

Supporting information

Development and characterization of novel and selective inhibitors of cytochrome P450 CYP26A1, the human liver retinoic acid hydroxylase

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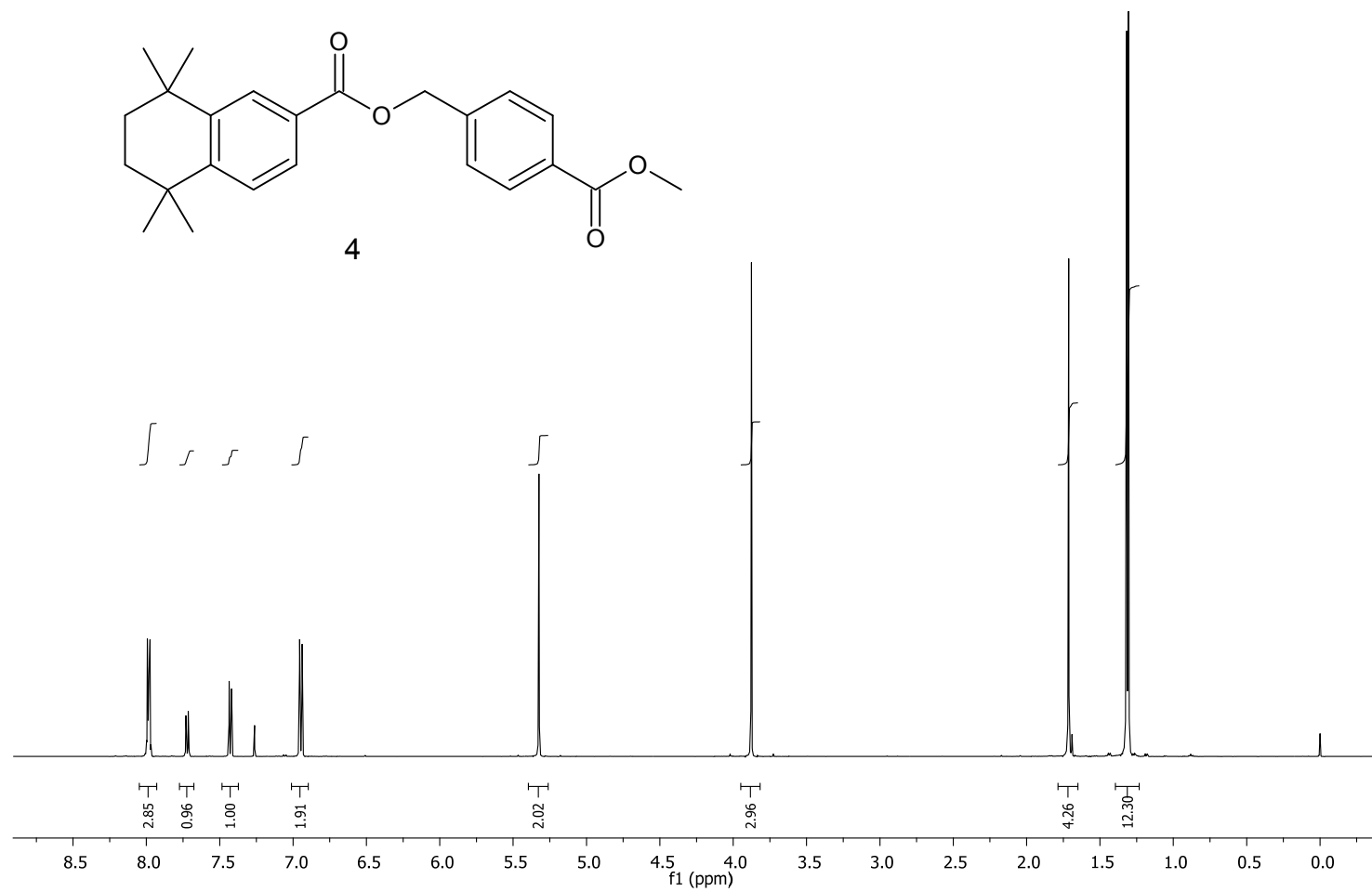
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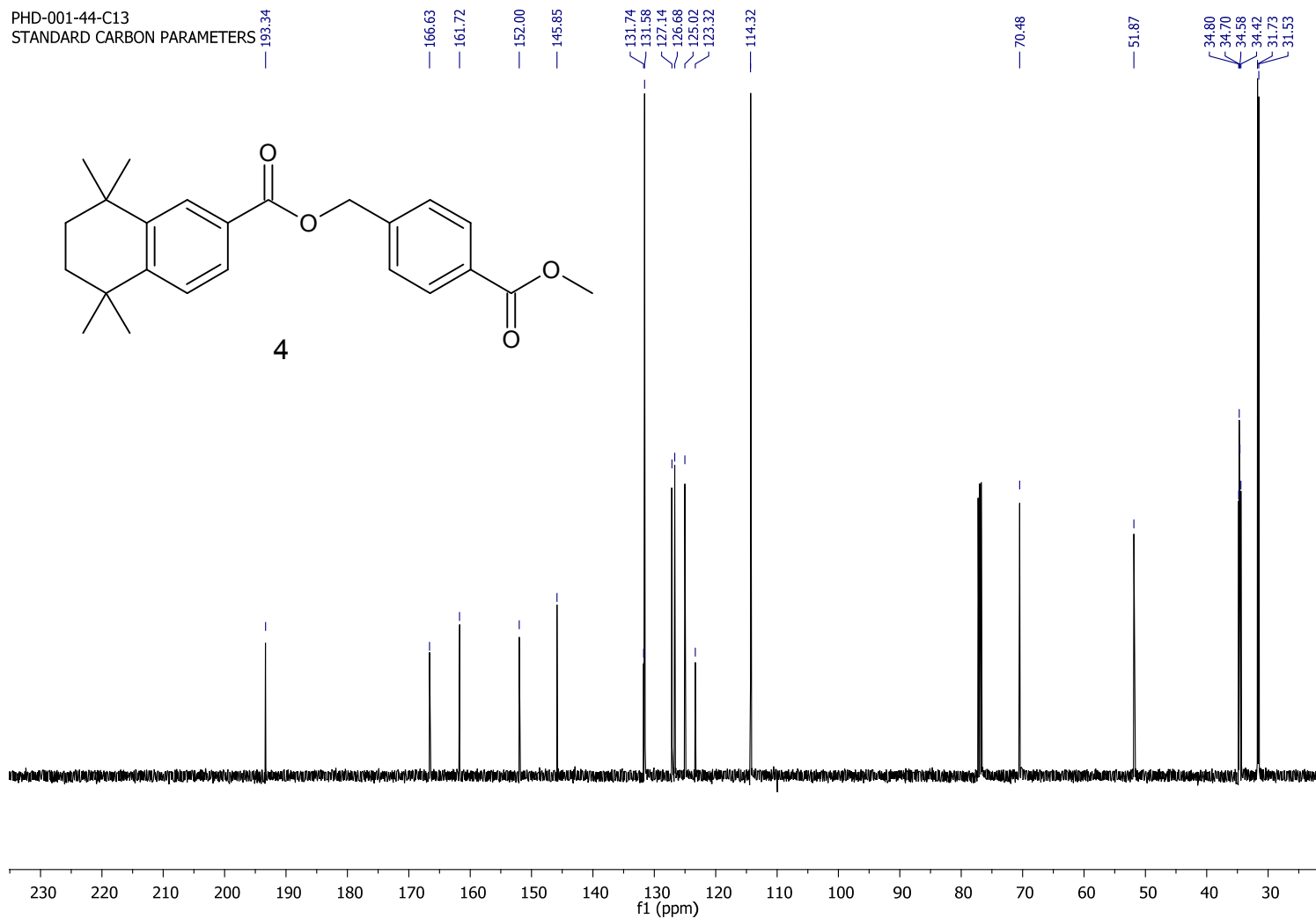
500 MHz ¹H-NMR of compound 4 in CDCl₃

PHD-001-44
STANDARD PROTON PARAMETERS



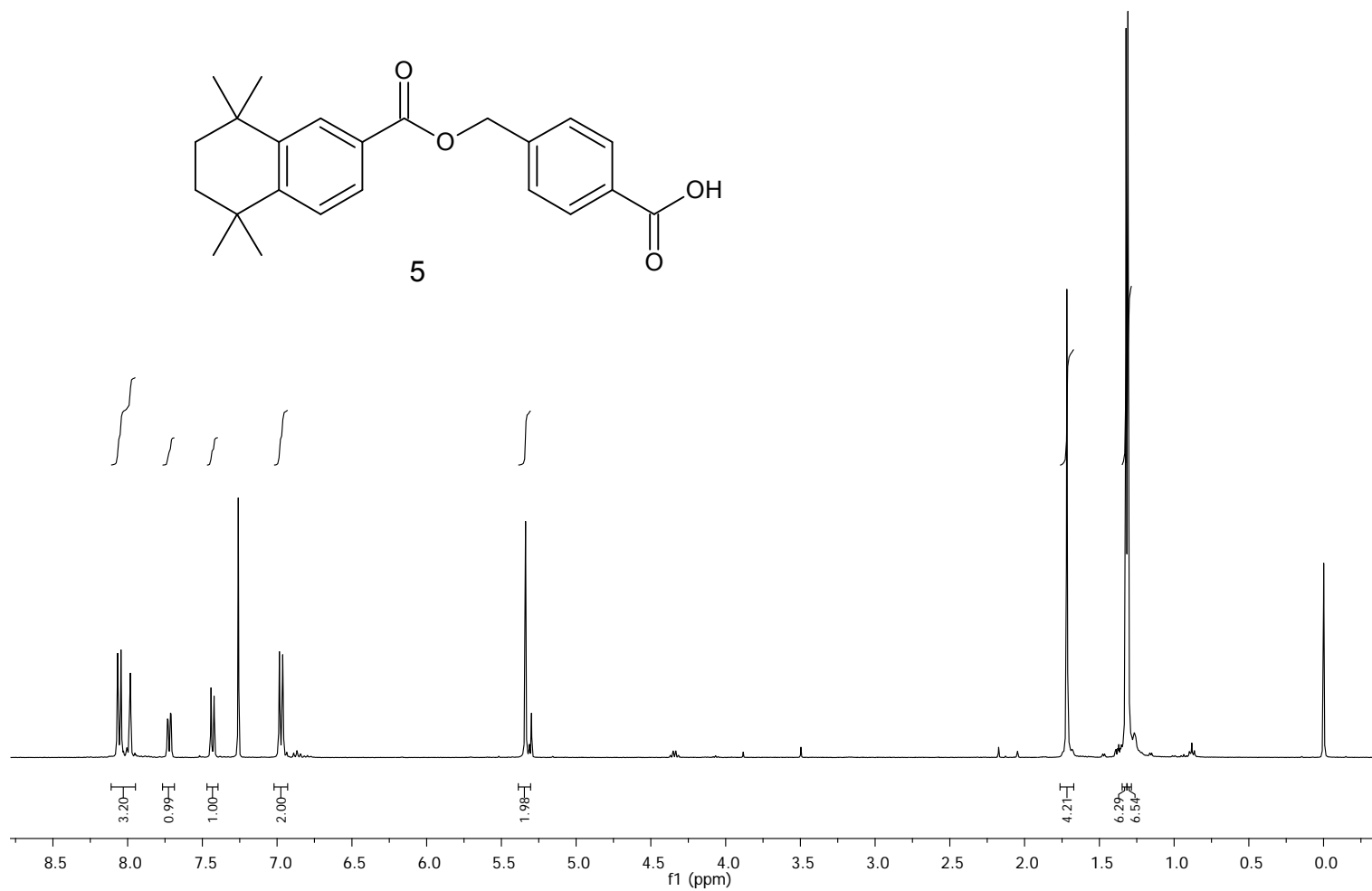
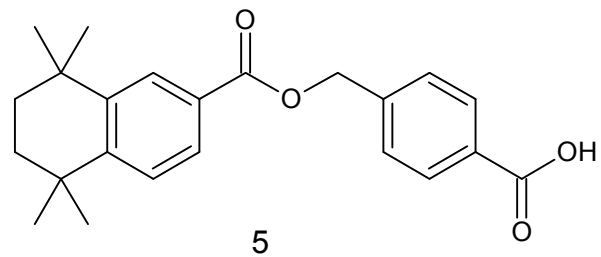
125 MHz ¹³C-NMR of compound 4 in CDCl₃

PHD-001-44-C13
STANDARD CARBON PARAMETERS



400 MHz $^1\text{H-NMR}$ of compound 5 in CDCl_3

PHD-02-43-proton



100 MHz ^{13}C -NMR of compound 5 in CDCl_3

PHD-02-43-C13

193.30

170.88

162.50

152.11

145.95

132.40

131.80

127.21

126.77

125.07

122.38

114.51

70.56

34.86

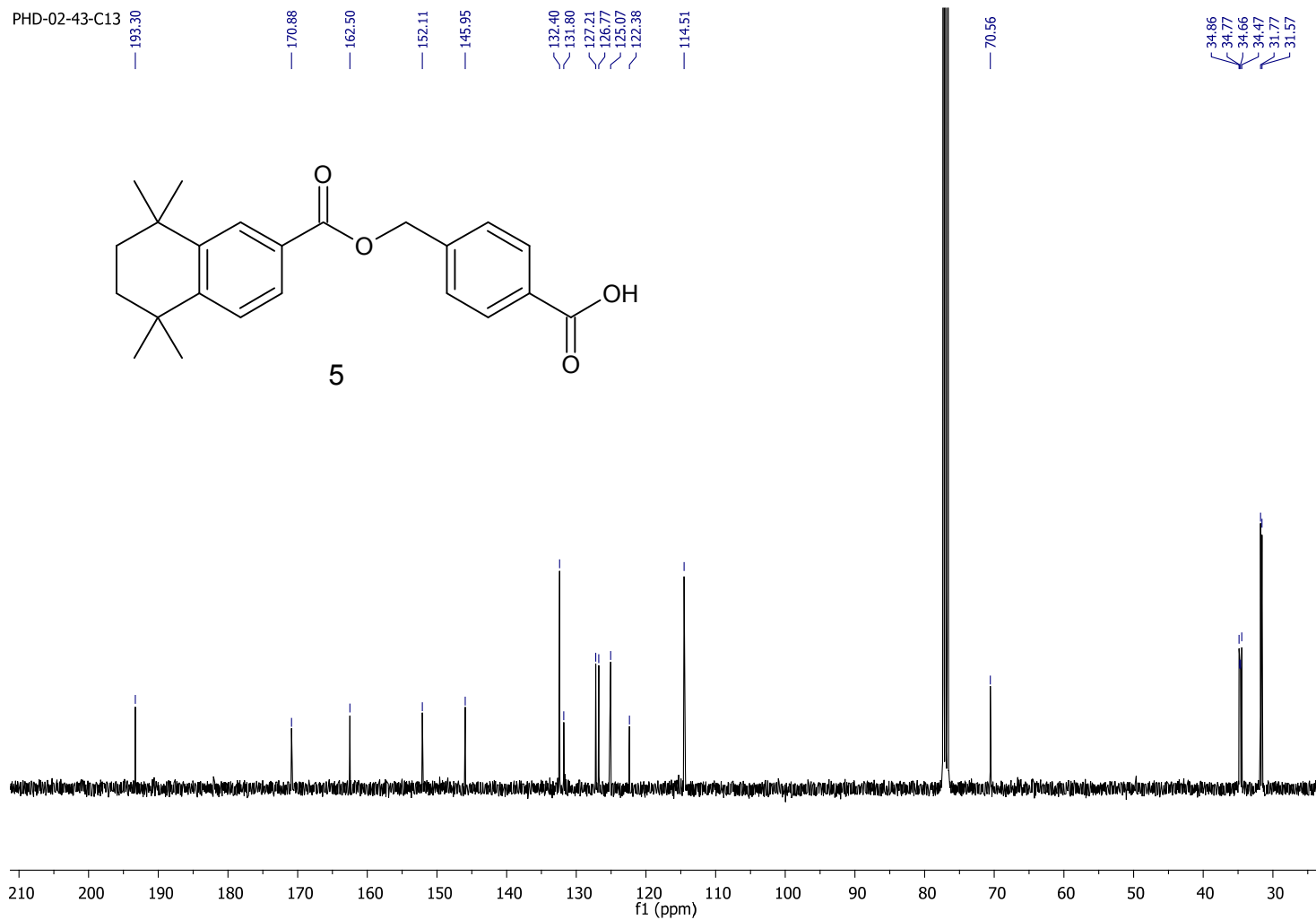
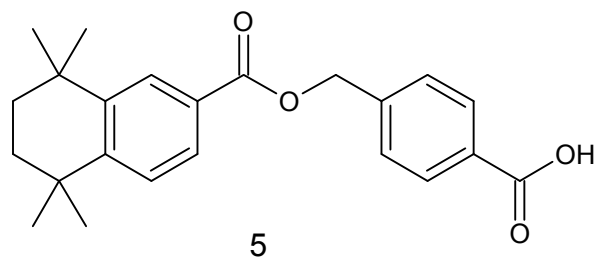
34.77

34.66

34.47

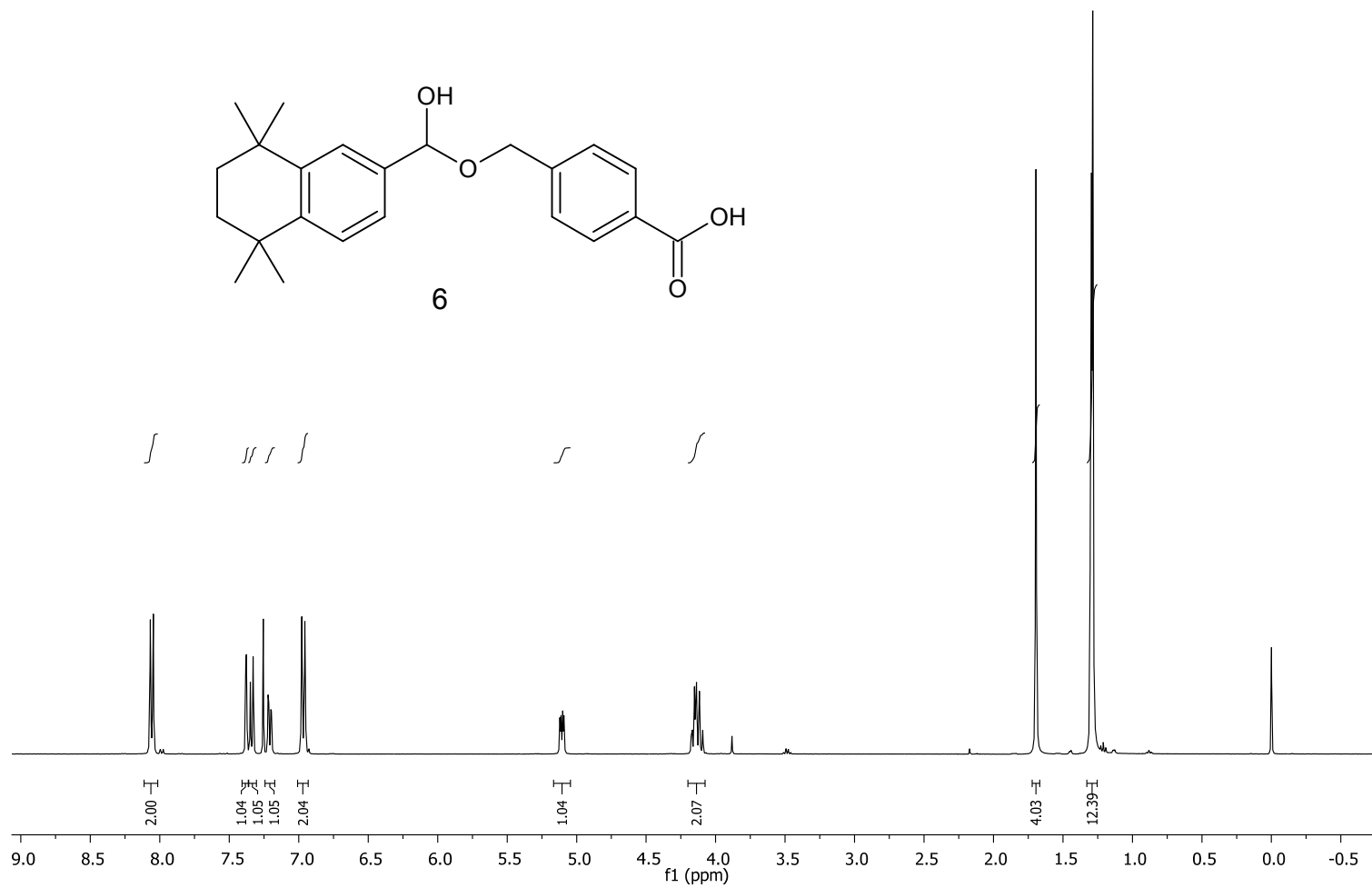
31.77

31.57



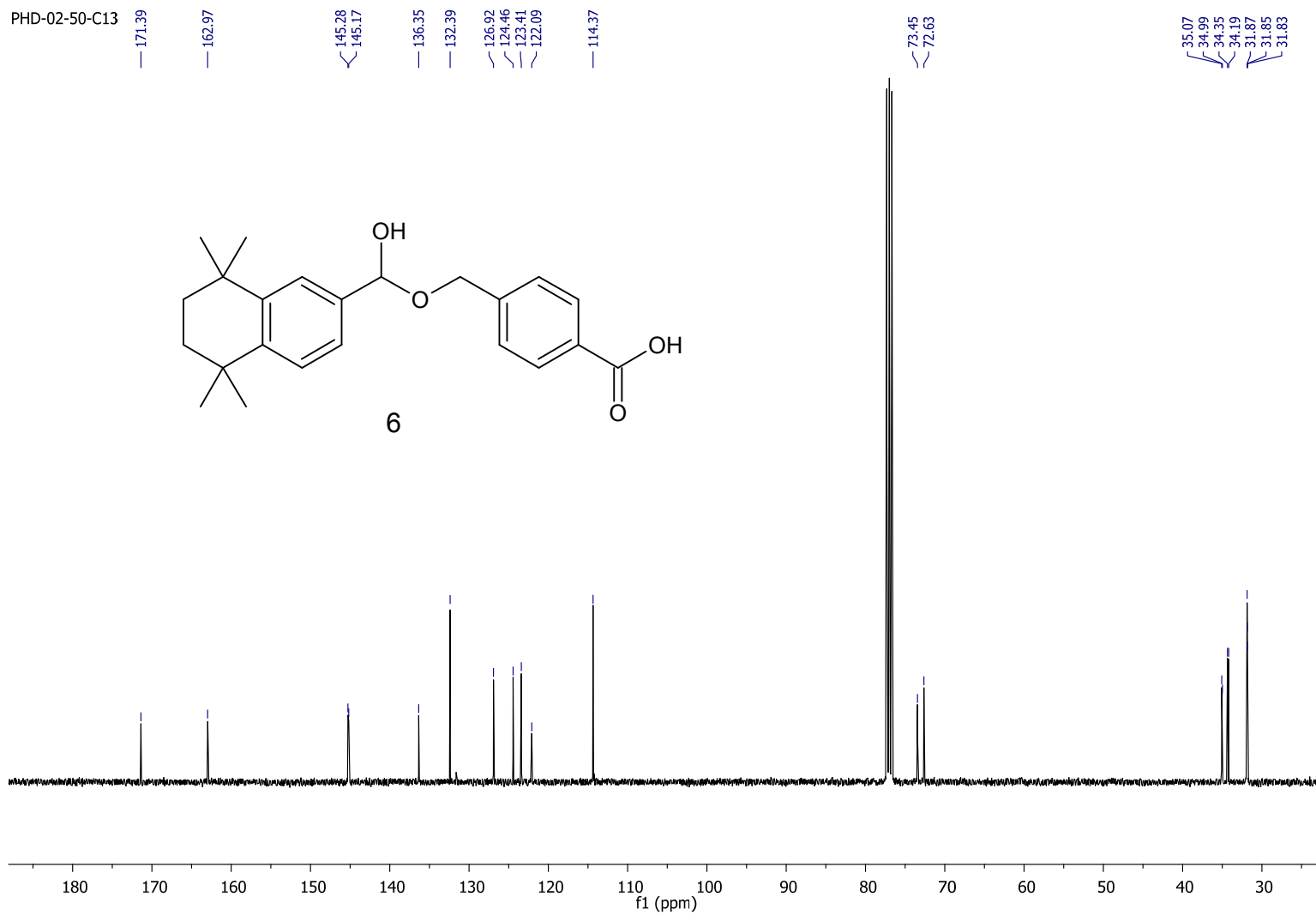
400 MHz ^1H -NMR of compound 6 in CDCl_3

PHD-02-50-proton



100 MHz ^{13}C -NMR of compound 6 in CDCl_3

PHD-02-50-C13



500 MHz $^1\text{H-NMR}$ of compound 7 in CDCl_3

CMK-02-3B-proton
STANDARD PROTON PARAMETERS

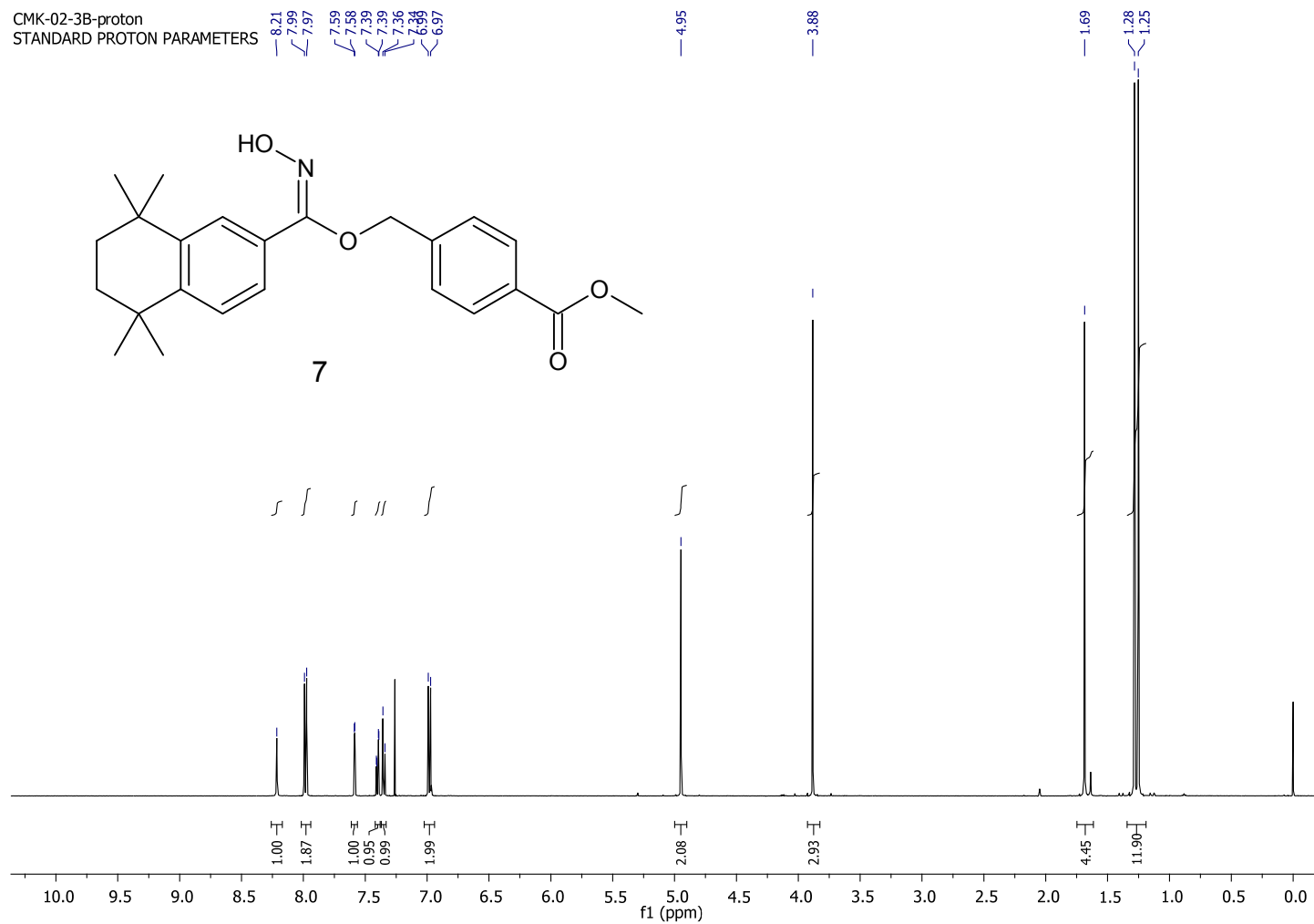
8.21
7.99
7.97
7.59
7.58
7.39
7.39
7.36
7.34
6.97

4.95

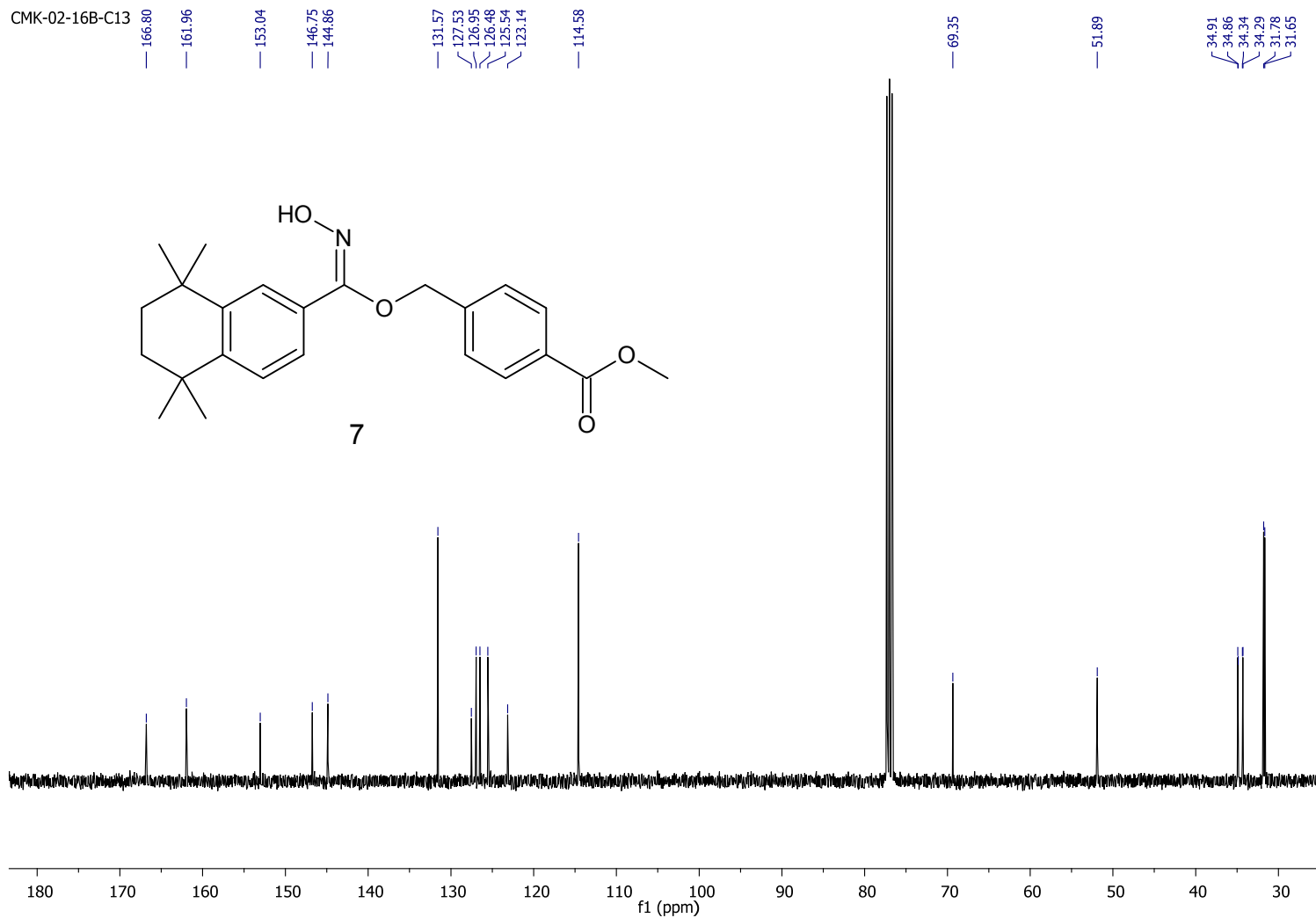
3.88

1.69

1.28
1.25

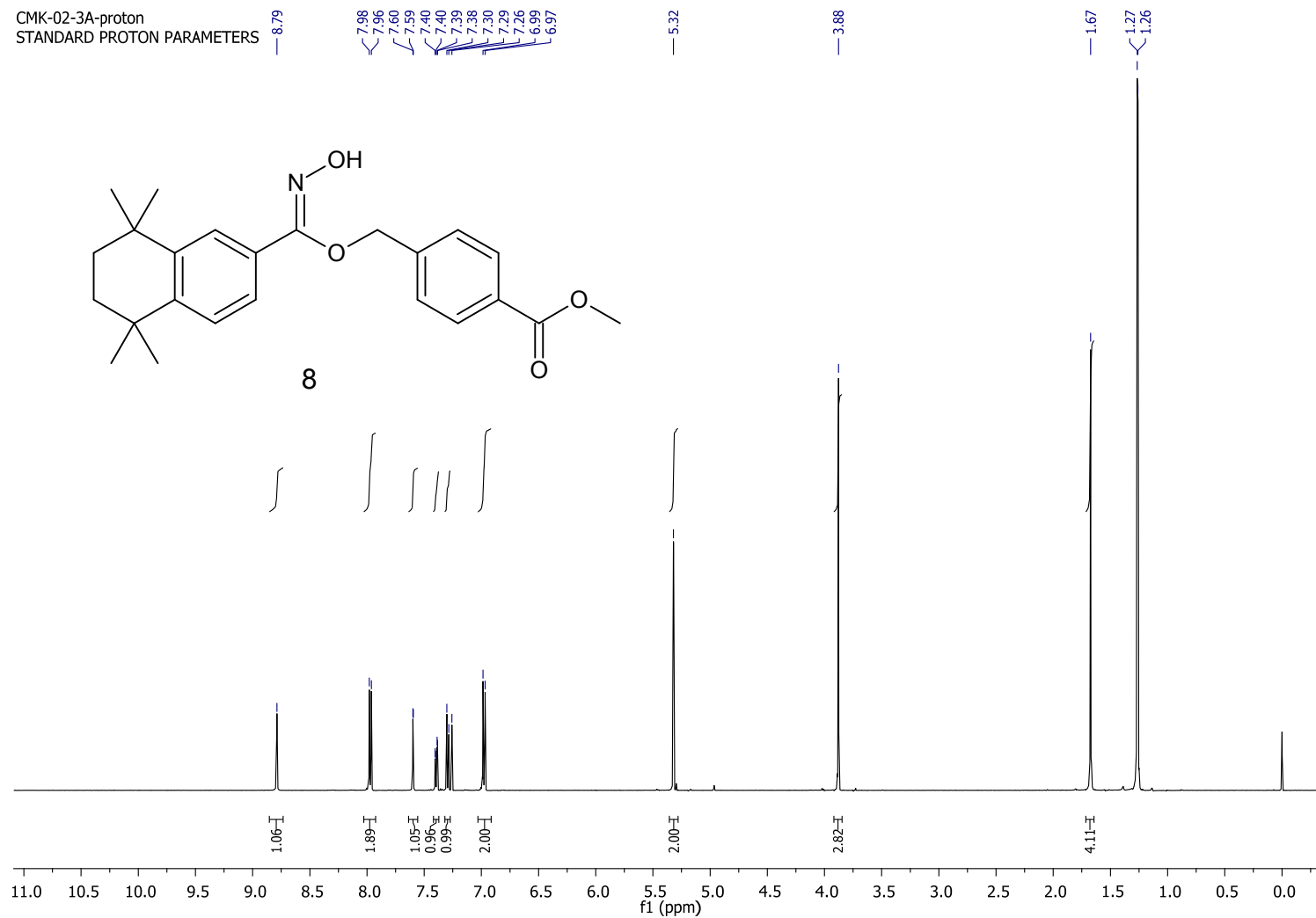


100 MHz ^{13}C -NMR of compound 7 in CDCl_3



500 MHz $^1\text{H-NMR}$ of Compound 8 in CDCl_3

CMK-02-3A-proton
STANDARD PROTON PARAMETERS



125 MHz ¹³C-NMR of compound 8 in CDCl₃

CMK-02-3A-C13
STANDARD CARBON PARAMETERS

166.81

161.81

155.20

146.73

144.91

131.59

130.12

126.69

125.35

124.01

123.09

114.25

59.86

51.91

34.93

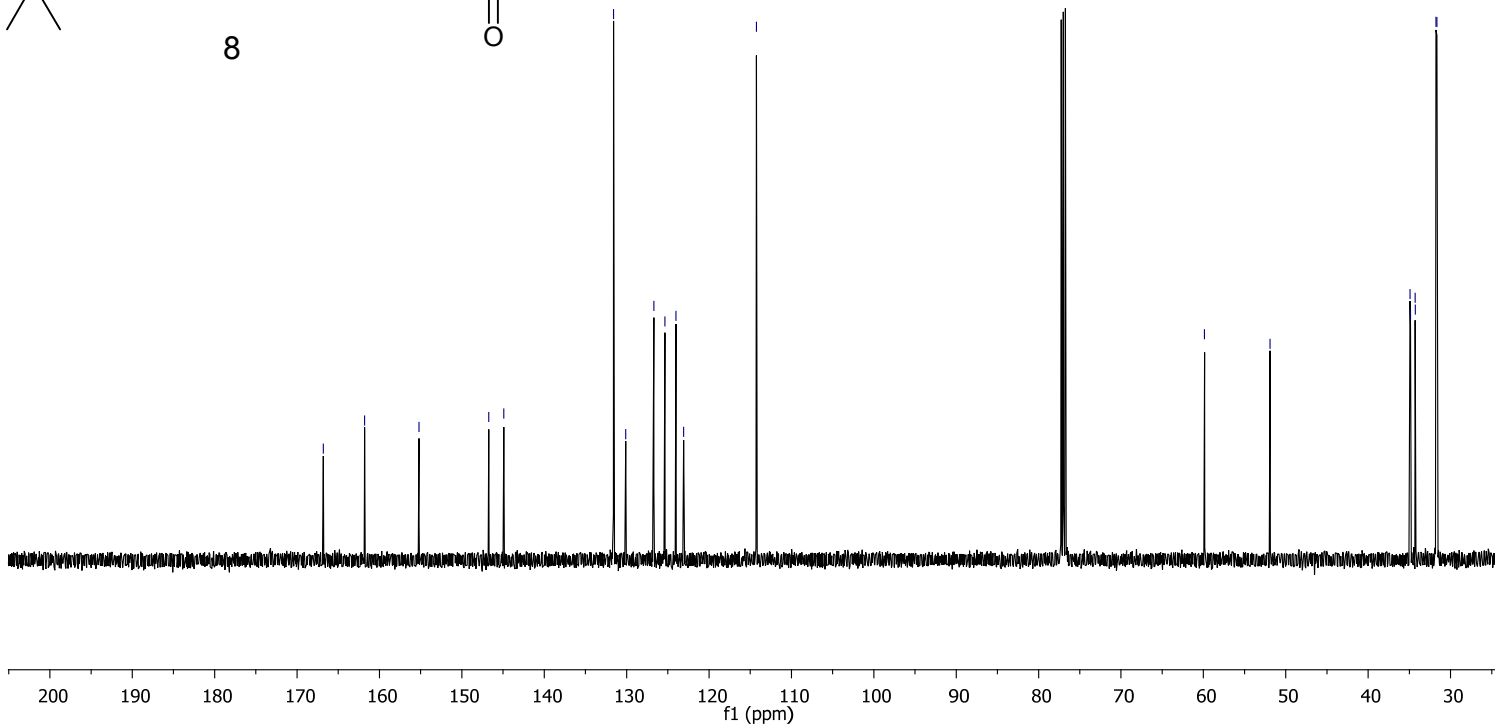
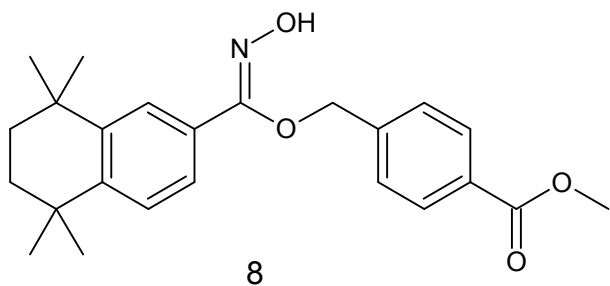
34.85

34.30

34.29

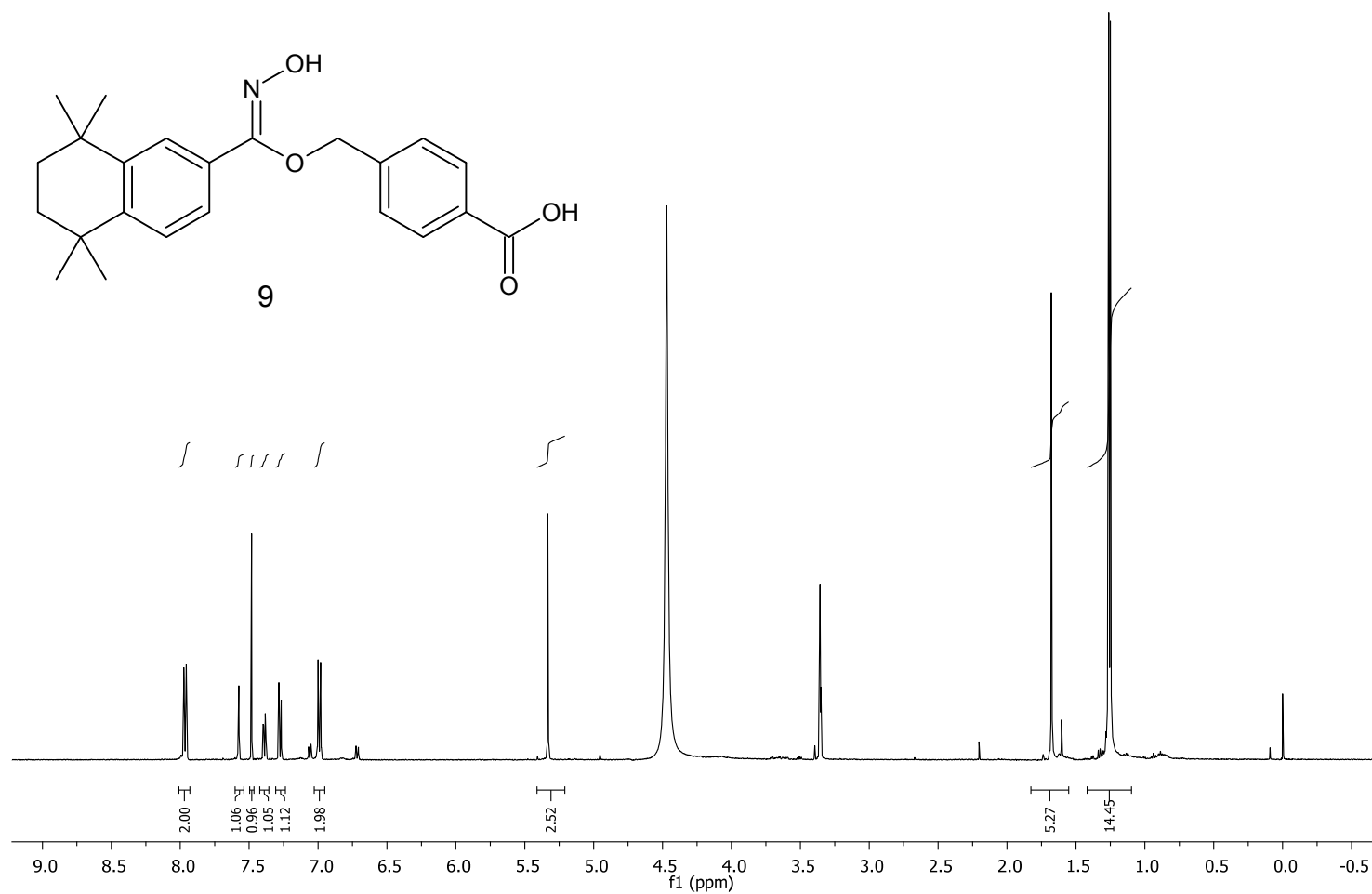
31.78

31.65



500 MHz $^1\text{H-NMR}$ of compound 9 in $\text{CDCl}_3/\text{CD}_3\text{OD}$

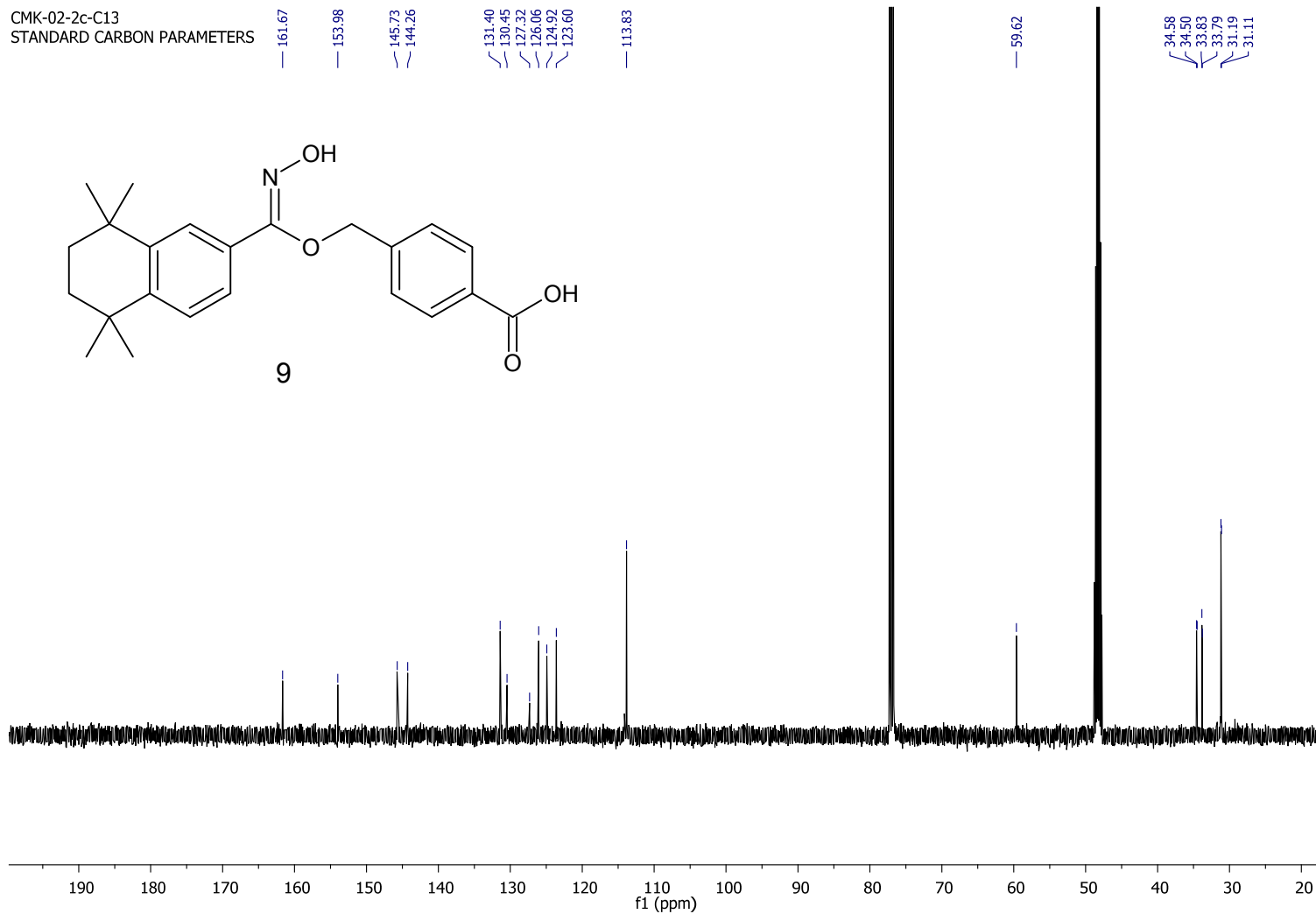
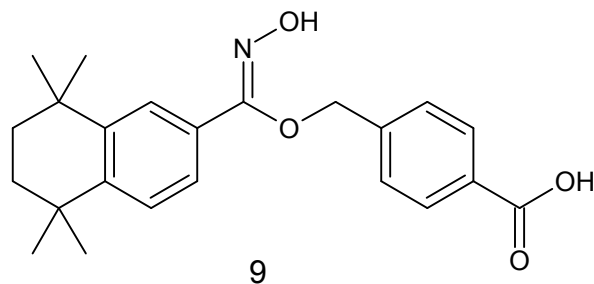
CMK-02-2c-proton
STANDARD PROTON PARAMETERS



125 MHz ¹³C-NMR of compound 9 in CDCl₃/CD₃OD

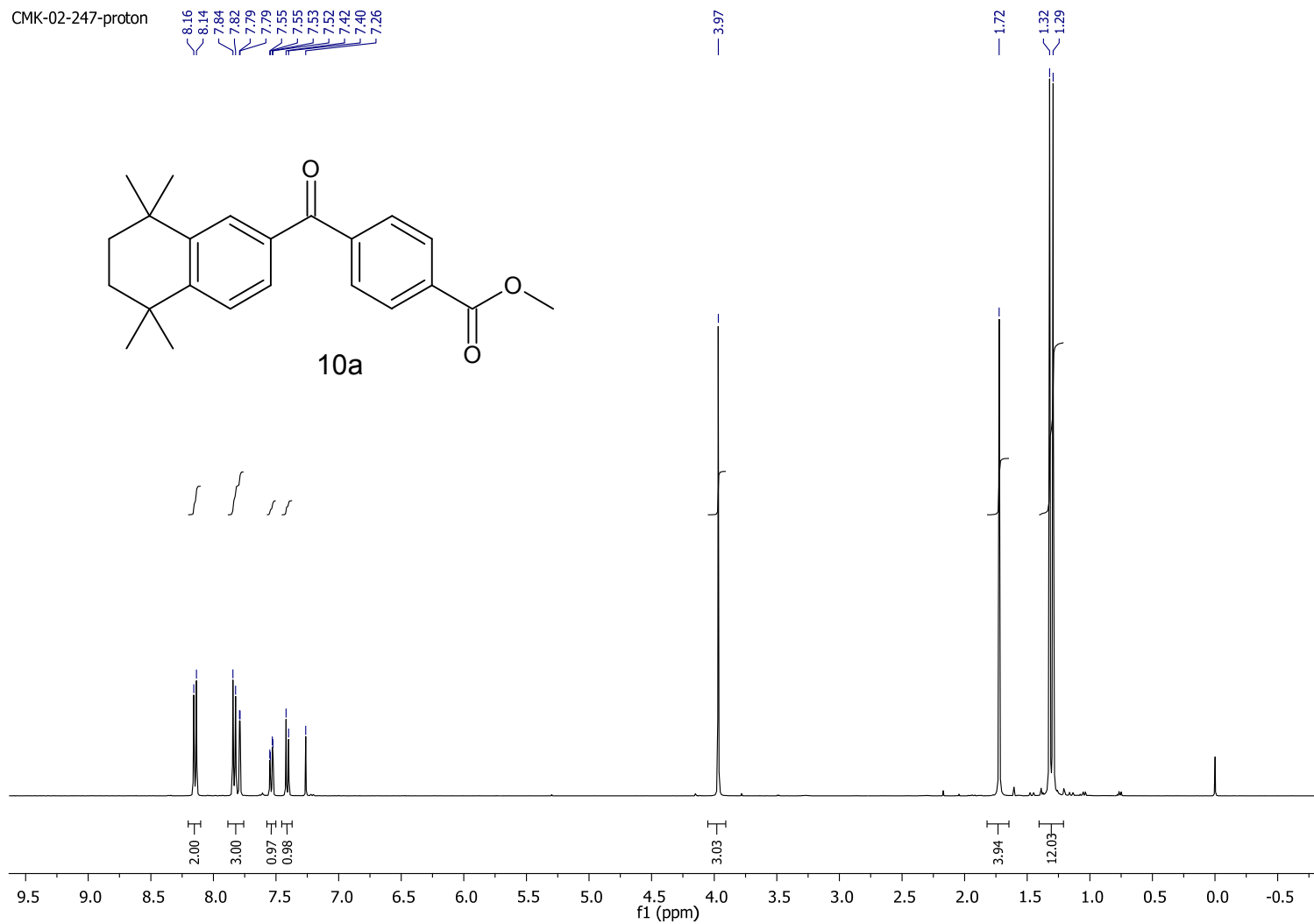
CMK-02-2c-C13
STANDARD CARBON PARAMETERS

— 161.67 — 153.98 — 145.73 — 144.26 — 131.40 — 130.45 — 127.32 — 126.06 — 124.92 — 123.60 — 113.83



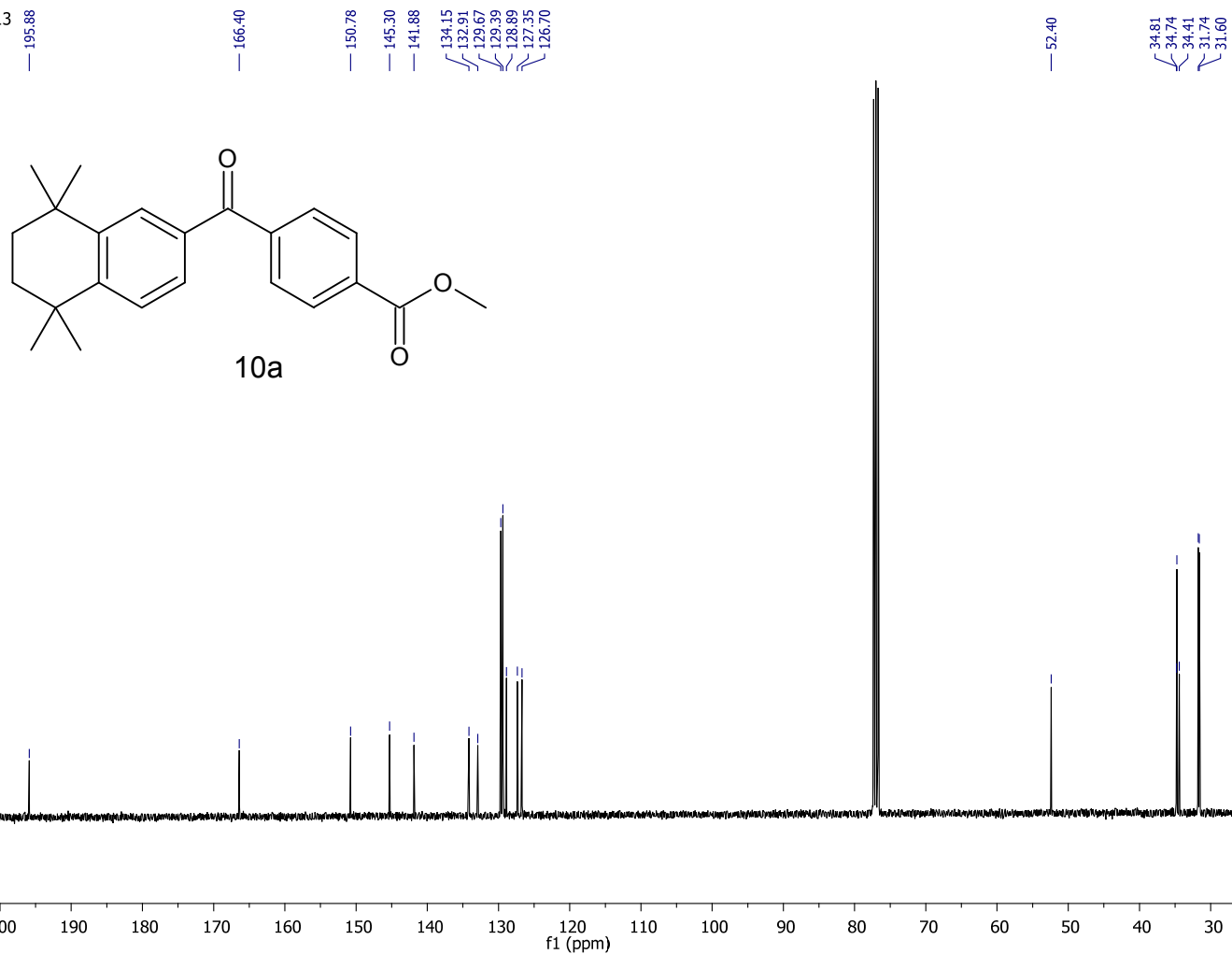
400 MHz ¹H-NMR of compound 10a in CDCl₃

CMK-02-247-proton



100 MHz ¹³C-NMR of compound 10a in CDCl₃

CMK-02-247C13

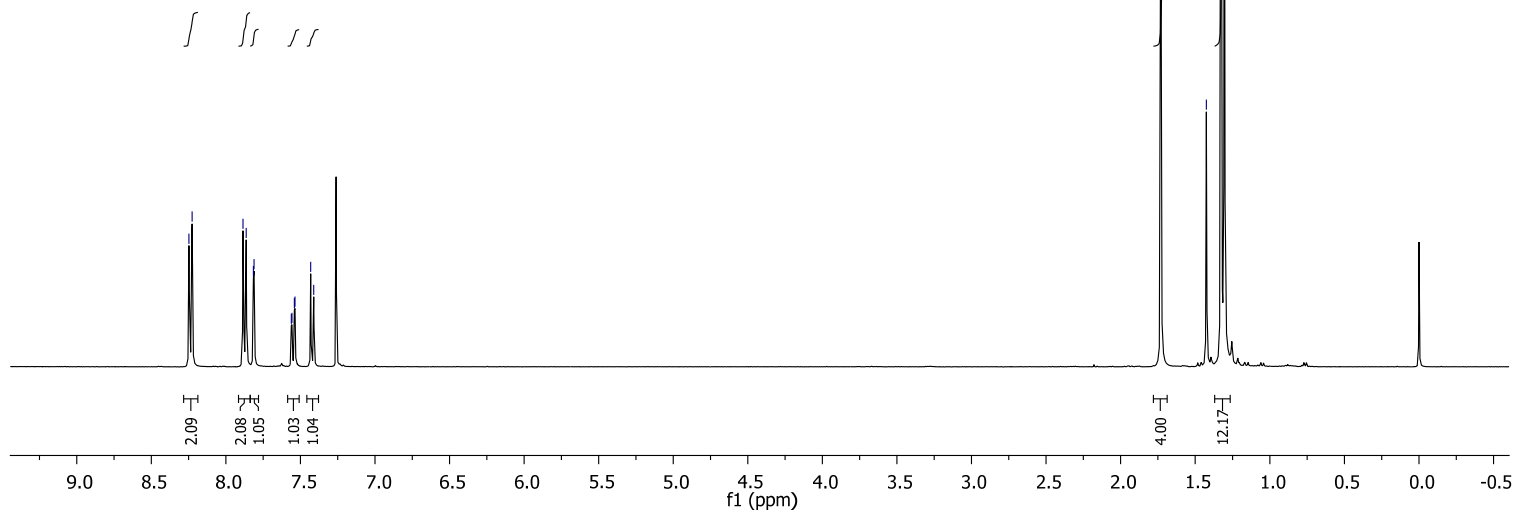
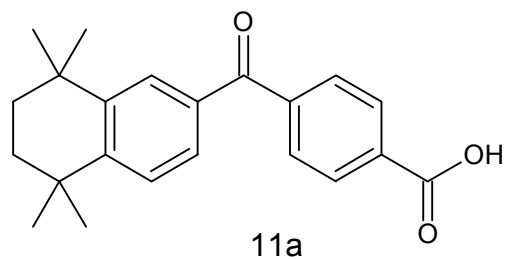


400 MHz $^1\text{H-NMR}$ of compound 11a in CDCl_3

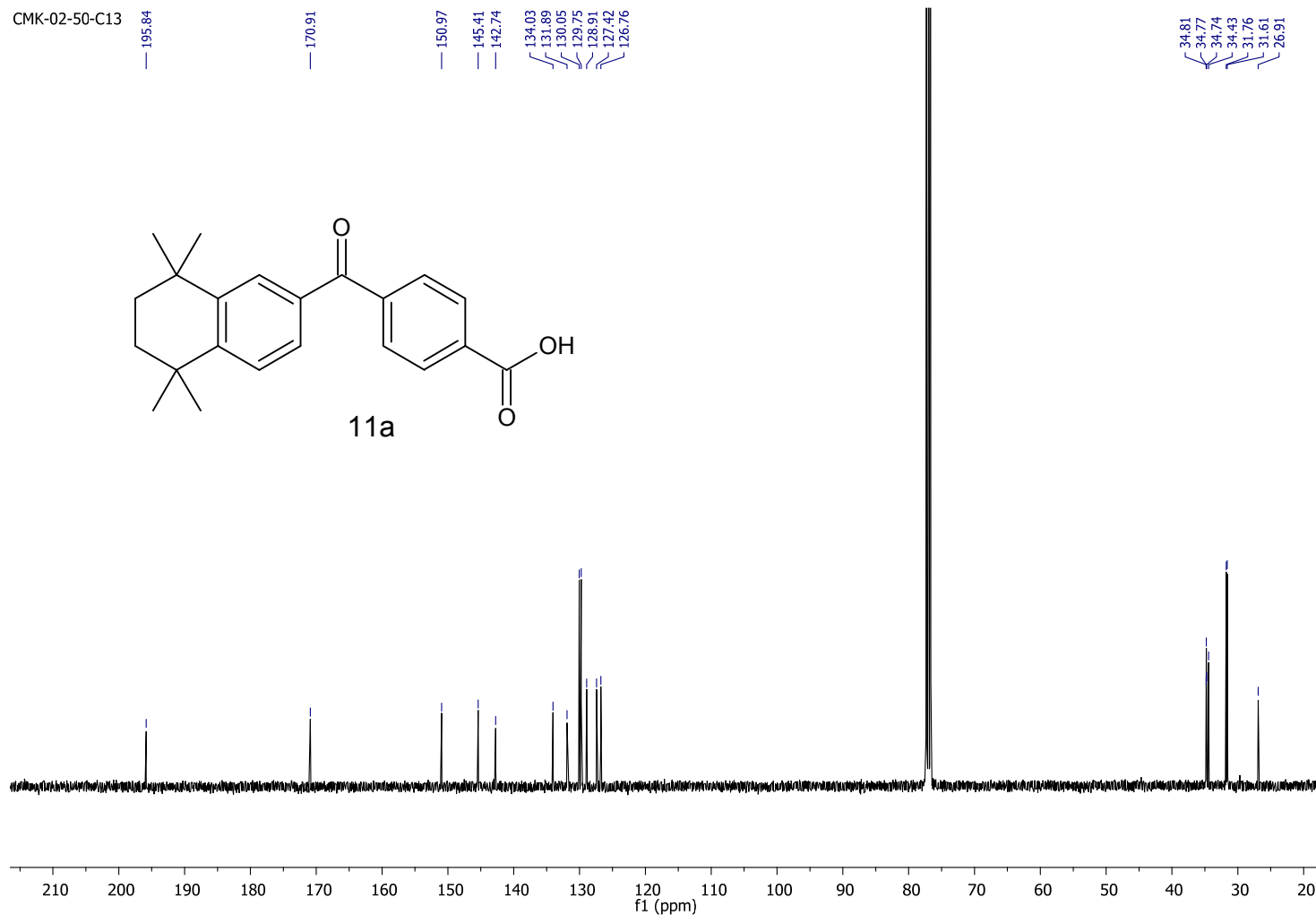
CMK-02-50-proton

8.25
8.23
7.89
7.86
7.81
7.56
7.54
7.54
7.43
7.41

1.73
1.43
1.33
1.30

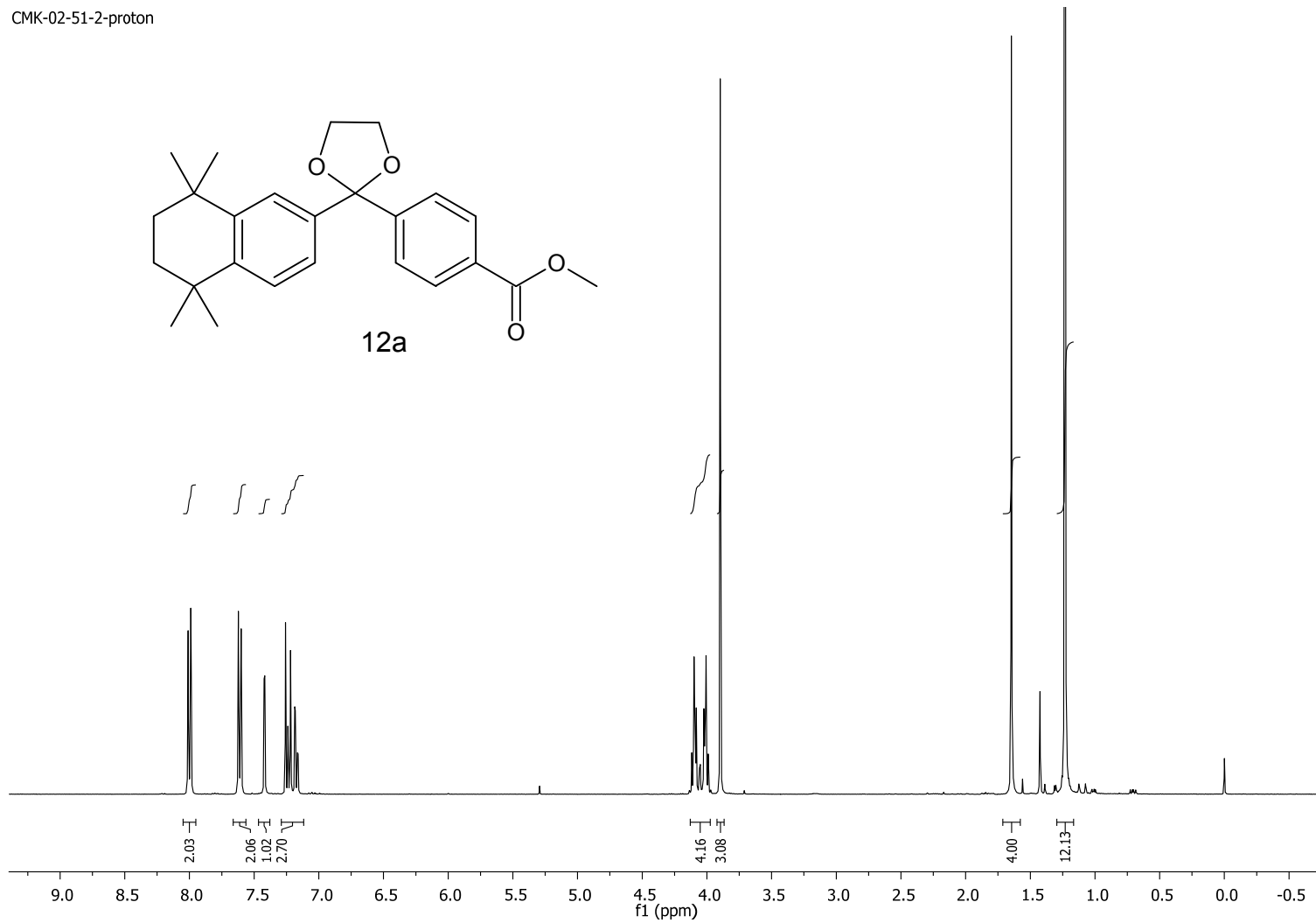
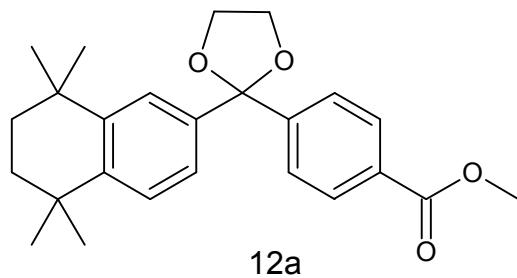


100 MHz ^{13}C -NMR of compound 11a in CDCl_3

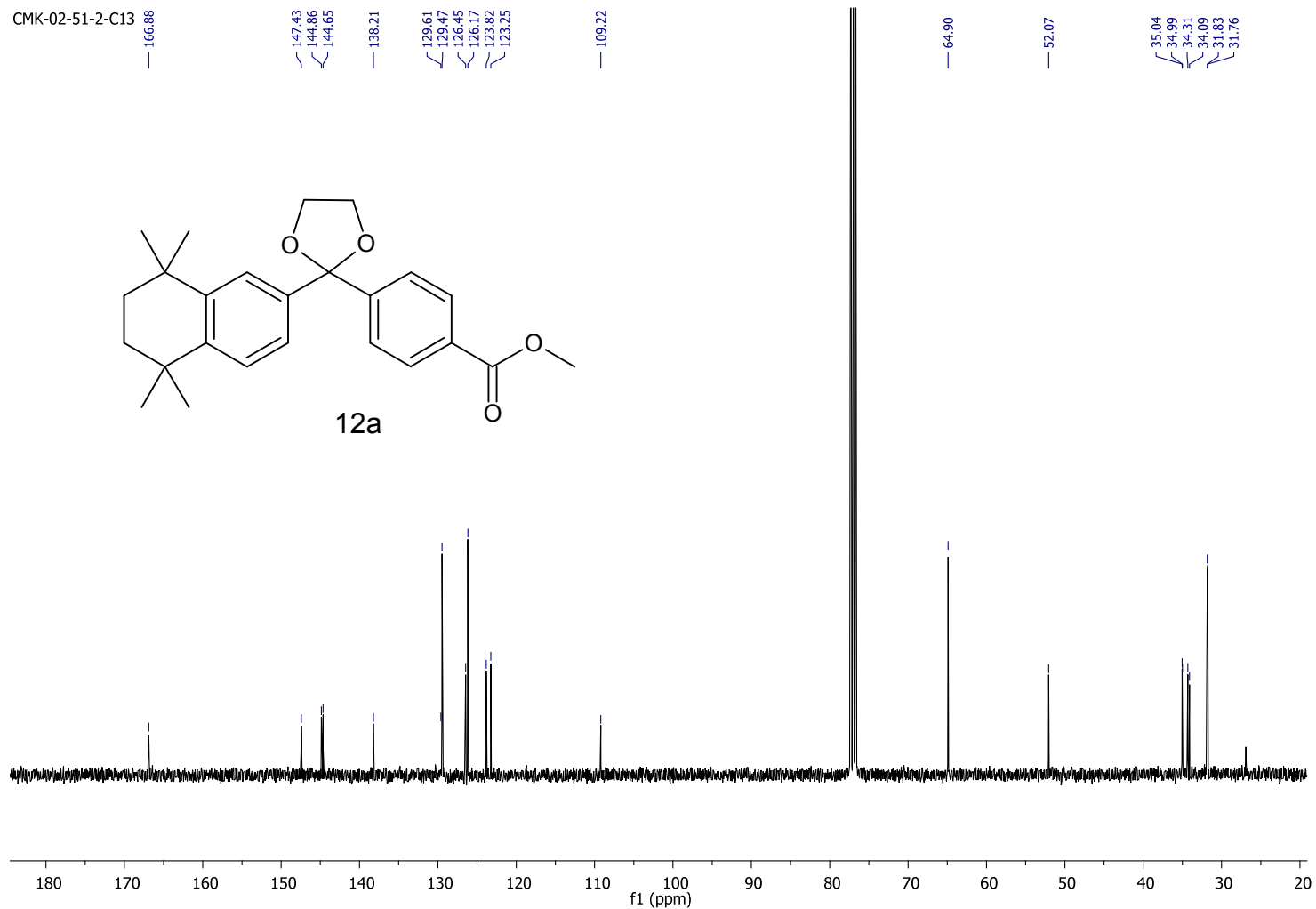


400 MHz $^1\text{H-NMR}$ of compound 12a in CDCl_3

CMK-02-51-2-proton

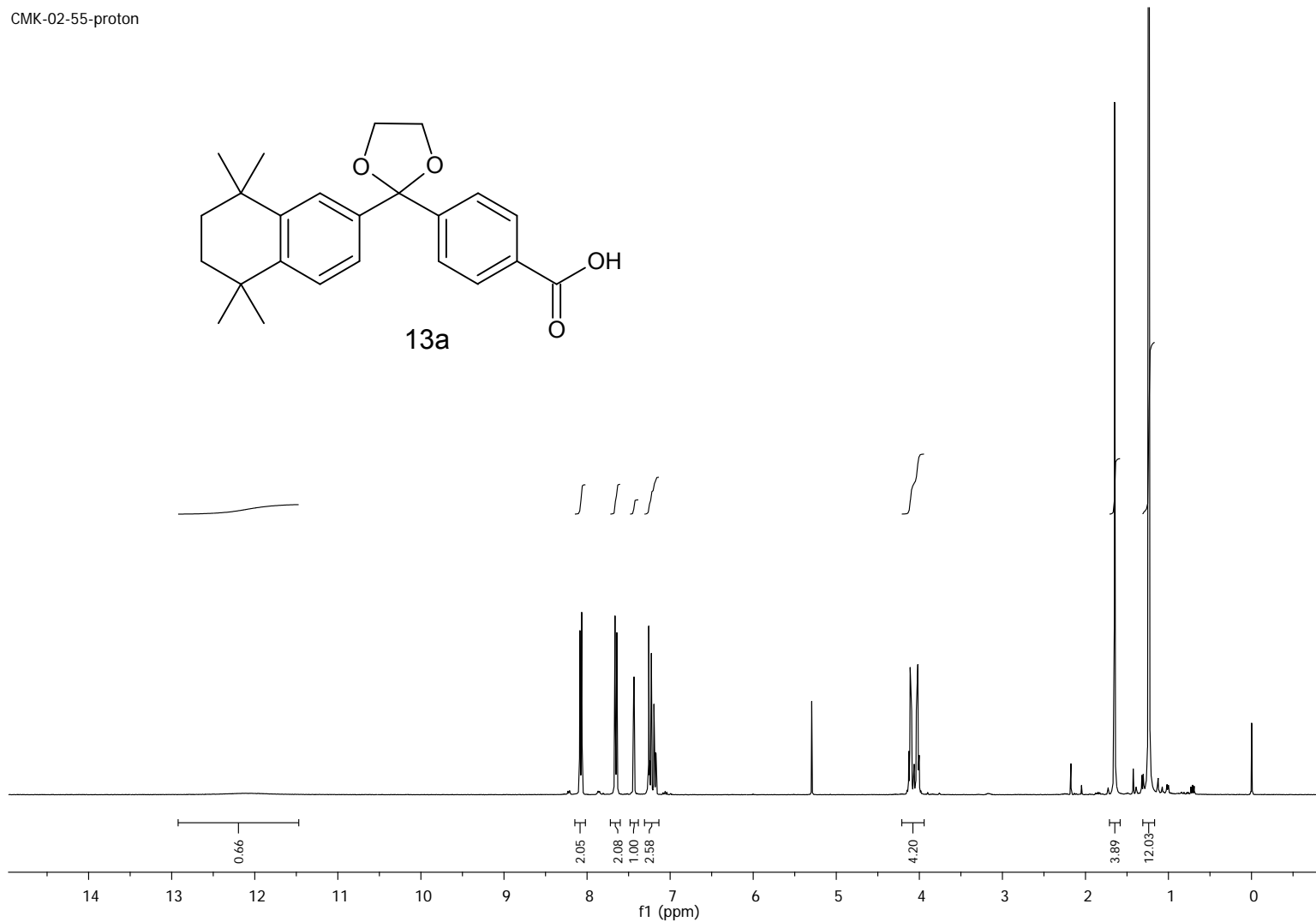
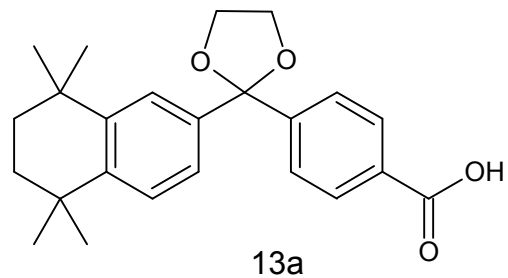


100 MHz ^{13}C -NMR of compound 12a in CDCl_3

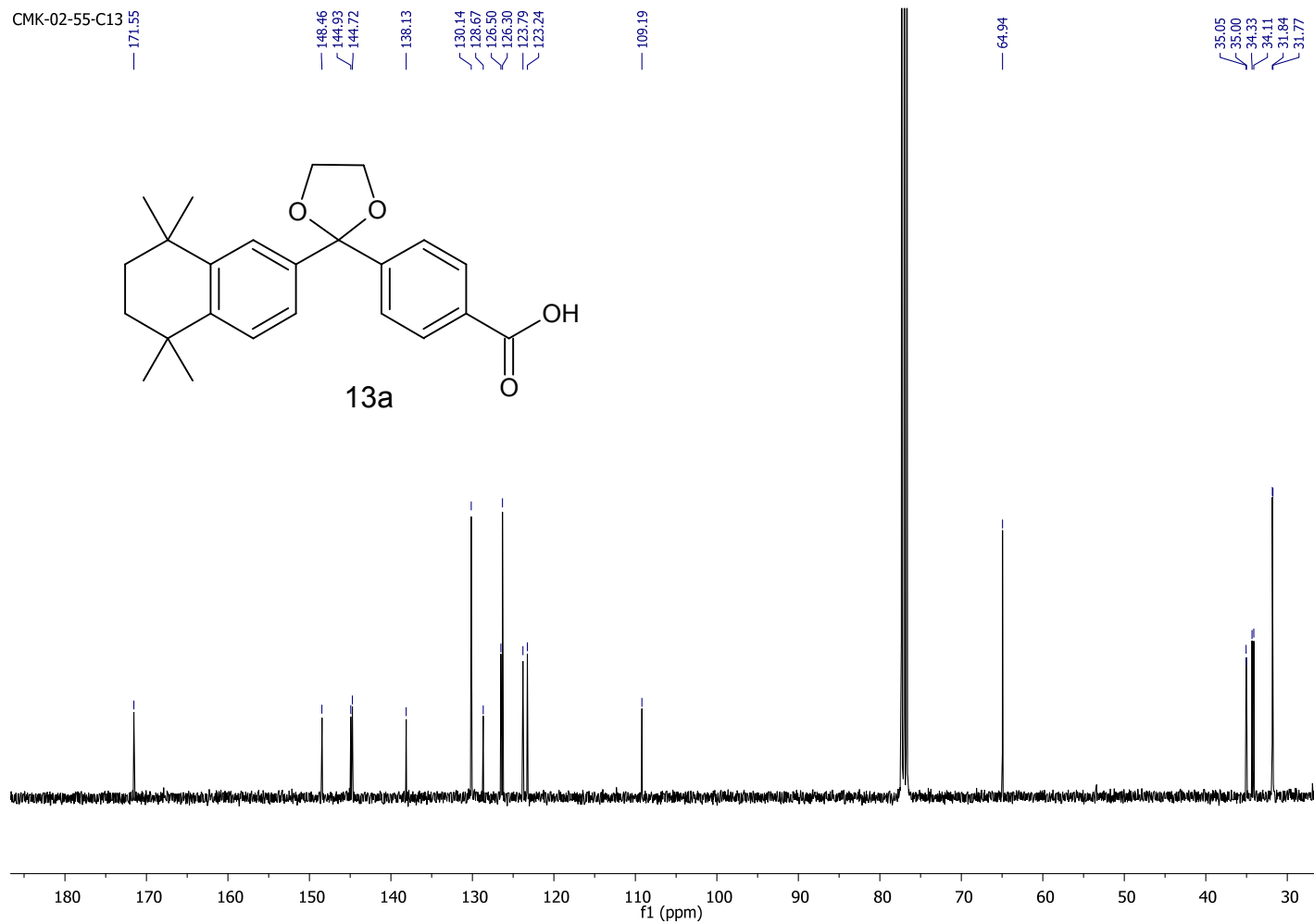


400 MHz ^1H -NMR of compound 13a in CDCl_3

CMK-02-55-proton

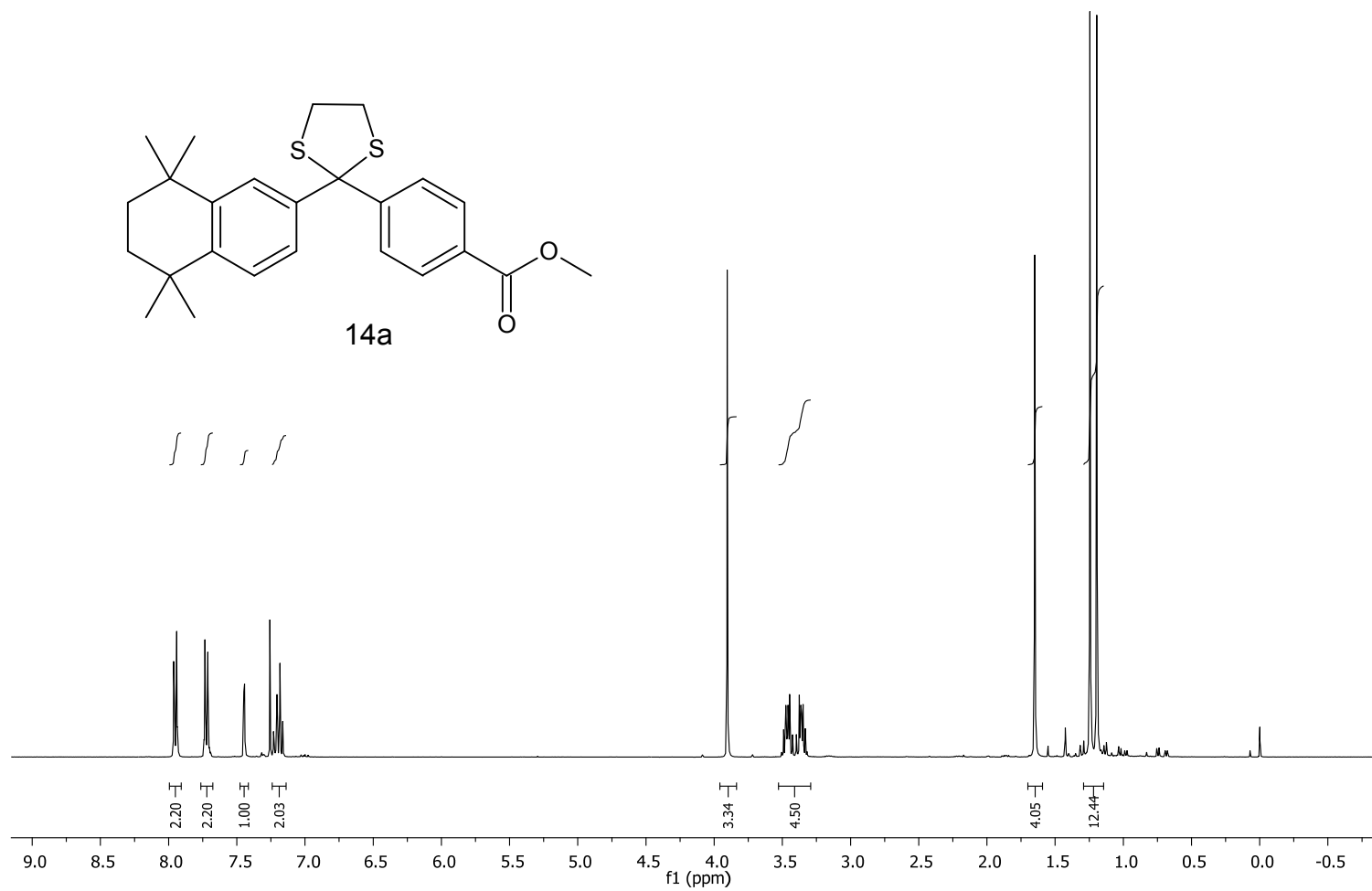


100 MHz ^{13}C -NMR of compound 13a in CDCl_3



400 MHz ^1H -NMR of compound 14a in CDCl_3

CMK-02-53-proton



100 MHz ^{13}C -NMR of compound 14a in CDCl_3

CMK-02-53-C13

166.79

150.39

144.37

144.03

140.08

129.12

128.77

128.37

126.24

125.34

52.07

40.23

35.02

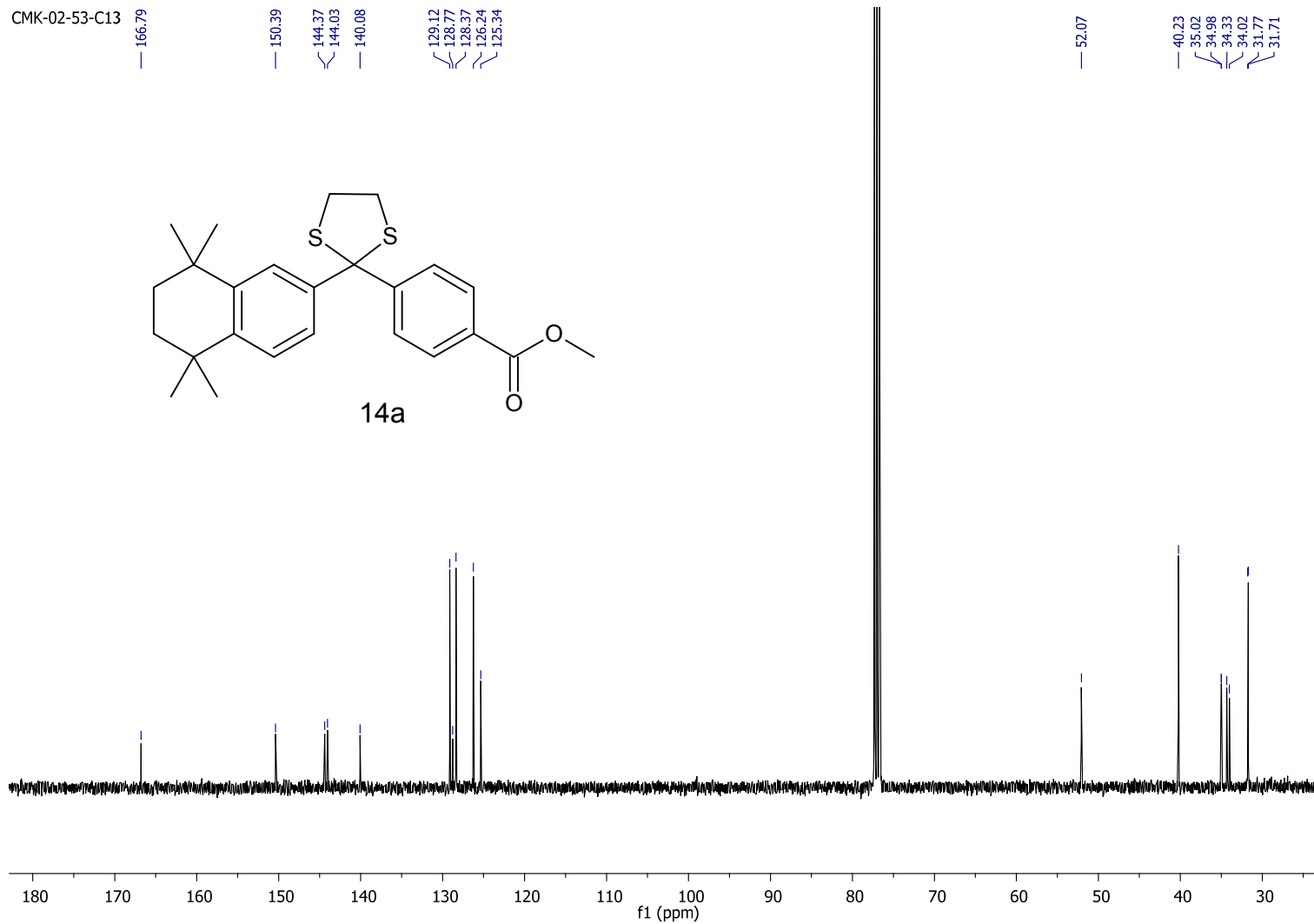
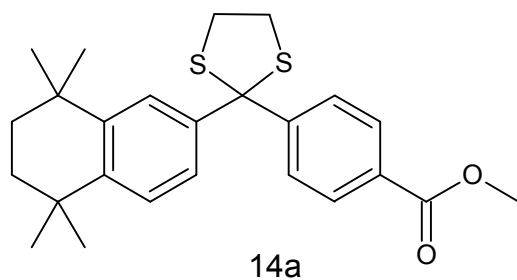
34.98

34.33

34.02

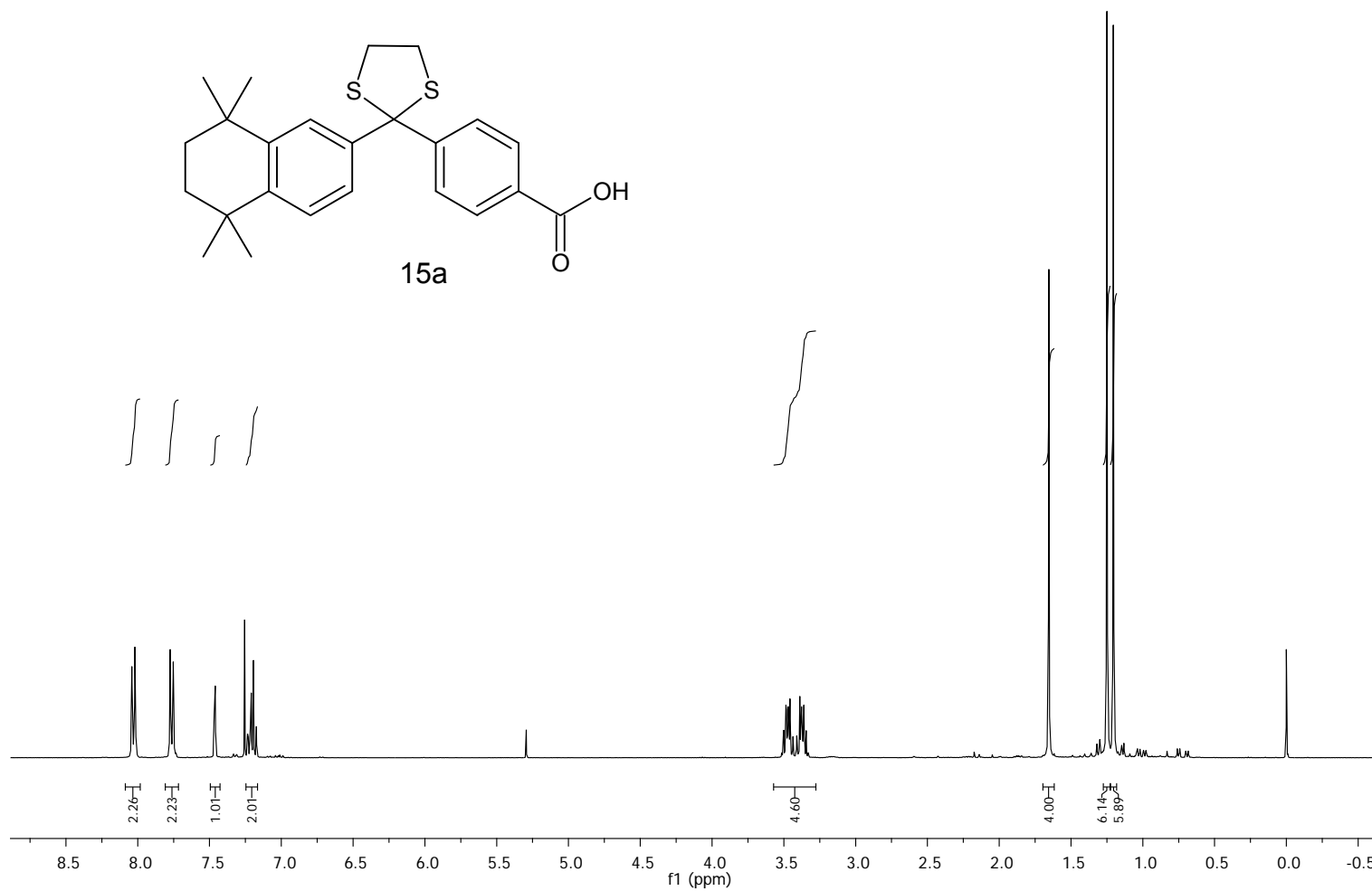
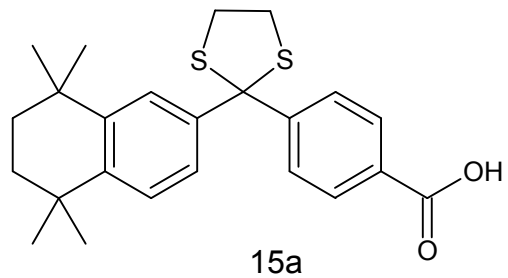
31.77

31.71



400 MHz $^1\text{H-NMR}$ of compound 15a in CDCl_3

CMK-02-54-proton



100 MHz ¹³C-NMR of compound 15a in CDCl₃

CMK-02-54-C13

171.58

151.50

144.44

144.10

139.98

129.80

128.49

127.83

126.30

126.21

125.33

40.28

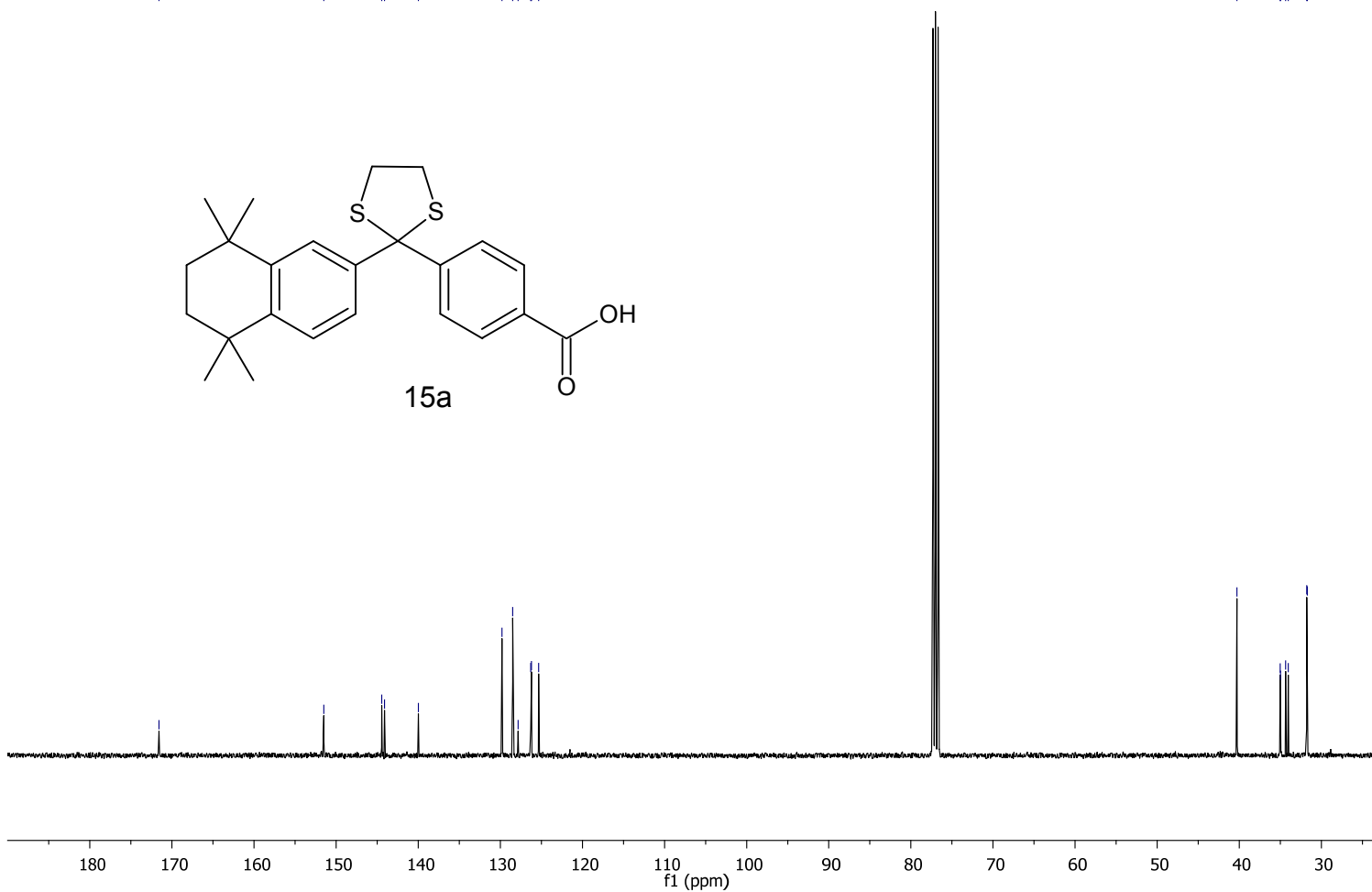
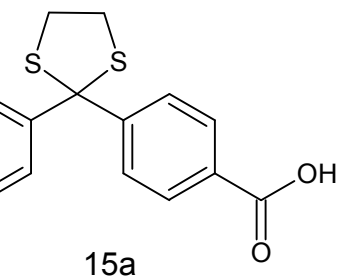
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34.99

34.35

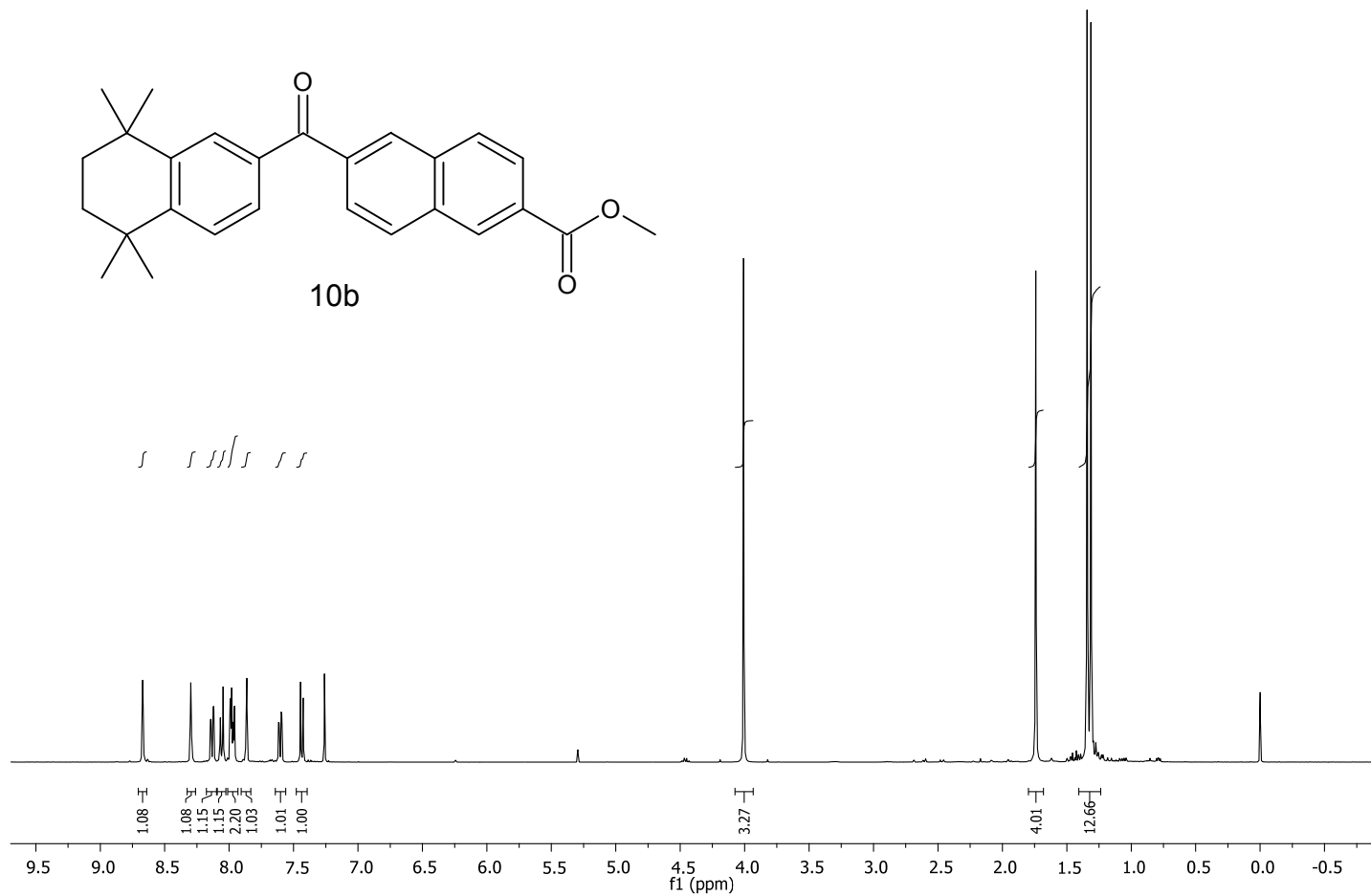
34.03

31.78



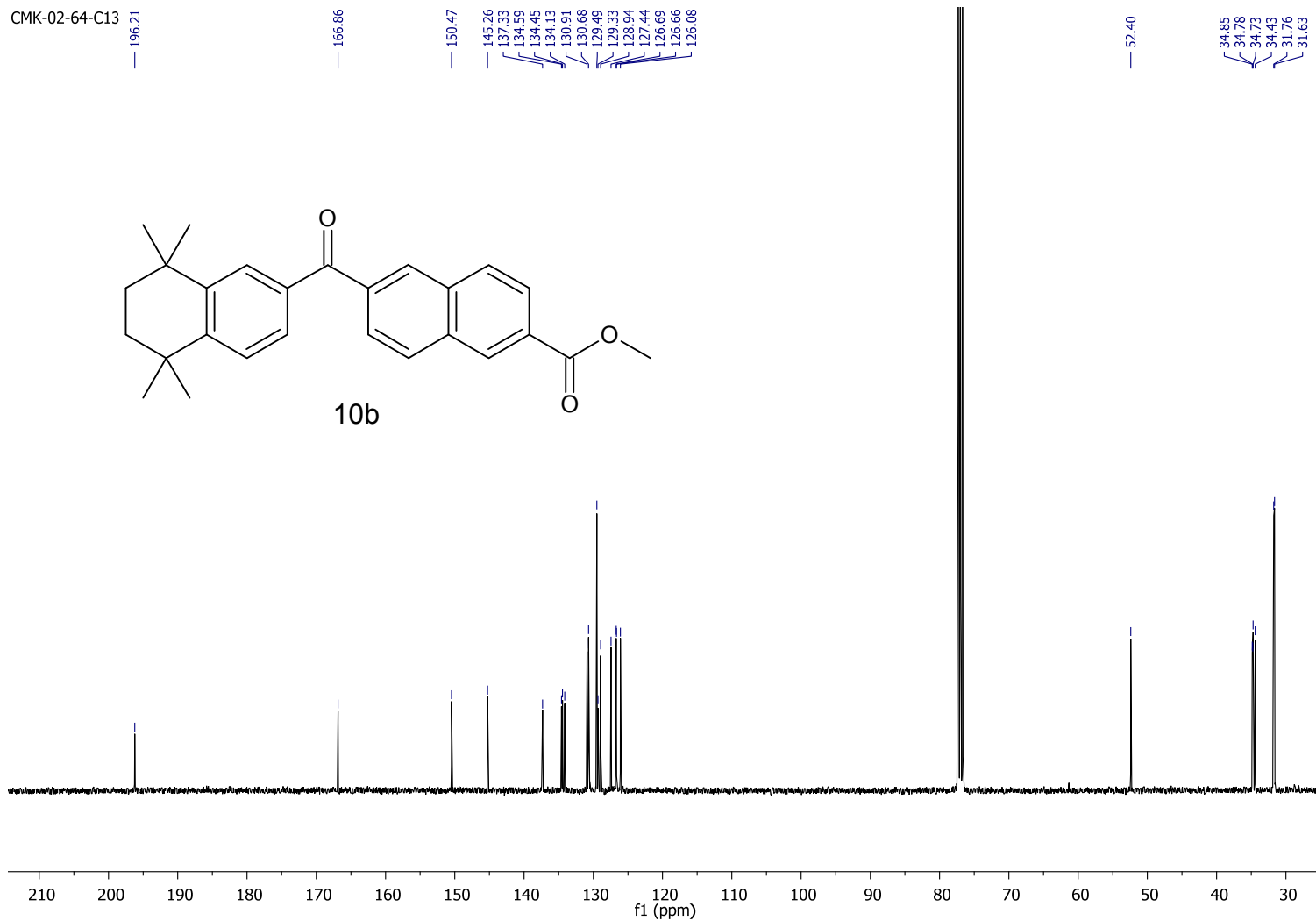
400 MHz ^1H -NMR of compound 10b in CDCl_3

CMK-02-64-proton



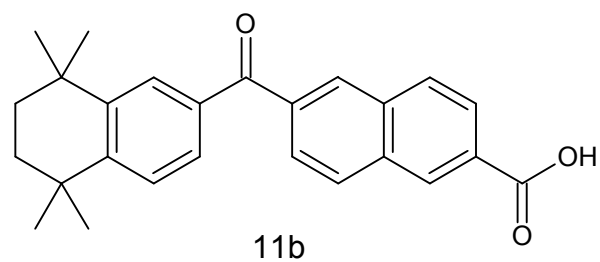
100 MHz ¹³C-NMR of compound 10b in CDCl₃

CMK-02-64-C13

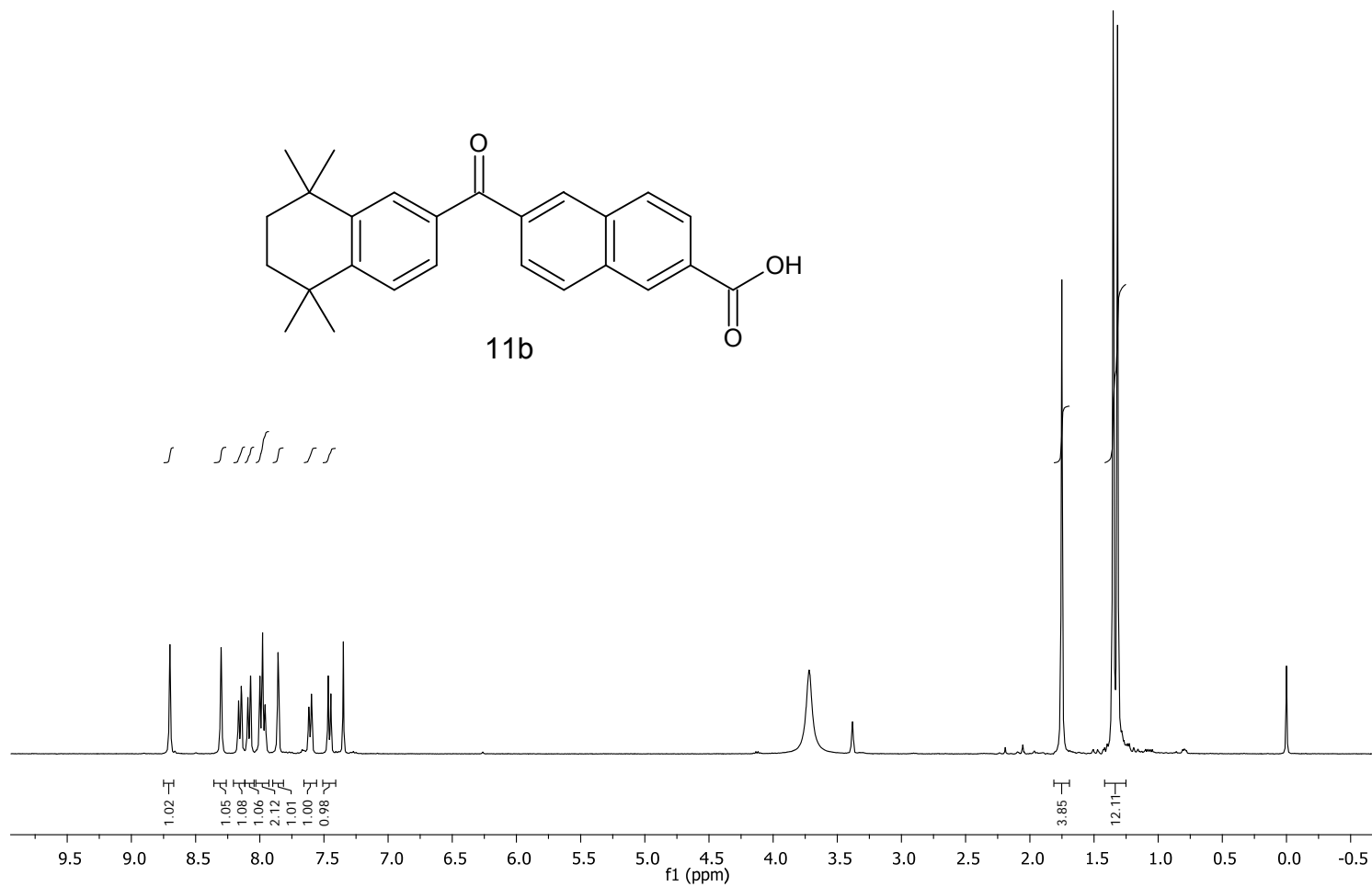


400 MHz ^1H -NMR of compound 11b in $\text{CDCl}_3/\text{CD}_3\text{OD}$ mixture

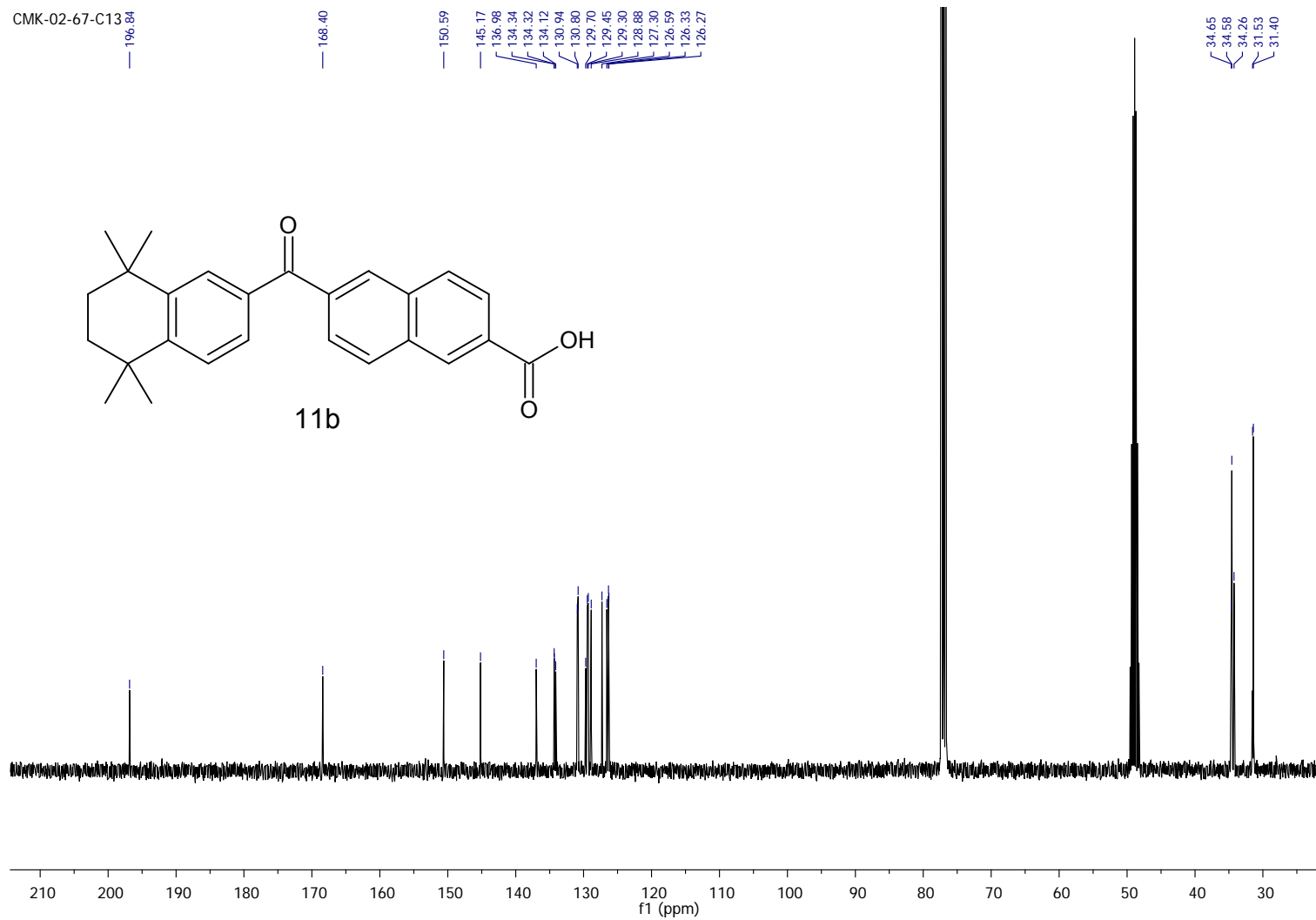
CMK-02-67-proton



s s s s s s

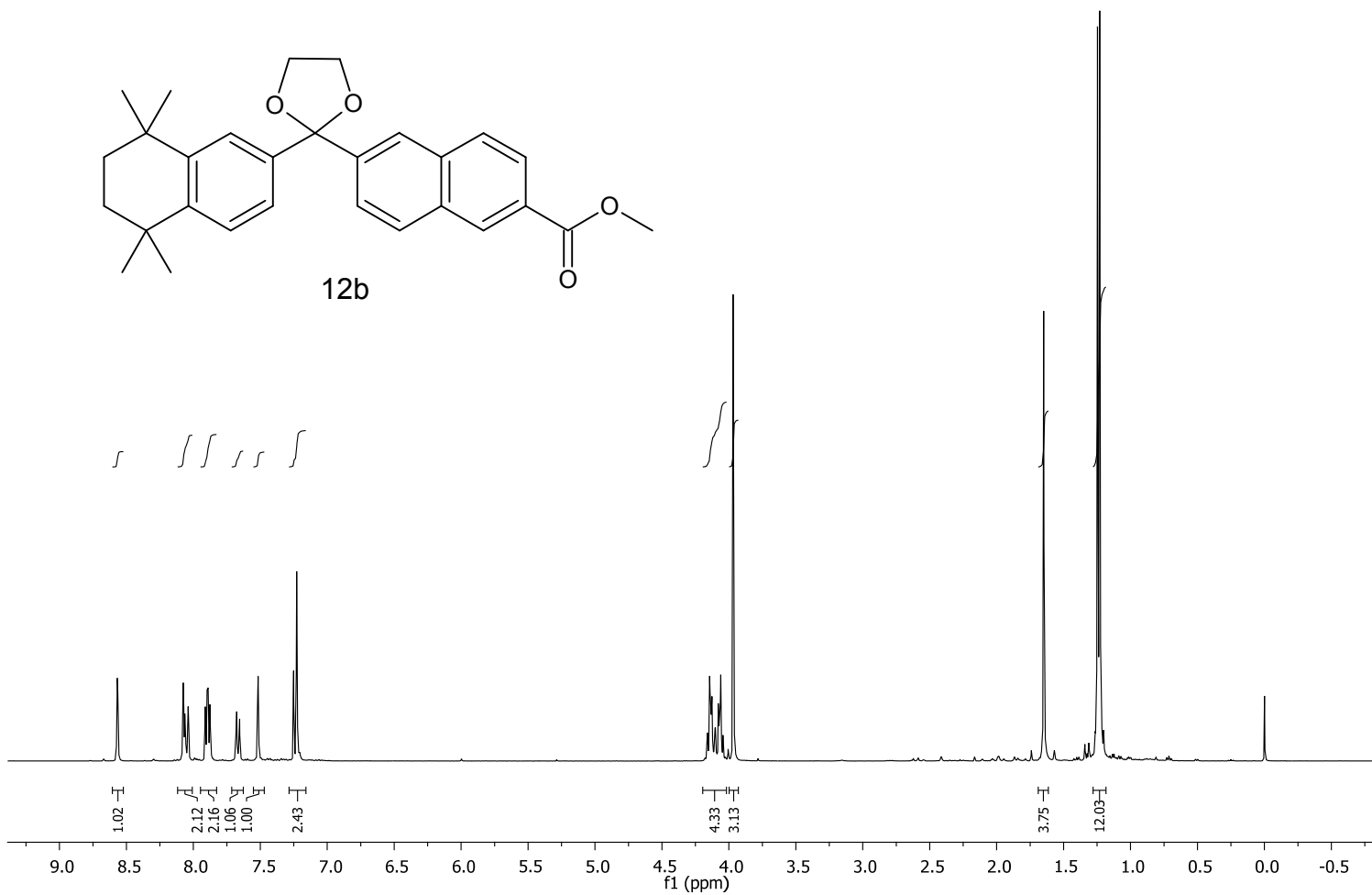


100 MHz ^{13}C -NMR of compound 11b in $\text{CDCl}_3/\text{CD}_3\text{OD}$ mixture

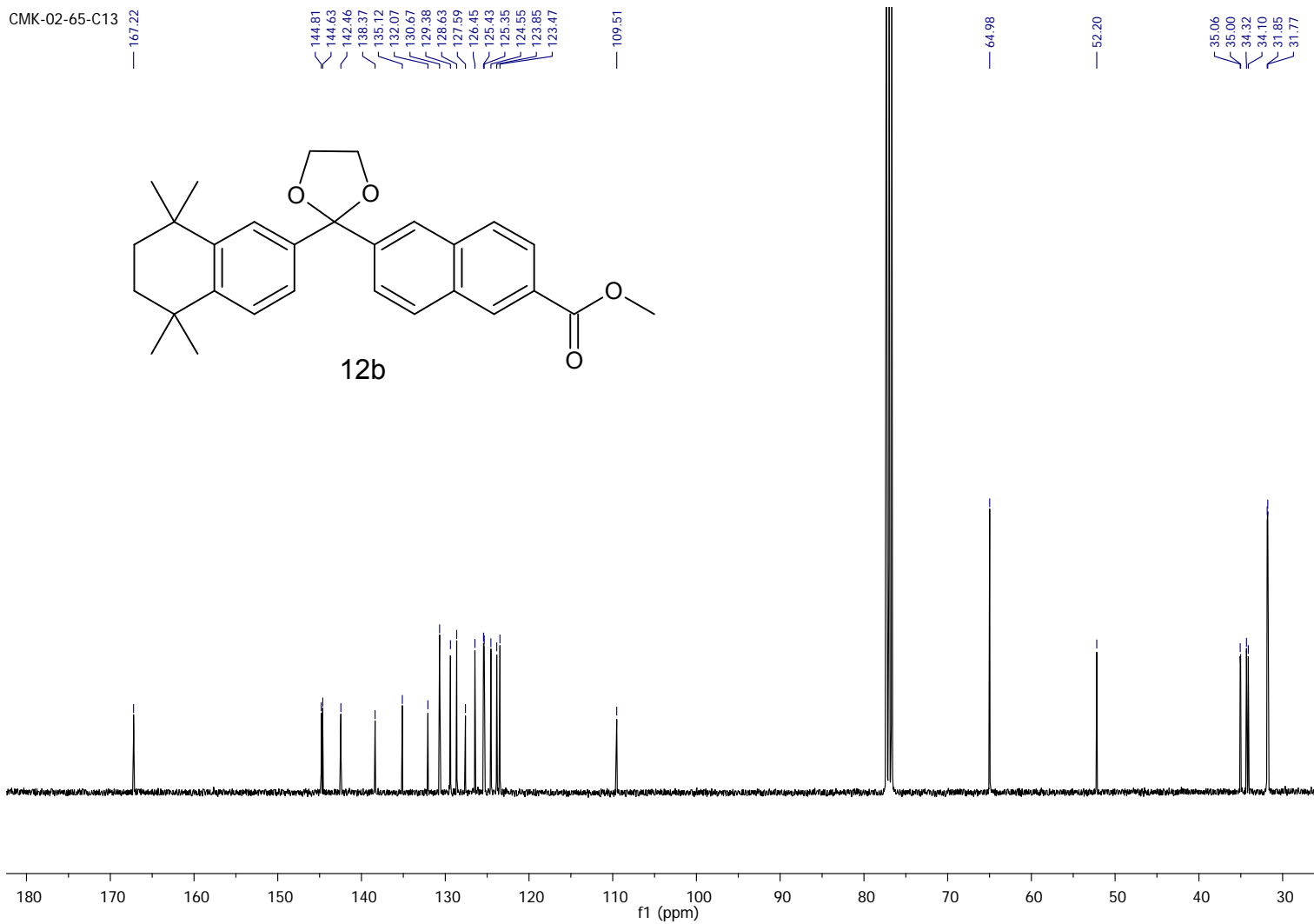


400 MHz $^1\text{H-NMR}$ of compound 12b in CDCl_3

CMK-02-65-proton



100 MHz ^{13}C -NMR of compound 12b in CDCl_3



100 MHz ^{13}C -NMR of compound 13b in CDCl_3

CMK-02-71-proton

172.09

144.86

144.67

142.95

138.32

135.60

132.02

131.77

129.58

128.82

126.68

126.48

125.60

125.52

124.60

123.87

123.46

109.50

65.01

35.06

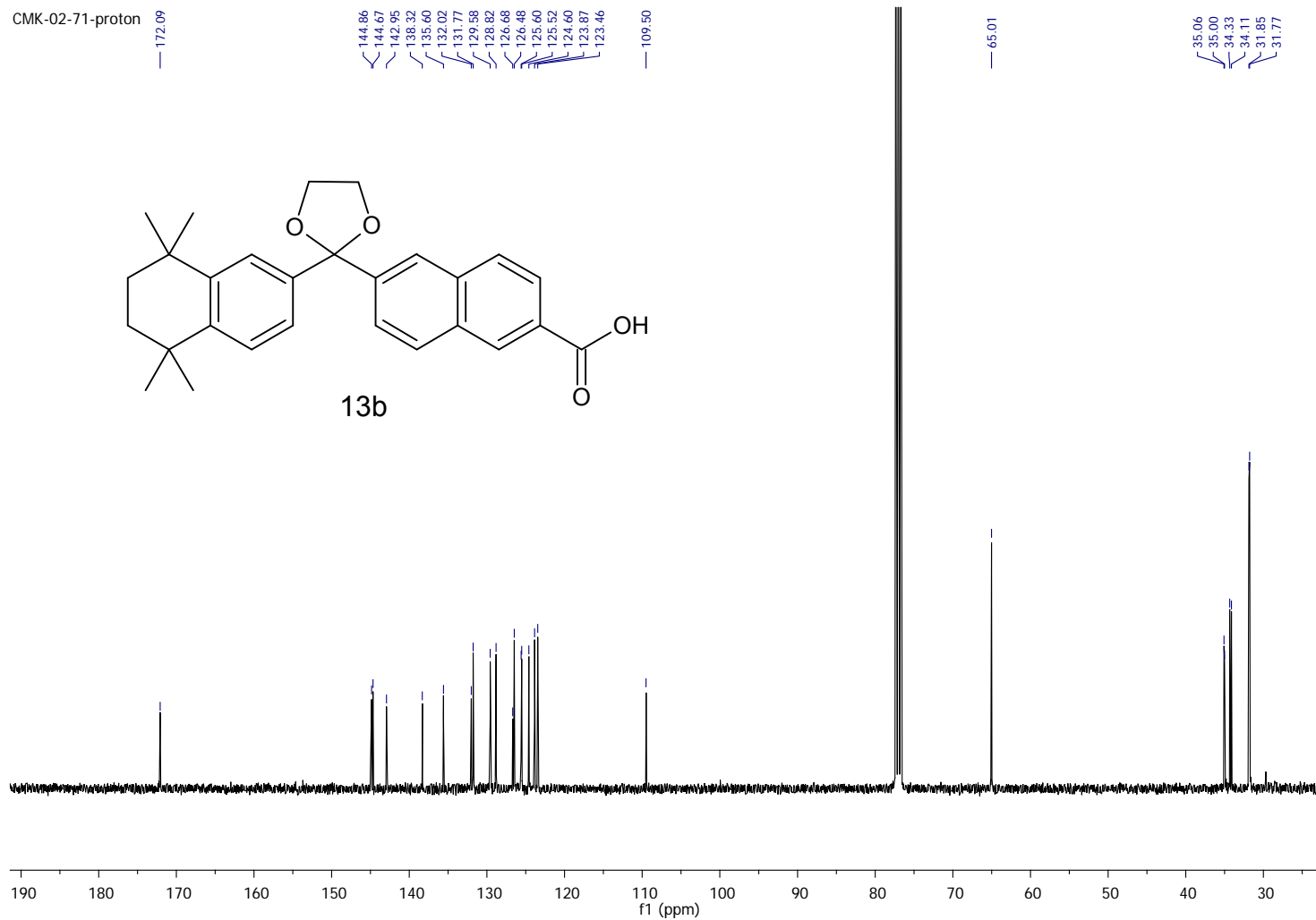
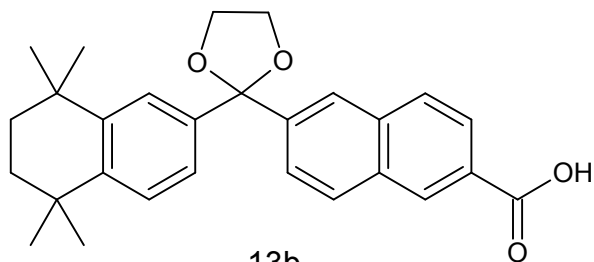
35.00

34.33

34.11

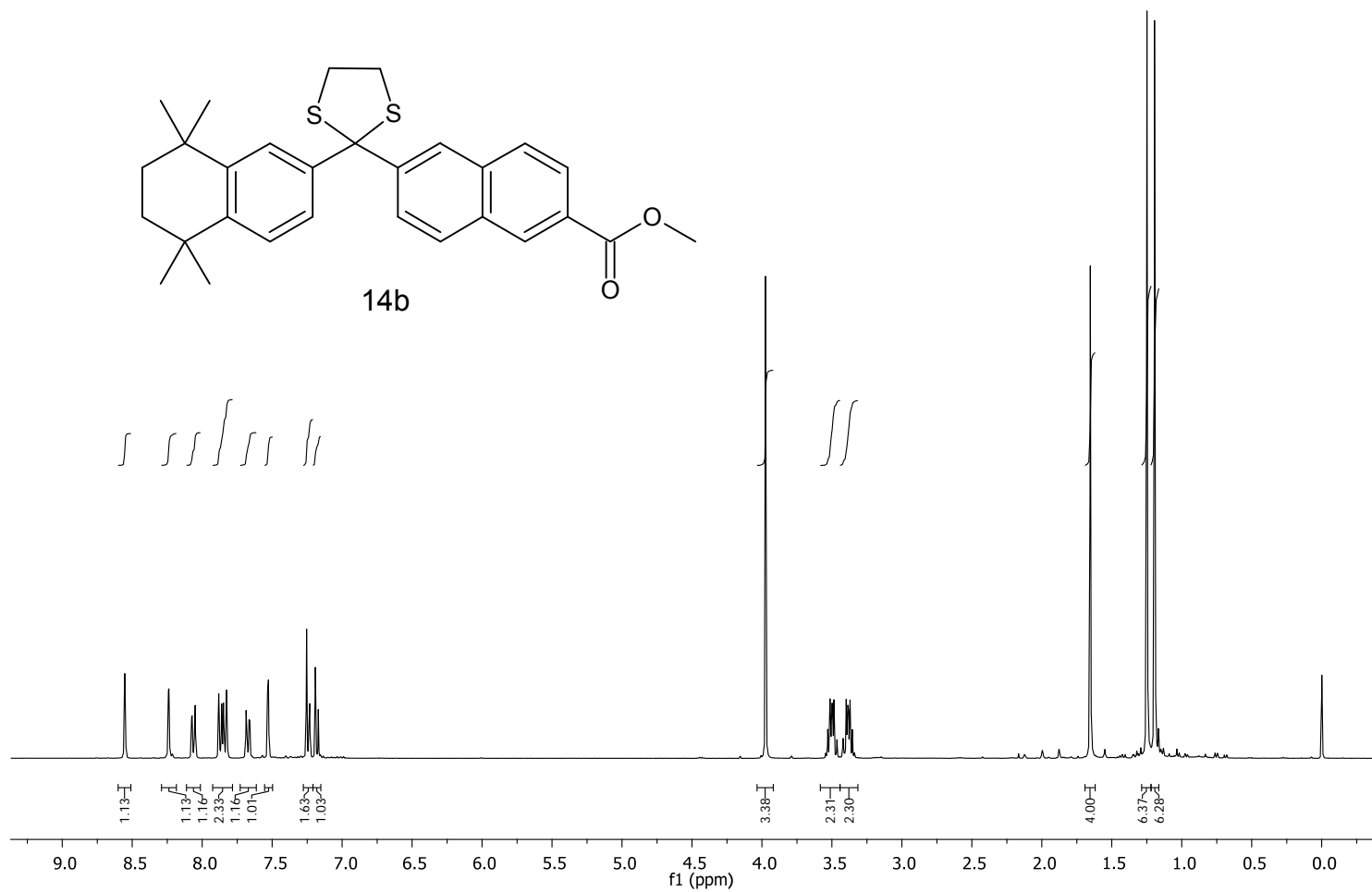
31.85

31.77



400 MHz ^1H -NMR of compound 14b in CDCl_3

CMK-02-66-proton



100 MHz ^{13}C -NMR of compound 14b in CDCl_3

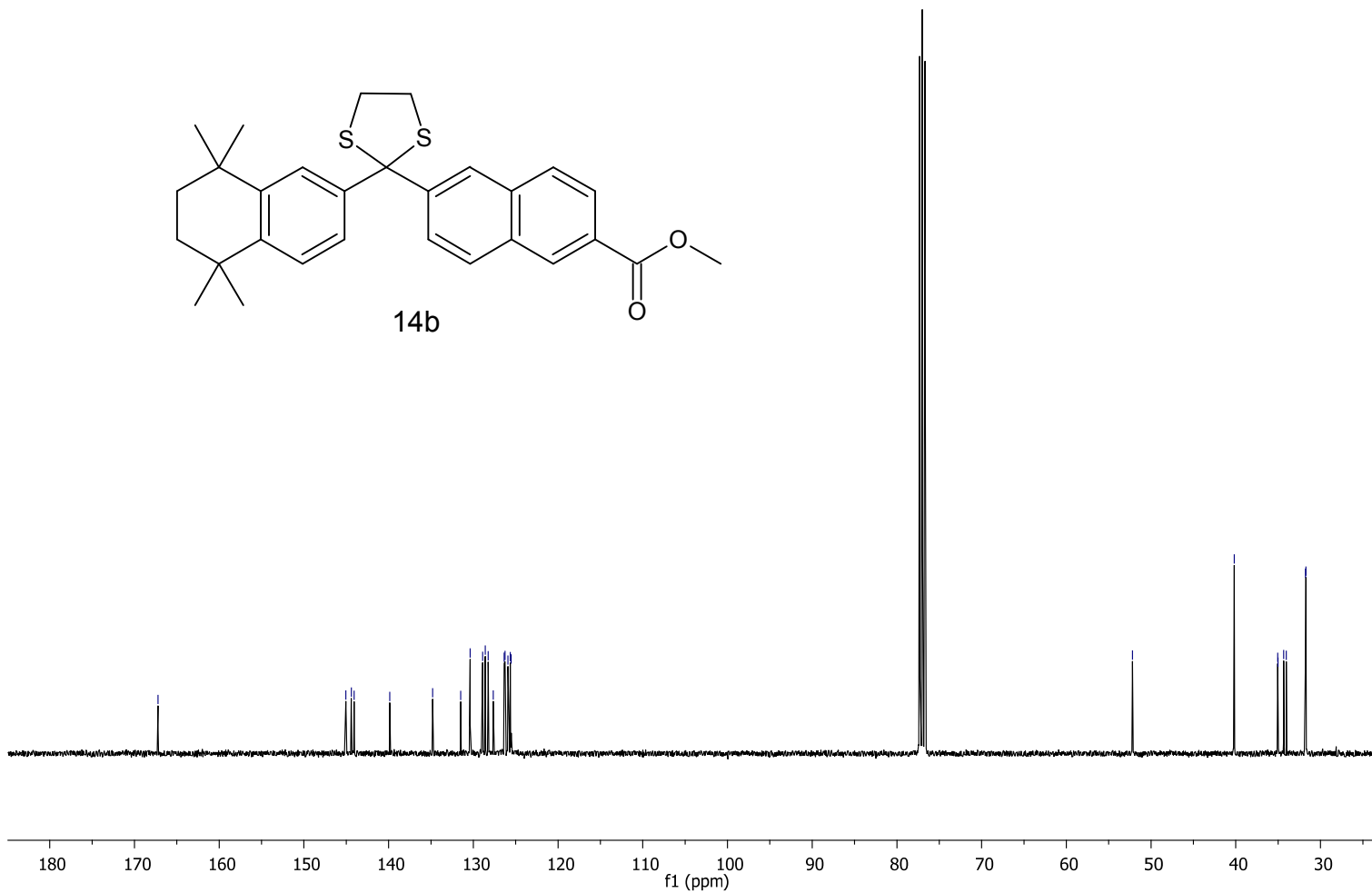
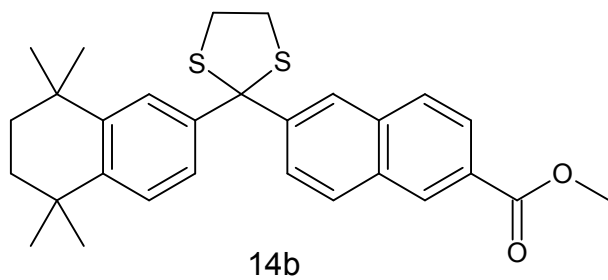
CMK-02-66-C13

167.23

145.05
144.41
144.06
139.86
134.81
131.49
130.38
128.91
128.60
128.24
127.64
126.36
126.26
125.92
125.63
125.53

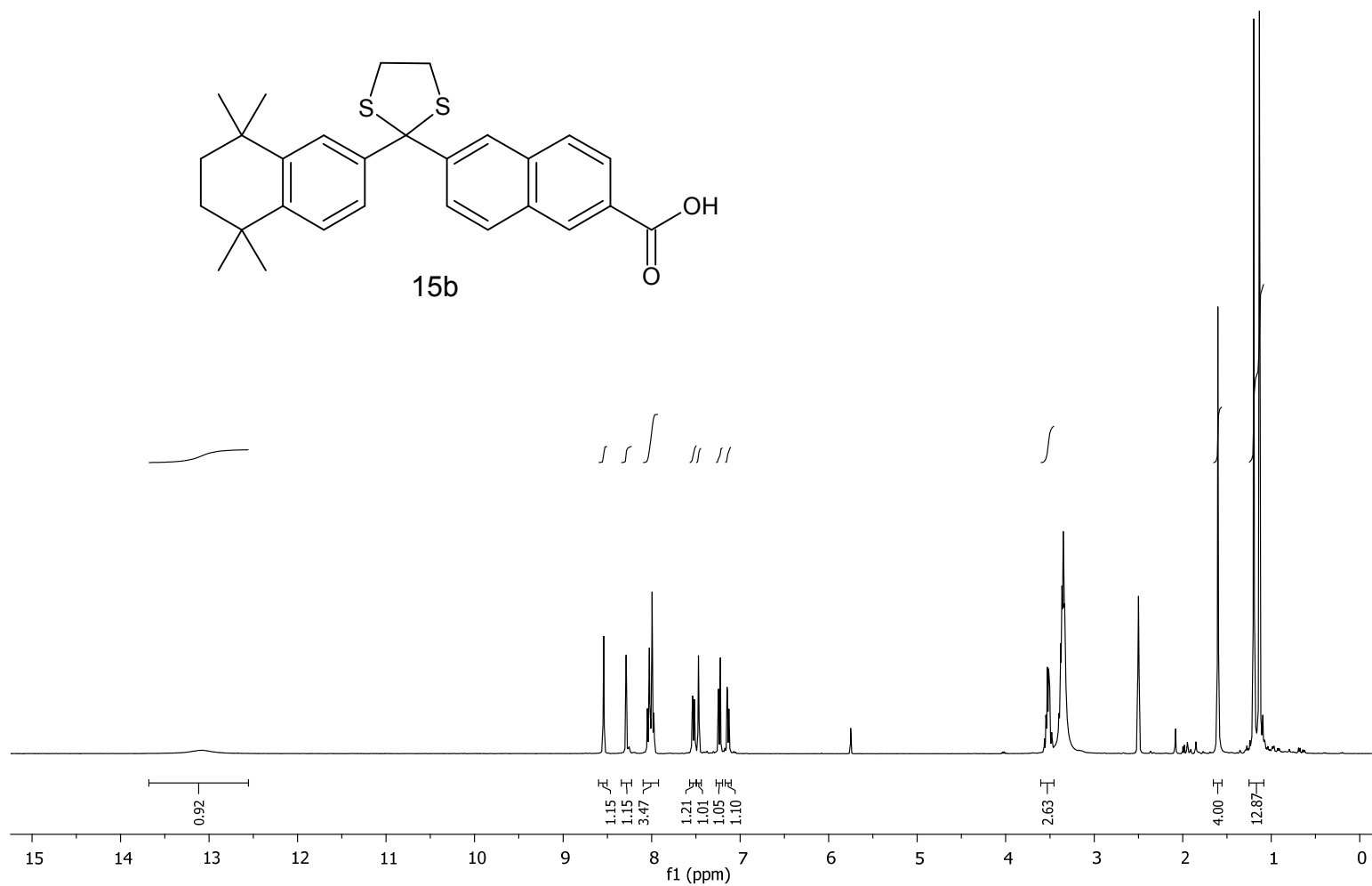
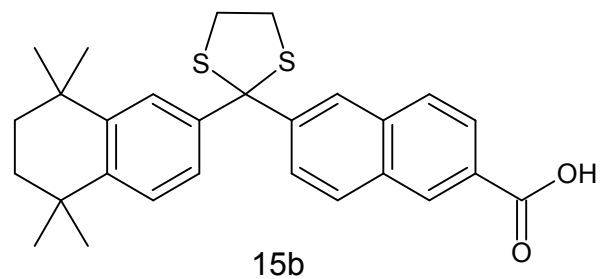
52.20

40.18
35.05
35.01
34.35
34.04
31.77
31.73



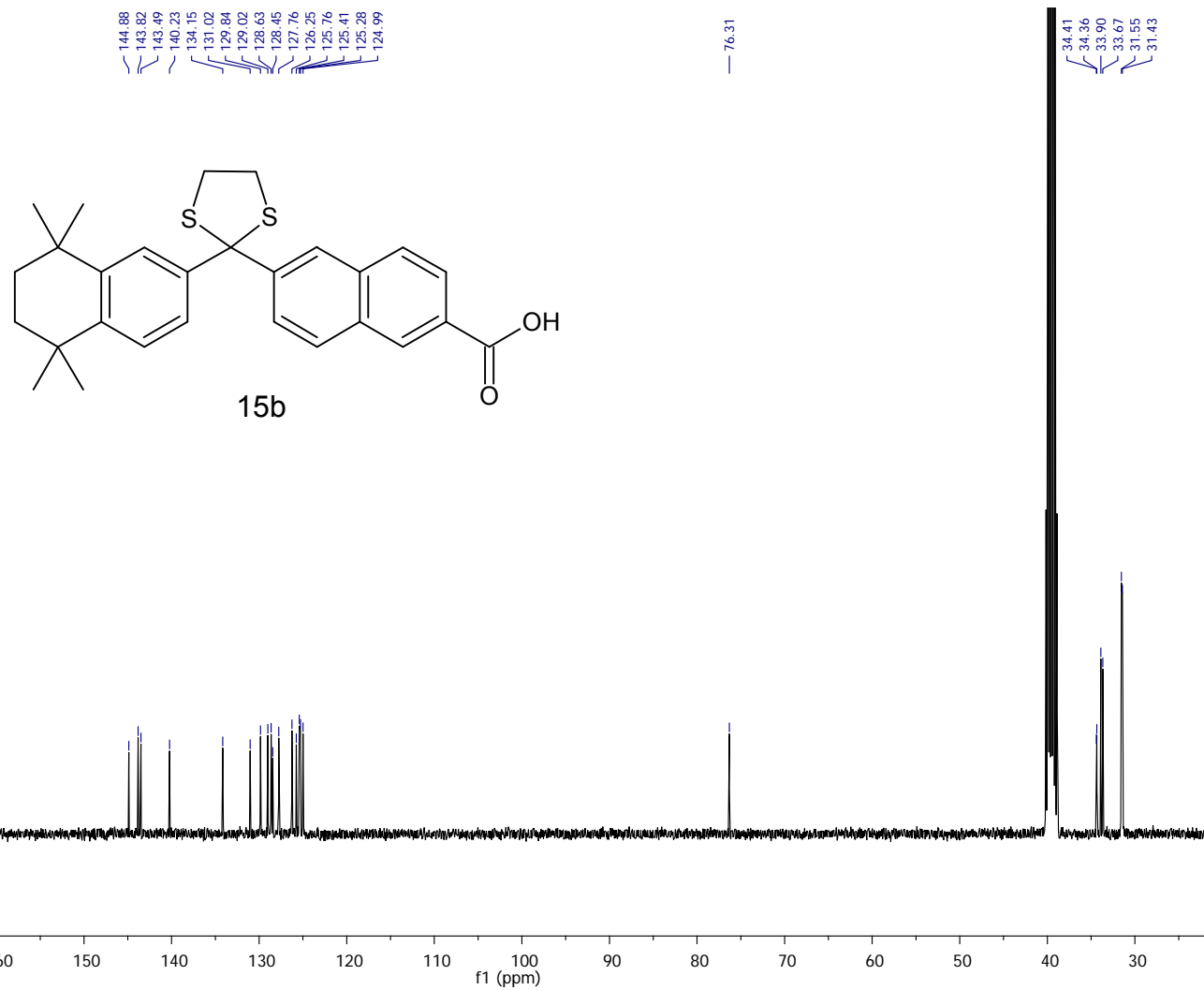
400 MHz $^1\text{H-NMR}$ of compound 15b in DMSO-D_6

CMK-02-69-proton



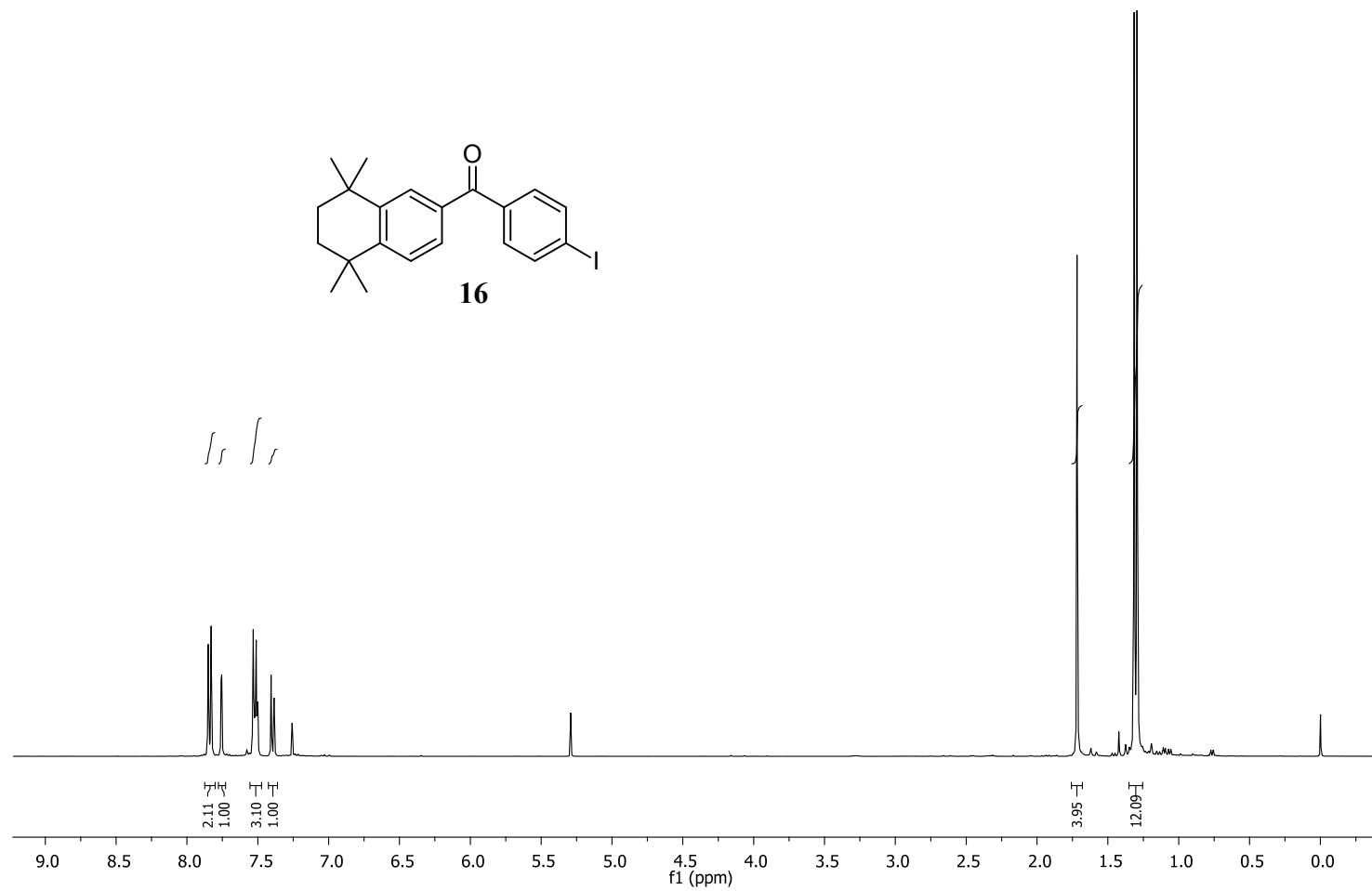
100 MHz ^{13}C -NMR of compound 15b in DMSO-D_6

CMK-02-69-C13



400 MHz ^1H -NMR of compound 16 in CDCl_3

CMK-02-100-proton



100 MHz ^{13}C -NMR of compound 16 in CDCl_3

CMK-02-100-

195.71

150.44

145.20

137.43

137.31

134.27

131.42

128.72

127.21

126.63

99.67

34.81

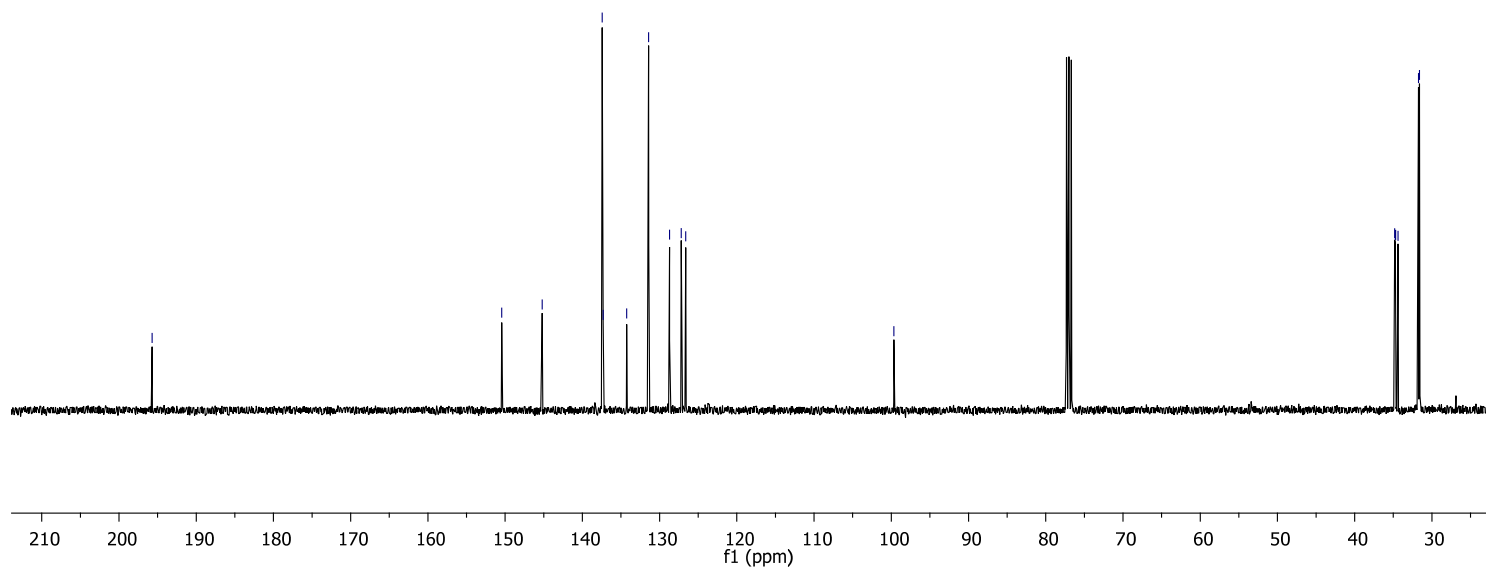
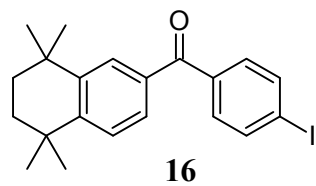
34.73

34.69

34.39

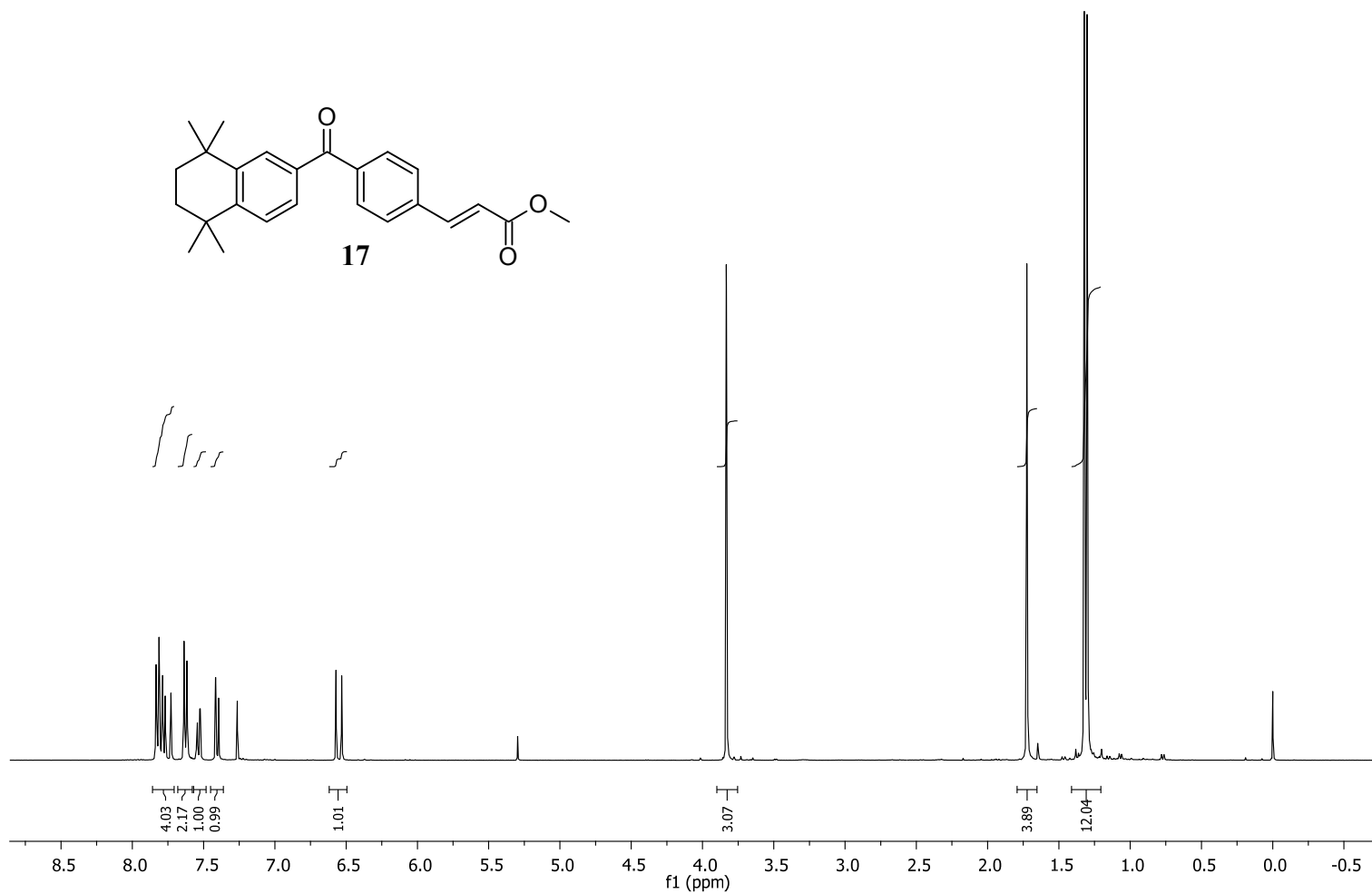
31.74

31.60

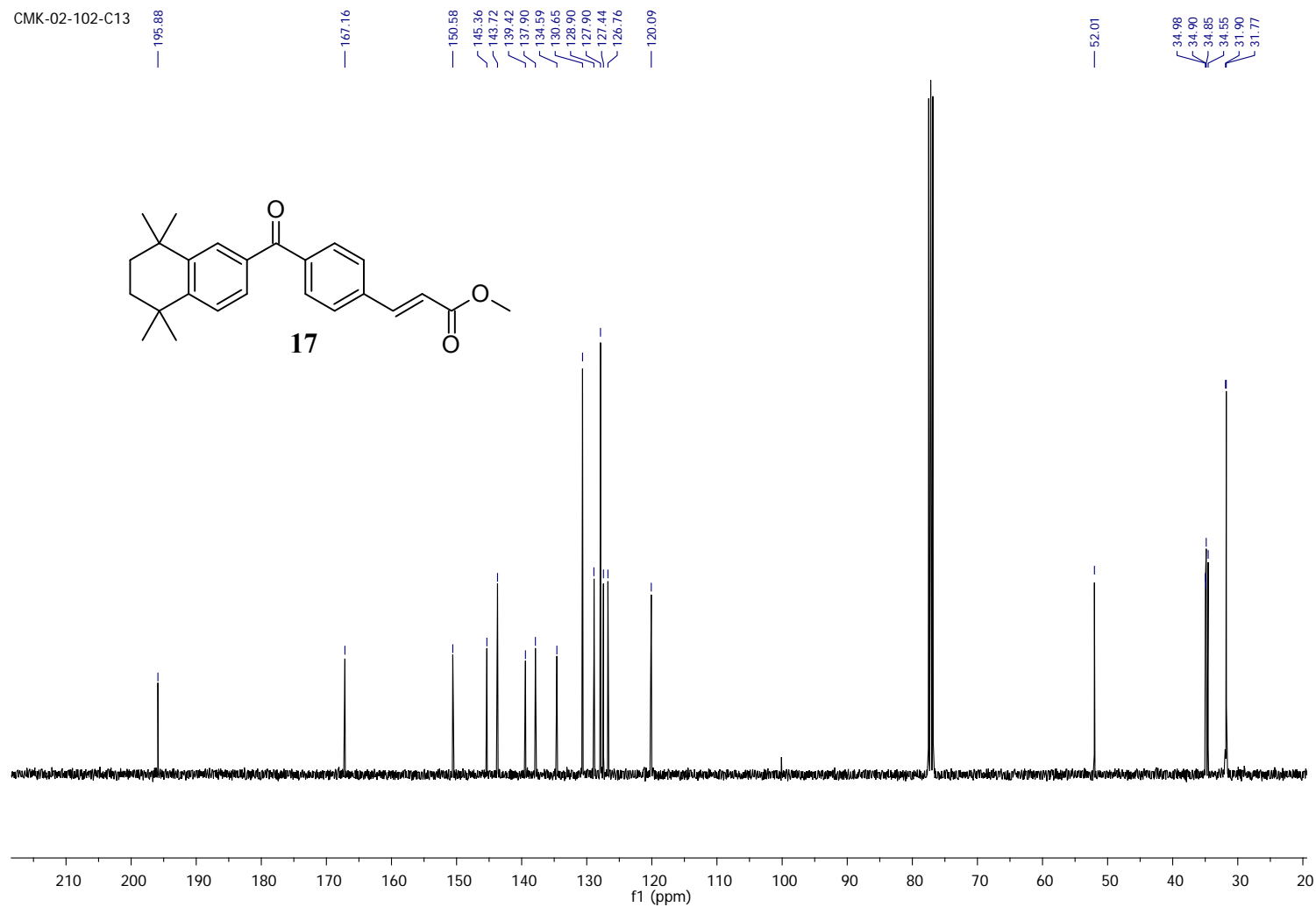


400 MHz $^1\text{H-NMR}$ of compound 17 in CDCl_3

CMK-02-102-proton

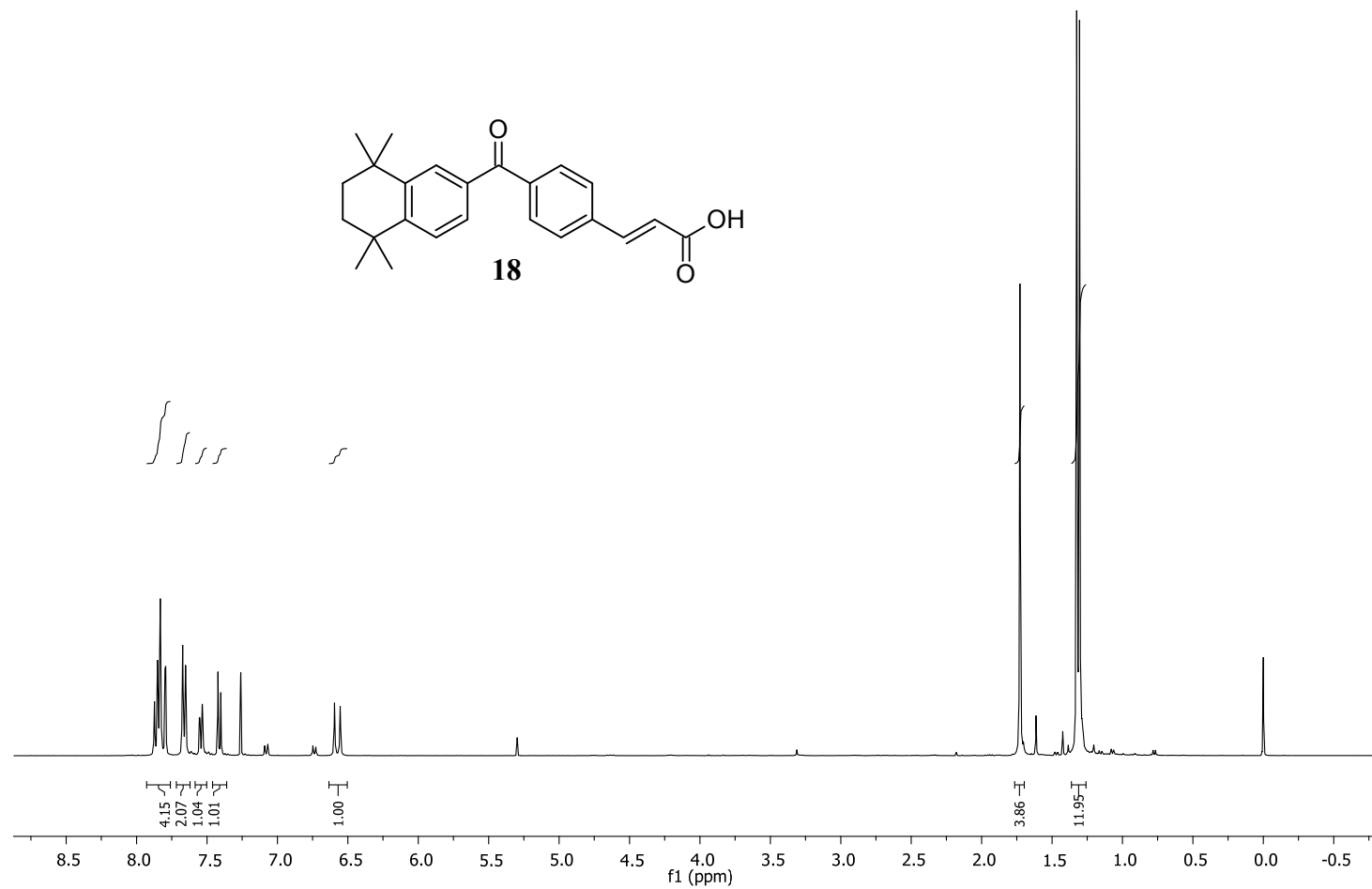


100 MHz ^{13}C -NMR of compound 17 in CDCl_3

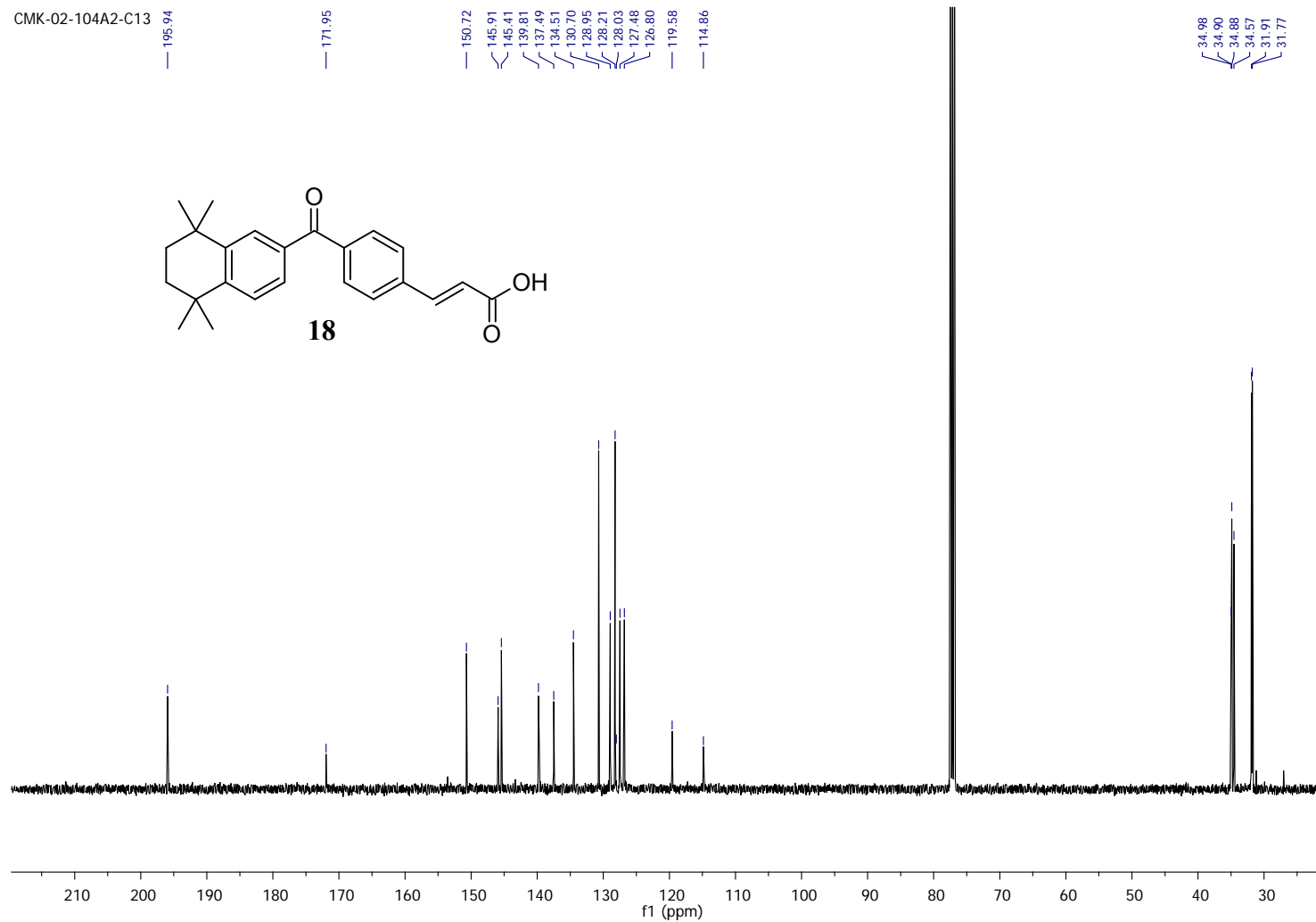


400 MHz $^1\text{H-NMR}$ of compound 18 in CDCl_3

CMK-02-104A2-proton

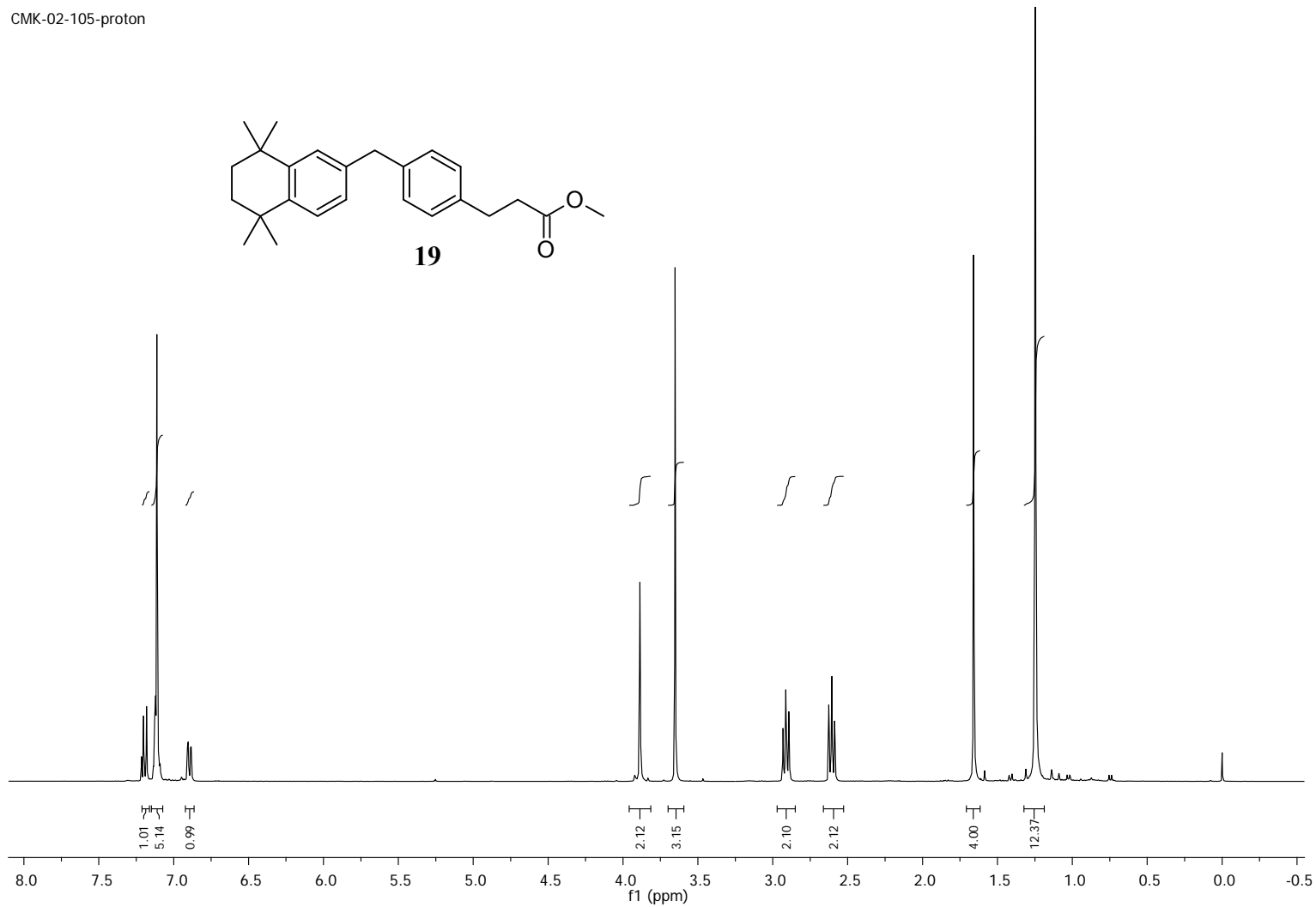
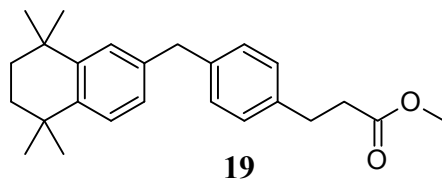


100 MHz ^{13}C -NMR of compound 18 in CDCl_3



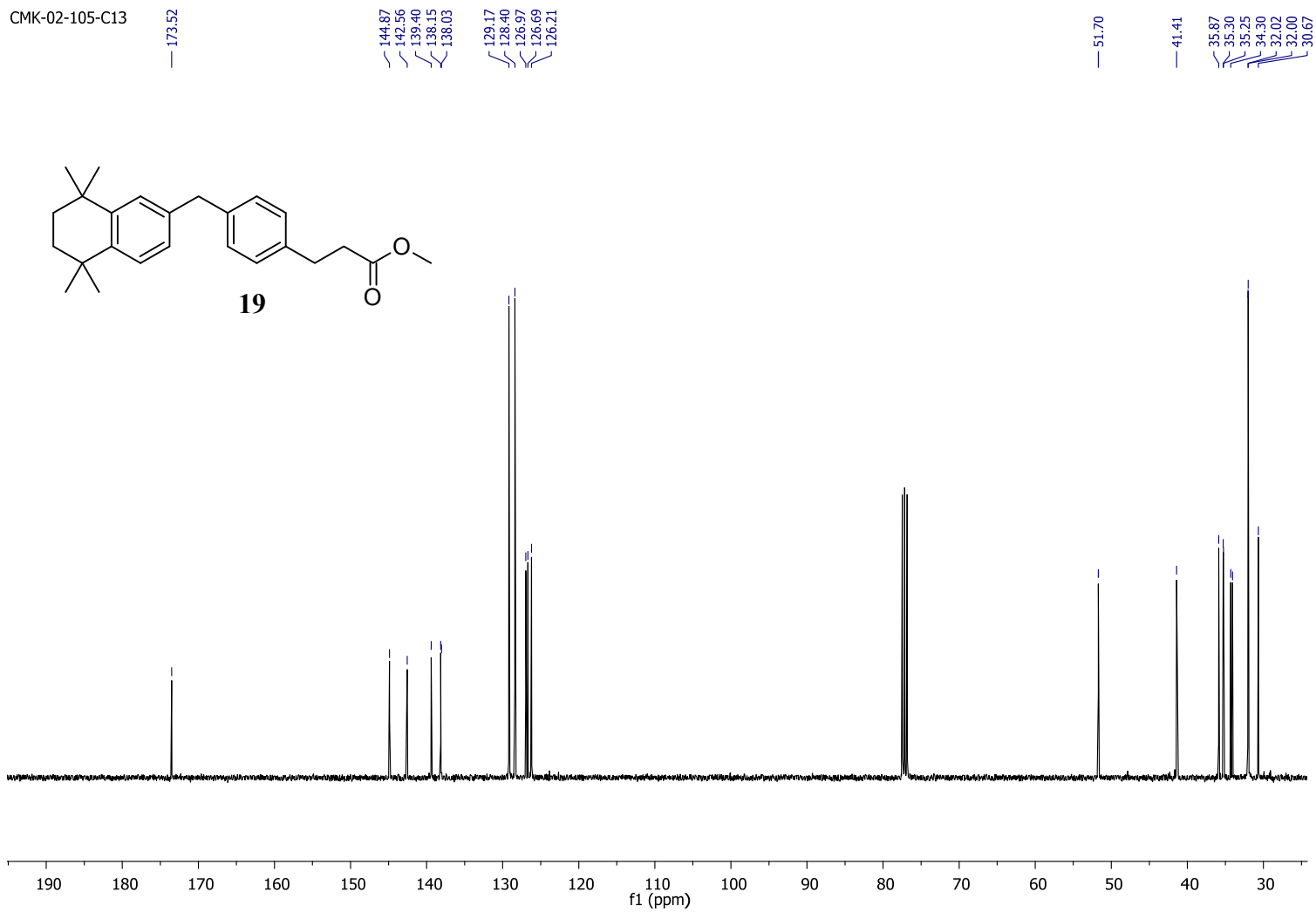
400 MHz $^1\text{H-NMR}$ of compound **19** in CDCl_3

CMK-02-105-proton



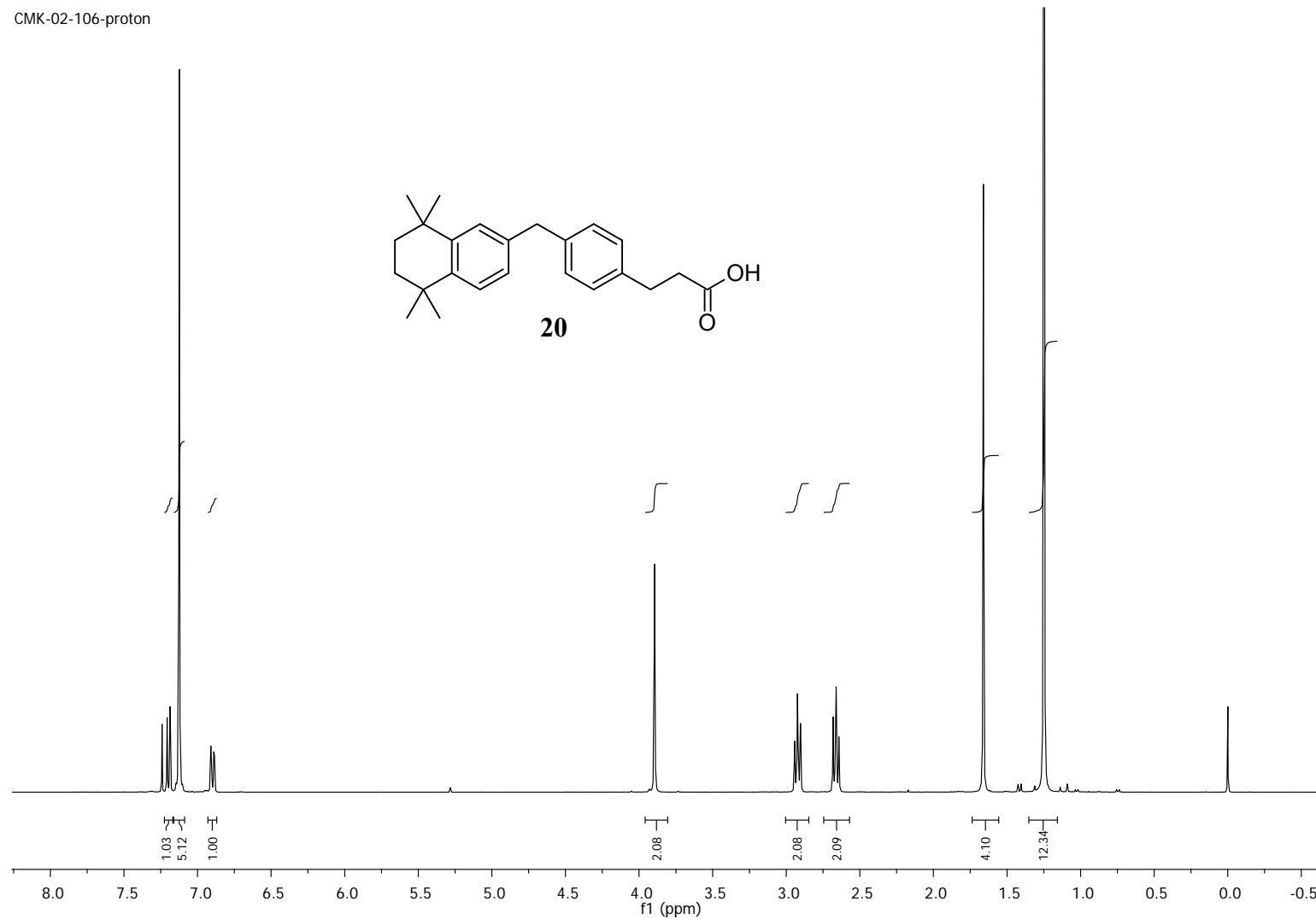
100 MHz ^{13}C -NMR of compound 19 in CDCl_3

CMK-02-105-C13



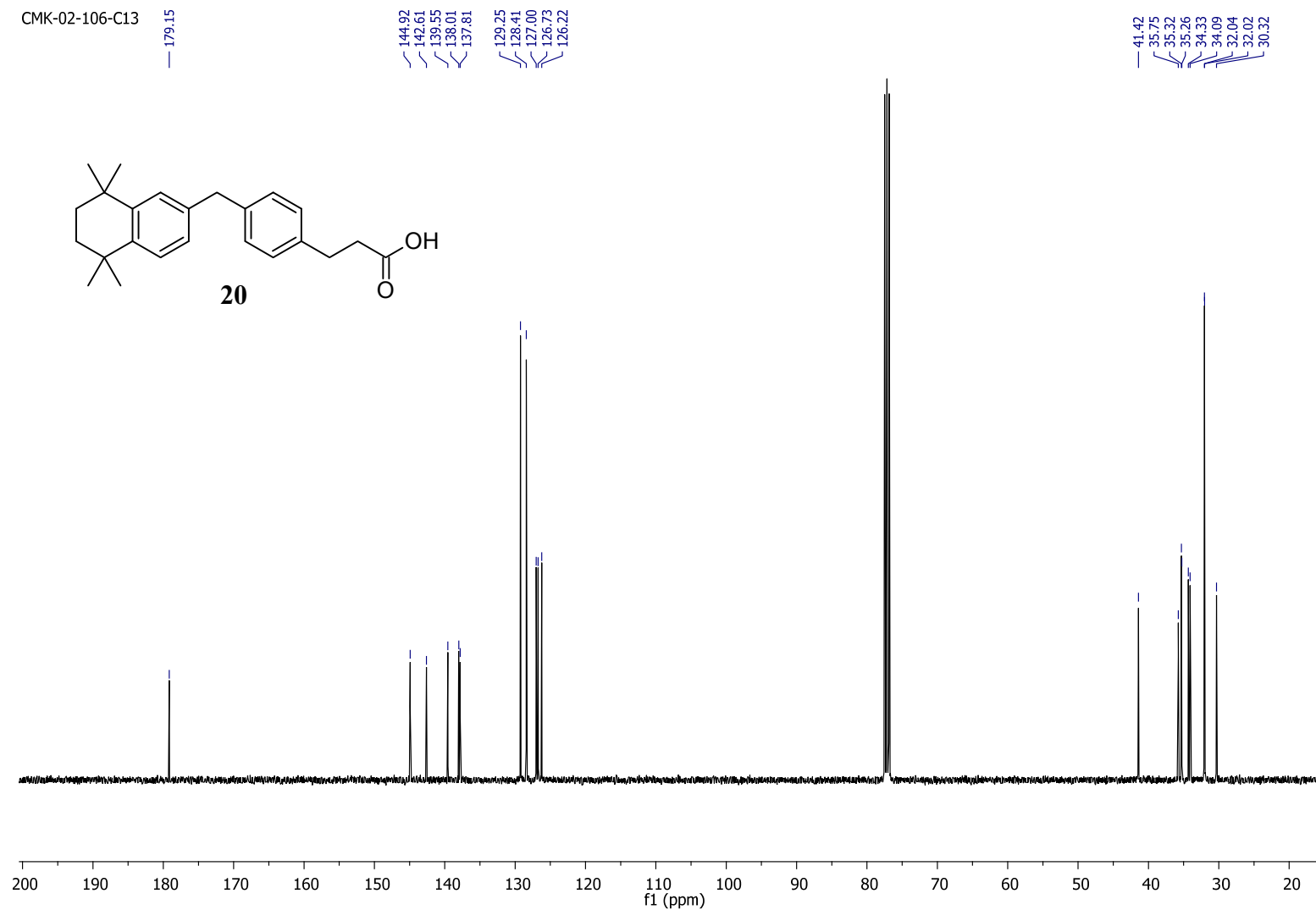
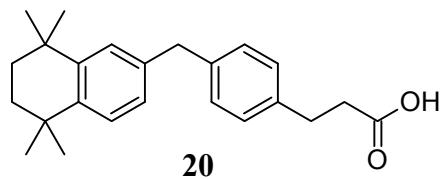
400 MHz ^1H -NMR of compound 20 in CDCl_3

CMK-02-106-proton



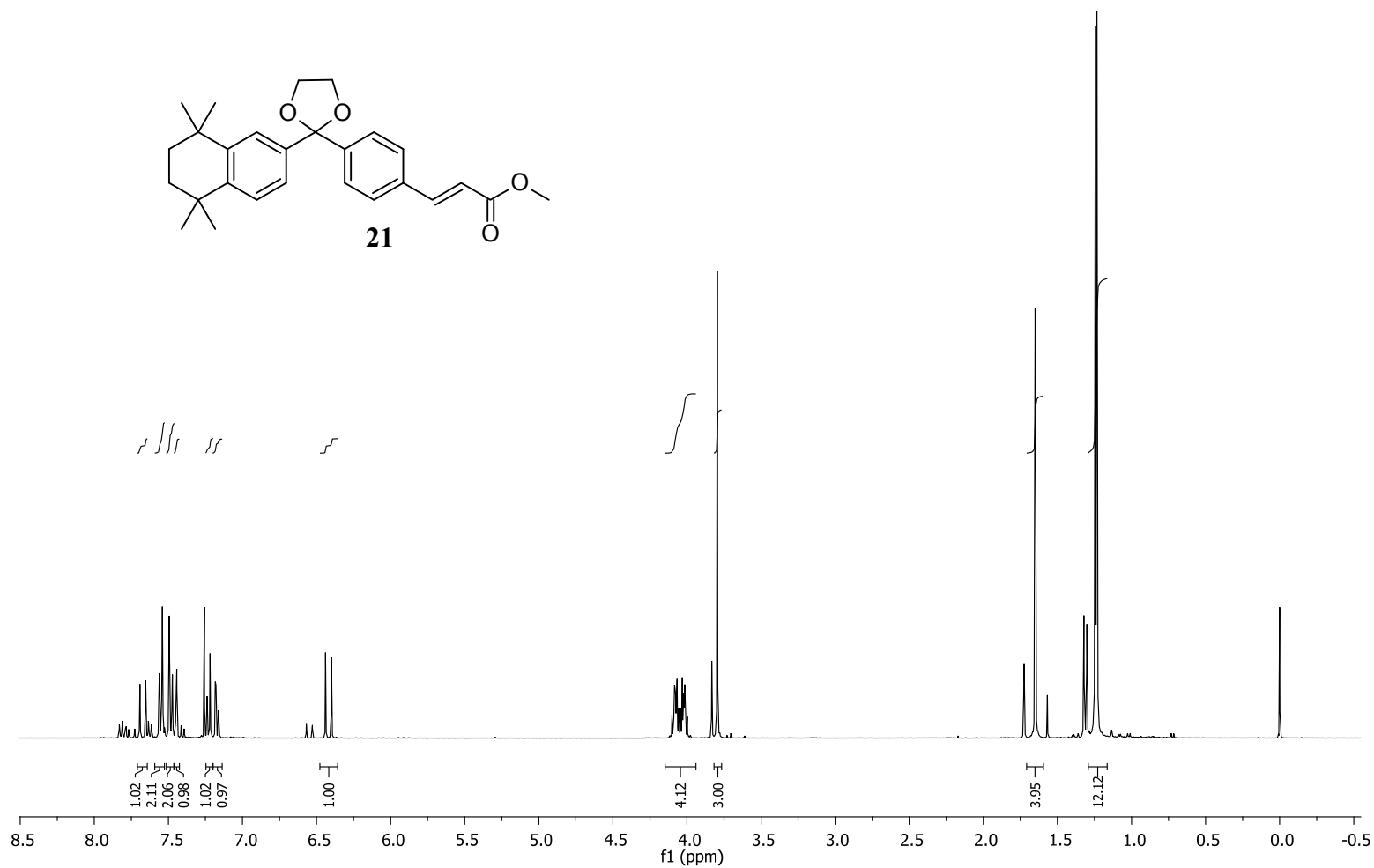
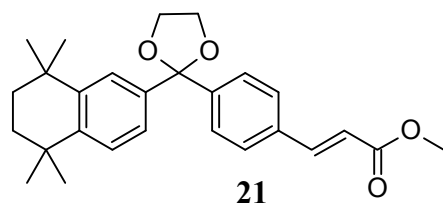
100 MHz ^{13}C -NMR of compound 20 in CDCl_3

CMK-02-106-C13



400 MHz $^1\text{H-NMR}$ of compound 21 in CDCl_3

CMK-02-115-proton



100 MHz ¹³C-NMR of compound 21 in CDCl₃

CMK-02-115-C13

167.56

144.94

144.92

144.77

144.62

138.51

134.09

128.06

126.86

126.60

123.91

123.47

118.03

109.42

77.48

77.16

76.84

65.02

51.84

35.22

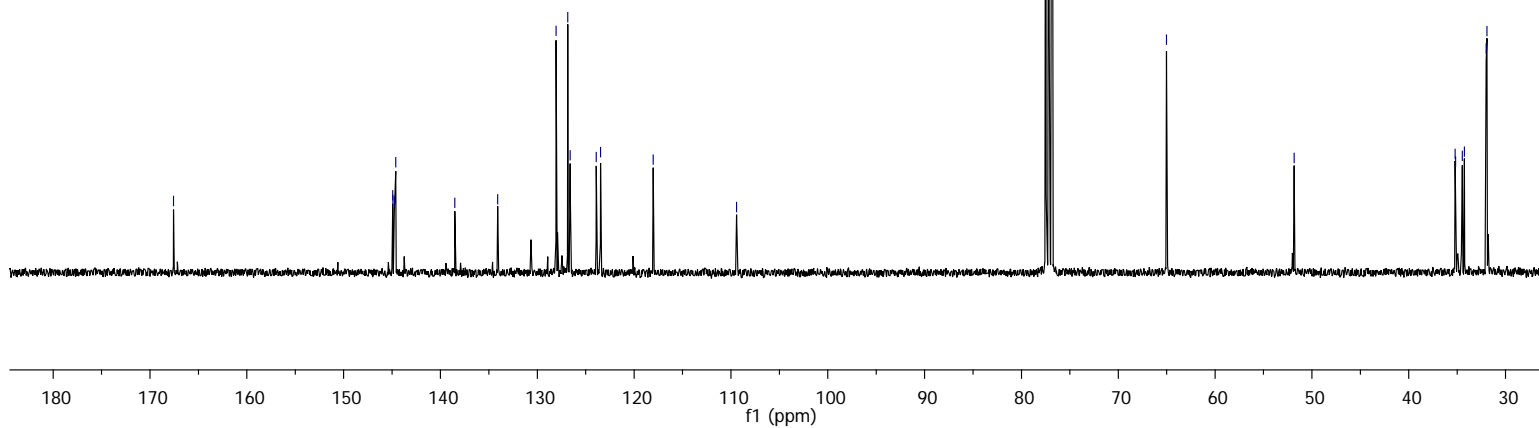
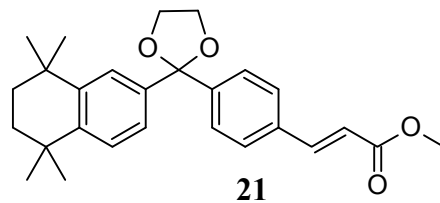
35.16

34.47

34.25

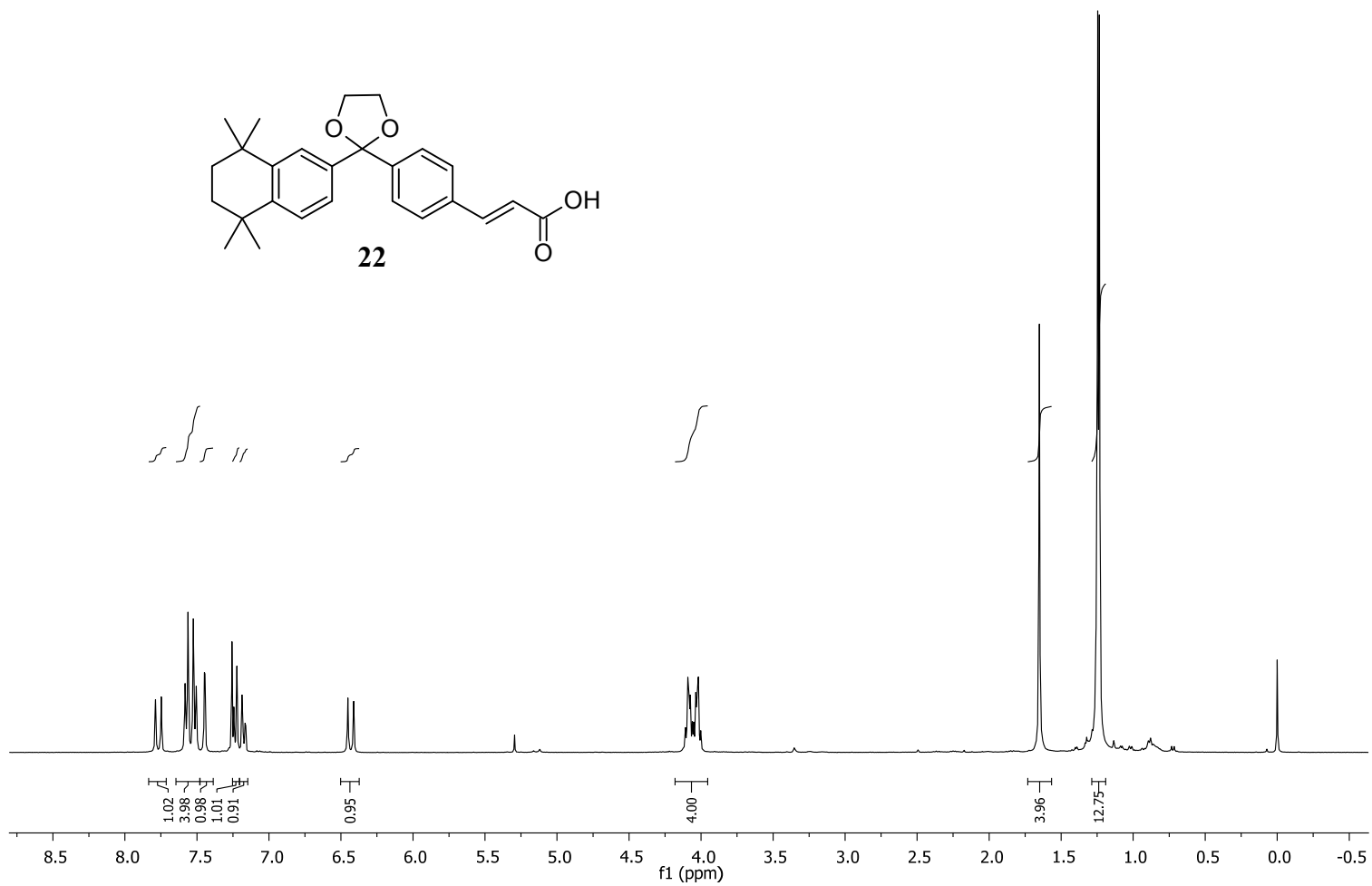
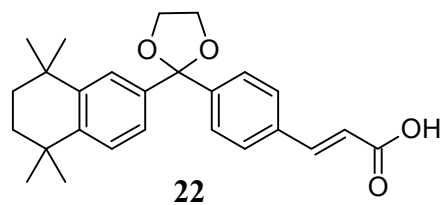
32.00

31.93



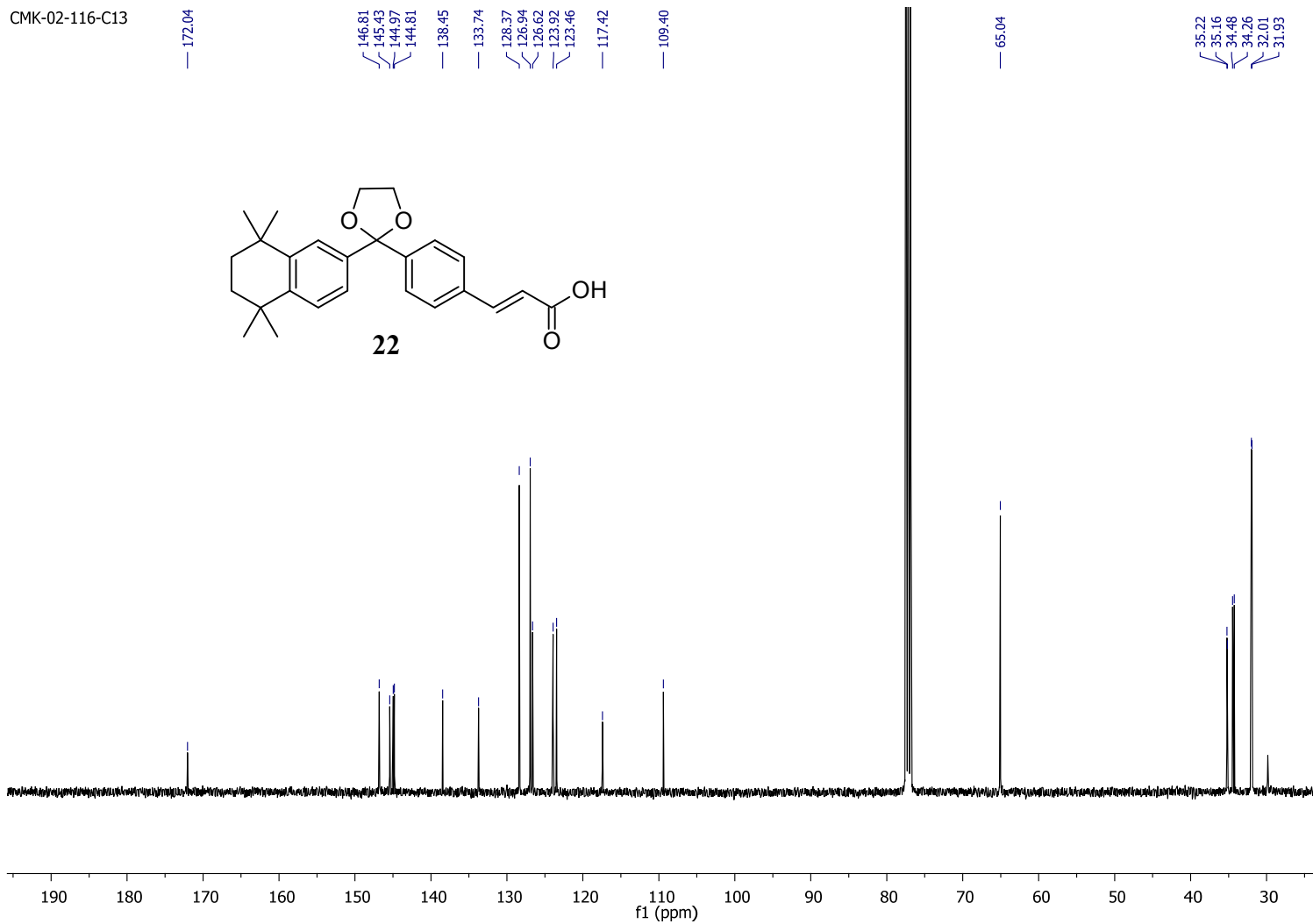
400 MHz $^1\text{H-NMR}$ of compound 22 in CDCl_3

CMK-02-116-proton

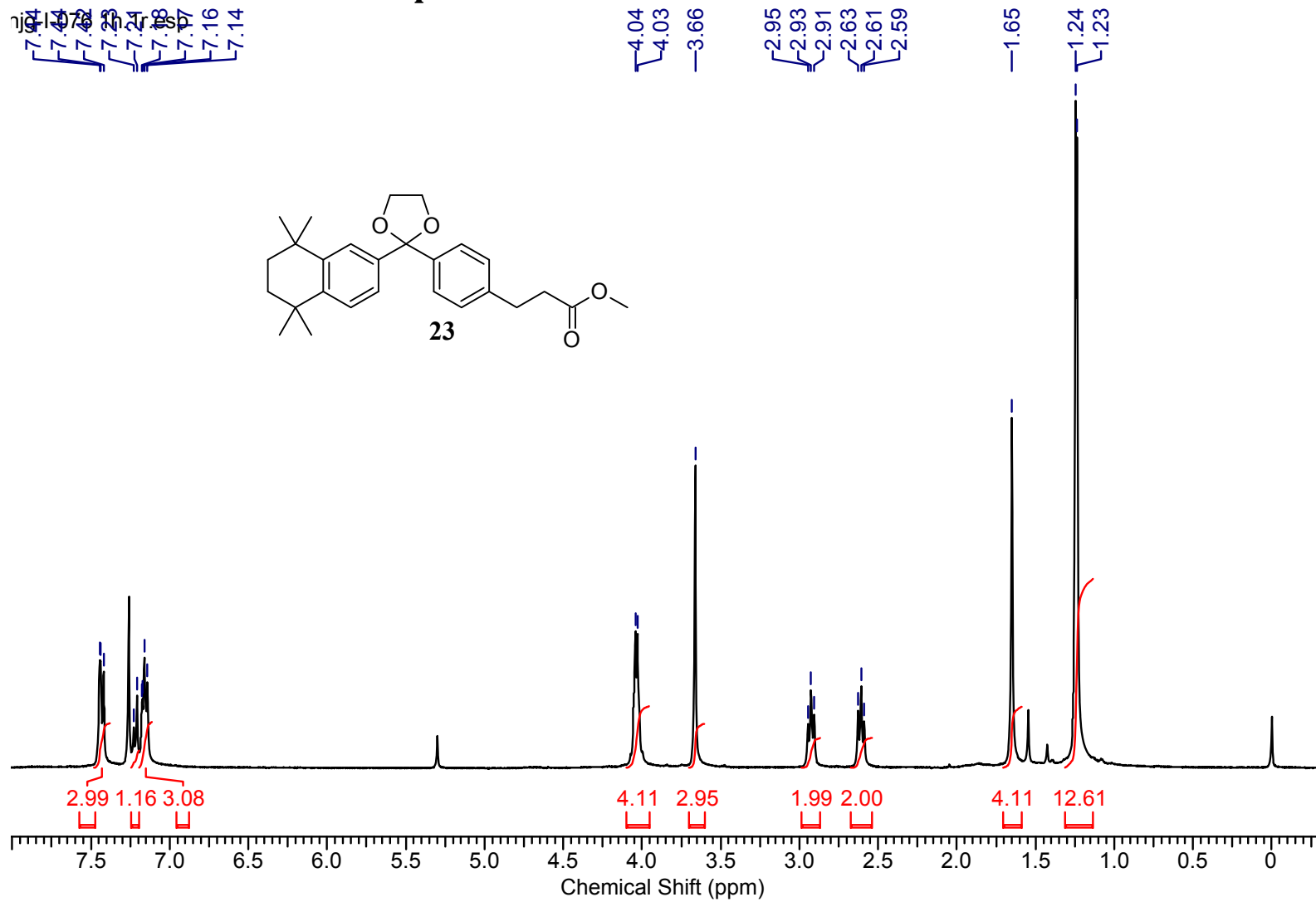


100 MHz ¹³C-NMR of compound 22 in CDCl₃

CMK-02-116-C13

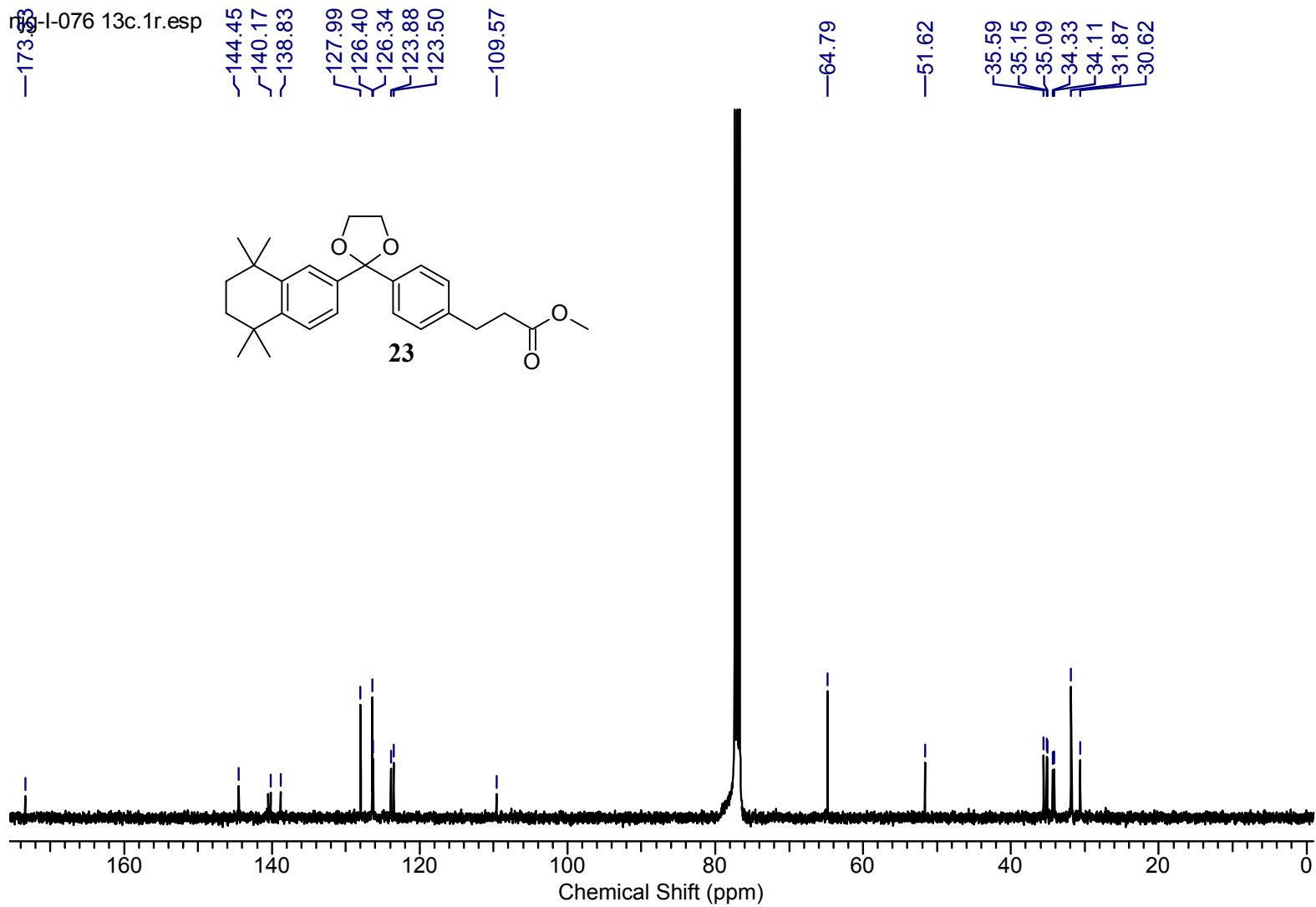


400 MHz ¹H-NMR of compound 23 in CDCl₃



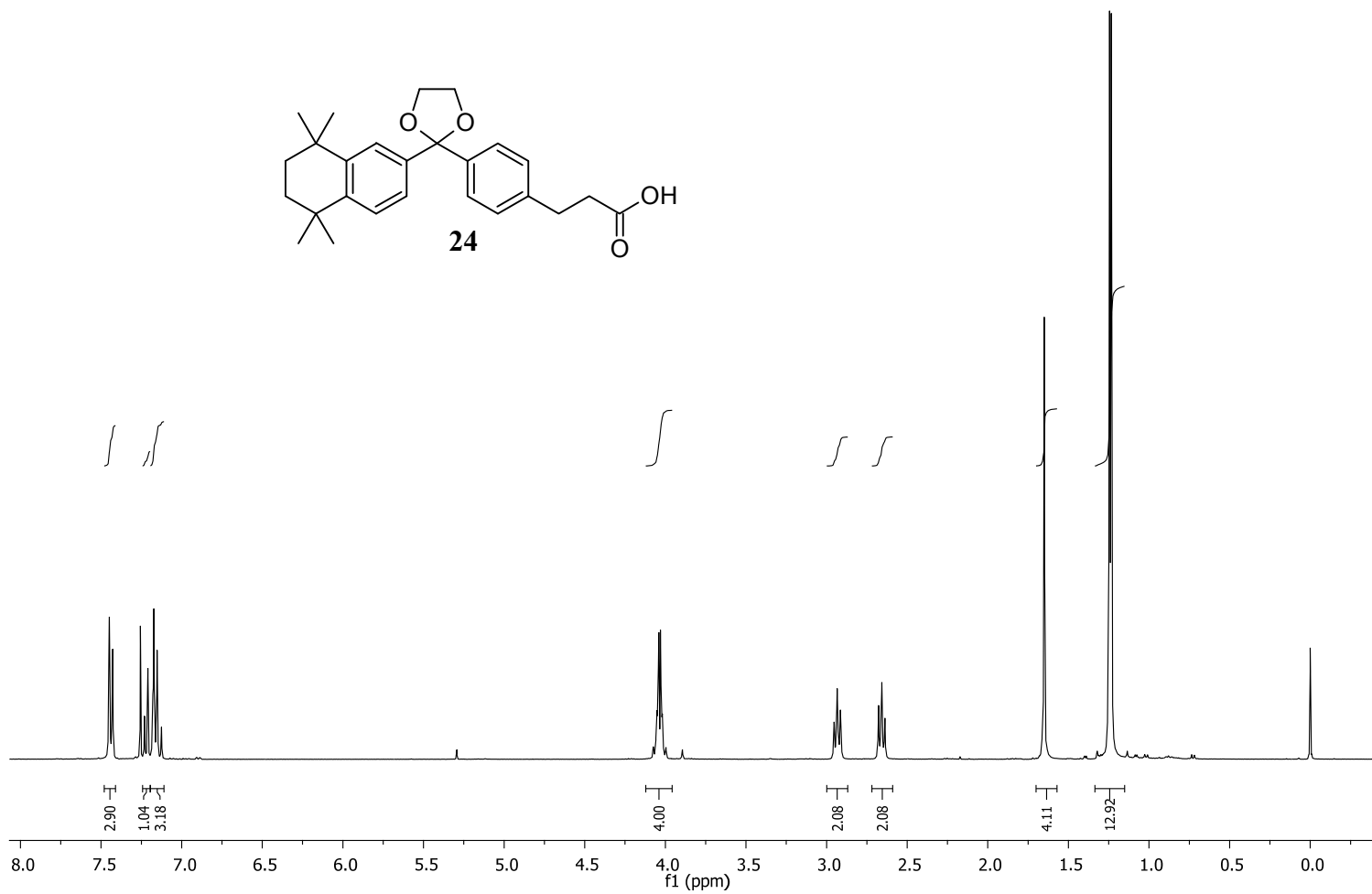
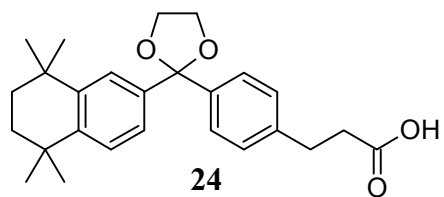
100 MHz ¹³C-NMR of compound 23 in CDCl₃

fig-I-076 13c.1r.esp



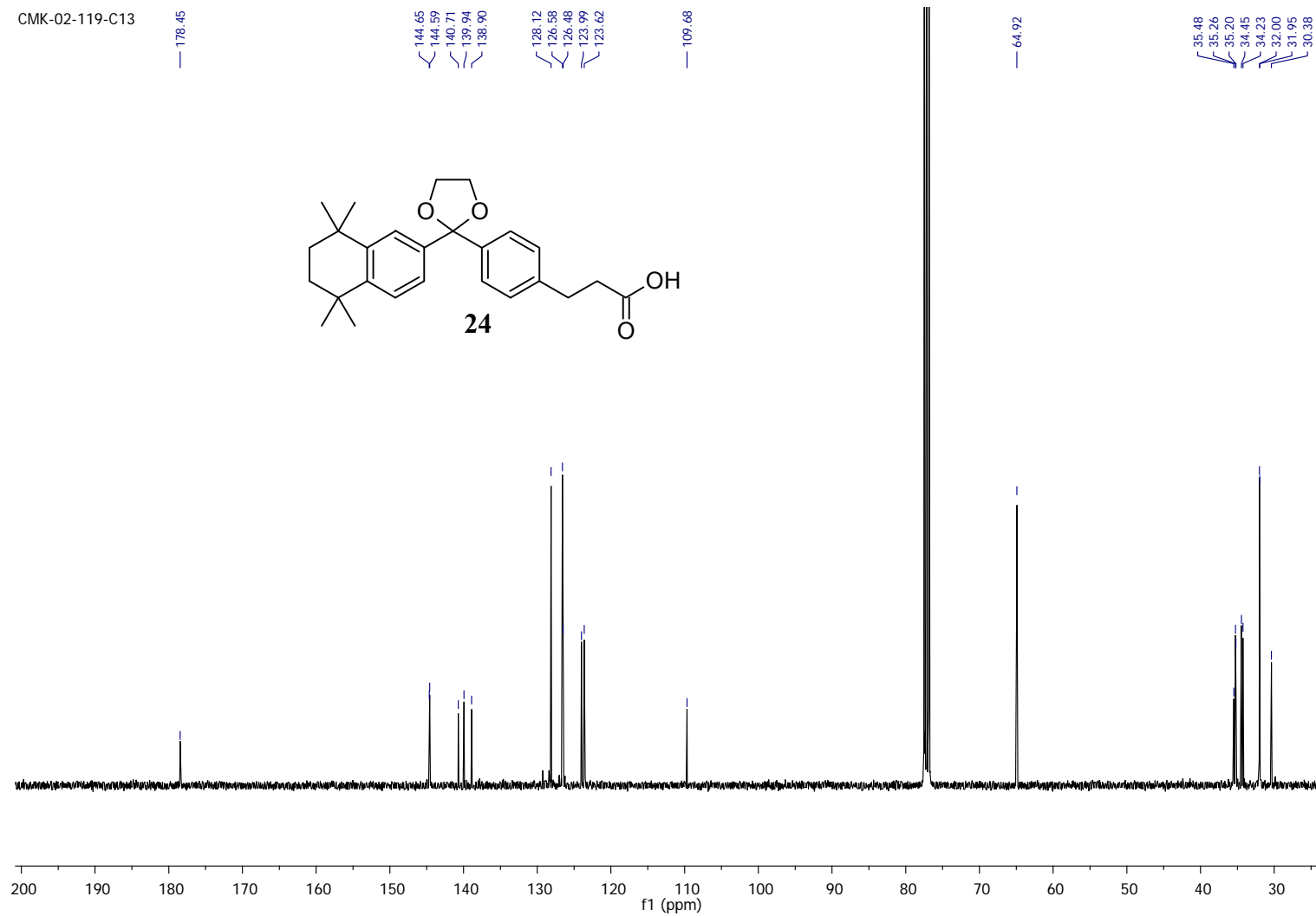
400 MHz ^1H -NMR of compound 24 in CDCl_3

CMK-02-119-proton

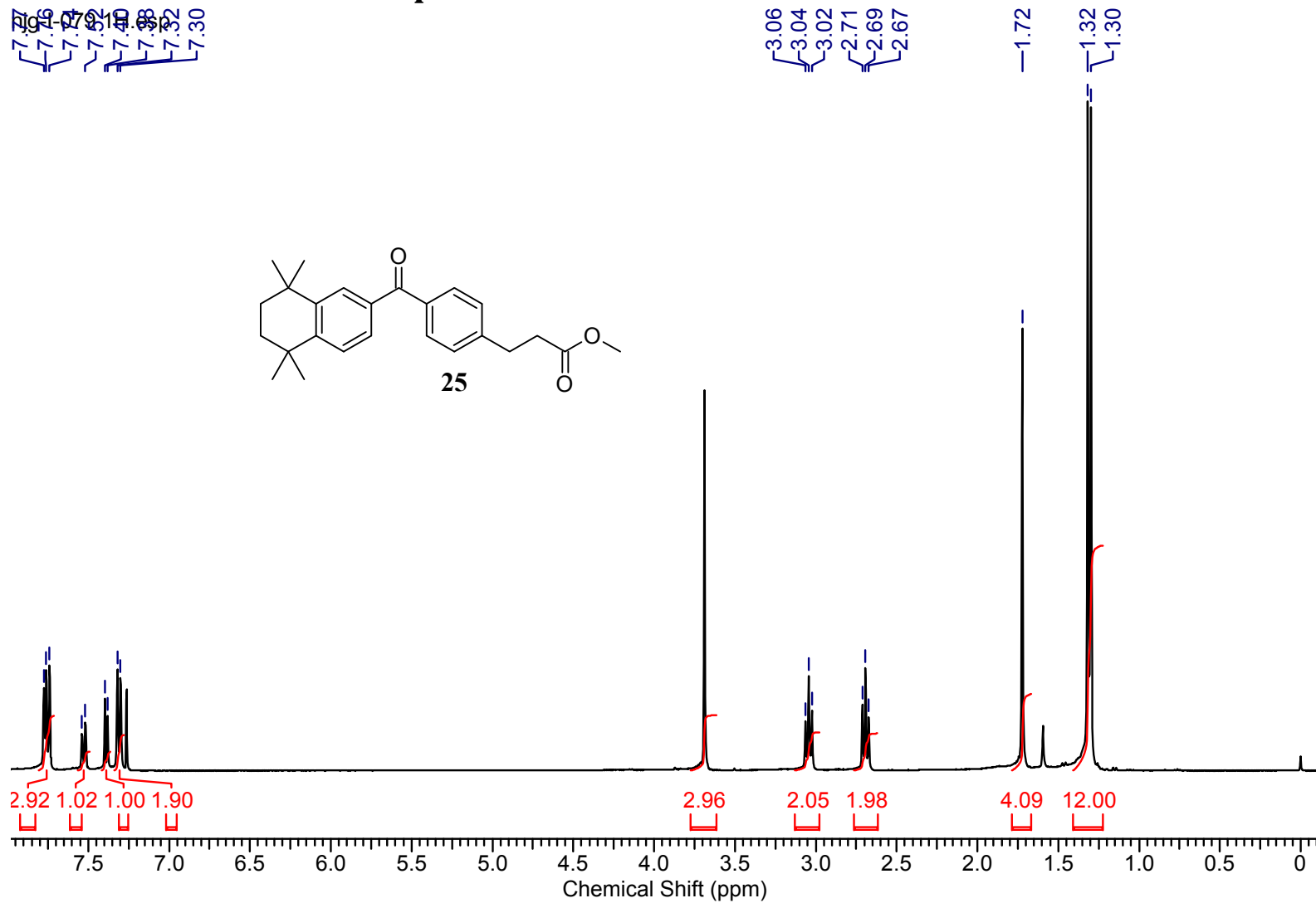


100 MHz ^{13}C -NMR of compound 24 in CDCl_3

CMK-02-119-C13

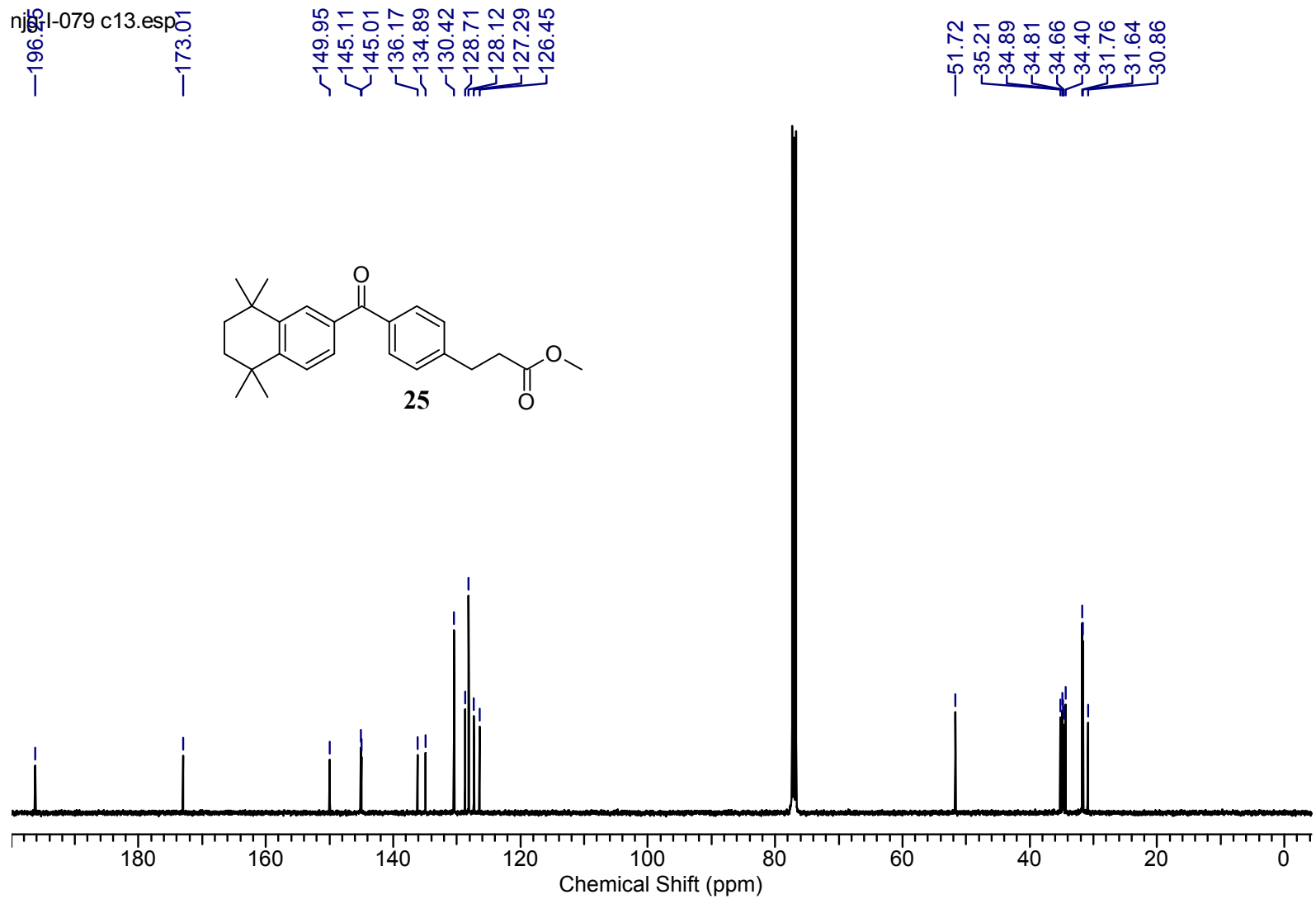


400 MHz ¹H-NMR of compound 25 in CDCl₃



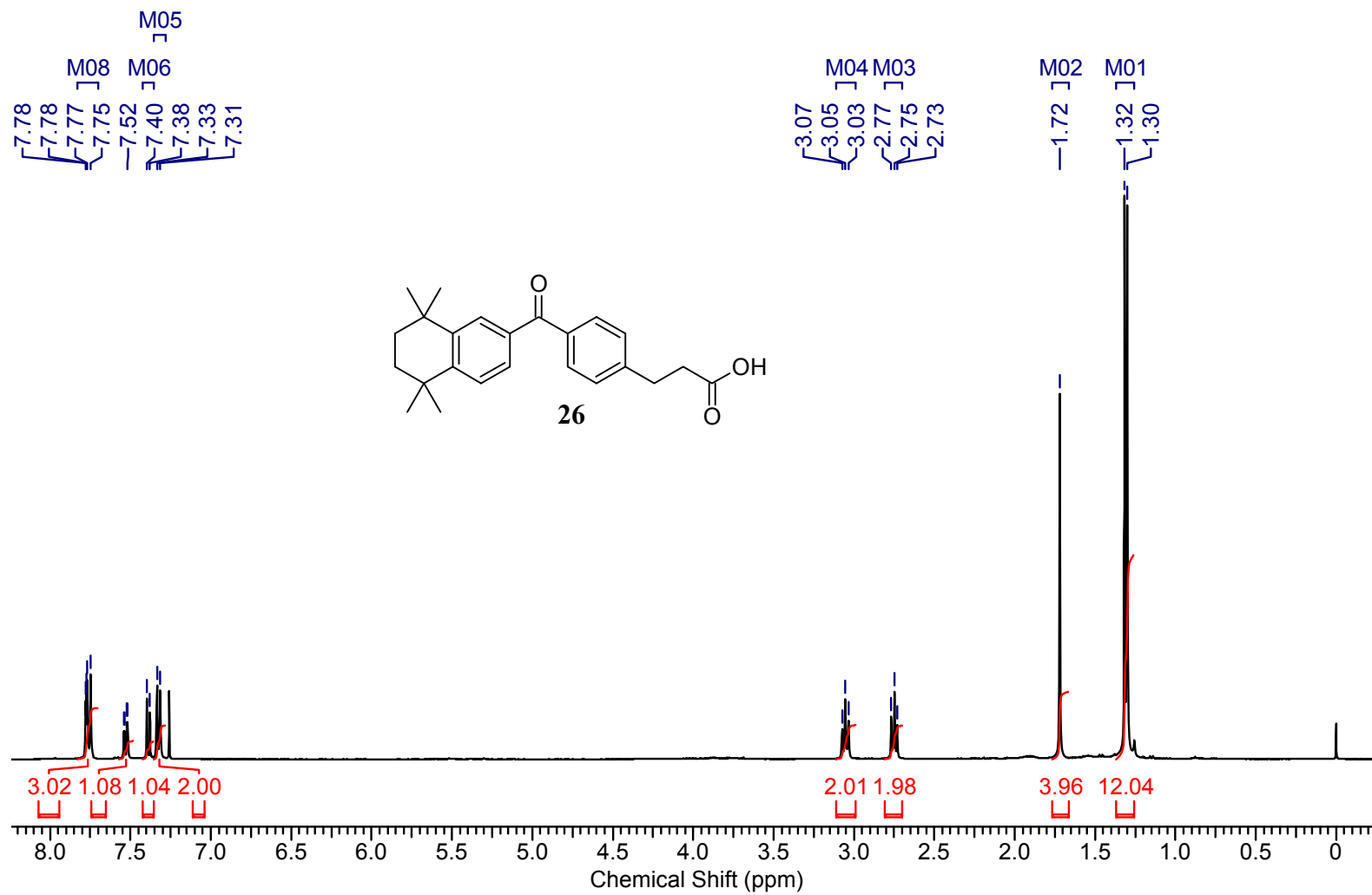
100 MHz ¹³C-NMR of compound 25 in CDCl₃

nj1-079 c13.esp



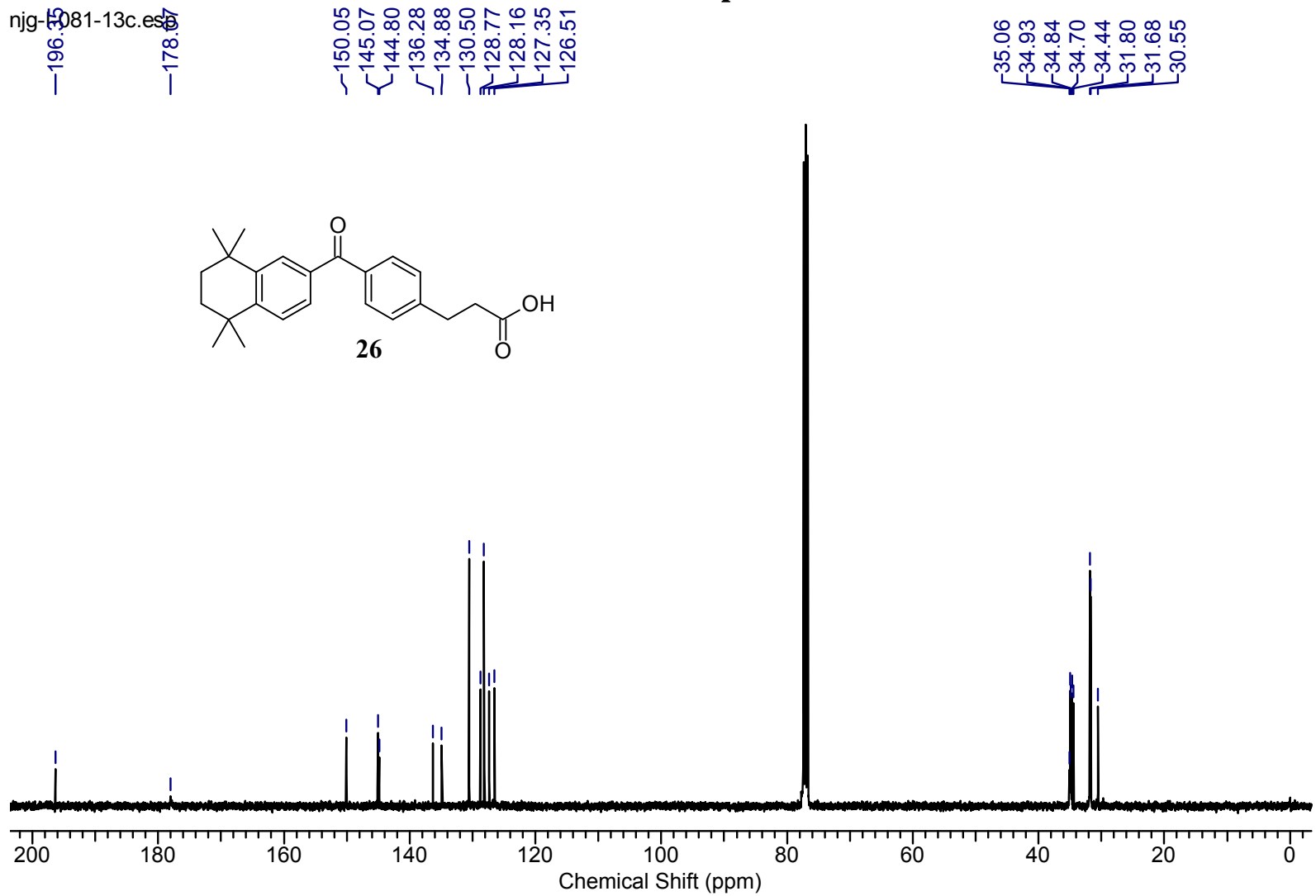
400 MHz ¹H-NMR of compound 26 in CDCl₃

njg-l-081-1071r.esp



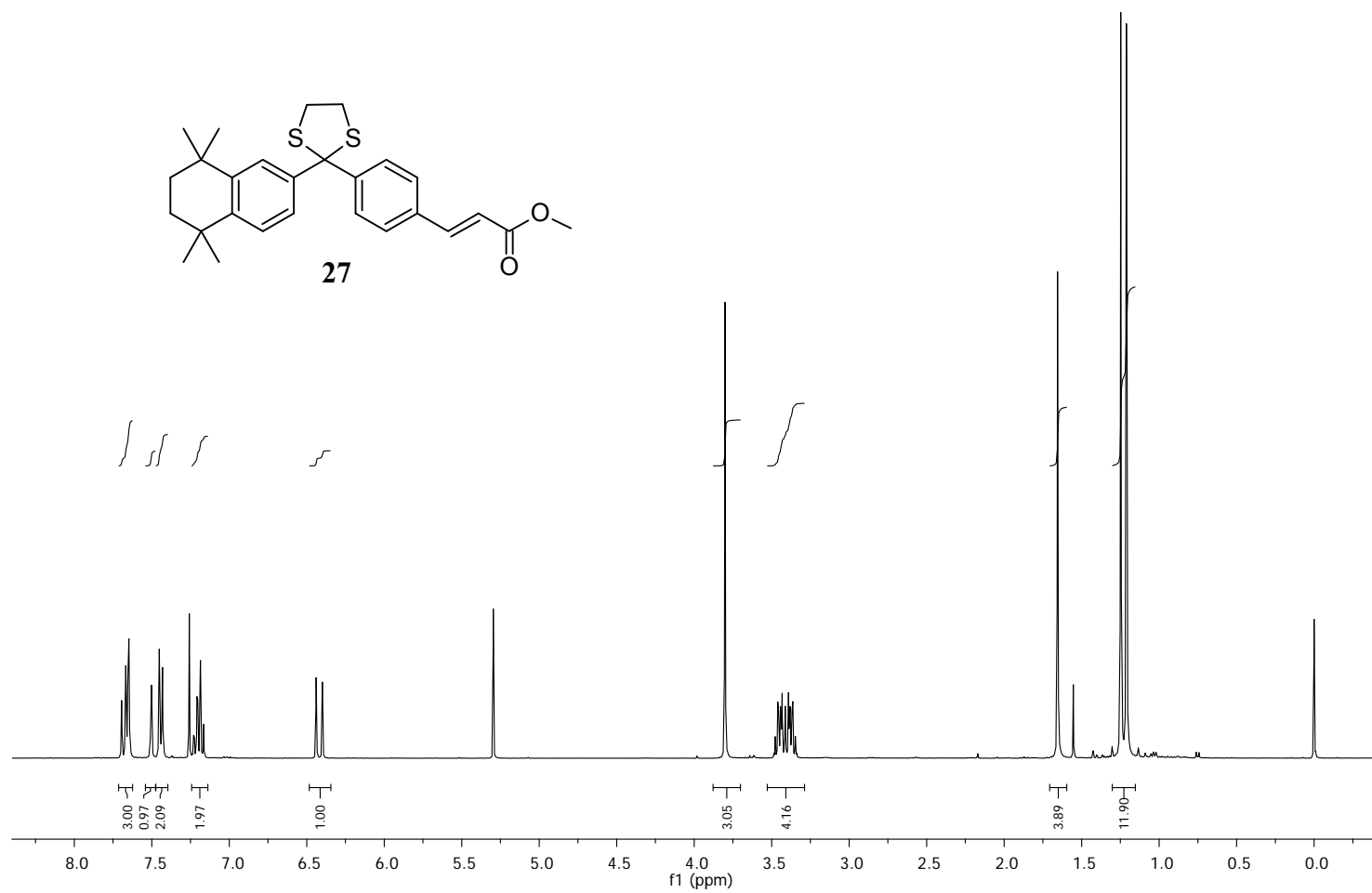
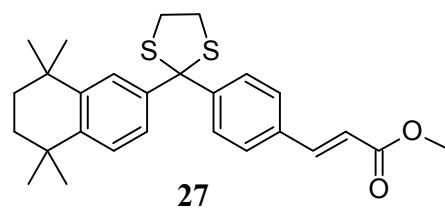
100 MHz ¹³C-NMR of compound 26 in CDCl₃

njg-081-13c.esf



400 MHz ^1H -NMR of compound 27 in CDCl_3

CMK-02-107-proton



100 MHz ¹³C-NMR of compound 27 in CDCl₃

CMK-02-107-C13

167.59

147.72

144.48

144.42

144.11

140.44

133.28

129.03

127.73

126.37

125.58

117.95

51.85

40.37

35.20

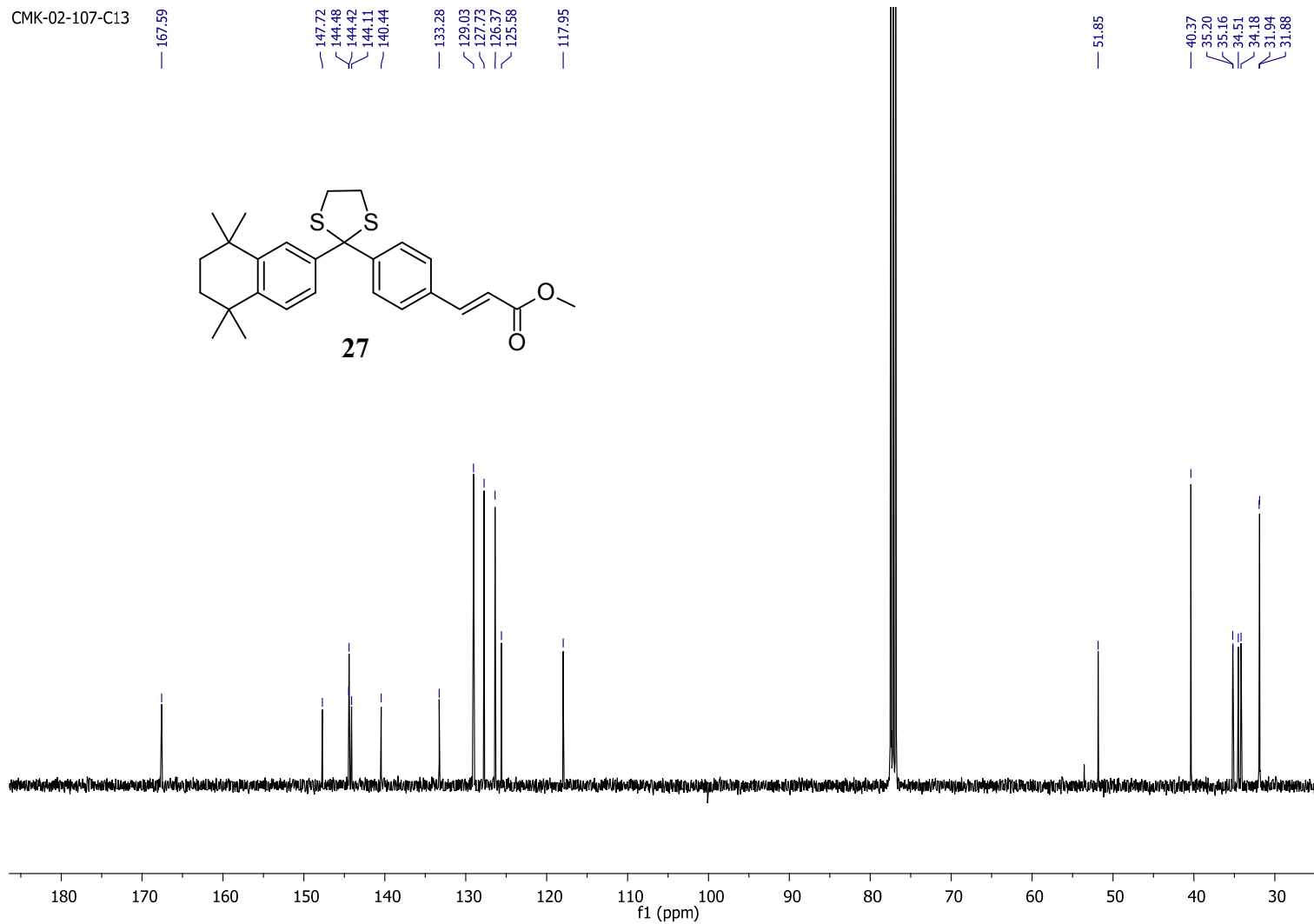
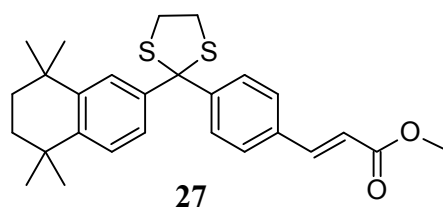
35.16

34.51

34.18

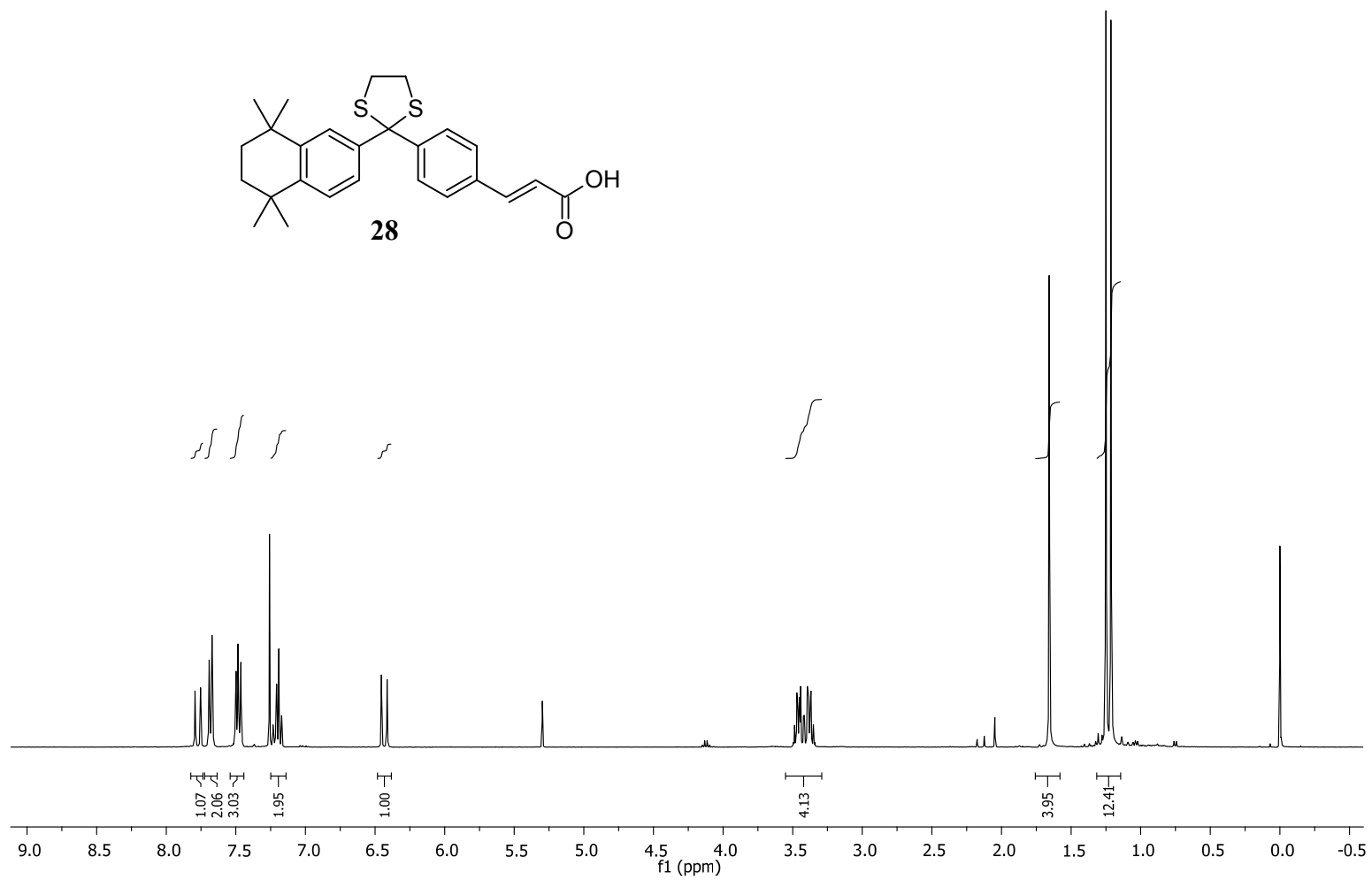
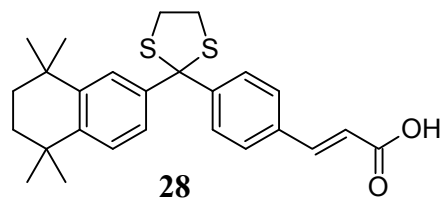
31.94

31.88



400 MHz $^1\text{H-NMR}$ of compound 28 in CDCl_3

CMK-02-108-proton



100 MHz ¹³C-NMR of compound 28 in CDCl₃

CMK-O2-108-C13

171.89

148.29

146.63

144.52

144.16

140.37

132.92

129.11

128.04

126.40

126.38

125.57

117.27

40.40

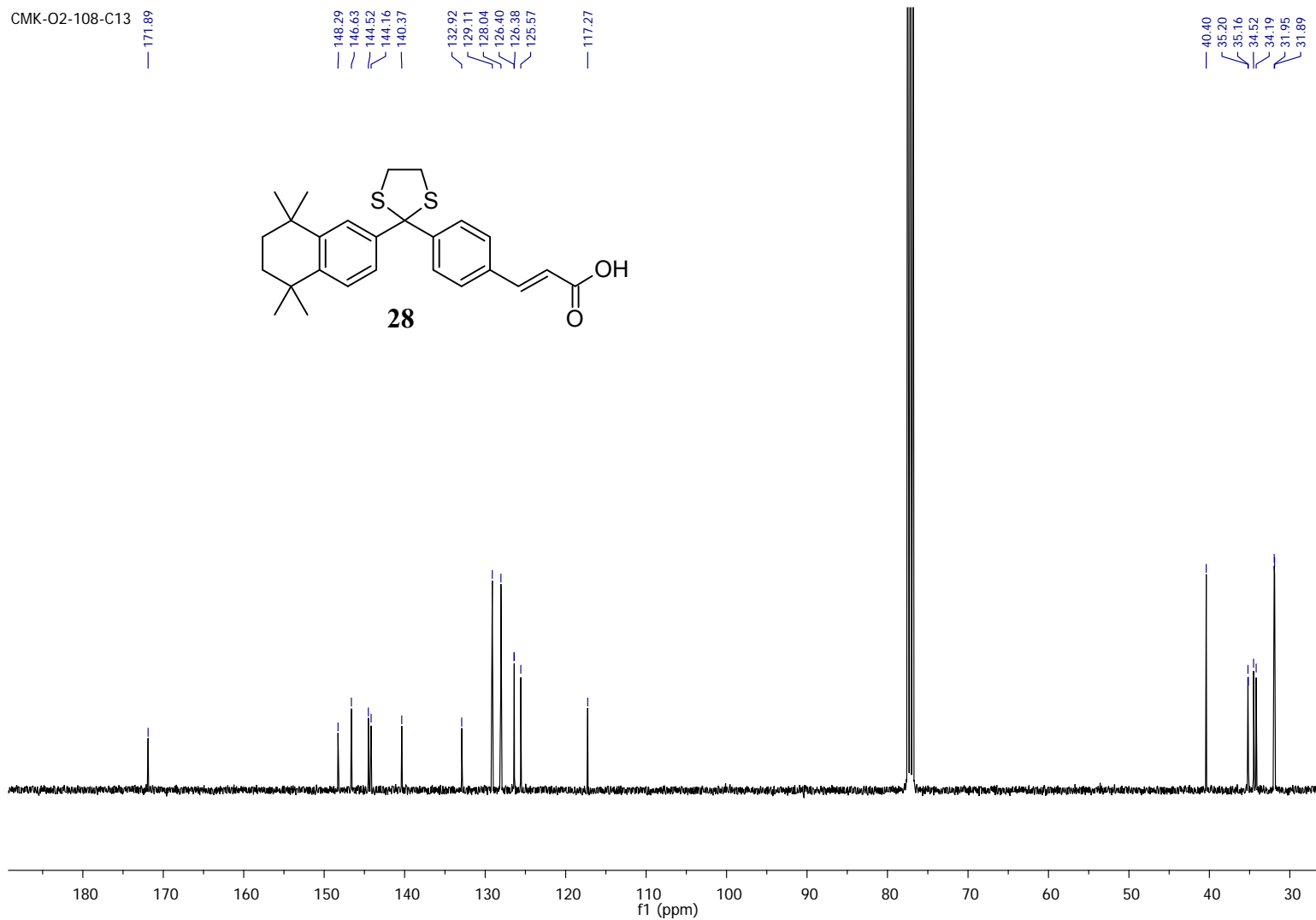
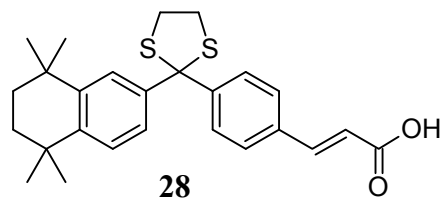
35.20

35.16

34.52

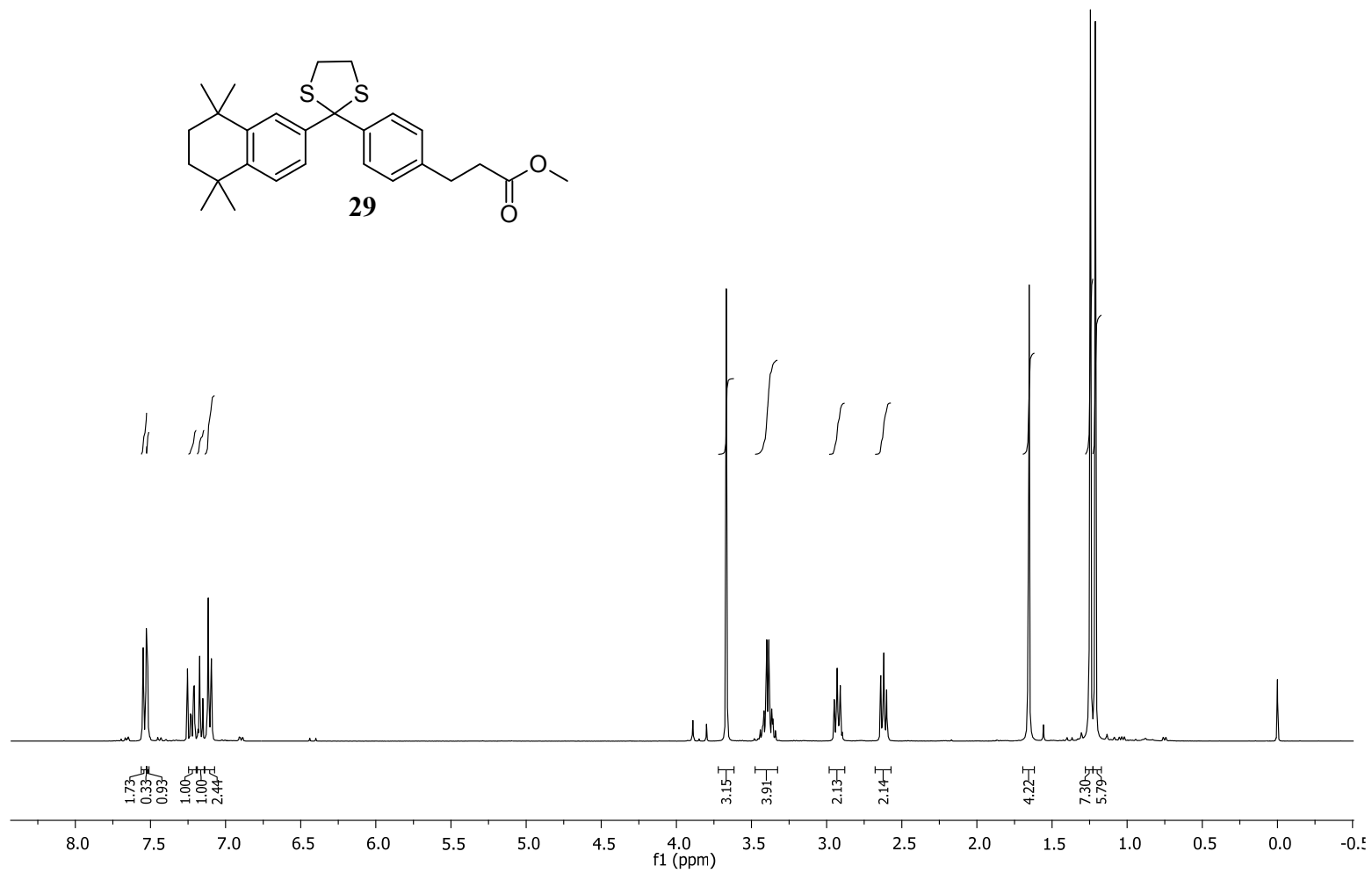
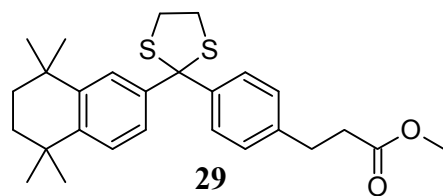
34.19

31.95



400 MHz $^1\text{H-NMR}$ of compound 29 in CDCl_3

CMK-02-120-proton



100 MHz ¹³C-NMR of compound 29 in CDCl₃

CMK-02-120-C13

173.47

144.19
143.77
142.99
141.05
139.45

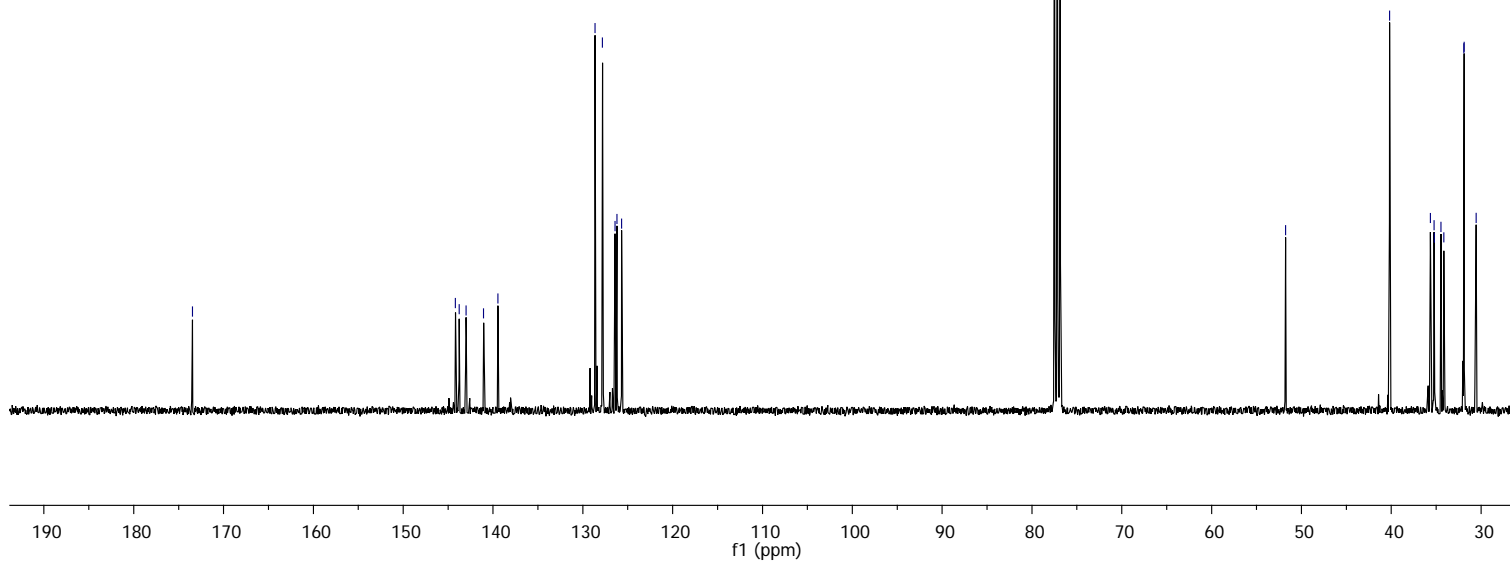
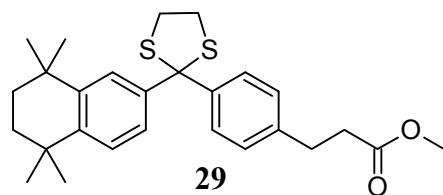
128.64
127.82
126.41
126.20
125.68

77.48
77.16
76.84

51.76

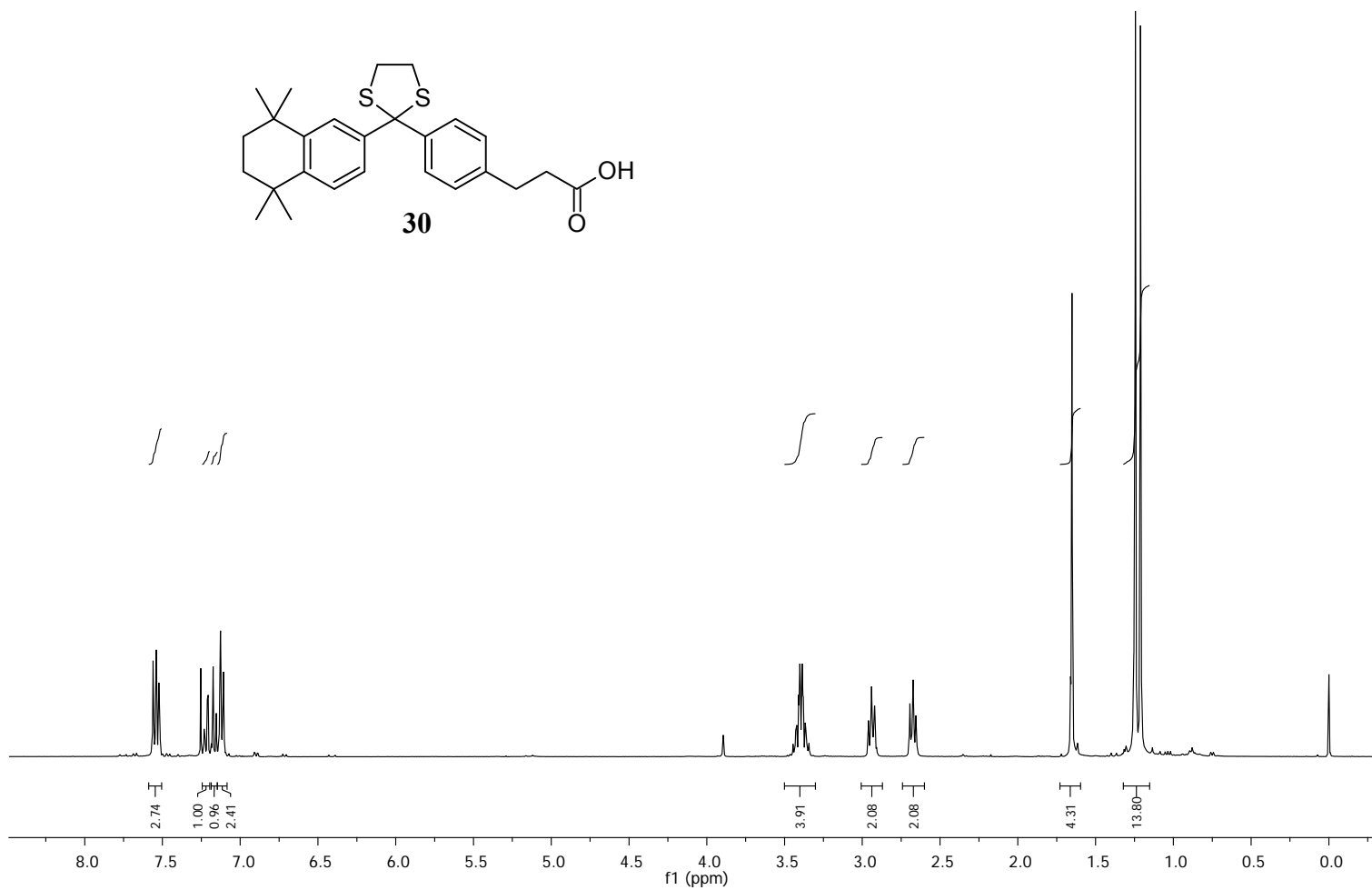
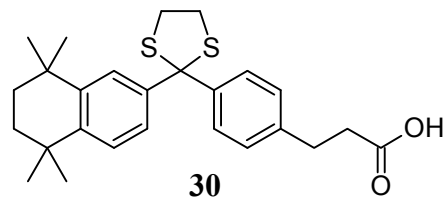
40.19

35.63
35.23
34.47
31.92
31.89
30.54



400 MHz $^1\text{H-NMR}$ of compound 30 in CDCl_3

CMK-02-124B-proton



100 MHz ¹³C-NMR of compound 30 in CDCl₃

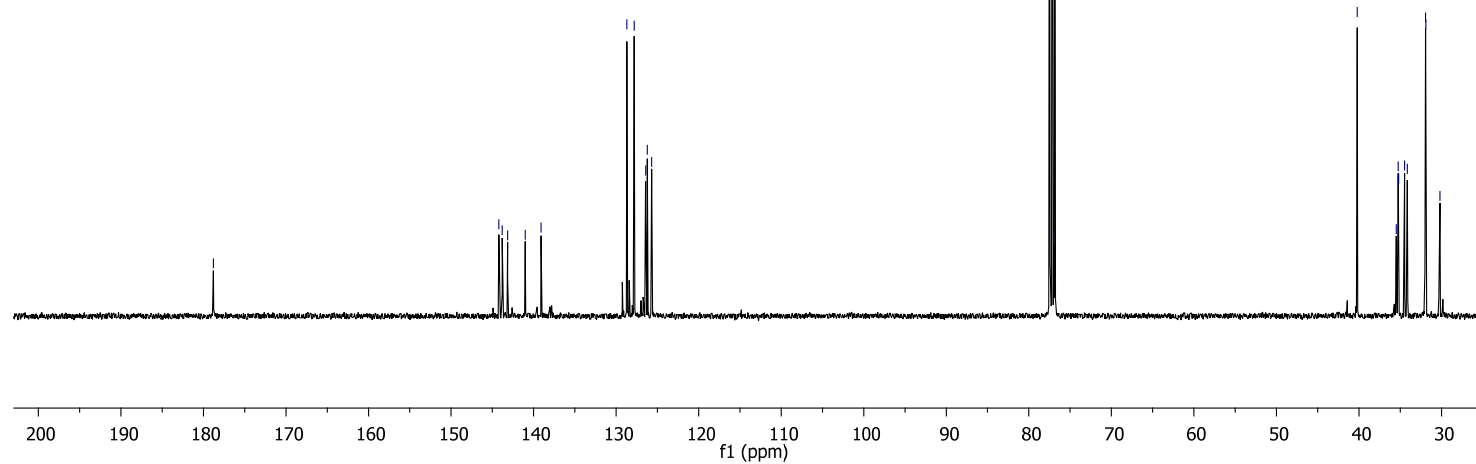
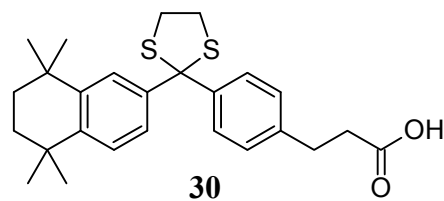
CMK-02-124B-C13

178.79

144.21
143.80
143.15
141.01
139.10
128.71
127.81
126.43
126.22
125.69

77.48
77.16
77.00
76.84

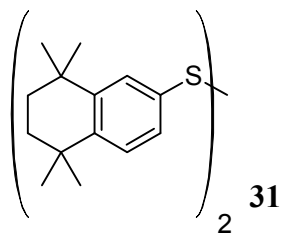
40.20
35.24
35.20
34.48
34.15
31.93
31.89



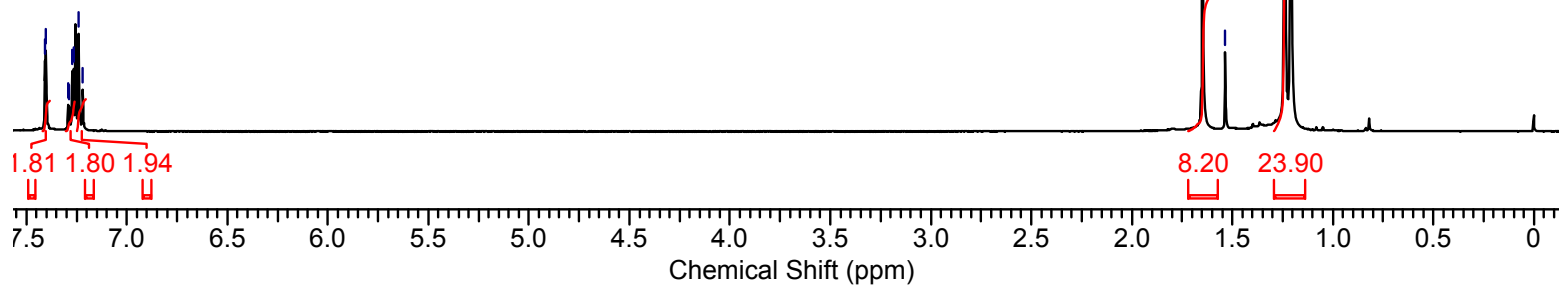
400 MHz ¹H-NMR of compound 31 in CDCl₃

njg-M05-1 1h.esp

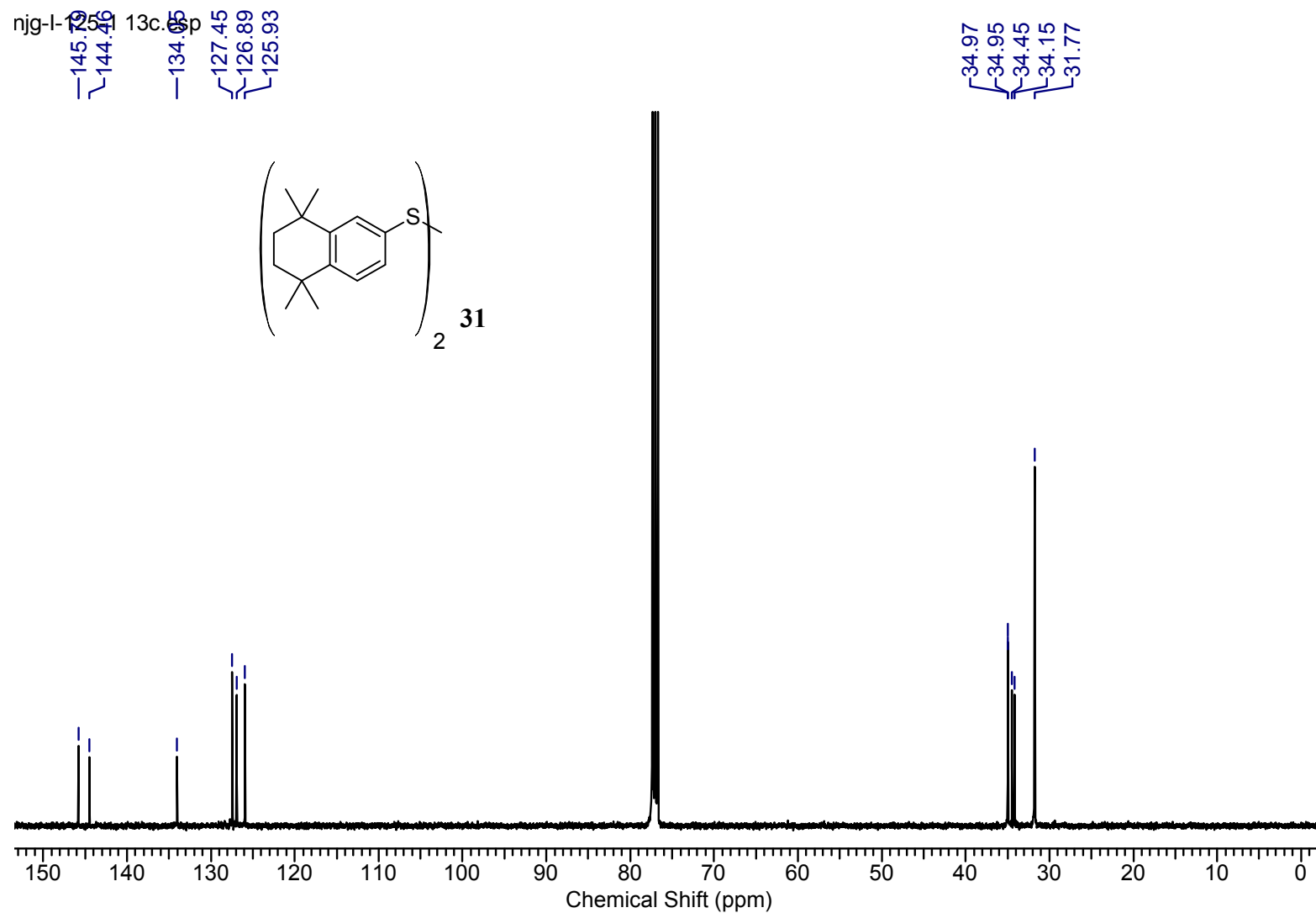
M05
M04
M05
7.41
7.40
7.27
7.27
7.24
7.22



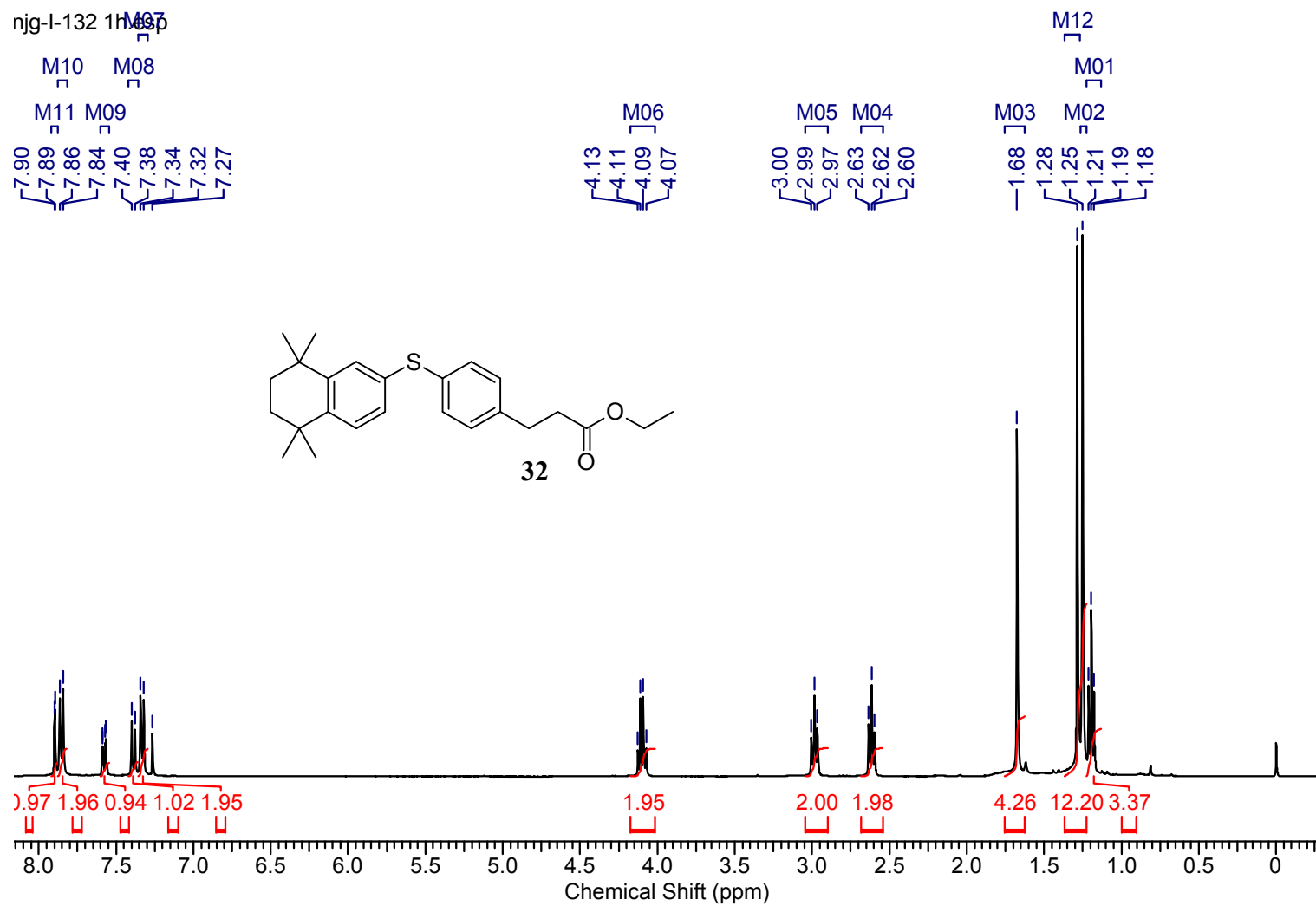
M02
M06
M01
1.65
1.53
1.24
1.21



100 MHz ^{13}C -NMR of compound 31 in CDCl_3



400 MHz ¹H-NMR of compound 32 in CDCl₃



100 MHz ¹³C-NMR of compound 32 in CDCl₃

njg-l-32 13c.esp

172.28

150.80

146.45

146.21

140.05

138.55

129.18

127.81

127.77

125.89

124.49

60.63

35.14

34.66

34.61

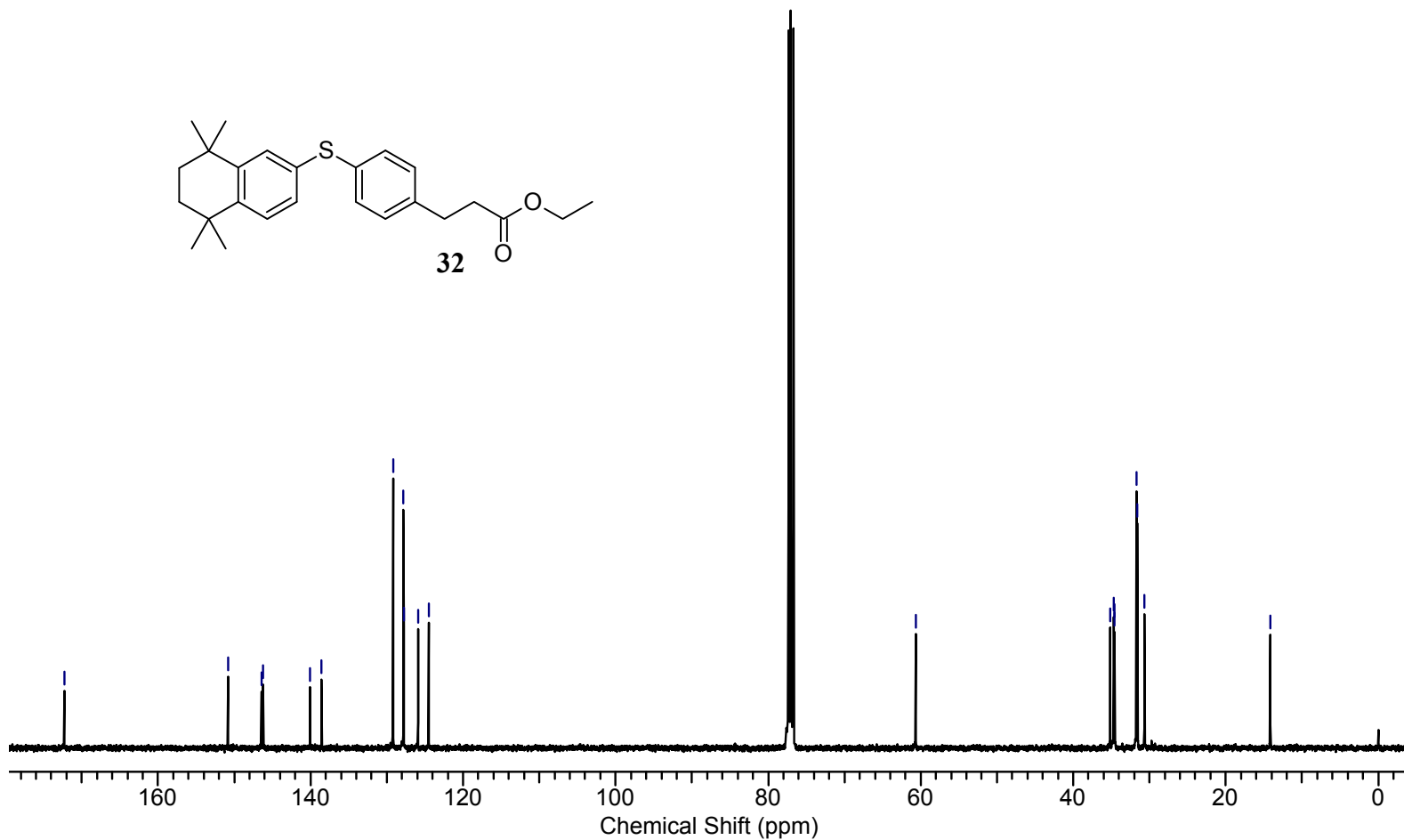
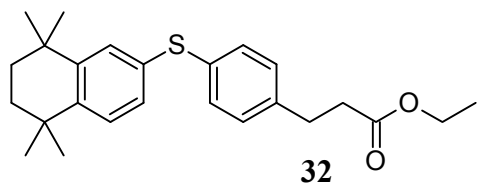
34.55

31.70

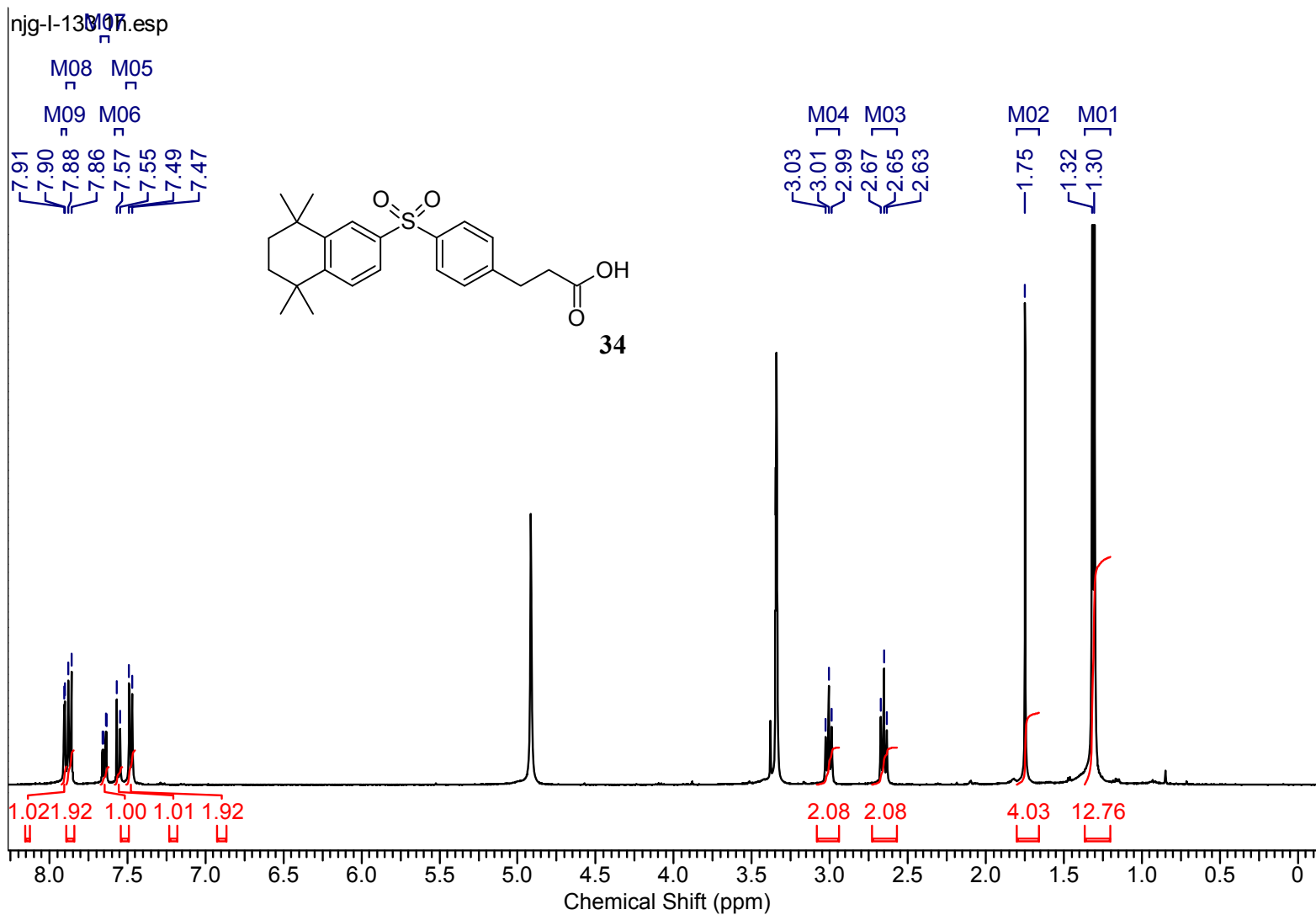
31.57

30.68

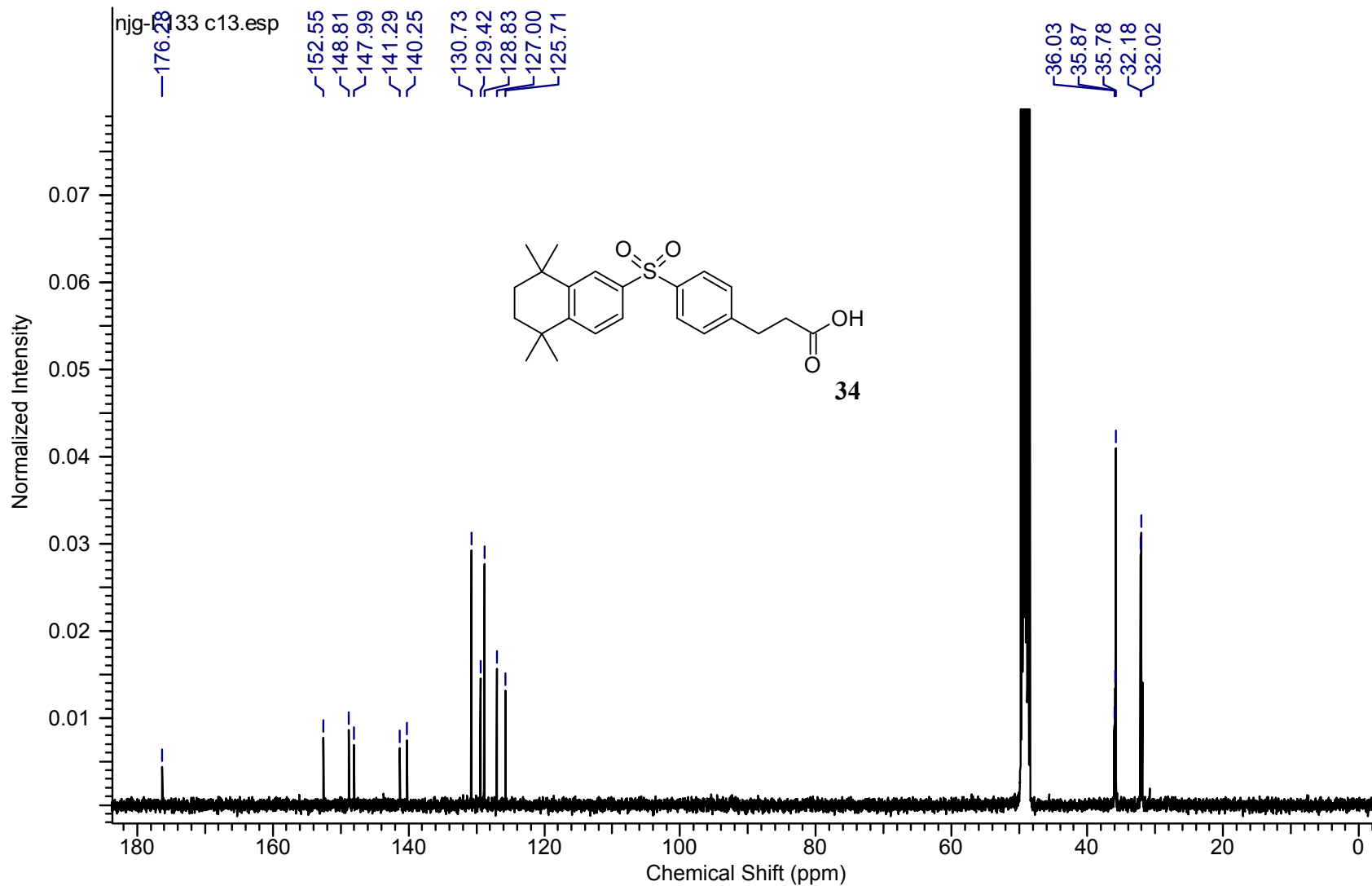
14.15



400 MHz ¹H-NMR of compound 34 in CDCl₃



100 MHz ¹³C-NMR of compound 34 in CDCl₃



400 MHz ¹H-NMR of compound 36a in CDCl₃

1jg-1006 1h.esp

M08 M05

M07

7.35
7.34
7.26
7.25
7.23
7.20

M04

M03

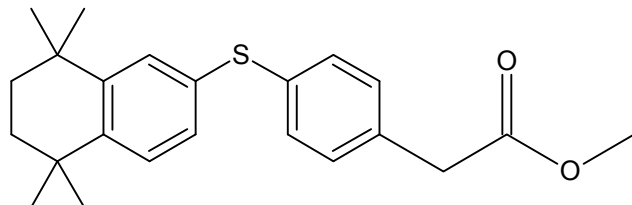
3.69
3.59

M02

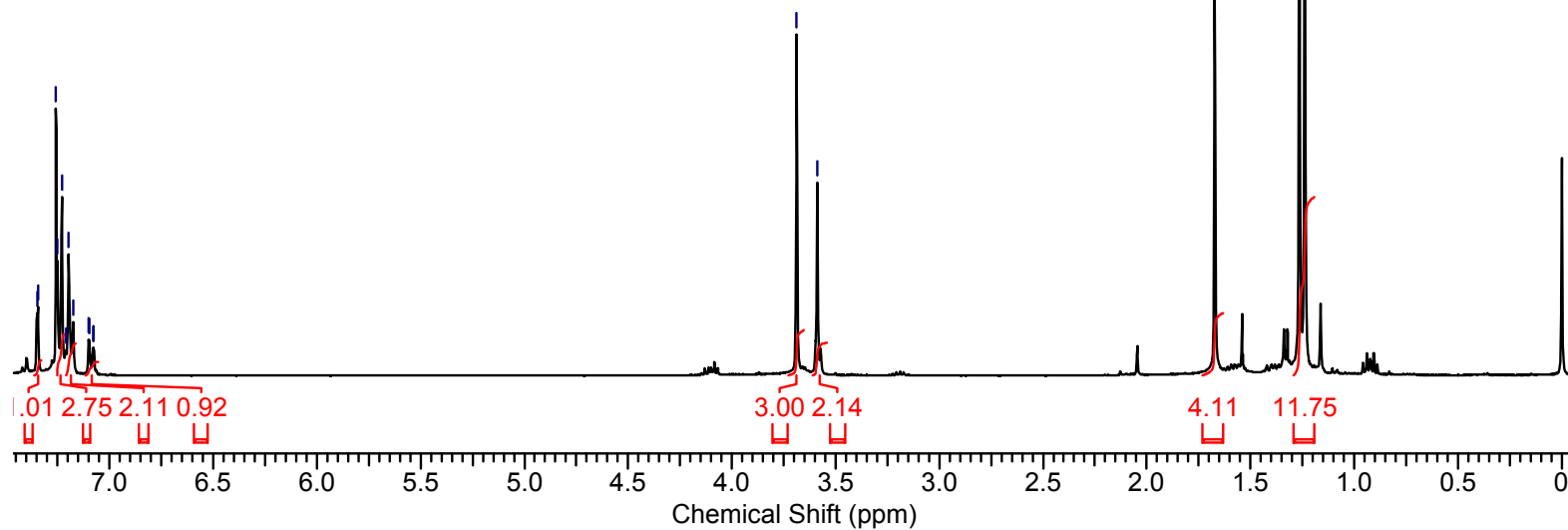
M01

M09

1.67
1.26
1.24

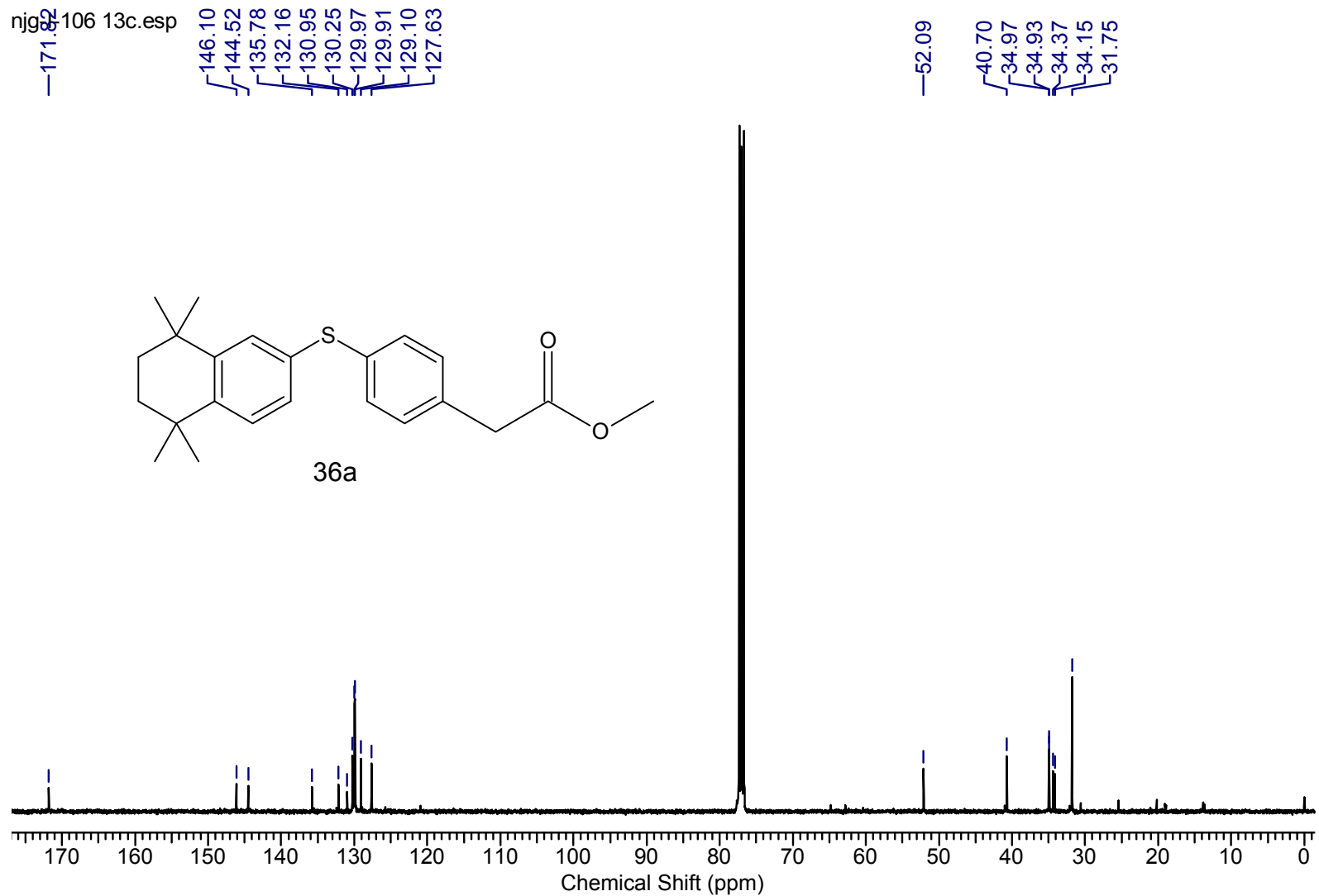


36a

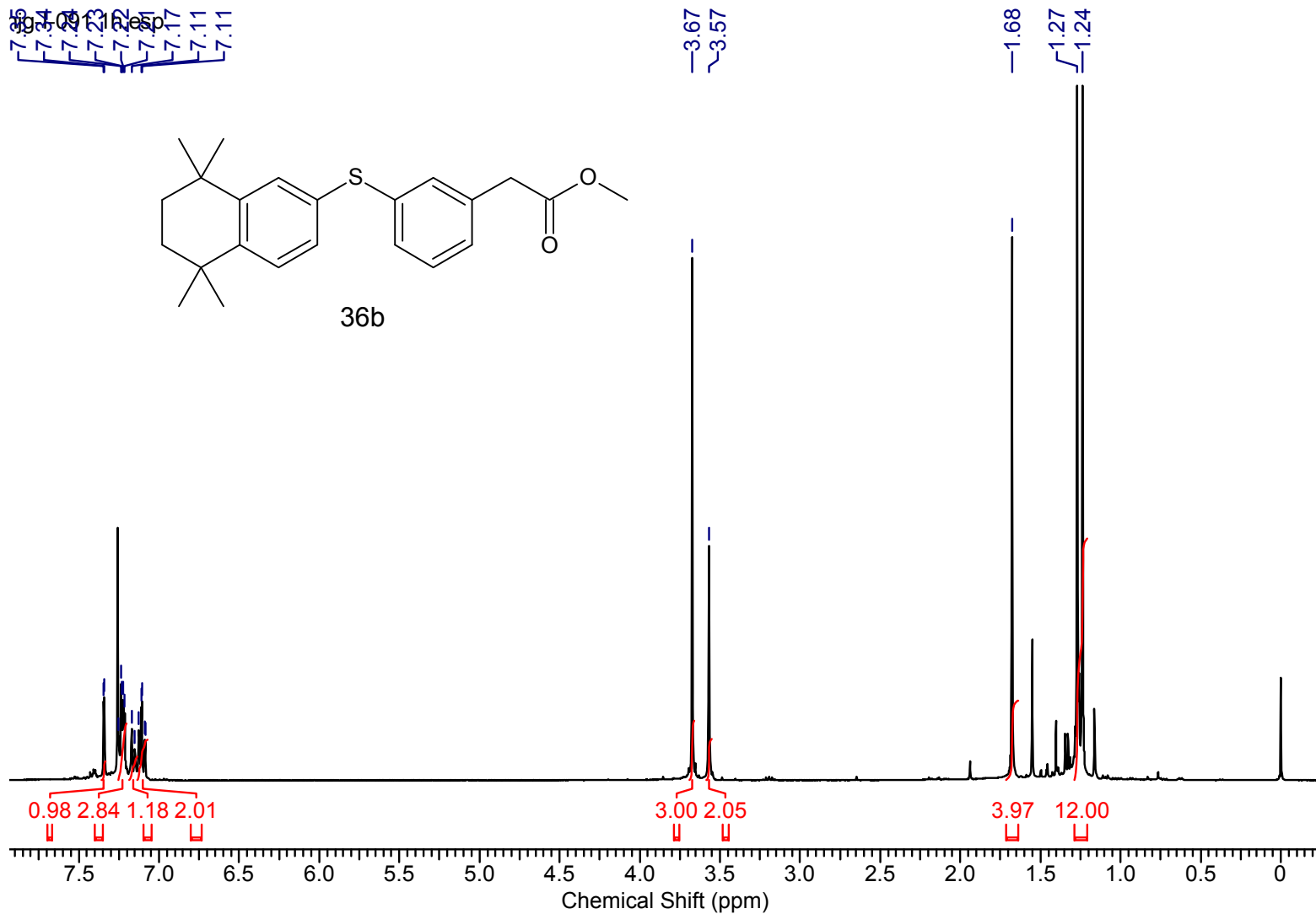


100 MHz ¹³C-NMR of compound 36a in CDCl₃

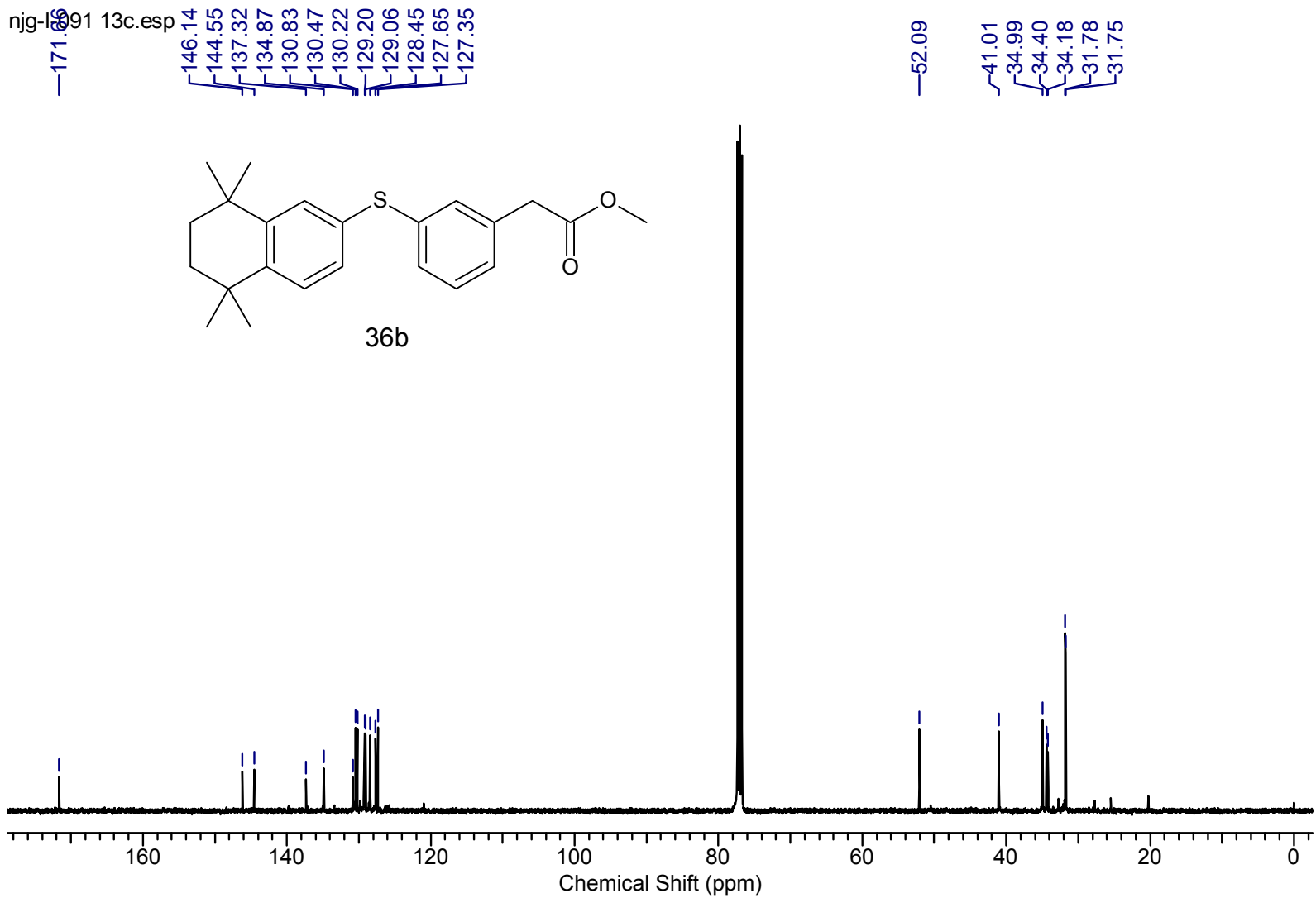
njg-106 13c.esp



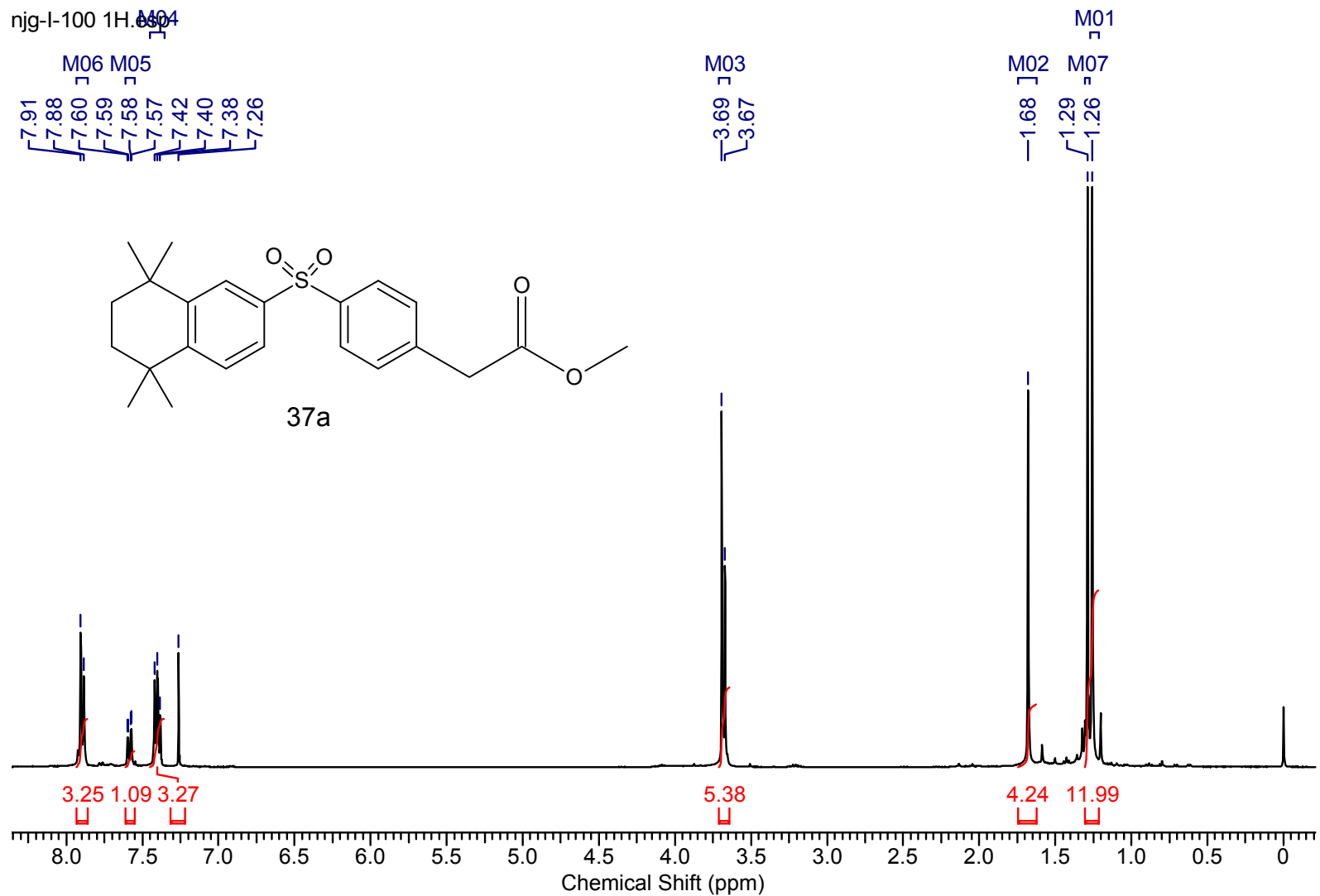
400 MHz $^1\text{H-NMR}$ of compound 36b in CDCl_3



100 MHz ¹³C-NMR of compound 36b in CDCl₃



400 MHz ¹H-NMR of compound 37a in CDCl₃



100 MHz ¹³C-NMR of compound 37a in CDCl₃

njg-l-10013c.esp

170.99

150.99

146.54

141.05

139.24

138.36

130.29

130.19

127.88

126.00

124.61

52.32

40.84

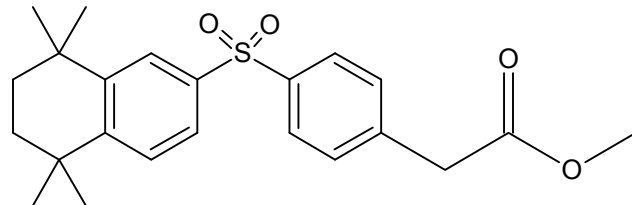
34.69

34.63

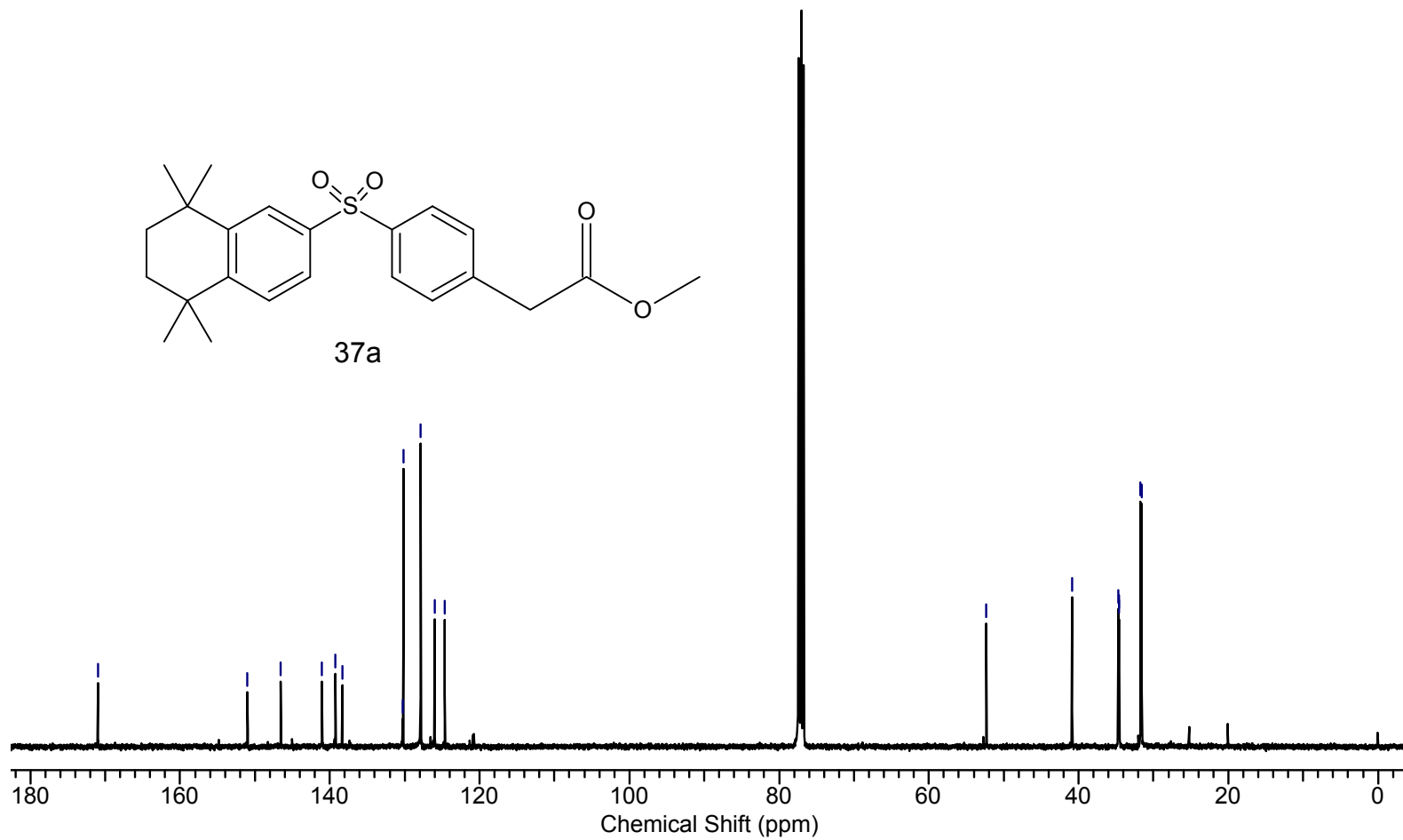
34.58

31.72

31.59

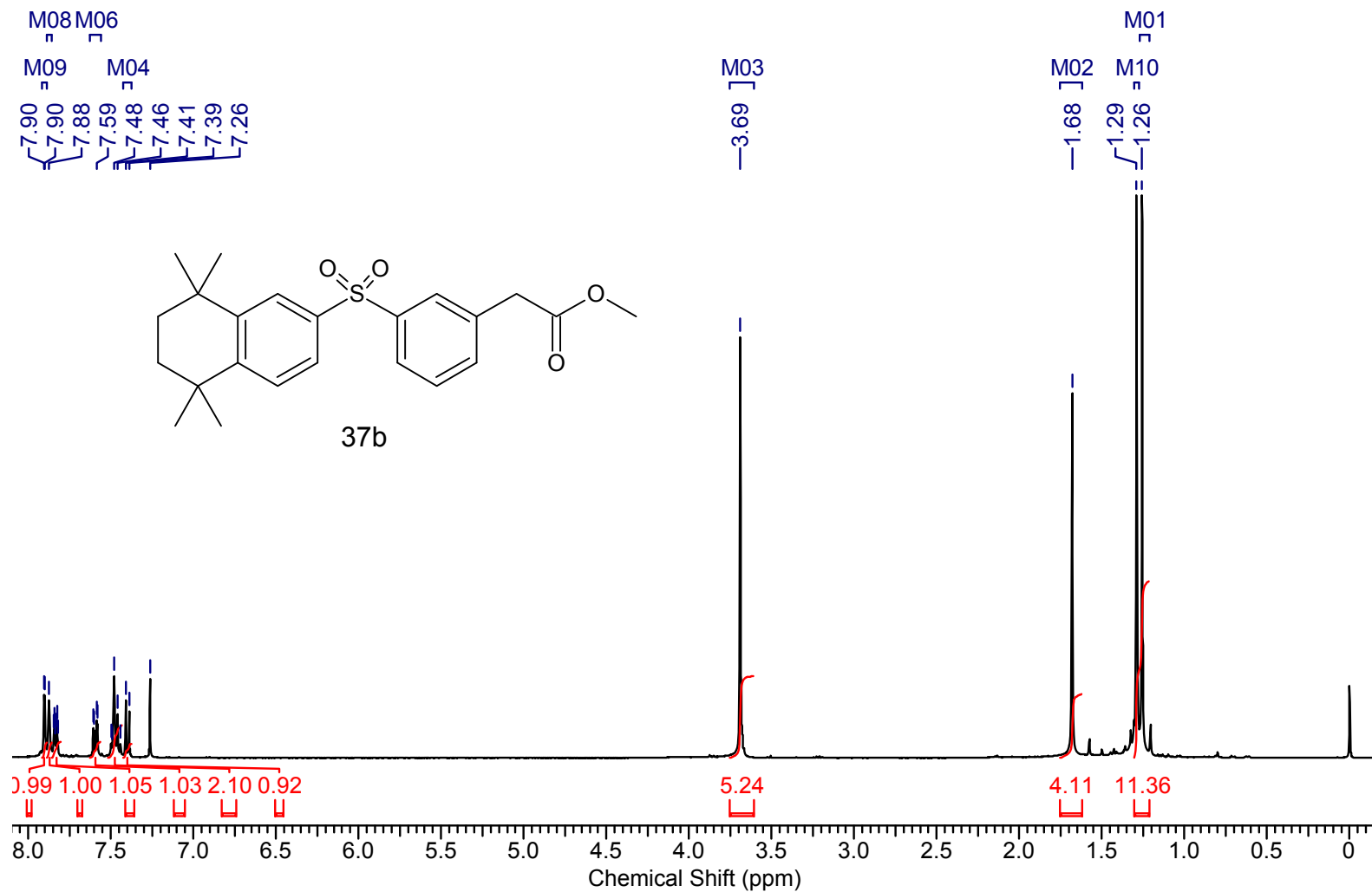


37a



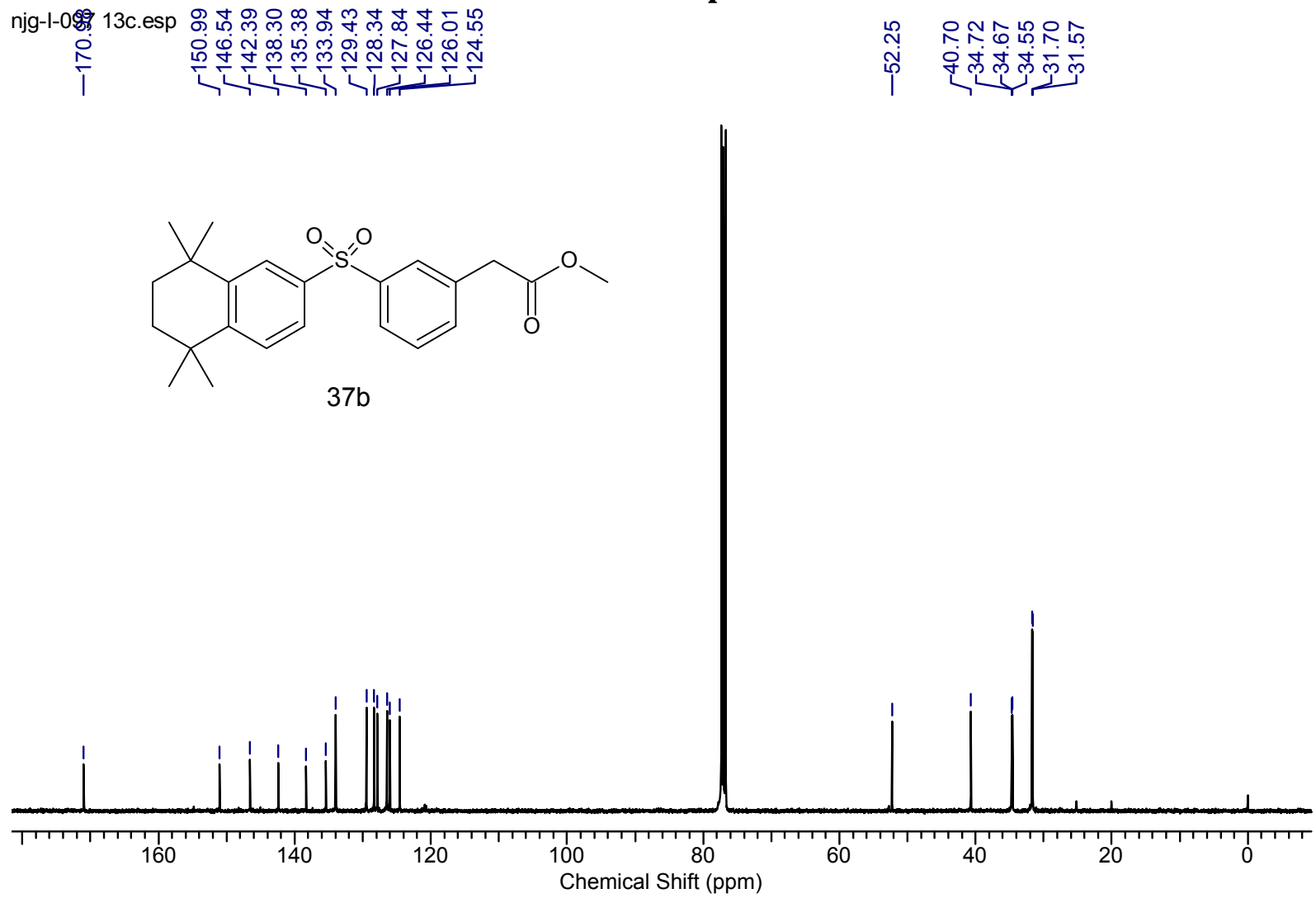
400 MHz ¹H-NMR of compound 37b in CDCl₃

njgM077M05.esp

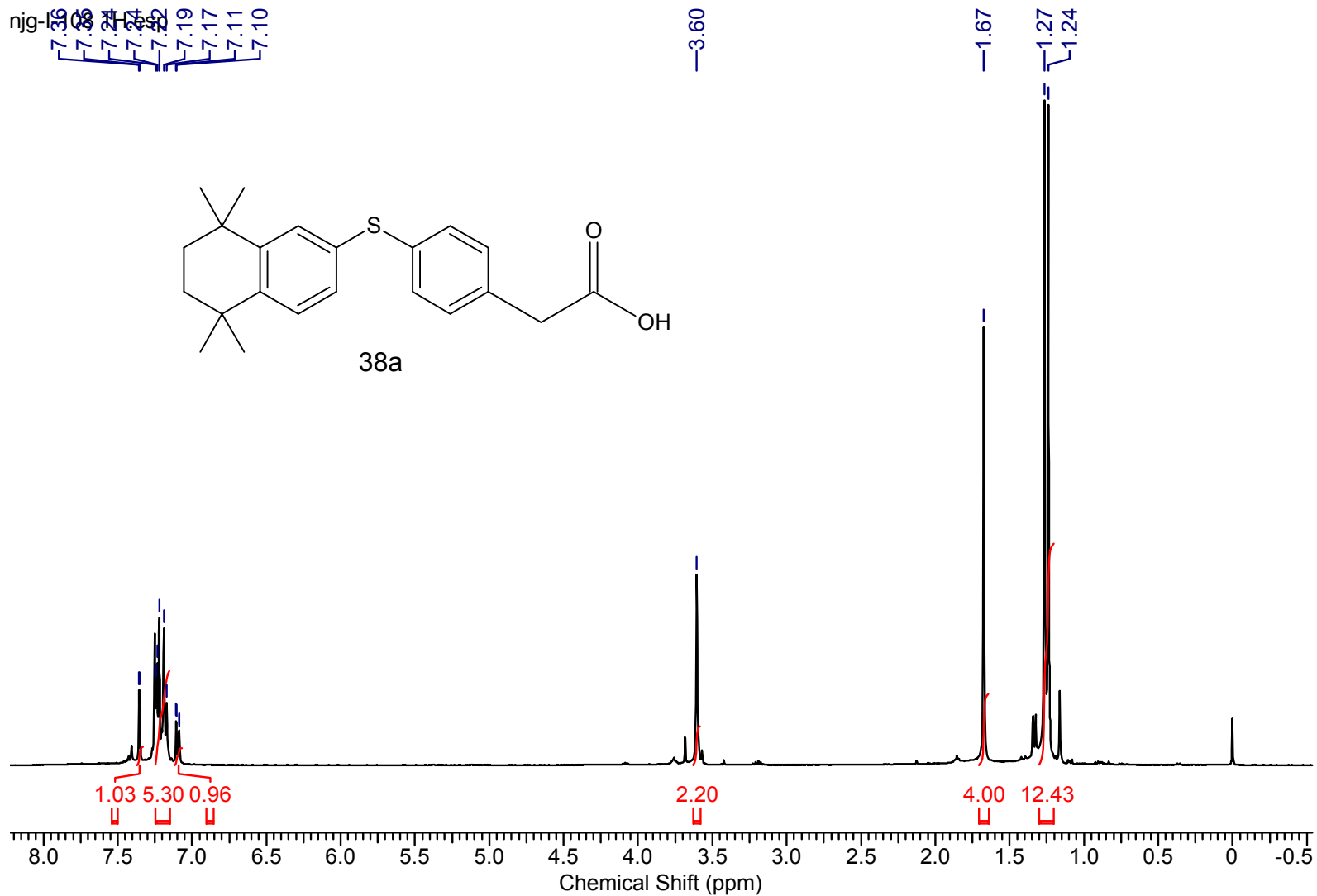


100 MHz ¹³C-NMR of compound 37b in CDCl₃

njg-l-009 13c.esp

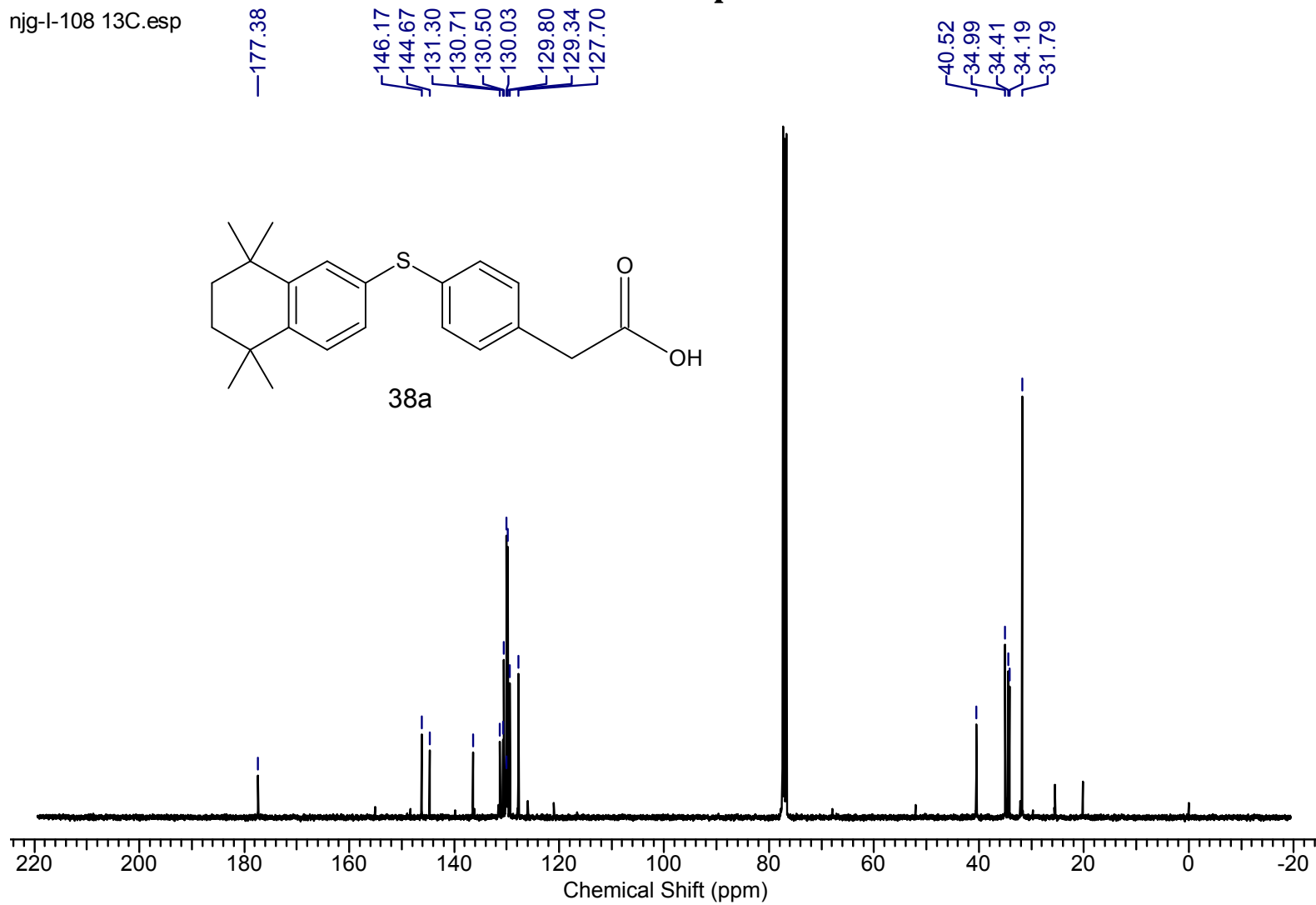


400 MHz ¹H-NMR of compound 38a in CDCl₃

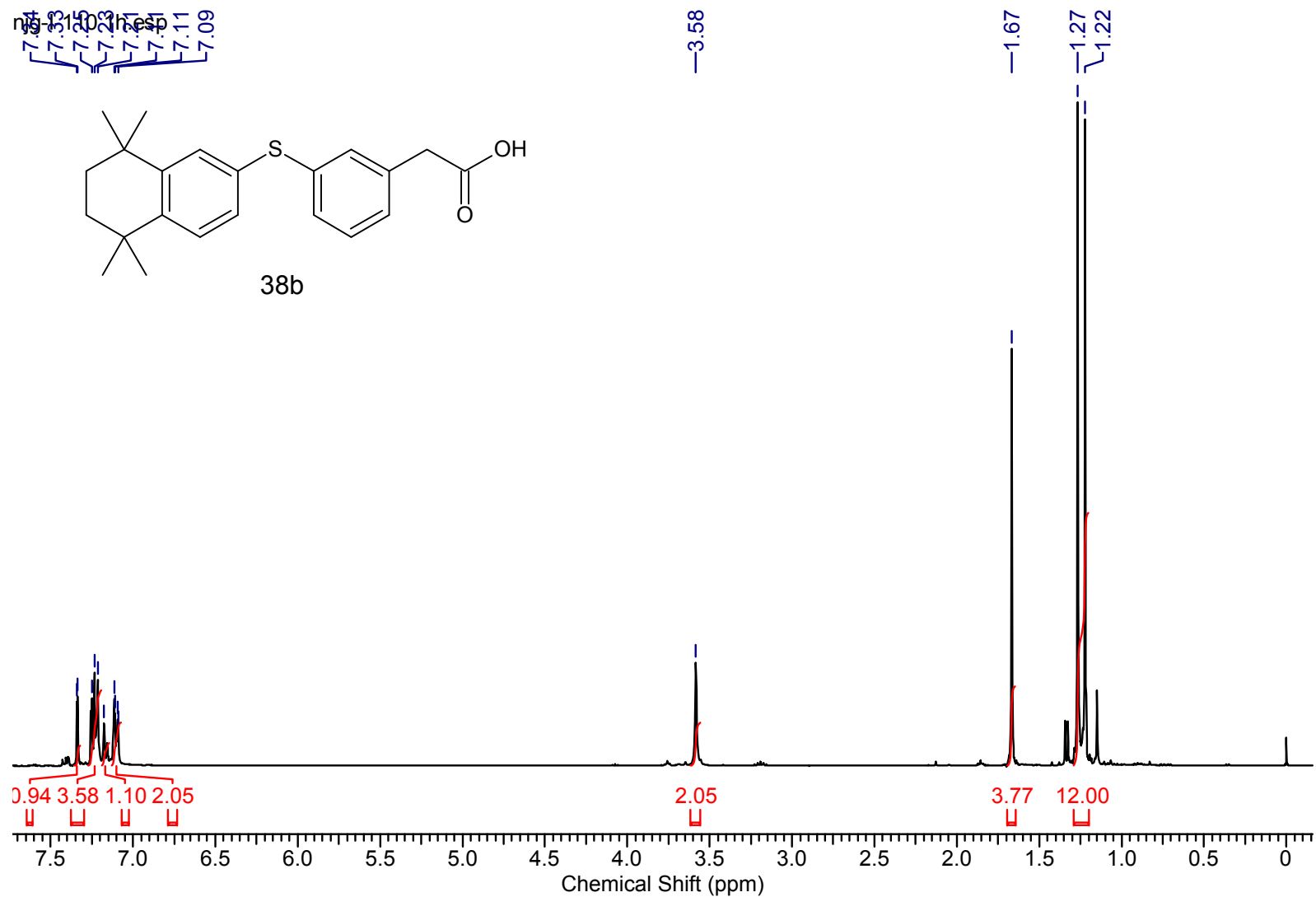


njg-l-108 13C.esp

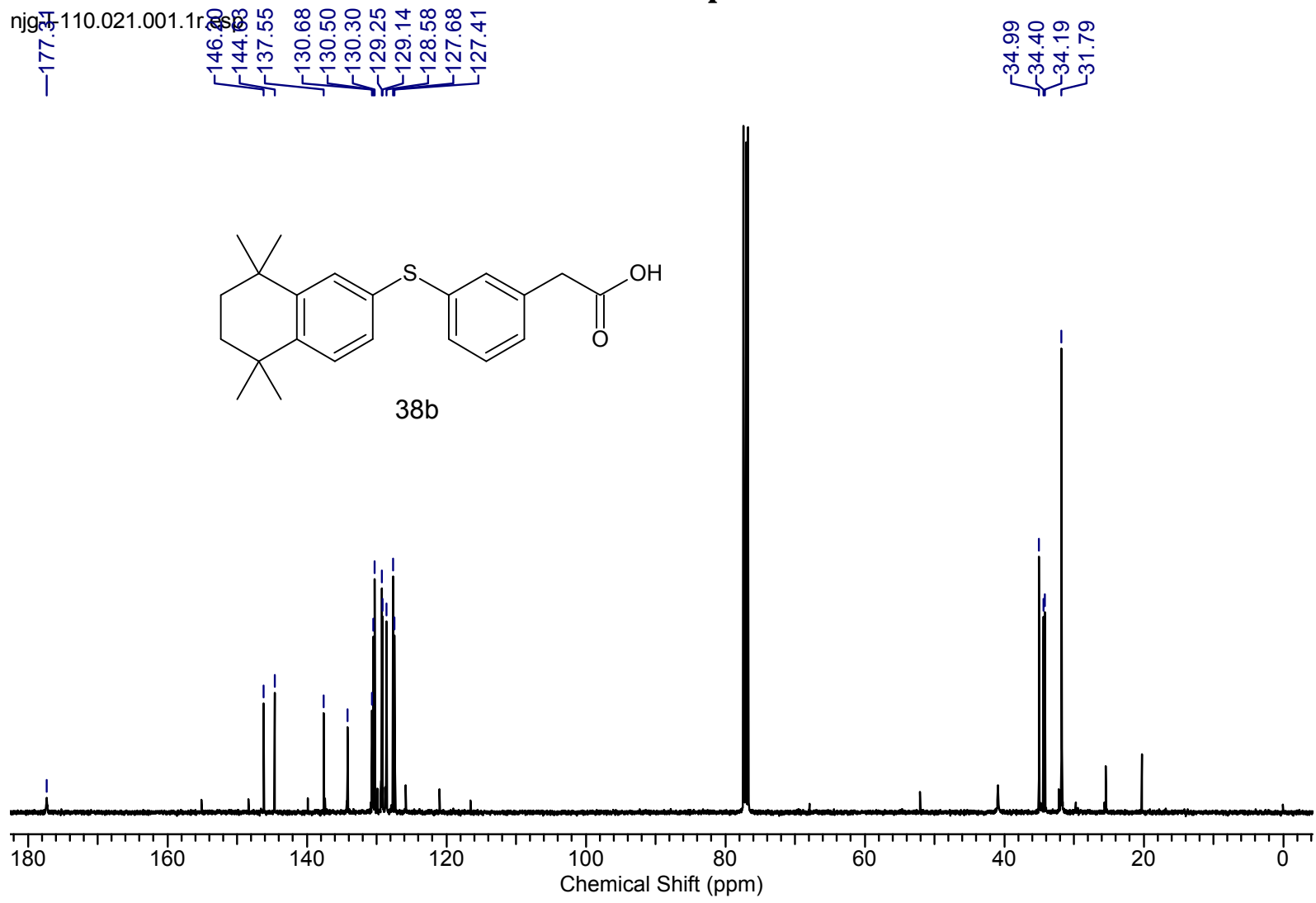
100 MHz ¹³C-NMR of compound 38a in CDCl₃



400 MHz ^1H -NMR of compound 38b in CDCl_3

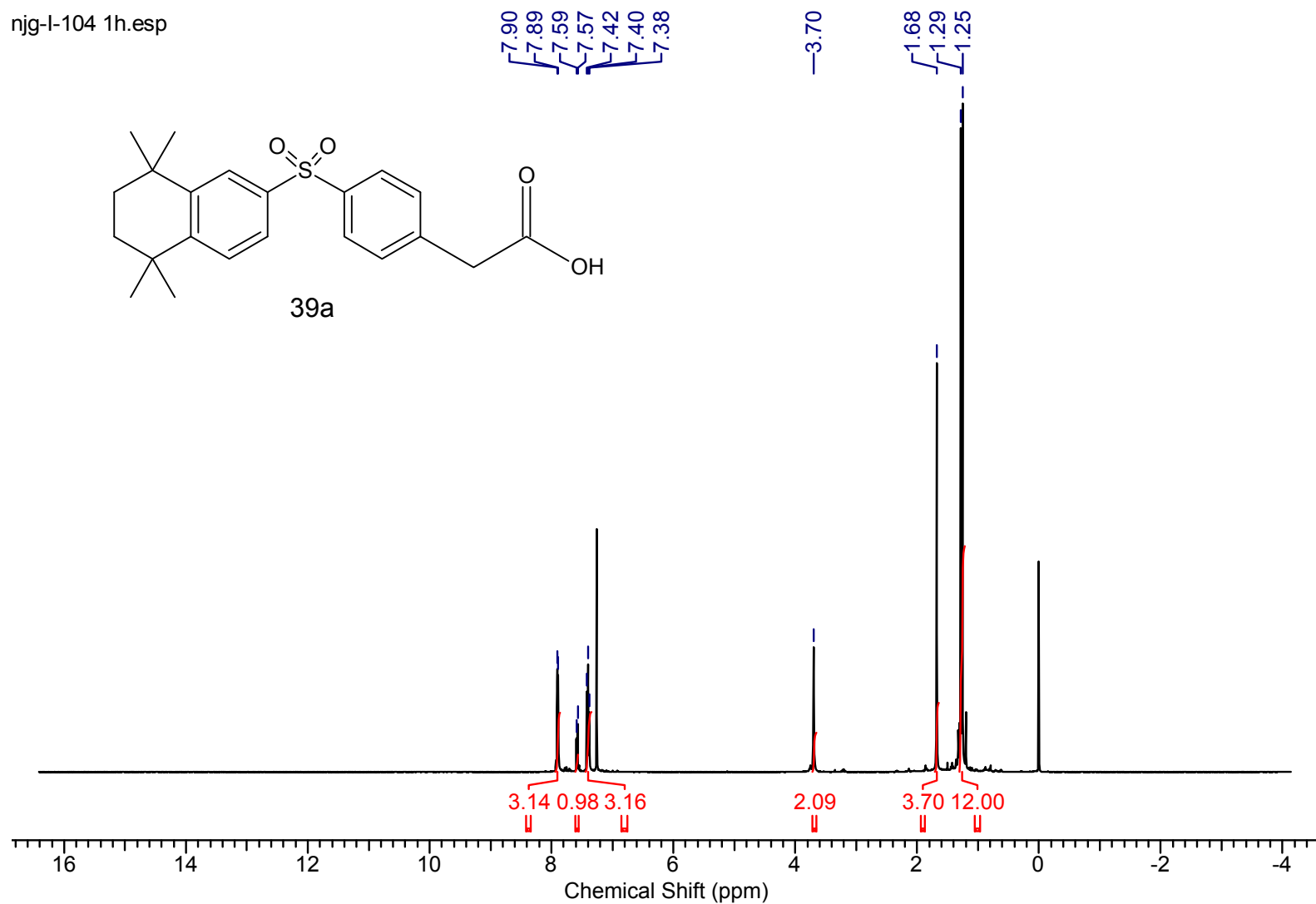


100 MHz ¹³C-NMR of compound 38b in CDCl₃



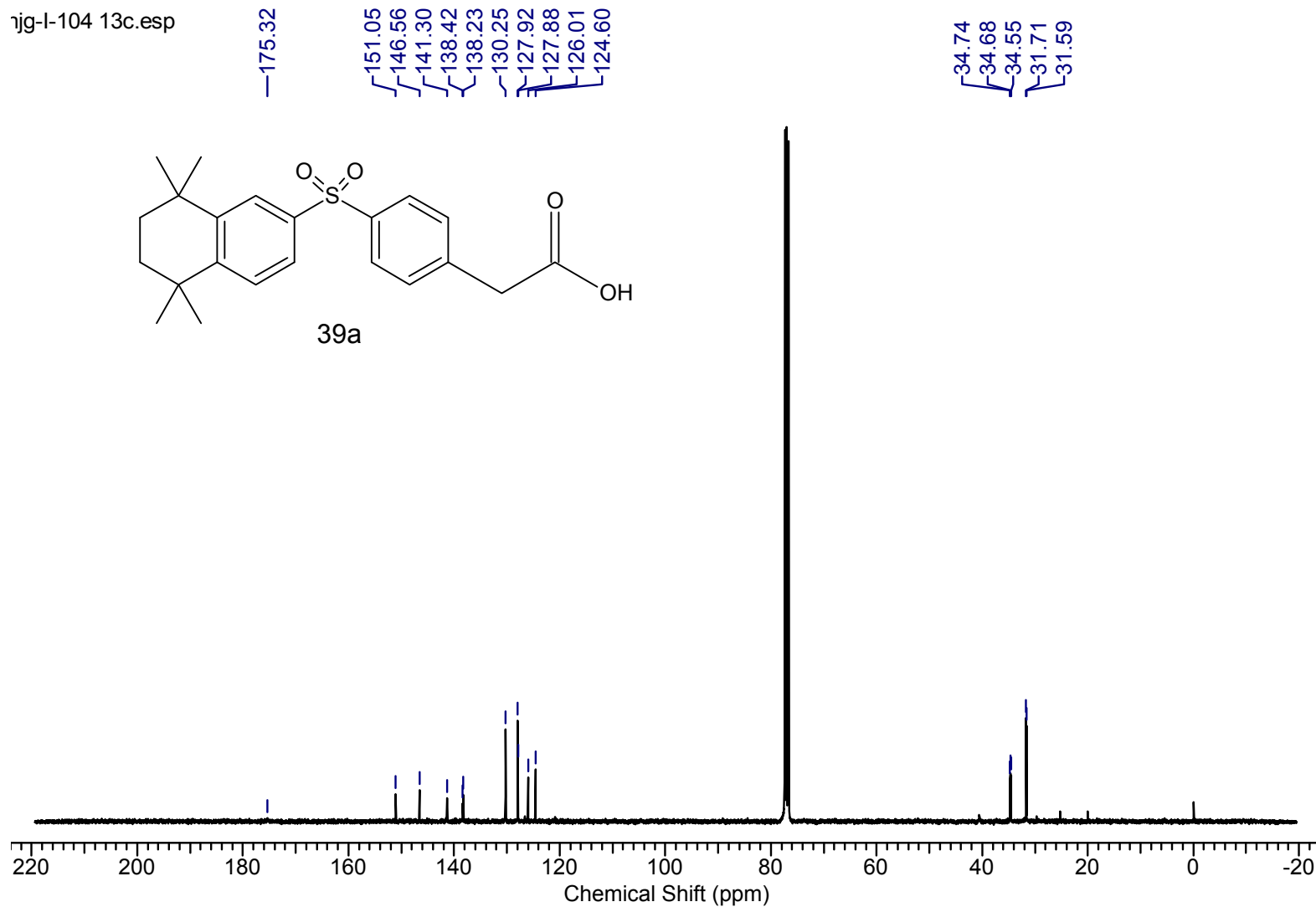
400 MHz ¹H-NMR of compound 39a in CDCl₃

njg-l-104 1h.esp



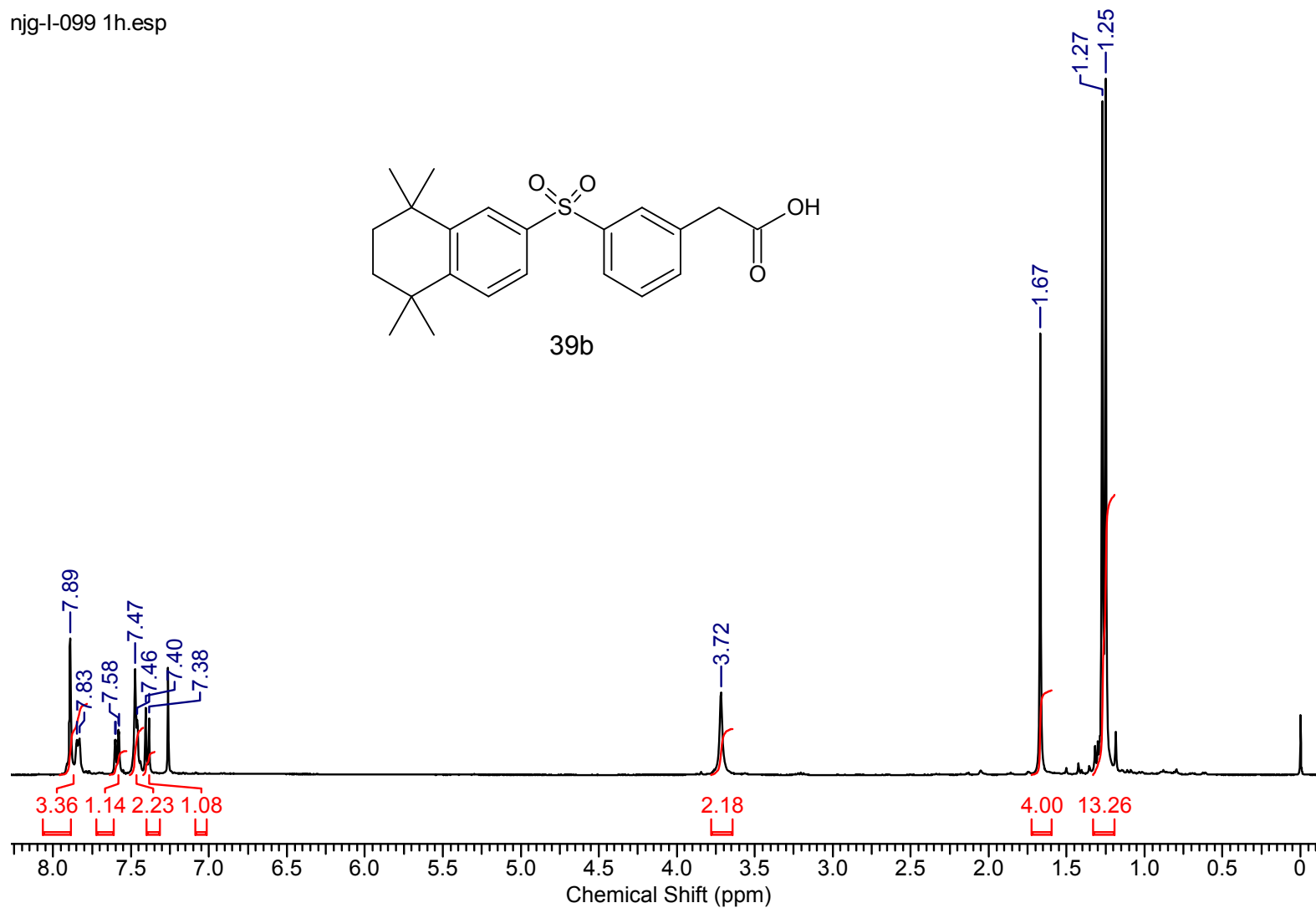
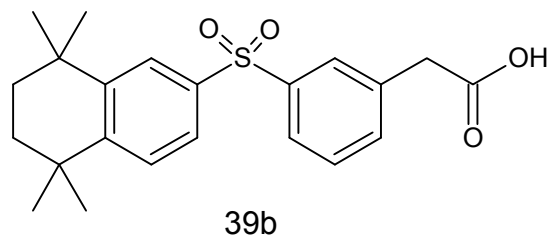
100 MHz ¹³C-NMR of compound 39a in CDCl₃

1jg-l-104 13c.esp

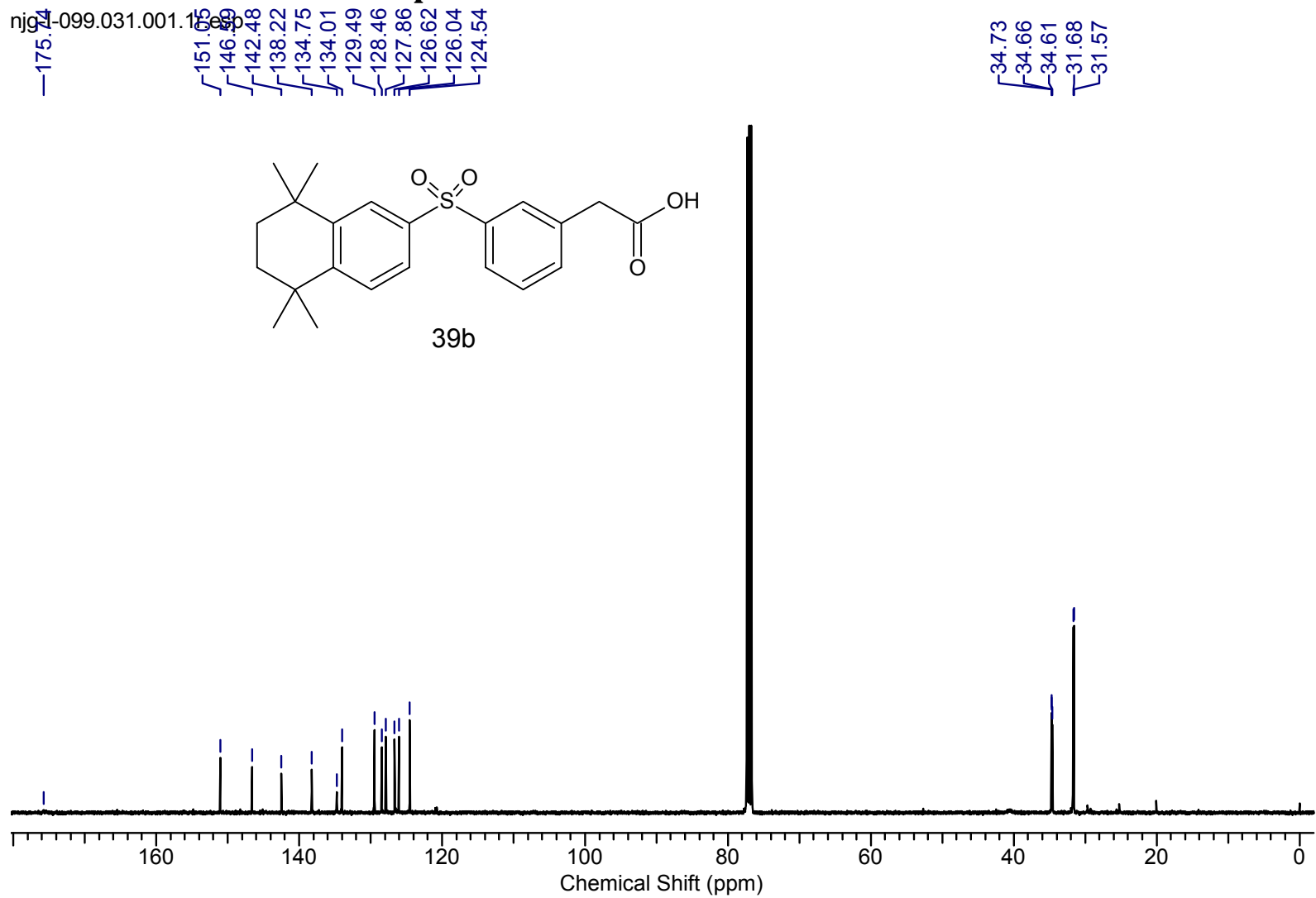


400 MHz ¹H-NMR of compound 39b in CDCl₃

njg-l-099 1h.esp



100 MHz ¹³C-NMR of compound 39b in CDCl₃



LC/MS analyses

LC/MS and HRMS analyses were obtained on a Waters ACQUITY UPLC-series liquid chromatography system equipped with a diode array detector and coupled to a LCT PREMIER XE™ time of flight (TOF) mass spectrometer with electrospray ionization (ESI). The liquid chromatography conditions were as follows: a Phenomenex column (NX, 3u, C18, 110A, 50.0x4.60 mm) was used, and it was eluted with the following gradient over 15 minutes at a rate of 0.4mL/min: solvent A: water (0.1% formic acid), solvent B: acetonitrile.

<u>Time (min)</u>	<u>Flow (mL/min)</u>	<u>%A</u>	<u>%B</u>
0.00	0.400	90.0	10.0
6.60	0.400	2.0	98.0
13.00	0.400	2.0	98.0
14.00	0.400	90.0	10.0
15.00	0.400	90.0	10.0

Compound purity was assigned on the basis of 254-nM detection data assessed by comparing relative peak areas of the signals.

Compound	Retention Time (min)	Purity (%)
5	8.12	96
6	8.34	97
9	8.14	95
11a	8.82	100
13a	8.85	98
15a	9.35	95
11b	9.40	95
13b	7.74	95
15b	9.97	94
18	10.06	80
20	9.83	93
22	9.60	100
24	9.13	99
26	8.96	99
28	10.02	100
30	9.67	99
34	8.44	99
38a	9.50	95
38b	9.45	95
39a	8.32	99
39b	8.27	99