Supporting Information
Journal of Organic Chemistry

"Synthesis of Carbapyochelins via Diastereoselective Azidation of 5-(Ethoxycarbonyl)methylproline Derivatives"

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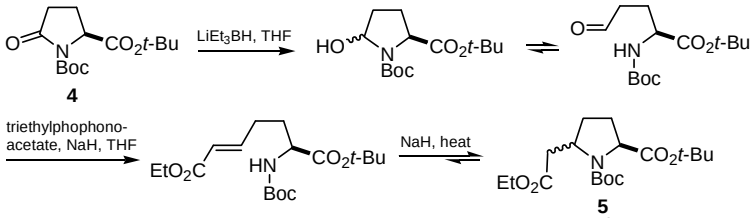
CONTENTS

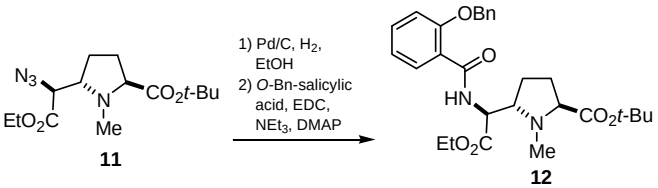
General. Unless stated otherwise, reactions were performed in flame-dried glassware under a positive pressure of argon using dry solvents. Commercial grade reagents and solvents were used without further purification except where noted. Diethyl ether, dichloromethane, and tetrahydrofuran were purified by solvent purification system. Other anhydrous solvents were purchased directly from chemical suppliers. Thin-layer chromatography (TLC) was performed using silica gel 60 F_{254} pre-coated plates (0.25 mm). Flash chromatography was performed using silica gel (40 μm particle size). The purity of all compounds was judged by TLC analysis (single spot/ two solvent systems) using a UV lamp or CAM or ninhydrin or basic $KMnO_4$ for detection purposes. NMR spectra were recorded on 300 MHz and 400 MHz spectrometers. ^1H and ^{13}C NMR chemical shifts are reported as δ using residual solvent as an internal standard. Analytical high performance liquid chromatography (HPLC) was performed C_{18} reverse phase analytical column. HPLC elution was carried out with a 20 min linear gradient of 0% - 90% MeCN in water (containing 0.1% formic acid buffer).



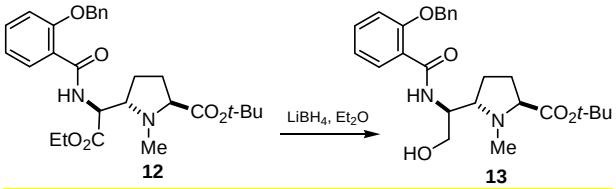
(2*S*,5*S*)-5-Ethoxycarbonylmethylpyrrolidine-1,2-dicarboxylic acid di-*tert*-butyl ester (5). A solution of **4** (3.00 g, 10.51 mmol) in THF (30 mL) was treated dropwise at -78 °C with $LiEt_3BH$ (1M solution in THF, 12.62 mL, 12.65 mmol), stirred at -78 °C for 30 min, and carefully quenched with sat. aq. $NaHCO_3$ (10 mL). The mixture was warmed to -15 °C and hydrogen peroxide (30% in water, 5.0 mL) was added dropwise. After stirring at 0 °C for 30 min, the solution was decanted from a white solid residue and concentrated. The crude material was taken up in Et_2O, washed with sat. aq. $NaHCO_3$, dried over $MgSO_4$, filtered, and evaporated under reduced pressure. The crude *N*-acyloxyaminal was used in the next step without further purification.

A suspension of sodium hydride (60% dispersion in mineral oil, 0.59 g, 14.72 mmol) in THF (30 mL) was treated dropwise with triethylphosphonoacetate (3.16 mL, 15.77 mmol) and stirred for 2 h at RT. The reaction was cooled to -15 °C and a solution of the above *N*-acyloxyaminal in THF (20 mL) was added dropwise. After the addition, the reaction was allowed to warm to RT and stirred 18 h, then treated with an additional 0.21 g of 60% NaH (5.26 mmol) and heated to 50 °C for 4 h, then cooled to RT and poured carefully into sat. aq. $NaHCO_3$ (failure to heat the reaction with additional NaH resulted in isolation of the ring-opened enoate as the major product). The mixture was extracted with EtOAc, dried over $MgSO_4$, fileterd, and evaporated under reduced pressure to furnish the crude diester. Purification by flash chromatography over silica gel (20% EtOAc/hexanes as eluant) gave **5** as white solid (3.16 g, 84% from **4**). ^1H NMR of crude **10** (*vide infra*) later revealed compound **5** to be a 16:1 trans:cis mixture of diastereomers. ^1H NMR (300MHz, $CDCl_3$) for the major trans isomer δ 4.41-4.25 (m, 1H), 4.20-4.08 (m, 3H), 2.97 and 2.87 (2dd, J = 15.2, 3.4 Hz, 1H, rotamers), 2.32-2.05 (m, 3H), 1.91 (q, J = 5.9 Hz, 1H), 1.74 (m, 1H), 1.47 and 1.43 (2s, 9H, rotamers), 1.44 and 1.42 (2s, 9H, rotamers), 1.25 (q, J = 7.0 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.9, 171.4, 154.0, 153.6,

90.0, 80.1, 79.9, 60.4, 60.3, 54.7, 39.4, 38.4, 28.4, 28.3, 28.0, 27.9, 14.2; ESI-MS (m/z) $[MH]^+$ calcd for $C_{18}H_{31}NO_6$ 358.22, found 358.35.

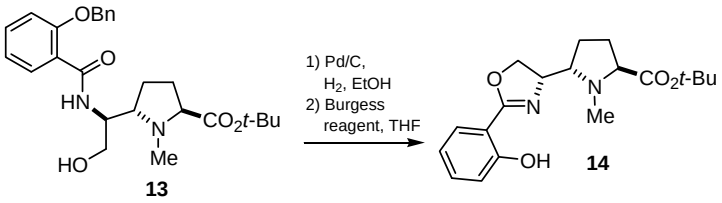


(2*S*,5*S*,1'*S*)-5-[(2-Benzyloxybenzoylamino)-ethoxycarbonylmethyl]-1-methylpyrrolidine-2-carboxylic acid *tert*-butyl ester (12): A solution of azide **11** (250 mg, 0.80 mmol) in EtOH (5 mL) was treated with a catalytic amount of 10% Pd/C and placed under H_2 atmosphere (balloon). After stirring at RT for 18 h, the mixture was purged with a stream of Ar and filtered through celite with EtOAc rinsing. Evaporation of the filtrate gave the intermediate diamine as a white solid (220 mg, 96% yield) which was taken up directly in CH_2Cl_2 (2mL) and treated with 2-benzyloxy-benzoic acid ethyl ester (200 mg, 0.90 mmol), EDC·HCl (180 mg, 0.96 mmol), NEt_3 (130 μL 0.96 mmol), and a catalytic amount of DMAP. The reaction was stirred 12 h at RT, concentrated, and the crude residue was purified by chromatography over silica gel (5-30% EtOAc/hexanes gradient as eluent) to give amide **12** as a sticky white foam (370 mg, 93% from **12**). ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.38 (d, J = 6.8 Hz, 1H), 8.23 (dd, J = 7.9, 2.1 Hz, 1H), 7.50-7.35 (m, 6H), 7.05 (m, 2H), 5.21 (m, 2H), 4.86 (dd, J = 6.7, 3.8 Hz, 1H), 4.19 (q, J = 7.0 Hz, 2H), 3.50 (ddd, J = 8.8, 5.0, 3.8 Hz, 1H), 3.37 (dd, J = 7.6, 1.5 Hz, 1H), 2.40 (s, 3H), 1.84-1.35 (m, 4H), 1.43 (s, 9H), 1.26 (t, J = 7.0 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 173.4, 171.2, 165.4, 157.0, 135.6, 132.8, 132.6, 128.9, 128.7, 128.2, 121.6, 121.4, 112.7, 80.8, 71.2, 67.2, 63.0, 61.0, 53.6, 34.8, 28.2, 27.5, 25.0, 14.2; HRMS (ESI-TOF) (m/z) $[MH]^+$ calcd for $C_{28}H_{36}N_2O_6$ 497.2646, found 497.2647.



(2*S*,5*S*,1'*S*)-5-[1-(2-Benzyloxybenzoylamino)-2-hydroxyethyl]-1-methylpyrrolidine-2-carboxylic acid *tert*-butyl ester (13). A solution of **12** (390 mg, 0.78 mmol) in Et_2O (4 mL) was treated with $LiBH_4$ (2M solution in THF, 580 μL, 1.16 mmol) and stirred for 12 h at RT. The reaction was quenched by addition of 1M NaOH (6 mL), stirred for an additional 10 min, then diluted with water and extracted with EtOAc. The combined organic layers were dried over Na_2SO_4, filtered, and evaporated and the crude residue purified by chromatography over silica gel (60% EtOAc/hexanes eluant) to give alcohol **13** as a sticky white foam (249 mg, 70%).^{1}H NMR (300 MHz, $CDCl_3$) δ 8.58 (d, J = 4.0 Hz, 1H), 8.19 (d, J = 8.0, 2.0 Hz, 1H), 7.41-7.34 (m, 6H), 7.06 (m, 1H), 6.98 (d, J = 8.4 Hz, 1H), 5.4 (bs, 1H), 5.23 (s, 2H), 4.04 (m, 1H), 3.78 (dd, J = 11.6, 5.6 Hz, 1H), 3.63 (dd, 11.6, 2.4 Hz, 1H), 3.42 (dd, J = 7.6, 1.6 Hz, 1H), 3.30 (ddd, J = 8.8, 5.6, 3.2 Hz, 1H), 2.31 (s, 3H), 2.00 (m, 1H), 1.84-1.62 (m, 2H), 1.49 (m, 1H), 1.43 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 172.9, 167.0, 156.6, 135.7, 132.8, 132.3, 128.9, 128.6, 127.4, 122.0,

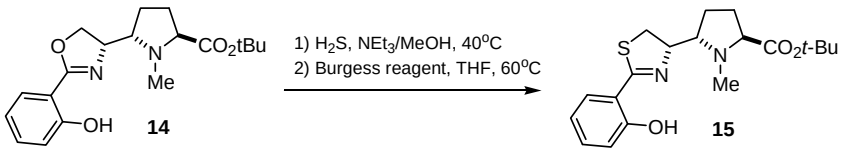
121.8, 121.5, 112.9, 81.1, 71.0, 67.3, 65.3, 63.6, 54.6, 35.4, 28.2, 27.6, 25.3; HRMS (ESI-TOF) (m/z) $[MH]^+$ calcd for $C_{26}H_{34}N_2O_5$ 455.2546, found 455.2550.



13 **14**

(2*S*,5*S*,4'*S*)-5-[2-(2-Hydroxyphenyl)-4,5-dihydrooxazol-4-yl]-1-methylpyrrolidine-2-carboxylic acid *tert*-butyl ester (14). A solution of **13** (190 mg, 0.42 mmol) in EtOH (2 mL) was treated with a catalytic amount of 10% Pd/C and placed under H_2 atmosphere (balloon). After stirring 12 h at RT, the mixture was purged with a stream of Ar, filtered over celite with EtOAc rinsing and evaporated to give the crude phenol intermediate. Purification by chromatography over silica gel (60% EtOAc/hexanes eluant) gave the pure phenol (120 mg, 78%). Data for (2*S*,5*S*,1'*S*)-5-[1-(2-Benzyloxybenzoylamino)-2-hydroxyethyl]-1-methylpyrrolidine-2-carboxylic acid *tert*-butyl ester: ^{1}H NMR (300 MHz, $CDCl_3$) δ 12.17 (s, 1H), 7.44-7.33 (m, 3H), 6.99 (dd, J = 8.0, 1.1 Hz, 1H), 6.86 (ddd, J = 8.0, 1.1 Hz, 1H), 3.97-3.92 (m, 2H), 3.75-3.68 (m. 2H), 3.52-3.47 (m, 1H), 2.28-2.01 (m, 2H), 1.94-1.87 (m, 1H), 1.72-1.62 (m, 1H), 1.47 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 172.5, 170.9, 161.4, 134.3, 125.6, 118.7, 114.24, 81.4, 67.5, 64.0, 63.9, 54.0, 36.1, 28.1, 28.0, 25.7; ESI-MS (m/z) $[MH]^+$ calcd for $C_{19}H_{28}N_2O_5$ 365.20, found 365.34.

A solution of the above phenol (130 mg, 0.36 mmol) in THF (2mL) was treated with 1.5 equiv. of Burgess reagent, stirred for 30min at RT, then at 60 °C for an additional 30min. The mixture was concentrated and the crude residue purified by chromatography over silica gel (60% EtOAc/hexanes eluant) to give **14** as a white solid (110 mg, 89%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 12.24 (s, 1H), 7.64 (dd, J = 7.9, 1.5 Hz, 1H), 7.37 (ddd, J = 8.5, 7.3, 1.8 Hz, 1H), 7.00 (m, 1H), 6.86 (ddd, J = 7.9, 7.3, 1.2 Hz, 1H), 4.49 (m, 2H), 4.22 (m, 1H), 3.65 (dd, J = 7.9, 1.8 Hz, 1H), 3.34 (m, 1H), 2.48 (s, 3H), 2.22 (m, 1H), 2.02 (m, 1H), 1.78 (ddt, 12.9, 8.8, 2.1 Hz, 1H), 1.58 (m, 1H), 1.47 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 173.6, 165.7, 159.9, 133.3, 128.0, 118.6, 116.7, 110.5, 80.8, 69.0, 68.7, 68.5, 65.4, 36.7, 28.2, 28.1, 26.7; mp = 75-78 °C; HRMS (ESI-TOF) (m/z) $[MH]^+$ calcd for $C_{19}H_{26}N_2O_4$ 347.1965, found 347.1974.

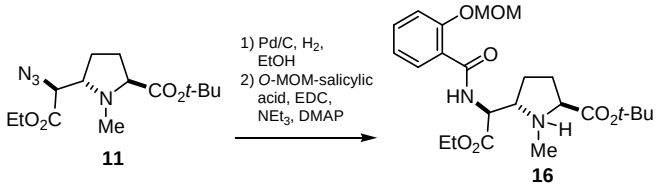


14 **15**

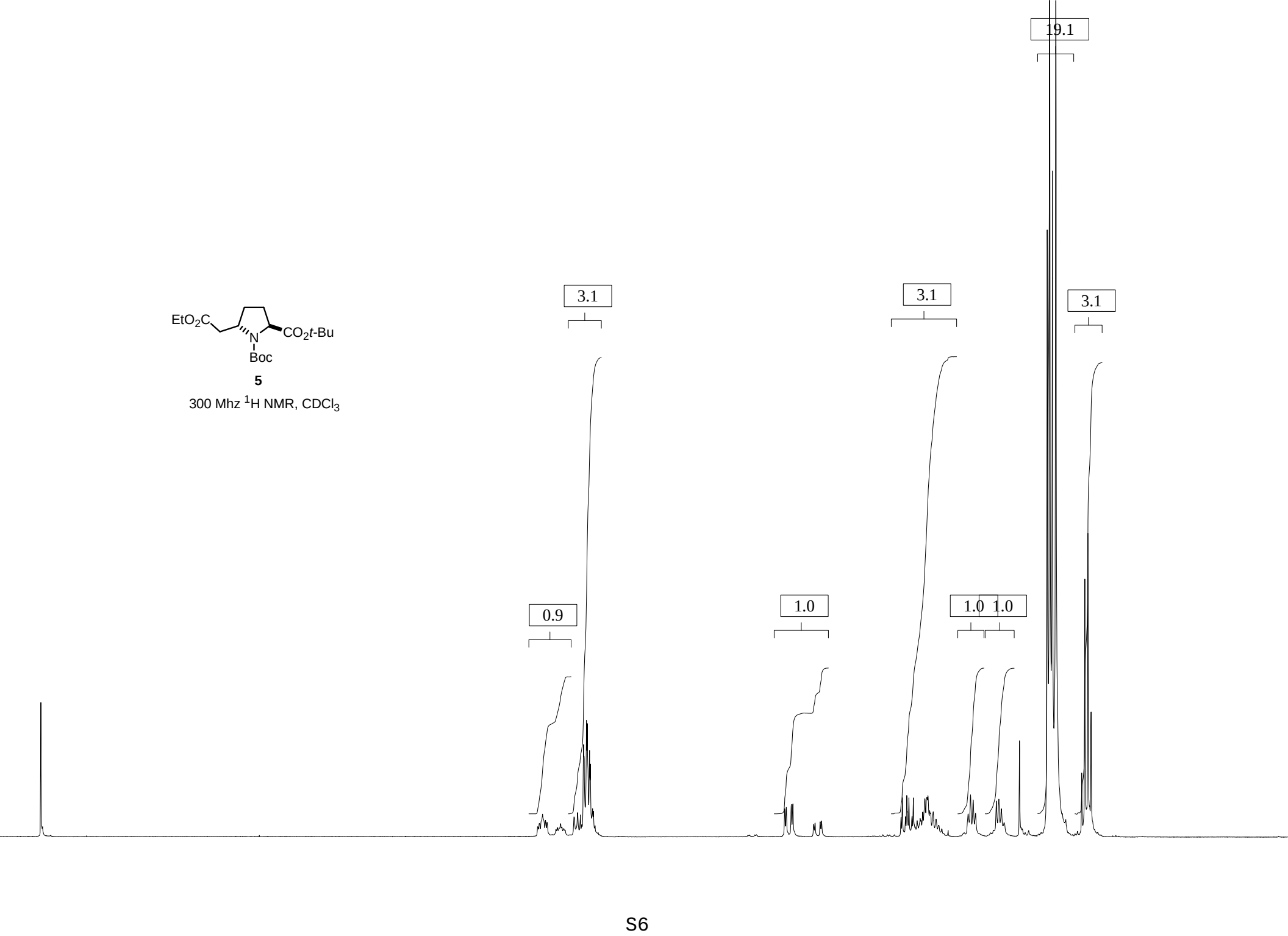
(2*S*,5*S*,4'*S*)-5-[2-(2-Hydroxyphenyl)-4,5-dihydrothiazol-4-yl]-1-methylpyrrolidine-2-carboxylic acid *tert*-butyl ester (15). A solution of **14** (120 mg, 0.35 mmol) was dissolved in 10 mL of 1:1 NEt_3:MeOH at RT in a pressure flask. Hydrogen sulfide gas was then bubbled through the stirring solution for 20 min, after which the pressure flask was closed and warmed to 40 °C. The reaction was monitored periodically by LCMS and stirred at 40 °C for 5d (> 90% consumption of starting material). The reaction was then purged of H_2S by bubbling a stream of

Ar through the solution for 15 min. Concentration and drying under vacuum gave a yellow solid which was used directly in the subsequent cyclodehydration.

The crude thioamide was taken up in 4 mL of THF, treated with Burgess reagent (124 mg, 0.52 mmol) and heated to 60 ^{o}C. After 1 hr, LCMS indicated complete consumption of the thioamide intermediate. The reaction was cooled to room temperature and filtered through a short silica pad with EtOAc rinsing. The filtrate was concentrated in vacuo and the crude residue purified by chromatography over silica gel (15% EtOAc/hexanes eluant) to give **15** as an off-white solid (48 mg, 38% from **14**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.80 (bs, 1H), 7.40-7.30 (m, 2H), 6.98 (dd, J = 8.2, 0.8 Hz, 1H), 6.86 (m, 1H), 4.81 (ddd, J = 10.2, 9.0, 3.1 Hz, 1H), 3.67 (dd, J = 7.8, 1.6 Hz, 1H), 3.52 (dt, J = 9.4, 3.9 Hz), 3.34 (dd, J = 10.5, 8.6 Hz, 1H), 3.21 (t, J = 10.5, 1H), 2.51 (s, 3H), 2.25 (dq, J = 12.9, 9.4 Hz, 1H), 2.03 (m, 1H), 1.79 (ddt, J = 12.9, 9.0, 2.0 Hz, 1H), 1.67 (m, 1H), 1.47 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.9, 171.5, 159.4, 133.0, 130.6, 118.9, 117.2, 116.6, 81.0, 79.6, 68.6, 66.0, 36.7, 33.0, 28.5, 28.4, 27.0; mp = 133 ^{o}C; HRMS (ESI-TOF) (m/z) $[MH]^+$ calcd for $C_{19}H_{26}N_2O_3S$ 363.1737, found 363.1753.



(2*S*,5*S*,1'*S*)-5-[(2-Methoxymethyloxybenzoylamino)-ethoxycarbonylmethyl]-1-methylpyrrolidine-2-carboxylic acid *tert*-butyl ester (16): Amide **16** was prepared from **11** (700 mg, 2.23 mmol) following the procedure described for the synthesis of **12**, and using 2-methoxymethyloxybenzoic acid in place of 2-benzyloxybenzoic acid. The crude amide was purified by chromatography over silica gel (35% EtOAc/hexane eluant) to give **16** as a white solid (850 mg, 84% from **11**). ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.47 (d, J = 5.3 Hz, 1H), 8.19 (dd, J = 7.9, 1.8 Hz, 1H), 7.43 (ddd, J = 8.5, 7.3, 1.8 Hz, 1H), 7.13 (m, 2H), 5.31 (m, 2H), 4.79 (dq, J = 7.0, 1.5 Hz, 2H), 3.65 (d, J = 6.7 Hz, 1H), 3.58 (m, 1H), 3.54 (s, 3H), 2.50 (s, 3H), 2.19-1.80 (m, 4H), 1.48 (s, 9H), 1.29 (t, J = 7.0 Hz, 3H); 13C NMR (75 MHz, $CDCl_3$) δ 173.2, 171.2, 165.3, 155.4, 132.8, 132.3, 122.3, 122.0, 114.6, 95.0, 81.1, 67.6, 62.9, 61.1, 56.6, 54.0, 35.0, 28.2, 27.6, 25.4, 14.2; mp = 79-81 ^{o}C; HRMS (ESI-TOF) (m/z) [MH+] calcd for $C_{23}H_{34}N_2O_7$ 451.2444, found 451.2431.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N
Boc
5
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1.0
1.0
7.0
6.5
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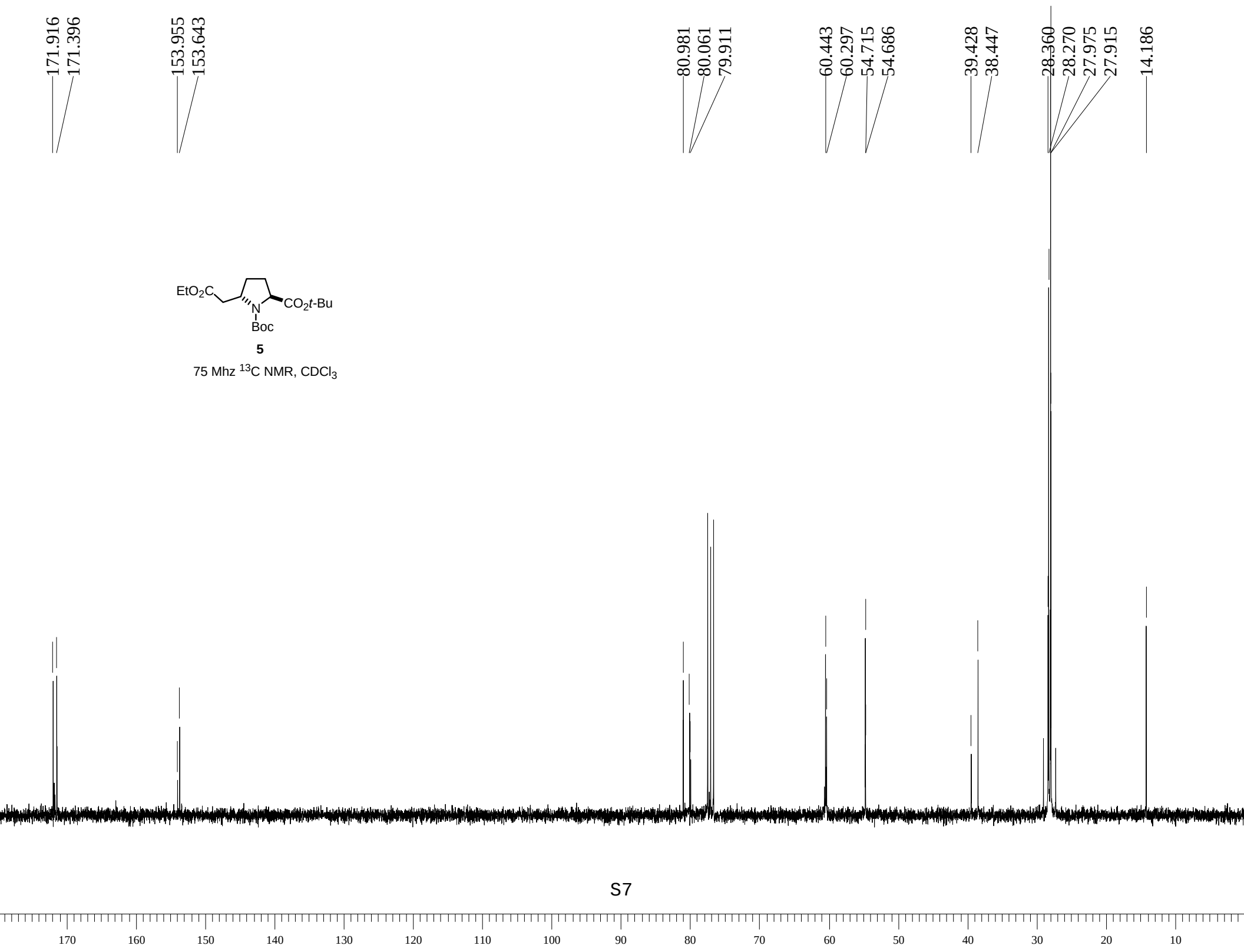
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38.447
28.360
28.270
27.975
27.915
14.186
EtO2C
CO2t-Bu
N
Boc
5
75 Mhz 13C NMR, CDCl3
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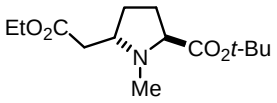
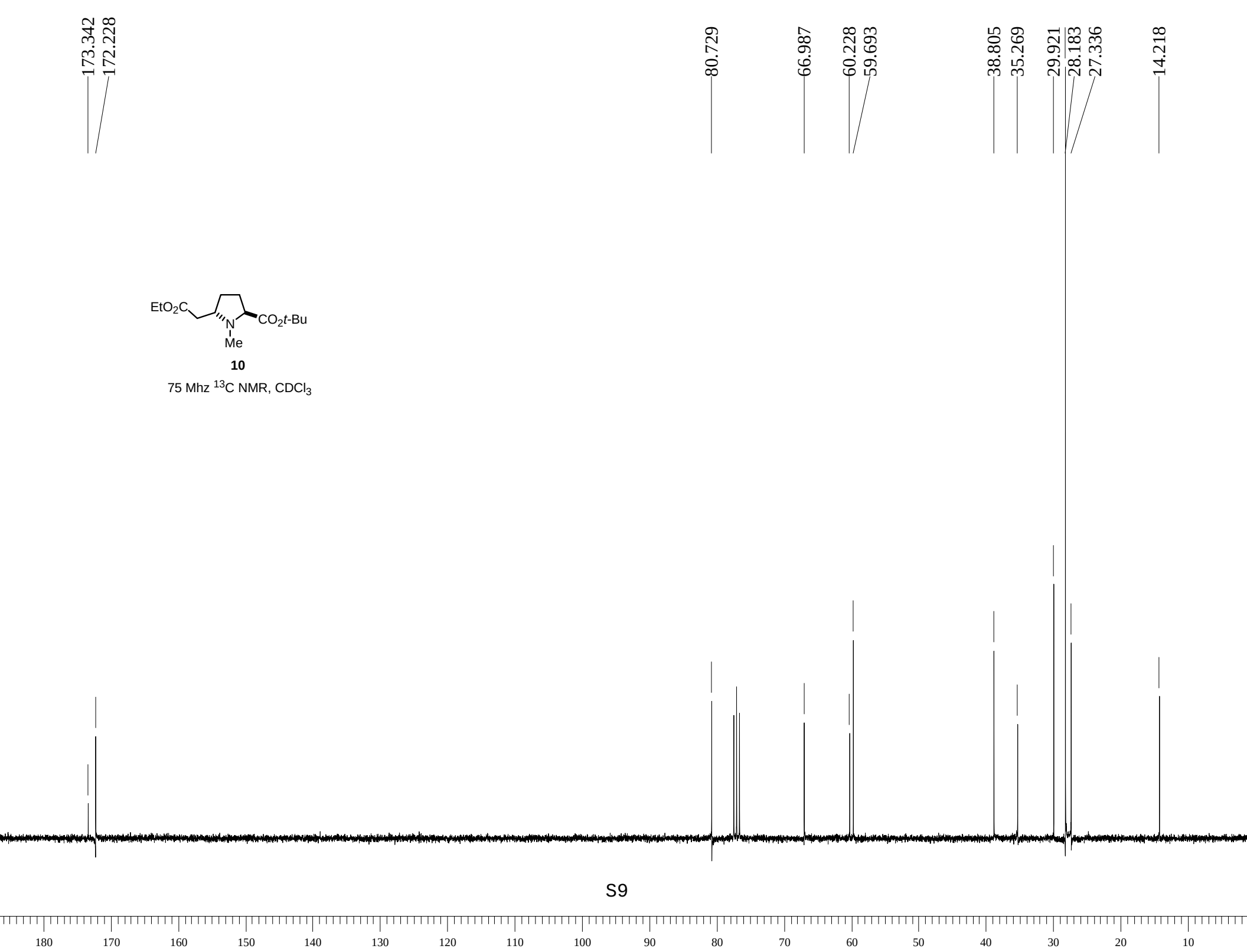
20
10



10

300 Mhz ^{1}H NMR, $CDCl_3$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59.693
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27.336
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EtO2C
CO2t-Bu
N
Me
10
75 Mhz 13C NMR, CDCl3
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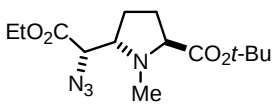
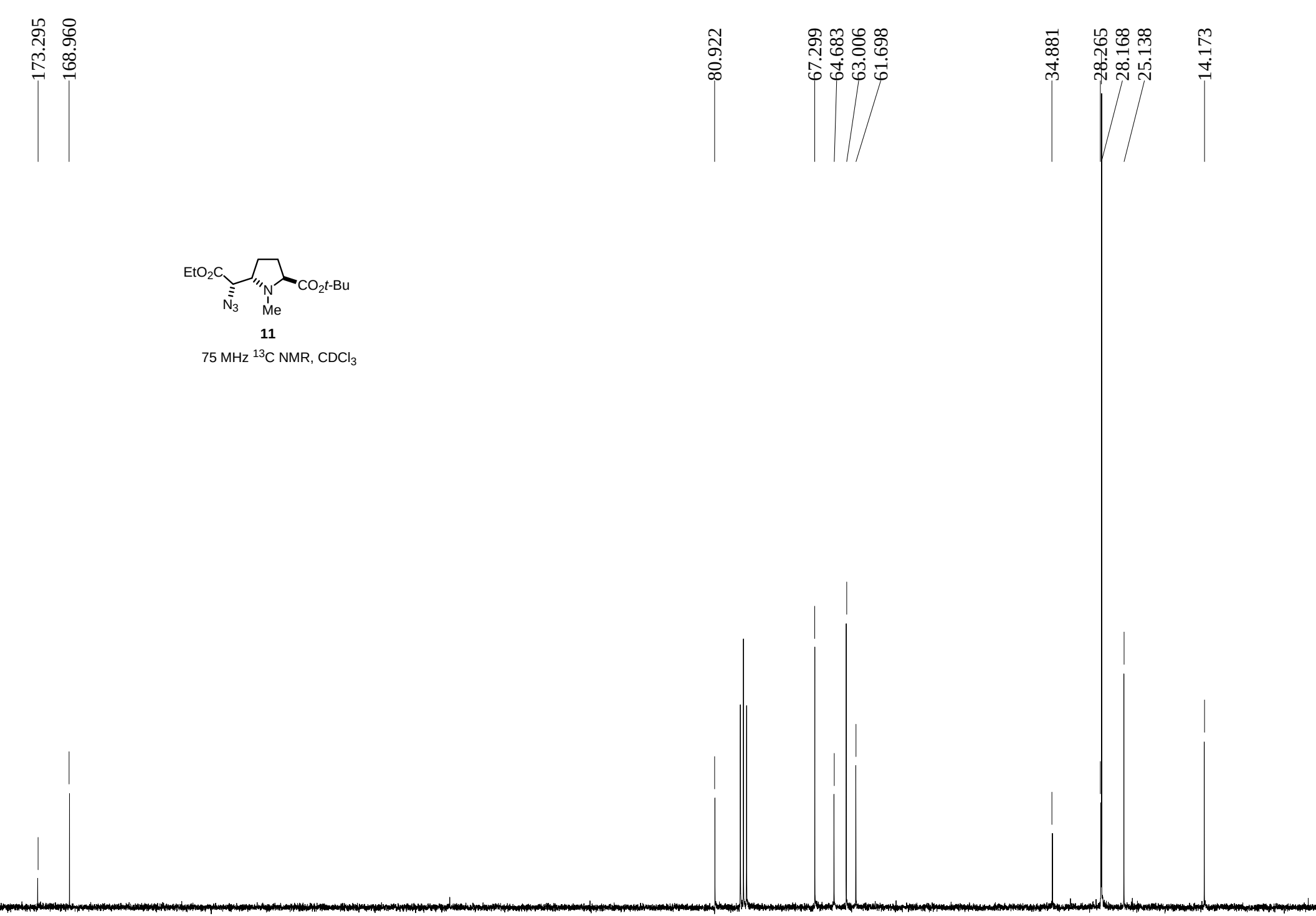


11

300 Mhz ^{1}H NMR, $CDCl_3$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67.299
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CO2t-Bu
N3
N
Me
11
75 MHz 13C NMR, CDCl3
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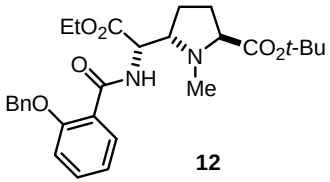
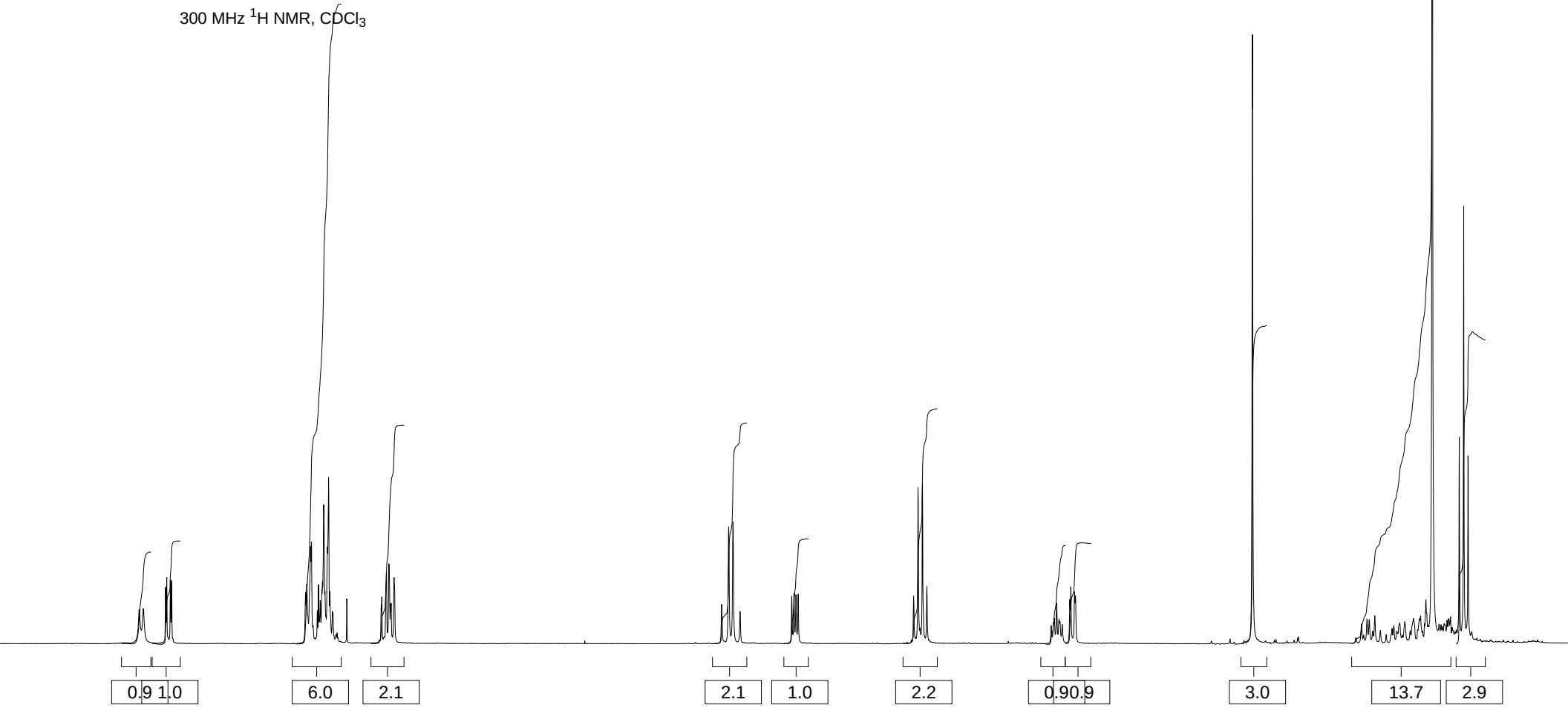
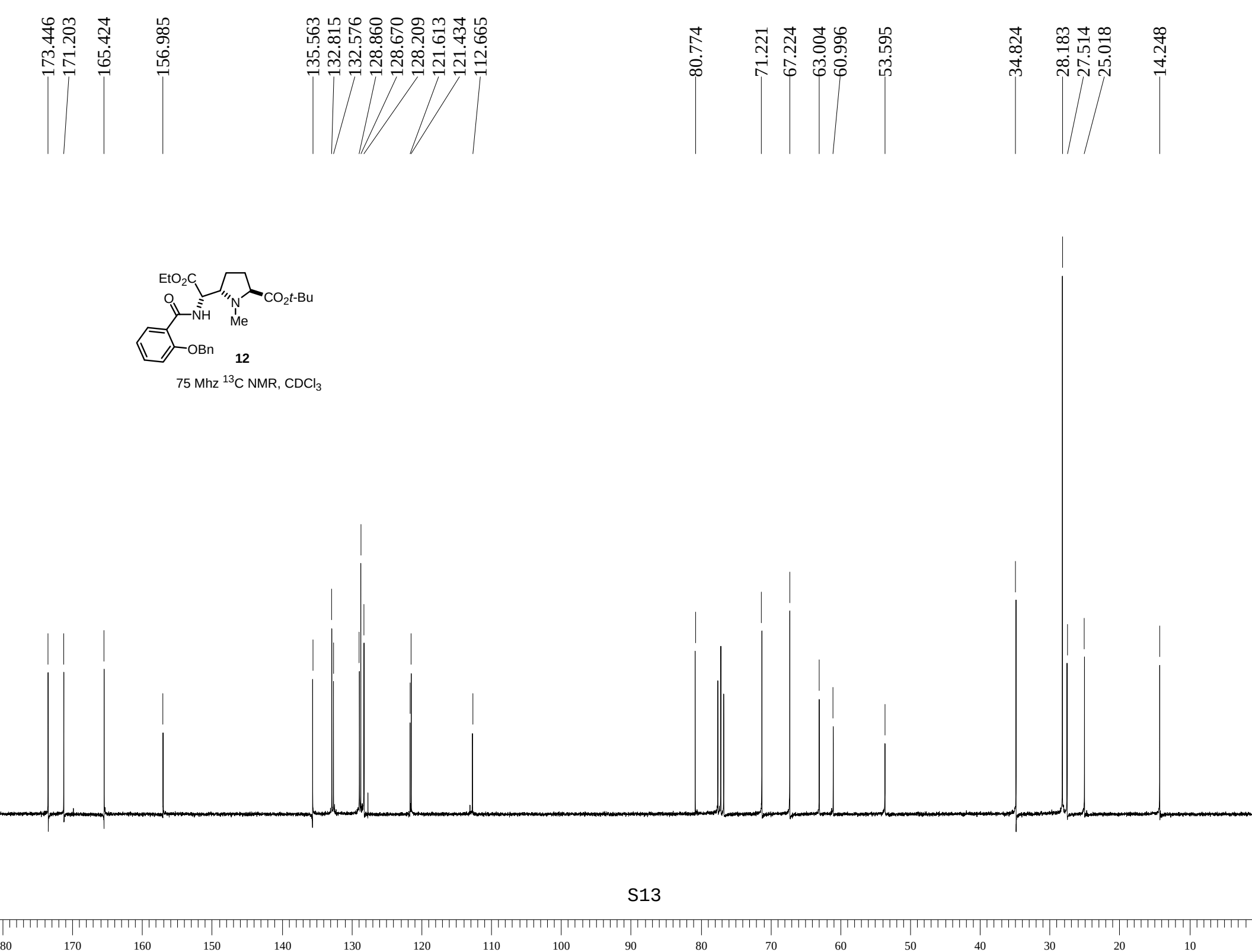


12

300 MHz ^{1}H NMR, $CDCl_3$

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80.774
71.221
67.224
63.004
60.996
53.595
34.824
28.183
27.514
25.018
14.248
EtO2C
O
NH
N
Me
CO2t-Bu
OBn
12
75 Mhz 13C NMR, CDCl3
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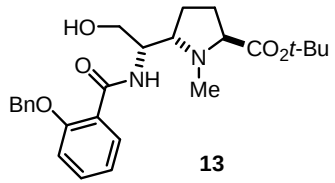
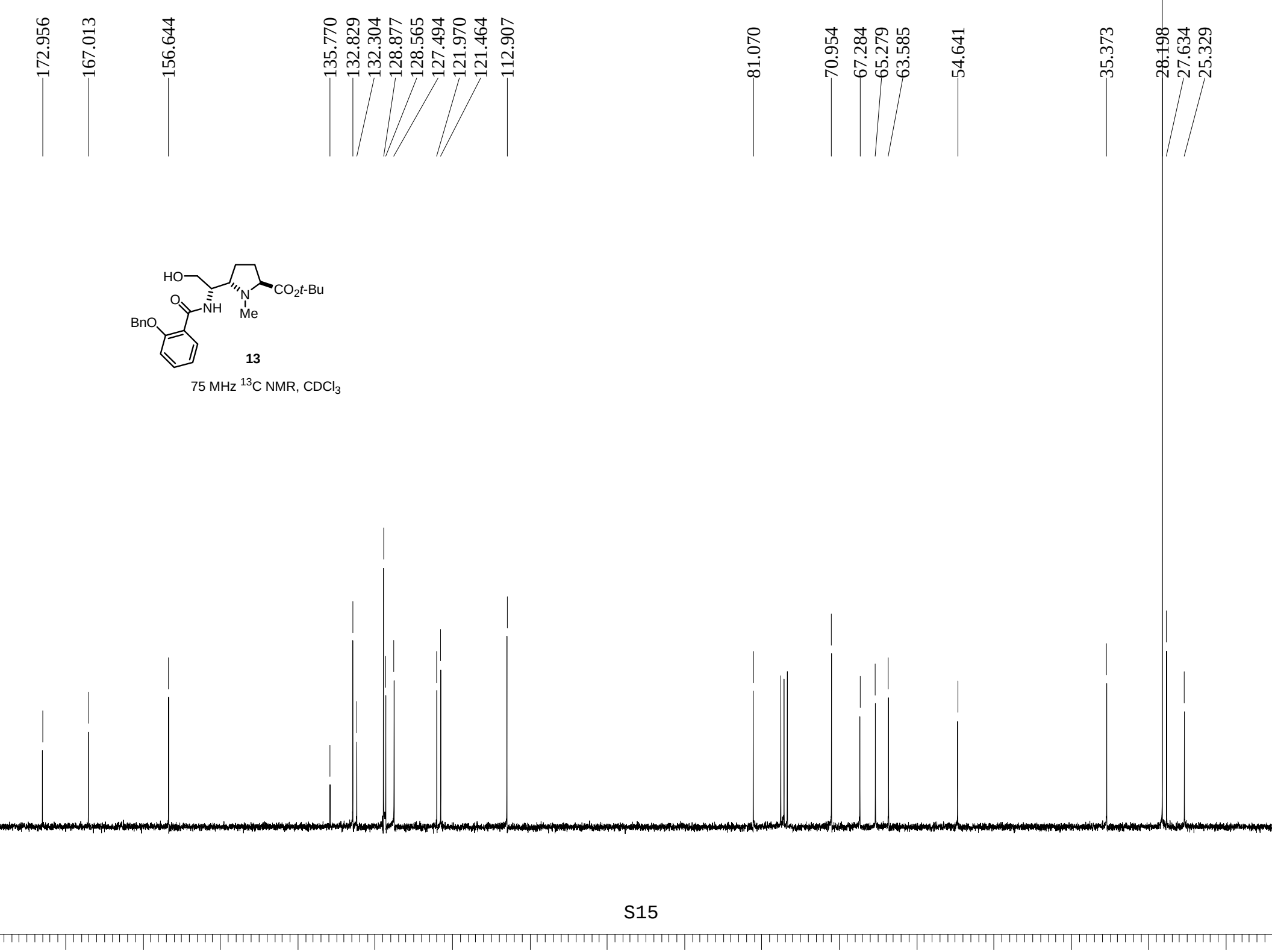


13

300 MHz ^{1}H NMR, $CDCl_3$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70.954
67.284
65.279
63.585
54.641
35.373
28.198
27.634
25.329
HO
O
NH
N
Me
CO2t-Bu
BnO
13
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160
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140
130
120
110
100
90
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70
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50
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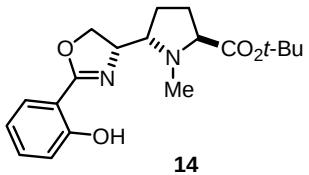
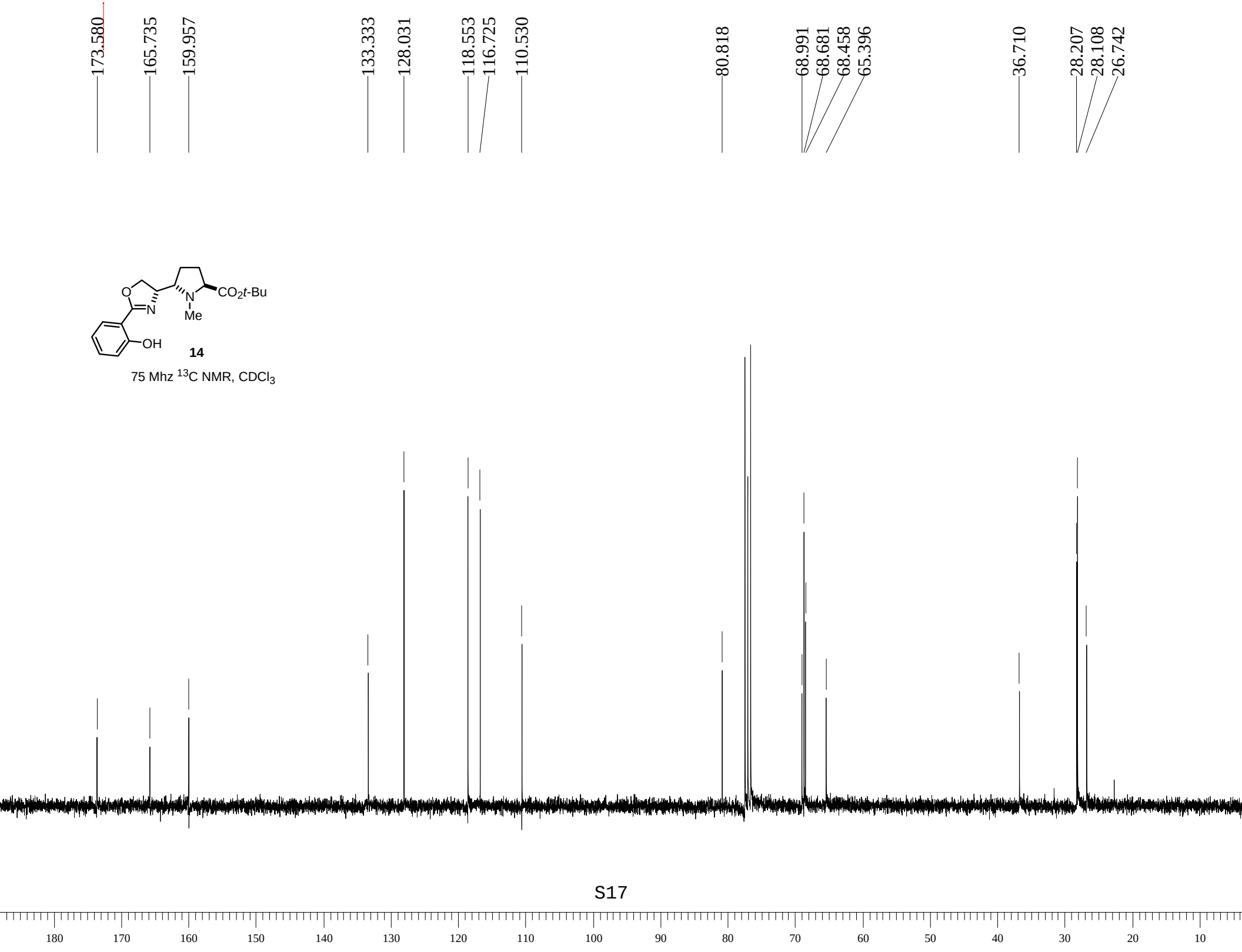
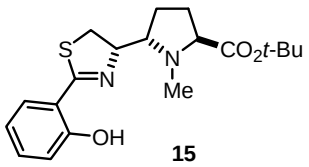


14

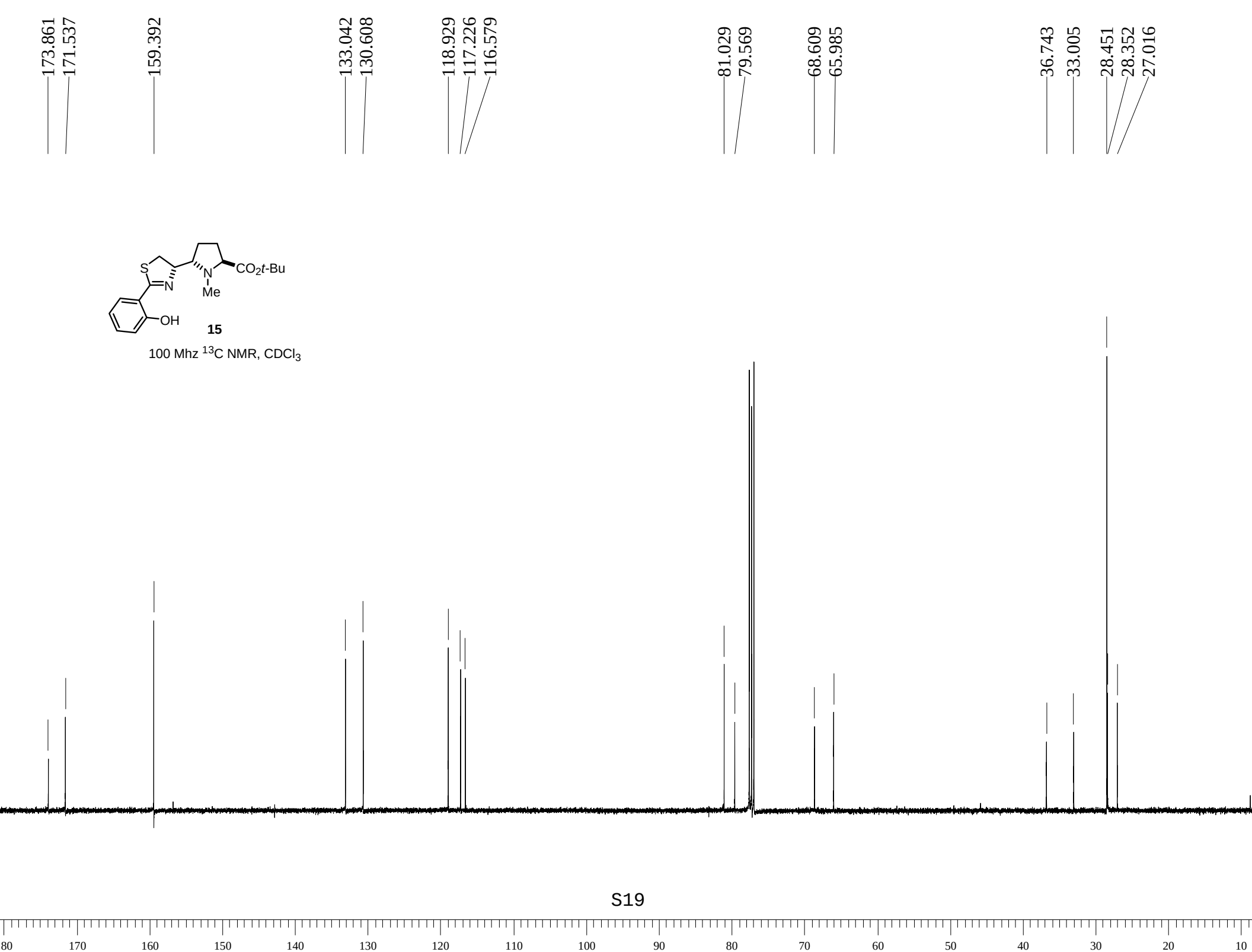
300 MHz ^{1}H NMR, $CDCl_3$

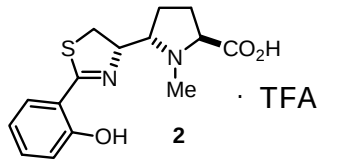

173.580
165.735
159.957
133.333
128.031
118.553
116.725
110.530
80.818
68.991
68.681
68.458
65.396
36.710
28.207
28.108
26.742
O
N
N
Me
CO2t-Bu
OH
14
75 Mhz 13C NMR, CDCl3
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10



15

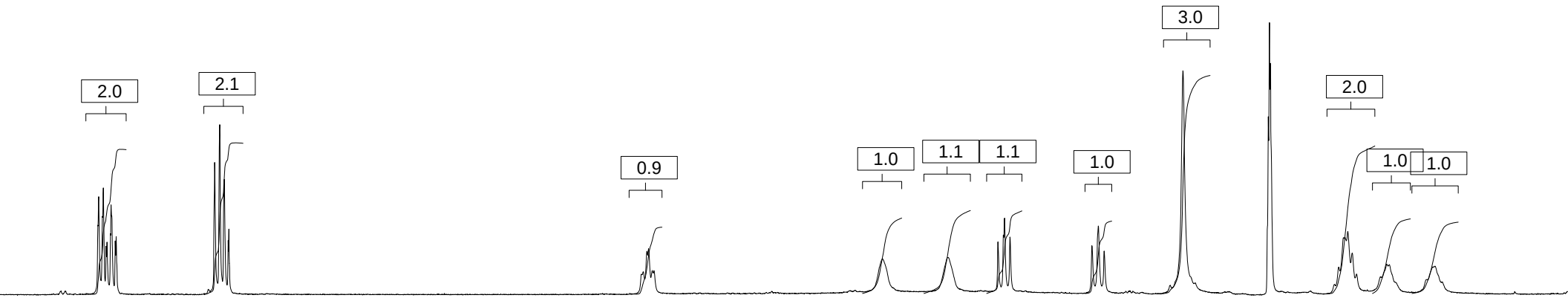
400 Mhz ^{1}H NMR, $CDCl_3$

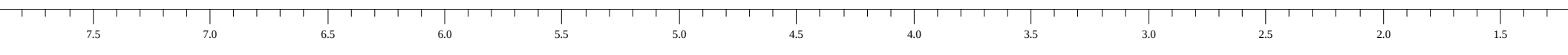

173.861
171.537
159.392
133.042
130.608
118.929
117.226
116.579
81.029
79.569
68.609
65.985
36.743
33.005
28.451
28.352
27.016
S
N
N
Me
CO2t-Bu
OH
15
100 Mhz 13C NMR, CDCl3
80
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

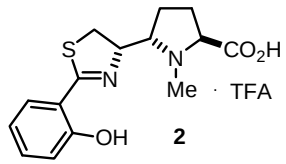


2 · TFA

400 MHz ^{1}H NMR, DMSO-d_6

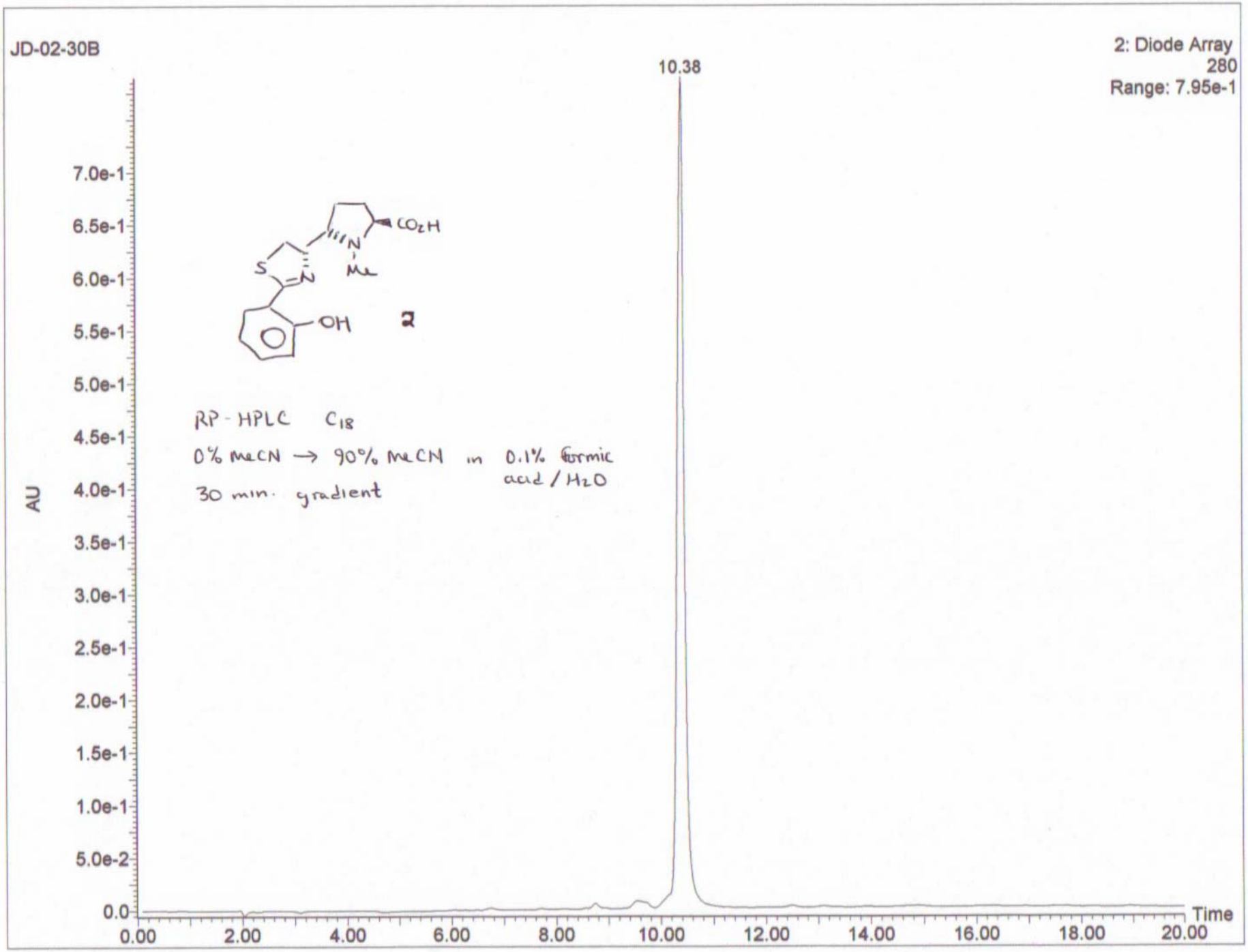


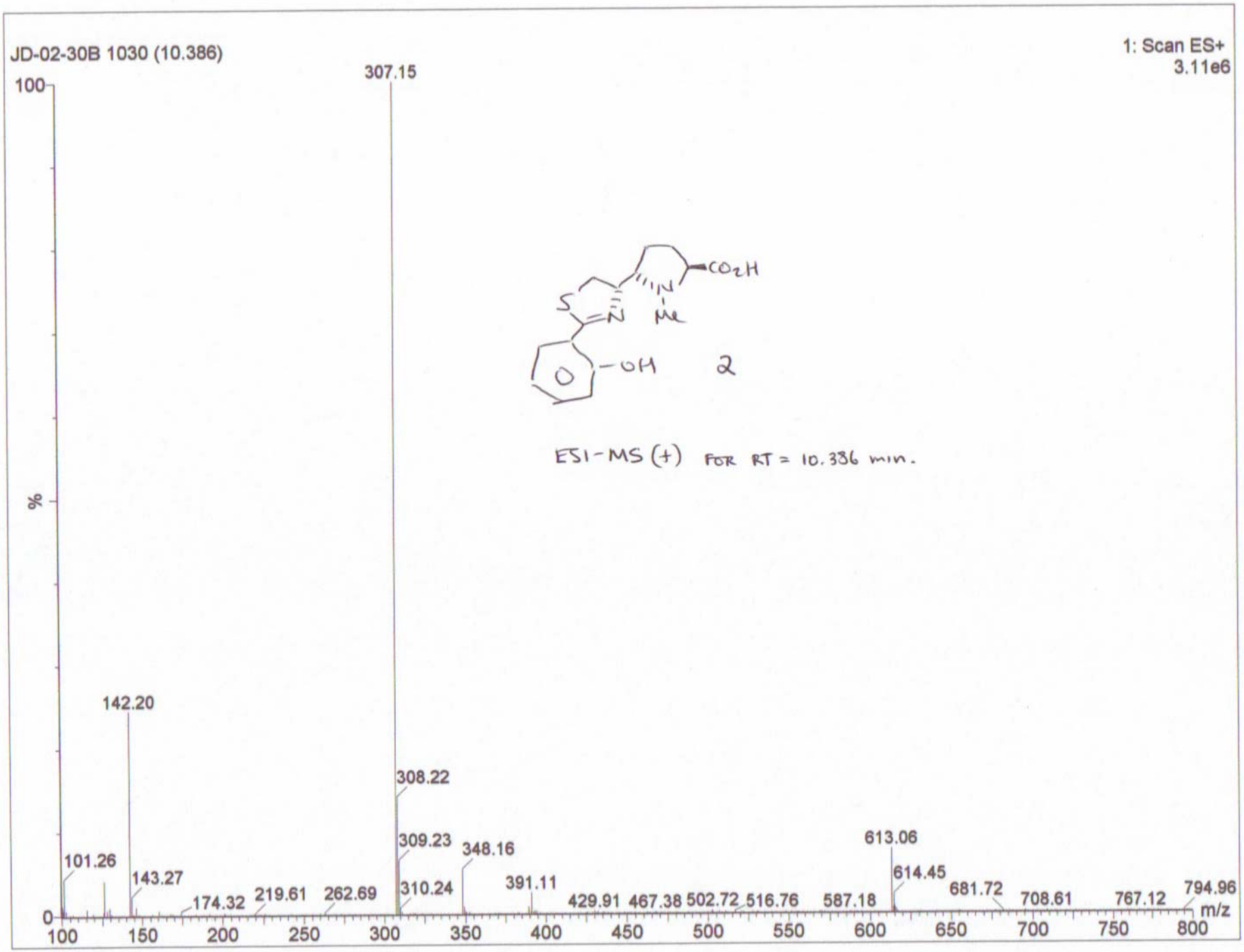


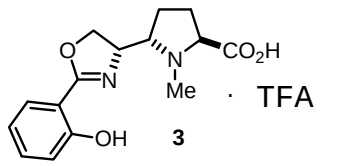


2 · TFA

100 MHz ^{13}C NMR, DMSO-d_6


JD-02-30B
2: Diode Array
280
Range: 7.95e-1
10.38
7.0e-1
6.5e-1
6.0e-1
5.5e-1
5.0e-1
4.5e-1
4.0e-1
3.5e-1
3.0e-1
2.5e-1
2.0e-1
1.5e-1
1.0e-1
5.0e-2
0.0
AU
CO2H
N
Me
S
N
OH
2
RP - HPLC C18
0% MeCN → 90% MeCN in 0.1% formic acid / H2O
30 min. gradient
0.00
2.00
4.00
6.00
8.00
10.00
12.00
14.00
16.00
18.00
20.00
Time

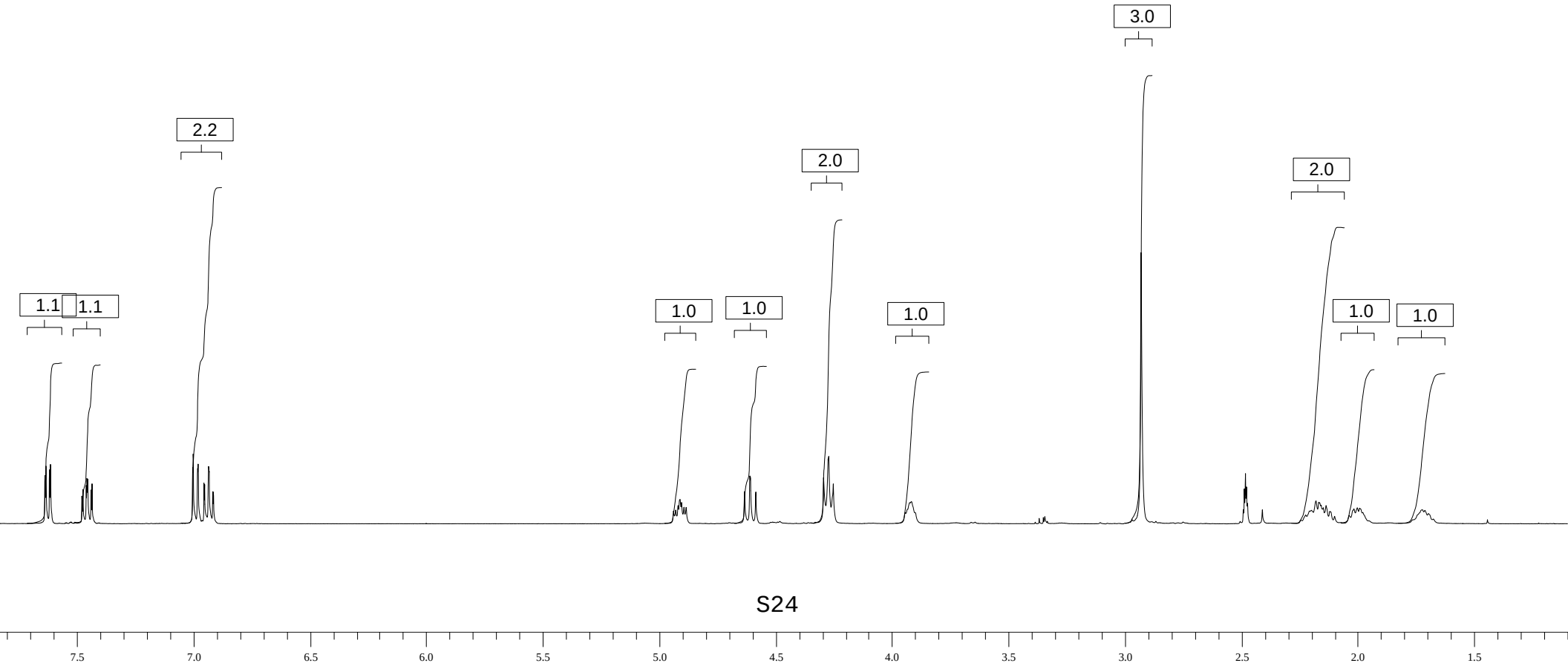

JD-02-30B 1030 (10.386)
1: Scan ES+
3.11e6
100
%
0
307.15
142.20
308.22
309.23
348.16
613.06
101.26
143.27
614.45
391.11
174.32
219.61
262.69
310.24
429.91
467.38
502.72
516.76
587.18
681.72
708.61
767.12
794.96
m/z
100
150
200
250
300
350
400
450
500
550
600
650
700
750
800
CO2H
N
Me
S
N
O
OH
2
ESI-MS (+) FOR RT = 10.386 min.

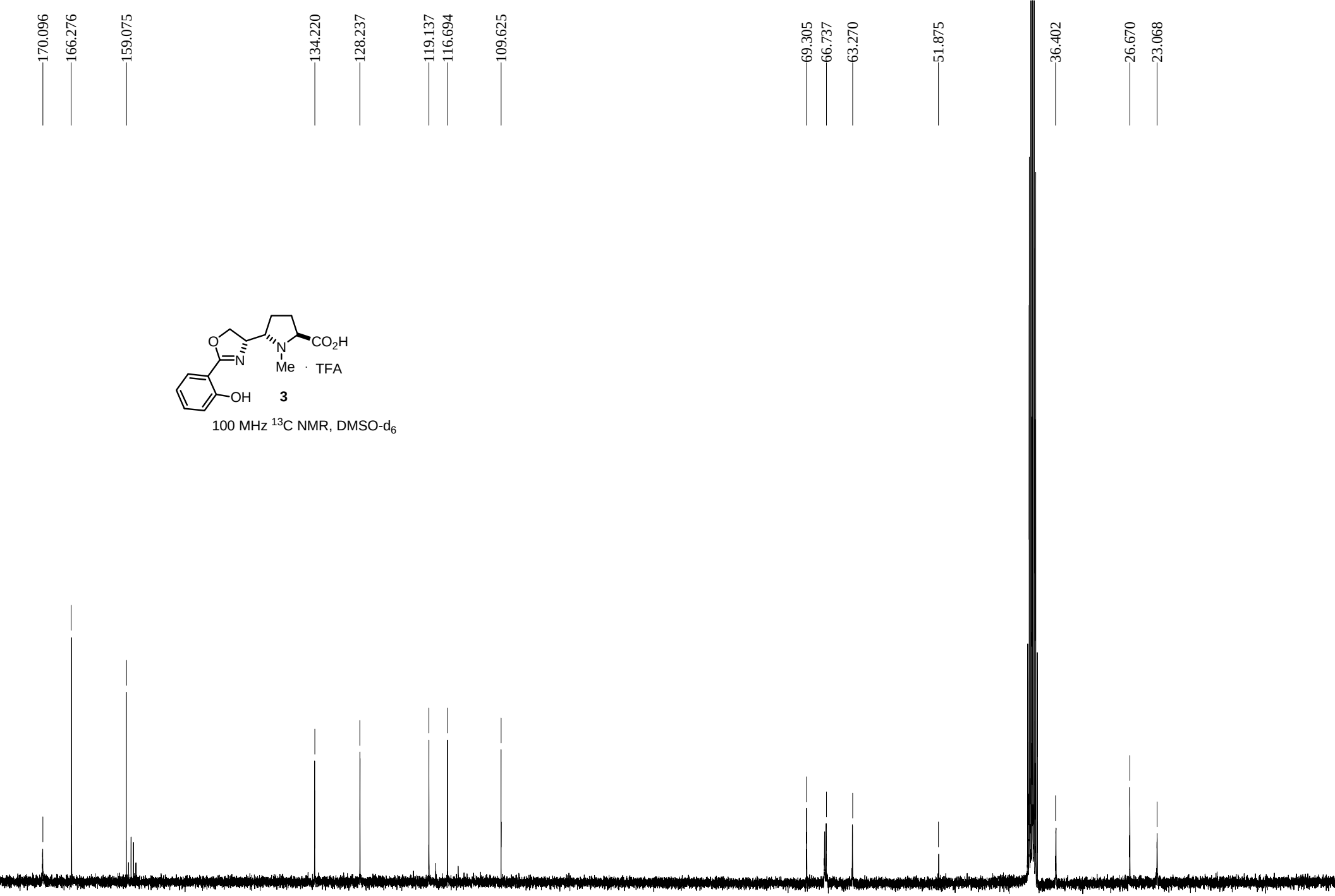


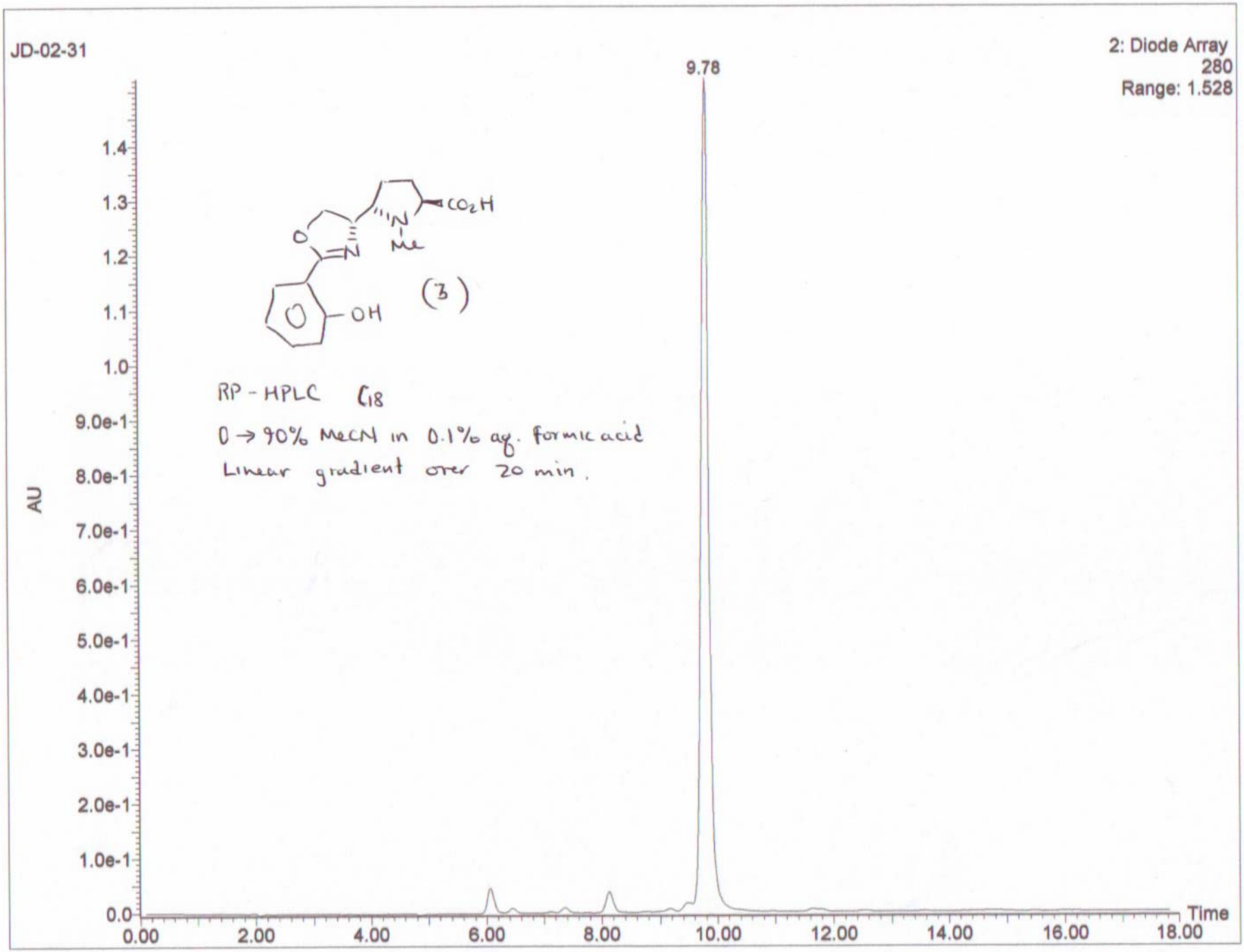
· TFA

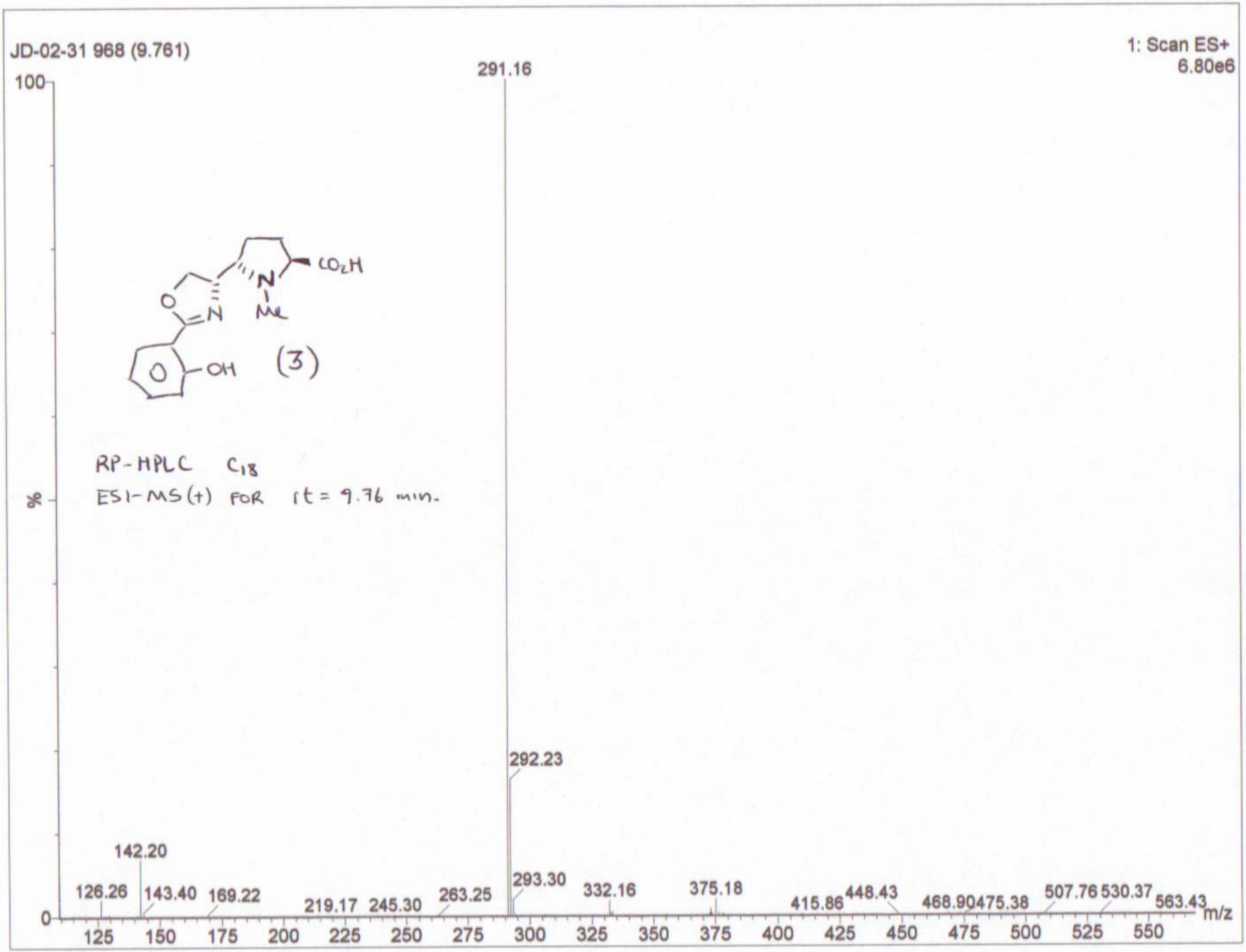
3

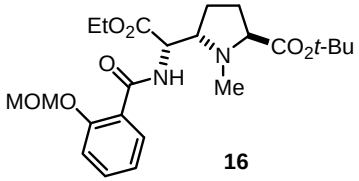
400 MHz ^{1}H NMR, DMSO-d_6




170.096
166.276
159.075
134.220
128.237
119.137
116.694
109.625
69.305
66.737
63.270
51.875
36.402
26.670
23.068
O
N
N
Me
CO2H
TFA
OH
3
100 MHz 13C NMR, DMSO-d6
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10


JD-02-31
2: Diode Array
280
Range: 1.528
9.78
CO2H
N
Me
O
N
OH
(3)
RP - HPLC C18
0 → 90% MeCN in 0.1% aq. formic acid
Linear gradient over 20 min.
AU
1.4
1.3
1.2
1.1
1.0
9.0e-1
8.0e-1
7.0e-1
6.0e-1
5.0e-1
4.0e-1
3.0e-1
2.0e-1
1.0e-1
0.0
0.00
2.00
4.00
6.00
8.00
10.00
12.00
14.00
16.00
18.00
Time


JD-02-31 968 (9.761)
1: Scan ES+
6.80e6
100
%
0
291.16
292.23
142.20
126.26
143.40
169.22
219.17
245.30
263.25
293.30
332.16
375.18
415.86
448.43
468.90
475.38
507.76
530.37
563.43
m/z
125
150
175
200
225
250
275
300
325
350
375
400
425
450
475
500
525
550
CO2H
N
Me
O
N
O
OH
(3)
RP-HPLC C18
ESI-MS(+) FOR rt = 9.76 min.



16

300 MHz ^{13}H NMR, $CDCl_3$

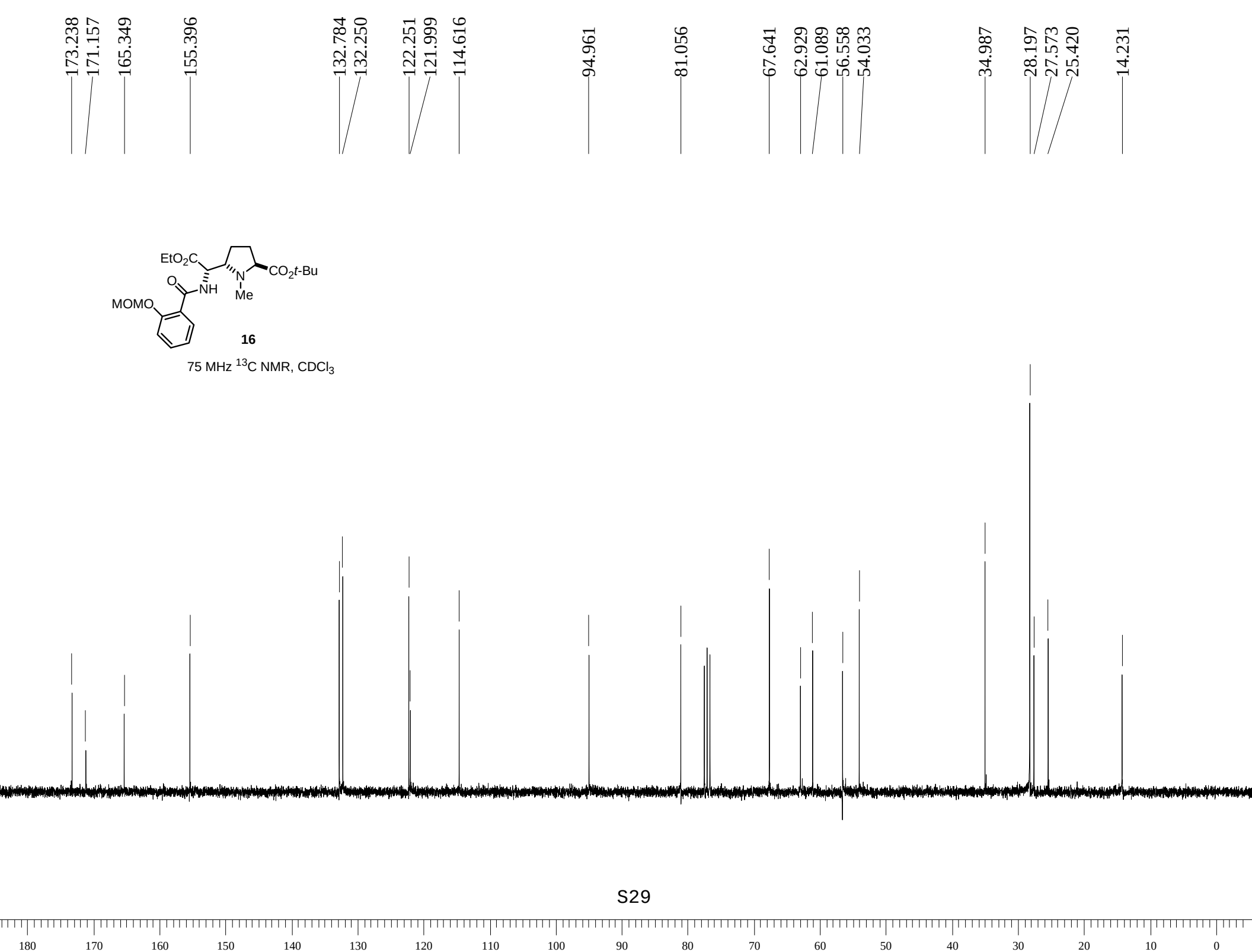
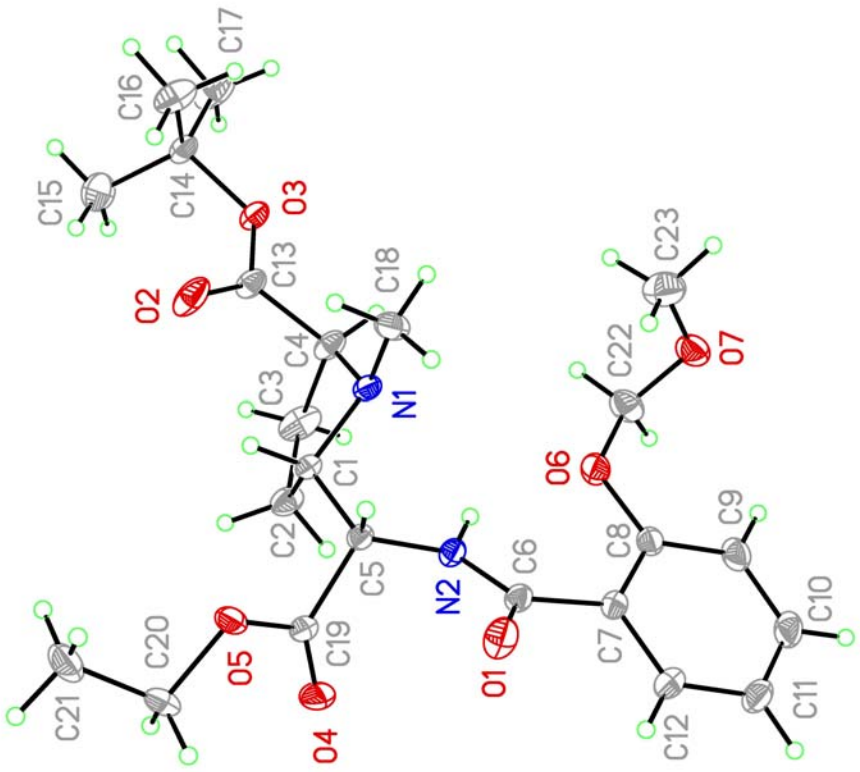

173.238
171.157
165.349
155.396
132.784
132.250
122.251
121.999
114.616
94.961
81.056
67.641
62.929
61.089
56.558
54.033
34.987
28.197
27.573
25.420
14.231
EtO2C
CO2t-Bu
O
NH
N
Me
MOMO
16
75 MHz 13C NMR, CDCl3
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

Table 1. Crystal data and structure refinement for **16**.

Identification code	**16**	
Empirical formula	C23 H34 N2 O7	
Formula weight	450.52	
Temperature	228(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P2(1)2(1)2(1)	
Unit cell dimensions	a = 9.0823(6) Å	α= 90°.
	b = 9.2605(7) Å	β= 90°.
	c = 29.233(2) Å	γ = 90°.
Volume	2458.7(3) $Å^3$	
Z	4	
Density (calculated)	1.217 Mg/m^3	
Absorption coefficient	0.090 mm^{-1}	
F(000)	968	
Crystal size	0.46 x 0.41 x 0.37 mm^3	
Theta range for data collection	3.07 to 30.50°.	
Index ranges	-12<=h<=12, -13<=k<=13, -41<=l<=41	
Reflections collected	65942	
Independent reflections	4224 [R(int) = 0.0374]	
Completeness to theta = 30.50°	99.8 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.970 and 0.960	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4224 / 0 / 307	
Goodness-of-fit on F^2	1.038	
Final R indices [I>2sigma(I)]	R1 = 0.0397, wR2 = 0.1024	
R indices (all data)	R1 = 0.0496, wR2 = 0.1106	
Largest diff. peak and hole	0.219 and -0.157 $e.Å^{-3}$	



Compound **16** single molecule view, all thermal ellipsoids at 20% probability