1 = 400.119995 MHz

F2 = 1.000000 MHz

SW1 = 8223.68 Hz

AT1 = 3.98 sec

Hz per Pt1stD = 0.25 Hz

SW2 = 1.00 Hz

Hz per Pt2ndD = 1.00 Hz

O1 = 2470.9036 Hz

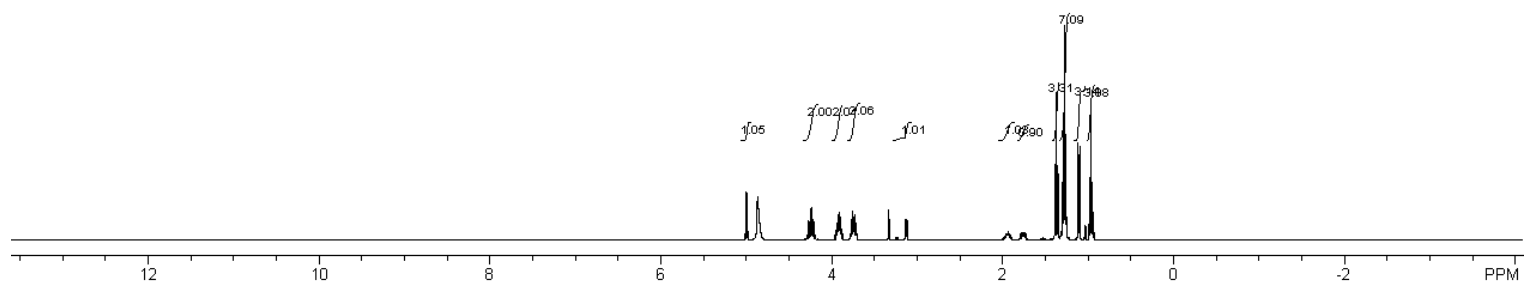
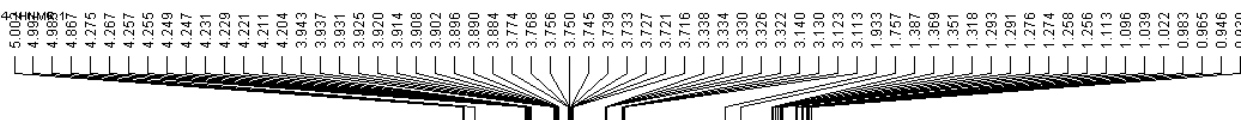
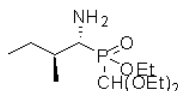
O2 = -1.0000 Hz

LB1 = 0.30 Hz

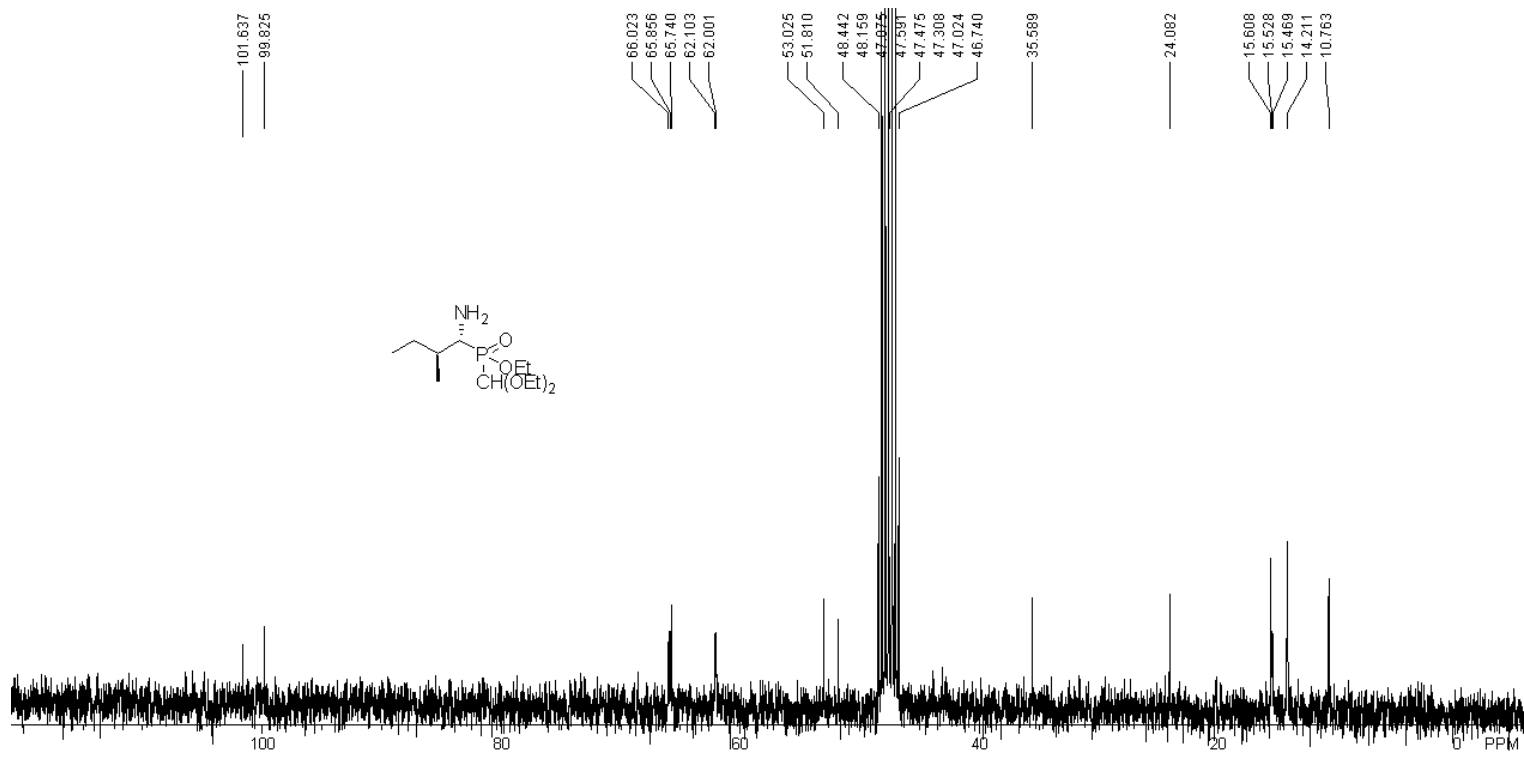
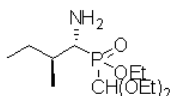
TP A = 0.00

B = 0.00

C = 0.00



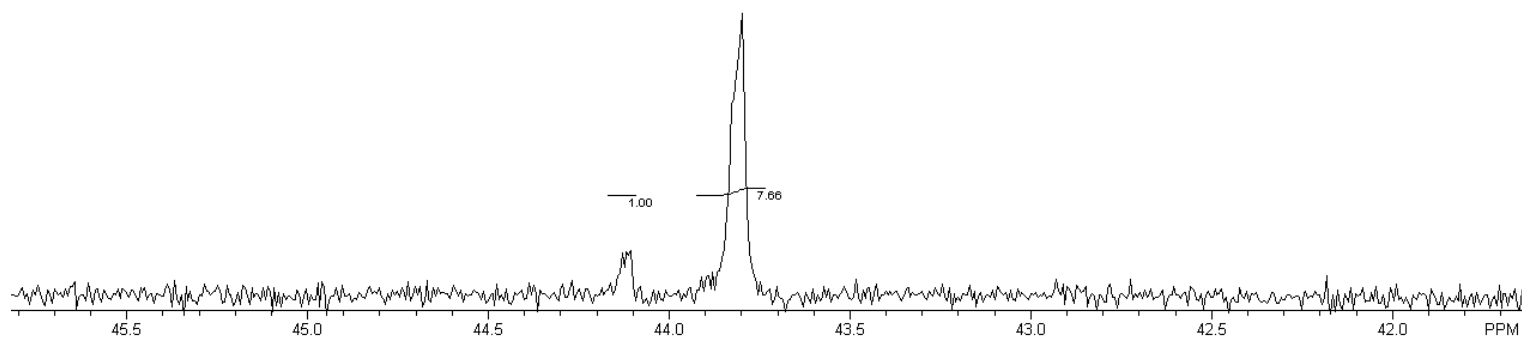
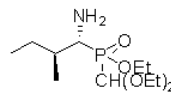
¹³C NMR:



³¹P NMR:

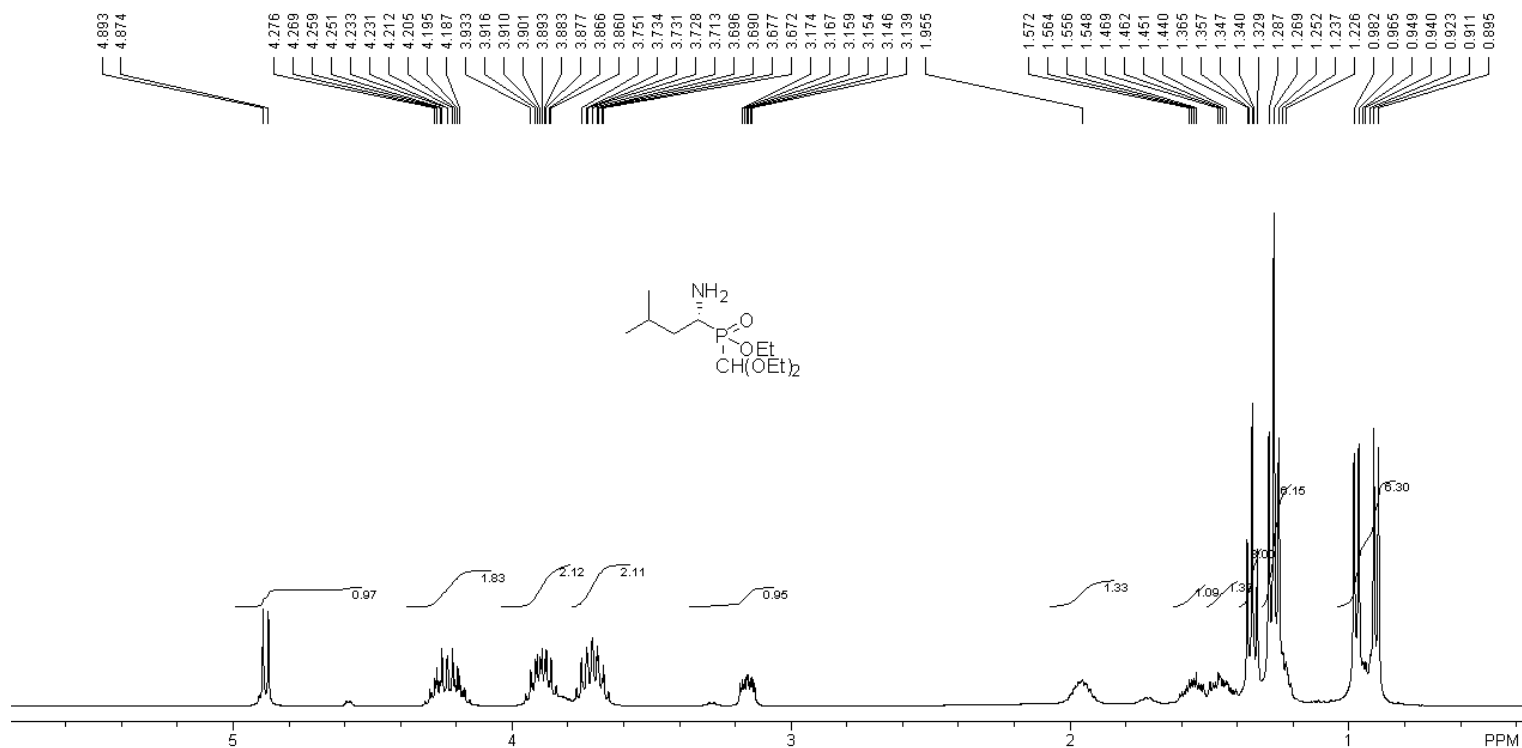
D:\software\nuts\DATA\3-94-1-p.fid
 3-94-1
 Jun 3 2010
 USER:
 SOLVENT: CD3OD
 Experiment = s2pul
 Pulse length = 3.889 usec
 Recycle delay = 1.000 sec
 NA = 88
 Solvent = CD3OD
 FID PTS1d = 60000
 PTS1d = 65536
 F1 = 121.463766 MHz
 F2 = 300.029541 MHz
 SW1 = 50000.00 Hz
 AT1 = 1.00 sec
 Hz per Pt1st0 = 0.76 Hz
 SW2 = 1.00 Hz
 Hz per Pt2nd0 = 1.00 Hz
 O1 = 338.4102 Hz
 O2 = -0.6000 Hz
 LB1 = 0.00 Hz
 TP A = -147.66
 B = 130.78
 C = 0.00

44.105
 43.797

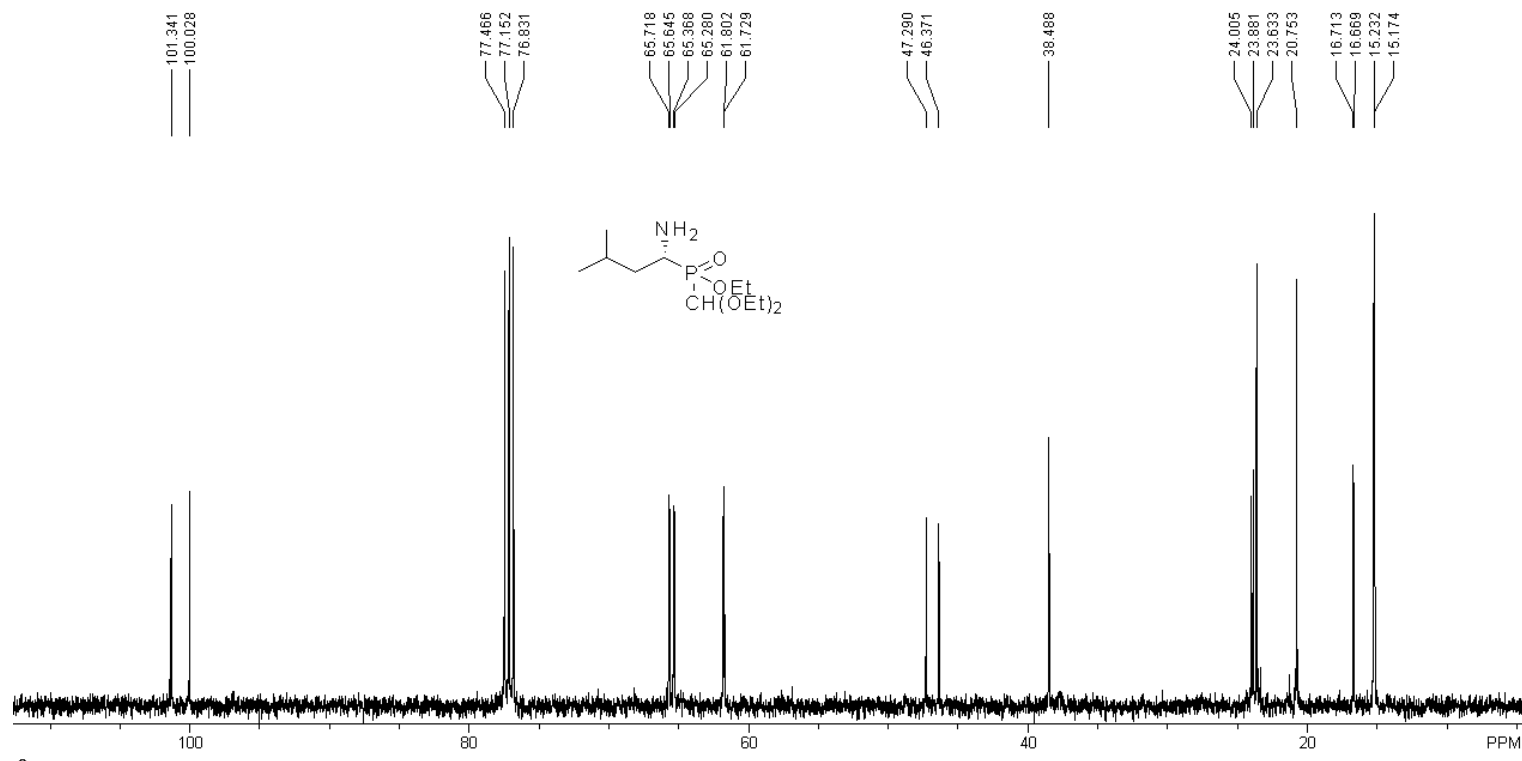


Compound 3g:

¹H NMR:



¹³C NMR:



³¹P NMR:

D:\software\nuts\DATA\峰补充数据_12-58-P_1

20:36:40.125 +0800 nmr@EP-ZH107708

USER: nmr

SOLVENT: CDCl3

Experiment = zgpg30

Pulse length = 8.300 usec

Recycle delay = 2.000 sec

NA = 8

Solvent = CDCl3

PTSD = 32768

F1 = 161.971542 MHz

F2 = 1.000000 MHz

SWH = 64102.56 Hz

AT1 = 0.51 sec

Hz per Pt1s0 = 1.96 Hz

SW2 = 1.00 Hz

Hz per Pt2nd0 = 1.00 Hz

Q1 = -8098.6865 Hz

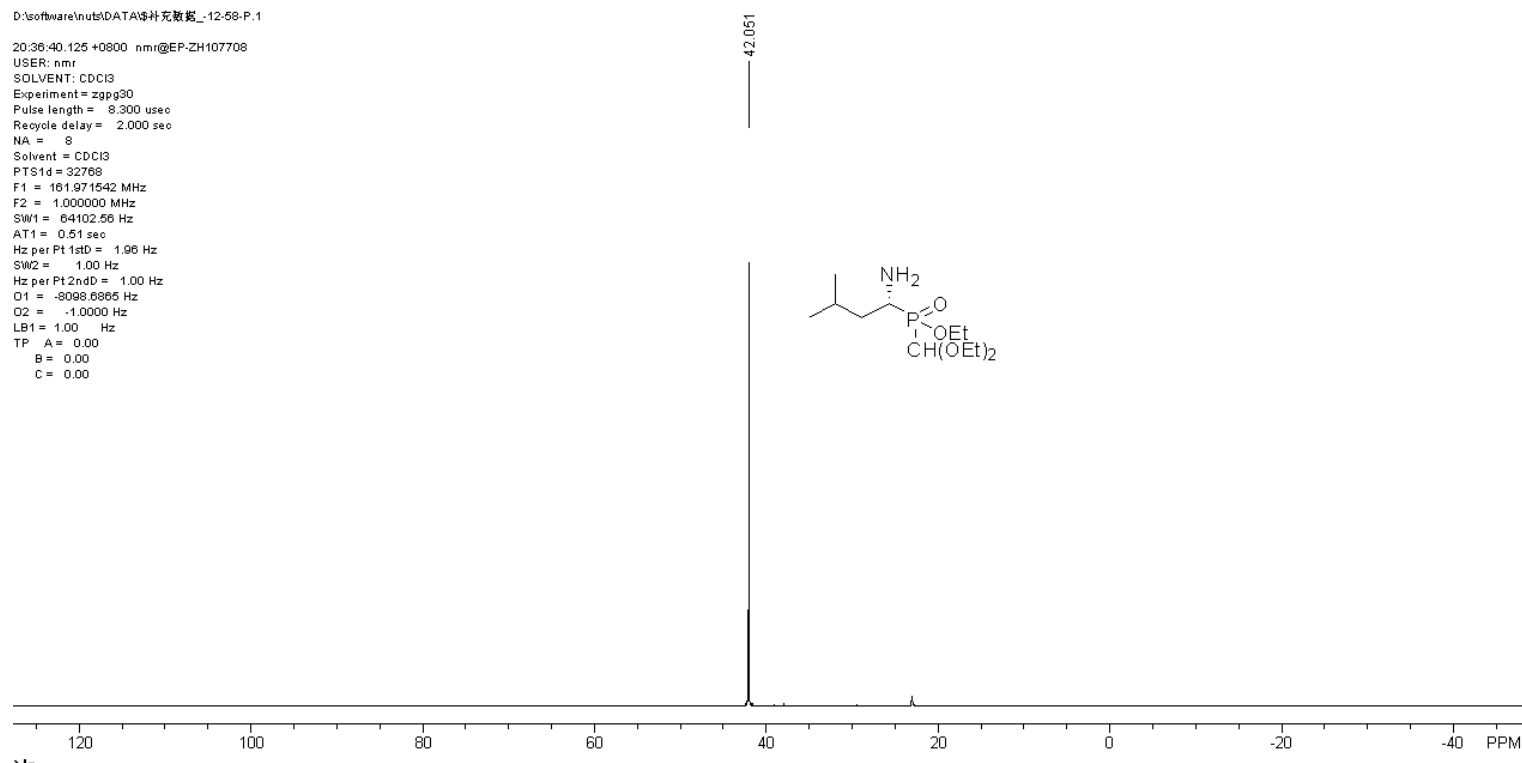
Q2 = -1.0000 Hz

LB1 = 1.00 Hz

TP A = 0.00

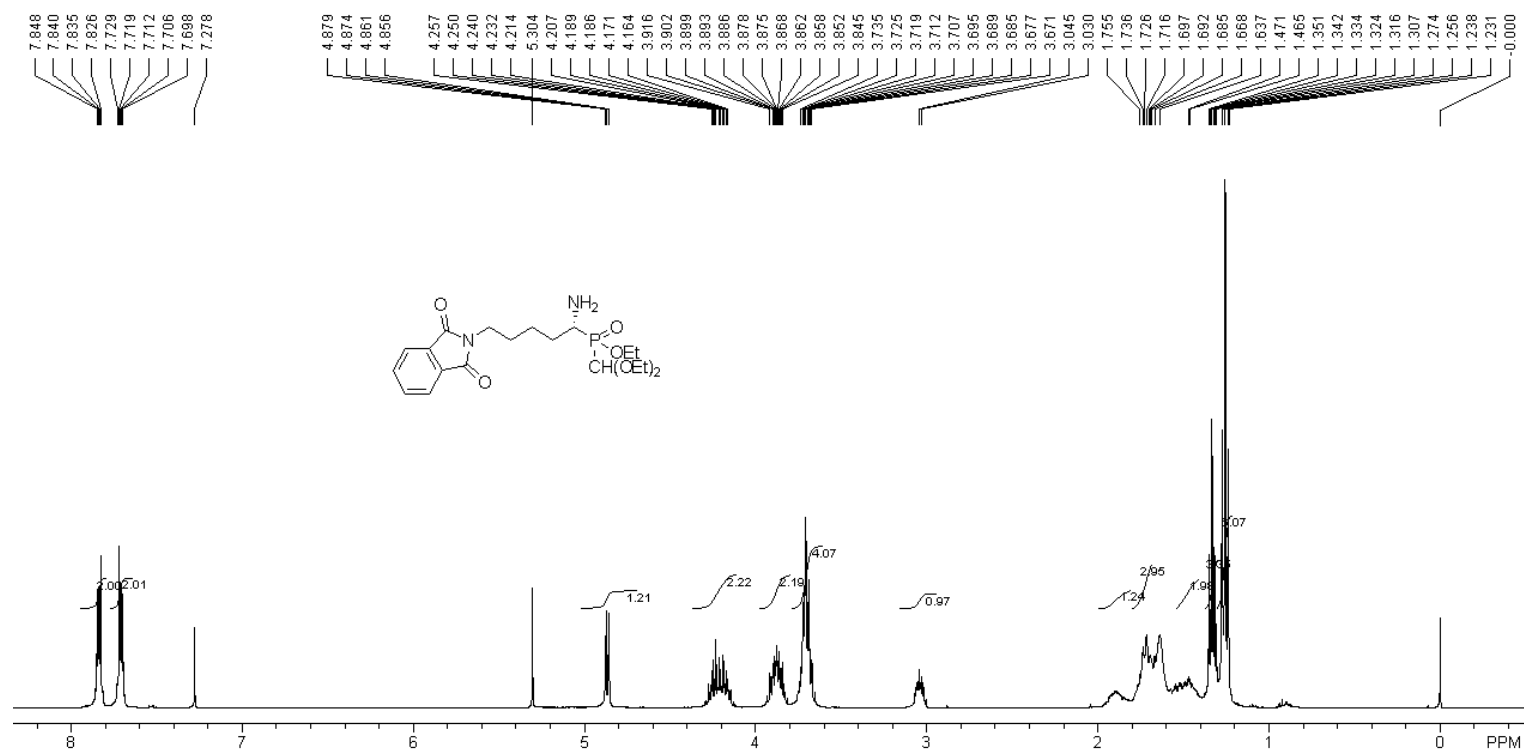
B = 0.00

C = 0.00

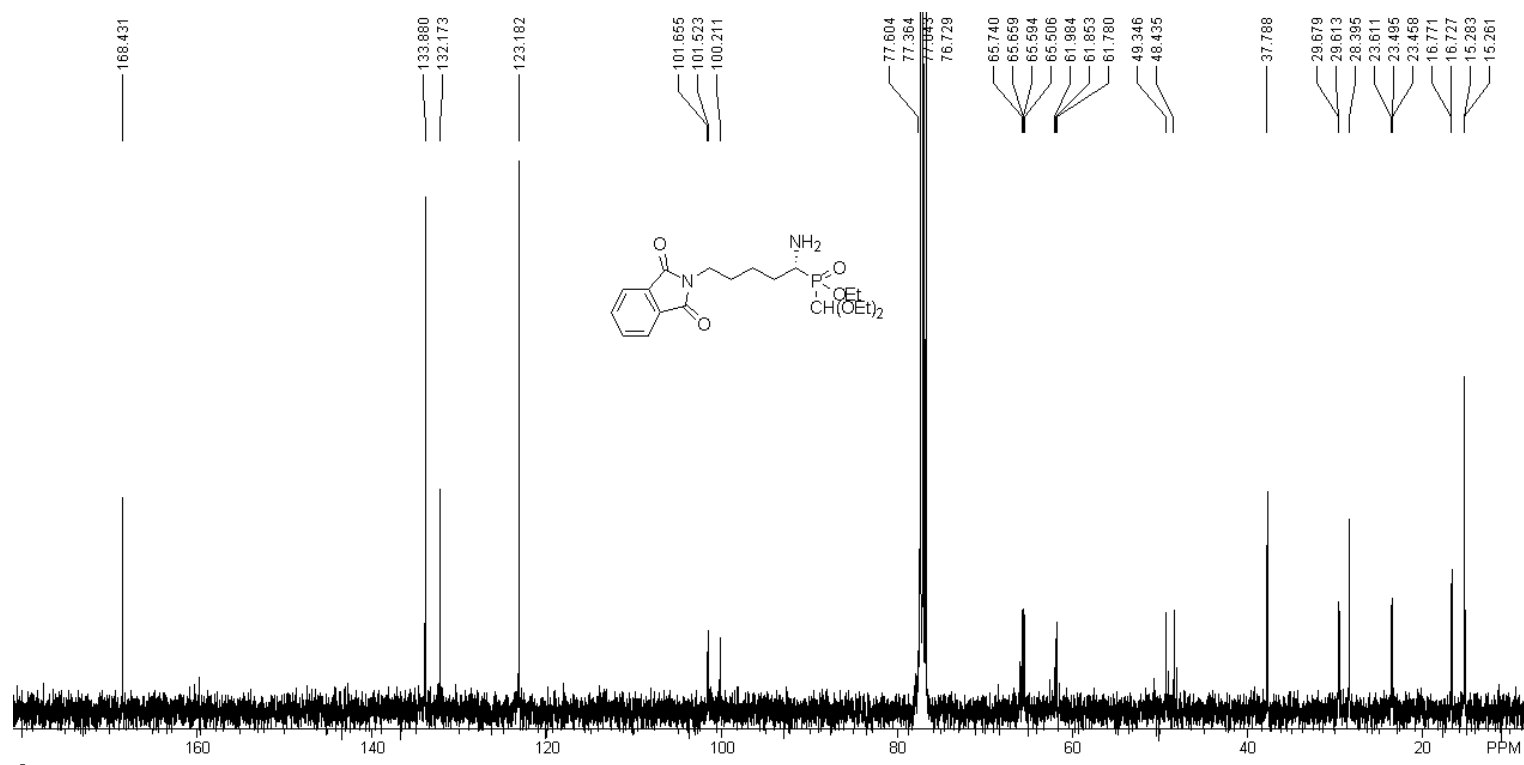


Compound 3h:

¹H NMR:

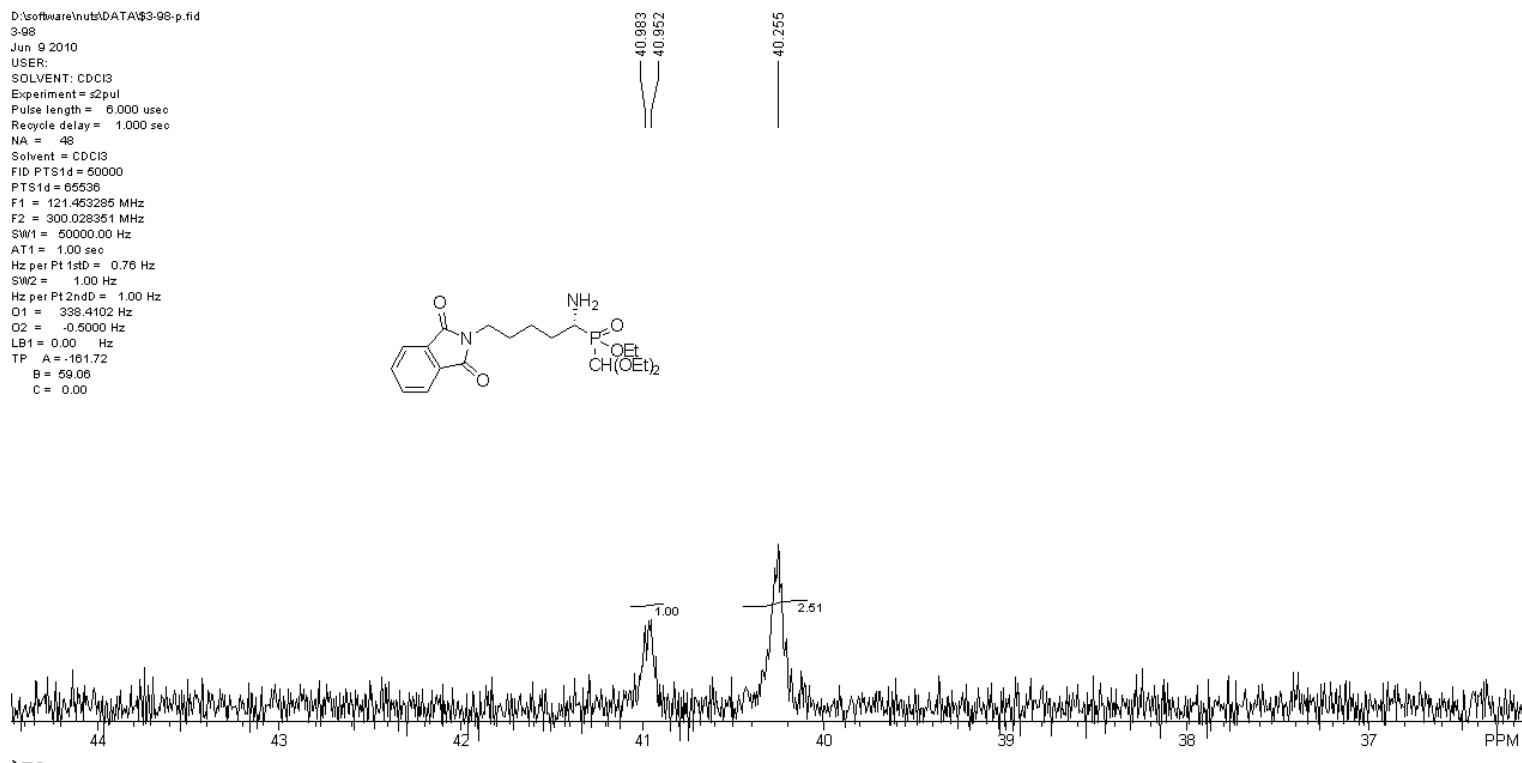
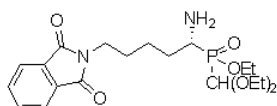


¹³C NMR:



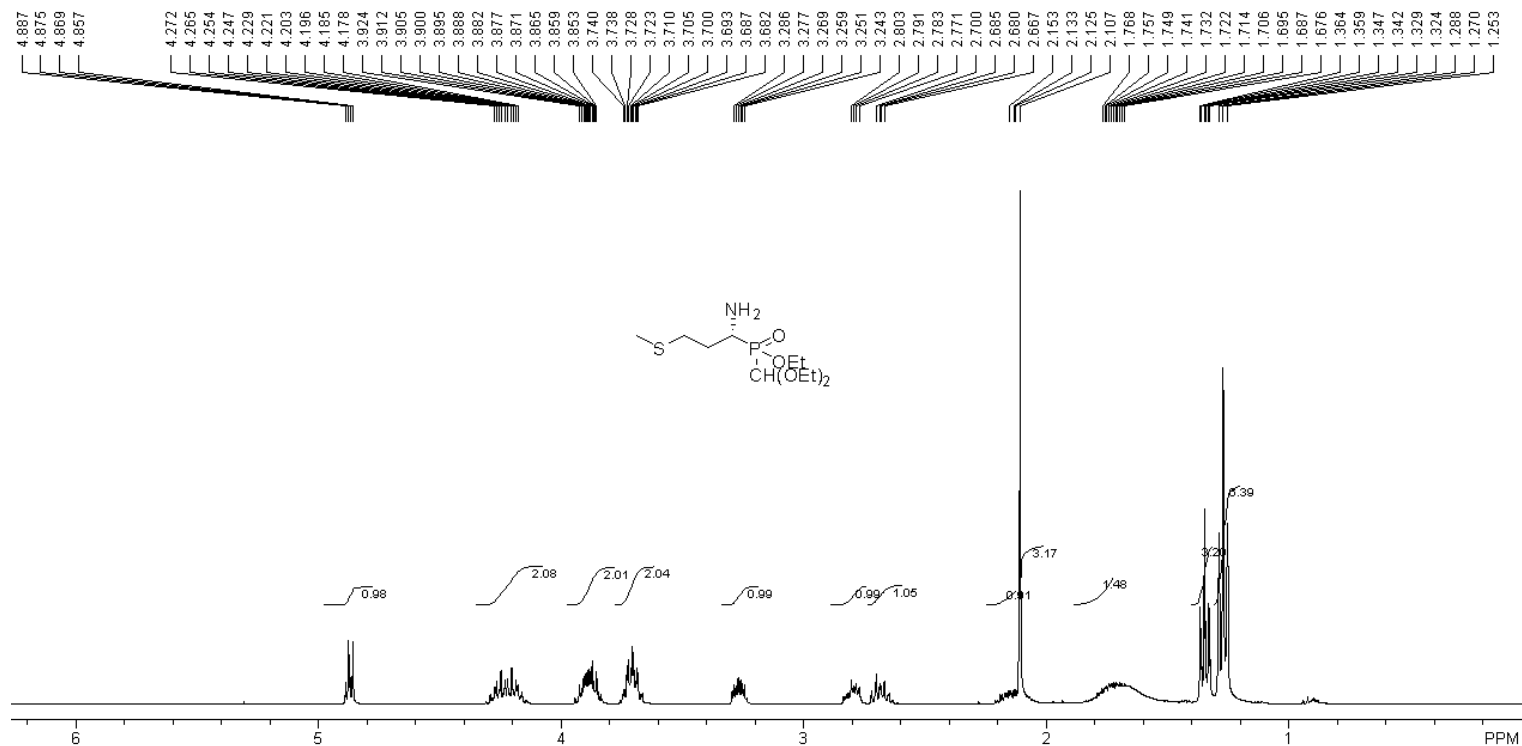
³¹P NMR:

D:\software\nuts\DATA\3-98-p.fid
 3-98
 Jun 9 2010
 USER:
 SOLVENT: CDCl3
 Experiment = s2pul
 Pulse length = 6.000 usec
 Recycle delay = 1.000 sec
 NA = 48
 Solvent = CDCl3
 FID PTS1d = 60000
 PTS1d = 65536
 F1 = 121.463285 MHz
 F2 = 300.028351 MHz
 SW1 = 50000.00 Hz
 AT1 = 1.00 sec
 Hz per Pt1st0 = 0.76 Hz
 SW2 = 1.00 Hz
 Hz per Pt2nd0 = 1.00 Hz
 O1 = 338.4102 Hz
 O2 = -0.6000 Hz
 LB1 = 0.00 Hz
 TP A = -181.72
 B = 59.06
 C = 0.00

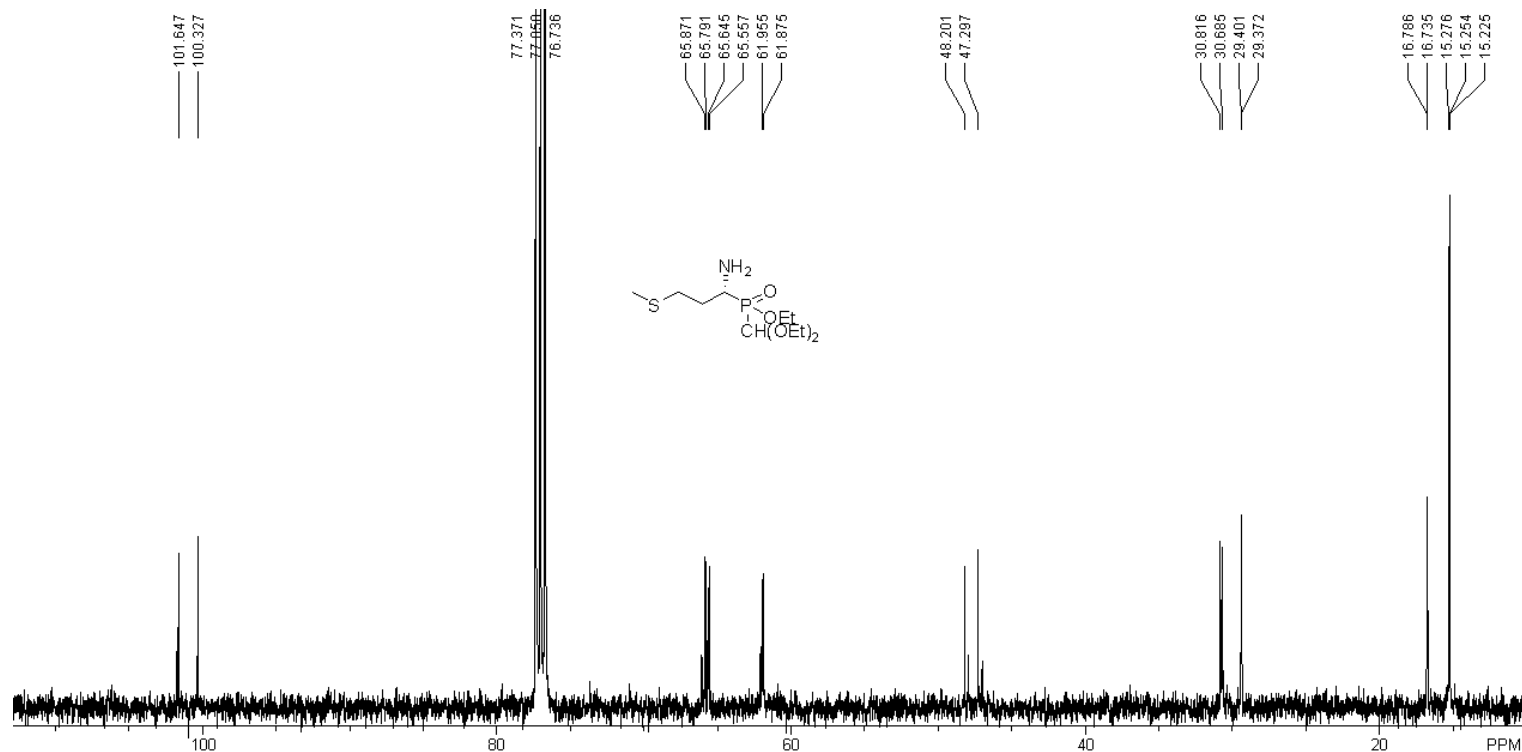


Compound 3i:

¹H NMR:

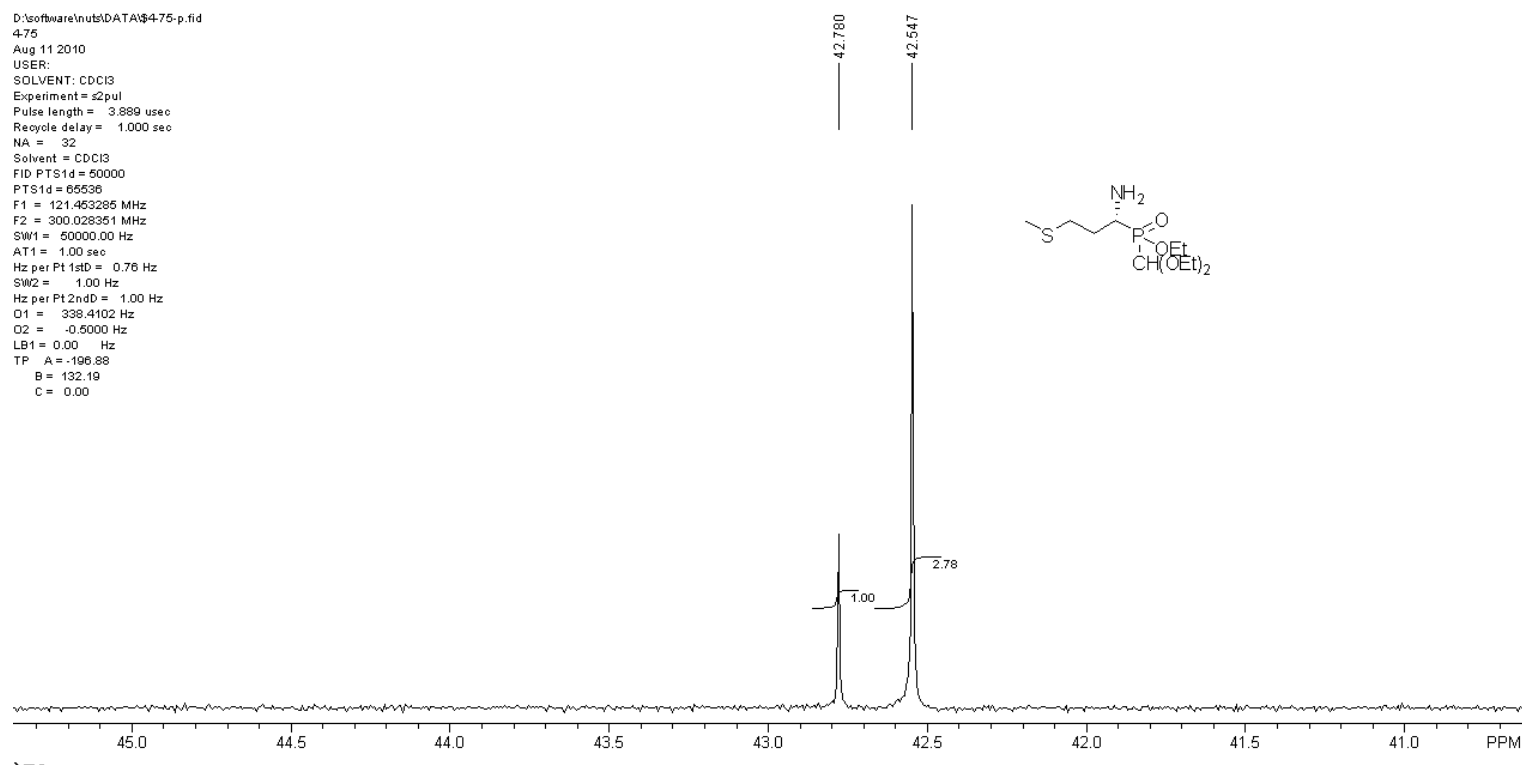


¹³C NMR:



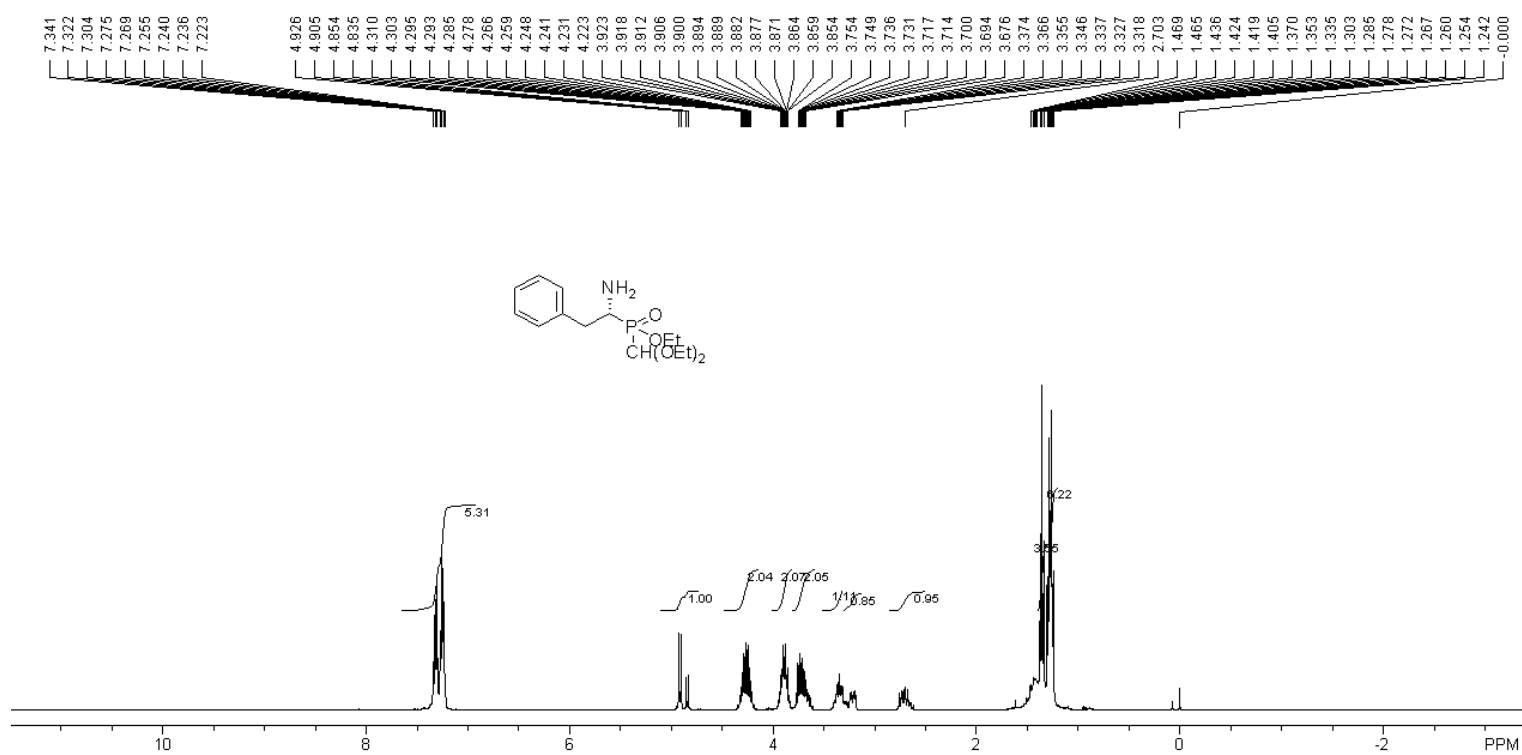
³¹P NMR:

D:\software\nuts\DATA\4-75-p.fid
4-75
Aug 11 2010
USER:
SOLVENT: CDCl3
Experiment = s2pul
Pulse length = 3.889 usec
Recycle delay = 1.000 sec
NA = 32
Solvent = CDCl3
FID PTS1d = 50000
PTS1d = 65536
F1 = 121.463285 MHz
F2 = 300.028351 MHz
SW1 = 50000.00 Hz
AT1 = 1.00 sec
Hz per Pt1s0 = 0.76 Hz
SW2 = 1.00 Hz
Hz per Pt2nd0 = 1.00 Hz
Q1 = 338.4102 Hz
Q2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = -196.88
B = 132.19
C = 0.00

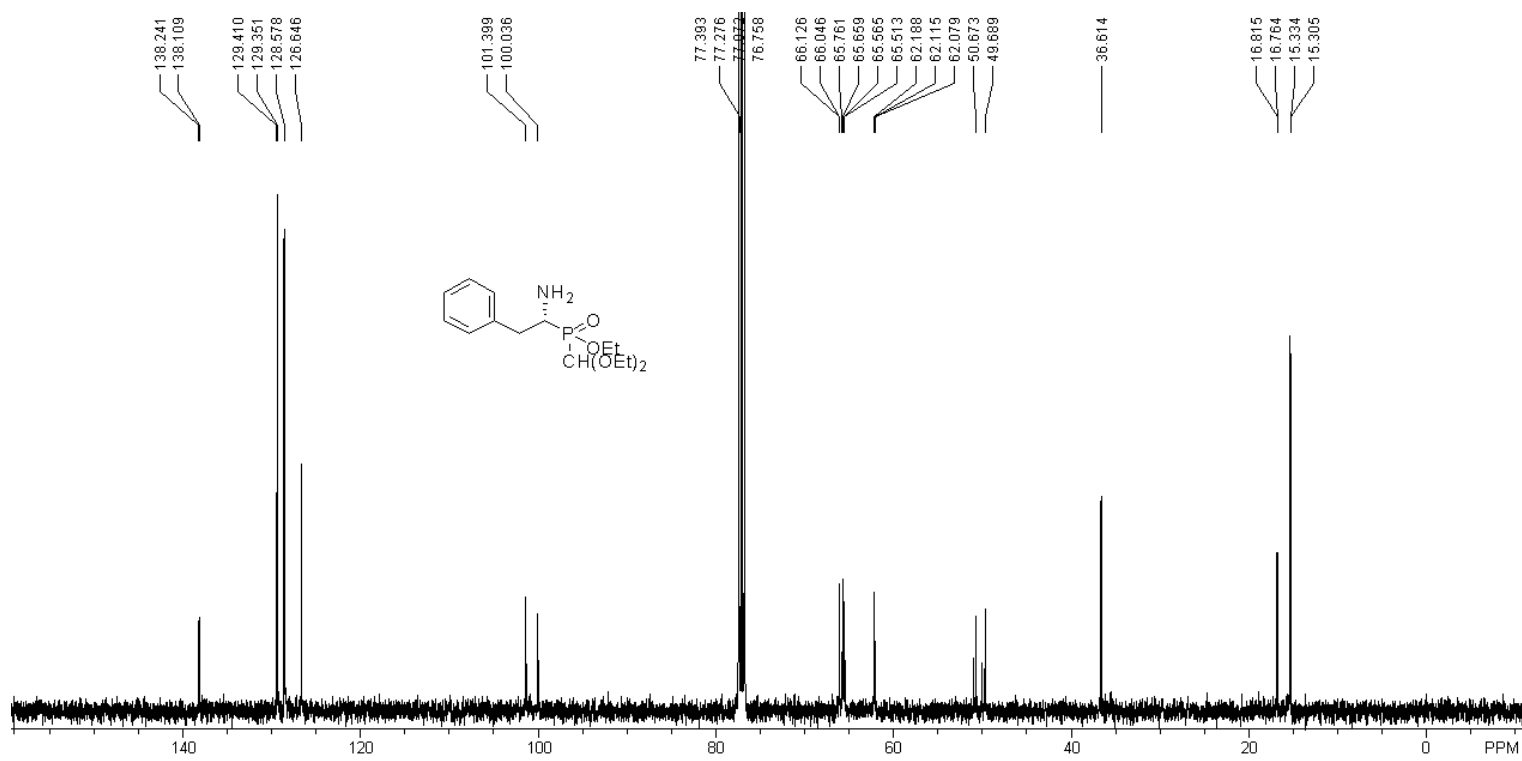


Compound 3j:

¹H NMR:



¹³C NMR:



³¹P NMR:

D:\software\nuts\DATA\峰补充数据_5-28-P.1

15:20:19.625 +0800 nmri@EP-ZH107708

USER: nmri

SOLVENT: CDCl3

Experiment = zgpg30

Pulse length = 8.300 usec

Recycle delay = 2.000 sec

NA = 8

Solvent = CDCl3

PTS1d = 32768

F1 = 161.971542 MHz

F2 = 1.000000 MHz

SW1 = 64102.56 Hz

AT1 = 0.51 sec

Hz per Pt1stD = 1.96 Hz

SW2 = 1.00 Hz

Hz per Pt2ndD = 1.00 Hz

O1 = -8098.6865 Hz

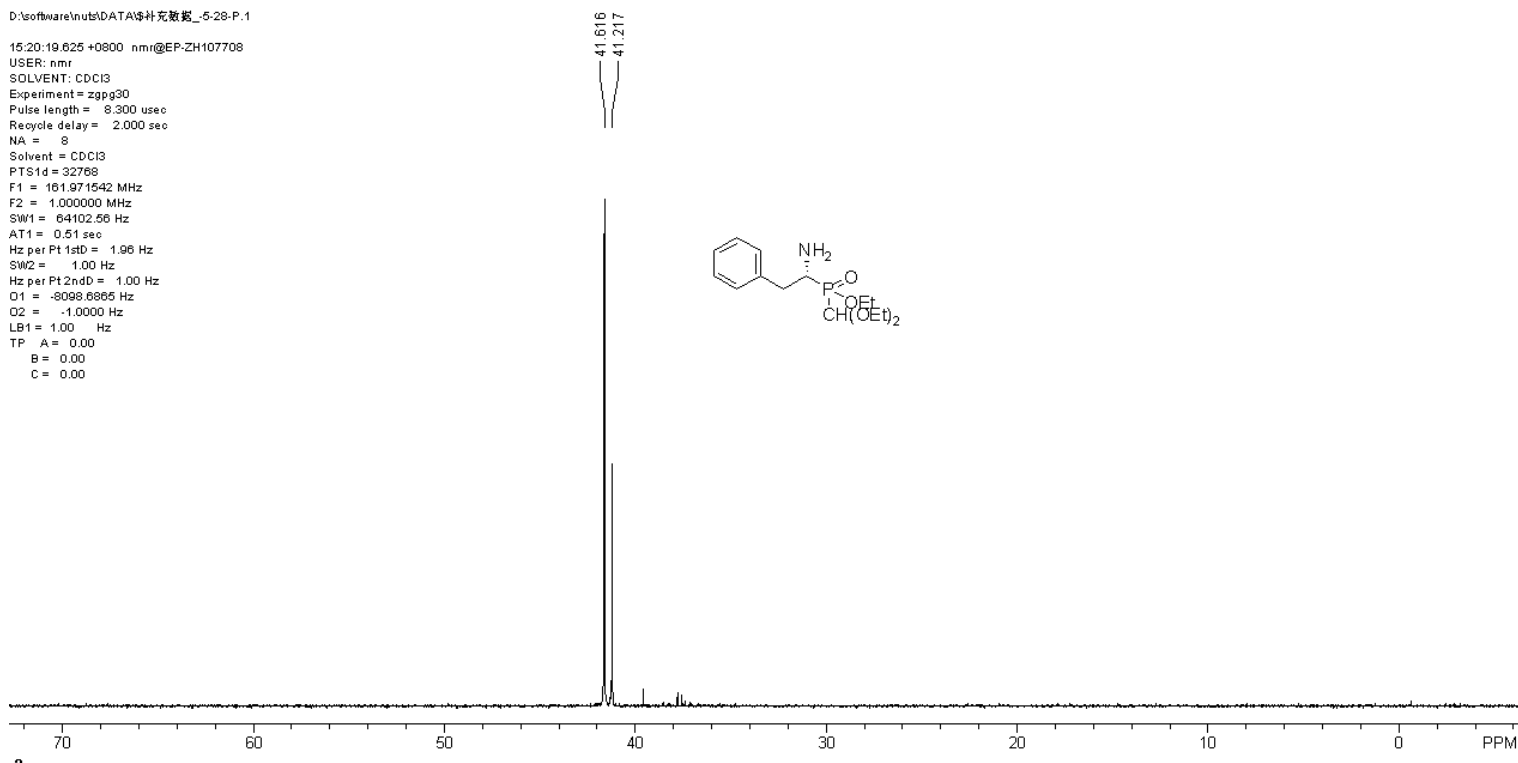
O2 = -1.0000 Hz

LB1 = 1.00 Hz

TP A = 0.00

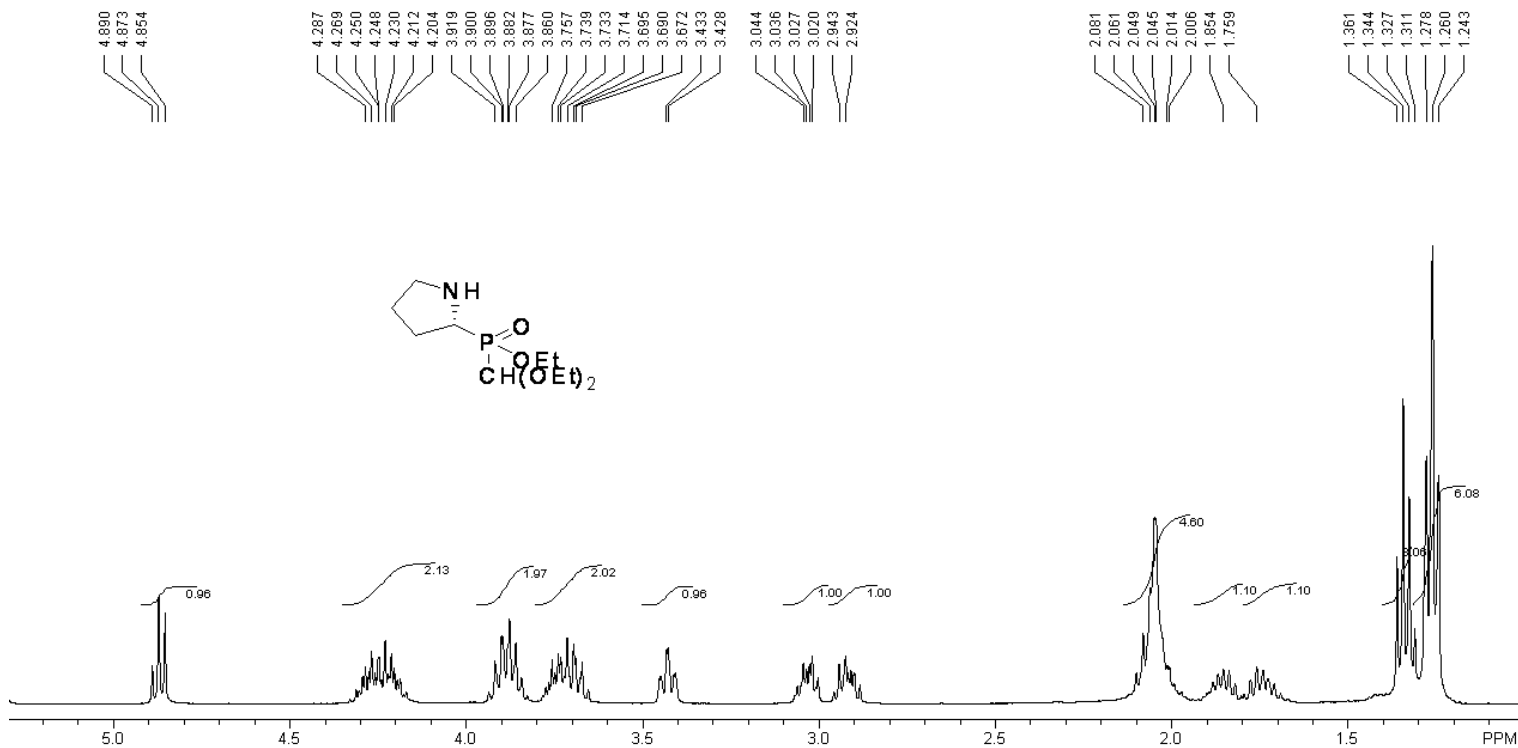
B = 0.00

C = 0.00

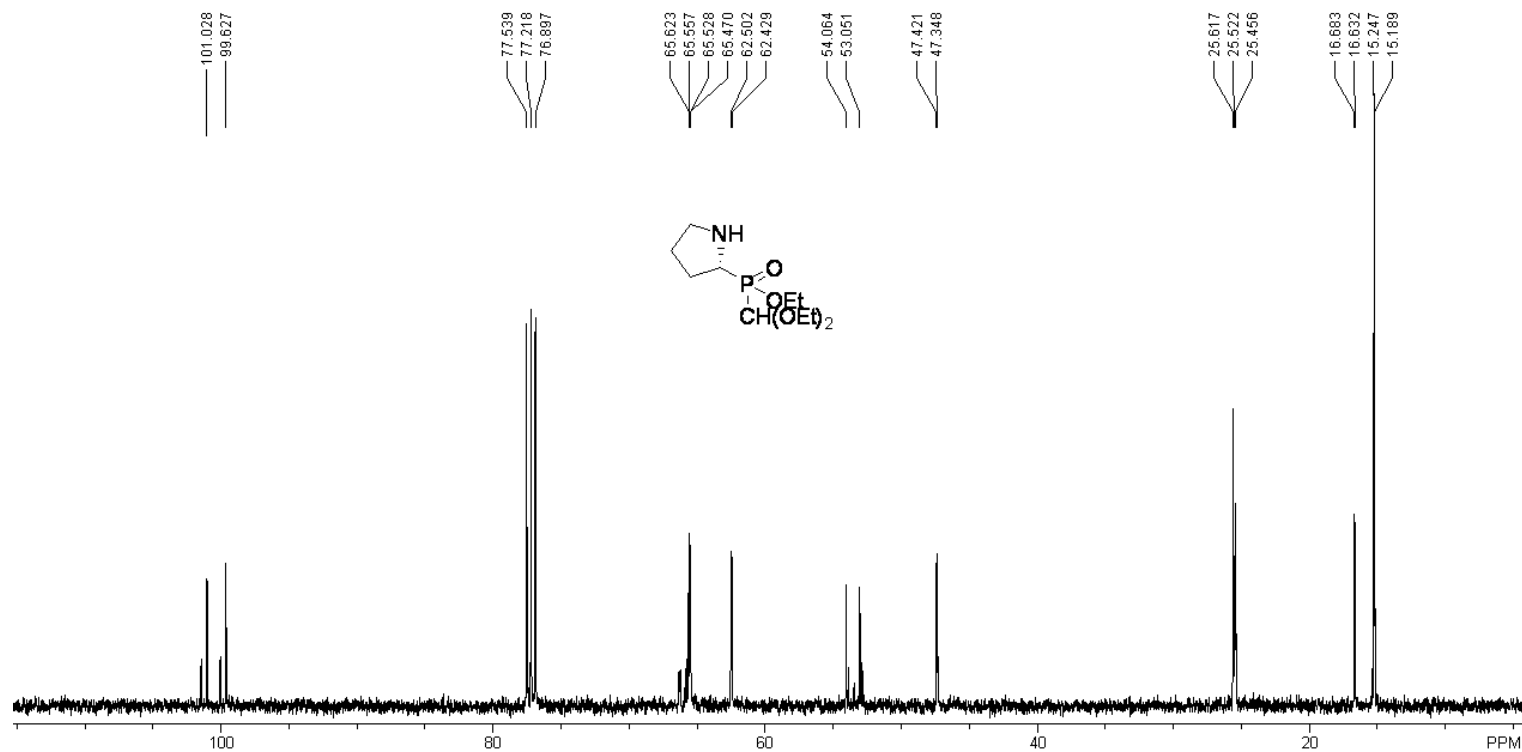


Compound 3k:

¹H NMR:

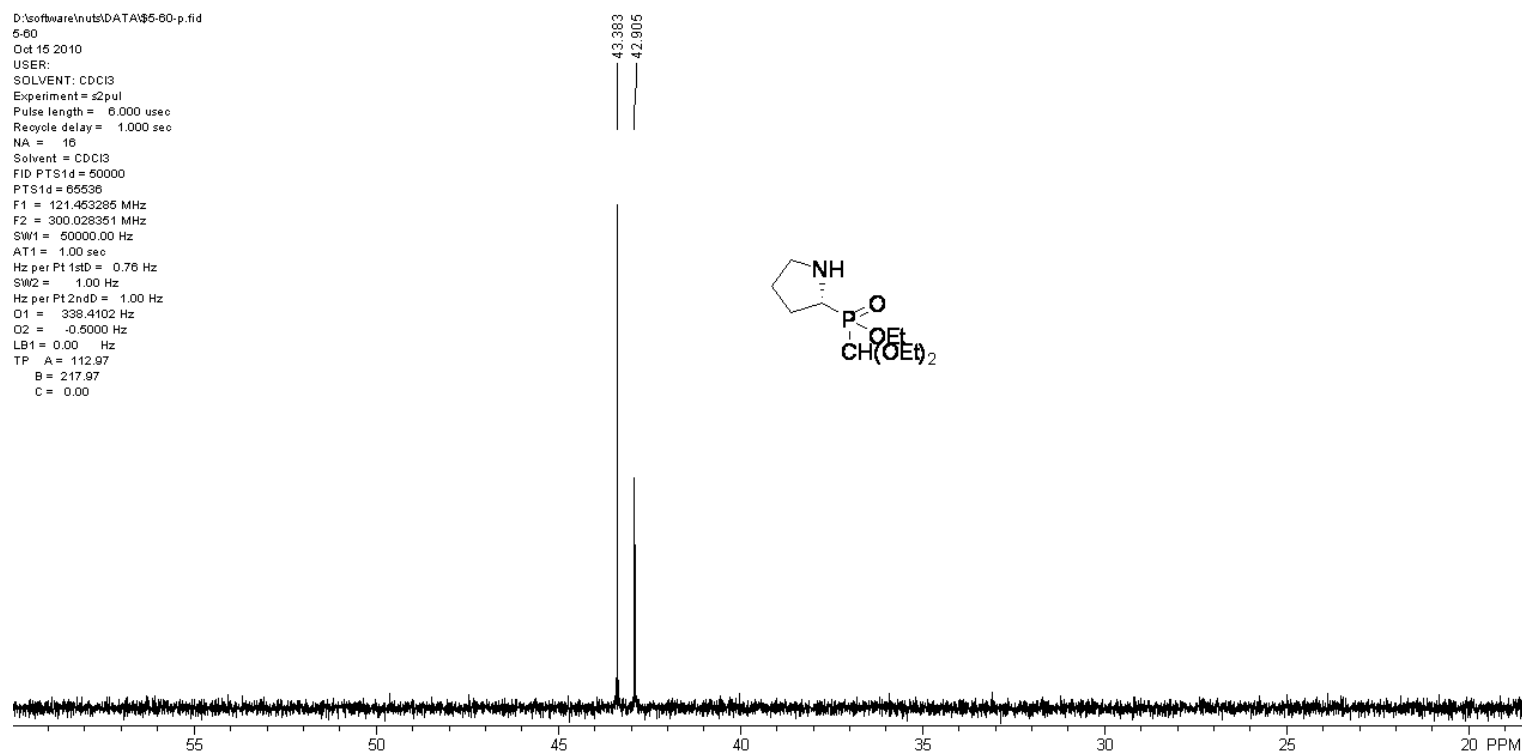


¹³C NMR:



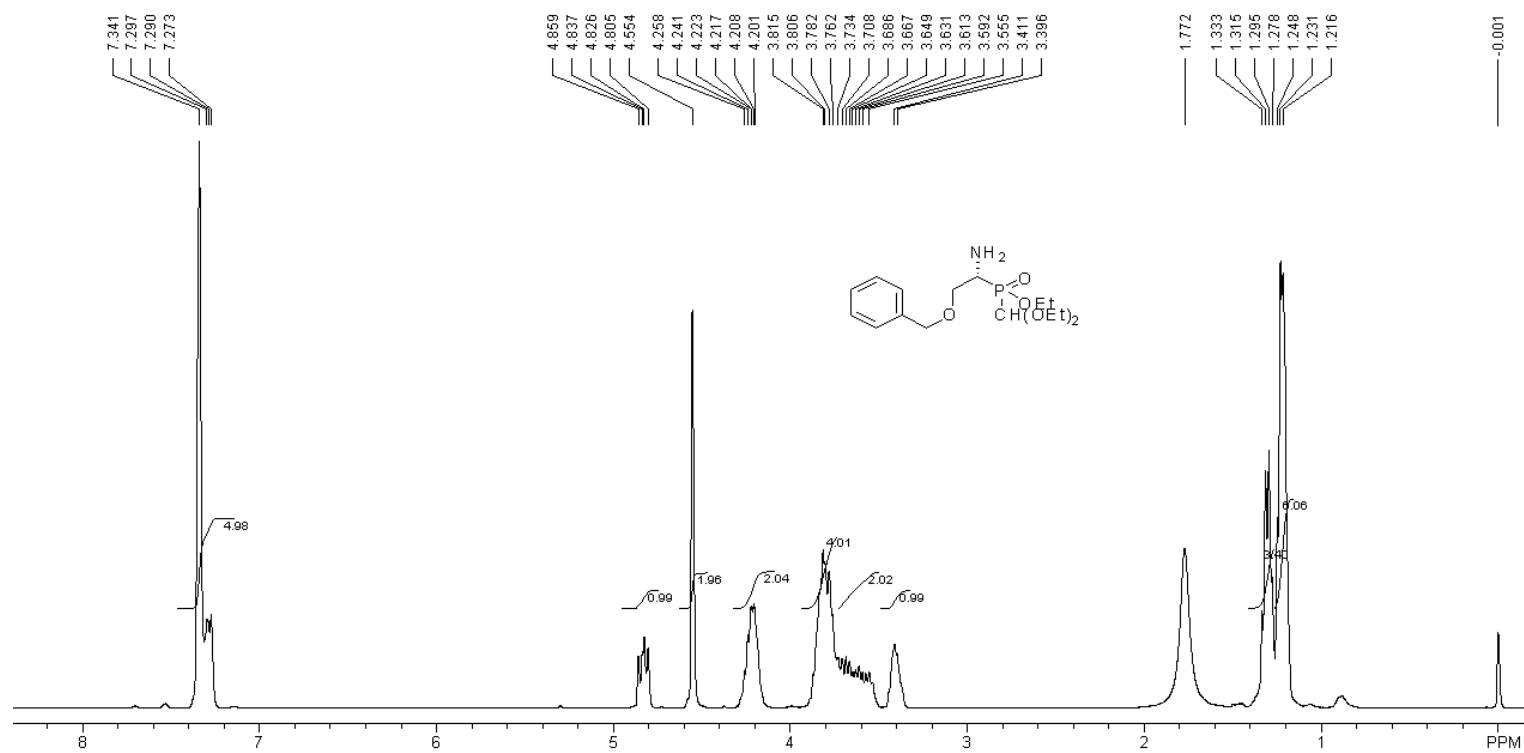
³¹P NMR:

D:\software\nuts\DATA\5-60-p.fid
 5-60
 Oct 15 2010
 USER:
 SOLVENT: CDCl3
 Experiment = s2pul
 Pulse length = 6.000 usec
 Recycle delay = 1.000 sec
 NA = 16
 Solvent = CDCl3
 FID PTS1d = 50000
 PTS1d = 65536
 F1 = 121.463285 MHz
 F2 = 300.028351 MHz
 SW1 = 50000.00 Hz
 AT1 = 1.00 sec
 Hz per Pt1s0 = 0.76 Hz
 SW2 = 1.00 Hz
 Hz per Pt2nd0 = 1.00 Hz
 Q1 = 338.4102 Hz
 Q2 = -0.5000 Hz
 LB1 = 0.00 Hz
 TP A = 112.97
 B = 217.97
 C = 0.00

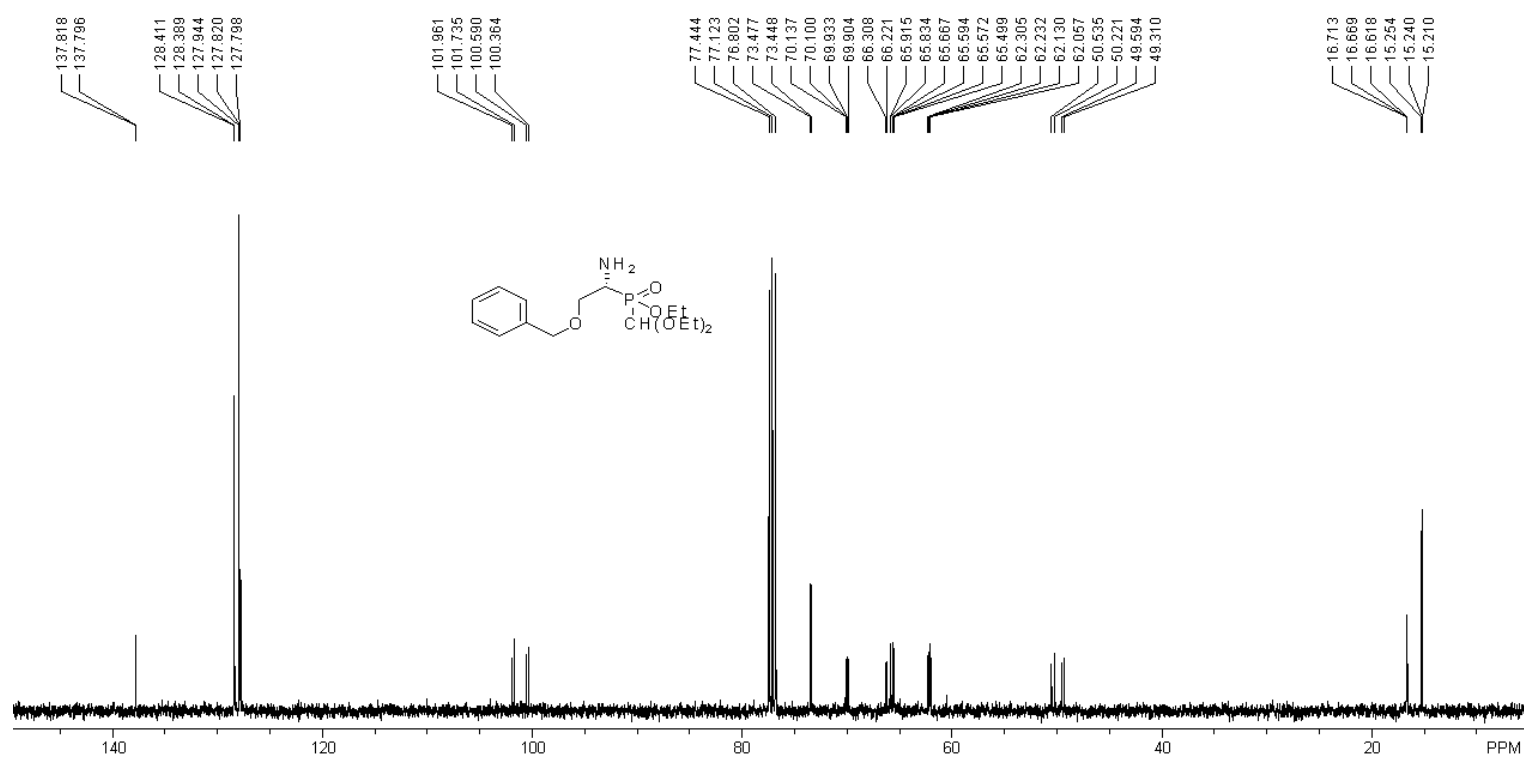


Compound 3l:

¹H NMR:



¹³C NMR:



³¹P NMR:

D:\software\nuts\DATA\2008119-9-34-2_11.1

09:10:46.953 +0800 nmr@EP-ZH107708

USER: nmr

SOLVENT: CDCl3

Experiment = zgpg30

Pulse length = 8.300 usec

Recycle delay = 2.000 sec

NA = 8

Solvent = CDCl3

PTS1d = 32768

F1 = 161.971542 MHz

F2 = 1.000000 MHz

SW1 = 64102.56 Hz

AT1 = 0.51 sec

Hz per Pt1stD = 1.96 Hz

SW2 = 1.00 Hz

Hz per Pt2ndD = 1.00 Hz

O1 = -8098.6865 Hz

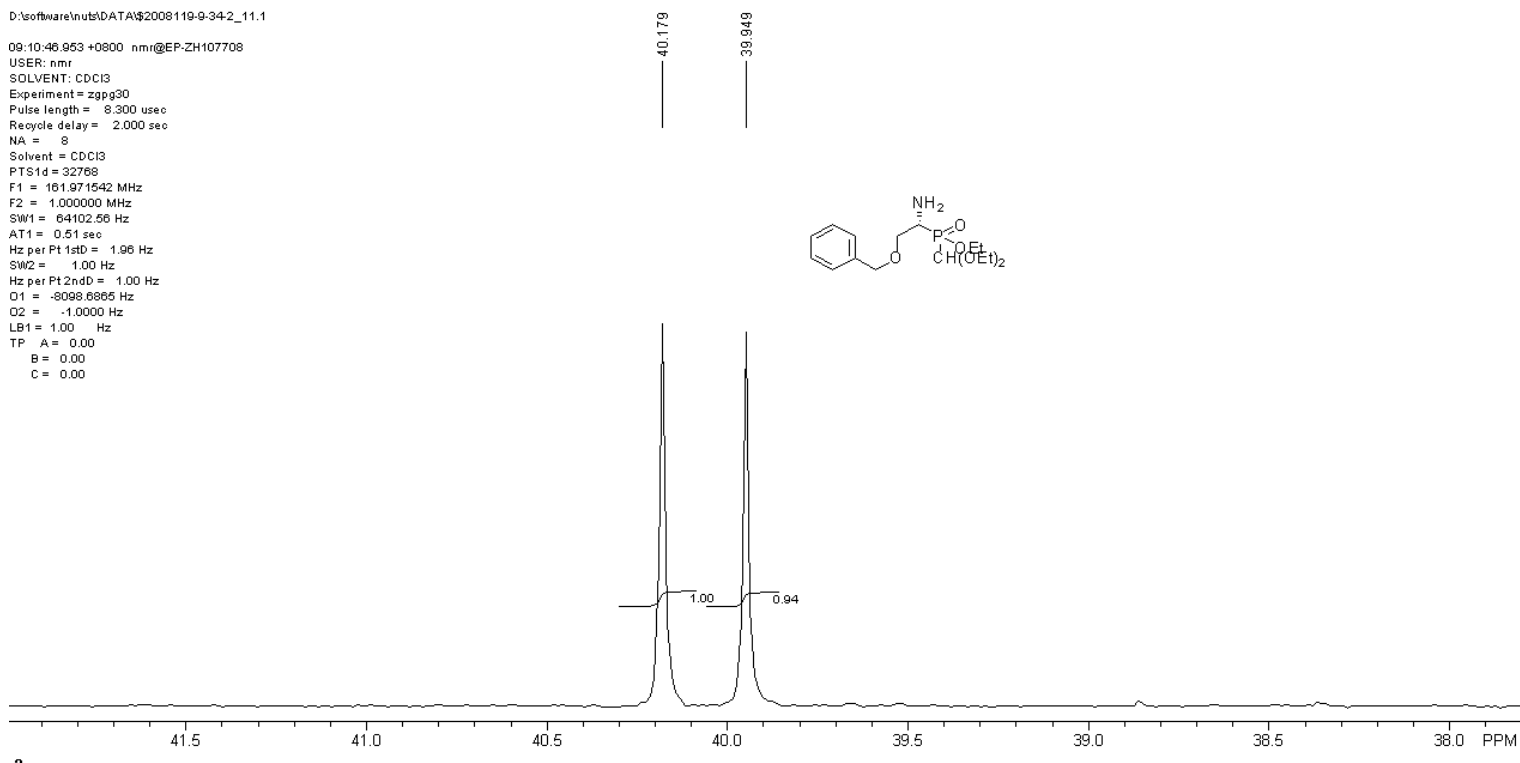
O2 = -1.0000 Hz

LB1 = 1.00 Hz

TP A = 0.00

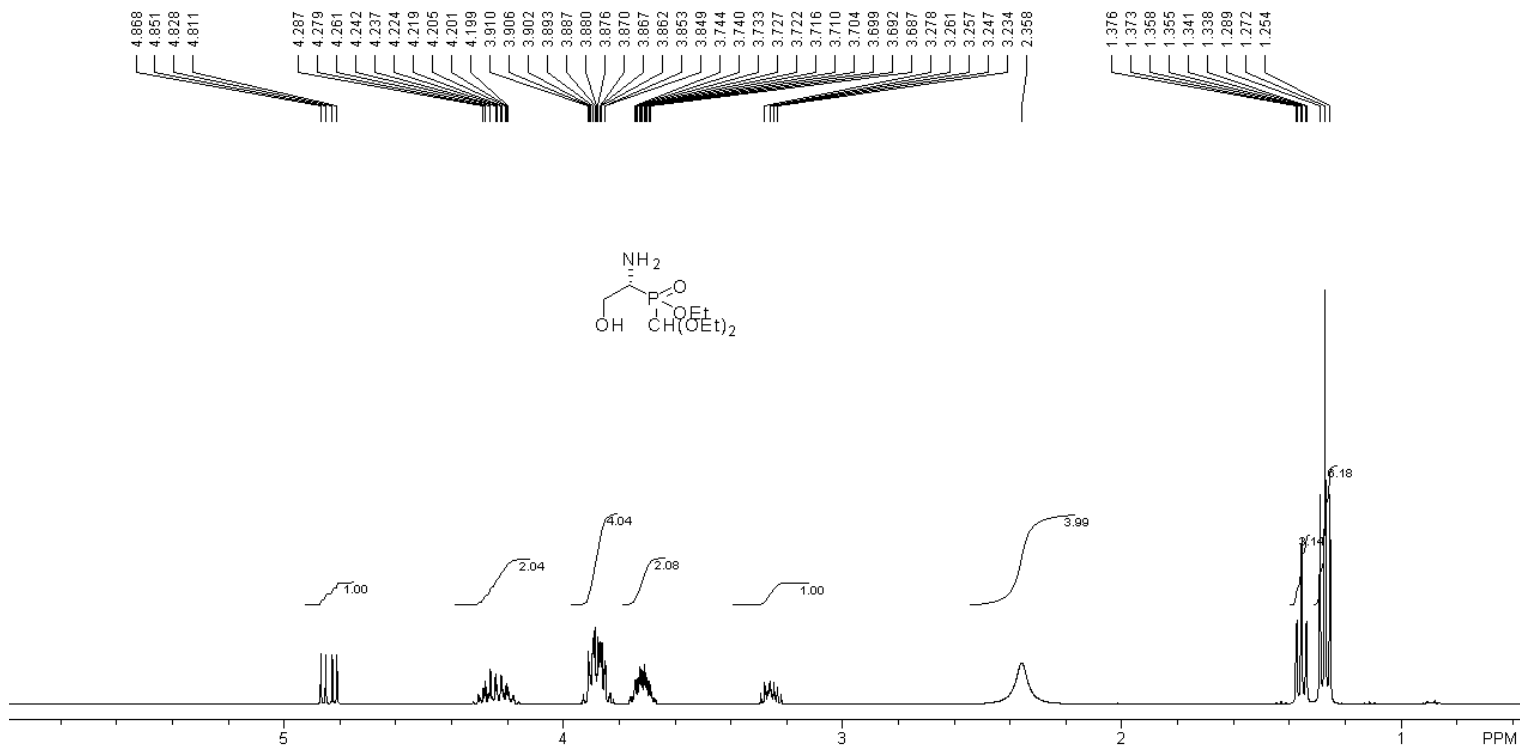
B = 0.00

C = 0.00

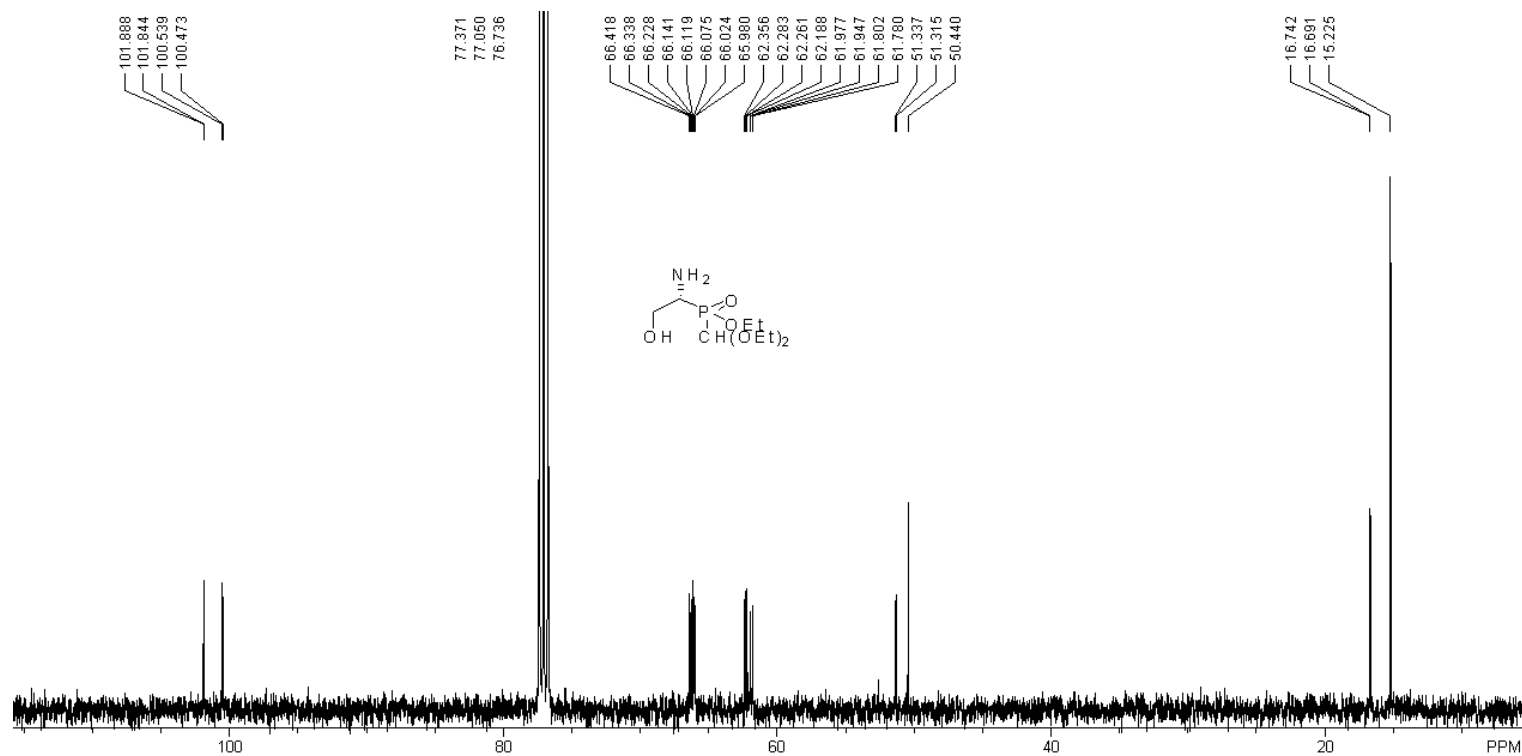


Compound 3s:

¹H NMR:



¹³C NMR:



³¹P NMR:

D:\software\nuts\DATA\20081109-9-43_11.1

13:44:08.765 +0800 nmr@EP.ZH107708

USER: nmr

SOLVENT: CDCl3

Experiment = zgpg30

Pulse length = 8.300 usec

Recycle delay = 2.000 sec

NA = 16

Solvent = CDCl3

PTS1d = 32768

F1 = 161.971542 MHz

F2 = 1.000000 MHz

SW1 = 64102.56 Hz

AT1 = 0.51 sec

Hz per Pt1stD = 1.96 Hz

SW2 = 1.00 Hz

Hz per Pt2ndD = 1.00 Hz

O1 = -8098.6865 Hz

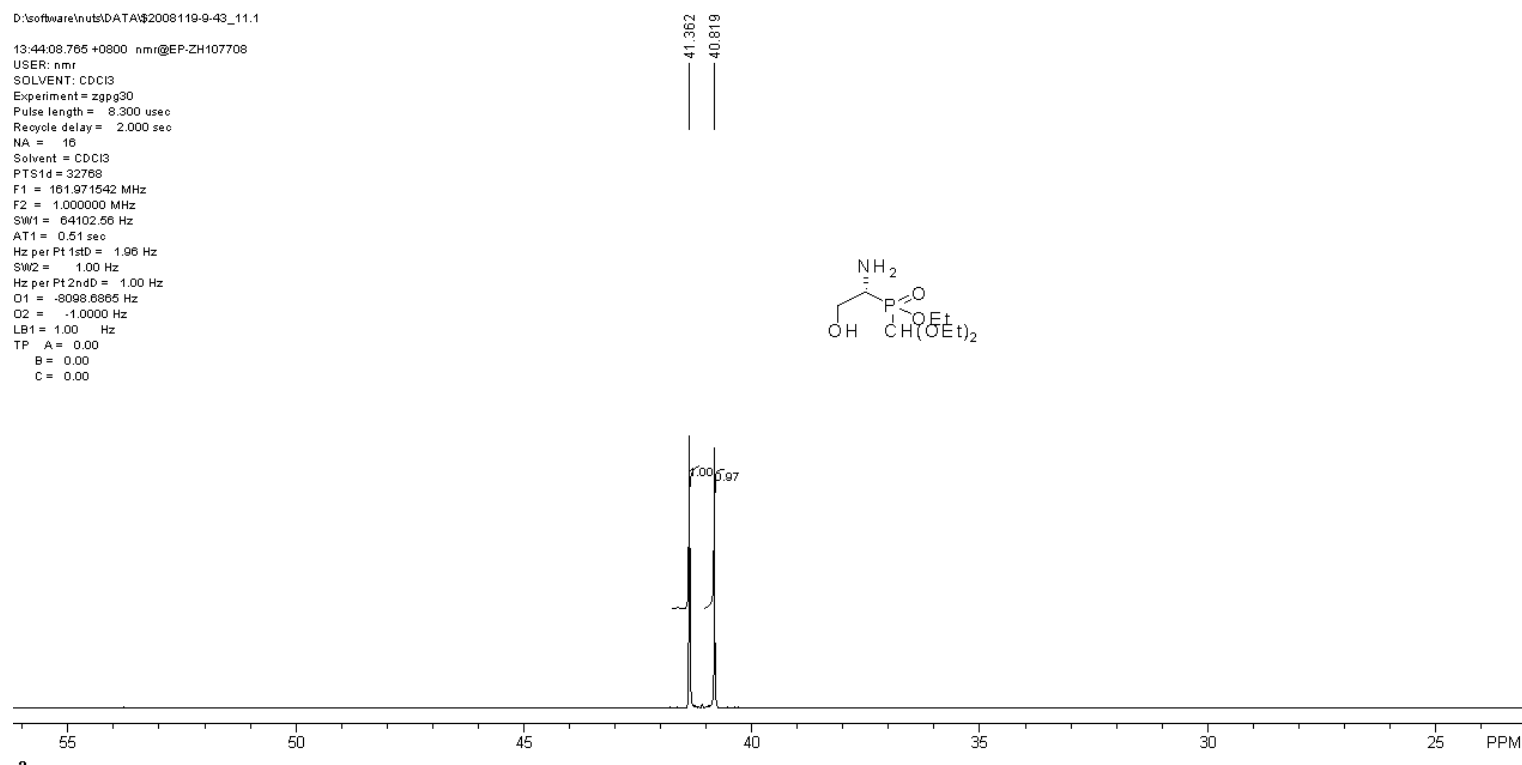
O2 = -1.0000 Hz

LB1 = 1.00 Hz

TP A = 0.00

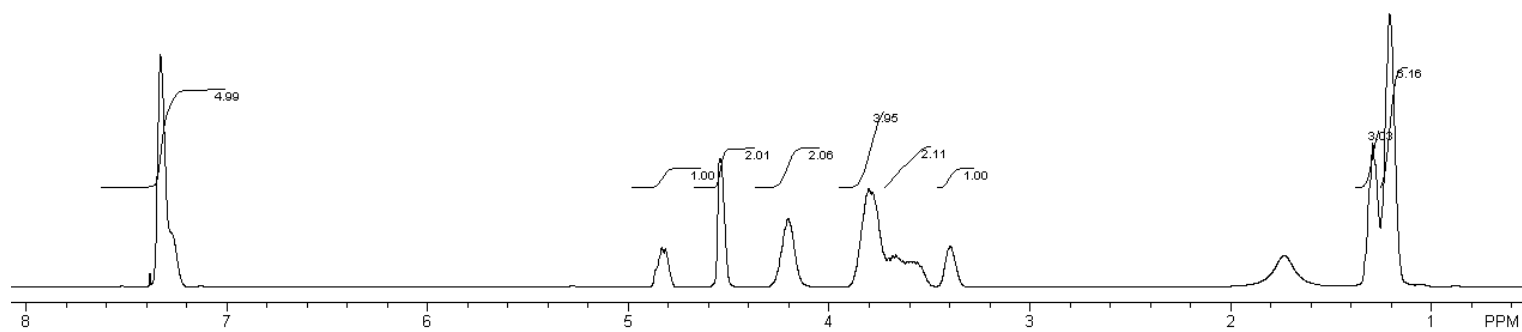
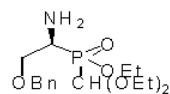
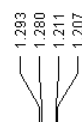
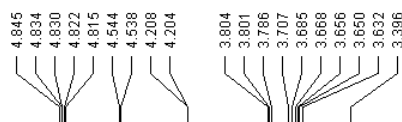
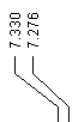
B = 0.00

C = 0.00

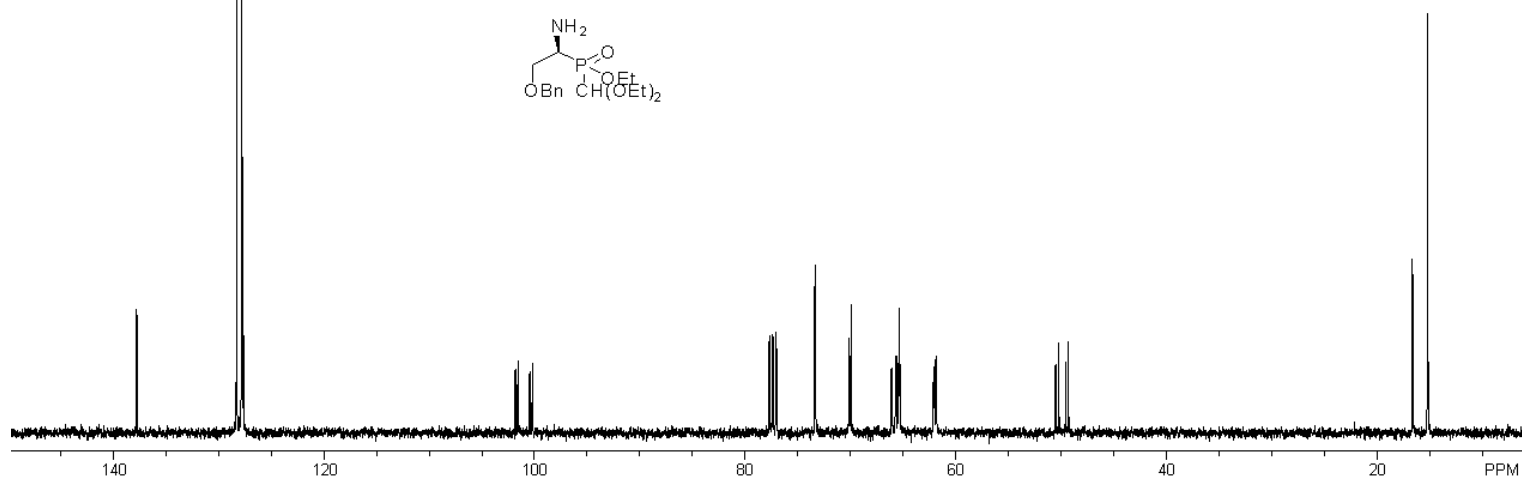
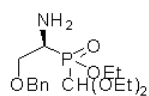
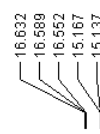
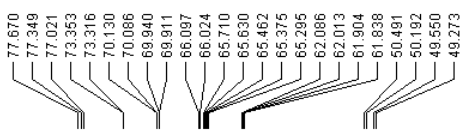
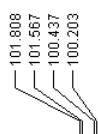
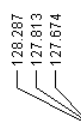
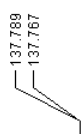


Compound (-)-3l:

¹H NMR:



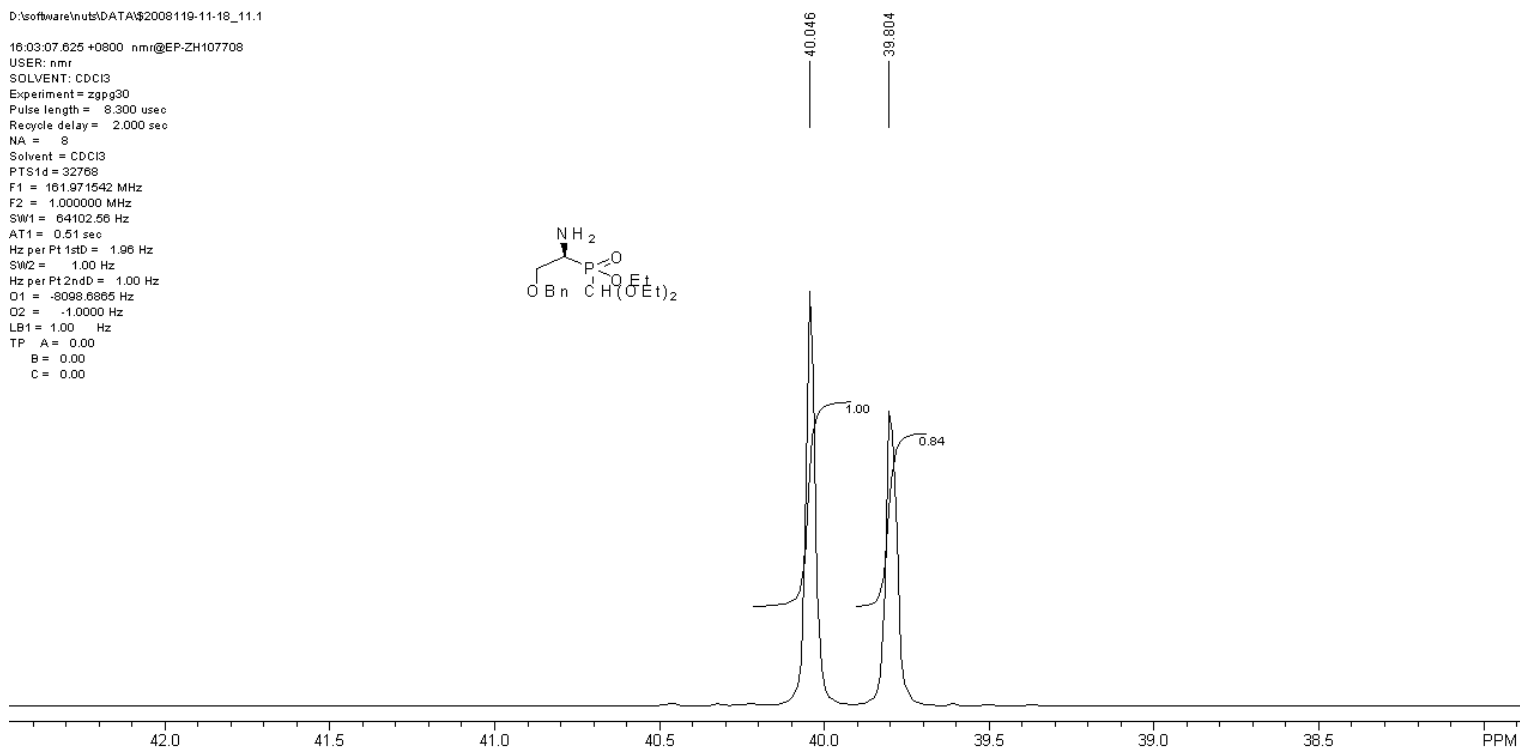
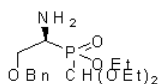
¹³C NMR:



³¹P NMR:

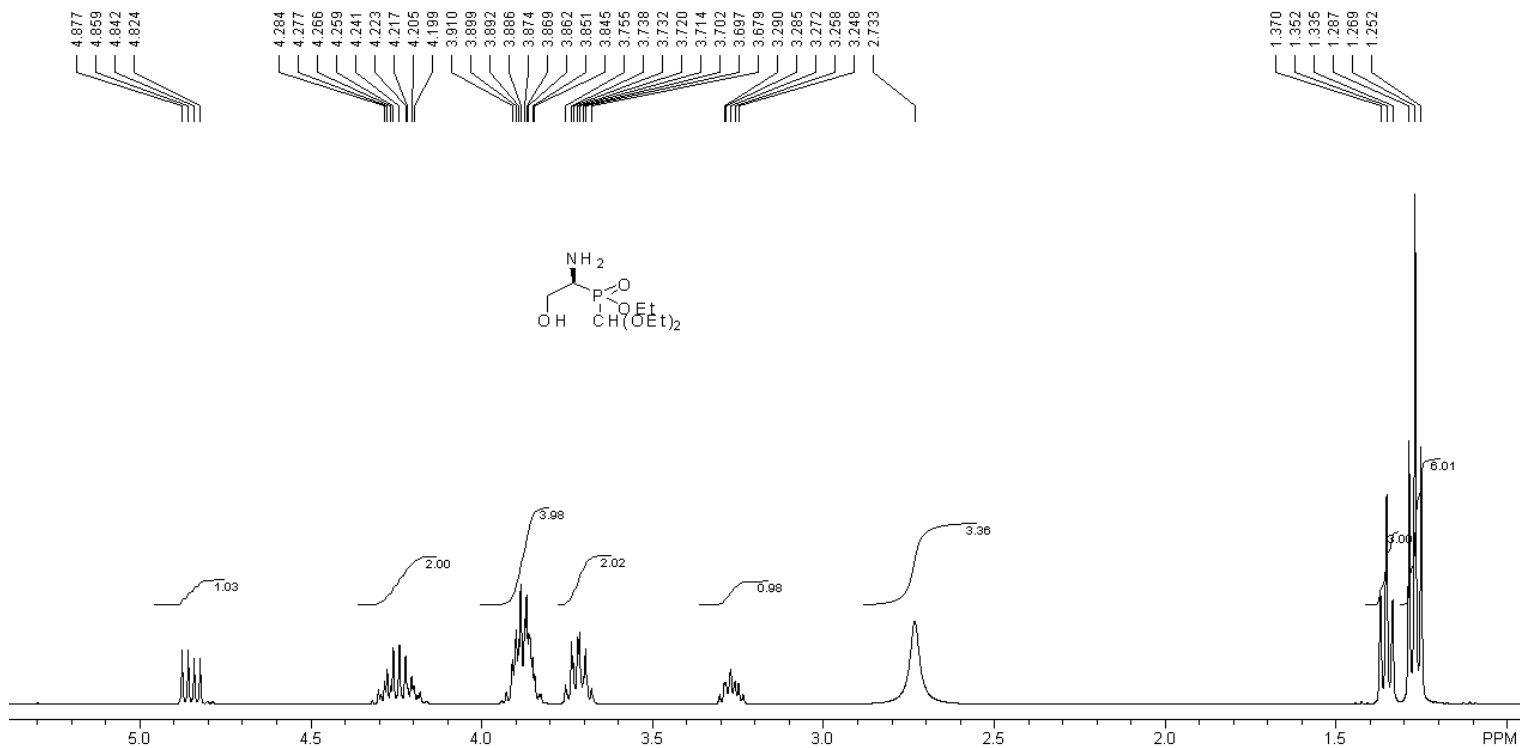
D:\software\nuts\DATA\2008119-11-18_11.1

16.03.07.625 +0800 nmr@EP-ZH107708
 USER: nmr
 SOLVENT: CDCl3
 Experiment: zgpg30
 Pulse length = 8.300 usec
 Recycle delay = 2.000 sec
 NA = 8
 Solvent = CDCl3
 PTS1d = 32768
 F1 = 161.971542 MHz
 F2 = 1.000000 MHz
 SW1 = 64102.56 Hz
 AT1 = 0.51 sec
 Hz per Pt1stD = 1.96 Hz
 SW2 = 1.00 Hz
 Hz per Pt2ndD = 1.00 Hz
 O1 = -8098.6865 Hz
 O2 = -1.0000 Hz
 LB1 = 1.00 Hz
 TP A = 0.00
 B = 0.00
 C = 0.00

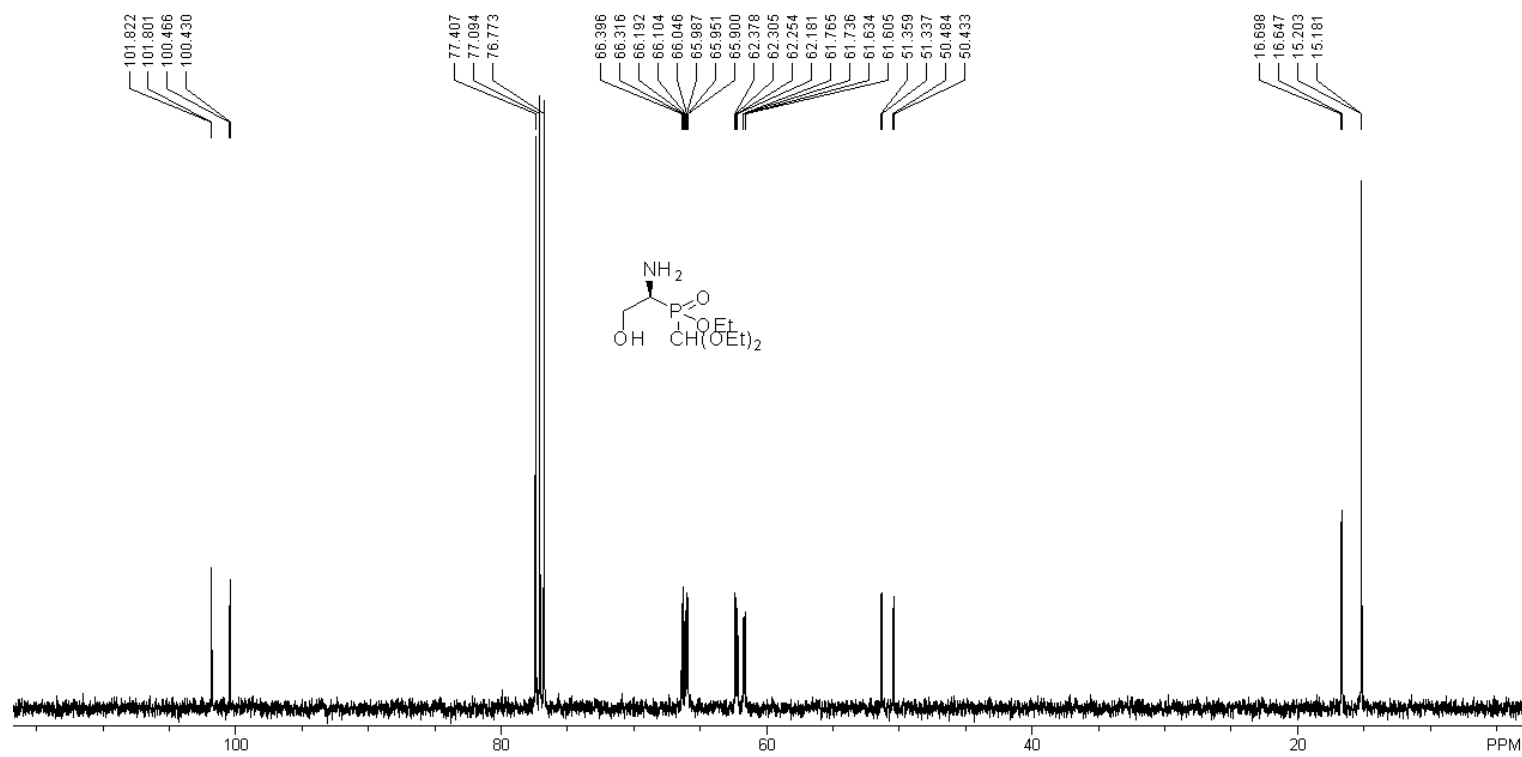


Compound (-)-3s:

¹H NMR:



¹³C NMR:



³¹P NMR:

D:\software\nuts\DATA\2008110-11-19H_11.1

21:09:06.828 +0800 nmri@EP.ZH107708

USER: nmri

SOLVENT: CDCl3

Experiment = zgpg30

Pulse length = 8.300 usec

Recycle delay = 2.000 sec

NA = 8

Solvent = CDCl3

PTS1d = 32768

F1 = 161.971542 MHz

F2 = 1.000000 MHz

SW1 = 64102.56 Hz

AT1 = 0.51 sec

Hz per Pt1s0 = 1.96 Hz

SW2 = 1.00 Hz

Hz per Pt2nd0 = 1.00 Hz

Q1 = -8098.6865 Hz

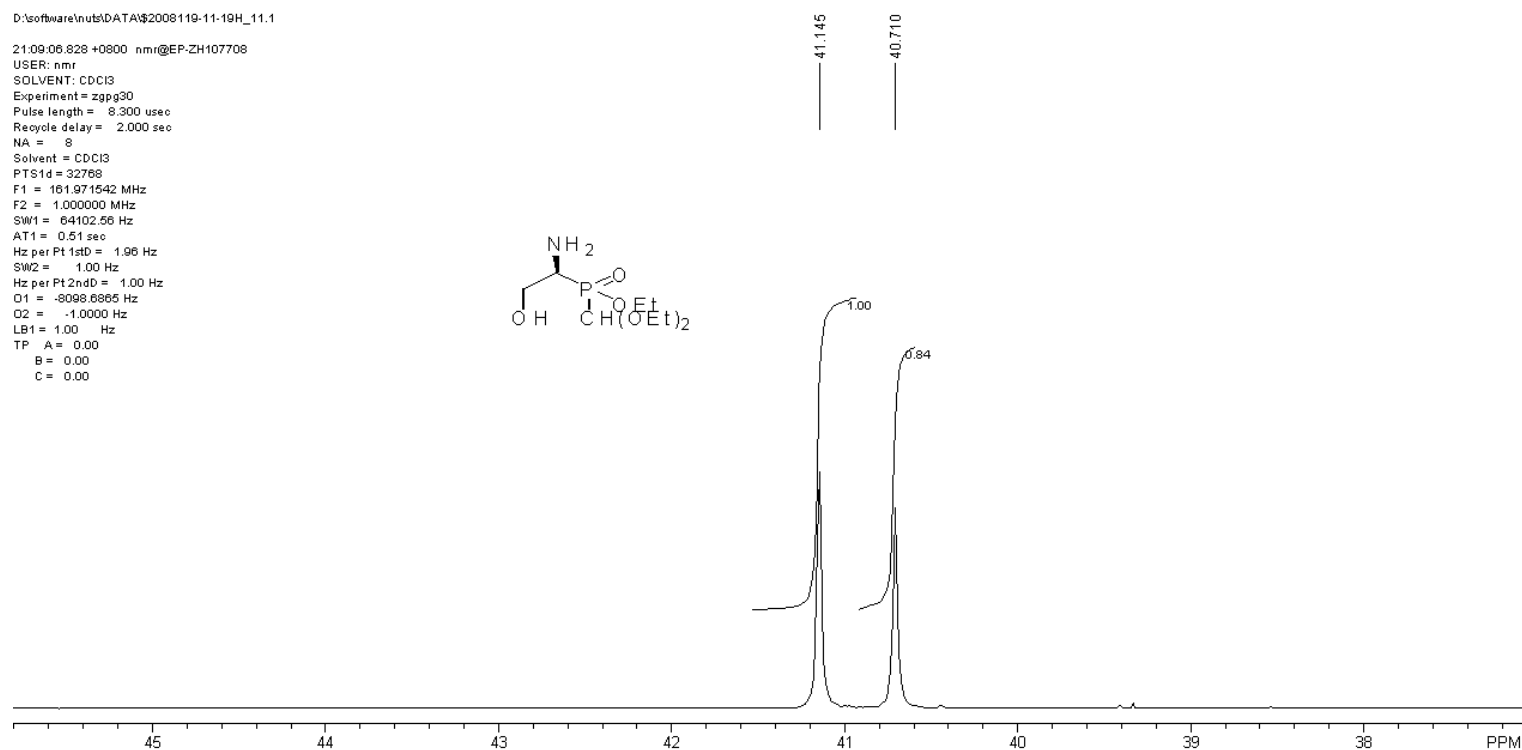
Q2 = -1.0000 Hz

LB1 = 1.00 Hz

TP A = 0.00

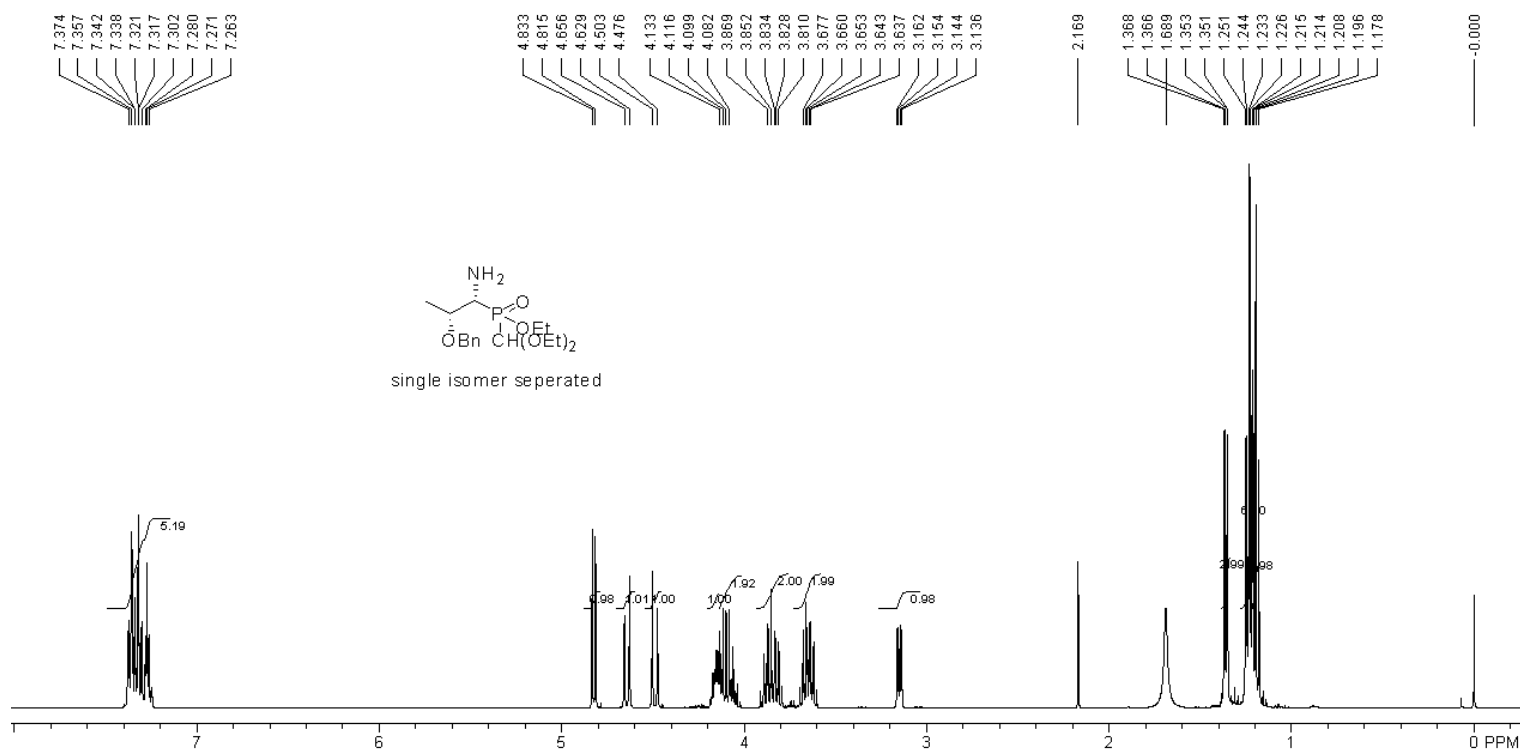
B = 0.00

C = 0.00

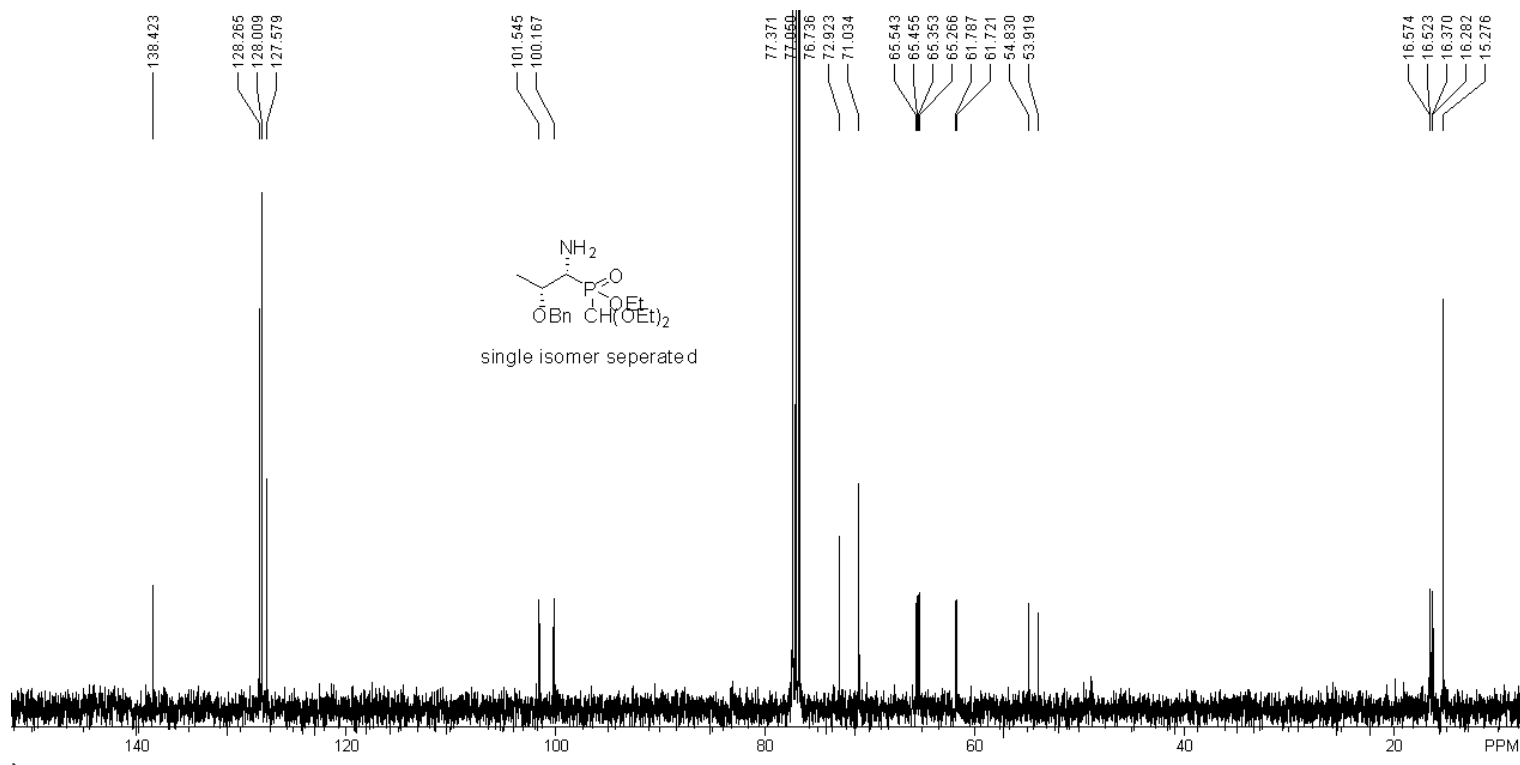


Compound 3m:

¹H NMR:



¹³C NMR:



³¹P NMR:

D:\software\nuts\DATA\2008119-9-92-1_21.1

09:52:18.000 +0800 nmr@EP-ZH107708

USER: nmr

SOLVENT: CDCl3

Experiment = zgpg30

Pulse length = 8.300 usec

Recycle delay = 2.000 sec

NA = 7

Solvent = CDCl3

PTS1d = 32768

F1 = 161.971542 MHz

F2 = 1.000000 MHz

SW1 = 64102.56 Hz

AT1 = 0.51 sec

Hz per Pt1stD = 1.96 Hz

SW2 = 1.00 Hz

Hz per Pt2ndD = 1.00 Hz

O1 = -8098.6865 Hz

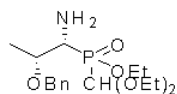
O2 = -1.0000 Hz

LB1 = 1.00 Hz

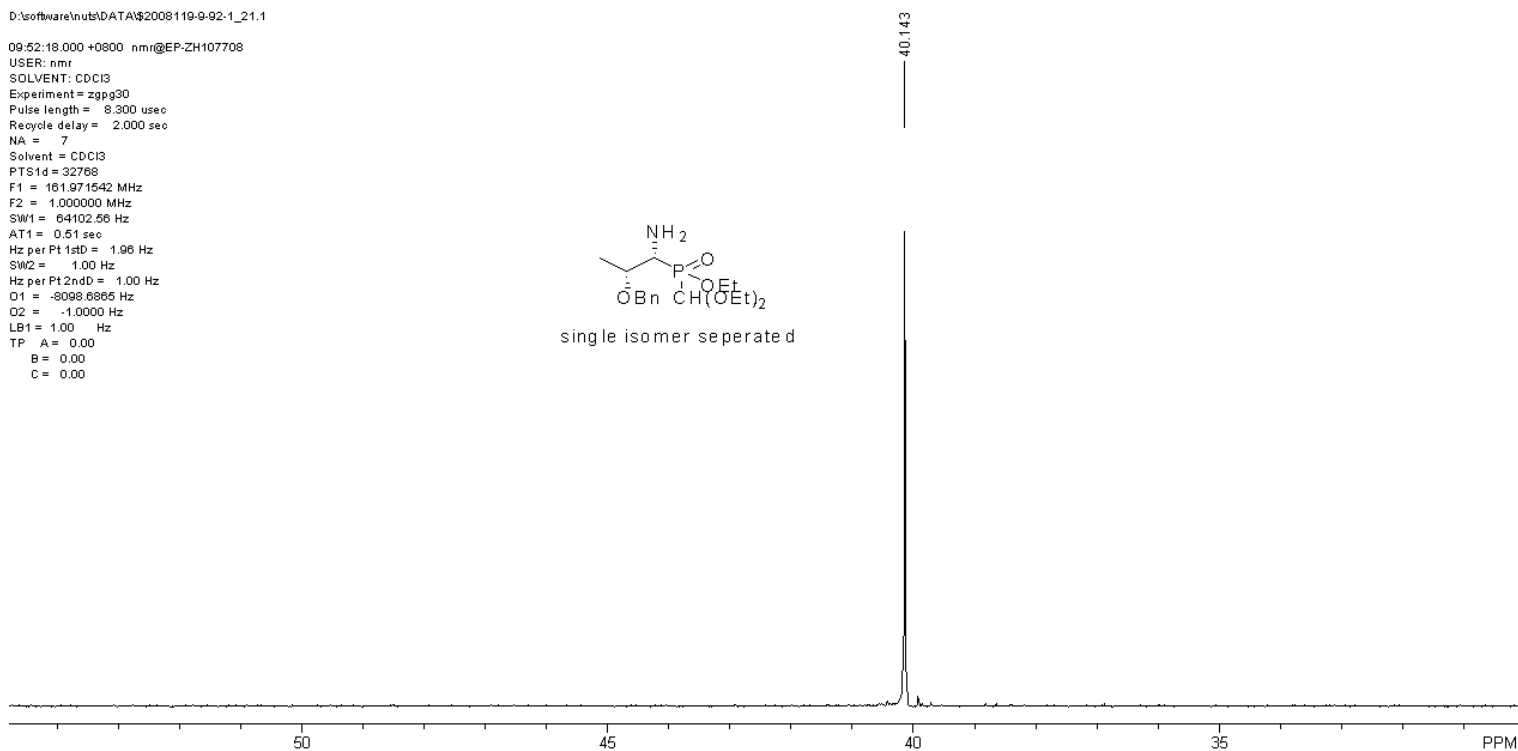
TP A = 0.00

B = 0.00

C = 0.00

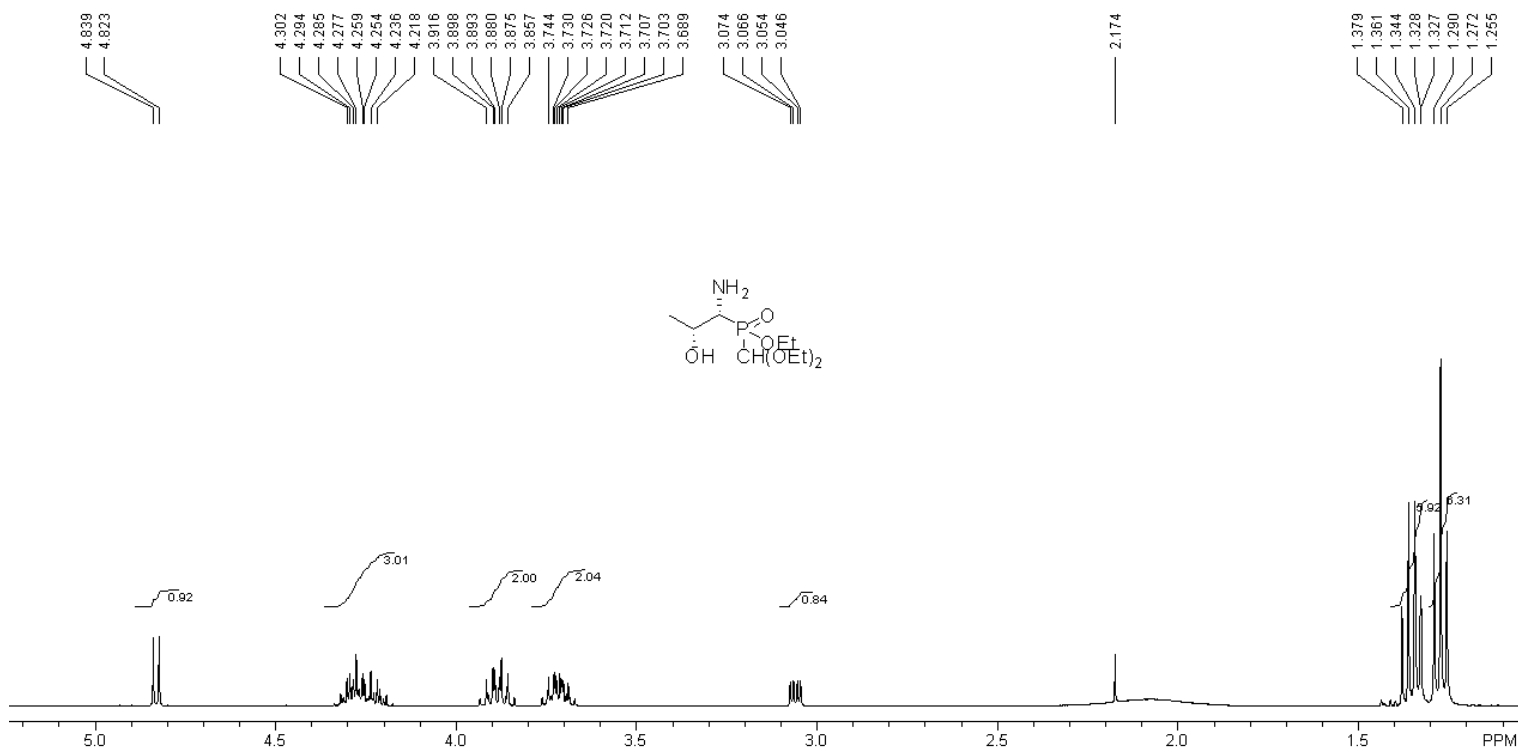


single isomer separated

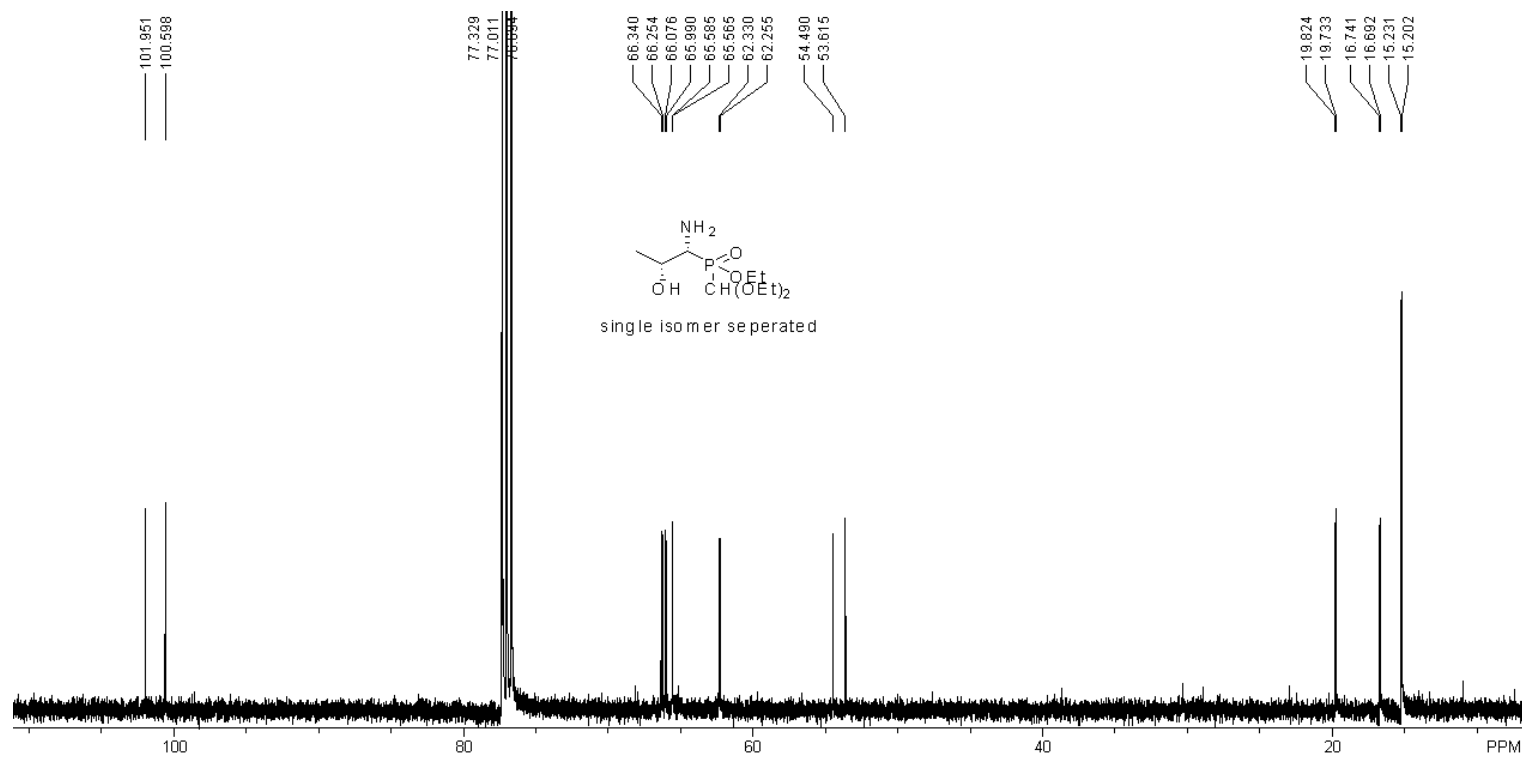


Compound 3t:

¹H NMR:



¹³C NMR:



³¹P NMR:

D:\software\nmr\DATA\2008119-9-98_11.1

09:15:15.234 +0800 nmri@EP.ZH107708

USER: nmri

SOLVENT: CDCl3

Experiment = zgpg30

Pulse length = 8.300 usec

Recycle delay = 2.000 sec

NA = 16

Solvent = CDCl3

PTS1d = 32768

F1 = 161.971542 MHz

F2 = 1.000000 MHz

SW1 = 64102.56 Hz

AT1 = 0.51 sec

Hz per Pt1s0 = 1.96 Hz

SW2 = 1.00 Hz

Hz per Pt2nd0 = 1.00 Hz

Q1 = -8098.6865 Hz

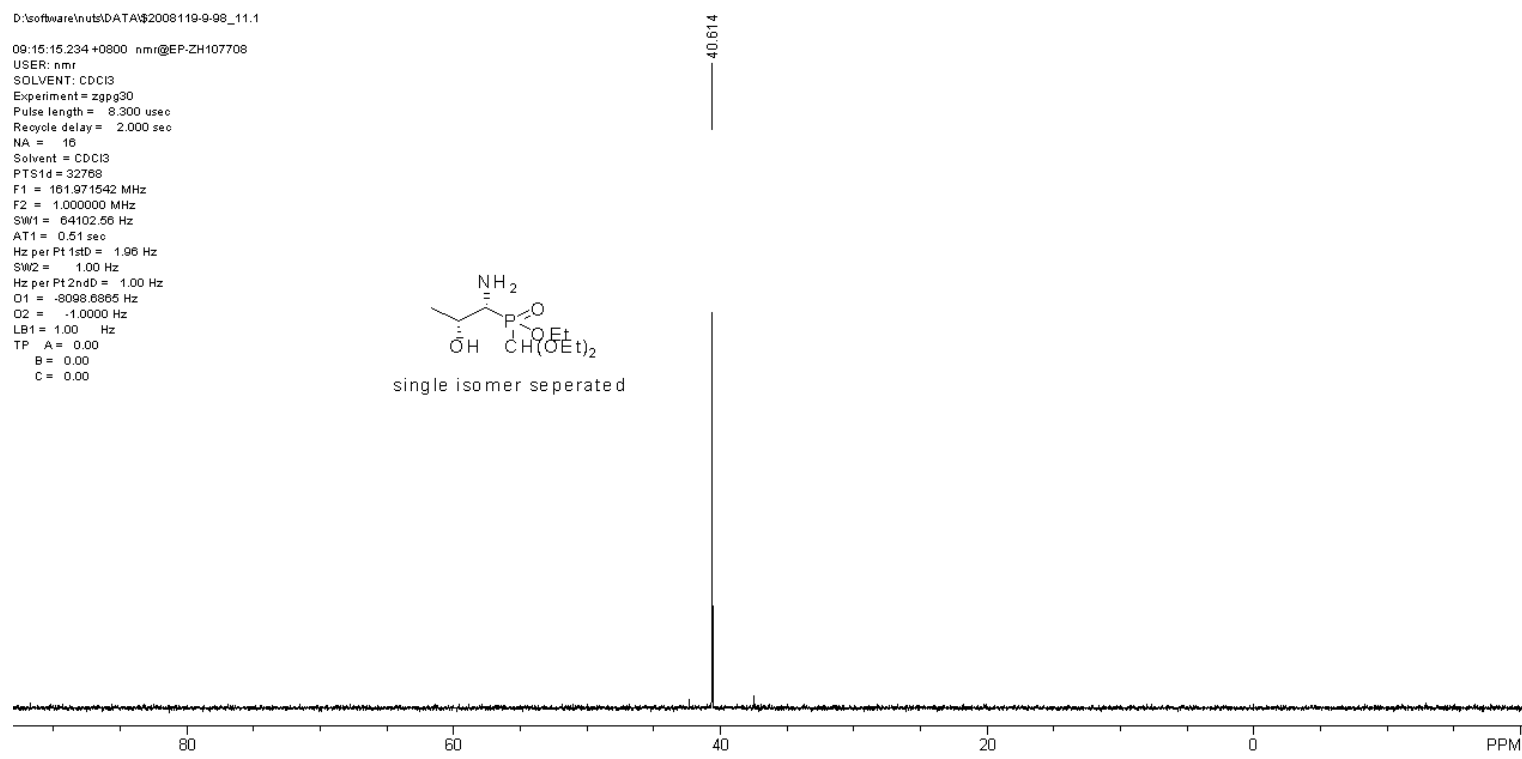
Q2 = -1.0000 Hz

LB1 = 1.00 Hz

TP A = 0.00

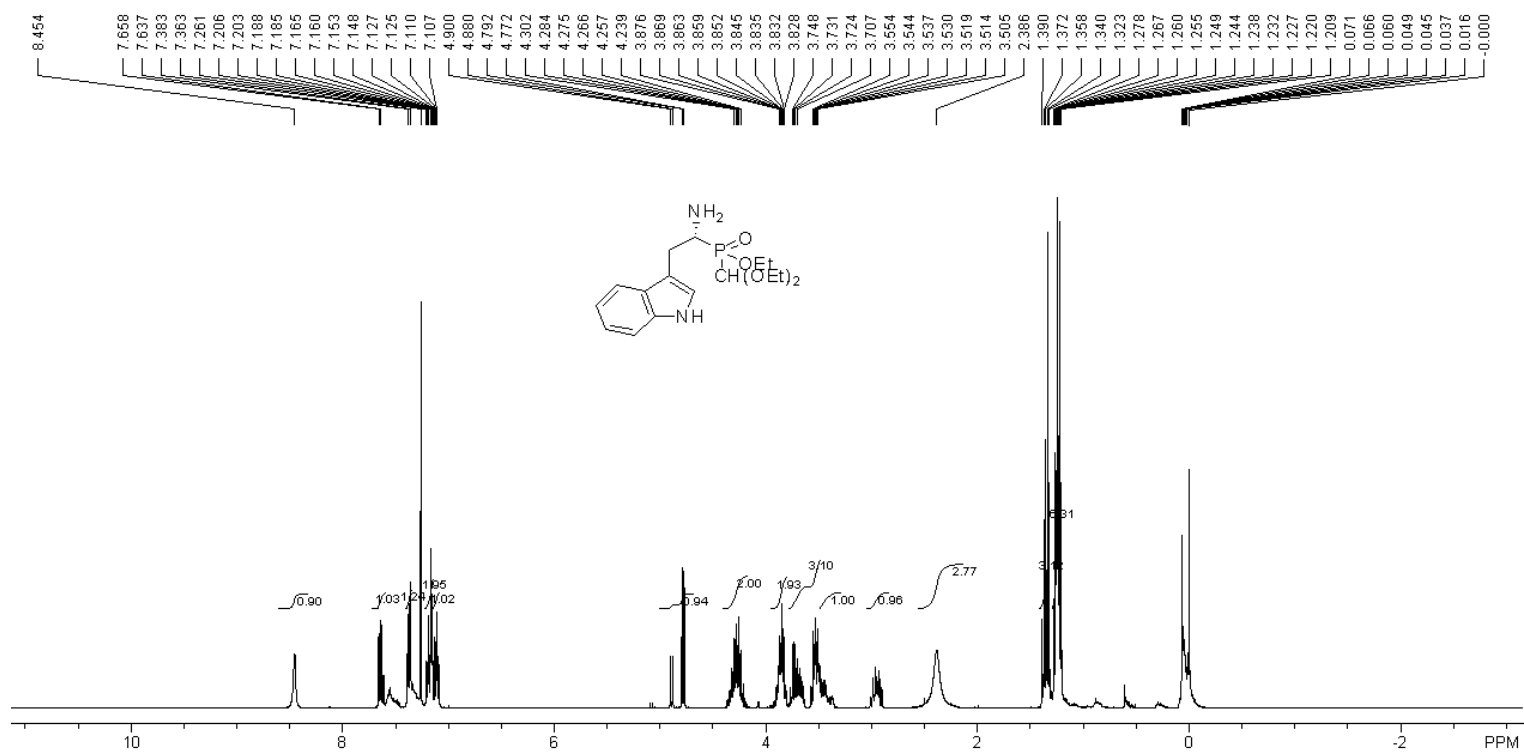
B = 0.00

C = 0.00

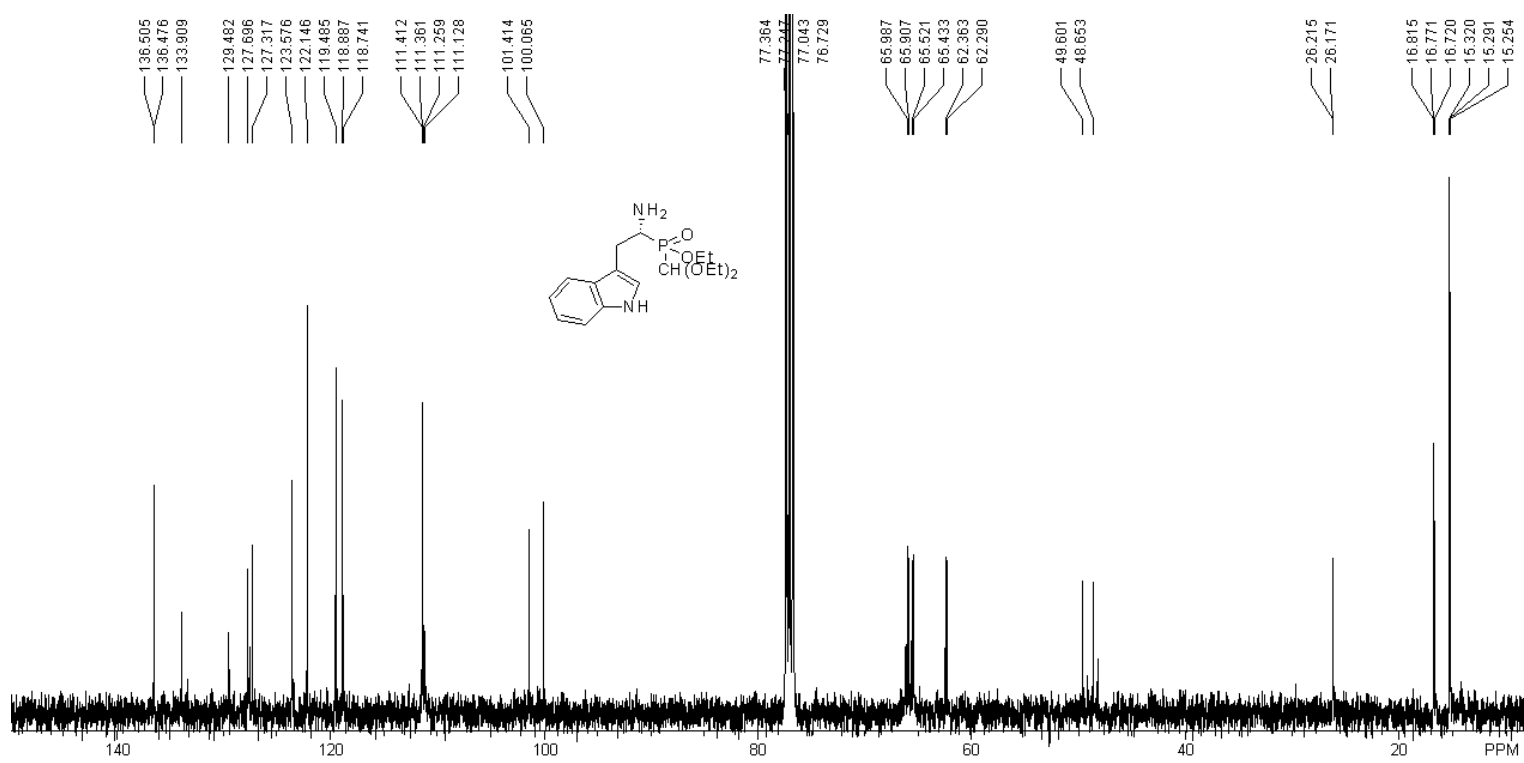


Compound 3n:

¹H NMR:



¹³C NMR:



³¹P NMR:

D:\software\nuts\DATA\2008119-10-26_11.1

09:08:20.140 +0800 nmri@EP-ZH107708

USER: nmri

SOLVENT: CDCl3

Experiment = zgpg30

Pulse length = 8.300 usec

Recycle delay = 2.000 sec

NA = 16

Solvent = CDCl3

PTS1d = 32768

F1 = 161.971542 MHz

F2 = 1.000000 MHz

SW1 = 64102.56 Hz

AT1 = 0.51 sec

Hz per Pt1stD = 1.96 Hz

SW2 = 1.00 Hz

Hz per Pt2ndD = 1.00 Hz

O1 = -8098.6865 Hz

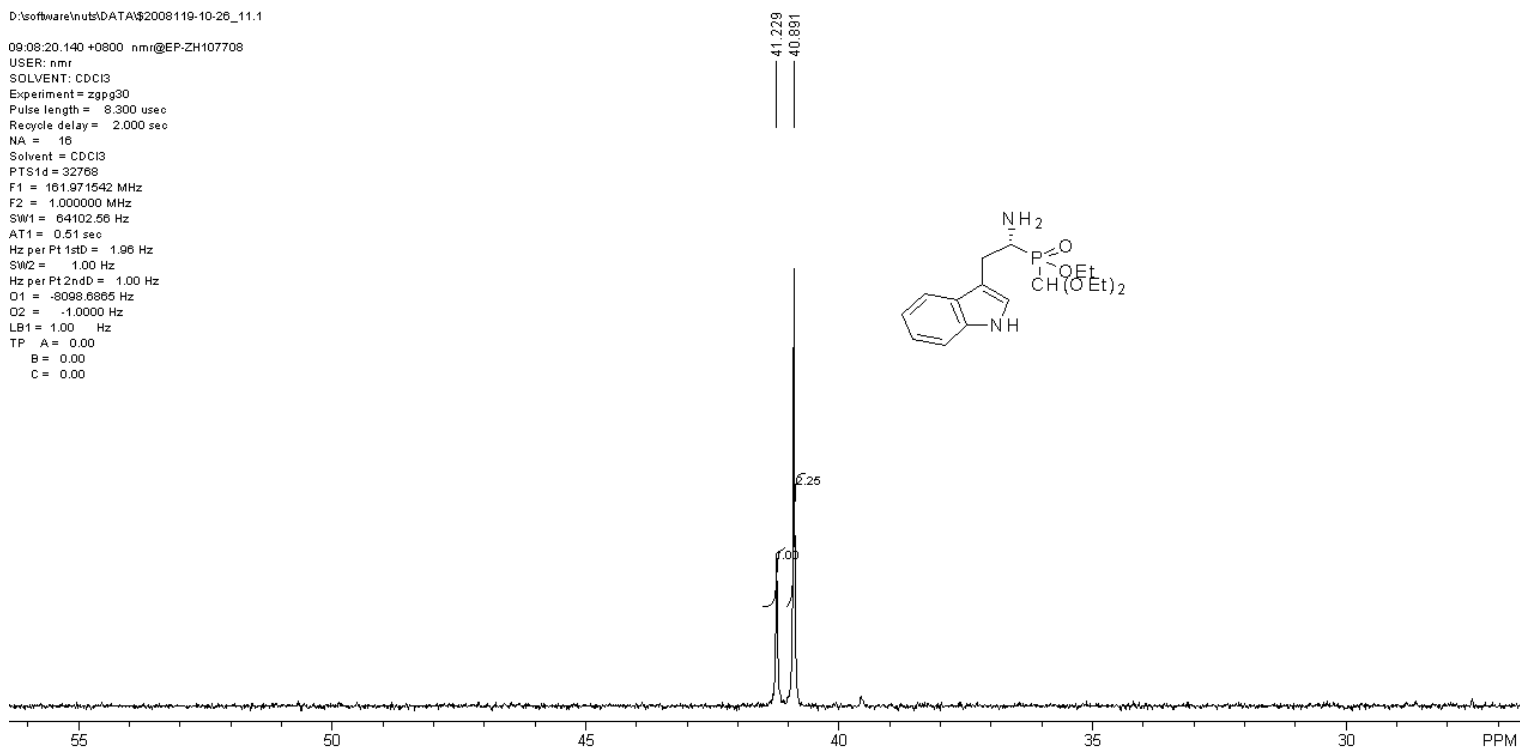
O2 = -1.0000 Hz

LB1 = 1.00 Hz

TP A = 0.00

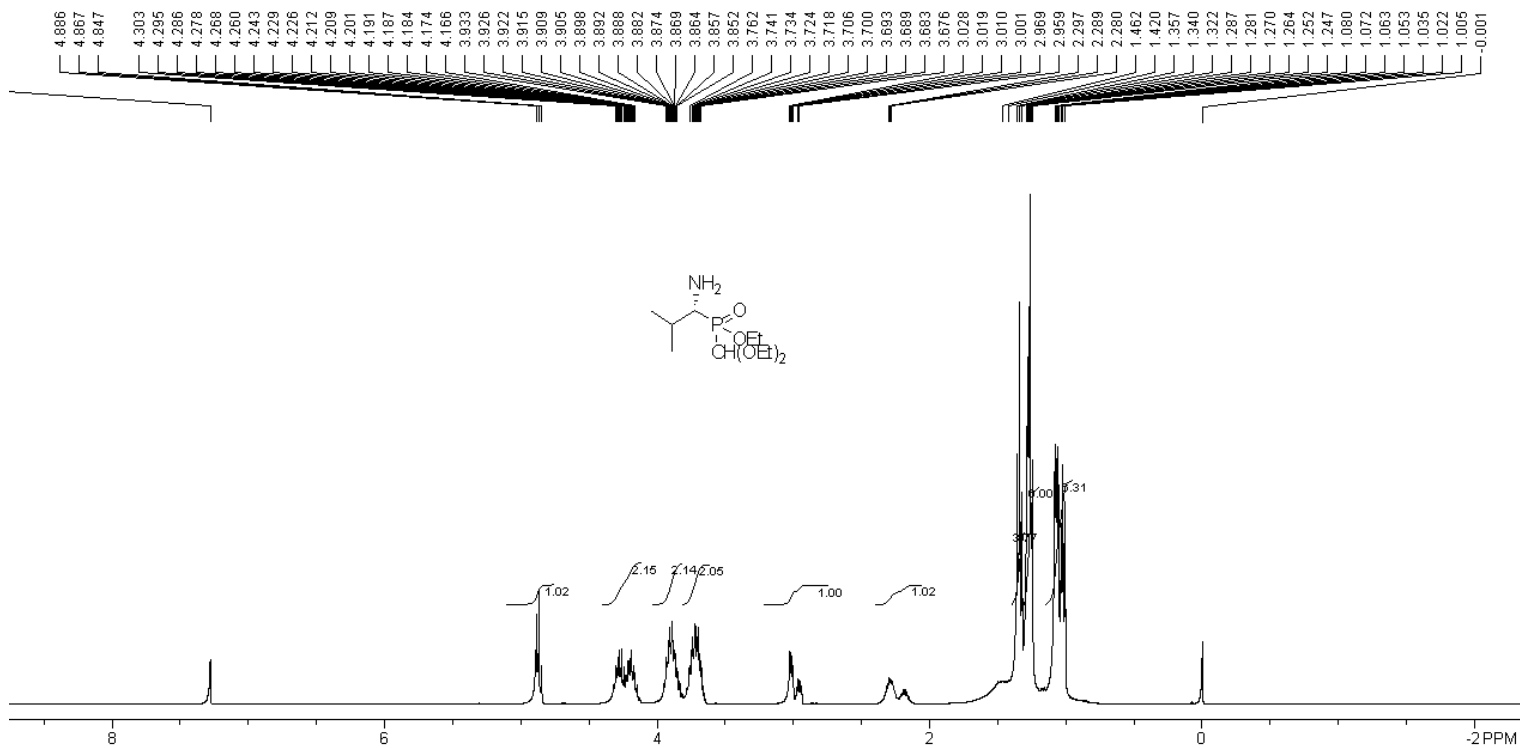
B = 0.00

C = 0.00

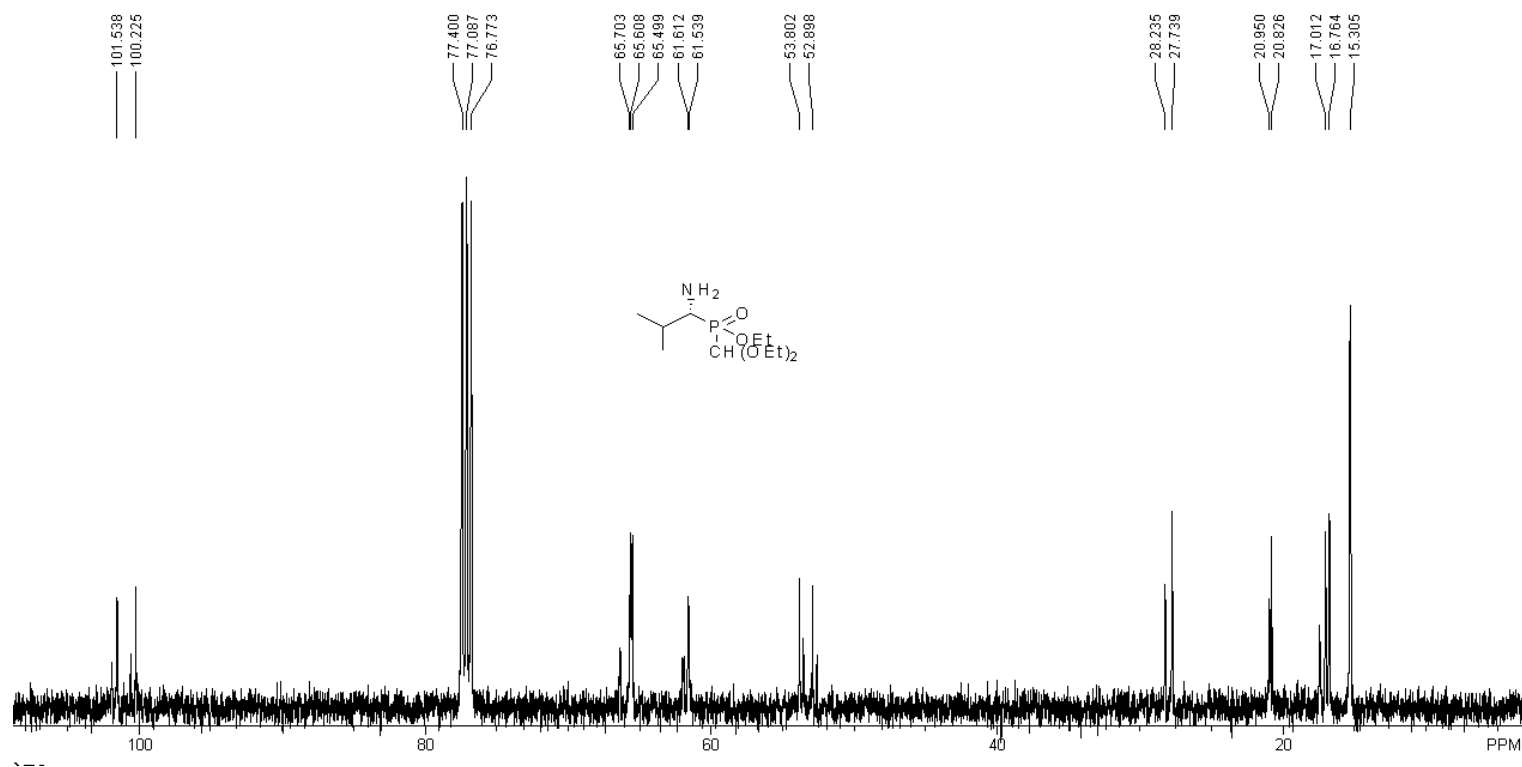


Compound 3p:

¹H NMR:



¹³C NMR:



³¹P NMR:

D:\software\nuts\DATA\补充数据_12-59-P_1

15:26:33.531 +0800 nmri@EP.ZH107708

USER: nmri

SOLVENT: CDCl₃

Experiment = zgpg30

Pulse length = 8.300 usec

Recycle delay = 2.000 sec

NA = 16

Solvent = CDCl₃

PTSD = 32768

F1 = 161.971542 MHz

F2 = 1.000000 MHz

SWH = 64102.56 Hz

AT1 = 0.51 sec

Hz per Pt1s0 = 1.96 Hz

SW2 = 1.00 Hz

Hz per Pt2nd = 1.00 Hz

Q1 = -8098.6965 Hz

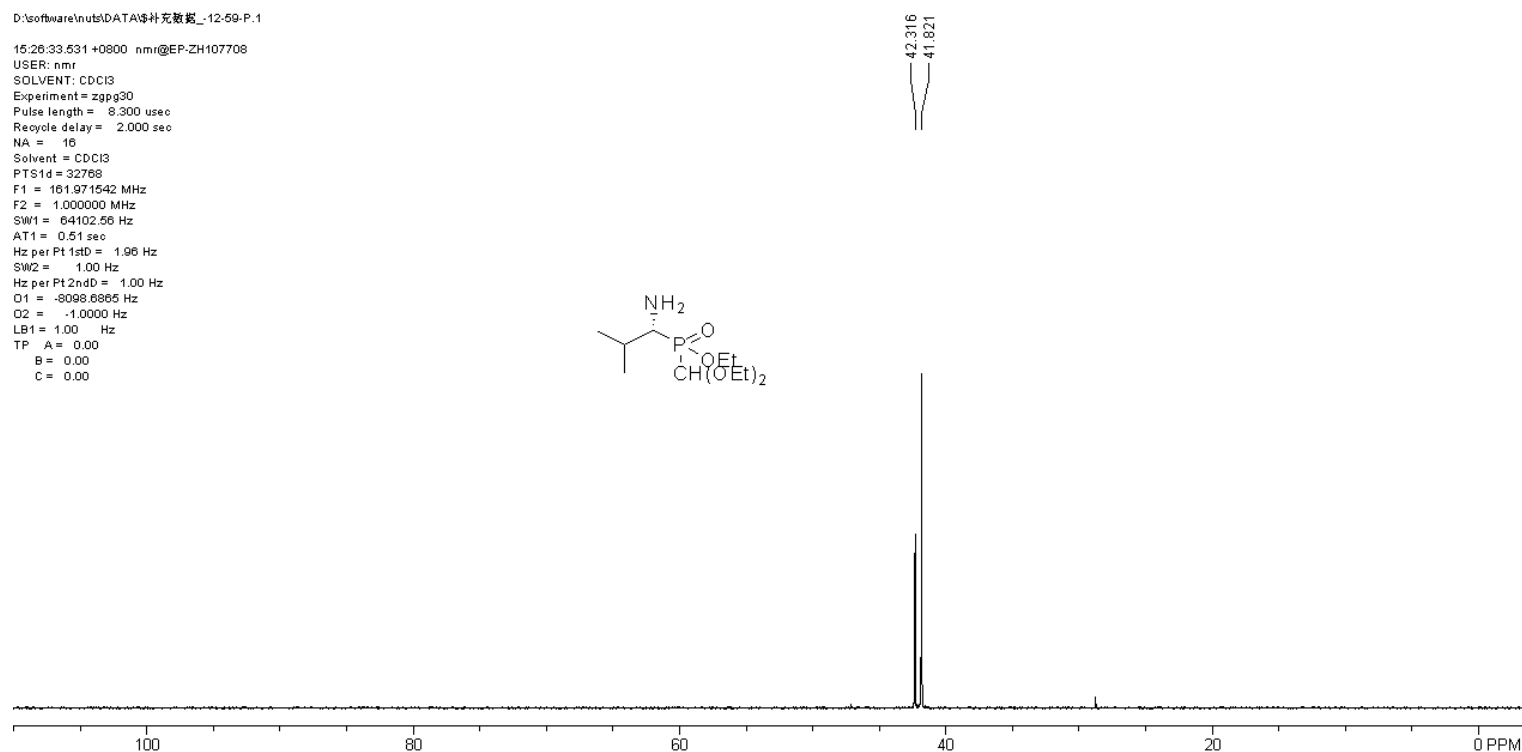
Q2 = -1.0000 Hz

LB1 = 1.00 Hz

TP A = 0.00

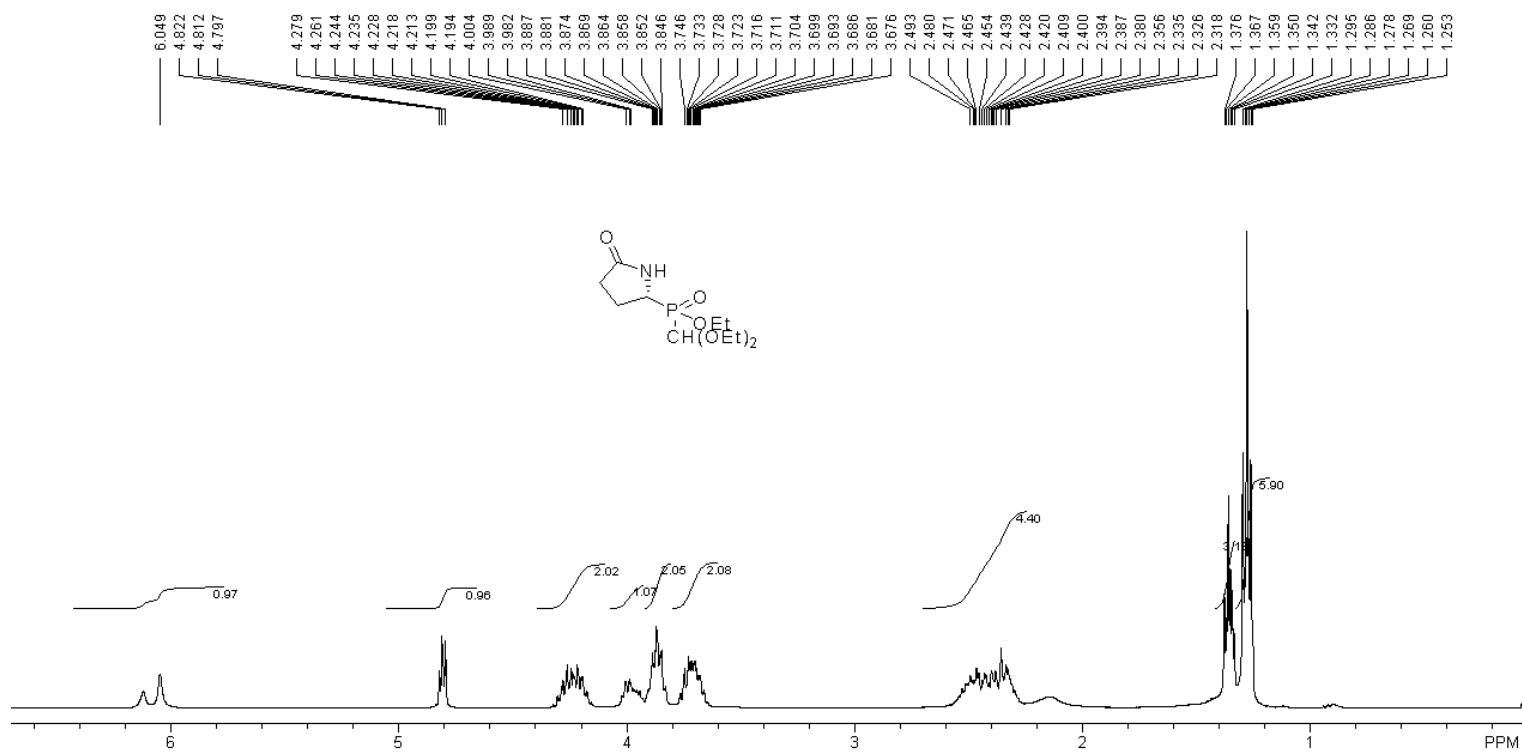
B = 0.00

C = 0.00

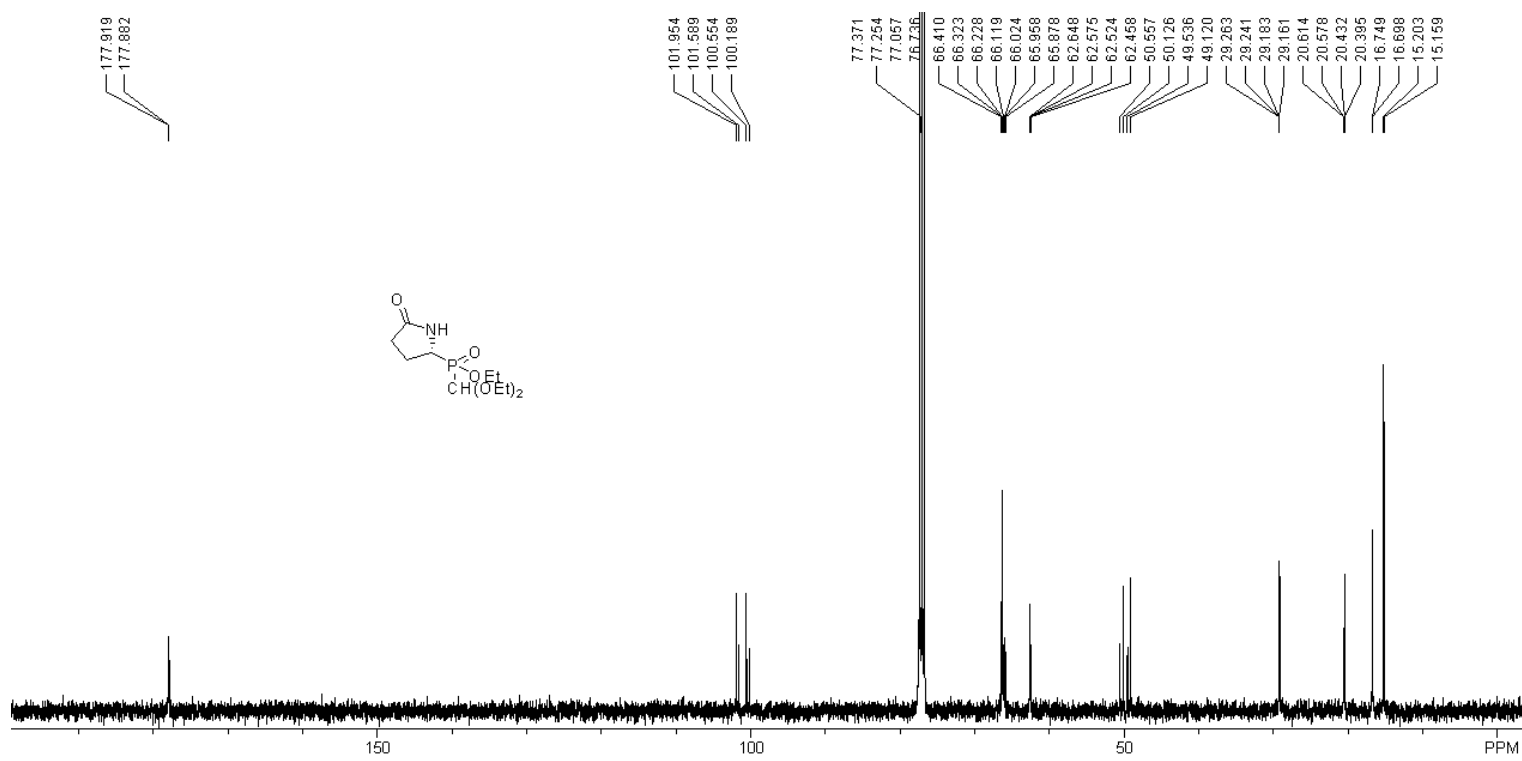


Compound 3q:

¹H NMR:

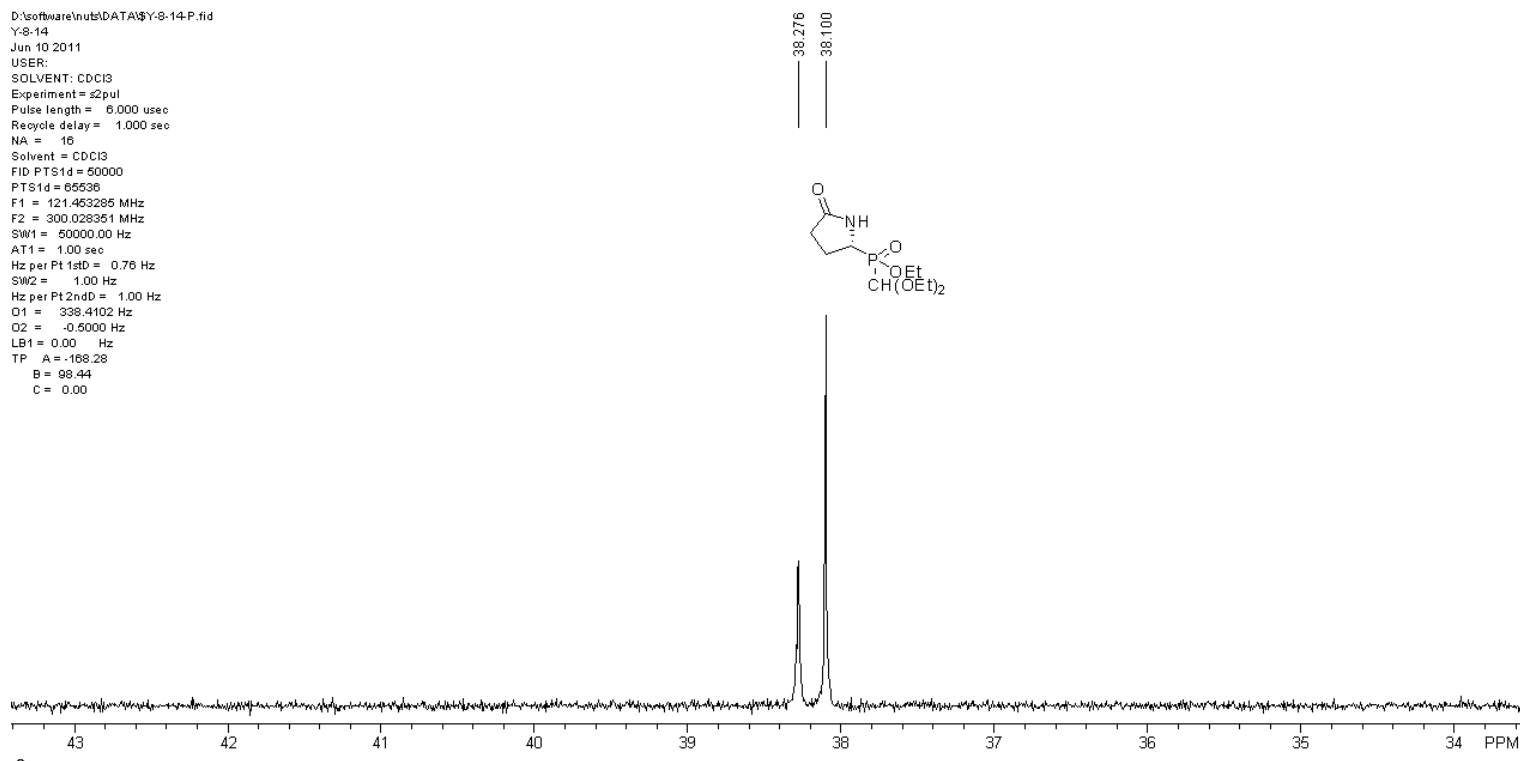


¹³C NMR:



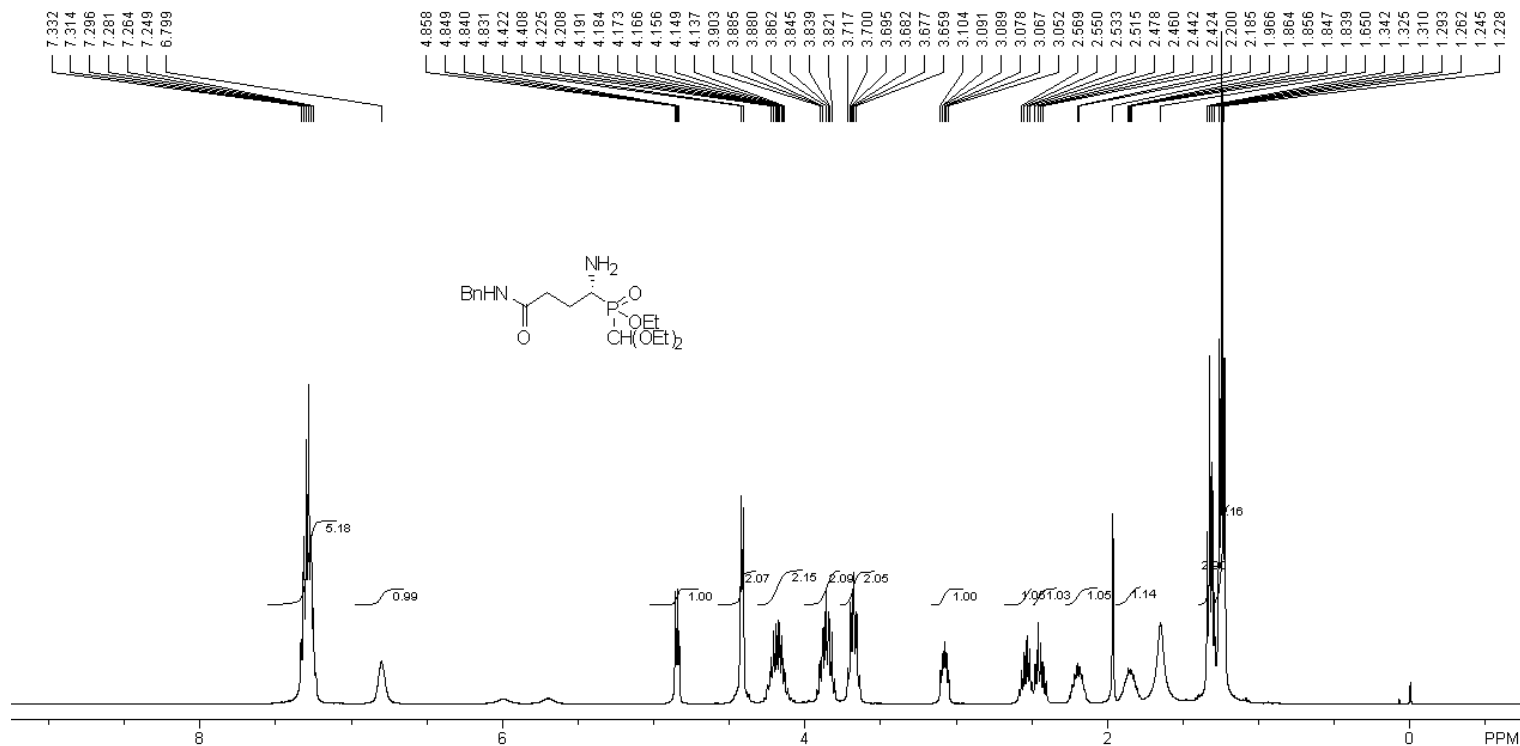
³¹P NMR:

D:\software\nuts\DATA\Y-8-14-P.fid
 Y-8-14
 Jun 10 2011
 USER:
 SOLVENT: CDCl3
 Experiment = s2pul
 Pulse length = 6.000 usec
 Recycle delay = 1.000 sec
 NA = 16
 Solvent = CDCl3
 FID PTS1d = 60000
 PTS1d = 65536
 F1 = 121.463285 MHz
 F2 = 300.028351 MHz
 SW1 = 50000.00 Hz
 AT1 = 1.00 sec
 Hz per Pt1st0 = 0.76 Hz
 SW2 = 1.00 Hz
 Hz per Pt2nd0 = 1.00 Hz
 O1 = 338.4102 Hz
 O2 = -0.6000 Hz
 LB1 = 0.00 Hz
 TP A = -188.28
 B = 98.44
 C = 0.00

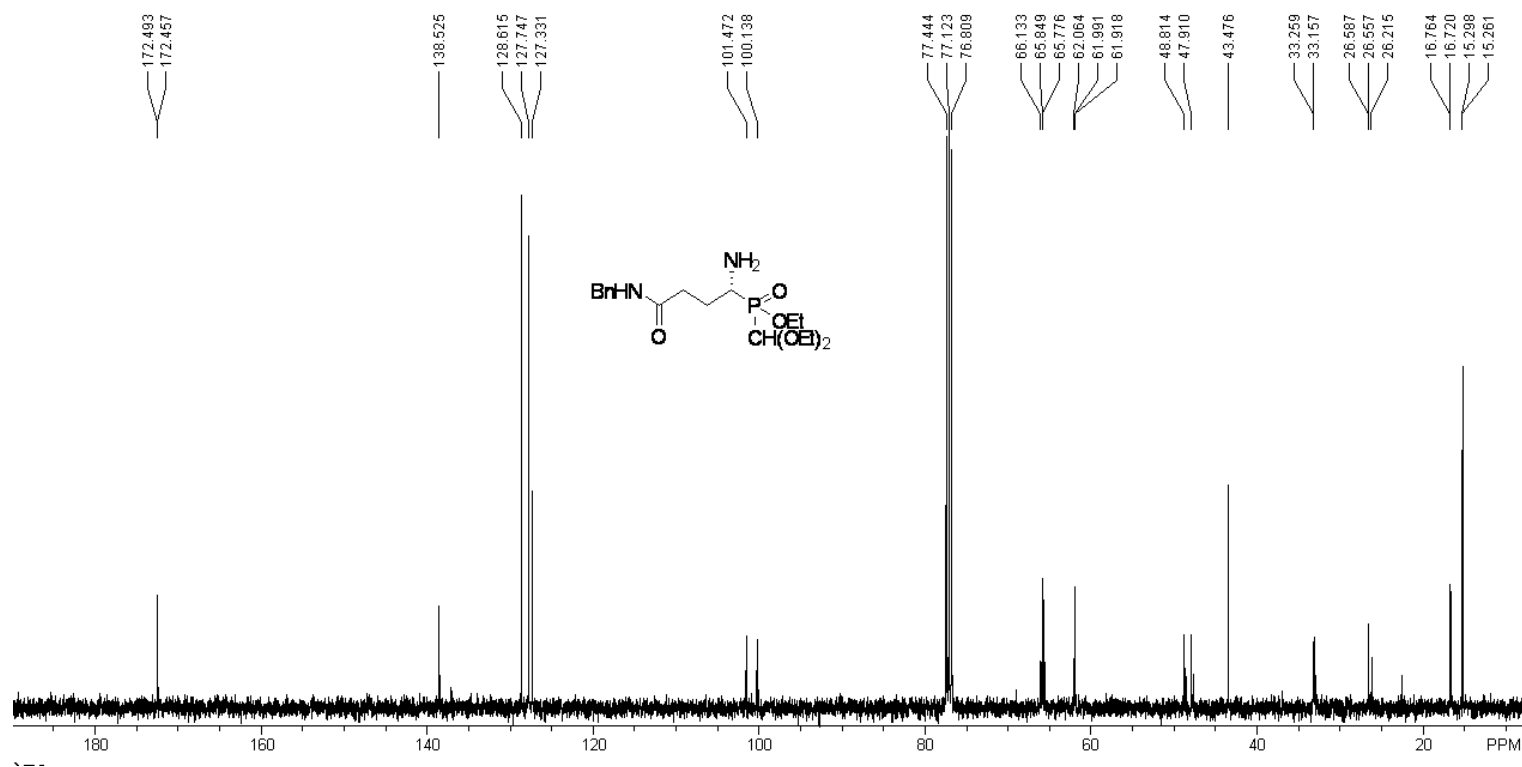


Compound 3r:

¹H NMR:



¹³C NMR:



³¹P NMR:

D:\software\nuts\DATA\补充数据_8-59-P.1

15:05:48.984 +0800 nmr@EP.ZH107708

USER: nmr

SOLVENT: CDCl3

Experiment = zgpg30

Pulse length = 8.300 usec

Recycle delay = 2.000 sec

NA = 8

Solvent = CDCl3

PTSD = 32768

F1 = 161.971542 MHz

F2 = 1.000000 MHz

SWH = 64102.56 Hz

AT1 = 0.51 sec

Hz per Pt1s0 = 1.96 Hz

SW2 = 1.00 Hz

Hz per Pt2nd0 = 1.00 Hz

O1 = -8098.6865 Hz

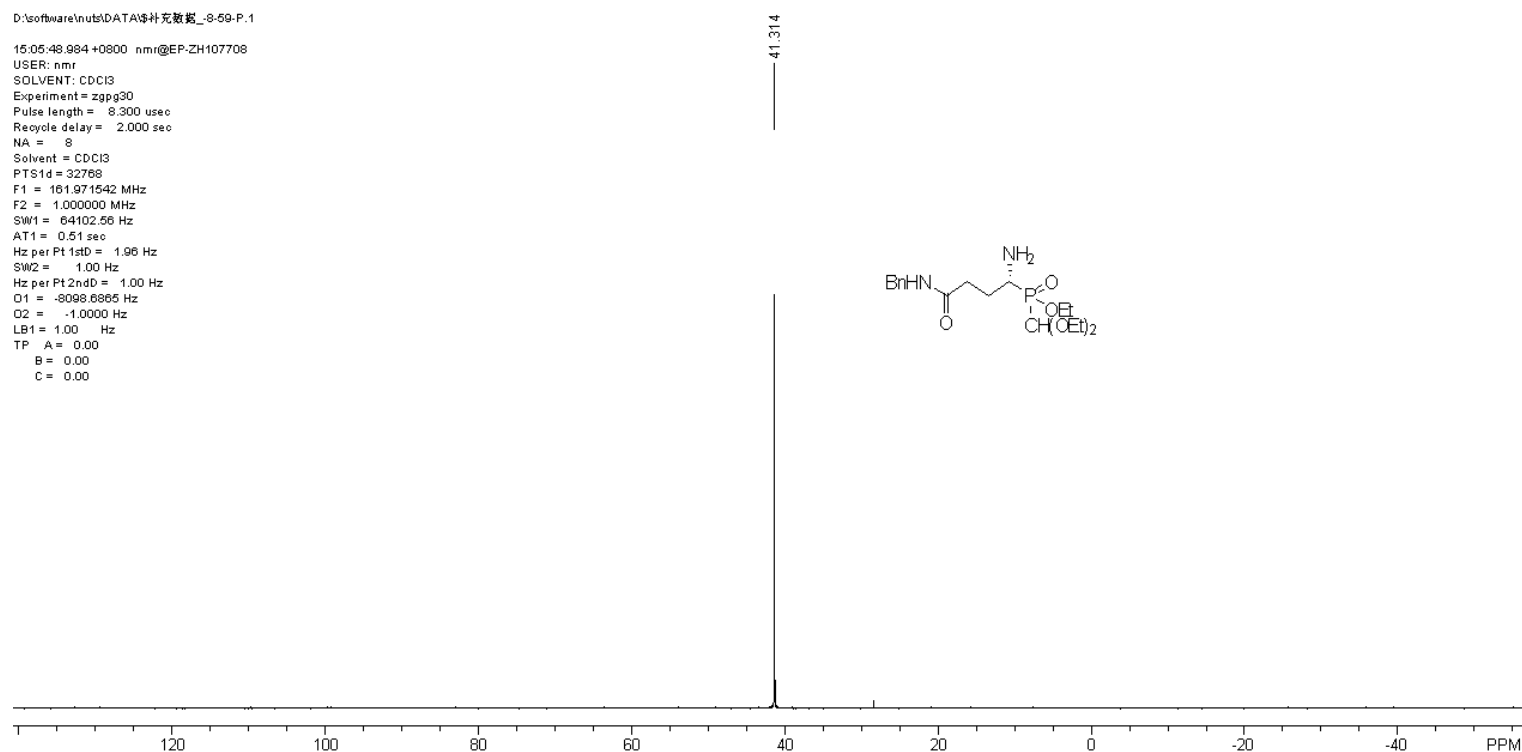
O2 = -1.0000 Hz

LB1 = 1.00 Hz

TP A = 0.00

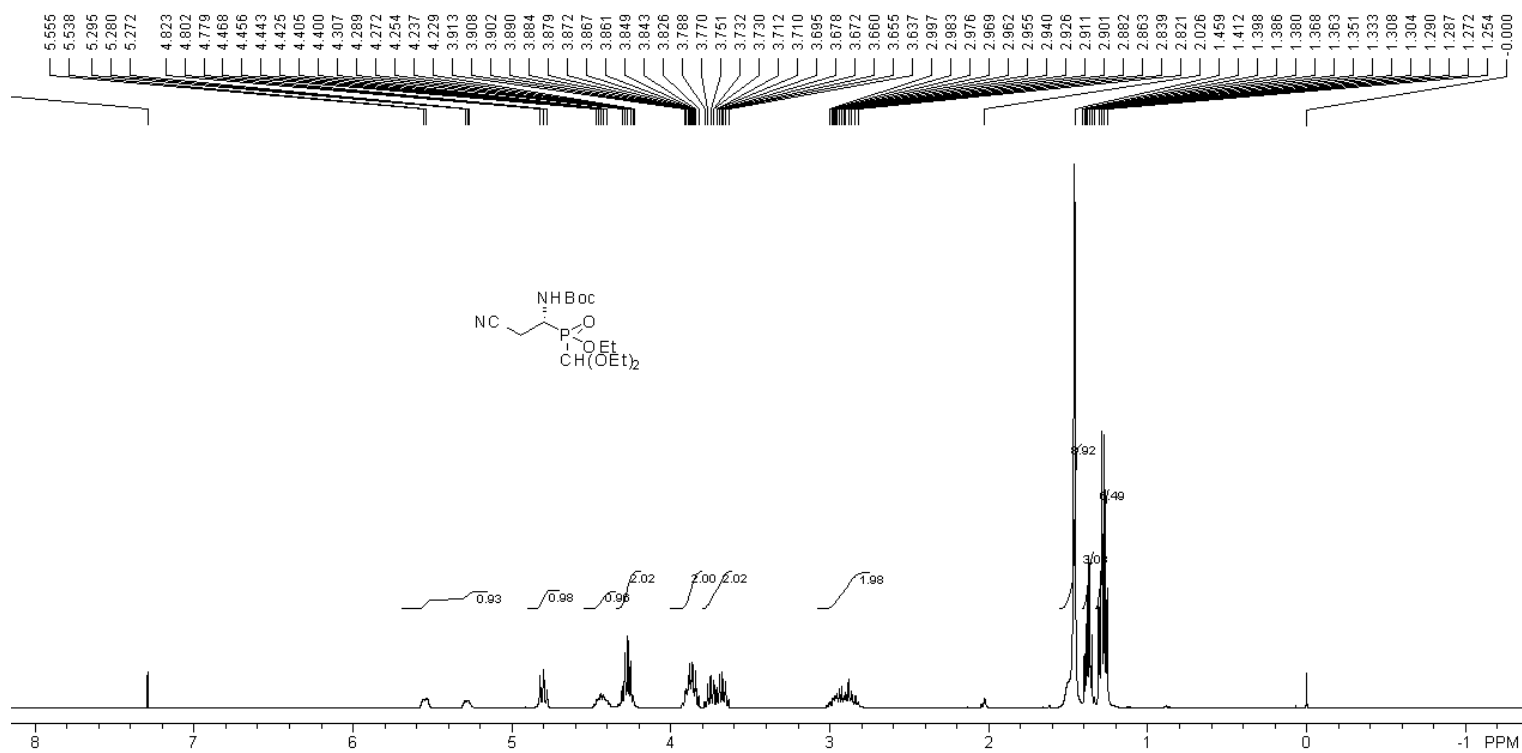
B = 0.00

C = 0.00

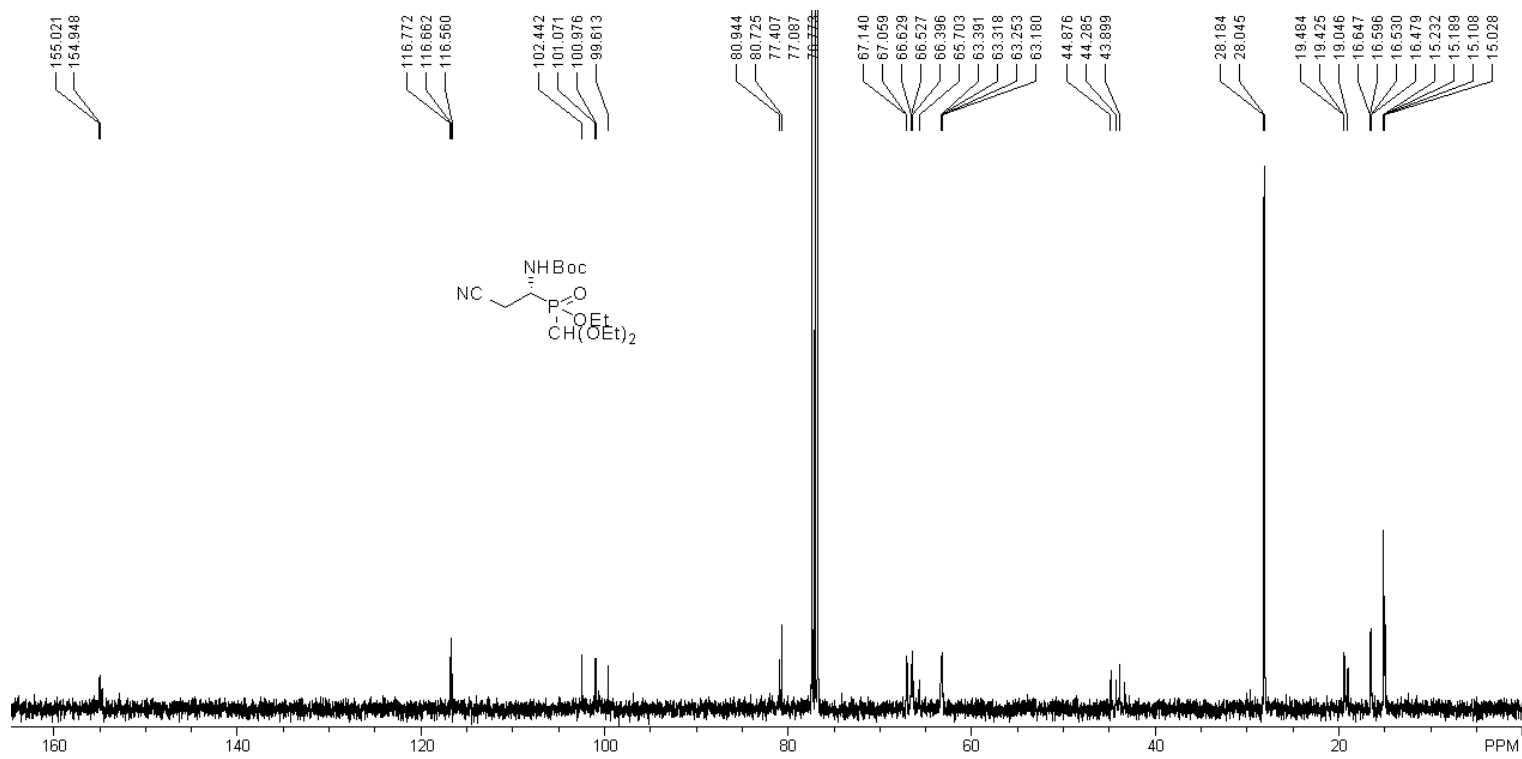


Compound 9a:

¹H NMR:



¹³C NMR:



³¹P NMR:

D:\software\nuts\DATA\2008119-10-6c_11.1

22:49:11.500 +0800 nmr@EP-ZH107708

USER: nmr

SOLVENT: CDCl3

Experiment = zgpg30

Pulse length = 8.300 usec

Recycle delay = 2.000 sec

NA = 9

Solvent = CDCl3

PTS1d = 32768

F1 = 161.971542 MHz

F2 = 1.000000 MHz

SWH = 64102.56 Hz

AT1 = 0.51 sec

Hz per Pt1stD = 1.96 Hz

SW2 = 1.00 Hz

Hz per Pt2ndD = 1.00 Hz

O1 = -8098.6865 Hz

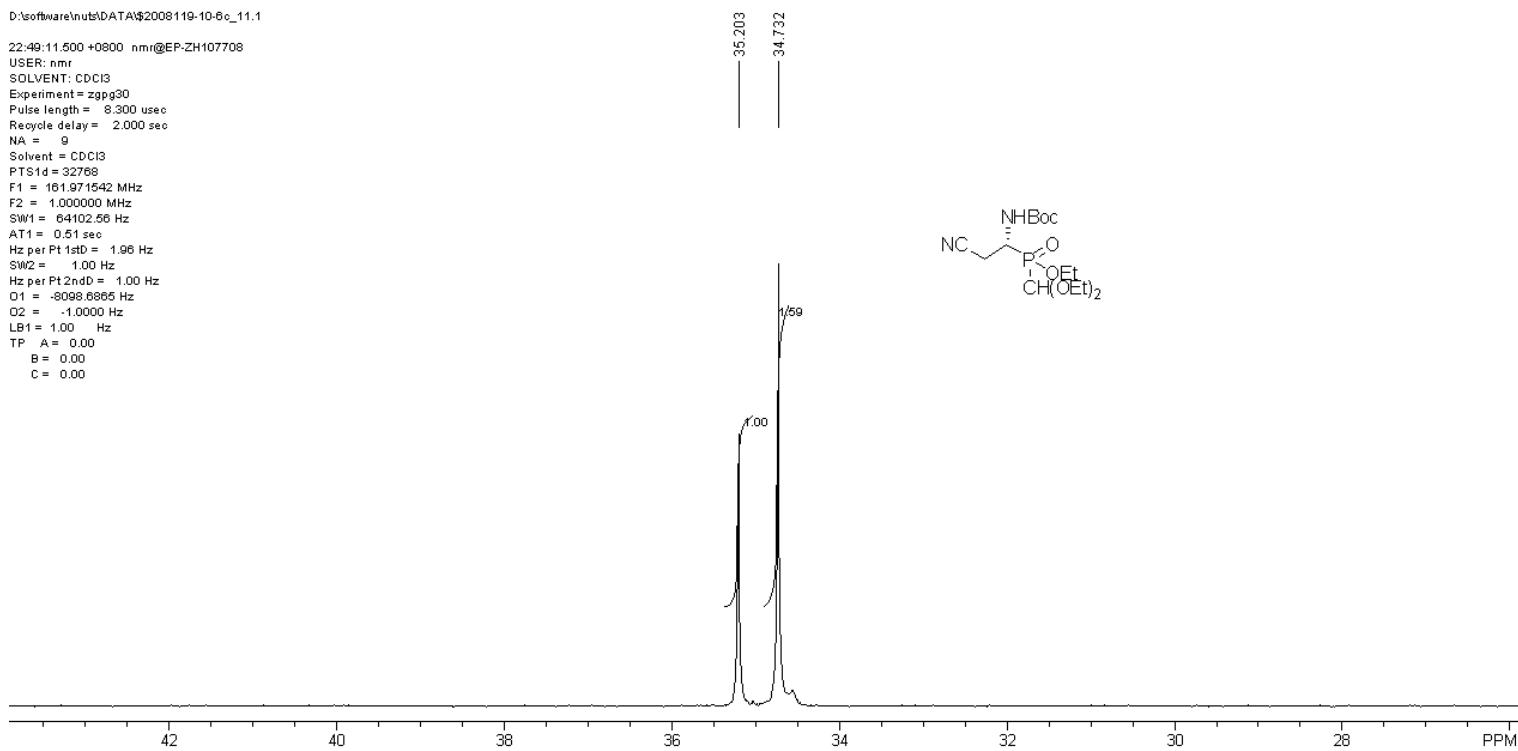
O2 = -1.0000 Hz

LB1 = 1.00 Hz

TP A = 0.00

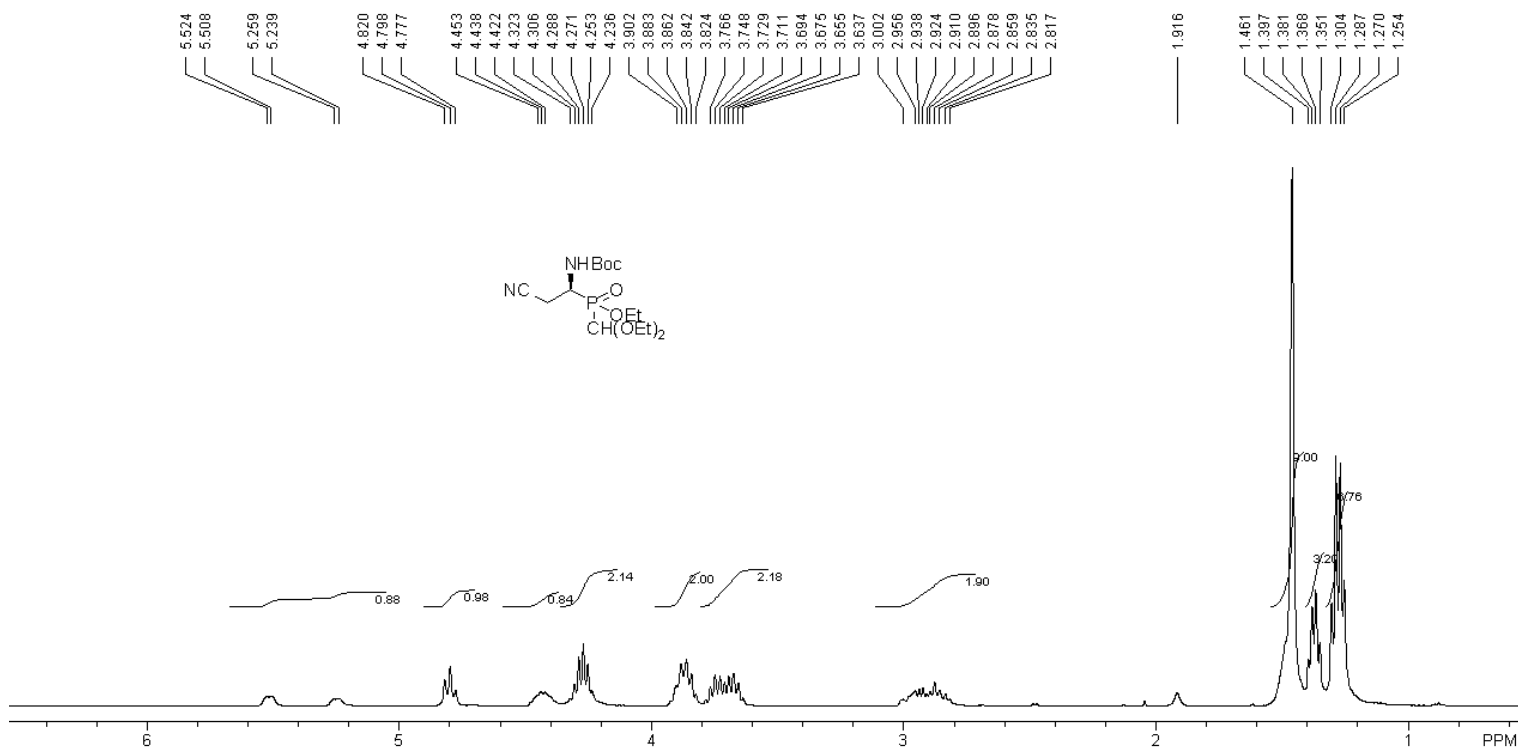
B = 0.00

C = 0.00

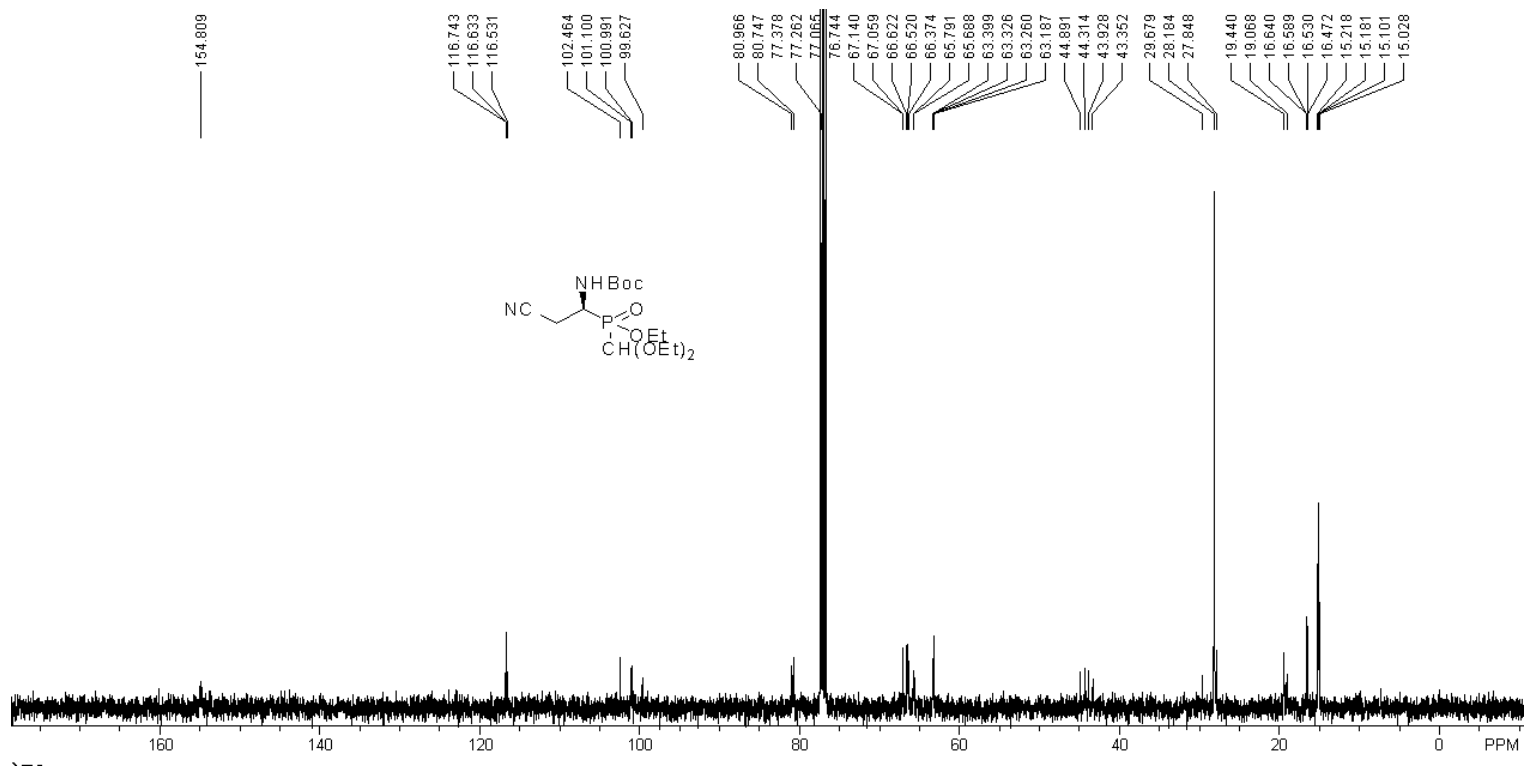


Compound (-)-9a:

¹H NMR:



¹³C NMR:



³¹P NMR:

D:\software\nuts\DATA\2008110-11-33pH_11.1

23:02:06.546 +0800 nmri@EP.ZH107708

USER: nmri

SOLVENT: CDCl3

Experiment = zgpg30

Pulse length = 8.300 usec

Recycle delay = 2.000 sec

NA = 8

Solvent = CDCl3

PTS1d = 32768

F1 = 161.971542 MHz

F2 = 1.000000 MHz

SWH = 64102.56 Hz

AT1 = 0.51 sec

Hz per Pt1s0 = 1.96 Hz

SW2 = 1.00 Hz

Hz per Pt2nd0 = 1.00 Hz

Q1 = -8098.6865 Hz

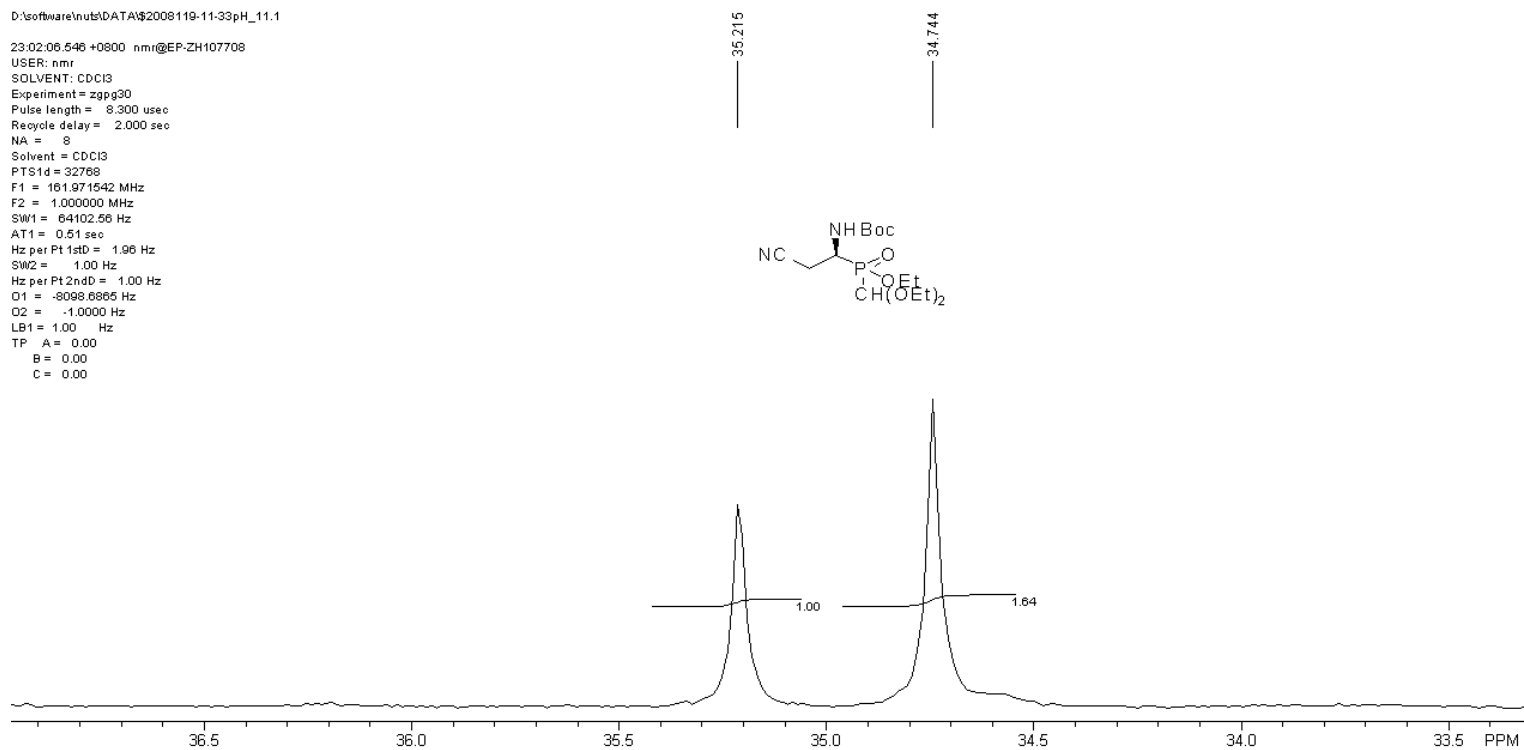
Q2 = -1.0000 Hz

LB1 = 1.00 Hz

TP A = 0.00

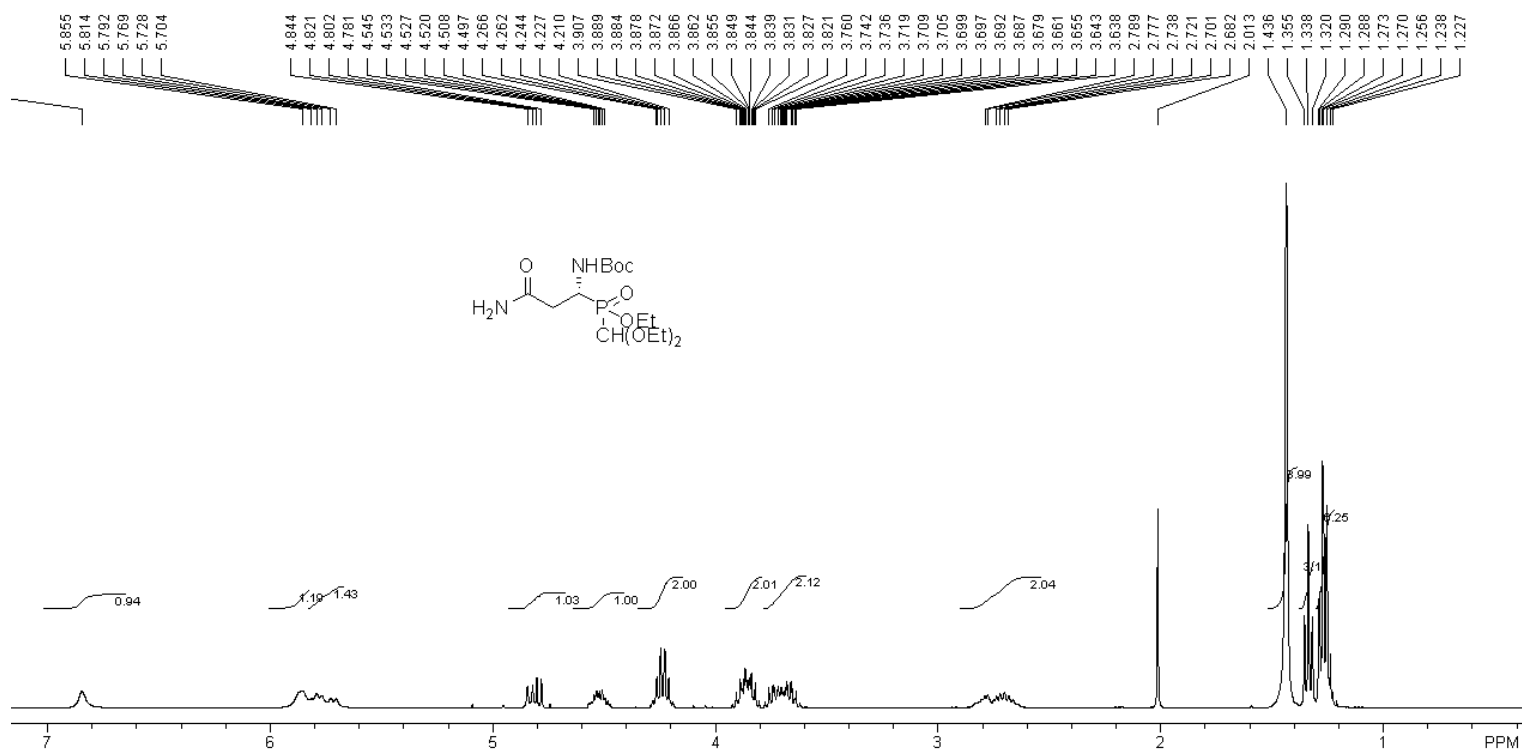
B = 0.00

C = 0.00

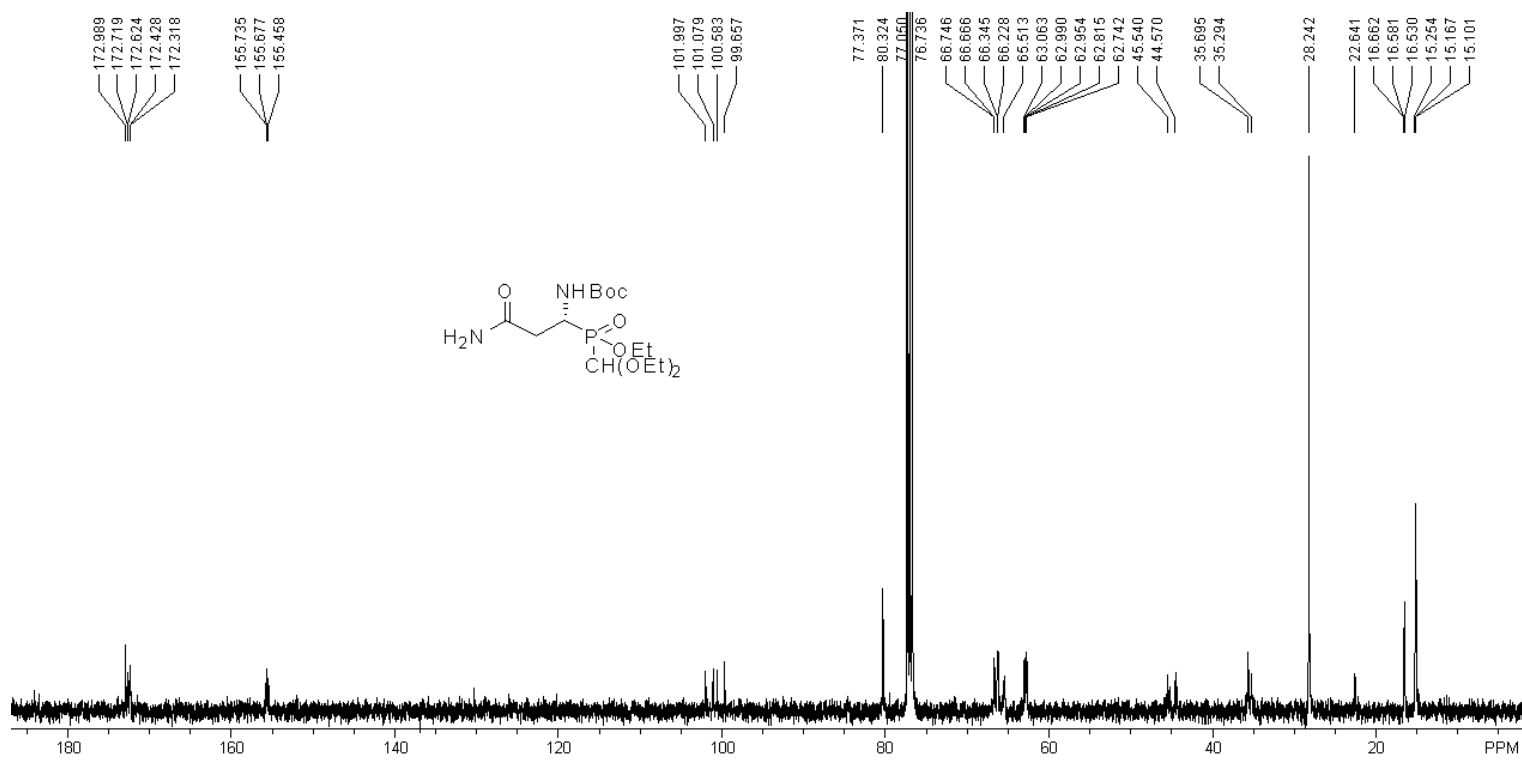


Compound 9b:

¹H NMR:



¹³C NMR:



³¹P NMR:

D:\software\nuts\DATA\2008119-10-1-2_12.1

12:52:49.390 +0800 nmr@EP-ZH107708

USER: nmr

SOLVENT: CDCl3

Experiment = zgpg30

Pulse length = 8.300 usec

Recycle delay = 2.000 sec

NA = 16

Solvent = CDCl3

PTS1d = 32768

F1 = 161.971542 MHz

F2 = 1.000000 MHz

SW1 = 64102.56 Hz

AT1 = 0.51 sec

Hz per Pt1stD = 1.96 Hz

SW2 = 1.00 Hz

Hz per Pt2ndD = 1.00 Hz

O1 = -8098.6865 Hz

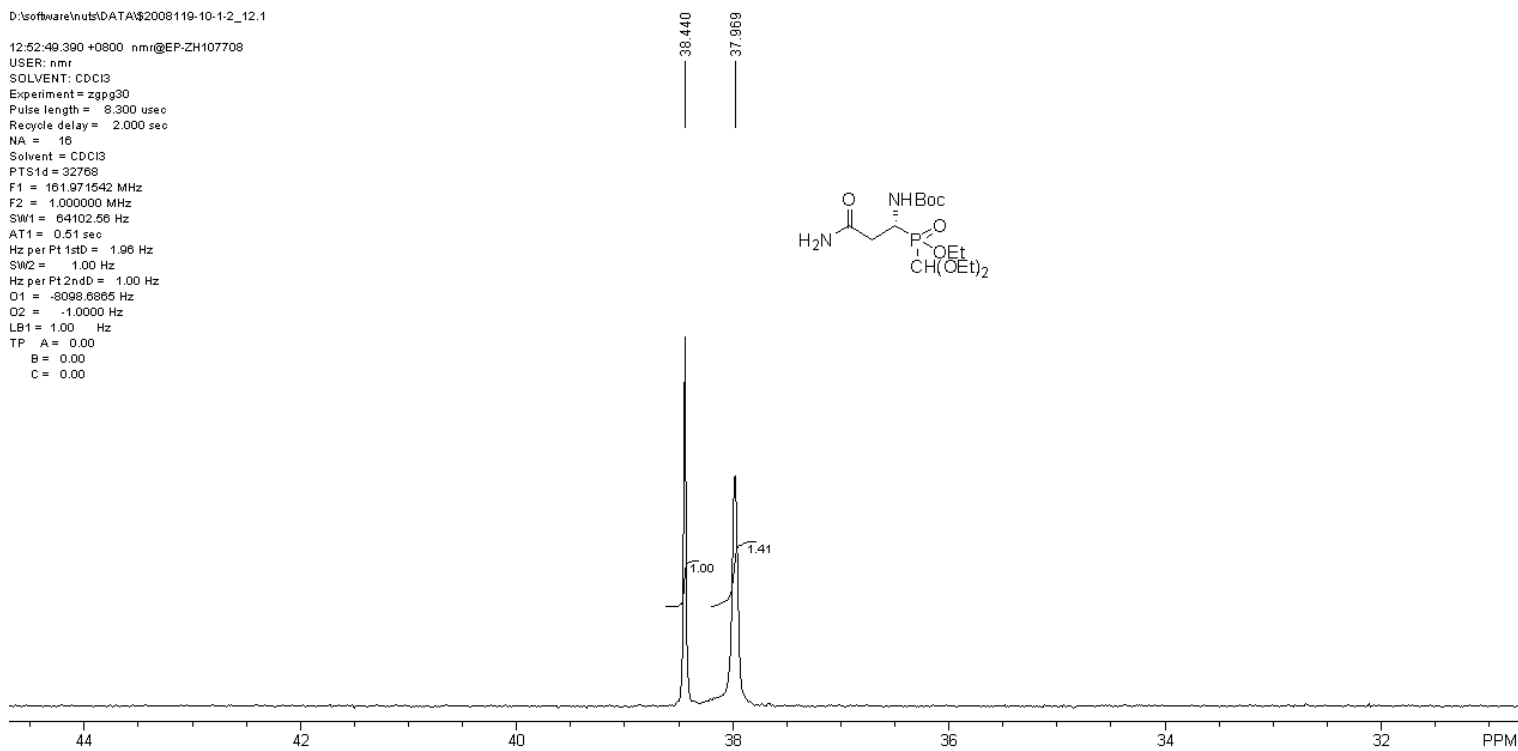
O2 = -1.0000 Hz

LB1 = 1.00 Hz

TP A = 0.00

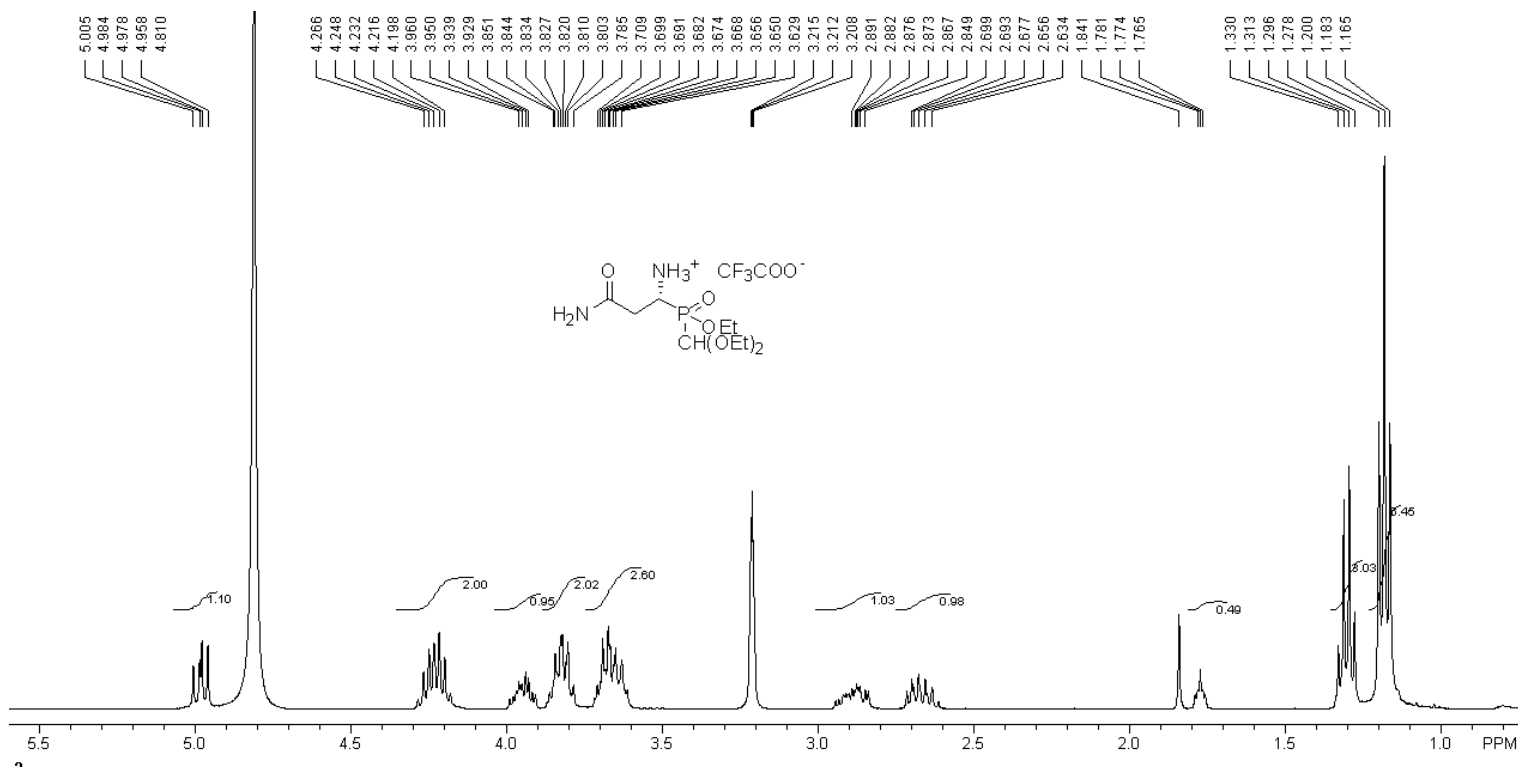
B = 0.00

C = 0.00

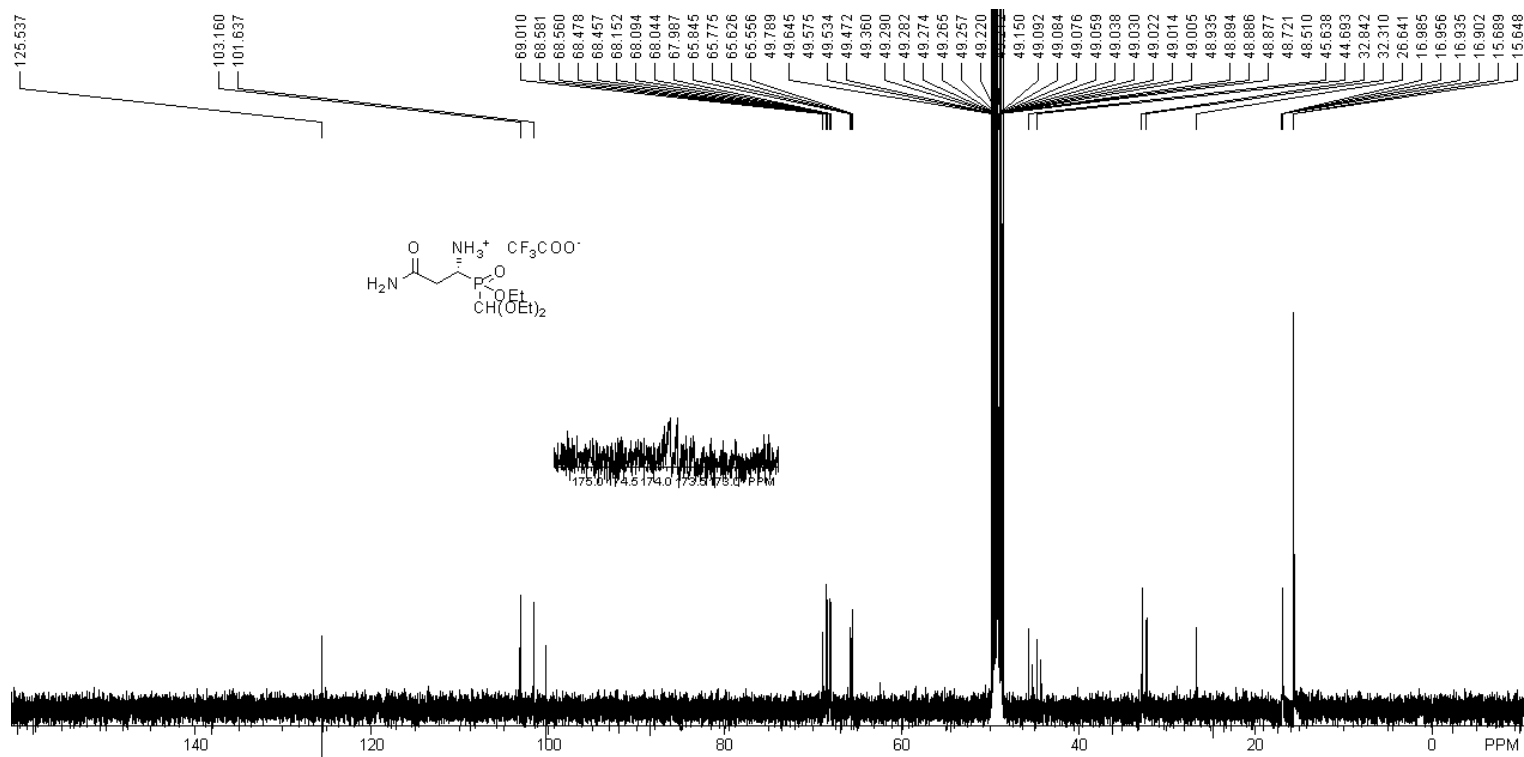


Compound 3u:

¹H NMR:



¹³C NMR:



¹⁹F NMR:

D:\software\nuts\DATA\2008119-10-10F_10.1

11:22:23.015 +0800 nmr@EP-ZH107708

USER: nmr

SOLVENT: MeOD

Experiment = zgfhgqn

Pulse length = 12.800 usec

Recycle delay = 1.000 sec

NA = 16

Solvent = MeOD

PTS1d = 65536

F1 = 376.488953 MHz

F2 = 1.000000 MHz

SW1 = 89285.71 Hz

AT1 = 0.73 sec

Hz per Pt1stD = 1.36 Hz

SW2 = 1.00 Hz

Hz per Pt2ndD = 1.00 Hz

O1 = -37649.0820 Hz

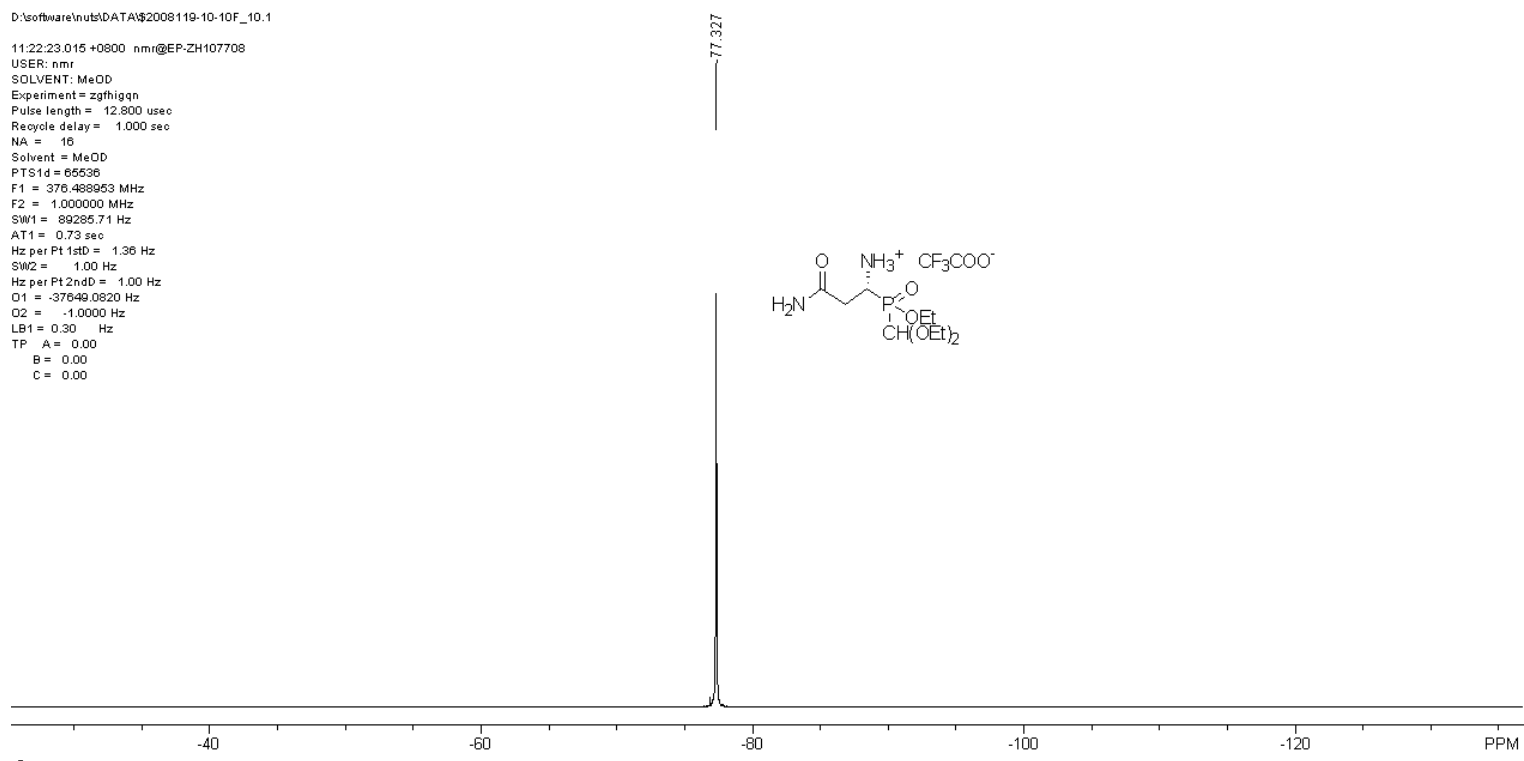
O2 = -1.0000 Hz

LB1 = 0.30 Hz

TP A = 0.00

B = 0.00

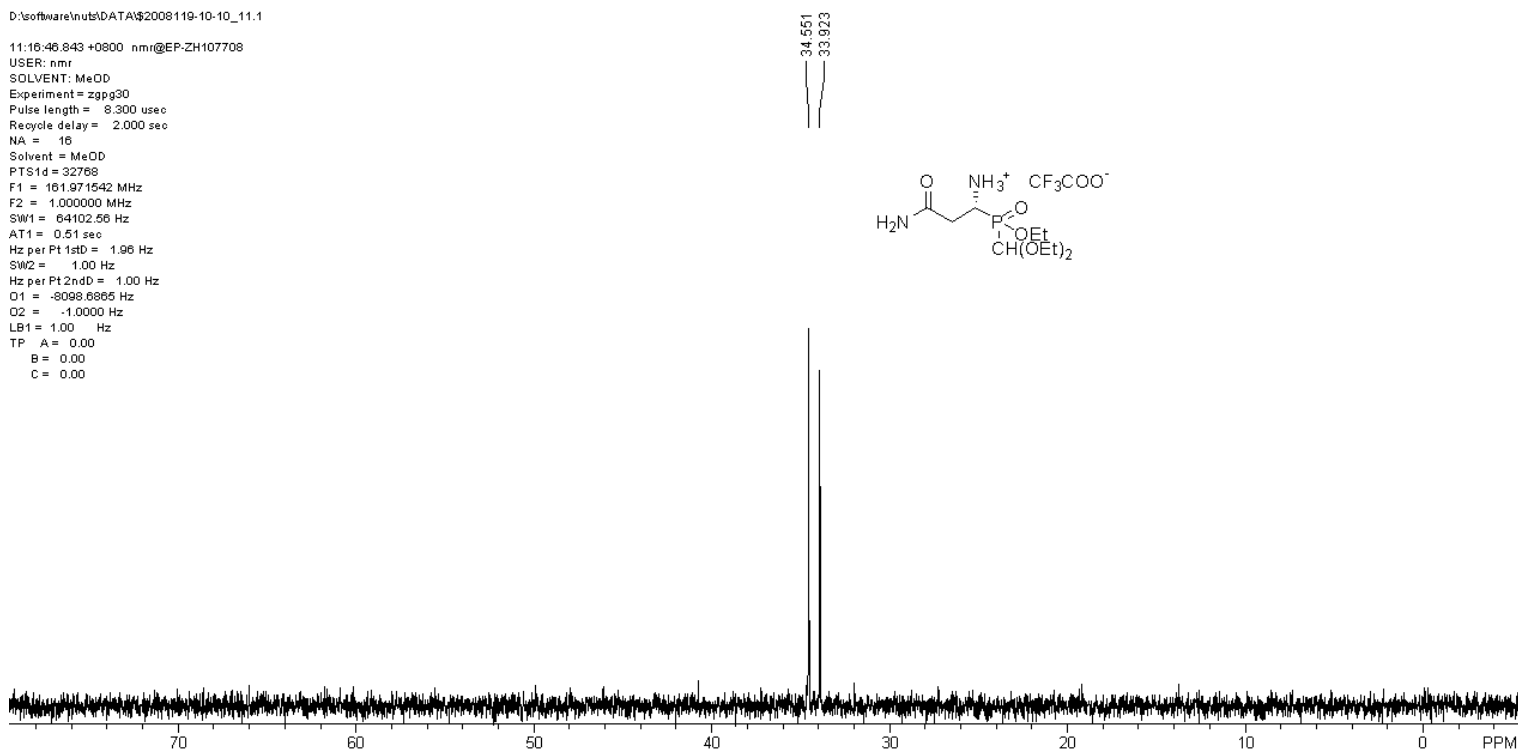
C = 0.00



³¹P NMR:

D:\software\nuts\DATA\2008119-10-10_11.1

11:16:46.843 +0800 nmri@EP-ZH107708
 USER: nmri
 SOLVENT: MeOD
 Experiment = zgpg30
 Pulse length = 8.300 usec
 Recycle delay = 2.000 sec
 NA = 16
 Solvent = MeOD
 PTS1d = 32768
 F1 = 161.971542 MHz
 F2 = 1.000000 MHz
 SW1 = 64102.56 Hz
 AT1 = 0.51 sec
 Hz per Pt1stD = 1.96 Hz
 SW2 = 1.00 Hz
 Hz per Pt2ndD = 1.00 Hz
 O1 = -8098.6865 Hz
 O2 = -1.0000 Hz
 LB1 = 1.00 Hz
 TP A = 0.00
 B = 0.00
 C = 0.00

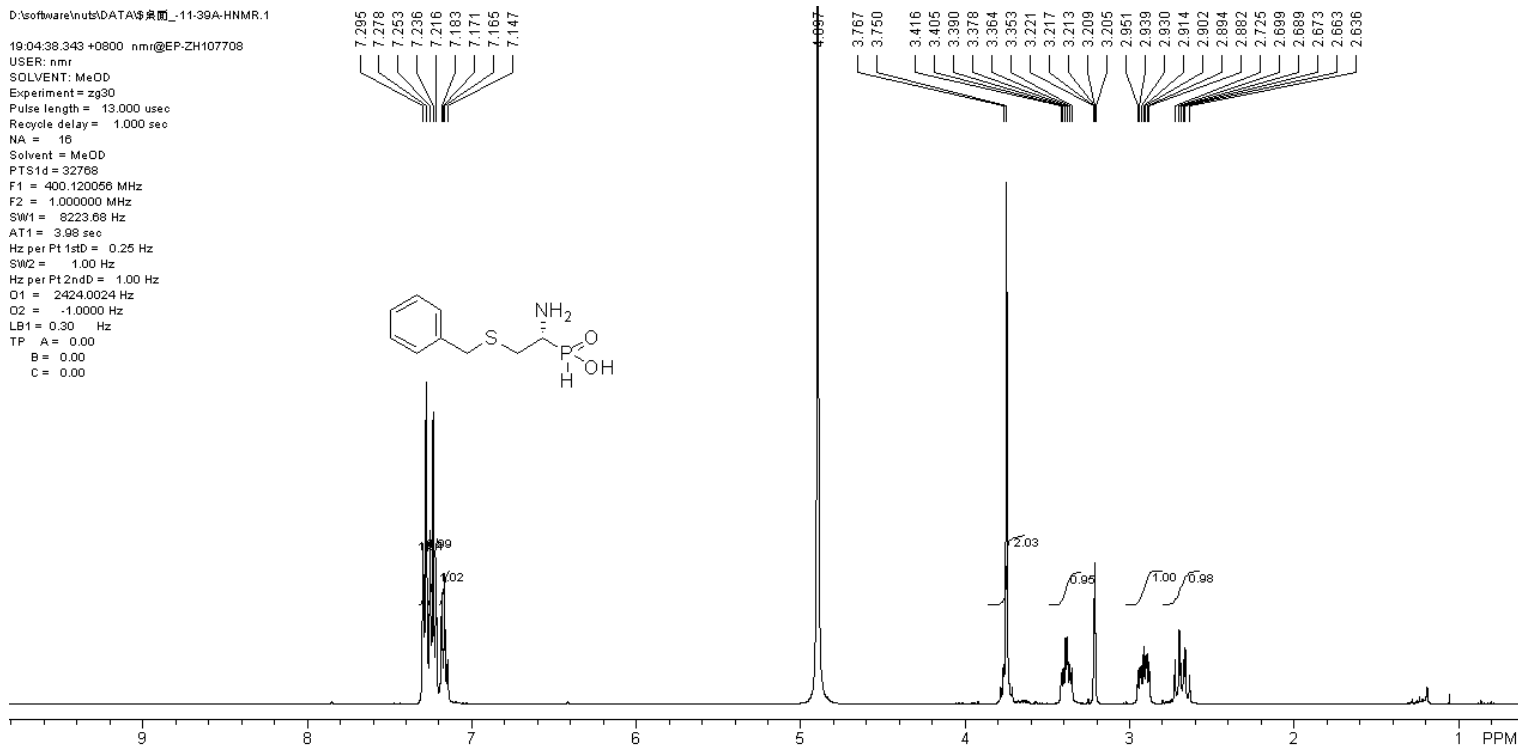


Compound 4c:

¹H NMR:

D:\software\nuts\DATA\2008119-10-10_11.1-39A-HNMR.1

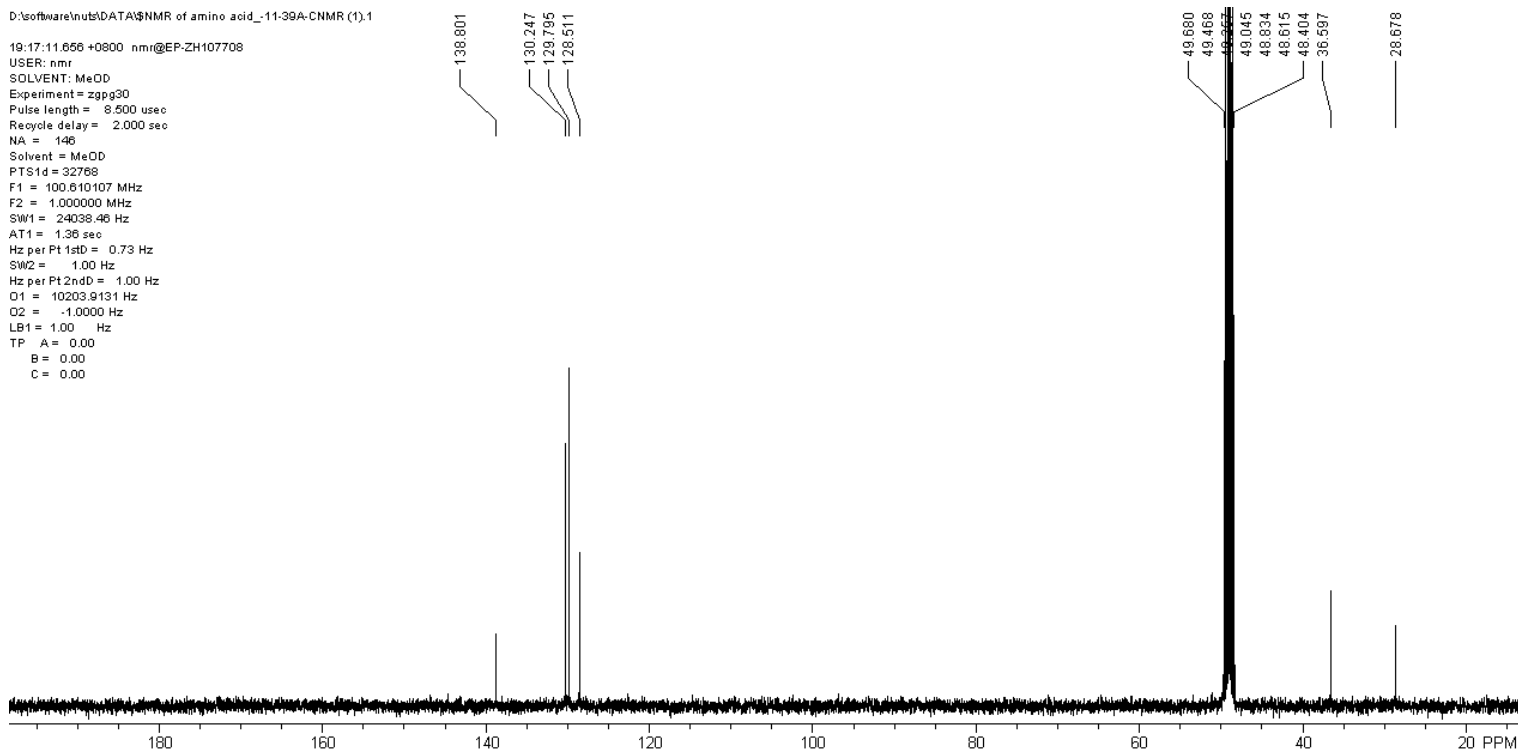
19:04:38.343 +0800 nmri@EP-ZH107708
 USER: nmri
 SOLVENT: MeOD
 Experiment = zg30
 Pulse length = 13.000 usec
 Recycle delay = 1.000 sec
 NA = 16
 Solvent = MeOD
 PTS1d = 32768
 F1 = 400.120058 MHz
 F2 = 1.000000 MHz
 SW1 = 8223.68 Hz
 AT1 = 3.98 sec
 Hz per Pt1stD = 0.25 Hz
 SW2 = 1.00 Hz
 Hz per Pt2ndD = 1.00 Hz
 O1 = 2424.0024 Hz
 O2 = -1.0000 Hz
 LB1 = 0.30 Hz
 TP A = 0.00
 B = 0.00
 C = 0.00



¹³C NMR:

D:\software\nuts\DATA\\$NMR of amino acid_11-39A-CNMR (1).1

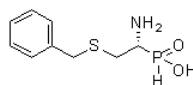
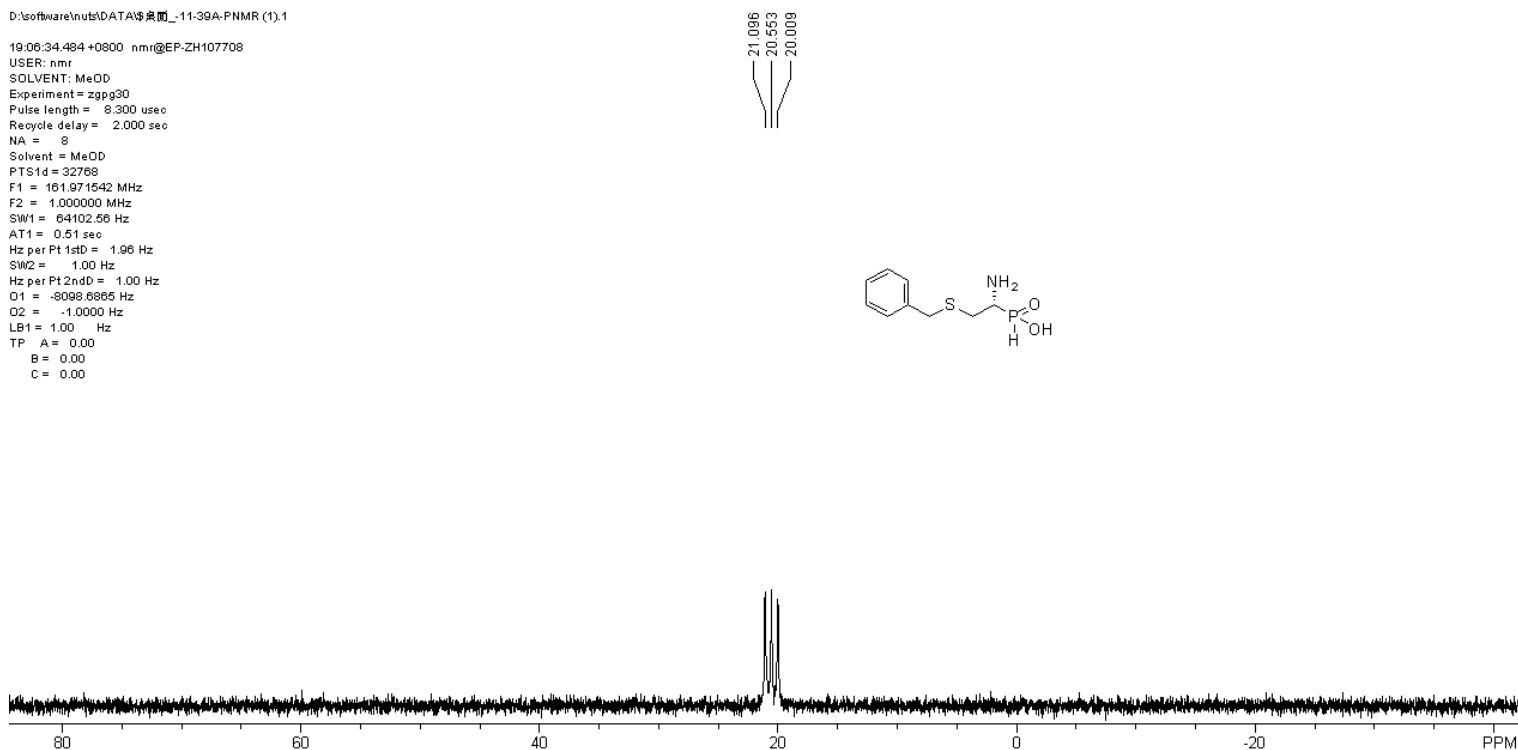
19:17:11.656 +0800 nmr@EP-ZH107708
 USER: nmr
 SOLVENT: MeOD
 Experiment = zgpg30
 Pulse length = 8.500 usec
 Recycle delay = 2.000 sec
 NA = 146
 Solvent = MeOD
 PTS1d = 32768
 F1 = 100.610107 MHz
 F2 = 1.000000 MHz
 SW1 = 24038.46 Hz
 AT1 = 1.36 sec
 Hz per Pt1stD = 0.73 Hz
 SW2 = 1.00 Hz
 Hz per Pt2ndD = 1.00 Hz
 O1 = 10203.8131 Hz
 O2 = -1.0000 Hz
 LB1 = 1.00 Hz
 TP A = 0.00
 B = 0.00
 C = 0.00



³¹P NMR:

D:\software\nuts\DATA\$奥面_11-39A-PNMR (1).1

19:06:34.484 +0800 nmr@EP-ZH107708
 USER: nmr
 SOLVENT: MeOD
 Experiment = zgpg30
 Pulse length = 8.300 usec
 Recycle delay = 2.000 sec
 NA = 8
 Solvent = MeOD
 PTS1d = 32768
 F1 = 161.971542 MHz
 F2 = 1.000000 MHz
 SW1 = 64102.56 Hz
 AT1 = 0.51 sec
 Hz per Pt1stD = 1.96 Hz
 SW2 = 1.00 Hz
 Hz per Pt2ndD = 1.00 Hz
 O1 = -8098.6865 Hz
 O2 = -1.0000 Hz
 LB1 = 1.00 Hz
 TP A = 0.00
 B = 0.00
 C = 0.00



Compound 4f:

¹H NMR:

D:\software\nuts\DATA\\$NMR of amino acid_4-31HNMR.1

14:17:03.390 +0800 nmr@EP-ZH107708

USER: nmr

SOLVENT: MeOD

Experiment = zg30

Pulse length = 9.300 usec

Recycle delay = 1.000 sec

NA = 16

Solvent = MeOD

PTS1d = 32768

F1 = 400.120056 MHz

F2 = 1.0000000 MHz

SW1 = 8223.68 Hz

AT1 = 3.98 sec

Hz per Pt1stD = 0.25 Hz

SW2 = 1.00 Hz

Hz per Pt2ndD = 1.00 Hz

O1 = 2424.4429 Hz

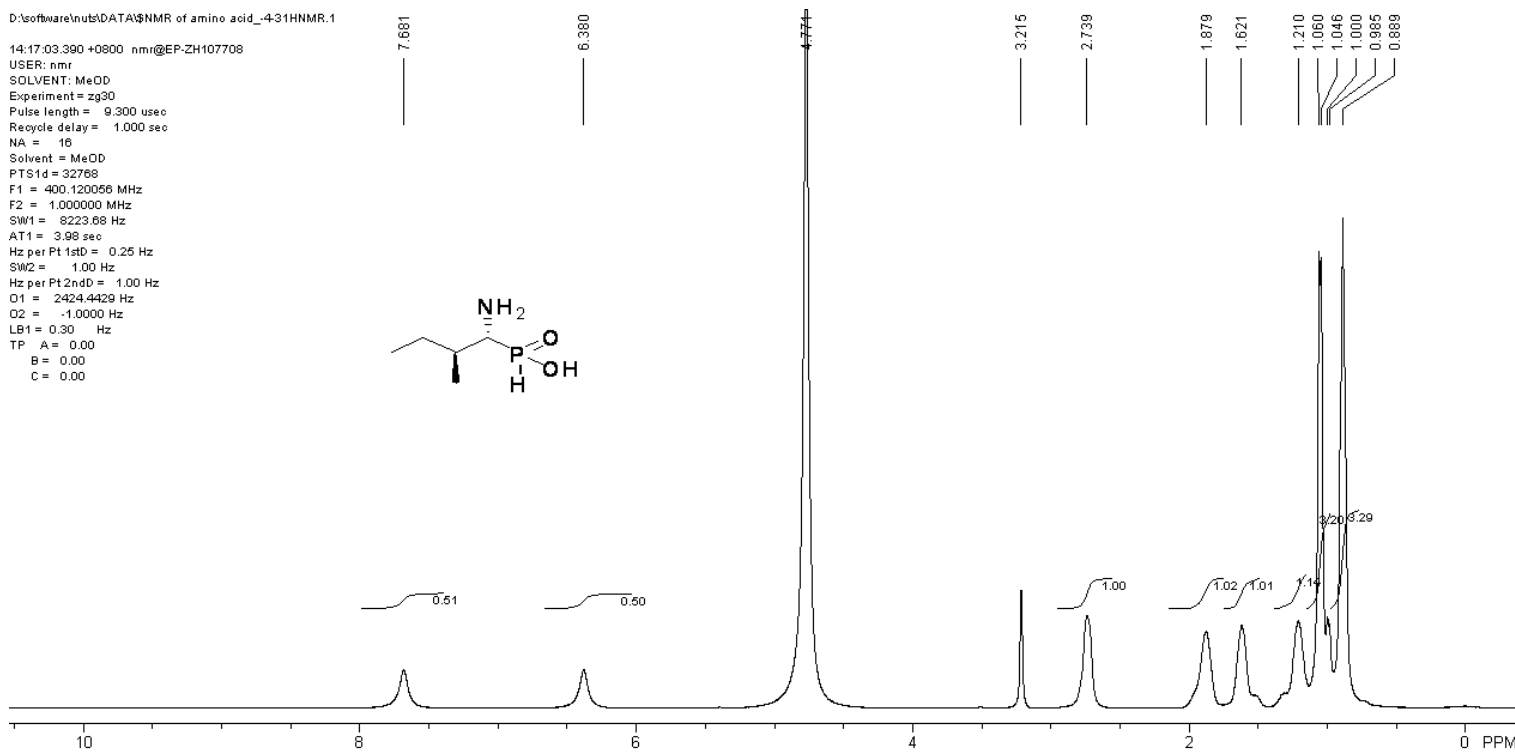
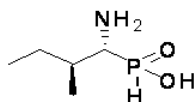
O2 = -1.0000 Hz

LB1 = 0.30 Hz

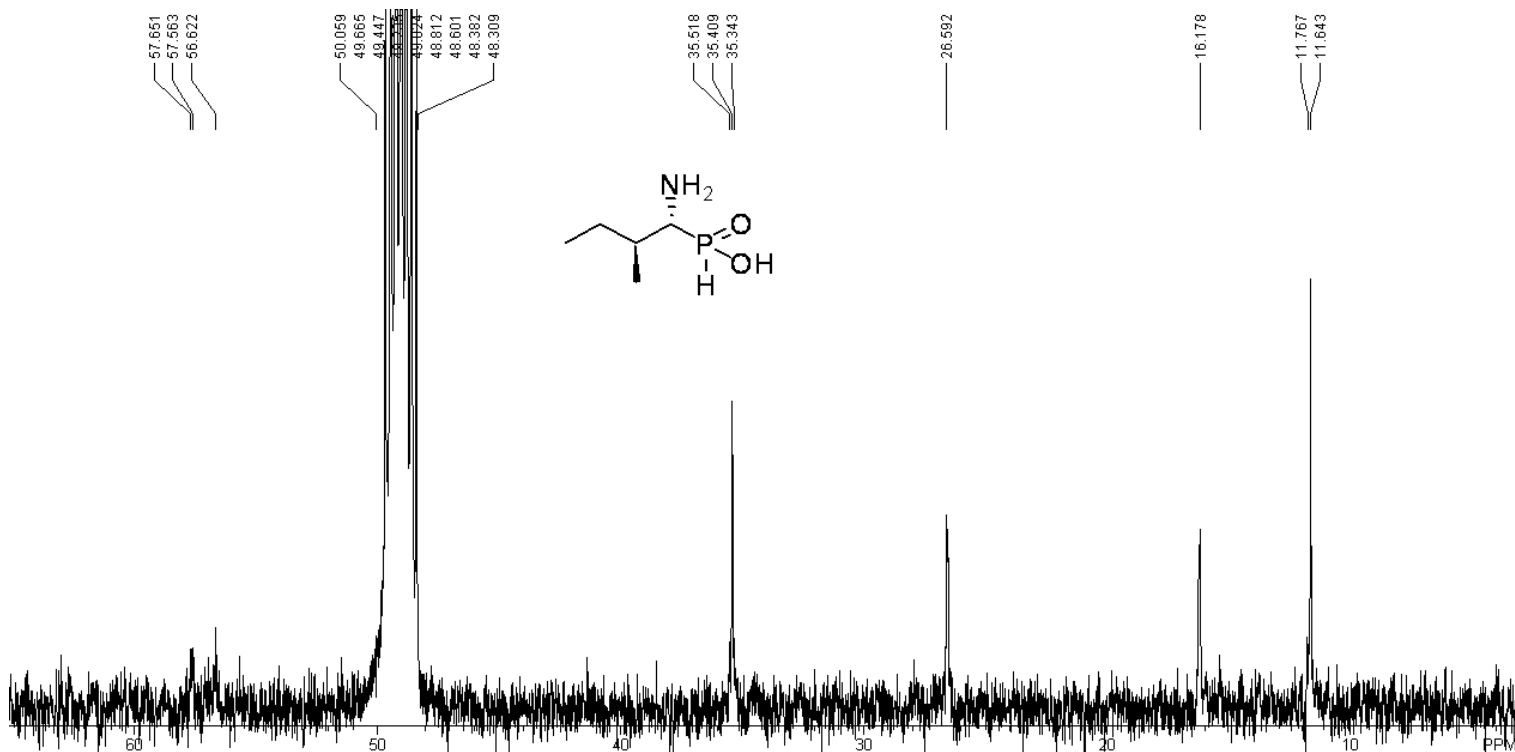
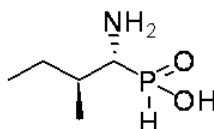
TP A = 0.00

B = 0.00

C = 0.00

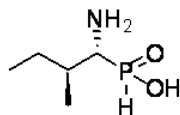


¹³C NMR:

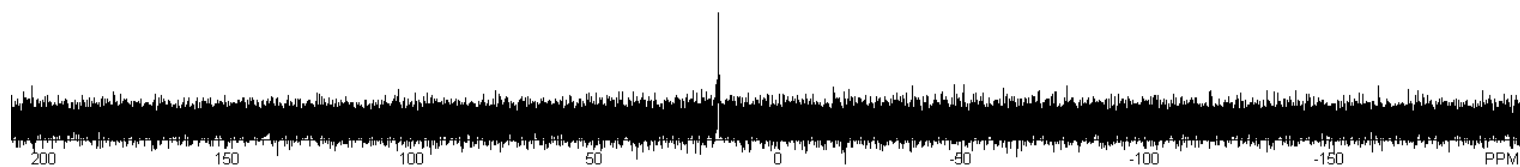


³¹P NMR:

D:\software\nuts\DATA\YQL-4-31PNMR.fid
 YQL-4-31
 Jul 2 2010
 USER:
 SOLVENT: CD3OD
 Experiment = s2pul
 Pulse length = 3.889 usec
 Recycle delay = 1.000 sec
 NA = 200
 Solvent = CD3OD
 FID PTS1d = 60000
 PTS1d = 65536
 F1 = 121.463766 MHz
 F2 = 300.029541 MHz
 SW1 = 50000.00 Hz
 AT1 = 1.00 sec
 Hz per Pt1stD = 0.76 Hz
 SW2 = 1.00 Hz
 Hz per Pt2ndD = 1.00 Hz
 O1 = 338.4102 Hz
 O2 = -0.6000 Hz
 LB1 = 0.00 Hz
 TP A = -10.31
 B = -94.38
 C = 0.00



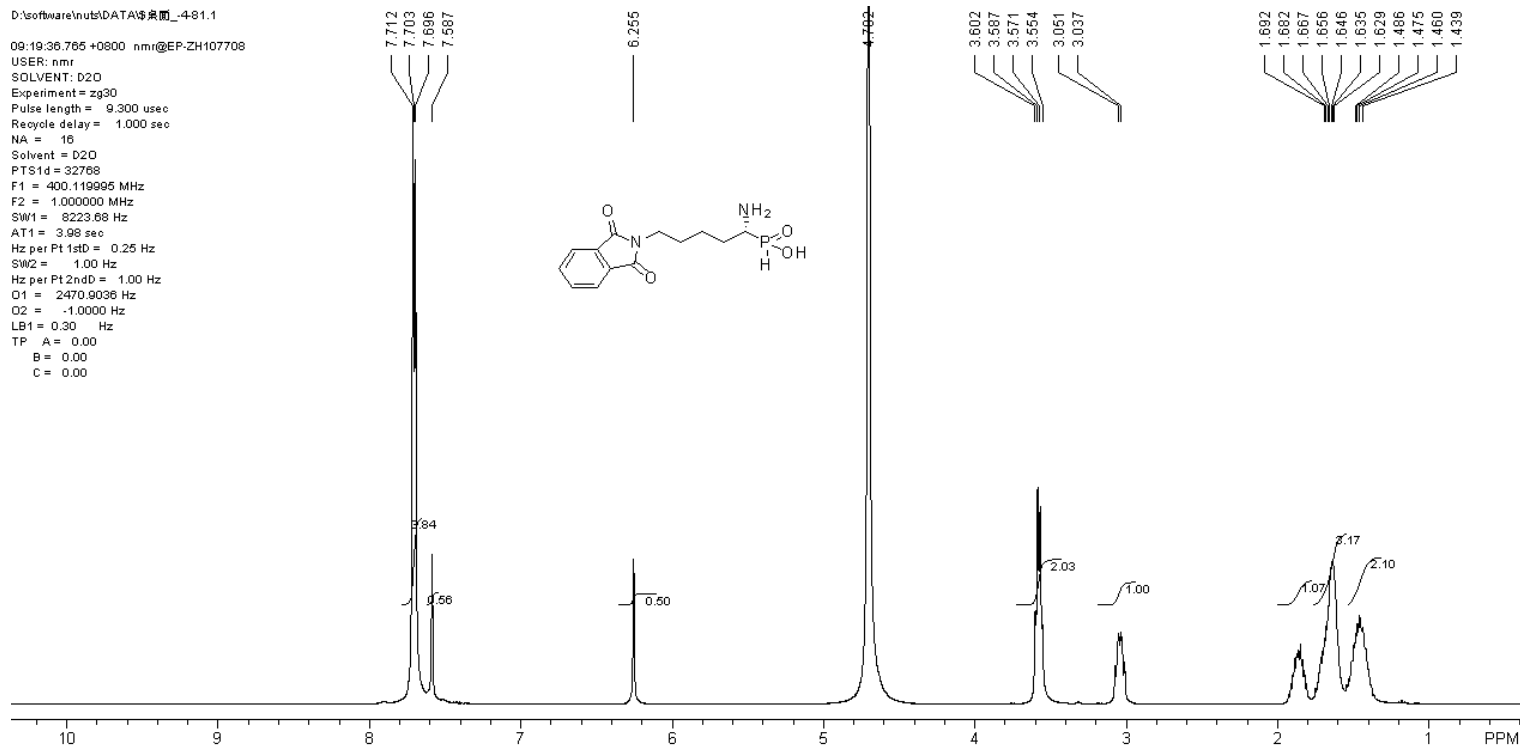
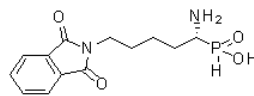
16.182



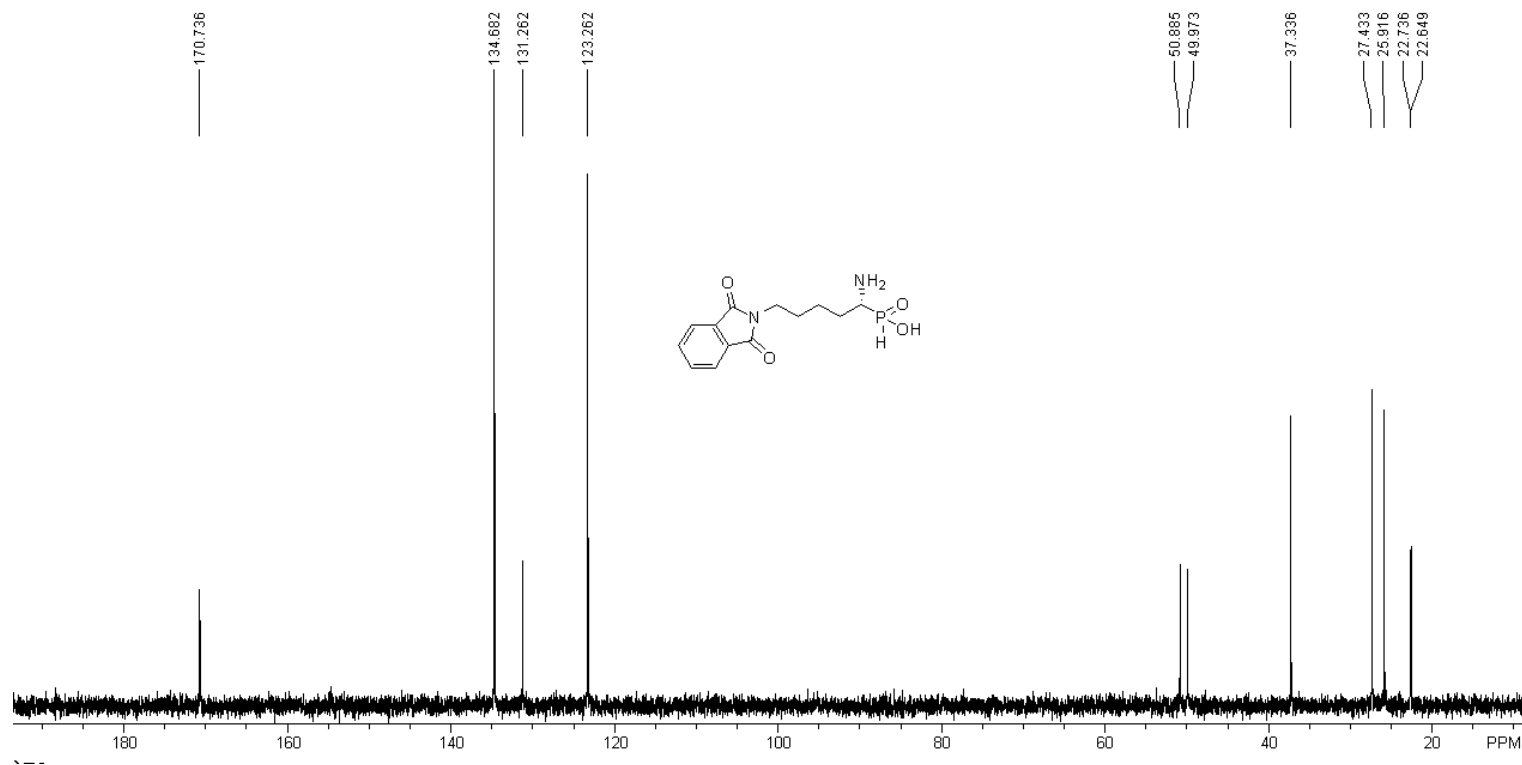
Compound 4h:

¹H NMR:

D:\software\nuts\DATA\谱图面_481.1
 09:19:36.765 +0800 nmr@EP-ZH107708
 USER: nmr
 SOLVENT: D2O
 Experiment = zg30
 Pulse length = 9.300 usec
 Recycle delay = 1.000 sec
 NA = 16
 Solvent = D2O
 PTS1d = 32768
 F1 = 400.119995 MHz
 F2 = 1.000000 MHz
 SW1 = 8223.68 Hz
 AT1 = 3.98 sec
 Hz per Pt1stD = 0.25 Hz
 SW2 = 1.00 Hz
 Hz per Pt2ndD = 1.00 Hz
 O1 = 2470.9036 Hz
 O2 = -1.0000 Hz
 LB1 = 0.30 Hz
 TP A = 0.00
 B = 0.00
 C = 0.00

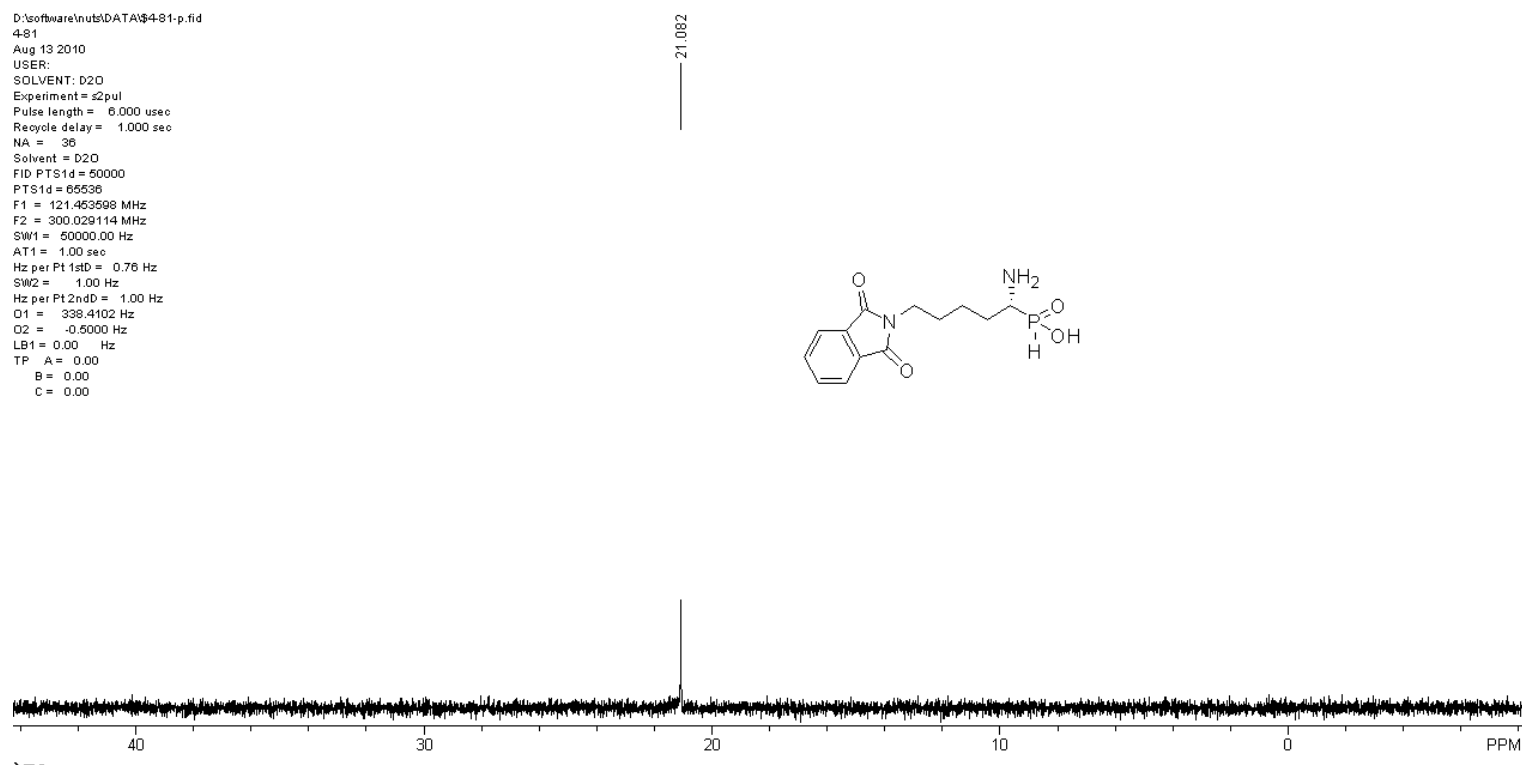


¹³C NMR:



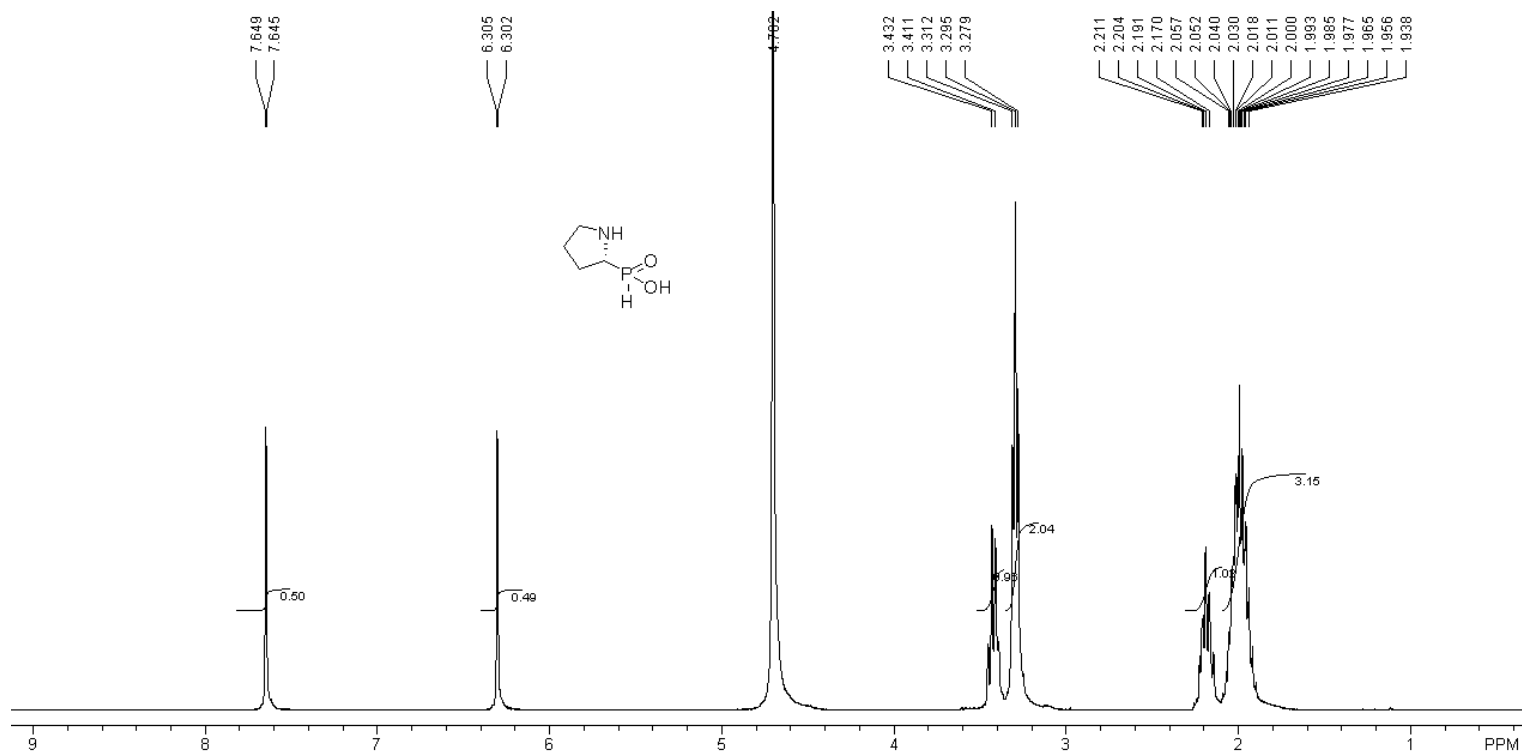
³¹P NMR:

D:\software\nuts\DATA\4-81-p.fid
 4-81
 Aug 13 2010
 USER:
 SOLVENT: D2O
 Experiment = s2pul
 Pulse length = 6.000 usec
 Recycle delay = 1.000 sec
 NA = 36
 Solvent = D2O
 FID PTS1d = 50000
 PTS1d = 65536
 F1 = 121.453599 MHz
 F2 = 300.029114 MHz
 SW1 = 50000.00 Hz
 AT1 = 1.00 sec
 Hz per Pt1s0 = 0.76 Hz
 SW2 = 1.00 Hz
 Hz per Pt2nd0 = 1.00 Hz
 Q1 = 338.4102 Hz
 Q2 = -0.5000 Hz
 LB1 = 0.00 Hz
 TP A = 0.00
 B = 0.00
 C = 0.00



Compound 4k:

¹H NMR:



¹³C NMR:

D:\software\nmr\DATA\2008119-5-66_o_10.1

18:48:34.468 +0800 nmr@EP-ZH107708

USER: nmr

SOLVENT: D2O

Experiment = zgpg30

Pulse length = 8.600 usec

Recycle delay = 2.000 sec

NA = 166

Solvent = D2O

PTS1d = 32768

F1 = 100.610252 MHz

F2 = 1.000000 MHz

SW1 = 24038.46 Hz

AT1 = 1.36 sec

Hz per Pt1stD = 0.73 Hz

SW2 = 1.00 Hz

Hz per Pt2ndD = 1.00 Hz

O1 = 10061.0479 Hz

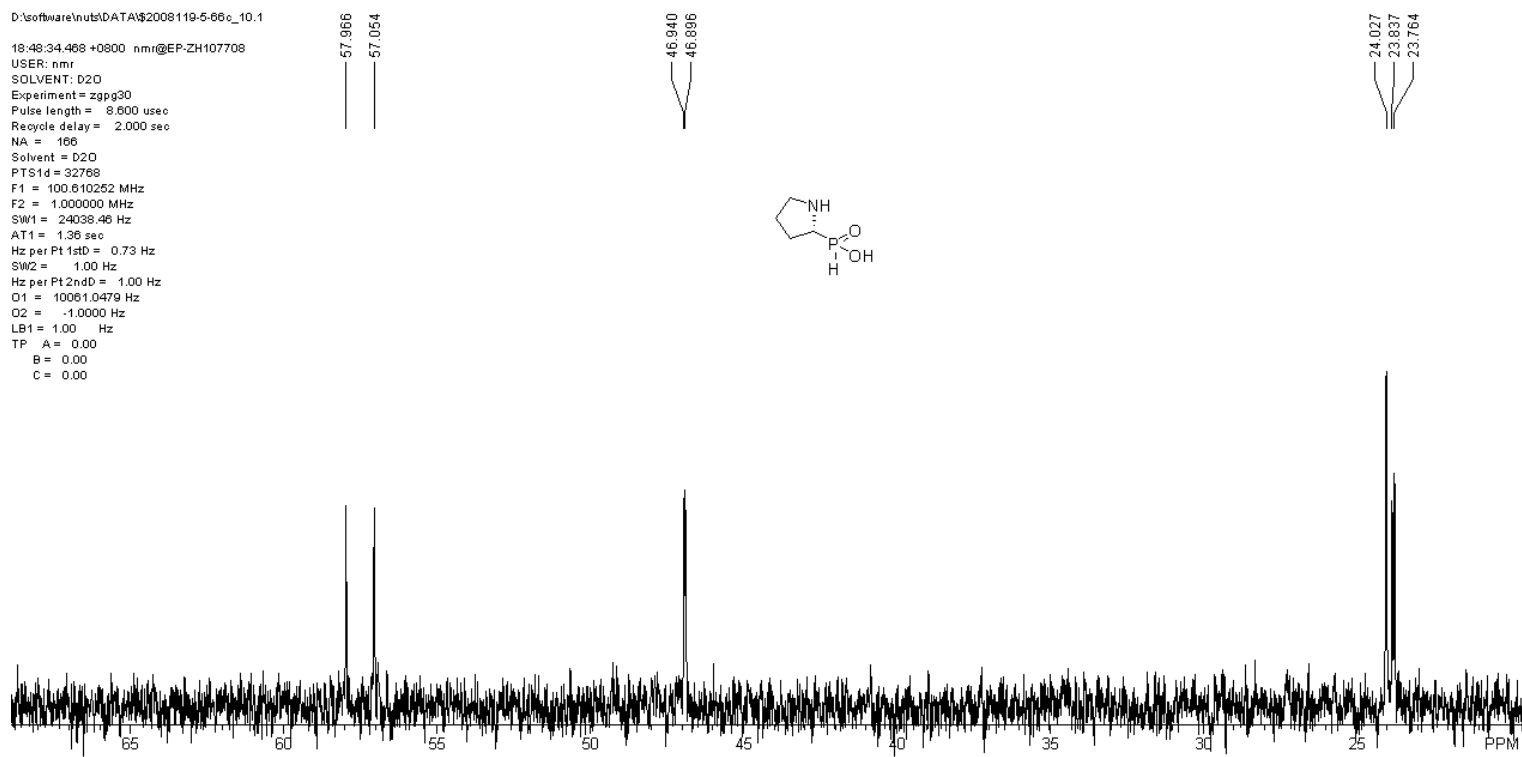
O2 = -1.0000 Hz

LB1 = 1.00 Hz

TP A = 0.00

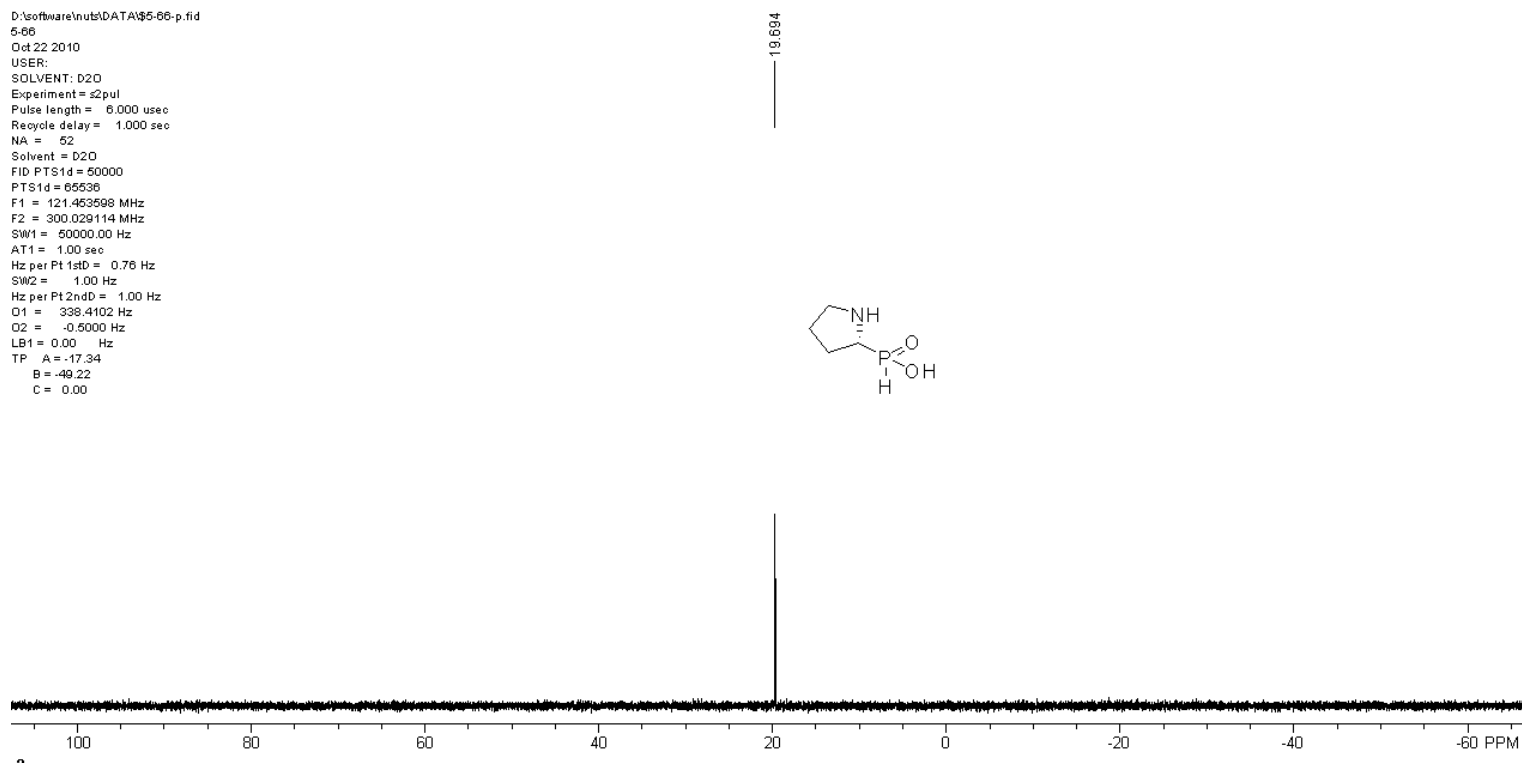
B = 0.00

C = 0.00



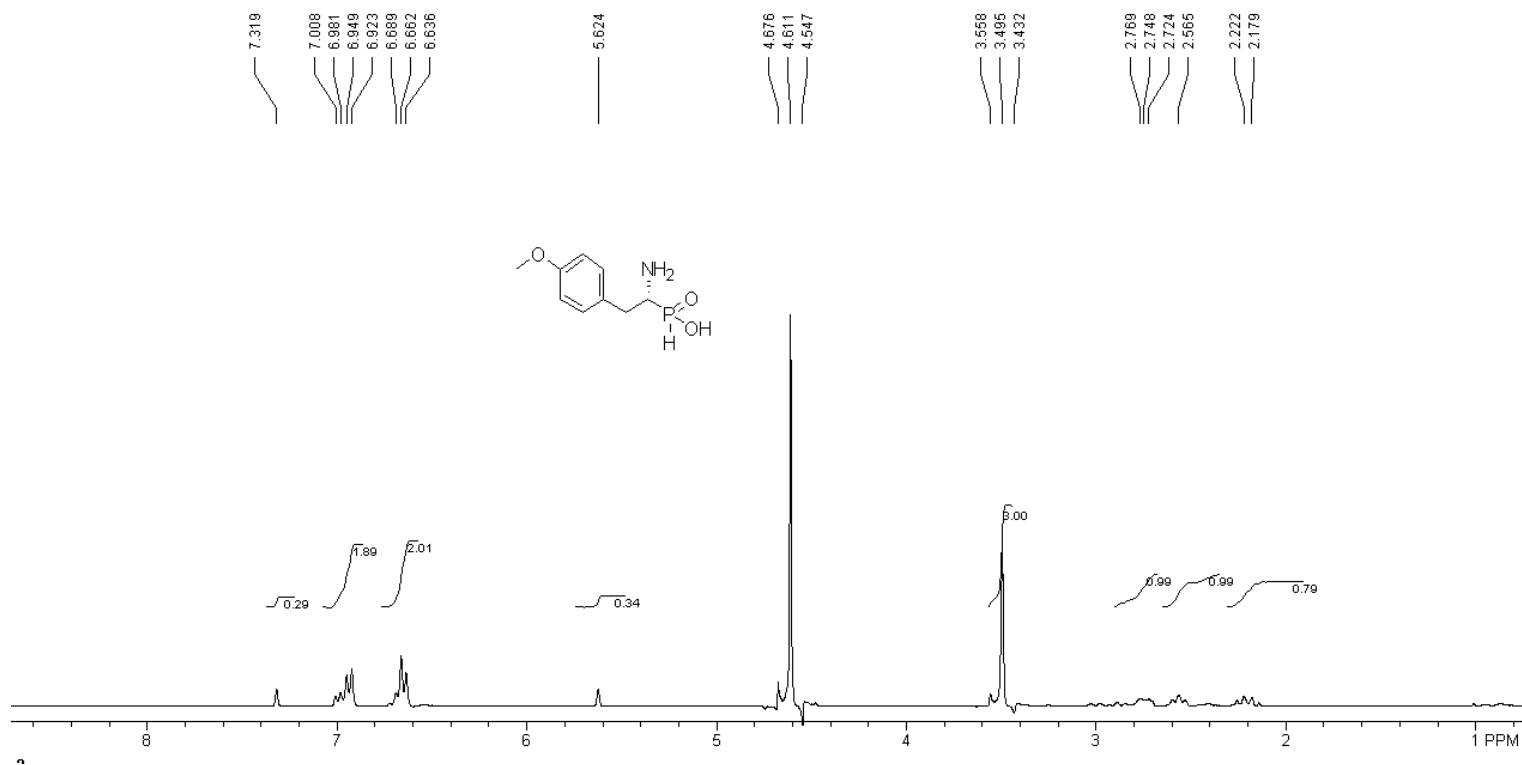
³¹P NMR:

D:\software\nuts\DATA\5-06-p.rid
 5-06
 Oct 22 2010
 USER:
 SOLVENT: D2O
 Experiment = s2pul
 Pulse length = 6.000 usec
 Recycle delay = 1.000 sec
 NA = 52
 Solvent = D2O
 FID PTS1d = 60000
 PTS1d = 65536
 F1 = 121.463598 MHz
 F2 = 300.029114 MHz
 SW1 = 50000.00 Hz
 AT1 = 1.00 sec
 Hz per Pt1st0 = 0.76 Hz
 SW2 = 1.00 Hz
 Hz per Pt2nd0 = 1.00 Hz
 O1 = 338.4102 Hz
 O2 = -0.6000 Hz
 LB1 = 0.00 Hz
 TP A = -17.34
 B = -49.22
 C = 0.00



Compound 4o:

¹H NMR:



¹³C NMR:

D:\software\inputs\DATA\NMR of amino acid_3-23c.1

13:35:01.515 +0800 nmr@EP-ZH107708

USER: nmr

SOLVENT: D2O

Experiment = zgpg30

Pulse length = 8.600 usec

Recycle delay = 2.000 sec

NA = 256

Solvent = D2O

PTS1d = 32768

F1 = 100.610252 MHz

F2 = 1.000000 MHz

SW1 = 24038.46 Hz

AT1 = 1.36 sec

Hz per Pt1stD = 0.73 Hz

SW2 = 1.00 Hz

Hz per Pt2ndD = 1.00 Hz

O1 = 10061.0479 Hz

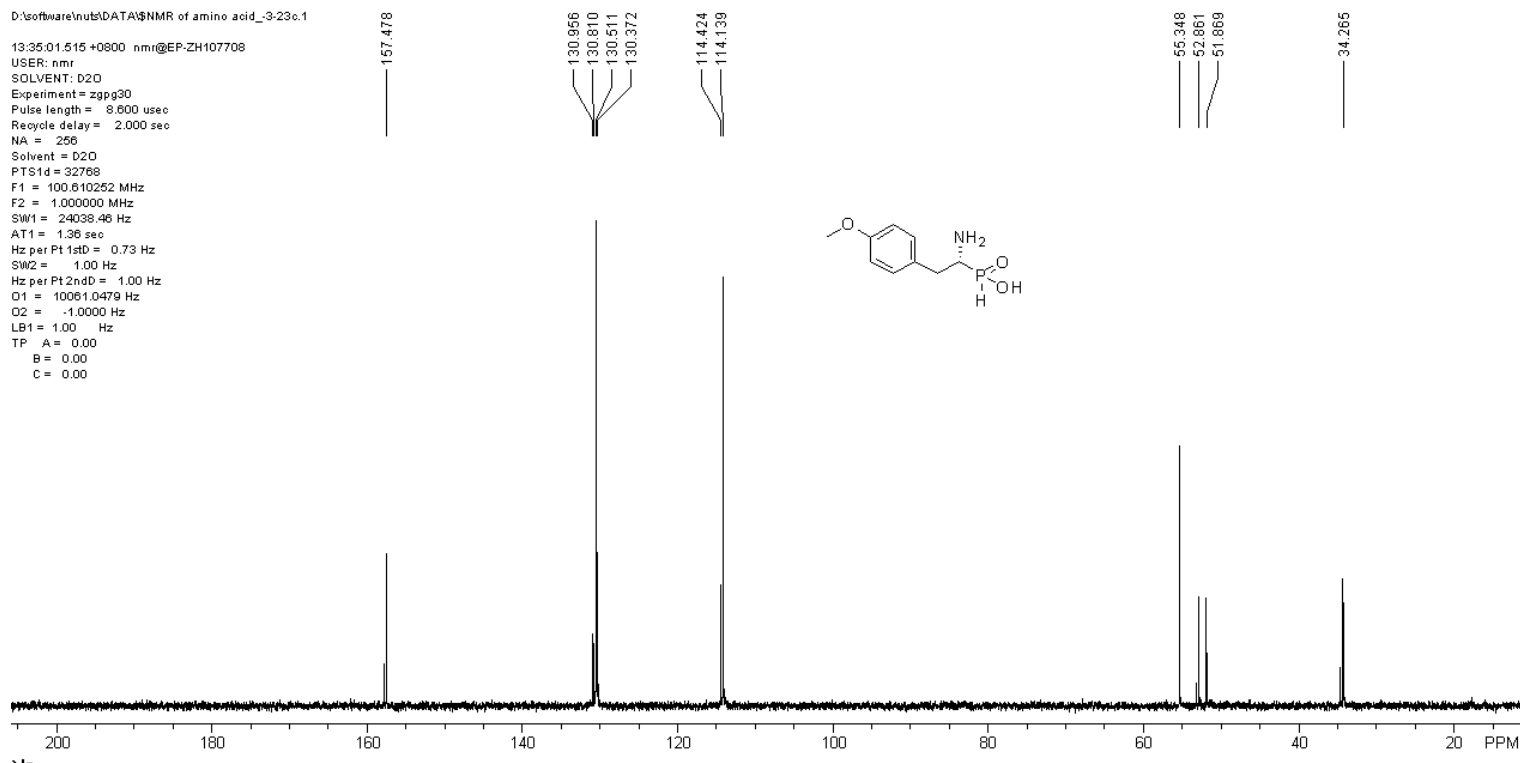
O2 = -1.0000 Hz

LB1 = 1.00 Hz

TP A = 0.00

B = 0.00

C = 0.00



Compound 4p:

¹H NMR:

D:\software\inputs\DATA\2-76_26Jan2010

:blank line

Jan 26 2010

USER:

SOLVENT: D2O

Experiment = 2pul

Pulse length = 5.850 usec

Recycle delay = 1.000 sec

NA = 8

Solvent = D2O

FID PTS1d = 9587

PTS1d = 16384

F1 = 300.055389 MHz

F2 = 75.456078 MHz

SW1 = 4803.07 Hz

AT1 = 2.00 sec

Hz per Pt1stD = 0.29 Hz

SW2 = 1.00 Hz

Hz per Pt2ndD = 1.00 Hz

O1 = 1800.3159 Hz

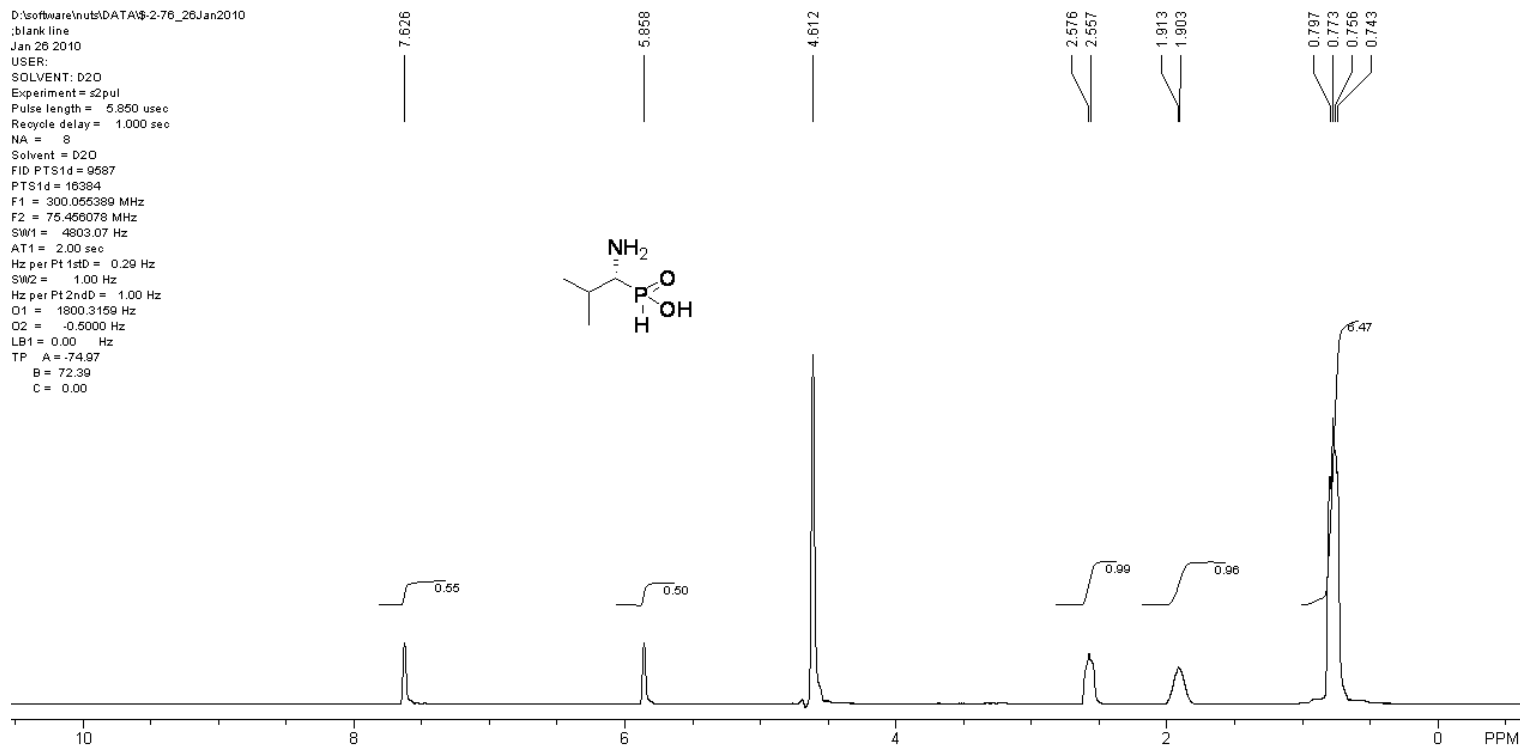
O2 = -0.6000 Hz

LB1 = 0.00 Hz

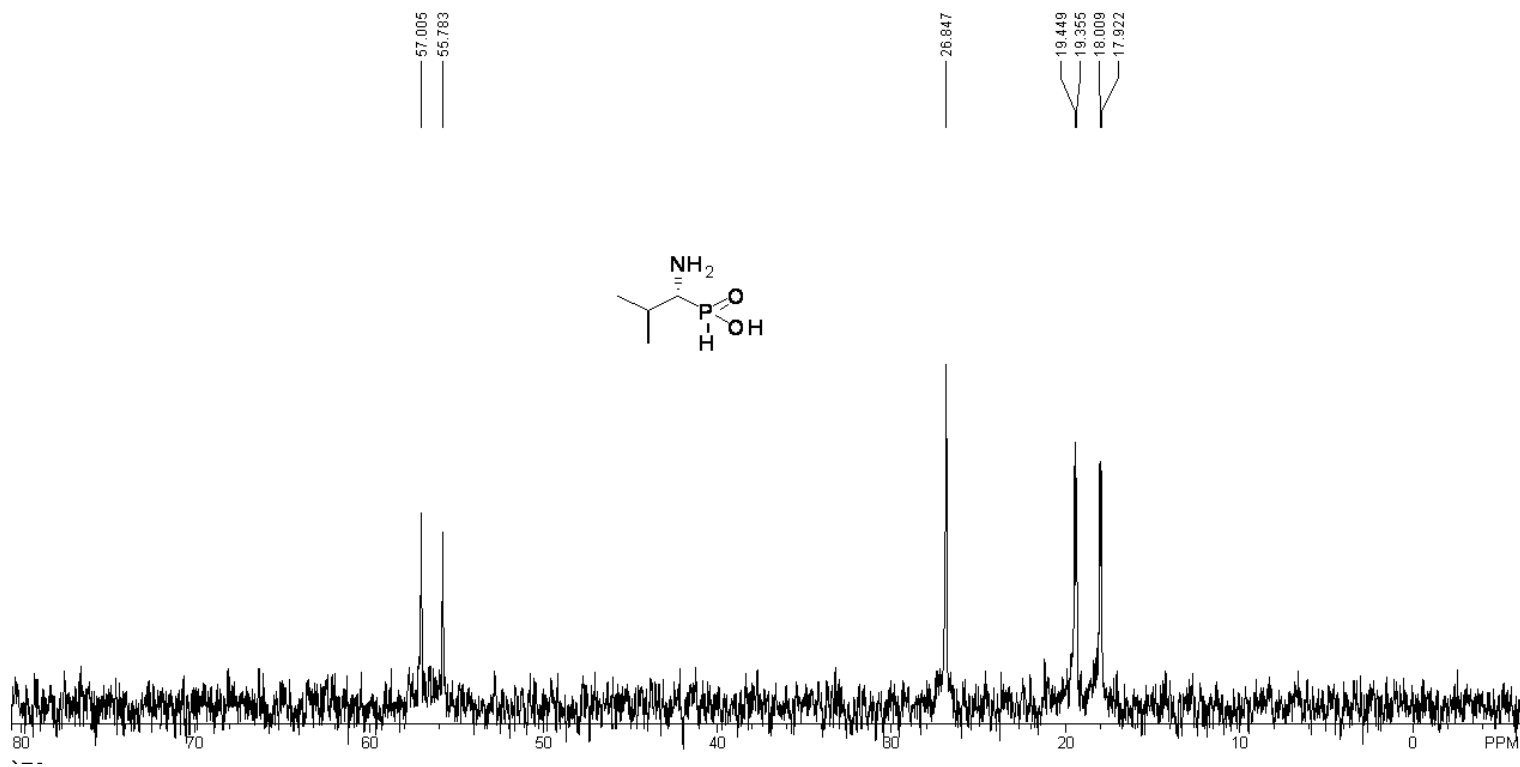
TP A = -74.97

B = 72.39

C = 0.00

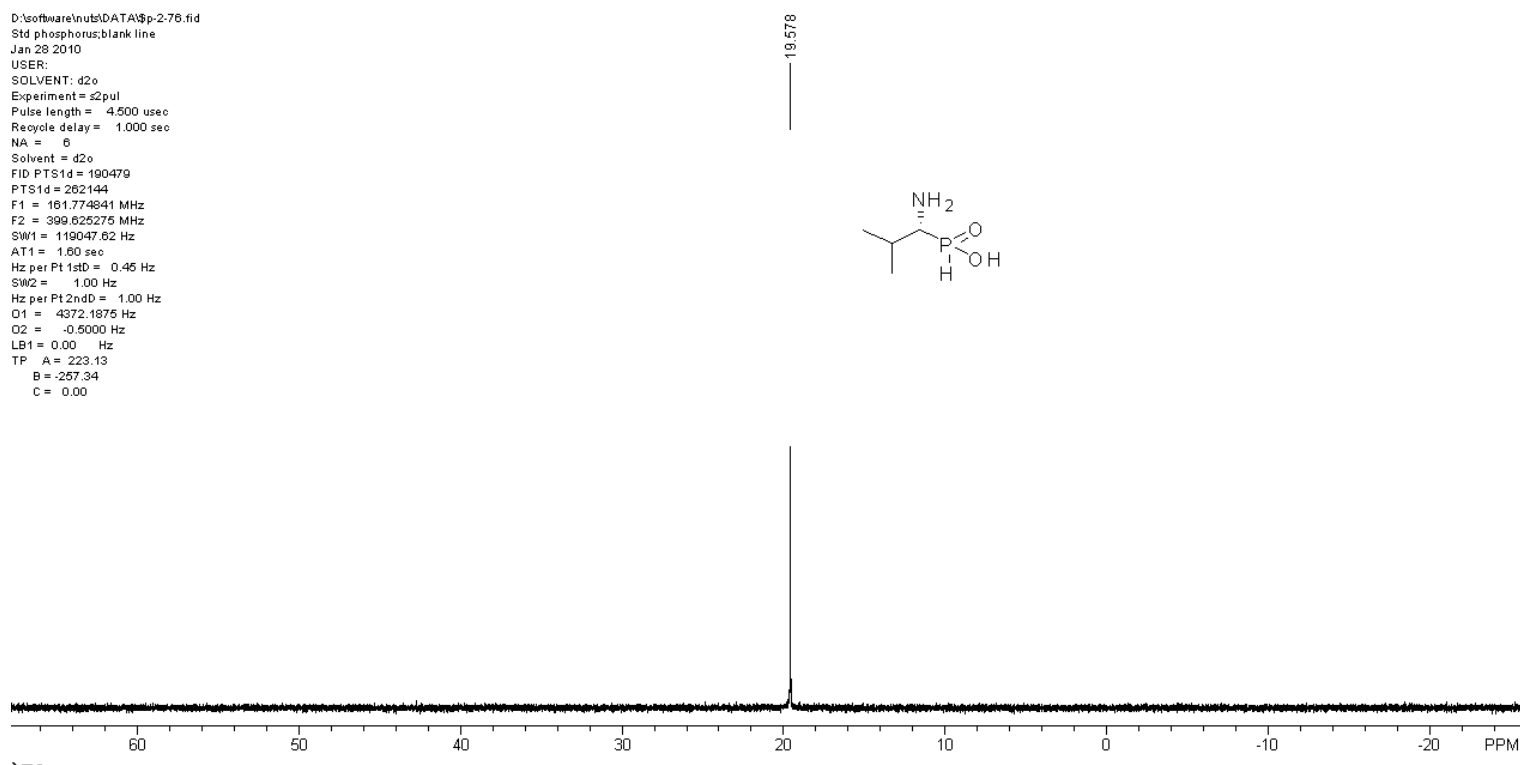


¹³C NMR:



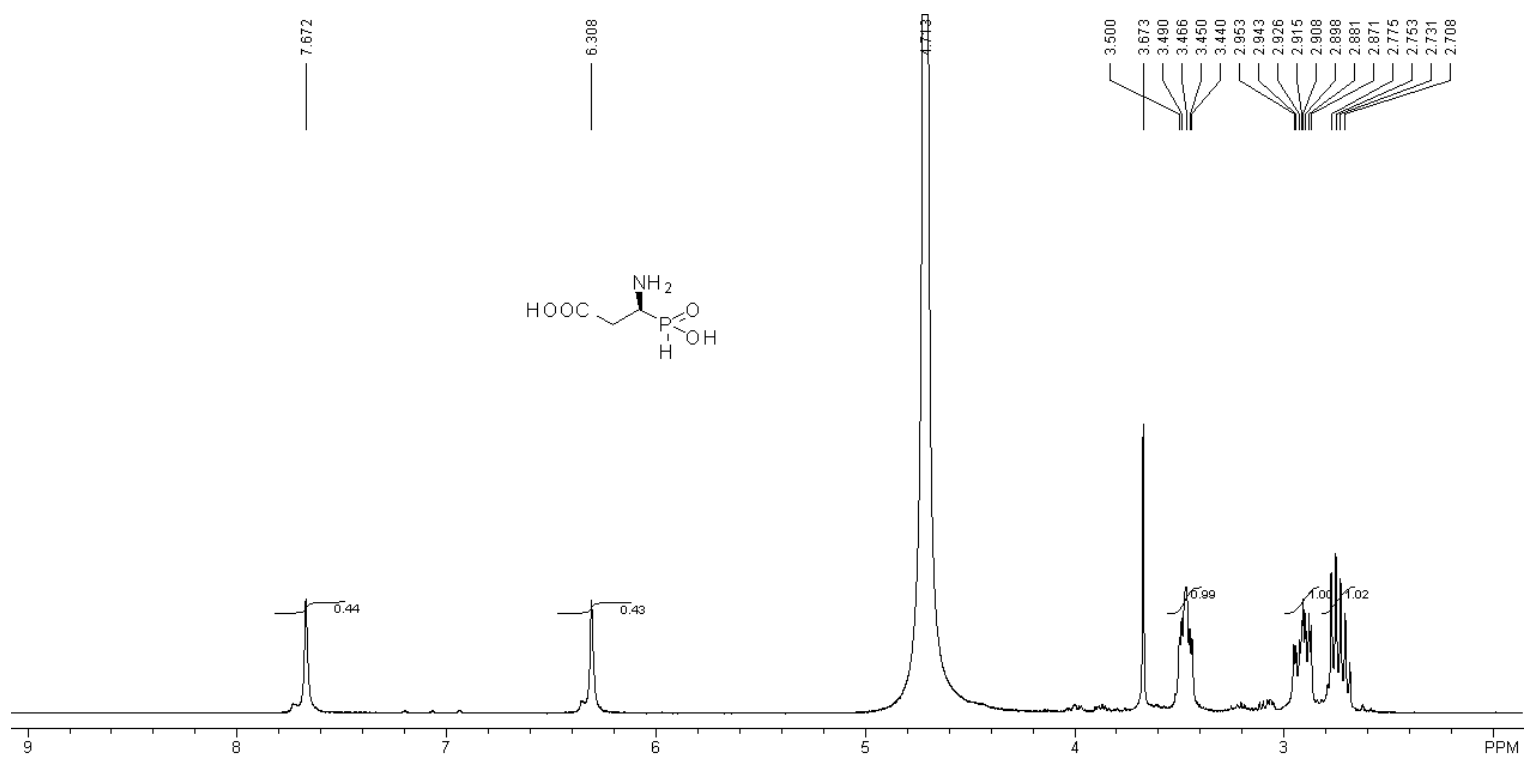
³¹P NMR:

D:\software\nuts\DATA\p-2-76.fid
 Std phosphorus;blank line
 Jan 28 2010
 USER:
 SOLVENT: d2o
 Experiment = s2pul
 Pulse length = 4.500 usec
 Recycle delay = 1.000 sec
 NA = 6
 Solvent = d2o
 FID PTS1d = 190479
 PTS1d = 262144
 F1 = 161.774841 MHz
 F2 = 399.625275 MHz
 SW1 = 119047.62 Hz
 AT1 = 1.60 sec
 Hz per Pt1s0 = 0.45 Hz
 SW2 = 1.00 Hz
 Hz per Pt2nd0 = 1.00 Hz
 Q1 = 4372.1875 Hz
 Q2 = -0.5000 Hz
 LB1 = 0.00 Hz
 TP A = 223.13
 B = -257.34
 C = 0.00

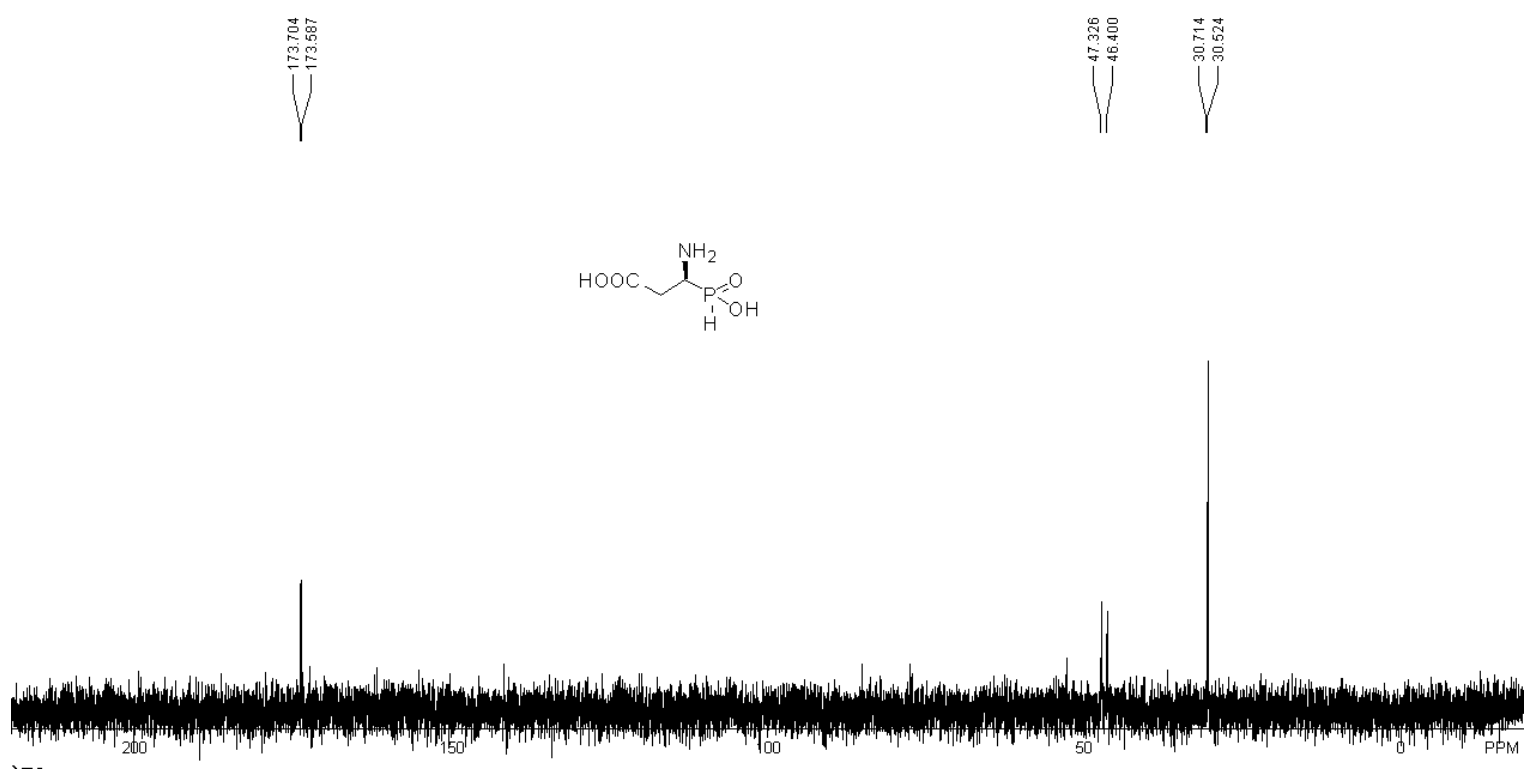


Compound 4v:

¹H NMR:



¹³C NMR:



³¹P NMR:

D:\software\nuts\DATA\2008119-11-37-2H_11.1

10:07:36.125 +0800 nmr@EP-ZH107708

USER: nmr

SOLVENT: D2O

Experiment = zgpg30

Pulse length = 8.300 usec

Recycle delay = 2.000 sec

NA = 16

Solvent = D2O

PTS1d = 32768

F1 = 161.971542 MHz

F2 = 1.000000 MHz

SW1 = 64102.56 Hz

AT1 = 0.51 sec

Hz per Pt1stD = 1.96 Hz

SW2 = 1.00 Hz

Hz per Pt2ndD = 1.00 Hz

O1 = -8098.6865 Hz

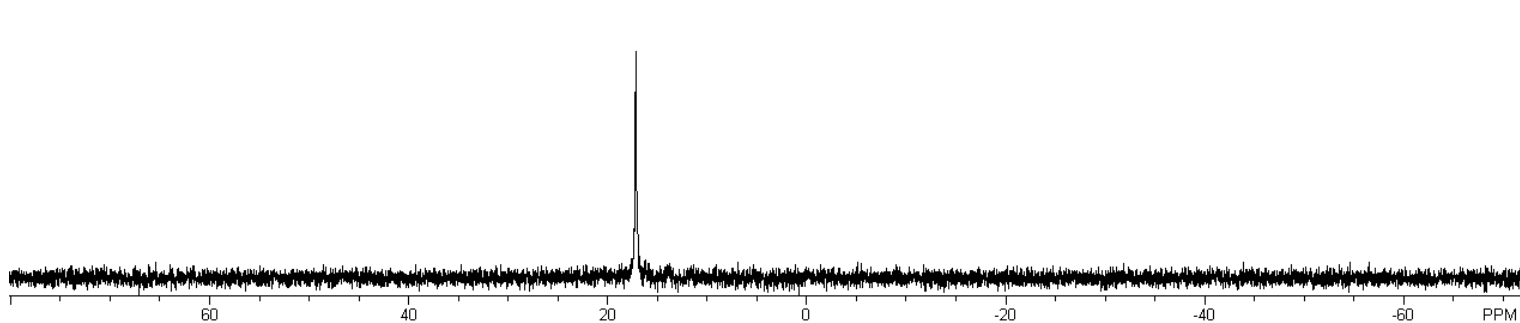
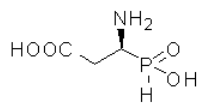
O2 = -1.0000 Hz

LB1 = 1.00 Hz

TP A = 0.00

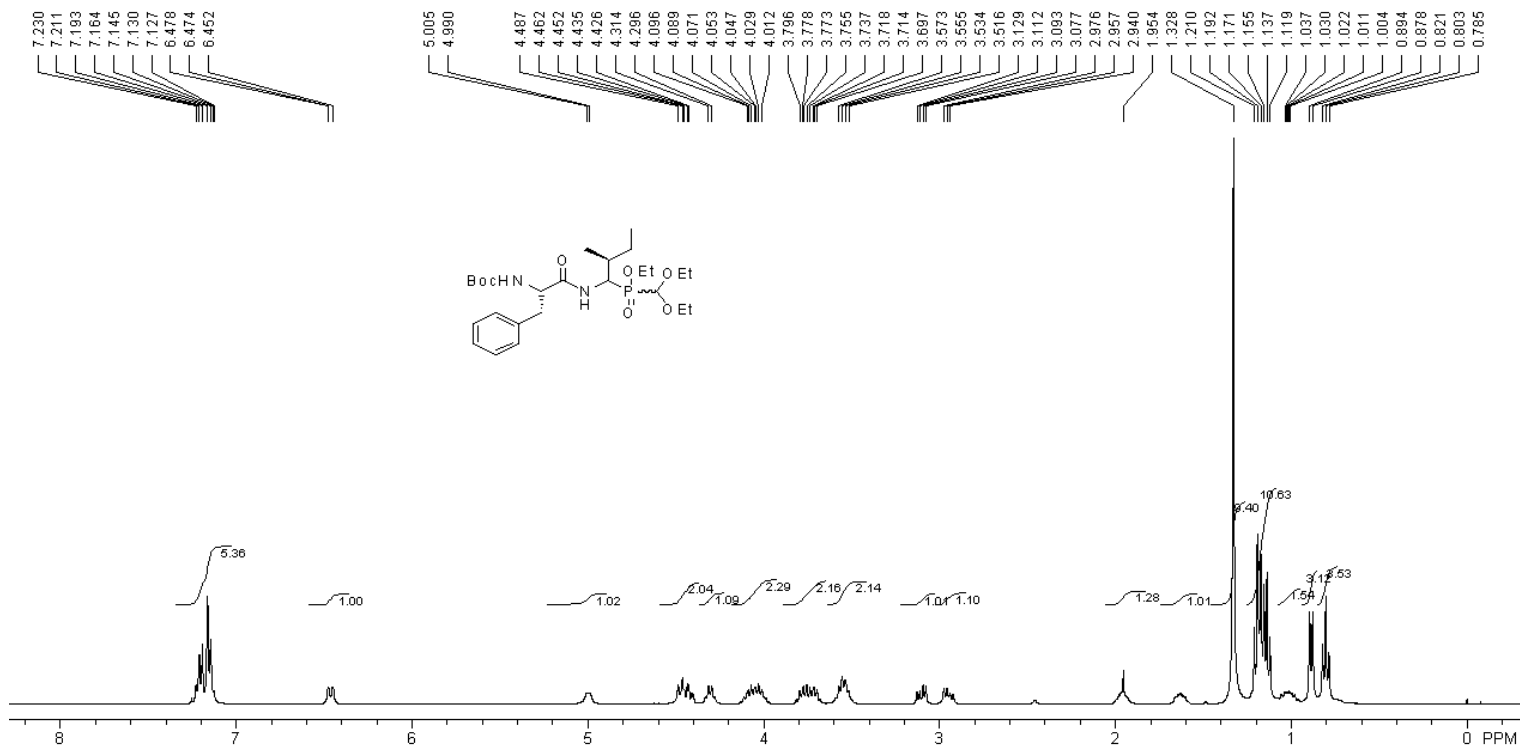
B = 0.00

C = 0.00

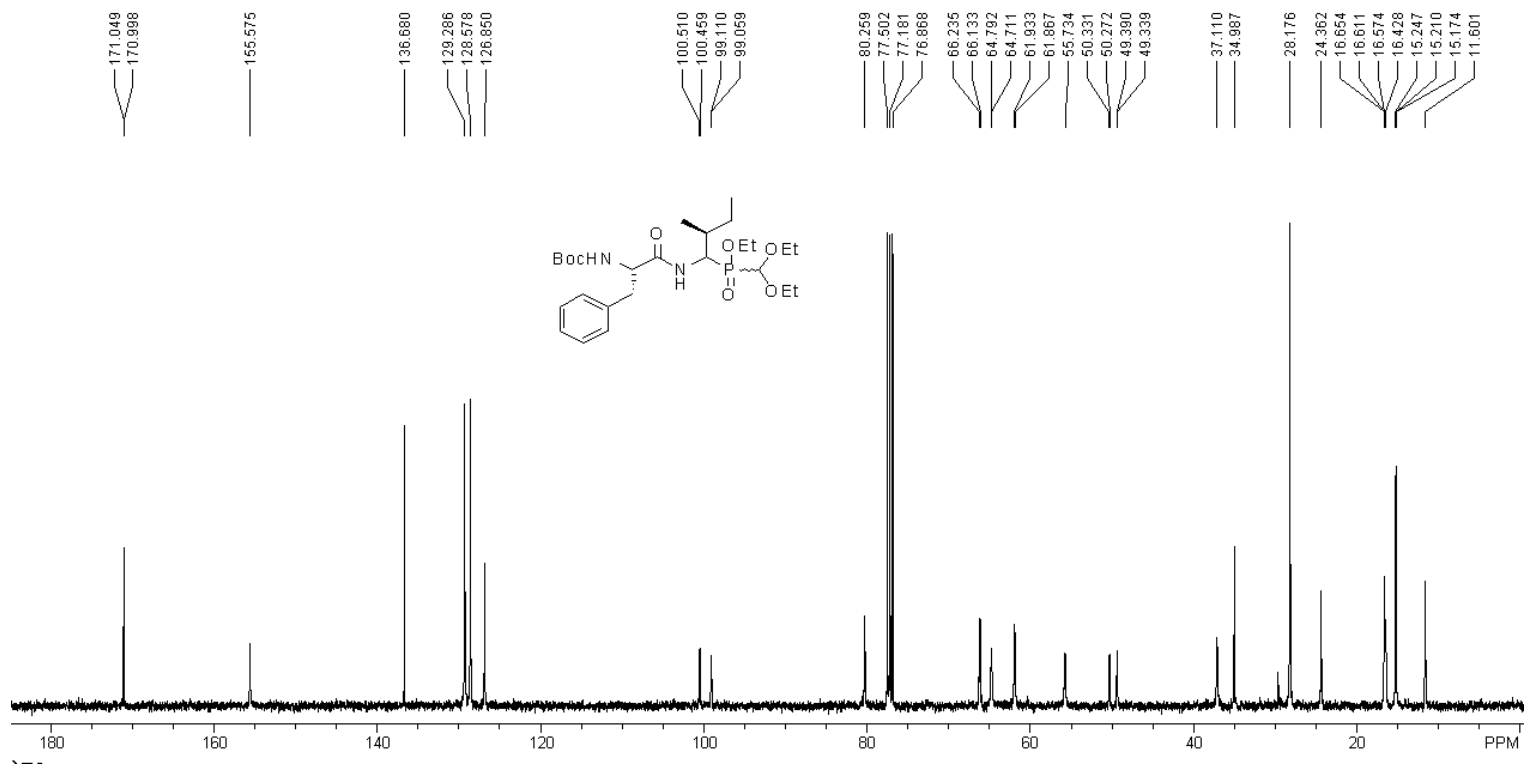


Compound 10a:

¹H NMR:



¹³C NMR:



³¹P NMR:

D:\software\nuts\DATA\nmr_12-63P.1

11:22:15.875 +0800 nmr@EP.ZH107708

USER: nmr

SOLVENT: CDCl3

Experiment = zgpg30

Pulse length = 8.300 usec

Recycle delay = 2.000 sec

NA = 16

Solvent = CDCl3

PTSD = 32768

F1 = 161.971542 MHz

F2 = 1.000000 MHz

SWH = 64102.56 Hz

AT1 = 0.51 sec

Hz per Pt1sd = 1.96 Hz

SW2 = 1.00 Hz

Hz per Pt2ndd = 1.00 Hz

O1 = -8098.6865 Hz

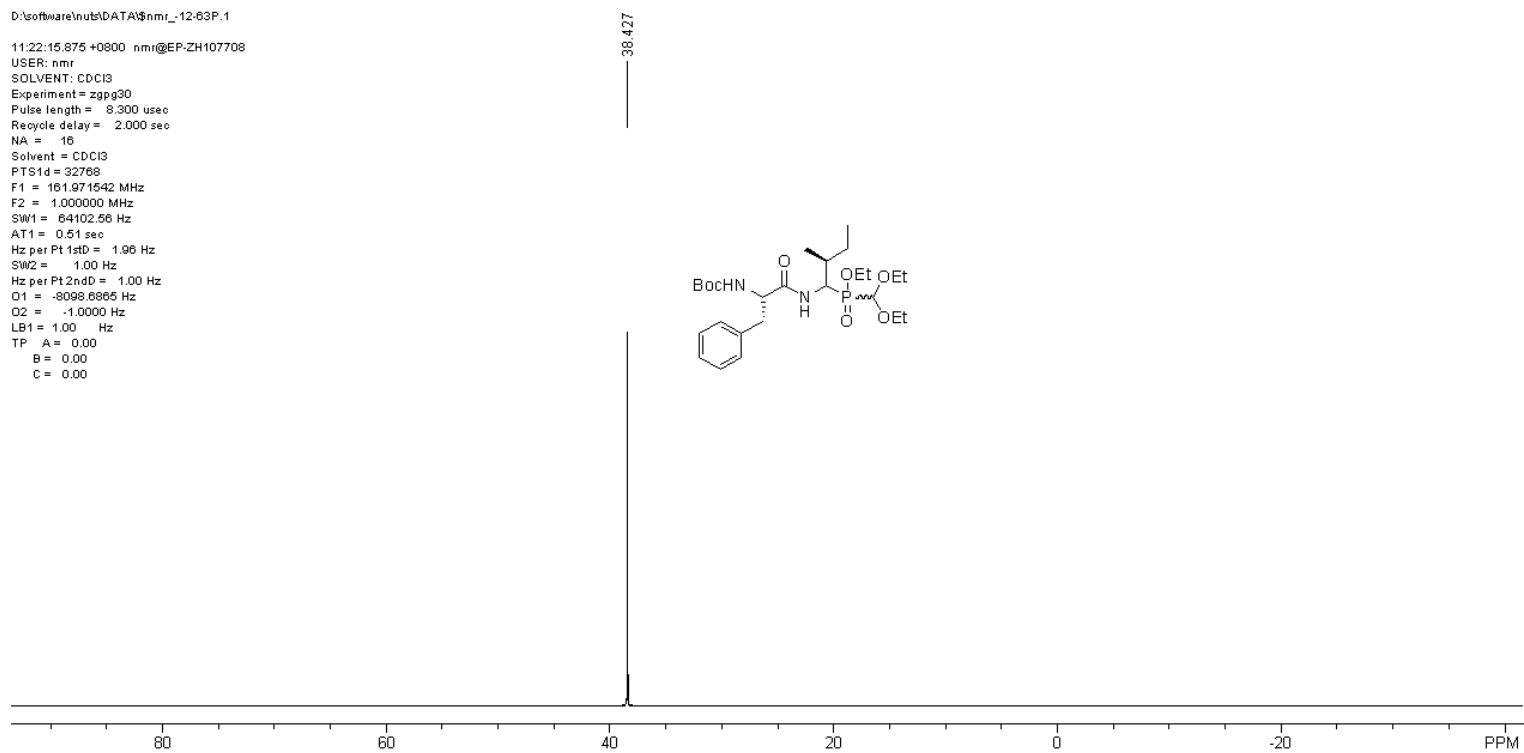
O2 = -1.0000 Hz

LB1 = 1.00 Hz

TP A = 0.00

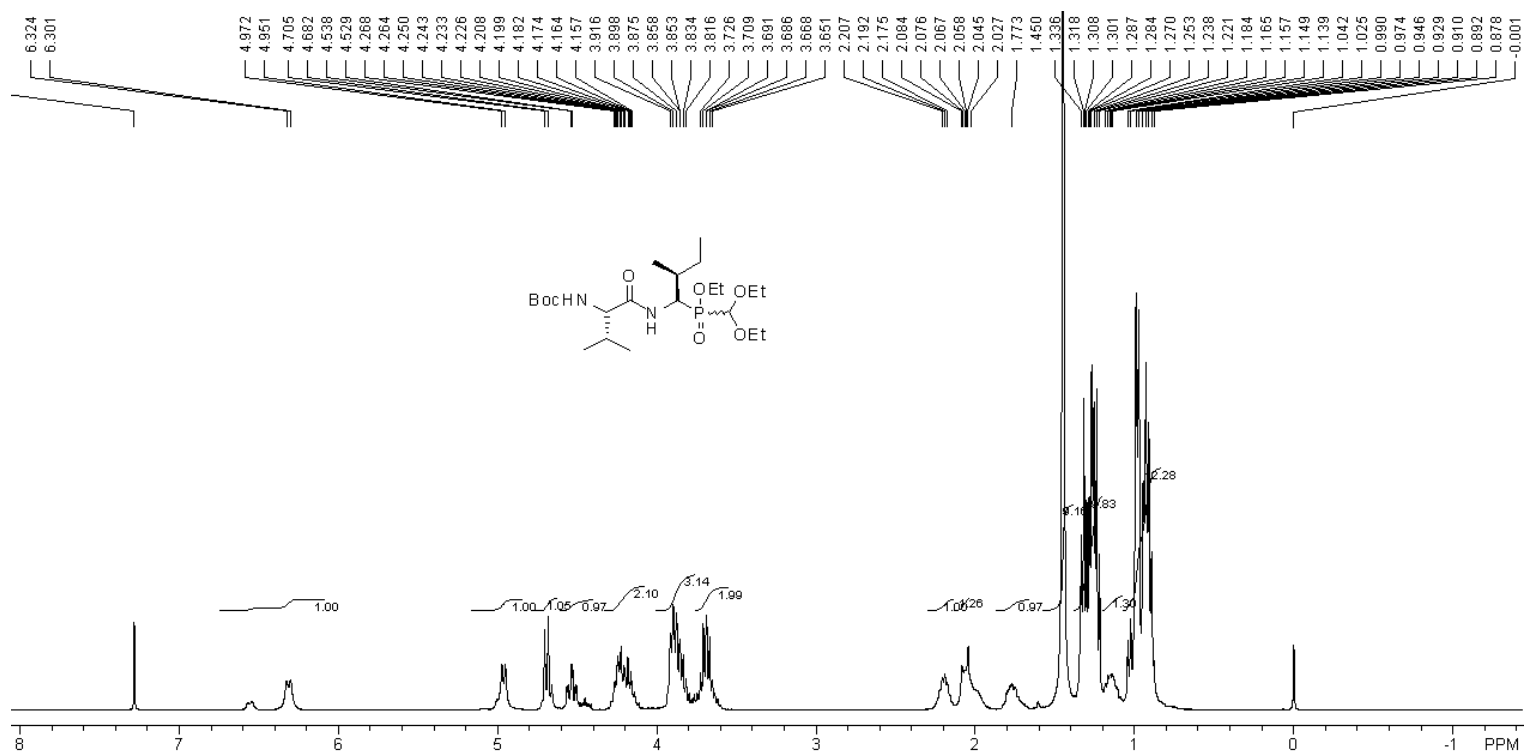
B = 0.00

C = 0.00

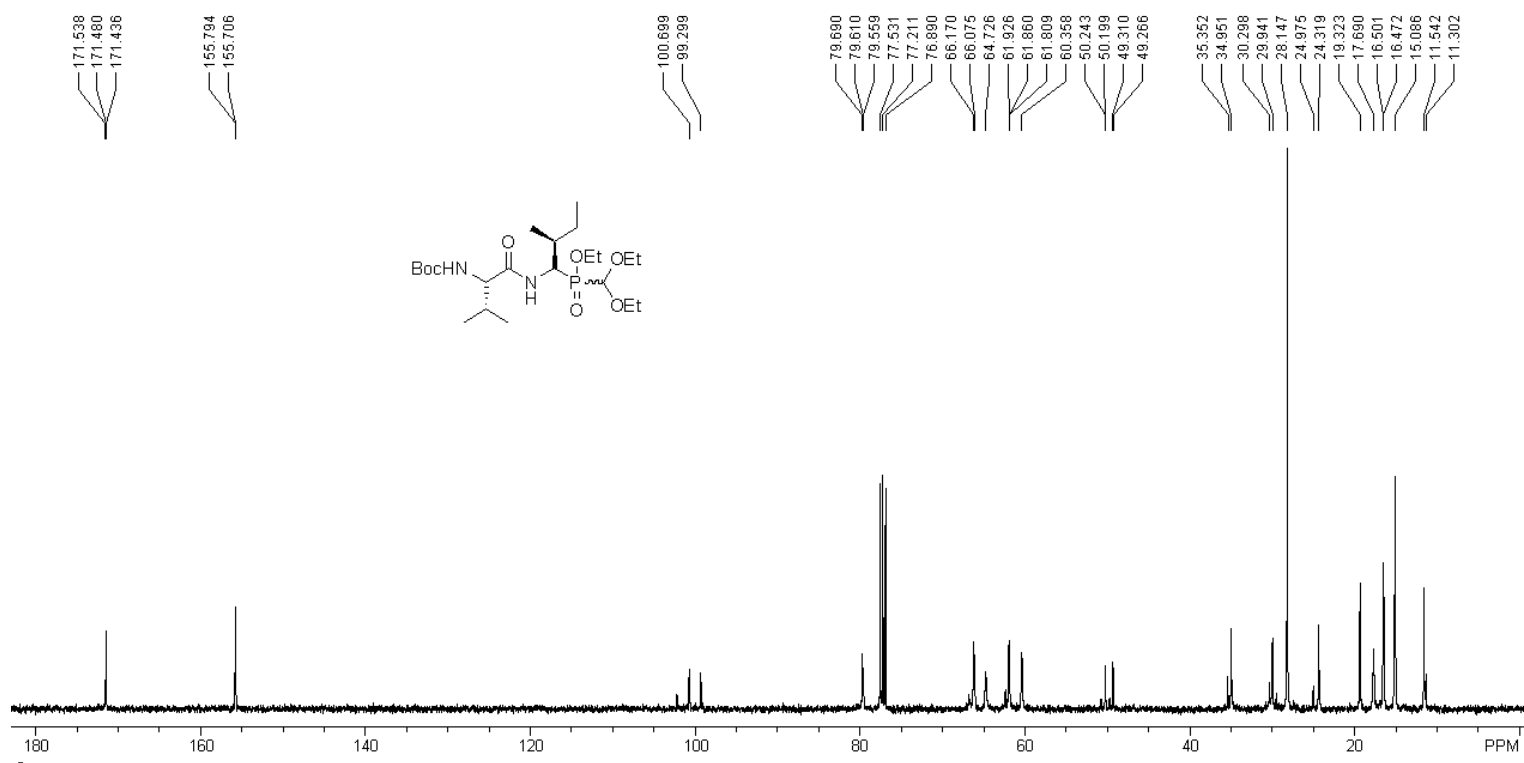


Compound 10b:

¹H NMR:

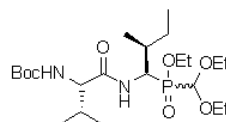
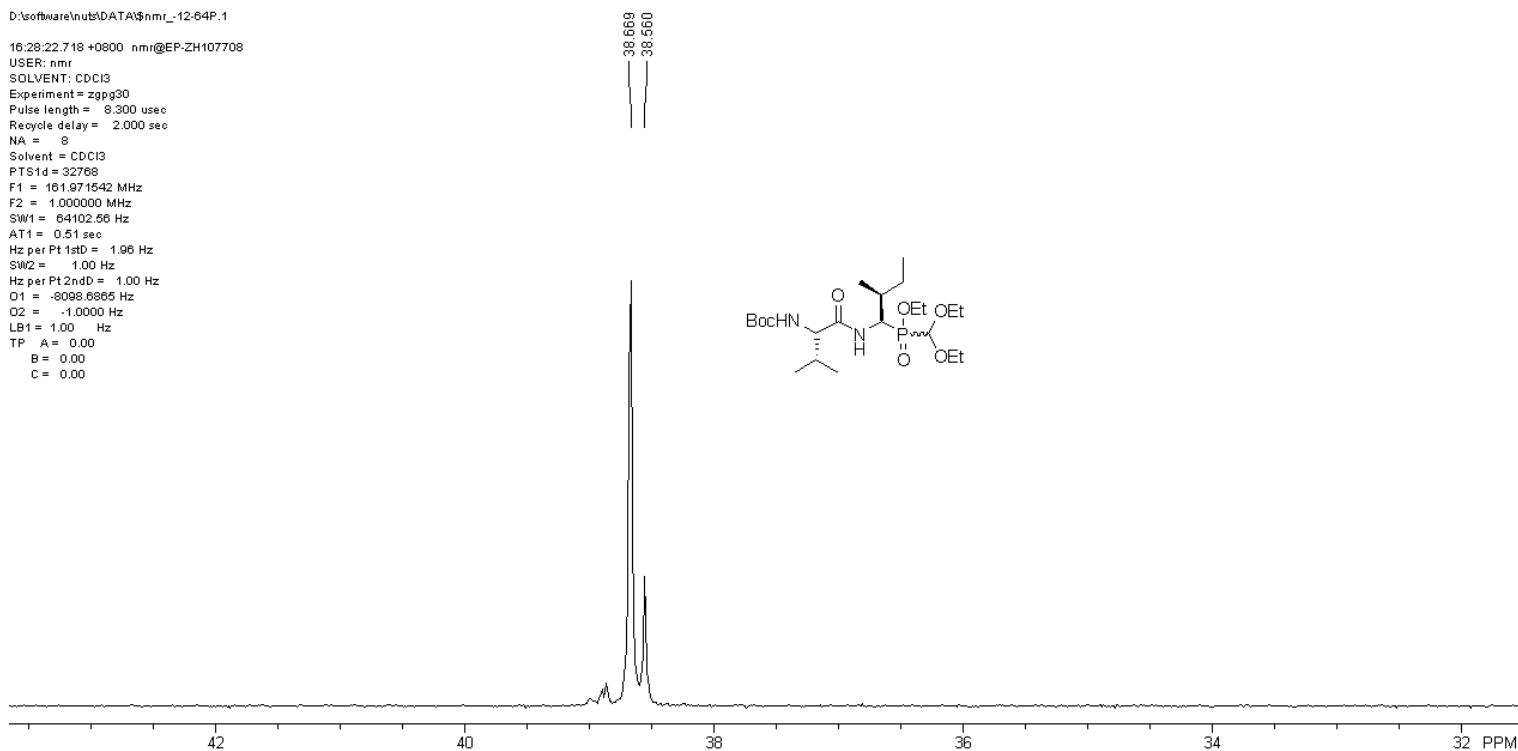


¹³C NMR:



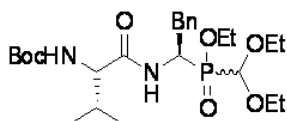
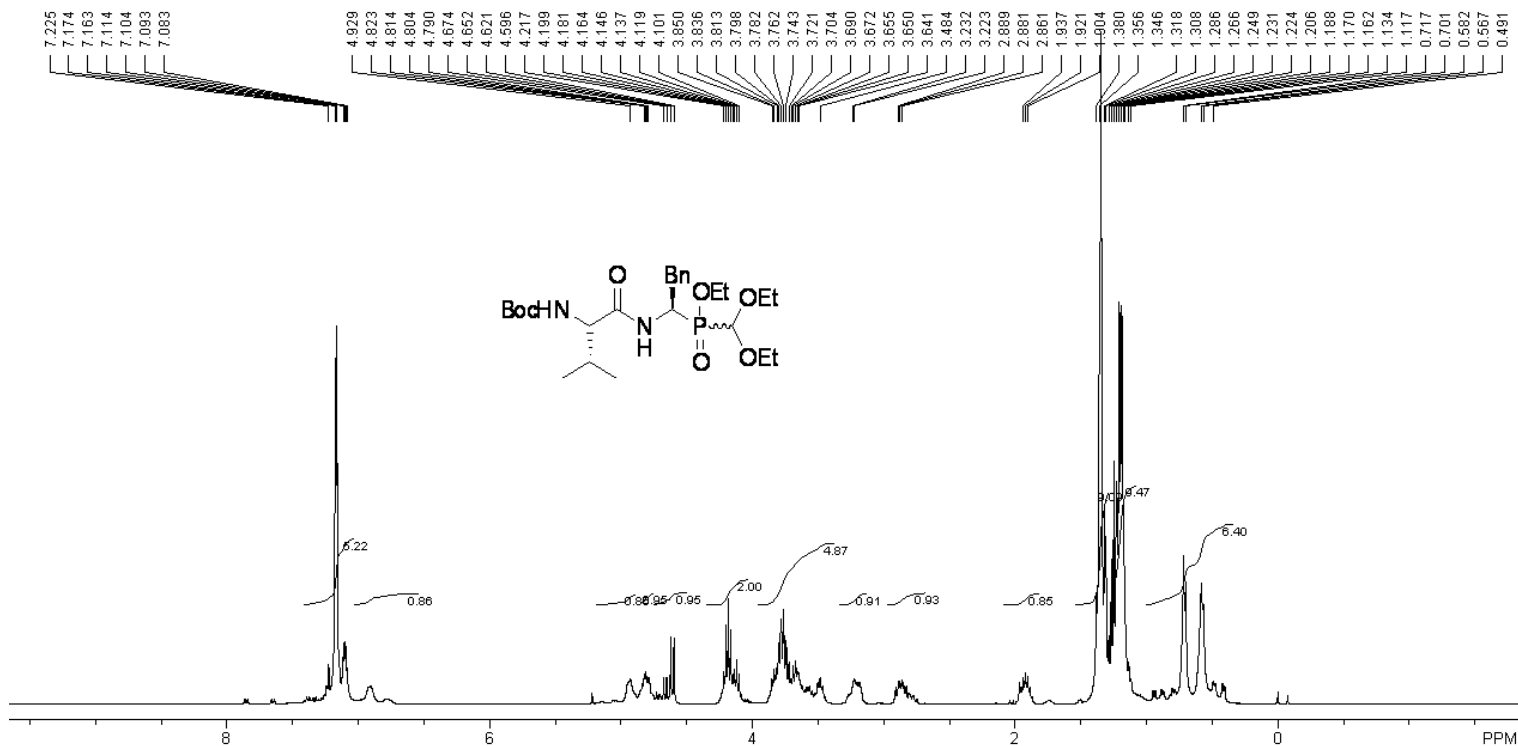
³¹P NMR:

D:\software\nuts\DATA\nmr_12-64P.1
 16:28:22.718 +0800 nmr@EP-ZH107708
 USER: nmr
 SOLVENT: CDCl3
 Experiment = zgpg30
 Pulse length = 8.300 usec
 Recycle delay = 2.000 sec
 NA = 8
 Solvent = CDCl3
 P1 = 161.971542 MHz
 F2 = 1.000000 MHz
 SW1 = 64102.56 Hz
 AT1 = 0.51 sec
 Hz per Pt1stD = 1.96 Hz
 SW2 = 1.00 Hz
 Hz per Pt2ndD = 1.00 Hz
 O1 = -8098.6865 Hz
 O2 = -1.0000 Hz
 LB1 = 1.00 Hz
 TP A = 0.00
 B = 0.00
 C = 0.00

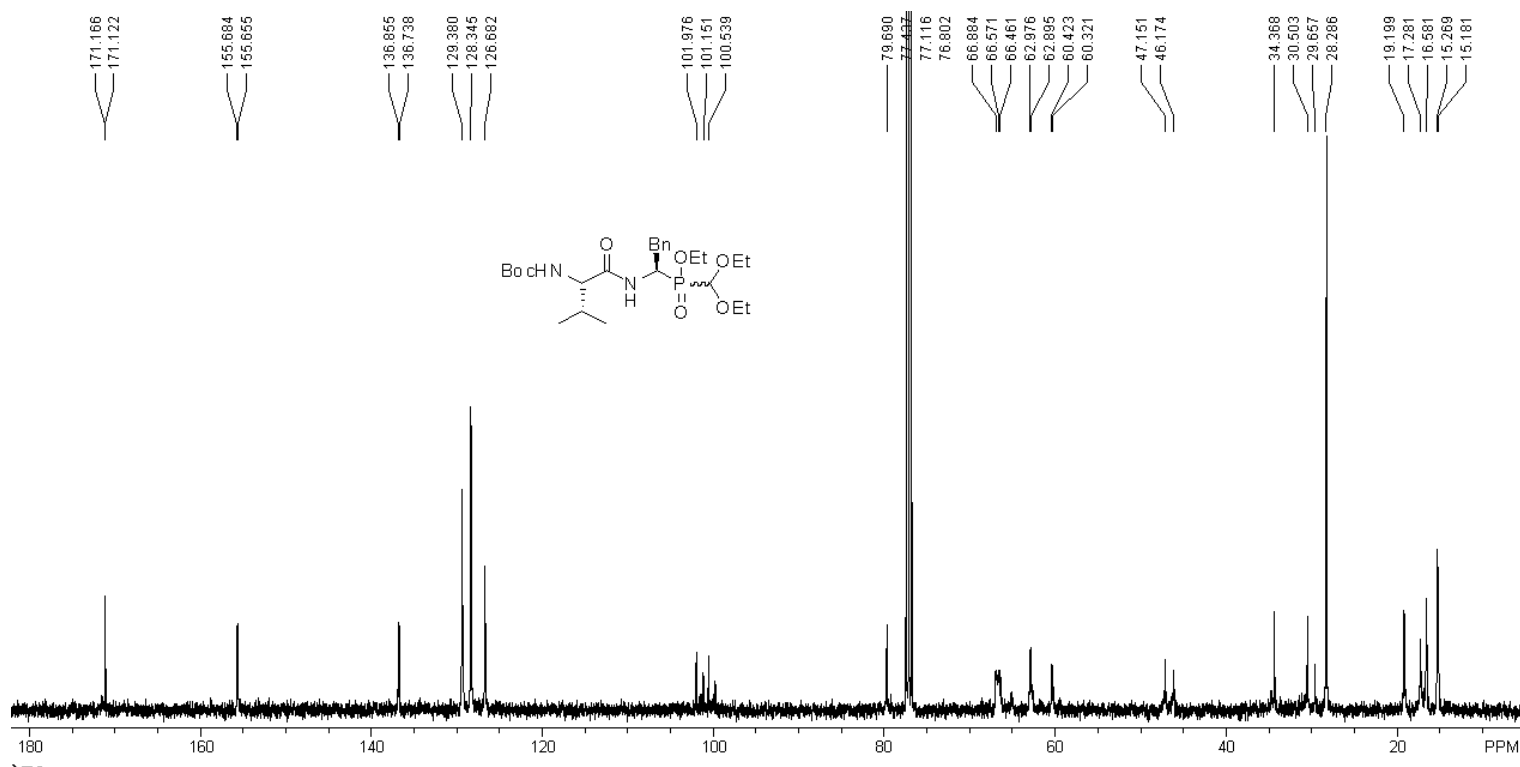


Compound 10c:

¹H NMR:



¹³C NMR:



³¹P NMR:

D:\software\nuts\DATA\nmr_12-67P_1

07:44:56.953 +0800 nmri@EP-ZH107708

USER: nmri

SOLVENT: CDCl3

Experiment = zgpg30

Pulse length = 8.300 usec

Recycle delay = 2.000 sec

NA = 16

Solvent = CDCl3

PTS1d = 32768

F1 = 161.971542 MHz

F2 = 1.000000 MHz

SW1 = 64102.56 Hz

AT1 = 0.51 sec

Hz per Pt1s0 = 1.96 Hz

SW2 = 1.00 Hz

Hz per Pt2ndD = 1.00 Hz

O1 = -8098.6965 Hz

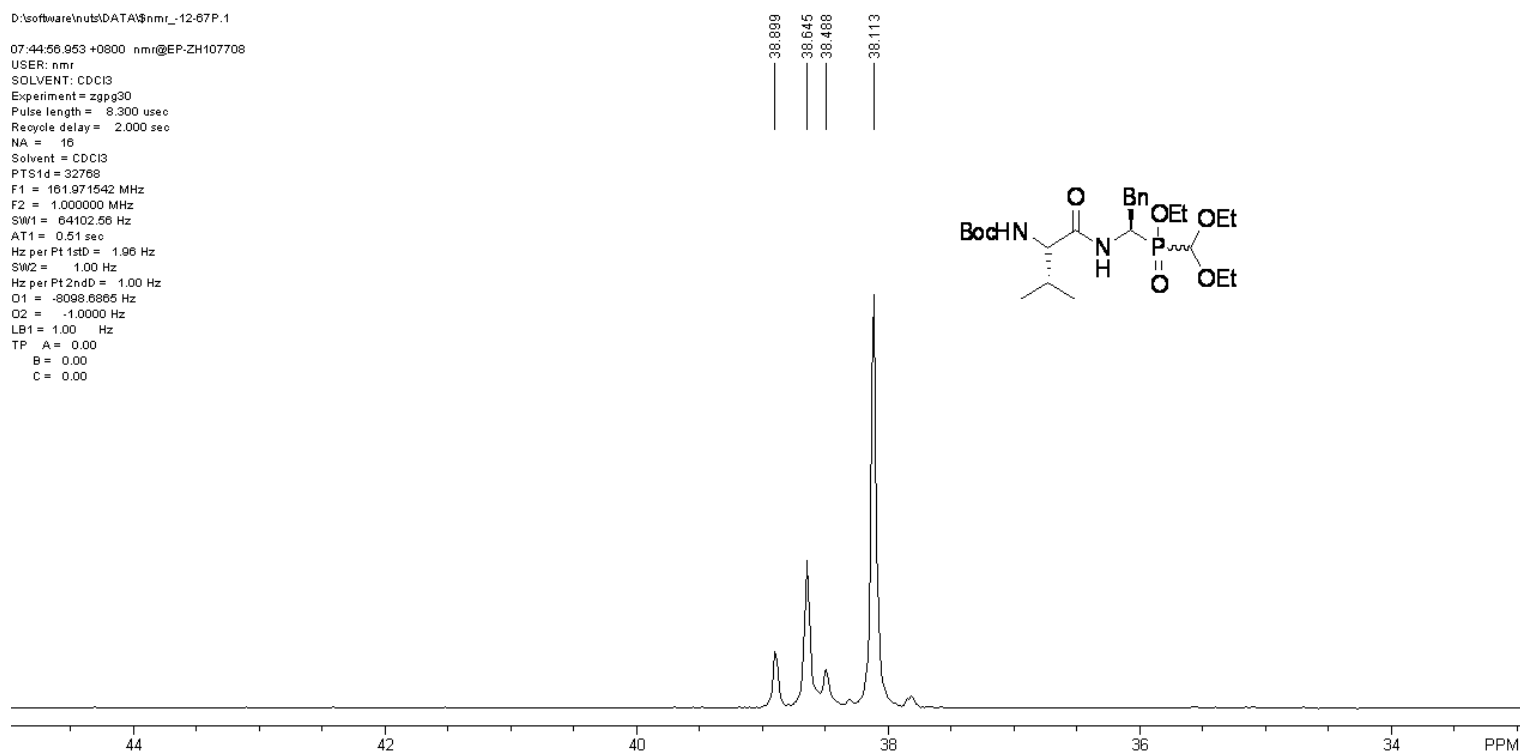
O2 = -1.0000 Hz

LB1 = 1.00 Hz

TP A = 0.00

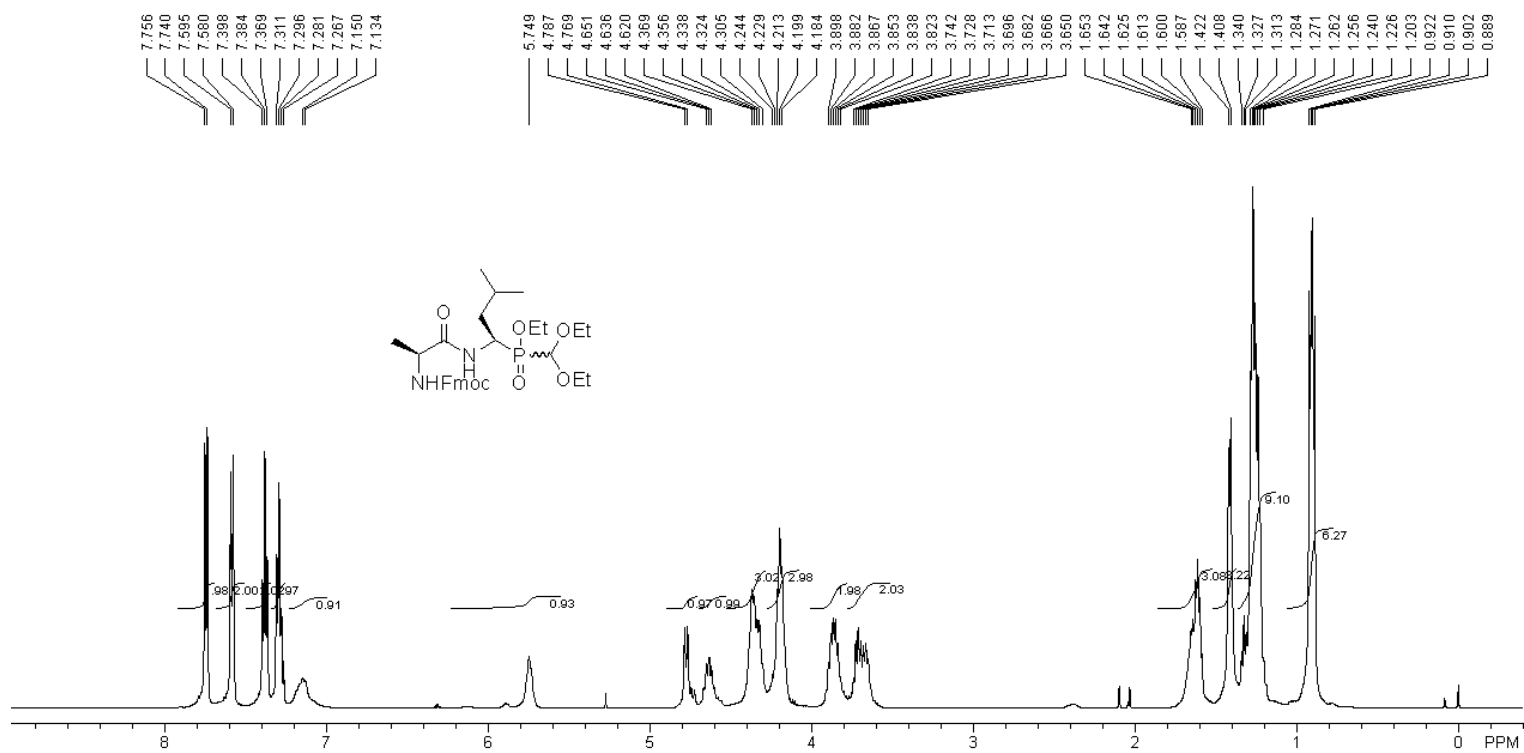
B = 0.00

C = 0.00

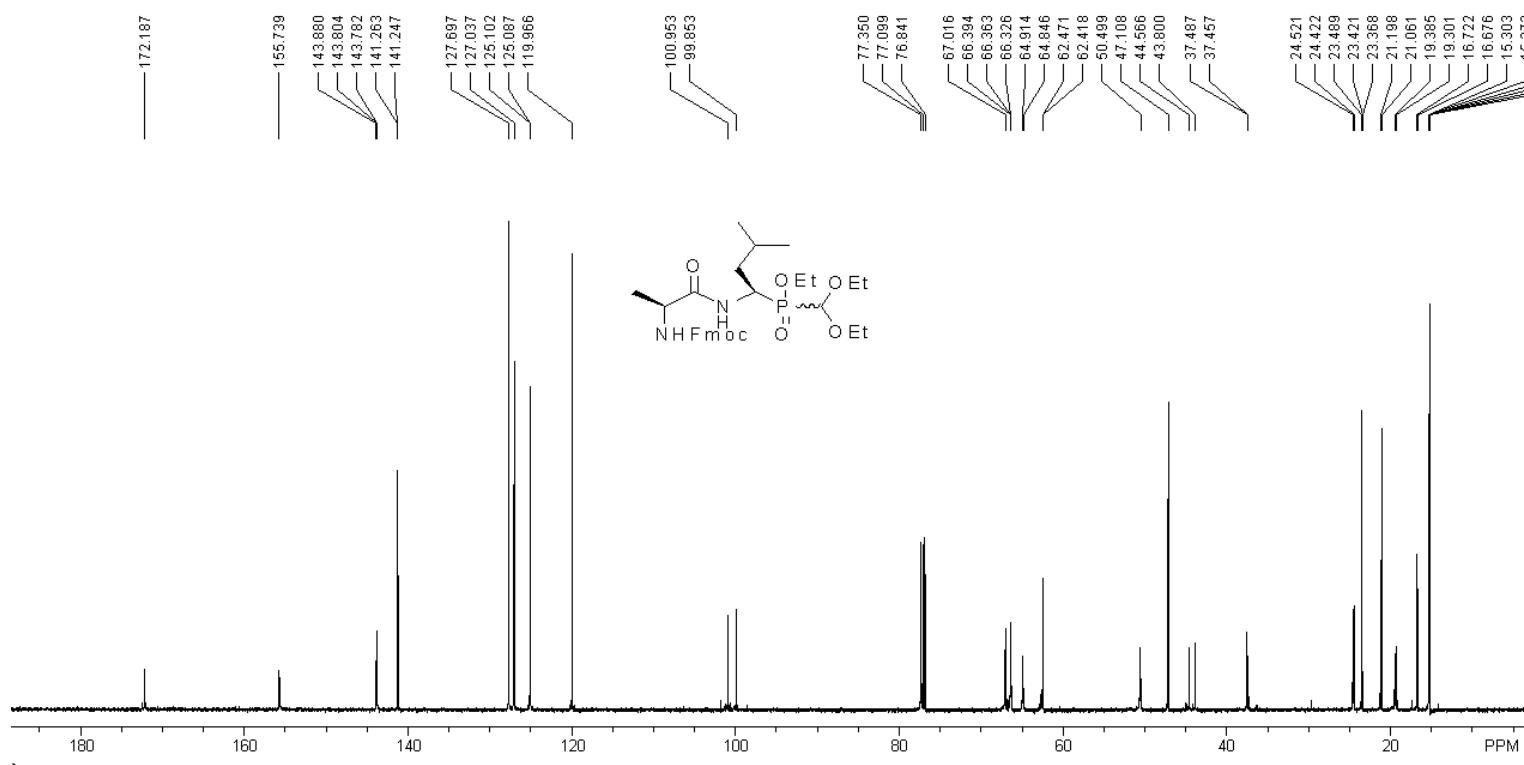


Compound 10d:

¹H NMR:



¹³C NMR:

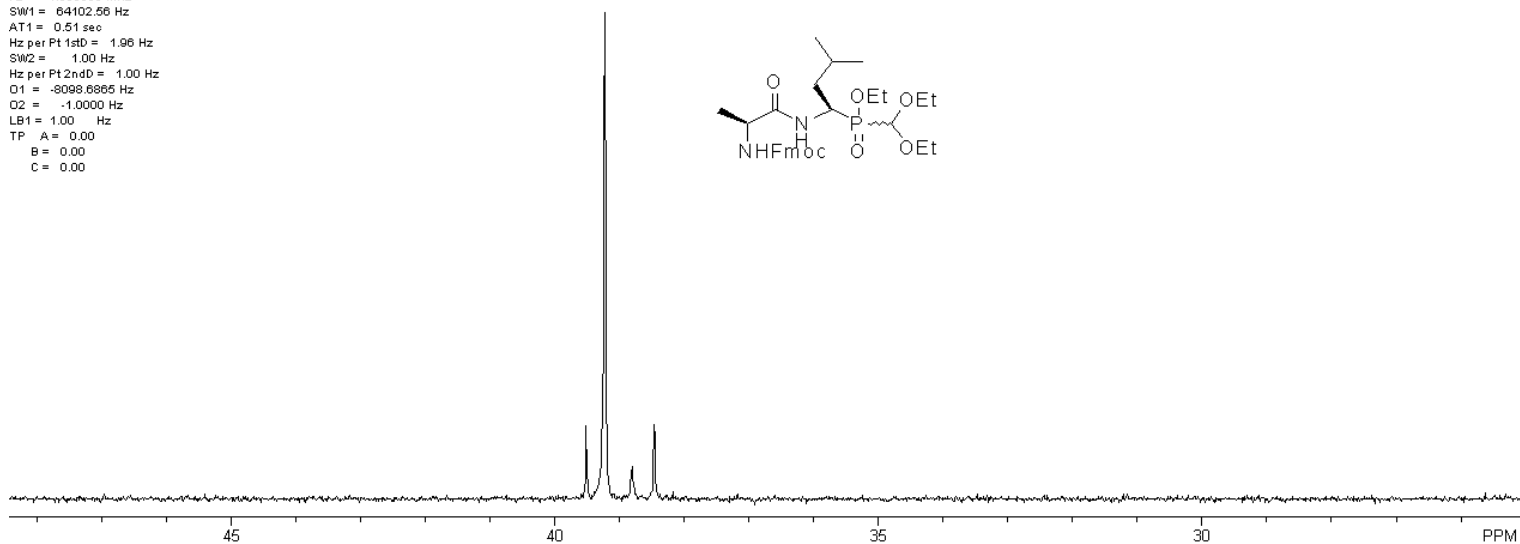
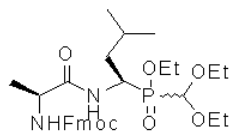


³¹P NMR:

D:\software\nuts\DATA\2008119-11-48H_11.1

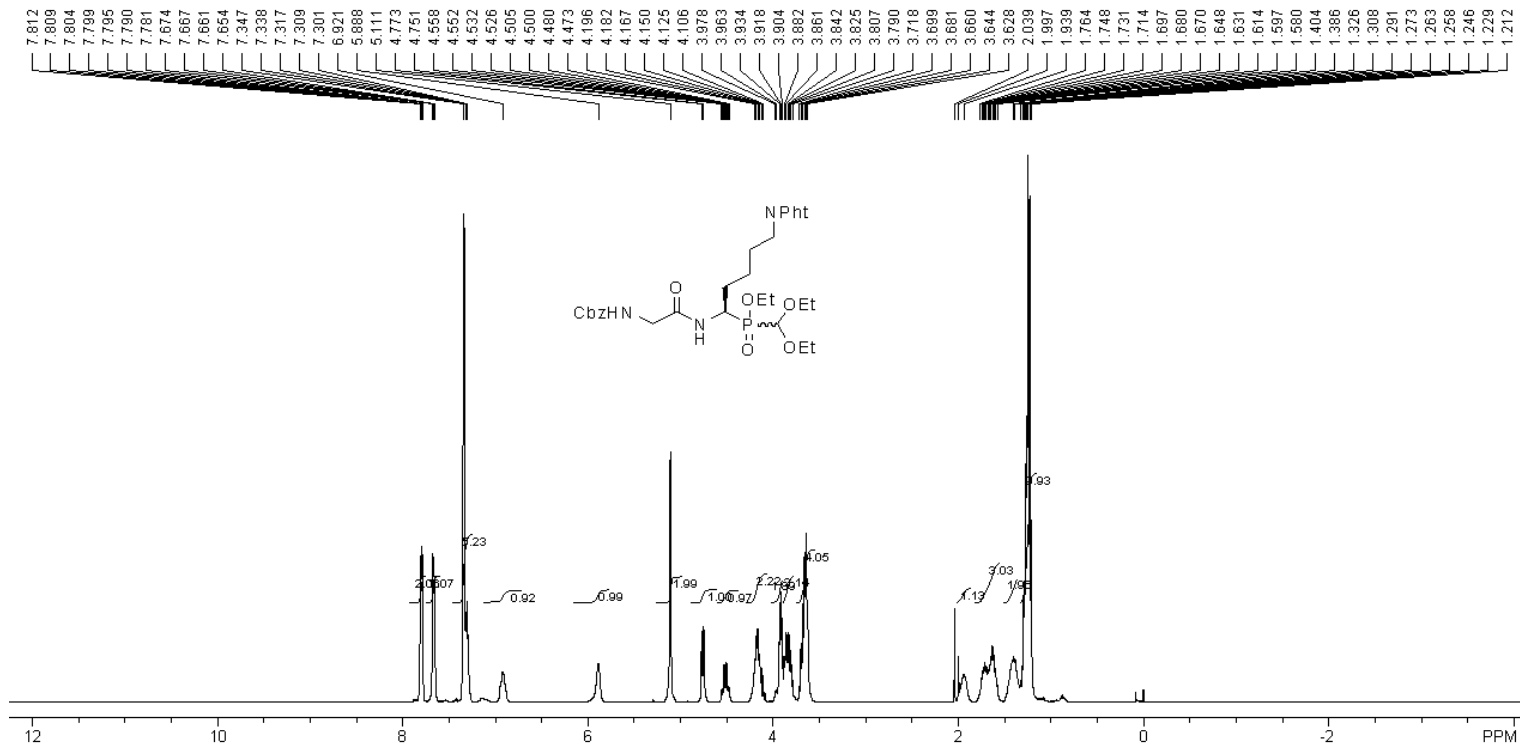
17:36:15.312+0800 nmr@EP-ZH107708
 USER: nmr
 SOLVENT: CDCl3
 Experiment = zgpg30
 Pulse length = 8.300 usec
 Recycle delay = 2.000 sec
 NA = 8
 Solvent = CDCl3
 PTS1d = 32768
 F1 = 161.971542 MHz
 F2 = 1.000000 MHz
 SW1 = 64102.56 Hz
 AT1 = 0.51 sec
 Hz per Pt1stD = 1.96 Hz
 SW2 = 1.00 Hz
 Hz per Pt2ndD = 1.00 Hz
 O1 = -8098.6865 Hz
 O2 = -1.0000 Hz
 LB1 = 1.00 Hz
 TP A = 0.00
 B = 0.00
 C = 0.00

39.514
 39.225
 38.802
 38.464

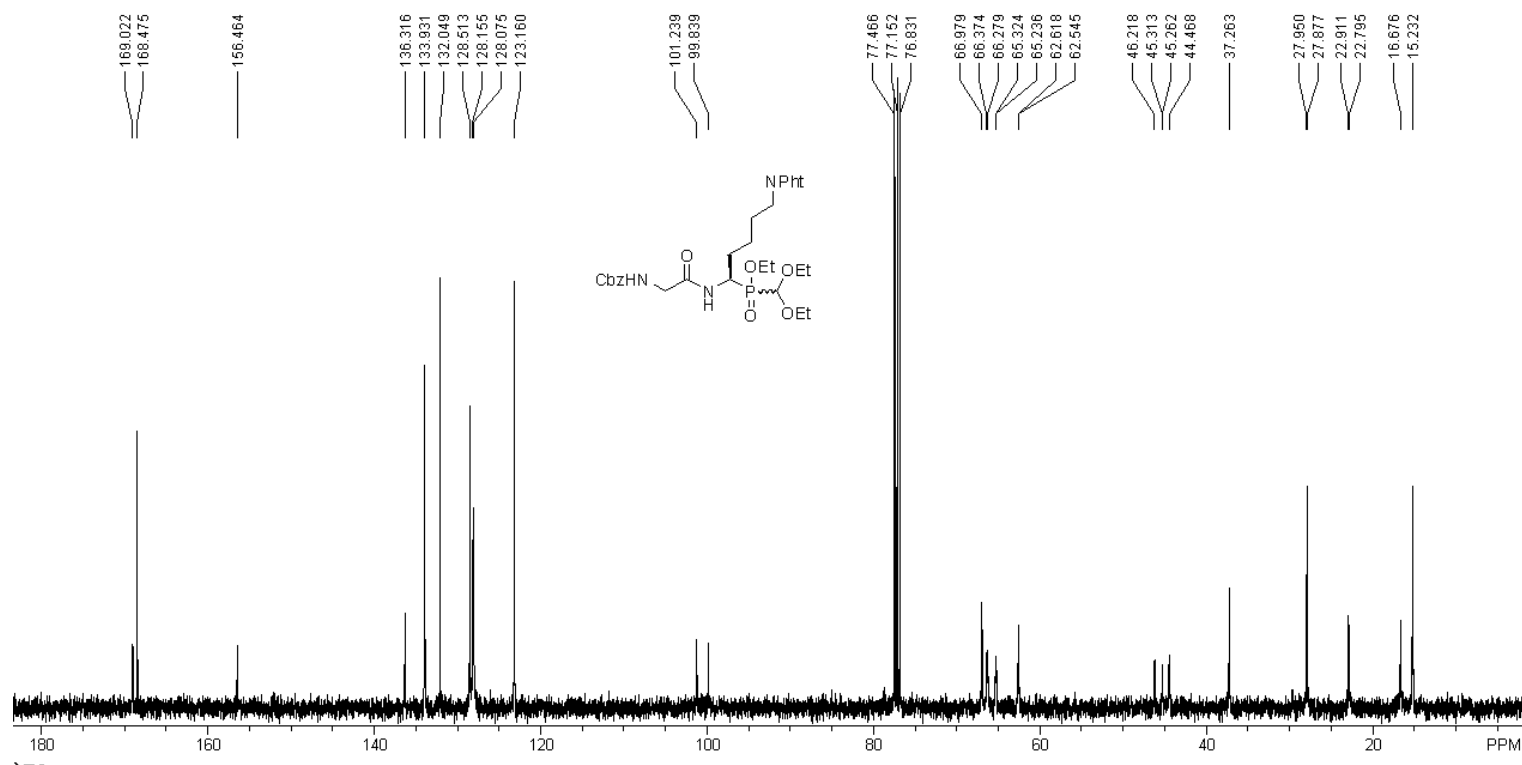


Compound 10c:

¹H NMR:



¹³C NMR:



³¹P NMR:

D:\software\nuts\DATA\2008119-11-11_11.1

16:38:00.921 +0800 nmri@EP.ZH107708

USER: nmri

SOLVENT: CDCl3

Experiment = zgpg30

Pulse length = 8.300 usec

Recycle delay = 2.000 sec

NA = 8

Solvent = CDCl3

PTS1d = 32768

F1 = 161.971542 MHz

F2 = 1.000000 MHz

SWH = 64102.56 Hz

AT1 = 0.51 sec

Hz per Pt1s0 = 1.96 Hz

SW2 = 1.00 Hz

Hz per Pt2nd0 = 1.00 Hz

Q1 = -8098.6965 Hz

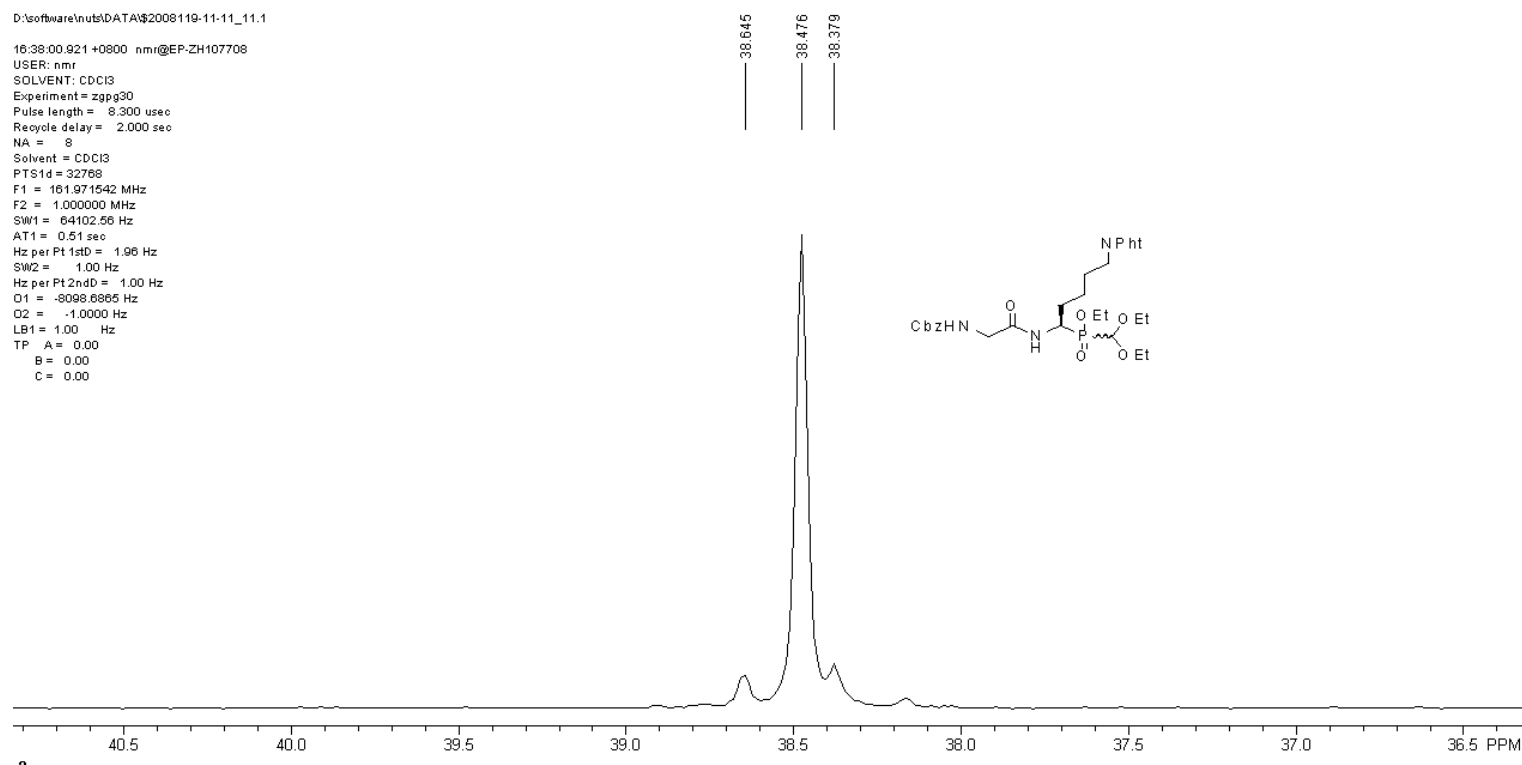
Q2 = -1.0000 Hz

LB1 = 1.00 Hz

TP A = 0.00

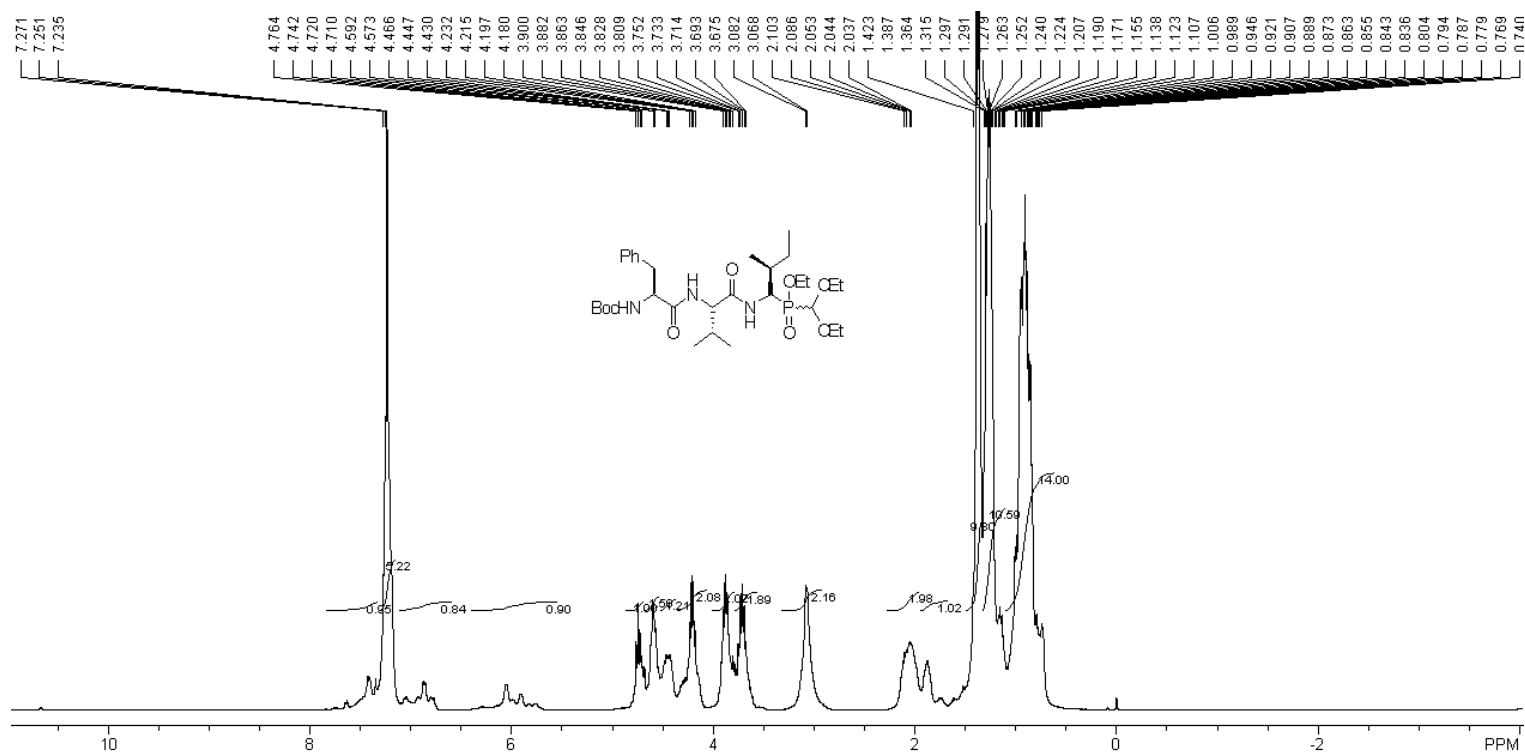
B = 0.00

C = 0.00

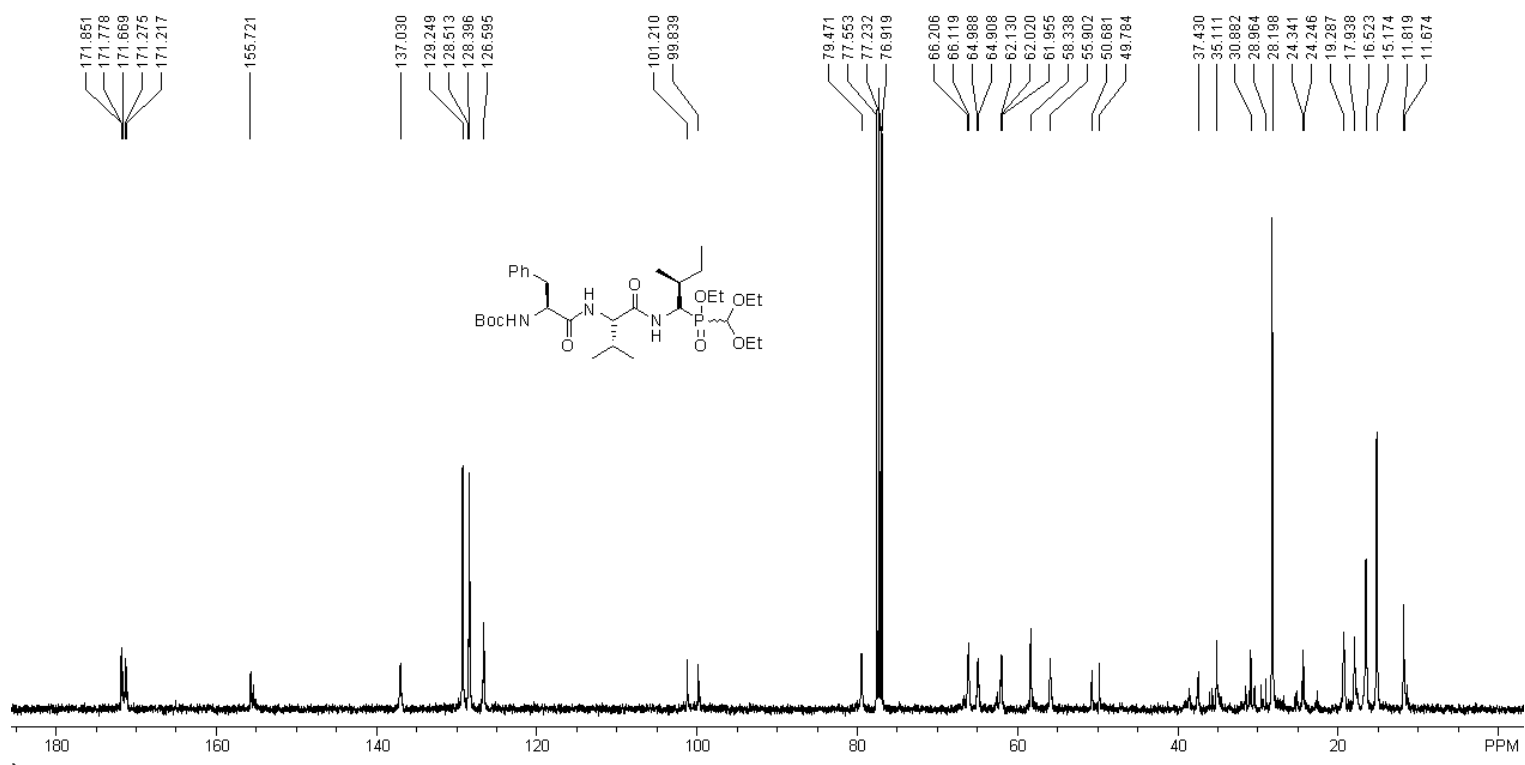


Compound 10f:

¹H NMR:

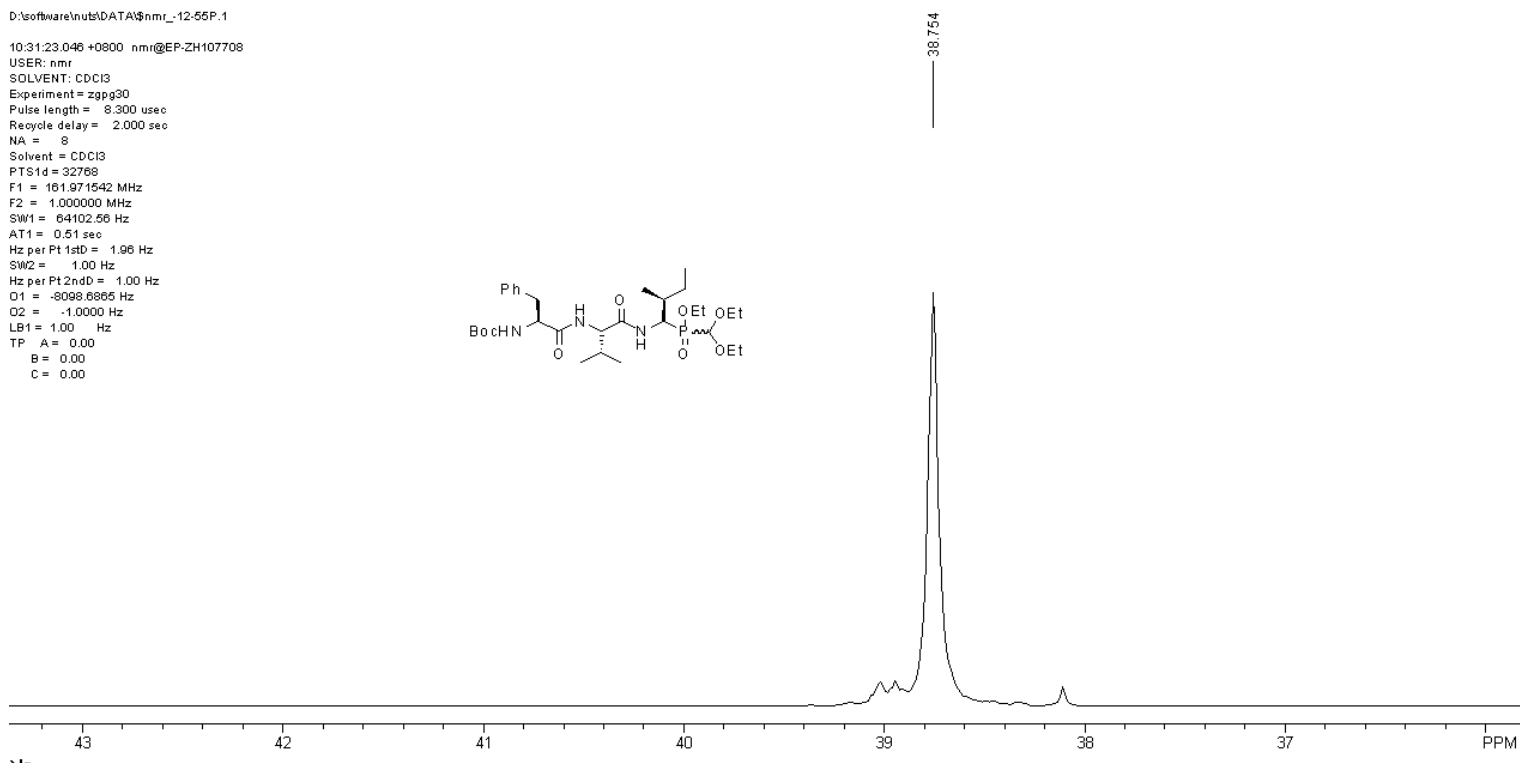
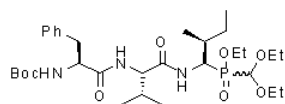


¹³C NMR:



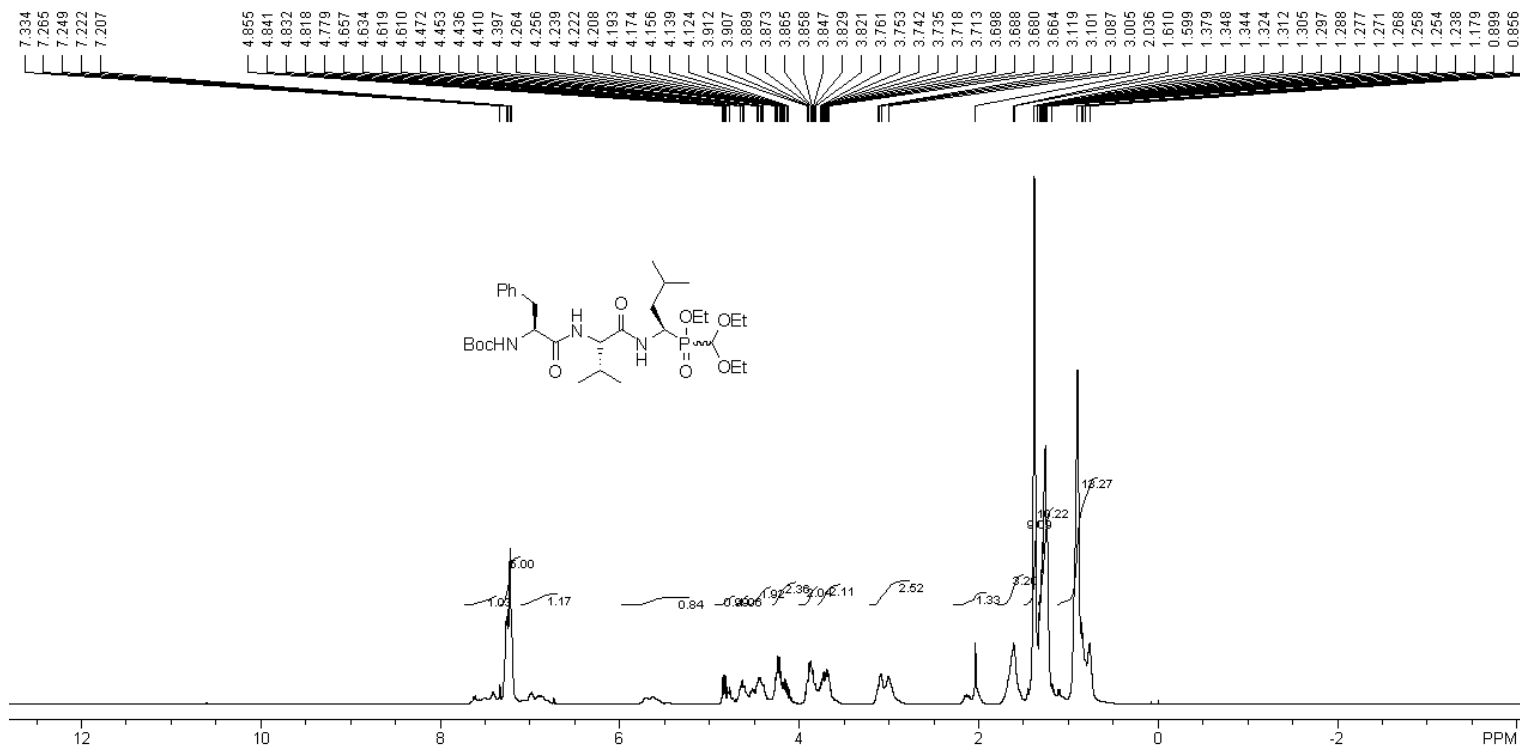
³¹P NMR:

D:\software\nmr\DATA\nmr_12-55P.1
 10:31:23.046 +0800 nmr@EP-ZH107708
 USER: nmr
 SOLVENT: CDCl3
 Experiment: zgpg30
 Pulse length = 8.300 usec
 Recycle delay = 2.000 sec
 NA = 8
 Solvent = CDCl3
 PTS1d = 32768
 F1 = 161.971542 MHz
 F2 = 1.000000 MHz
 SW1 = 64102.56 Hz
 AT1 = 0.51 sec
 Hz per Pt1stD = 1.96 Hz
 SW2 = 1.00 Hz
 Hz per Pt2ndD = 1.00 Hz
 O1 = -8098.6865 Hz
 O2 = -1.0000 Hz
 LB1 = 1.00 Hz
 TP A = 0.00
 B = 0.00
 C = 0.00

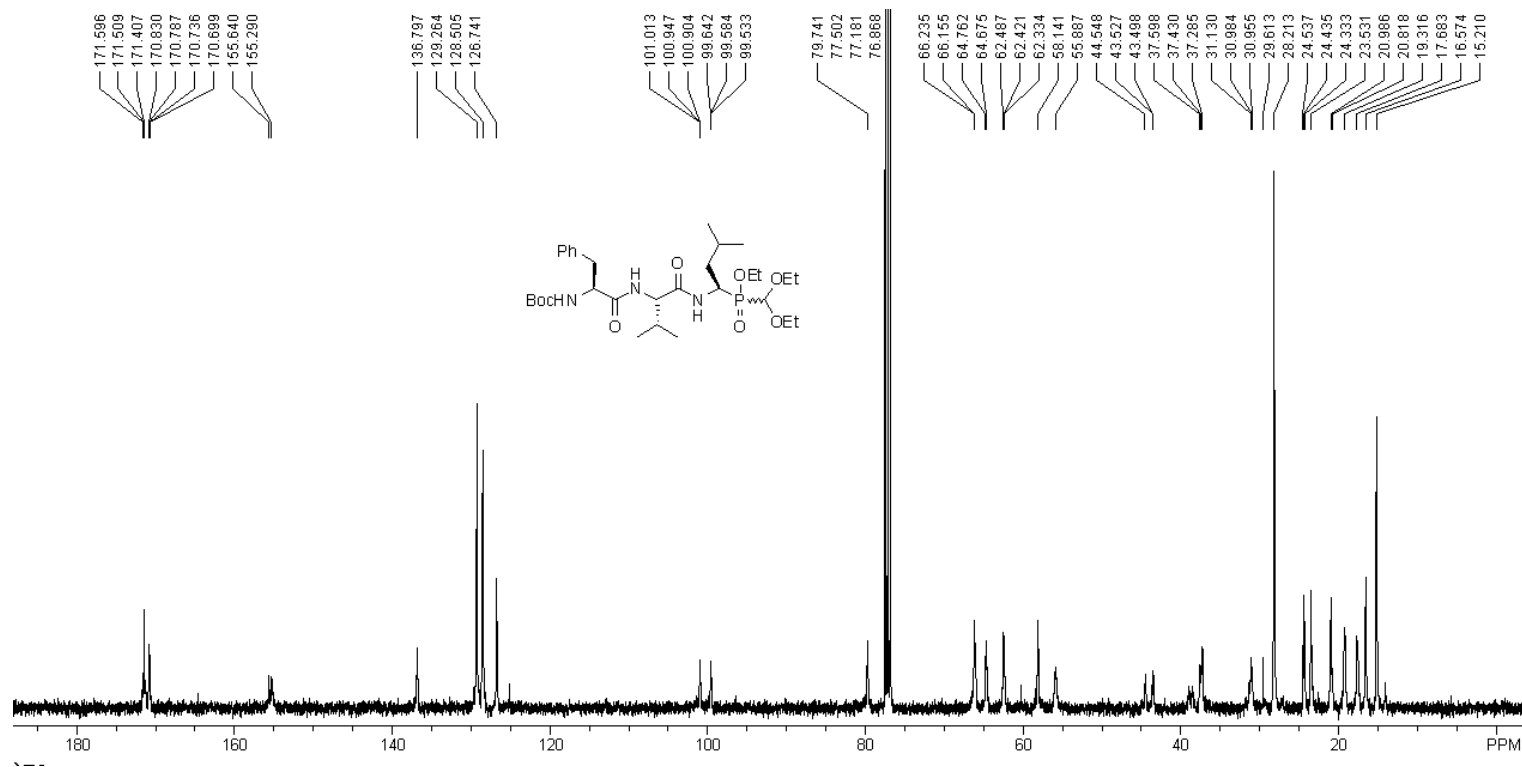


Compound 10g:

¹H NMR:



¹³C NMR:



³¹P NMR:

D:\software\nmr\DATA\nmr_12-61P_1

22.02:49.625 +0800 nmr@EP.ZH107708

USER: nmr

SOLVENT: CDCl3

Experiment = zgpg30

Pulse length = 8.300 usec

Recycle delay = 2.000 sec

NA = 8

Solvent = CDCl3

PTSD = 32768

F1 = 161.971542 MHz

F2 = 1.000000 MHz

SWH = 64102.56 Hz

AT1 = 0.51 sec

Hz per Pt1s0 = 1.96 Hz

SW2 = 1.00 Hz

Hz per Pt2nd0 = 1.00 Hz

O1 = -8098.6865 Hz

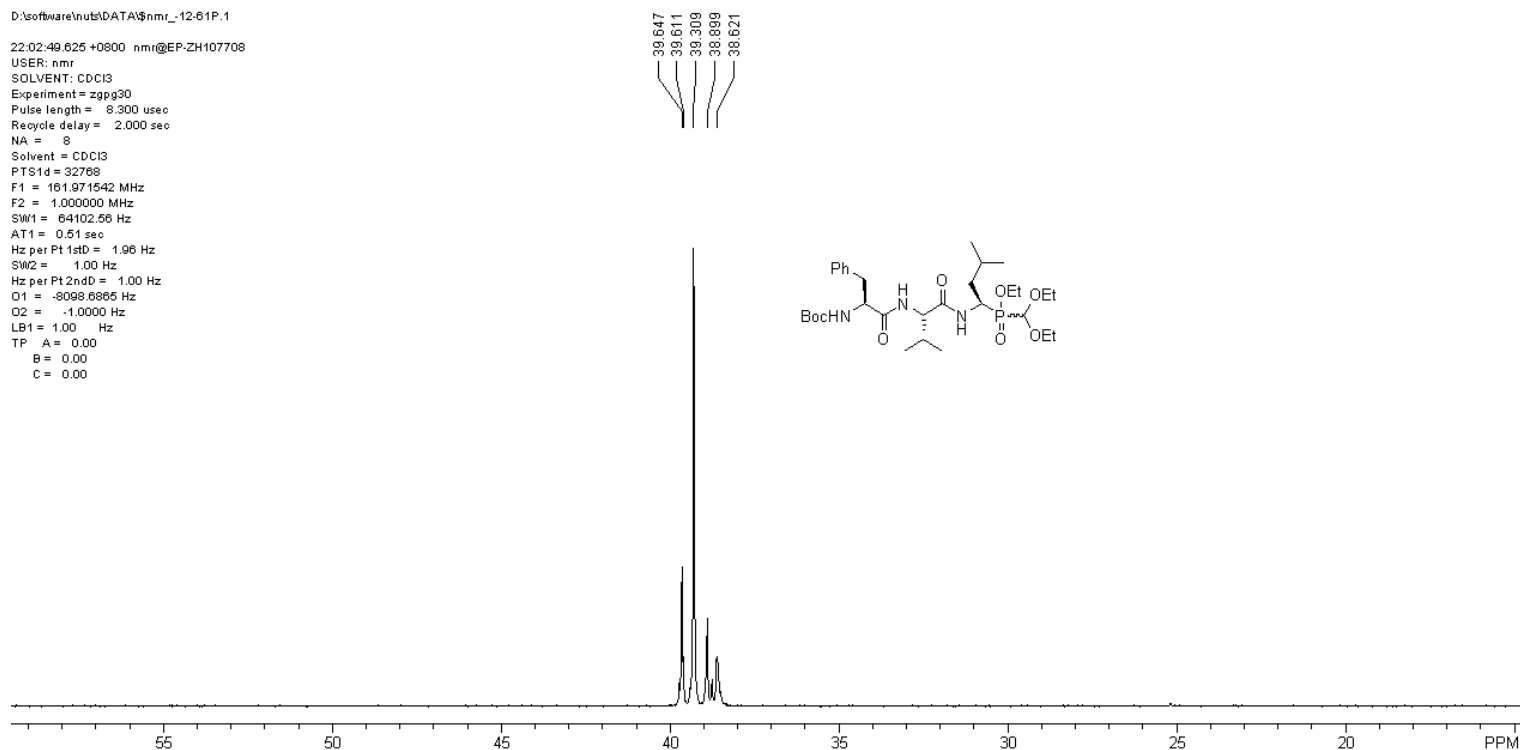
O2 = -1.0000 Hz

LB1 = 1.00 Hz

TP A = 0.00

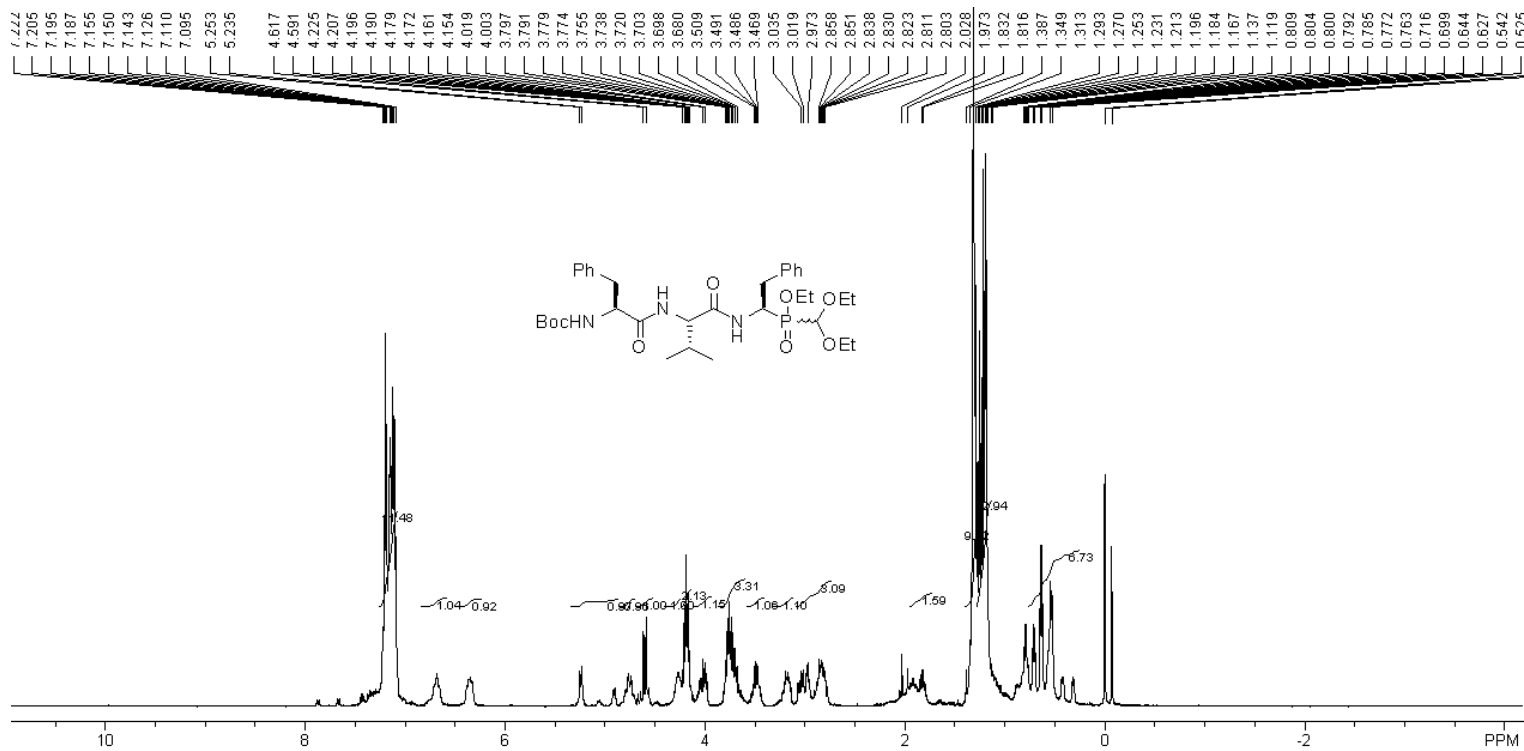
B = 0.00

C = 0.00

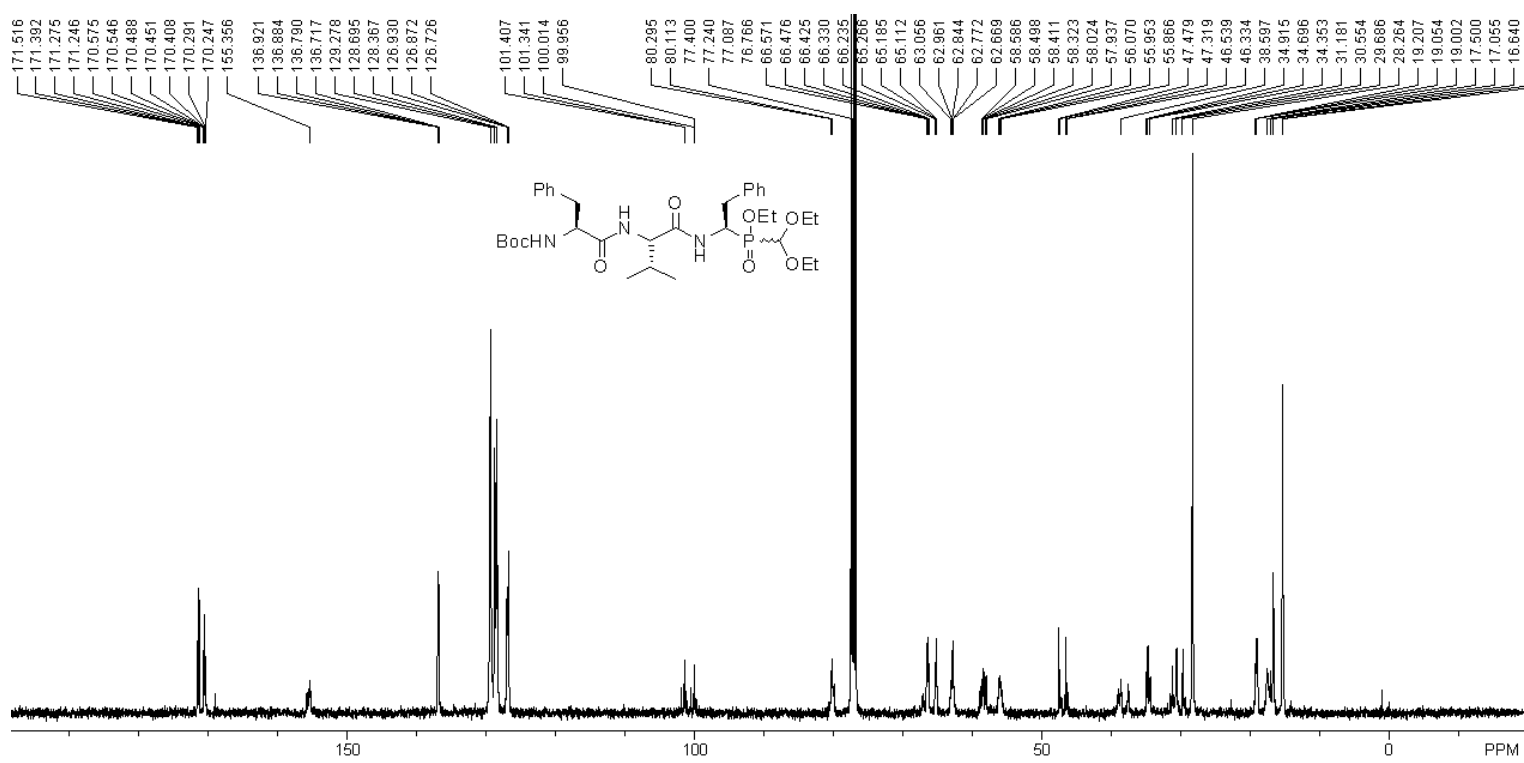


Compound 10h:

¹H NMR:



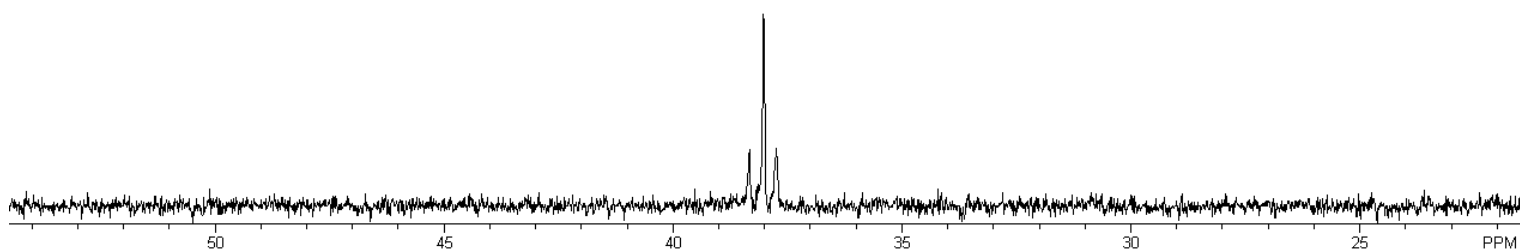
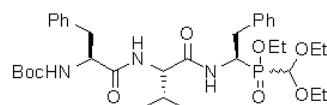
¹³C NMR:



³¹P NMR:

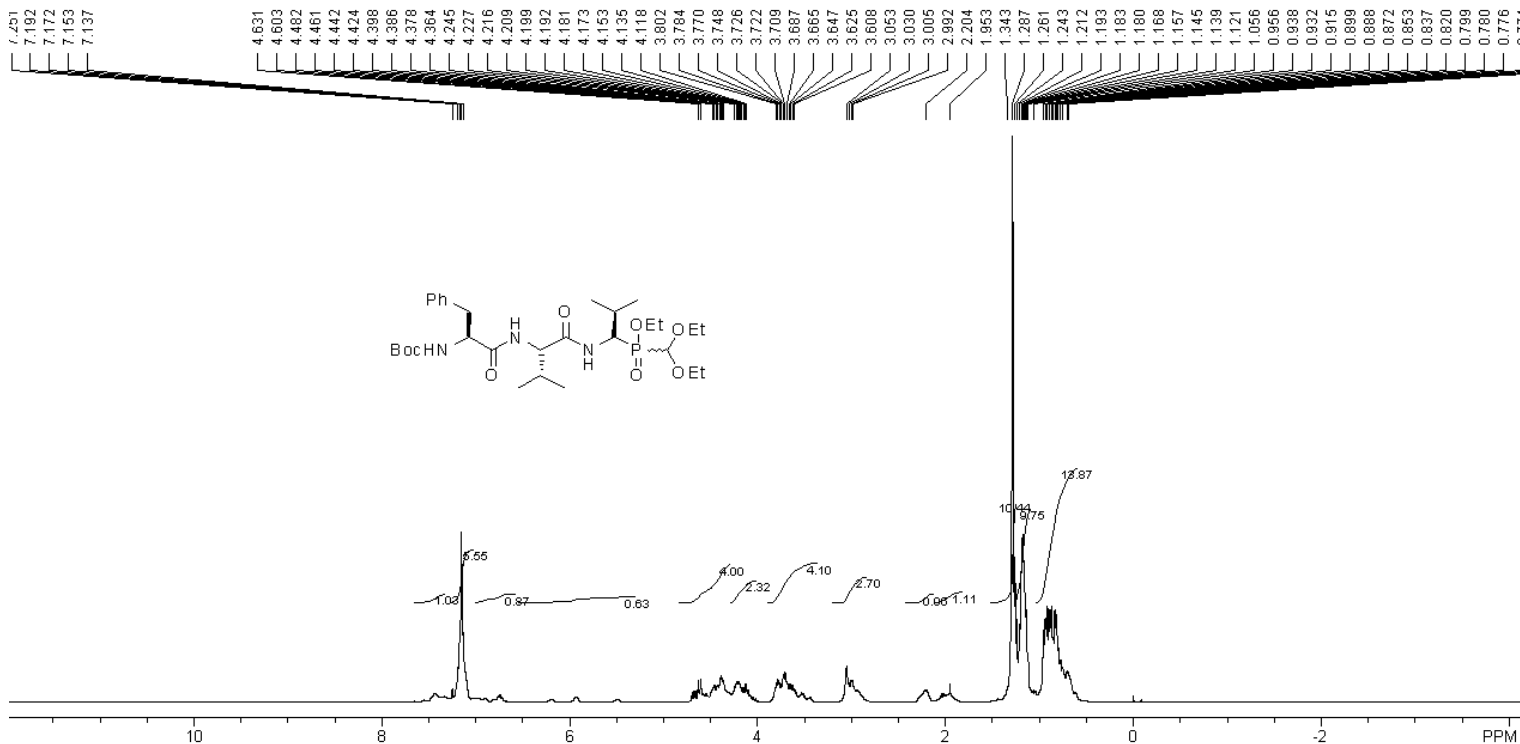
D:\software\nuts\DATA\nmr_12-66P.1
 07:37:29.343 +0800 nmri@EP-ZH107708
 USER: nmri
 SOLVENT: CDCl3
 Experiment = zgpg30
 Pulse length = 8.300 usec
 Recycle delay = 2.000 sec
 NA = 8
 Solvent = CDCl3
 PTS1d = 32768
 F1 = 161.971542 MHz
 F2 = 1.000000 MHz
 SW1 = 64102.56 Hz
 AT1 = 0.51 sec
 Hz per Pt1stD = 1.96 Hz
 SW2 = 1.00 Hz
 Hz per Pt2ndD = 1.00 Hz
 O1 = -8098.6865 Hz
 O2 = -1.0000 Hz
 LB1 = 1.00 Hz
 TP A = 0.00
 B = 0.00
 C = 0.00

38.331
 38.029
 37.739

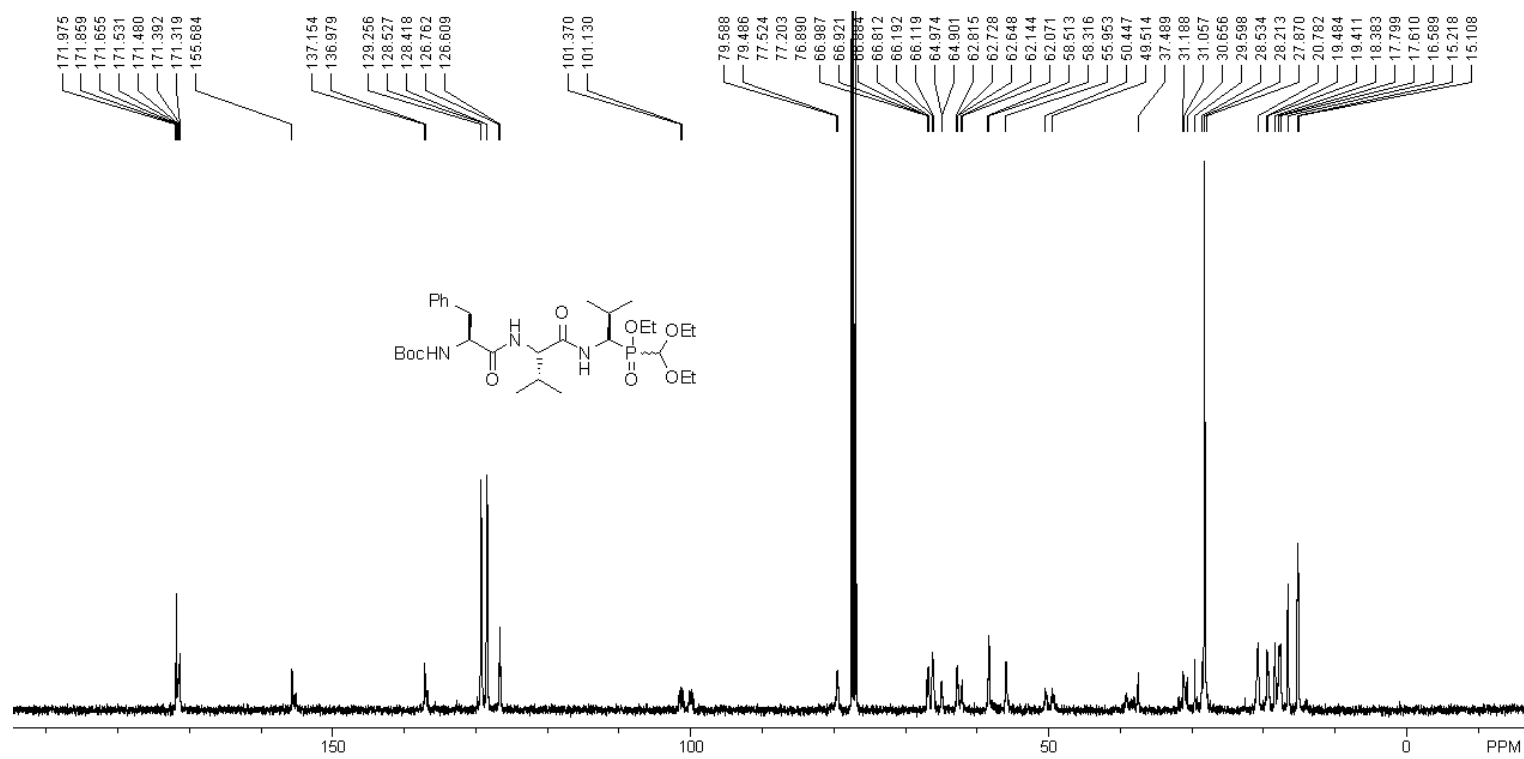


Compound 10i:

¹H NMR:



¹³C NMR:



³¹P NMR:

D:\software\nmr\DATA\nmr_12-60P.1

22:14:21.703 +0800 nmri@EP.ZH107708

USER: nmri

SOLVENT: CDCl₃

Experiment = zgpg30

Pulse length = 8.300 usec

Recycle delay = 2.000 sec

NA = 8

Solvent = CDCl₃

PTSD = 32768

F1 = 161.971542 MHz

F2 = 1.000000 MHz

SWH = 64102.56 Hz

AT1 = 0.51 sec

Hz per Pt1s0 = 1.96 Hz

SW2 = 1.00 Hz

Hz per Pt2nd0 = 1.00 Hz

Q1 = -8098.6965 Hz

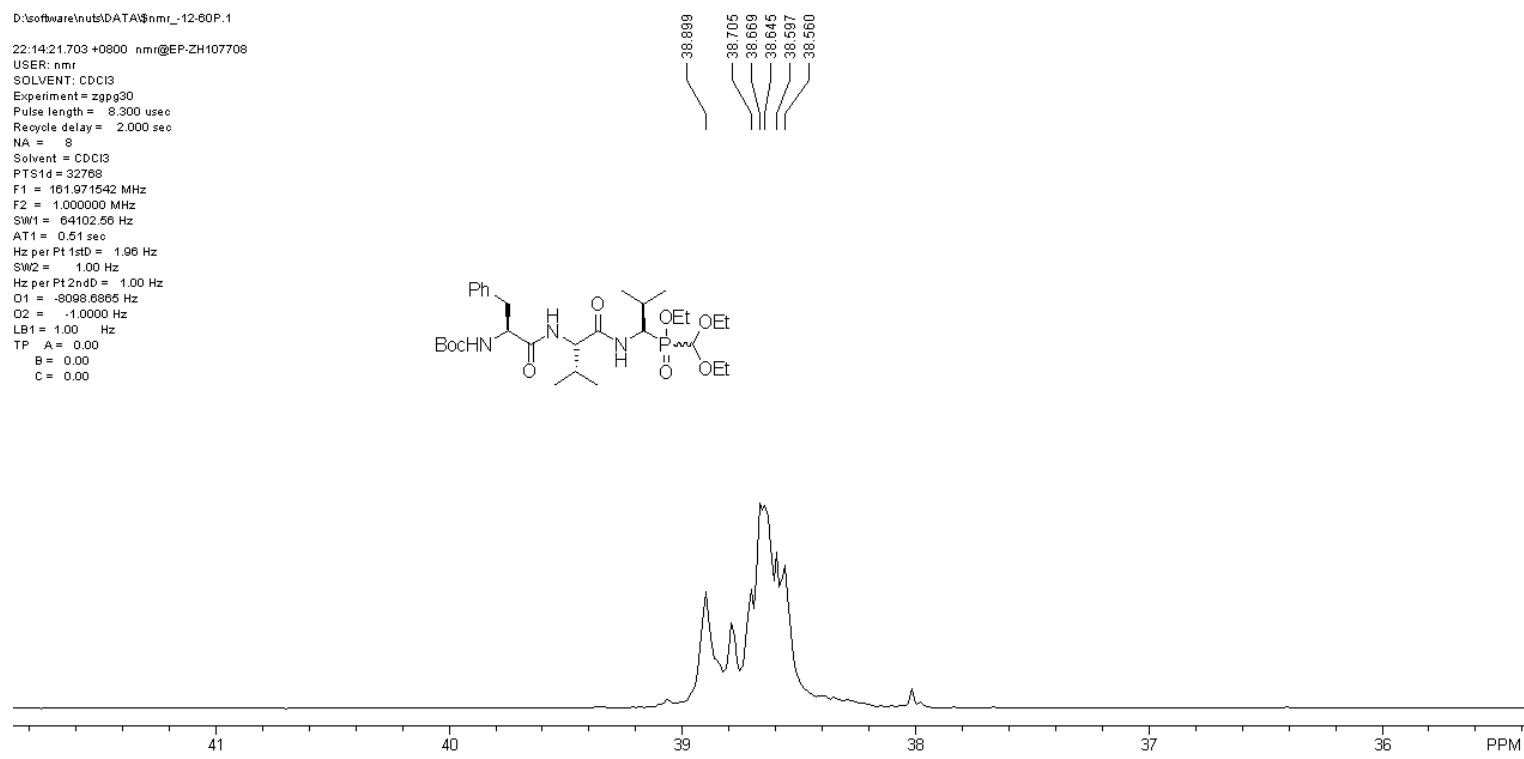
Q2 = -1.0000 Hz

LB1 = 1.00 Hz

TP A = 0.00

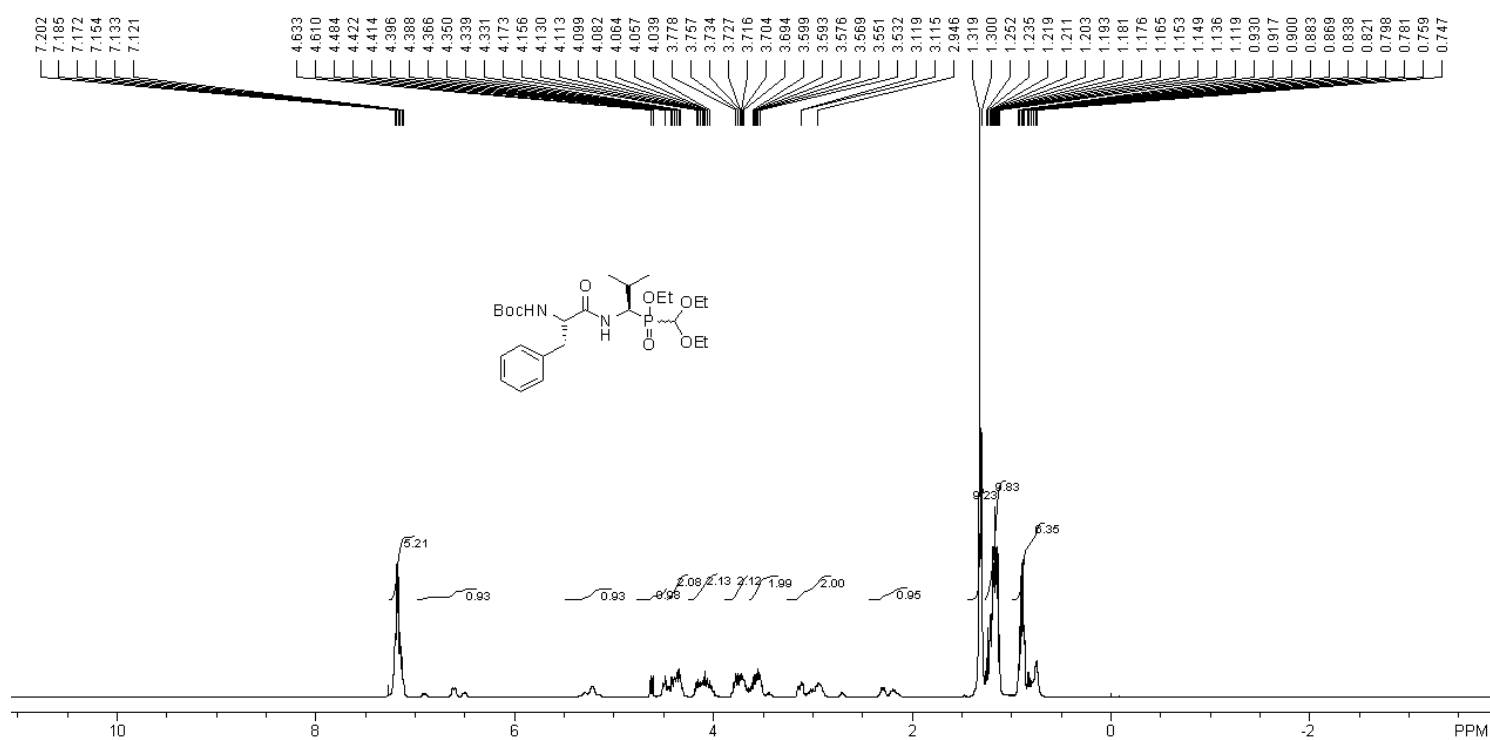
B = 0.00

C = 0.00

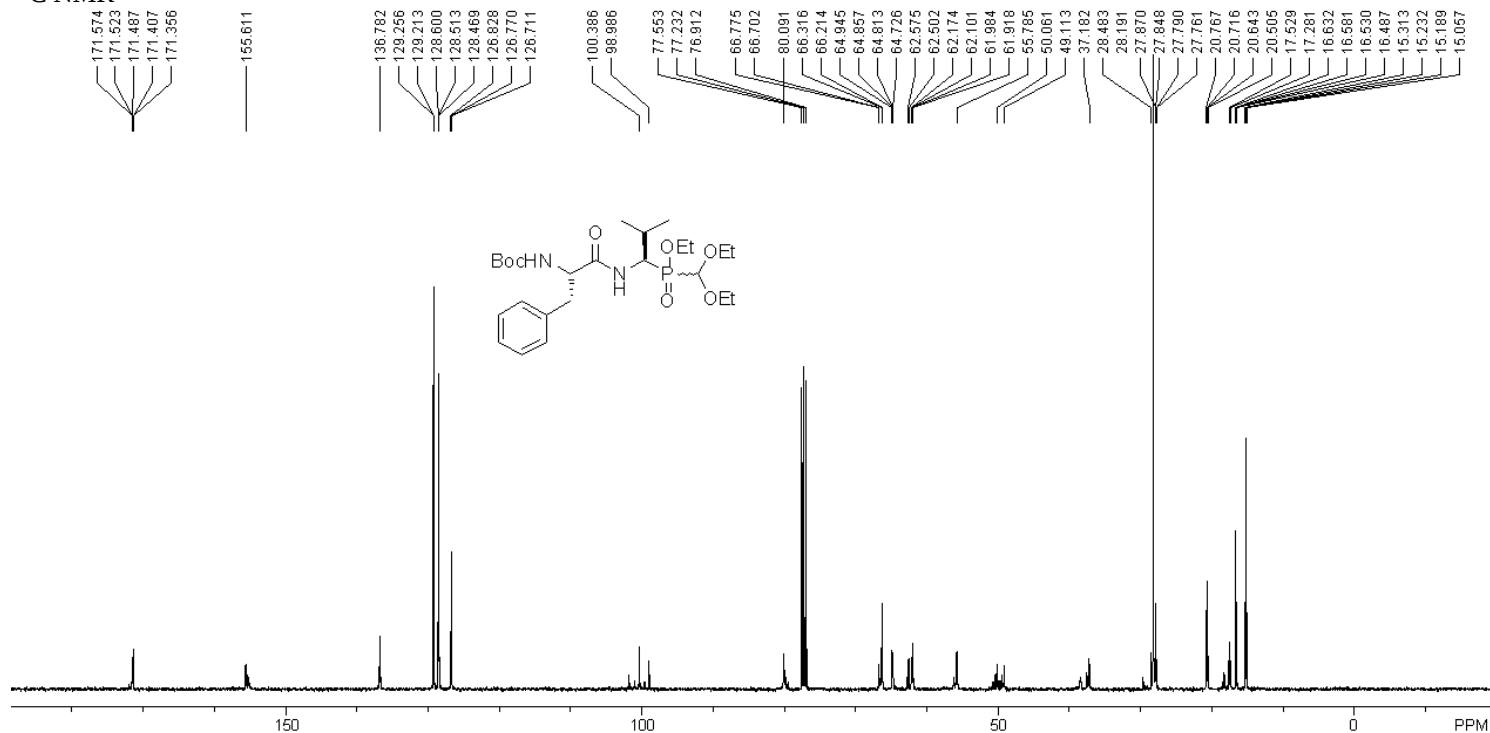


Compound 10j:

¹H NMR



¹³C NMR



³¹P NMR

D:\software\inputs\DATA\nmr_13-39P.1

18:36:12.062 +0800 nmr@EP-ZH107708

USER: nmr

SOLVENT: CDCl3

Experiment = zgpg30

Pulse length = 8.300 usec

Recycle delay = 2.000 sec

NA = 16

Solvent = CDCl3

PTS1d = 32768

F1 = 161.971542 MHz

F2 = 1.000000 MHz

SW1 = 64102.56 Hz

AT1 = 0.51 sec

Hz per Pt1sd = 1.96 Hz

SW2 = 1.00 Hz

Hz per Pt2nd = 1.00 Hz

O1 = -8098.6865 Hz

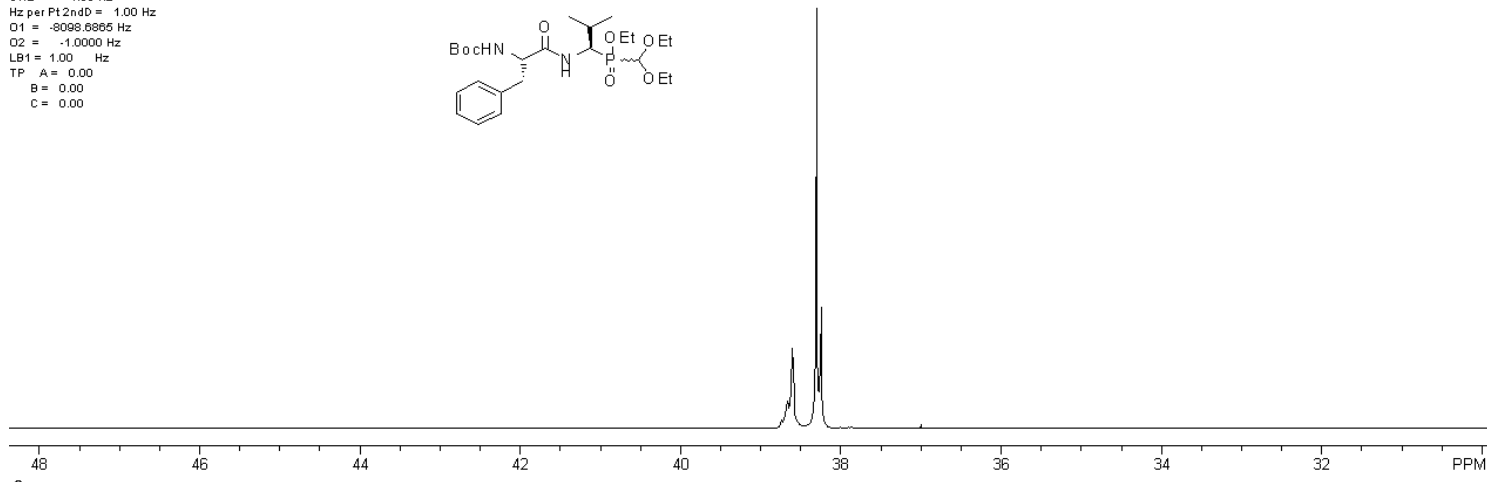
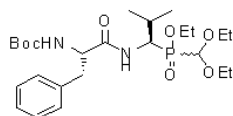
O2 = -1.0000 Hz

LB1 = 1.00 Hz

TP A = 0.00

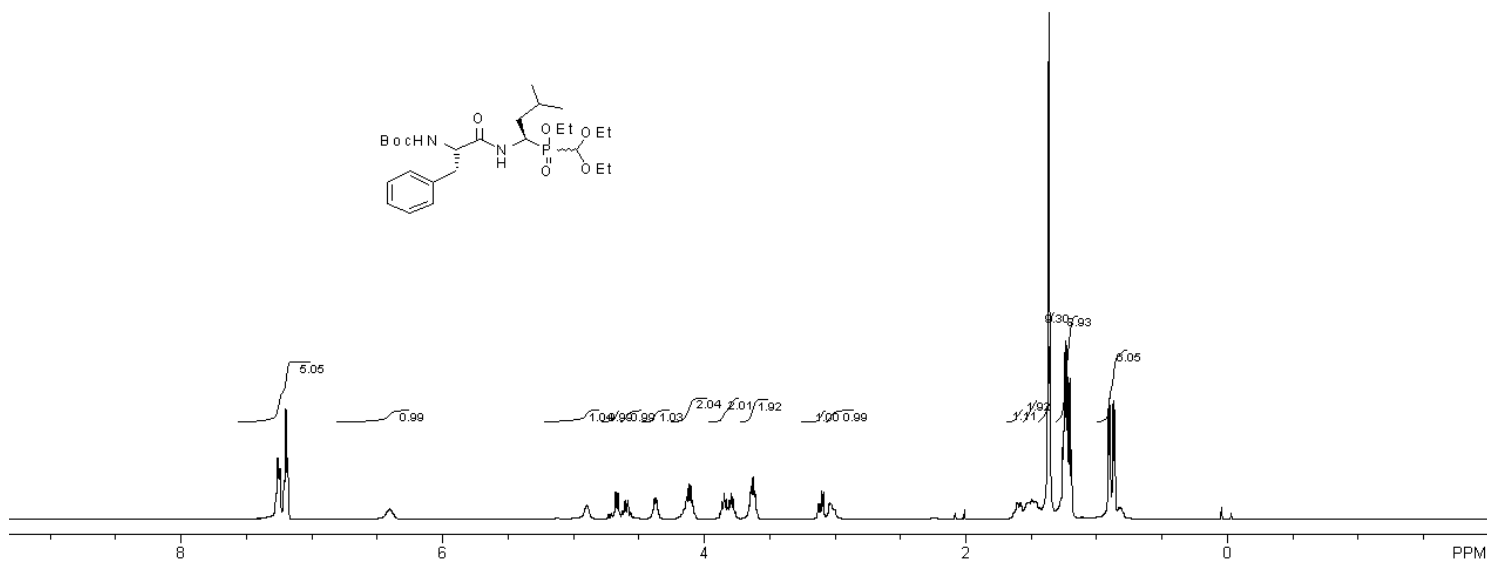
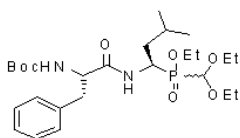
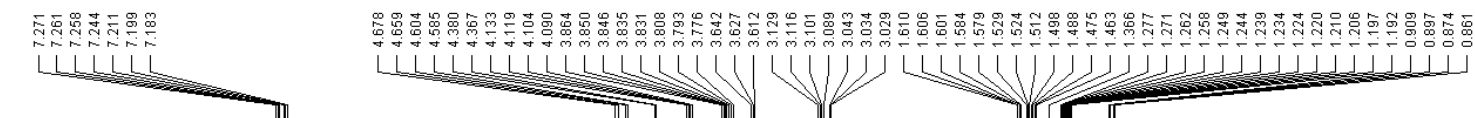
B = 0.00

C = 0.00

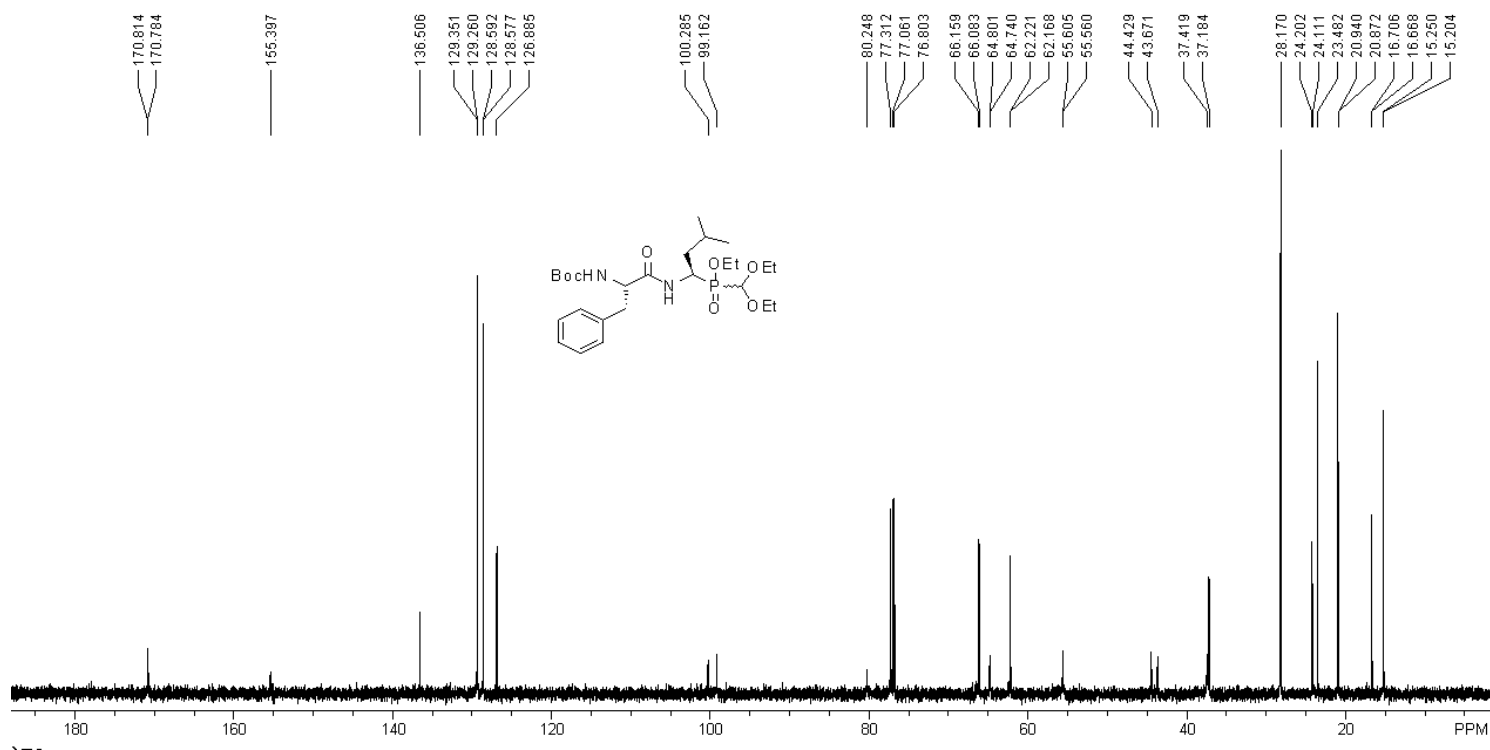


Compound 10k:

¹H NMR:



¹³C NMR:



³¹P NMR:

D:\software\nmr\DATA\nmr_13-40P.1

18:41:58.281 +0800 nmr@EP-ZH107708

USER: nmr

SOLVENT: CDCl3

Experiment = zgpg30

Pulse length = 8.300 usec

Recycle delay = 2.000 sec

NA = 16

Solvent = CDCl3

PTS1d = 32768

F1 = 161.971542 MHz

F2 = 1.000000 MHz

SW1 = 64102.56 Hz

AT1 = 0.51 sec

Hz per Pt 1stD = 1.96 Hz

SW2 = 1.00 Hz

Hz per Pt 2ndD = 1.00 Hz

O1 = -8098.6865 Hz

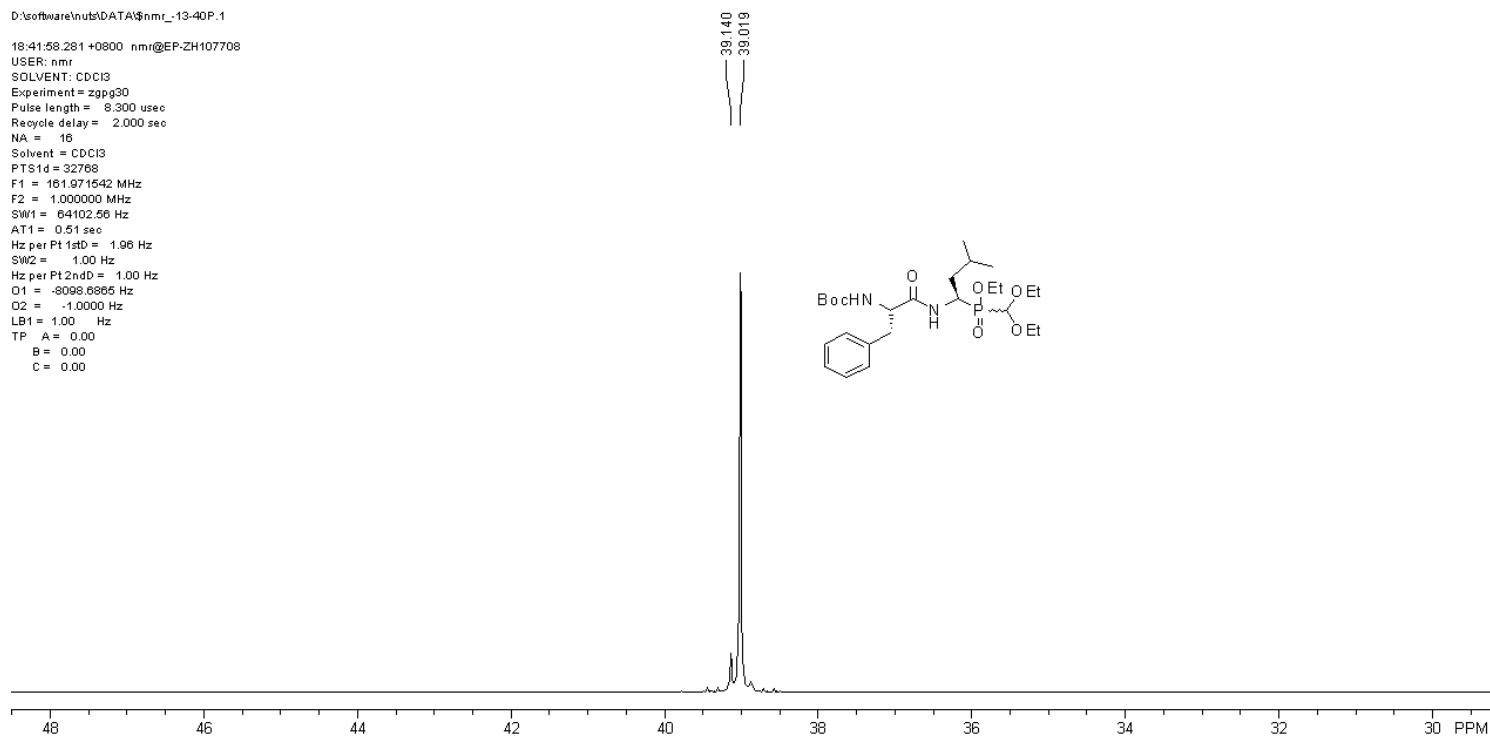
O2 = -1.0000 Hz

LB1 = 1.00 Hz

TP A = 0.00

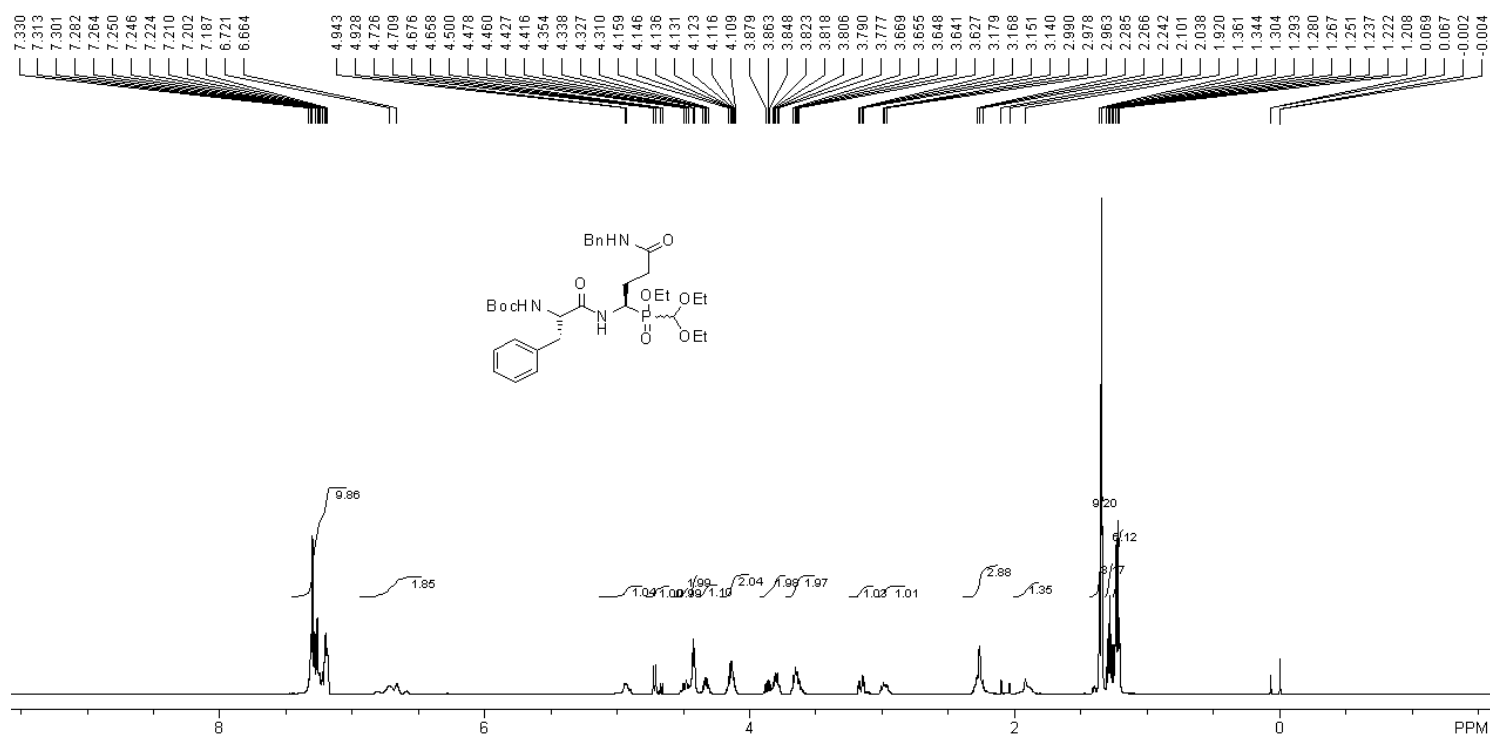
B = 0.00

C = 0.00

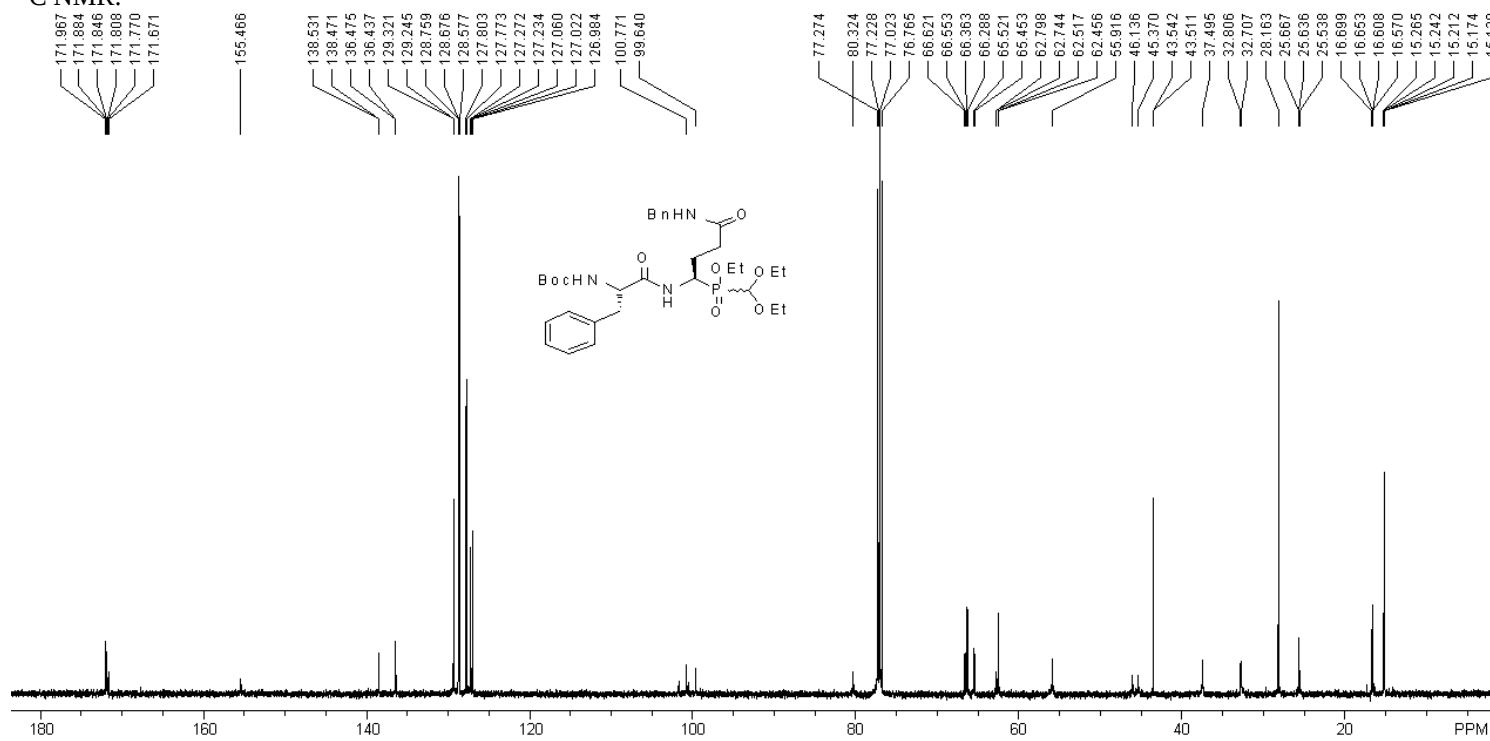


Compound 101:

¹H NMR:



¹³C NMR:

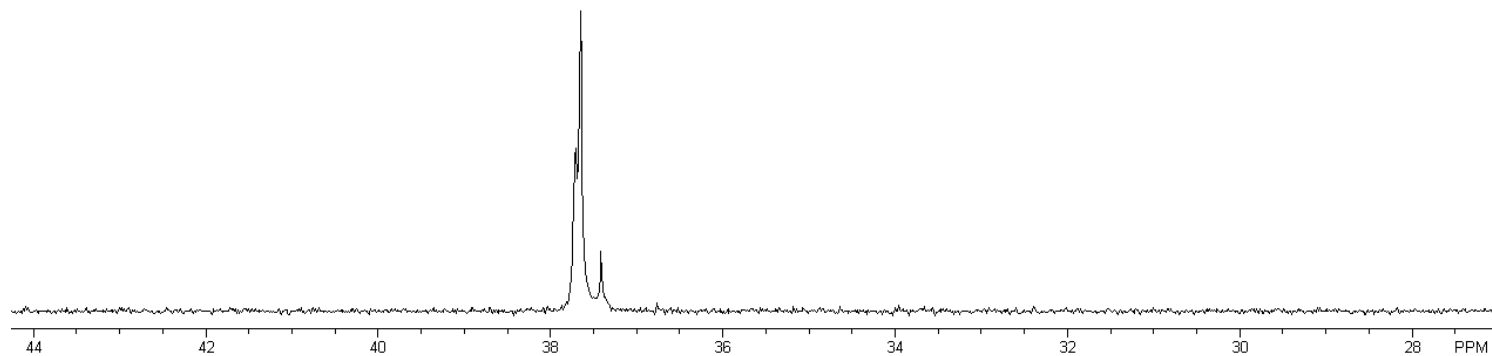
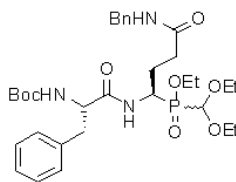
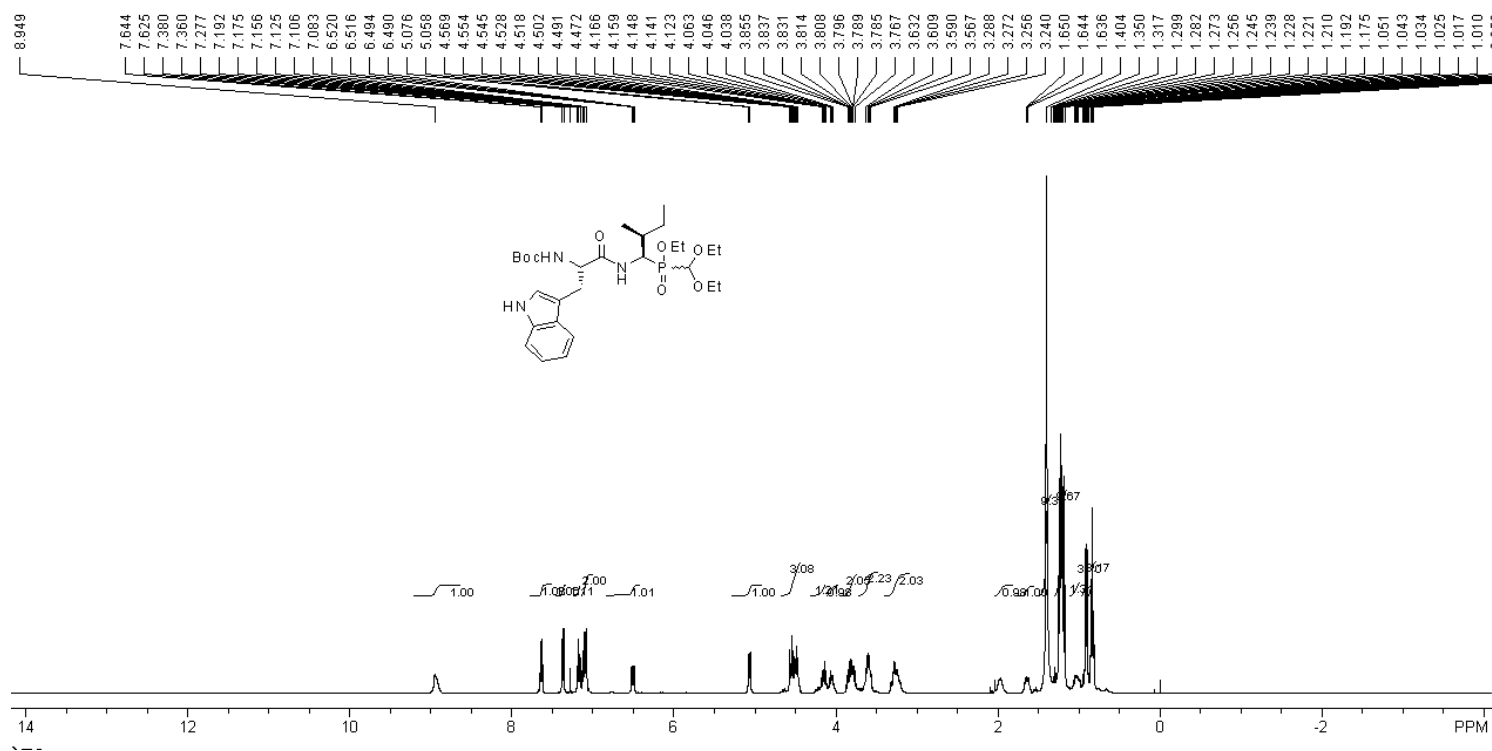


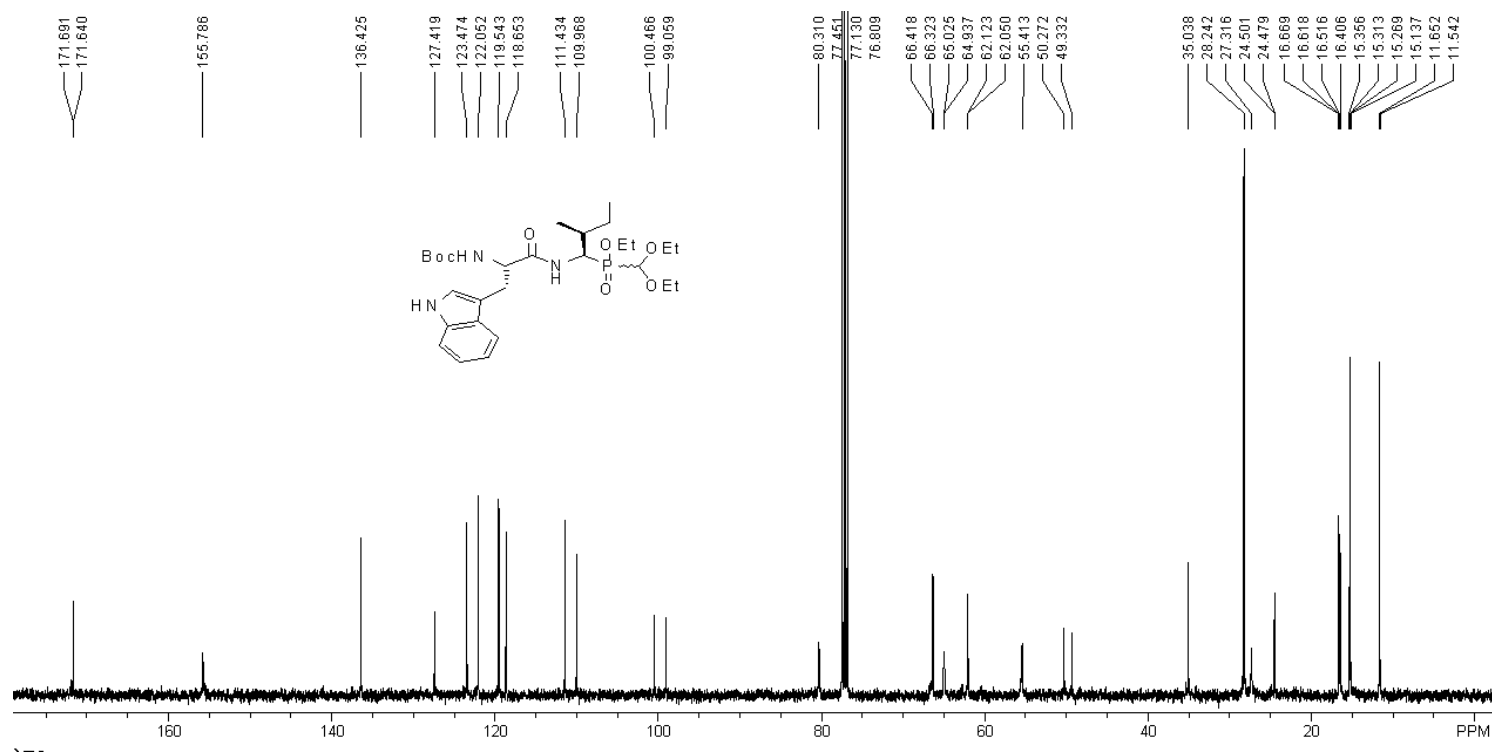
³¹P NMR:

```

18:30:47.140 +0800 nmri@EP-ZH107708
USER: nmri
SOLVENT: CDCI3
Experiment = zgpg30
Pulse length = 8.300 usec
Recycle delay = 2.000 sec
NA = 16
Solvent = CDCI3
PT1SD = 32768
F2 = 181971542 MHz
F1 = 1.00000000 MHz
SWH1 = 64102.56 Hz
AT1 = 0.51 sec
Hz per Pt1sd = 1.96 Hz
SWH2 = 1.00 Hz
Hz per Pt2nd = 1.00 Hz
Q1 = -8098.6865 Hz
D1 = -1.0000 Hz
LB1 = 1.00 Hz
TP_A = 0.00
B = 0.00
C = 0.00

```

¹H NMR:¹³C NMR:



³¹P NMR:

D:\software\nmr\DATA\nmr_13-44P.1

21:17:46.609 +0800 nmr@EP-ZH107708

USER: nmr

SOLVENT: CDCl3

Experiment = zgpg30

Pulse length = 8.300 usec

Recycle delay = 2.000 sec

NA = 8

Solvent = CDCl3

PTS1d = 32768

F1 = 161.971542 MHz

F2 = 1.000000 MHz

SW1 = 64102.56 Hz

AT1 = 0.51 sec

Hz per Pt 1stD = 1.96 Hz

SW2 = 1.00 Hz

Hz per Pt 2ndD = 1.00 Hz

O1 = -8098.6865 Hz

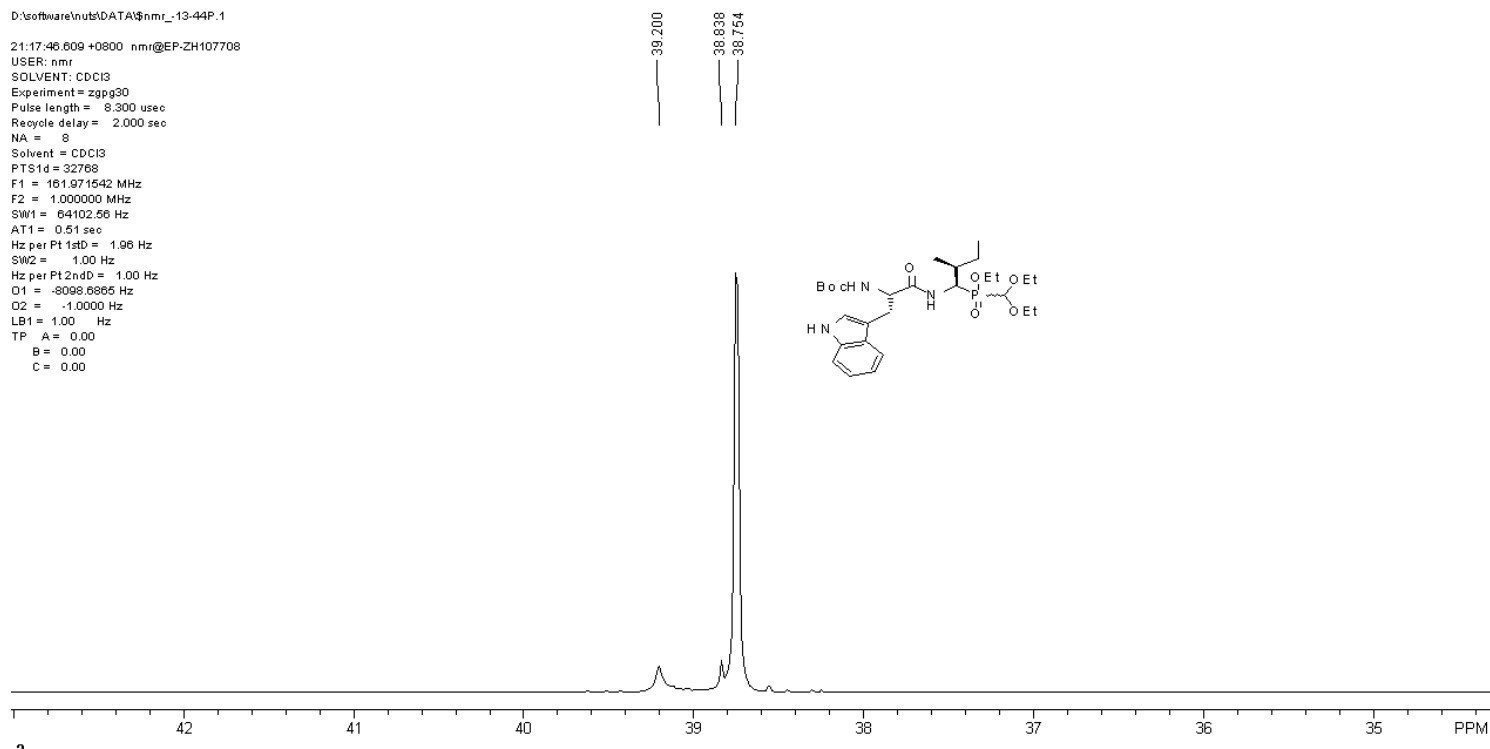
O2 = -1.0000 Hz

LB1 = 1.00 Hz

TP A = 0.00

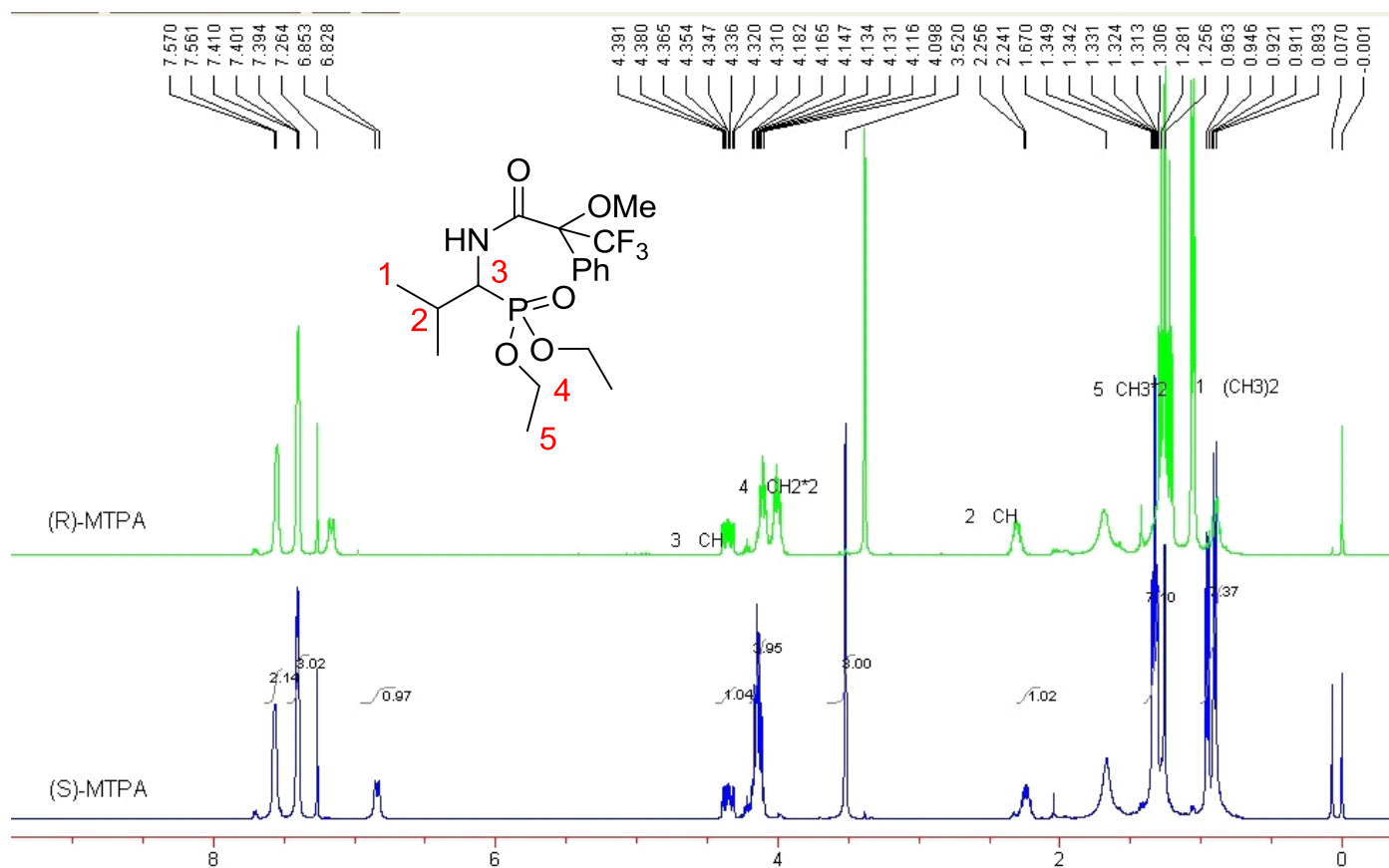
B = 0.00

C = 0.00



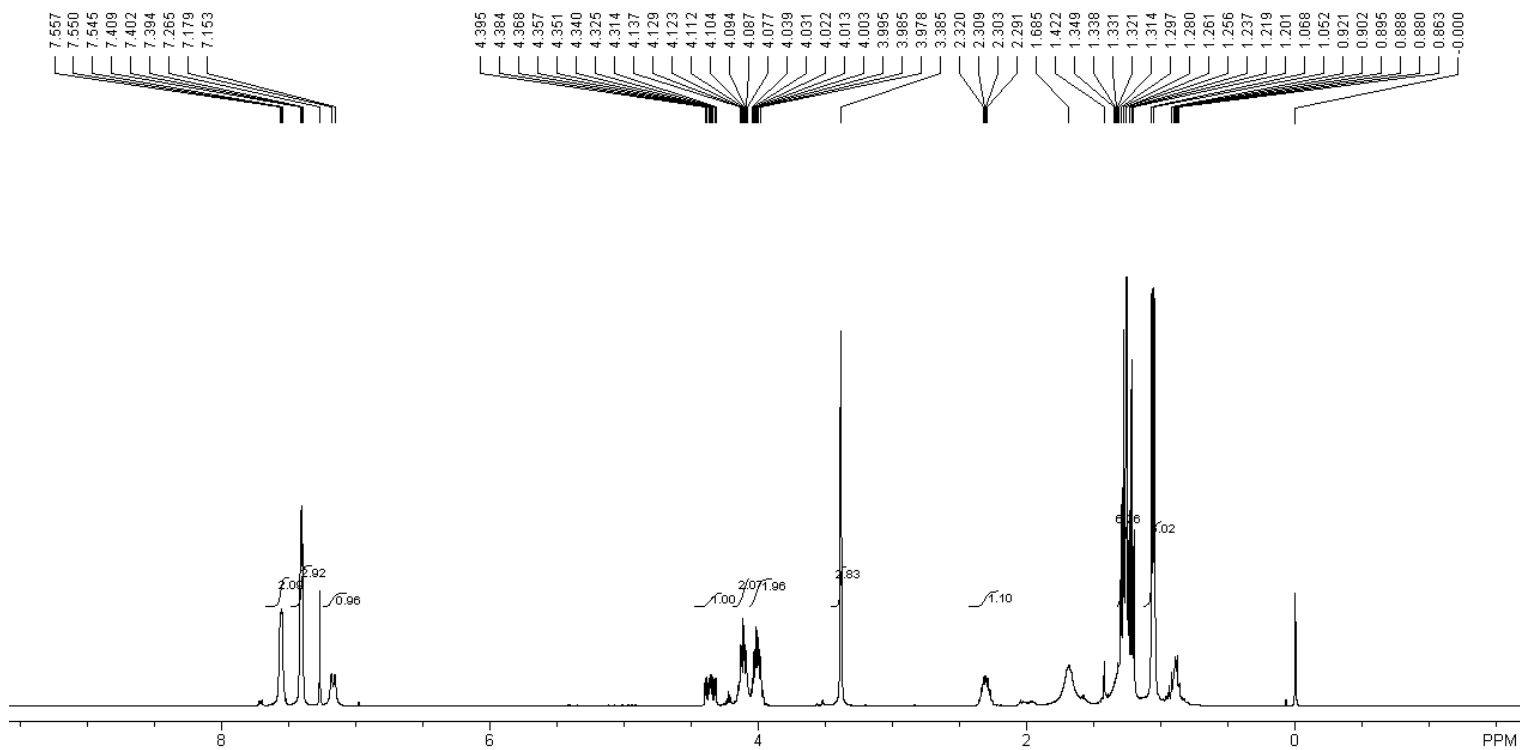
2. NMR Spectra for Mosher's Derivatives of 5p and the Major Isomer of 3m.

^1H NMR of (S) and (R)-Mosher's Derivative **6p**:



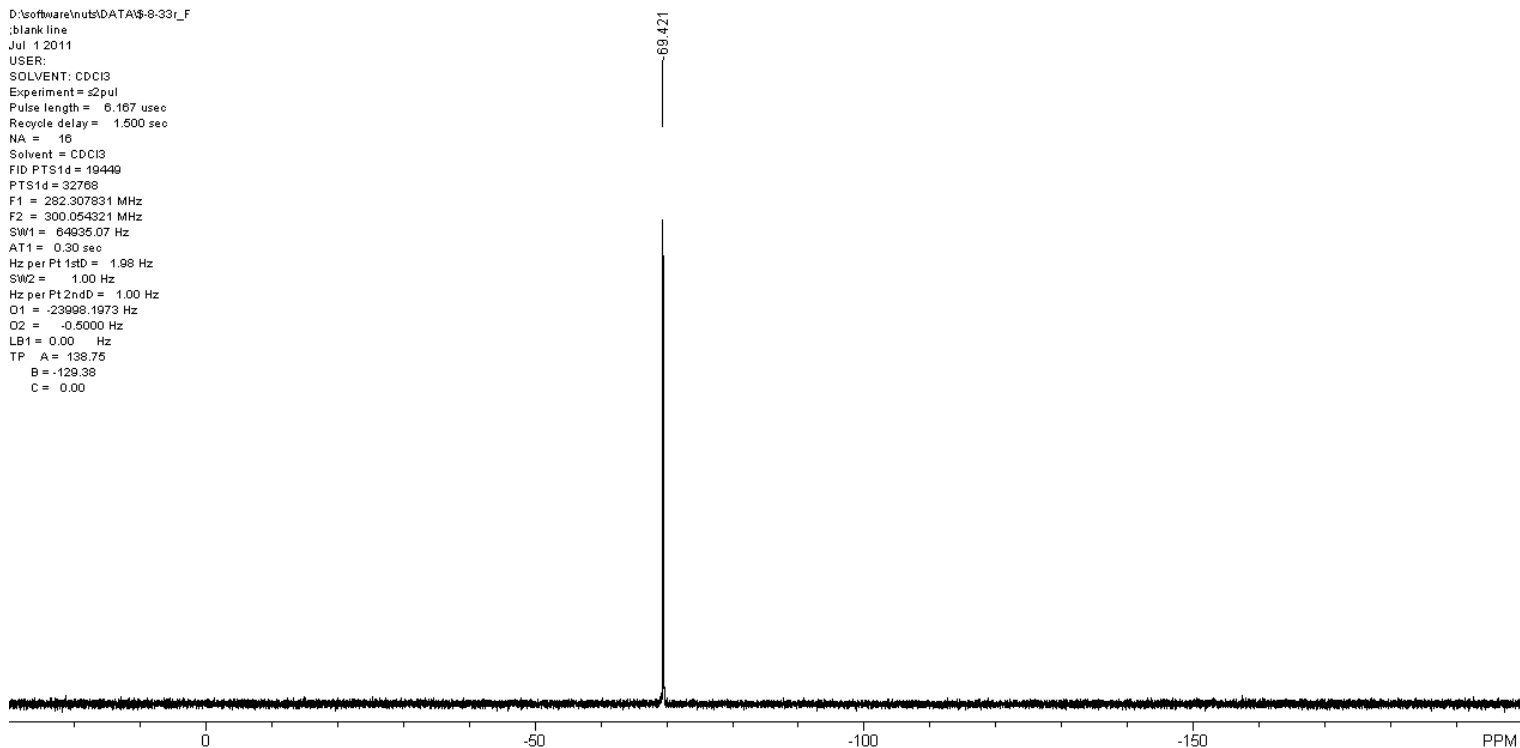
(R)-Mosher's derivative of **5p** (**6p**):

^1H NMR:



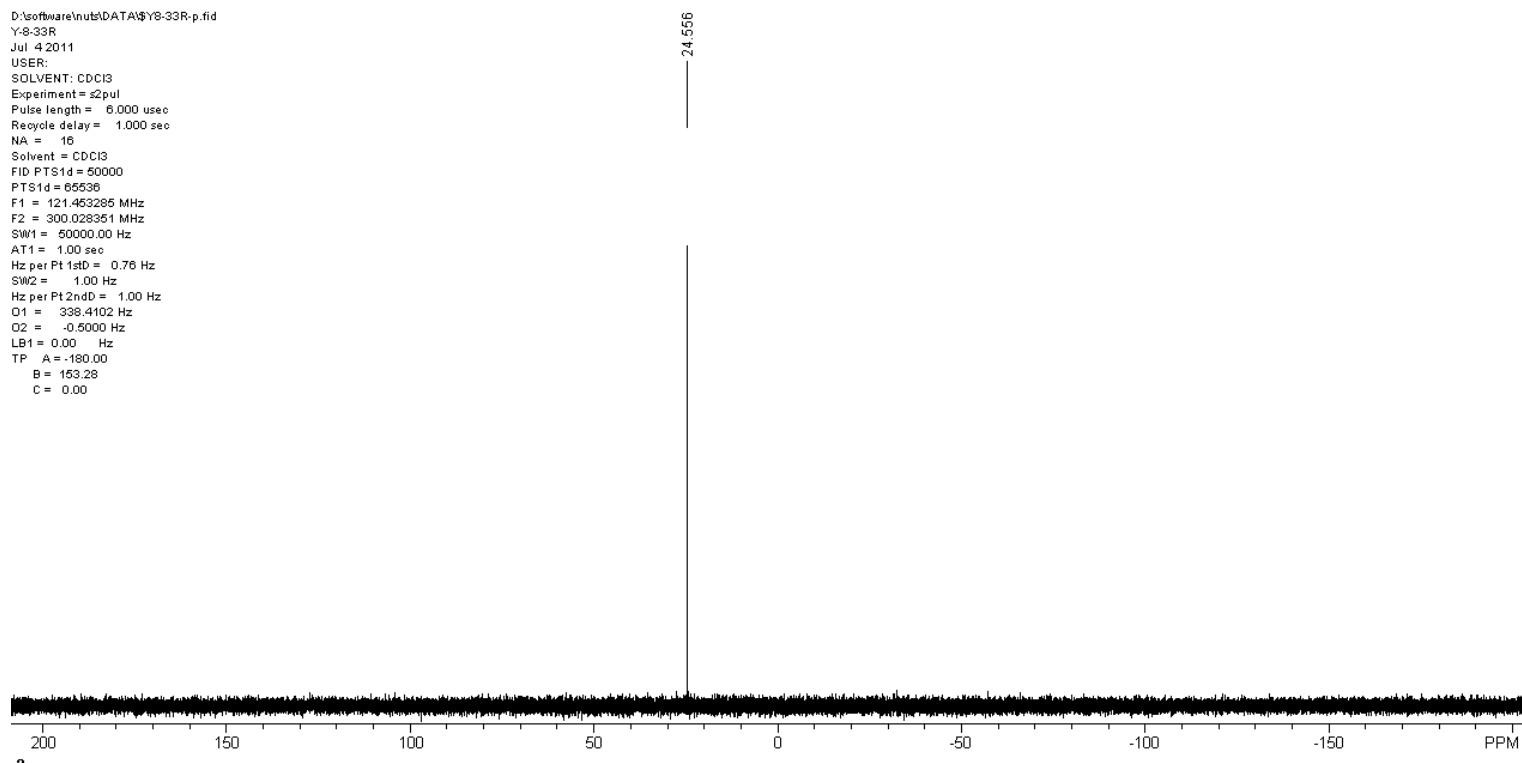
¹⁹F NMR:

D:\software\nuts\DATA\8-33r_F
:blank line
Jul 1 2011
USER:
SOLVENT: CDCl3
Experiment = s2pul
Pulse length = 6.167 usec
Recycle delay = 1.500 sec
NA = 16
Solvent = CDCl3
FID PTS1d = 19449
PTS1d = 32768
F1 = 282.307831 MHz
F2 = 300.054321 MHz
SW1 = 64835.07 Hz
AT1 = 0.30 sec
Hz per Pt1sD = 1.98 Hz
SW2 = 1.00 Hz
Hz per Pt2ndD = 1.00 Hz
Q1 = -23998.1973 Hz
Q2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = 138.75
B = -129.38
C = 0.00



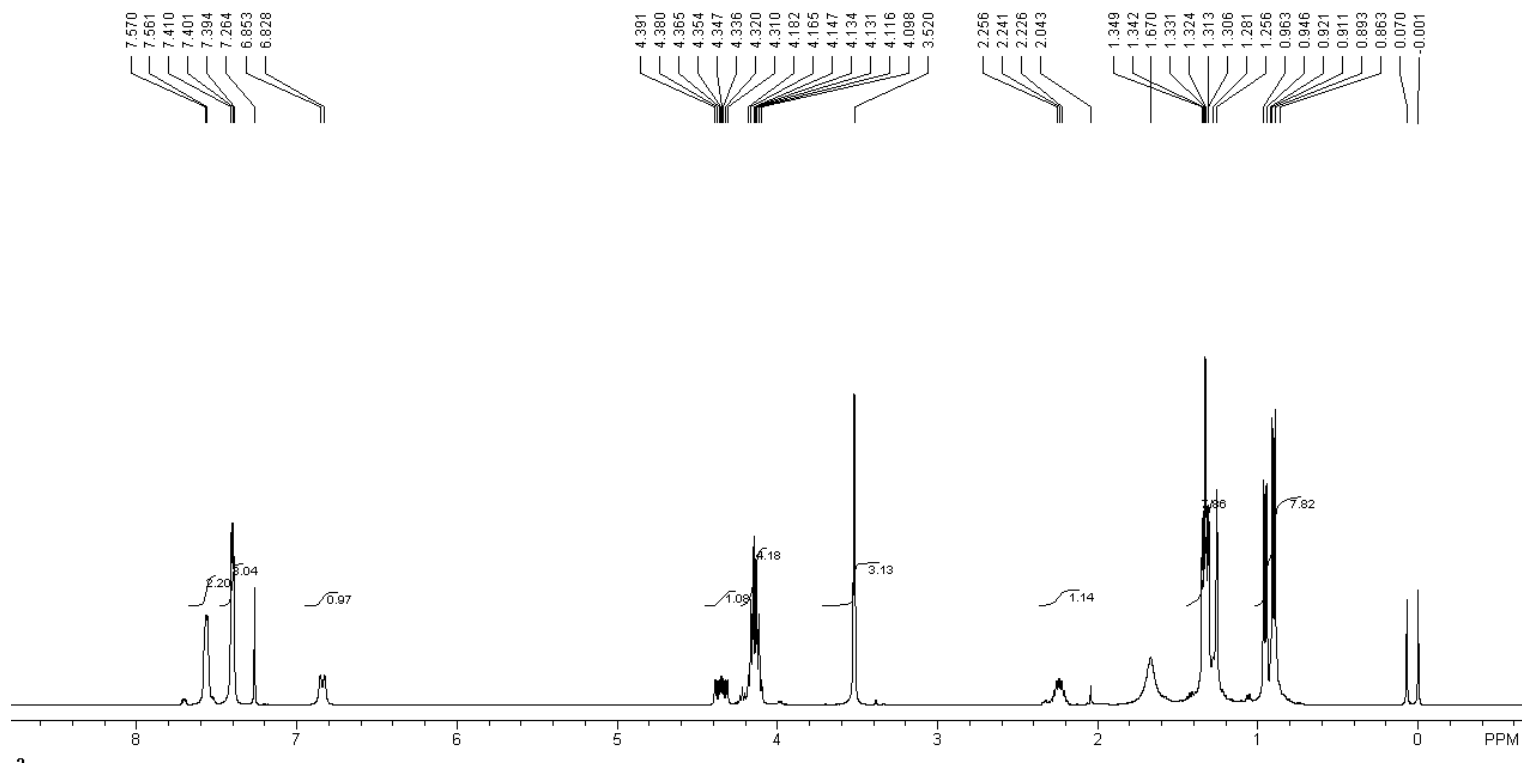
³¹P NMR:

D:\software\nuts\DATA\Y8-33R.p.fid
 Y-8-33R
 Jul 4 2011
 USER:
 SOLVENT: CDCl3
 Experiment = s2pul
 Pulse length = 6.000 usec
 Recycle delay = 1.000 sec
 NA = 16
 Solvent = CDCl3
 FID PTS1d = 60000
 PTS1d = 65536
 F1 = 121.463285 MHz
 F2 = 300.028351 MHz
 SW1 = 50000.00 Hz
 AT1 = 1.00 sec
 Hz per Pt1st0 = 0.76 Hz
 SW2 = 1.00 Hz
 Hz per Pt2nd0 = 1.00 Hz
 O1 = 338.4102 Hz
 O2 = -0.6000 Hz
 LB1 = 0.00 Hz
 TP A = -180.00
 B = 153.28
 C = 0.00



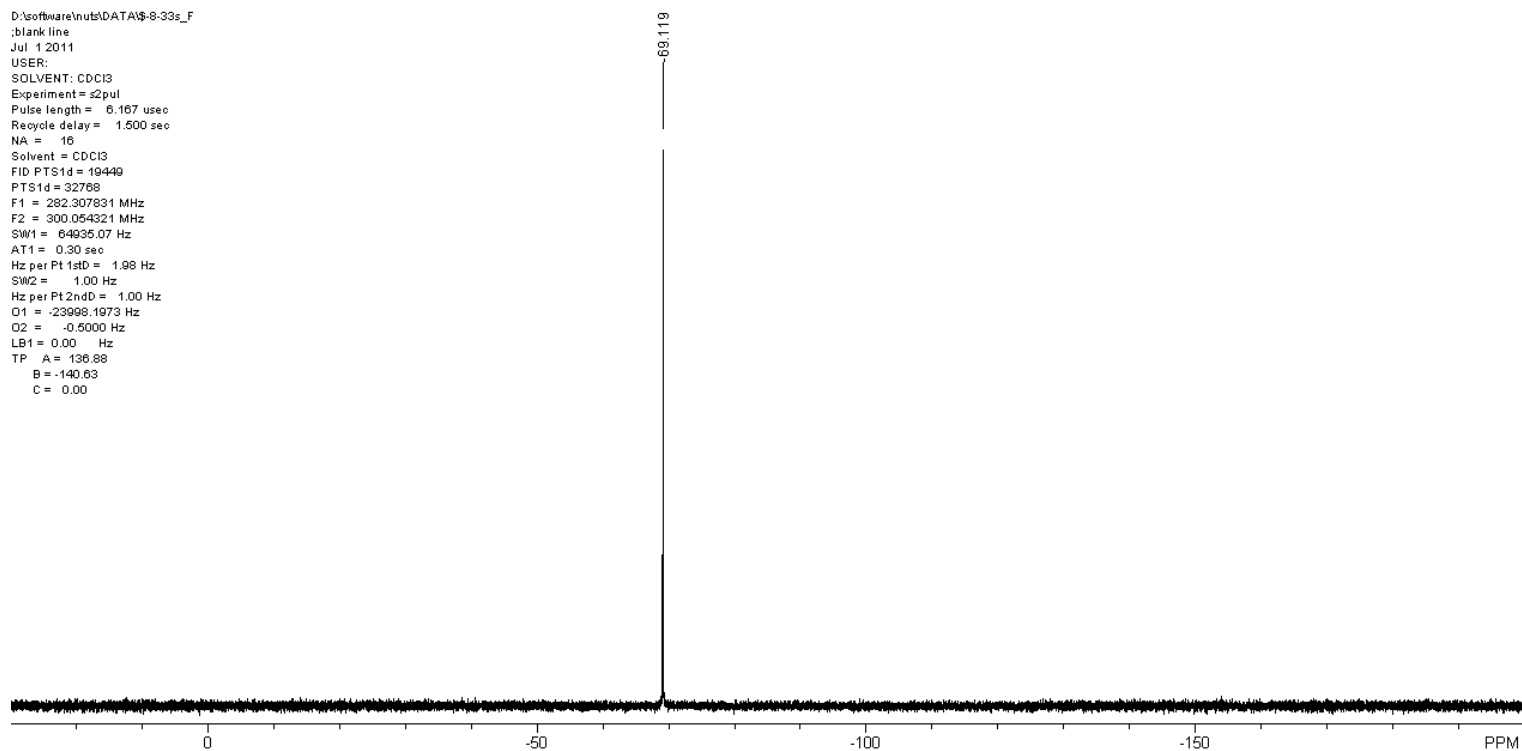
(S)-Mosher's derivative of **5p** ((-)-**6p**):

¹H NMR:



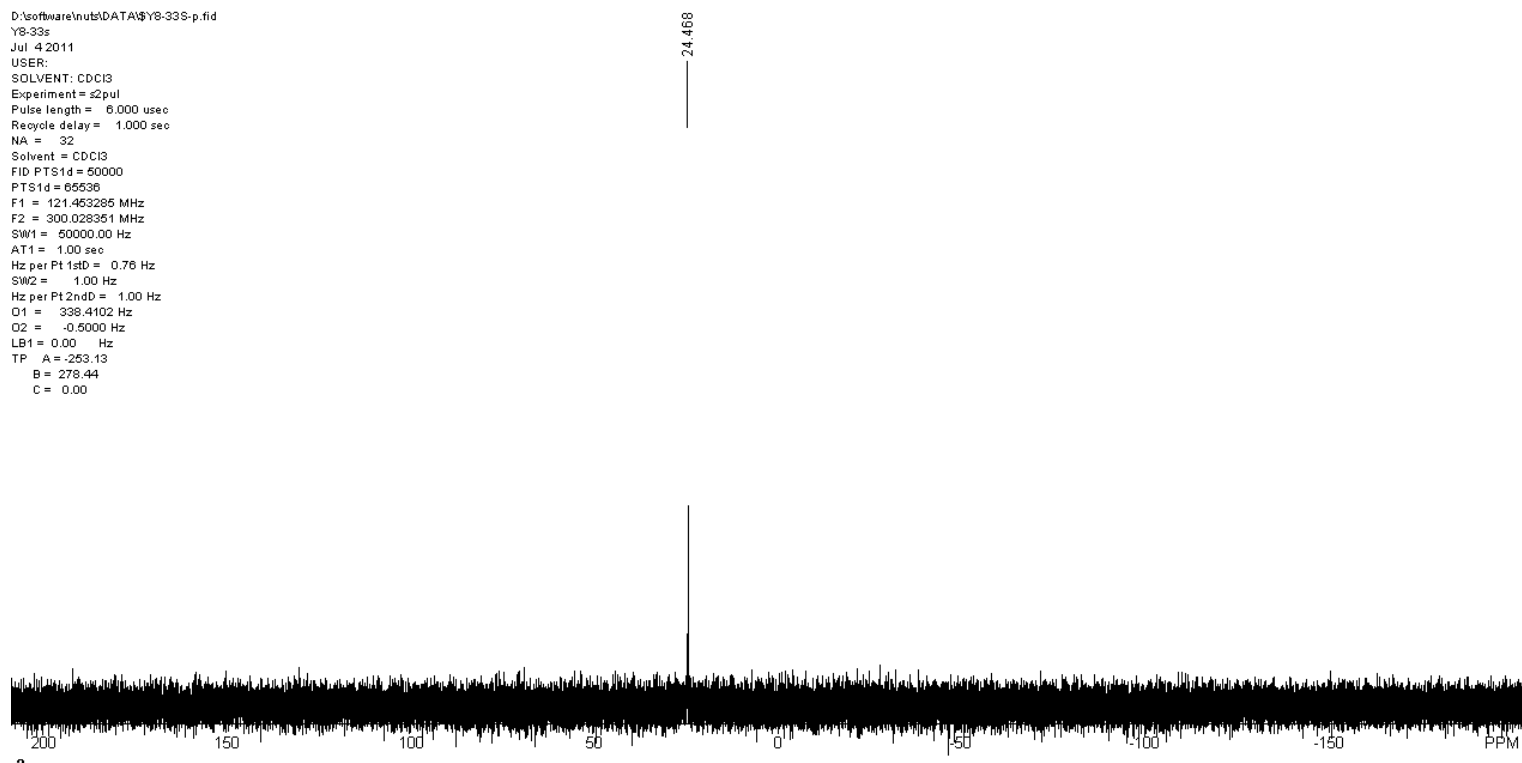
¹⁹F NMR:

D:\software\nuts\DATA\8-33s_F
 :blank line
 Jul 1 2011
 USER:
 SOLVENT: CDCl₃
 Experiment = s2pul
 Pulse length = 6.167 usec
 Recycle delay = 1.500 sec
 NA = 16
 Solvent = CDCl₃
 FID PTS1d = 19446
 PTS1d = 32768
 F1 = 282.307831 MHz
 F2 = 300.054321 MHz
 SW1 = 64635.07 Hz
 AT1 = 0.30 sec
 Hz per Pt1stD = 1.98 Hz
 SW2 = 1.00 Hz
 Hz per Pt2ndD = 1.00 Hz
 O1 = -23998.1973 Hz
 O2 = -0.5000 Hz
 LB1 = 0.00 Hz
 TP A = 136.88
 B = -140.63
 C = 0.00



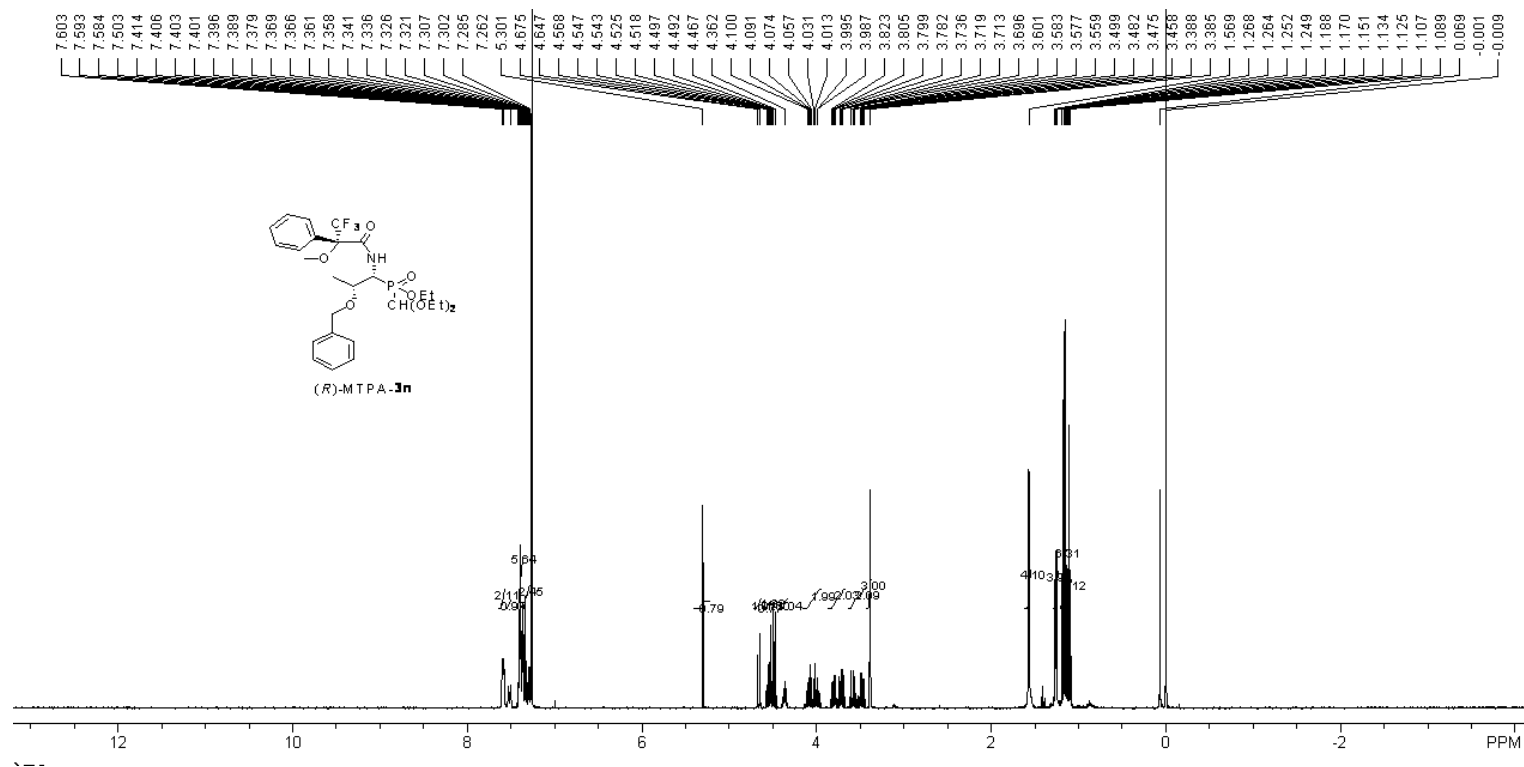
³¹P NMR:

D:\software\nuts\DATA\Y8-33S-p.fid
 Y8-33s
 Jul 4 2011
 USER:
 SOLVENT: CDCl₃
 Experiment = s2pul
 Pulse length = 6.000 usec
 Recycle delay = 1.000 sec
 NA = 32
 Solvent = CDCl₃
 FID PTS1d = 50000
 PTS1d = 65536
 F1 = 121.463285 MHz
 F2 = 300.028351 MHz
 SW1 = 50000.00 Hz
 AT1 = 1.00 sec
 Hz per Pt1stD = 0.76 Hz
 SW2 = 1.00 Hz
 Hz per Pt2ndD = 1.00 Hz
 O1 = 338.4102 Hz
 O2 = -0.5000 Hz
 LB1 = 0.00 Hz
 TP A = -253.13
 B = 278.44
 C = 0.00



(R)-Mosher's derivative of the major isomer of **3m**:

¹H NMR:

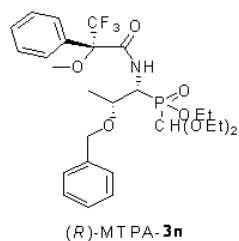


¹⁹F NMR:

D:\software\nmr\DATA\20081109-9-99-2R-H,P,F_12FNMR.1

22:25:59.578 +0800 nmr@EP.ZH107708
 USER: nmr
 SOLVENT: CDCl3
 Experiment = zgfhgqn
 Pulse length = 12.800 usec
 Recycle delay = 1.000 sec
 NA = 16
 Solvent = CDCl3
 P1 = 376.468953 MHz
 F2 = 1.000000 MHz
 SW1 = 89285.71 Hz
 AT1 = 0.73 sec
 Hz per Pt1s0 = 1.36 Hz
 SW2 = 1.00 Hz
 Hz per Pt2nd0 = 1.00 Hz
 Q1 = -37649.0820 Hz
 Q2 = -1.0000 Hz
 LB1 = 0.30 Hz
 TP A = 0.00
 B = 0.00
 C = 0.00

89.131



³¹P NMR:

D:\software\nmr\DATA\2008119-9-99-2R-H,P_F_11PNMR.1

22:24:14.500 +0800 nmri@EP-ZH107708

USER: nmri

SOLVENT: CDCl3

Experiment = zgpg30

Pulse length = 8.300 usec

Recycle delay = 2.000 sec

NA = 16

Solvent = CDCl3

PTS1d = 32768

F1 = 161.971542 MHz

F2 = 1.000000 MHz

SW1 = 64102.56 Hz

AT1 = 0.51 sec

Hz per Pt1stD = 1.96 Hz

SW2 = 1.00 Hz

Hz per Pt2ndD = 1.00 Hz

O1 = -8098.6865 Hz

O2 = -1.0000 Hz

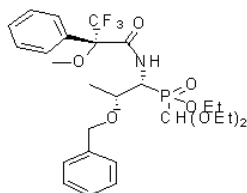
LB1 = 1.00 Hz

TP A = 0.00

B = 0.00

C = 0.00

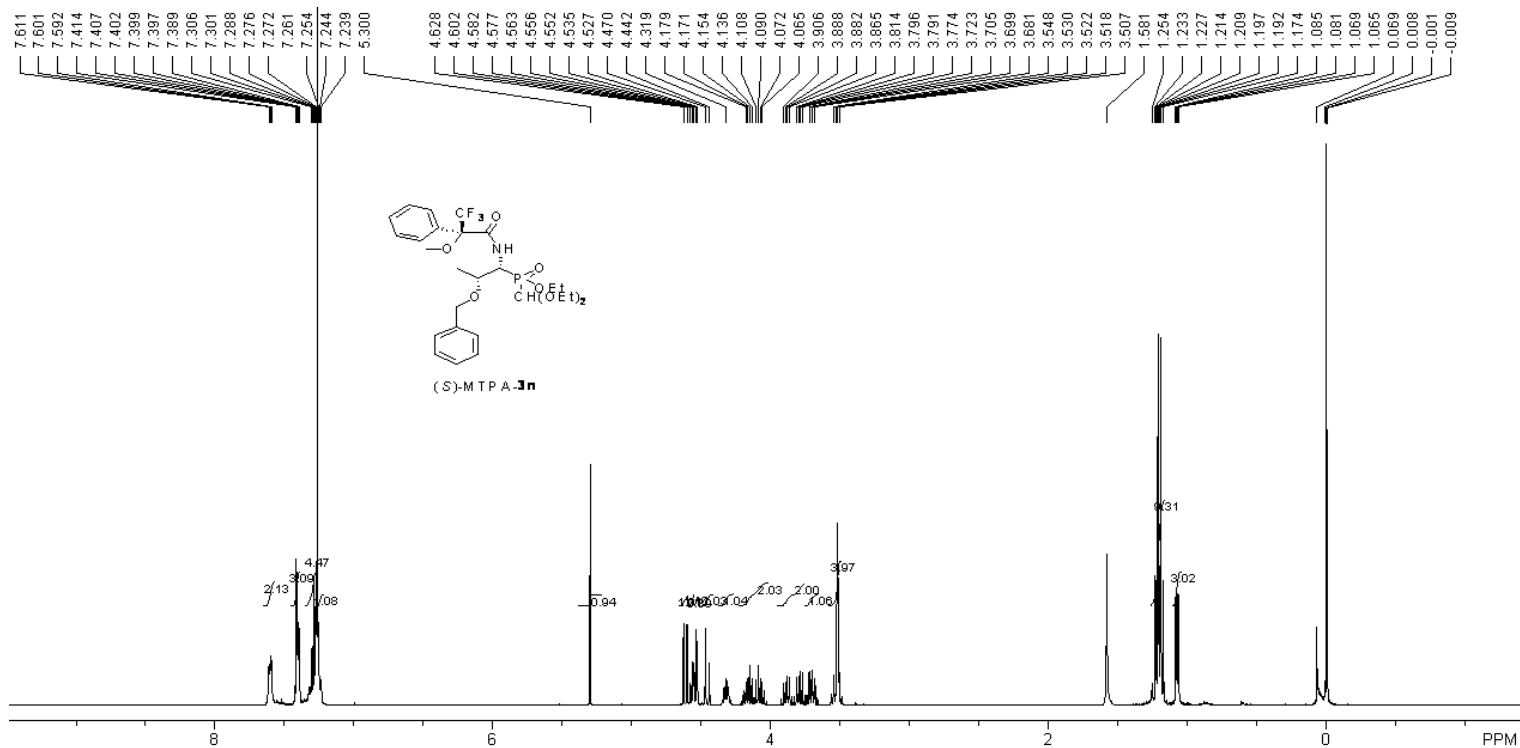
36.228



(R)-MTPA-3n

(S)-Mosher's derivative of the major isomer of 3m:

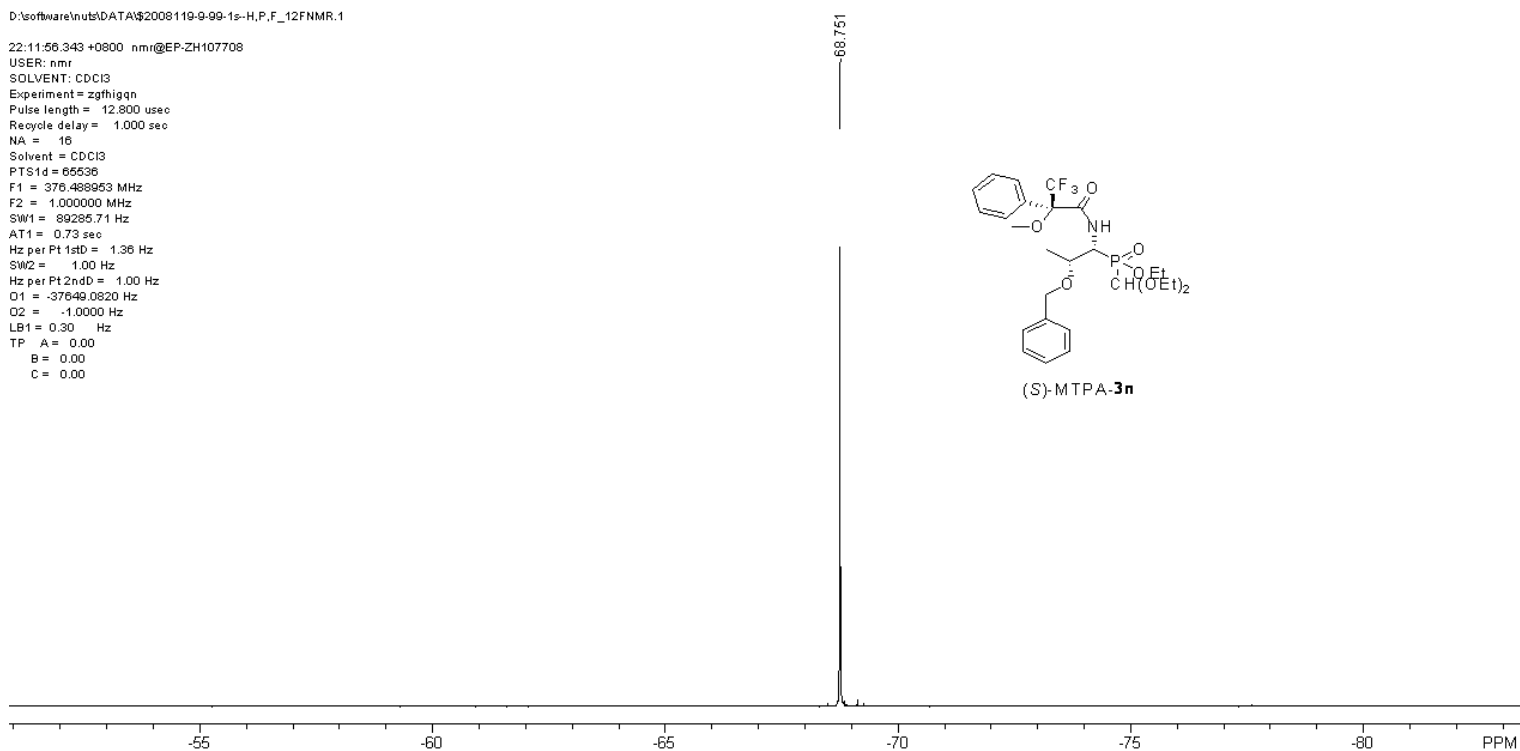
¹H NMR:



¹⁹F NMR:

D:\software\nuts\DATA\2008119-9-99-1s-H,P,F_12FNMNR.1

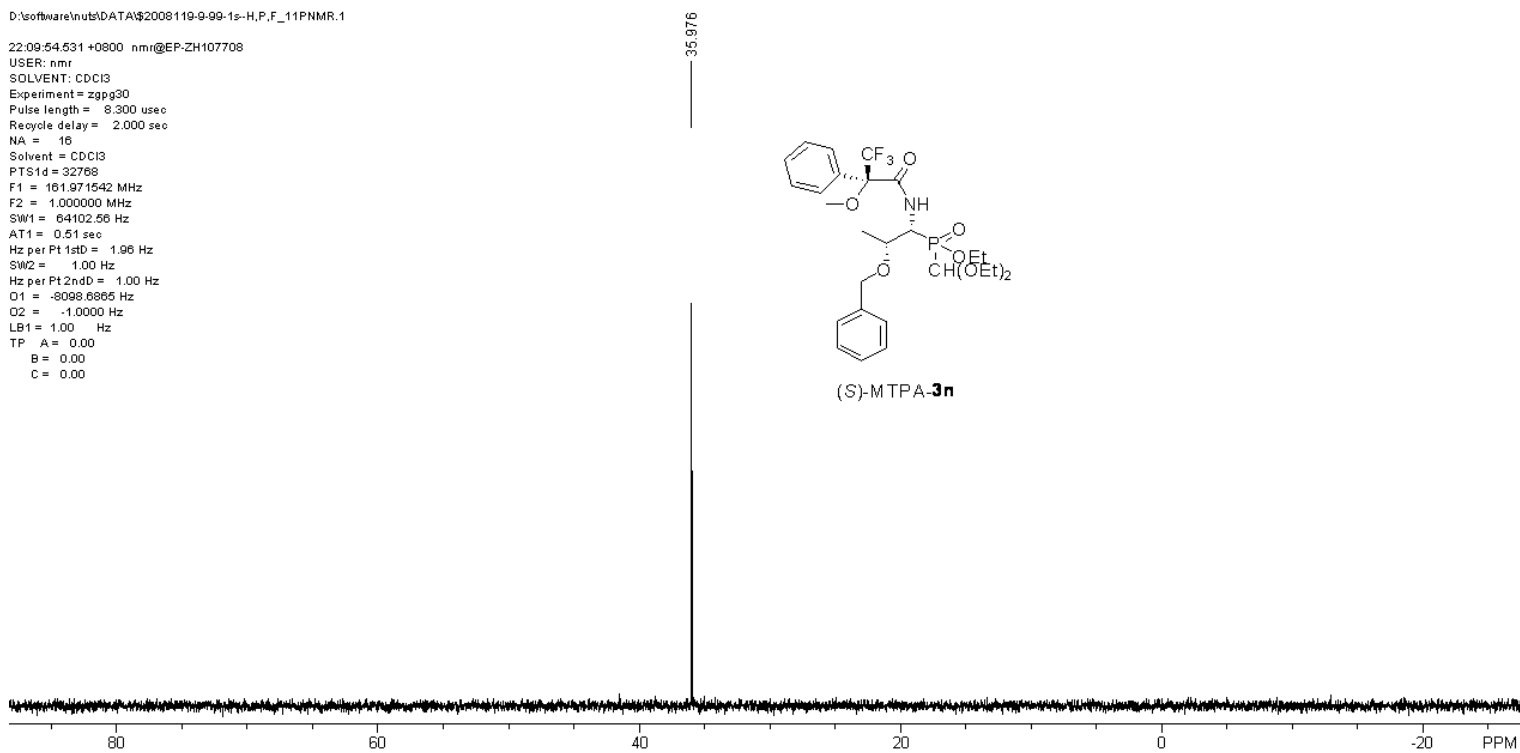
22:11:56.343 +0800 nmri@EP-ZH107708
 USER: nmri
 SOLVENT: CDCl3
 Experiment = zgfhgqn
 Pulse length = 12.800 usec
 Recycle delay = 1.000 sec
 NA = 16
 Solvent = CDCl3
 PTS1d = 65536
 F1 = 376.488953 MHz
 F2 = 1.000000 MHz
 SW1 = 89285.71 Hz
 AT1 = 0.73 sec
 Hz per Pt1stD = 1.36 Hz
 SW2 = 1.00 Hz
 Hz per Pt2ndD = 1.00 Hz
 O1 = -37649.0820 Hz
 O2 = -1.0000 Hz
 LB1 = 0.30 Hz
 TP A = 0.00
 B = 0.00
 C = 0.00



³¹P NMR:

D:\software\nuts\DATA\2008119-9-99-1s-H,P,F_11PNMNR.1

22:09:54.531 +0800 nmri@EP-ZH107708
 USER: nmri
 SOLVENT: CDCl3
 Experiment = zgpg30
 Pulse length = 8.300 usec
 Recycle delay = 2.000 sec
 NA = 16
 Solvent = CDCl3
 PTS1d = 32768
 F1 = 161.971542 MHz
 F2 = 1.000000 MHz
 SW1 = 64102.56 Hz
 AT1 = 0.51 sec
 Hz per Pt1stD = 1.96 Hz
 SW2 = 1.00 Hz
 Hz per Pt2ndD = 1.00 Hz
 O1 = -8098.6965 Hz
 O2 = -1.0000 Hz
 LB1 = 1.00 Hz
 TP A = 0.00
 B = 0.00
 C = 0.00

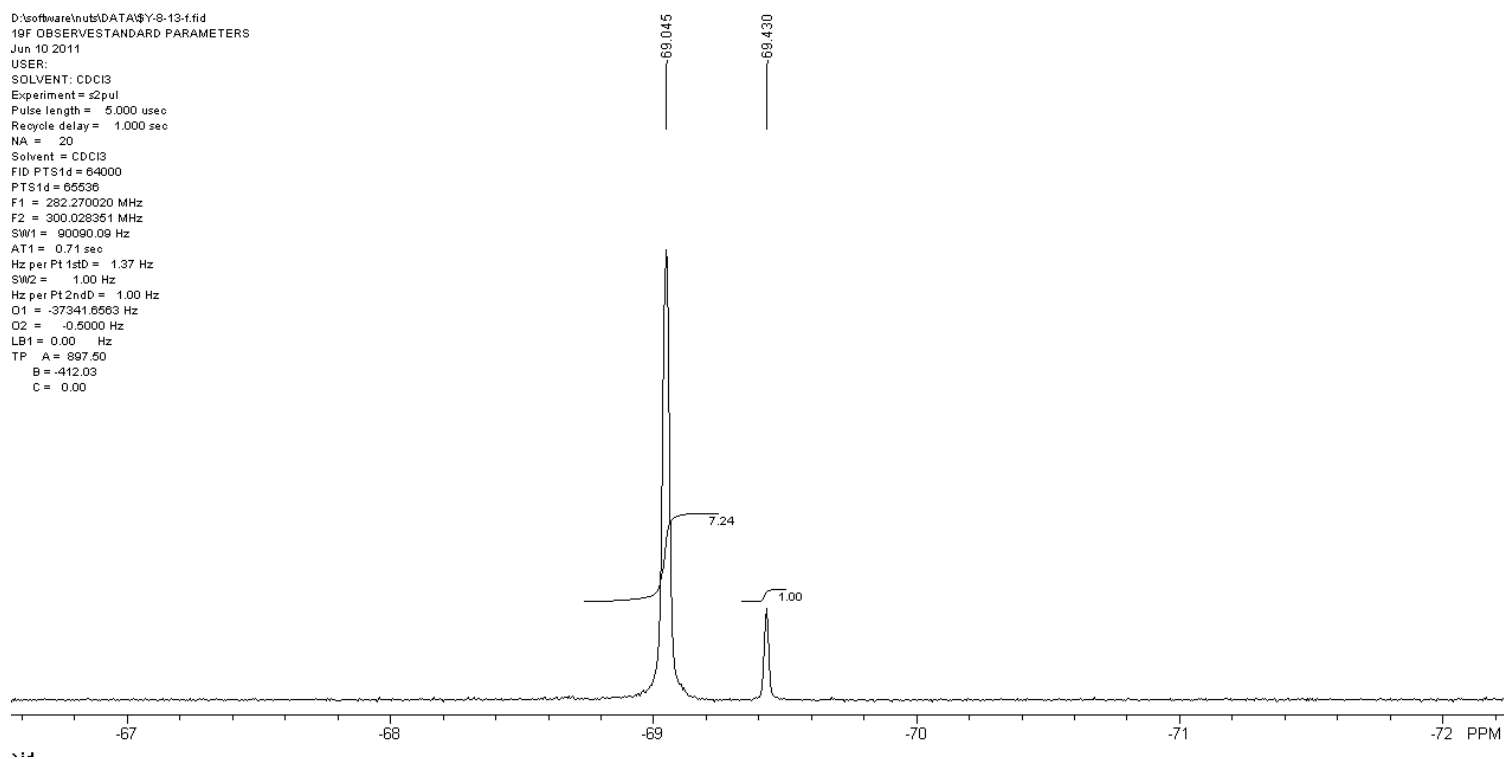


3. NMR Spectra or HPLC Data of Compounds 6 and 7.

NMR spectra of 6:

¹⁹F NMR of 6e:

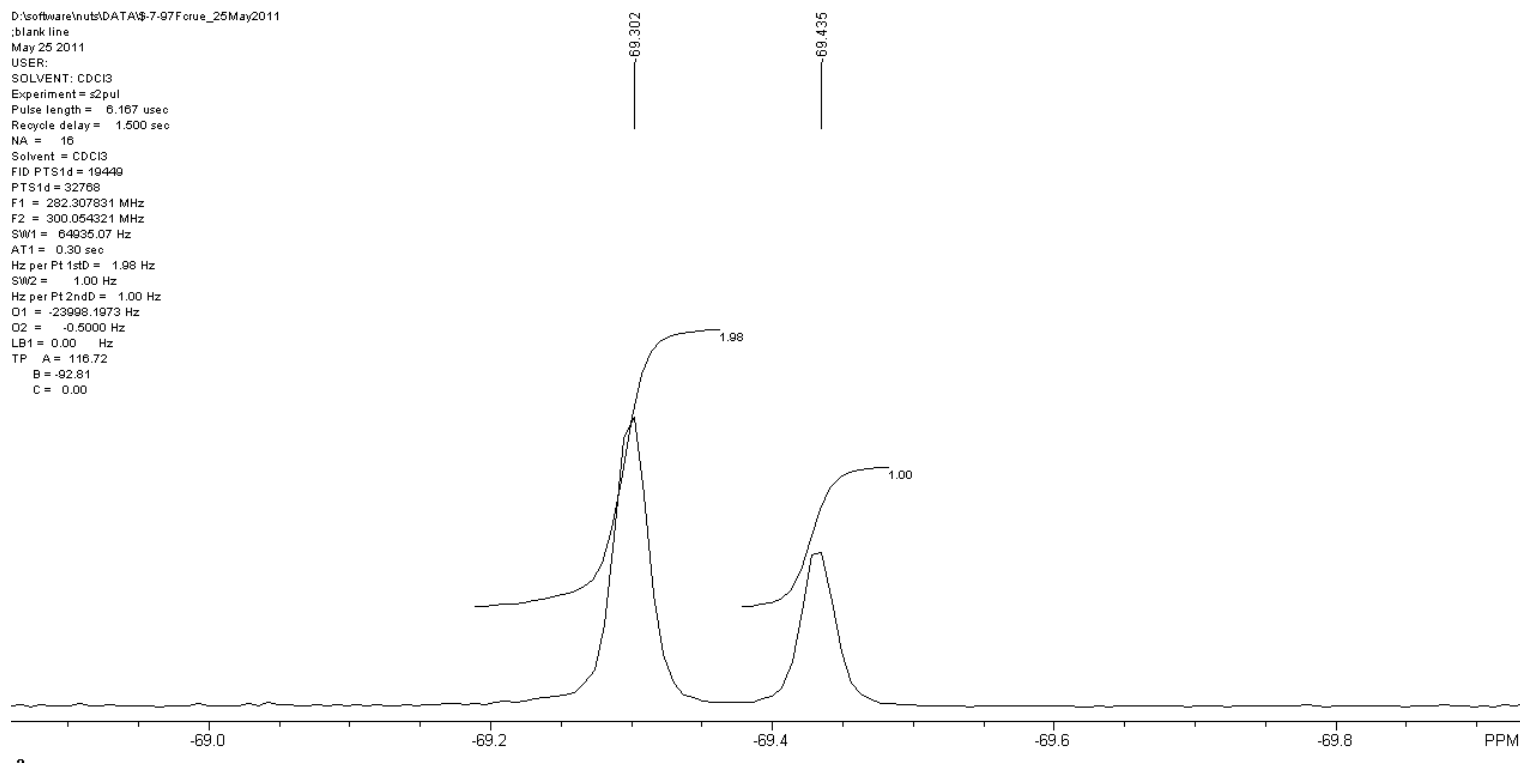
D:\software\nut\DATA\Y-S-13-1.fid
19F OBSERVESTANDARD PARAMETERS
Jun 10 2011
USER:
SOLVENT: CDCl3
Experiment = s2pul
Pulse length = 5.000 usec
Recycle delay = 1.000 sec
NA = 20
Solvent = CDCl3
FID PTS1d = 64000
PTS1d = 65536
F1 = 282.270020 MHz
F2 = 300.028351 MHz
SW1 = 90090.09 Hz
AT1 = 0.71 sec
Hz per Pt1stD = 1.37 Hz
SW2 = 1.00 Hz
Hz per Pt2ndD = 1.00 Hz
O1 = -37341.6563 Hz
O2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = 897.50
B = -412.03
C = 0.00



¹⁹F NMR of 6l:

D:\software\nuts\DATA\7-97F\Forue_25May2011

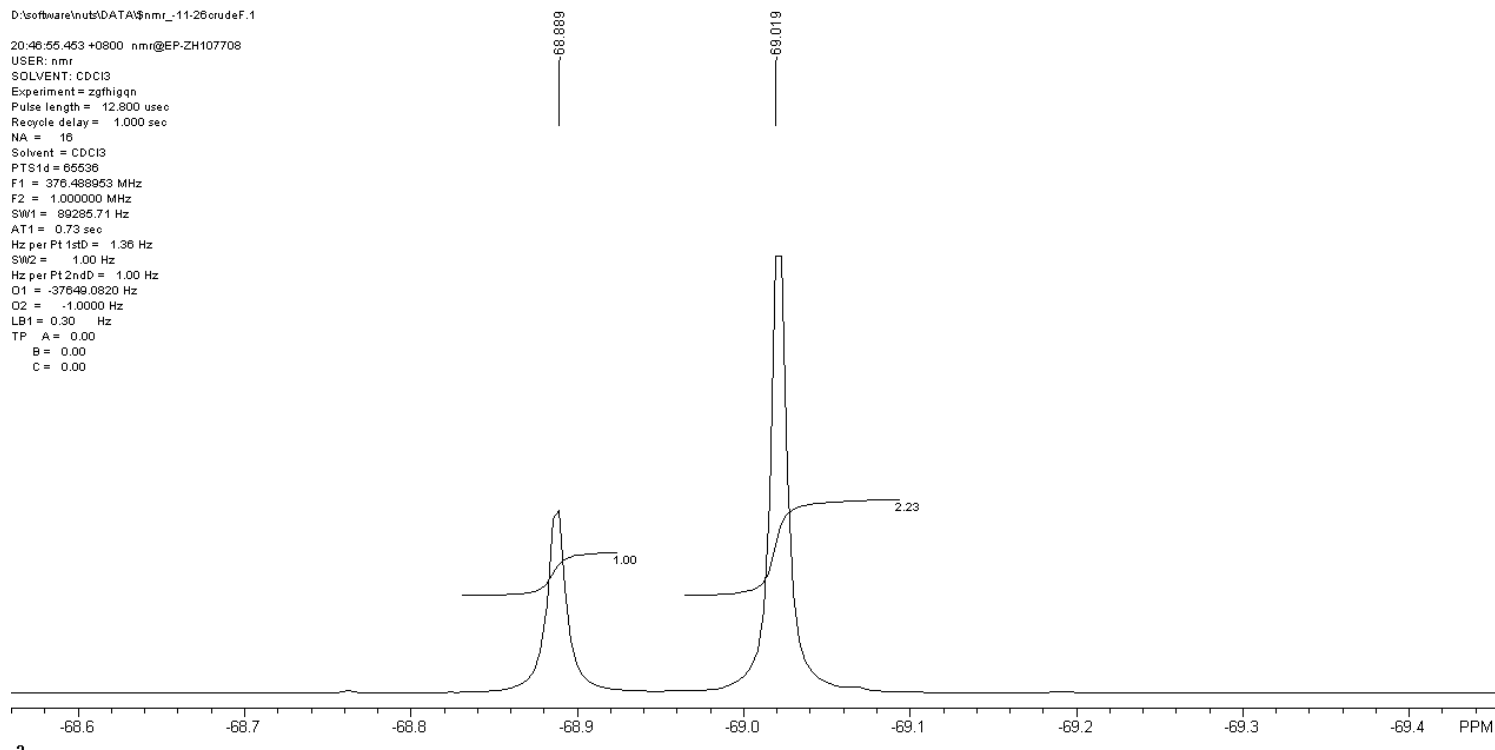
:blank line
May 25 2011
USER:
SOLVENT: CDCl3
Experiment = s2pul
Pulse length = 6.167 usec
Recycle delay = 1.500 sec
NA = 16
Solvent = CDCl3
FID PTS1d = 19446
PTS1d = 32768
F1 = 282.307831 MHz
F2 = 300.054321 MHz
SW1 = 64635.07 Hz
AT1 = 0.30 sec
Hz per Pt1stD = 1.98 Hz
SW2 = 1.00 Hz
Hz per Pt2ndD = 1.00 Hz
O1 = -23998.1973 Hz
O2 = -0.5000 Hz
LB1 = 0.00 Hz
TP A = 116.72
B = -92.81
C = 0.00



¹⁹F NMR of (-)-6l:

D:\software\nuts\DATA\nmr_11-26\crudeF.1

20:46:55.453 +0800 nmr@EP-ZH107708
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 12.800 usec
Recycle delay = 1.000 sec
NA = 16
Solvent = CDCl3
PTS1d = 65536
F1 = 376.488953 MHz
F2 = 1.000000 MHz
SW1 = 89285.71 Hz
AT1 = 0.73 sec
Hz per Pt1stD = 1.36 Hz
SW2 = 1.00 Hz
Hz per Pt2ndD = 1.00 Hz
O1 = -37649.0820 Hz
O2 = -1.0000 Hz
LB1 = 0.30 Hz
TP A = 0.00
B = 0.00
C = 0.00



¹⁹F NMR of 6n:

D:\software\nuts\DATA\10_10-34F.1

20:29:29.640 +0800 nmri@EP.ZH107708

USER: nmri

SOLVENT: CDCl3

Experiment = zgfhigqn

Pulse length = 12.800 usec

Recycle delay = 1.000 sec

NA = 16

Solvent = CDCl3

PTS1d = 65536

F1 = 376.488953 MHz

F2 = 1.000000 MHz

SW1 = 89285.71 Hz

AT1 = 0.73 sec

Hz per Pt1stD = 1.36 Hz

SW2 = 1.00 Hz

Hz per Pt2ndD = 1.00 Hz

O1 = -37649.0820 Hz

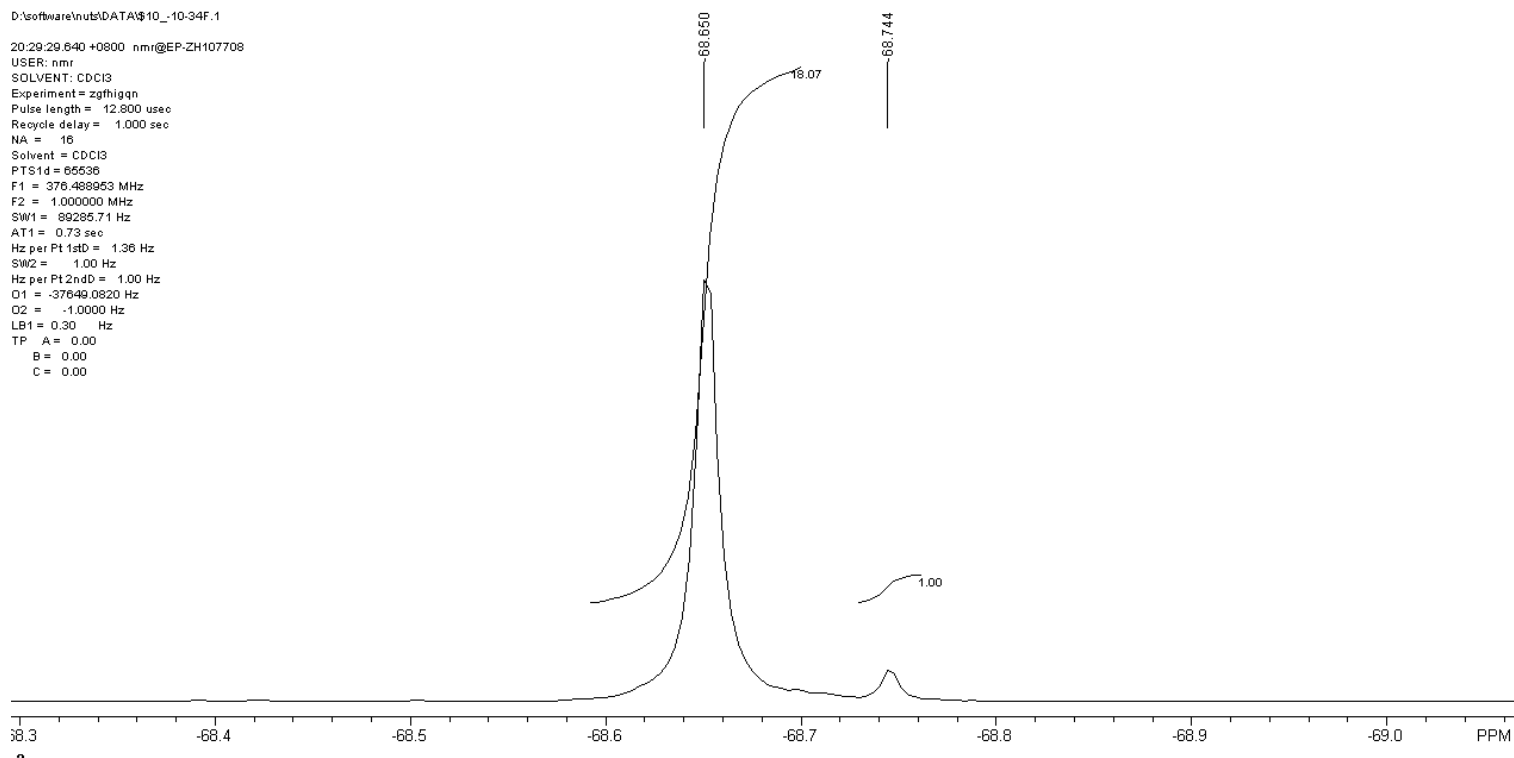
O2 = -1.0000 Hz

LB1 = 0.30 Hz

TP A = 0.00

B = 0.00

C = 0.00

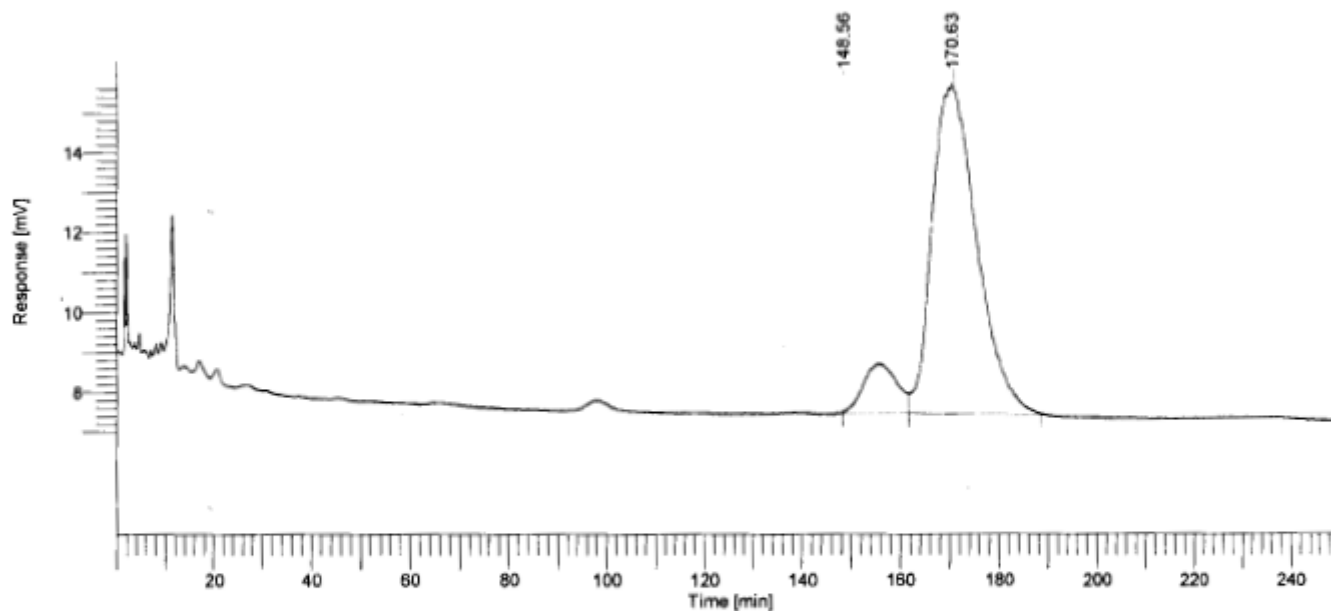


HPLC data for 6b.

Software Version : 6.3.0.0445
Sample Name : 7-73
Instrument Name : Perkin Elmer LC
Rack/Vial : 0/0
Sample Amount : 1.000000
Cycle : 1

Date : 2011-5-16 13:43:38
Data Acquisition Time : 2011-5-16 9:18:07
Channel : A
Operator : manager
Dilution Factor : 1.000000

Result File : D:\YZR\7-73.rst
Sequence File : D:\YZR\20110511-.seq



HPLC REPORT

Peak #	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	BL
1	148.560	585731.15	42.64	10.127	*BV
2	170.632	5197930.82	8308.32	89.873	*VB
		5783661.98	8350.96	1e+02	

HPLC data for 7f.

AD-H, n-hexane/2-propanol .50:50, $\lambda = 214 \text{ nm}$, flow rate = 0.7 mL min^{-1} , retention time = 5.18 and 6.18 min.

Racemate:

HPLC Report

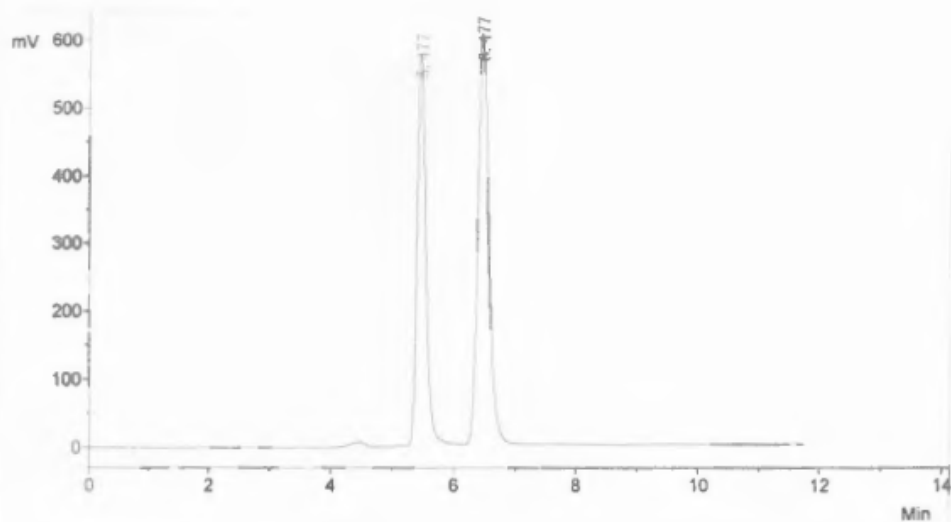
Sample Name:

Data File: 5-63+-AD-H550.7214.che

Operator:

Date: 2010-10-20

Time: 08:25



No.	PeakNo	R. Time	PeakHeight	PeakArea	PerCent
1	1	5.177	532217.2	5805852.3	12.8142
2	2	6.177	596499.6	7751709.9	57.1858
Total			1128716.8	13560562.2	100.0000

7f:

色谱分析报告

样品名称:

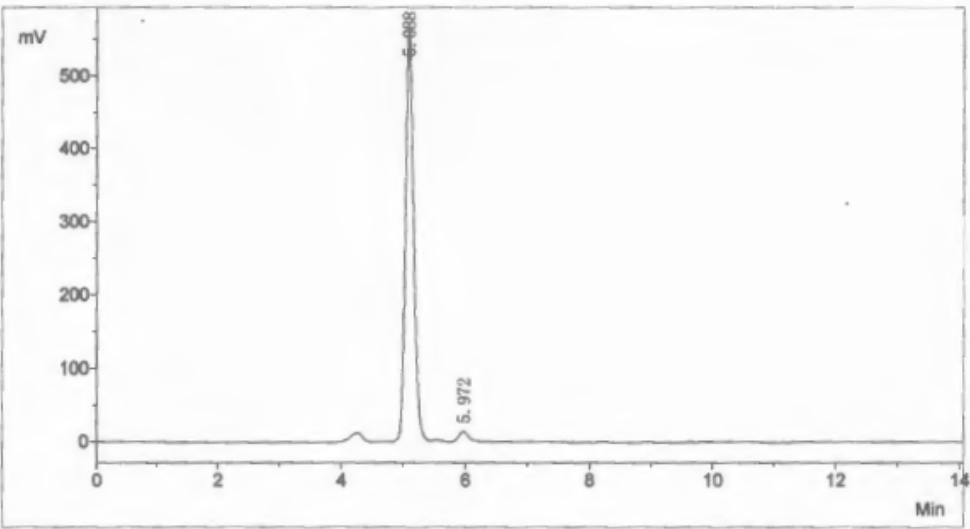
样品批号:

分析日期:2010-10-12

样品文件名:5-46. che

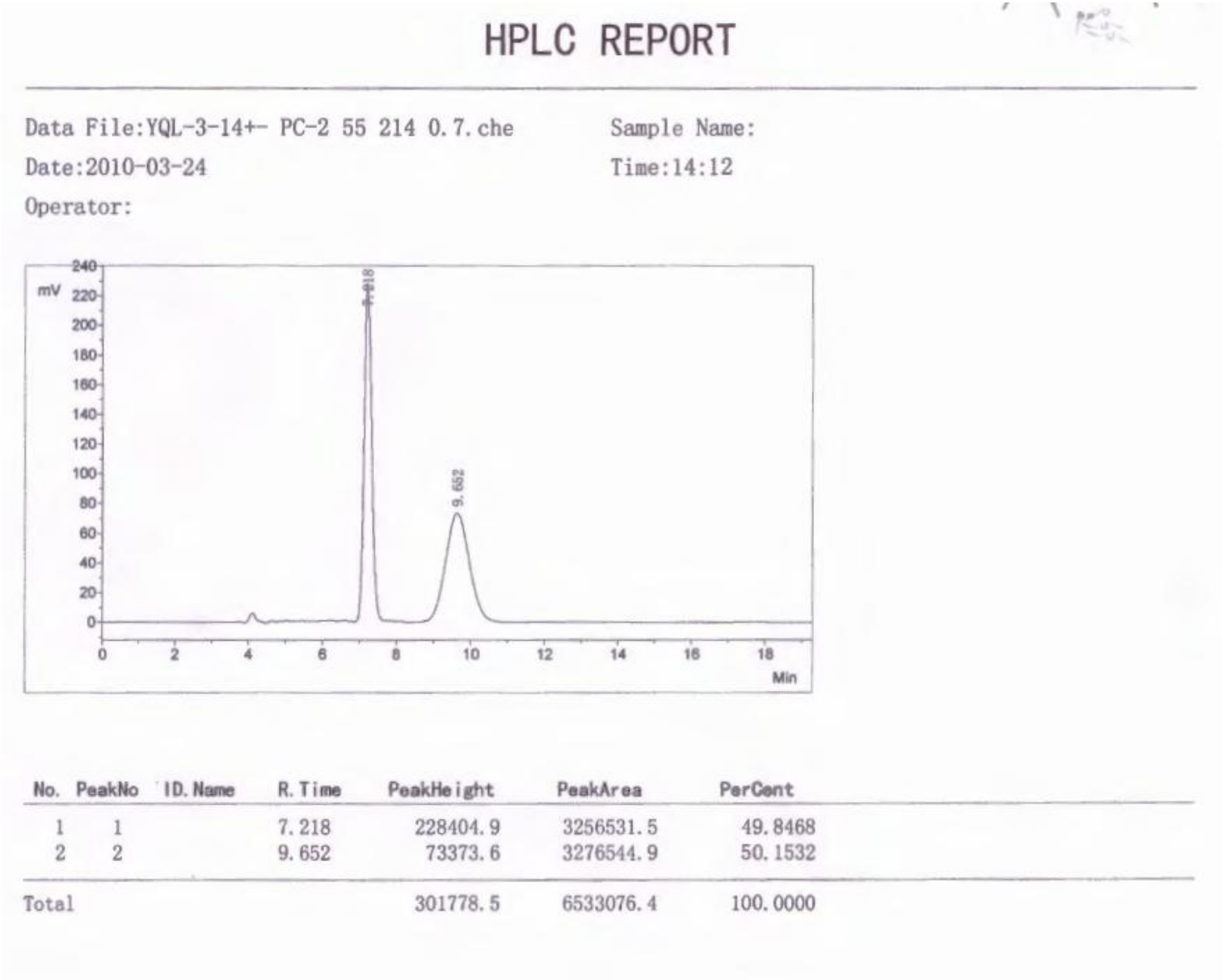
分析者:

分析时间:14:05



序号	峰号	组份名	保留时间	峰高	峰面积	面积百分比(%)
1	1		5.088	563256.2	5098322.3	97.2054
2	2		5.972	13911.3	146576.4	2.7946
合计:				577167.4	5244898.7	100.0000

HPLC data for 7g.
Chiralpak PC-2, n-hexane/2-propanol 50:50, $\lambda = 214 \text{ nm}$, flow rate = 0.7 mL min^{-1} , retention time = 9.6 and 7.2 min.
Racemate:



7g:

HPLC REPORT

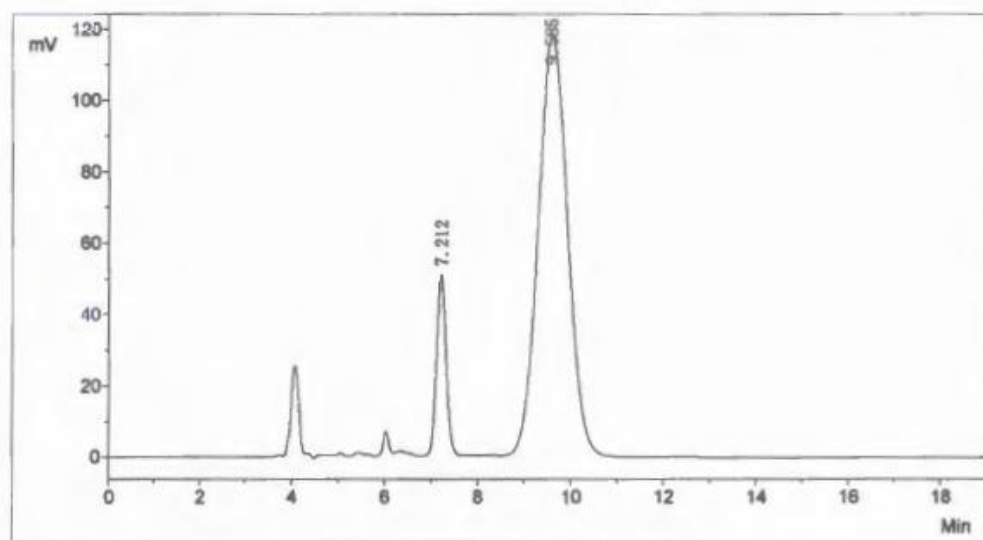
Data File: YQL-3-24. che

Sample Name:

Date: 2010-03-24

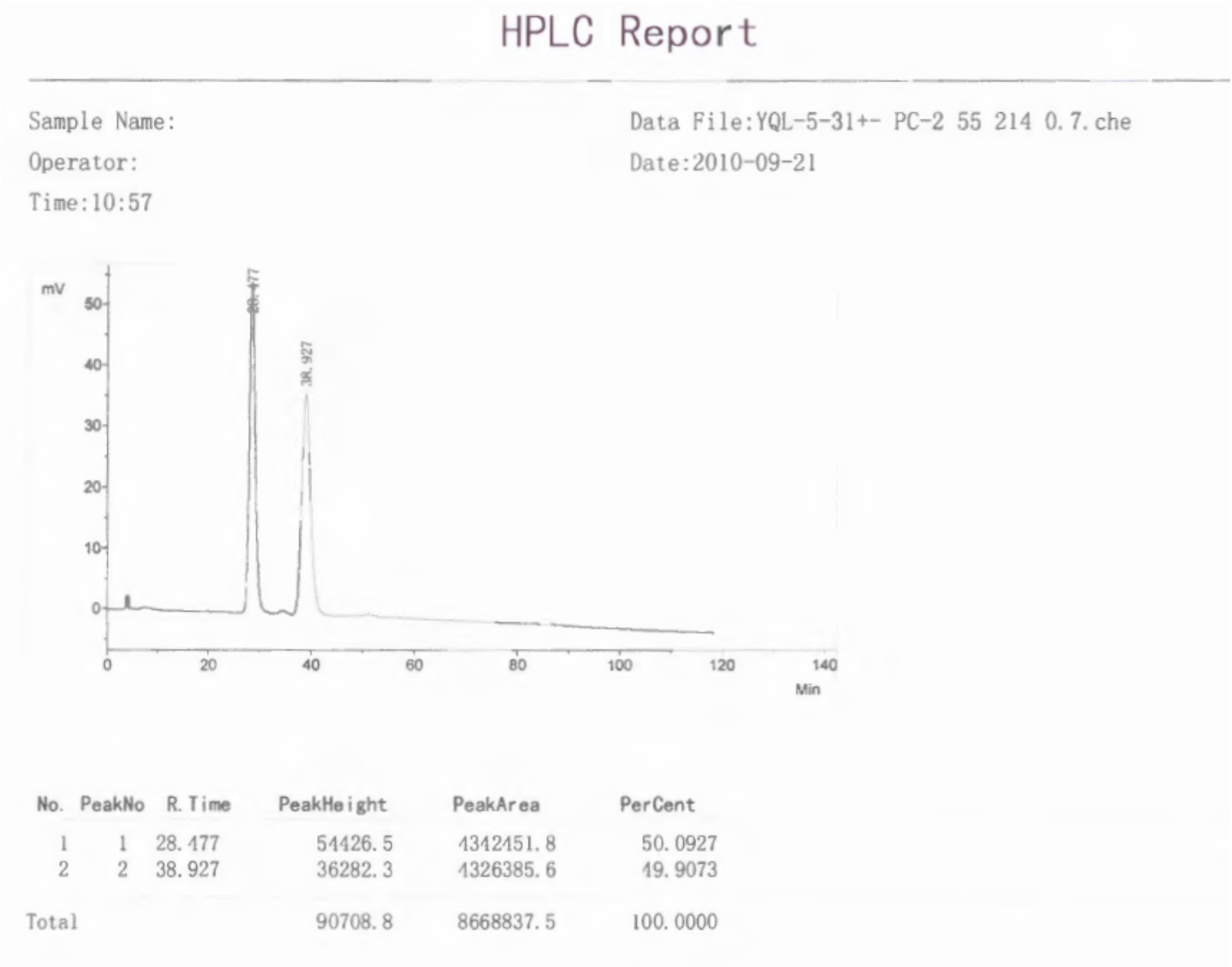
Time: 14:38

Operator:



No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1		7.212	50774.5	725088.9	12.2653
2	2		9.585	117803.6	5186601.1	87.7347
Total				168578.1	5911690.0	100.0000

HPLC data for 7h.
Chiralpak PC-2, n-hexane/2-propanol 50:50, $\lambda = 214 \text{ nm}$, flow rate = 0.7 mL min^{-1} , retention time = 39.4 and 28.8 min.
Racemate:



7h:

HPLC Report

ce 887.

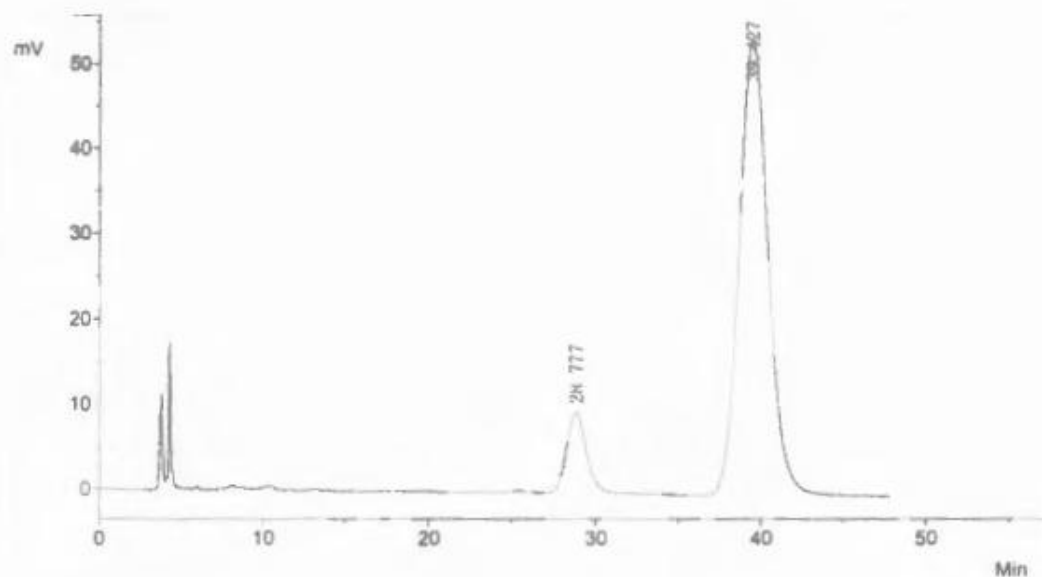
Sample Name:

Data File:YQL-5-27-3.che

Operator:

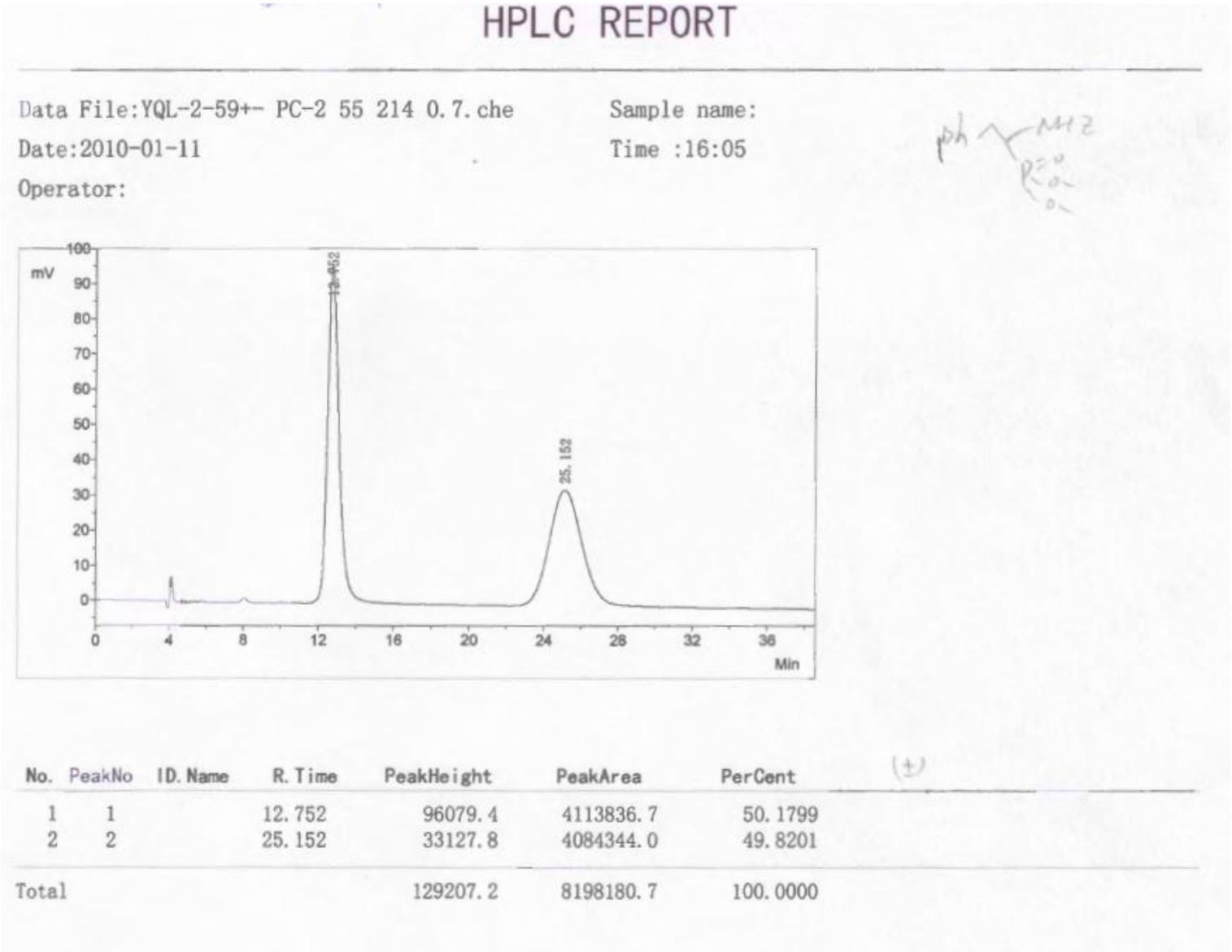
Date:2010-09-21

Time:13:01



No.	PeakNo	R. Time	PeakHeight	PeakArea	PerCent
1	1	28.777	9436.4	772754.6	10.5604
2	2	39.427	53708.4	6541713.7	89.4396
Total			63144.8	7317468.3	100.0000

HPLC data for 7j.
Chiralpak PC-2, n-hexane/2-propanol 50:50, $\lambda = 214 \text{ nm}$, flow rate = 0.7 mL min^{-1} , retention time = 23.0 and 11.9 min.
Racemate:



7j:

HPLC REPORT

CHILL

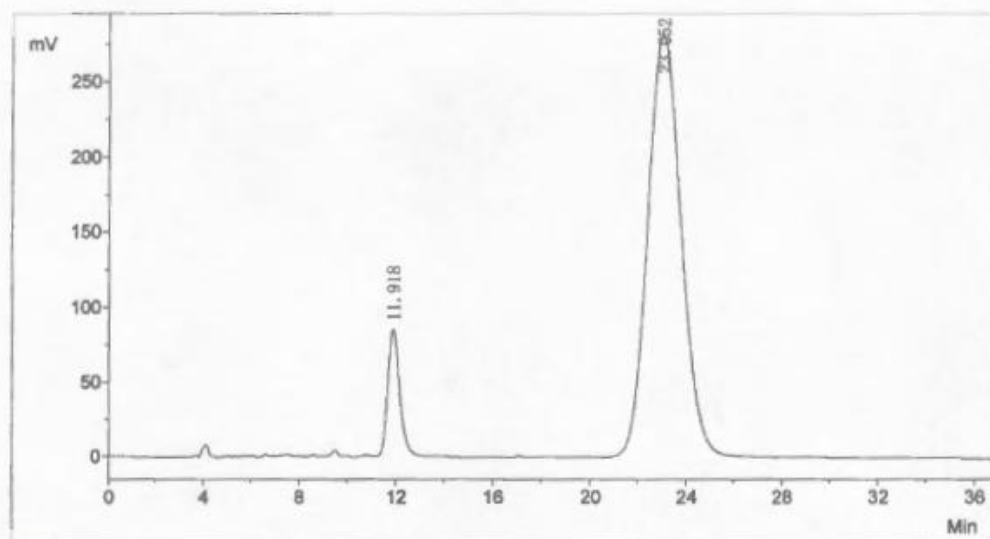
Data File:YQL-2-65. che

Sample name:

Date:2010-01-12

Time :11:01

Operator:



No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1		11.918	85503.3	2950166.4	9.7209
2	2		23.052	282112.0	27398521.9	90.2791
Total				367615.3	30348688.4	100.0000

HPLC data for 7k.

Chiralpak PA-2, n-hexane/2-propanol 50:50, $\lambda = 214$ nm, flow rate = 0.7 mL min⁻¹, retention time = 17.7 and 15.1 min.

Racemate:

HPLC Report

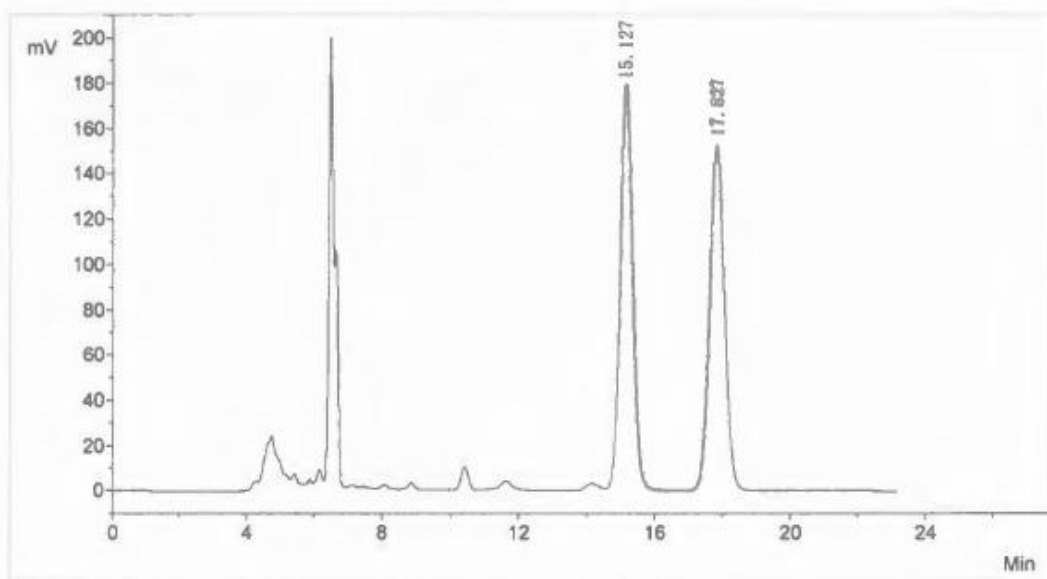
Sample Name:

Data File:5-82+-PA-2550.7214.che

Operator:

Date:2010-11-10

Time:13:08



No.	PeakNo	R. Time	PeakHeight	PeakArea	PerCent
1	1	15.127	178216.5	4577516.0	49.5098
2	2	17.827	152391.7	4668167.8	50.4902
Total			330608.2	9245683.8	100.0000

7k:

HPLC Report

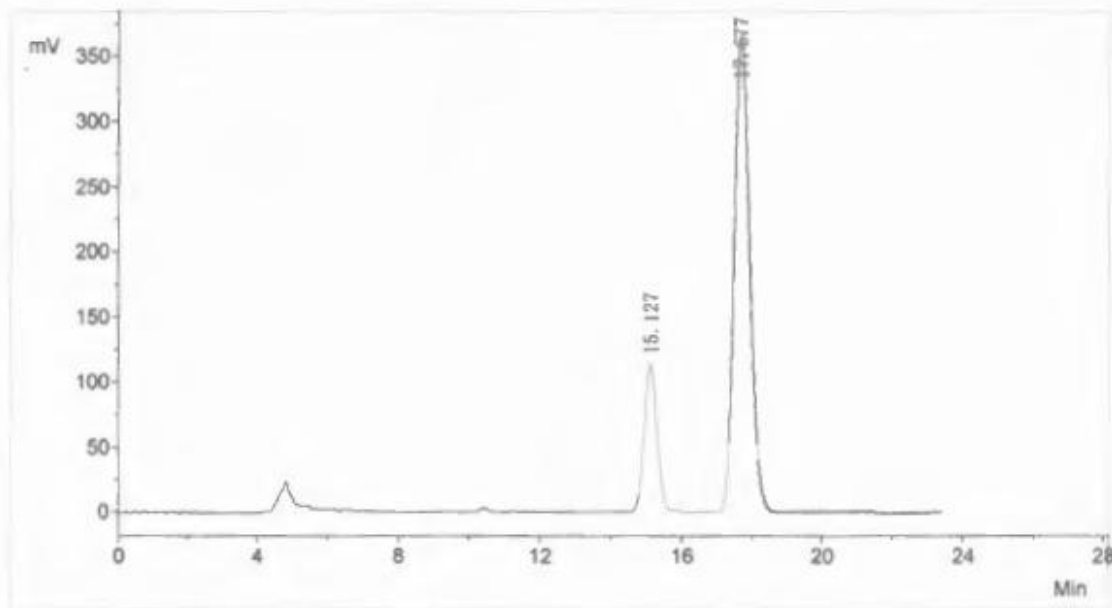
Sample Name:

Data File:5-81.che

Operator:

Date:2010-11-10

Time:13:35



No.	PeakNo	R. Time	PeakHeight	PeakArea	PerCent
1	1	15.127	113872.6	2928463.7	20.6252
2	2	17.677	364845.4	11270025.9	79.3748
Total			478718.0	14198489.6	100.0000

58.87.

HPLC data for 7o.

Chiralpak PC-2, n-hexane/2-propanol 50:50, $\lambda = 214$ nm, flow rate = 0.7 mL min^{-1} , retention time = 26.5 and 17.0 min.

Racemate:

HPLC REPORT

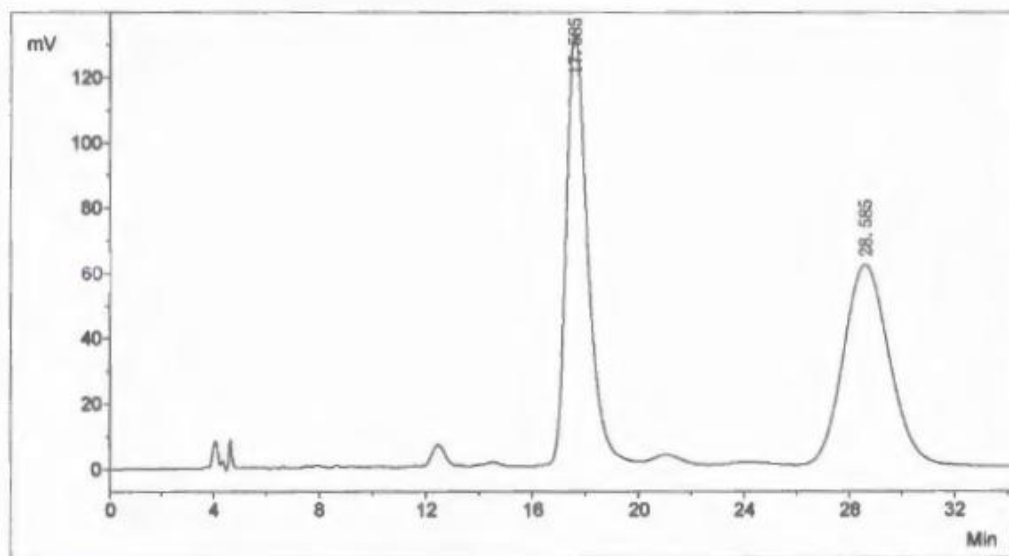
Data File: YQL-3-20+- PC-2 55 214 0.7. che

Sample Name:

Date: 2010-03-24

Time: 11:48

Operator:



No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1		17.585	131680.6	7380205.4	49.9689
2	2		28.585	61418.2	7389381.1	50.0311
Total				193098.9	14769586.5	100.0000

HPLC REPORT

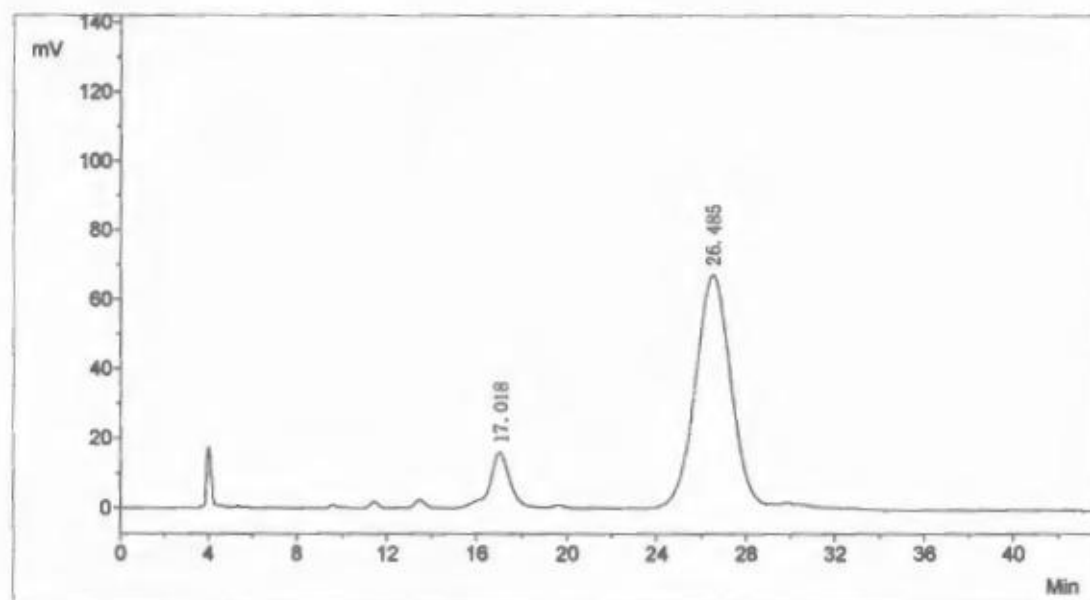
Data File: YQL-3-67. che

Sample Name:

Date: 2010-05-10

Time: 16:41

Operator:



No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1		17.018	15965.1	992469.0	12.0368
2	2		26.485	66646.7	7252821.8	87.9632
Total				82611.9	8245290.8	100.0000

HPLC data for 7p.

Chiralpak PC-2, n-hexane/2-propanol 50:50, $\lambda = 214$ nm, flow rate = 0.7 mL min^{-1} , retention time = 8.9 and 7.5 min.

Racemate:

HPLC REPORT

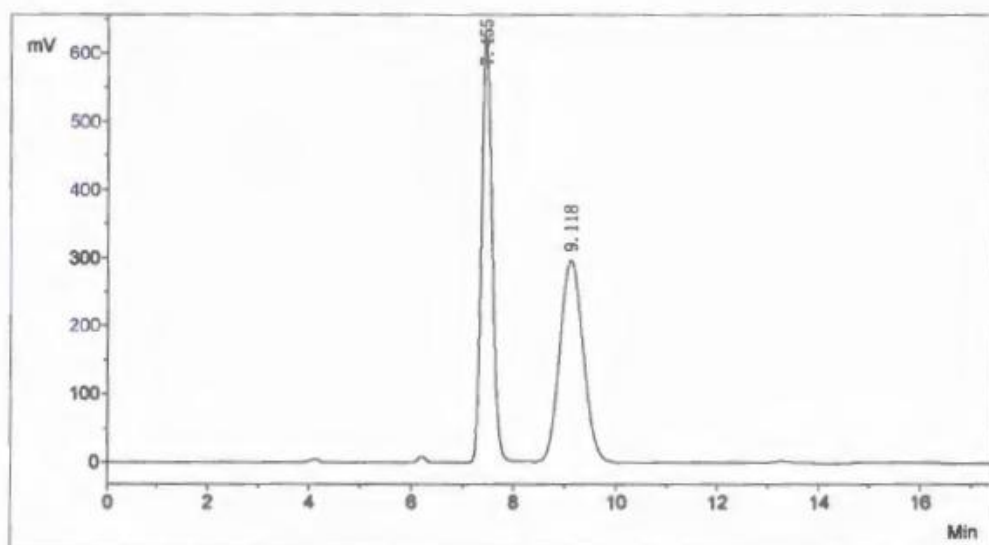
Data File: YQL-2-49+- PC-2 55 214 0.7. che

Sample Name:

Date: 2010-03-03

Time: 16:40

Operator:



No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1		7.455	624336.6	9009234.9	49.1671
2	2		9.118	295614.5	9314475.8	50.8329
Total				919951.1	18323710.7	100.0000

HPLC REPORT

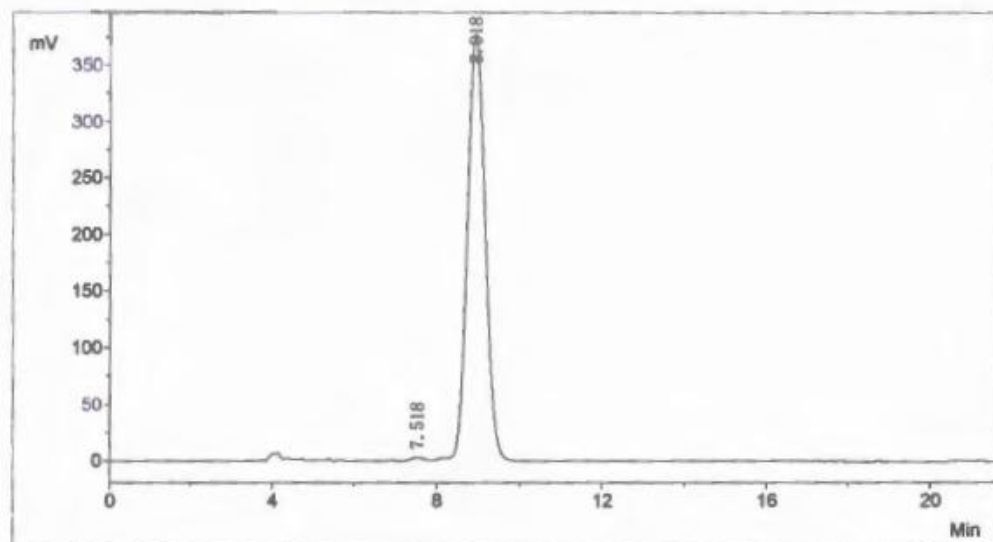
Data File: YQL-3-6..che

Sample Name:

Date: 2010-03-24

Time: 15:05

Operator:



No.	PeakNo	ID. Name	R. Time	PeakHeight	PeakArea	PerCent
1	1		7.518	2583.6	47181.7	0.4240
2	2		8.918	374352.4	11081865.1	99.5760
Total				376936.0	11129046.8	100.0000