Supporting Information for:

Synthesis of fluorescent enone derived α-amino acids

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Table Of Contents

1. Experimental Procedures and Spectroscopic Data For All Known Compounds

Dimethyl (2*S*)-2-aminobutandioate hydrochloride 5.[1] To a suspension of L-aspartic acid **4** (5.0 g, 37.56 mmol) in methanol (30 mL) at 0 °C under argon was added dropwise thionyl chloride (4 mL, 52.58 mmol). The reaction mixture was allowed to warm to room temperature and heated under reflux for 3 h. The solution was concentrated *in vacuo* to give a colourless oil and triturated with diethyl ether to give **5** as an colourless solid (7.55 g, 100%). mp 115–116 °C (lit.,[1] mp 114–115 °C); $[\alpha]_D^{24}$ +22.0 (*c* 1.0, MeOH); δ_H (400 MHz, DMSO-d_6) 2.99 (1H, dd, *J* 18.0, 5.5 Hz, 3-*H*H), 3.05 (1H, dd, *J* 18.0, 5.5 Hz, 3-H*H*), 3.66 (3H, s, OMe), 3.74 (3H, s, OMe), 4.35 (1H, t, *J* 5.5 Hz, 2-H), 8.72 (3H, s, CHNH_3^+); δ_C (100 MHz, DMSO-d_6) 34.0 (CH_2), 48.4 (CH), 52.2 (CH_3), 53.0 (CH_3), 168.7 (C), 169.6 (C); *m/z* (CI) 162 (MH^+, 100%), 148 (5), 102 (20).

Dimethyl (2*S*)-2-(tritylamino)butandioate 6.[2] To a solution of dimethyl (2*S*)-2-aminobutandioate hydrochloride **5** (7.46 g, 37.72 mmol) in dichloromethane (150 mL) at 0 °C under argon was added dropwise triethylamine (11 mL, 75.44 mmol) and triphenylmethyl chloride (10.50 g, 37.72 mmol). The reaction mixture was allowed to warm to room temperature and stirred for 24 h. The reaction mixture was washed with citric acid (2 M, 100 mL), water (100 mL), brine (100 mL) then dried (MgSO$_4$) and concentrated *in vacuo* to give a colourless oil. The crude product was purified by column chromatography on silica eluting with 50% diethyl ether in petroleum ether to give **6** a colourless solid (15.16 g, 100%). mp 71–72 °C (lit.,[2] mp 70–71 °C); $[\alpha]_D^{24}$ +36.6 (*c* 1.0, CHCl$_3$); δ_H (400 MHz, CDCl$_3$) 2.51 (1H, dd, *J* 14.7, 7.0 Hz, 3-*H*H), 2.66 (1H, dd, *J* 14.7, 5.4 Hz, 3-H*H*), 2.93 (1H, d, *J* 10.1 Hz, NH), 3.25 (3H, s, OMe), 3.67 (3H, s, OMe), 3.68–3.73 (1H, m, 2-H), 7.15–7.20 (3H, m, ArH), 7.23–7.28 (6H, m, ArH), 7.46–7.51 (6H, m, ArH); δ_C (100 MHz, CDCl$_3$) 39.0 (CH_2), 50.5 (CH), 50.7 (CH_3), 52.4 (CH_3), 69.9 (C), 125.2 (CH), 126.6 (CH), 127.5 (CH), 144.4 (C), 169.7 (C), 172.6 (C); *m/z* (EI) 403 (M^+, 1%), 326 (35), 243 (100), 165 (30), 83 (70).

Methyl (2*S*)-5-(dimethoxyphosphoryl)-4-oxo-2-(tritylamino)pentanoate 7.[2] A solution of dimethyl methylphosphonate (3.0 mL, 27.26 mmol) in THF (50 mL) was cooled to −78 °C under argon. *n*-Butyl lithium (2.5 M in hexane, 11 mL, 28.62 mmol) was added dropwise and the reaction mixture stirred for 1 h. In a separate reaction vessel, a solution of dimethyl (2*S*)-2-(tritylamino)butandioate **6** (5.0 g, 12.39 mmol) in THF (100 mL) was cooled to −78 °C under argon and then the dimethyl methylphosphonate/*n*-butyl lithium solution was cannulated into the flask and the reaction mixture stirred at −78 °C for 2 h to give a yellow solution. The reaction was quenched with a saturated solution of ammonium chloride (3 mL) and allowed to warm to room temperature. The

mixture was concentrated *in vacuo*. The resulting residue was diluted with ethyl acetate (100 mL), washed with water (2 × 100 mL), brine (100 mL) then dried (MgSO$_4$) and concentrated *in vacuo*. The crude product was purified by column chromatography on silica eluting with 75% ethyl acetate in petroleum ether to give **7** as a colourless solid (4.93 g, 80%). mp 117–118 °C (lit.,[2] mp 117–118.5 °C); $[\alpha]_D^{24}$ +31.1 (*c* 1.0, CHCl$_3$); δ_H (400 MHz, CDCl$_3$) 2.78 (1H, dd, *J* 16.7, 6.9 Hz, 3-*H*H), 2.85–2.95 (2H, m, 3-H*H* and NH), 3.06 (2H, d, $J_{H–C–P}$ 22.7 Hz, 5-H$_2$), 3.29 (3H, s, OMe), 3.65–3.73 (1H, m, 2-H), 3.76 (3H, s, OMe), 3.79 (3H, s, OMe), 7.15–7.21 (3H, m, ArH), 7.26 (6H, t, *J* 7.7 Hz, ArH), 7.47 (6H, d, *J* 7.7 Hz, ArH); δ_C (100 MHz, CDCl$_3$) 41.8 (d, $J_{C–P}$ 128 Hz, CH$_2$), 48.7 (CH$_2$), 52.0 (CH$_3$), 52.9 (CH$_3$), 53.0 (CH$_3$), 53.1 (CH), 71.2 (C), 126.5 (CH), 127.9 (CH), 128.7 (CH), 145.6 (C), 174.0 (C), 199.2 (C); *m/z* (CI) 496 (MH$^+$, 1%), 301 (5), 254 (90), 243 (100), 237 (55), 167 (45).

4-(4'-benzyloxycarbonylaminophenyl)benzaldehyde (Table 1, entry 13). To a solution of 4-bromobenzaldehyde (0.30 g, 1.62 mmol) in *N,N*-dimethylformamide/water (10 mL, 95:5) was added 4-(benzyloxycarbonylamino)phenylboronic acid (0.66 g, 2.43 mmol), tetrakis(triphenylphosphine)palladium (0.04 g, 0.03 mmol) and potassium carbonate (0.56 g, 4.05 mmol). The reaction mixture was heated to 110 °C and stirred for 4 h. The solution was allowed to cool to room temperature then concentrated *in vacuo*. The residue was dissolved in chloroform, filtered through celite$^®$, and then the filtrate was concentrated *in vacuo*. The resultant solid was dissolved in diethyl ether (100 mL), then washed with water (6 × 100 mL), brine (2 × 100 mL), dried (MgSO$_4$), and concentrated *in vacuo*. The crude product was purified by column chromatography on silica eluting with 15% ethyl acetate in petroleum ether to give a colourless solid (0.19 g, 36%). mp 132–133 °C; ν_{max}/cm^{-1} (neat) 3331(NH), 1730 (CO), 1697 (CO), 1593, 1568, 1533, 1514, 1425, 1219; δ_H (400 MHz, CDCl$_3$) 5.23 (2H, s, PhC*H$_2$*), 6.81 (1H, s, NH), 7.31–7.45 (5H, m, ArH), 7.51 (2H, d, *J* 8.2 Hz, ArH), 7.60 (2H, d, *J* 8.2 Hz, ArH), 7.72 (2H, d, *J* 8.0 Hz, ArH), 7.93 (2H, d, *J* 8.0 Hz, ArH), 10.04 (1H, s, CHO); δ_C (100 MHz, CDCl$_3$) 67.2 (CH$_2$), 119.1 (CH), 127.2 (CH), 128.0 (CH), 128.4 (CH), 128.5 (CH), 128.7 (CH), 130.4 (CH), 134.5 (C), 134.9 (C), 136.0 (C), 138.5 (C), 146.5 (C), 153.4 (C), 192.1 (CH); *m/z* (EI) 331.1207 (M$^+$. C$_{21}$H$_{17}$NO$_3$ requires 331.1208), 287 (31%), 223 (92), 166 (22), 139 (21), 91 (100).

4-(3'-Nitrophenyl)benzaldehyde (Table 1, entry 12). Prepared using the procedure described above. Spectroscopic data as previously reported.[3]

5-(4'-Fluorophenyl)-2-furaldehyde (Table 1, entry 14). Prepared using the procedure described above. Spectroscopic data as previously reported.[4]
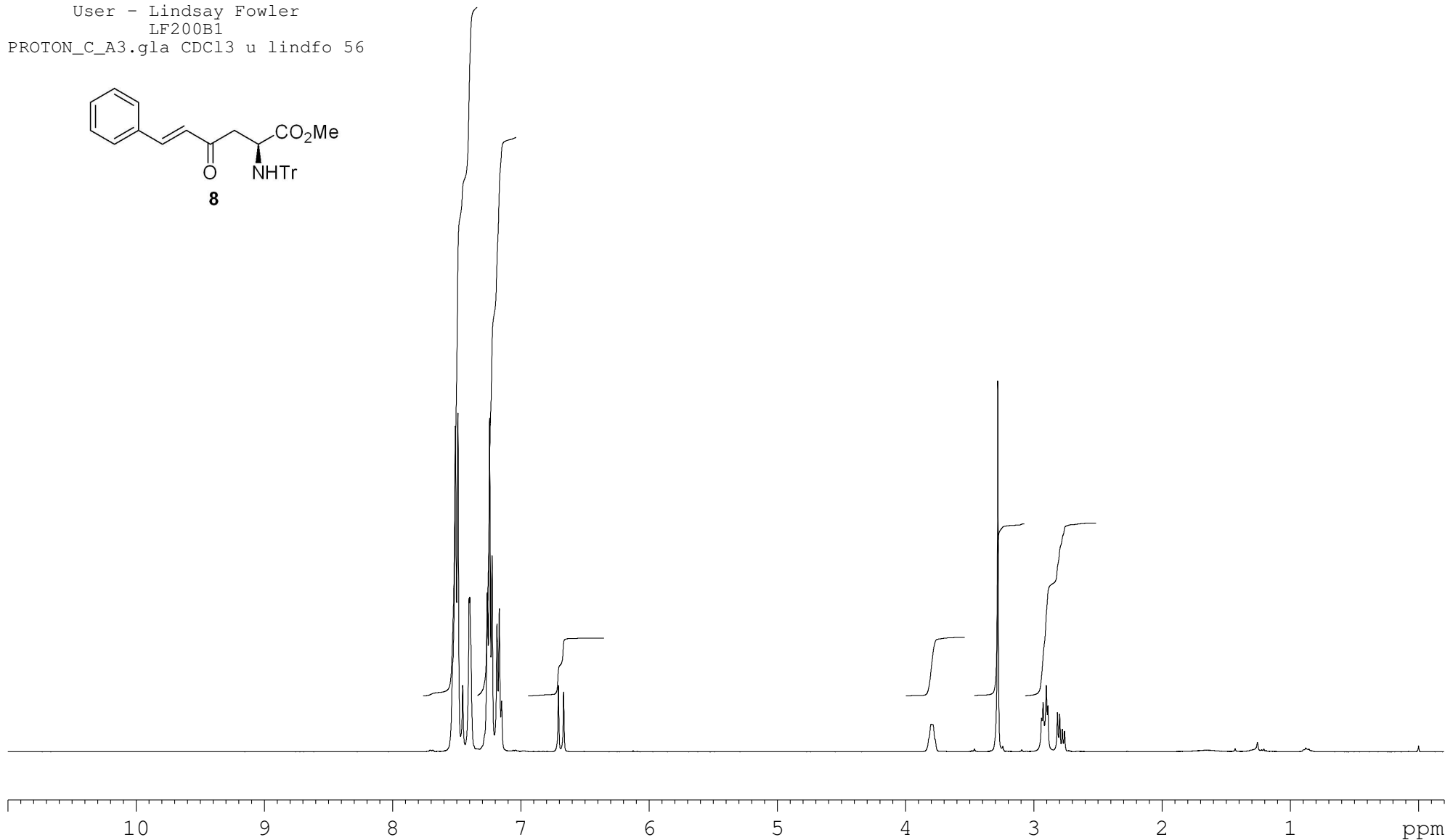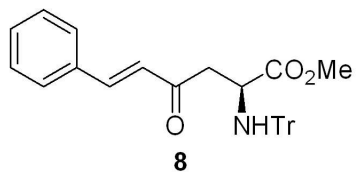
3. References

1. P. Gmeiner, P. L. Feldman, M. Y. Chu-Moyer and H. Rapoport, *J. Org. Chem.*, 1990, **55**, 3068.

2. D. E. Rudisill and J. P. Whitten, *Synthesis*, 1994, 851.

3. B. Tao and D. W. Boykin, *J. Org. Chem.*, 2004, **69**, 4330.

4. T. Hosoya, H. Aoyama, T. Ikemoto, Y. Kihara, T. Hiramatsu, M. Endo and M. Suzuki, *Bioorg. Med. Chem.*, 2003, **11**, 663.
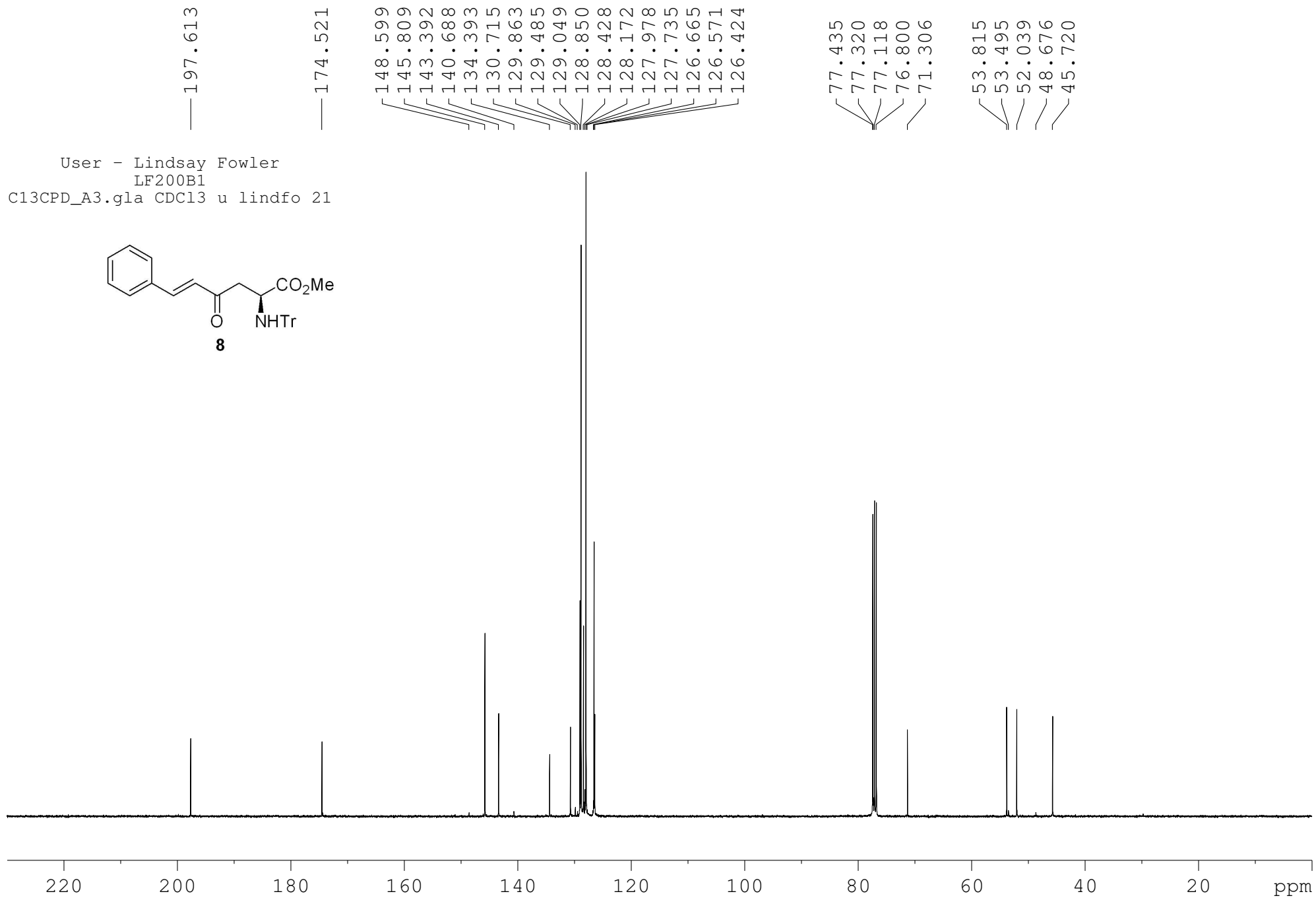
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2.760

0.000

8

10   9   8   7   6   5   4   3   2   1   ppm

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45.720

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8

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LF075B2
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7.420
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7.287
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7.191
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7.091
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3.281
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9

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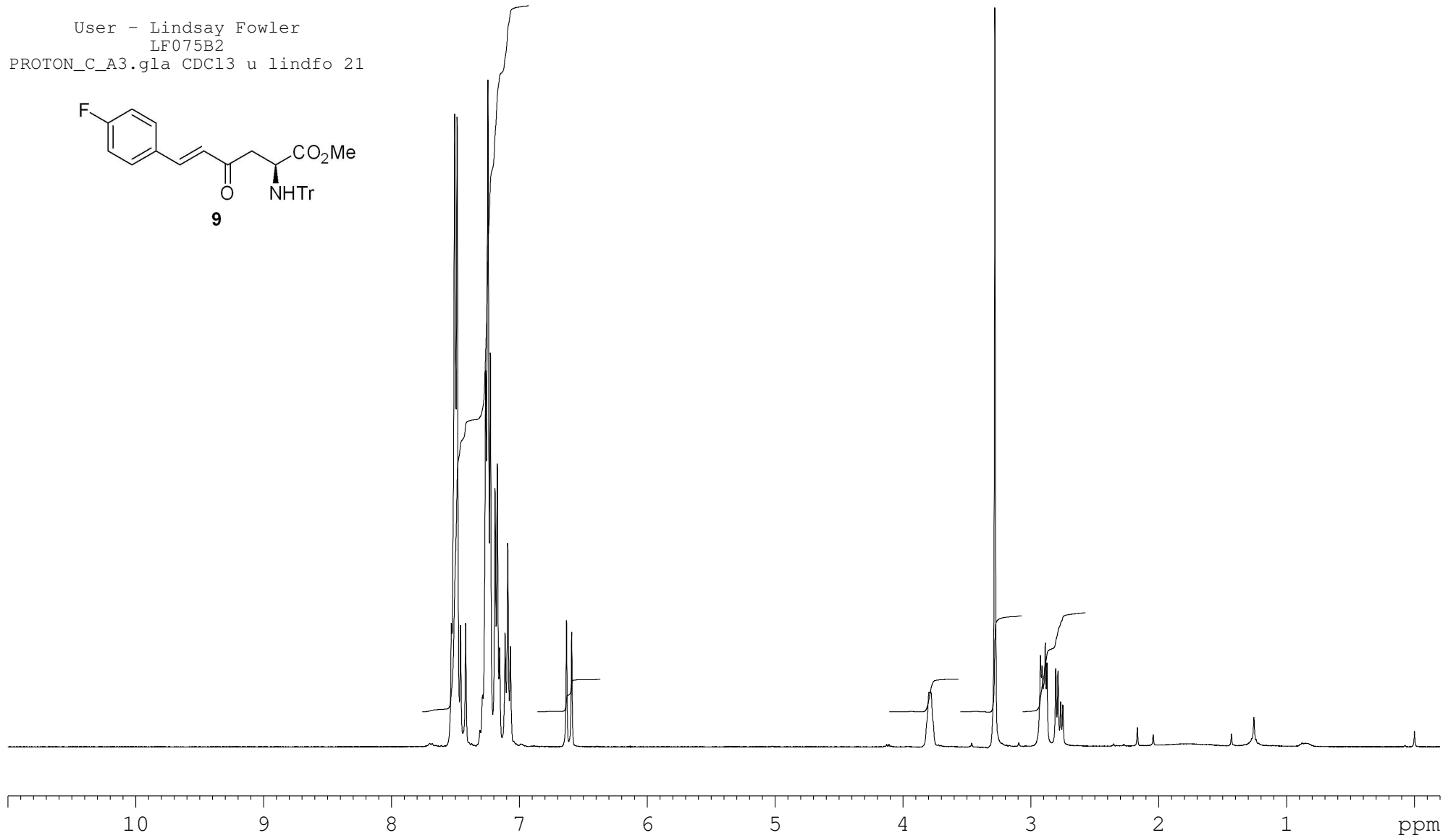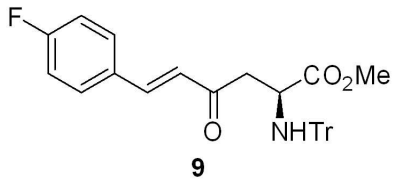
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126.081
126.063
116.329
116.113

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77.067
76.750
71.289

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52.021
45.825

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LF075B2
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9

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0.000

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70.143

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User – Lindsay Fowler
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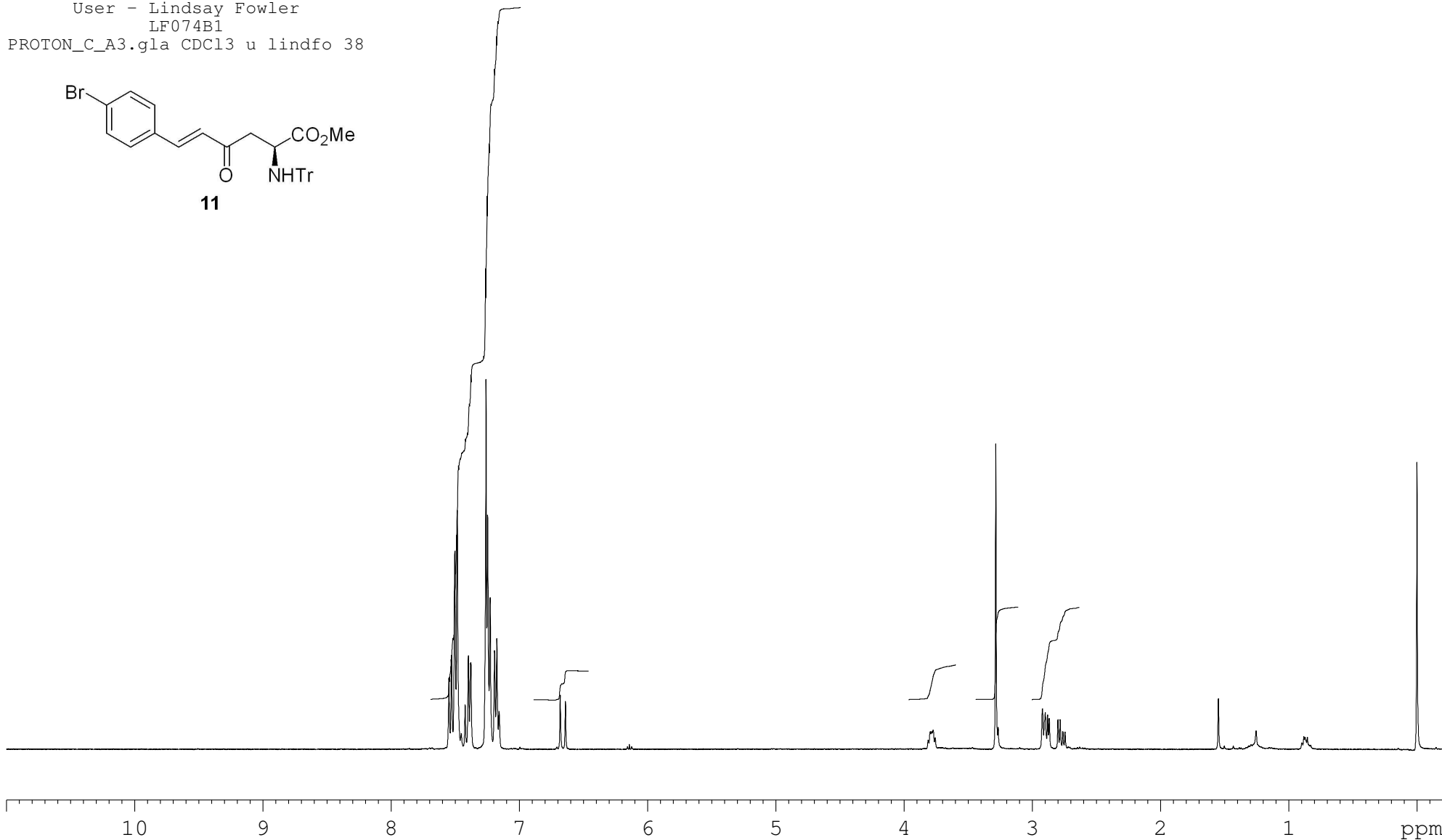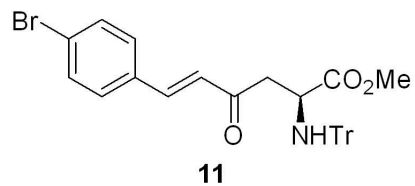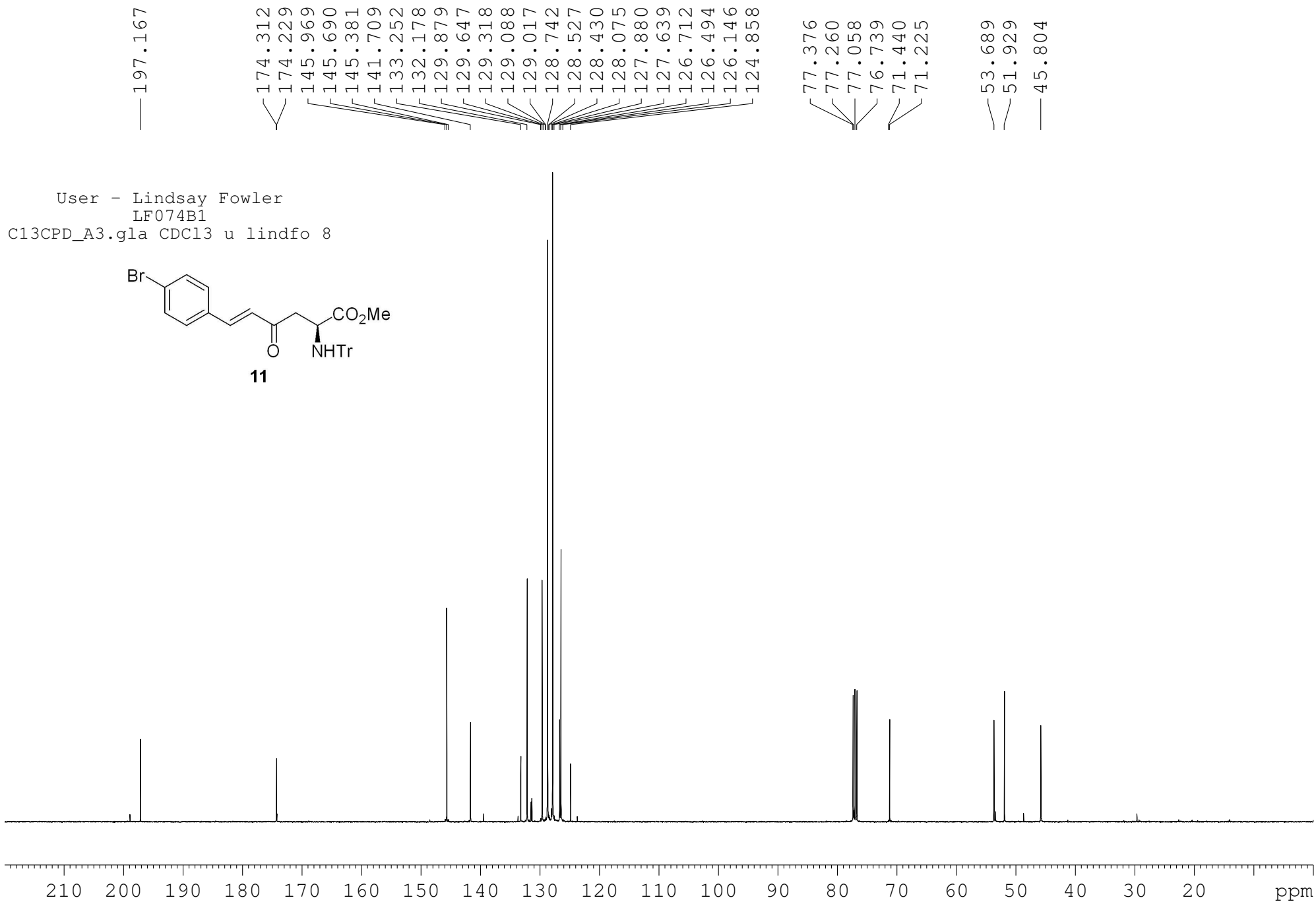
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11

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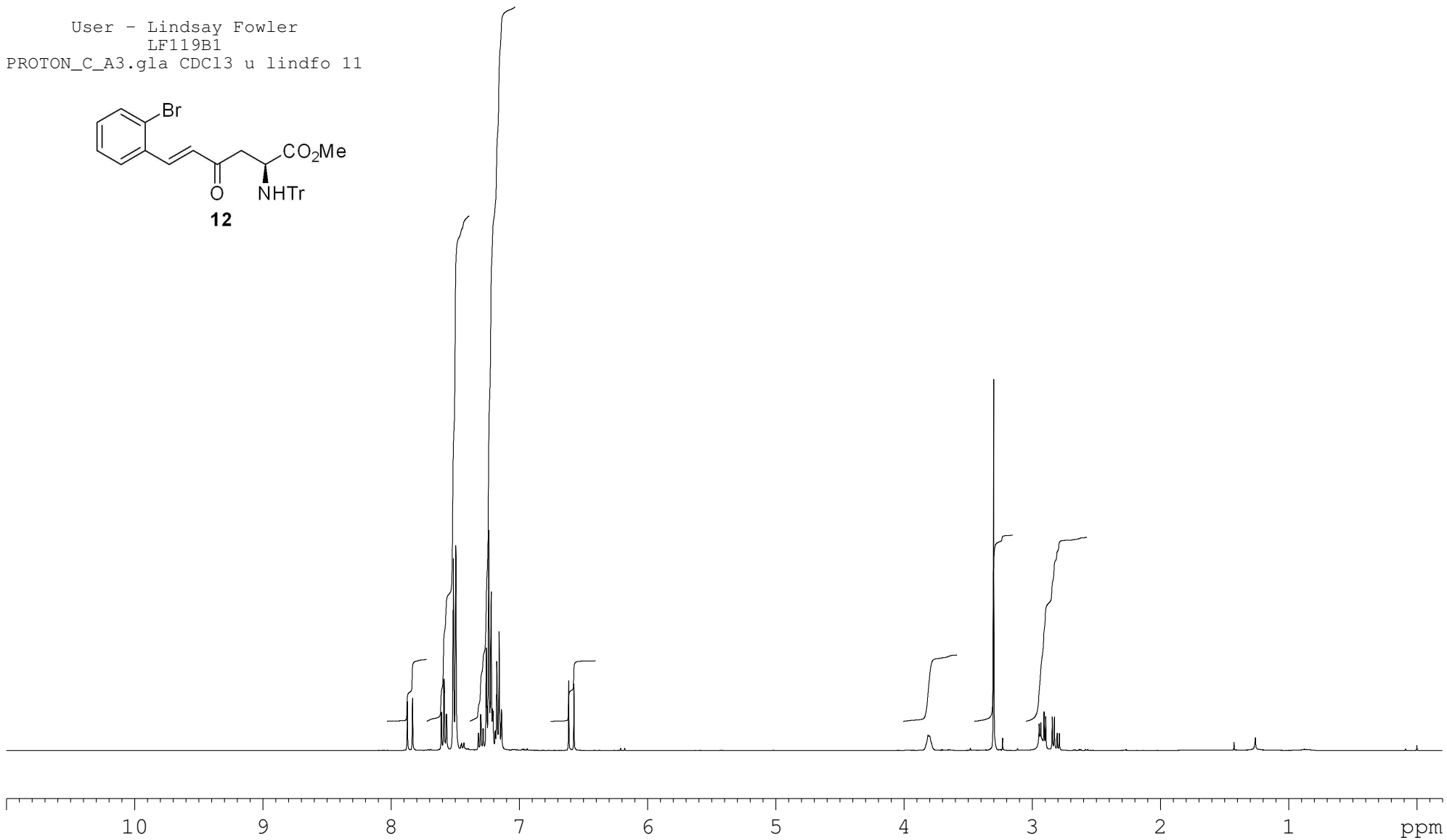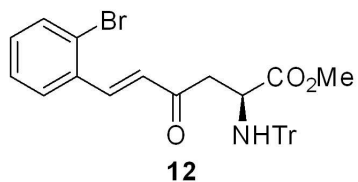
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LF119B1
C13CPD_A3.gla CDCl3 u lindfo 4

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O   NHTr

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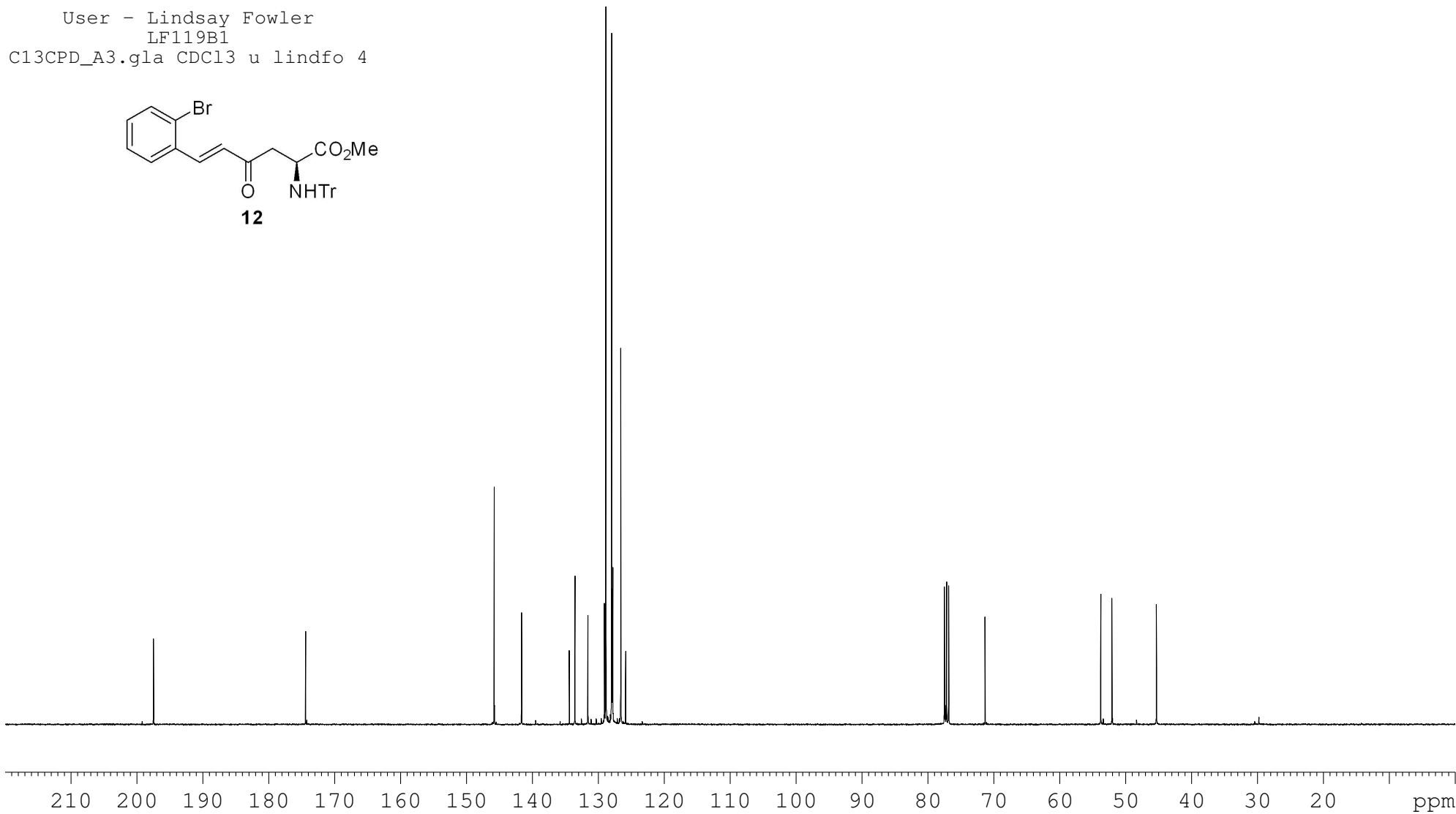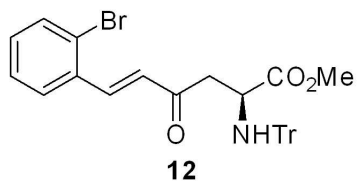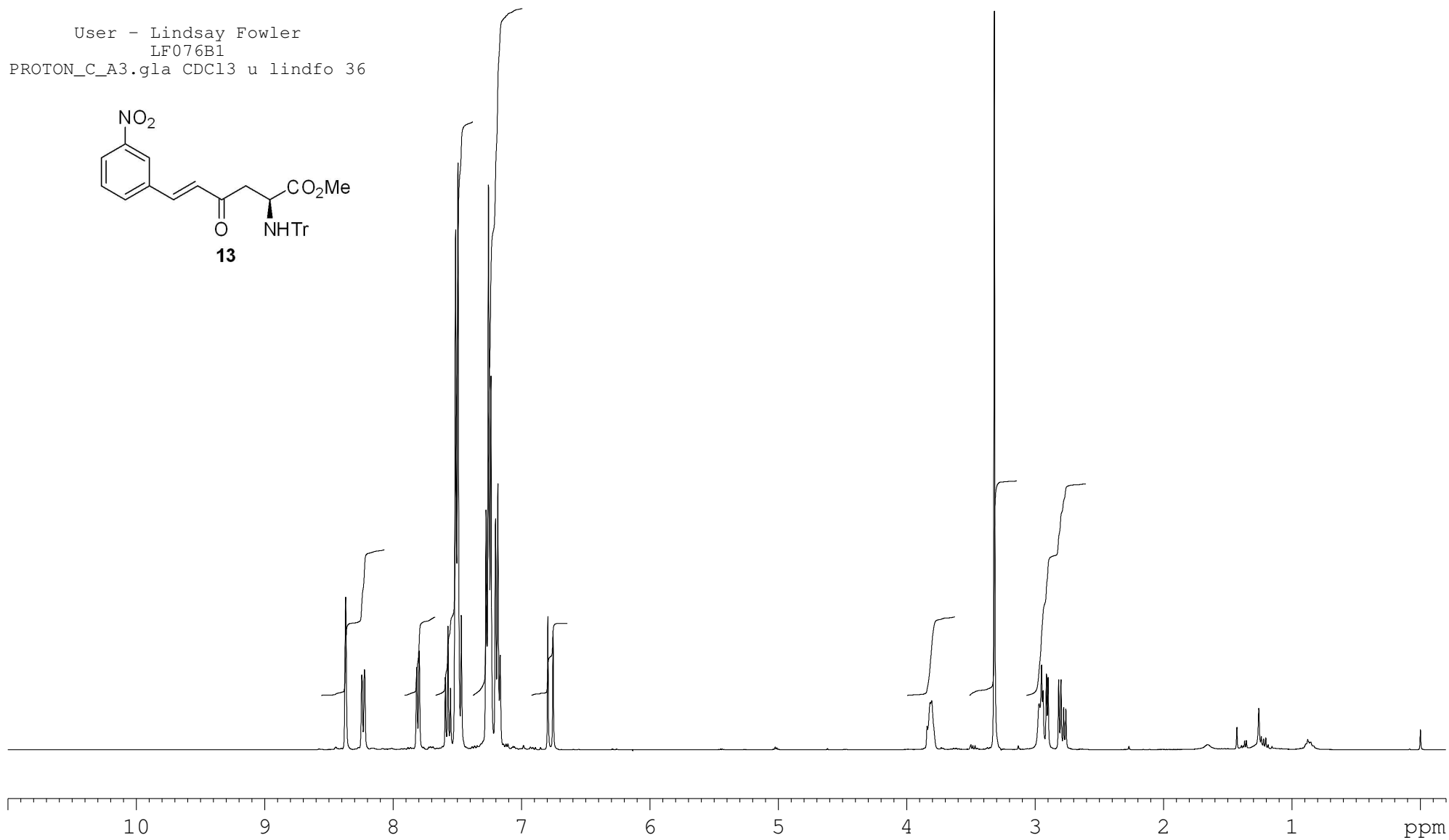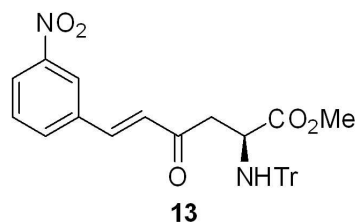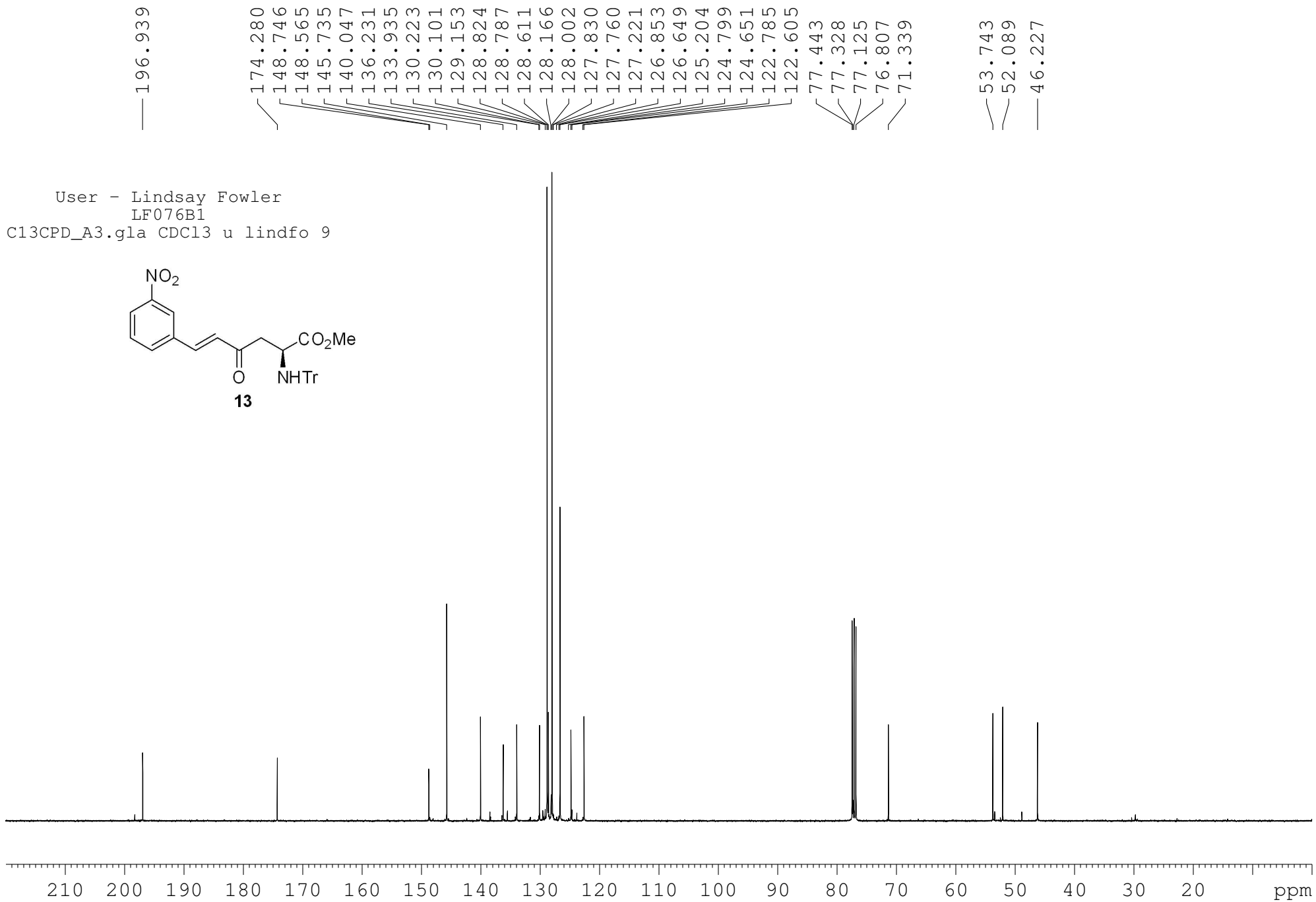
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0.000

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LF076B1
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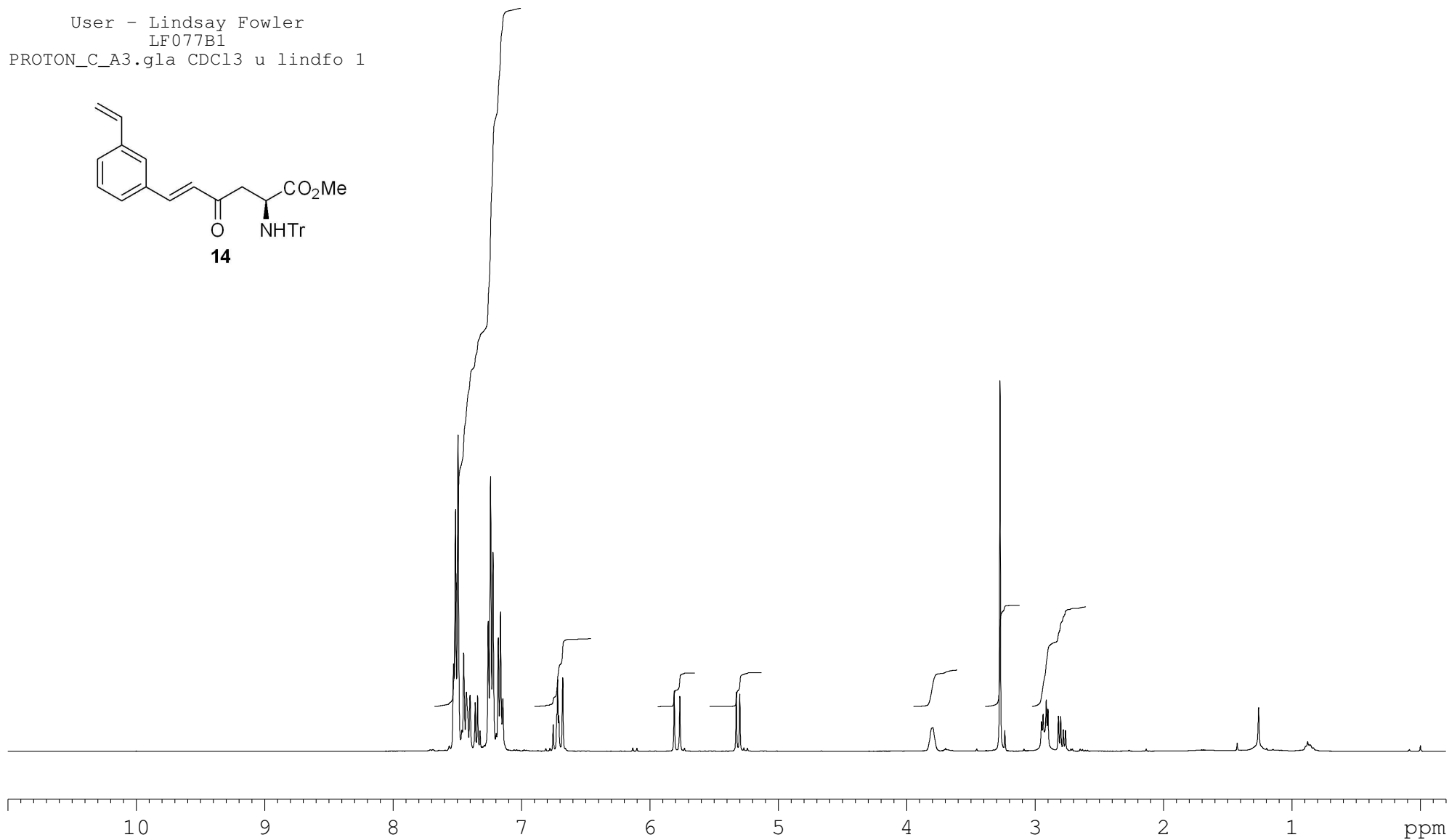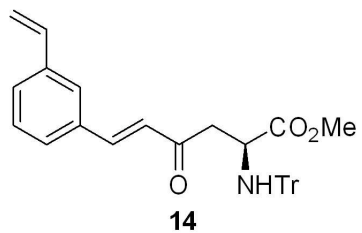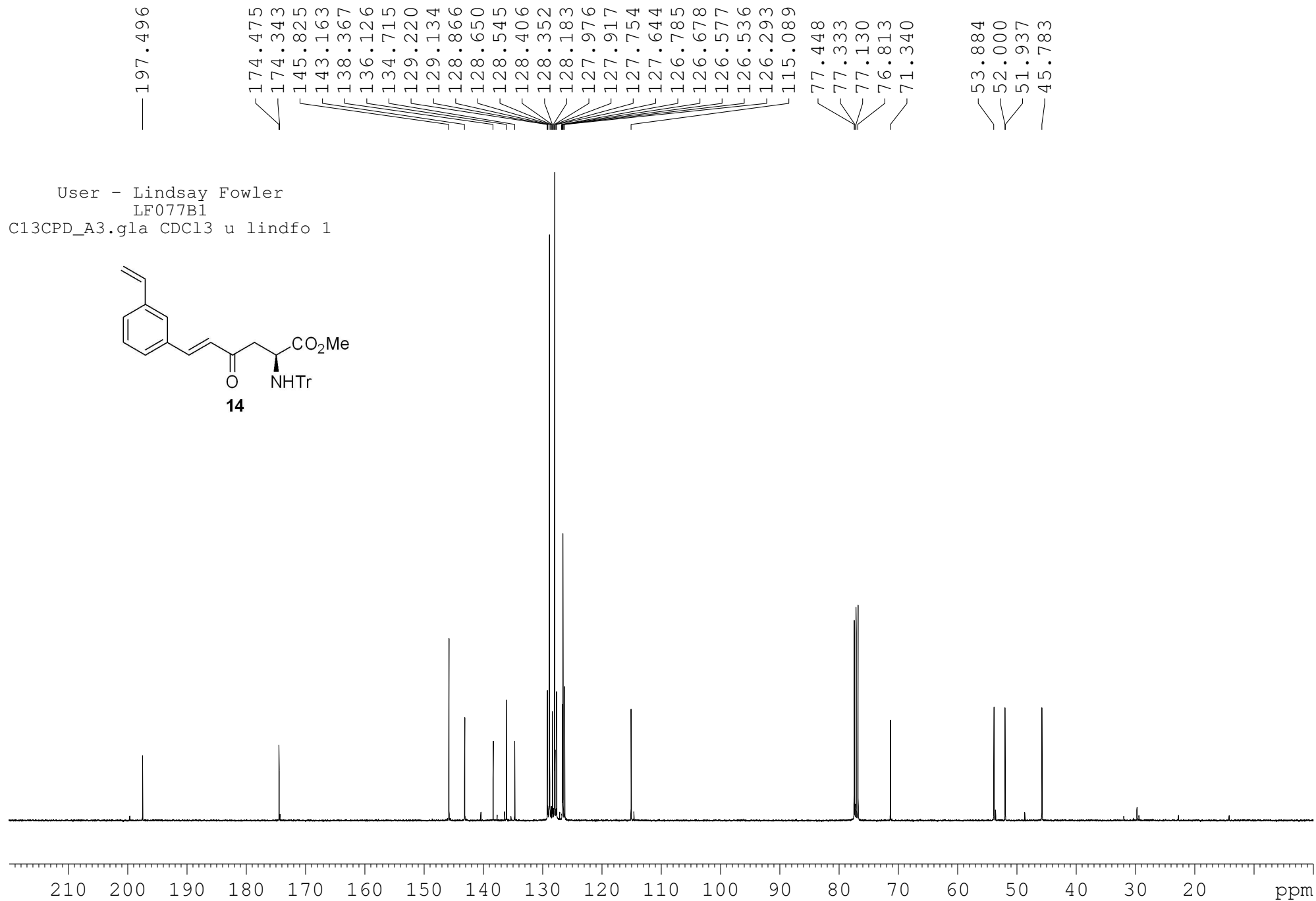
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User – Lindsay Fowler
LF077B1
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14

CO₂Me

NHTr

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

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LF123B1
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LF123B1
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15

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3.706 3.249 2.852 2.790 2.777 2.761 2.741 2.723 2.688 2.652 2.635 2.614 2.597 2.531 2.513 2.494 2.476

0.000

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LF079B1
PROTON_C_A3.gla CDCl3 u lindfo 9

16

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75.030
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32.408

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    LF079B1
C13CPD_A3.gla CDCl3 u lindfo 9

16

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

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LF080B1
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Compound **17**

197.595

174.535

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77.056
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45.604

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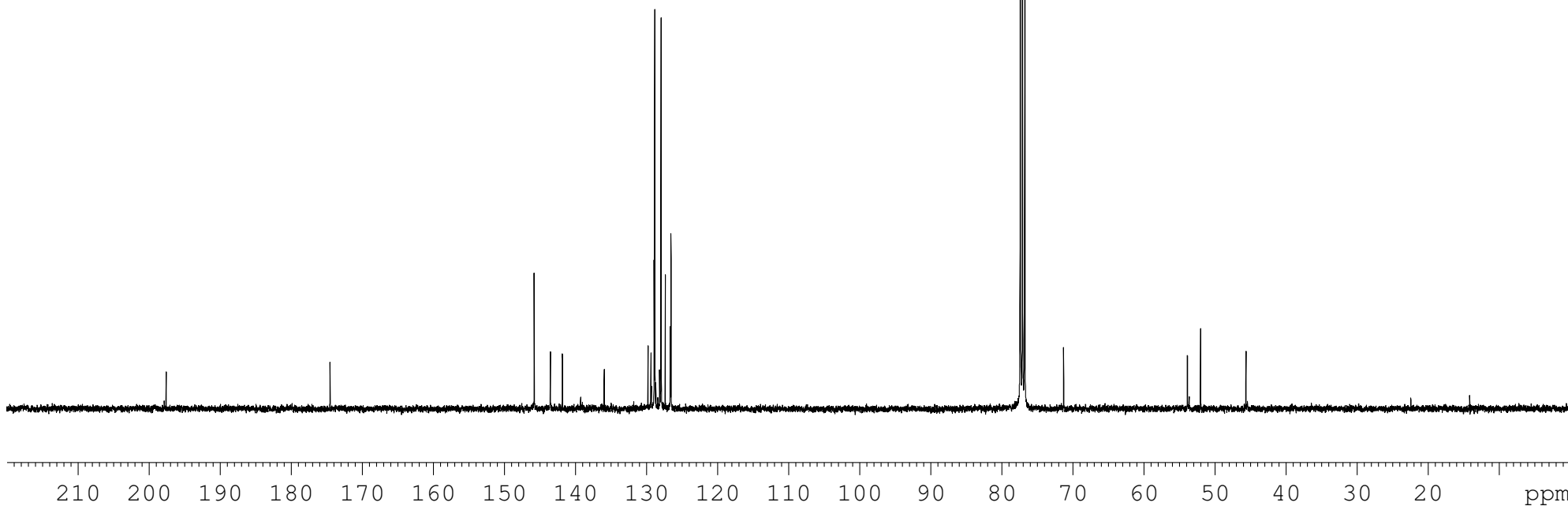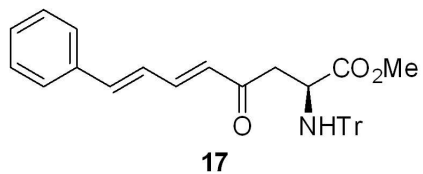
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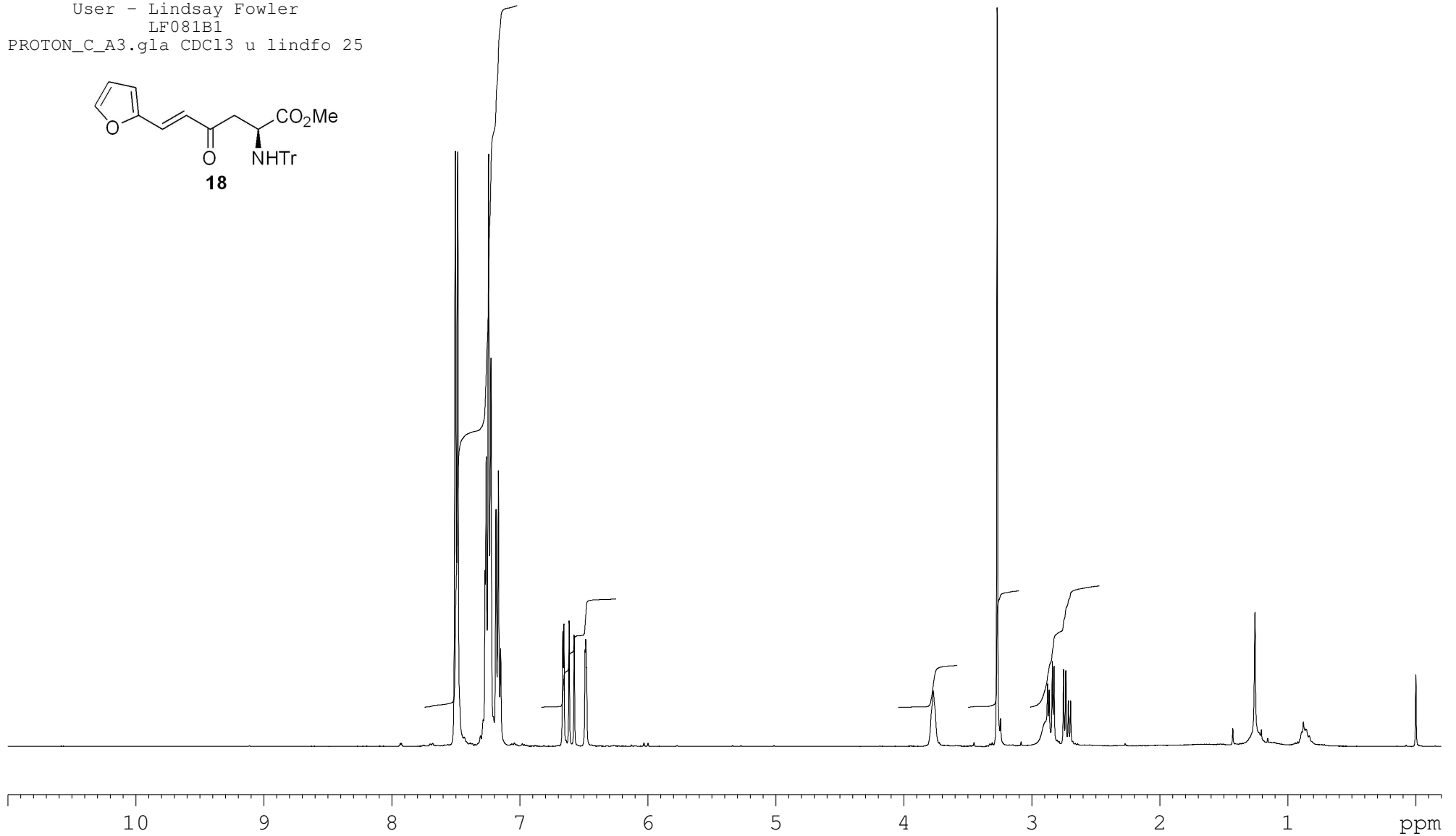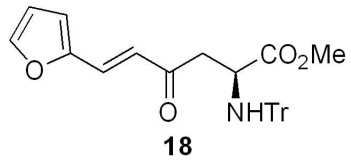
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145.812
145.503
145.194
144.931
130.113
129.820
129.751
129.310
129.124
128.852
128.631
128.583
128.476
128.351
128.267
127.947
127.270
126.947
126.757
126.551
123.611
116.178
113.023
112.812
112.694

77.450
77.334
77.132
76.814
71.311

53.863
52.371
51.984
46.189

User – Lindsay Fowler
LF081B1
C13CPD_A3.gla CDCl3 u lindfo 6

18

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm
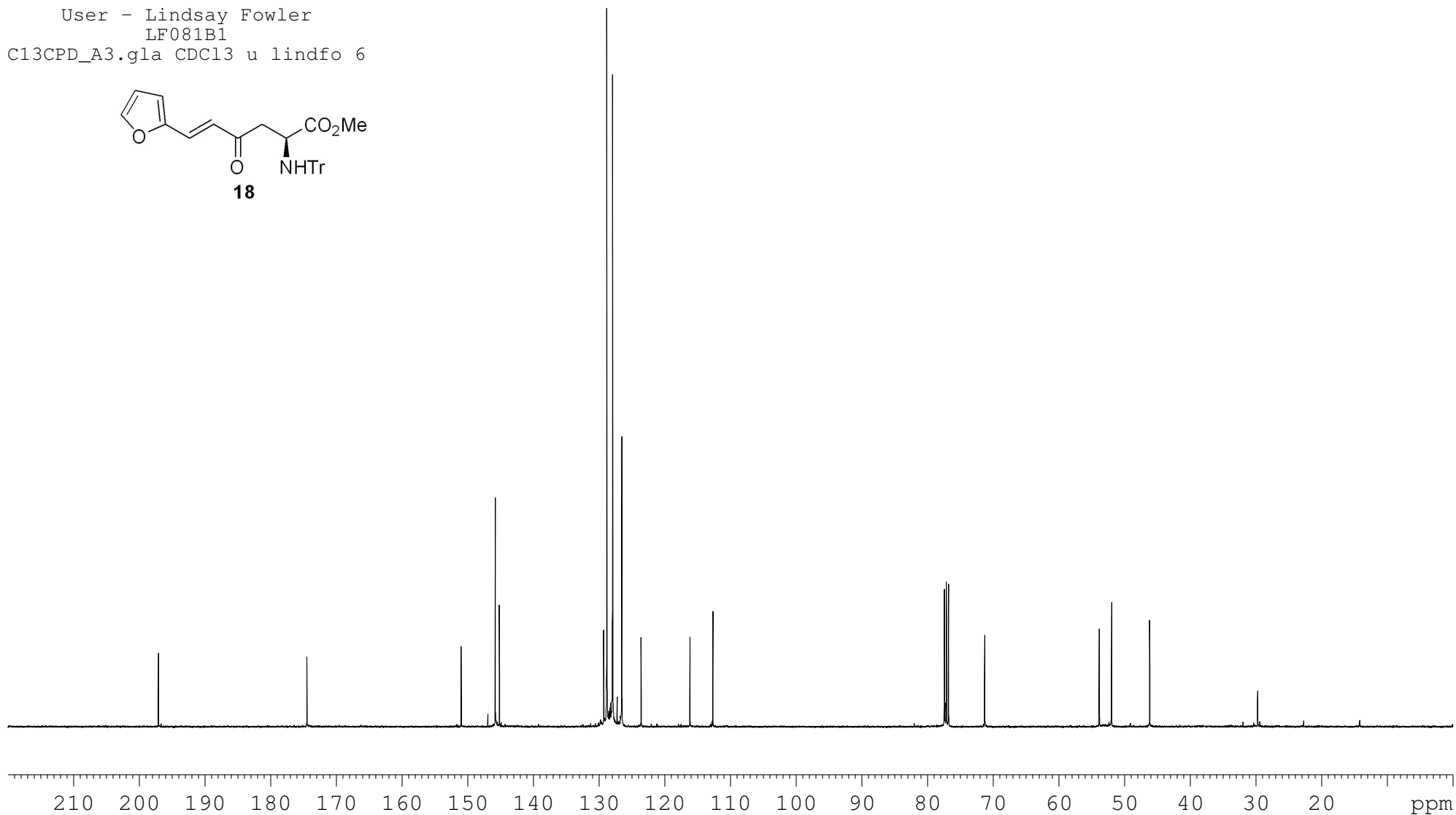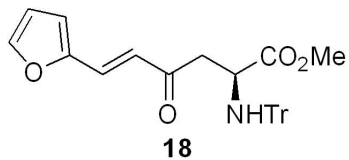
7.534 7.494 7.424 7.421 7.416 7.403 7.386 7.301 7.288 7.210 7.188 7.186 7.176 7.174 7.156 7.141 7.136 7.125 7.117 7.105 7.098 7.095 7.092 7.082 7.077 7.072 7.062 7.059 7.056 6.970 6.961 6.957 6.948 6.434 6.394

3.723 3.692

3.188

2.816 2.801 2.787 2.763 2.750 2.687 2.673 2.656 2.636 2.618

0.000

User – Lindsay Fowler
LF084B1
PROTON_C_A3.gla CDCl3 u lindfo 52

10   9   8   7   6   5   4   3   2   1   ppm
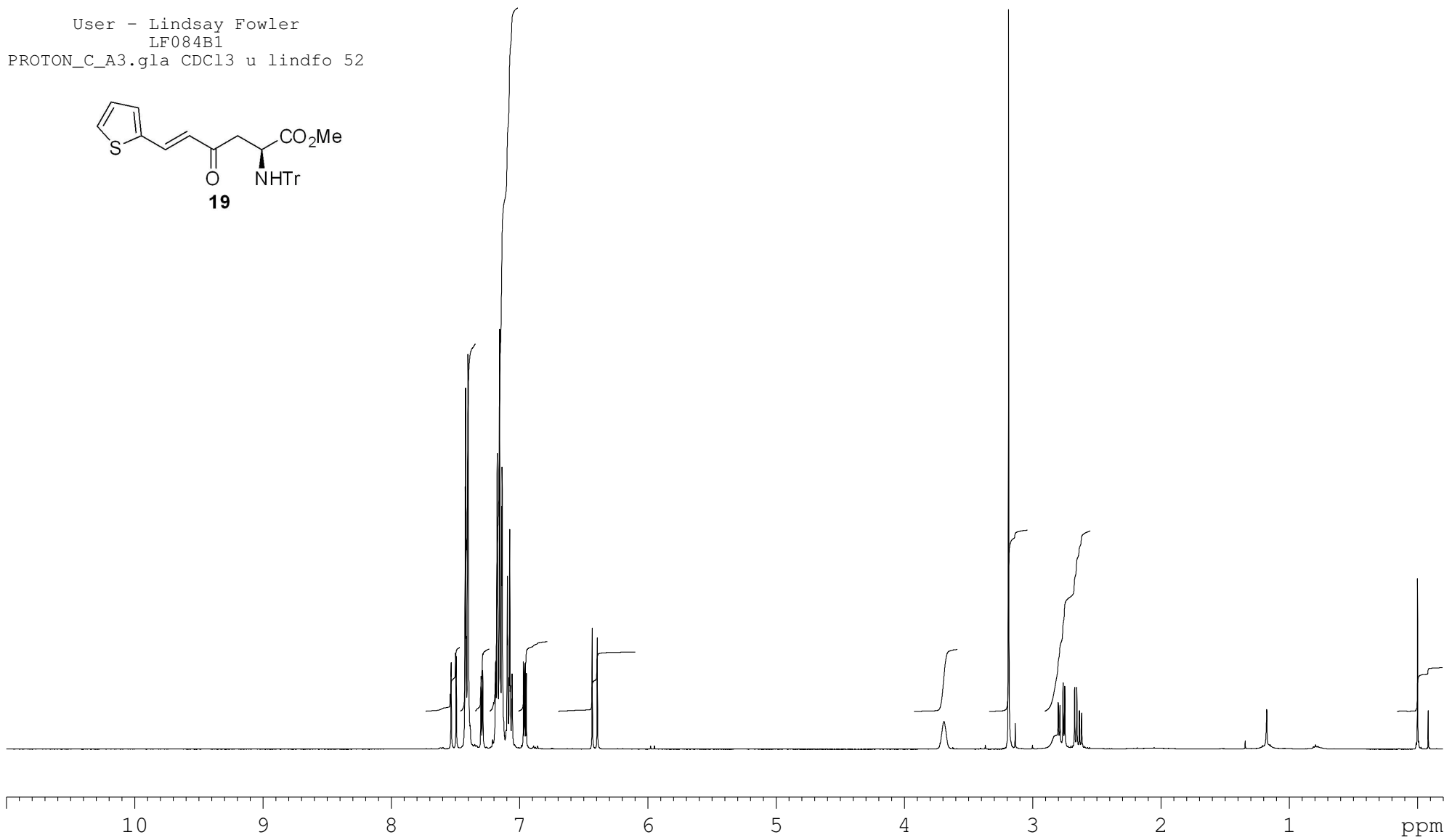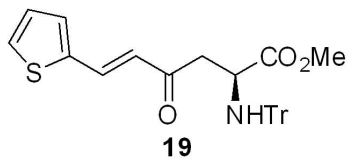
195.742

173.228

144.567
138.586
134.454
130.699
127.912
127.618
127.401
127.154
126.941
126.736
126.493
125.695
125.539
125.413
125.334
123.925

76.194
76.079
75.876
75.559
70.079

52.769
52.652
50.778
50.701
44.716

User – Lindsay Fowler
LF084B1
C13CPD_A3.gla CDCl3 u lindfo 8

19

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20   ppm
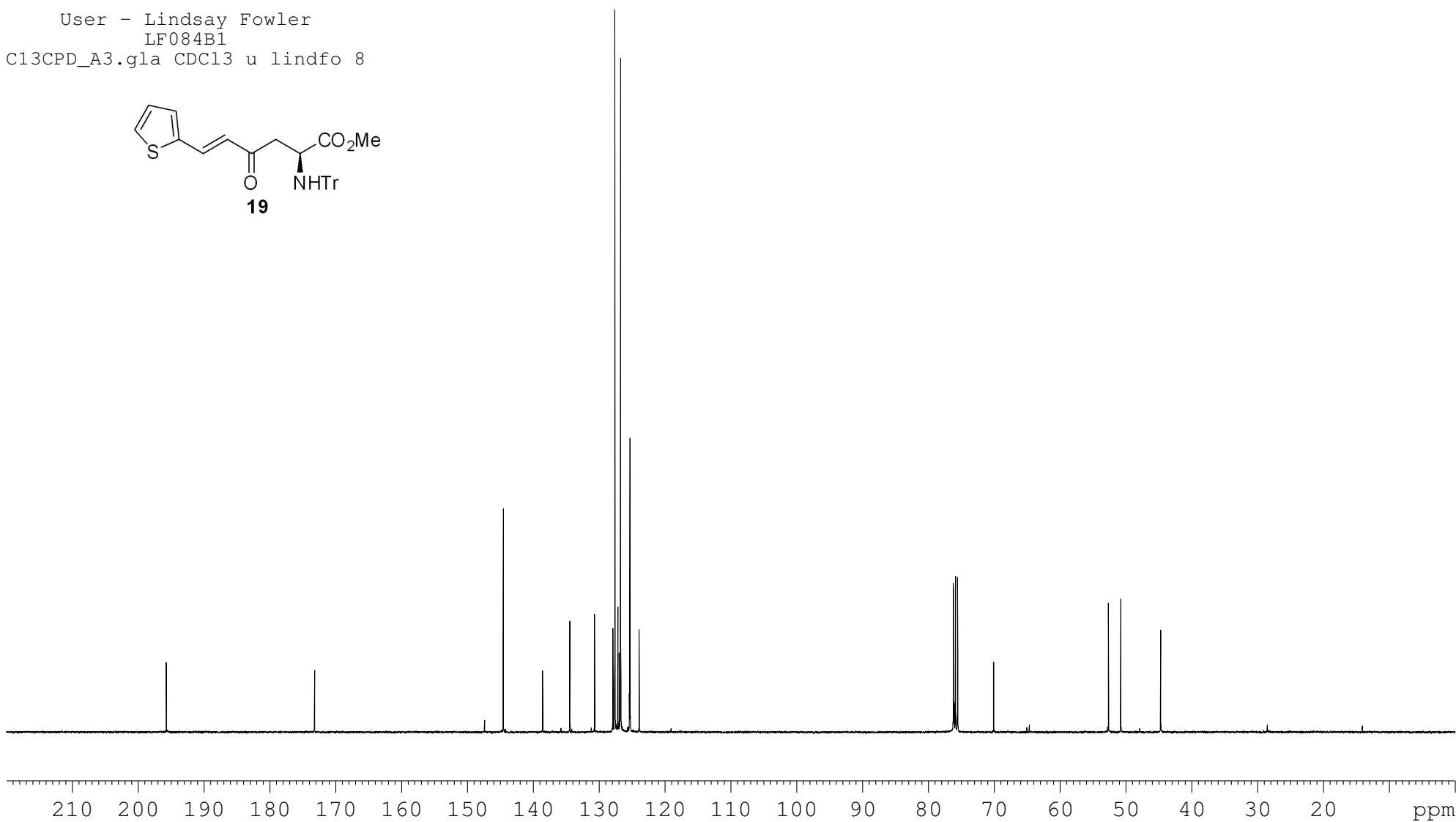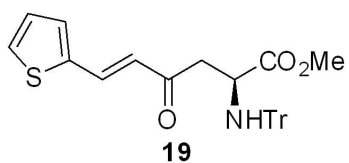
8.476
8.232
8.212
7.943
7.924
7.664
7.660
7.646
7.626
7.606
7.540
7.522
7.502
7.466
7.273
7.255
7.248
7.236
7.196
7.178
7.160
6.773
6.732

3.817
3.300
2.969
2.956
2.931
2.919
2.846
2.828
2.808
2.790

0.000

User – Lindsay Fowler
LF214B1
PROTON_C_A3.gla CDCl3 u lindfo 20

20

O$_2$N

CO$_2$Me

O   NHTr

10   9   8   7   6   5   4   3   2   1   ppm
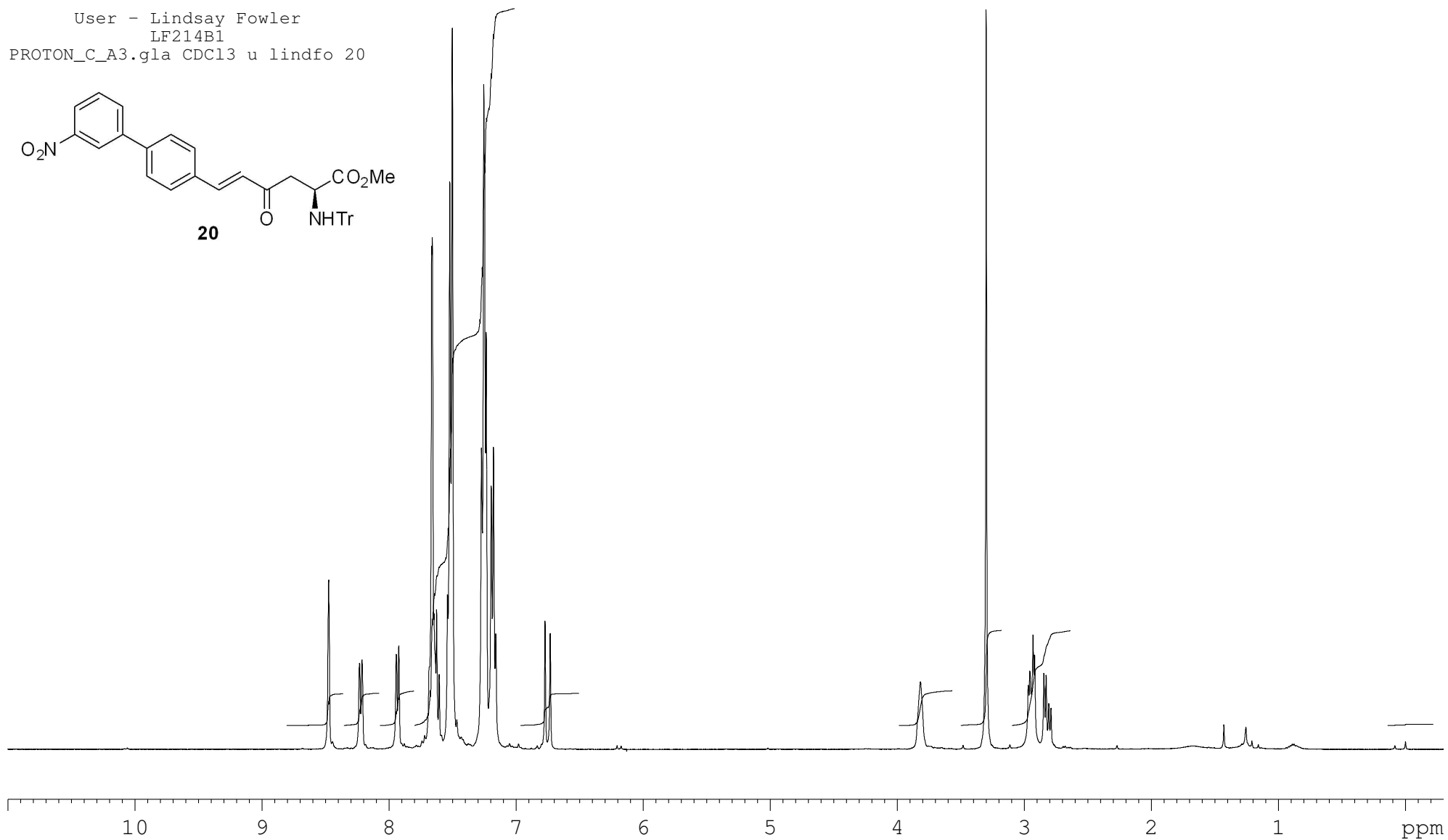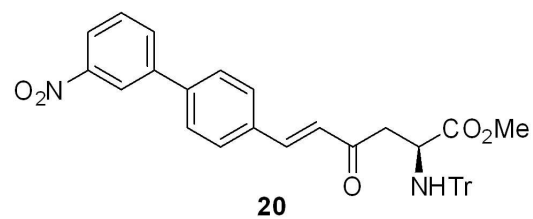
197.418

174.469

148.823
145.810
142.183
141.737
140.581
134.668
132.939
130.007
130.183
129.183
128.854
128.330
128.168
127.990
127.736
126.986
126.804
126.598
122.641
122.401
121.885

77.464
77.347
77.146
76.828
71.328

53.823
52.061

45.876

User – Lindsay Fowler
LF214B1
C13CPD_A3.gla CDCl3 u lindfo 2

20

O_2N

CO_2Me

O NHTr

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

User – Lindsay Fowler
LF218B1
PROTON_C_A3.gla CDCl3 u lindfo 21
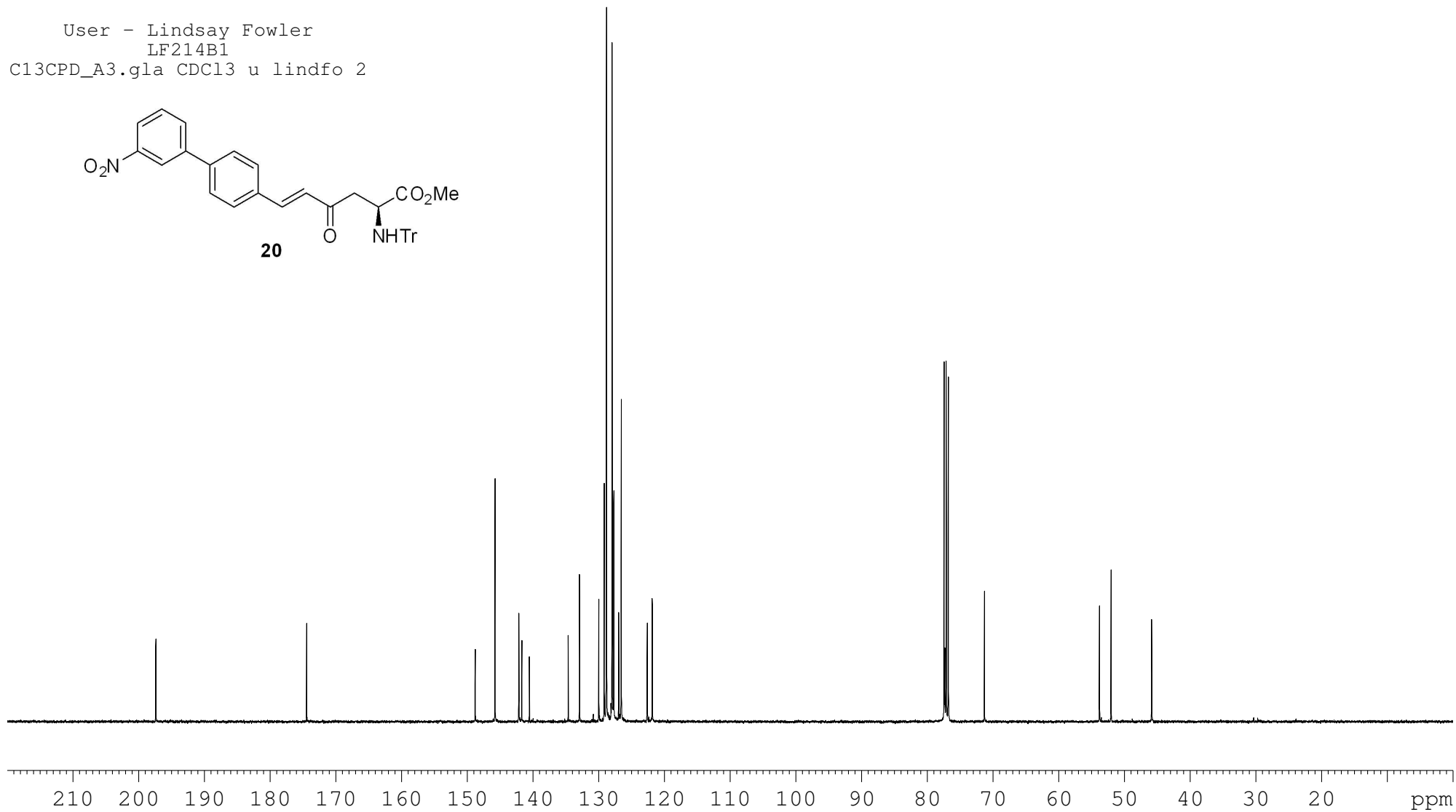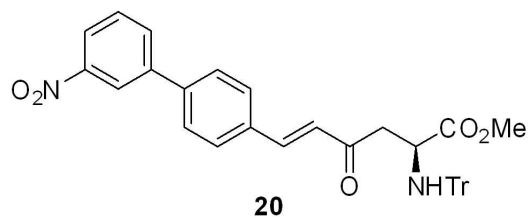
7.600
7.591
7.571
7.515
7.496
7.479
7.417
7.397
7.378
7.259
7.234
7.195
7.177
7.159
6.741
6.728
6.688

5.230

3.796
3.289
2.939
2.915
2.826
2.808
2.788
2.770

0.000

21

10   9   8   7   6   5   4   3   2   1   ppm
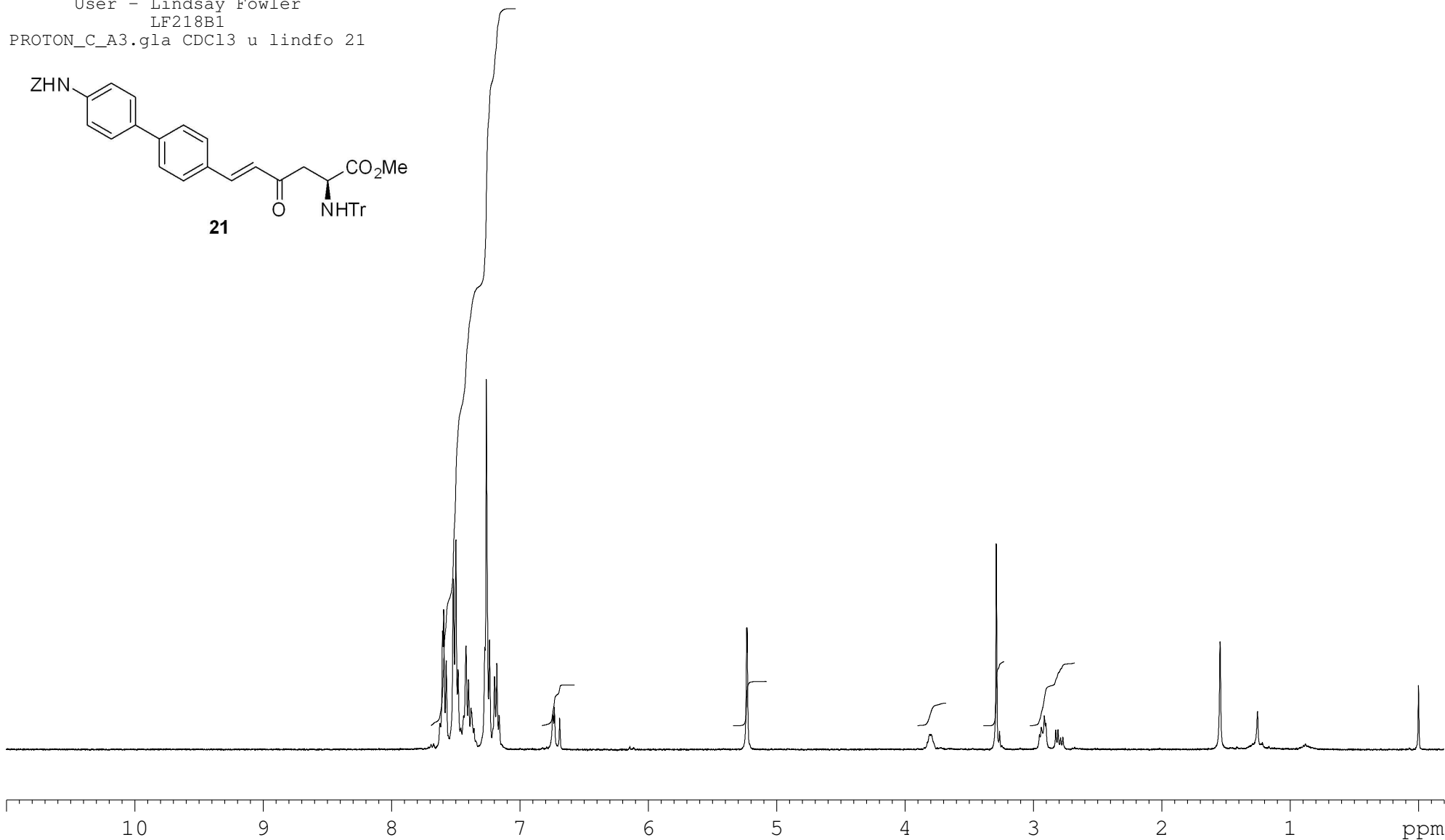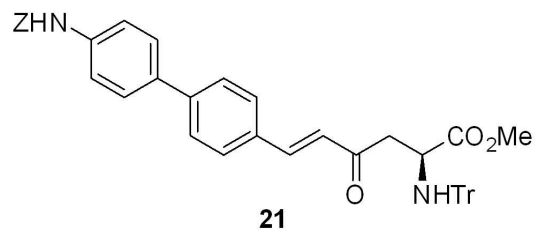
197.570

174.564

153.294
145.810
142.953
142.703
137.854
135.980
135.060
133.053
128.969
128.849
128.696
128.475
128.398
127.966
127.681
127.191
126.562
126.079
119.019

77.404
77.289
77.086
76.769
71.310
67.187

53.867
52.026

45.740

User – Lindsay Fowler
LF218B1
C13CPD_A3.gla CDCl3 u lindfo 7

21

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20   ppm

User – Lindsay Fowler
LF216B1
PROTON_C_A3.gla CDCl3 u lindfo 22
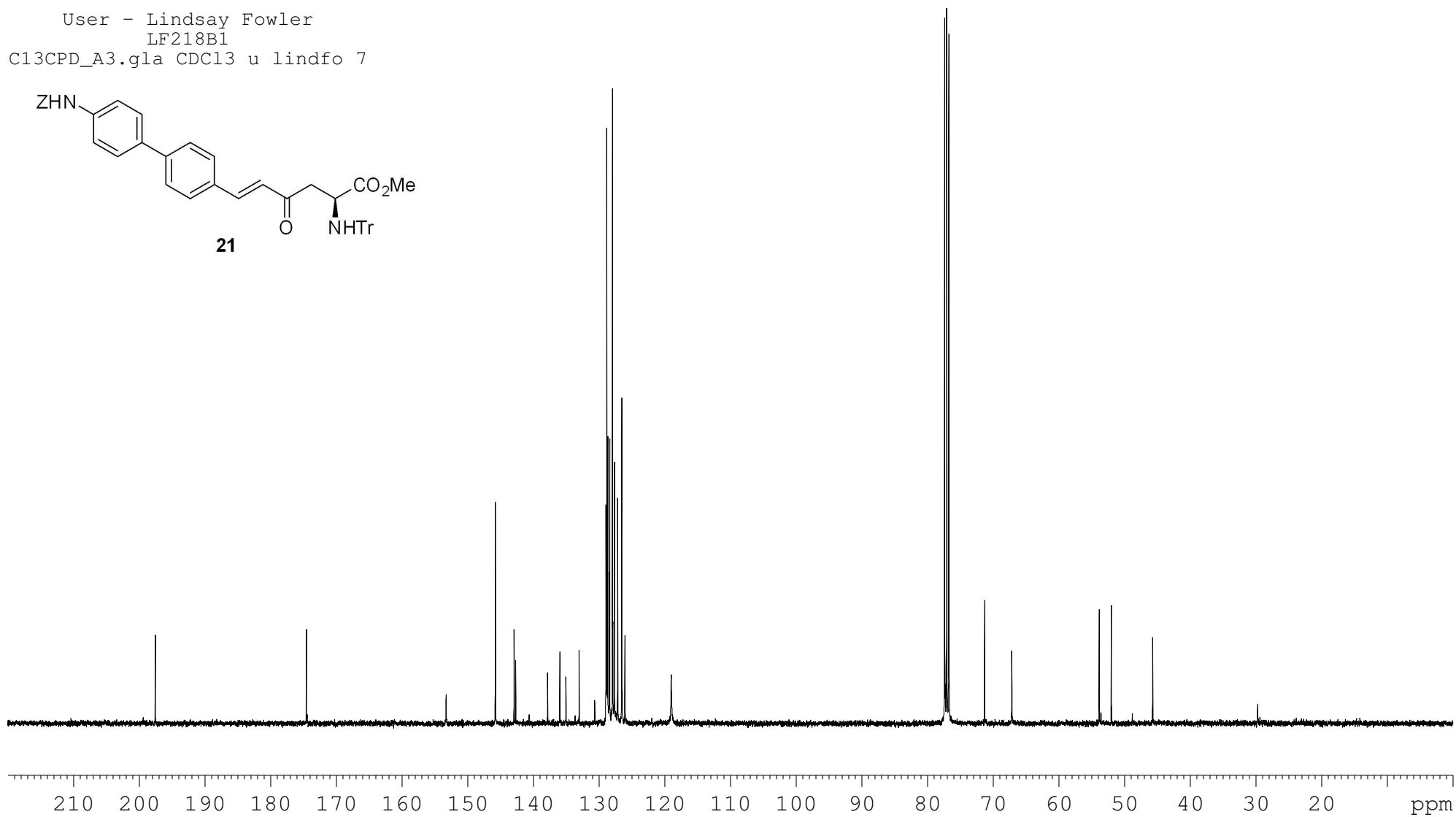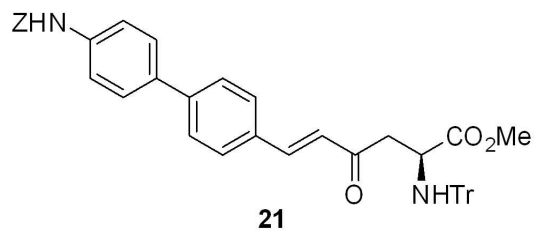
7.731 7.710 7.697 7.513 7.494 7.282 7.269 7.252 7.232 7.189 7.172 7.148 7.126 7.104 6.752 6.744 6.705 6.696 6.690 6.650

3.796 3.272 2.930 2.917 2.893 2.880 2.795 2.777 2.757 2.740

0.000

22
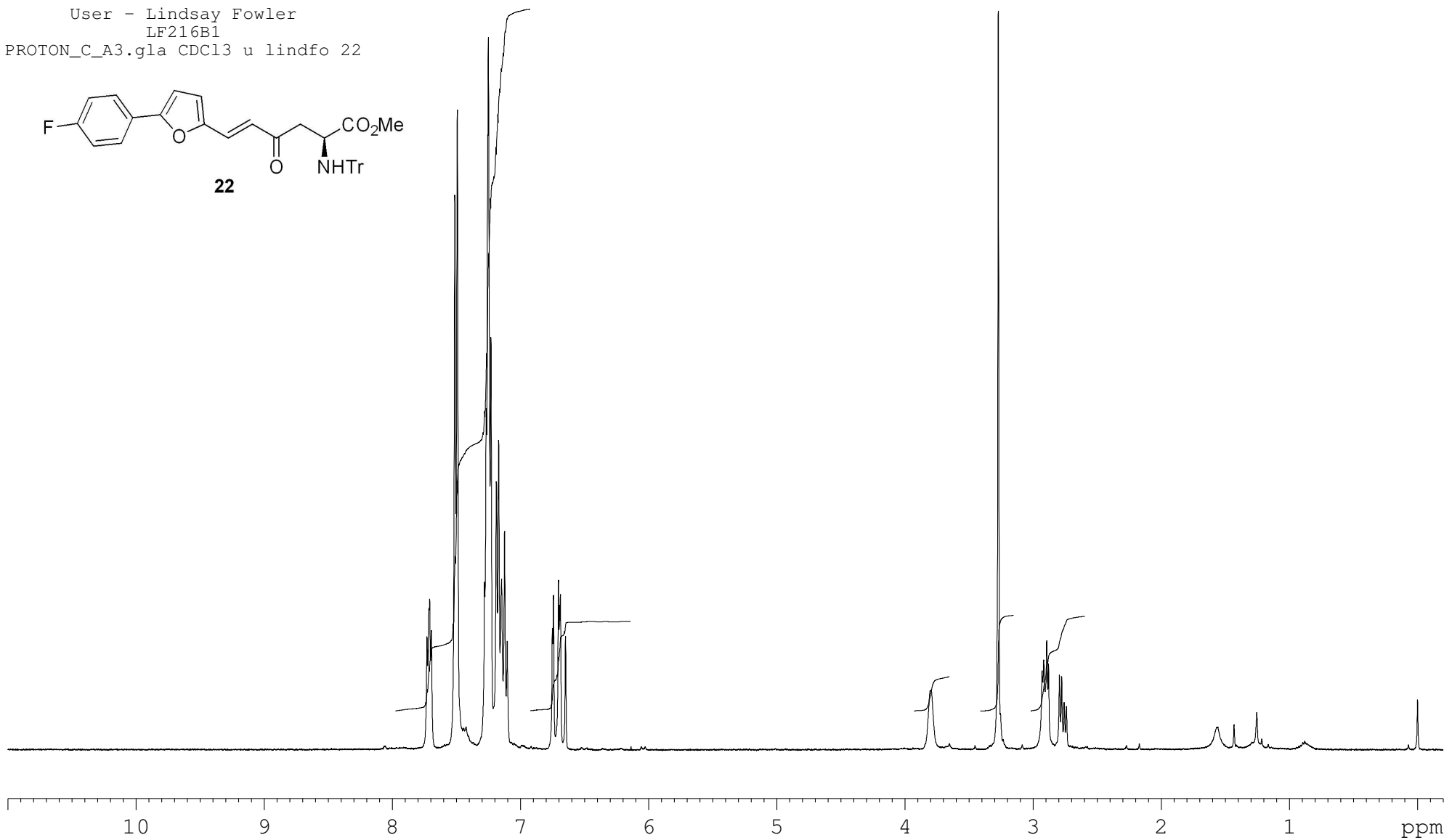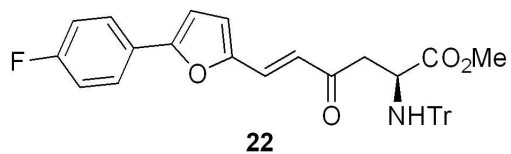
196.896

174.496
164.141
161.661
155.646
150.429
145.826
128.990
128.870
128.806
128.649
128.553
128.178
127.961
127.717
126.765
126.674
126.561
126.397
126.314
126.179
126.148
123.071
118.787
116.199
115.979
107.889
77.453
77.337
77.135
76.817
71.336
53.991
51.991
46.270

User – Lindsay Fowler
C13CPD_A3.gla CDCl3 u lindfo 3
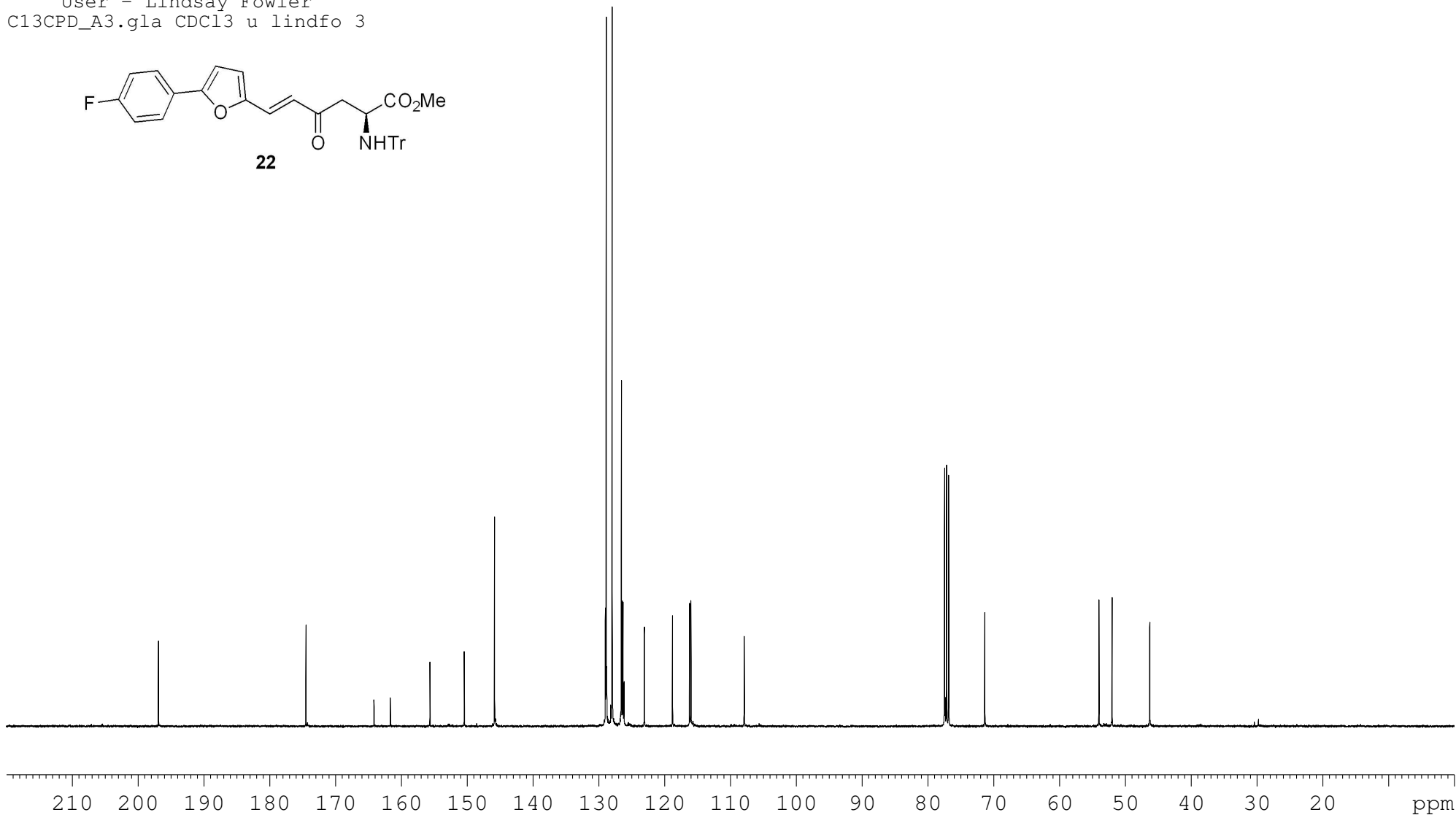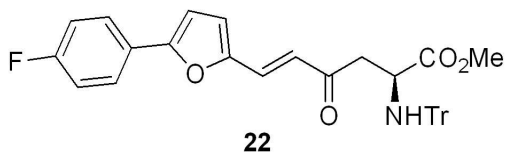
22

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

User Lindsay Fowler
LF196B3
PROTON_C_A3.gla CDCl3 u lindfo 12

30
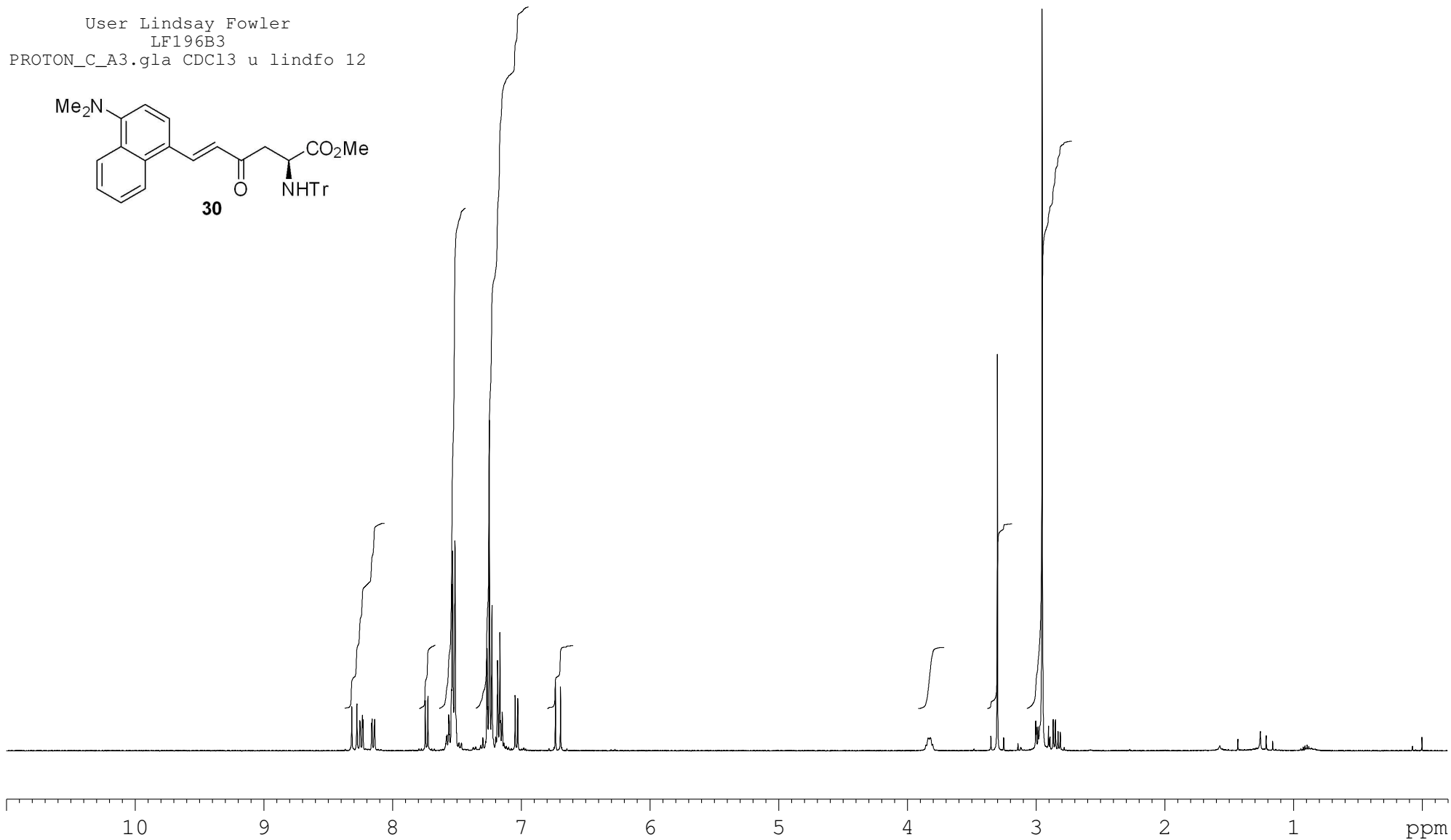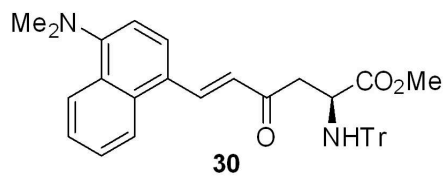
197.414

174.657

153.762
145.848
140.200
133.080
128.859
128.357
127.926
126.847
126.557
126.511
125.843
125.415
125.274
125.197
123.592
113.349

77.358
77.244
77.041
76.723
71.305

53.966
51.977
45.805
44.917

User Lindsay Fowler
LF196B3
C13CPD_A3.gla CDCl3 u lindfo 12

Me₂N

CO₂Me

O   NHTr

30

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20   ppm
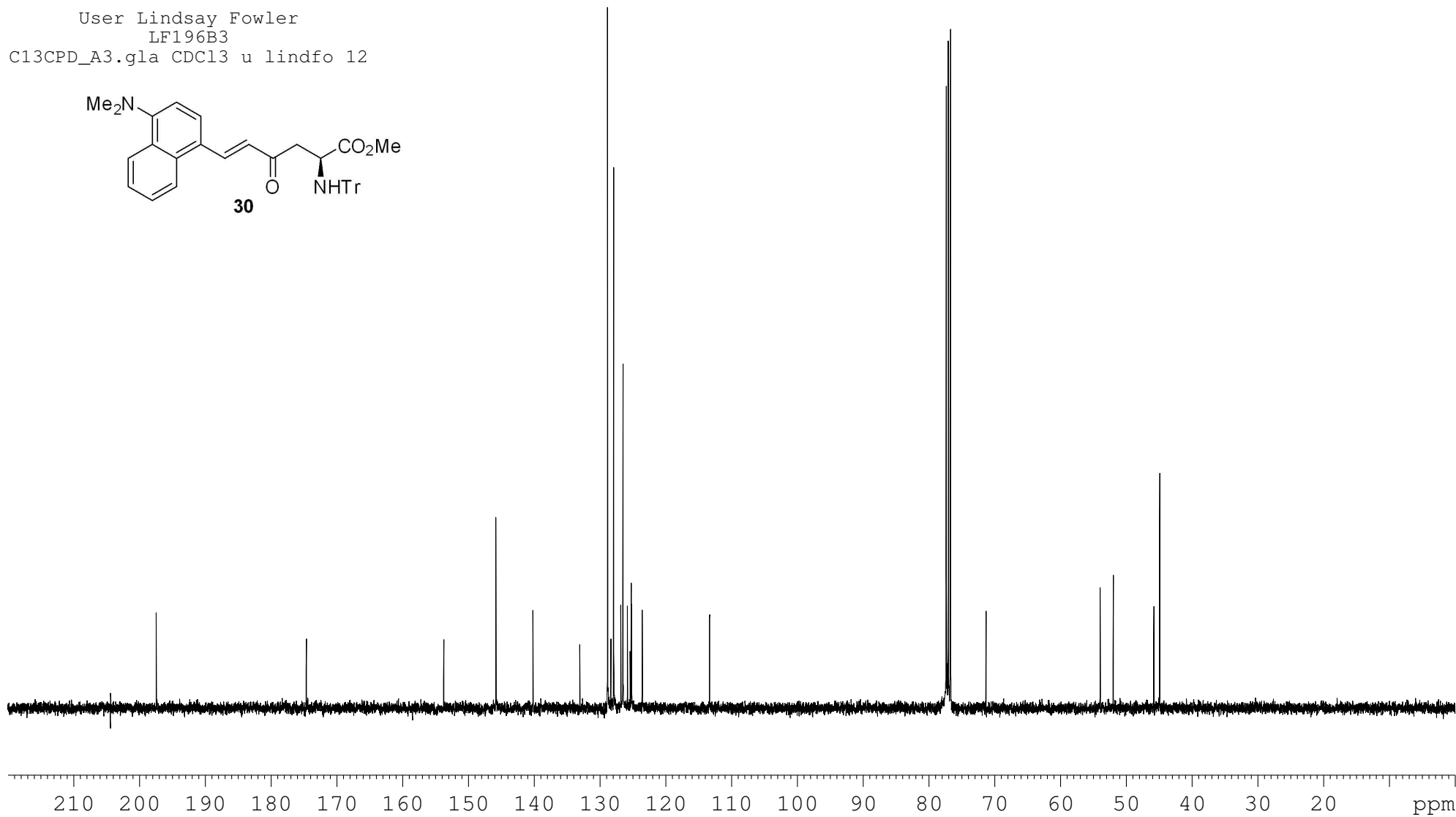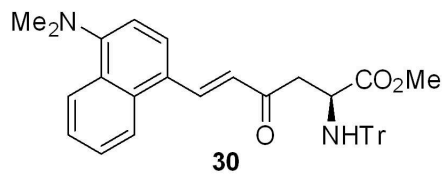
7.588
7.554
7.548
7.540
7.530
7.517
7.411
7.406
7.398
7.394
7.384
7.370
7.361
7.287
6.730
6.689
5.639
5.617
4.648
4.637
4.626
4.615
4.605
3.744
3.476
3.465
3.431
3.420
3.267
3.256
3.222
3.211
1.445
0.000

User Lindsay Fowler
LF173B1
PROTON_C_A3.gla CDCl3 u lindfo 13

23

10   9   8   7   6   5   4   3   2   1   ppm
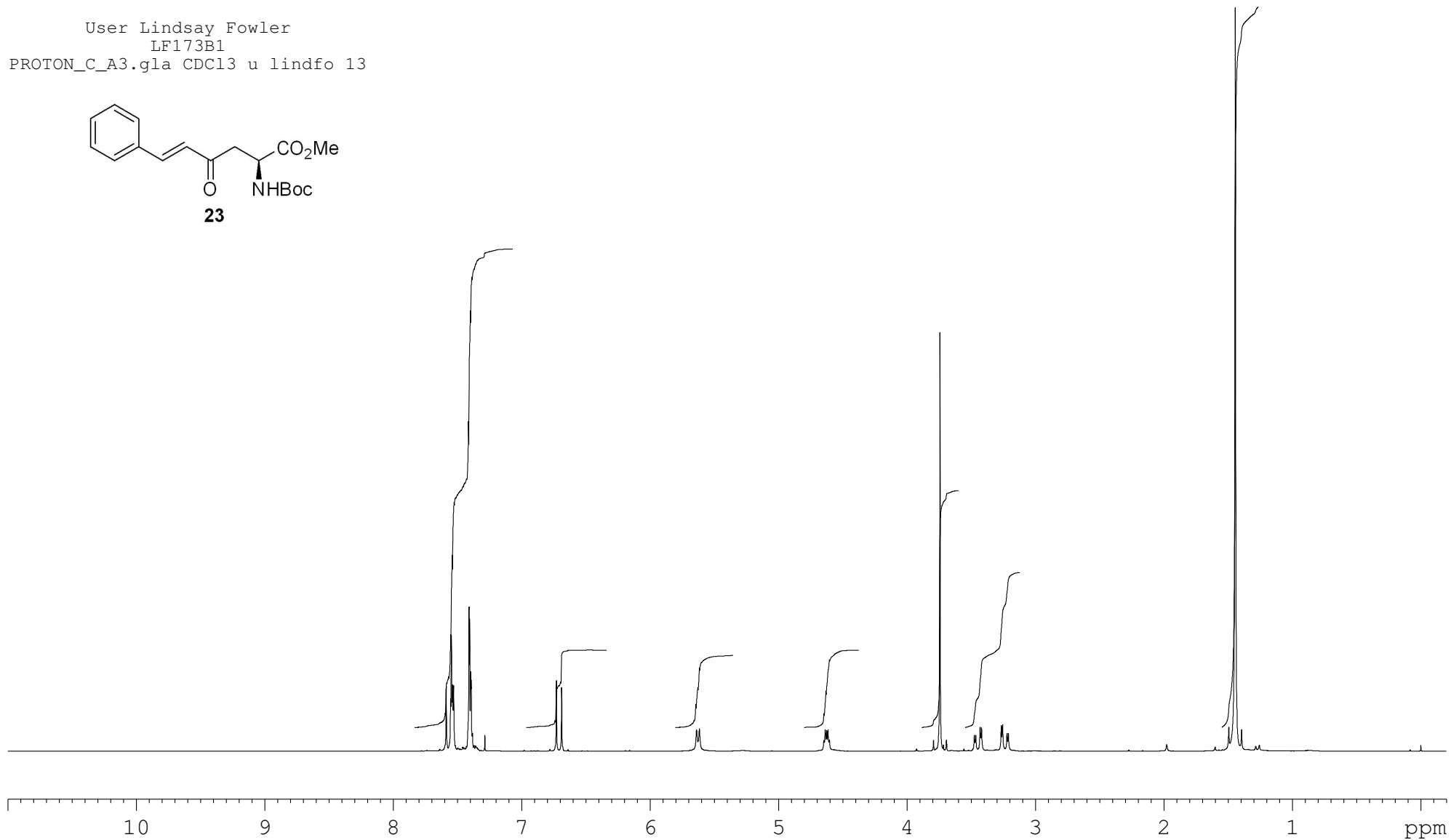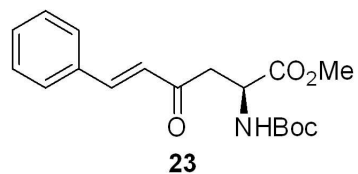
197.615

171.984

155.559

143.918
134.117
130.841
130.589
129.229
129.011
128.825
128.678
128.572
128.421
125.585

79.923
77.452
77.336
77.134
76.815

52.580
49.627

42.430

28.497
28.311
28.105

User – Lindsay Fowler
LF173B1
C13CPD_A3.gla CDCl3 u lindfo 6

23

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm
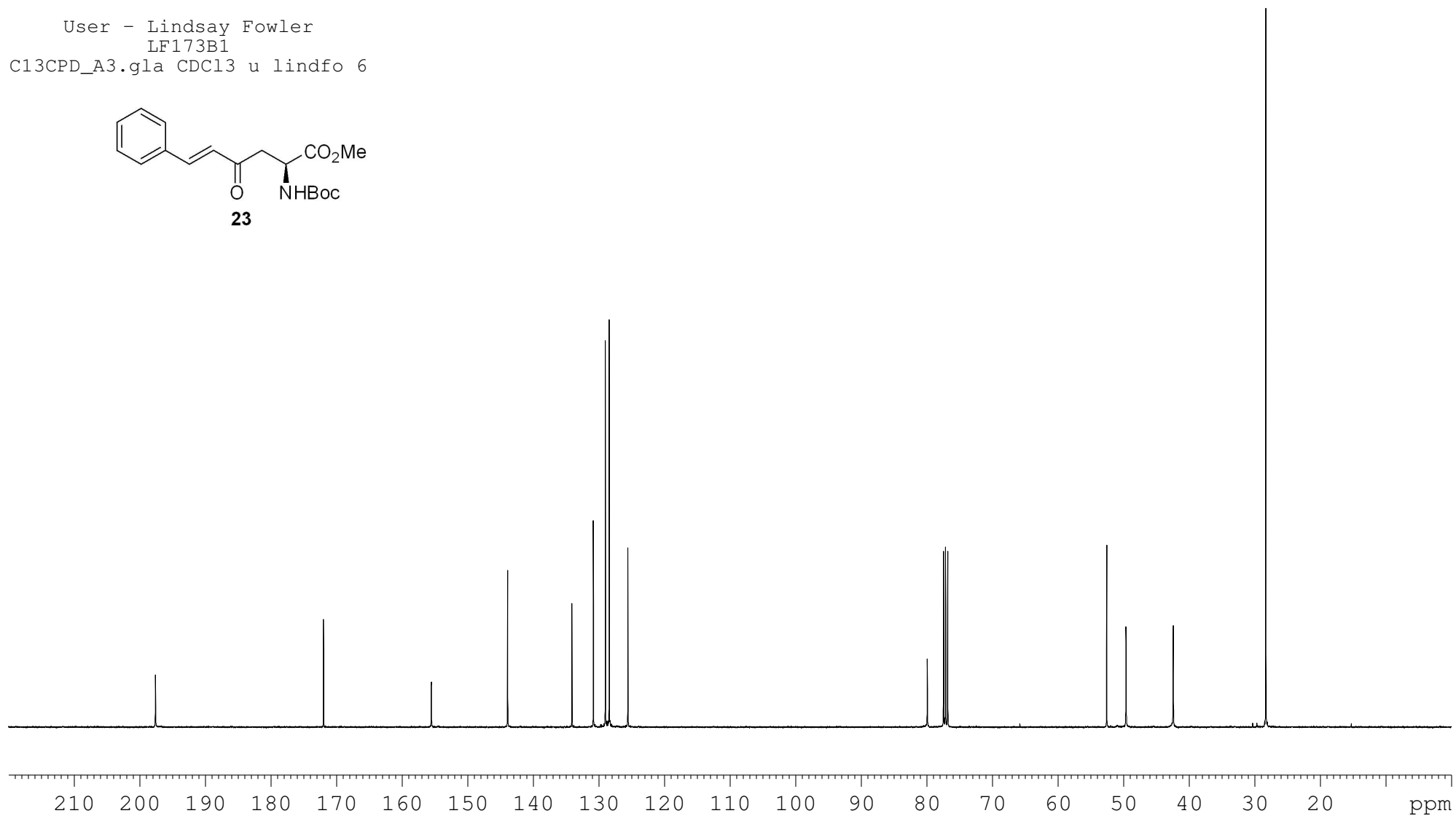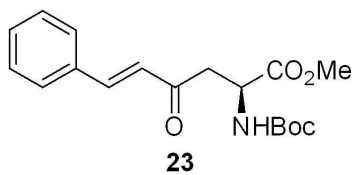
7.548
7.528
7.474
7.413
7.393
7.261
6.709
6.668

5.570
5.550

4.607

3.748
3.444
3.399
3.244
3.201

1.442

0.000

User – Lindsay Fowler
LF183B1
PROTON_C_A3.gla CDCl3 u lindfo 18

24

10   9   8   7   6   5   4   3   2   1   ppm
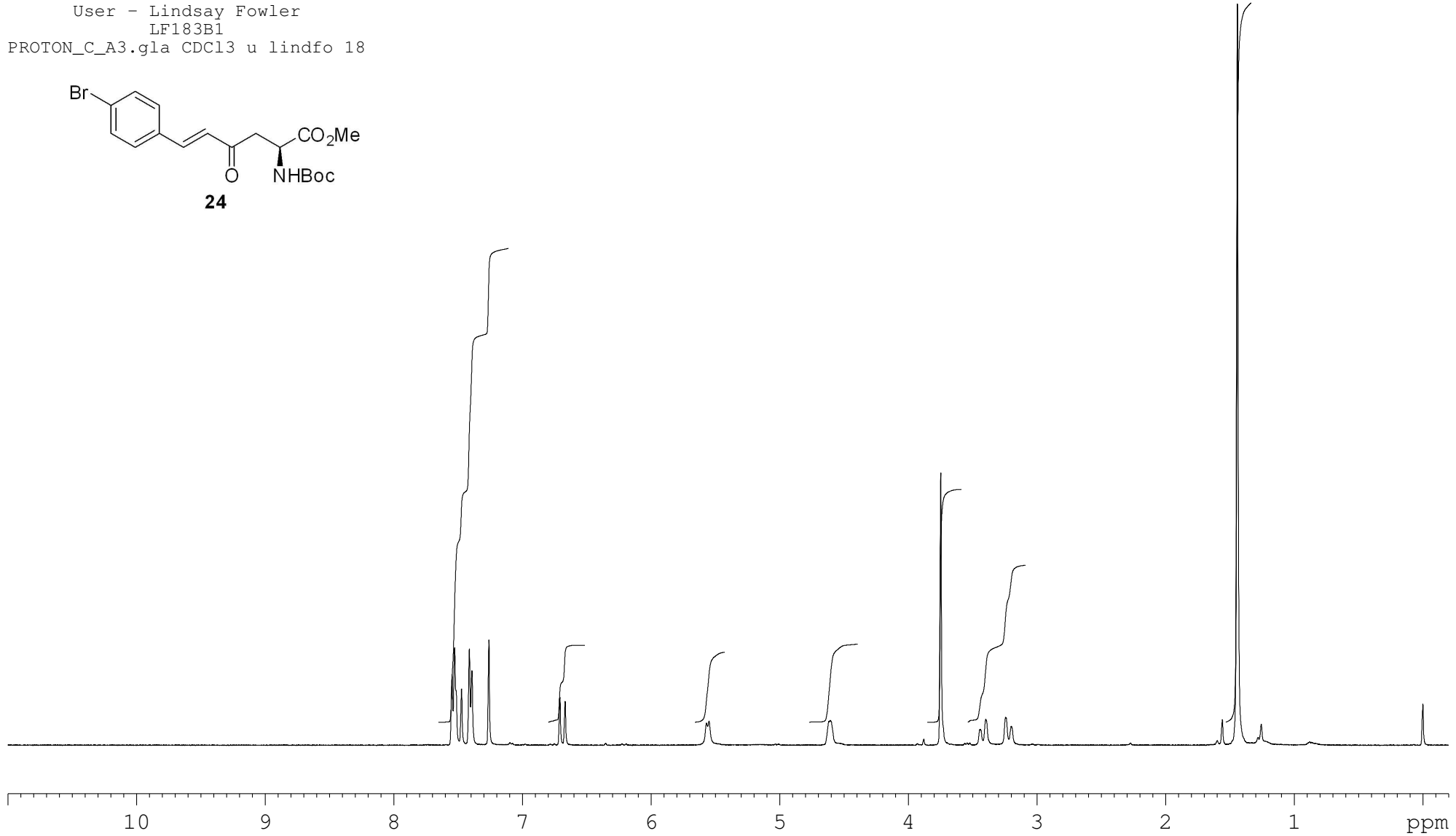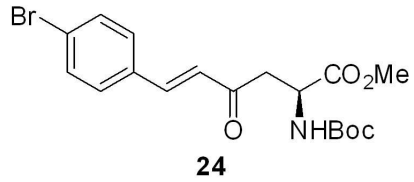
197.494

171.961

155.558

142.505
133.016
132.293
130.028
129.785
125.954
125.205

80.033
77.434
77.318
77.116
76.798

52.699
49.549

42.626

28.334

User – Lindsay Fowler
LF183B1
C13CPD_A3.gla CDCl3 u lindfo 6

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm
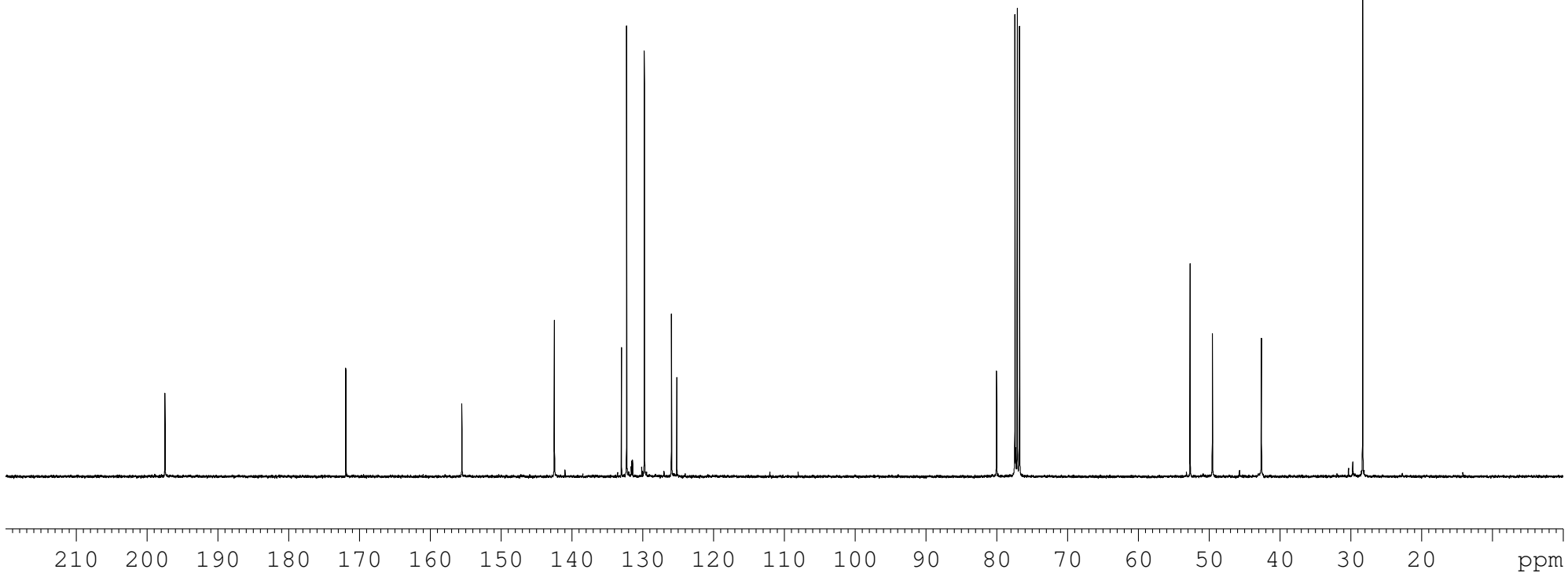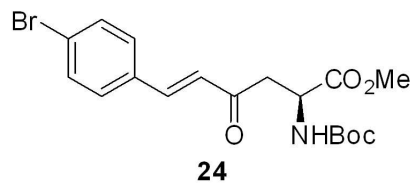
7.312
7.308
7.294
7.275
7.264
7.244
7.223
7.205
7.200
7.180
7.176
7.159
6.914
6.897
6.880
6.874
6.857
6.840
6.112
6.072
5.539
5.517
5.555
4.544
4.533
3.716
3.306
3.296
3.261
3.251
3.084
3.073
3.039
3.029
2.799
2.780
2.760
2.575
2.572
2.555
2.538
2.535
2.520
1.440

0.000

User Lindsay Fowler
LF189B1
PROTON_C_A3.gla CDCl3 u lindfo 23

25

10   9   8   7   6   5   4   3   2   1   ppm
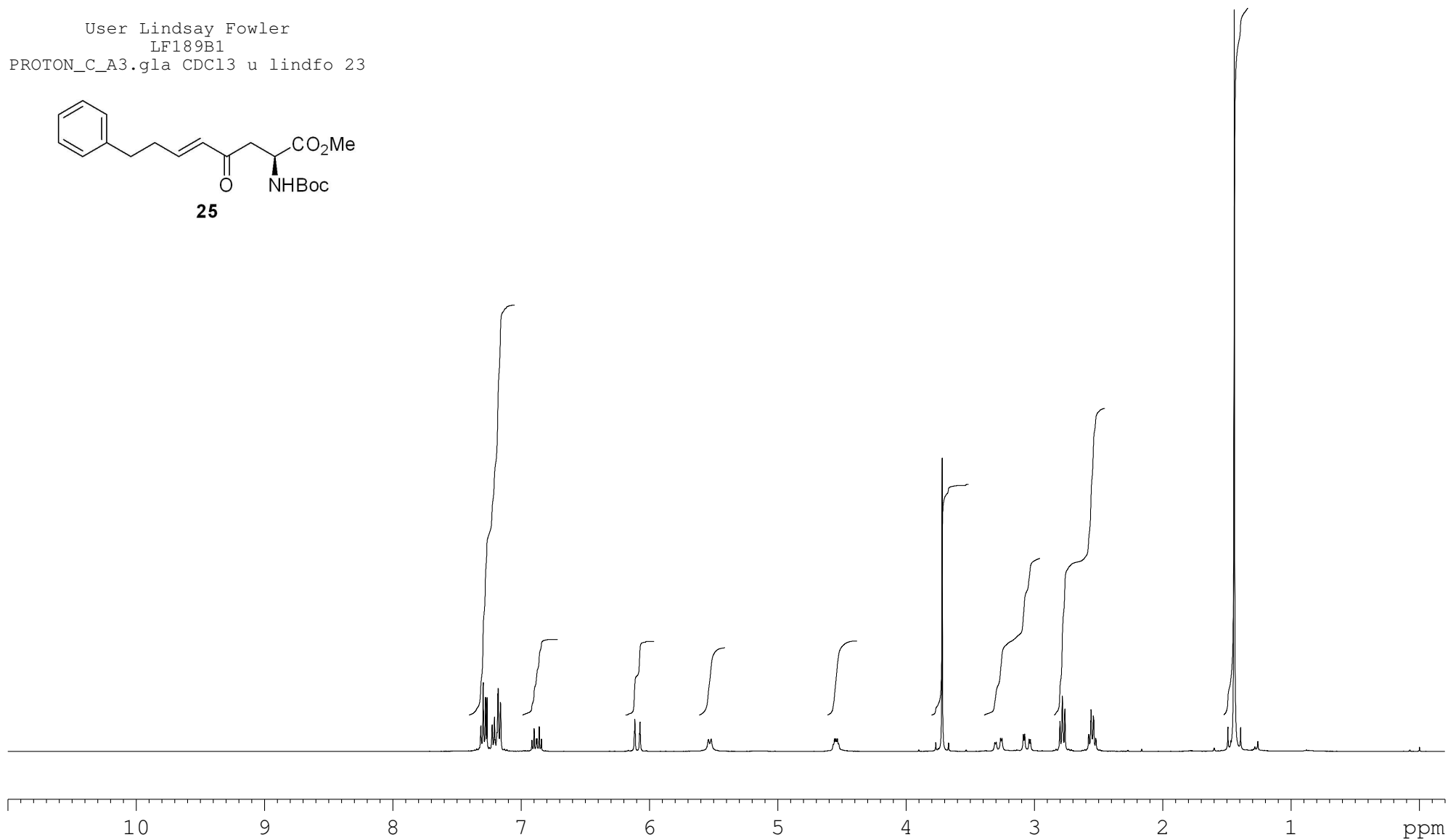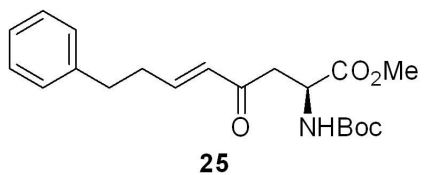
197.715

171.978

155.547

147.782

140.489

130.251
128.550
128.298
126.298

79.915
77.392
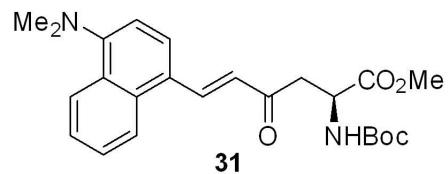77.276
77.074
76.756

52.555
49.488

41.798

34.261
34.191
28.311

User Lindsay Fowler
LF189B1
C13CPD_A3.gla CDCl3 u lindfo 23

25

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm
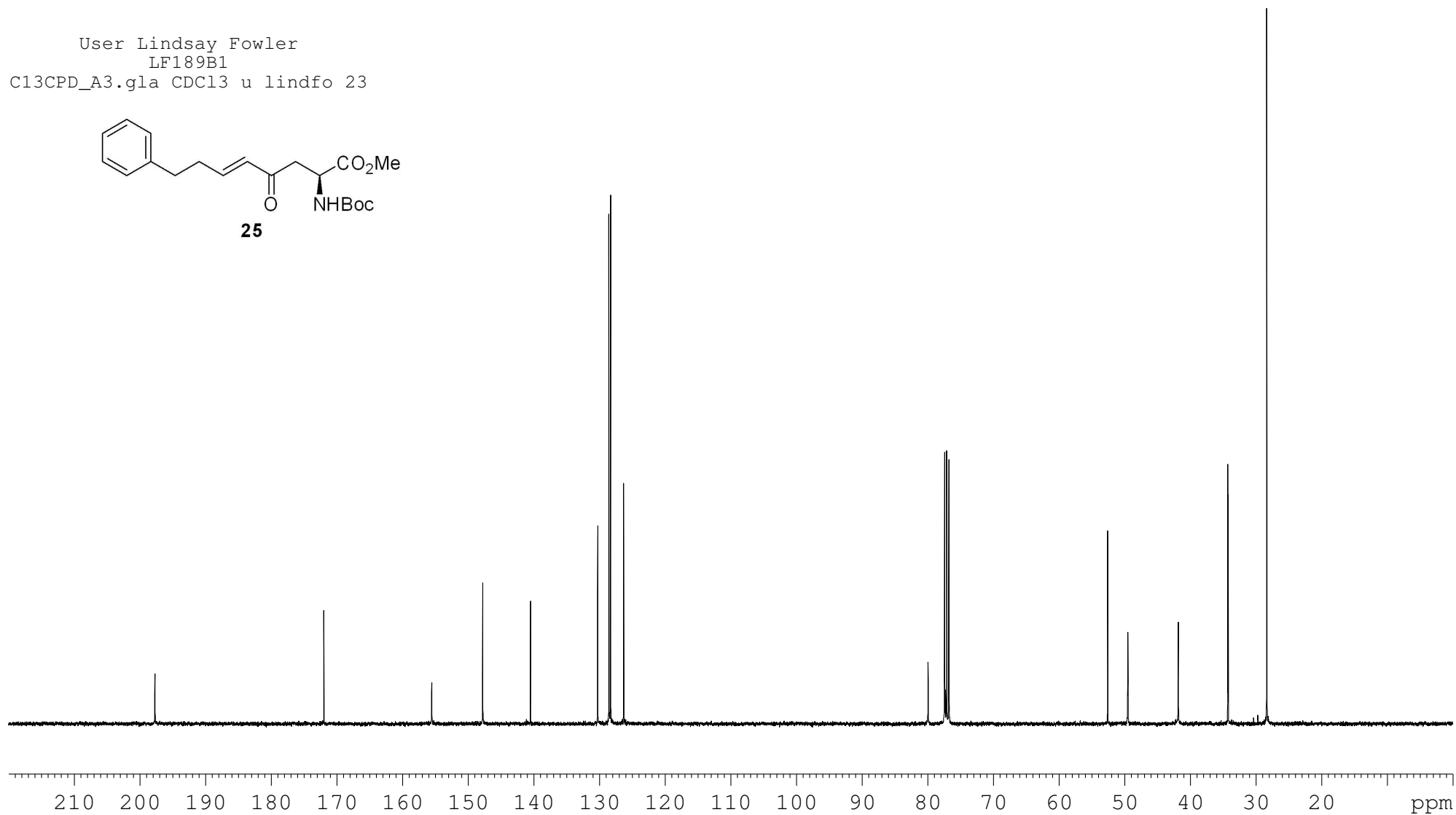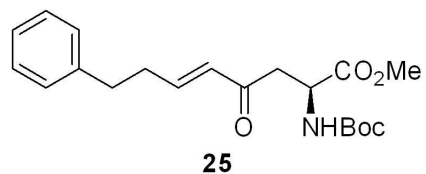
8.428 8.389 8.247 8.226 8.188 8.167 7.777 7.757 7.591 7.574 7.548 7.526 7.509 7.262 7.049 7.029 6.998 6.768 6.729 5.655 5.634

4.660 4.650 4.639 3.773 3.744 3.550 3.541 3.506 3.496 3.312 3.303 3.268 3.258 2.959 2.940

1.453 1.417

0.000

User – Lindsay Fowler
LF203B1
PROTON_C_A3.gla CDCl3 u lindfo 32

31

197.460

172.150

155.637
153.994

140.780
133.091
128.282
126.920
126.000
125.341
125.284
125.246
124.984
123.449
113.278

79.948
77.392
77.276
77.074
76.756

52.642
49.744
44.867
42.709

28.494

User Lindsay Fowler
LF203B3
C13CPD_A3.gla CDCl3 u lindfo 21

Me$_2$N

CO$_2$Me

O   NHBoc

31

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm
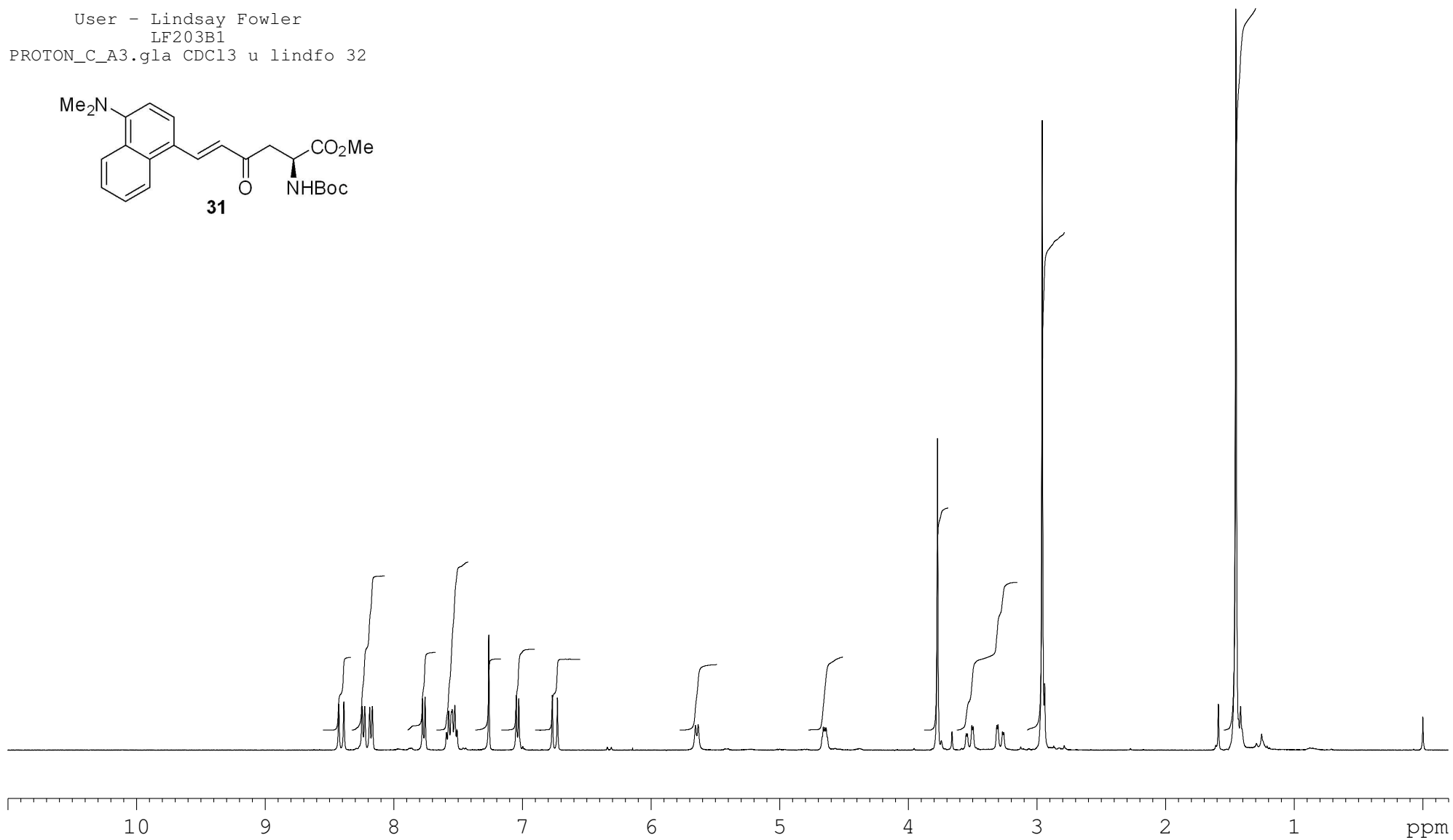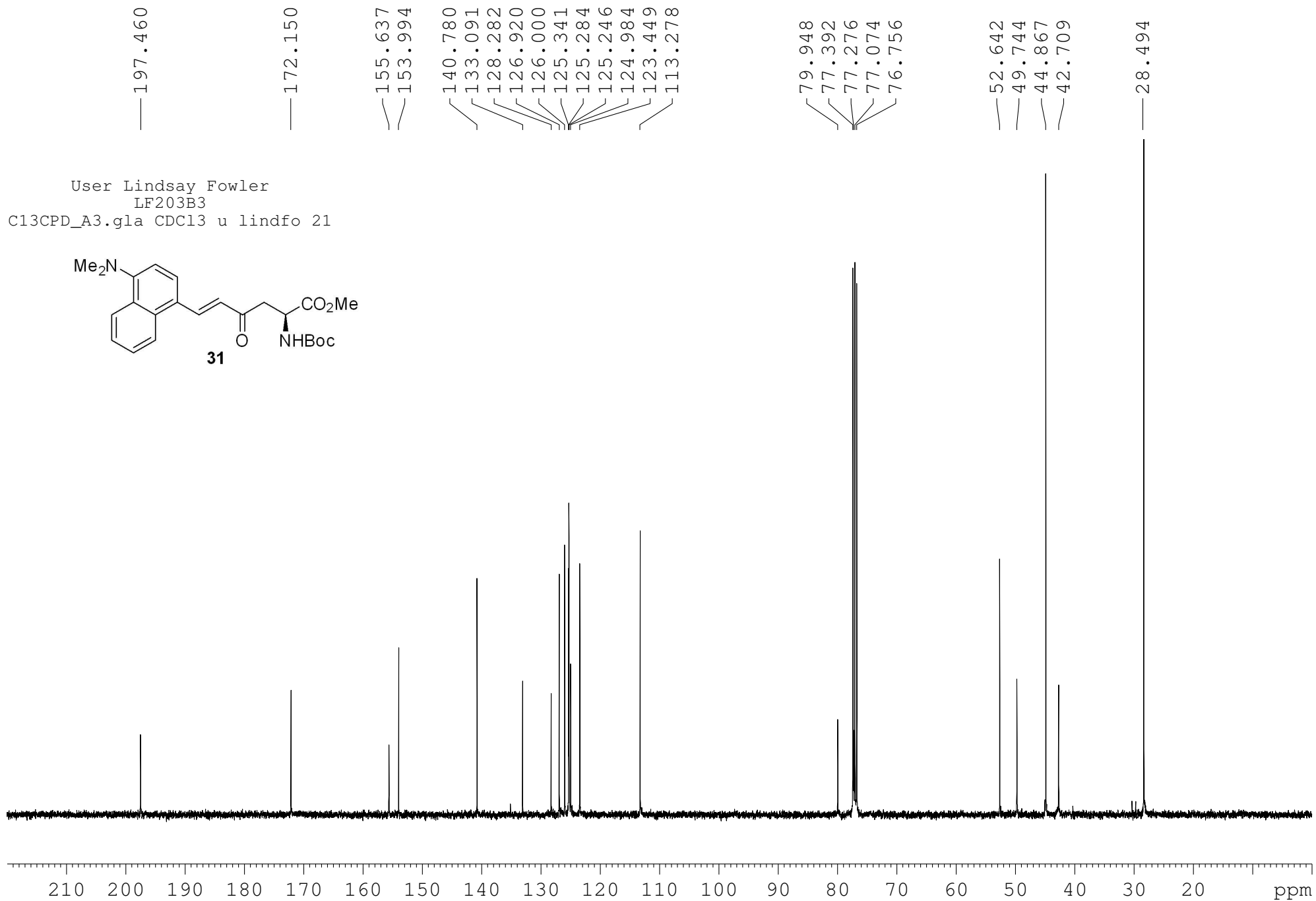
7.770
7.729
7.670
7.442
7.352
6.933
6.892

4.930

4.281

3.523
3.475
3.447
3.429
3.400
3.382
3.305

0.000

User - Lindsay Fowler
LF184B1
PROTON_C_A3.gla MeOD u lindfo 34

26

10   9   8   7   6   5   4   3   2   1   ppm
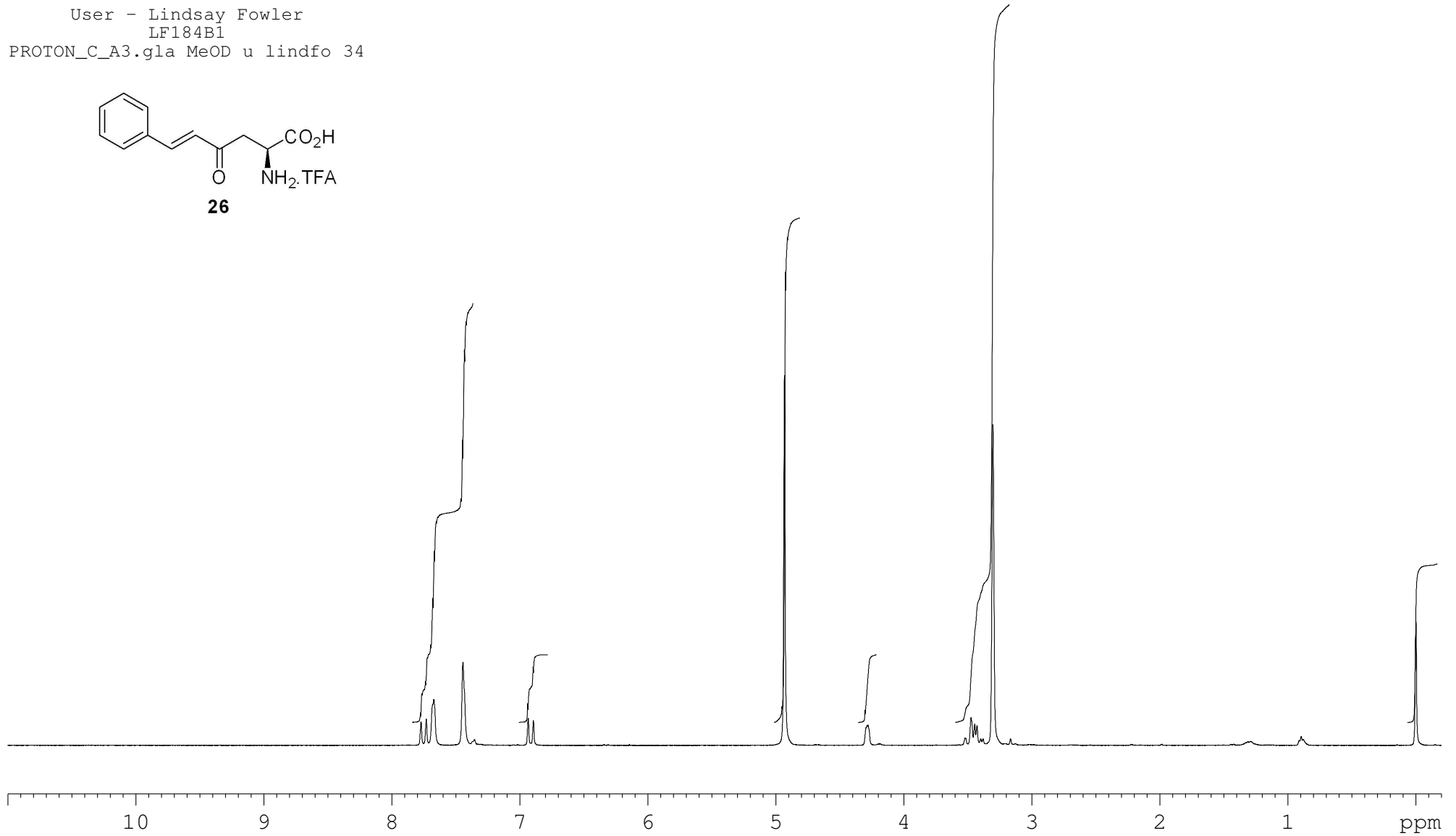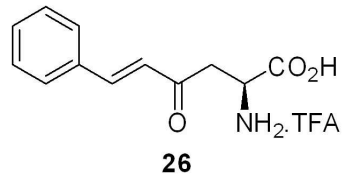
197.984

171.770

146.034

135.613
132.136
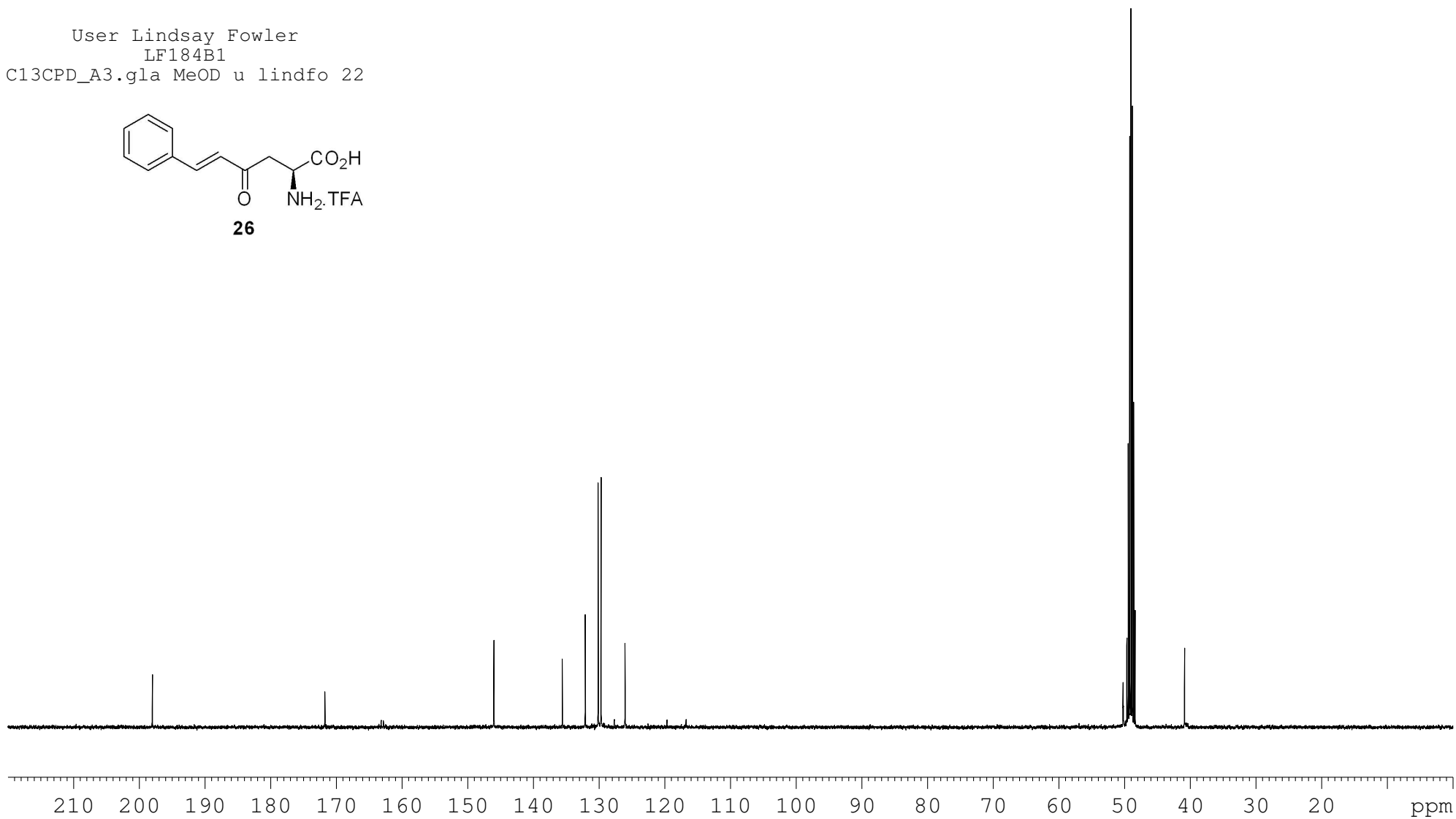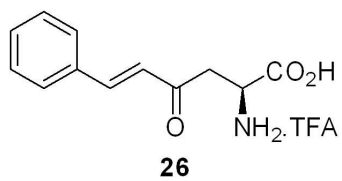130.161
129.720
126.058

50.242
49.672
49.458
49.245
49.032
48.819
48.606
48.393
40.877

User Lindsay Fowler
LF184B1
C13CPD_A3.gla MeOD u lindfo 22

26

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

7.694
7.654
7.589

6.929
6.888

4.883

3.956
3.941
3.484
3.445
3.305
3.271
3.248
3.224
3.201
3.175

0.000

User – Lindsay Fowler
LF212B1
PROTON_C_A3.gla MeOD u lindfo 41

27

10 9 8 7 6 5 4 3 2 1 ppm
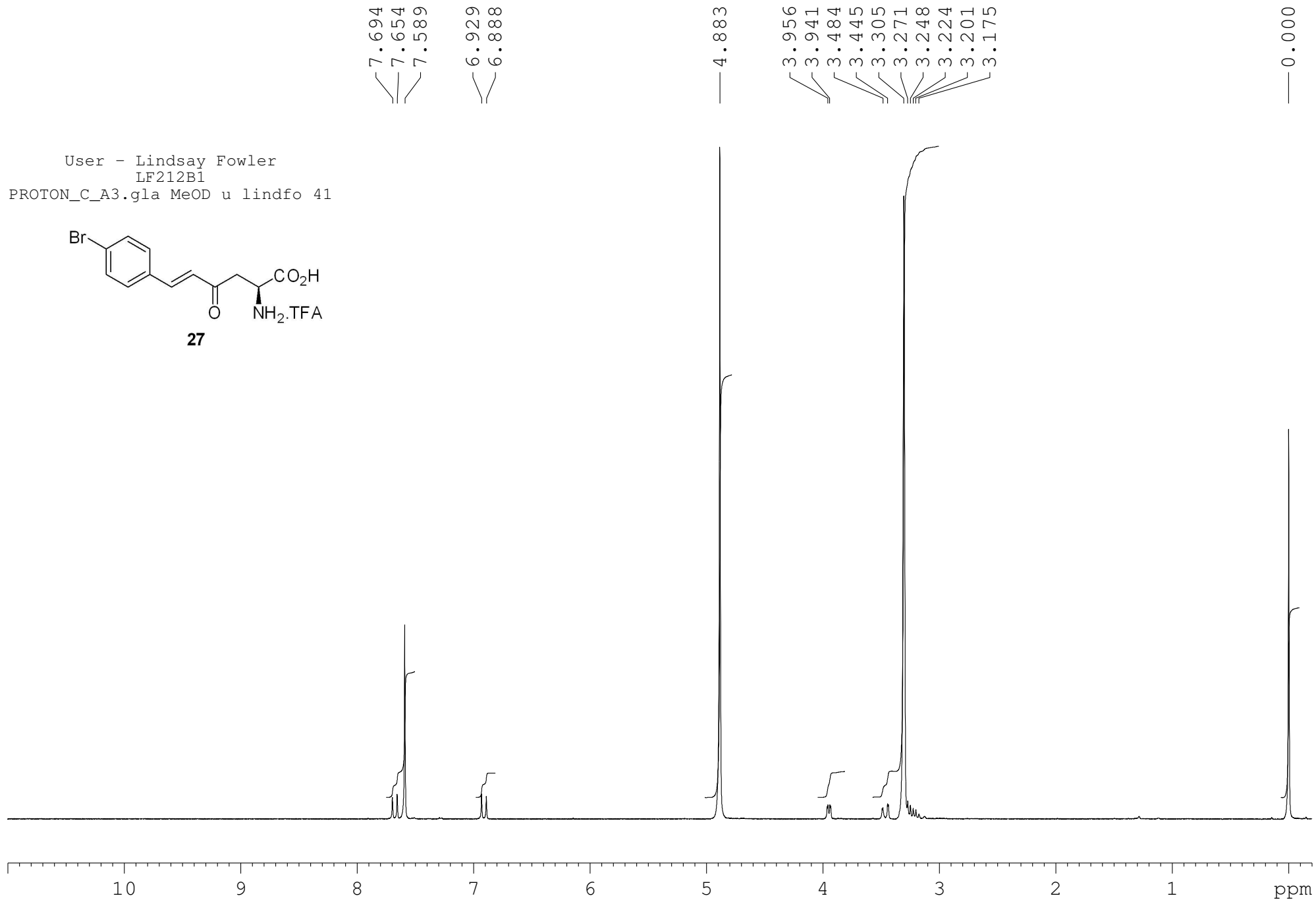
197.681

171.514

144.574
134.819
133.426
131.374
129.749
126.691
126.226
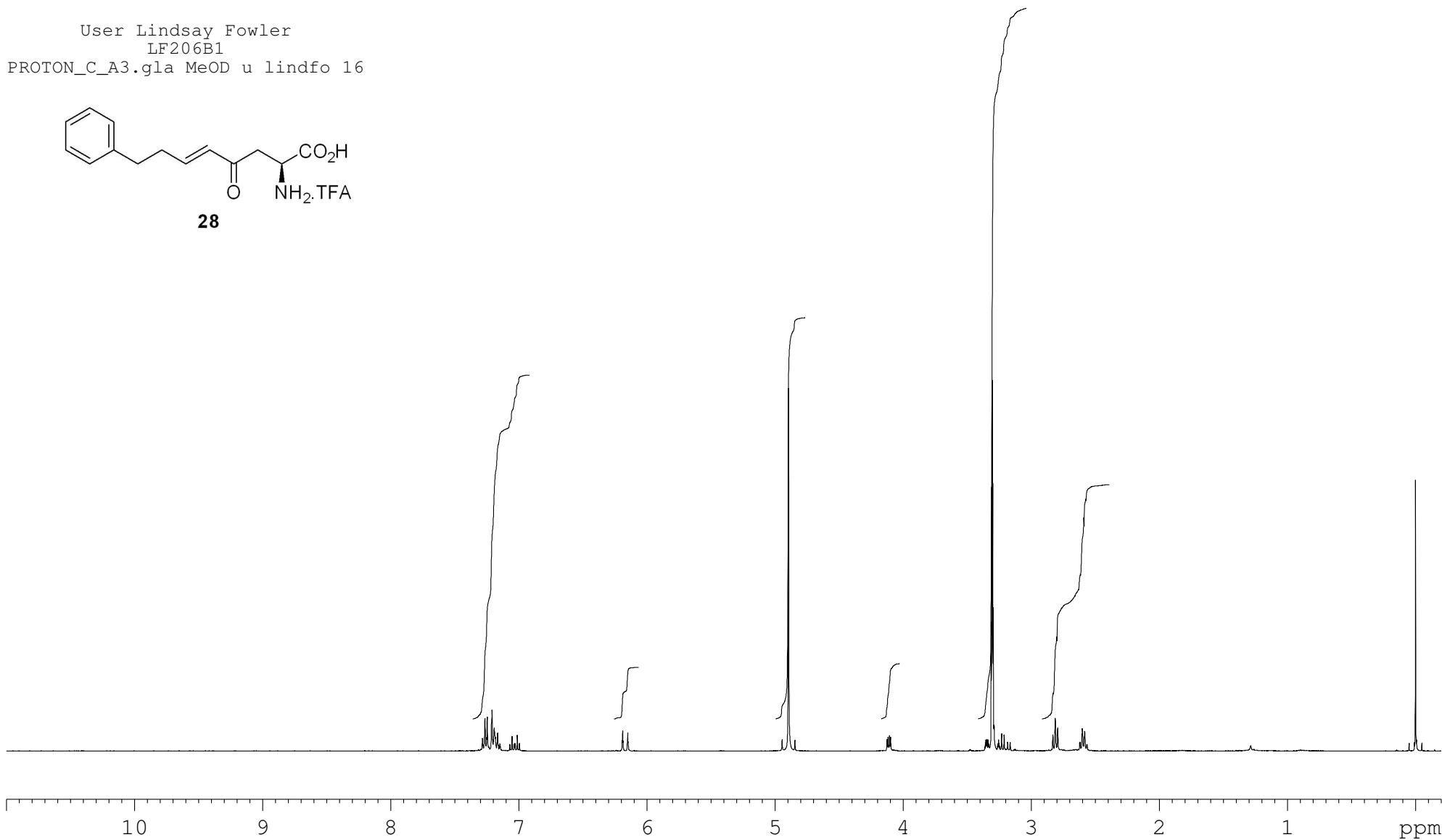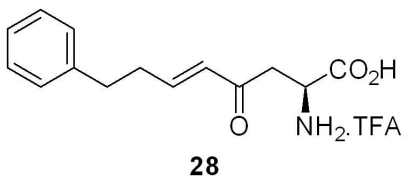
49.686
49.473
49.261
49.048
48.835
48.623
48.410
40.914

User – Lindsay Fowler
LF212B1
C13CPD_A3.gla MeOD u lindfo 14
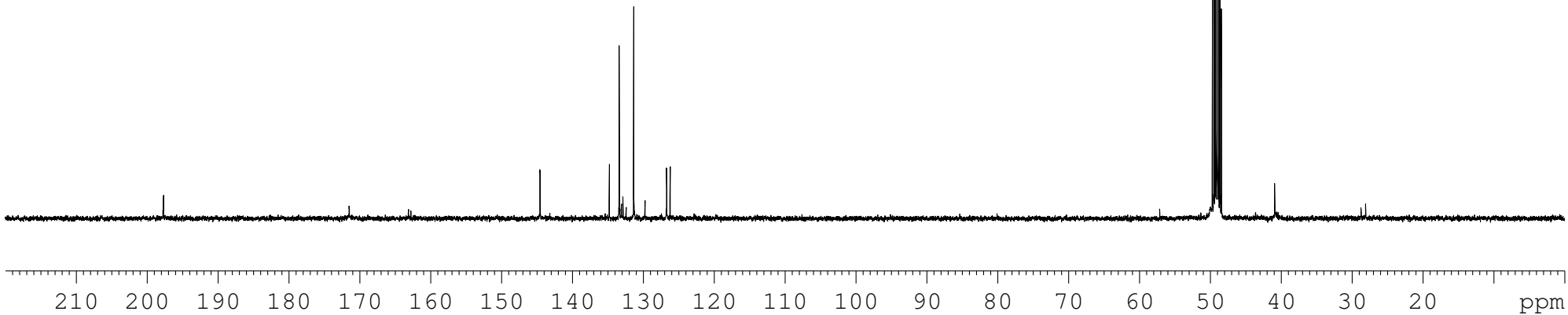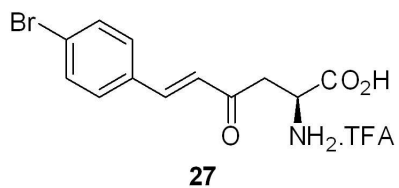
27

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20   ppm

User Lindsay Fowler
LF206B1
PROTON_C_A3.gla MeOD u lindfo 16

28

198.113

150.506

142.131

130.836
129.542
129.482
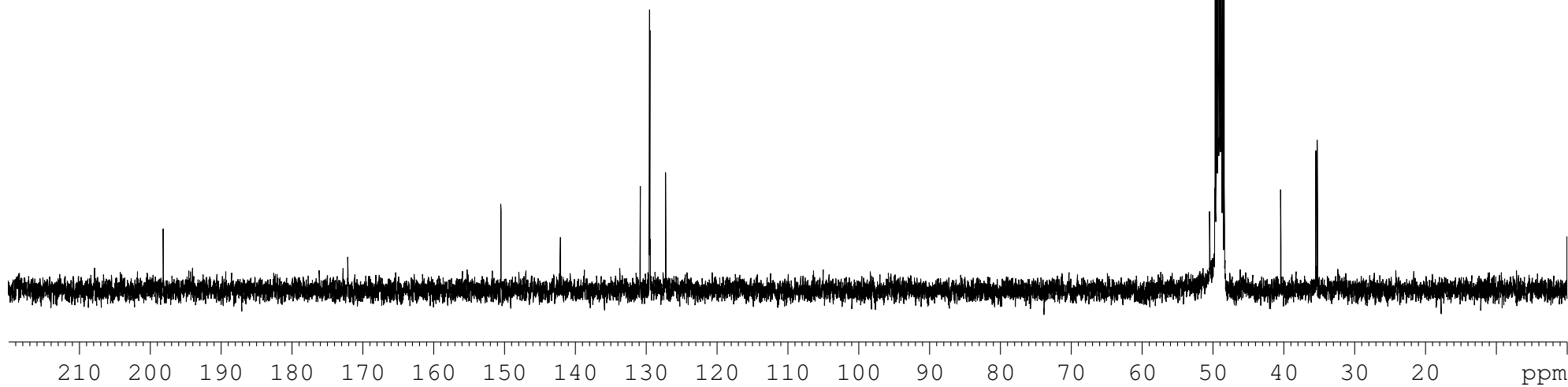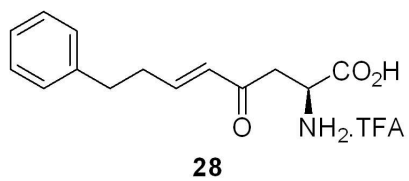127.246

50.463
49.671
49.459
49.246
49.033
48.821
48.608
48.395
40.419
35.464
35.250

User – Lindsay Fowler
LF206B1
C13CPD_A3.gla MeOD u lindfo 3

28

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

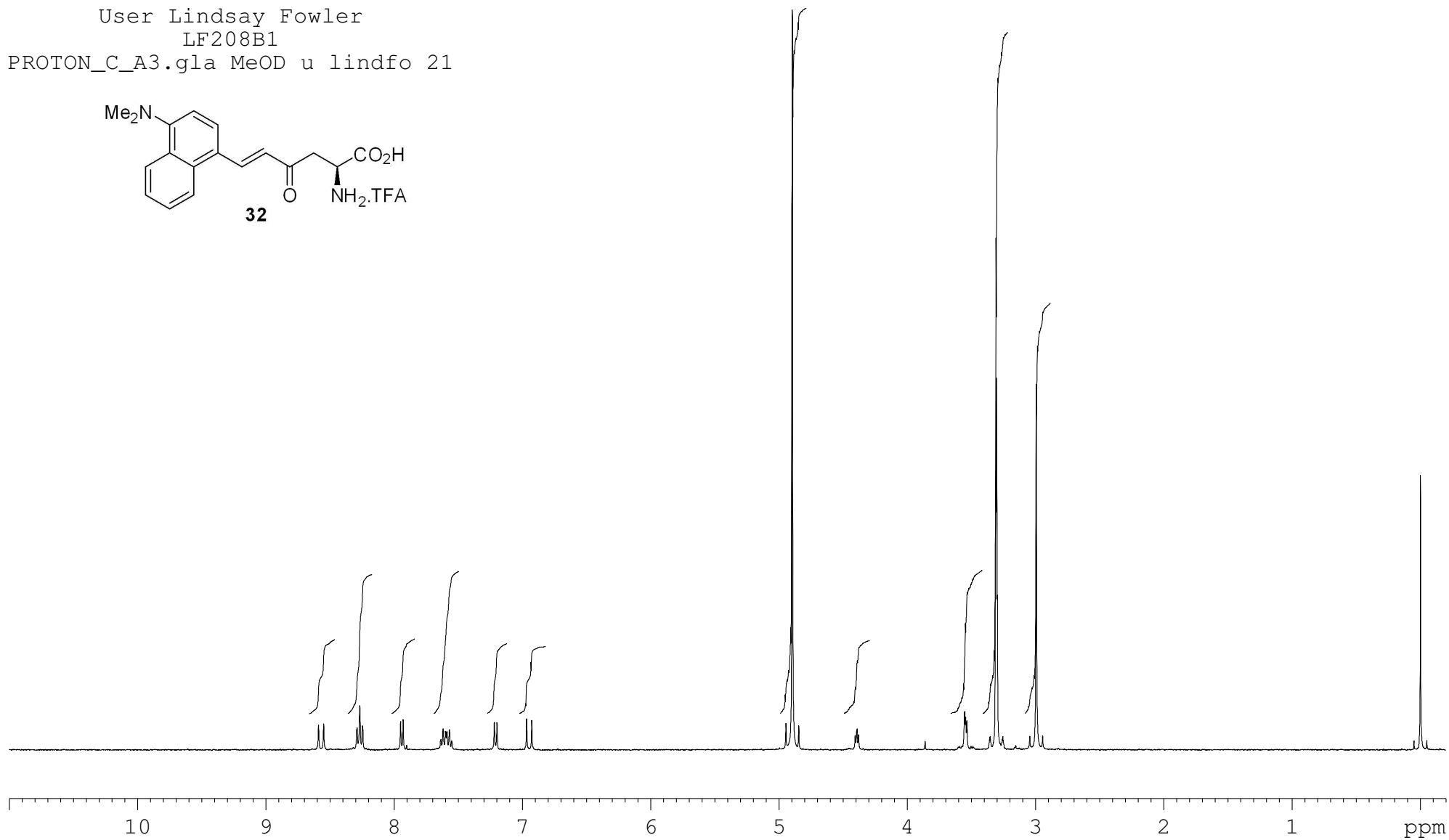User Lindsay Fowler
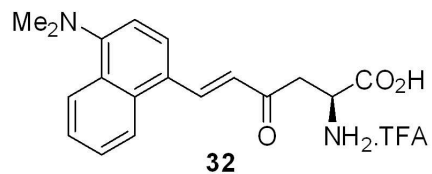LF208B1
PROTON_C_A3.gla MeOD u lindfo 21

8.588 8.548 8.288 8.267 8.246 7.948 7.928 7.636 7.619 7.602 7.591 7.571 7.553 7.220 7.200 6.968 6.929

4.947 4.897 4.407 4.392 4.381 3.552 3.545 3.536 3.314 3.310 3.306 3.302 3.298 2.995

0.000

197.447

171.325

152.818
142.339
134.477
128.986
128.438
128.055
127.209
127.065
126.595
125.432
124.742
115.483

49.998
49.681
49.468
49.255
49.042
48.829
48.617
48.404
45.665
40.987

User Lindsay Fowler
LF208B1
C13CPD_A3.gla MeOD u lindfo 14

32

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20   ppm