

Supporting Information

Microwave Assisted Chemoselective Peptide Alcohol Synthesis from C-terminal Amide

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1 Copies of NMR (¹H and ¹³C) and HRMS spectra:

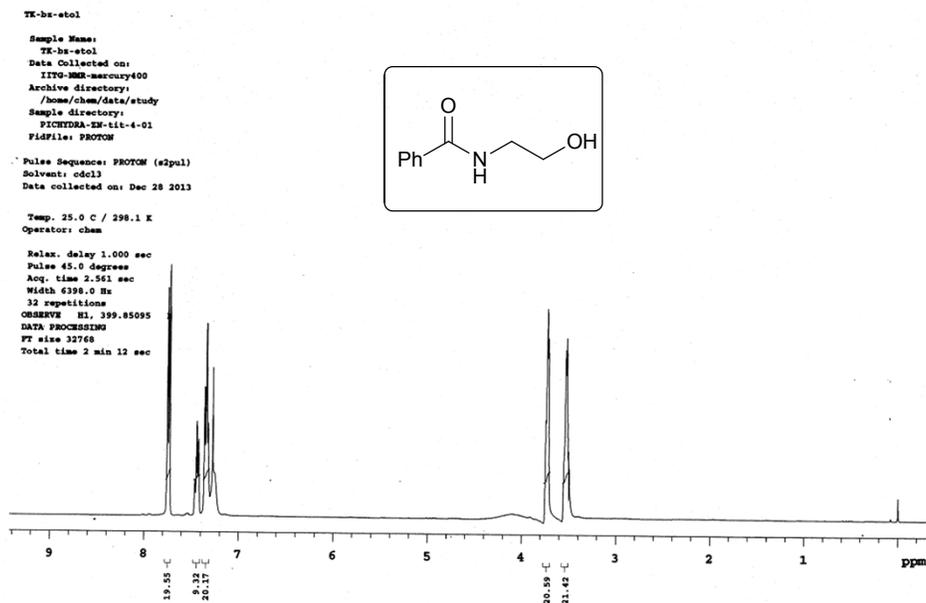


Figure S1. ¹H NMR spectra of the product of entry 1 in table 2

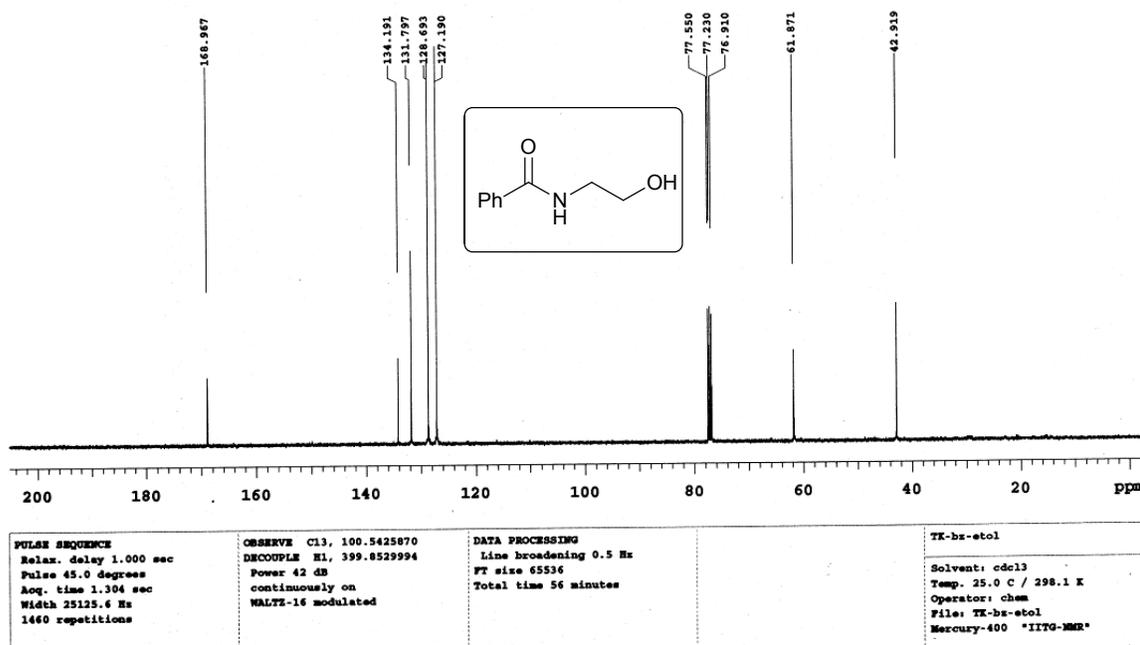


Figure S2. ¹³C NMR spectra of the product of entry 1 in table 2

Sample Name TK-BZALOL Position -1 Instrument Name Instrument 1 User Name
 Inj Vol -10 InjPosition SampleType Sample IRM Calibration Status Success
 Data Filename TK-BZALOL.d ACQ Method Comment Acquired Time 5/14/2013 3:43:28 PM

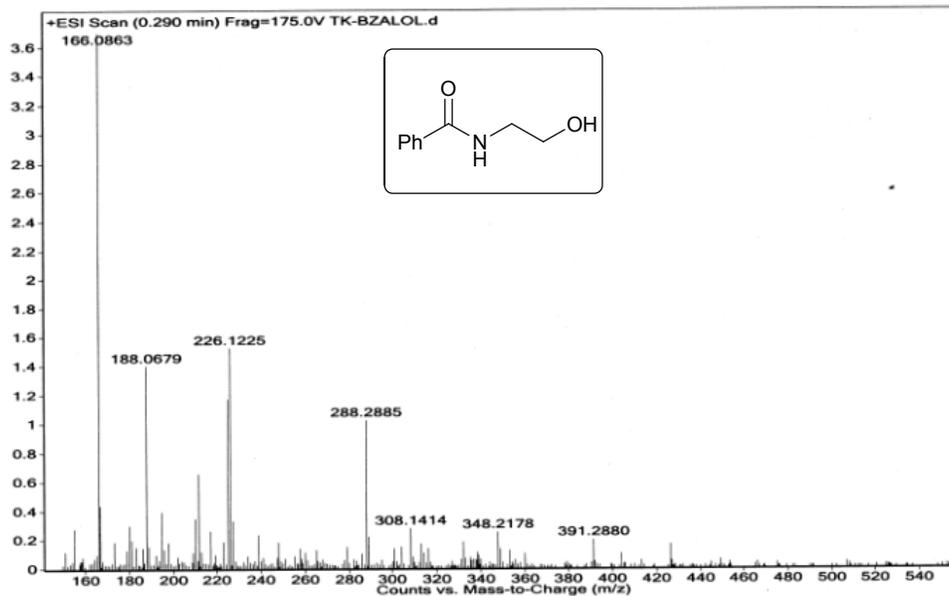


Figure S3. HRMS spectra of the product of entry 1 in table 2

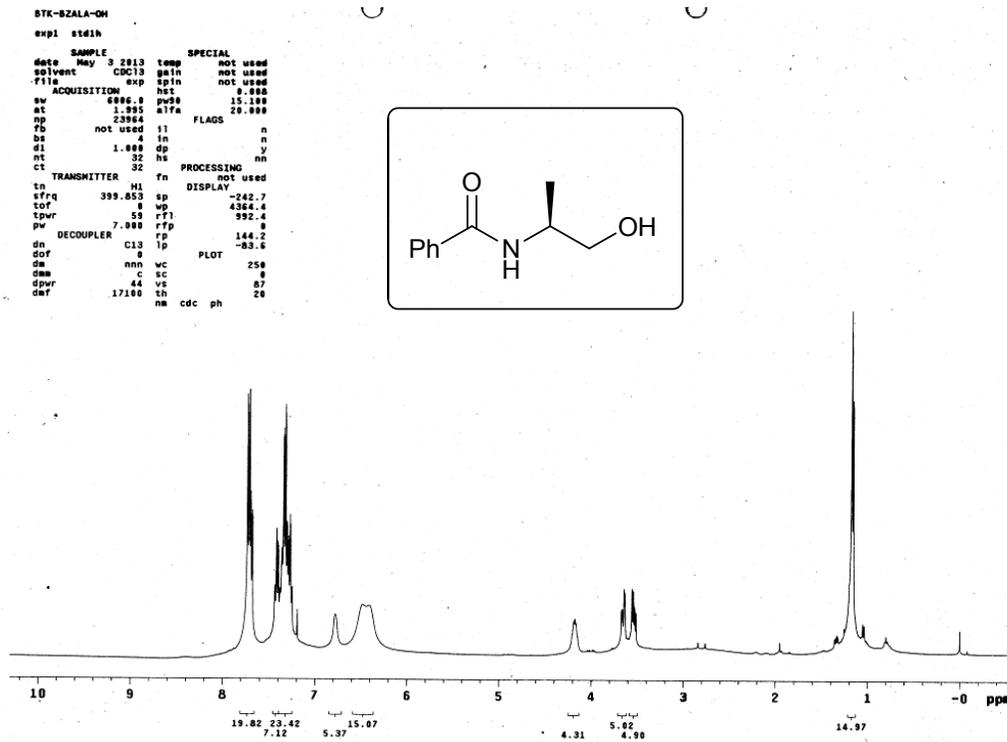


Figure S4. ¹H NMR spectra of the product of entry 2 in table 2

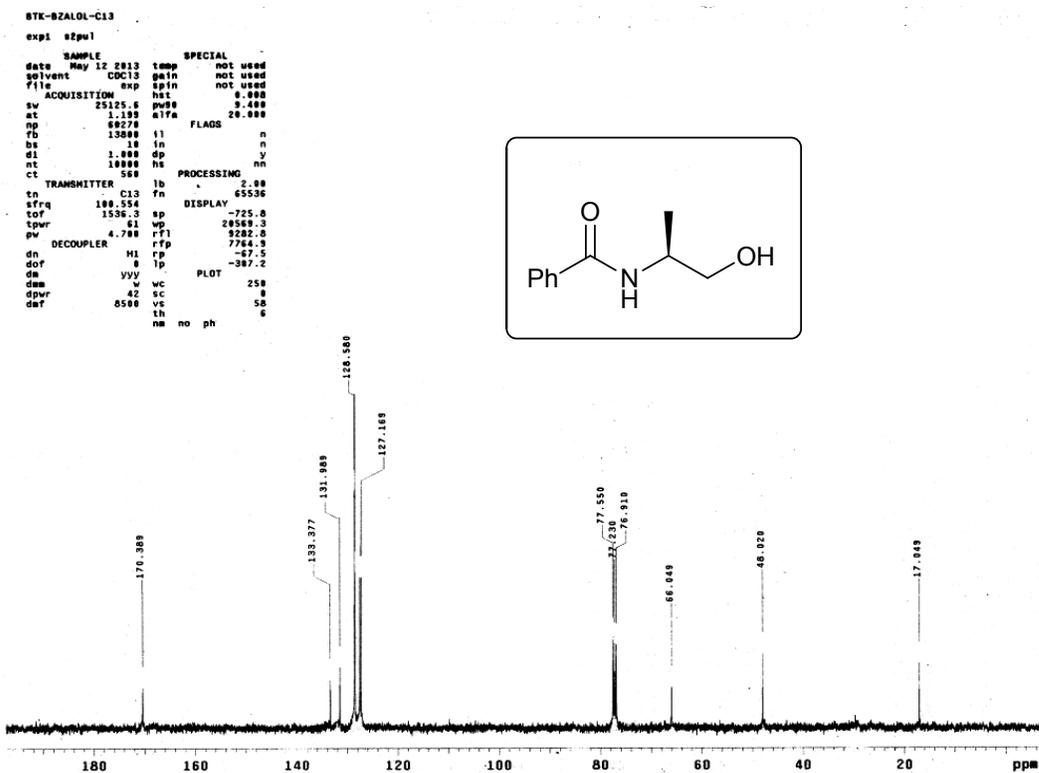


Figure S5. ^{13}C NMR spectra of the product of entry 2 in table 2

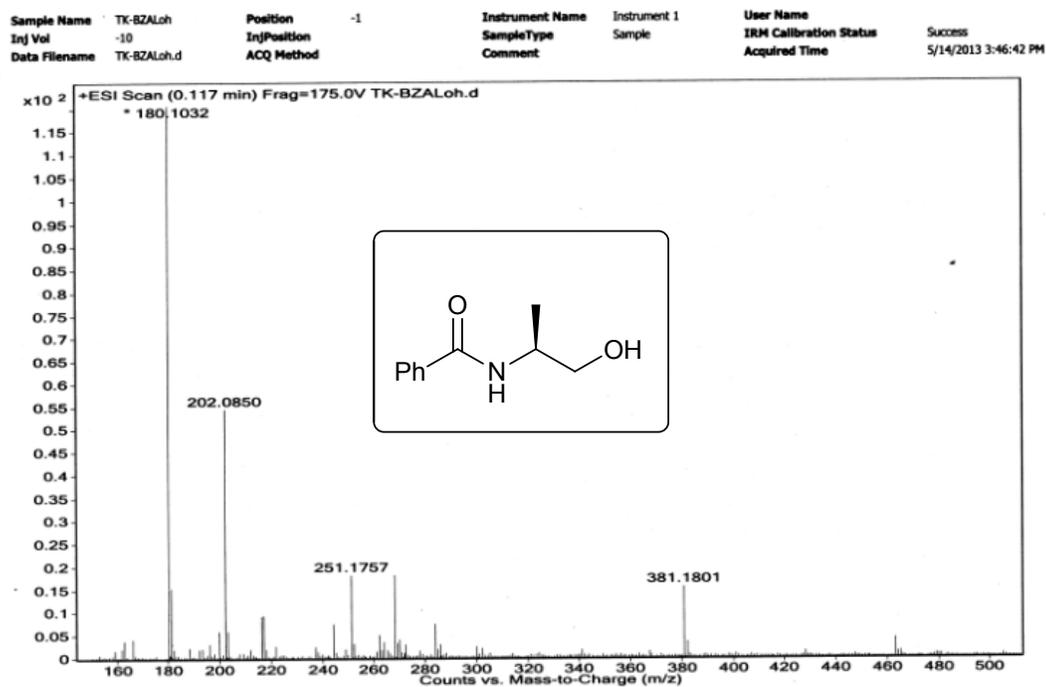


Figure S6. HRMS spectra of the product of entry 2 in table 2

```

BTK-PHMHCOHMET-OL
exp1 s2pu1
SAMPLE
date Jun 3 2013 temp not used
solvent CDCl3 gain not used
file not used
ACQUISITION exp spin not used
ht 0.000
sw 6389.6 pw9 15.148
at 1.888 a1fa 28.000
np 25528
fb not used f1 n
bs 4 in n
d1 1.000 dp y
nt 32 hs PROCESSING nn
ct 32
TRANSMITTER t1 fb 0.10
fn 65536
sfrq 399.653 DISPLAY
tof 362.8 sp -226.6
tpr 50 wp 4727.2
pw 7.550 rf1 789.0
DECOUPLER rfp 0
dn C13 rp 148.0
dof 0 lp PLOT -67.0
dm nnn
dmm C wc 250
dpr 44 sc 0
dpt 17100 vs 50
dat th 7
nm cdc ph

```

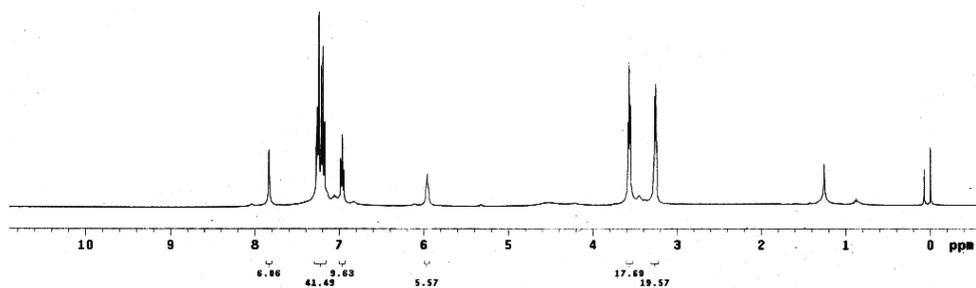
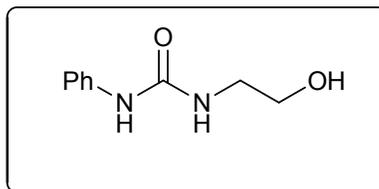


Figure S7. ¹H NMR spectra of the product of entry 3 in table 2

```

BTK-PHMHCOETOL
exp1 s2pu1
SAMPLE
date May 12 2013 temp not used
solvent CDCl3 gain not used
file /export/home/ spin not used
mercury/BTK-PHMHCO- hst 0.000
ACQUISITION pw9 9.400
a1fa 20.000
sw 25125.6 f1 n
at 1.150 f2 n
fb 60270 in n
np 13800 dp y
bs 19 hs PROCESSING nn
d1 1.000
nt 10000 lb 2.00
ct 780 fn 65536
TRANSMITTER C13 sp DISPLAY -524.9
fn 21229.5
sfrq 100.554 wp 21229.5
tof 1536.3 rf1 9279.8
tpr 61 rfp 7764.9
pw 4.700 rp -55.2
DECOUPLER H1 lp PLOT -338.1
dn 0 wc 250
dof 0 sc 0
dm vvv vs 52
dmm 42 th 3
dpr 8500 nm no ph

```

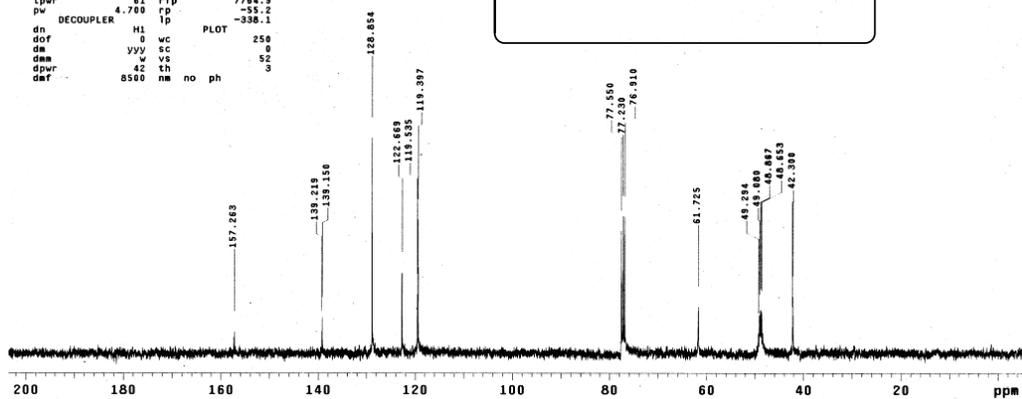
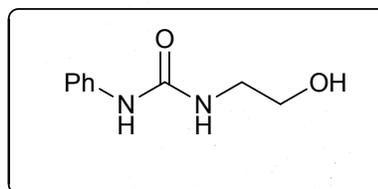


Figure S8. ¹³C NMR spectra of the product of entry 3 in table 2

Sample Name TK-ETHANOL-AMINE Position -1 Instrument Name Instrument 1 User Name
 Inj Vol -10 InjPosition SampleType Sample IRM Calibration Status Success
 Data Filename TK-ETHANOL-AMINE.d ACQ Method Comment Acquired Time 11/11/2013 10:49:52 AM

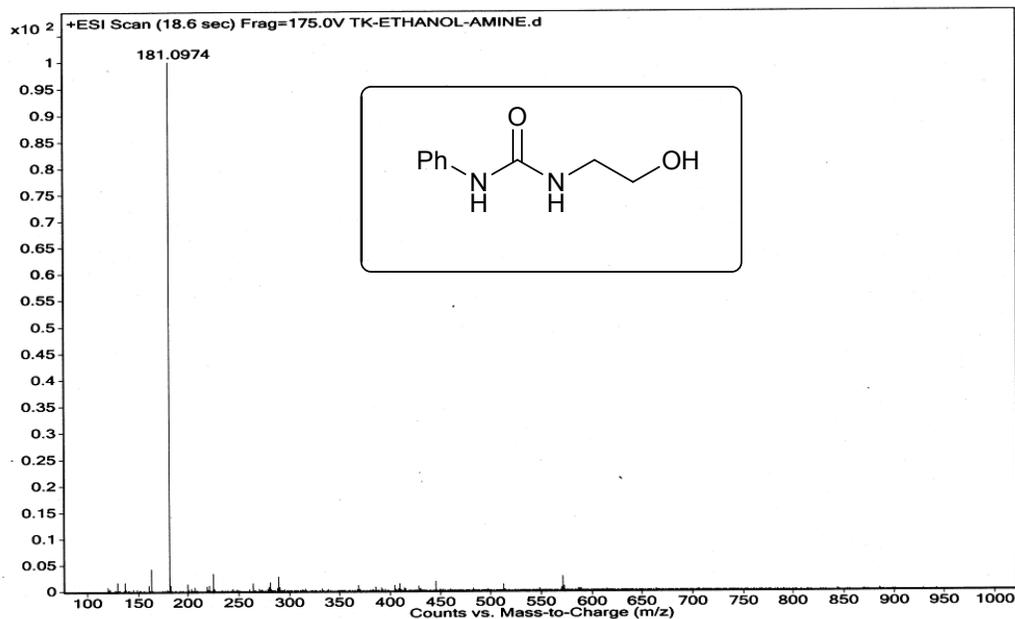


Figure S9. HRMS spectra of the product of entry 3 in table 2

```

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expi s2pul
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solvent CDCl3 gain not used
file /export/home/- spin not used
mercury/BTK-PTHETH- hst 0.000
NM2 p99 15.188
ACQUISITION alfa 28.000
sw 6380.0 FLAGS
at 1.990 i1 n
np 25520 in n
fb not used dp y
bs 4 hs nn
dl 1.000 PROCESSING 0.10
nt 32 lb 65536
ct 32 fn
TRANSMITTER H1 sp DISPLAY
tn 399.853 wp -200.0
tor 382.0 rfl 4802.0
tpwr 59 rfp 3897.0
pv 7.550 rp 2884.0
DECOUPLER lp -77.0
dn C13 PLOT
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da nnn sc 0
dmm c vs 48
dpar 44 tn 17
daf 17100 nm cdc ph
  
```

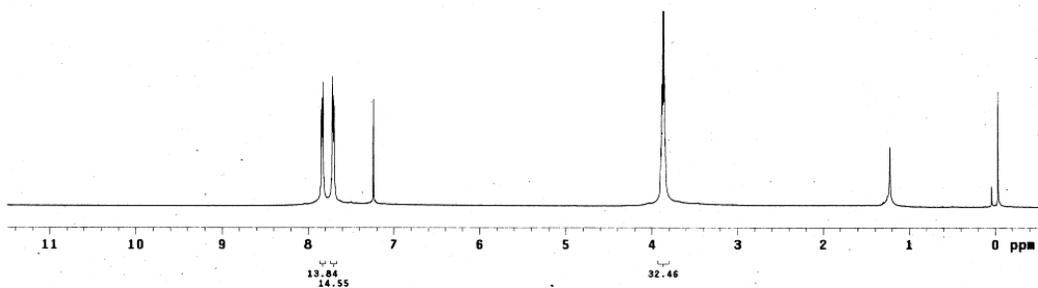
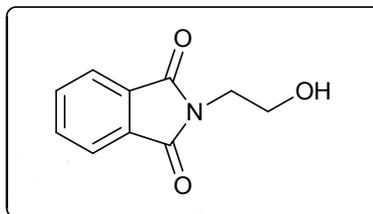


Figure S10. ¹H NMR spectra of the product of entry 4 in table 2

```

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SAMPLE          SPECIAL
date May 28 2013 temp not used
solvent CDCl3 gain not used
file /export/home/~ rpin not used
mercury/BTK-PHTHAETHAN-13
HMMH2-C13 pvt# 8.488
ACQUISITION    a1fa 28.000
sv 25125.8      FLAOS
ac 1.199        n
ap 84270        fn
fb 13890        dp
bs 19          mh
dl 1.000        PROCESSING
nt 18000        lb 2.00
ct 1720         fn 65536
TRANSMITTER C13 ep DISPLAY
tn          C13 ep -452.9
sfrq       100.554 wp 21262.5
tof        1536.3 rfl 3272.9
tpwr       61 rfp 7764.9
pv         4.799 rp -56.4
pc         1720 ip -281.5
dn DECOUPLER H1 PL
do         9 wc 258
dor        yyy ec
dm         42 th 8
dpr        8500 ne no ph 3
dft

```

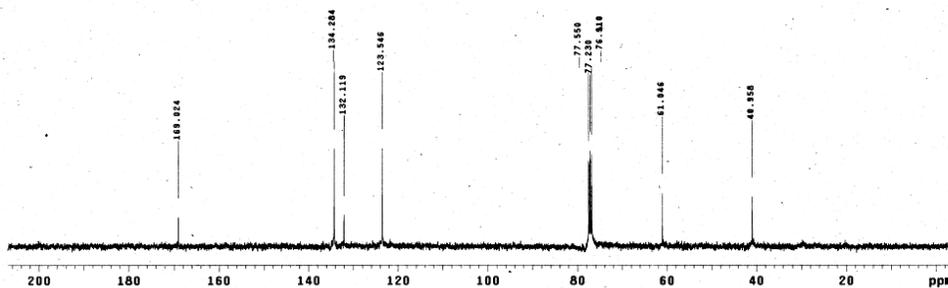
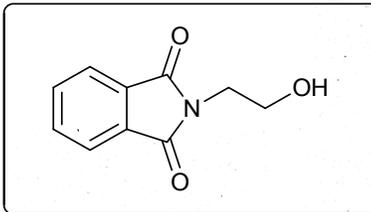


Figure S11. ^{13}C NMR spectra of the product of entry 4 in table 2

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Inj Vol	-10	InjPosition		SampleType	Sample	Acquired Time		5/14/2013 3:48:29 PM
Data Filename	TK-PHTA-etho.d	ACQ Method		Comment				

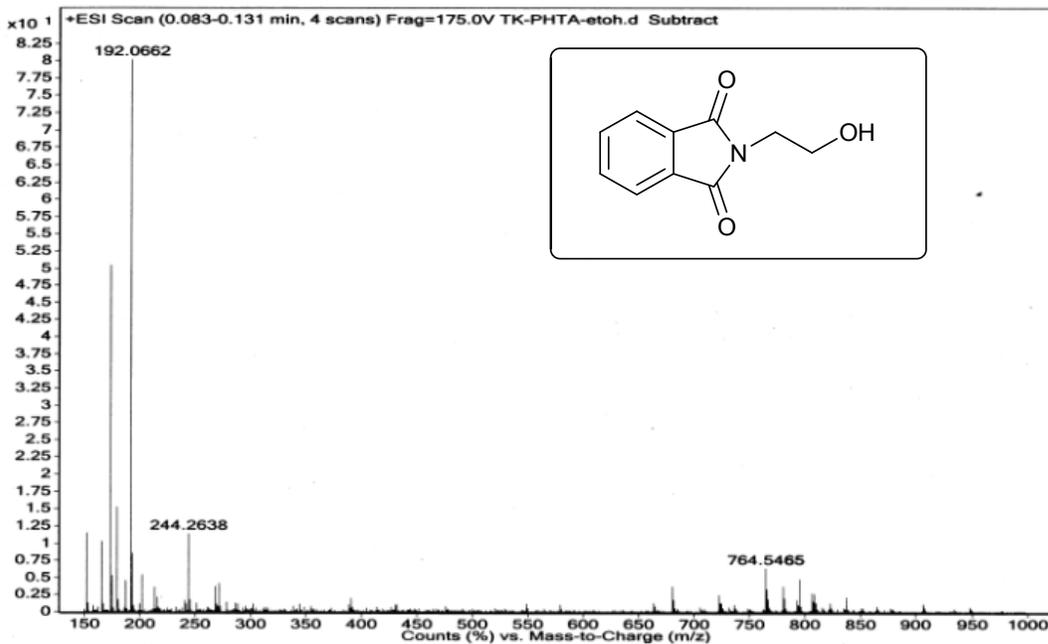


Figure S12. HRMS spectra of the product of entry 4 in table 2

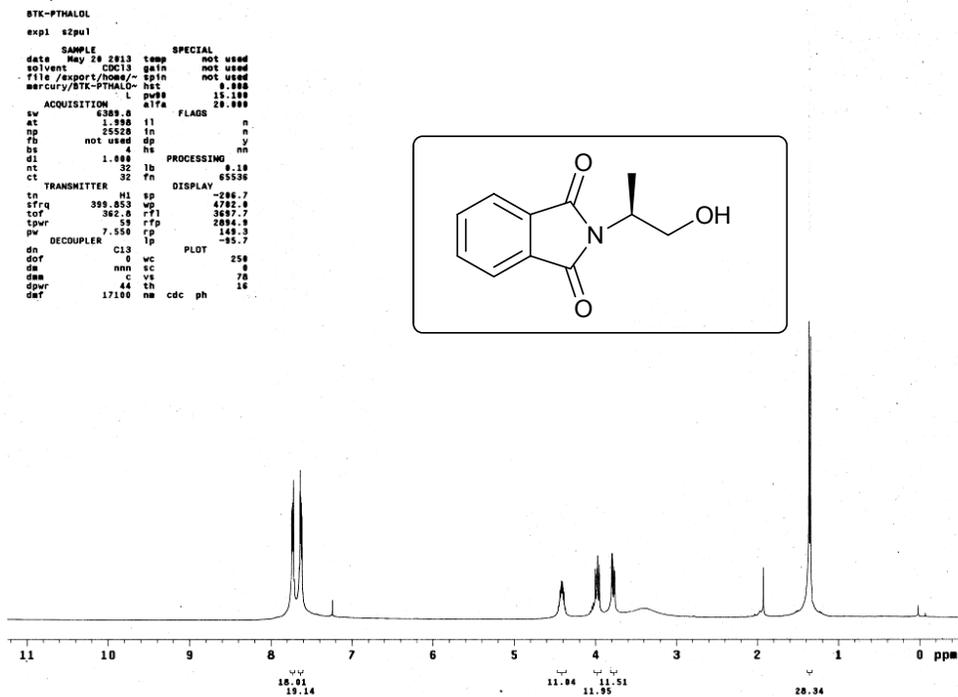


Figure S13. ^1H NMR spectra of the product of entry 5 in table 2

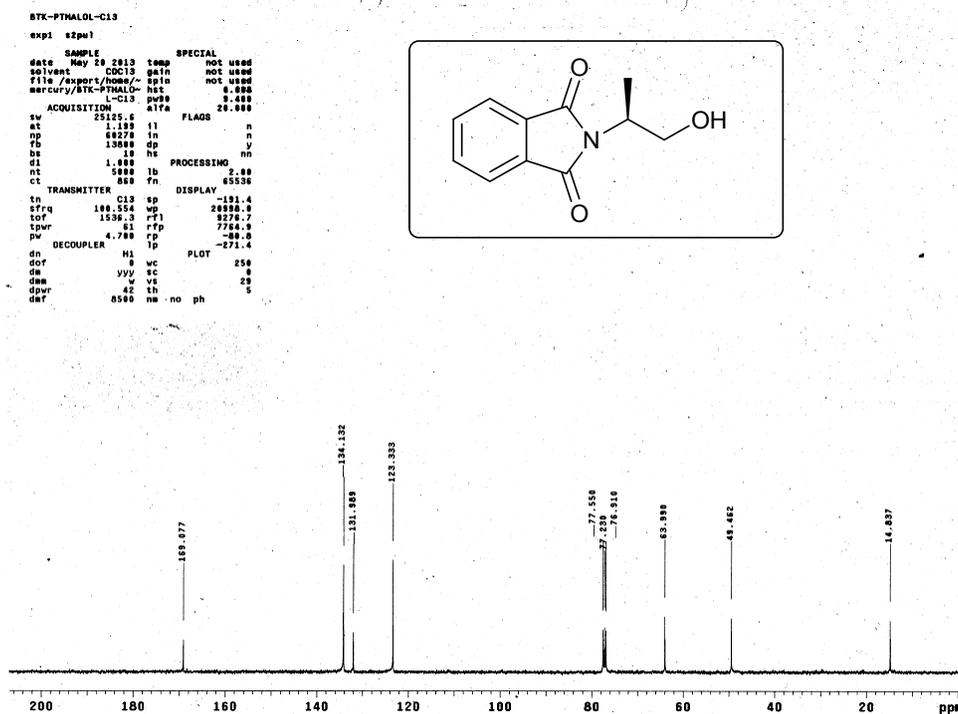


Figure S14. ^{13}C NMR spectra of the product of entry 5 in table 2

Sample Name TK-~~PTA-AL-OL~~ Position -1 Instrument Name Instrument 1 User Name
 Inj Vol -10 InjPosition Sample IRM Calibration Status Success
 Data Filename TK-~~PTA-AL-OL~~ ACQ Method Comment Sample Acquired Time 12/13/2013 11:54:54 AM

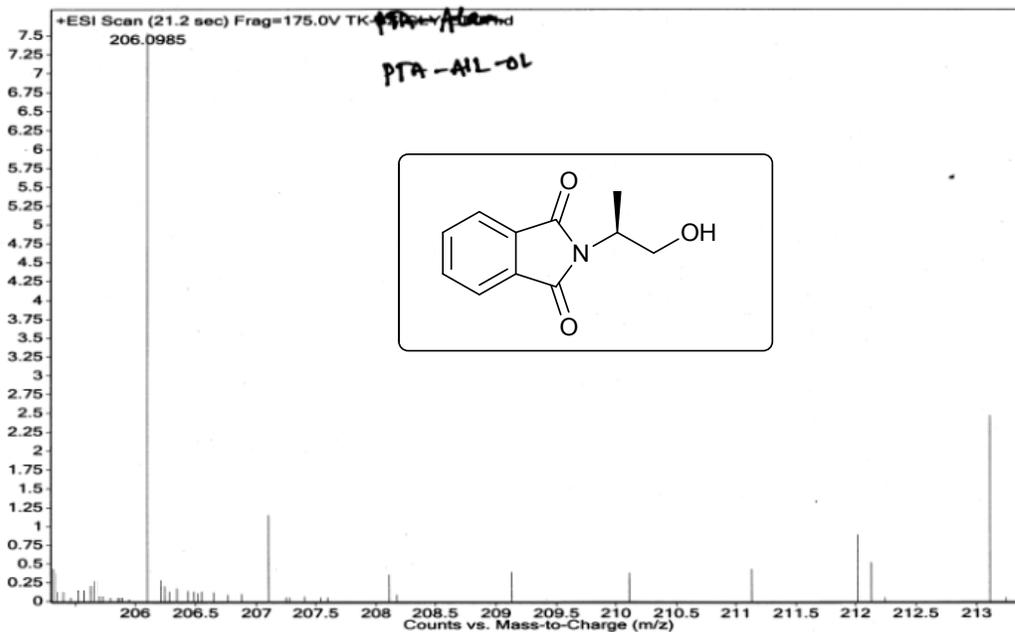


Figure S15. HRMS spectra of the product of entry 5 in table 2

TK-BN-gly-EtOL-1H

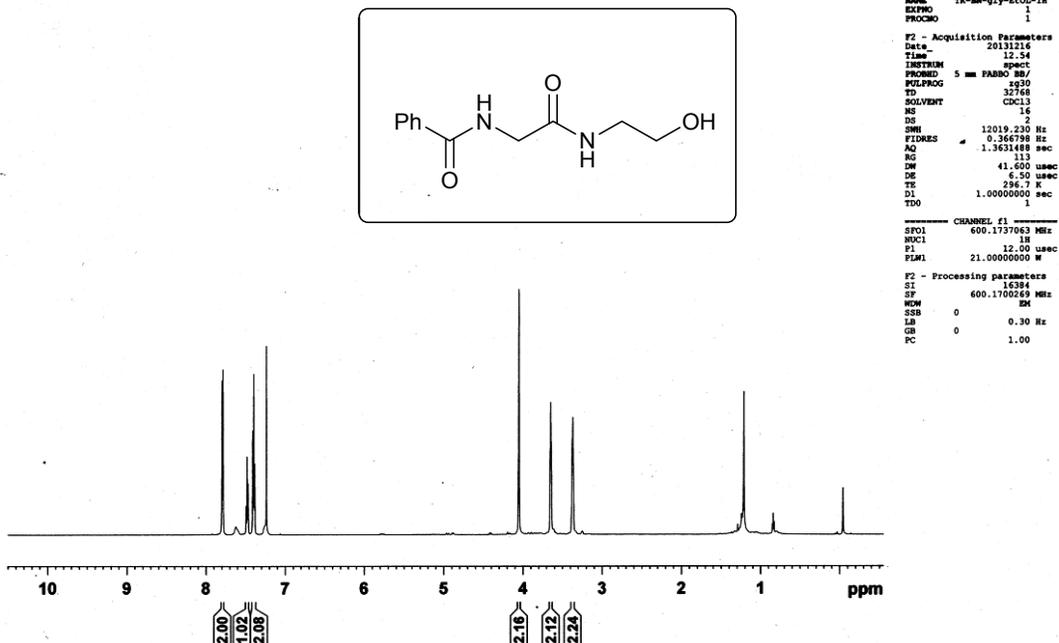


Figure S16. ¹H NMR spectra of the product of entry 6 in table 2

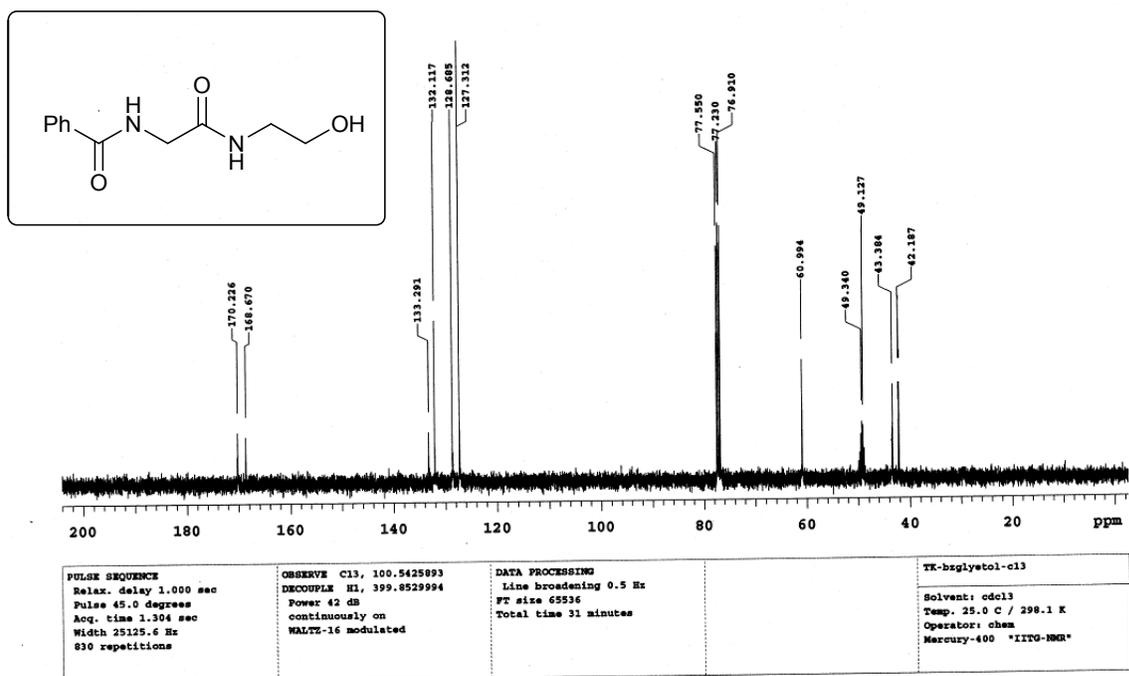


Figure S17. ¹³C NMR spectra of the product of entry 6 in table 2

Sample Name	Unavailable	Position	Unavailable	Instrument Name	Unavailable	User Name	Unavailable
Inj Vol	Unavailable	InjPosition	Unavailable	SampleType	Unavailable	IRM Calibration Status	Success
Data Filename	TK-BZ-GLY-ET01.d	ACQ Method		Comment	Sample information is unavailable	Acquired Time	Unavailable

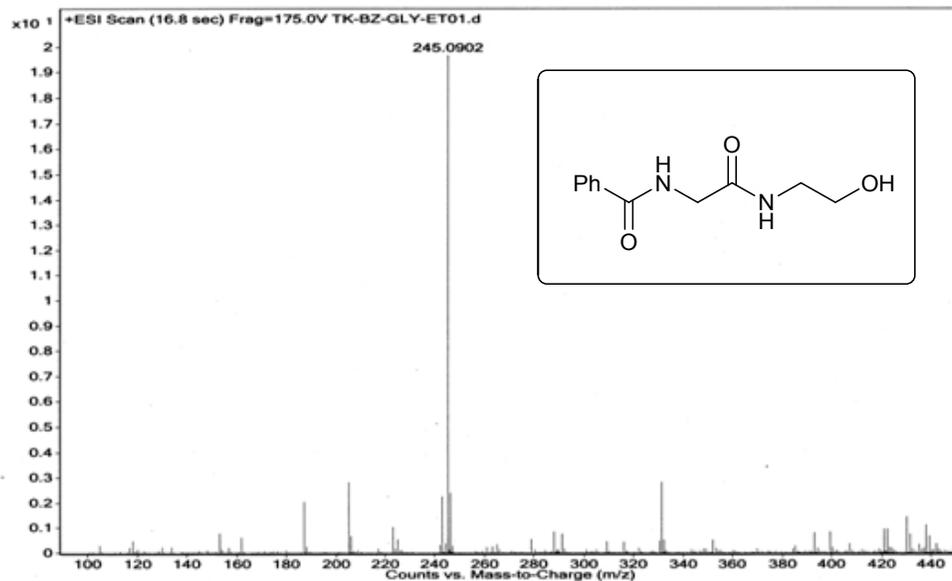


Figure S18. HRMS spectra of the product of entry 6 in table 2

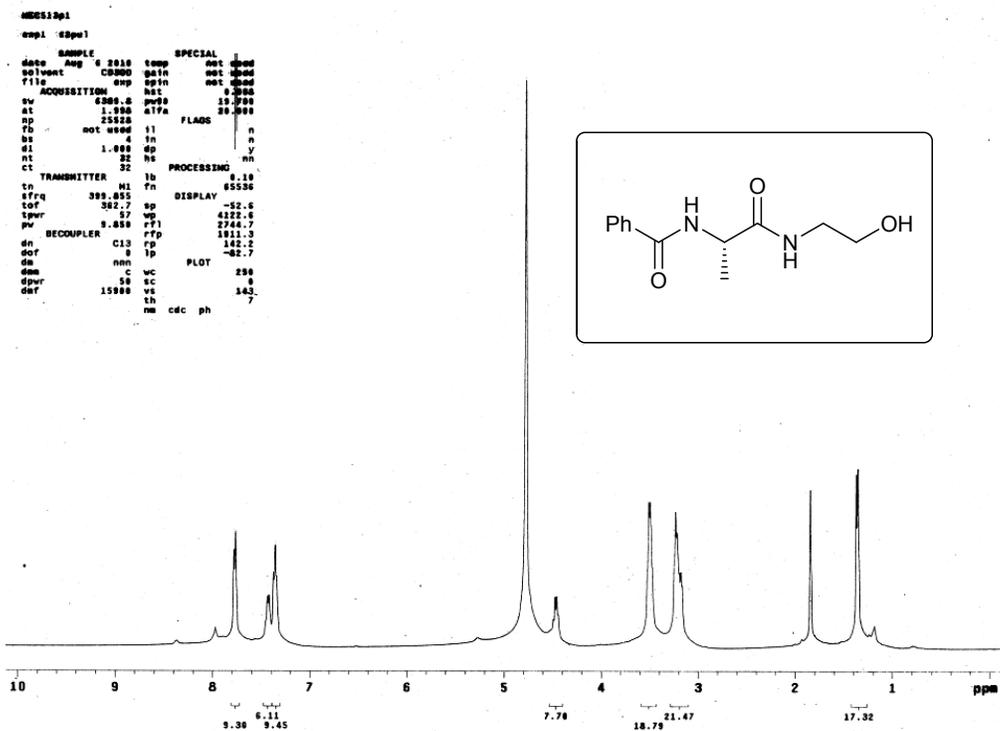


Figure S19. ¹H NMR spectra of the product of entry 7 in table 2

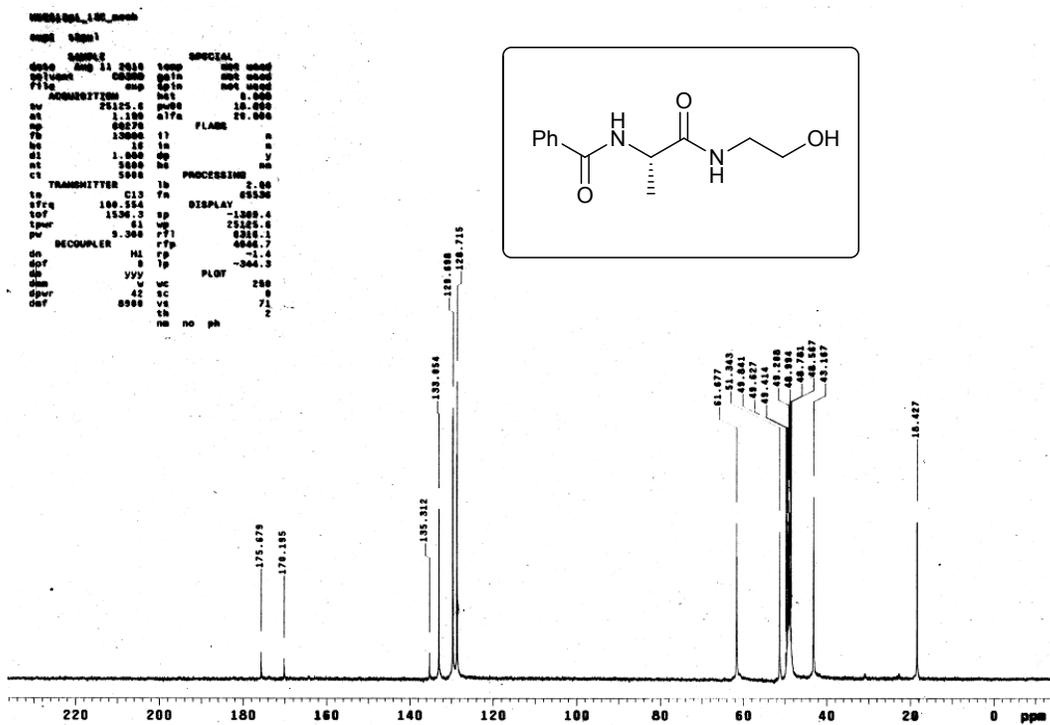


Figure S20. ¹³C NMR spectra of the product of entry 7 in table 2

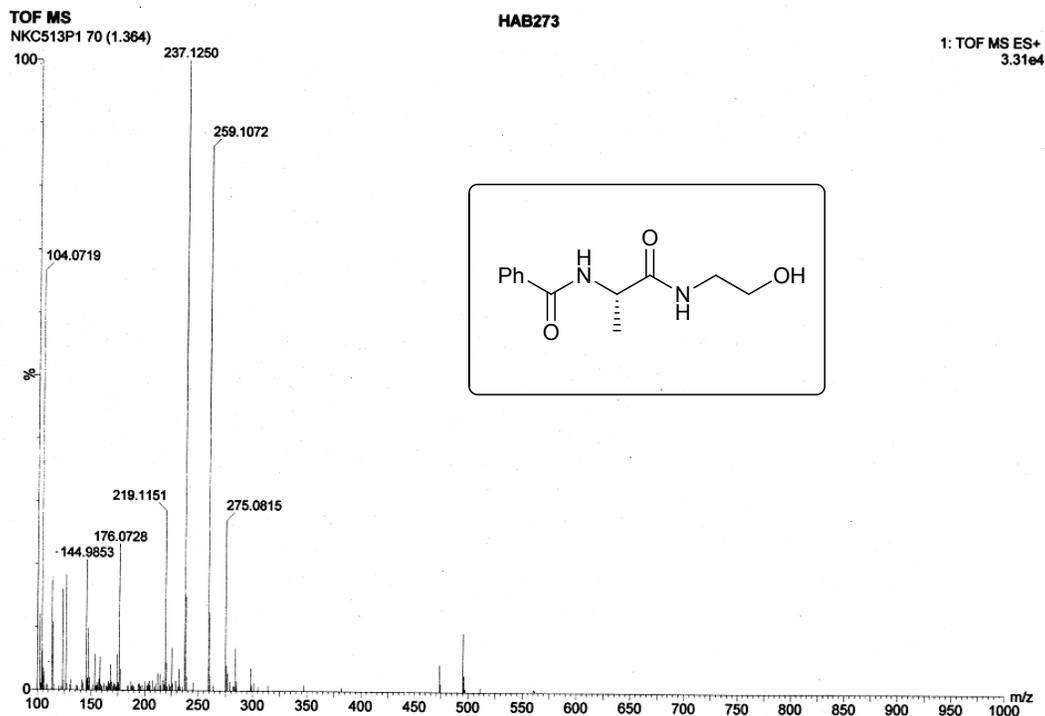


Figure S21. HRMS spectra of the product of entry 7 in table 2

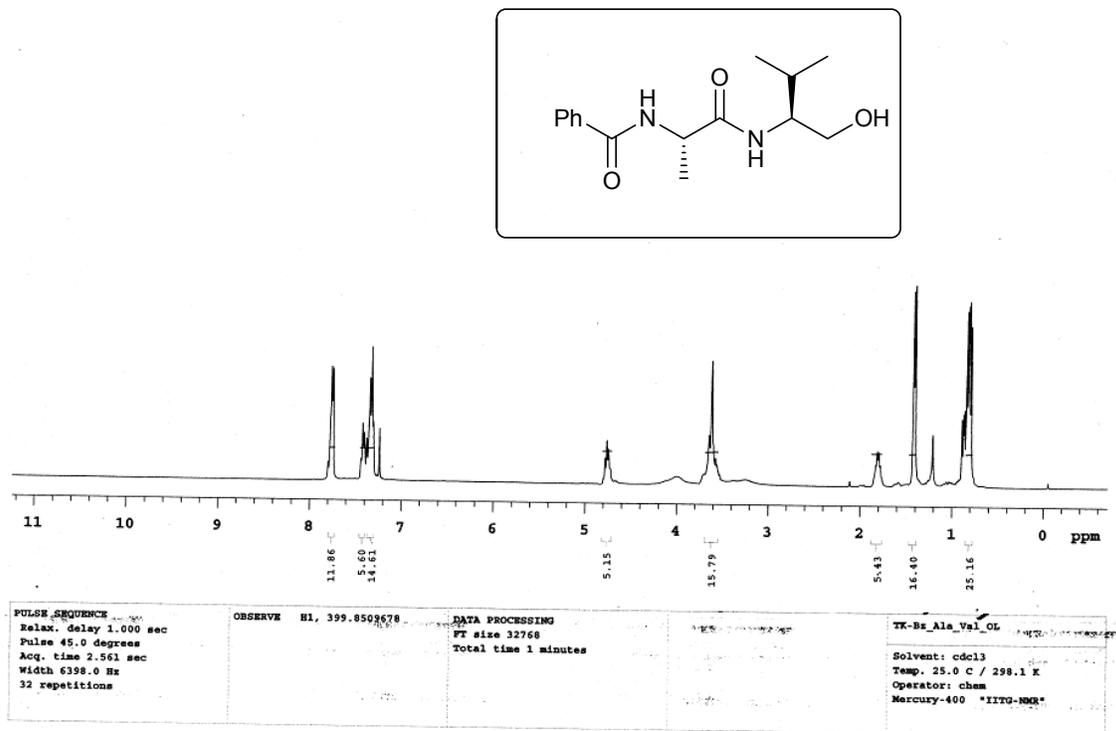
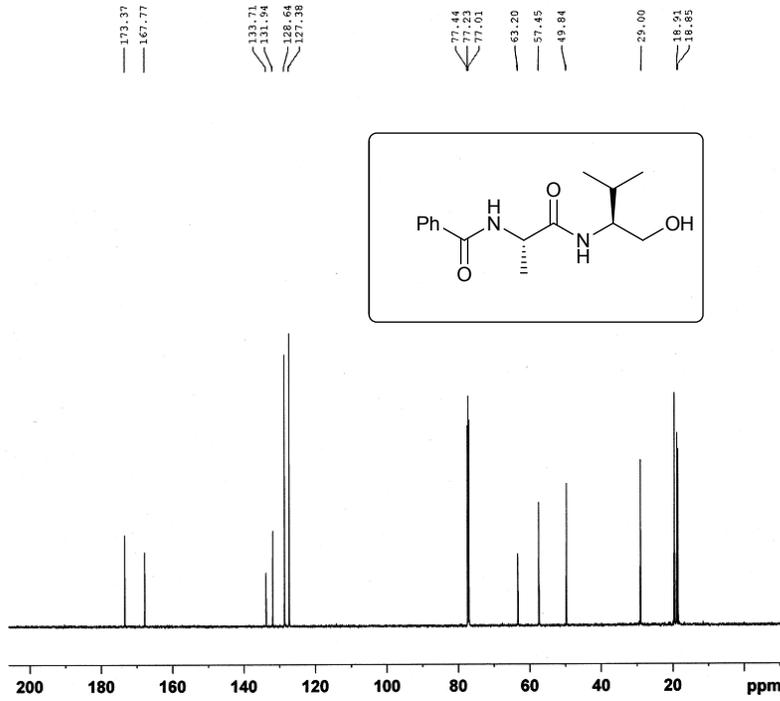


Figure S22. ¹H NMR spectra of the product of entry 8 in table 2

TK-BZ-ALA-VAL-OL-13C



Current Data Parameters
 NAME TK-BZ-ALA-VAL-OL-13C
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20131219
 Time 10.59
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 32768
 SOLVENT CDC13
 NS 280
 DS 2
 SWH 36057.691 Hz
 FIDRES 1.100393 Hz
 AQ 0.4543829 sec
 RG 65.24
 DE 13.867 usec
 DW 6.50 usec
 TE 297.8 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TDO 1

===== CHANNEL f1 =====
 SF01 150.9279571 MHz
 NUC1 13C
 P1 10.50 usec
 PLW1 95.0000000 W

===== CHANNEL f2 =====
 SF02 600.1724007 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCD2 70.00 usec
 PLW2 21.0000000 W
 PLW12 0.61714000 W
 PLW13 0.30239999 W

F2 - Processing parameters
 SI 16384
 SF 150.9128484 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure S23. ¹³C NMR spectra of the product of entry 8 in table 2

Sample Name	BZ-ALA-VAL-OL	Position	-1	Instrument Name	Instrument 1	User Name	
Inj Vol	-10	InjPosition		SampleType	Sample	IRM Calibration Status	Success
Data Filename	BZ-ALA-VAL-OL.d	ACQ Method		Comment		Acquired Time	12/23/2013 11:54:18 A

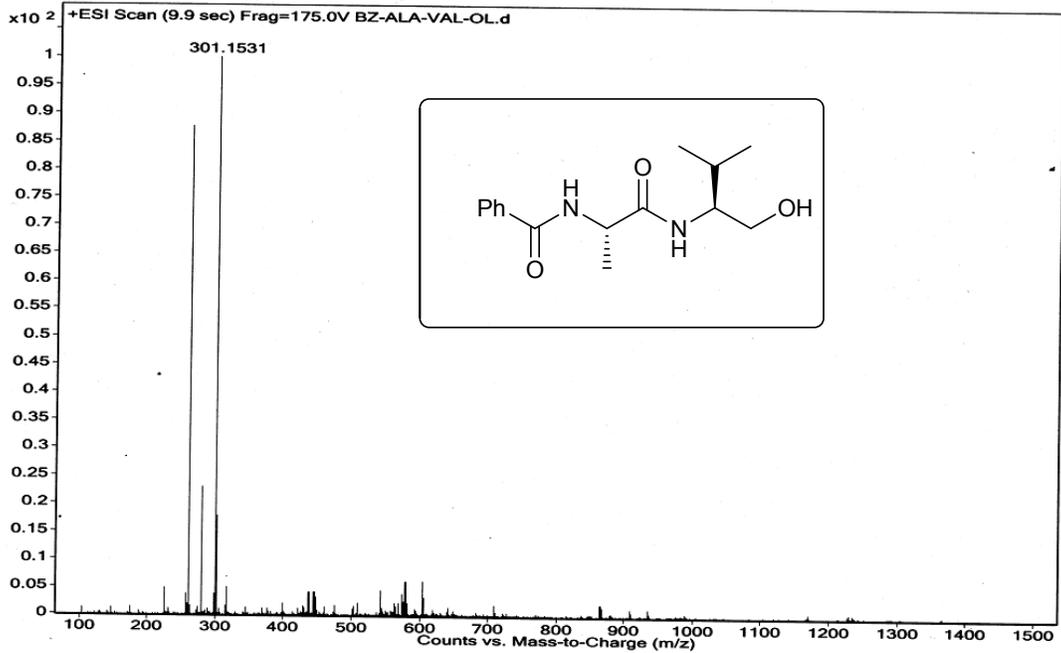


Figure S24. HRMS spectra of the product of entry 8 in table 2

TK_BzCOPGOL_1_13C

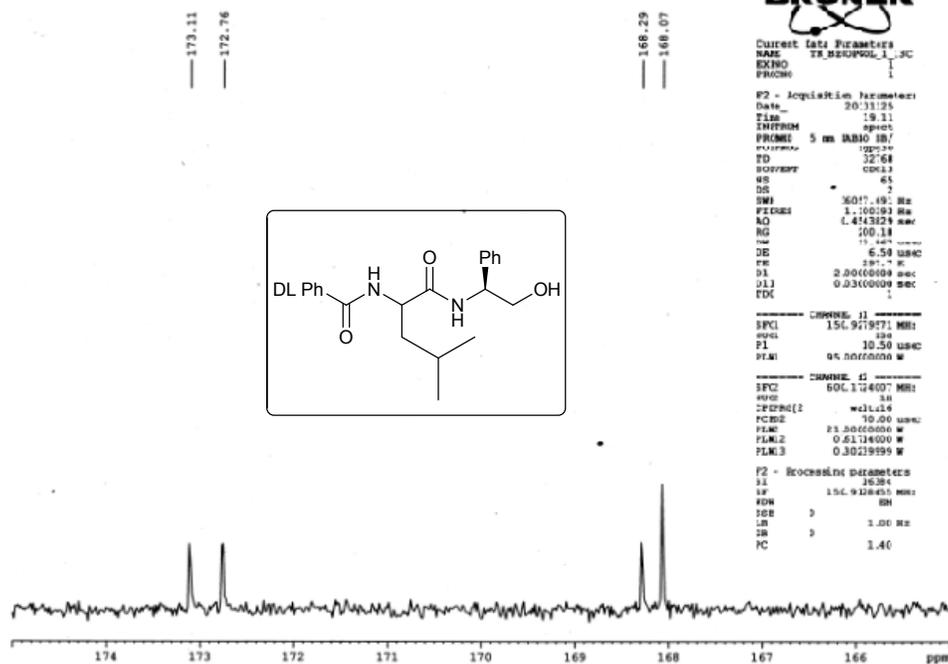


Figure S27. ¹³C NMR expanded spectra of the product of entry 9 in table 2

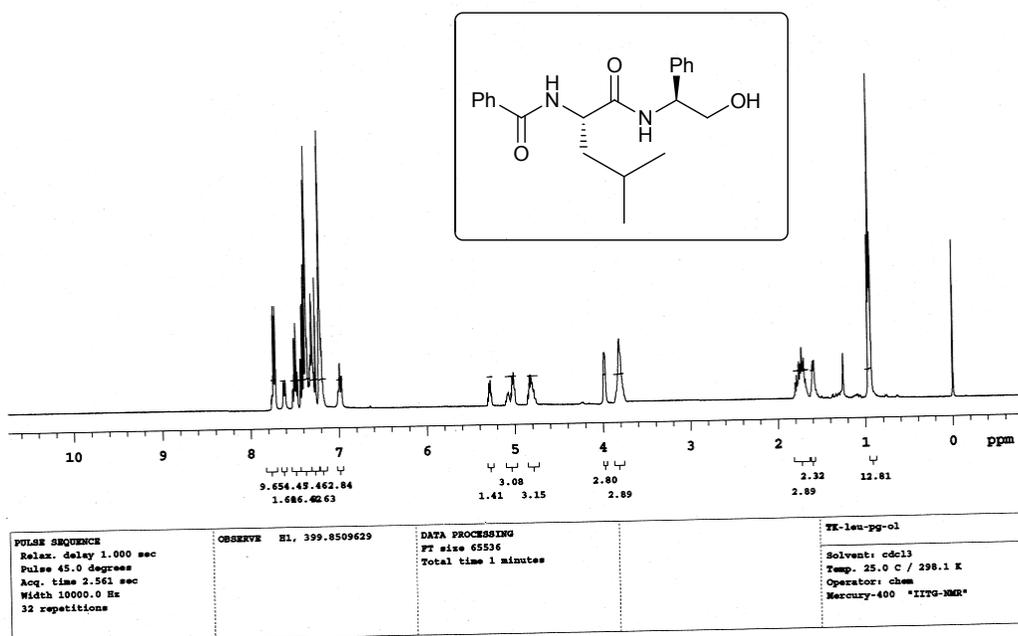


Figure S28. ¹H NMR spectra of the product of entry 10 in table 2

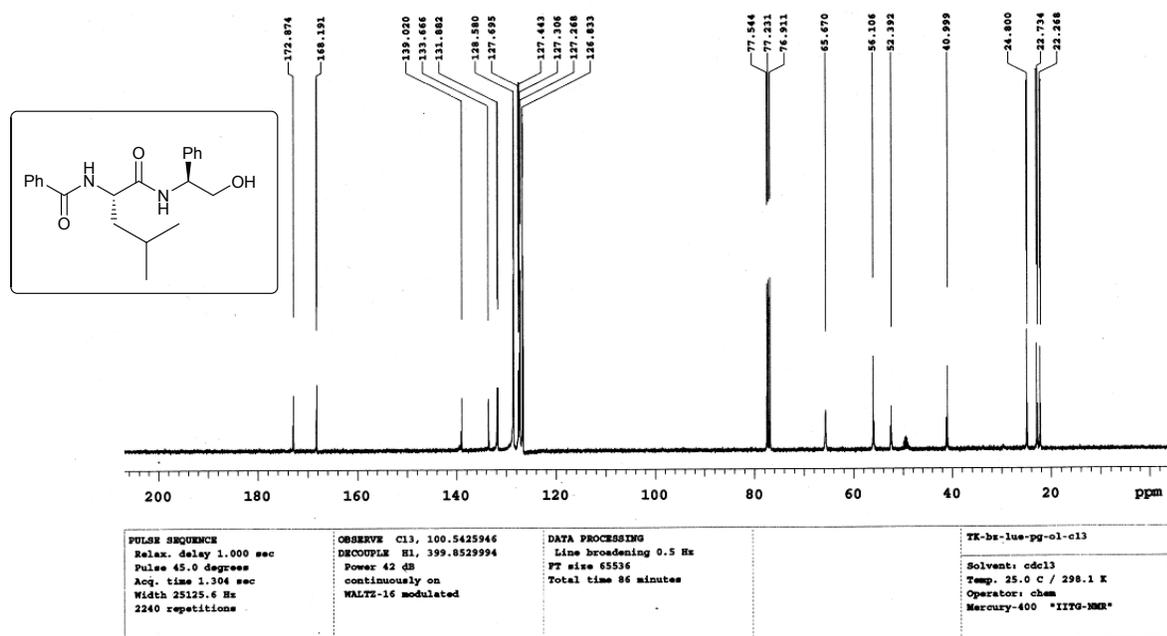


Figure S29. ¹³C NMR spectra of the product of entry 10 in table 2

Sample Name BZ-LU-PG-OL	Position -1	Instrument Name Instrument 1	User Name
Inj Vol -10	InjPosition	SampleType Sample	IRM Calibration Status Success
Data Filename BZ-LU-PG-OL.d	ACQ Method	Comment	Acquired Time 12/23/2013 11:56:03 AM

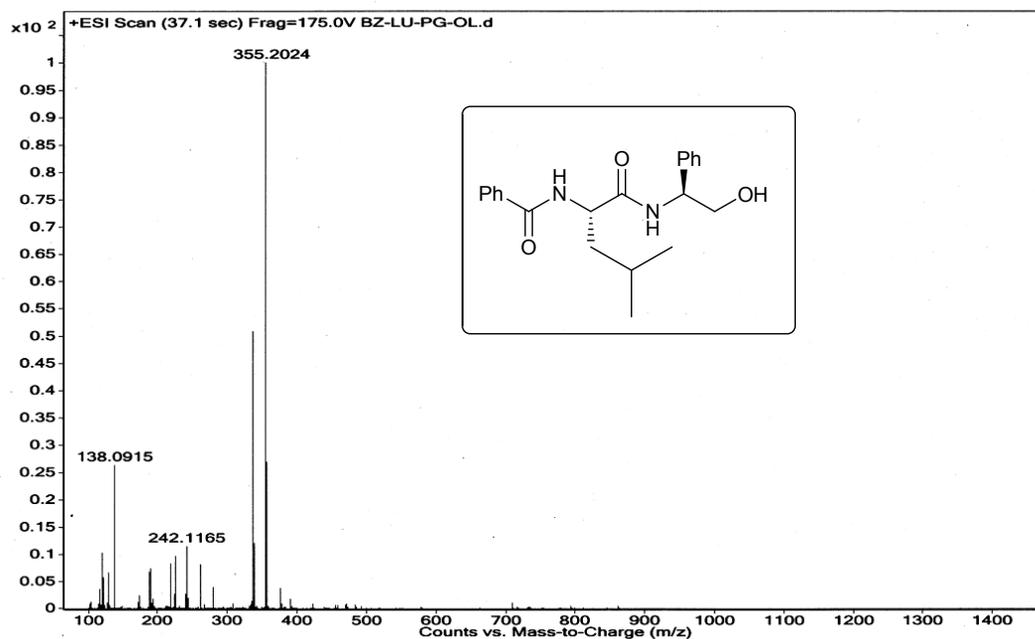


Figure S30. HRMS spectra of the product of entry 10 in table 2

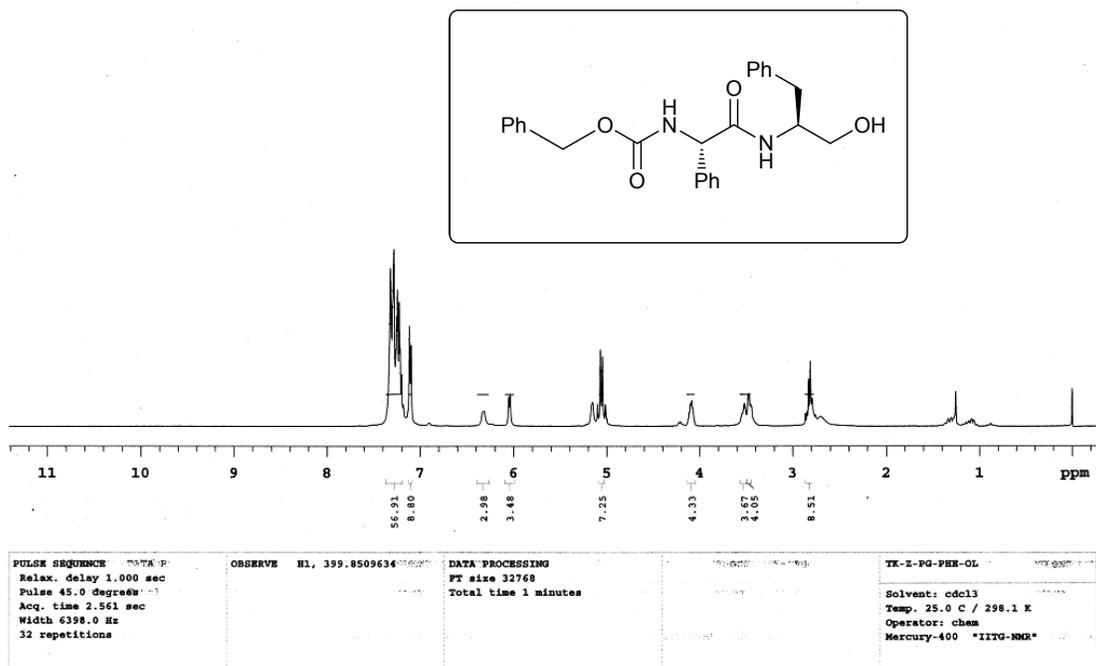


Figure S31. ¹H NMR spectra of the product of entry 11 in table 2

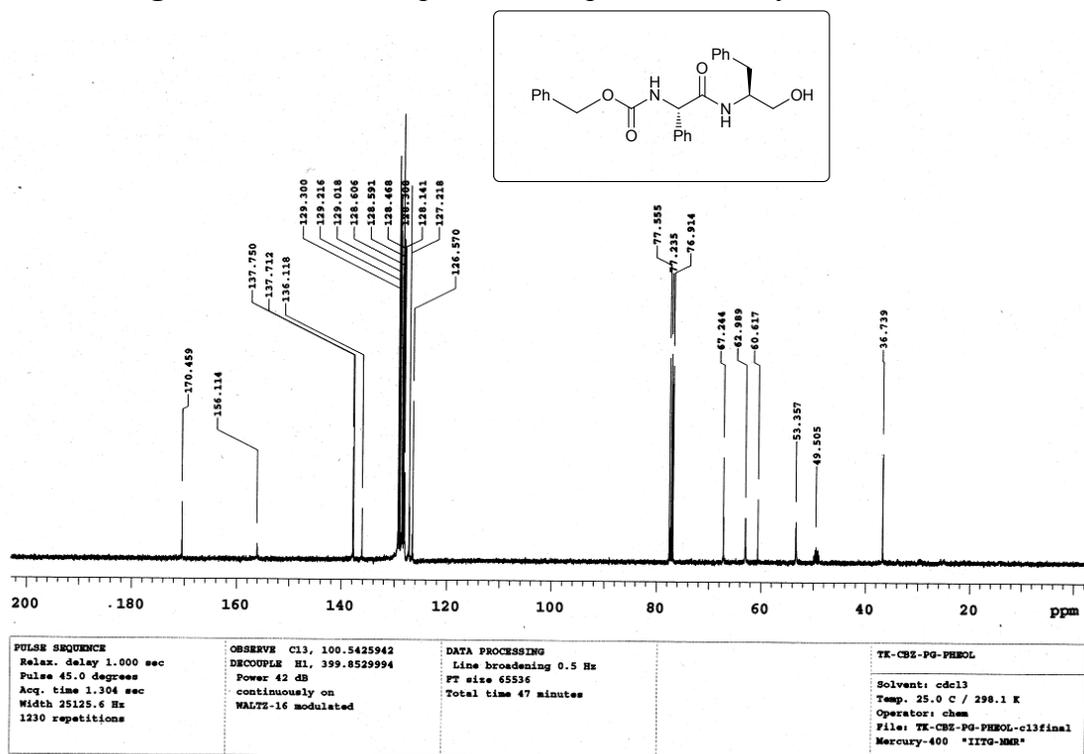


Figure S32. ¹³C NMR spectra of the product of entry 11 in table 2

Sample Name	Z-PHE-PG-OL	Position	-1	Instrument Name	Instrument 1	User Name	
Inj Vol	-10	InjPosition		SampleType	Sample	IRM Calibration Status	Success
Data Filename	Z-PHE-PG-OL.d	ACQ Method		Comment		Acquired Time	12/23/2013 12:01:11 PM

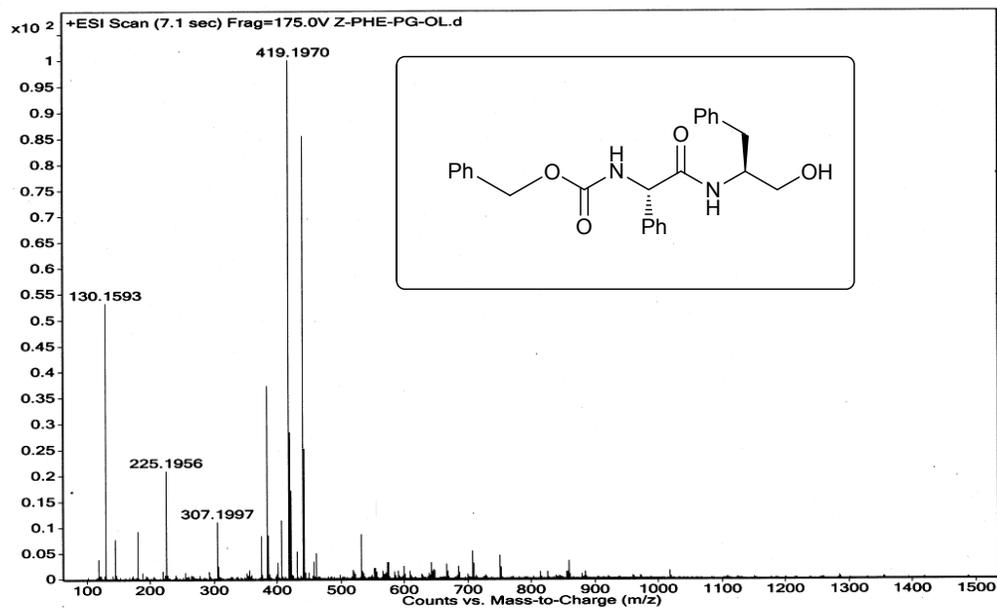


Figure S33. HRMS spectra of the product of entry 11 in table 2

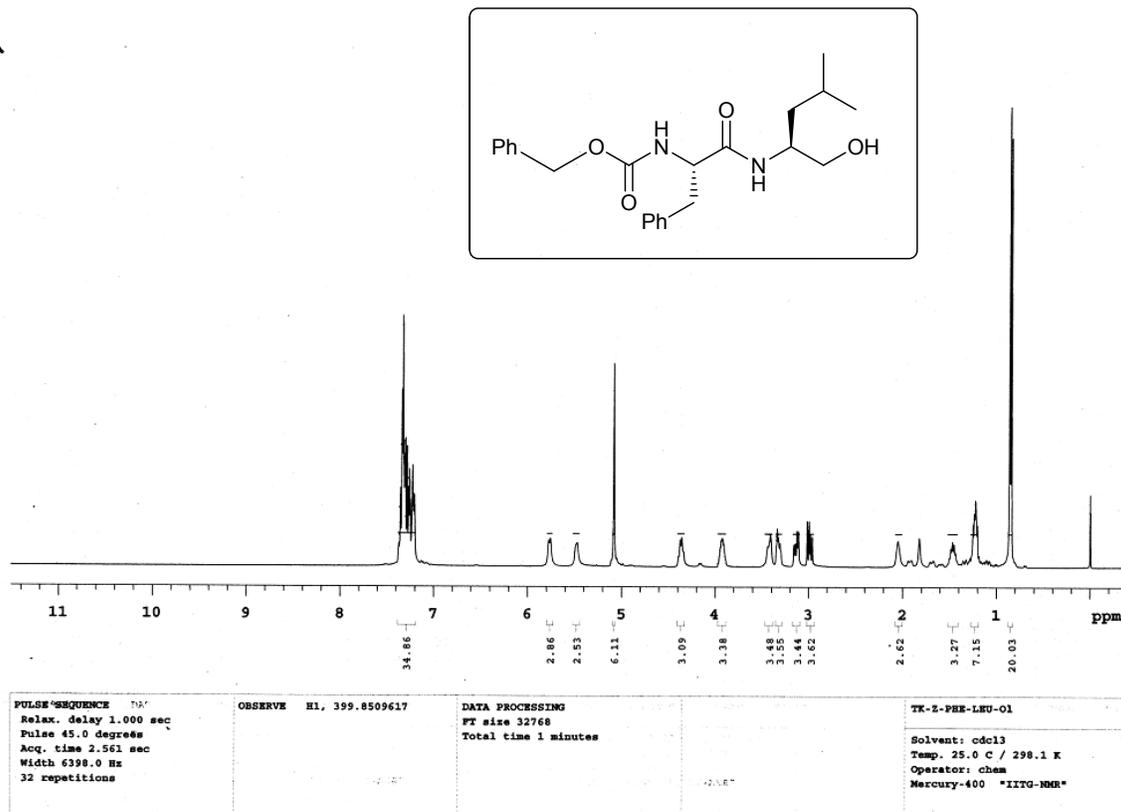


Figure S34. ¹H NMR spectra of the product of entry 12 in table 2

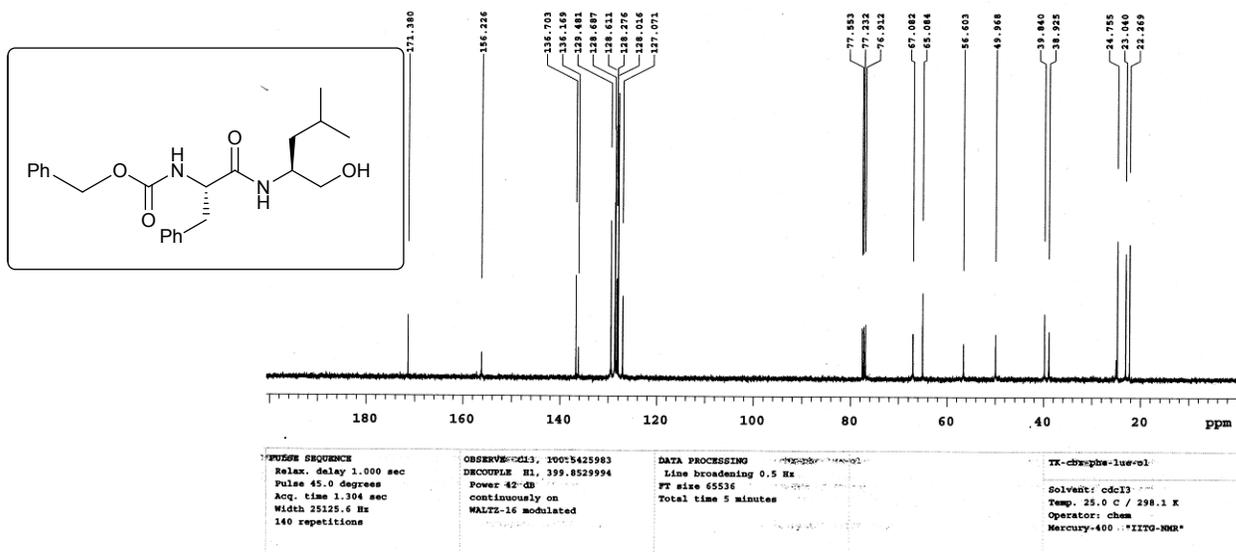


Figure S35. ^{13}C NMR spectra of the product of entry 12 in table 2

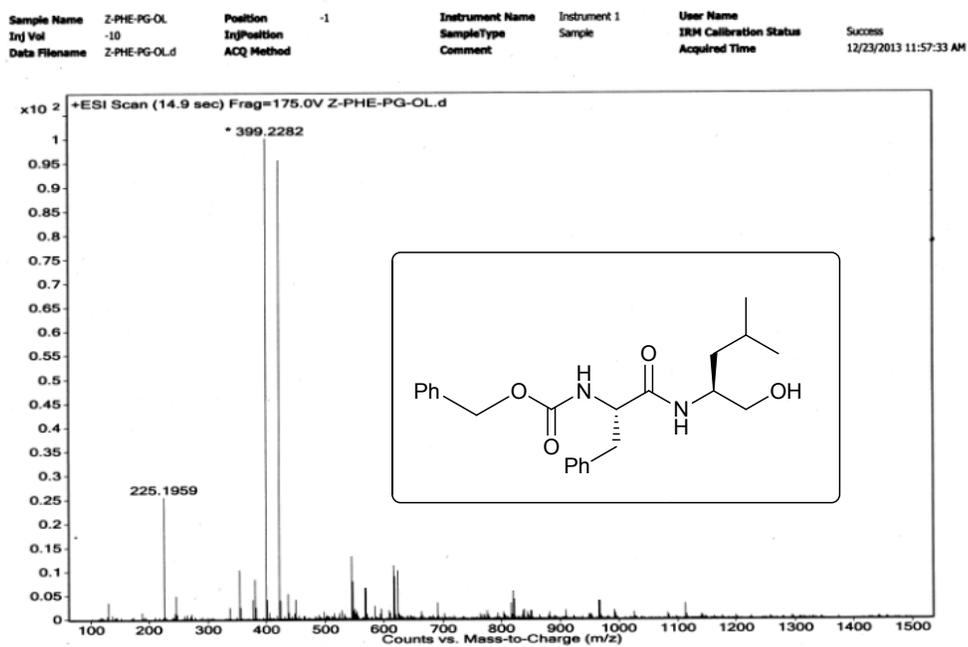


Figure S36. HRMS spectra of the product of entry 12 in table 2

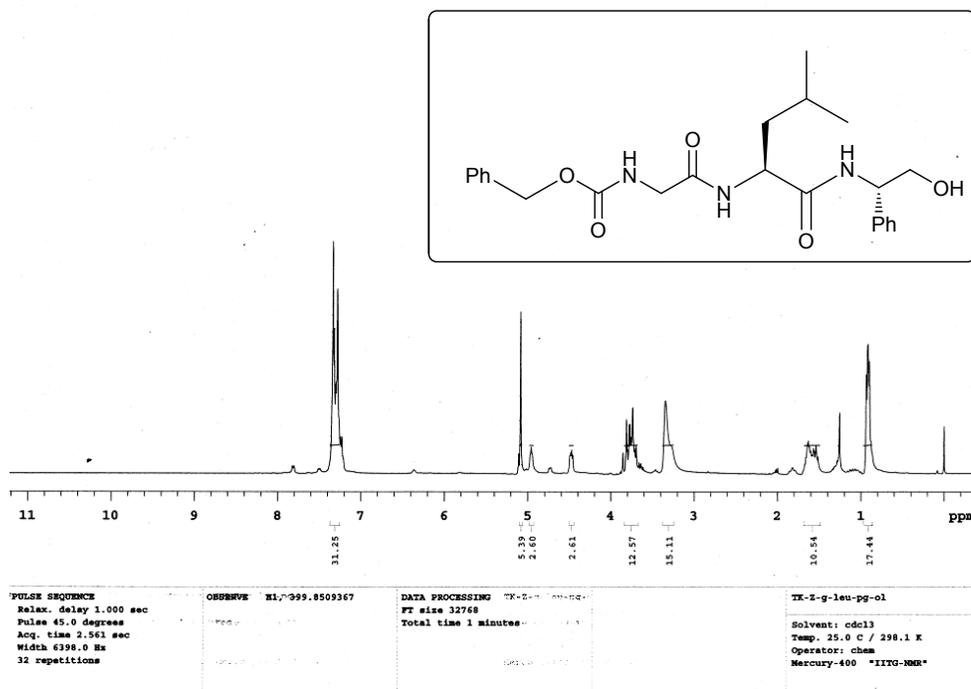


Figure S37. ¹H NMR spectra of the product of entry 13 in table 2

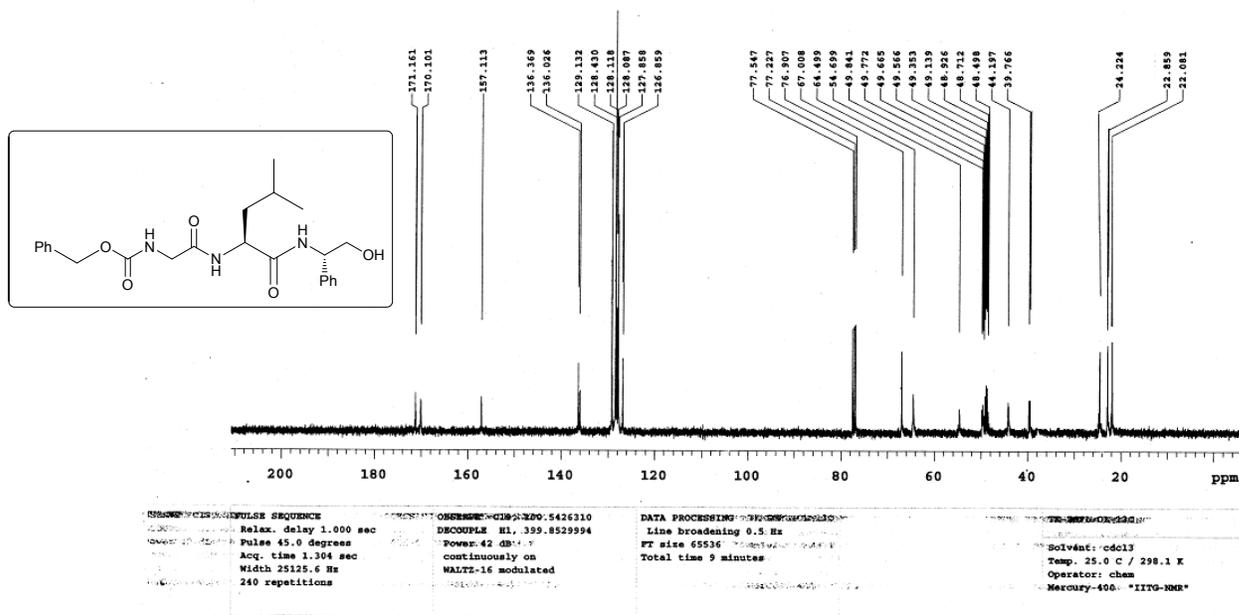


Figure S38. ¹³C NMR spectra of the product of entry 13 in table 2

Sample Name	Unavailable	Position	Unavailable	Instrument Name	Unavailable	User Name	Unavailable
Inj Vol	Unavailable	InjPosition	Unavailable	SampleType	Unavailable	IRM Calibration Status	Success
Data Filename	TK-GLPGOL-1.d	ACQ Method		Comment	Sample information is unavailable	Acquired Time	Unavailable

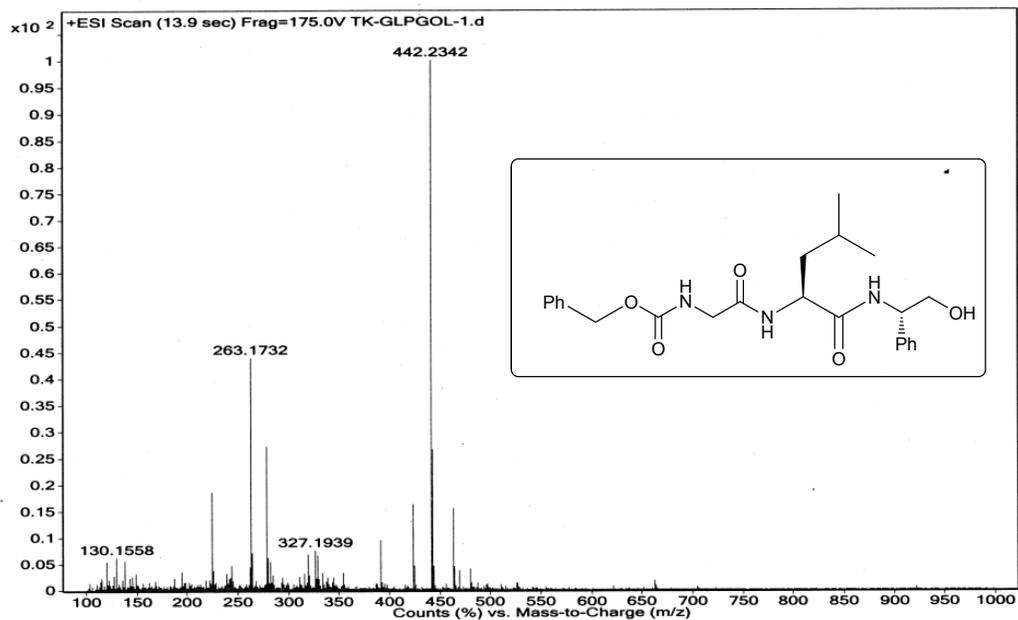


Figure S39. HRMS spectra of the product of entry 13 in table 2

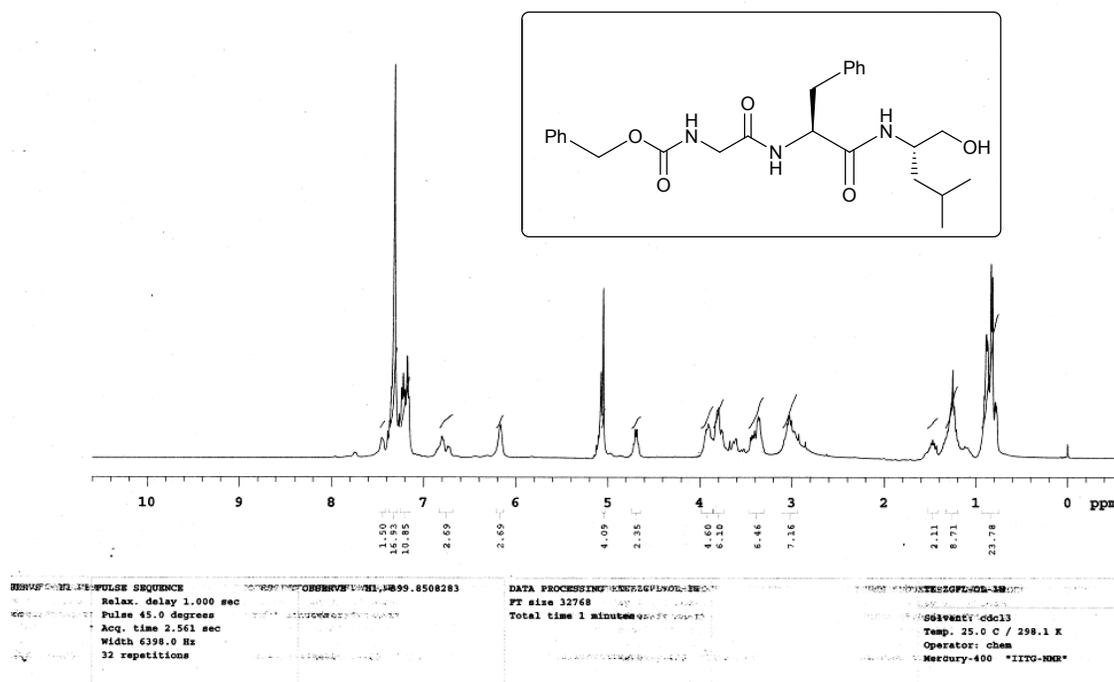


Figure S40. ¹H NMR spectra of the product of entry 14 in table 2

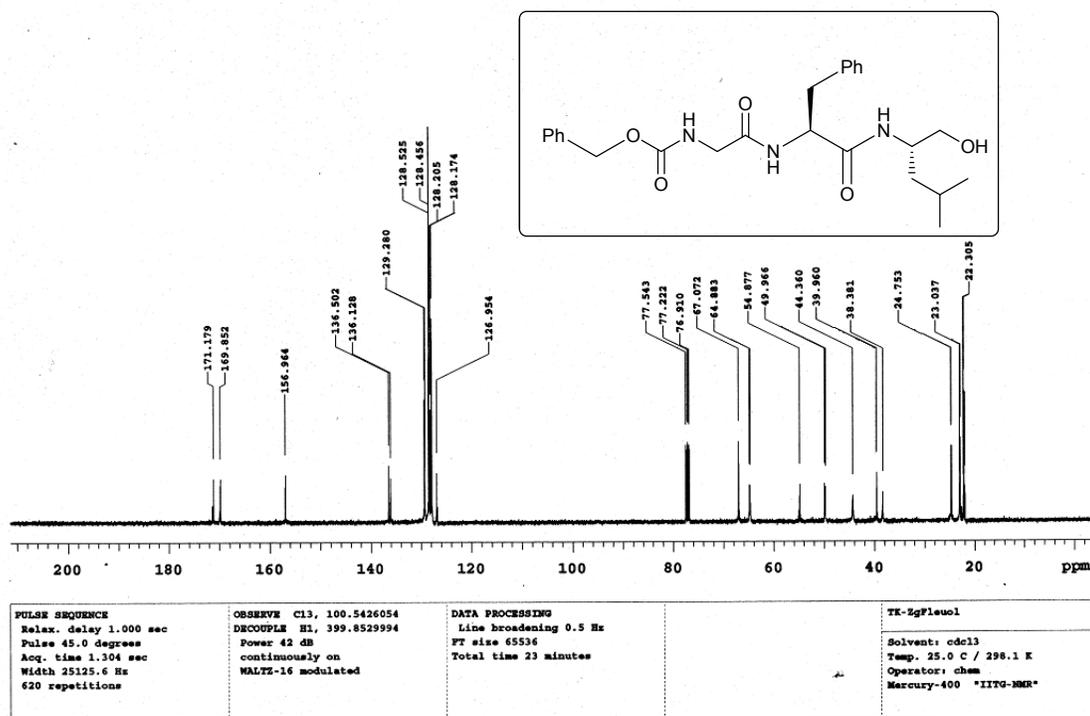


Figure S41. ¹³C NMR spectra of the product of entry 14 in table 2

Sample Name	Unavailable	Position	Unavailable	Instrument Name	Unavailable	User Name	Unavailable
Inj Vol	Unavailable	InjPosition	Unavailable	SampleType	Unavailable	IRM Calibration Status	Success
Data Filename	TK-ZGFLOL.d	ACQ Method		Comment	Sample information is unavailable	Acquired Time	Unavailable

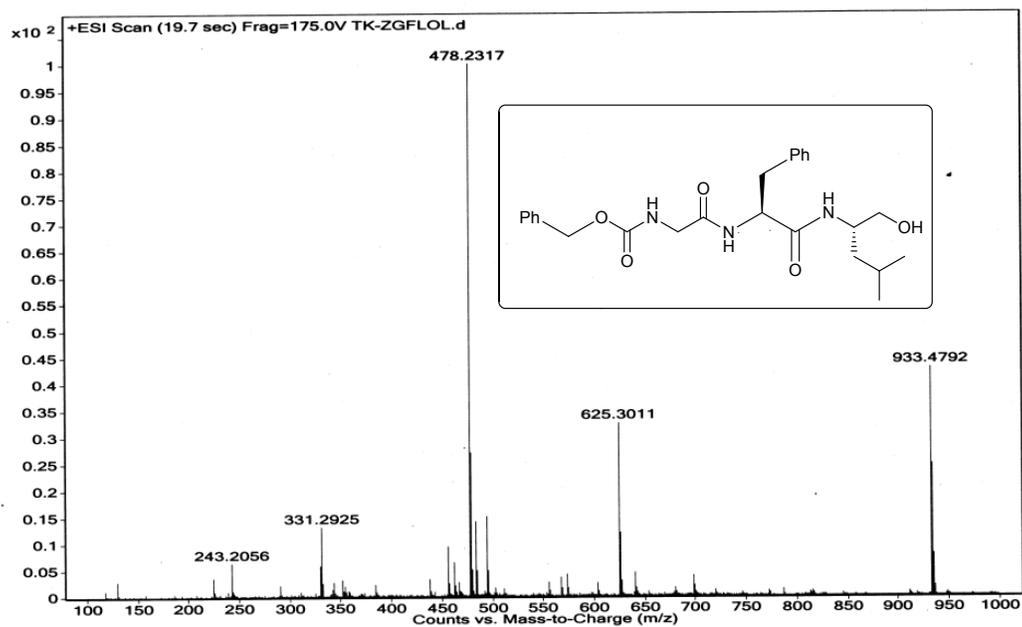


Figure S42. HRMS spectra of the product of entry 14 in table 2

Sample Name	ZGVFOL	Position	-1	Instrument Name	Instrument 1	User Name	
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Data Filename	ZGVFOL.d	ACQ Method		Comment		Acquired Time	12/23/2013 11:52:38 AM

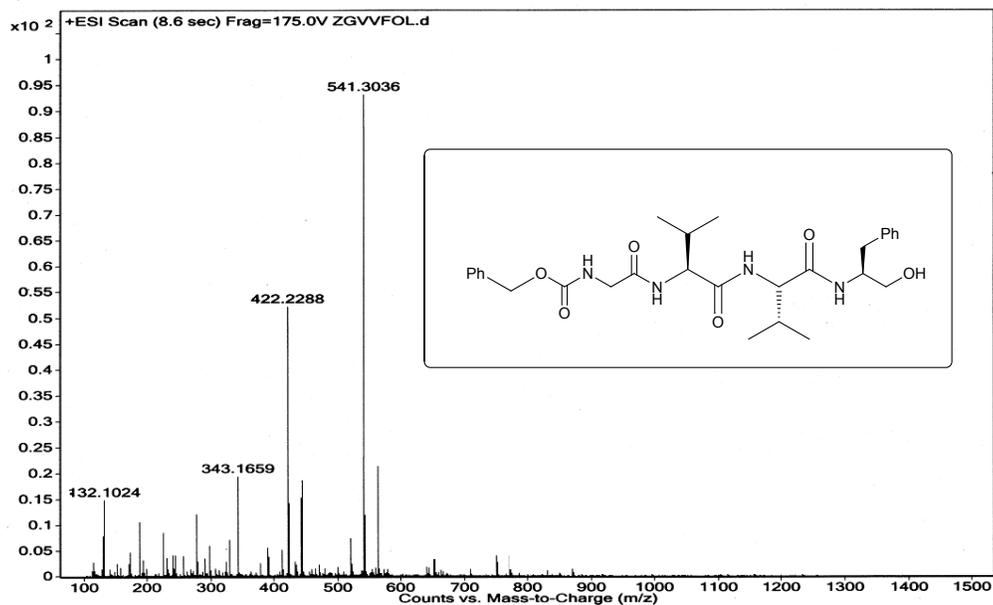


Figure S45. HRMS spectra of the product of entry 15 in table 2

2 HPLC data for racemization studies:

HPLC profiles of Peptide alcohols, reverse phase, run time 20 min (linear gradient of 0 to 100% CH₃CN in H₂O with 0.1% formic acid)

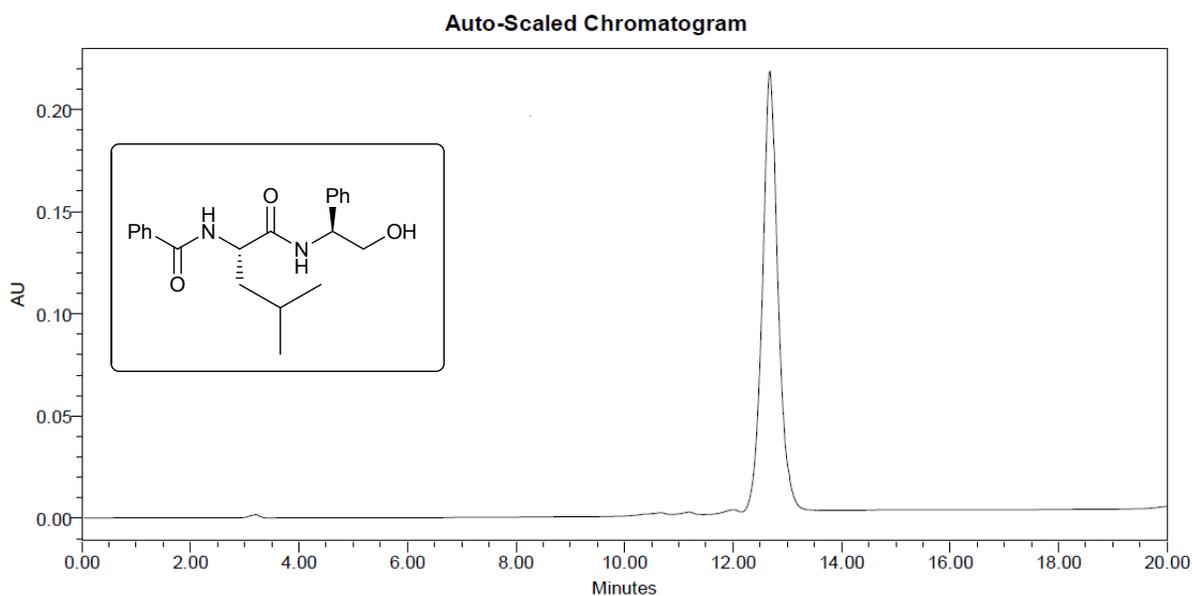
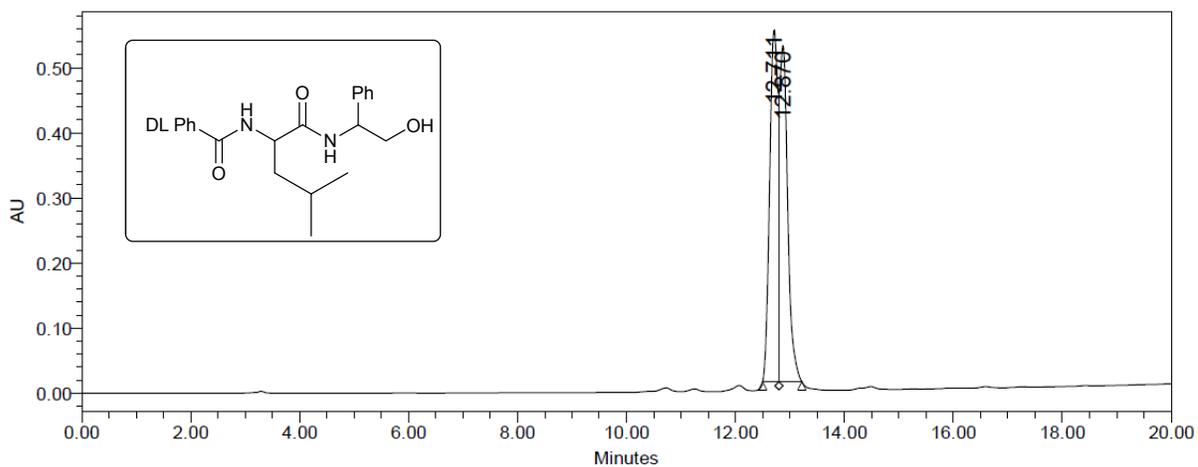


Figure S46 HPLC profile diagram of the product of entry 9 in table 2



	RT	Area	% Area	Height
1	12.711	5711231	51.75	541199
2	12.870	5323968	48.25	516790

Figure S47 HPLC profile diagram of the product of entry 10 in table 2

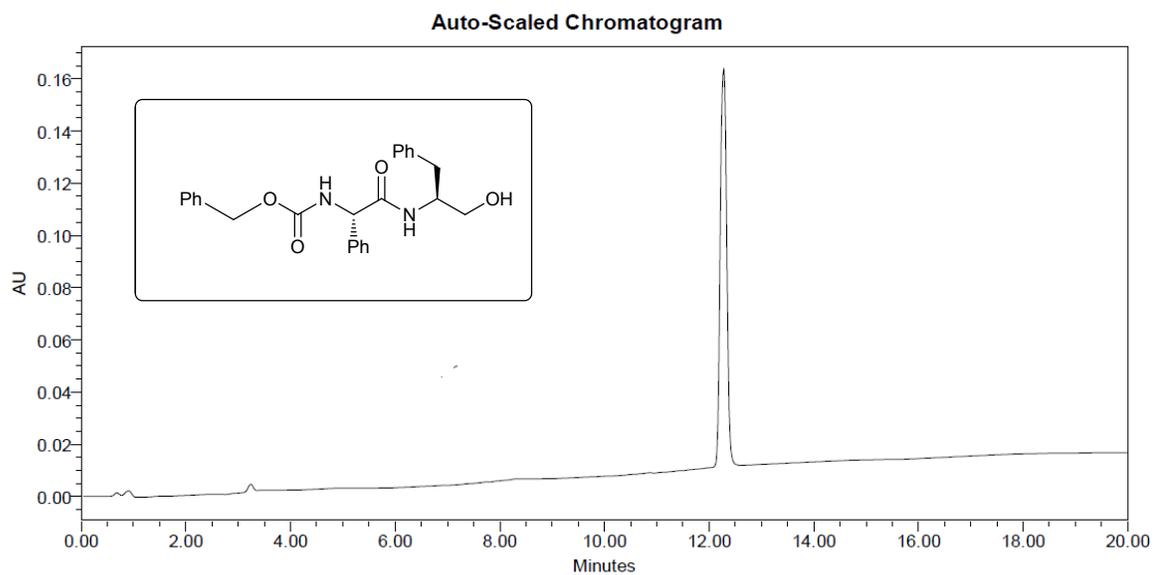


Figure S48 HPLC profile diagram of the product of entry 11 in table 2

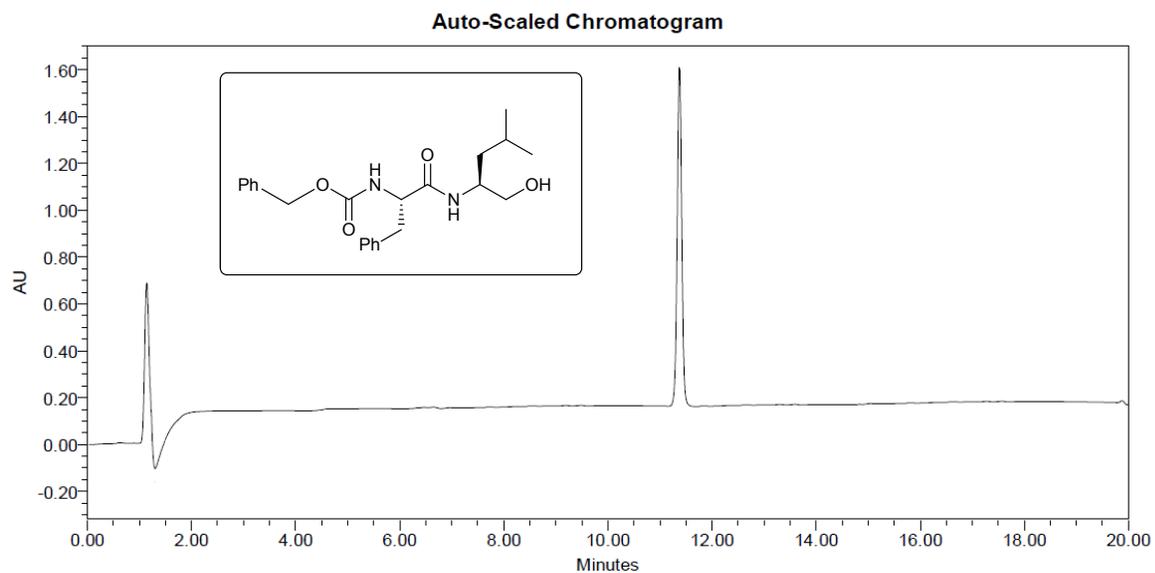
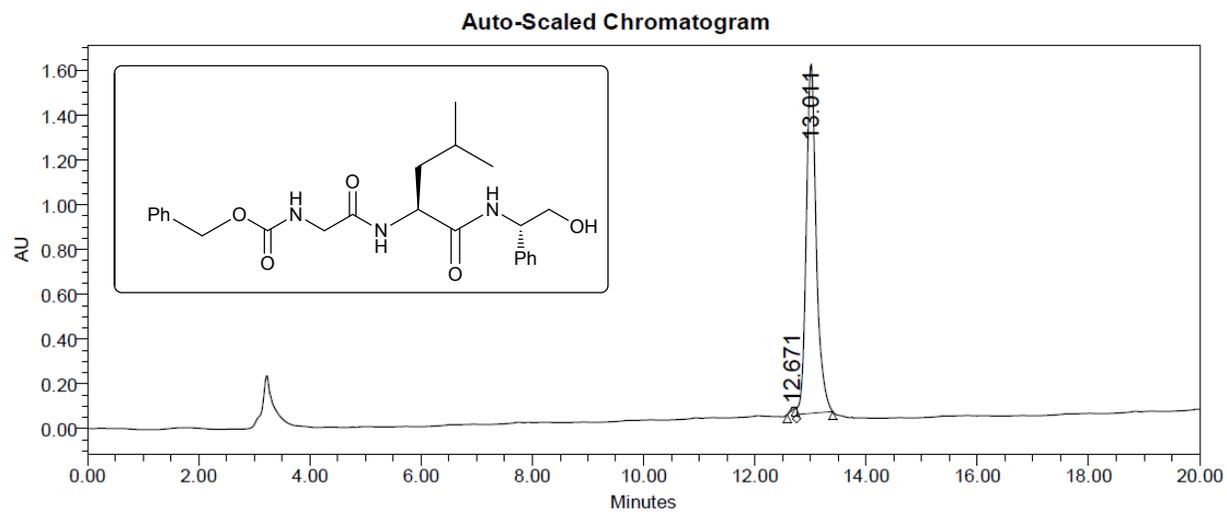
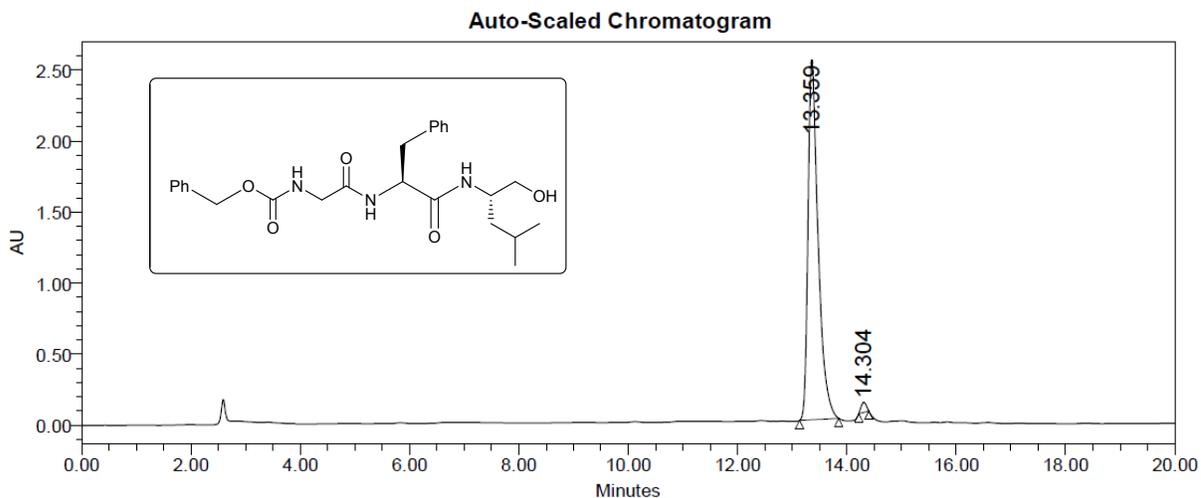


Figure S49 HPLC profile diagram of the product of entry 12 in table 2



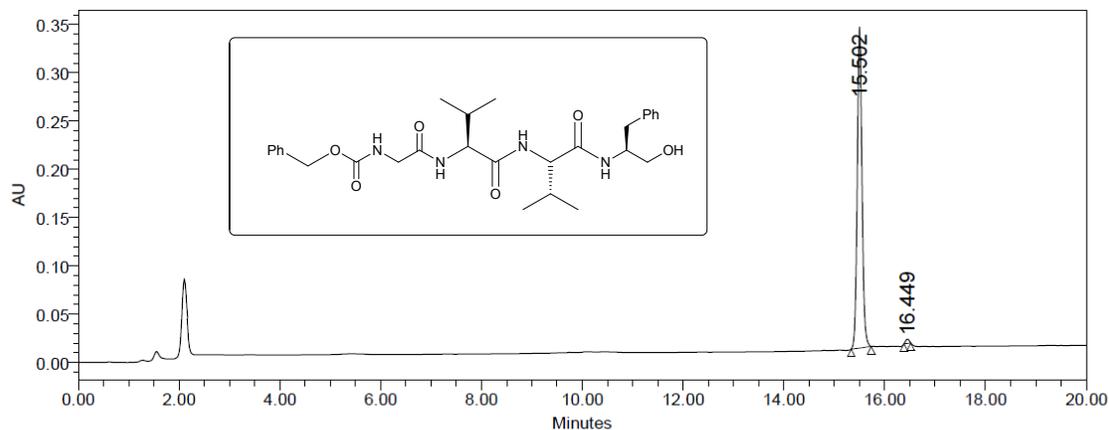
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1	12.671	55936	0.29	10882
2	13.011	19481136	99.71	1563010

Figure S50 HPLC profile diagram of the product of entry 13 in table 2



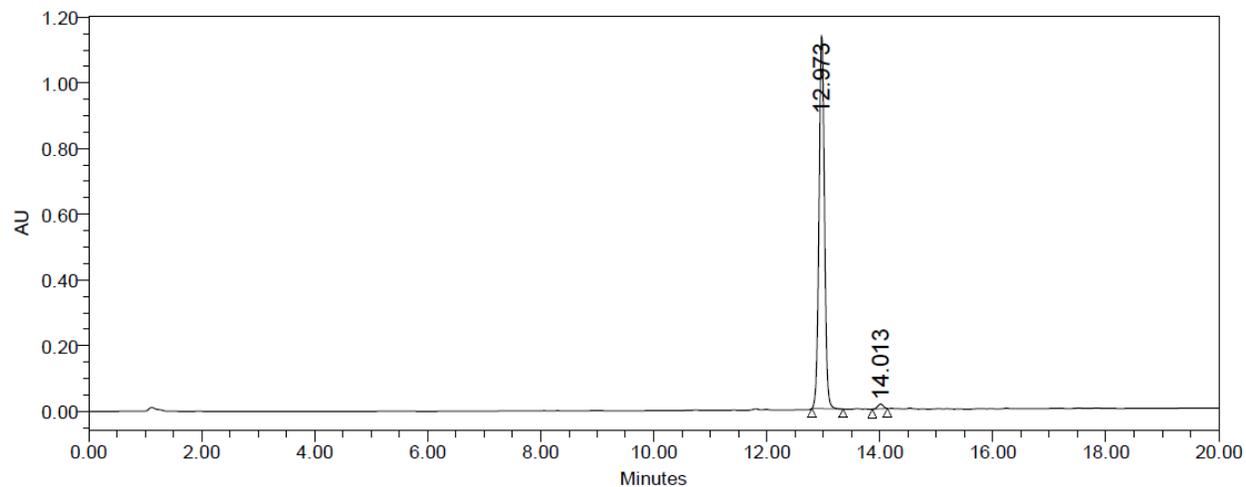
	RT	Area	% Area	Height
1	13.359	32560748	99.60	2549534
2	14.304	463261	0.40	70573

Figure S51 HPLC profile diagram of the product of entry 14 in table 2



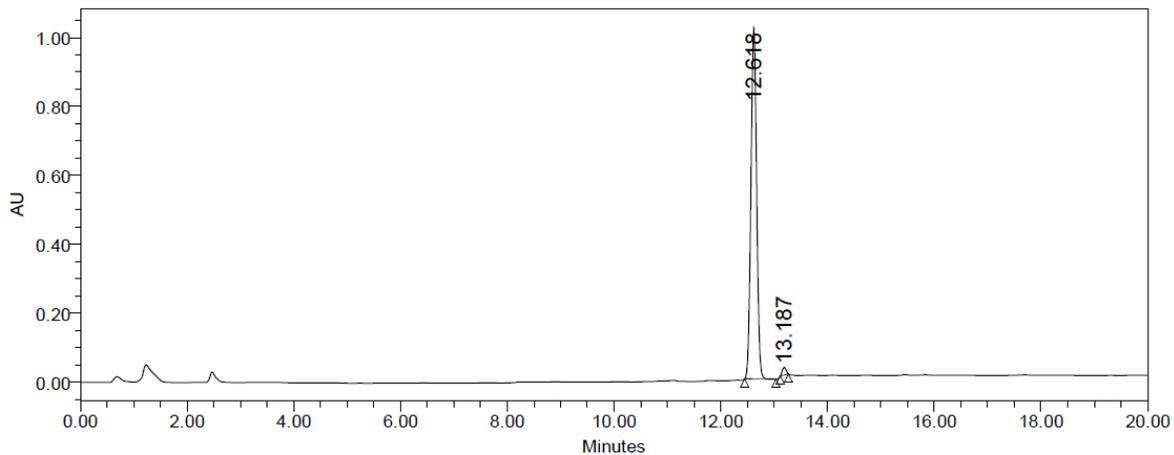
	RT	Area	% Area	Height
1	15.502	2169156	99.04	330702
2	16.449	21026	0.96	4354

Figure S52 HPLC profile diagram of the product of entry 15 in table 2



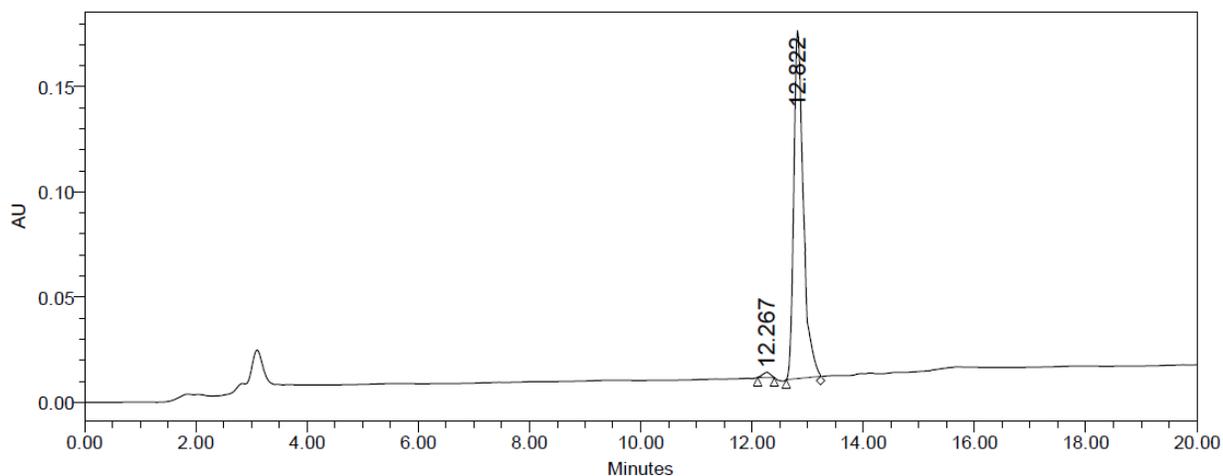
	RT	Area	% Area	Height
1	12.973	7644662	98.74	1140857
2	14.013	97751	1.26	14754

Figure S53 HPLC profile diagram of peptide alcohol **A** (Figure 1)



	RT	Area	% Area	Height
1	12.618	7524790	98.57	1018656
2	13.187	109328	1.43	21627

Figure S54 HPLC profile diagram of peptide alcohol **B** (Figure 1)



	RT	Area	% Area	Height
1	12.267	24372	1.09	2478
2	12.822	2221749	98.91	165780

Figure S55 HPLC profile diagram of peptide alcohol C (Figure 1)

3 Mass spectr of A, B and C

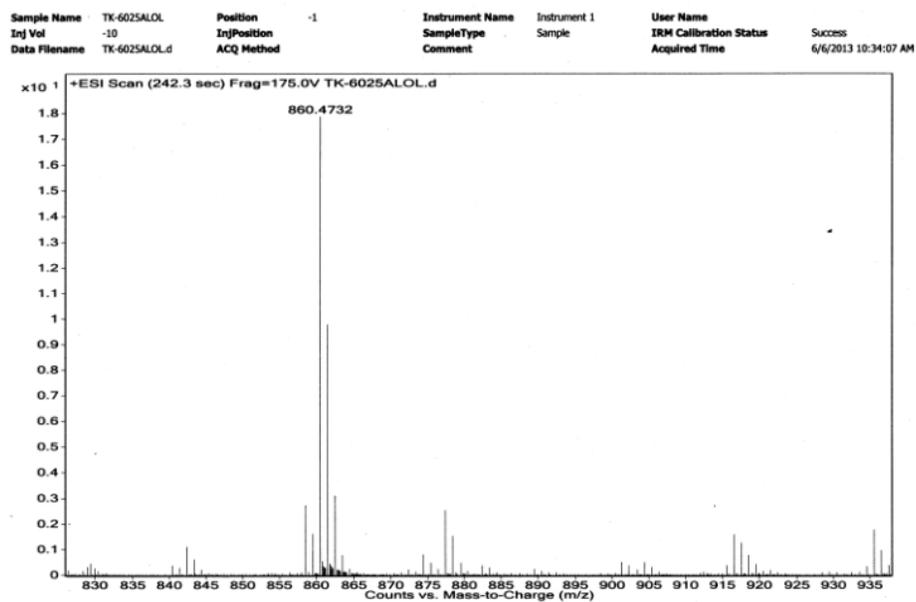


Figure S56 Mass spectra of peptide alcohol A (Figure 1)

Sample Name	TK-6025ALOL	Position	-1	Instrument Name	Instrument 1	User Name	
Inj Vol	-10	InjPosition		SampleType	Sample	IRM Calibration Status	Success
Data Filename	TK-6025ALOL.d	ACQ Method		Comment		Acquired Time	6/6/2013 10:34:07 AM

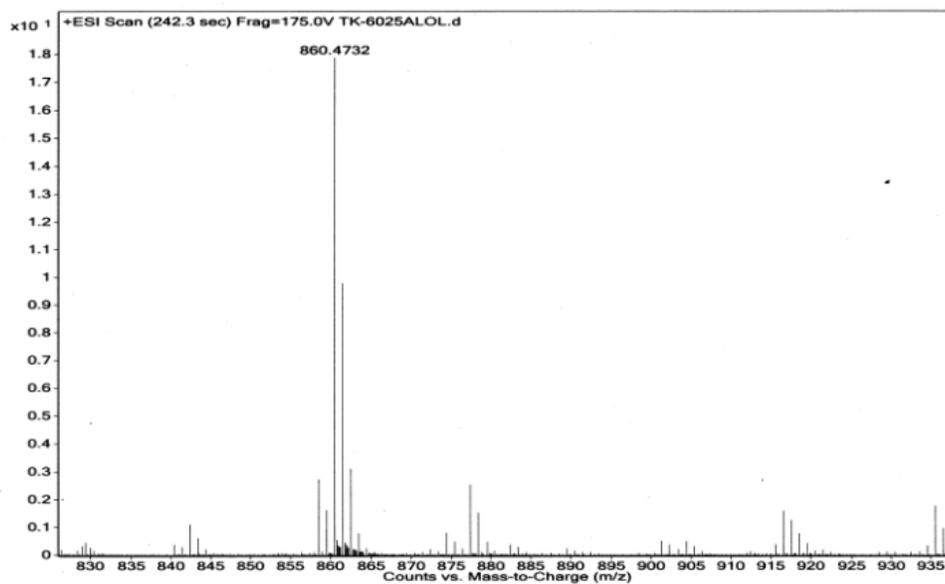


Figure S57 ESI-MS spectrum of the peptide alcohol **B** (Figure 1)

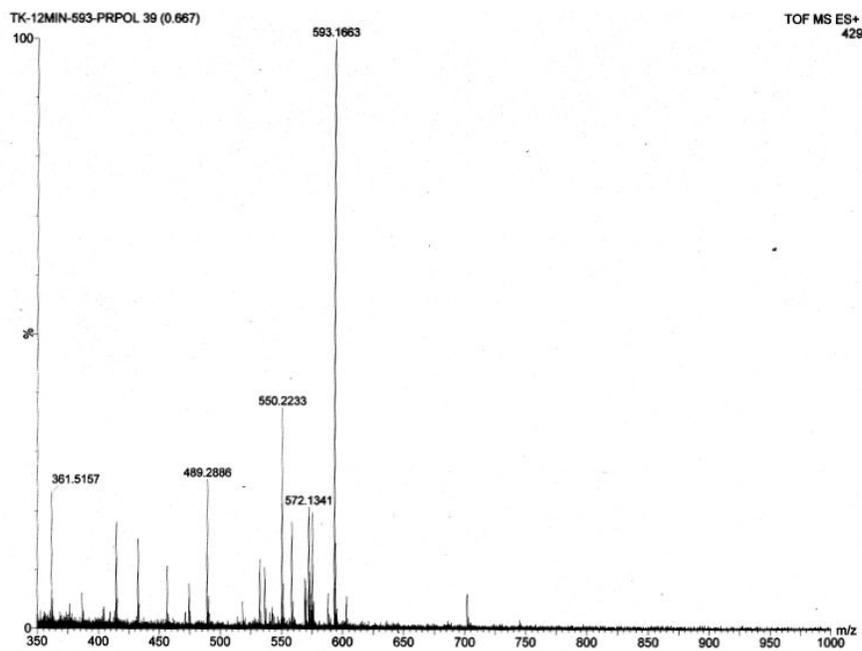


Figure S58 ESI-MS spectrum of peptide alcohol **C** (Figure 1)

4 Spectra for chemoselectivity studies:

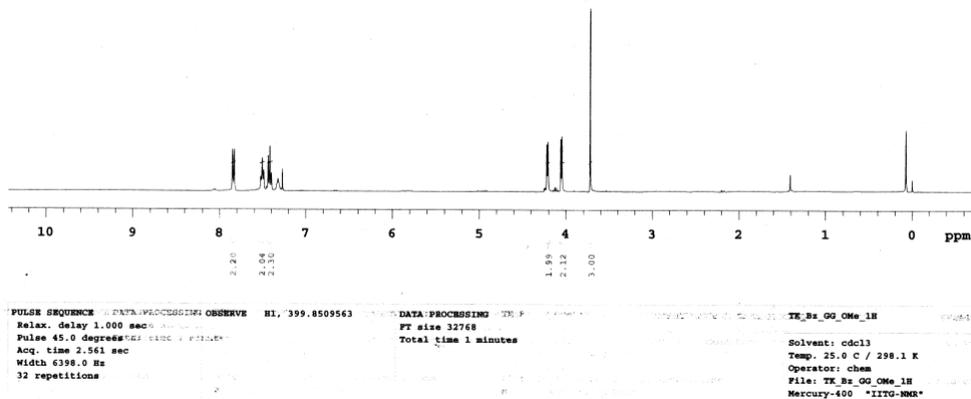


Figure S59. ¹H NMR spectra of compound Bz-Gly-Gly-OMe

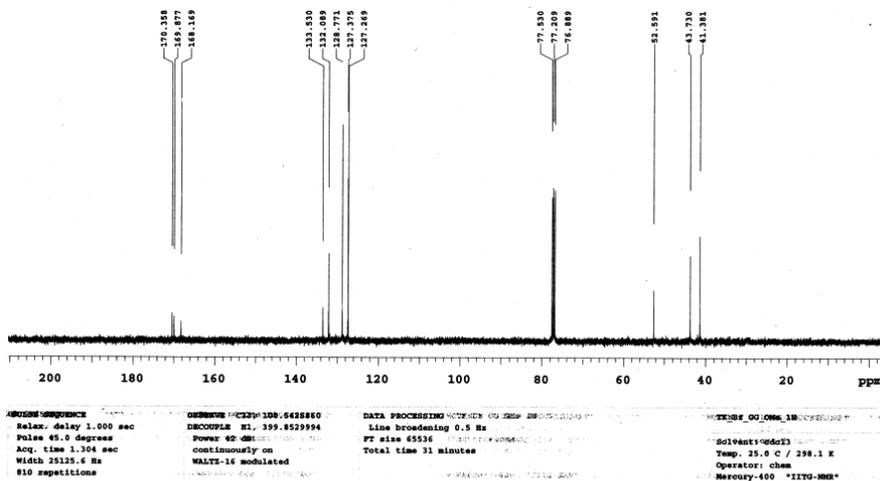


Figure S60. ¹³C NMR spectra of compound Bz-Gly-Gly-OMe

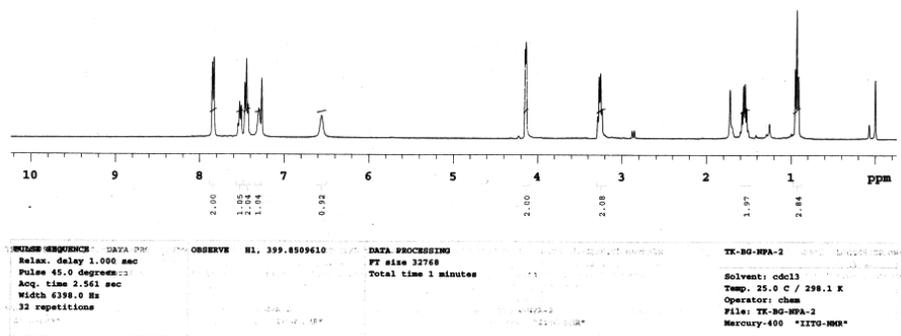


Figure S61. ¹H NMR spectra of compound N-(2-(diisopropylamino)-2-oxoethyl)benzamide

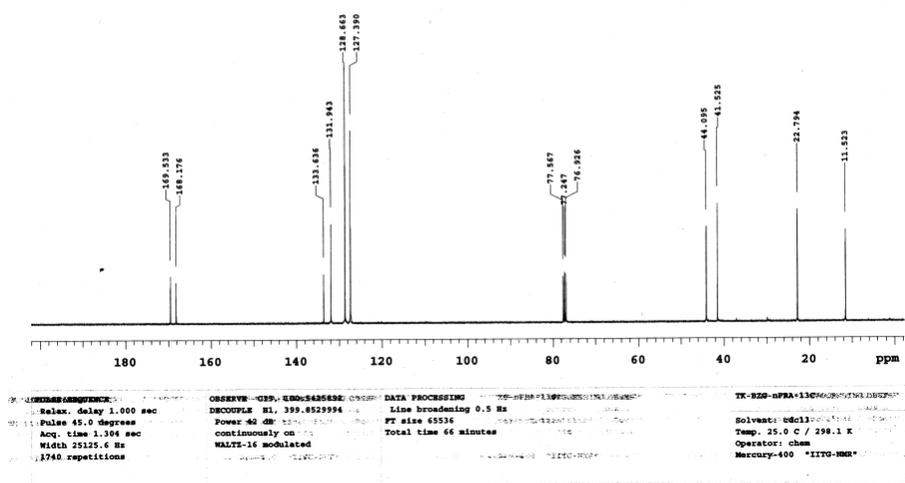


Figure S562. ¹³C NMR spectra of N-(2-(diisopropylamino)-2-oxoethyl)benzamide

5 Spectra for mechanism:

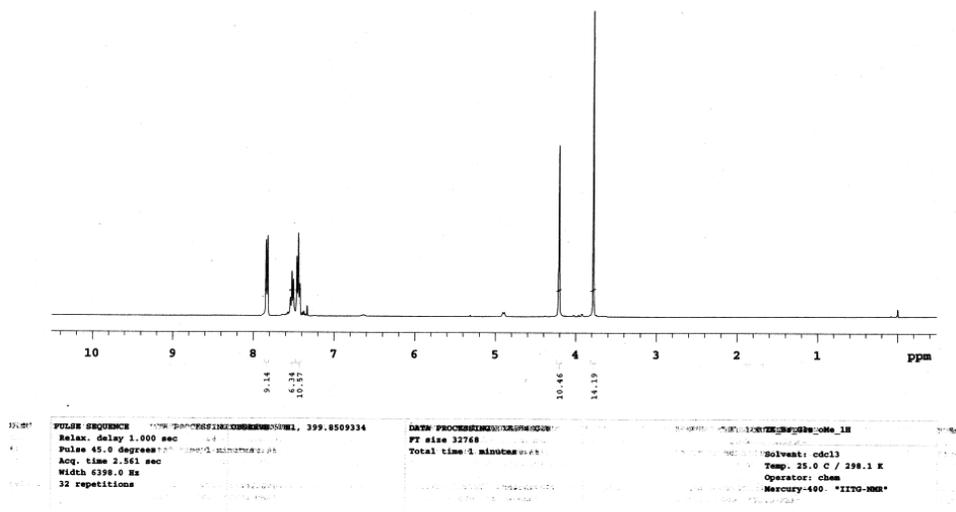


Figure S63. ¹H NMR spectra of compound Bz-Gly-OMe

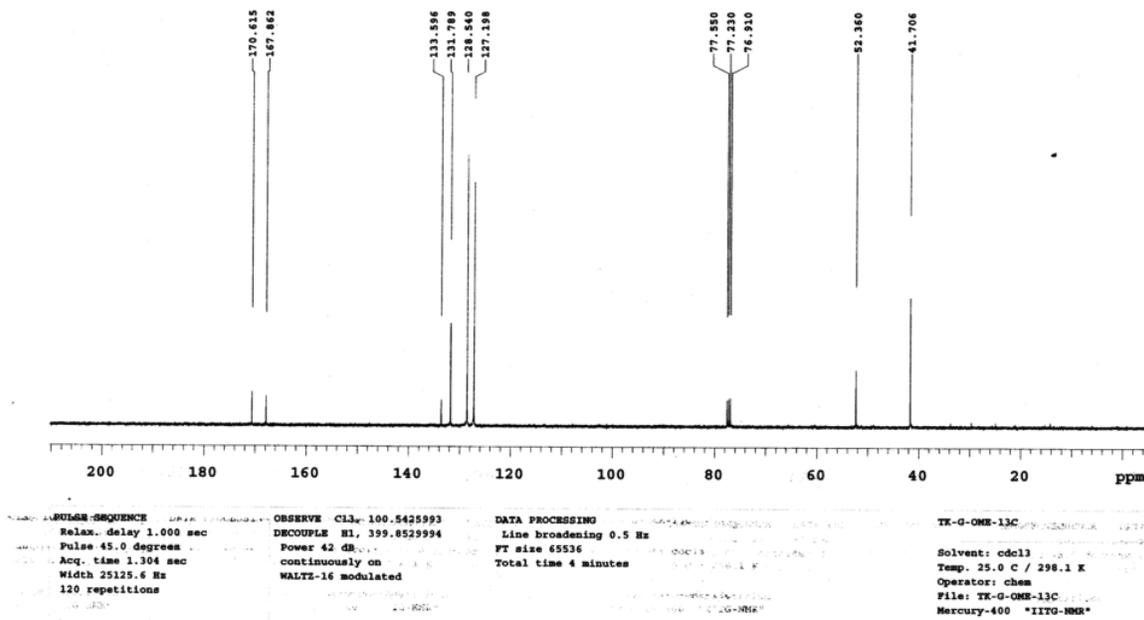


Figure S64. ¹³C NMR spectra of Bz-Gly-OMe

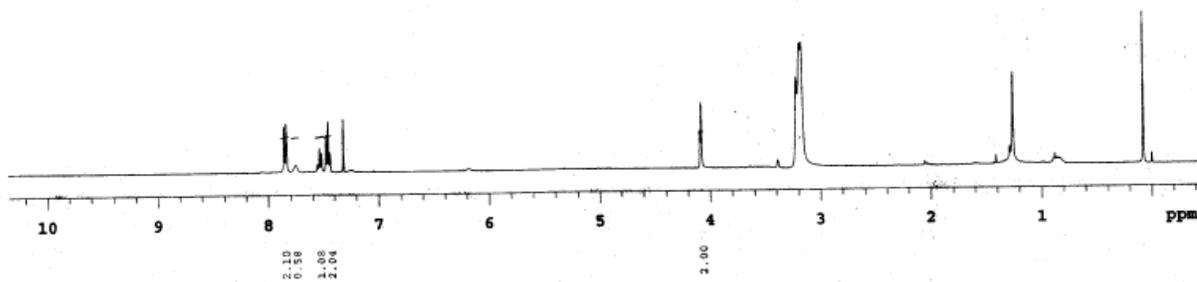


Figure S625. ¹H NMR spectra of compound Bz-Gly-NH₂ (Benzoylglycinamide)

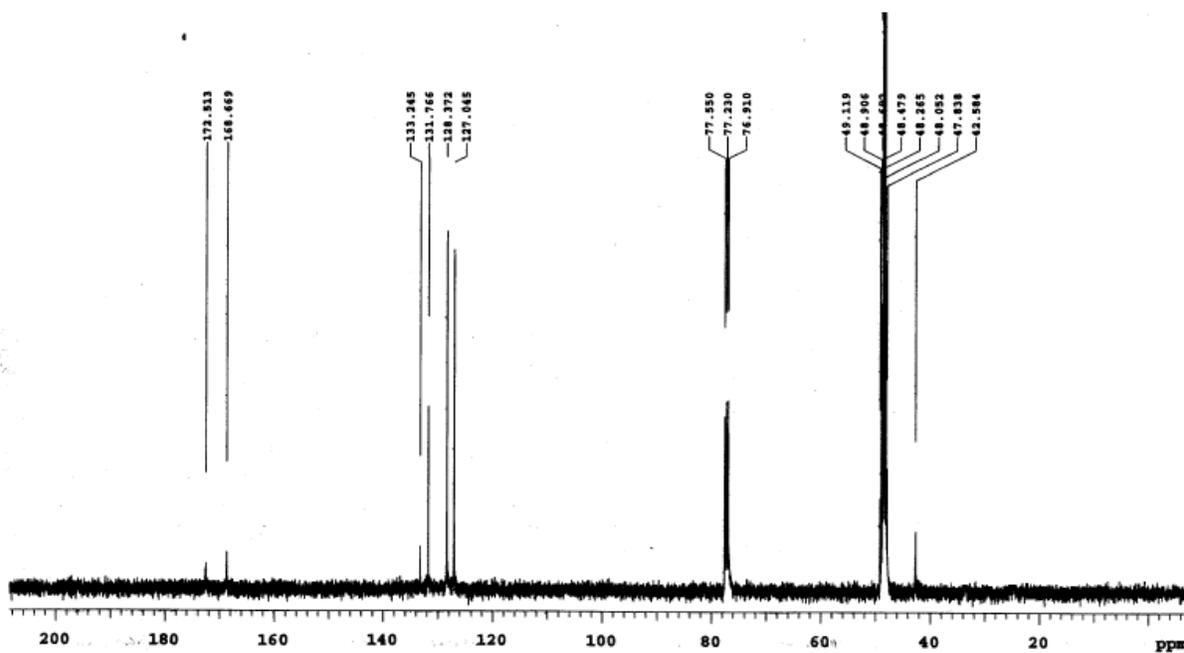


Figure S66. ¹³C NMR spectra of Bz-Gly-NH₂ (Benzoylglycinamide)