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SUPPORTING INFORMATION

Asymmetric transfer hydrogenation of α-amino β-keto ester hydrochlorides through dynamic kinetic resolution

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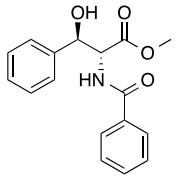
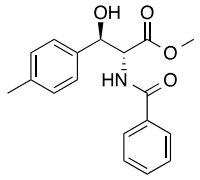
Methyl (2*R*,3*R*)-2-benzamido-3-hydroxy-3-phenylpropanoate 2a



2a

White solid, 76% yield, *anti*:*syn* = 83:17, er_{anti} = 99:1, $[\alpha]_D^{20}$ –113.7 (*c* 0.88, $CHCl_3$), ^1H NMR (300 MHz, $CDCl_3$) (*anti)*: δ=7.66–7.62 (m, 2H; CH), 7.44–7.37 (m, 1H; CH), 7.35–7.28 (m, 2H; CH), 7.23–7.15 (m, 5H; CH), 6.90 (d, *J*=7.4 Hz, 1H; NH), 5.24 (bs, 1H; CH), 5.10 (dd, *J*=7.4, 3.6 Hz, 1H; CH), 4.64 (bs, 1H; OH), 3.62 (s, 3H; CH_3), ^{13}C NMR (75 MHz, $CDCl_3$) (*anti)*: δ=169.9, 168.4, 139.0, 133.0, 132.0, 128.6, 128.2, 128.0, 127.1, 125.8, 75.0, 59.3, 52.5, SFC : Chiralpak AD-H, $scCO_2$/MeOH 85/15, 3 mL/min, P = 150 bar, λ = 215 nm, t_R [*syn*] = 7.75 min, t_R [*syn*] = 8.73 min, t_R [*anti*-(*R*,*R*)] = 10.38 min (major), t_R [*anti*-(*S*,*S*)] = 11.39 min.

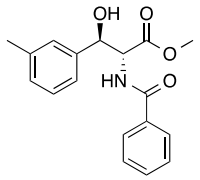
Methyl (2*R*,3*R*)-2-benzamido-3-hydroxy-3-(*p*-tolyl)propanoate 2b



2b

White solid, 90% yield, *anti*:*syn* = 79:21, er_{anti} > 94:6, $[\alpha]_D^{20}$ –105.9 (*c* 0.92, $CHCl_3$), ^1H NMR (300 MHz, $CDCl_3$) (*anti)*: δ=7.74 (d, *J*=7.0 Hz, 2H; CH), 7.55–7.26 (m, 4H; CH), 7.17–7.09 (m, 3H; CH), 6.91 (d, *J*=7.2 Hz, 1H; NH), 5.32 (bs, 1H; CH), 5.20 (dd, *J*=7.2, 3.6 Hz, 1H; CH), 4.52 (d, *J*=5.3 Hz, 1H; OH), 3.75 (s, 3H; CH_3), 2.32 (s, 3H; CH_3), ^{13}C NMR (75 MHz, $CDCl_3$) (*anti*): δ=170.0, 168.5, 137.8, 136.0, 133.1, 132.1, 129.0, 128.6, 127.2, 125.8, 75.1, 59.4, 52.6, 21.1, SFC : Chiralpak AD-H, $scCO_2$/MeOH 89/11, 3 mL/min, P = 100 bar, λ = 215 nm, t_R [*syn*] = 10.93 min, t_R [*syn*] = 22.87 min, t_R [*anti*-(*R*,*R*)] = 24.93 min (major), t_R [*anti*-(*S*,*S*)] = 27.02 min.

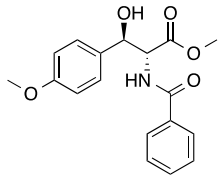
Methyl (2*R*,3*R*)-2-benzamido-3-hydroxy-3-(*m*-tolyl)propanoate 2c



2c

Pale yellow solid, 77% yield, *anti*:*syn* = 80:20, er_{anti} = 99:1, $[\alpha]_D^{20}$ –121.8 (*c* 0.65, $CHCl_3$), ^1H NMR (300 MHz, $CDCl_3$) (*anti)*: δ=7.64 (d, *J*=6.9 Hz, 2H; CH), 7.43–7.20 (m, 3H; CH), 7.13–7.07 (m, 2H; CH), 7.03–6.93 (m, 2H; CH), 6.90 (d, *J*=7.5 Hz, 1H; NH), 5.19 (bs, 1H; CH), 5.07 (dd, *J*=7.5, 3.7 Hz, 1H; CH), 4.52 (bs, 1H; OH), 3.61 (s, 3H; CH_3), 2.21 (s, 3H; CH_3), ^{13}C NMR (75 MHz, $CDCl_3$) (*anti)*: δ=170.0, 168.3, 139.0, 137.8, 133.1, 132.0, 128.8, 128.5, 128.1, 127.1, 126.5, 122.9, 74.9, 59.3, 52.4, 21.3, HPLC : Chiralpak IC, hexane/*i*PrOH 90/10, 1 mL/min, λ = 215 nm; t_R [*syn*] = 22.06 min, t_R [*syn*] = 34.44 min, t_R [*anti*-(*R*,*R*)] = 47.06 min (major), t_R [*anti*-(*S*,*S*)] = 59.18 min.

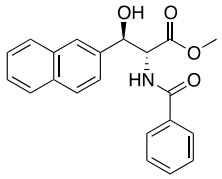
Methyl (2*R*,3*R*)-2-benzamido-3-hydroxy-3-(4-methoxyphenyl)propanoate 2e



2e

White solid, 69% yield, *anti*:*syn* = 79:21, er_{anti} = 97:3, $[\alpha]_D^{20}$ –109.7 (*c* 1.69, $CHCl_3$), ^1H NMR (300 MHz, $CDCl_3$) (*anti*): δ=7.73 (d, *J*=7.3 Hz, 2H; CH), 7.53–7.24 (m, 3H; CH), 7.19 (d, *J*=8.8 Hz, 2H; CH), 6.94 (d, *J*=7.3 Hz, 1H; NH), 6.83 (d, *J*=8.8 Hz, 2H; CH), 5.28 (s, 1H; CH), 5.16 (dd, *J*=7.3, 3.7 Hz, 1H; CH), 4.56 (bs, 1H; OH), 3.76 (s, 3H; CH_3), 3.72 (s, 3H; CH_3), ^{13}C NMR (75 MHz, $CDCl_3$) (*anti*): δ=170.1, 168.4, 159.3, 133.1, 132.0, 131.1, 128.6, 127.1 (2C), 113.7, 74.7, 59.3, 55.1, 52.6, HPLC : Chiralpak IA, hexane/*i*PrOH 90/10, 1 mL/min, λ = 215 nm; t_R [*syn*] = 37.10 min, t_R [*anti*-(*S*,*S*)] = 43.92 min, t_R [*syn*] = 51.40 min, t_R [*anti*-(*R*,*R*)] = 65.26 min (major).

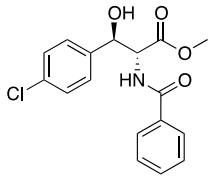
Methyl (2*R*,3*R*)-2-benzamido-3-hydroxy-3-(naphthalen-2-yl)propanoate 2f



2f

White solid, 90% yield, *anti*:*syn* = 78:22, er_{anti} = 96:4, $[\alpha]_D^{20}$ –112.4 (*c* 0.90, $CHCl_3$), ^1H NMR (300 MHz, $CDCl_3$) (*anti*): δ=7.86–7.63 (m, 6H; CH), 7.54–7.26 (m, 6H; CH), 6.97 (d, *J*=7.1 Hz, 1H; NH), 5.53 (bs, 1H; CH), 5.30 (dd, *J*=7.2, 3.4 Hz, 1H; CH), 4.75 (d, *J*=5.3 Hz, 1H; OH), 3.73 (s, 3H; CH_3), ^{13}C NMR (75 MHz, $CDCl_3$) (*anti*): δ=169.9, 168.7, 136.6, 133.6, 133.13, 133.1, 133.06, 132.2, 128.7, 128.1, 128.0, 127.7, 127.2, 126.2, 126.1, 125.0, 123.7, 75.4, 59.5, 52.7, SFC : Chiralpak AD-H, $scCO_2$/MeOH 85/15, 4 mL/min, P = 100 bar, λ = 215 nm, t_R [*syn*] = 8.98 min, t_R [*anti*-(*R*,*R*)] = 21.35 min (major), t_R [*syn*] = 23.76 min, t_R [*anti*-(*S*,*S*)] = 28.55 min.

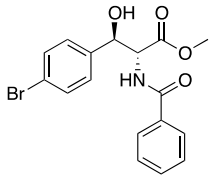
Methyl (2*R*,3*R*)-2-benzamido-3-(4-chlorophenyl)-3-hydroxypropanoate 2g



2 g

White solid, 82% yield, *anti*:*syn* = 76:24, er_{anti} = 93:7, $[\alpha]_D^{20}$ –100.5 (*c* 1.53, $CHCl_3$), ^1H NMR (300 MHz, $CDCl_3$) (*anti*): δ=7.65–7.61 (m, 2H; CH), 7.46–7.10 (m, 7H; CH), 6.93 (d, *J*=7.2 Hz, 1H; NH), 5.21 (bs, 1H; CH), 5.06 (dd, *J*=7.2, 3.4 Hz, 1H; CH), 4.84 (d, *J*=5.1 Hz, 1H; OH), 3.64 (s, 3H; CH_3), ^{13}C NMR (75 MHz, $CDCl_3$) (*anti*): δ=169.7, 168.6, 137.7, 133.7, 132.8, 132.4, 128.7, 128.4, 127.3, 127.1, 74.5, 59.4, 52.7, SFC : Chiralpak AD-H, $scCO_2$/MeOH 85/15, 4 mL/min, P = 100 bar, λ = 215 nm, t_R [*syn*] = 5.23 min, t_R [*syn*] = 8.36 min, t_R [*anti*-(*S*,*S*)] = 12.68 min, t_R [*anti*-(*R*,*R*)] = 15.26 min (major).

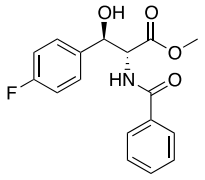
Methyl (2*R*,3*R*)-2-benzamido-3-(4-bromophenyl)-3-hydroxypropanoate 2h



2h

White solid, 66% yield, *anti*:*syn* = 74:26, er_{anti} = 95:5, $[\alpha]_D^{20}$ –87.0 (*c* 1.42, $CHCl_3$), ^1H NMR (300 MHz, $CDCl_3$) (*anti*): δ=7.63 (d, *J*=7.7 Hz, 2H; CH), 7.46–7.22 (m, 5H; CH), 7.05 (d, *J*=7.7 Hz, 2H; CH), 6.94 (d, *J*=7.2 Hz, 1H; NH), 5.19 (bs, 1H; CH), 5.05 (dd, *J*=7.2, 3.4 Hz, 1H; CH), 4.86 (s, 1H; OH), 3.63 (s, 3H; CH_3), ^{13}C NMR (75 MHz, $CDCl_3$) (*anti*): δ=169.6, 168.5, 138.3, 132.7, 132.2, 131.3, 128.6, 127.6, 127.1, 121.9, 74.5, 59.3, 52.7, SFC : Chiralpak AD-H, $scCO_2$/MeOH 80/20, 4 mL/min, P = 150 bar, λ = 215 nm, t_R [*syn*] = 3.46 min, t_R [*syn*] = 5.53 min, t_R [*anti*-(*S*,*S*)] = 8.68 min, t_R [*anti*-(*R*,*R*)] = 11.02 min (major).

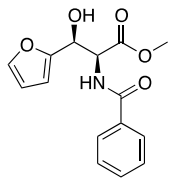
Methyl (2*R*,3*R*)-2-benzamido-3-(4-fluorophenyl)-3-hydroxypropanoate 2i



2i

White solid, 82% yield, *anti*:*syn* = 76:24, er_{anti} = 93:7, $[\alpha]_D^{20}$ –98.6 (*c* 0.98, $CHCl_3$), ^1H NMR (300 MHz, $CDCl_3$) (*anti*): δ=7.64 (d, *J*=7.0 Hz, 2H; CH), 7.45–7.13 (m, 5H; CH), 6.91 (t, *J*=8.6 Hz, 3H; CH,NH), 5.24 (d, *J*=3.5 Hz, 1H; CH), 5.07 (dd, *J*=7.1, 3.5 Hz, 1H; CH), 3.65 (s, 3H; CH_3), ^{13}C NMR (75 MHz, $CDCl_3$) (*anti*): δ=169.8, 168.6, 162.4 (d, *J*=246.2 Hz), 134.9 (d, *J*=3.0 Hz), 132.8, 132.2, 128.7, 127.5, 127.1, 115.2 (d, *J*=21.6 Hz), 74.5, 59.4, 52.7, SFC : Chiralpak AD-H, $scCO_2$/MeOH 85/15, 4 mL/min, P = 150 bar, λ = 215 nm, t_R [*syn*] = 3.04 min, t_R [*syn*] = 4.22 min, t_R [*anti*-(*S*,*S*)] = 5.42 min, t_R [*anti*-(*R*,*R*)] = 7.12 min (major).

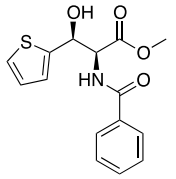
Methyl (2*S*,3*S*)-2-benzamido-3-(furan-2-yl)-3-hydroxypropanoate 2j



2j

Orange solid, 66% yield, *anti*:*syn* = 41:59, er_{anti} = 95:5, er_{syn} > 99:1, $[\alpha]_D^{20}$ –44.0 (*c* 0.66, $CHCl_3$), ^1H NMR (300 MHz, $CDCl_3$) (*syn)*: δ=7.75–7.71 (m, 2H; CH), 7.53–7.31 (m, 4H; CH), 7.18 (d, *J*=8.8 Hz, 1H; NH), 6.31–6.29 (m, 1H; CH), 6.25 (dd, *J*=3.3, 1.8 Hz, 1H; CH), 5.36–5.31 (m, 1H; CH), 5.17 (dd, *J*=8.8, 3.2 Hz, 1H), 4.24 (bs, 1H; OH), 3.73 (s, 3H; CH_3), ^{13}C NMR (75 MHz, $CDCl_3$) (*syn)*: δ=170.5, 167.8, 152.6, 142.4, 133.6, 131.7, 128.4, 127.1, 110.2, 107.2, 68.2, 56.4, 52.7, SFC (er measurement for the *anti* compound) : Chiralpak IC, $scCO_2$/MeOH 90/10, 4 mL/min, P = 150 bar, λ = 215 nm, t_R [*syn* × 2] = 4.49 min, t_R [*anti*] = 6.55 min, t_R [*anti*] = 7.59 min, SFC (er measurement for the *syn* compound) : Chiralcel OD-H, $scCO_2$/MeOH 95/5, 2 mL/min, P = 150 bar, λ = 215 nm, t_R [*syn*] = 10.74 min, t_R [*anti* × 2] = 11.78 min, t_R [*syn*] = 14.63 min.

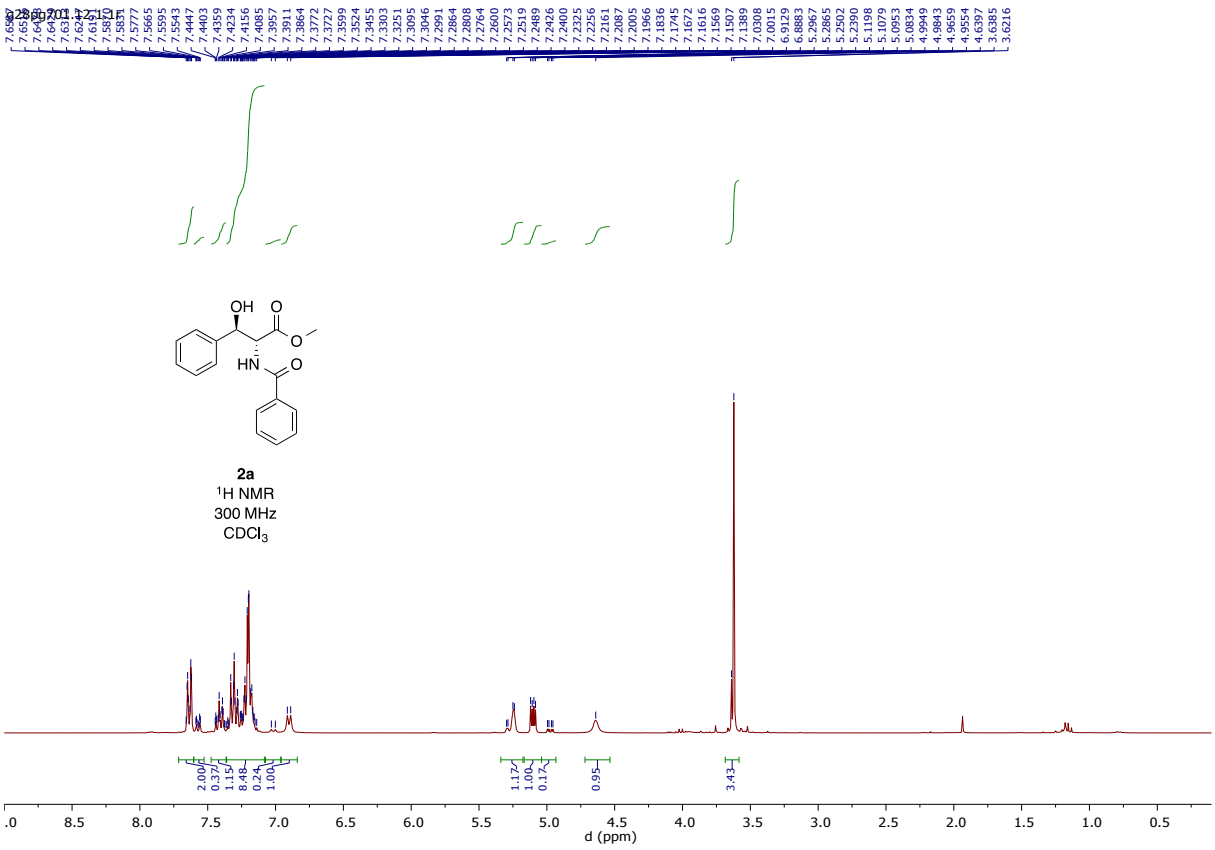
Methyl (2*S*,3*S*)-2-benzamido-3-hydroxy-3-(thiophen-2-yl)propanoate 2k



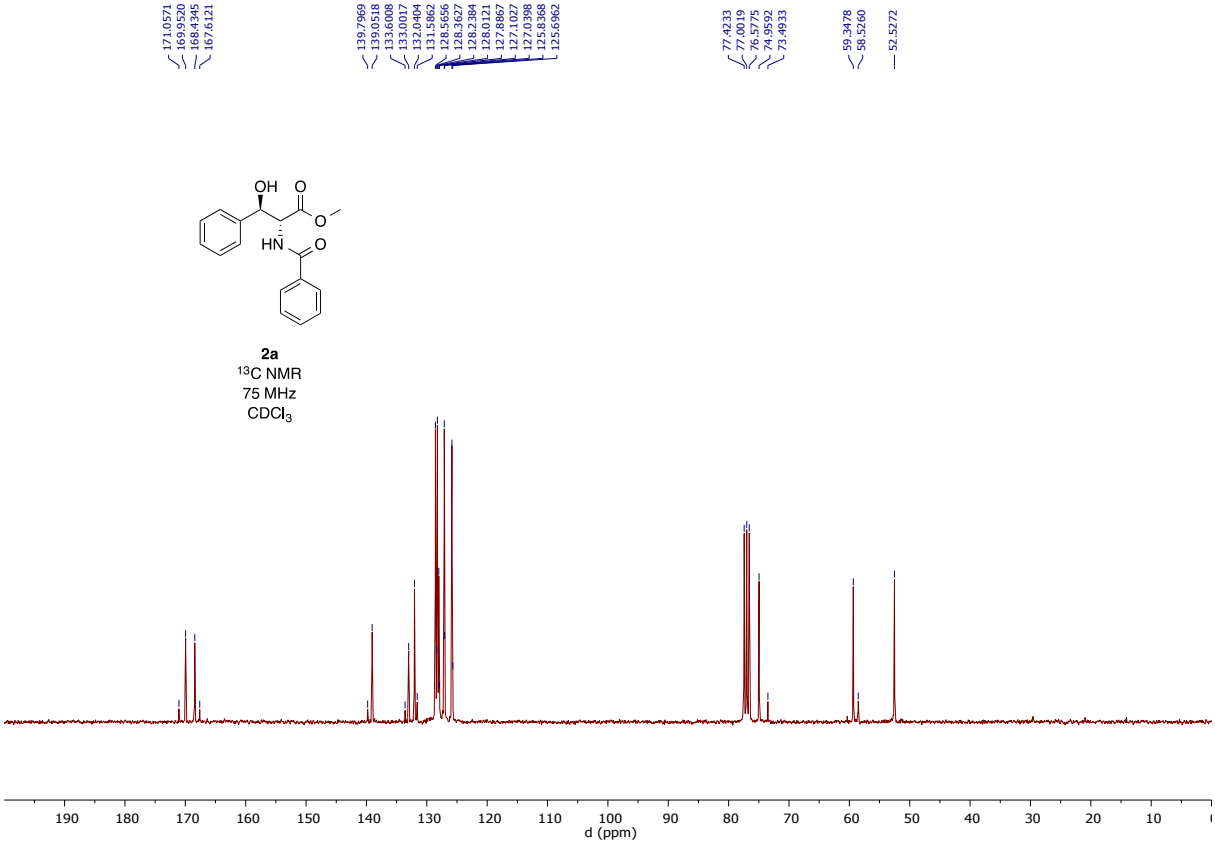
2k

Orange oil, 79% yield, *anti*:*syn* = 32:68, er_{syn} > 99:1, er_{anti} = 98:2, $[\alpha]_D^{20}$ –48.1 (*c* 0.80, $CHCl_3$), ^1H NMR (300 MHz, $CDCl_3$) (*syn*): δ=7.79–7.75 (m, 2H; CH), 7.56–7.35 (m, 4H; CH), 7.21 (dd, *J*=5.1, 1.3 Hz, 1H; CH), 7.15 (d, *J*=8.9 Hz, 1H; NH), 7.03–7.01 (m, 1H; CH), 6.97–6.88 (m, 1H; CH), 5.63 (bs, 1H; CH), 5.12 (dd, *J*=8.9, 2.7 Hz, 1H; CH), 3.76 (s, 3H; CH_3), ^{13}C NMR (75 MHz, $CDCl_3$) (*syn*): δ=170.6, 167.9, 143.2, 133.6, 131.8, 128.5, 127.2, 126.6, 125.5, 124.7, 70.3, 58.1, 52.8, SFC : Chiralpak AD-H, $scCO_2$/MeOH 80/20, 4 mL/min, P = 150 bar, λ = 215 nm, t_R [*syn*-(*R*,*R*)] = 3.02 min, t_R [*syn*-(*S*,*S*)] = 3.80 min (major), t_R [*anti*] = 5.32 min, t_R [*anti*] = 5.95 min.

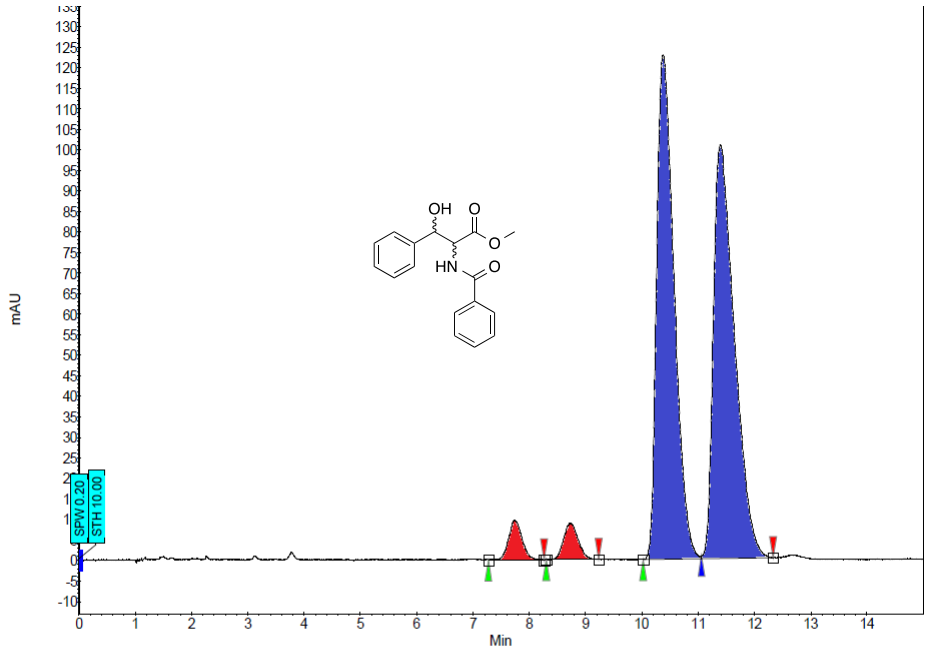
NMR and SFC/HPLC spectra of compounds 2a–2c and 2e–2k



2a
^{1}H NMR
300 MHz
$CDCl_3$



2a
^{13}C NMR
75 MHz
$CDCl_3$



Results Table:

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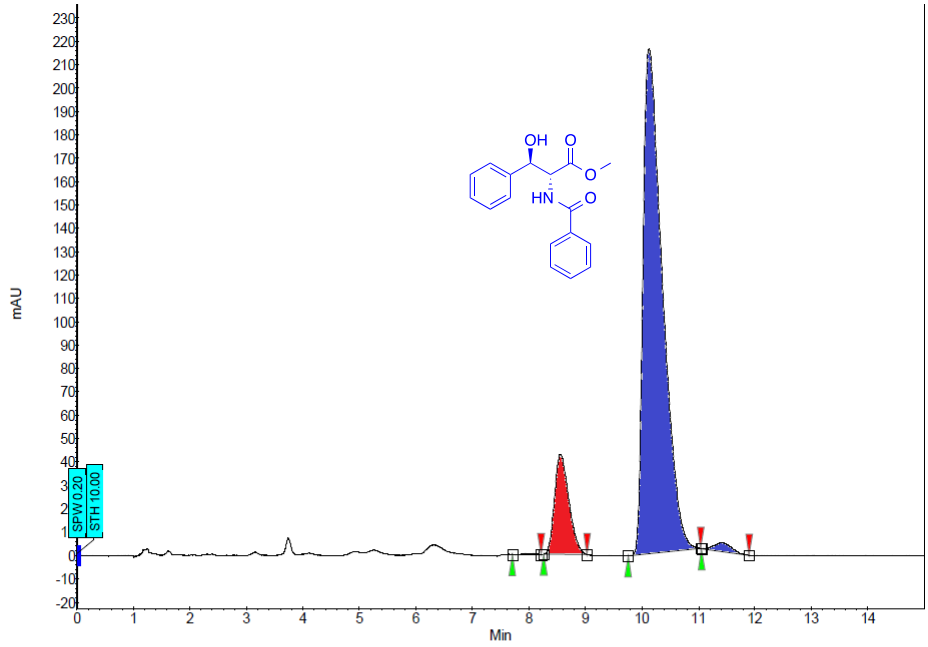
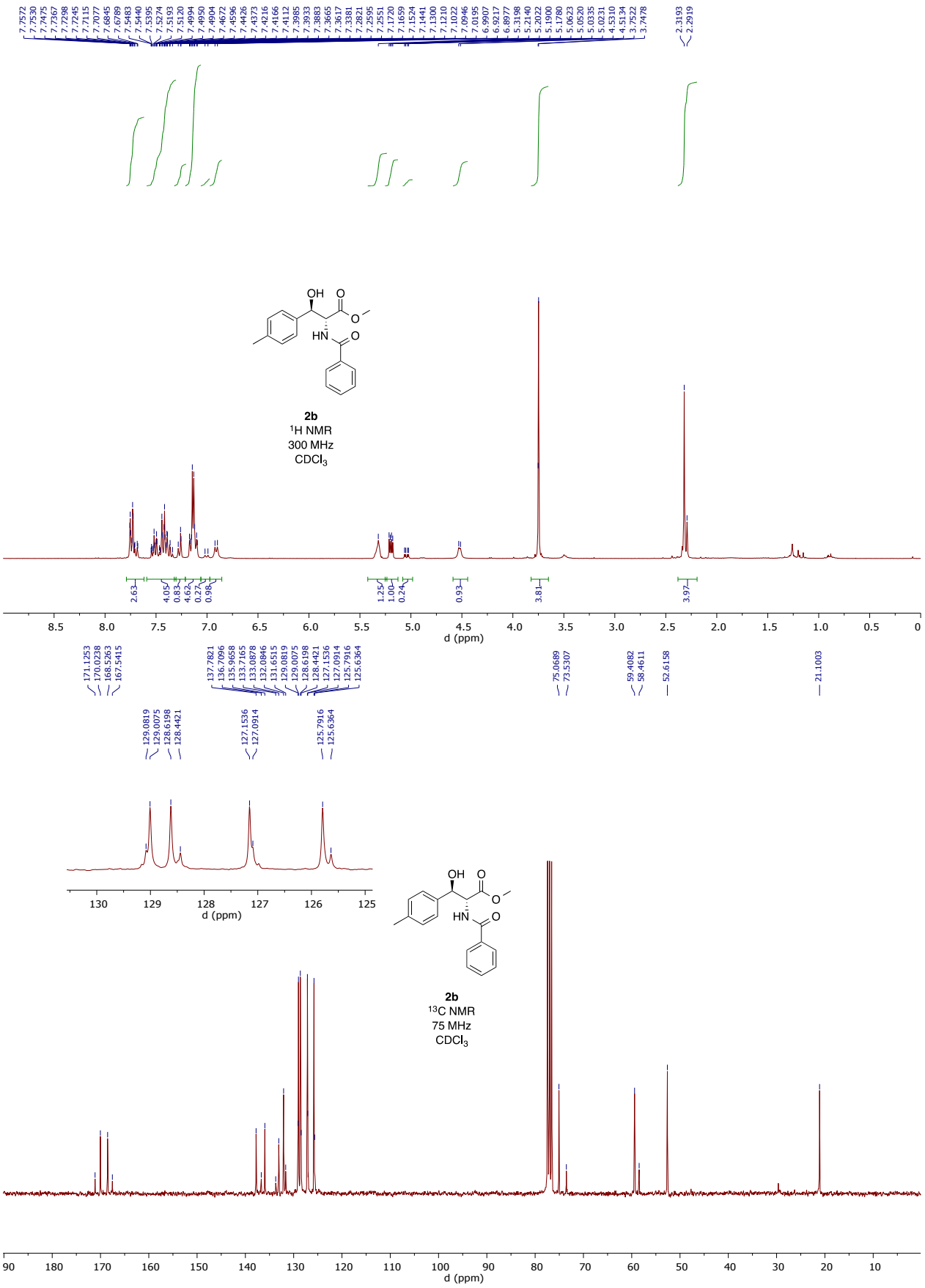
2	UNKNOWN	8.30	8.73	9.23	0.00	2.83	8.8	2.6	2.832
3	UNKNOWN	10.02	10.38	11.06	0.00	47.27	122.8	43.7	47.266
4	UNKNOWN	11.06	11.39	12.33	0.00	47.05	100.7	43.5	47.053
Total						100.00	241.9	92.5	100.000



Results Table:

Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	7.71	8.01	8.22	0.00	0.12	0.6	0.1	0.116
2	UNKNOWN	8.27	8.56	9.03	0.00	12.65	42.9	12.6	12.652
3	UNKNOWN	9.75	10.12	11.04	0.00	85.91	215.9	85.7	85.914
4	UNKNOWN	11.06	11.43	11.89	0.00	1.32	3.7	1.3	1.318
Total						100.00	263.1	99.8	100.000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HN
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2b
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3.97
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7.0
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130
129
128
127
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d (ppm)
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O
O
HN
O
2b
13C NMR
75 MHz
CDCl3
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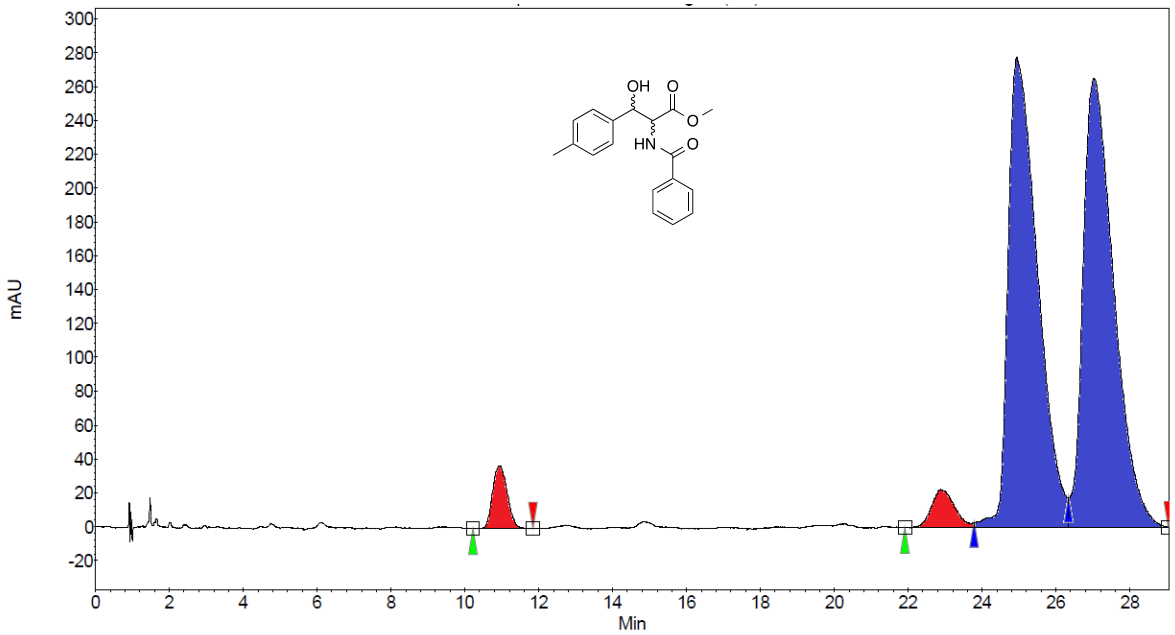
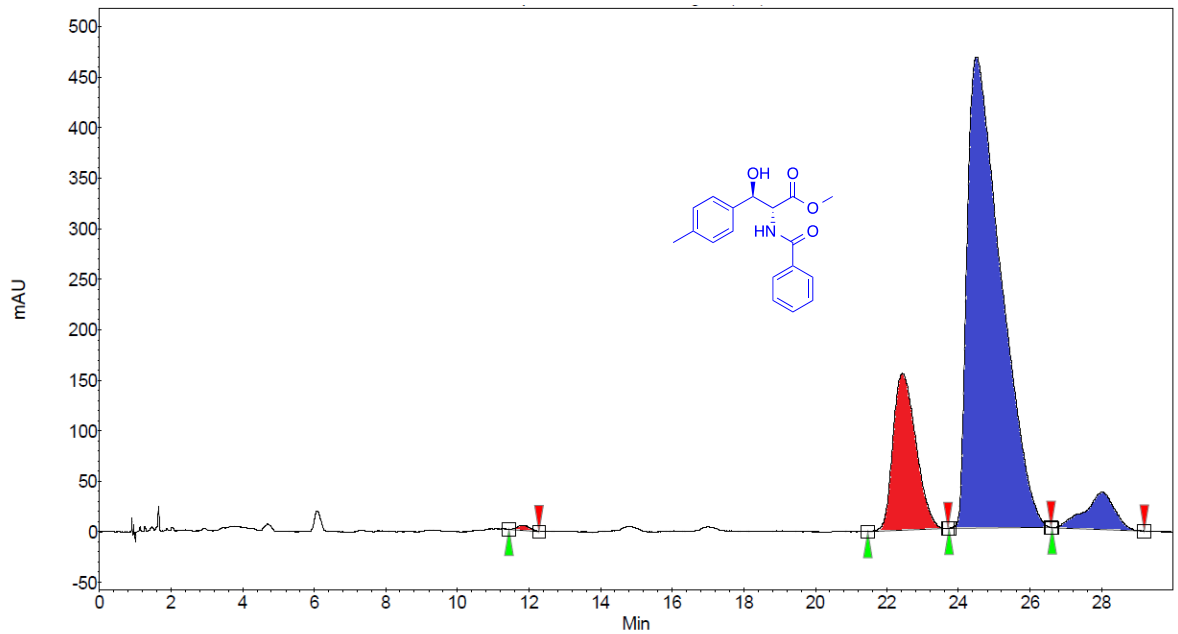
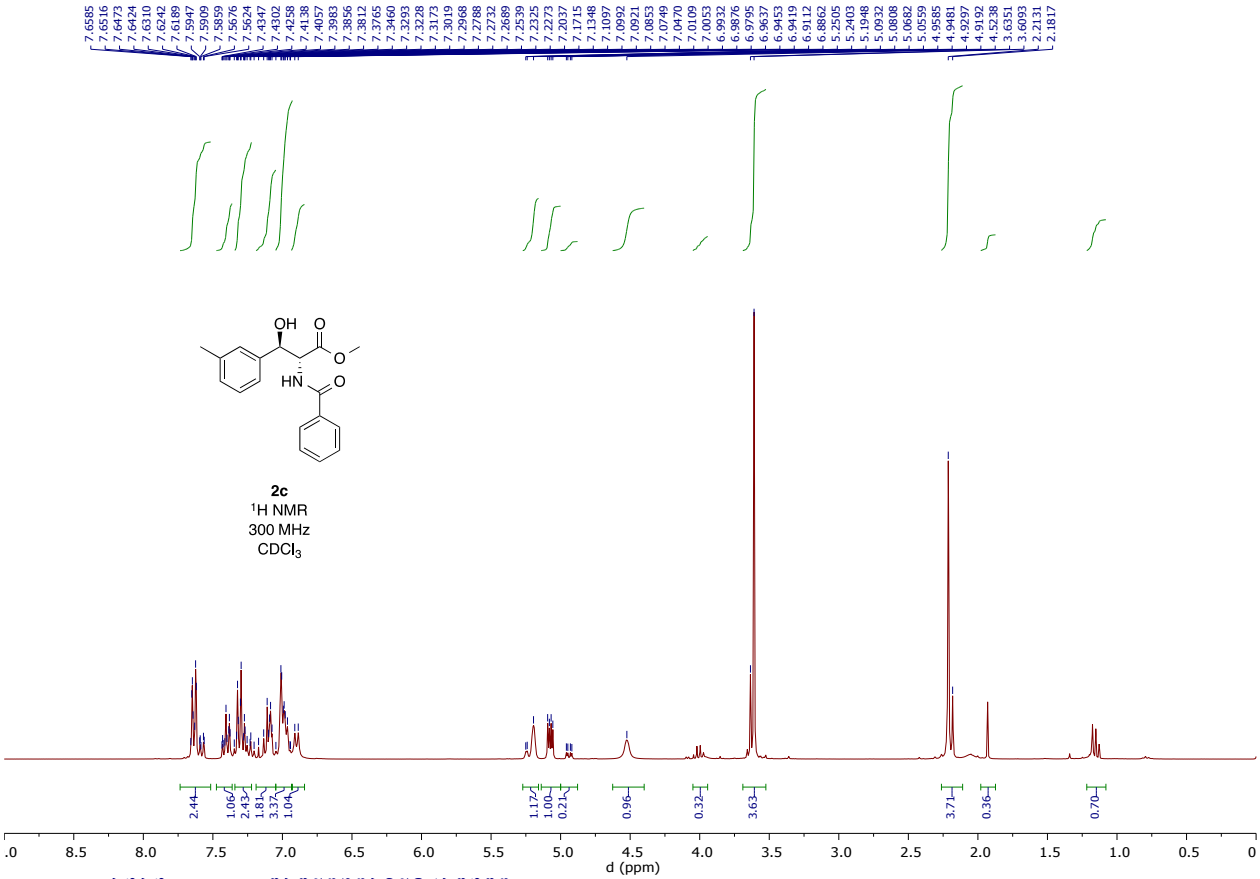
d (ppm)



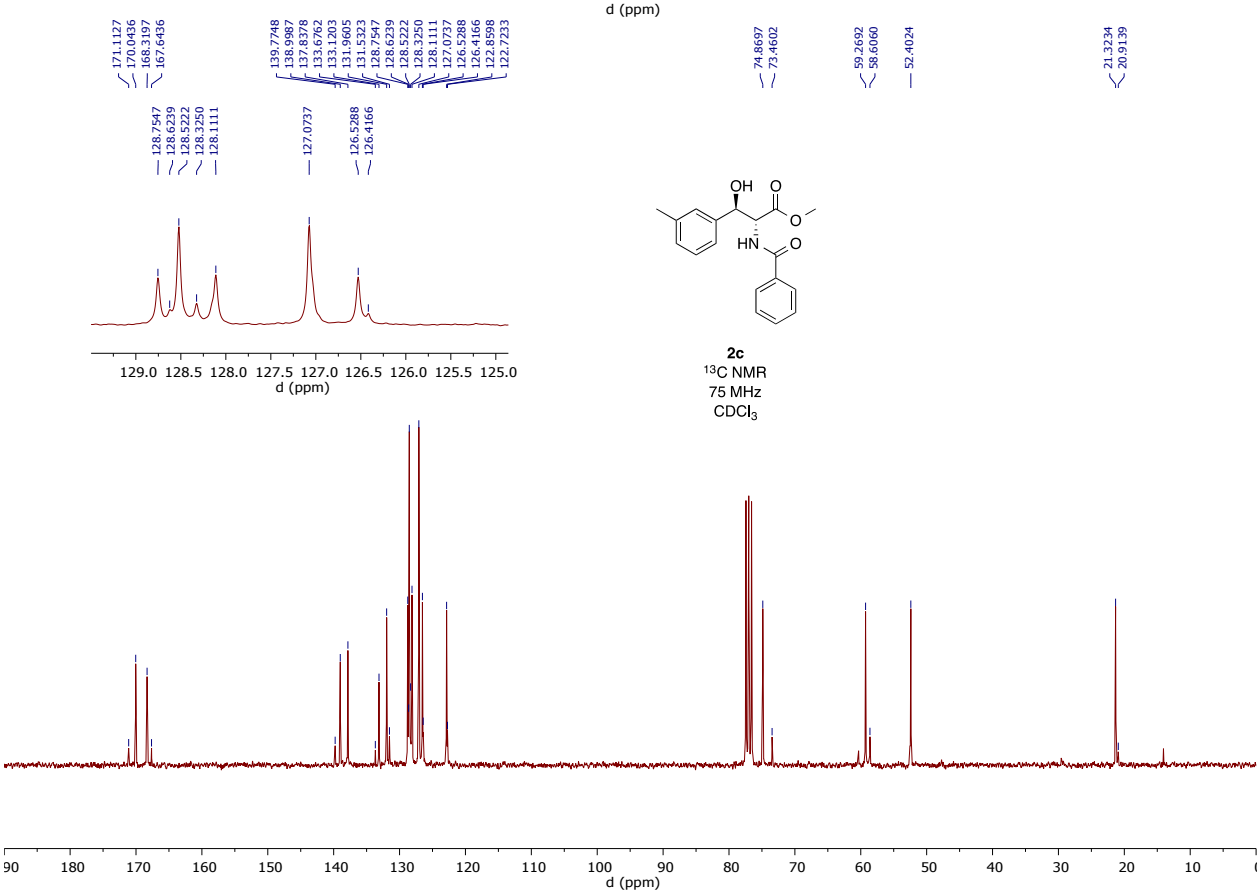
Index	Name	Time	Height	Area	Area	Selectivity	Res. HW
		[Min]	[μV]	[μV.Min]	[%]		
1	UNKNOWN	10.93	37.2	17.0	3.097	0.00	0.00
2	UNKNOWN	22.87	22.5	15.9	2.913	2.09	12.76
3	UNKNOWN	24.93	277.8	257.6	47.055	1.09	1.59
4	UNKNOWN	27.02	265.2	256.9	46.936	1.08	1.40
Total			602.8	547.4	100.000		



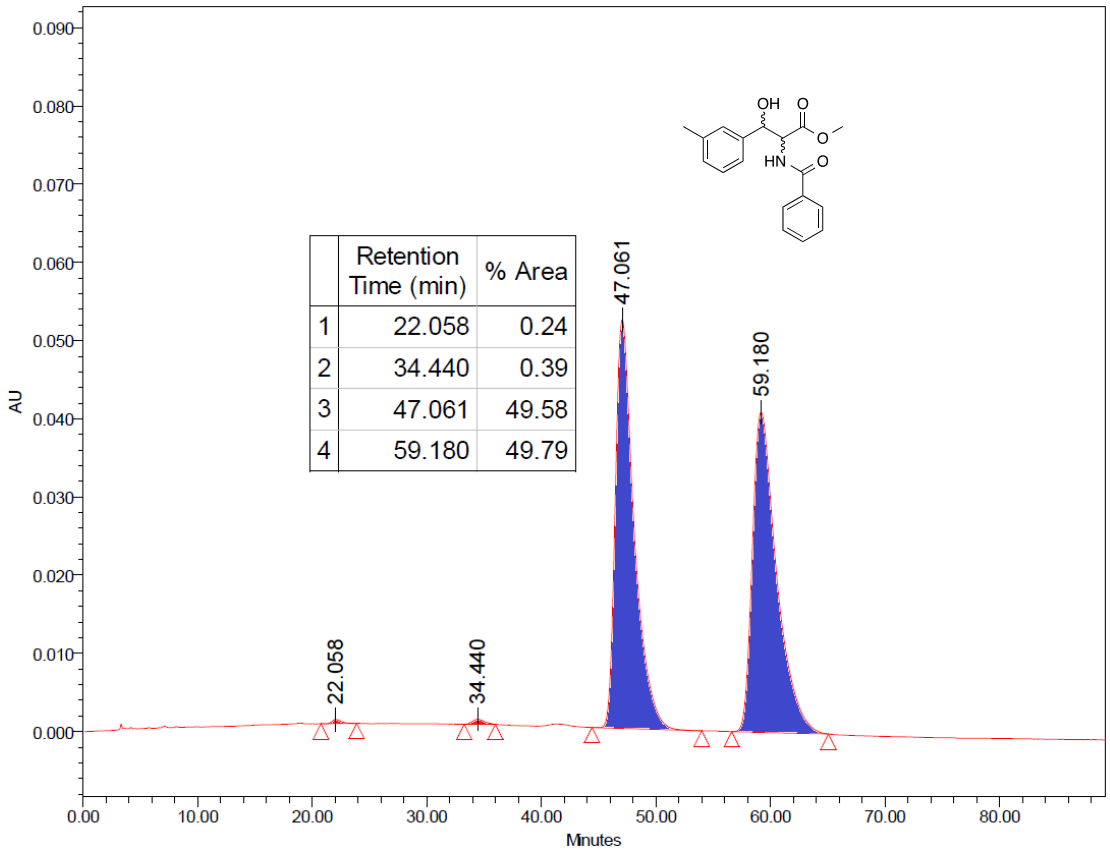
Index	Name	Time	Height	Area	Area	Selectivity	Res. HW
		[Min]	[μV]	[μV.Min]	[%]		
3	UNKNOWN	11.83	5.3	2.0	0.300	0.00	0.00
1	UNKNOWN	22.43	155.7	116.3	17.543	1.90	11.70
2	UNKNOWN	24.53	466.2	508.5	76.689	1.09	1.41
4	UNKNOWN	28.01	37.1	36.3	5.469	1.14	2.21
Total			664.4	663.0	100.000		



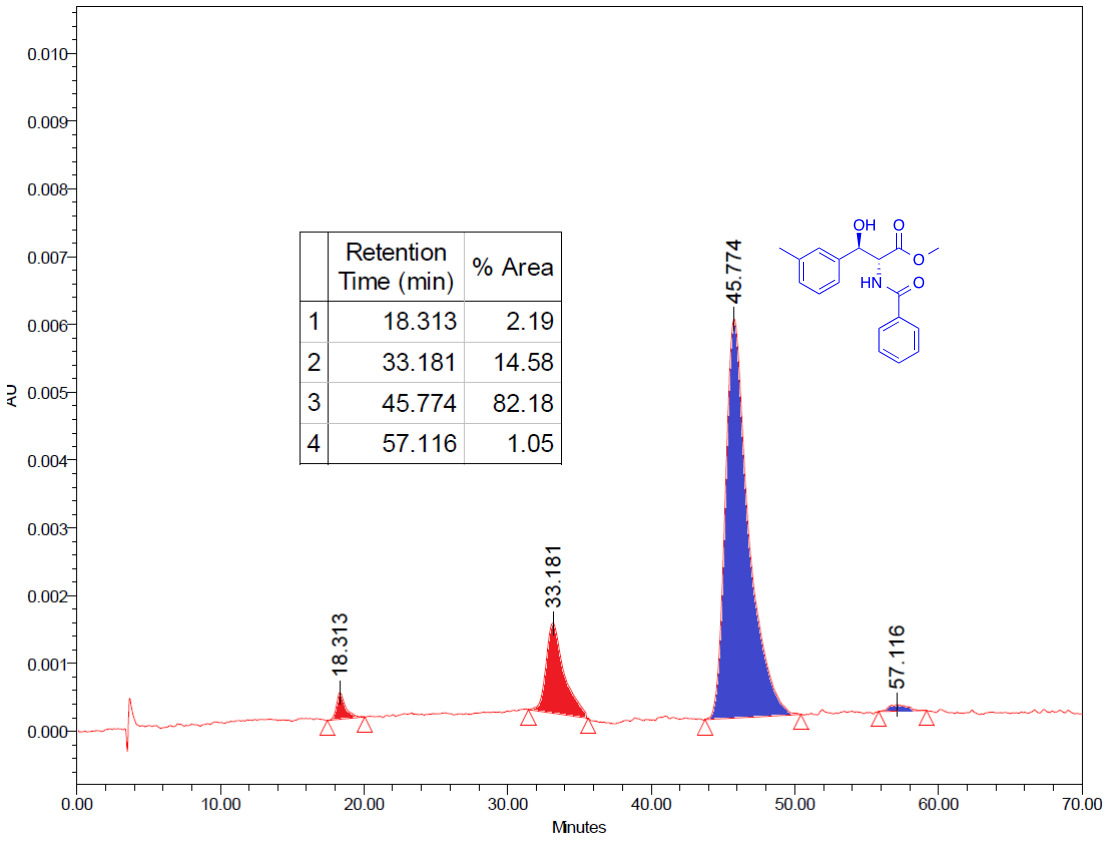
2c
^{1}H NMR
300 MHz
$CDCl_3$



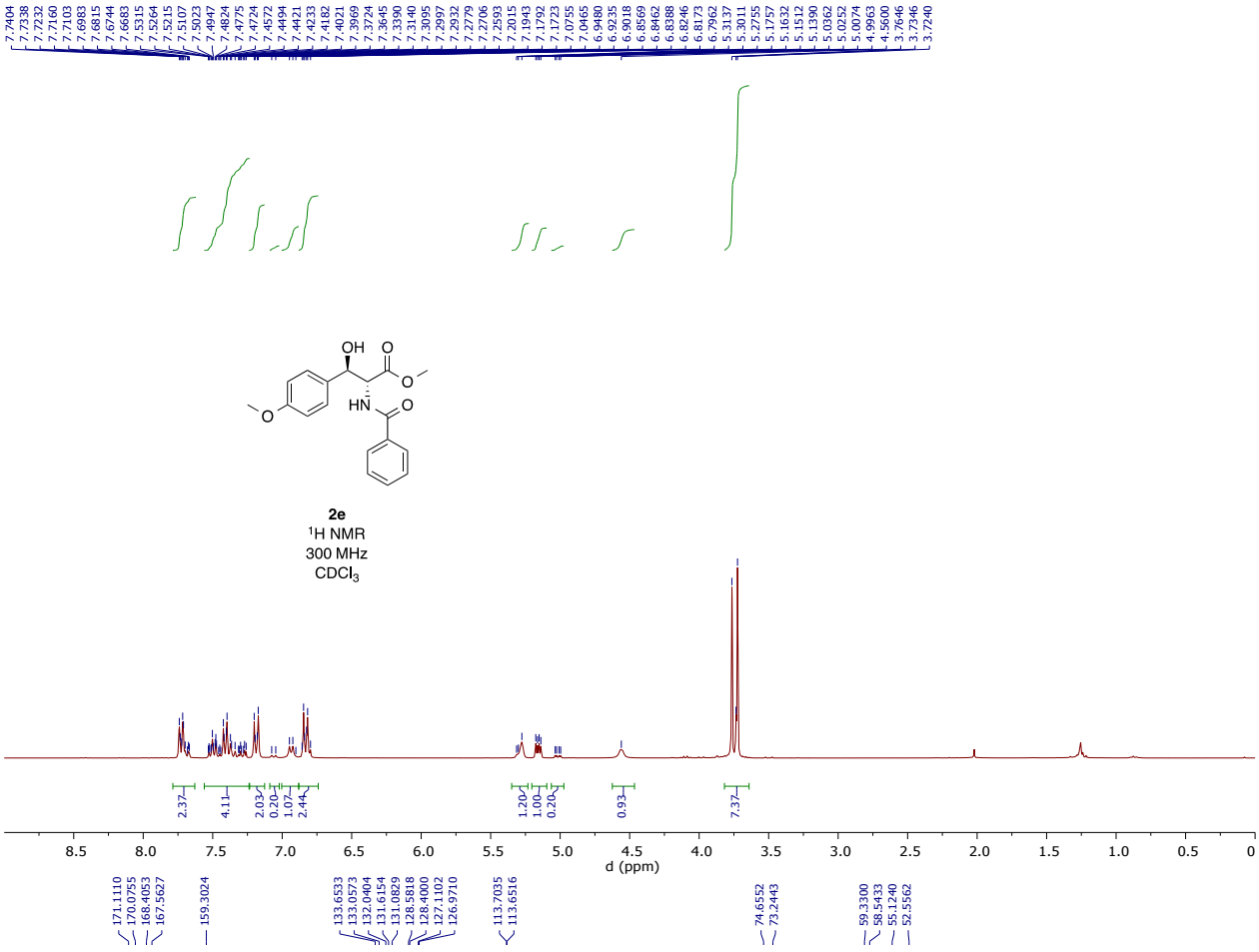
2c
^{13}C NMR
75 MHz
$CDCl_3$

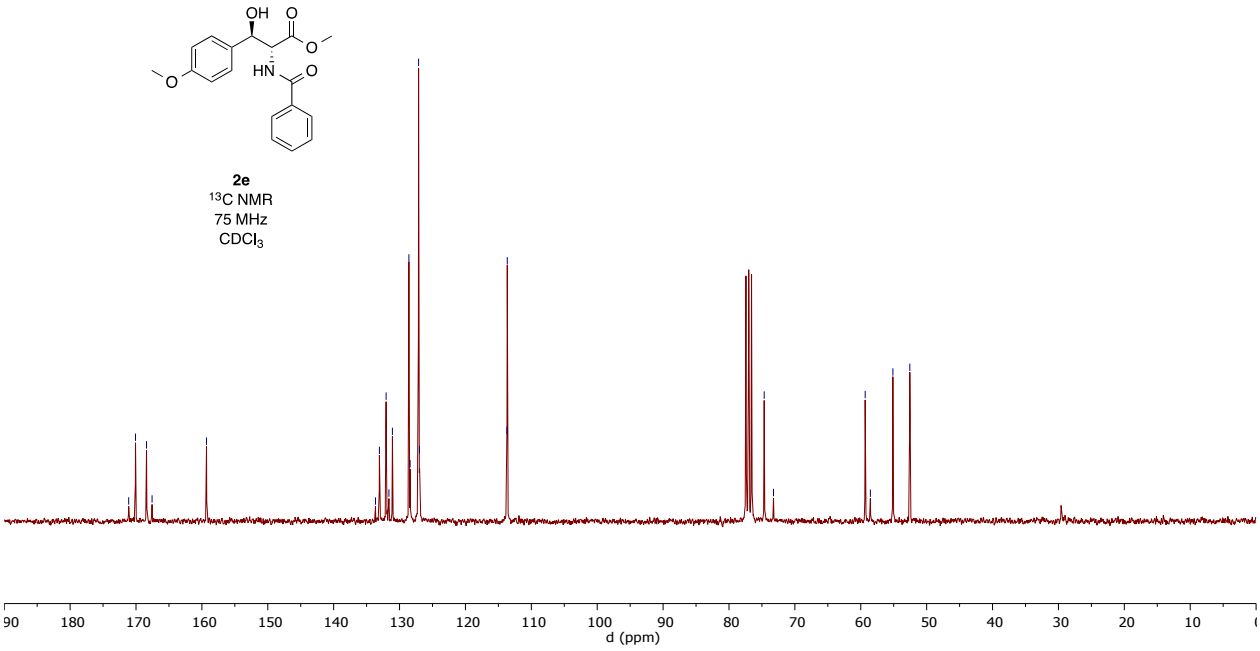


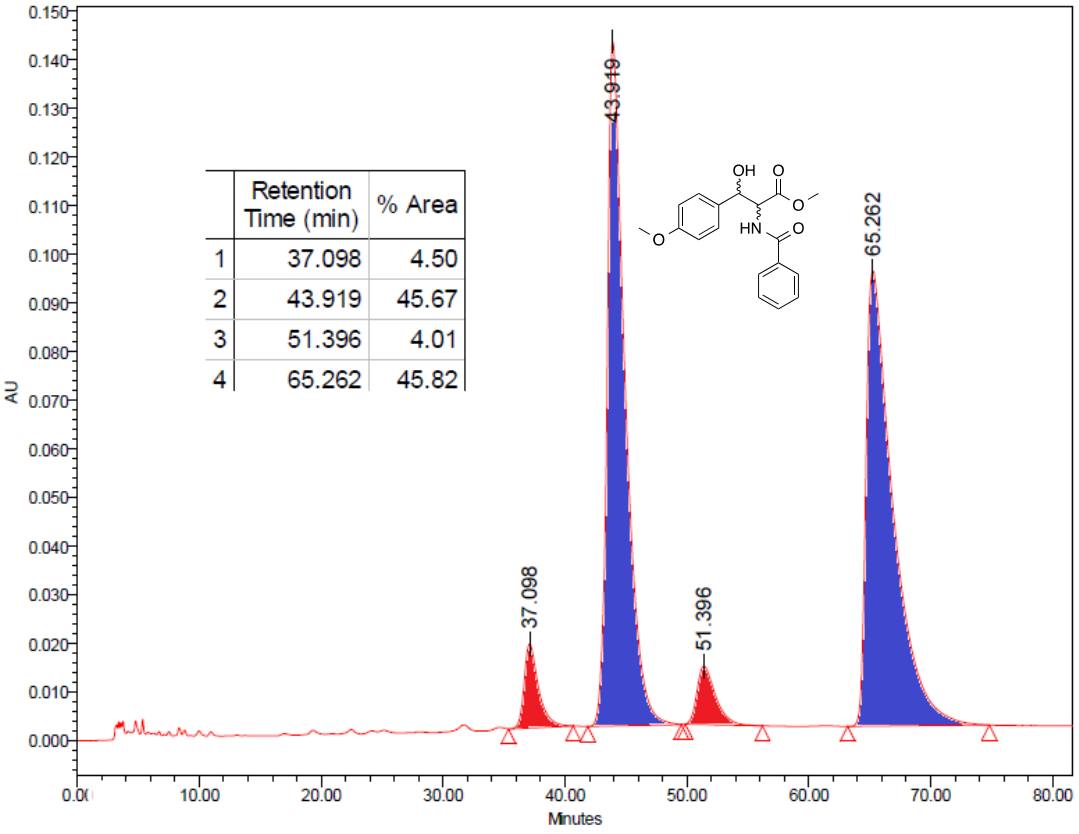
	Retention Time (min)	% Area
1	22.058	0.24
2	34.440	0.39
3	47.061	49.58
4	59.180	49.79



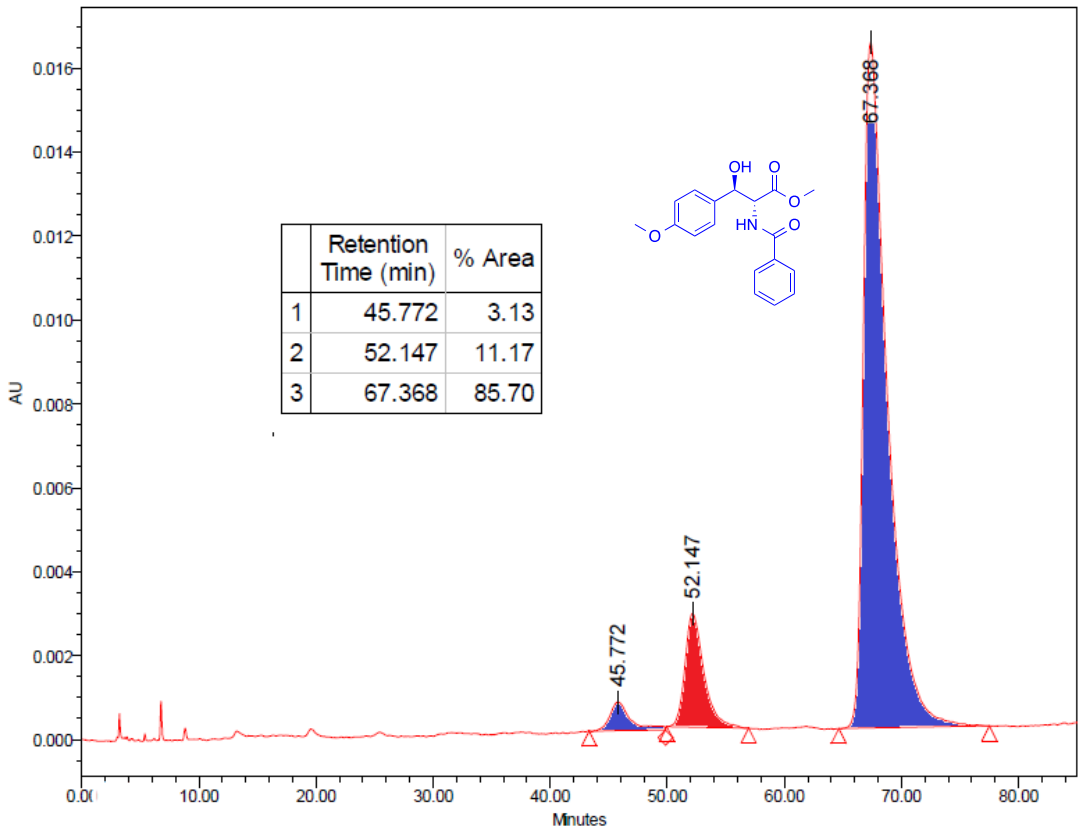
	Retention Time (min)	% Area
1	18.313	2.19
2	33.181	14.58
3	45.774	82.18
4	57.116	1.05


7.7404
7.7338
7.7232
7.7160
7.7103
7.6983
7.6815
7.6744
7.6683
7.5315
7.5264
7.5215
7.5107
7.5023
7.4947
7.4824
7.4775
7.4724
7.4572
7.4494
7.4421
7.4233
7.4182
7.4021
7.3969
7.3724
7.3645
7.3390
7.3140
7.3095
7.2997
7.2932
7.2779
7.2706
7.2593
7.2015
7.1943
7.1792
7.1723
7.0755
7.0465
6.9480
6.9235
6.9018
6.8569
6.8462
6.8388
6.8246
6.8173
6.7962
5.3137
5.3011
5.2755
5.1757
5.1632
5.1512
5.1390
5.0362
5.0252
5.0074
4.9963
4.5600
3.7646
3.7346
3.7240
OH
O
O
HN
O
O
2e
1H NMR
300 MHz
CDCl3
2.37
4.11
2.03
0.20
1.07
2.44
1.20
1.00
0.20
0.93
7.37
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
d (ppm)
171.1110
170.0755
168.4053
167.5627
159.3024
133.6533
133.0573
132.0404
131.6154
131.0829
128.5818
128.4000
127.1102
126.9710
113.7035
113.6516
74.6552
73.2443
59.3300
58.5433
55.1240
52.5562

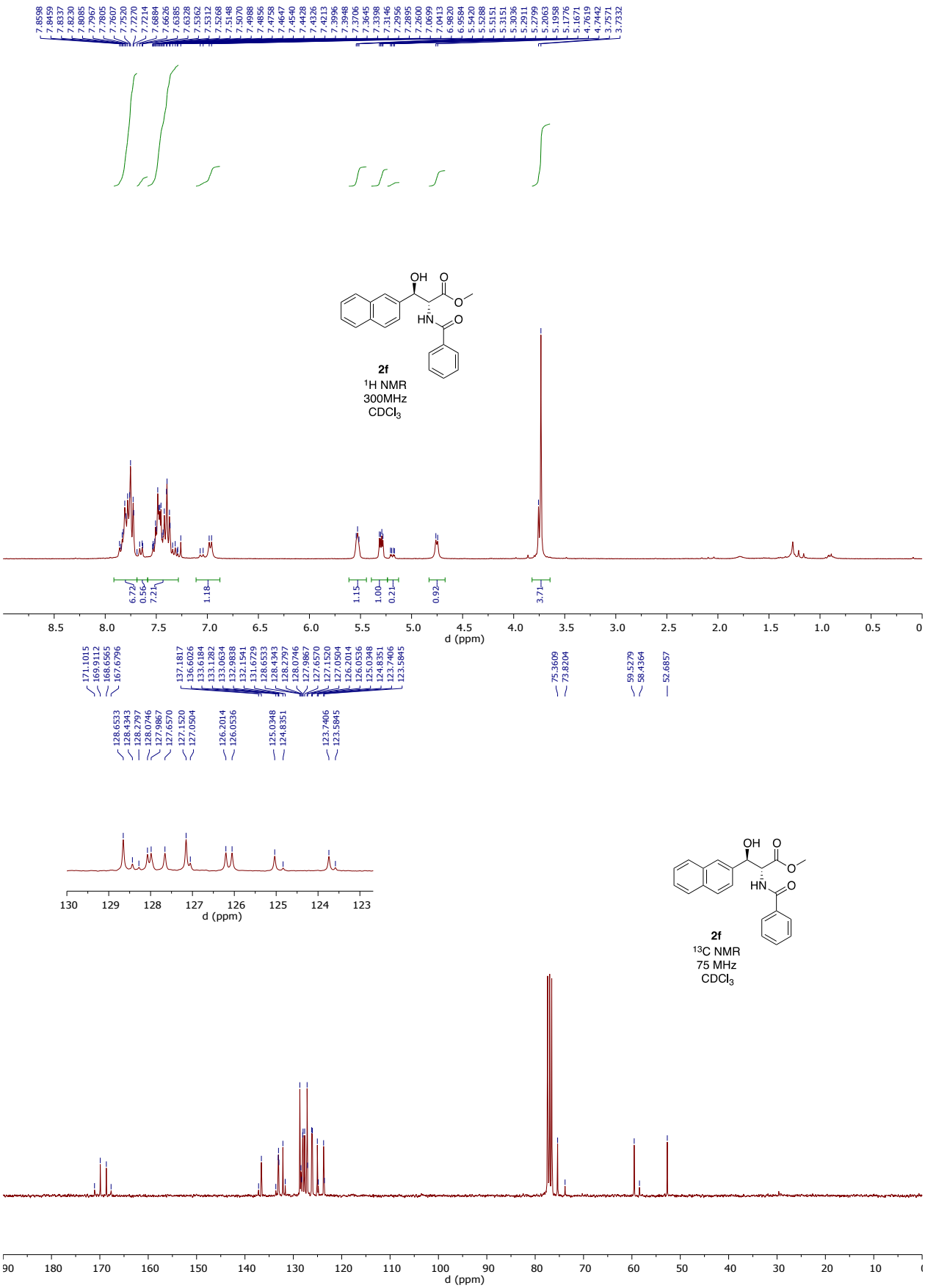

OH
O
O
HN
O
O
2e
13C NMR
75 MHz
CDCl3
90
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
d (ppm)

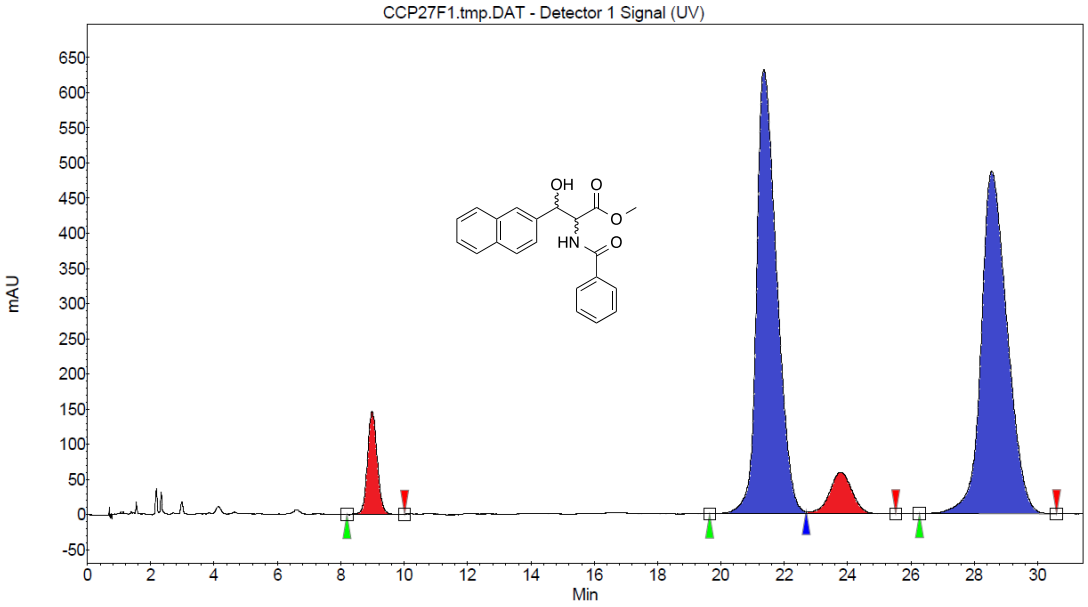


	Retention Time (min)	% Area
1	37.098	4.50
2	43.919	45.67
3	51.396	4.01
4	65.262	45.82

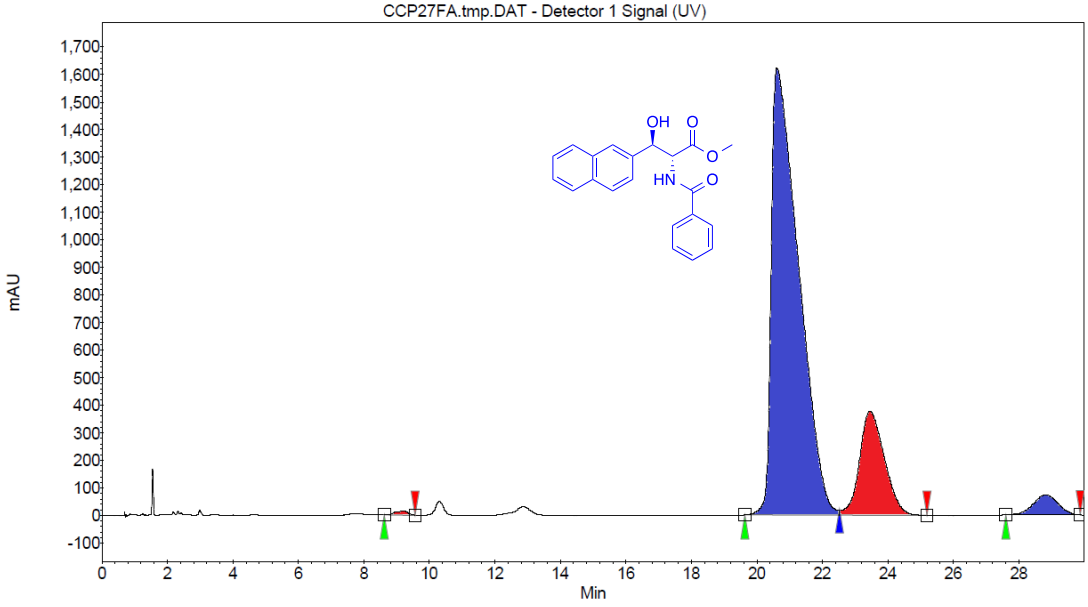


	Retention Time (min)	% Area
1	45.772	3.13
2	52.147	11.17
3	67.368	85.70

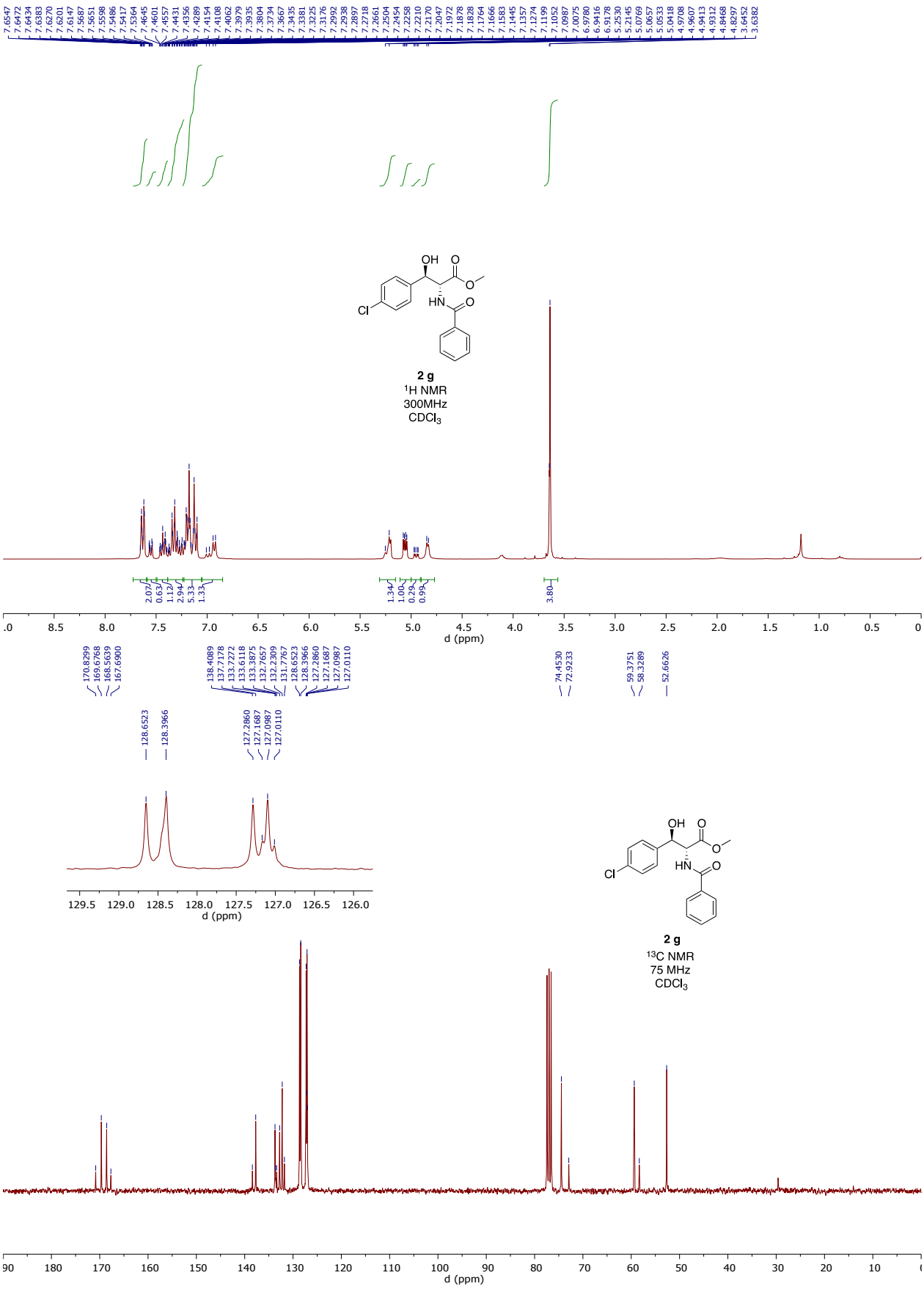

7.8598
7.8459
7.8337
7.8230
7.8085
7.7967
7.7805
7.7607
7.7520
7.7270
7.7214
7.6884
7.6626
7.6385
7.6328
7.5362
7.5312
7.5268
7.5148
7.5070
7.4988
7.4856
7.4758
7.4647
7.4540
7.4428
7.4326
7.4213
7.3996
7.3948
7.3706
7.3645
7.3398
7.3146
7.2956
7.2895
7.2600
7.0699
7.0413
6.9820
6.9584
5.5420
5.5288
5.5151
5.3151
5.3036
5.2911
5.2799
5.2063
5.1958
5.1776
5.1671
4.7619
4.7442
3.7571
3.7332
OH
O
O
HN
O
2f
1H NMR
300MHz
CDCl3
6.72
0.56
7.21
1.18
1.15
1.00
0.21
0.92
3.71
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
d (ppm)
171.1015
169.9112
168.6565
167.6796
137.1817
136.6026
133.6184
133.1282
133.0634
132.9838
132.1541
131.6729
128.6533
128.4343
128.2797
128.0746
127.9867
127.6570
127.1520
127.0504
126.2014
126.0536
125.0348
124.8351
123.7406
123.5845
75.3609
73.8204
59.5279
58.4364
52.6857
130
129
128
127
126
125
124
123
d (ppm)
OH
O
O
HN
O
2f
13C NMR
75 MHz
CDCl3
90
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
d (ppm)

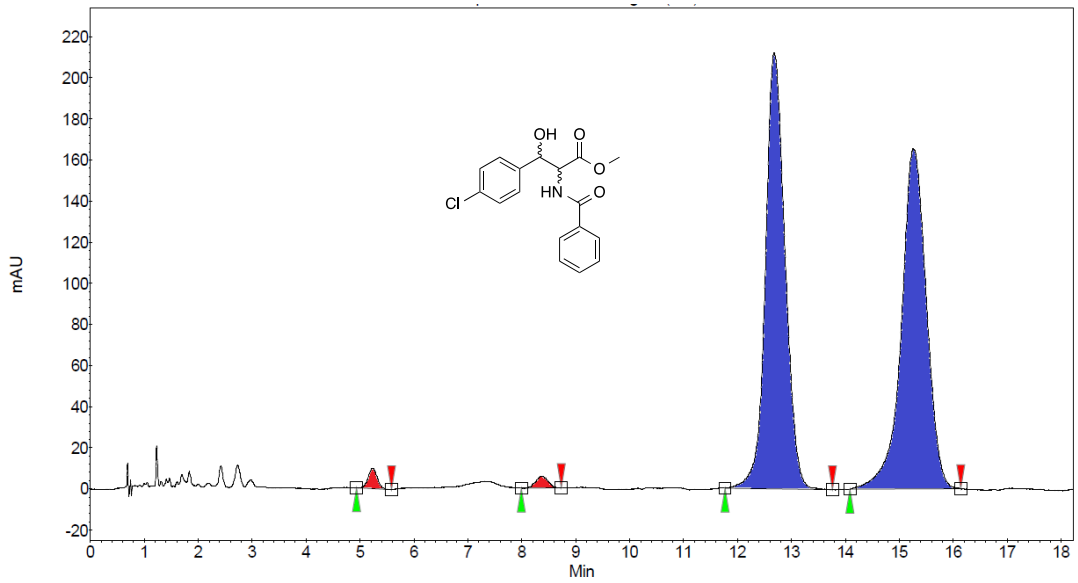


Index	Name	Time	Height	Area	Area	Selectivity	Res. HW
		[Min]	[µV]	[µV.Min]	[%]		
1	UNKNOWN	8.98	146.4	52.1	5.051	0.00	0.00
2	UNKNOWN	21.35	632.0	464.9	45.099	2.38	14.55
4	UNKNOWN	23.76	58.9	48.6	4.710	1.11	2.00
3	UNKNOWN	28.55	487.1	465.3	45.140	1.20	3.50
Total			1324.5	1030.8	100.000		

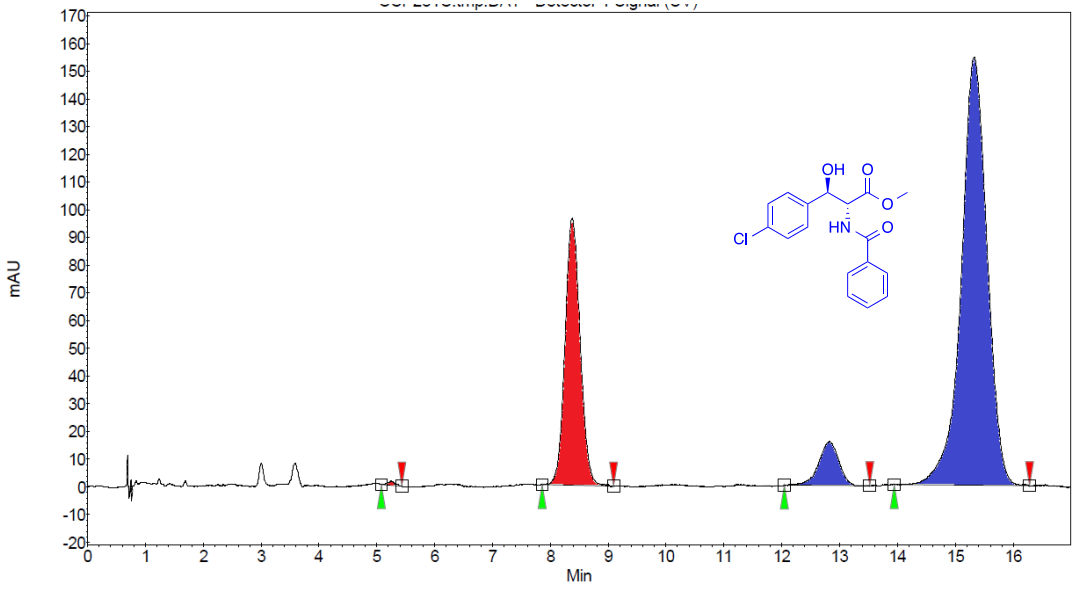


Index	Name	Time	Height	Area	Area	Selectivity	Res. HW
		[Min]	[µV]	[µV.Min]	[%]		
1	UNKNOWN	9.21	16.0	6.8	0.345	0.00	0.00
2	UNKNOWN	20.61	1622.5	1569.4	79.505	2.24	9.80
4	UNKNOWN	23.46	377.5	333.4	16.891	1.14	1.93
3	UNKNOWN	28.82	73.4	64.3	3.260	1.23	3.89
Total			2089.3	1973.9	100.000		

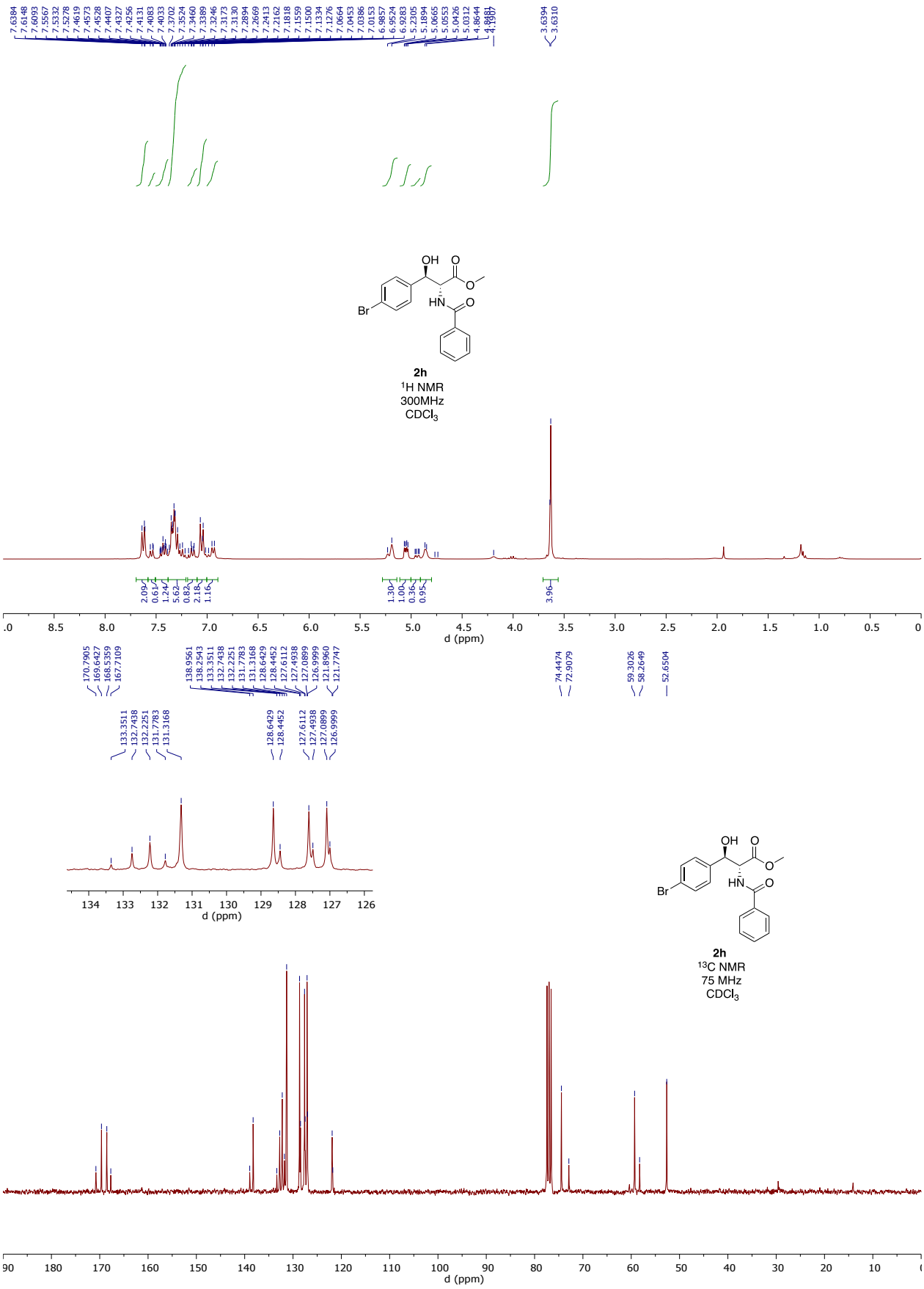

OH
O
O
HN
O
Cl
2 g
1H NMR
300MHz
CDCl3
d (ppm)
2 g
13C NMR
75 MHz
CDCl3
d (ppm)

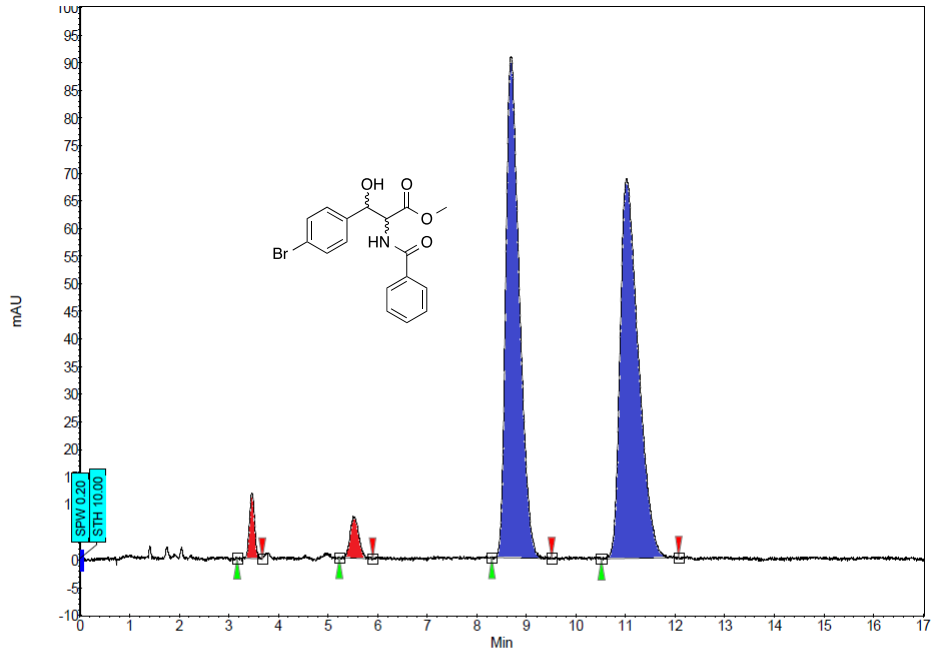


Index	Name	Time	Height	Area	Area	Selectivity	Res. HW
		[Min]	[μV]	[μV.Min]	[%]		
1	UNKNOWN	5.23	9.9	1.9	1.046	0.00	0.00
2	UNKNOWN	8.36	5.7	1.5	0.806	1.60	8.48
3	UNKNOWN	12.68	212.2	89.3	49.119	1.52	8.01
4	UNKNOWN	15.26	165.5	89.1	49.028	1.20	3.55
Total			393.3	181.8	100.000		



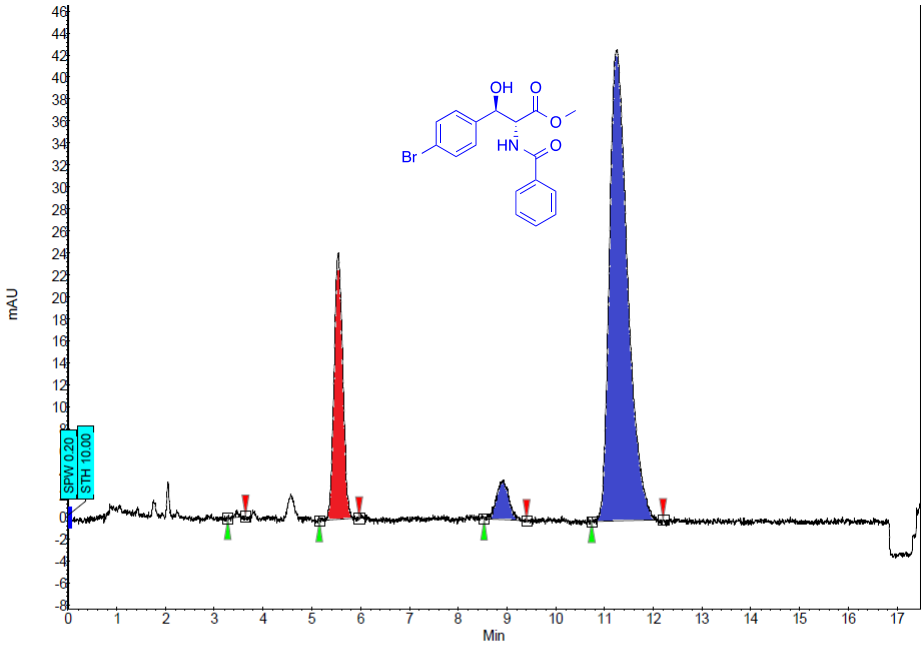
Index	Name	Time	Height	Area	Area	Selectivity	Res. HW
		[Min]	[μV]	[μV.Min]	[%]		
4	UNKNOWN	5.25	1.5	0.2	0.187	0.00	0.00
1	UNKNOWN	8.37	96.3	29.3	25.224	1.60	8.89
2	UNKNOWN	12.82	15.8	6.4	5.474	1.53	8.09
3	UNKNOWN	15.32	154.5	80.2	69.114	1.20	3.56
Total			268.0	116.1	100.000		


7.6384
7.6148
7.6093
7.5567
7.5532
7.5278
7.4619
7.4573
7.4528
7.4407
7.4327
7.4256
7.4131
7.4083
7.4033
7.3702
7.3524
7.3460
7.3389
7.3246
7.3173
7.3130
7.2894
7.2669
7.2413
7.2162
7.1818
7.1559
7.1500
7.1334
7.1276
7.0664
7.0453
7.0386
7.0153
6.9857
6.9524
6.9283
5.2305
5.1894
5.0665
5.0553
5.0426
5.0312
4.8644
3.6394
3.6310
OH
O
O
HN
O
Br
2h
1H NMR
300MHz
CDCl3
2.09
0.61
1.24
5.62
0.82
2.18
1.16
1.30
1.00
0.36
0.95
3.96
d (ppm)
170.7905
169.6427
168.5359
167.7109
138.9561
138.2543
133.3511
132.7438
132.2251
131.7783
131.3168
128.6429
128.4452
127.6112
127.4938
127.0899
126.9999
121.8960
121.7747
74.4474
72.9079
59.3026
58.2649
52.6504
d (ppm)
2h
13C NMR
75 MHz
CDCl3
d (ppm)



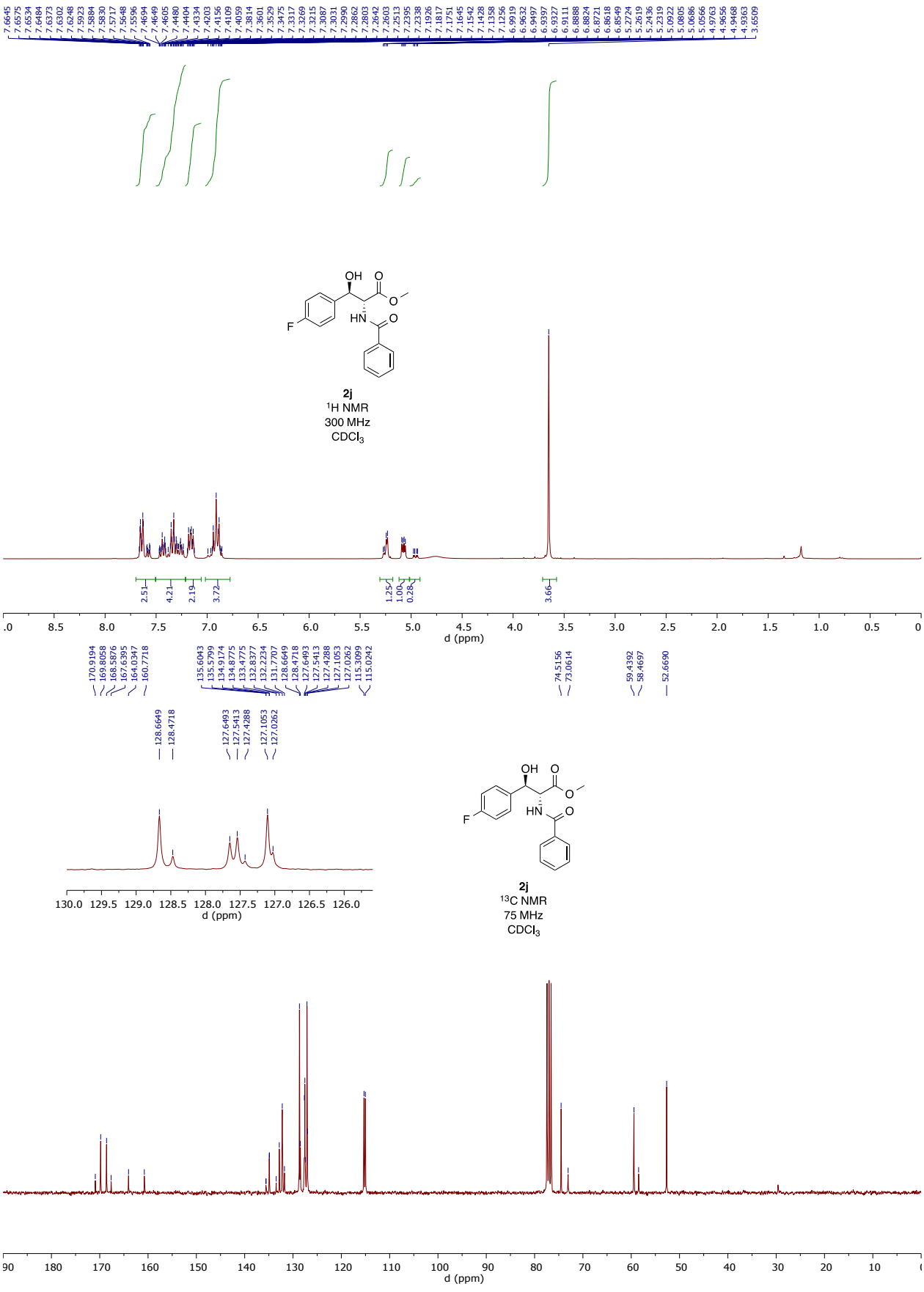
Results Table:

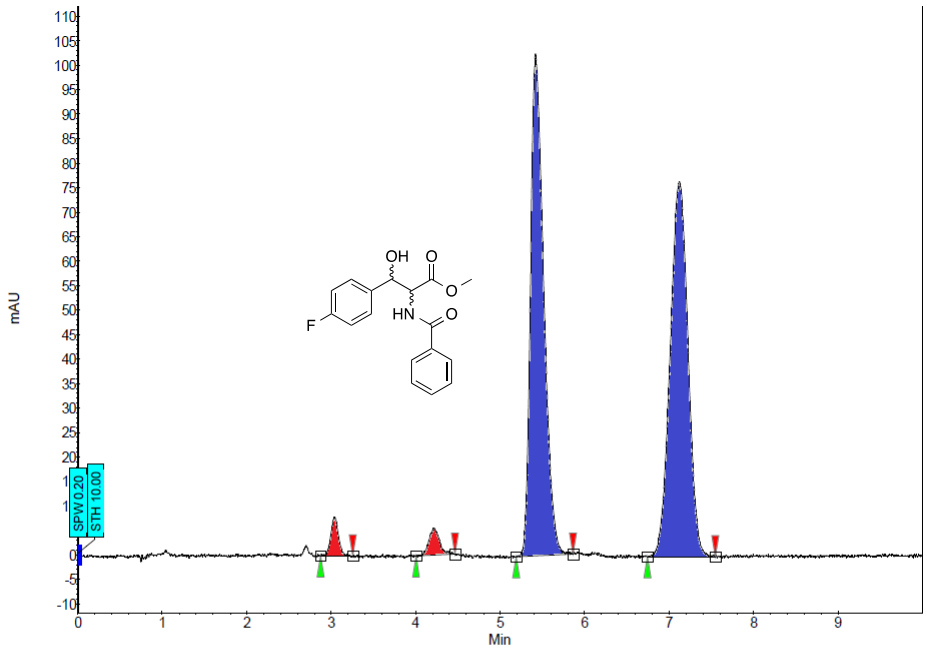
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
4	UNKNOWN	3.17	3.46	3.68	0.00	2.34	11.9	1.4	2.343
3	UNKNOWN	5.23	5.53	5.90	0.00	2.47	7.4	1.5	2.475
2	UNKNOWN	8.31	8.68	9.50	0.00	47.33	90.7	29.1	47.327
1	UNKNOWN	10.51	11.02	12.07	0.00	47.86	68.7	29.4	47.855
Total						100.00	178.6	61.4	100.000



Results Table:

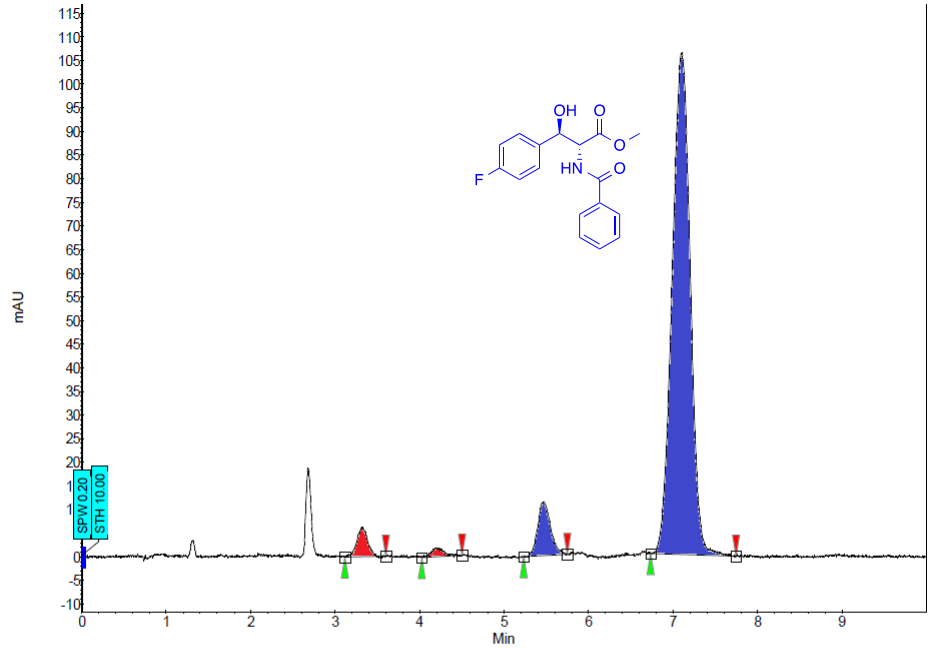
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	3.27	3.47	3.63	0.00	0.11	0.6	0.0	0.107
2	UNKNOWN	5.15	5.53	5.96	0.00	20.28	24.1	5.0	20.284
3	UNKNOWN	8.53	8.92	9.40	0.00	4.30	3.6	1.1	4.302
4	UNKNOWN	10.73	11.24	12.20	0.00	75.31	42.7	18.5	75.307
Total						100.00	71.0	24.5	100.000


2j
1H NMR
300 MHz
CDCl3
d (ppm)
2j
13C NMR
75 MHz
CDCl3
d (ppm)



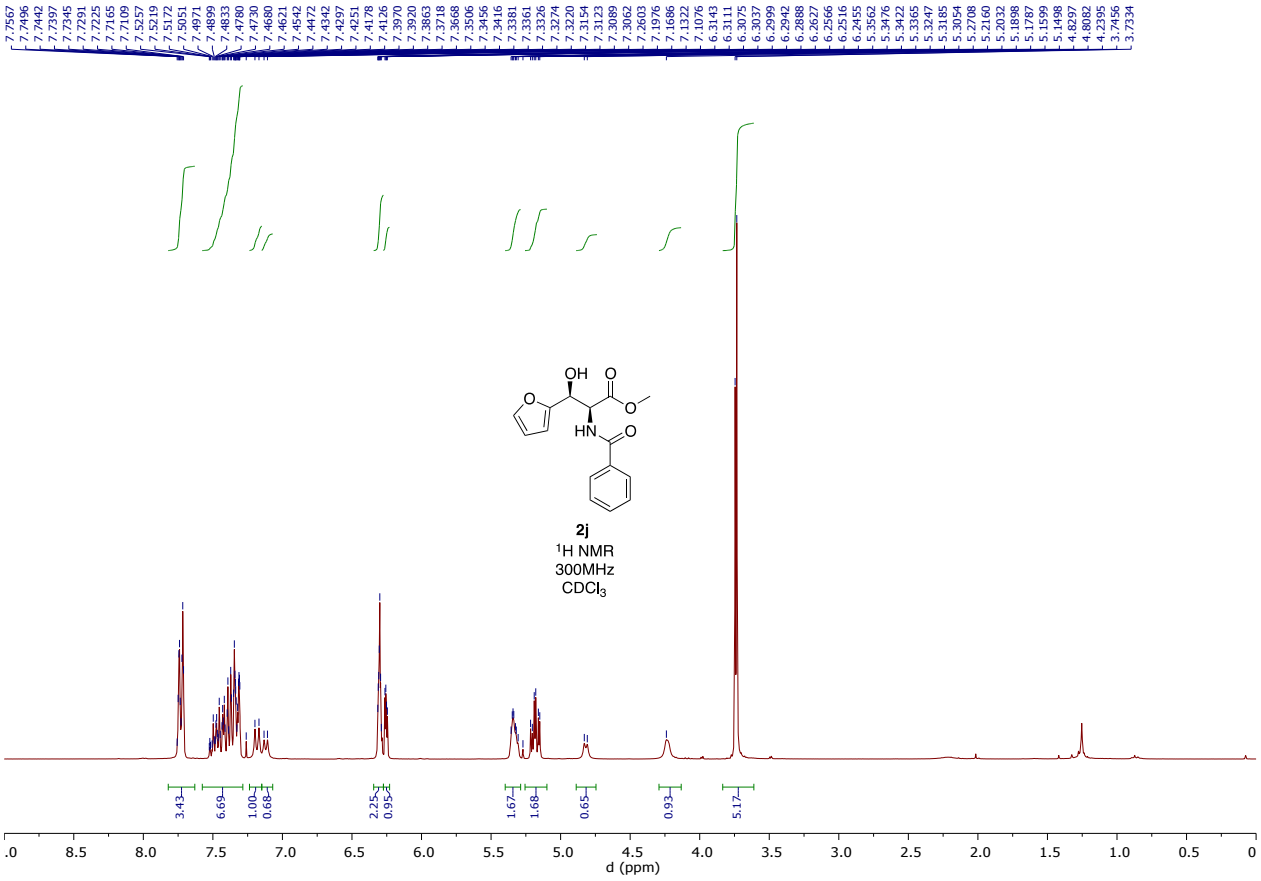
Results Table:

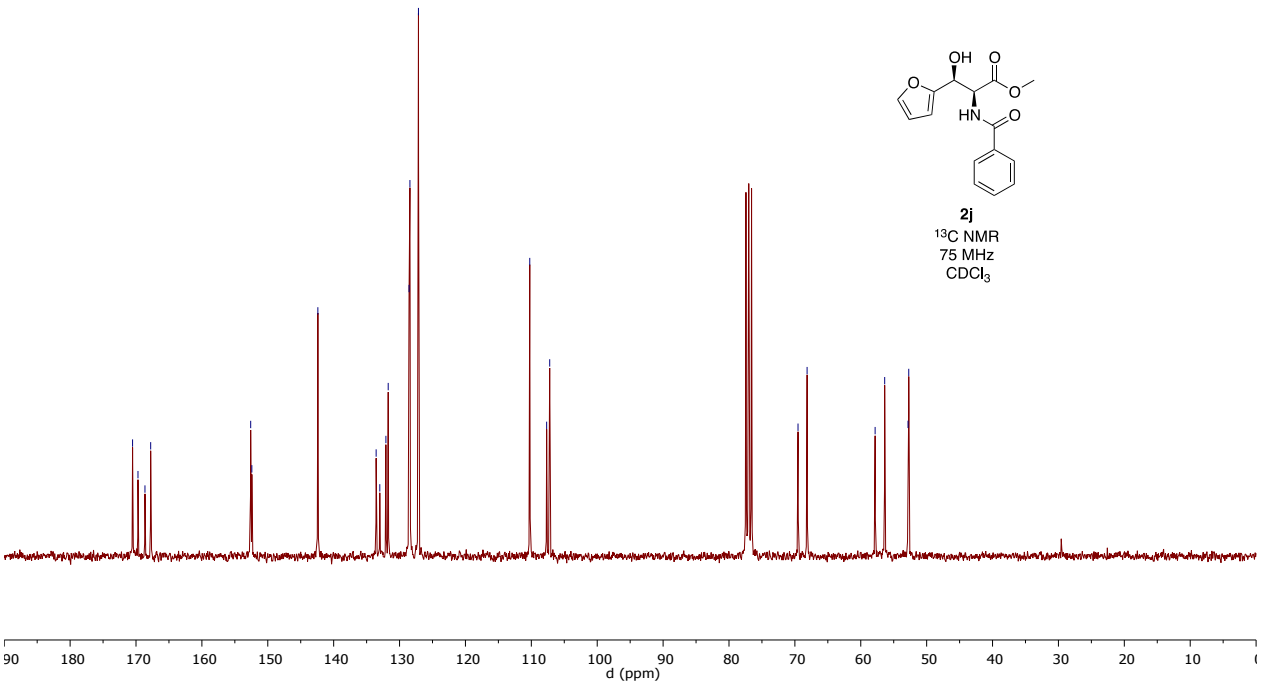
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	2.87	3.04	3.26	0.00	2.10	7.8	0.8	2.096
2	UNKNOWN	4.00	4.22	4.47	0.00	1.97	5.5	0.8	1.971
3	UNKNOWN	5.19	5.42	5.86	0.00	47.91	102.4	18.7	47.912
4	UNKNOWN	6.74	7.12	7.55	0.00	48.02	76.7	18.7	48.020
Total						100.00	192.3	39.0	100.000



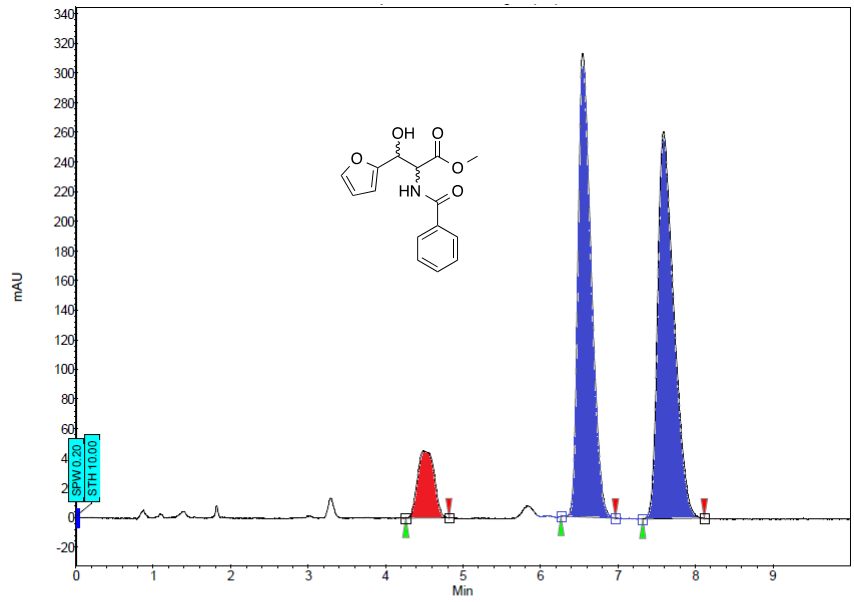
Results Table:

Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
4	UNKNOWN	3.11	3.31	3.60	0.00	2.99	6.1	0.9	2.985
3	UNKNOWN	4.02	4.20	4.50	0.00	1.01	1.6	0.3	1.008
2	UNKNOWN	5.23	5.46	5.75	0.00	6.78	11.3	2.0	6.775
1	UNKNOWN	6.73	7.10	7.74	0.00	89.23	106.1	26.5	89.232
Total						100.00	125.1	29.7	100.000


OH
O
O
O
HN
O
2j
1H NMR
300MHz
CDCl3
d (ppm)

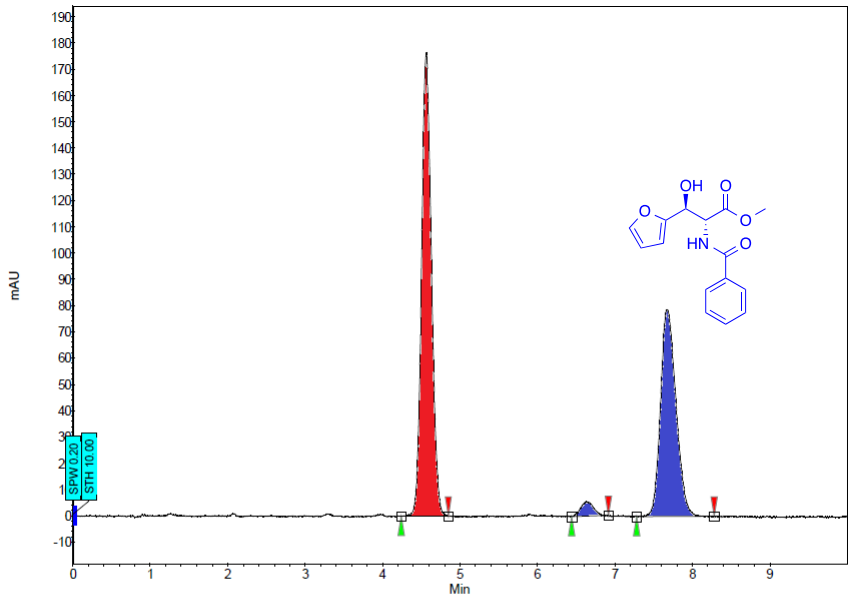

OH
O
O
O
HN
O
2j
13C NMR
75 MHz
CDCl3
d (ppm)

Measure of the enantiomeric excess for the *anti* compound :



Results Table:

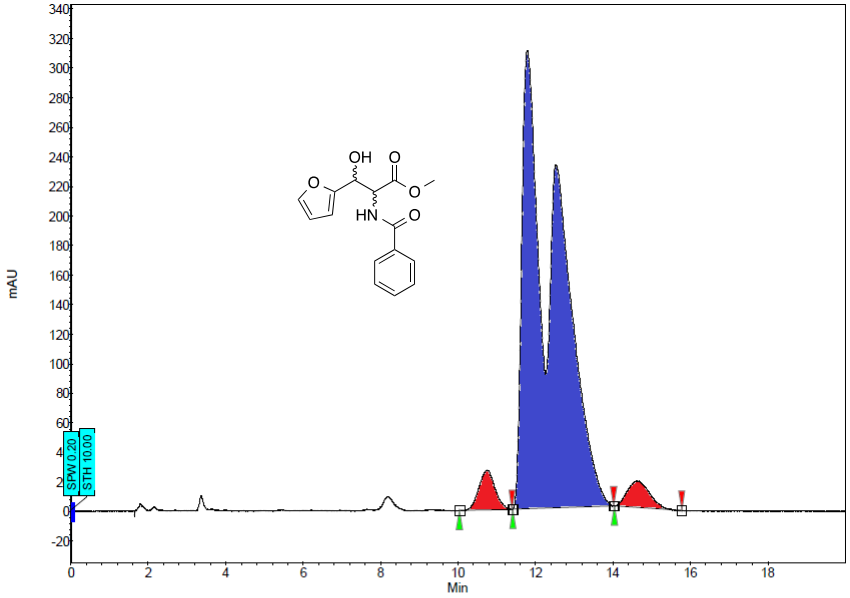
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	4.26	4.49	4.82	0.00	8.25	45.6	11.4	8.252
2	UNKNOWN	6.26	6.55	6.97	0.00	45.38	312.6	62.8	45.378
3	UNKNOWN	7.32	7.59	8.12	0.00	46.37	260.7	64.1	46.370
Total						100.00	618.9	138.3	100.000



Results Table:

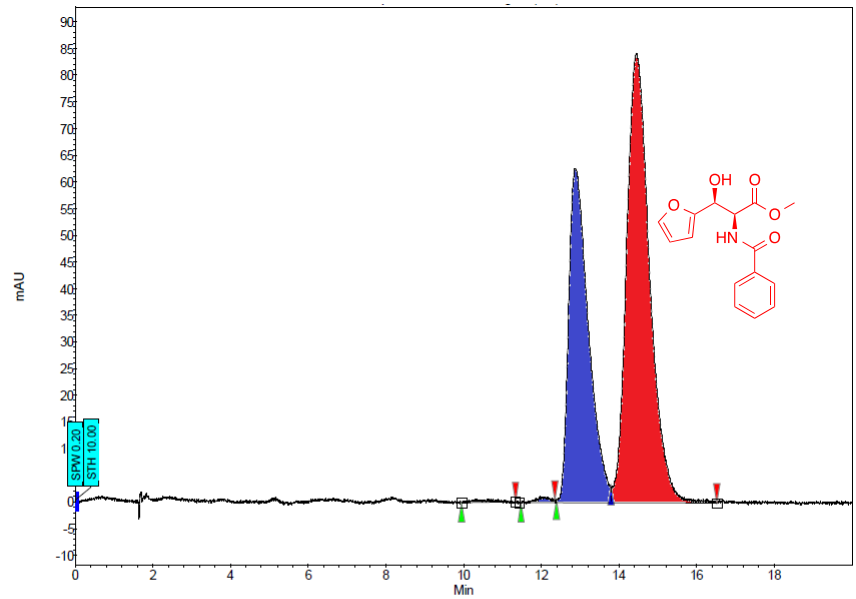
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	4.24	4.56	4.85	0.00	57.85	176.6	26.4	57.854
2	UNKNOWN	6.44	6.63	6.92	0.00	2.30	5.5	1.1	2.300
3	UNKNOWN	7.28	7.68	8.28	0.00	39.85	78.7	18.2	39.846
Total						100.00	260.8	45.7	100.000

Measure of the enantiomeric excess for the *syn* compound :



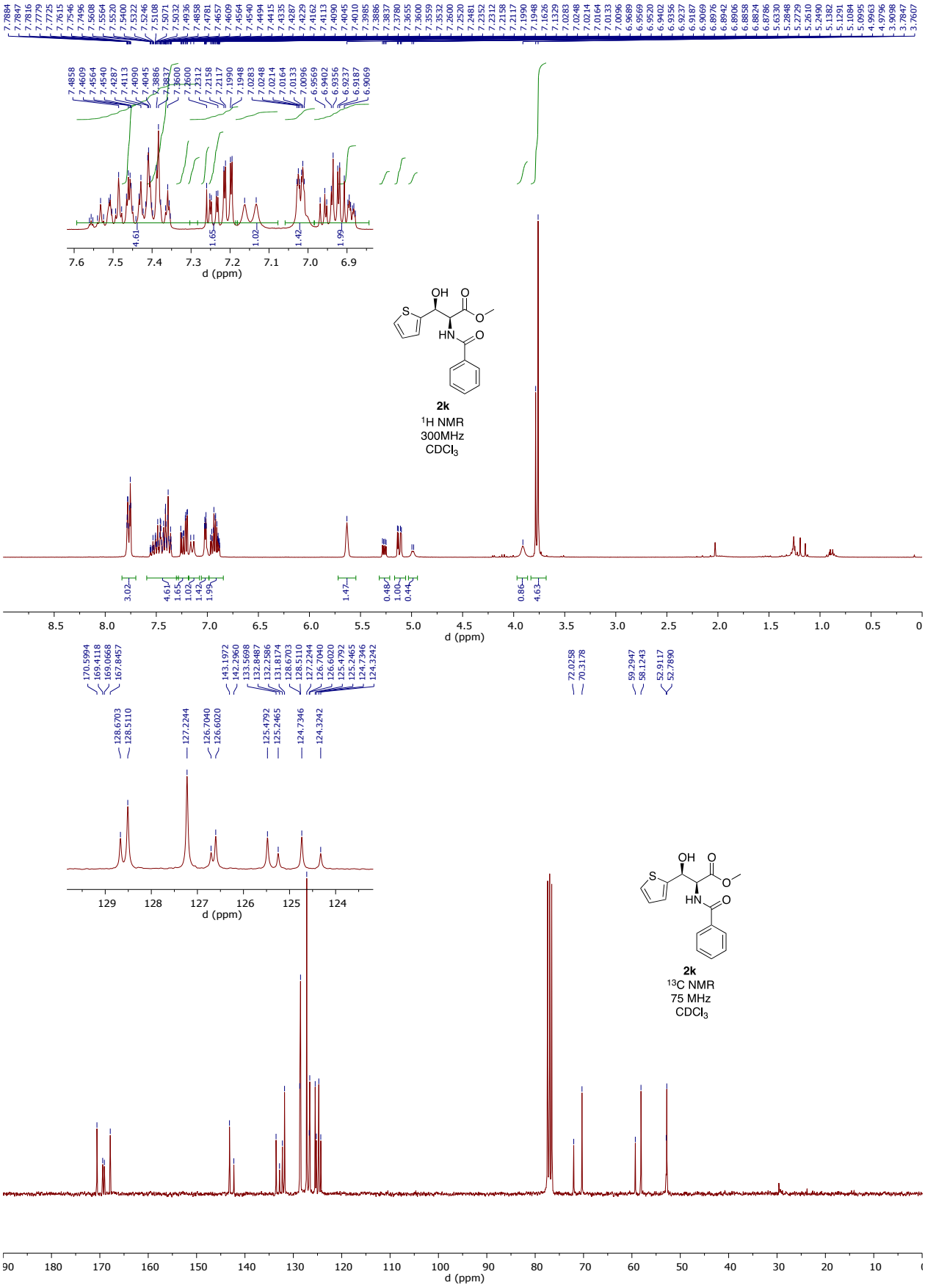
Results Table:

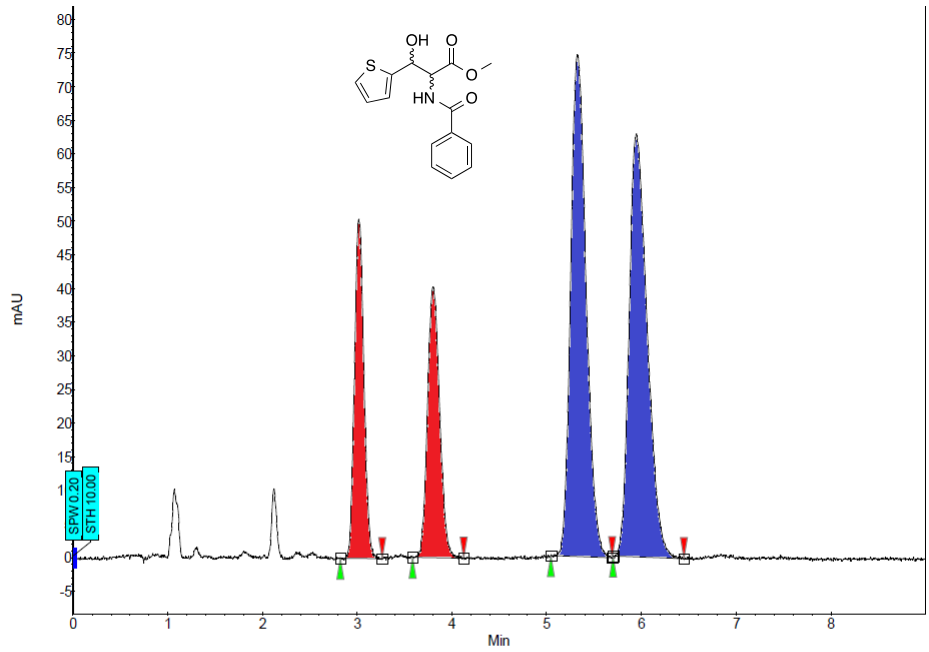
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	10.03	10.74	11.39	0.00	3.81	26.9	12.8	3.811
2	UNKNOWN	11.41	11.78	14.02	0.00	92.64	310.1	311.5	92.640
3	UNKNOWN	14.04	14.63	15.78	0.00	3.55	18.0	11.9	3.548
Total						100.00	355.0	336.2	100.000



Results Table:

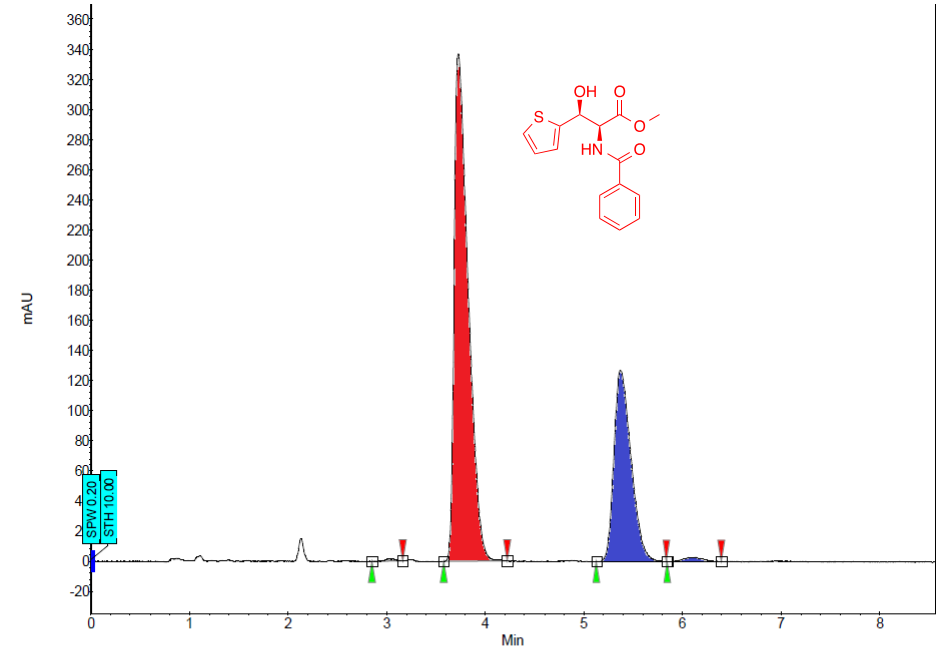
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
4	UNKNOWN	9.95	10.64	11.33	0.00	0.28	0.4	0.3	0.280
2	UNKNOWN	11.48	12.05	12.35	0.00	0.44	0.9	0.4	0.444
3	UNKNOWN	12.39	12.88	13.79	0.00	38.64	62.6	36.8	38.638
1	UNKNOWN	13.79	14.45	16.52	0.00	60.64	84.1	57.8	60.638
Total						100.00	147.9	95.4	100.000


2k
1H NMR
300MHz
CDCl3
d (ppm)
2k
13C NMR
75 MHz
CDCl3
d (ppm)



Results Table:

Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	2.82	3.02	3.27	0.00	14.38	50.4	5.7	14.378
2	UNKNOWN	3.58	3.80	4.12	0.00	14.89	40.3	5.9	14.889
3	UNKNOWN	5.04	5.32	5.69	0.00	35.48	74.5	14.1	35.483
4	UNKNOWN	5.70	5.95	6.45	0.00	35.25	62.9	14.0	35.249
Total						100.00	228.2	39.6	100.000



Results Table:

Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	2.85	3.03	3.16	0.00	0.23	1.4	0.2	0.229
2	UNKNOWN	3.58	3.73	4.22	0.00	67.85	336.9	55.5	67.849
3	UNKNOWN	5.13	5.37	5.84	0.00	31.22	127.0	25.5	31.224
4	UNKNOWN	5.84	6.09	6.39	0.00	0.70	2.8	0.6	0.698
Total						100.00	468.1	81.8	100.000