Visible-light-promoted hydroxysulfenylation of alkenes for the synthesis of β-hydroxysulfides

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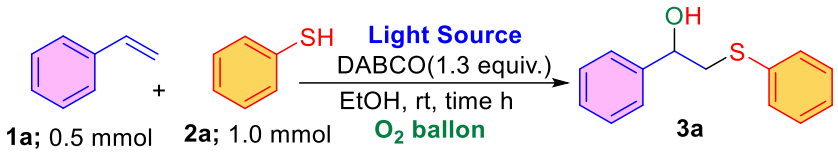
Light on-off experiment[S1]

For the on-off experiments, five oven-dried 10 mL Schlenk tubes were charged with styrene **1a** (0.5 mmol), thiophenol **2a** (1.0 equiv.), DABCO (1.3 equiv.), and EtOH (2 mL). An oxygen-filled balloon was attached to each tube, after which the reaction mixtures were stirred and irradiated under 410 nm Blue-light emitting diode at room temperature. After 1h, the LED was turn off, and one tube was removed from the irradiation setup for analysis. The remaining four tubes were stirred in the absence of light for an additional 1 h. We than, one tube was removed for analysis, and light turn back to on to irradiated remaining three reaction mixtures. After an additional 1h of irradiation, the LED was turn off, and one tube removed for analysis. The remaining two tubes were stirred in the absence of light for an additional 1h. Then, one tube was removed for analysis and the blue LED was turned back on to irradiate the last one reaction mixture. After an additional 1h irradiation, the last one was analyzed. Yield was determined by ^{1}H NMR spectroscopy using 1,3,5 trimethoxy benzene as internal standard.



Entry	Time (h)	Experiments	3a yield (%)
1	0	-	0
2	1	on	49
3	2	off	50
4	3	on	76
5	4	off	77
6	5	on	90

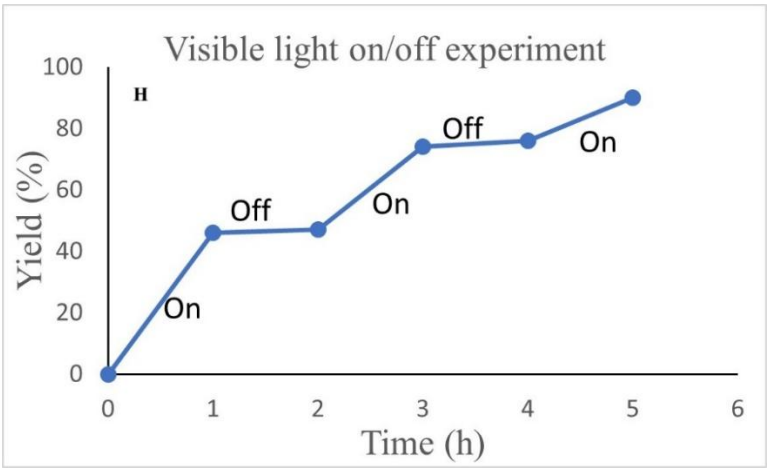
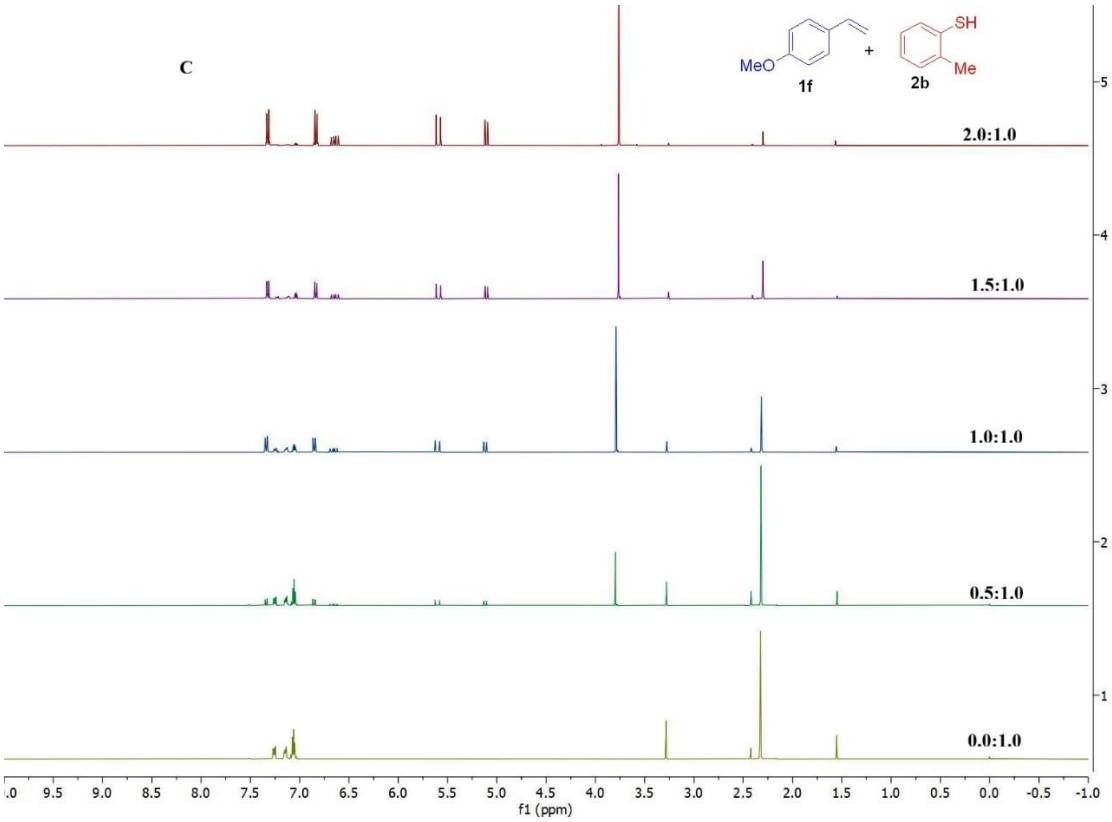
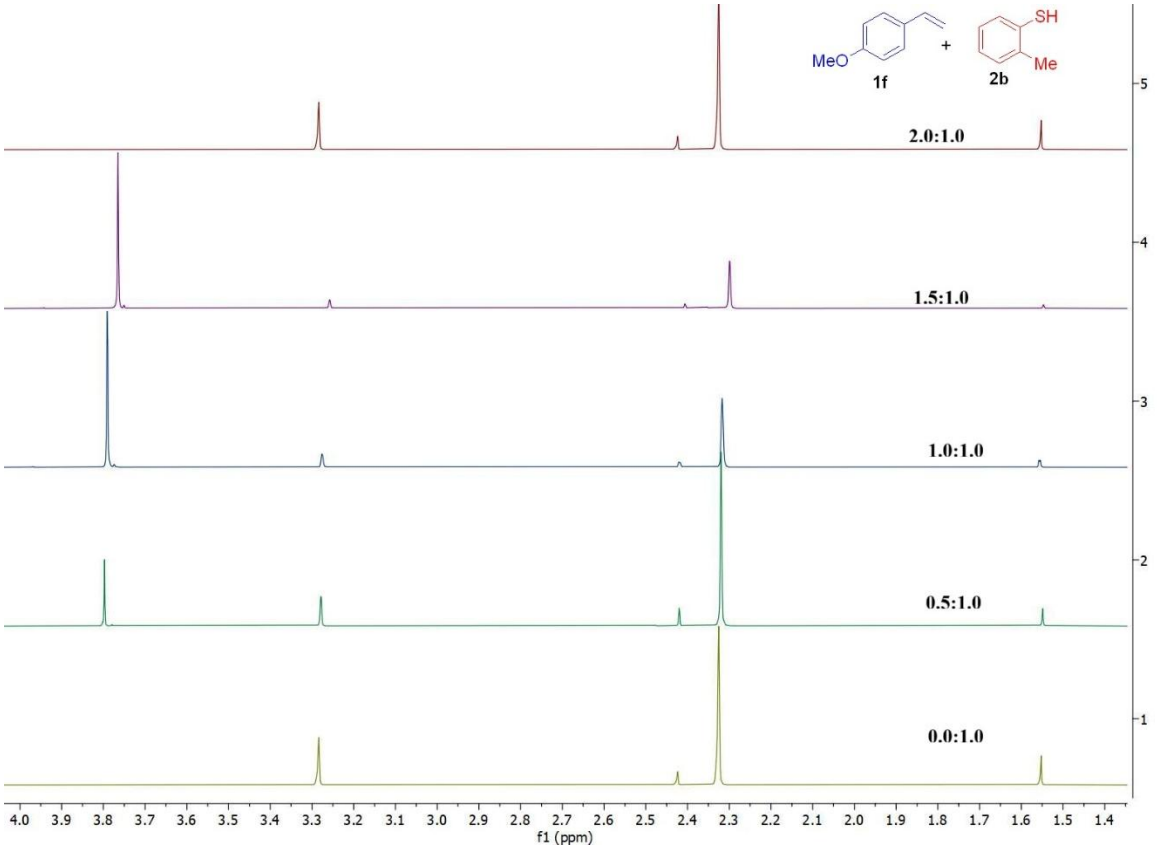


Fig S1: Light on-off experiments

^{1}H-NMR titration experiment to detect EDA complex formation[S1]

^{1}H NMR experiments were carried out in $CDCl_3$ solution containing 4-methoxy styrene **1f** and 2-methyl thiol **2b** in varying ratio. As the concentration of 4-methoxy styrene **2f** increased, the methyl proton of **2b** shifted little up-field, while the methoxy protons of **1f** shifted little downfield. These reciprocal changes confirmed the formation of an electron donor-acceptor (EDA) complex between **1f** and **2b**

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1f
+
SH
Me
2b
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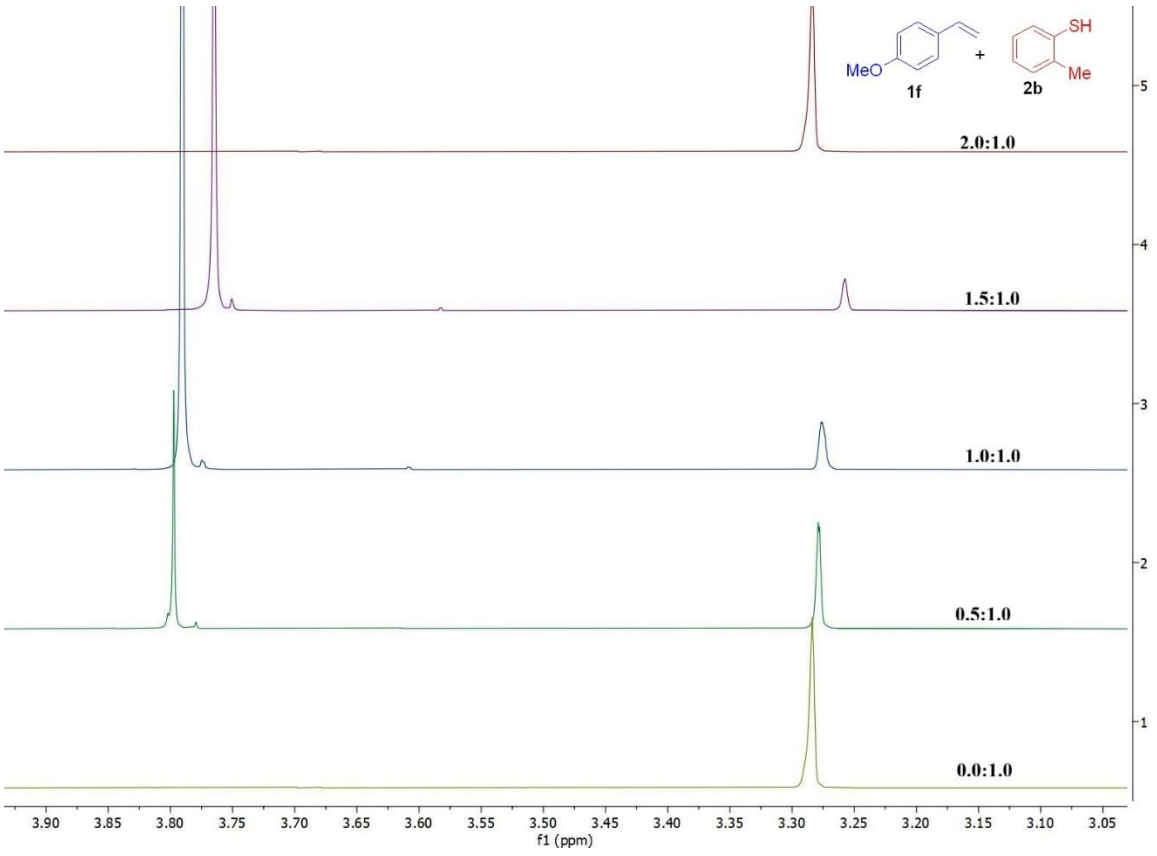
1.5:1.0
1.0:1.0
0.5:1.0
0.0:1.0
f1 (ppm)


MeO
1f
+
SH
Me
2b
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1.5:1.0
1.0:1.0
0.5:1.0
0.0:1.0
f1 (ppm)

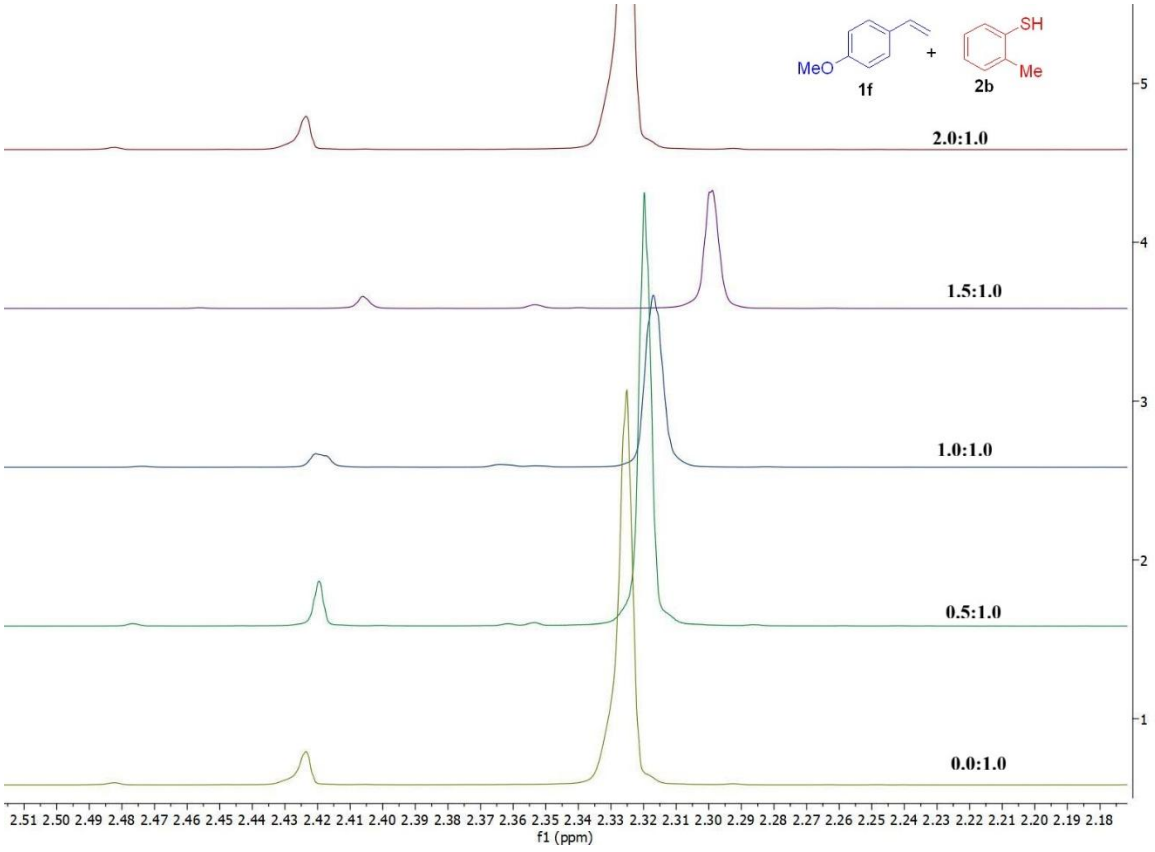


Fig S2: Evidence for the formation of Electron-donor-acceptor complex through ^{1}H NMR

Spectroscopic studies[S1]

The UV-Vis absorption spectra of styrene **1a** (25 mM), 2-methoxythiophenol **2d** (50 mM), and DABCO (50 mM) in ethanol were recorded using 1 cm quartz cuvettes on a U-3900 spectrophotometer (Fig. S3-S4). The spectra were obtained for each component individually as well as for their 1:1 binary mixture. In the mixed samples, two new absorption bands appear at wavelength above 300 nm, which were absent in the spectra of the individual components. The appearance of these additional bands suggests the formation of an EDA (electron donor-acceptor) complex between thiolate species and the styrene **1a**, thereby supporting the involvement of an EDA complex in this blue-LED driven reaction pathway.

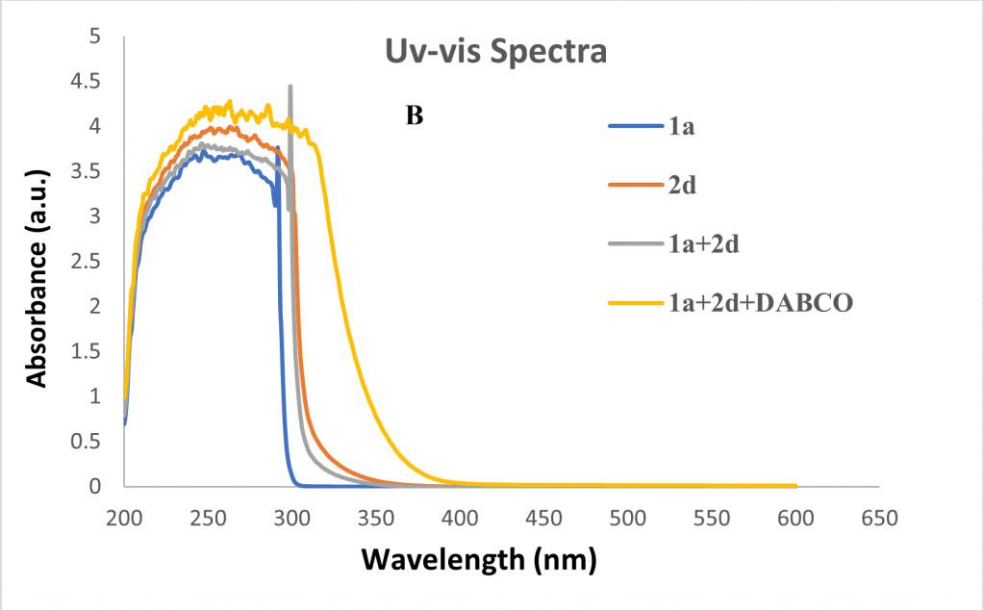


Fig. S3: UV-vis spectroscopic measurements on various **1a**, **2d**, and DABCO and their combination in EtOH

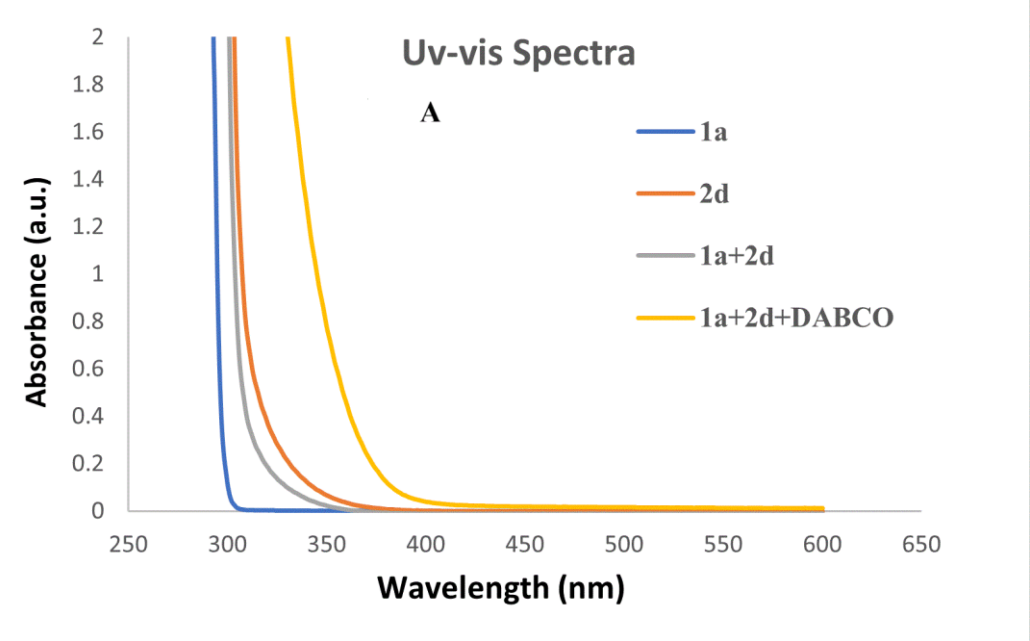


Fig. S4: UV-vis spectroscopic measurements on various **1a**, **2d** and DABCO their combination in EtOH

Fluorescence studies (Stern–Volmer Studies)

The fluorescence emission intensities were recorded on a F-7000 FL Spectrophotometer. The excitation wavelength was set at 240 nm, and the emission wavelength was measured over the range of 250-470 nm. Samples were prepared by mixing thiophenol **2a** (3.0×10^{-5} mol/L) with varying concentration of styrene **1a** and DABCO in EtOH (total volume = 2.0 mL) in a quartz fluorescence cuvette. For each quenching experiment, 1.0 mL of styrene **1a** or DABCO stock solution at different concentration was added to 1.0 mL of the thiophenol solution, yielding a total volume of 2.0 mL. Then the emission

intensity was collected and the data were presented in Figure S5 and S6.

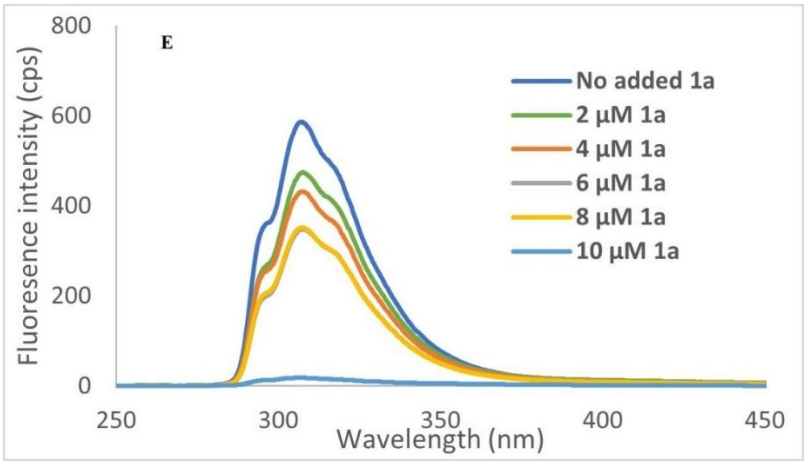


Fig S5: Fluorescence emission in the presence of Styrene (**1a**).

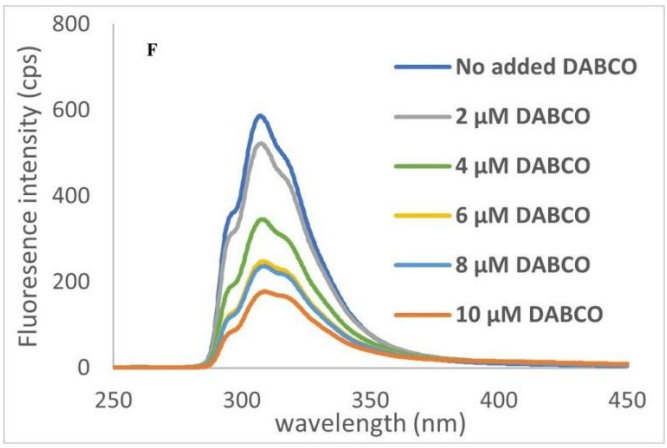


Fig S6: Fluorescence emission in the presence of DABCO

EPR experiments[S1]

Without Blue-LED irradiation: A mixture of styrene **1a** (0.5 mmol), thiophenol **2a** (1.0 mmol), DABCO (1.3 equiv.), EtOH (3 mL), and DMPO (12 µL) was prepared and directly analyzed by EPR.

Standard reaction Conditions:

A mixture of styrene **1a** (0.5 mmol), thiophenol **2a** (1.0 mmol), DABCO (1.3 equiv.), EtOH (3 mL), and DMPO (12 µL) was stirred under Blue LED irradiation under oxygen atmosphere at room temperature. Aliquots were taken and analyzed by EPR after 1, 5, 10, 15, and 30 minutes of irradiation.

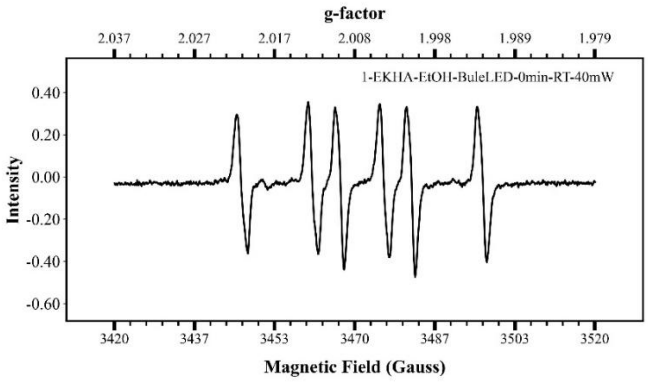


Fig S7: EPR Without Blue-LED irradiation

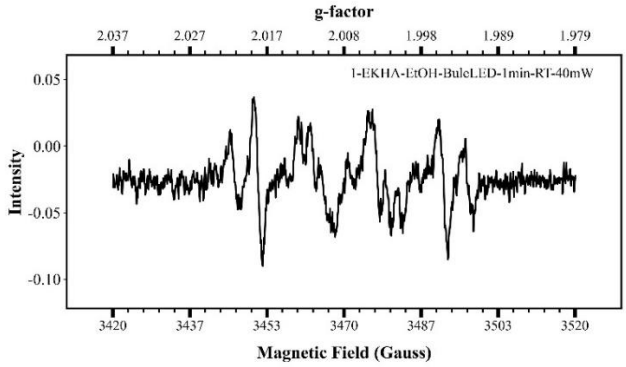


Fig S8: EPR under Blue-LED irradiation after 1 min.

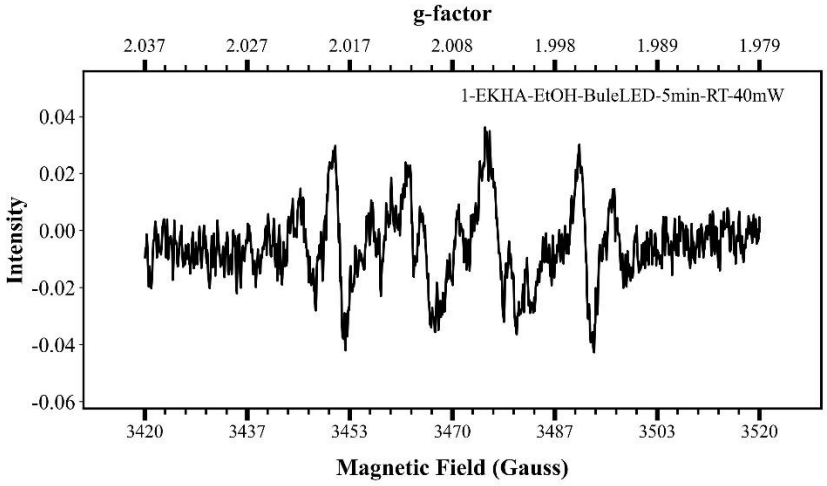


Fig S9: EPR under Blue-LED irradiation after 5 min.

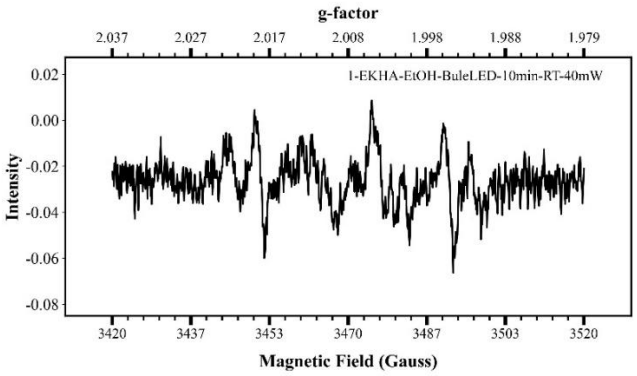


Fig S9: EPR under Blue-LED irradiation after 10 min.

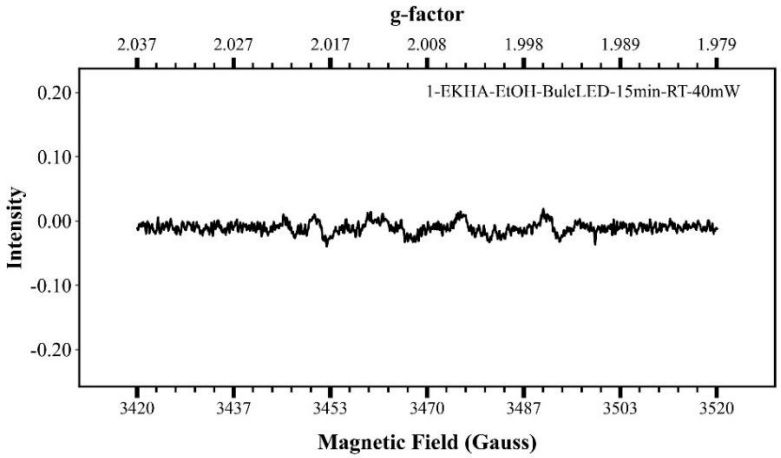


Fig S10: EPR under Blue-LED irradiation after 15 min.

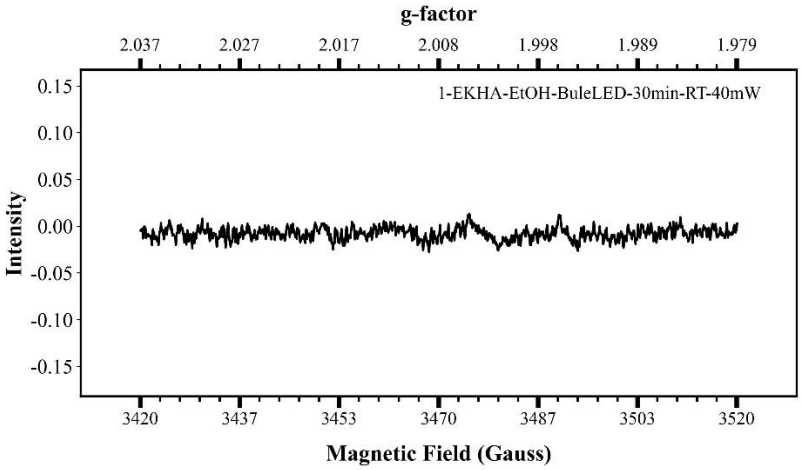
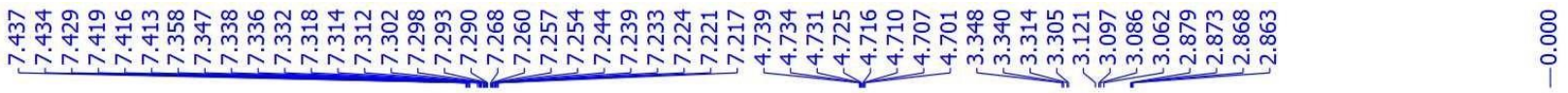


Fig S11: EPR under Blue-LED irradiation after 30 min.

References:

S1 (a) H. Ruan, L.-G. Meng, L. Zhu and L. Wang, *Adv. Synth. Catal*., 2019, **361**, 3217; (b) Z. Chen, F. Xue, T. Liu, B. Wang, Y. Zhang, W. Jin, Y. Xia, and C. Liu, *Green Chem.*, 2022, **24**, 3250; (c) J. Shi, X. W. Gao, Q. X. Tong, and J. J. Zhong, *J. Org. Chem.,* 2021, **86**, 12922; (d) J. E. Hong, Y. Jung, D. Min, M. Jang, S. Kim, J. Park, and Y. Park, *J. Org. Chem.*, 2022, **87**, 7378; (e) A. Das, and K. J. Thomas, ACS omega, 2023, **8**, 18275. (f) Z. Xing, M. Yang, H. Sun, Z. Wang, P. Chen, L. Liu, X. Wang, X. Xie and X. She, *Green Chem.,* 2018, **20**, 5117.

Copies of ^{1}H, ^{13}C, ^{19}F-NMR and Mass



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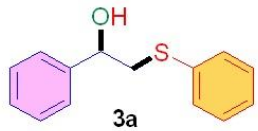
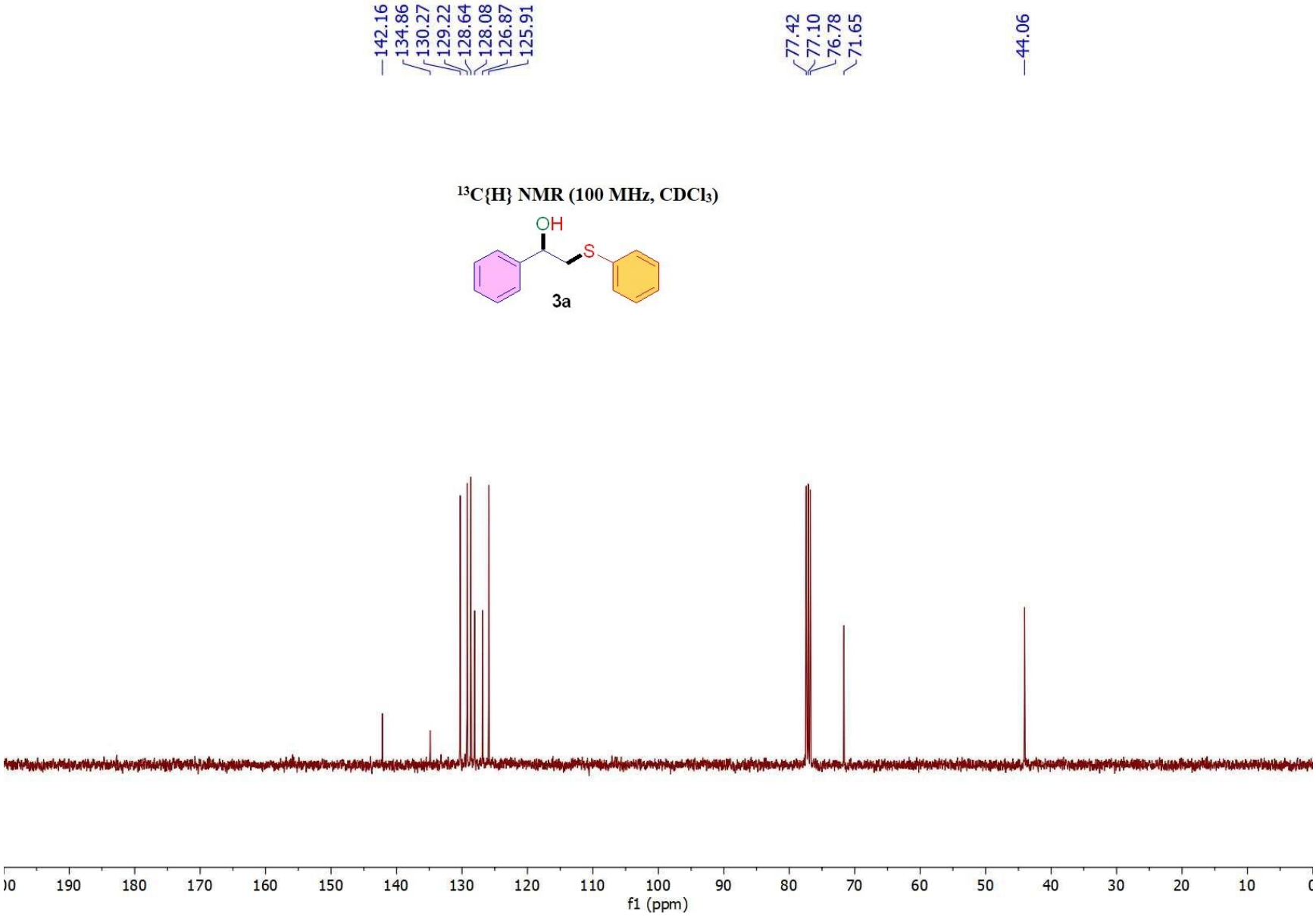
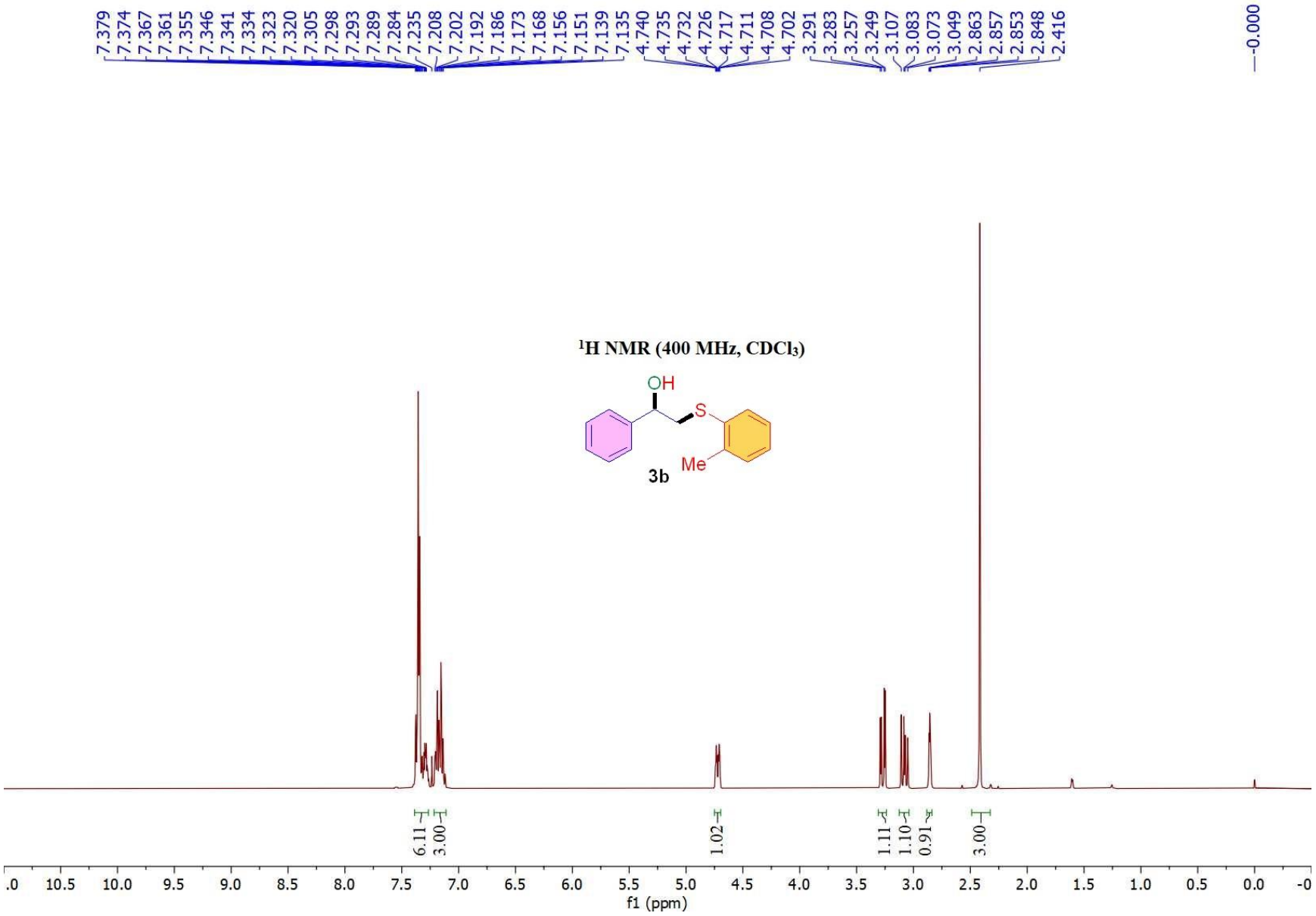


3a



$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)

3a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7.293
7.289
7.284
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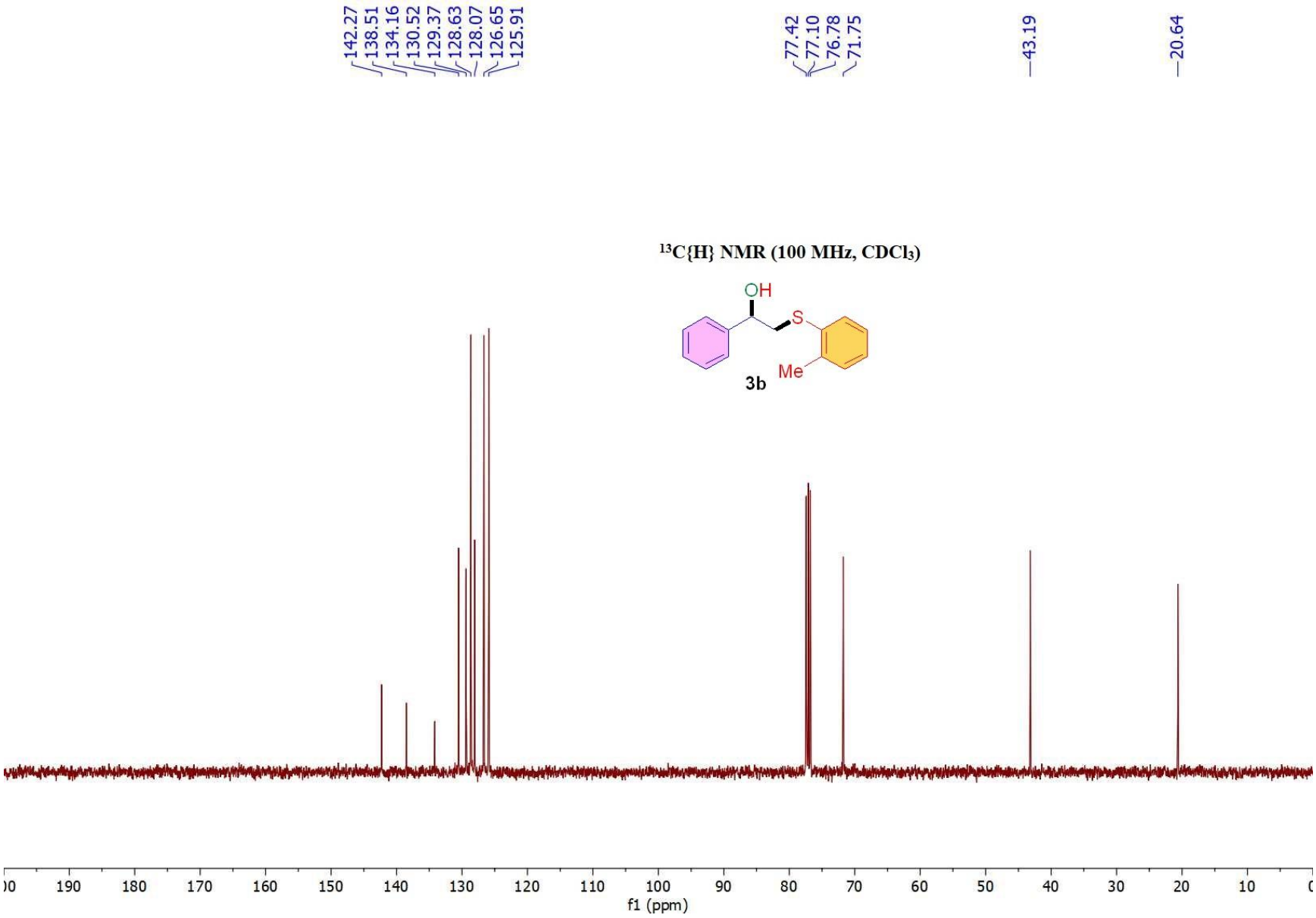
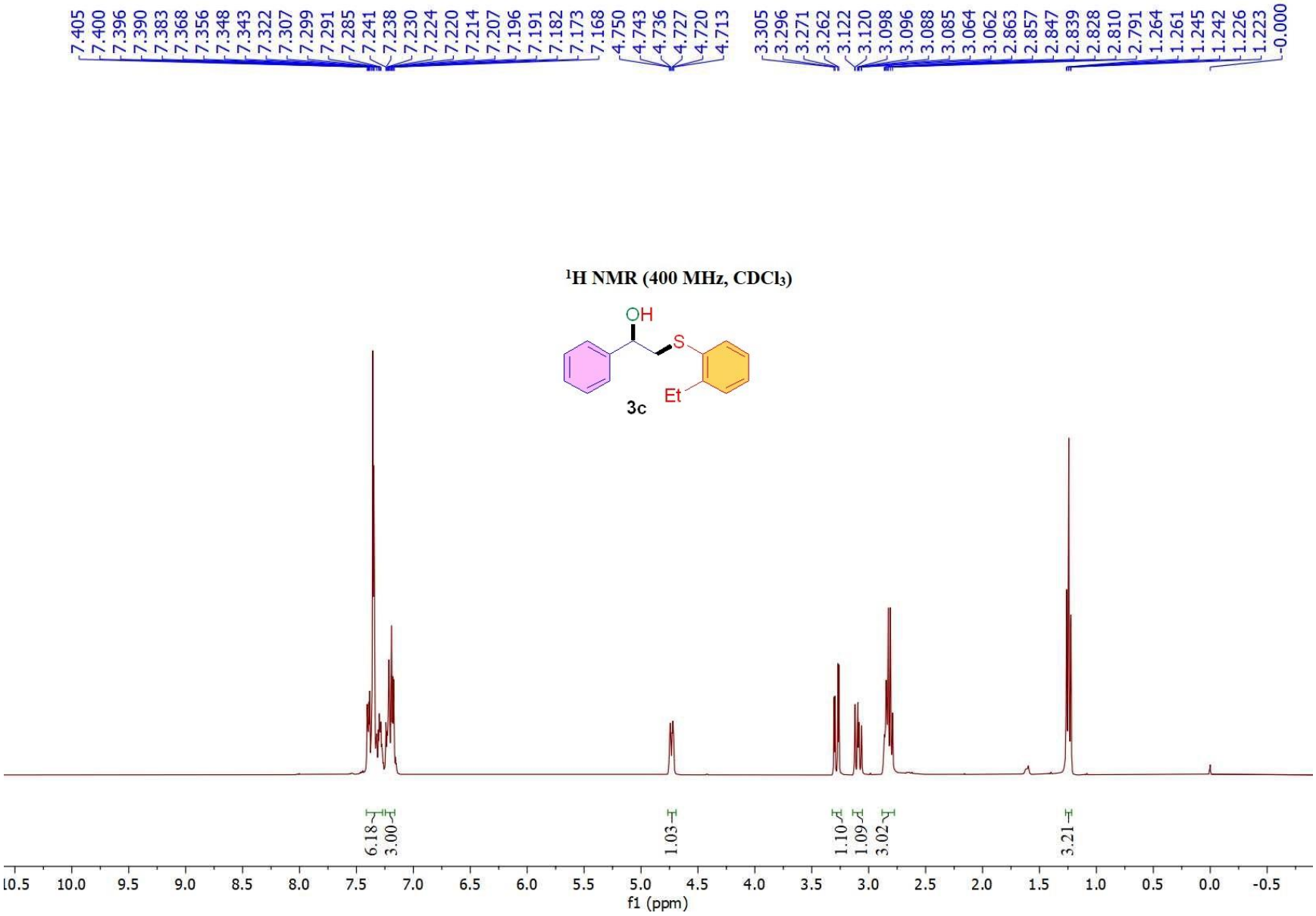
7.173
7.168
7.156
7.151
7.139
7.135
4.740
4.735
4.732
4.726
4.717
4.711
4.708
4.702
3.291
3.283
3.257
3.249
3.107
3.083
3.073
3.049
2.863
2.857
2.853
2.848
2.416
-0.000
1H NMR (400 MHz, CDCl3)
OH
S
Me
3b
6.11
3.00
1.02
1.11
1.10
0.91
3.00
f1 (ppm)



$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)

3b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7.291
7.285
7.241
7.238
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7.207
7.196
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7.182
7.173
7.168
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3.122
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2.857
2.847
2.839
2.828
2.810
2.791
1.264
1.261
1.245
1.242
1.226
1.223
-0.000
1H NMR (400 MHz, CDCl3)
OH
S
Et
3c
6.18
3.00
1.03
1.10
1.09
3.02
3.21
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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4.0
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0.0
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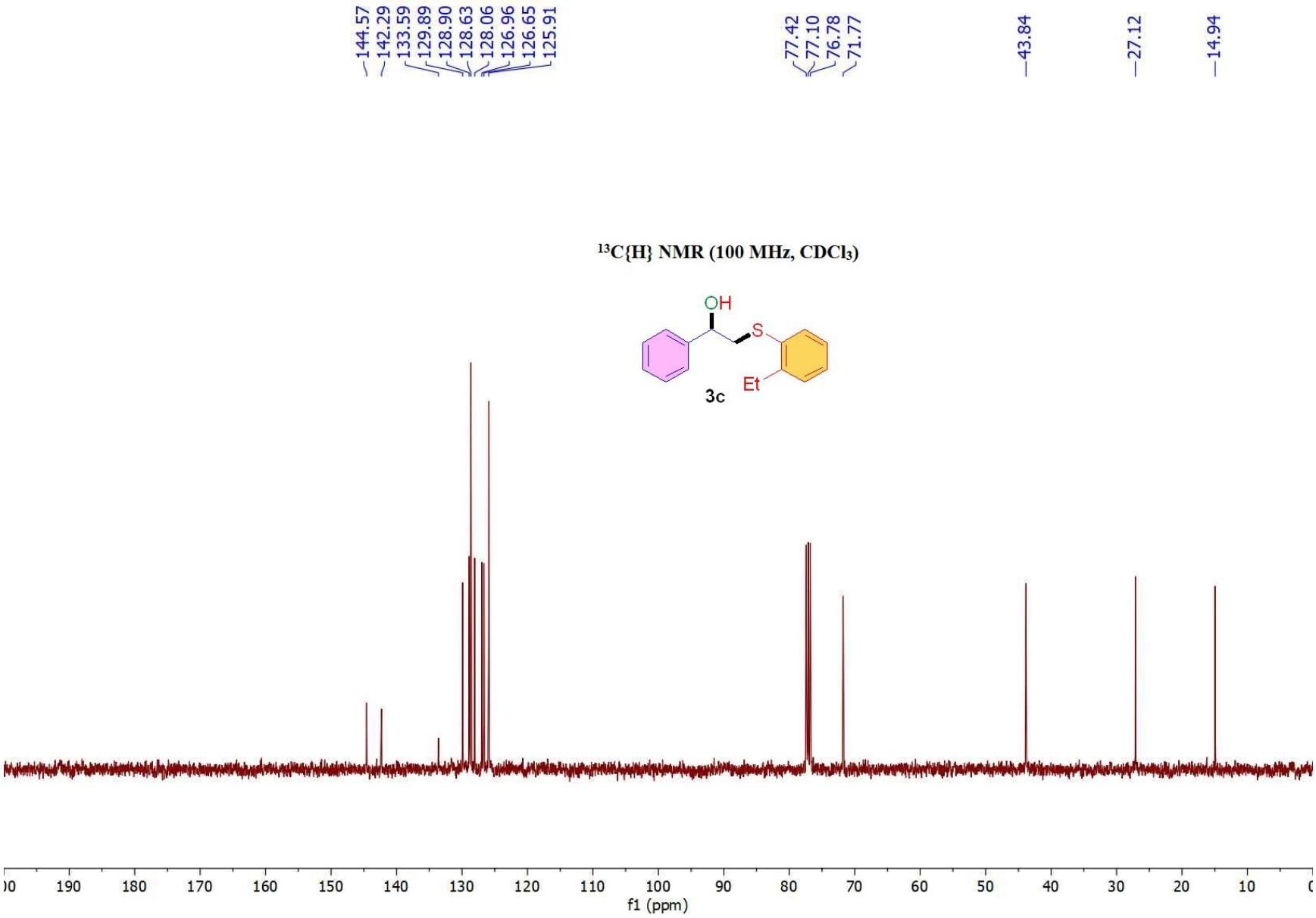
f1 (ppm)



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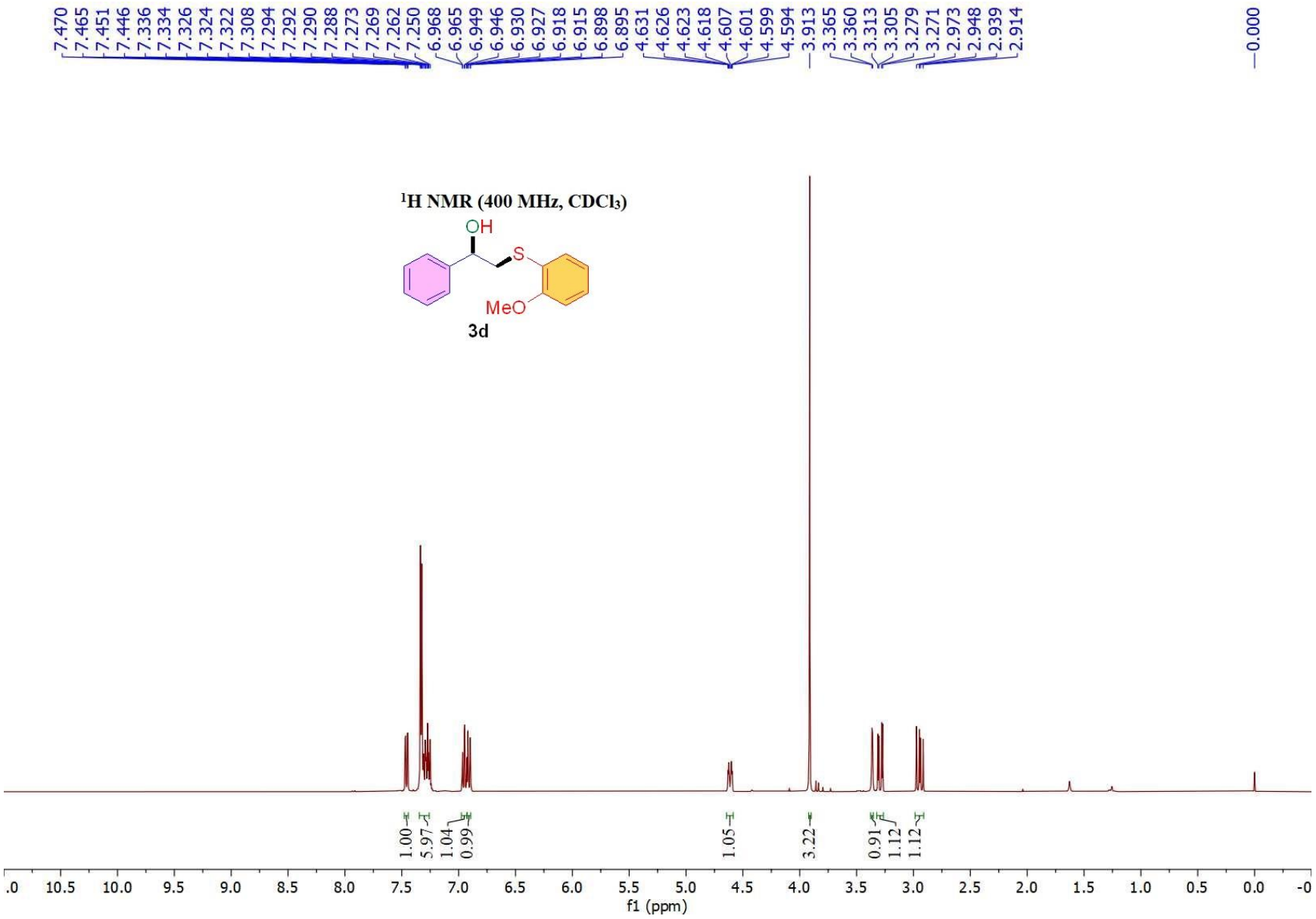
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7.288
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6.949
6.946
6.930
6.927
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3.365
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3.279
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2.914
0.000
1H NMR (400 MHz, CDCl3)
OH
S
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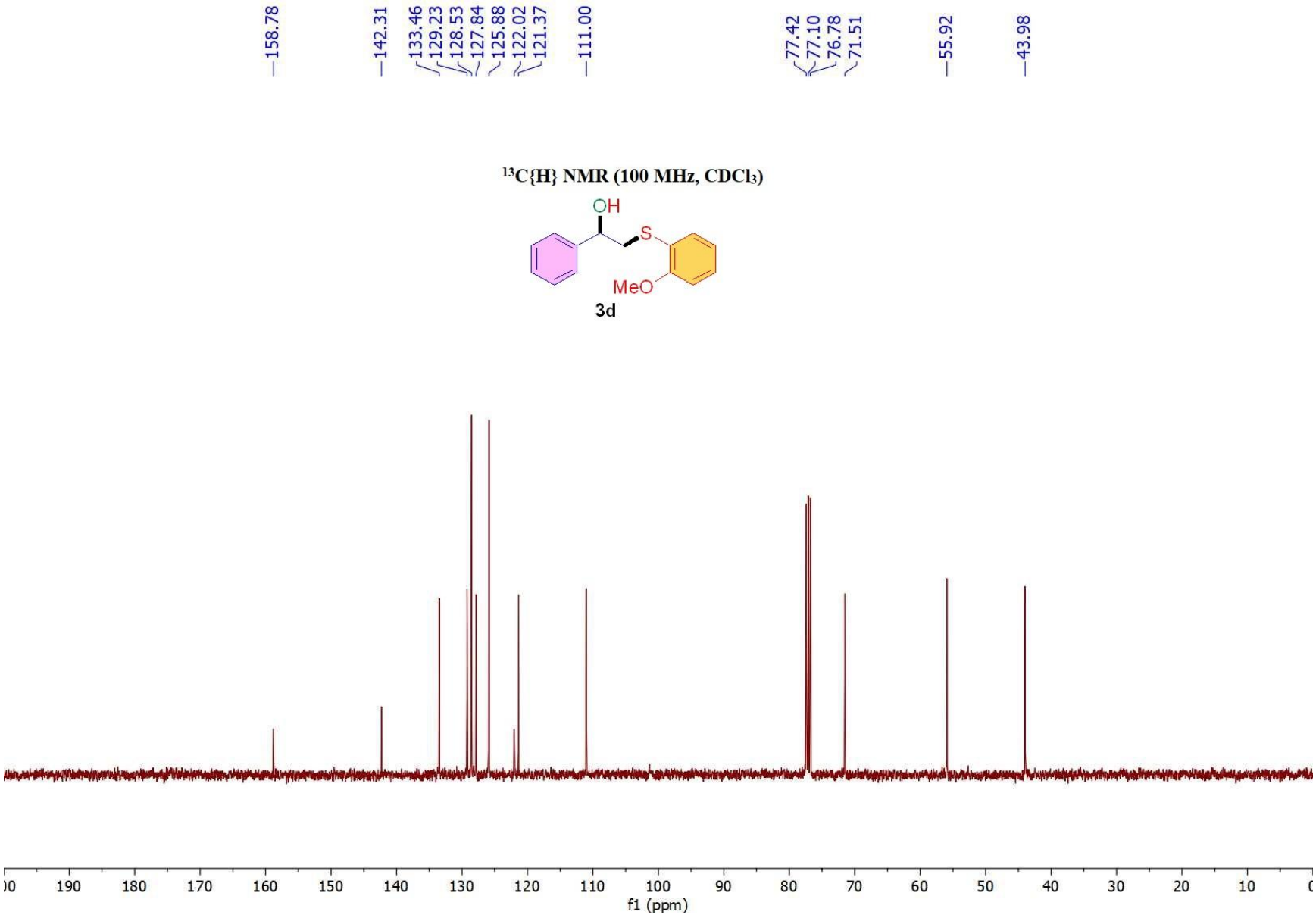
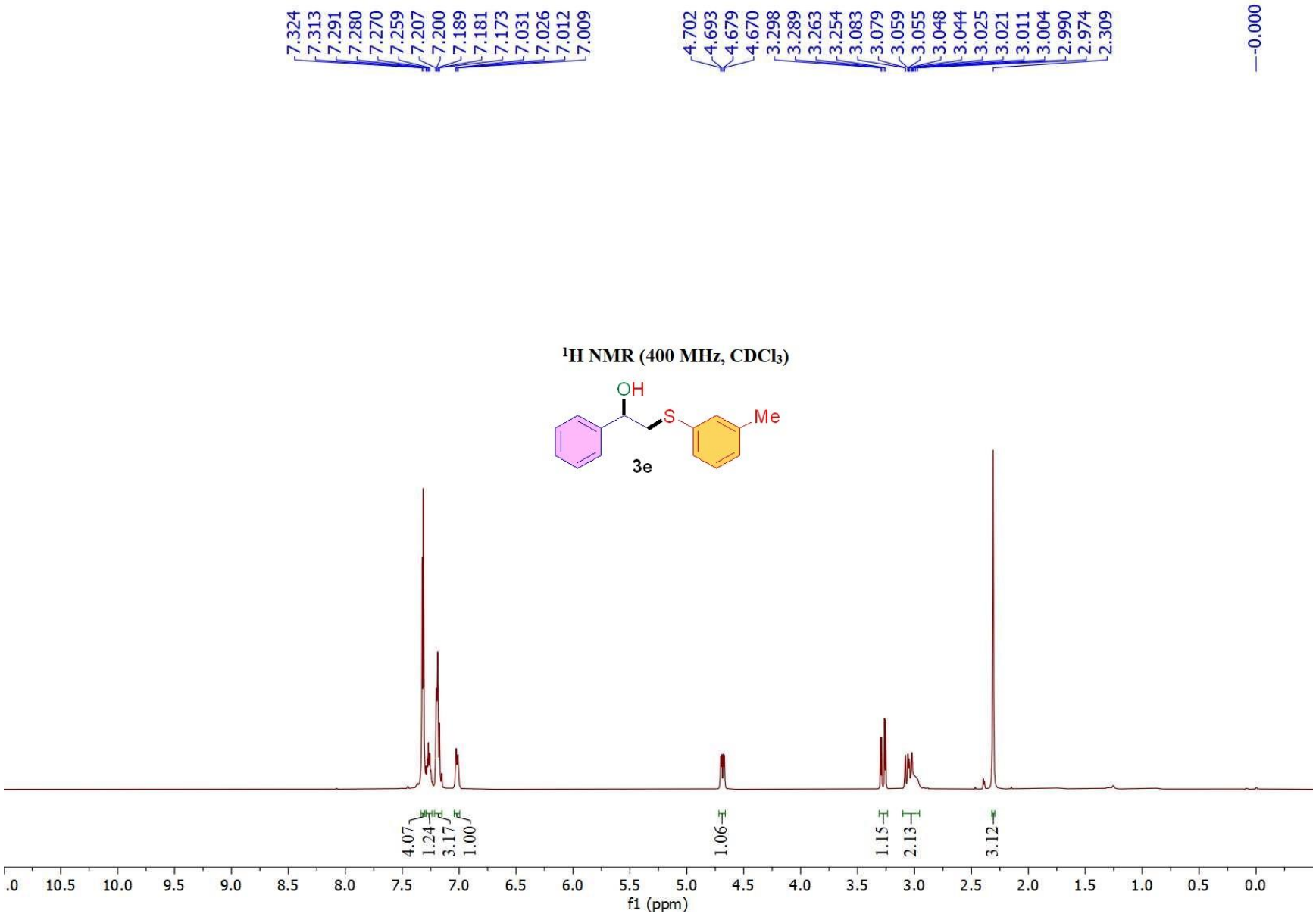
1.12
f1 (ppm)



$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)

3d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1H NMR (400 MHz, CDCl3)
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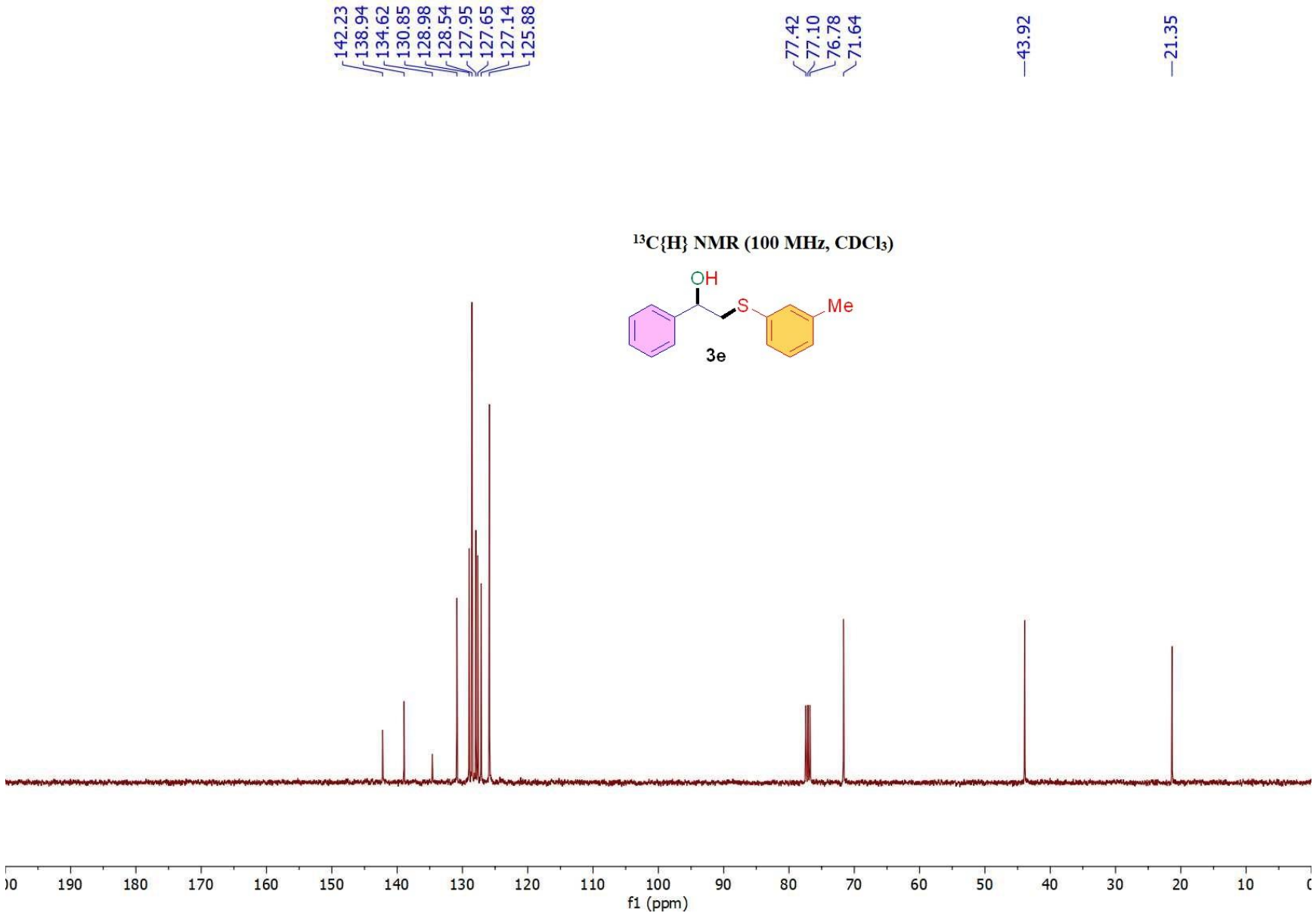
f1 (ppm)



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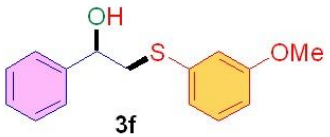
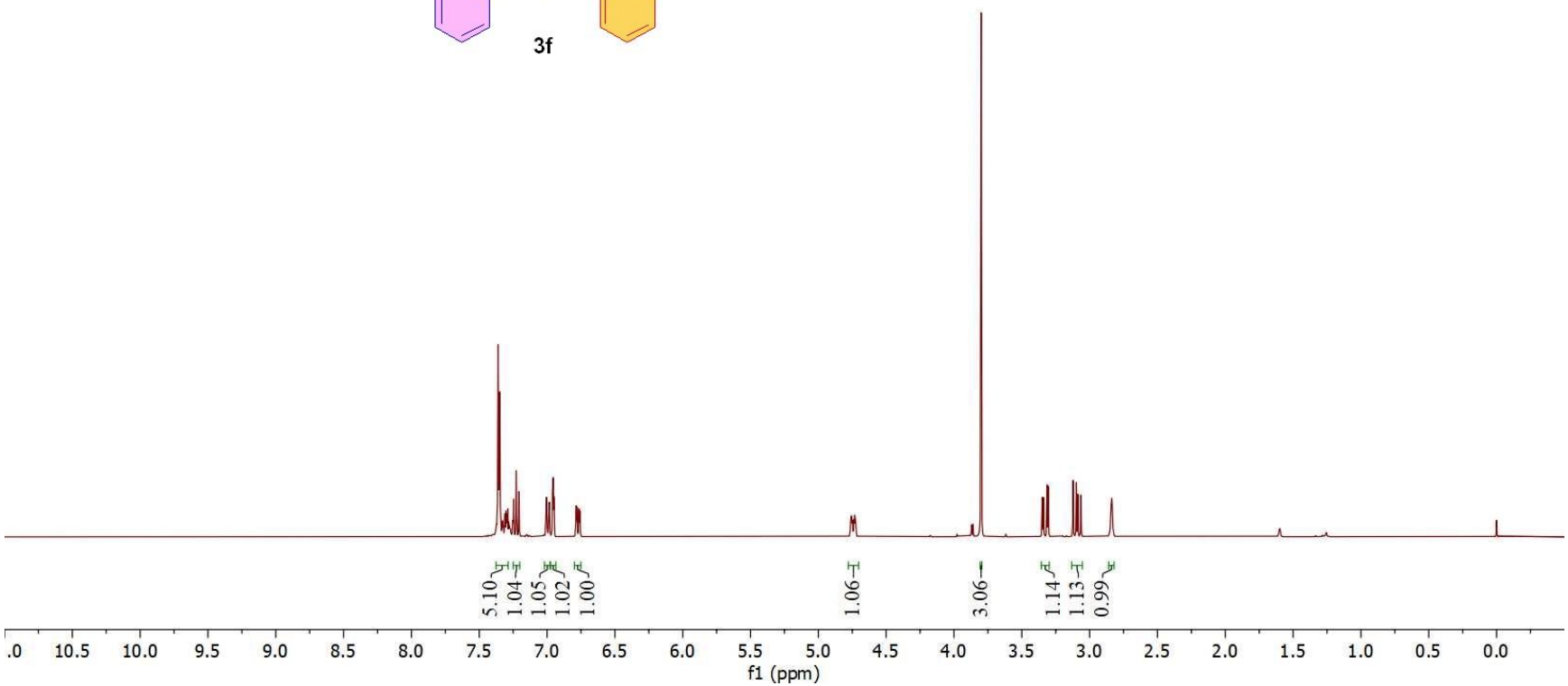
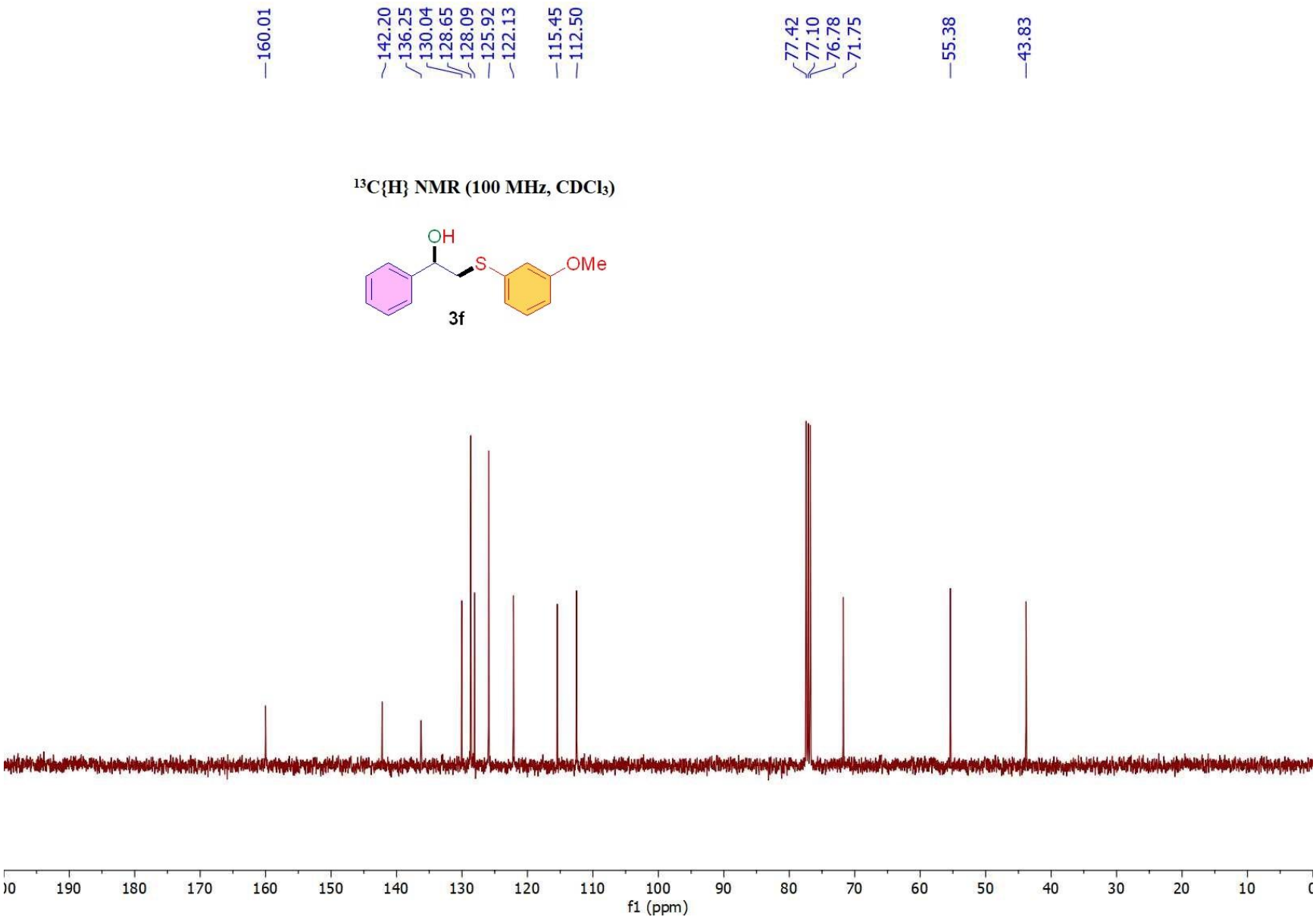
^{1}H NMR (400 MHz, $CDCl_3$)



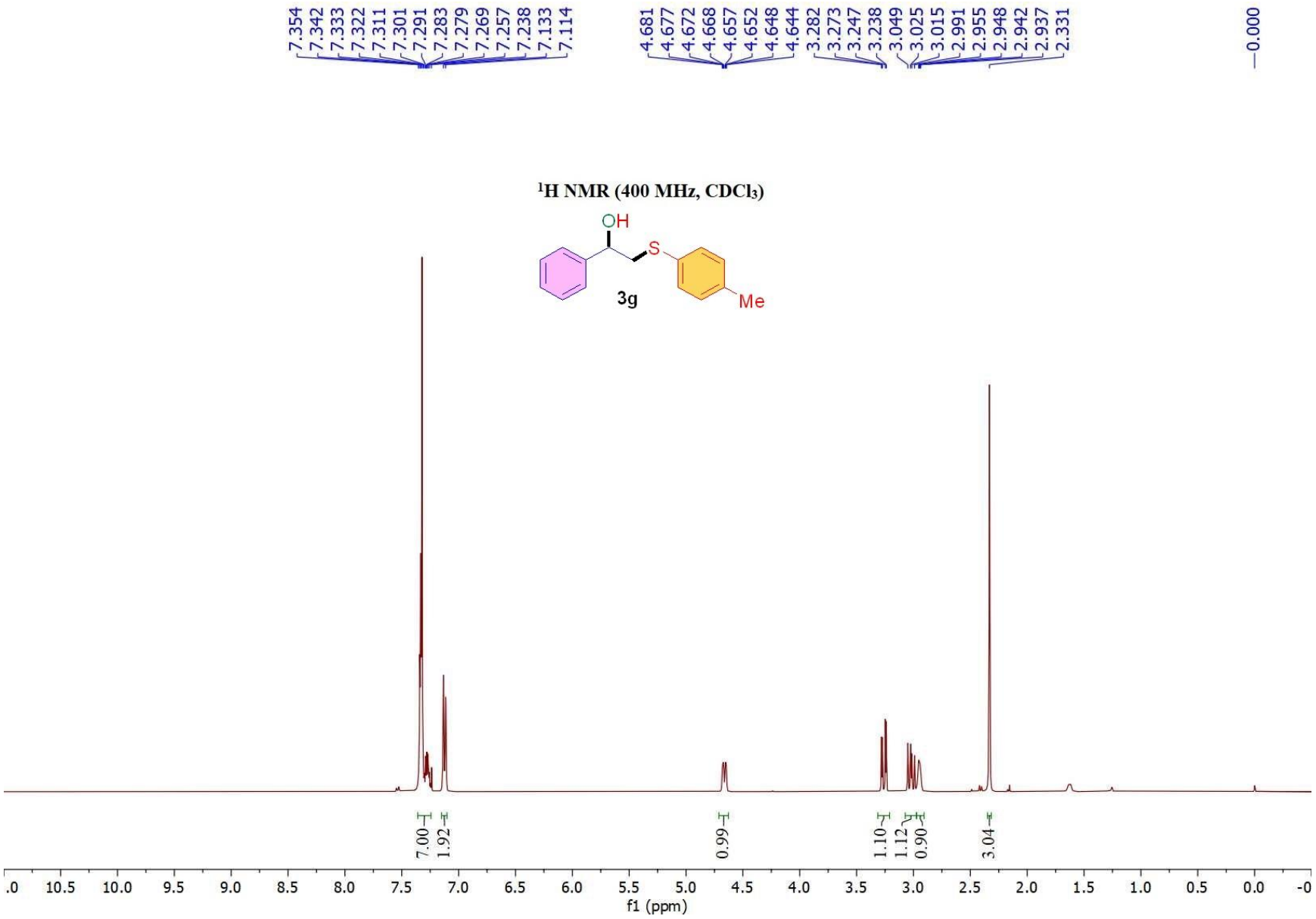
3f





$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)

3f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2.948
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1H NMR (400 MHz, CDCl3)
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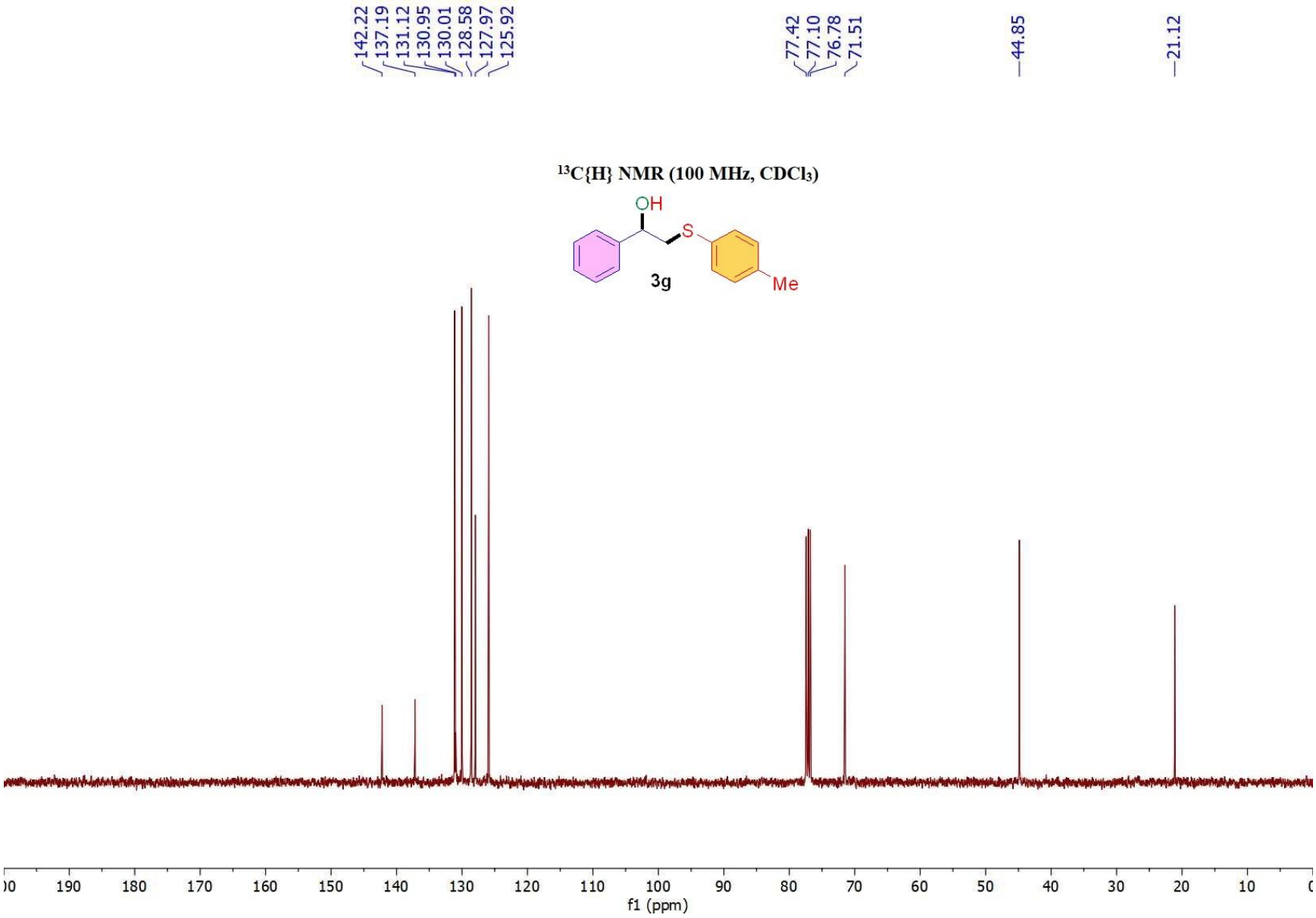
f1 (ppm)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21.12
13C{H} NMR (100 MHz, CDCl3)
OH
S
Me
3g
190
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140
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120
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80
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60
50
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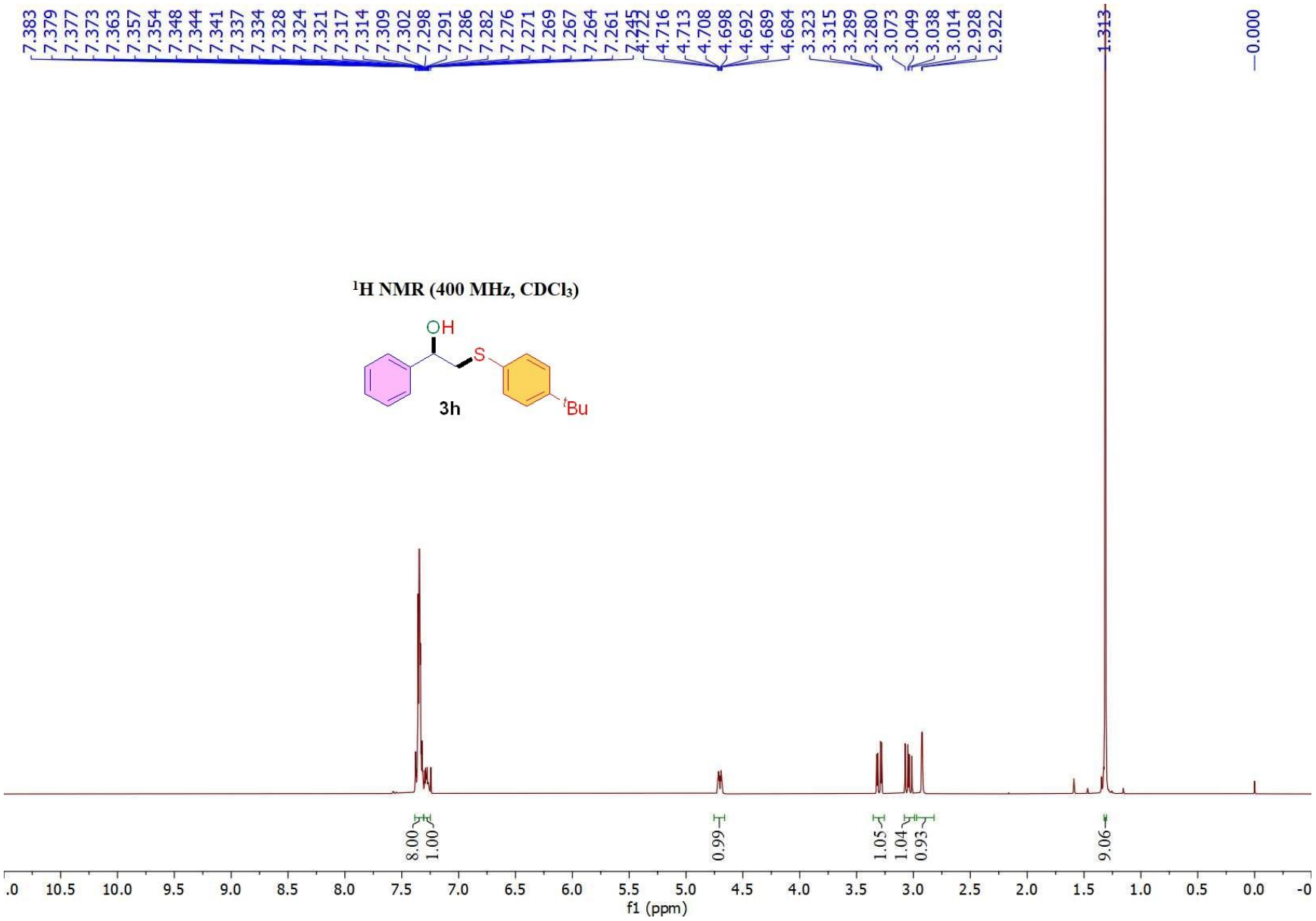
30
20
10
f1 (ppm)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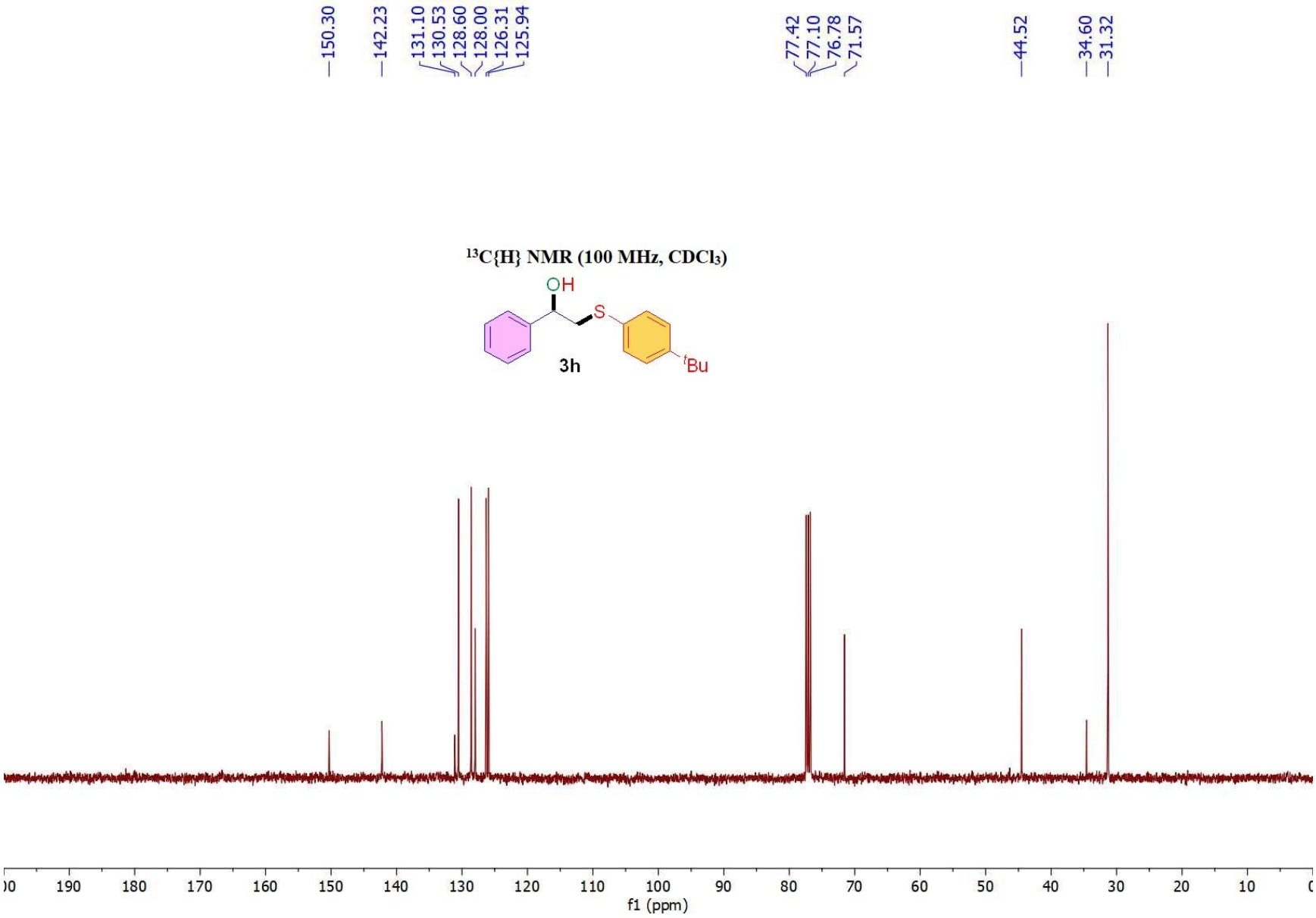
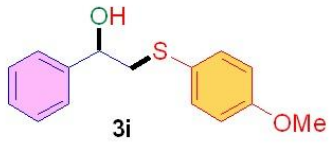
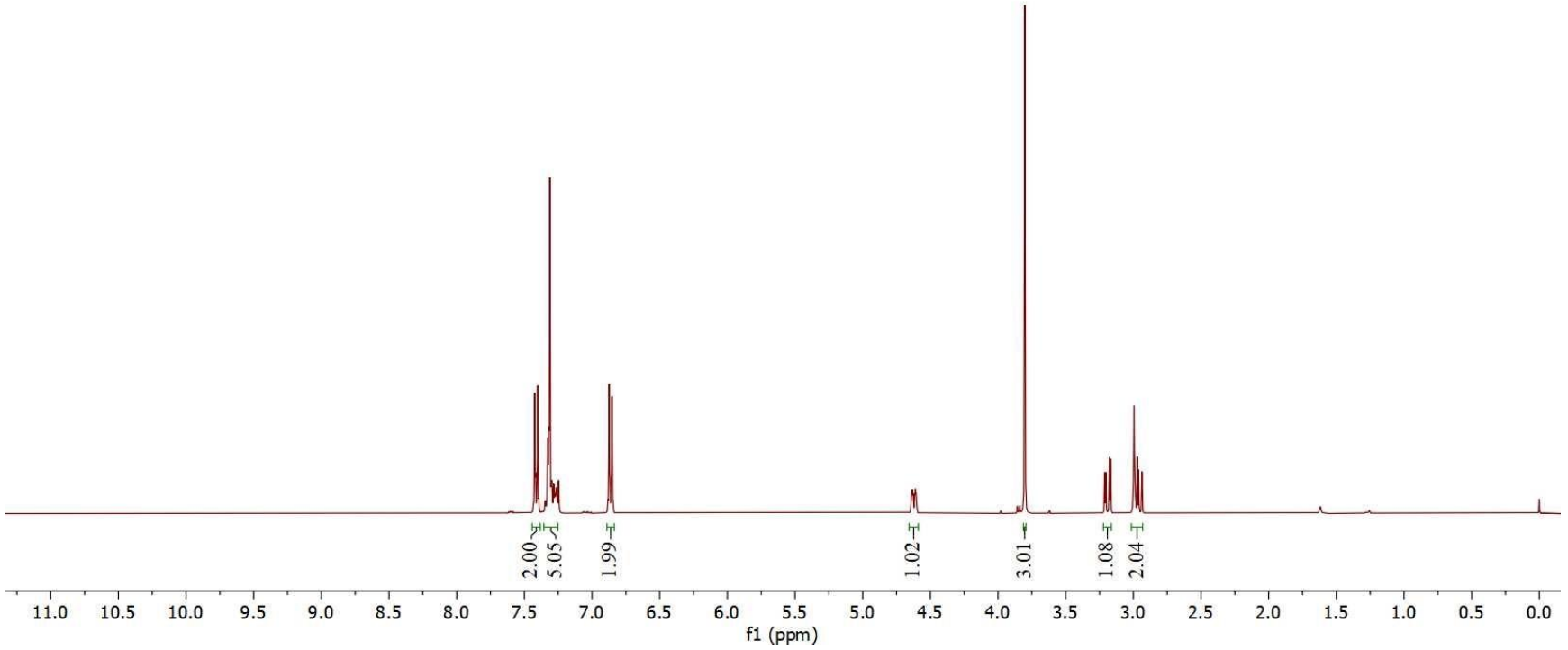
7.324
7.321
7.317
7.314
7.309
7.302
7.298
7.291
7.286
7.282
7.276
7.271
7.269
7.267
7.264
7.261
4.716
4.713
4.708
4.698
4.692
4.689
4.684
3.323
3.315
3.289
3.280
3.073
3.049
3.038
3.014
2.928
2.922
1.313
0.000
1H NMR (400 MHz, CDCl3)
OH
S
tBu
3h
8.00
1.00
0.99
1.05
1.04
0.93
9.06
f1 (ppm)

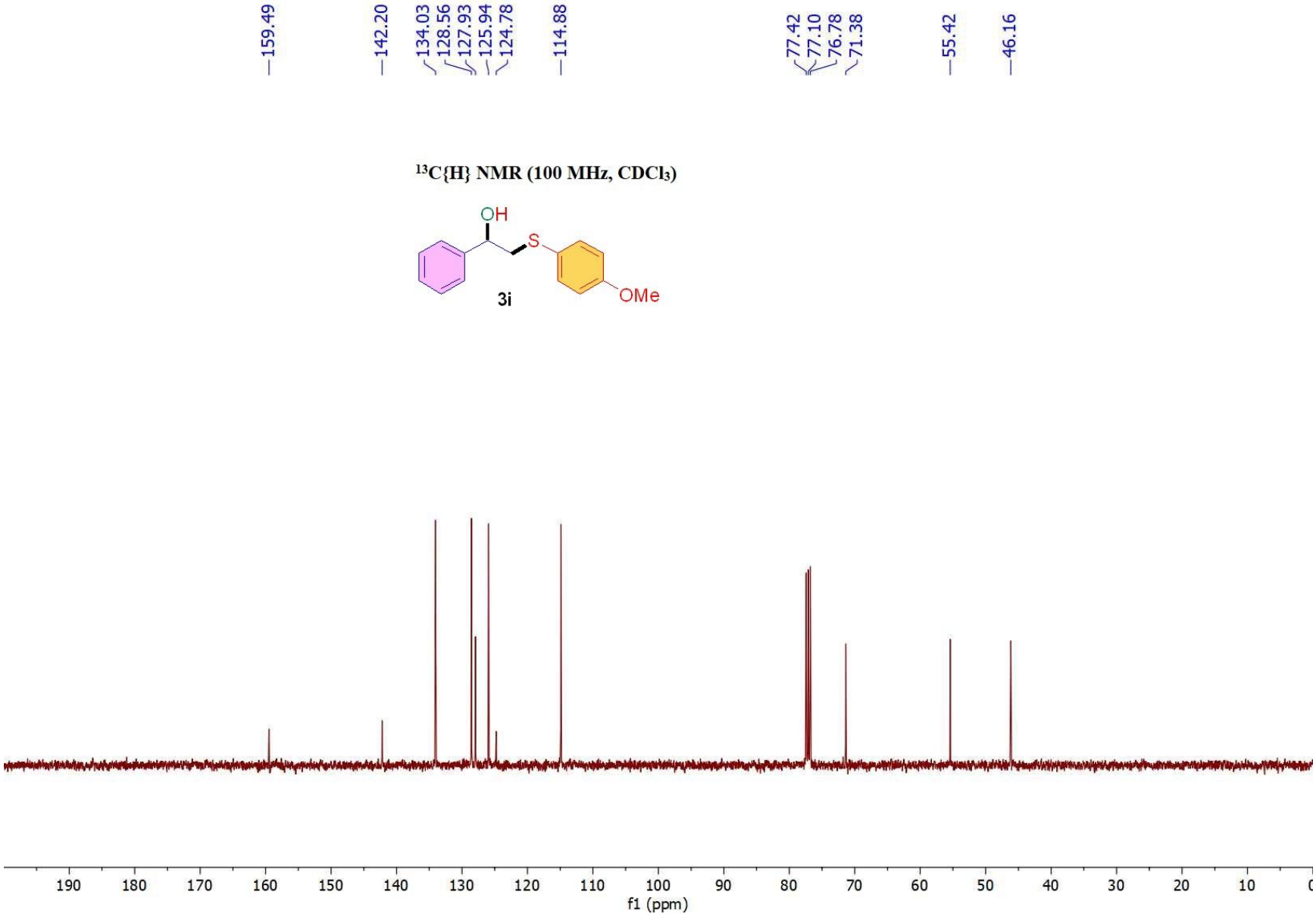

150.30
142.23
131.10
130.53
128.60
128.00
126.31
125.94
77.42
77.10
76.78
71.57
44.52
34.60
31.32
13C{H} NMR (100 MHz, CDCl3)
OH
S
tBu
3h
f1 (ppm)

^{1}H NMR (400 MHz, $CDCl_3$)



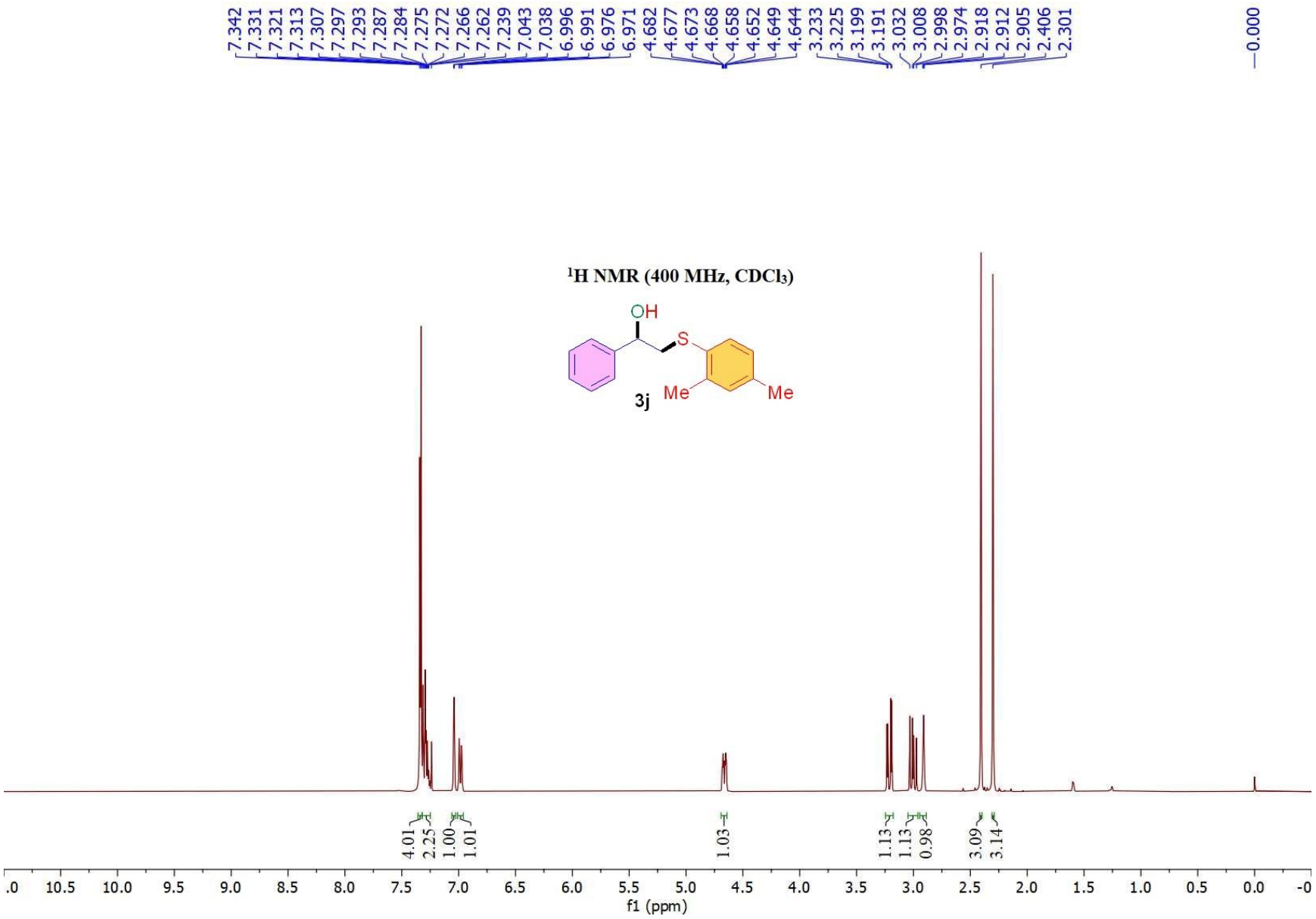
3i





$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)

3i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7.331
7.321
7.313
7.307
7.297
7.293
7.287
7.284
7.275
7.272
7.266
7.262
7.239
7.043
7.038
6.996
6.991
6.976
6.971
4.682
4.677
4.673
4.668
4.658
4.652
4.649
4.644
3.233
3.225
3.199
3.191
3.032
3.008
2.998
2.974
2.918
2.912
2.905
2.406
2.301
0.000
1H NMR (400 MHz, CDCl3)
OH
S
Me
Me
3j
4.01
2.25
1.00
1.01
1.03
1.13
1.13
0.98
3.09
3.14
f1 (ppm)

142.28
139.11
137.12
131.51
130.92
130.14
128.60
127.99
127.45
125.92

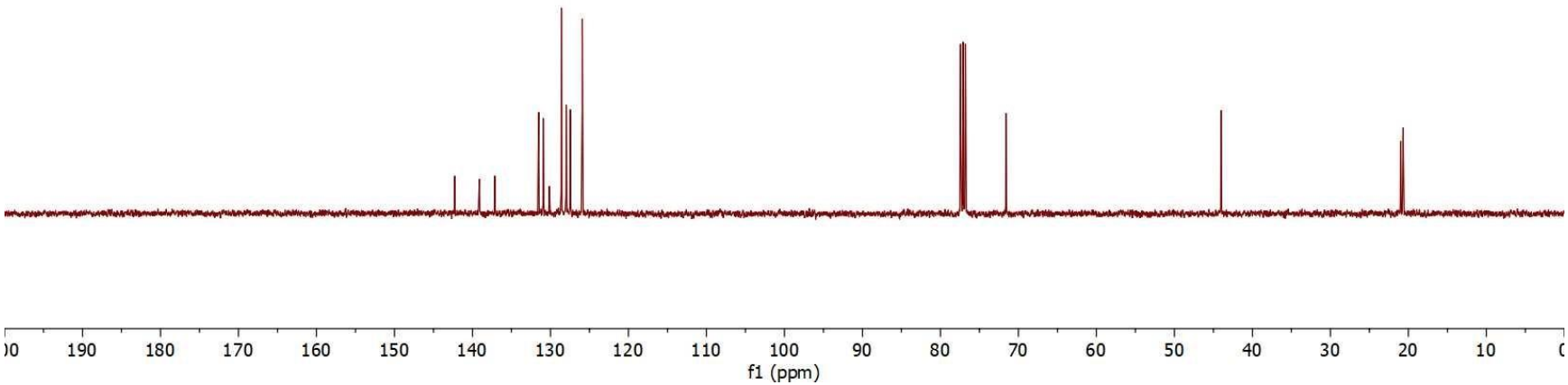
77.42
77.10
76.78
71.60

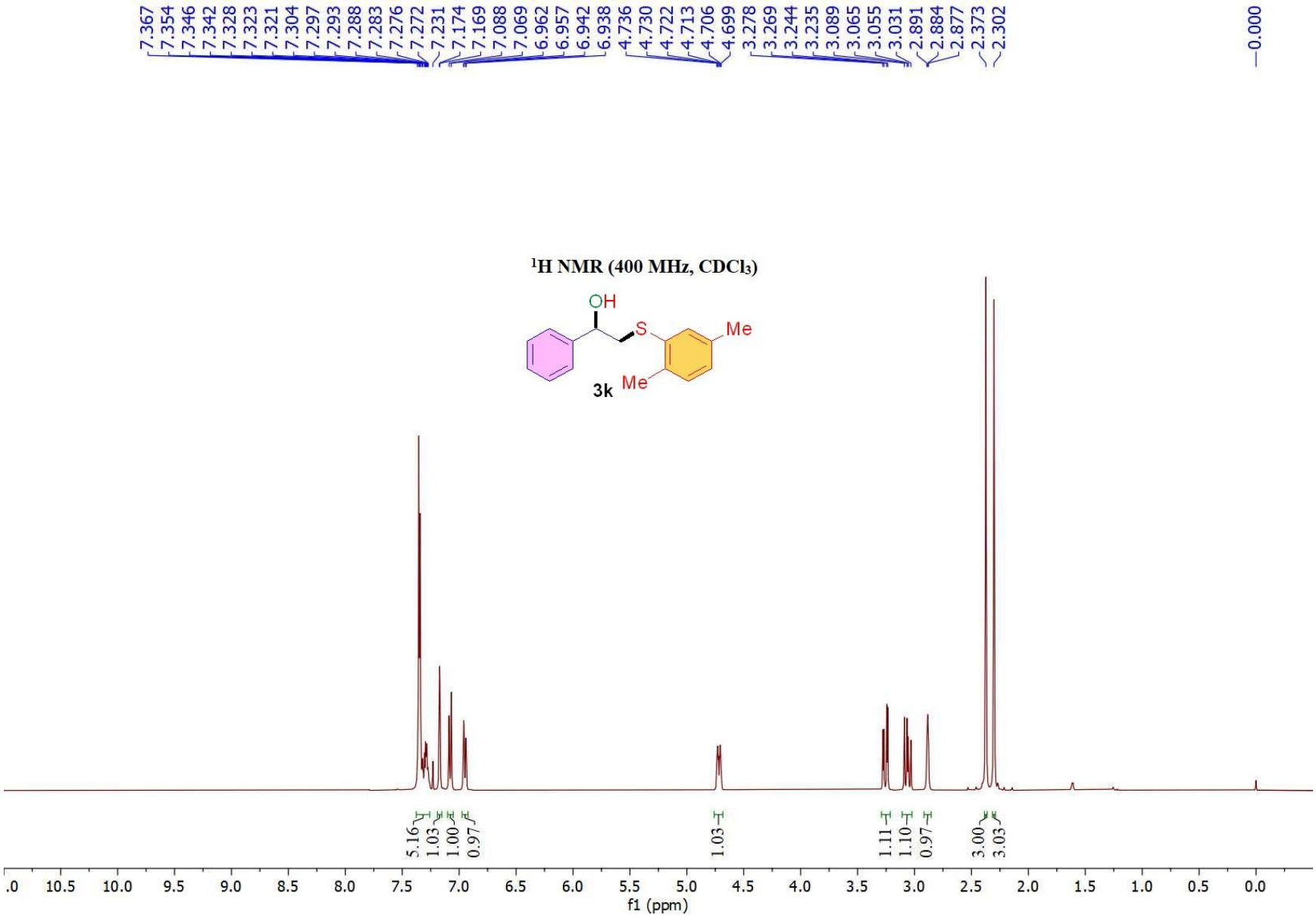
44.02

21.00
20.67

$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)

3j

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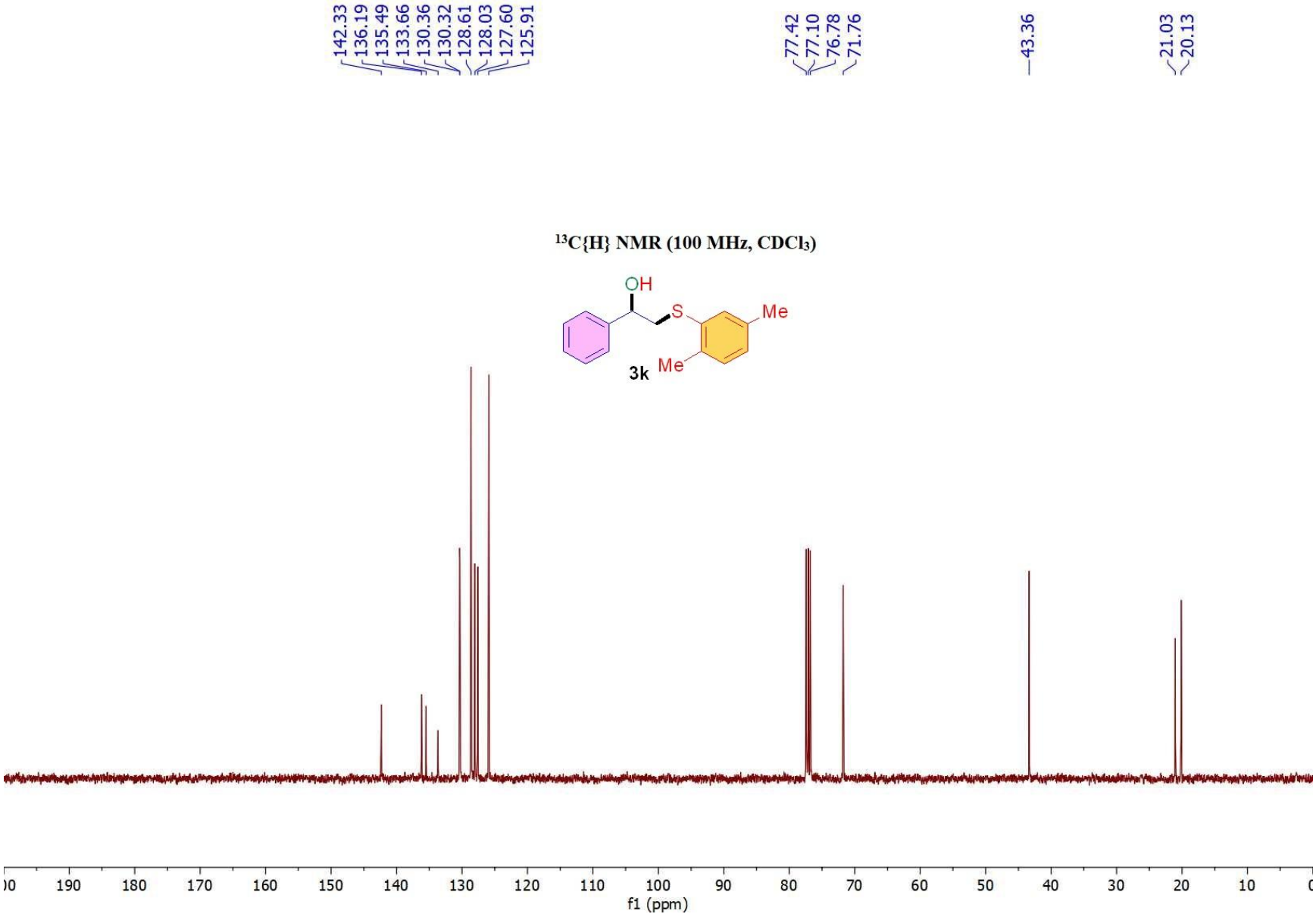
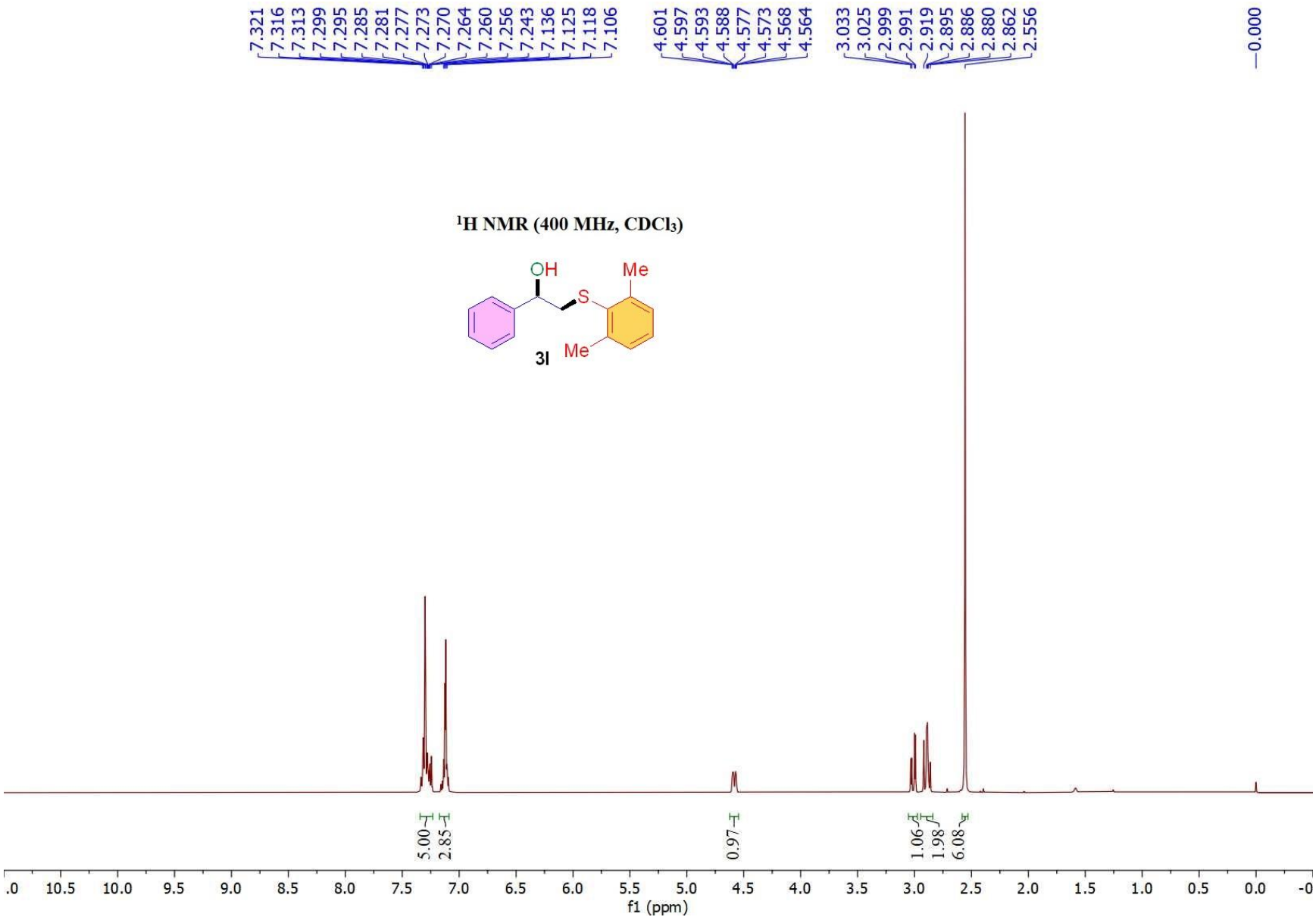
7.354
7.346
7.342
7.328
7.323
7.321
7.304
7.297
7.293
7.288
7.283
7.276
7.272
7.231
7.174
7.169
7.088
7.069
6.962
6.957
6.942
6.938
4.736
4.730
4.722
4.713
4.706
4.699
3.278
3.269
3.244
3.235
3.089
3.065
3.055
3.031
2.891
2.884
2.877
2.373
2.302
0.000
1H NMR (400 MHz, CDCl3)
OH
S
Me
Me
3k
5.16
1.03
1.00
0.97
1.03
1.11
1.10
0.97
3.00
3.03
f1 (ppm)

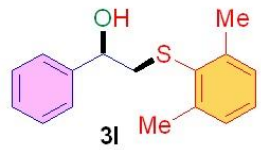


$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)

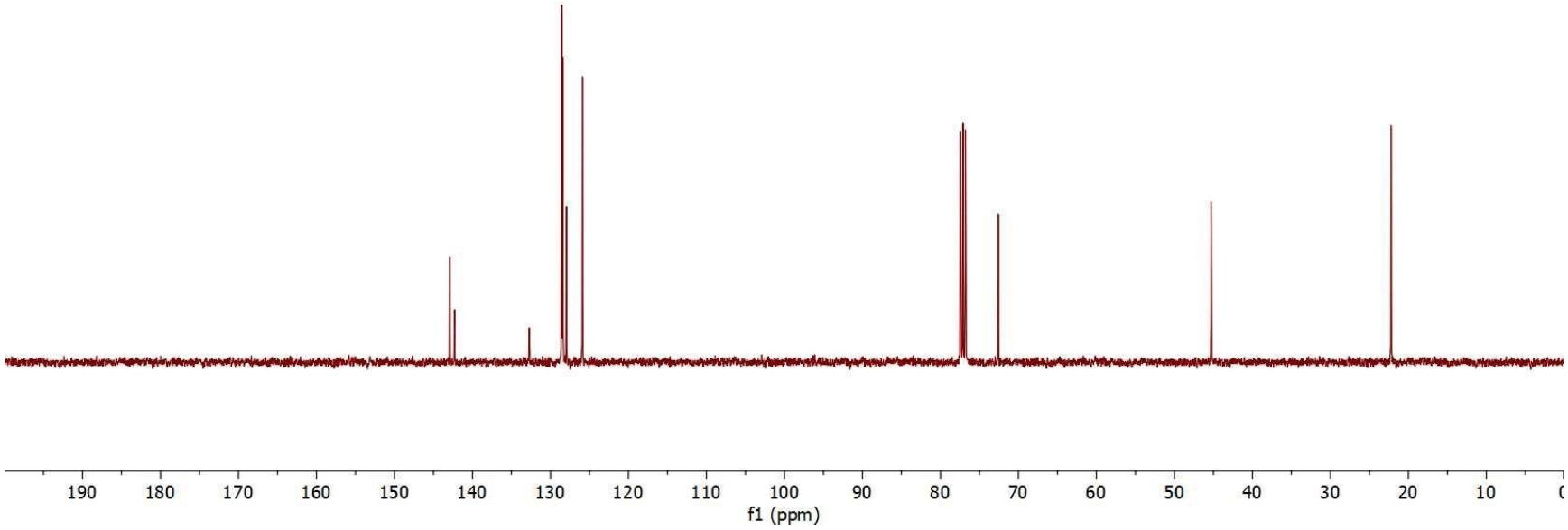

7.321
7.316
7.313
7.299
7.295
7.285
7.281
7.277
7.273
7.270
7.264
7.260
7.256
7.243
7.136
7.125
7.118
7.106
4.601
4.597
4.593
4.588
4.577
4.573
4.568
4.564
3.033
3.025
2.999
2.991
2.919
2.895
2.886
2.880
2.862
2.556
0.000
1H NMR (400 MHz, CDCl3)
OH
Me
S
Me
3l
5.00
2.85
0.97
1.06
1.98
6.08
f1 (ppm)

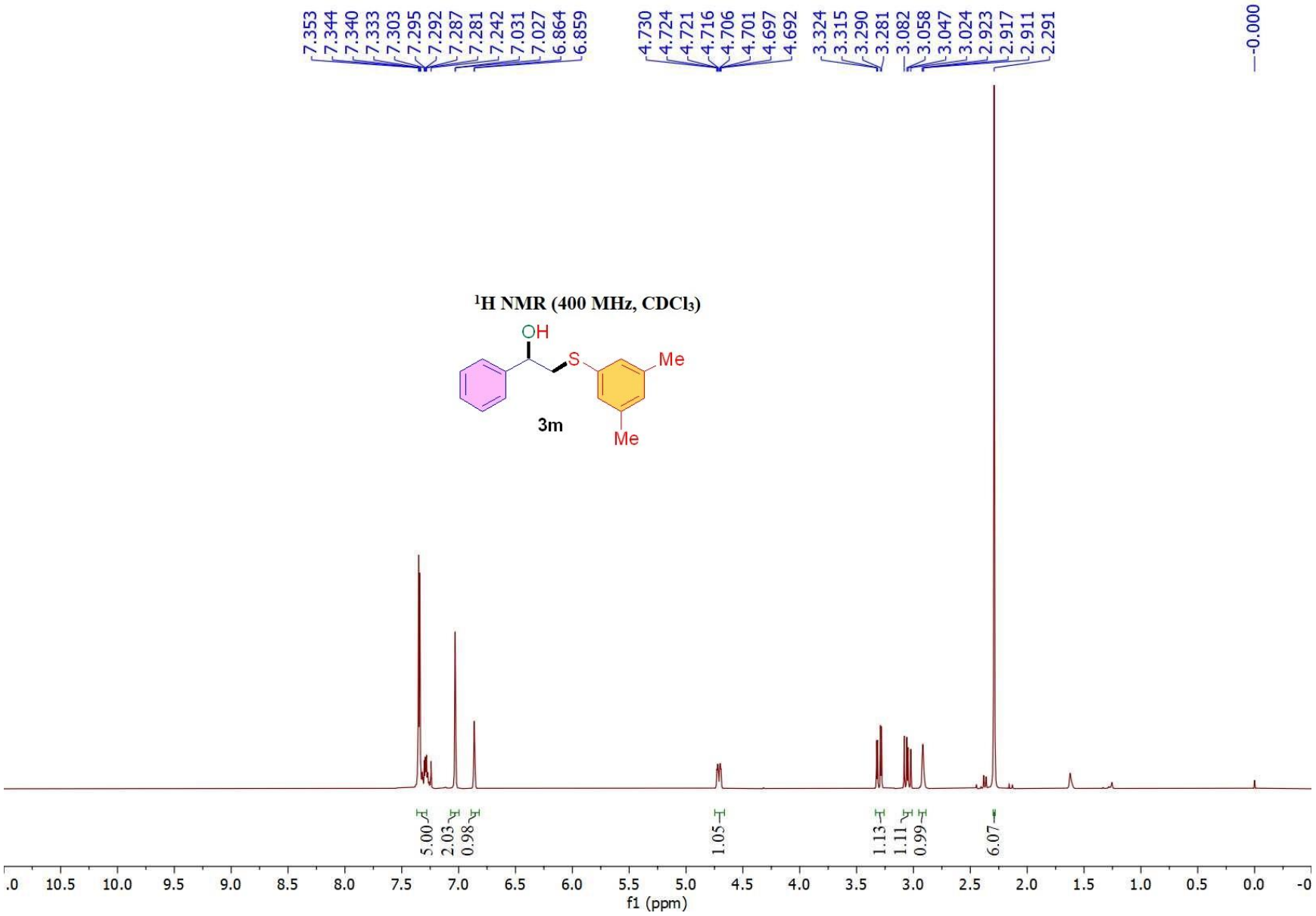
142.93
142.29
132.74
128.56
128.43
127.96
125.90
77.42
77.10
76.78
72.57
45.29
22.22

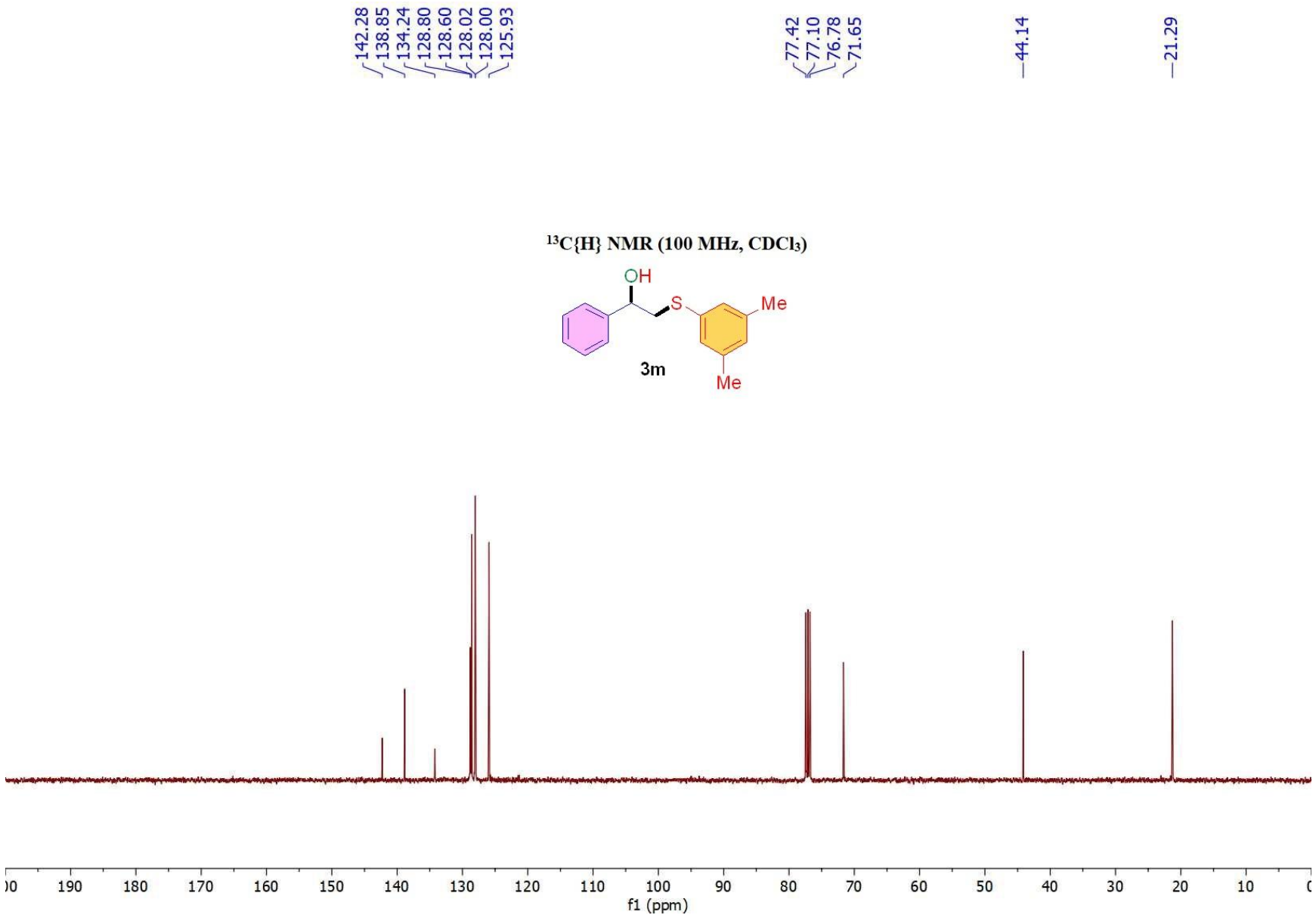
$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)



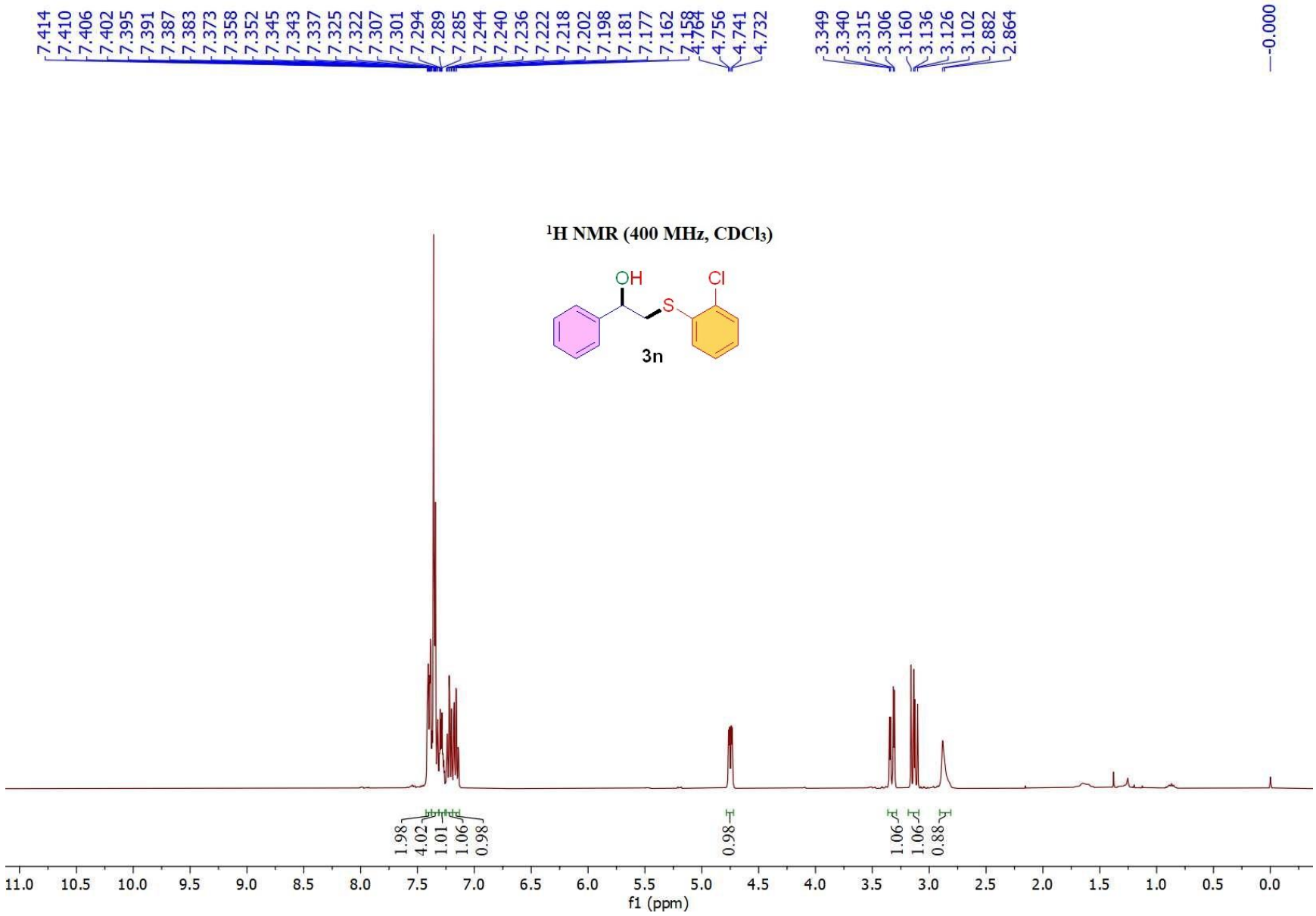
3l

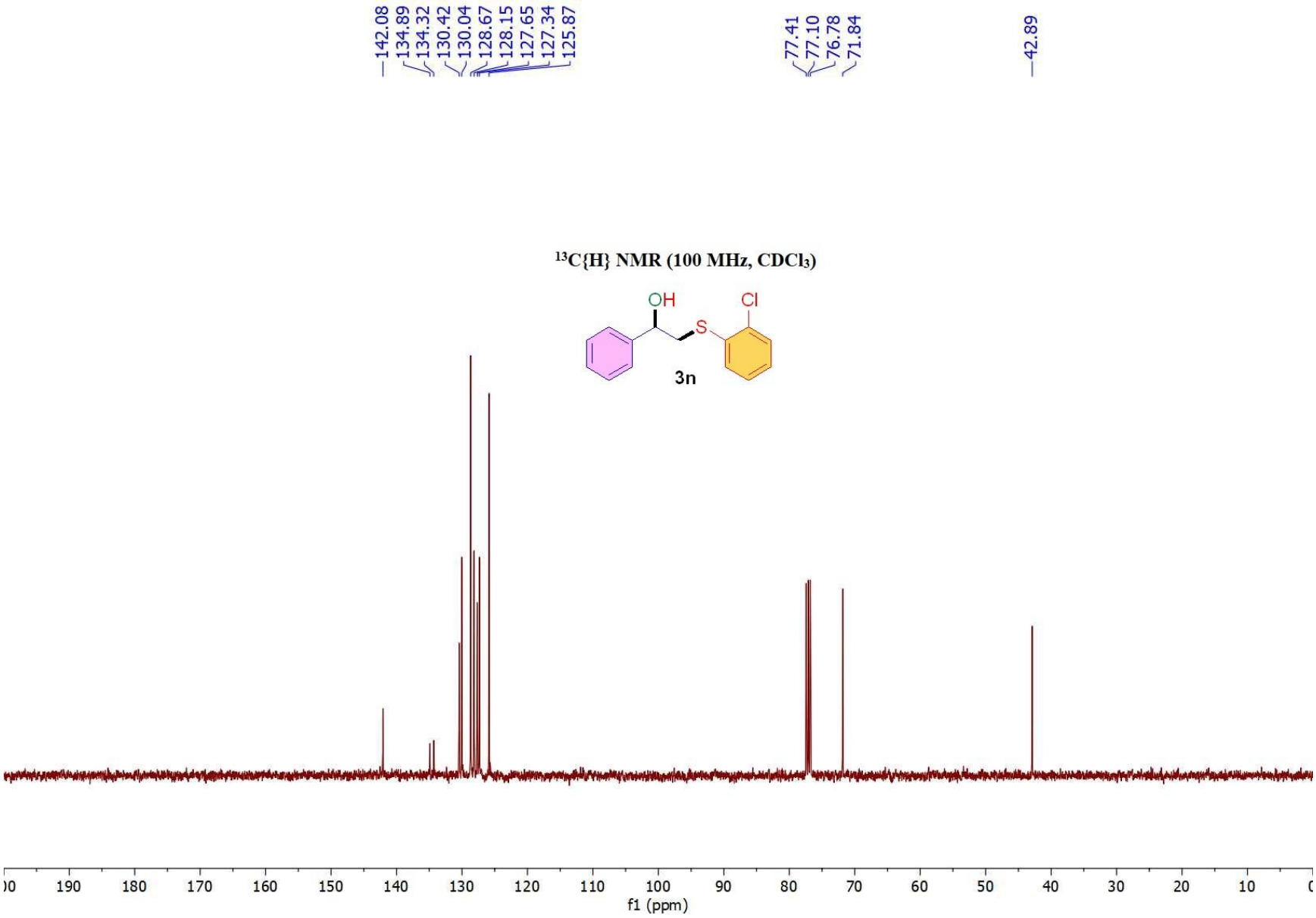



7.353
7.344
7.340
7.333
7.303
7.295
7.292
7.287
7.281
7.242
7.031
7.027
6.864
6.859
4.730
4.724
4.721
4.716
4.706
4.701
4.697
4.692
3.324
3.315
3.290
3.281
3.082
3.058
3.047
3.024
2.923
2.917
2.911
2.291
-0.000
1H NMR (400 MHz, CDCl3)
OH
S
Me
Me
3m
5.00
2.03
0.98
1.05
1.13
1.11
0.99
6.07
f1 (ppm)



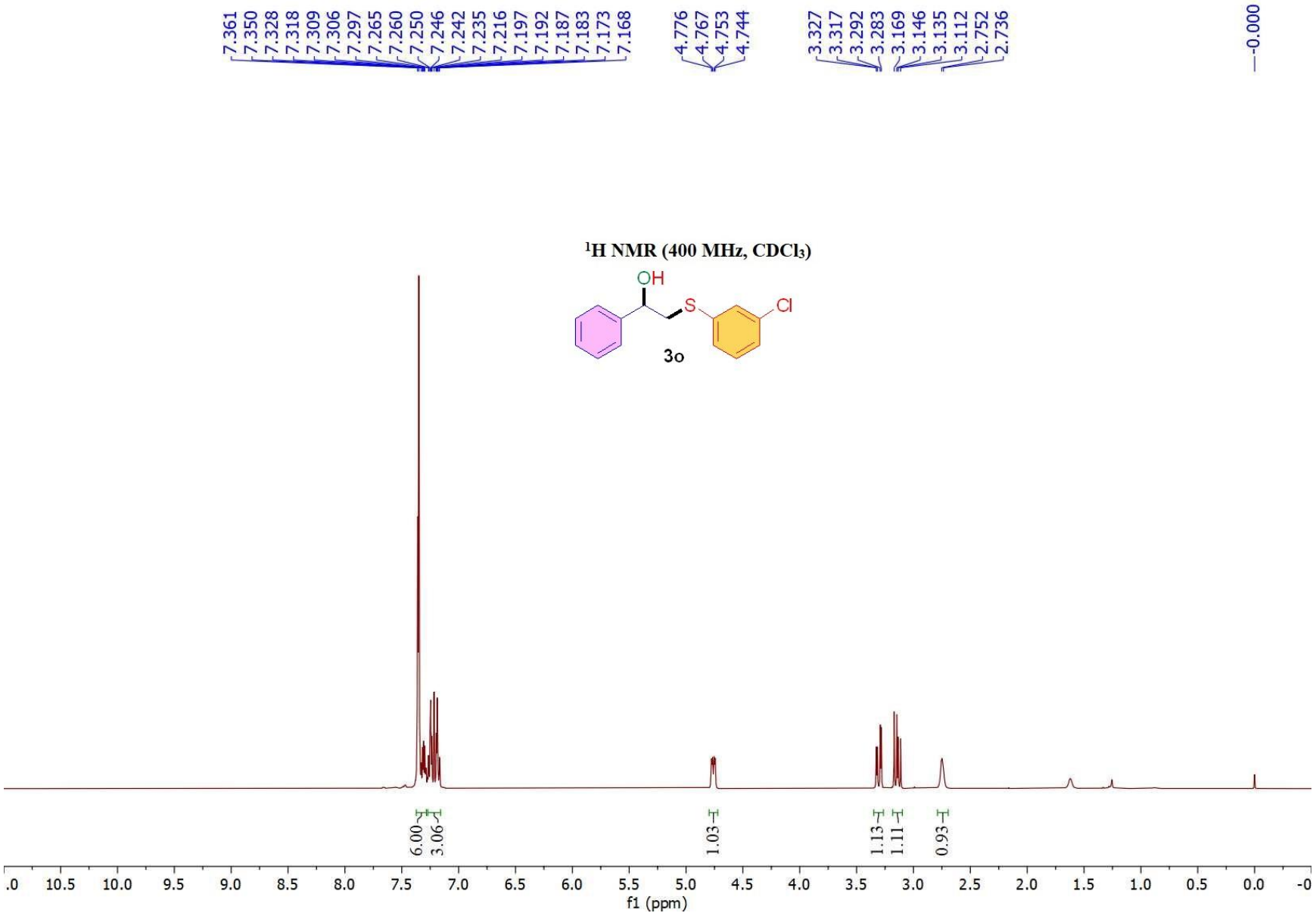
$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)

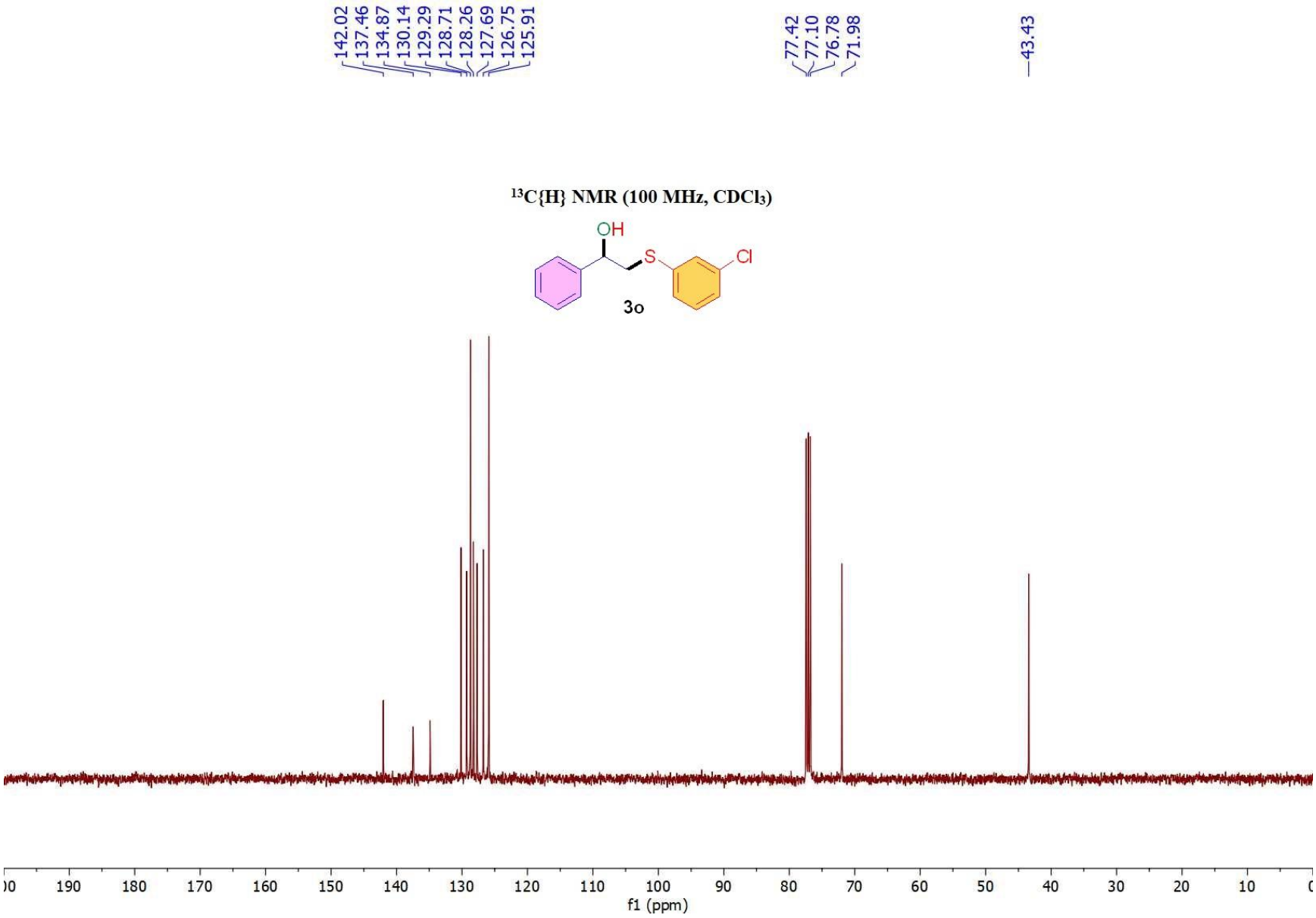

7.414
7.410
7.406
7.402
7.395
7.391
7.387
7.383
7.373
7.358
7.352
7.345
7.343
7.337
7.325
7.322
7.307
7.301
7.294
7.289
7.285
7.244
7.240
7.236
7.222
7.218
7.202
7.198
7.181
7.177
7.162
7.158
4.764
4.756
4.741
4.732
3.349
3.340
3.315
3.306
3.160
3.136
3.126
3.102
2.882
2.864
-0.000
1H NMR (400 MHz, CDCl3)
OH
Cl
S
3n
1.98
4.02
1.01
1.06
0.98
0.98
1.06
1.06
0.88
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)



$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)

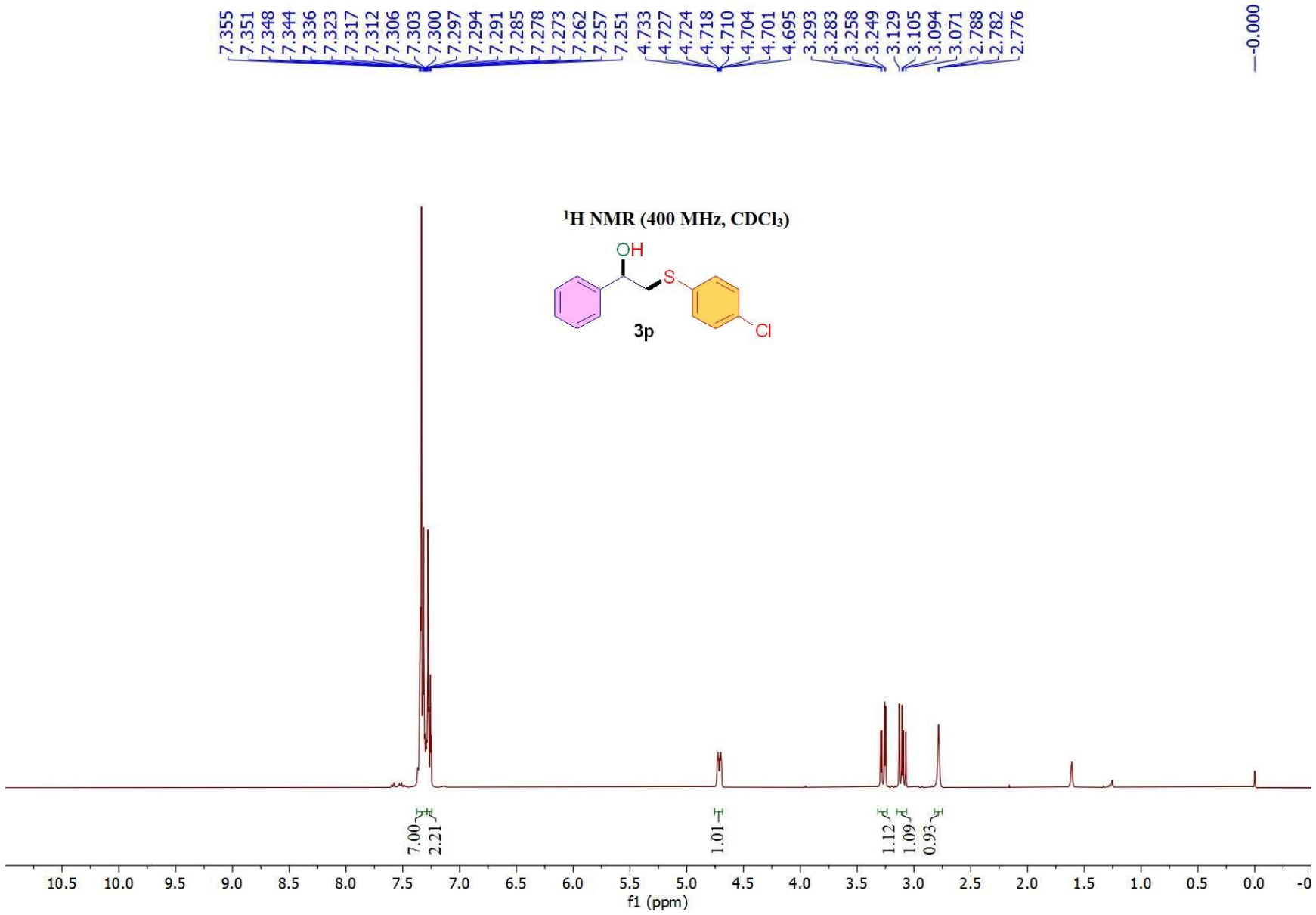
3n

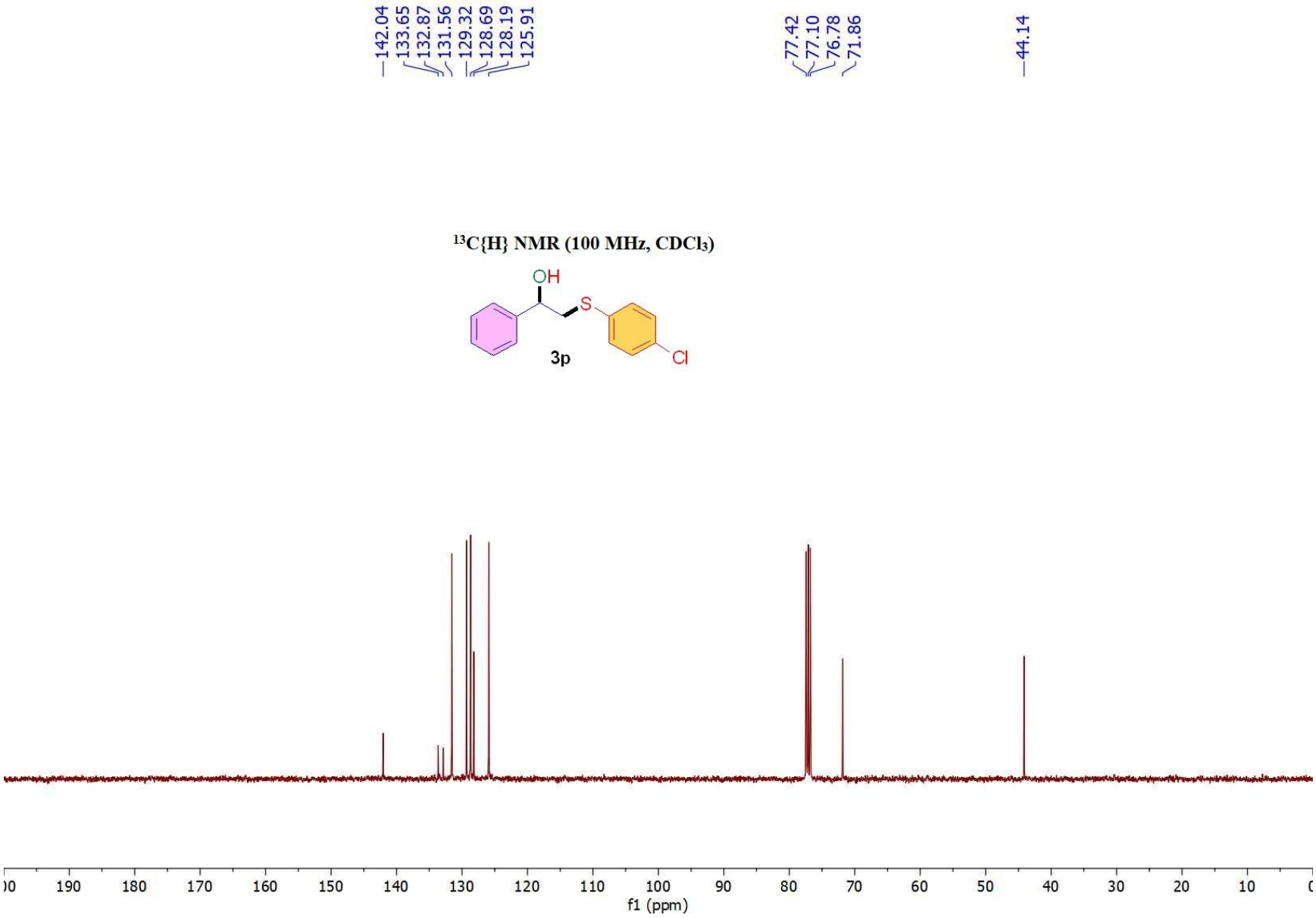

7.361
7.350
7.328
7.318
7.309
7.306
7.297
7.265
7.260
7.250
7.246
7.242
7.235
7.216
7.197
7.192
7.187
7.183
7.173
7.168
4.776
4.767
4.753
4.744
3.327
3.317
3.292
3.283
3.169
3.146
3.135
3.112
2.752
2.736
-0.000
1H NMR (400 MHz, CDCl3)
OH
S
Cl
3o
6.00
3.06
1.03
1.13
1.11
0.93
f1 (ppm)



$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)

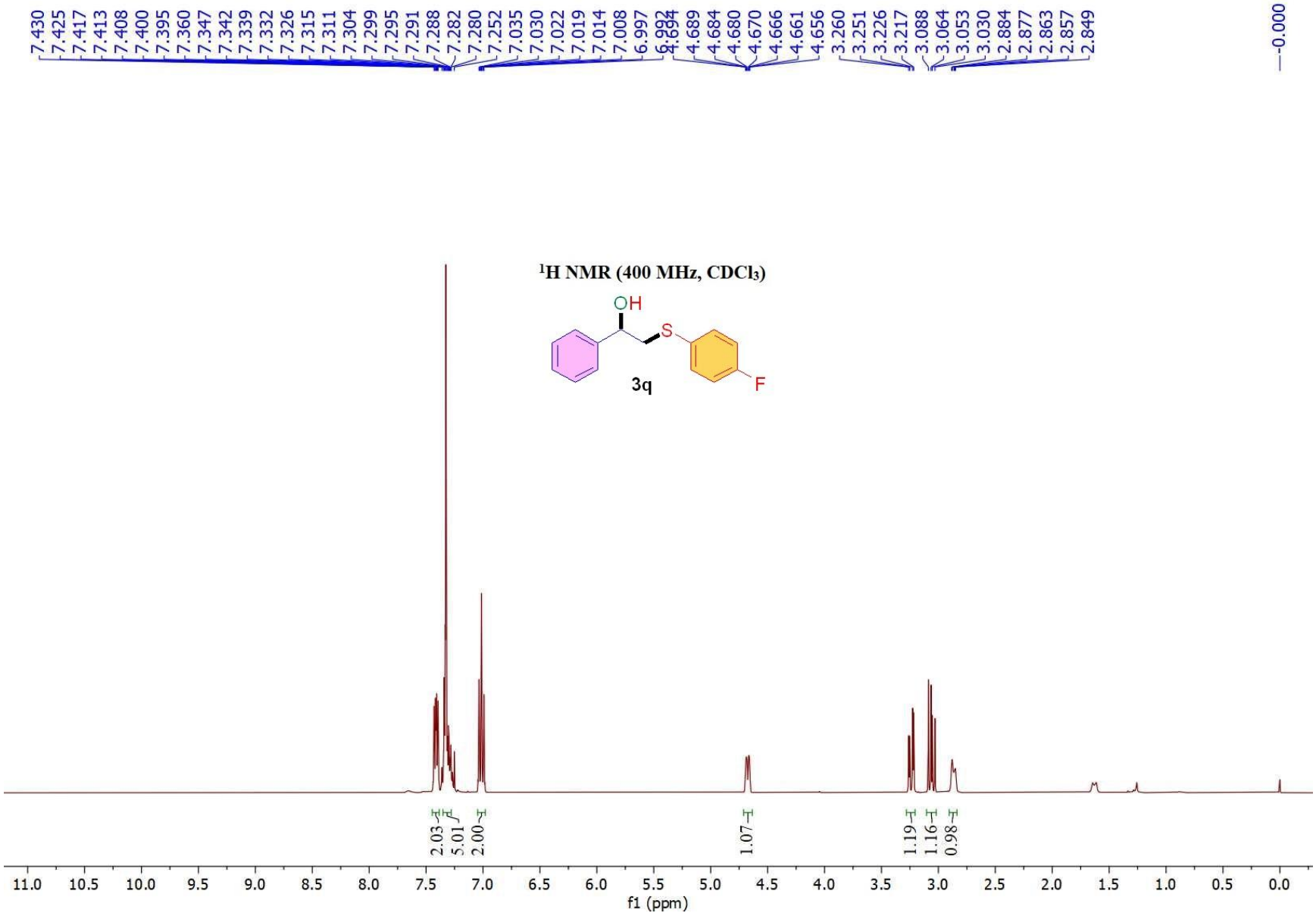
3o

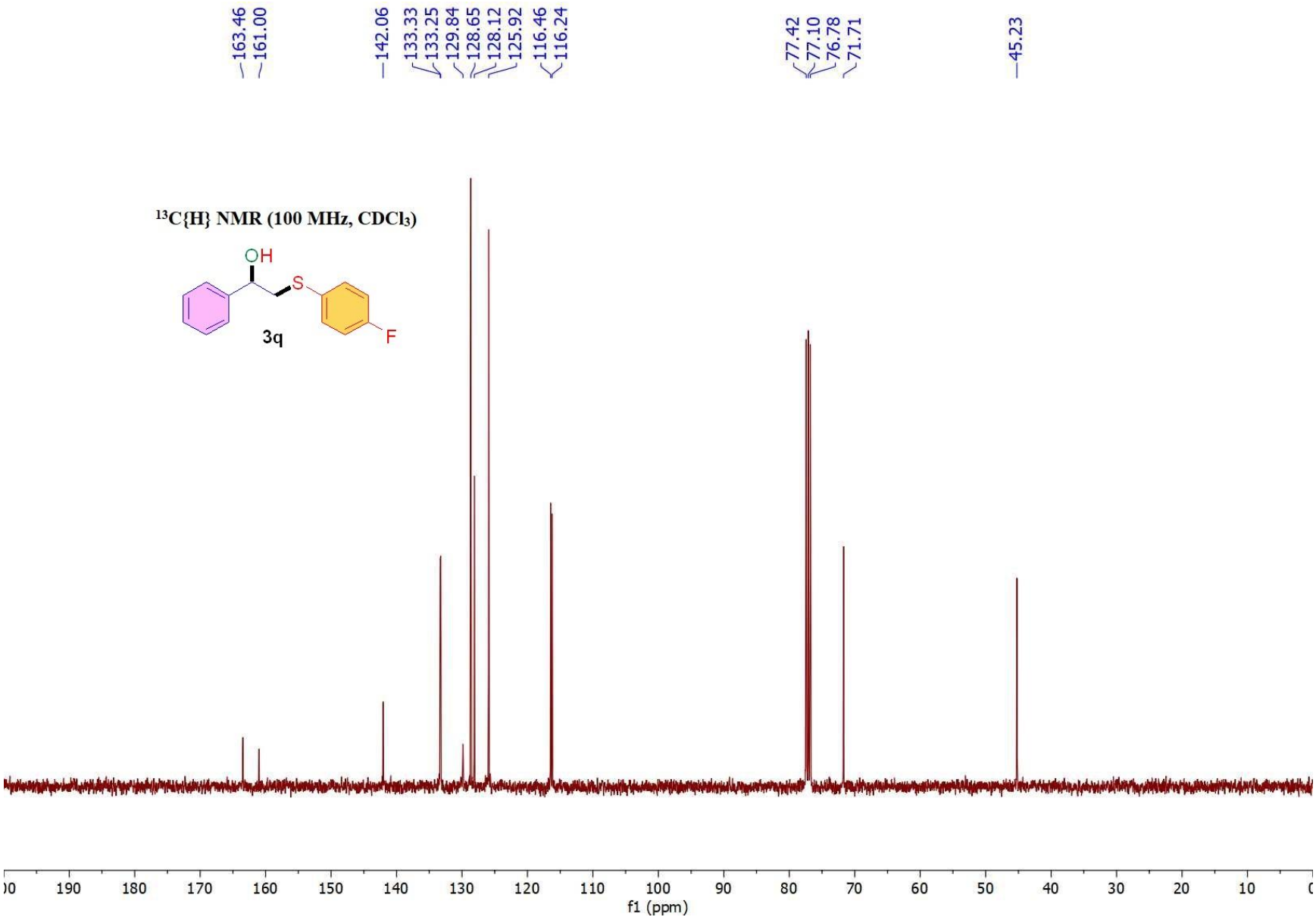

7.355
7.351
7.348
7.344
7.336
7.323
7.317
7.312
7.306
7.303
7.300
7.297
7.294
7.291
7.285
7.278
7.273
7.262
7.257
7.251
4.733
4.727
4.724
4.718
4.710
4.704
4.701
4.695
3.293
3.283
3.258
3.249
3.129
3.105
3.094
3.071
2.788
2.782
2.776
-0.000
1H NMR (400 MHz, CDCl3)
OH
S
Cl
3p
7.00
2.21
1.01
1.12
1.09
0.93
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)

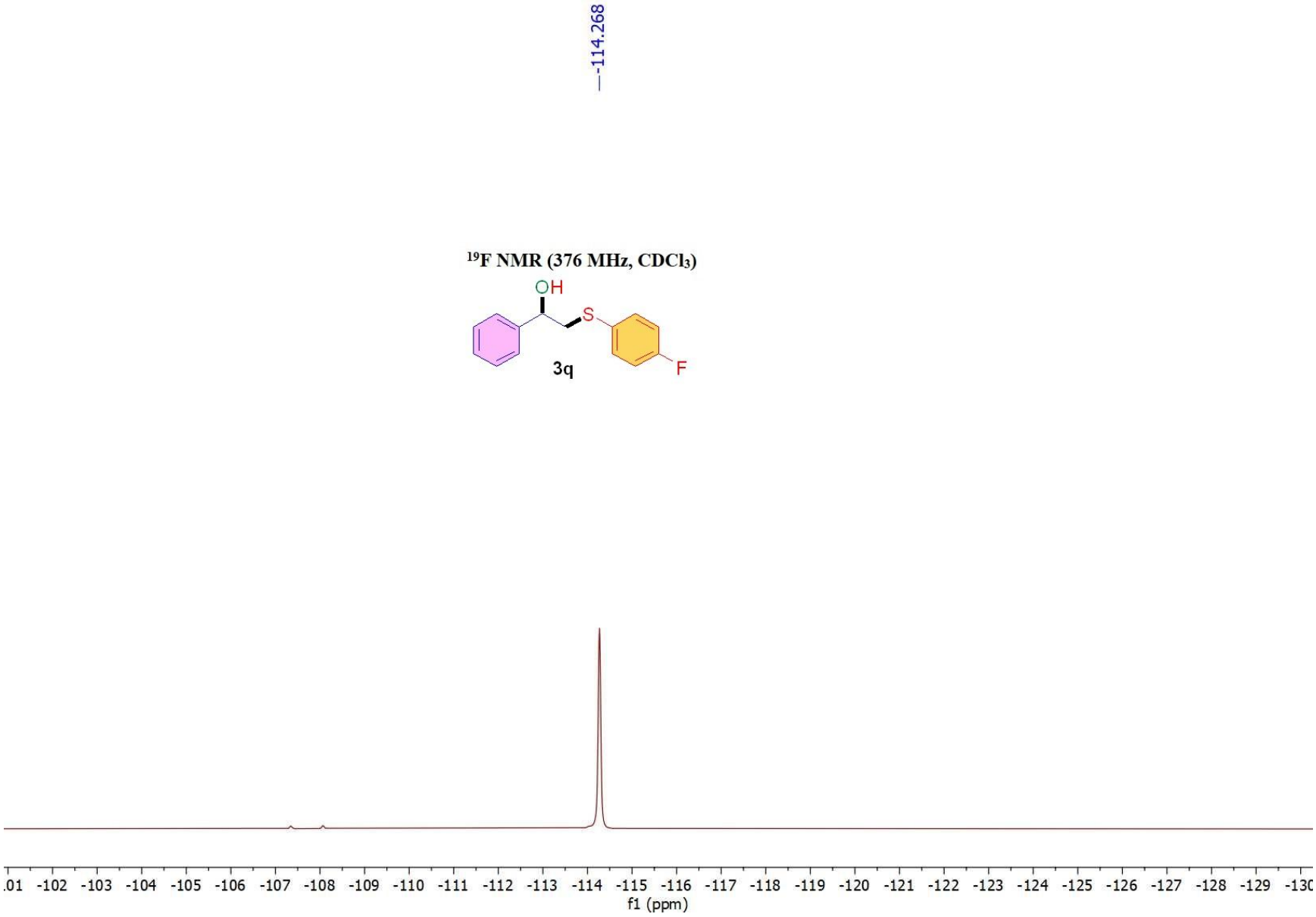


$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)

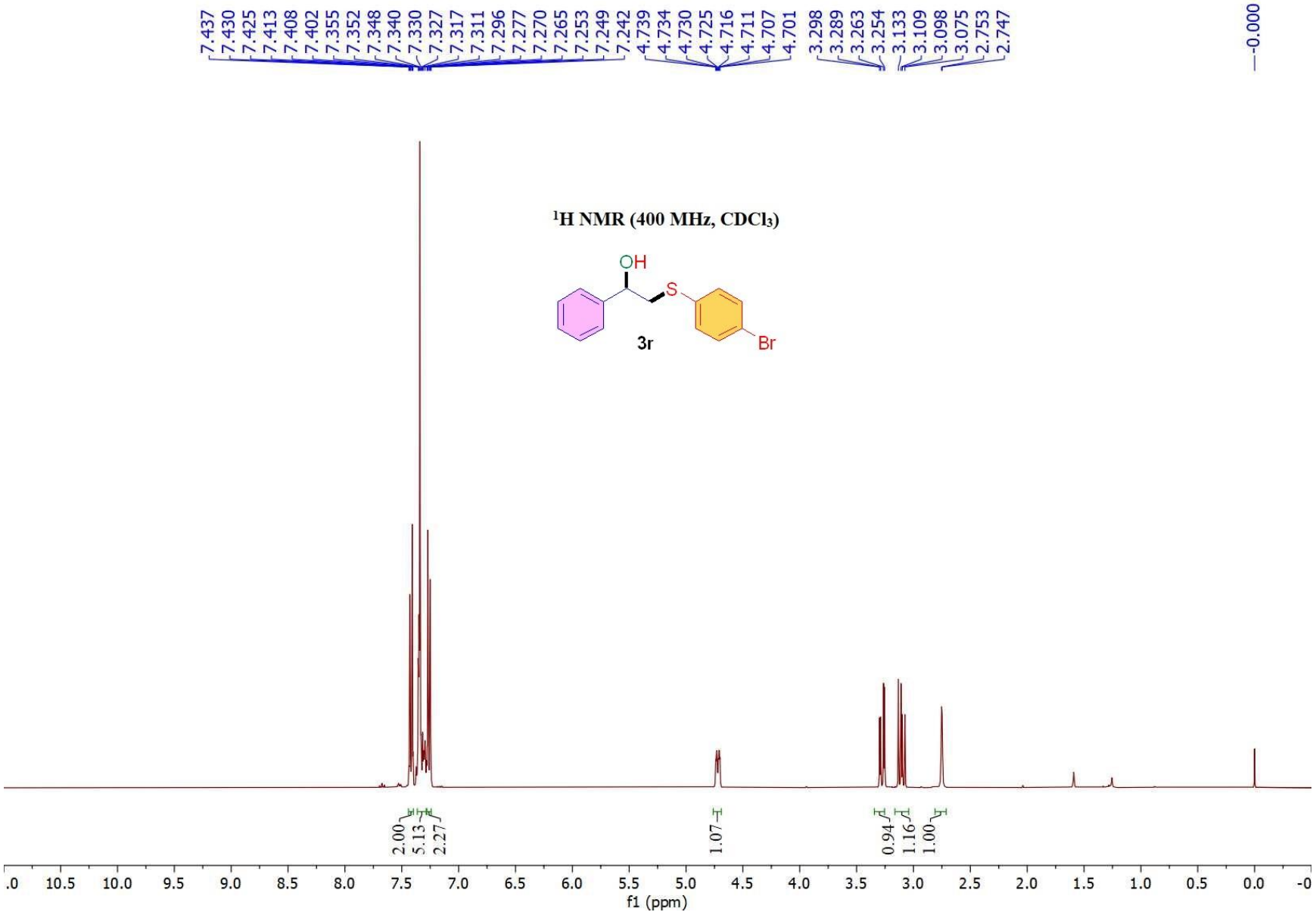
3p


7.430
7.425
7.417
7.413
7.408
7.400
7.395
7.360
7.347
7.342
7.339
7.332
7.326
7.315
7.311
7.304
7.299
7.295
7.291
7.288
7.282
7.280
7.252
7.035
7.030
7.022
7.019
7.014
7.008
6.997
6.992
4.694
4.689
4.684
4.680
4.670
4.666
4.661
4.656
3.260
3.251
3.226
3.217
3.088
3.064
3.053
3.030
2.884
2.877
2.863
2.857
2.849
-0.000
1H NMR (400 MHz, CDCl3)
OH
S
F
3q
2.03
5.01
2.00
1.07
1.19
1.16
0.98
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


163.46
161.00
142.06
133.33
133.25
129.84
128.65
128.12
125.92
116.46
116.24
77.42
77.10
76.78
71.71
45.23
13C{H} NMR (100 MHz, CDCl3)
OH
S
F
3q
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)



^{19}F NMR (376 MHz, $CDCl_3$)


7.437
7.430
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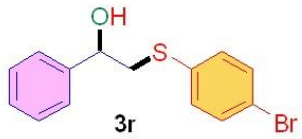
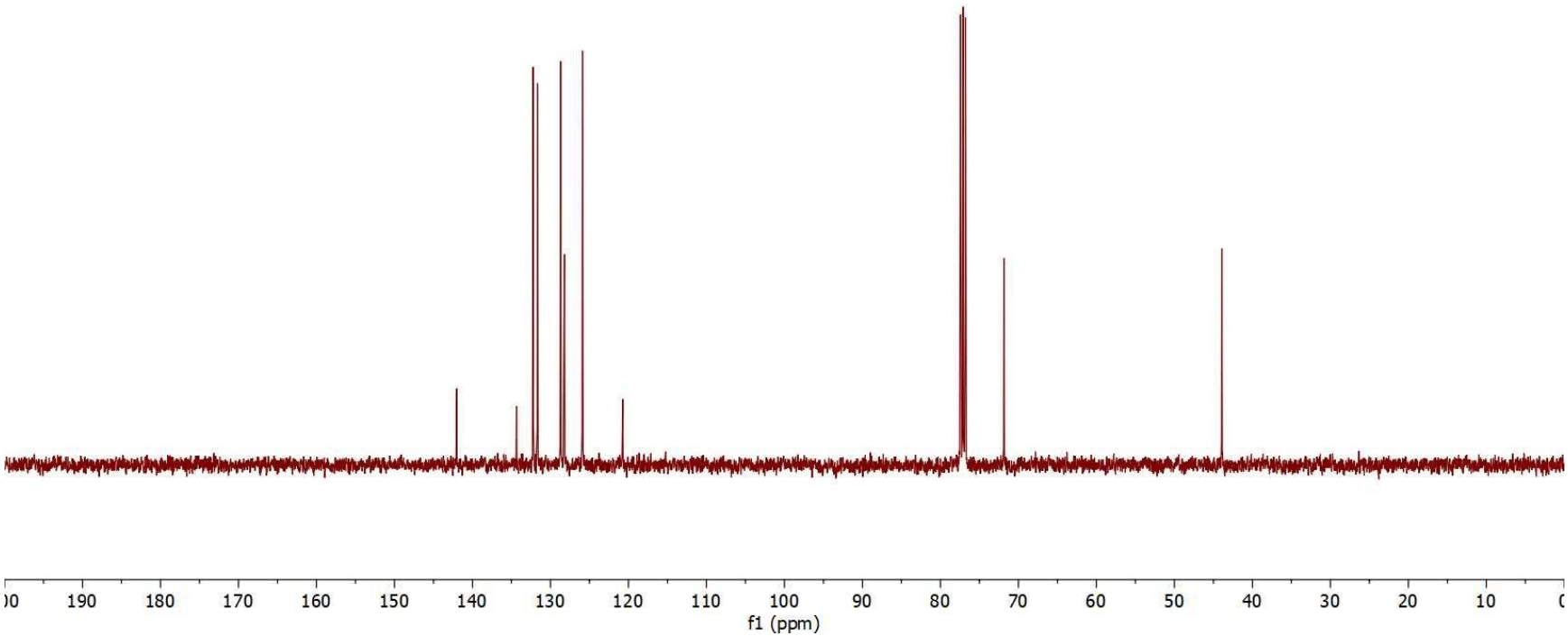
7.413
7.408
7.402
7.355
7.352
7.348
7.340
7.330
7.327
7.317
7.311
7.296
7.277
7.270
7.265
7.253
7.249
7.242
4.739
4.734
4.730
4.725
4.716
4.711
4.707
4.701
3.298
3.289
3.263
3.254
3.133
3.109
3.098
3.075
2.753
2.747
-0.000
1H NMR (400 MHz, CDCl3)
OH
S
Br
3r
2.00
5.13
2.27
1.07
0.94
1.16
1.00
f1 (ppm)

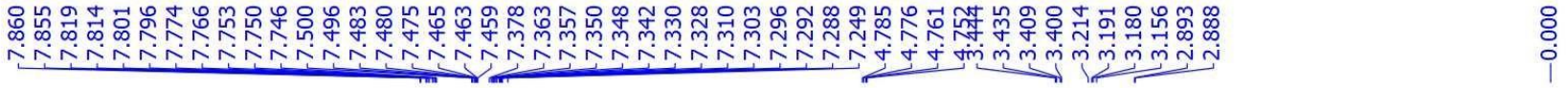
142.02
134.35
132.23
131.67
128.71
128.21
125.90
120.73
77.42
77.10
76.78
71.86
43.92

$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)

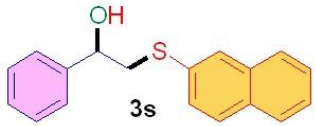


3r

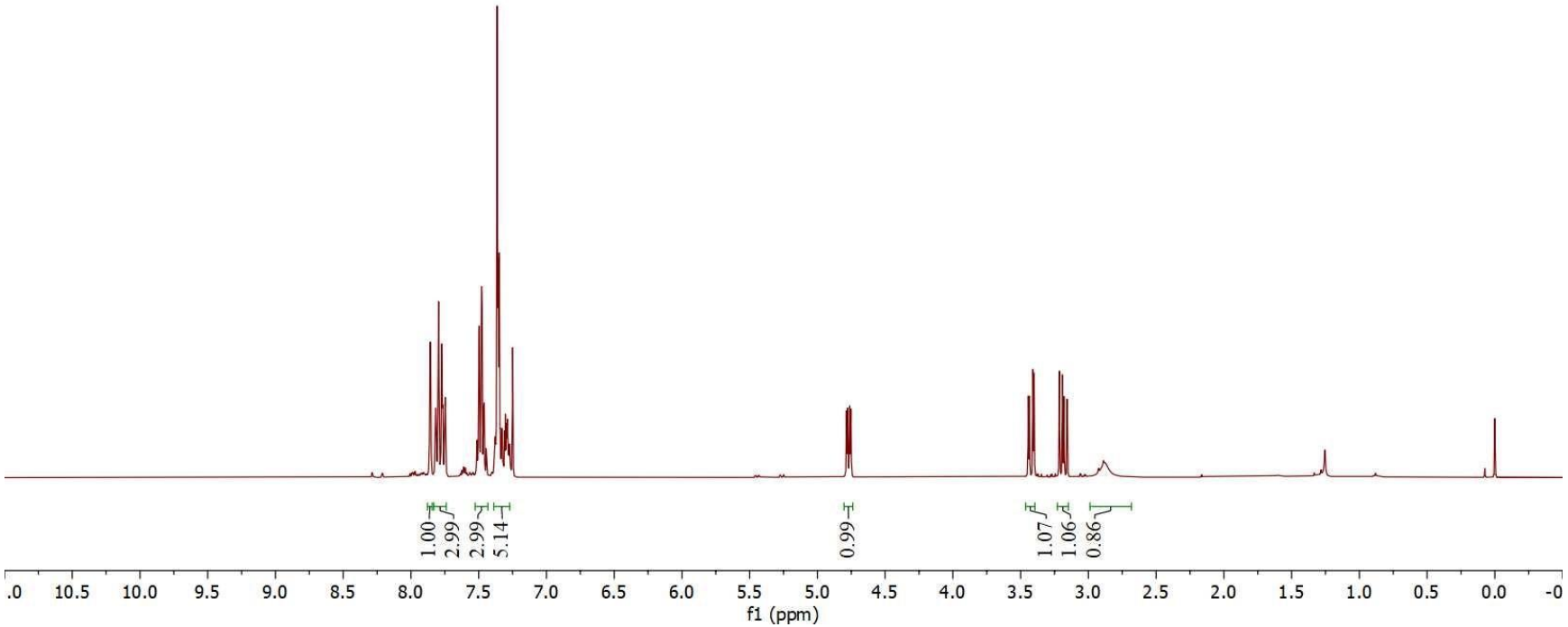




^{1}H NMR (400 MHz, $CDCl_3$)

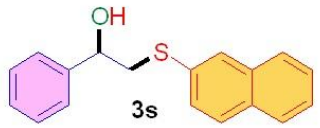


3s

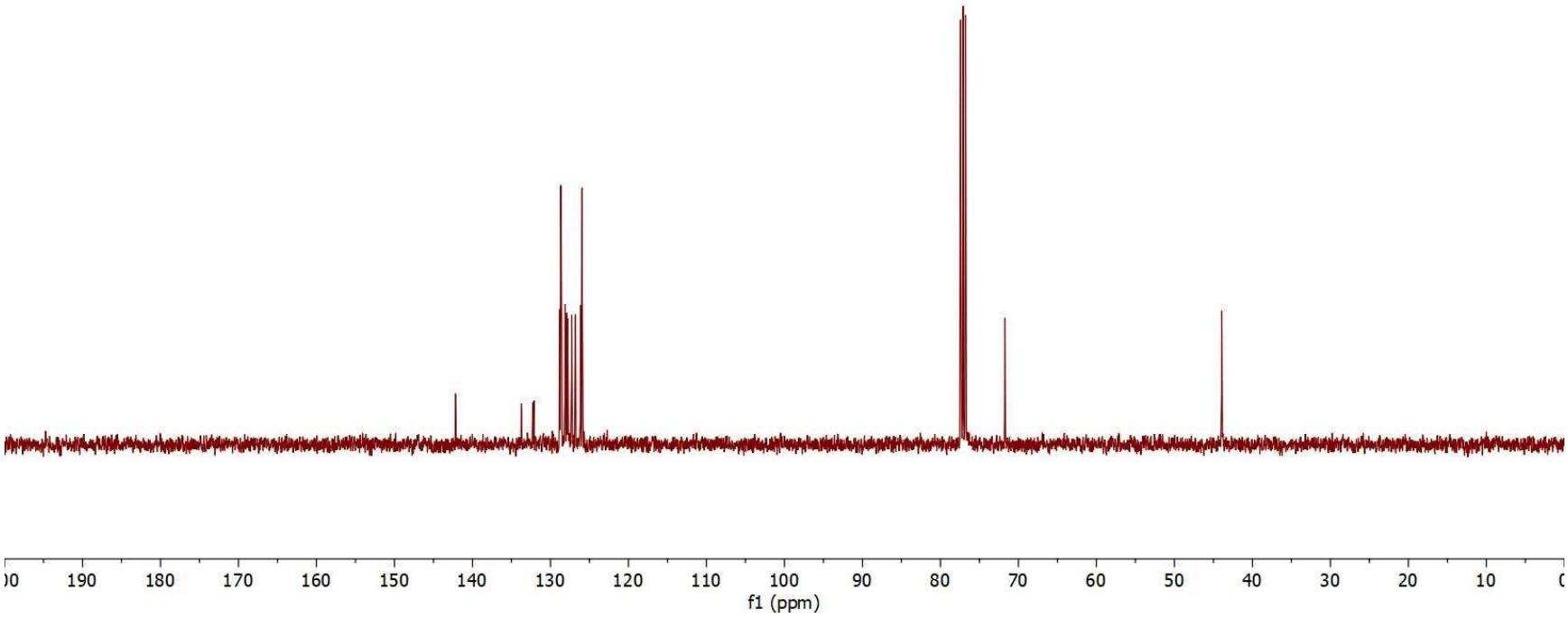


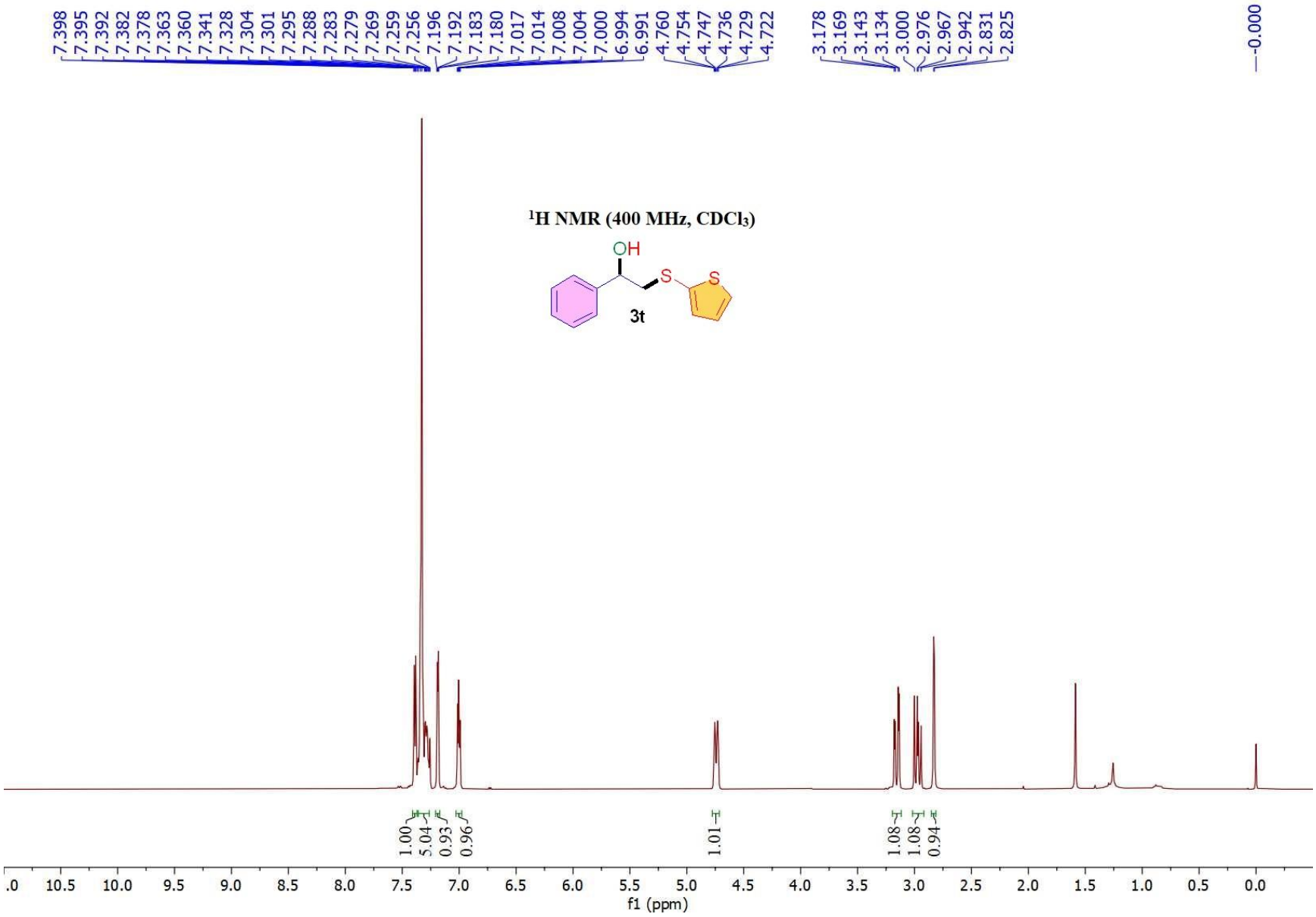
142.19
133.74
132.26
132.12
128.85
128.67
128.57
128.12
127.92
127.81
127.27
126.79
126.15
125.94
77.42
77.10
76.78
71.75
43.93

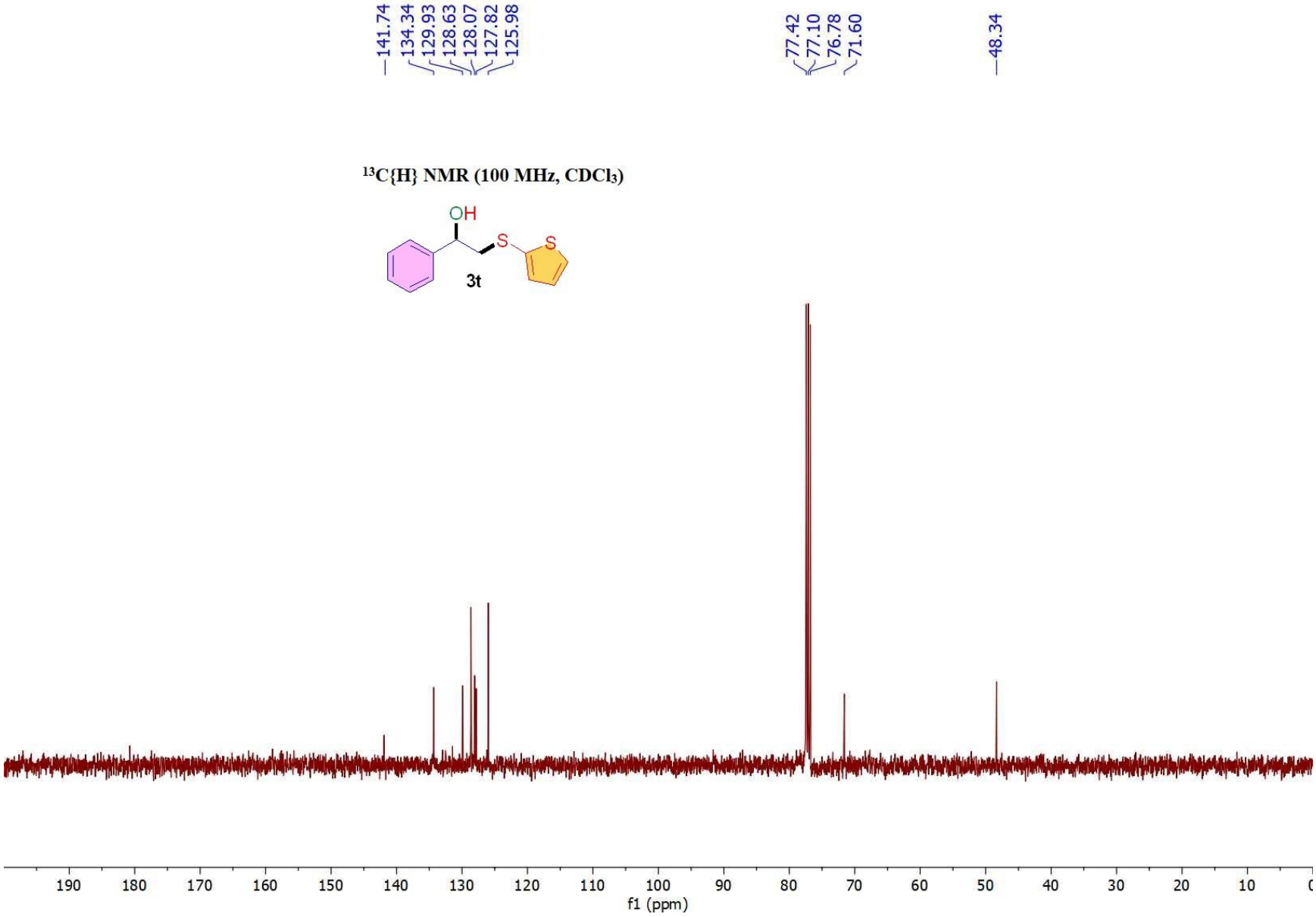
$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)



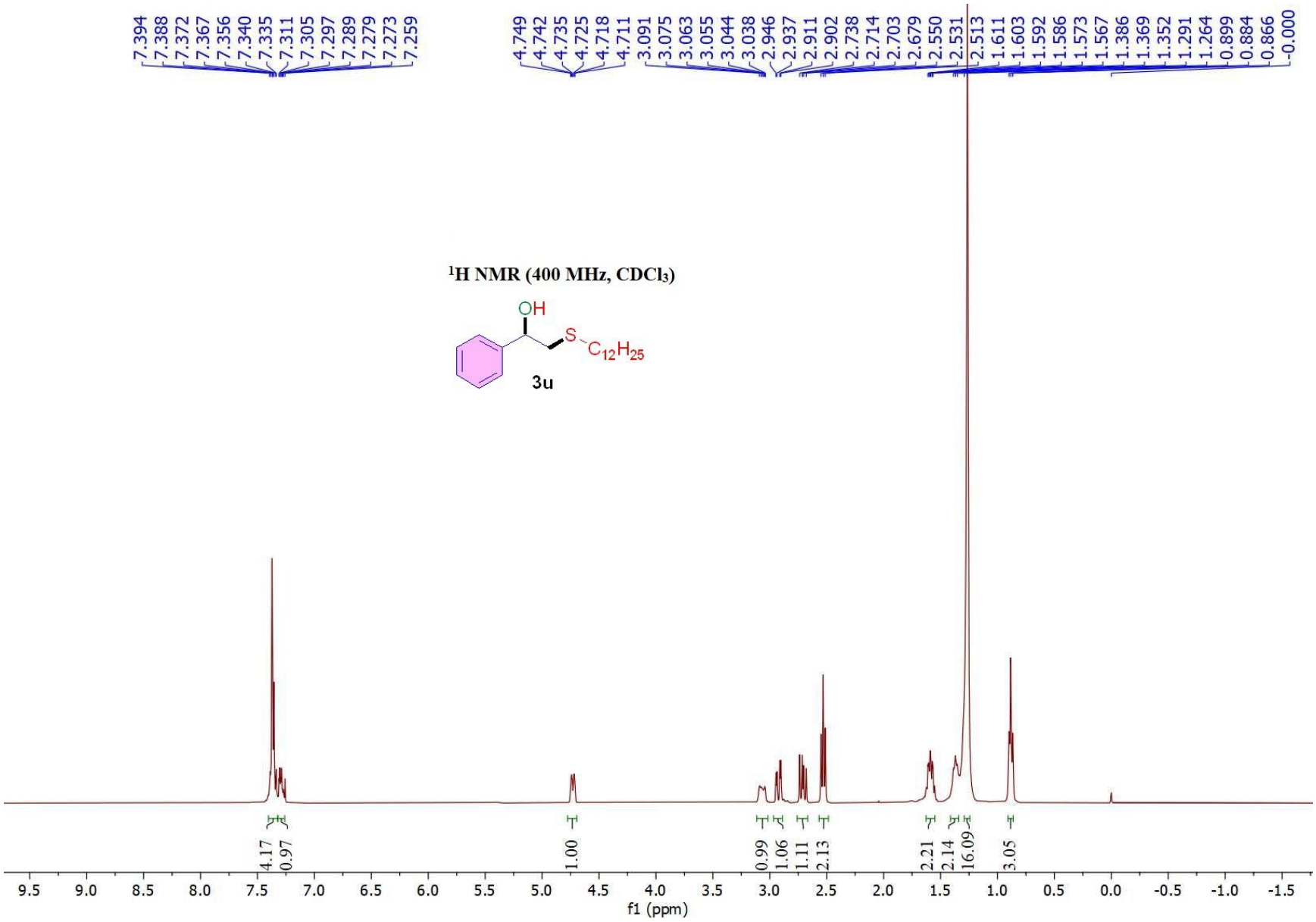
3s

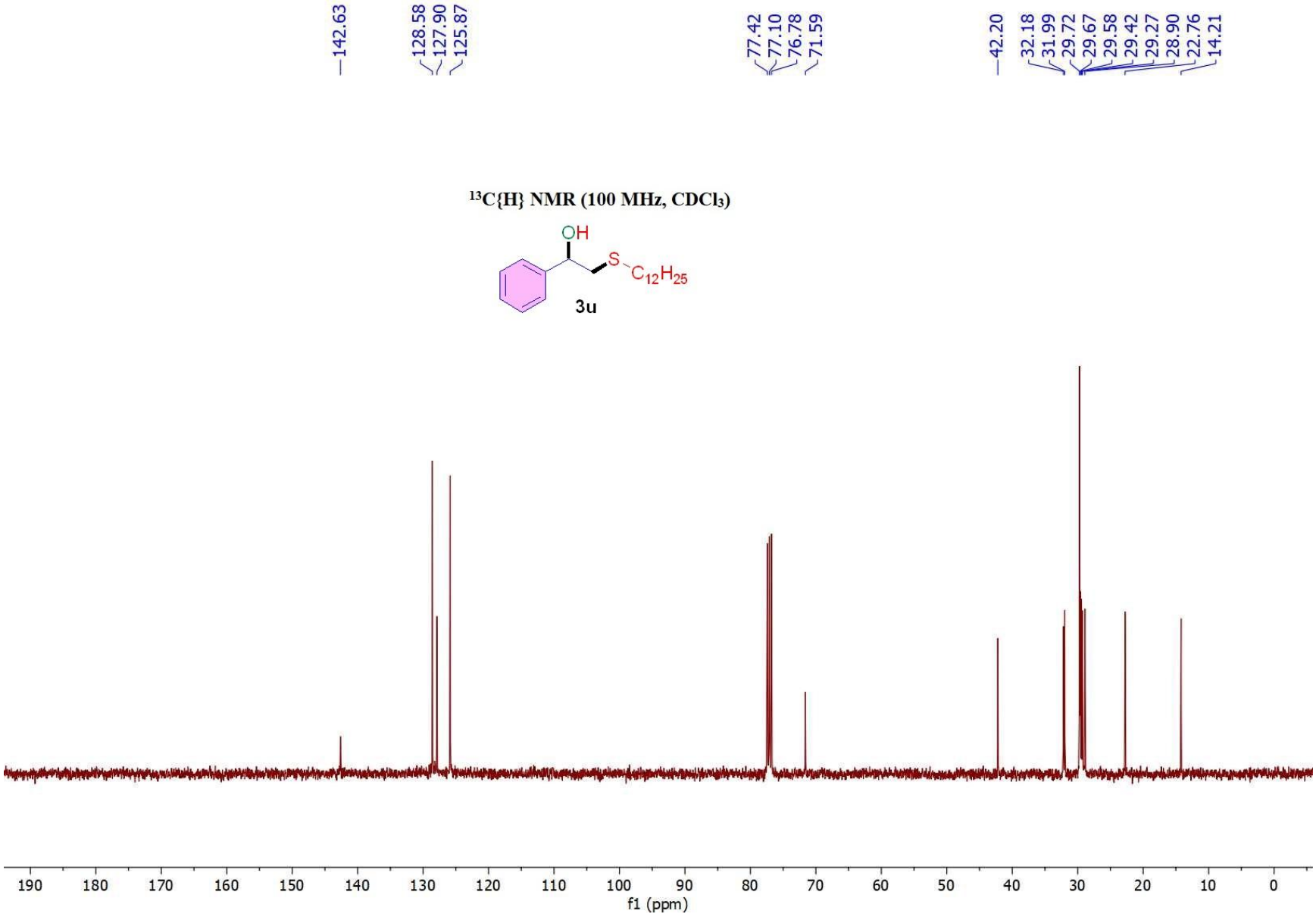



7.398
7.395
7.392
7.382
7.378
7.363
7.360
7.341
7.328
7.304
7.301
7.295
7.288
7.283
7.279
7.269
7.259
7.256
7.196
7.192
7.183
7.180
7.017
7.014
7.008
7.004
7.000
6.994
6.991
4.760
4.754
4.747
4.736
4.729
4.722
3.178
3.169
3.143
3.134
3.000
2.976
2.967
2.942
2.831
2.825
-0.000
1H NMR (400 MHz, CDCl3)
OH
S
S
3t
1.00
5.04
0.93
0.96
1.01
1.08
1.08
0.94
f1 (ppm)



$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)

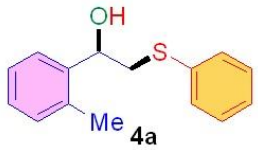

7.394
7.388
7.372
7.367
7.356
7.340
7.335
7.311
7.305
7.297
7.289
7.279
7.273
7.259
4.749
4.742
4.735
4.725
4.718
4.711
3.091
3.075
3.063
3.055
3.044
3.038
2.946
2.937
2.911
2.902
2.738
2.714
2.703
2.679
2.550
2.531
2.513
1.611
1.603
1.592
1.586
1.573
1.567
1.386
1.369
1.352
1.291
1.264
0.899
0.884
0.866
-0.000
1H NMR (400 MHz, CDCl3)
OH
S
C12H25
3u
4.17
0.97
1.00
0.99
1.06
1.11
2.13
2.21
2.14
16.09
3.05
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
-1.5
f1 (ppm)



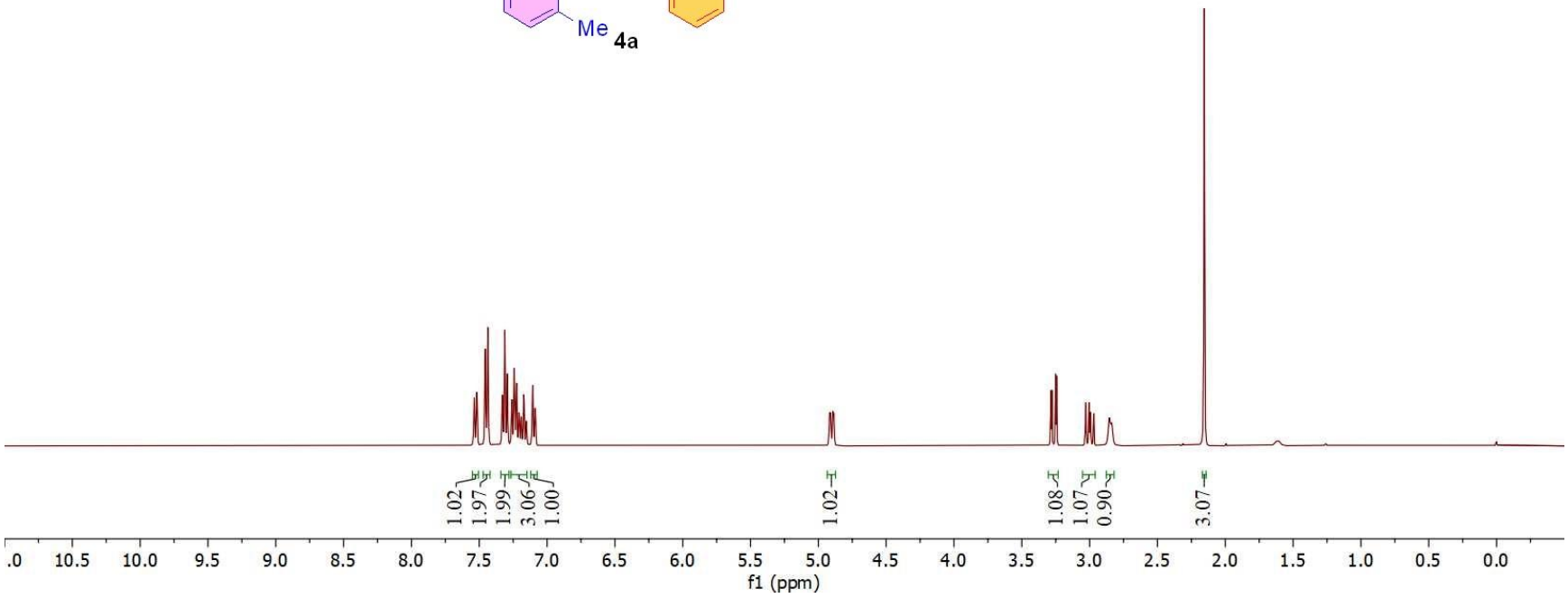
$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)

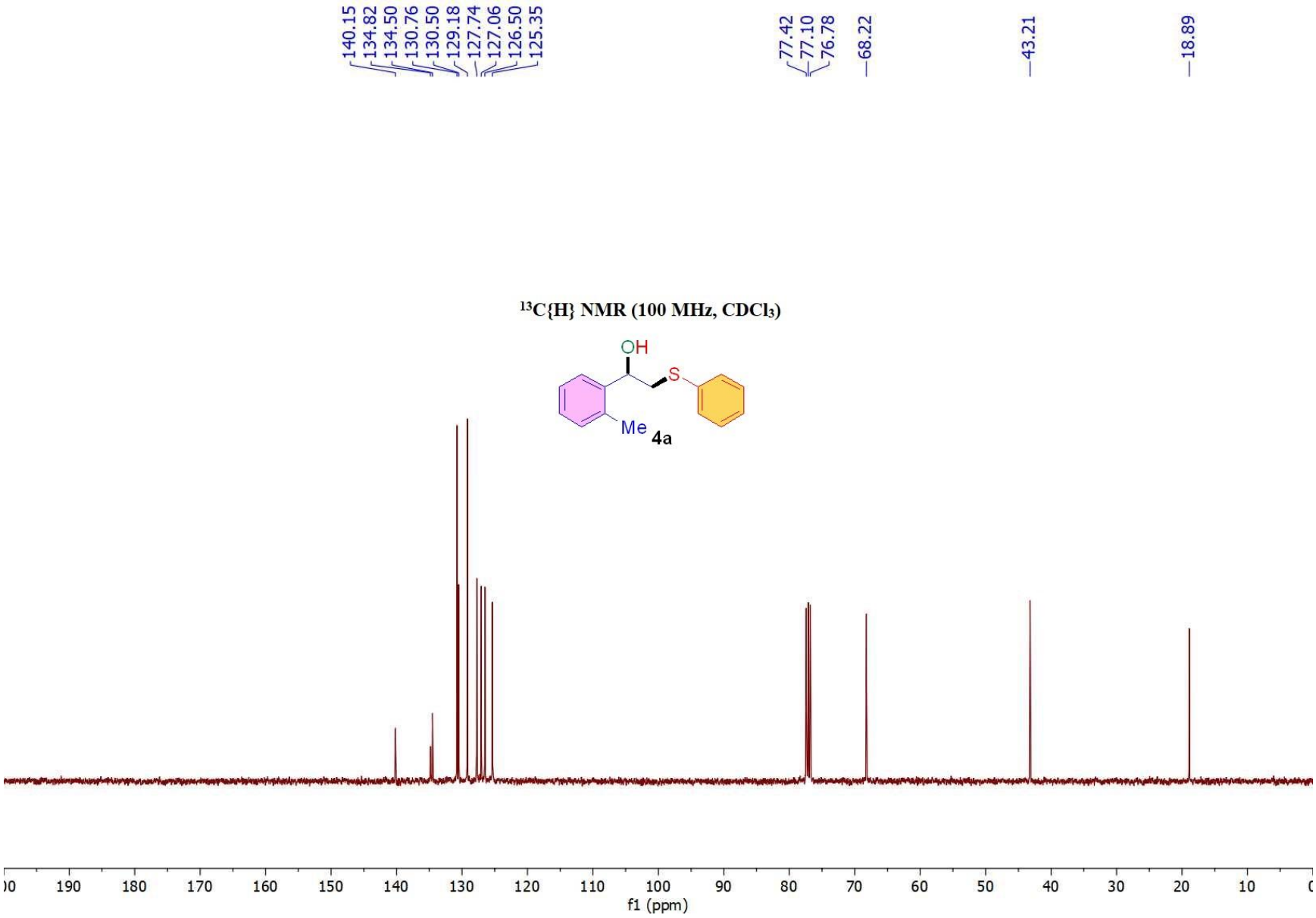
3u

^{1}H NMR (400 MHz, $CDCl_3$)

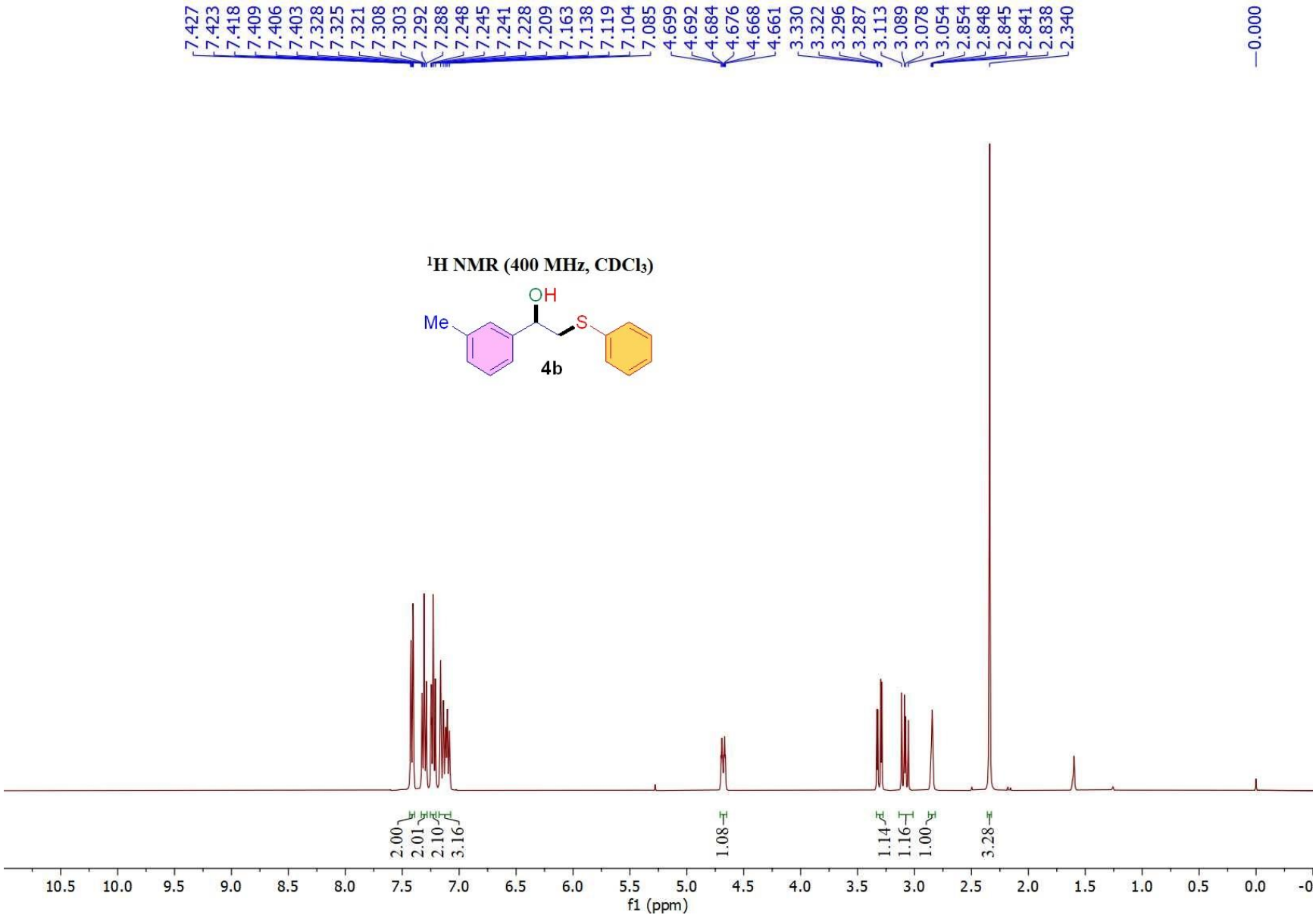


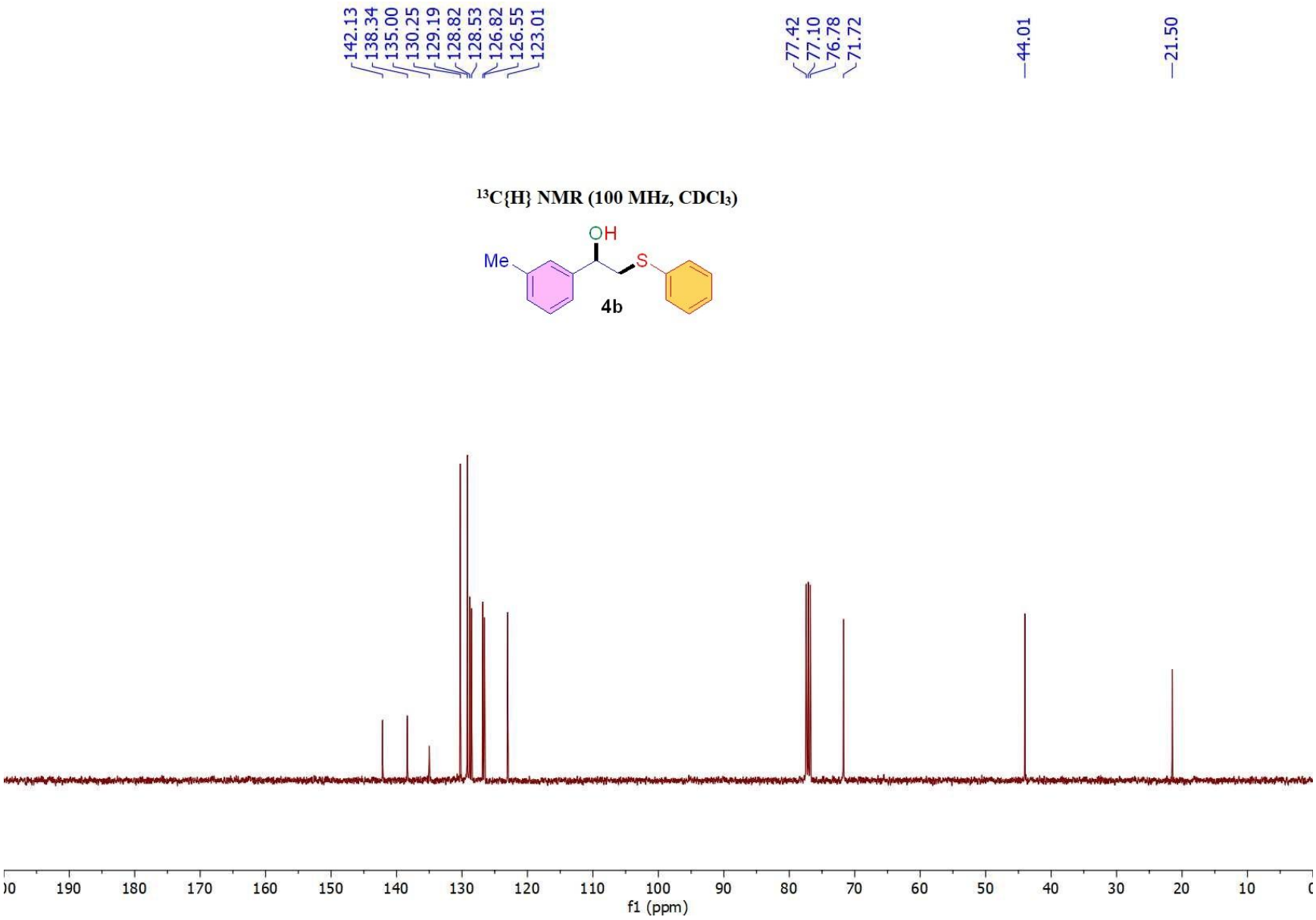
4a





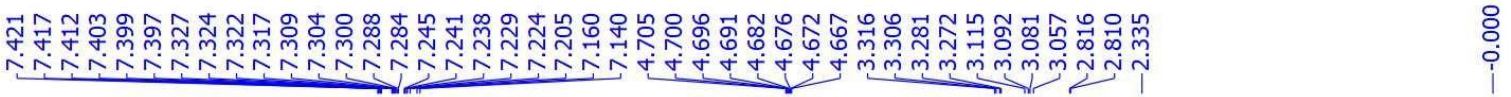
$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)


7.427
7.423
7.418
7.409
7.406
7.403
7.328
7.325
7.321
7.308
7.303
7.292
7.288
7.248
7.245
7.241
7.228
7.209
7.163
7.138
7.119
7.104
7.085
4.699
4.692
4.684
4.676
4.668
4.661
3.330
3.322
3.296
3.287
3.113
3.089
3.078
3.054
2.854
2.848
2.845
2.841
2.838
2.340
0.000
1H NMR (400 MHz, CDCl3)
OH
Me
S
4b
2.00
2.01
2.10
3.16
1.08
1.14
1.16
1.00
3.28
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)

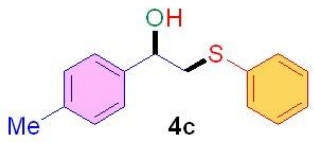


$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)

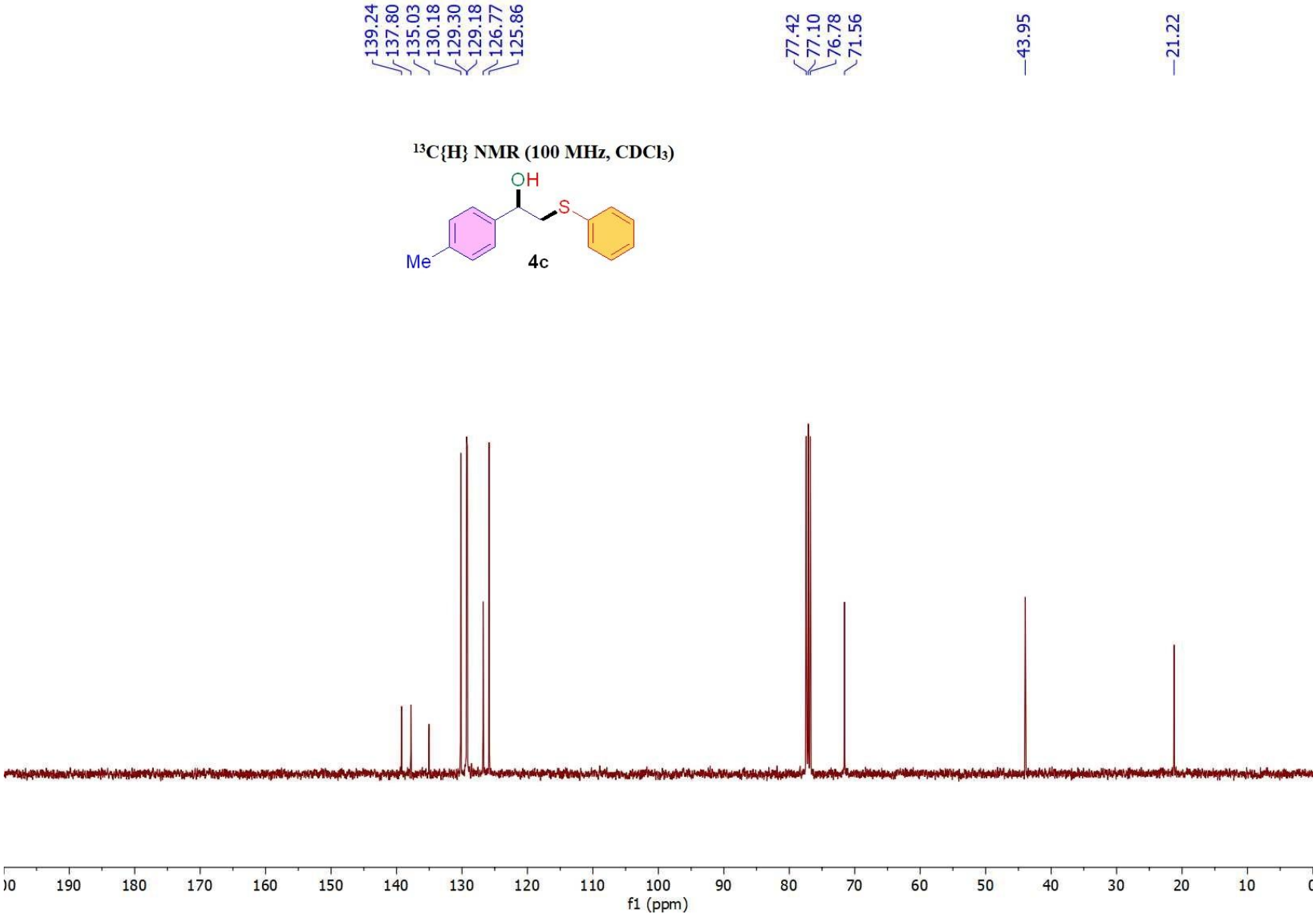
4b



^{1}H NMR (400 MHz, $CDCl_3$)

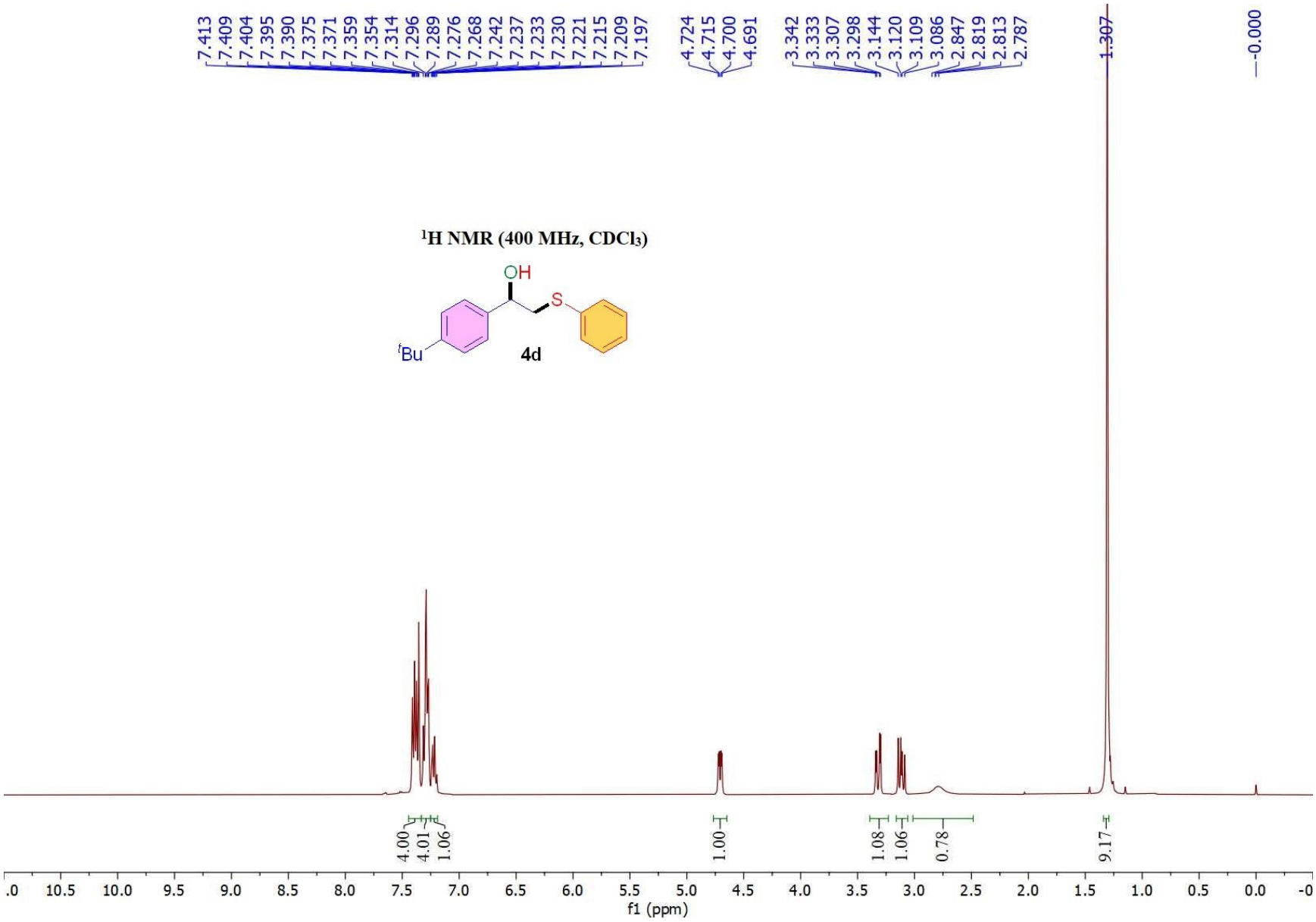


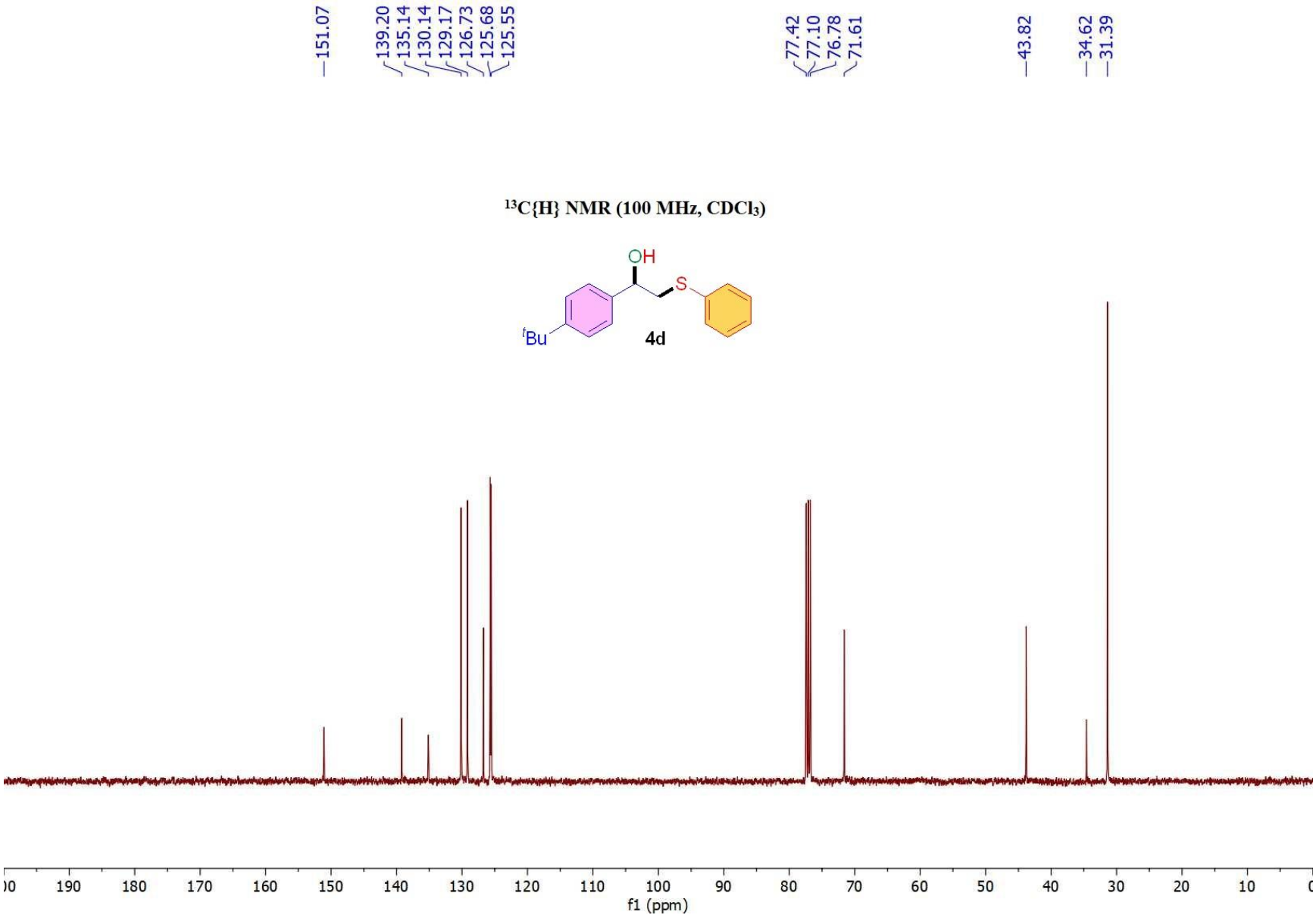
4c



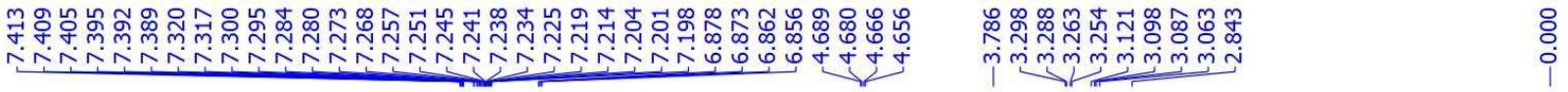
$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)

4c

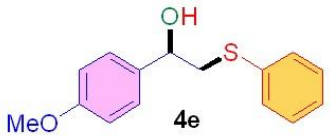

7.413
7.409
7.404
7.395
7.390
7.375
7.371
7.359
7.354
7.314
7.296
7.289
7.276
7.268
7.242
7.237
7.233
7.230
7.221
7.215
7.209
7.197
4.724
4.715
4.700
4.691
3.342
3.333
3.307
3.298
3.144
3.120
3.109
3.086
2.847
2.819
2.813
2.787
1.307
-0.000
^{1}H NMR (400 MHz, CDCl$_3$)
OH
S
^{t}Bu
4d
4.00
4.01
1.06
1.00
1.08
1.06
0.78
9.17
f1 (ppm)



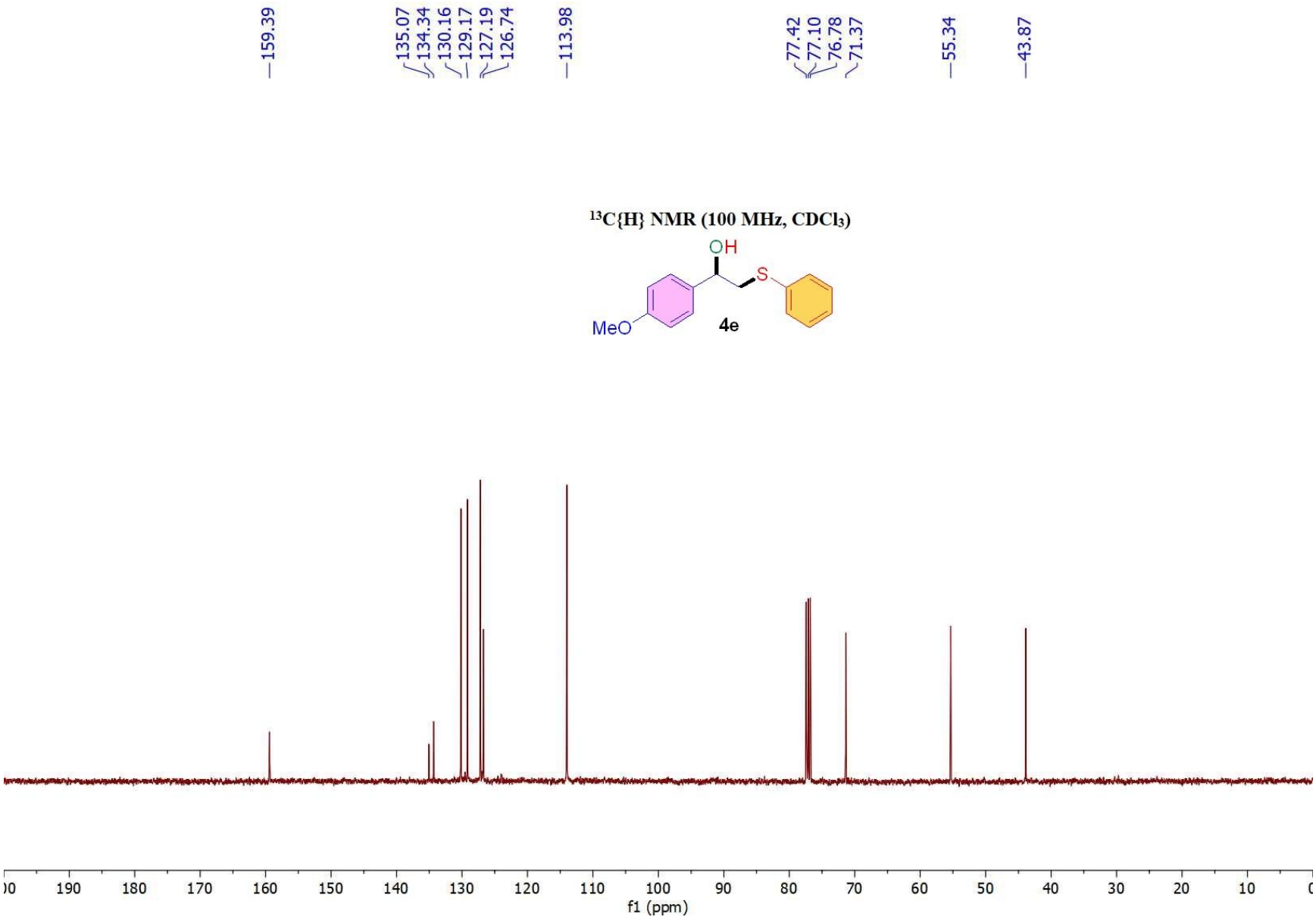
$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)



^{1}H NMR (400 MHz, $CDCl_3$)

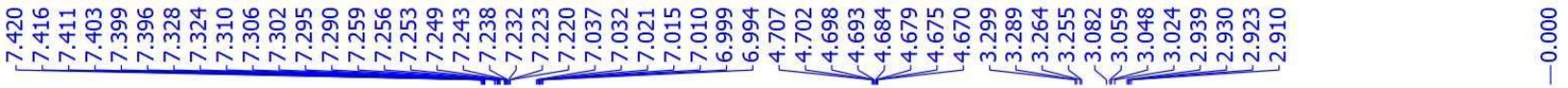


4e

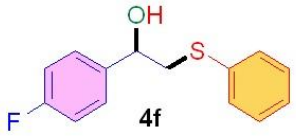


$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)

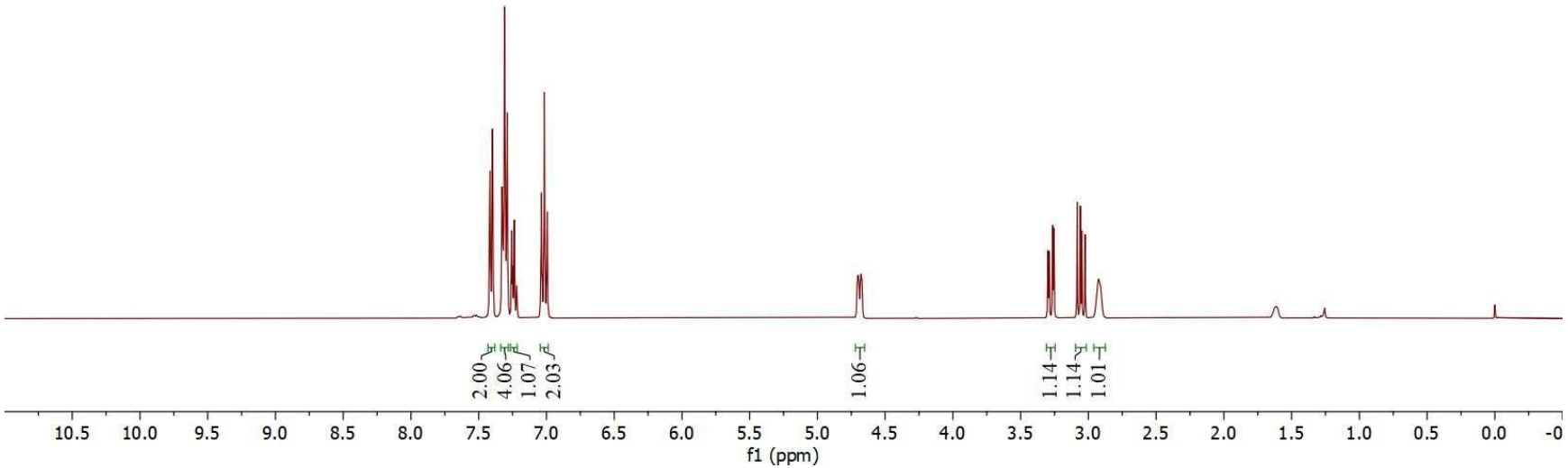
4e

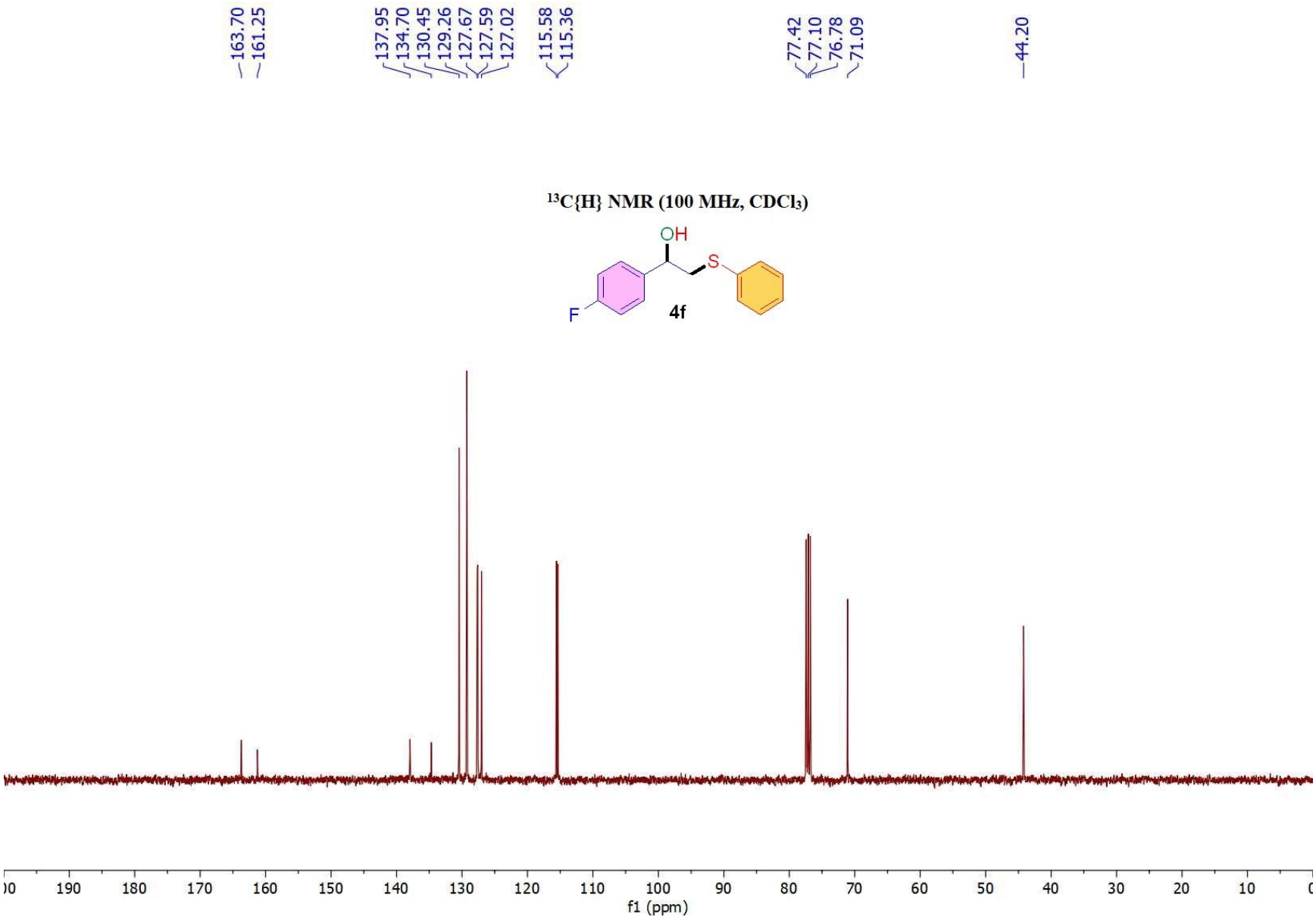


^{1}H NMR (400 MHz, $CDCl_3$)



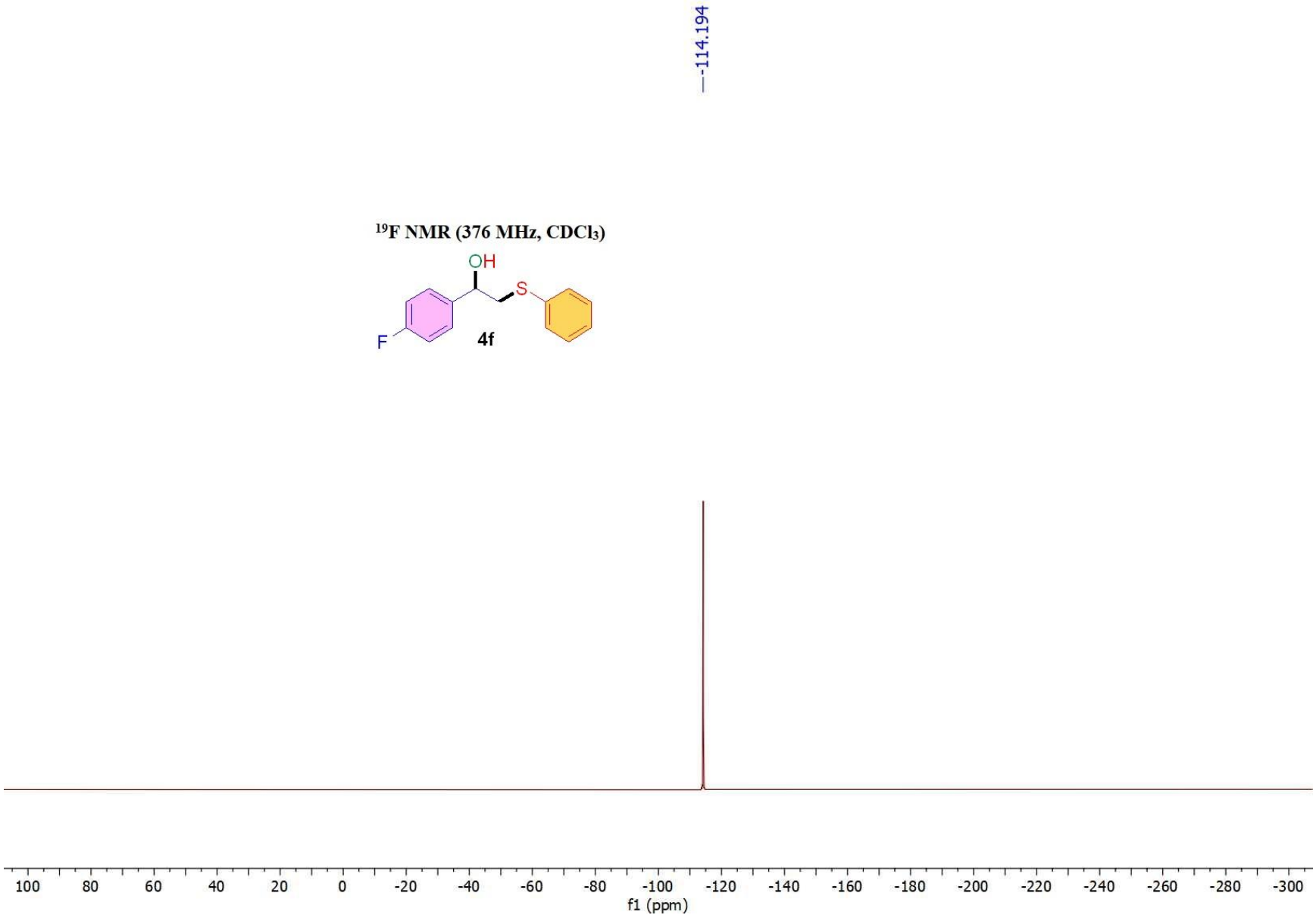
4f





$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)

4f

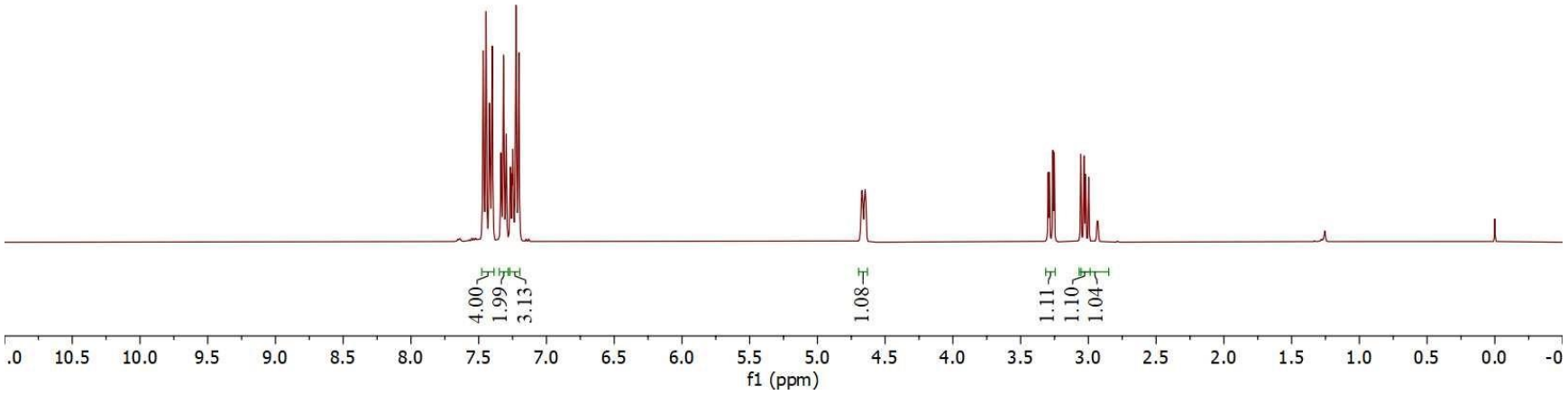


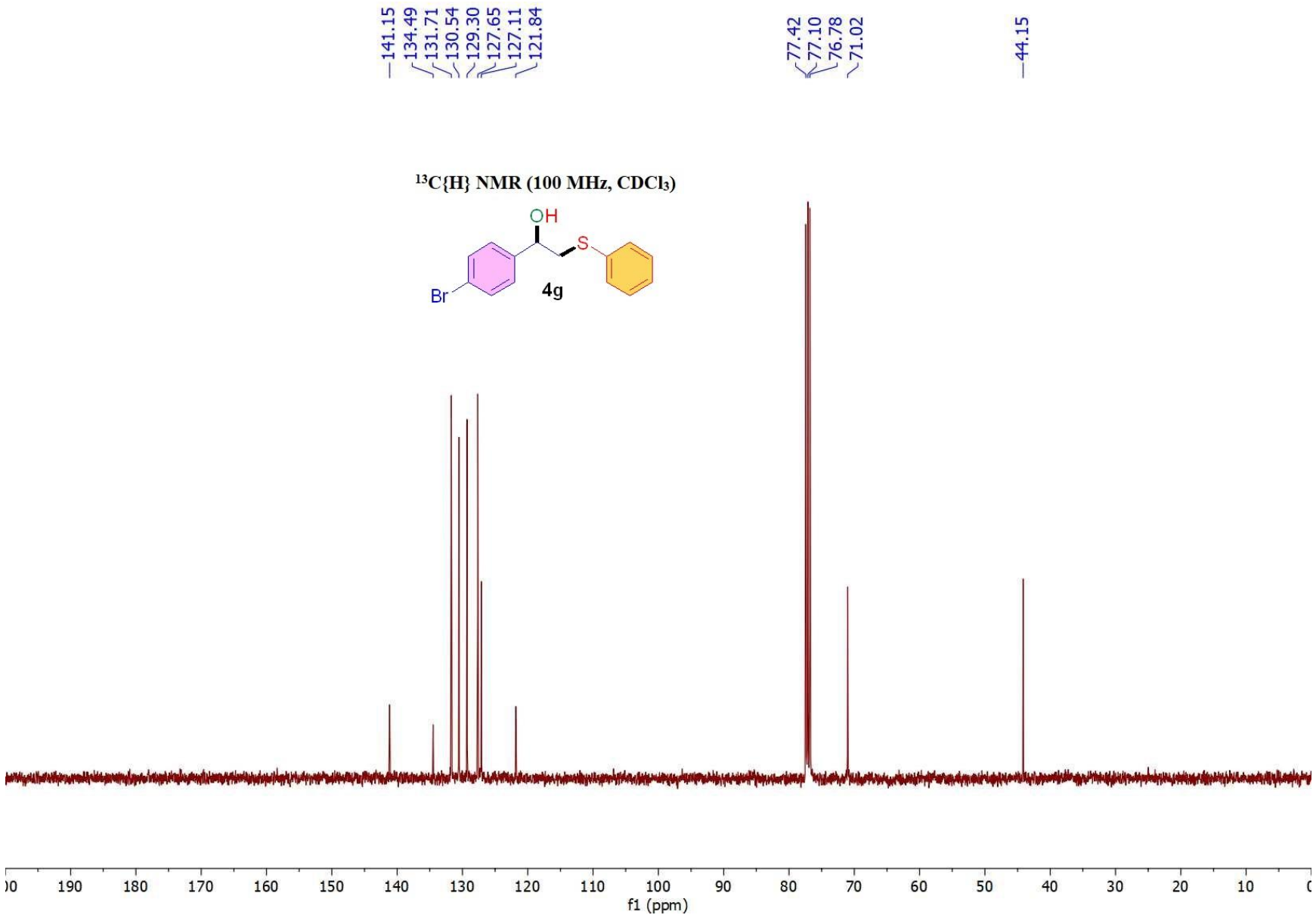
^{19}F NMR (376 MHz, $CDCl_3$)

4f

^{1}H NMR (400 MHz, $CDCl_3$)

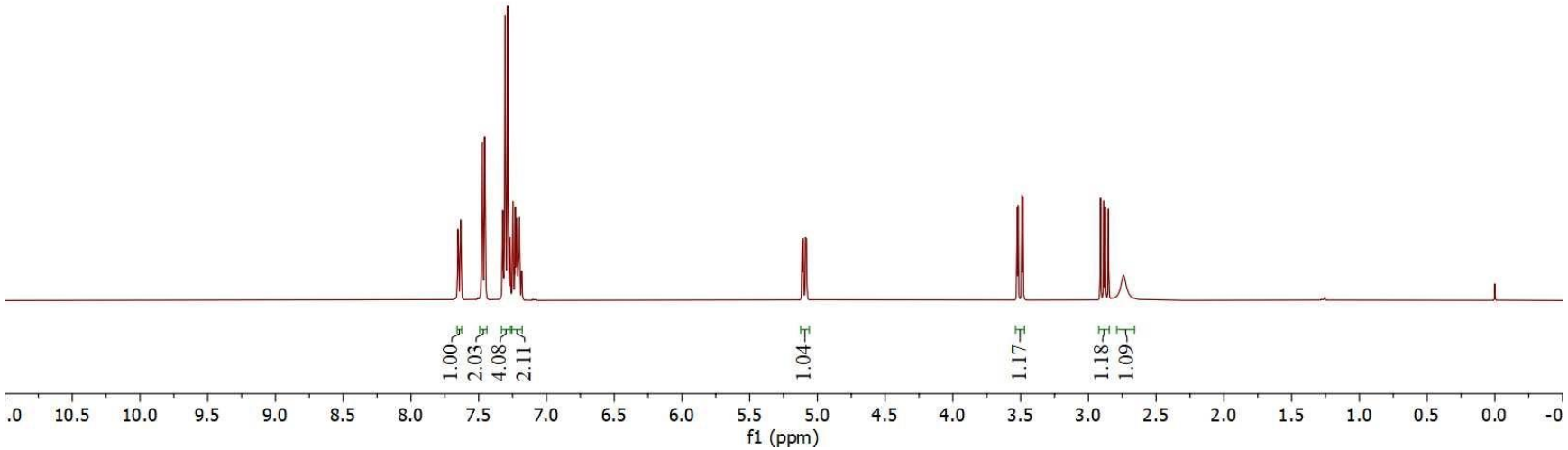
4g




141.15
134.49
131.71
130.54
129.30
127.65
127.11
121.84
77.42
77.10
76.78
71.02
44.15
13C{H} NMR (100 MHz, CDCl3)
OH
S
Br
4g
f1 (ppm)

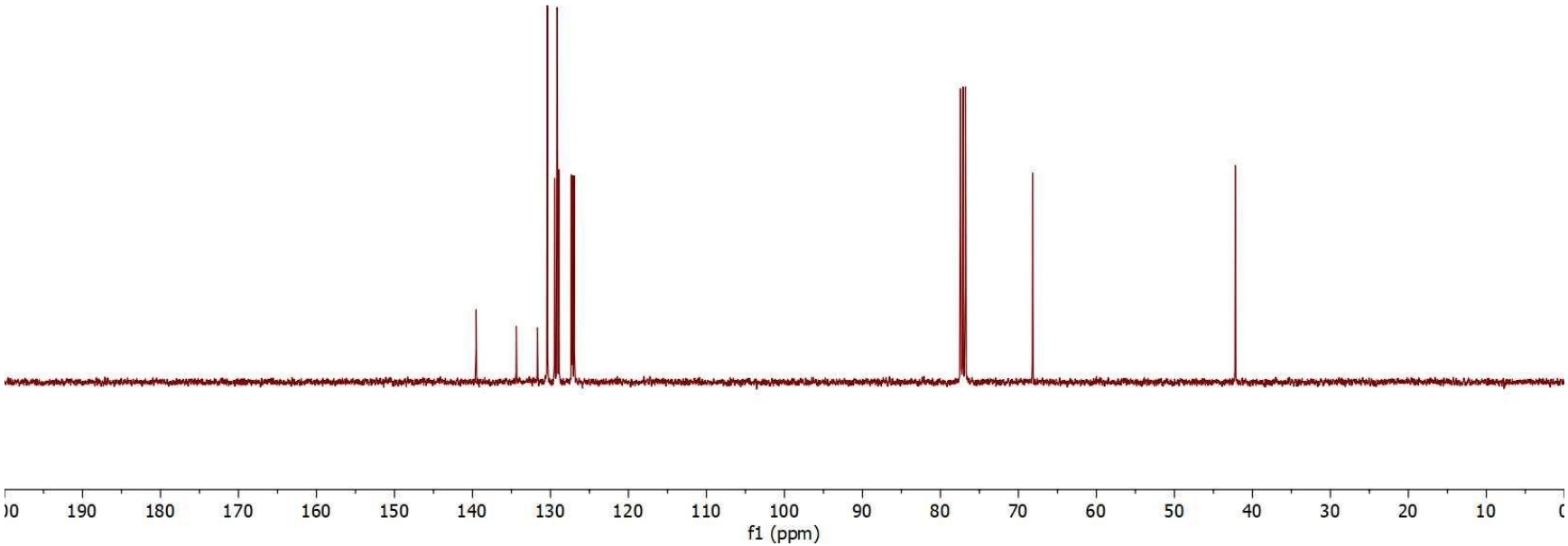
^{1}H NMR (400 MHz, $CDCl_3$)

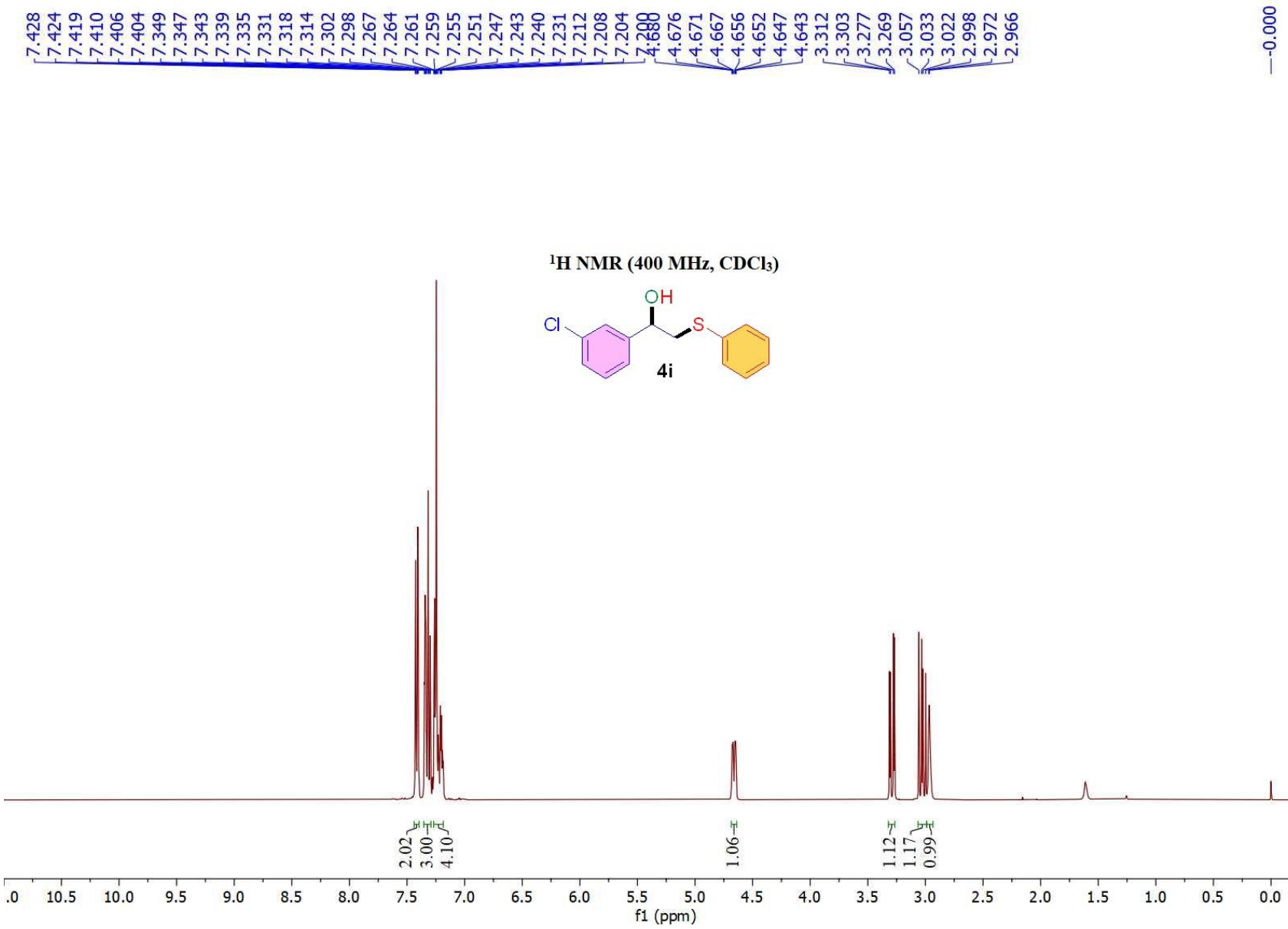
4h



$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)

4h



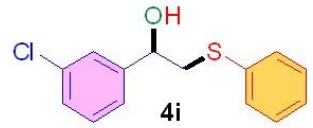

7.428
7.424
7.419
7.410
7.406
7.404
7.349
7.347
7.343
7.339
7.335
7.331
7.318
7.314
7.302
7.298
7.267
7.264
7.261
7.259
7.255
7.251
7.247
7.243
7.240
7.231
7.212
7.208
7.204
4.676
4.671
4.667
4.656
4.652
4.647
4.643
3.312
3.303
3.277
3.269
3.057
3.033
3.022
2.998
2.972
2.966
-0.000
1H NMR (400 MHz, CDCl3)
OH
Cl
S
4i
2.02
3.00
4.10
1.06
1.12
1.17
0.99
f1 (ppm)

144.23
134.55
134.47
130.59
129.89
129.30
128.15
127.15
126.15
124.08

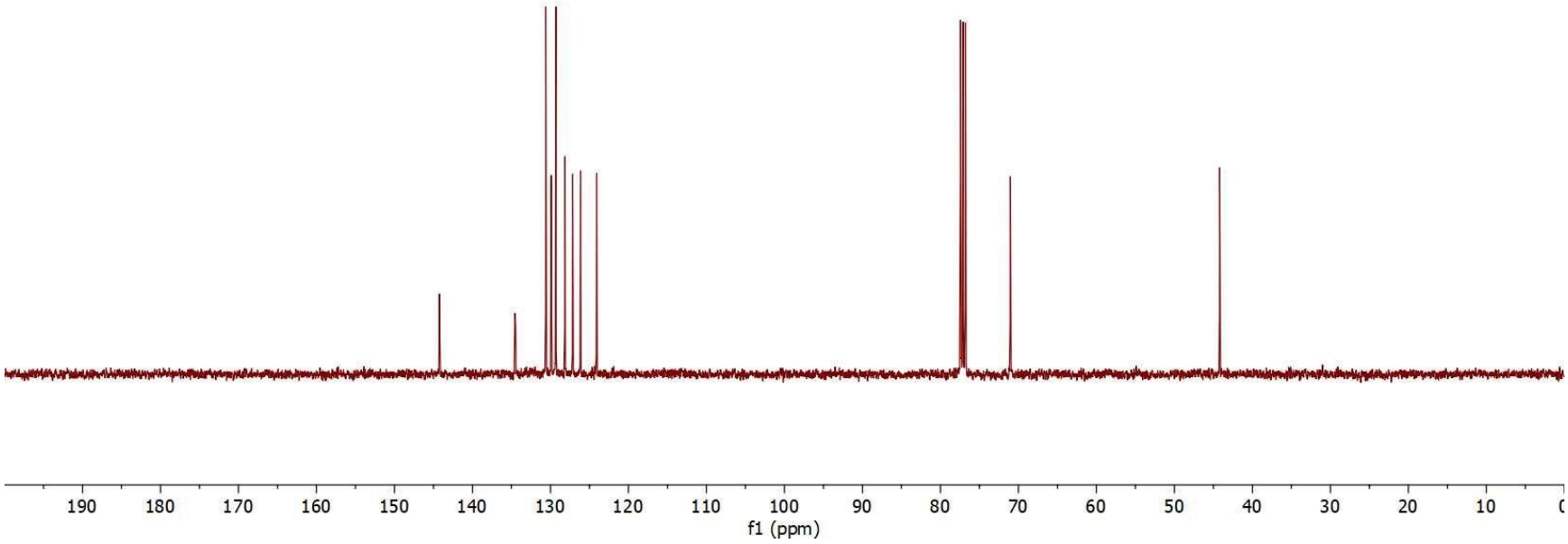
77.42
77.10
76.78
71.04

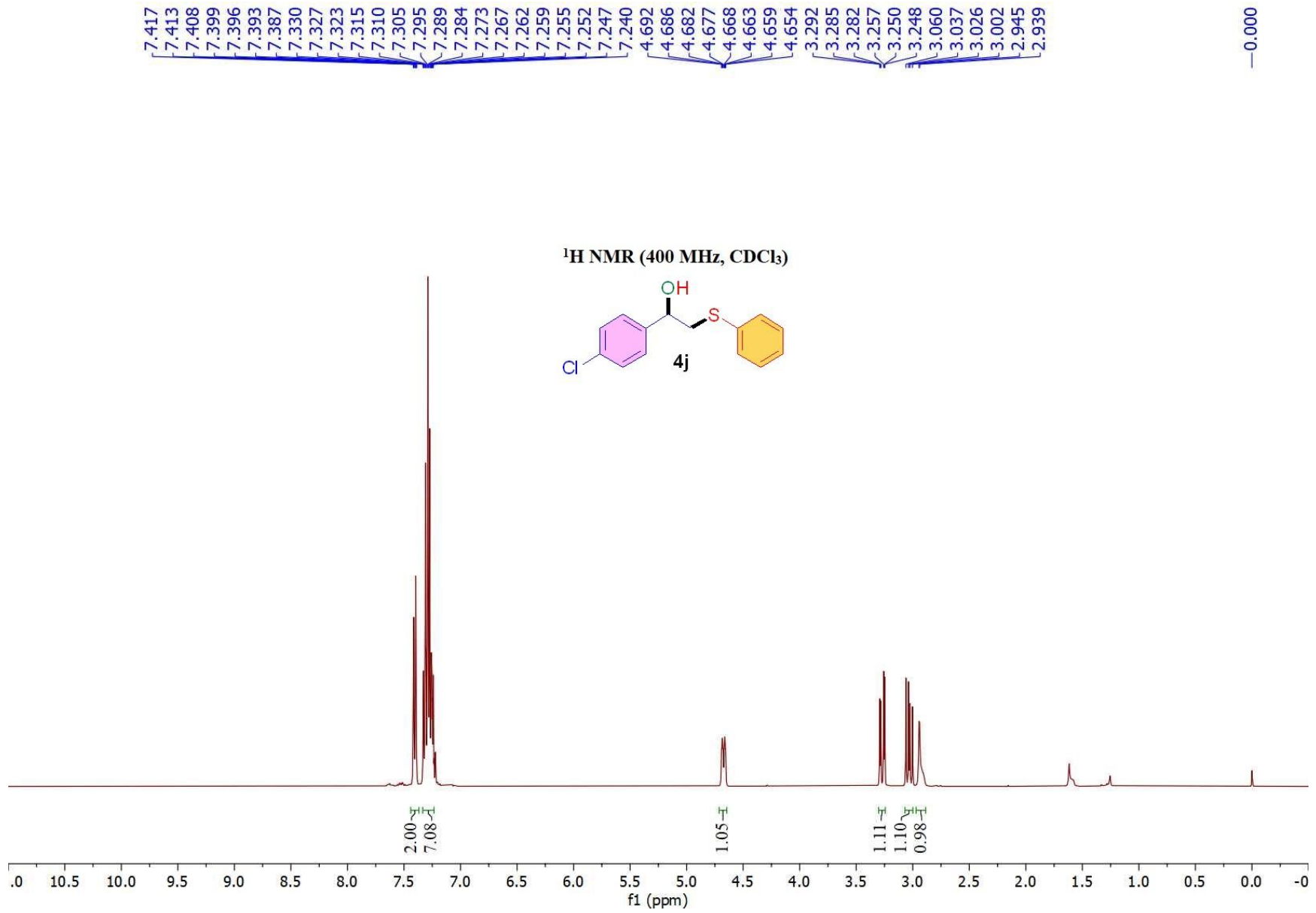
44.20

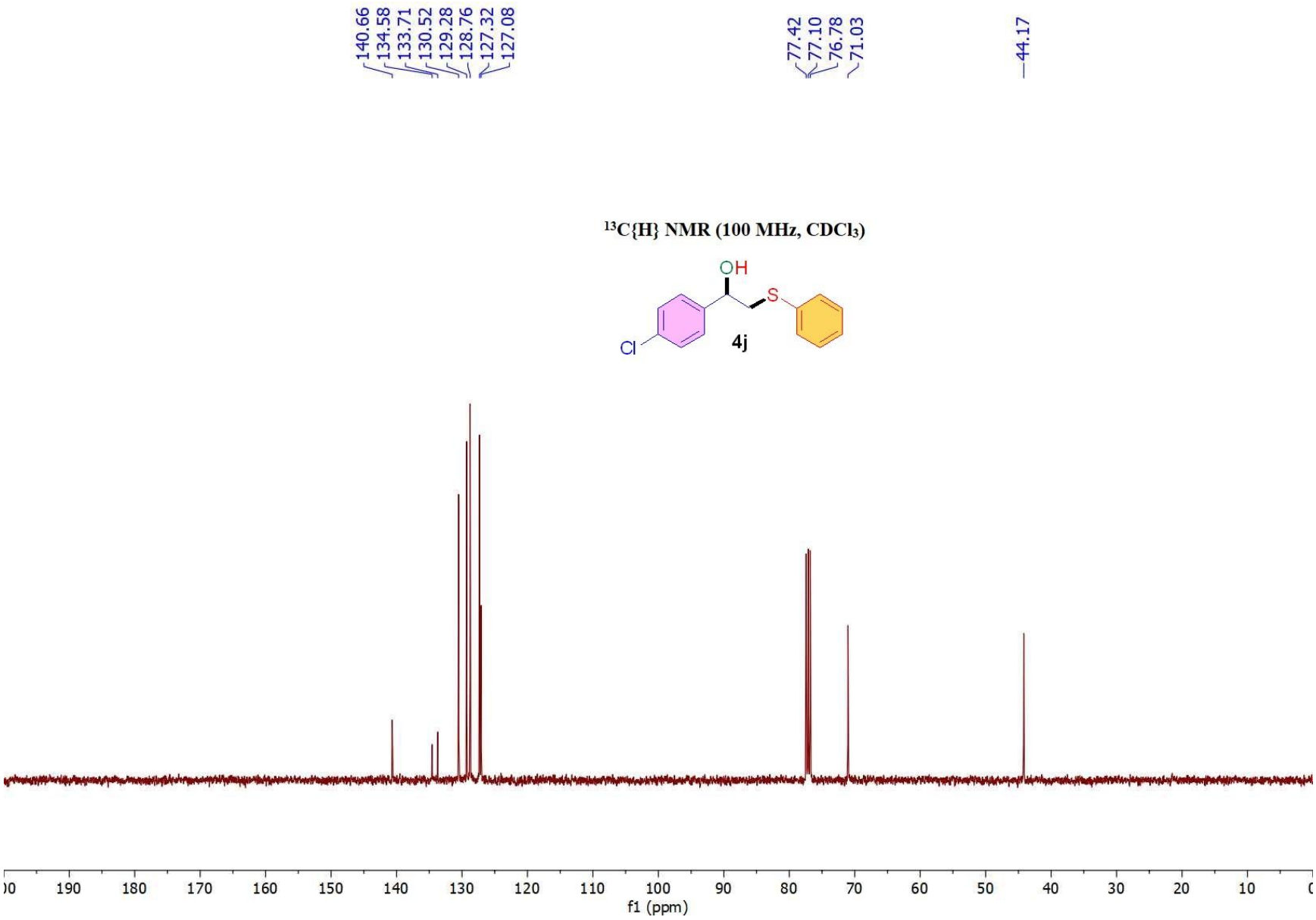
$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)



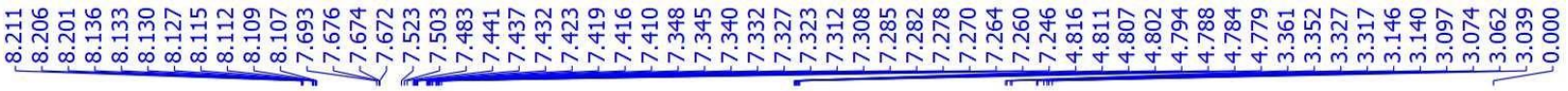
4i



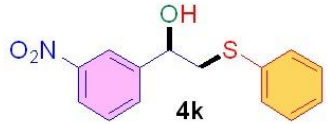

7.417
7.413
7.408
7.399
7.396
7.393
7.387
7.330
7.327
7.323
7.315
7.310
7.305
7.295
7.289
7.284
7.273
7.267
7.262
7.259
7.255
7.252
7.247
7.240
4.692
4.686
4.682
4.677
4.668
4.663
4.659
4.654
3.292
3.285
3.282
3.257
3.250
3.248
3.060
3.037
3.026
3.002
2.945
2.939
0.000
1H NMR (400 MHz, CDCl3)
OH
S
Cl
4j
2.00
7.08
1.05
1.11
1.10
0.98
f1 (ppm)



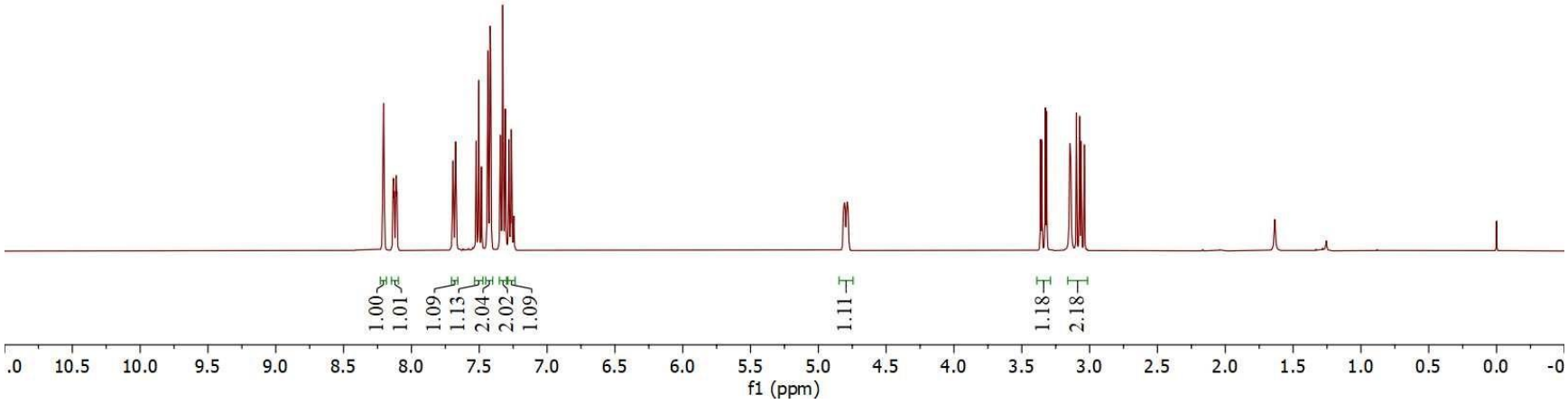
$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)



^{1}H NMR (400 MHz, $CDCl_3$)

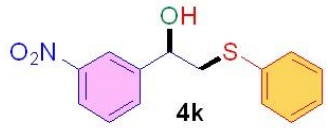


4k

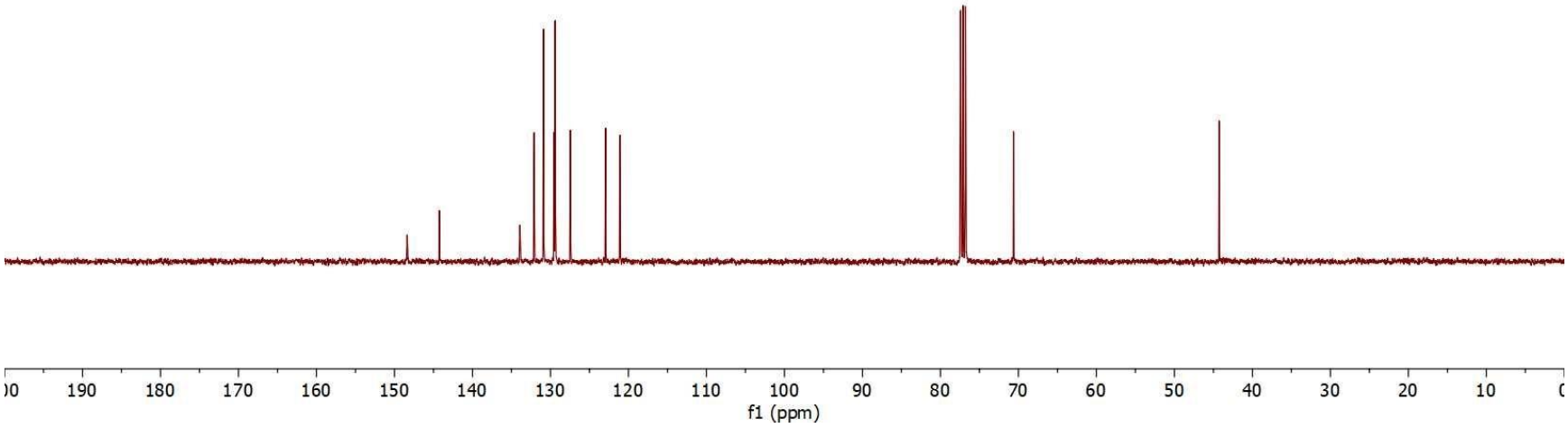


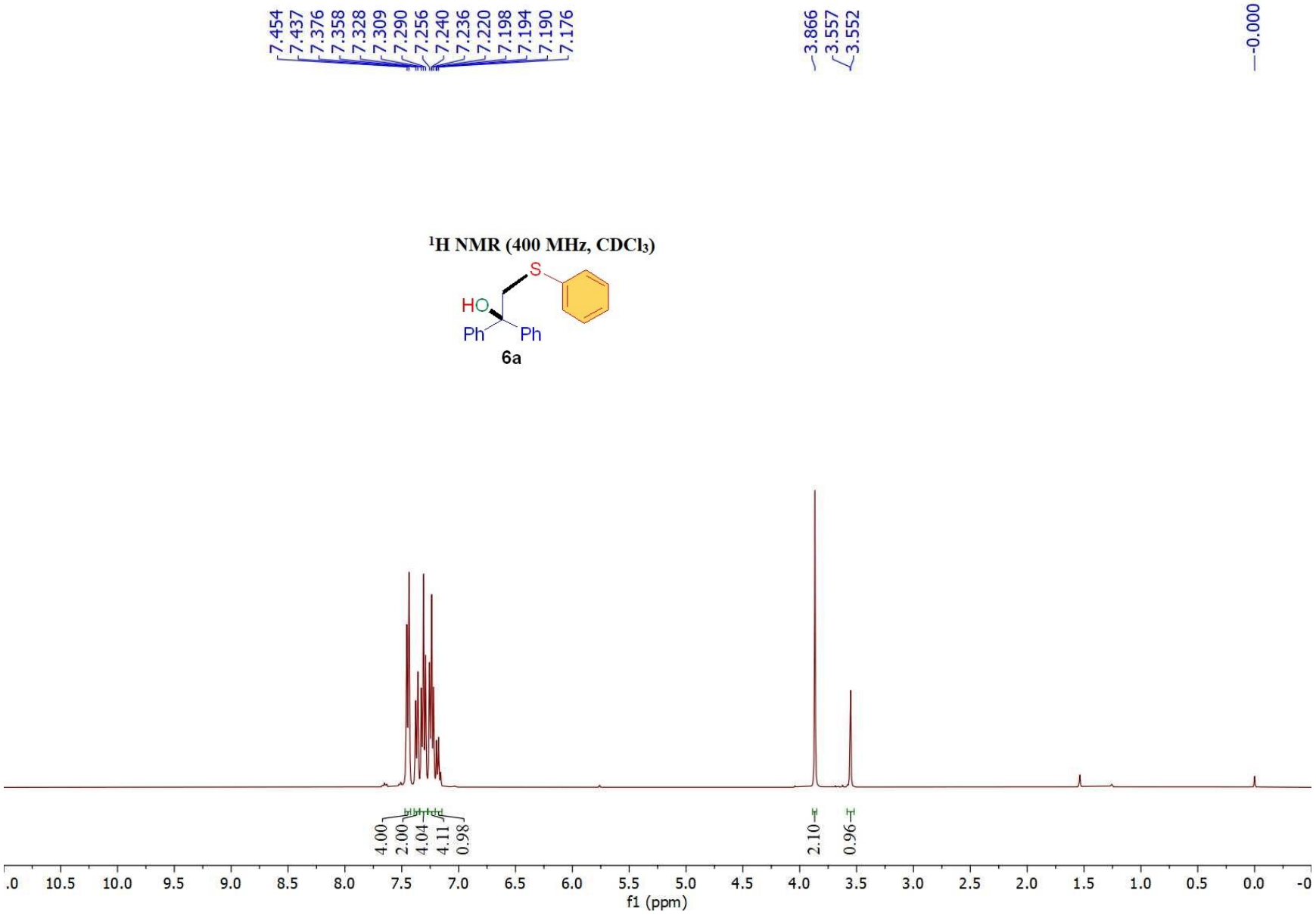
148.37
144.25
133.92
132.09
130.89
129.54
129.40
127.44
122.93
121.10
77.42
77.10
76.78
70.63
44.26

$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)



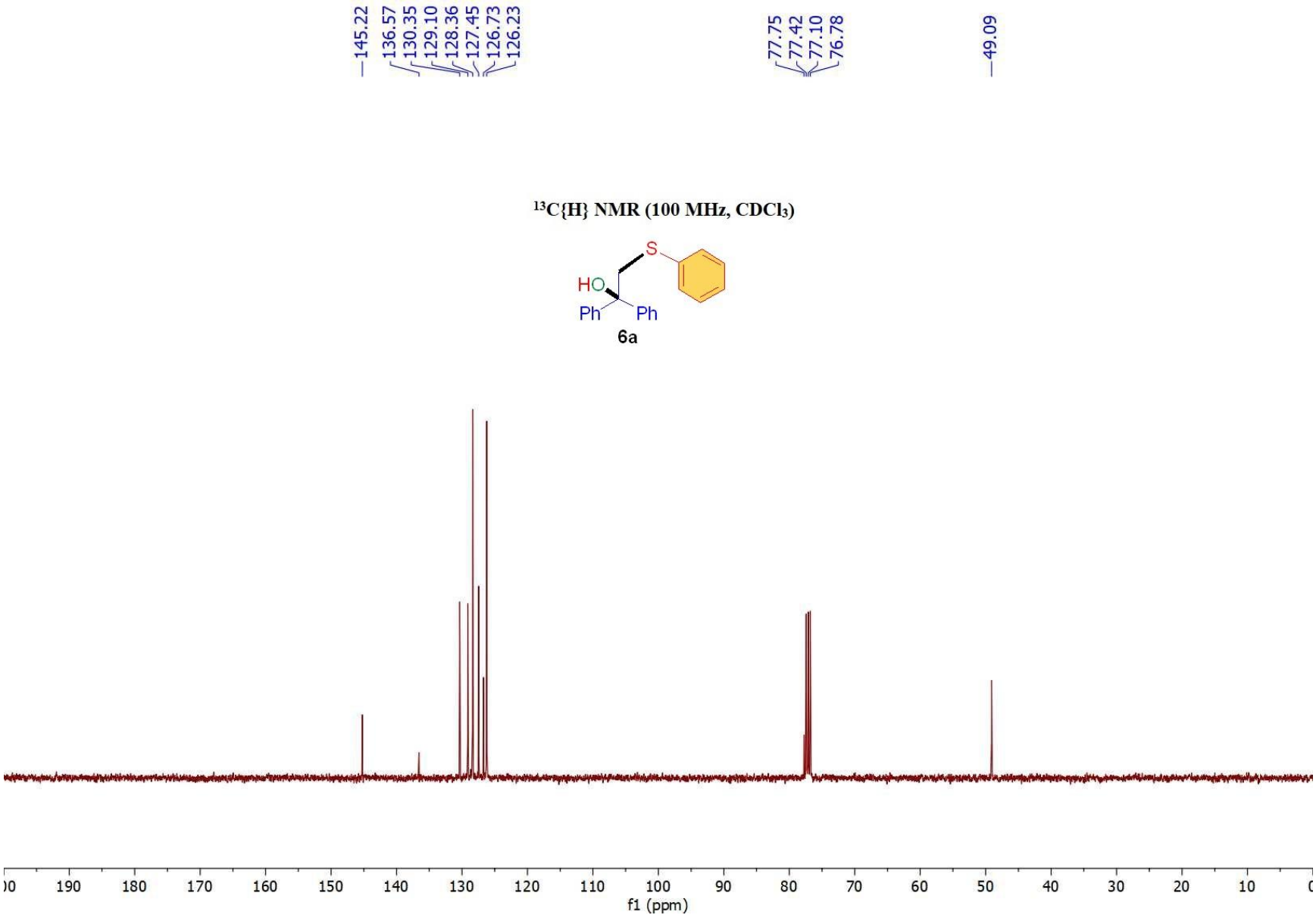
4k





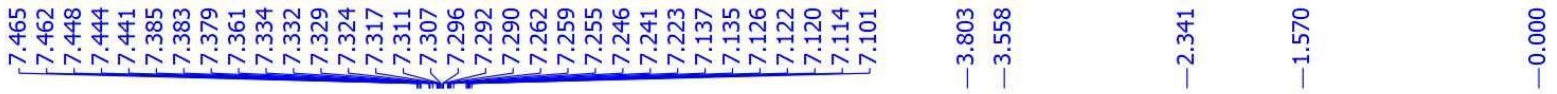
^{1}H NMR (400 MHz, $CDCl_3$)

6a

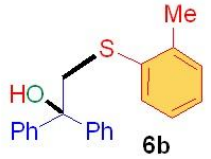


$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)

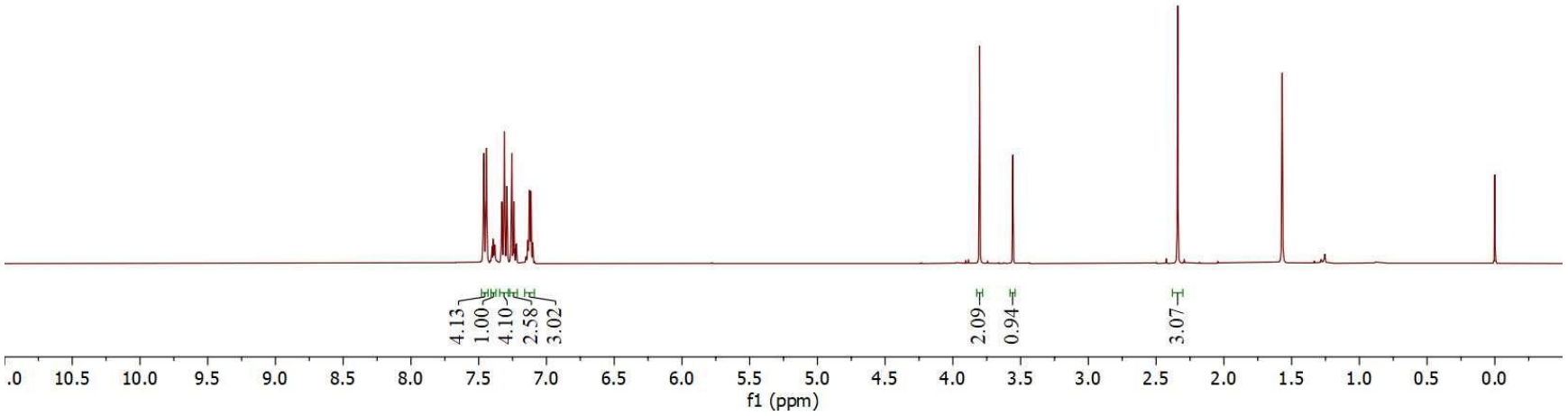
6a

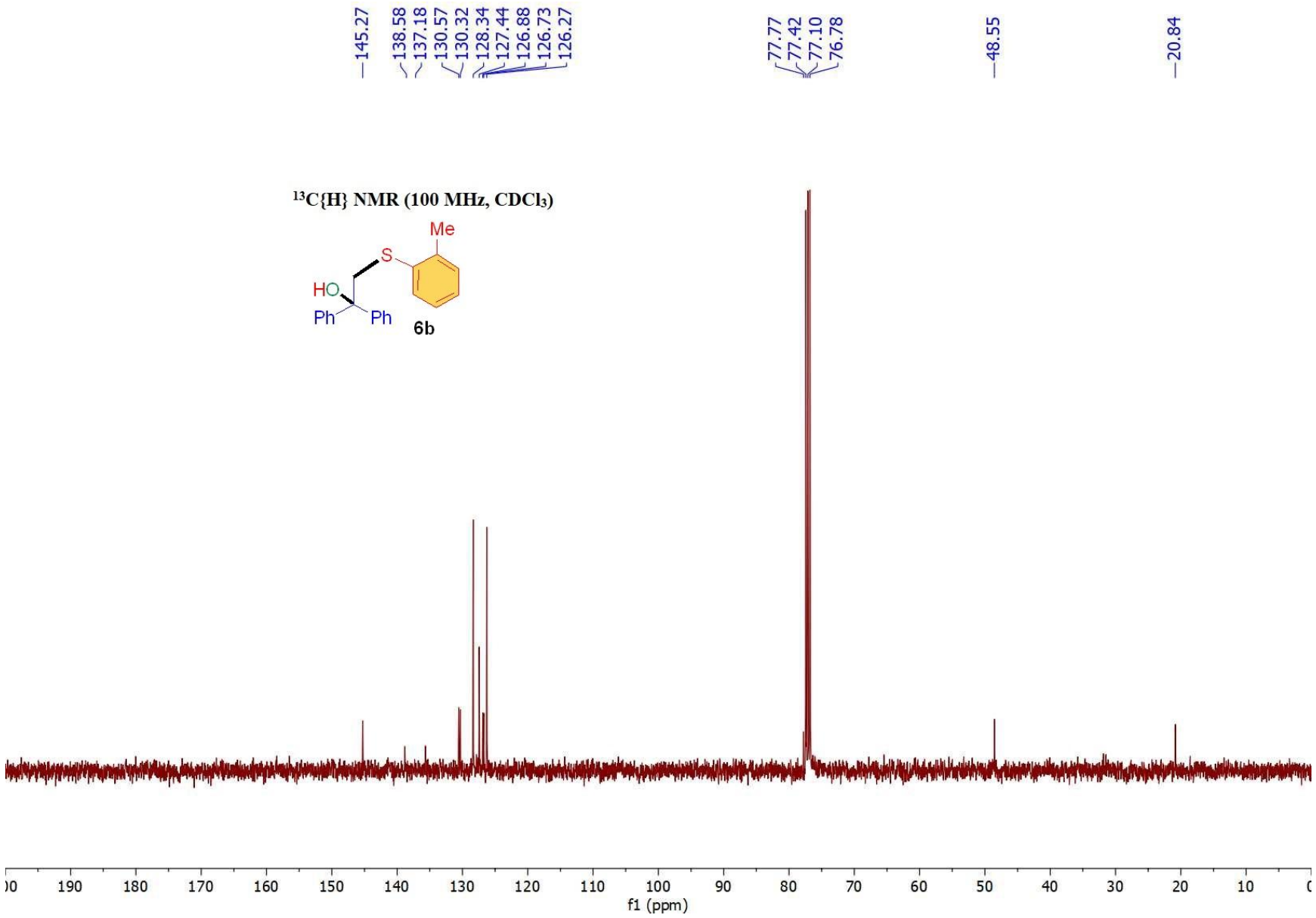


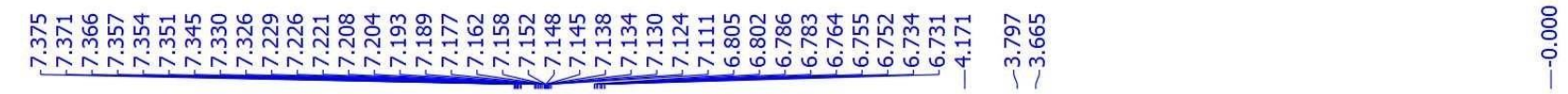
^{1}H NMR (400 MHz, $CDCl_3$)



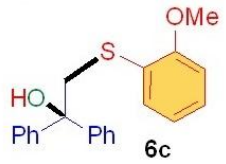
6b



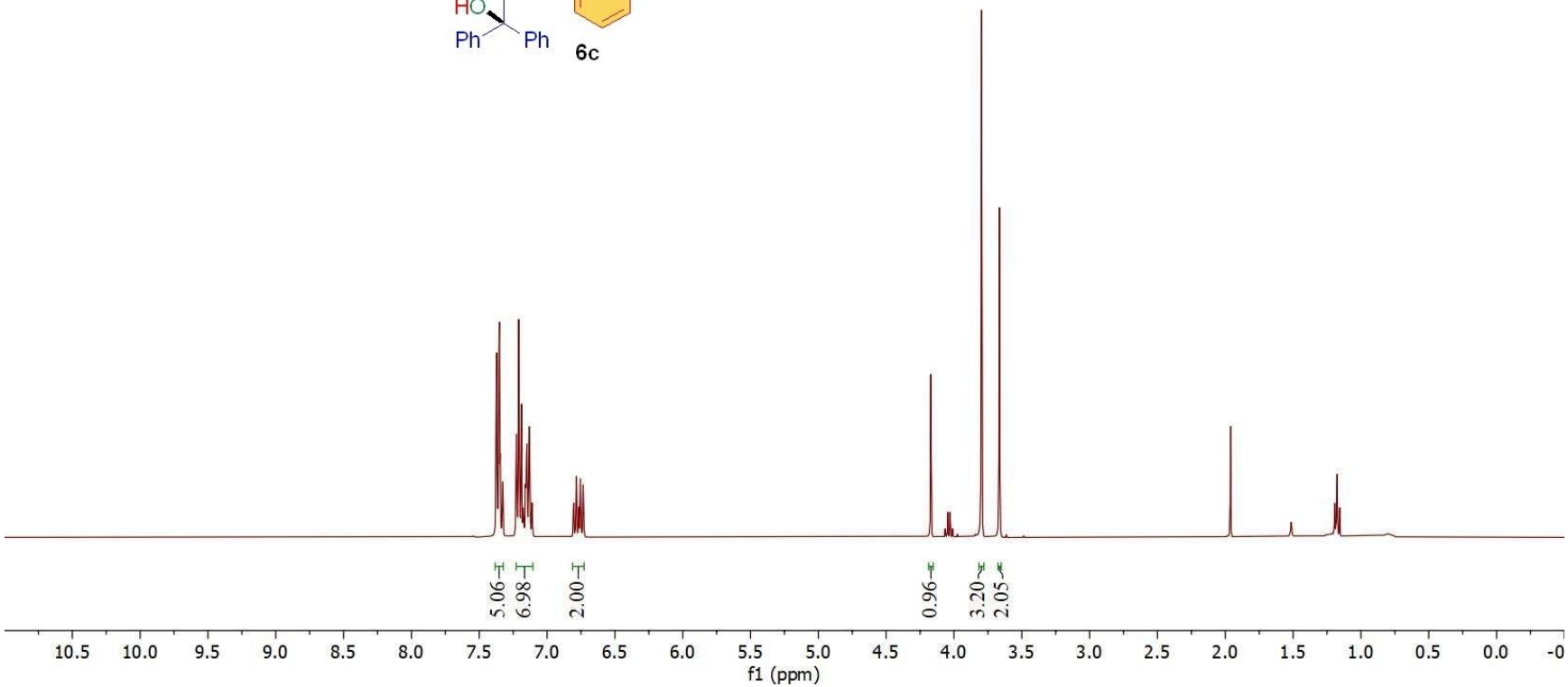

145.27
138.58
137.18
130.57
130.32
128.34
127.44
126.88
126.73
126.27
77.77
77.42
77.10
76.78
48.55
20.84
13C{H} NMR (100 MHz, CDCl3)
Me
S
HO
Ph
Ph
6b
f1 (ppm)



^{1}H NMR (400 MHz, $CDCl_3$)

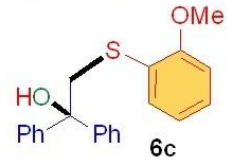


6c

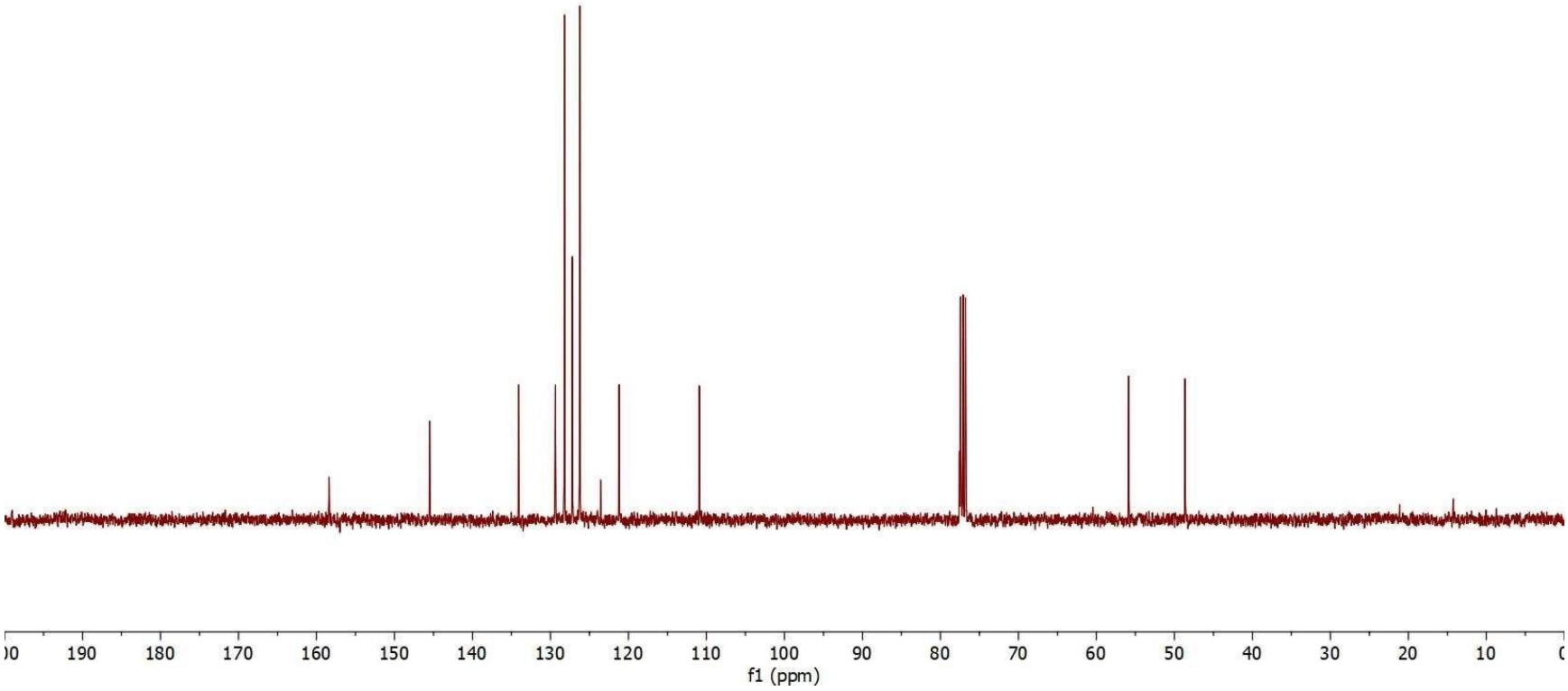


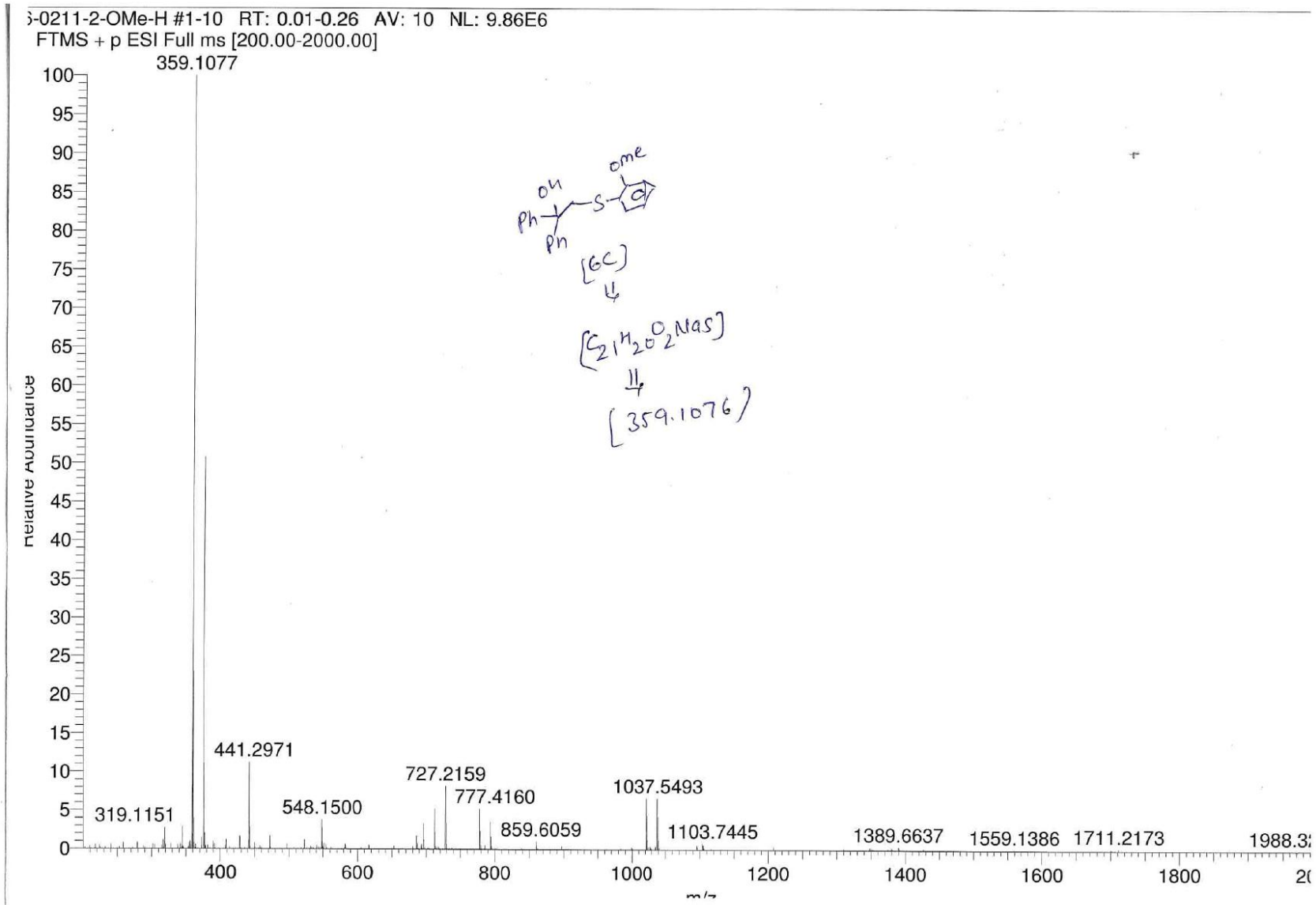
158.39
145.50
134.10
129.36
128.21
127.20
126.25
123.57
121.21
110.92
77.54
77.42
77.10
76.78
55.86
48.64

$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)

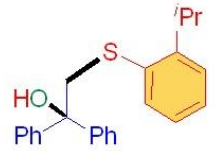


6c

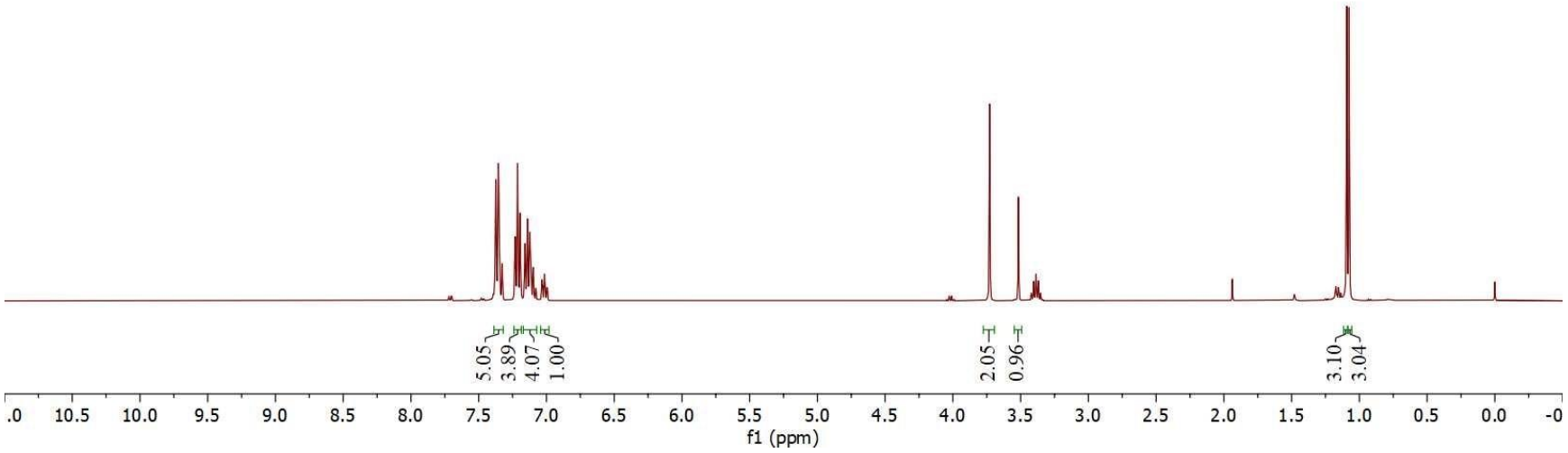


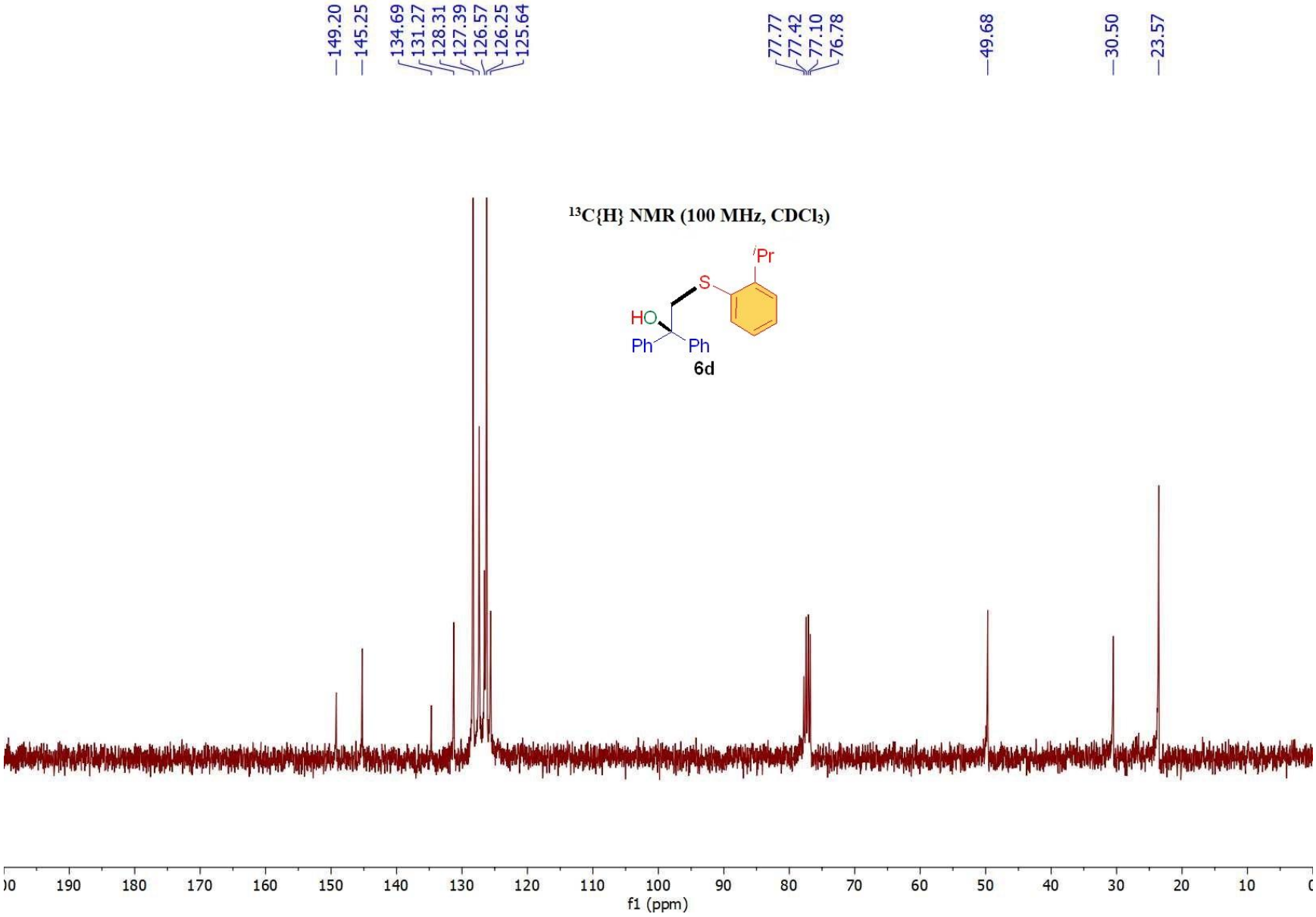

S-0211-2-OMe-H #1-10 RT: 0.01-0.26 AV: 10 NL: 9.86E6
FTMS + p ESI Full ms [200.00-2000.00]
359.1077
441.2971
319.1151
548.1500
727.2159
777.4160
859.6059
1037.5493
1103.7445
1389.6637
1559.1386
1711.2173
Relative Abundance
m/z
OH
Ph
Ph
S
OMe
[GC]
[C21H20O2NaS]
[359.1076]

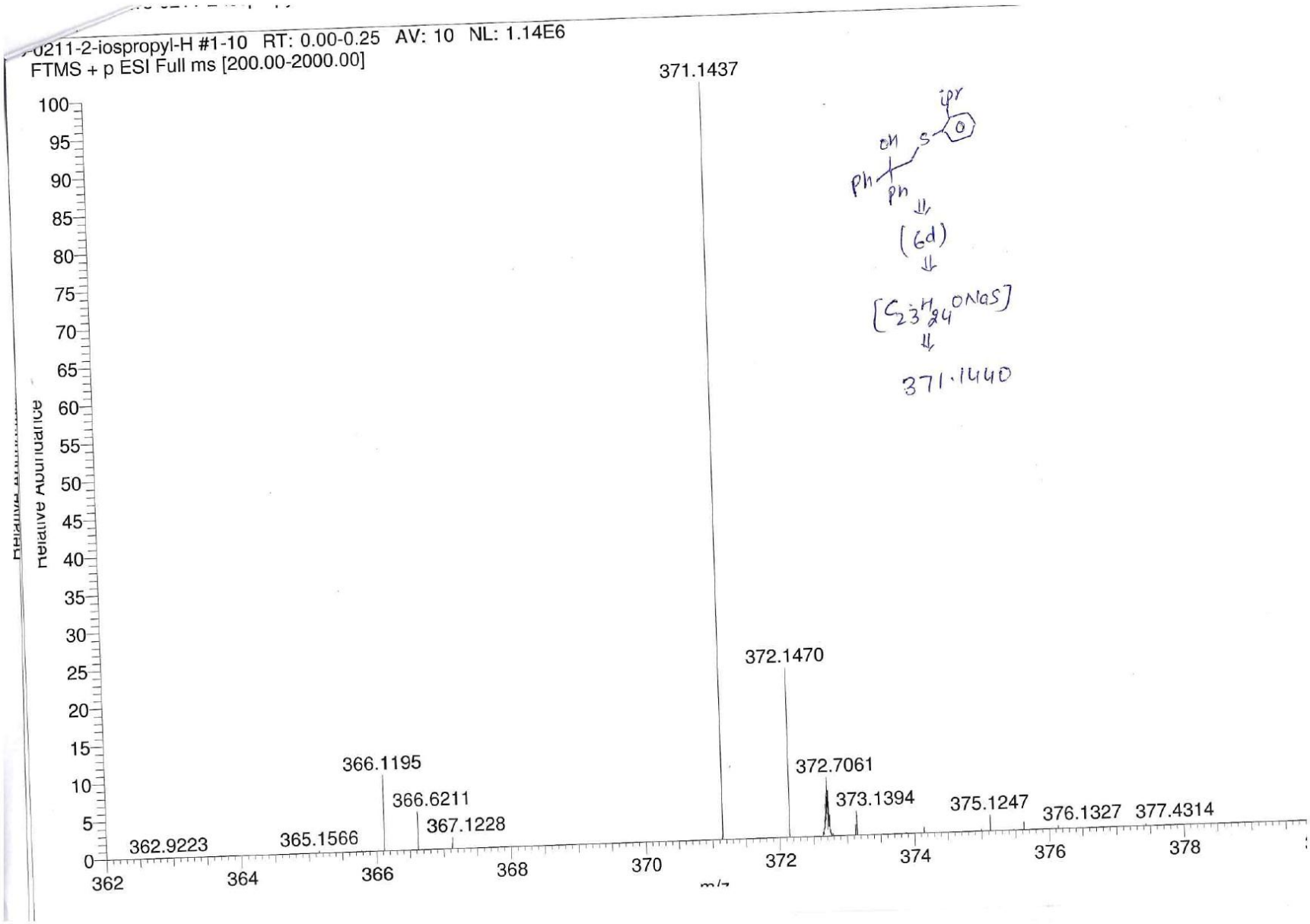
^{1}H NMR (400 MHz, $CDCl_3$)

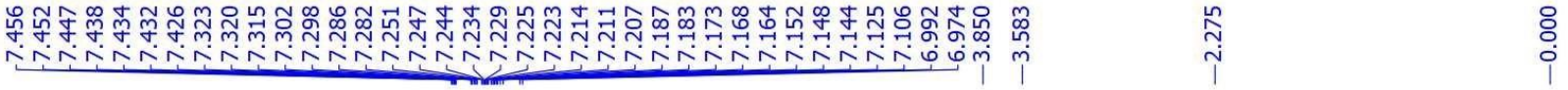


6d

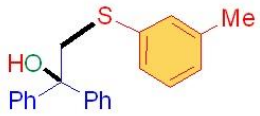



149.20
145.25
134.69
131.27
128.31
127.39
126.57
126.25
125.64
77.77
77.42
77.10
76.78
49.68
30.50
23.57
13C{H} NMR (100 MHz, CDCl3)
iPr
S
HO
Ph
Ph
6d
f1 (ppm)

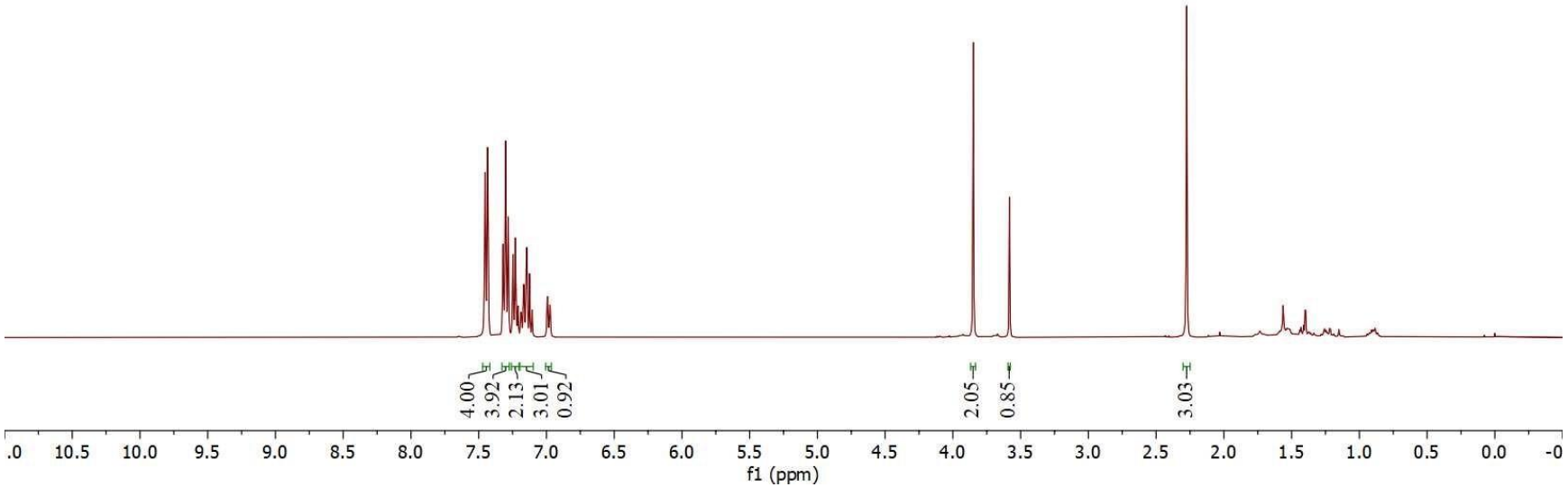

0211-2-iospropyl-H #1-10 RT: 0.00-0.25 AV: 10 NL: 1.14E6
FTMS + p ESI Full ms [200.00-2000.00]
Relative Abundance
m/z
371.1437
372.1470
366.1195
366.6211
367.1228
362.9223
365.1566
372.7061
373.1394
375.1247
376.1327
377.4314
OH
S
ipr
O
Ph
Ph
(6d)
[C23H24ONaS]
371.1440

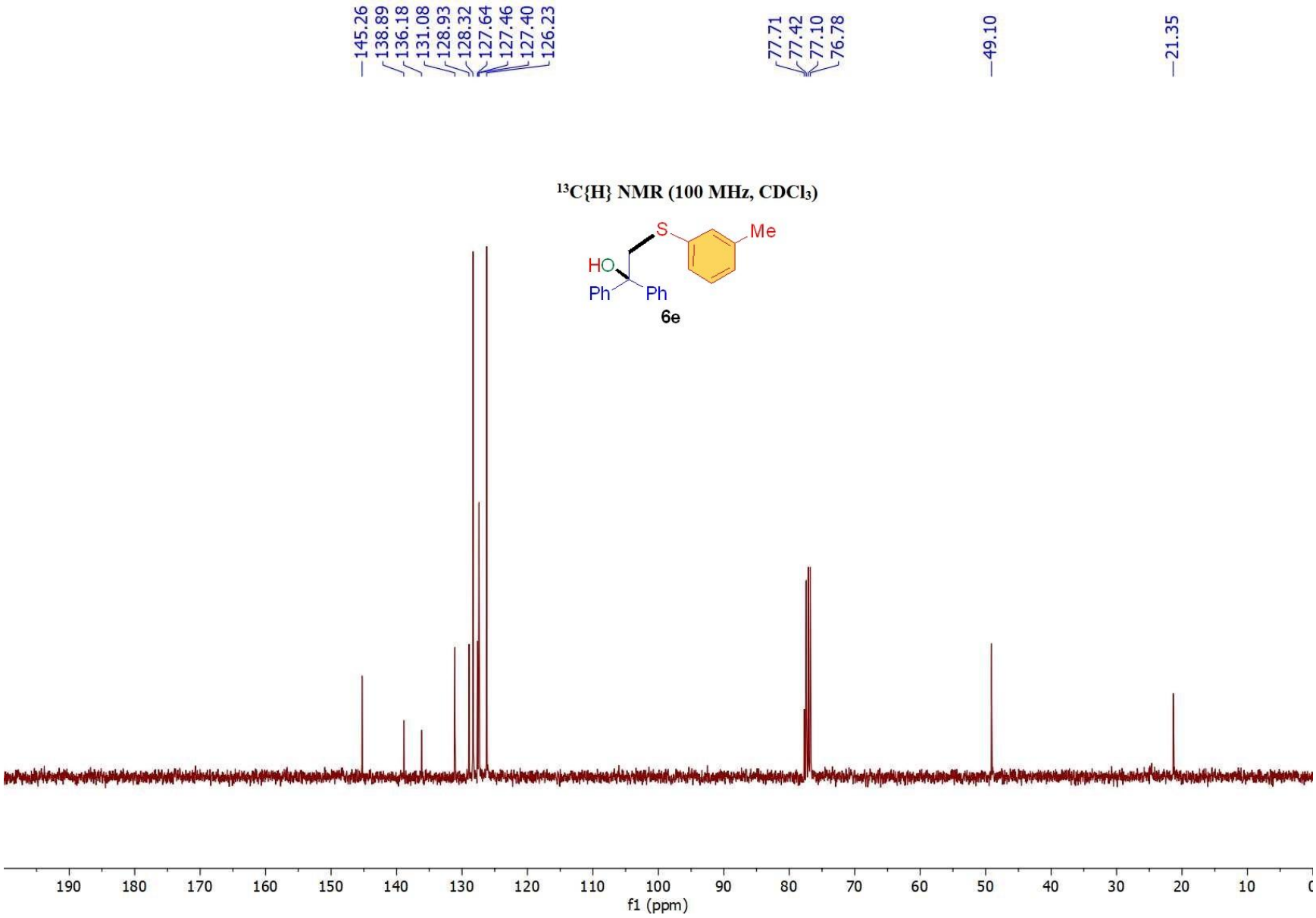


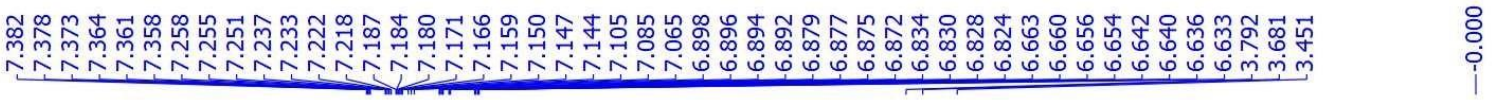
^{1}H NMR (400 MHz, $CDCl_3$)



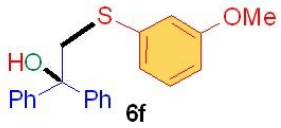
6e




145.26
138.89
136.18
131.08
128.93
128.32
127.64
127.46
127.40
126.23
77.71
77.42
77.10
76.78
49.10
21.35
13C{H} NMR (100 MHz, CDCl3)
S
Me
HO
Ph
Ph
6e
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)



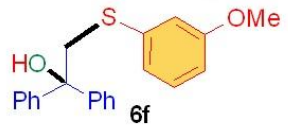
^{1}H NMR (400 MHz, $CDCl_3$)



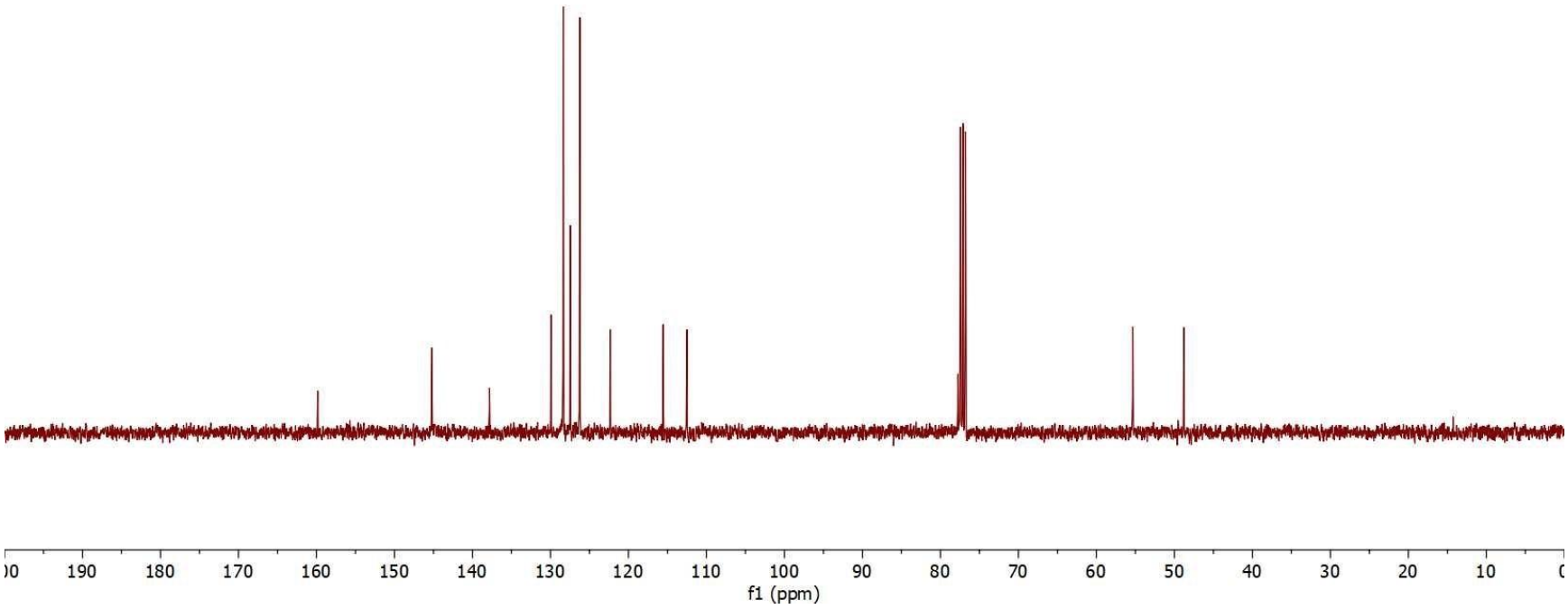
6f

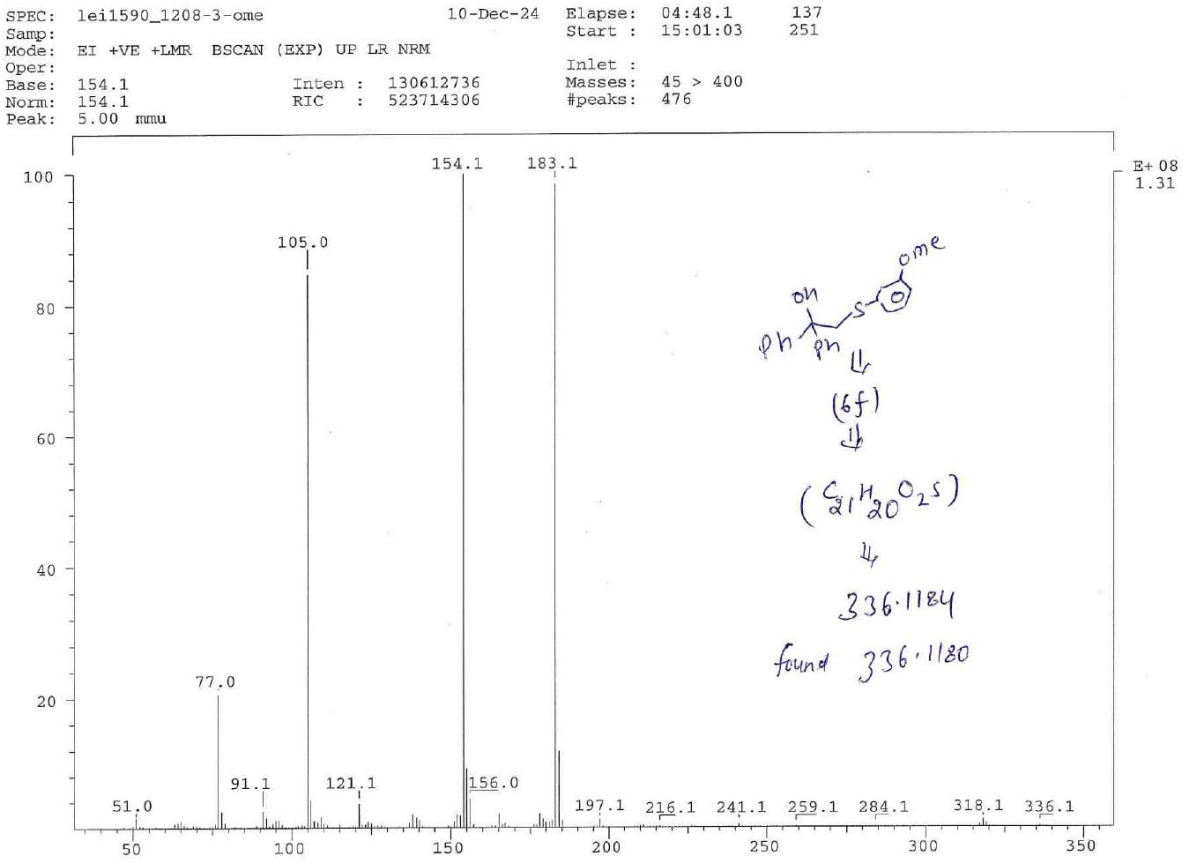
159.84
145.19
137.82
129.93
128.36
127.46
126.23
122.34
115.54
112.49
77.77
77.42
77.10
76.78
55.34
48.79

$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)

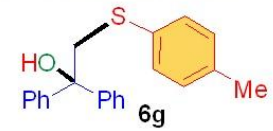


6f

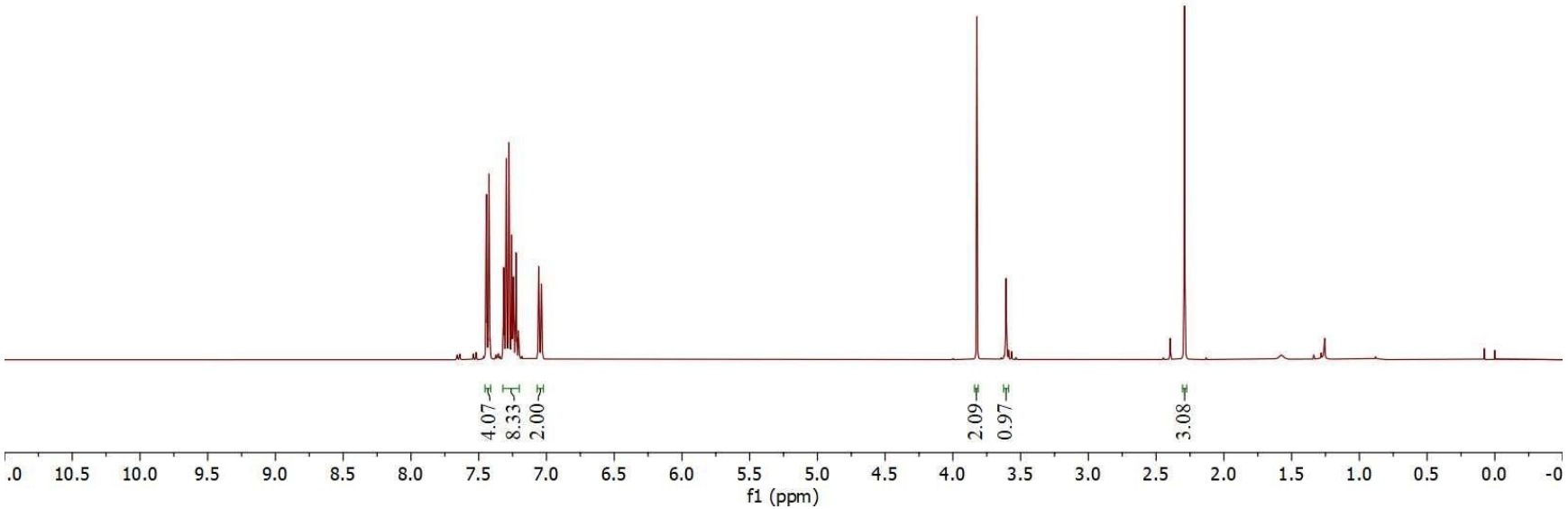



SPEC: lei1590_1208-3-ome
10-Dec-24
Elapse: 04:48.1 137
Samp:
Start : 15:01:03 251
Mode: EI +VE +LMR BSCAN (EXP) UP LR NRM
Oper:
Inlet :
Base: 154.1 Inten : 130612736
Masses: 45 > 400
Norm: 154.1 RIC : 523714306
#peaks: 476
Peak: 5.00 mmu
E+ 08
1.31
100
80
60
40
20
154.1
183.1
105.0
77.0
91.1
121.1
156.0
51.0
197.1
216.1
241.1
259.1
284.1
318.1
336.1
50
100
150
200
250
300
350
OH
Ph
Ph
S
O
OMe
(6f)
($C_{21}H_{20}O_2S$)
336.1184
found 336.1180

^{1}H NMR (400 MHz, $CDCl_3$)

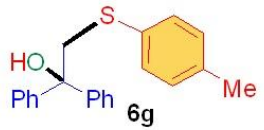


6g

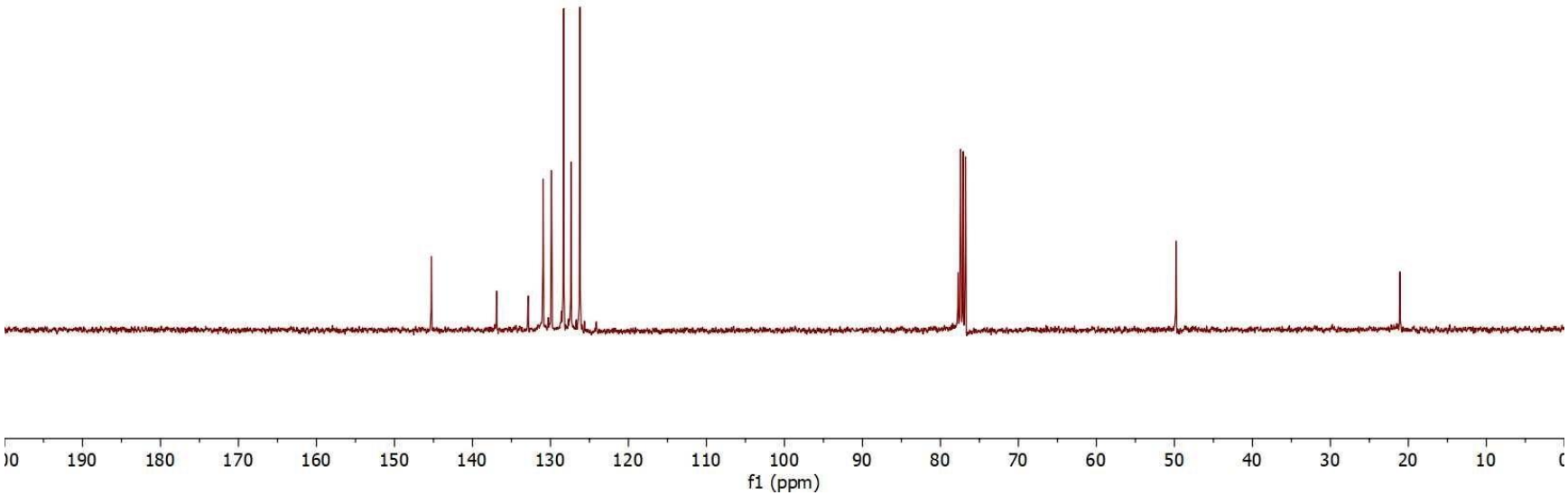


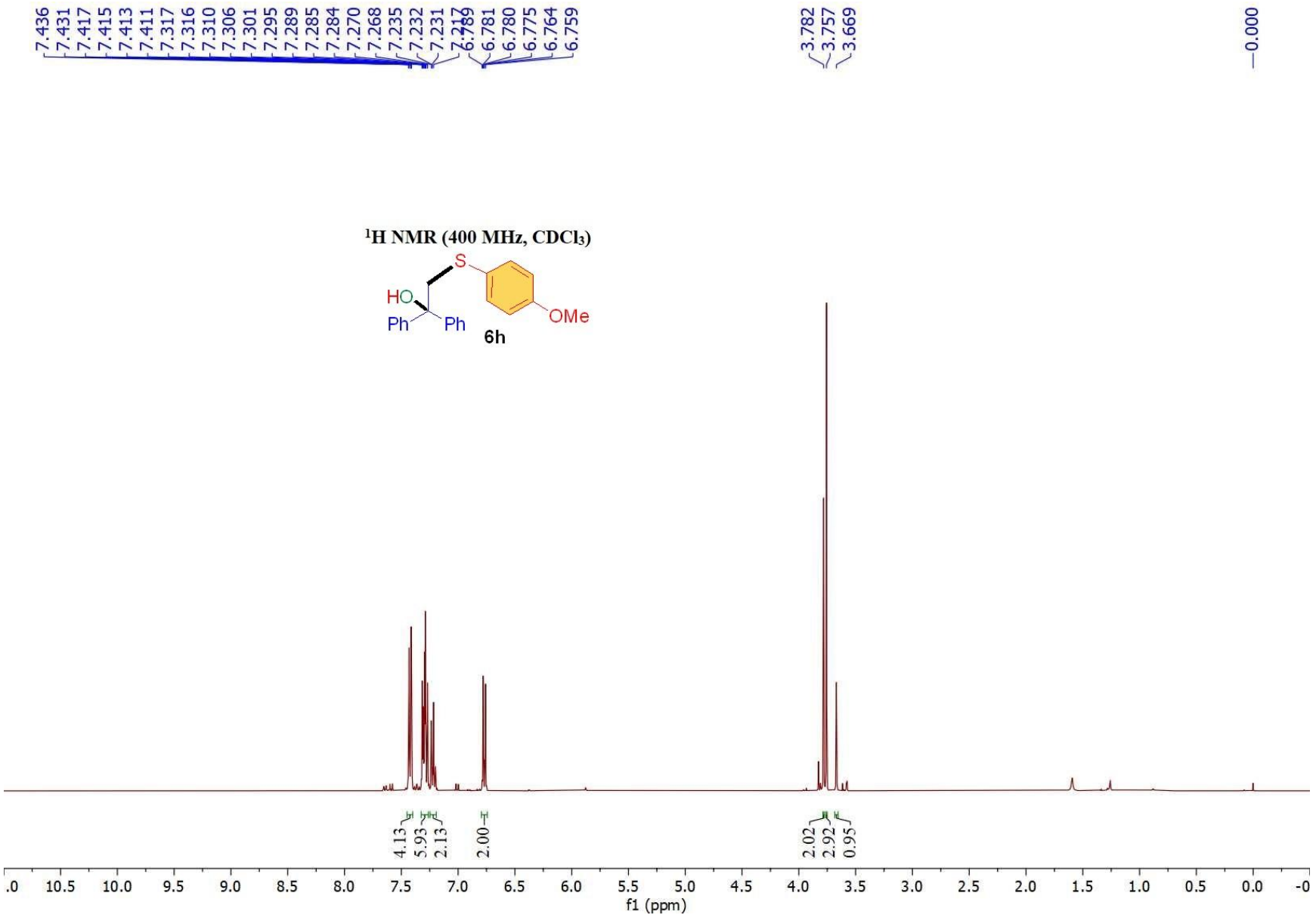
145.28
136.91
132.86
130.95
129.87
128.31
127.36
126.22
77.75
77.41
77.10
76.78
49.79
21.07

$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)



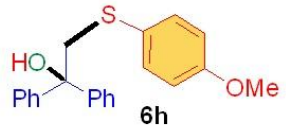
6g



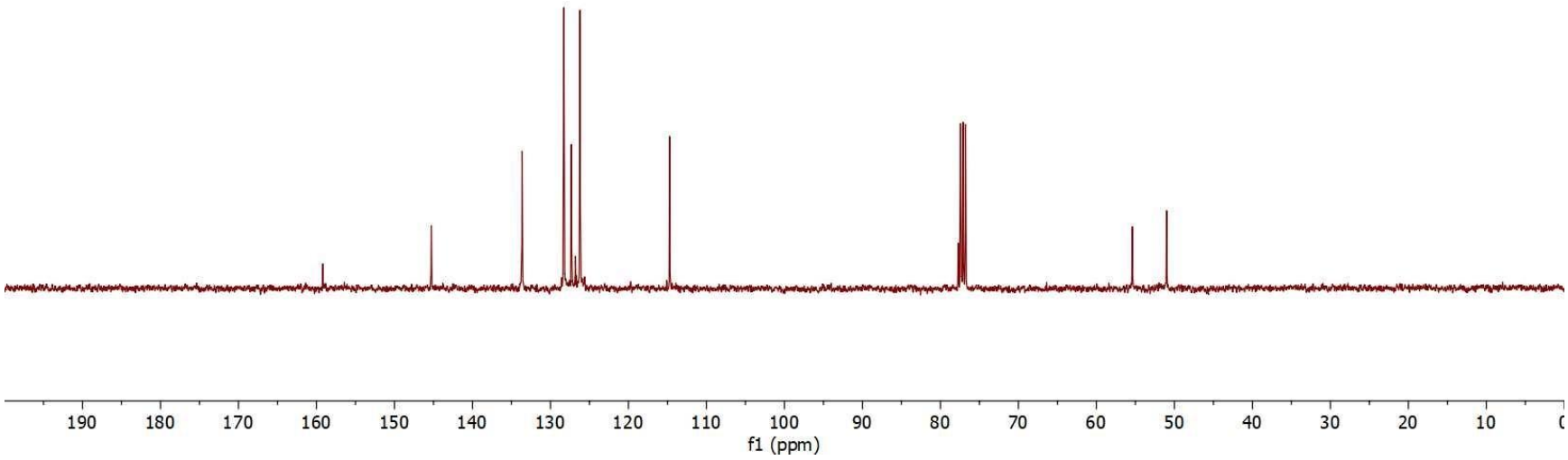

7.436
7.431
7.417
7.415
7.413
7.411
7.317
7.316
7.310
7.306
7.301
7.295
7.289
7.285
7.284
7.270
7.268
7.235
7.232
7.231
7.217
6.789
6.781
6.780
6.775
6.764
6.759
3.782
3.757
3.669
0.000
1H NMR (400 MHz, CDCl3)
S
HO
Ph
Ph
OMe
6h
4.13
5.93
2.13
2.00
2.02
2.92
0.95
f1 (ppm)

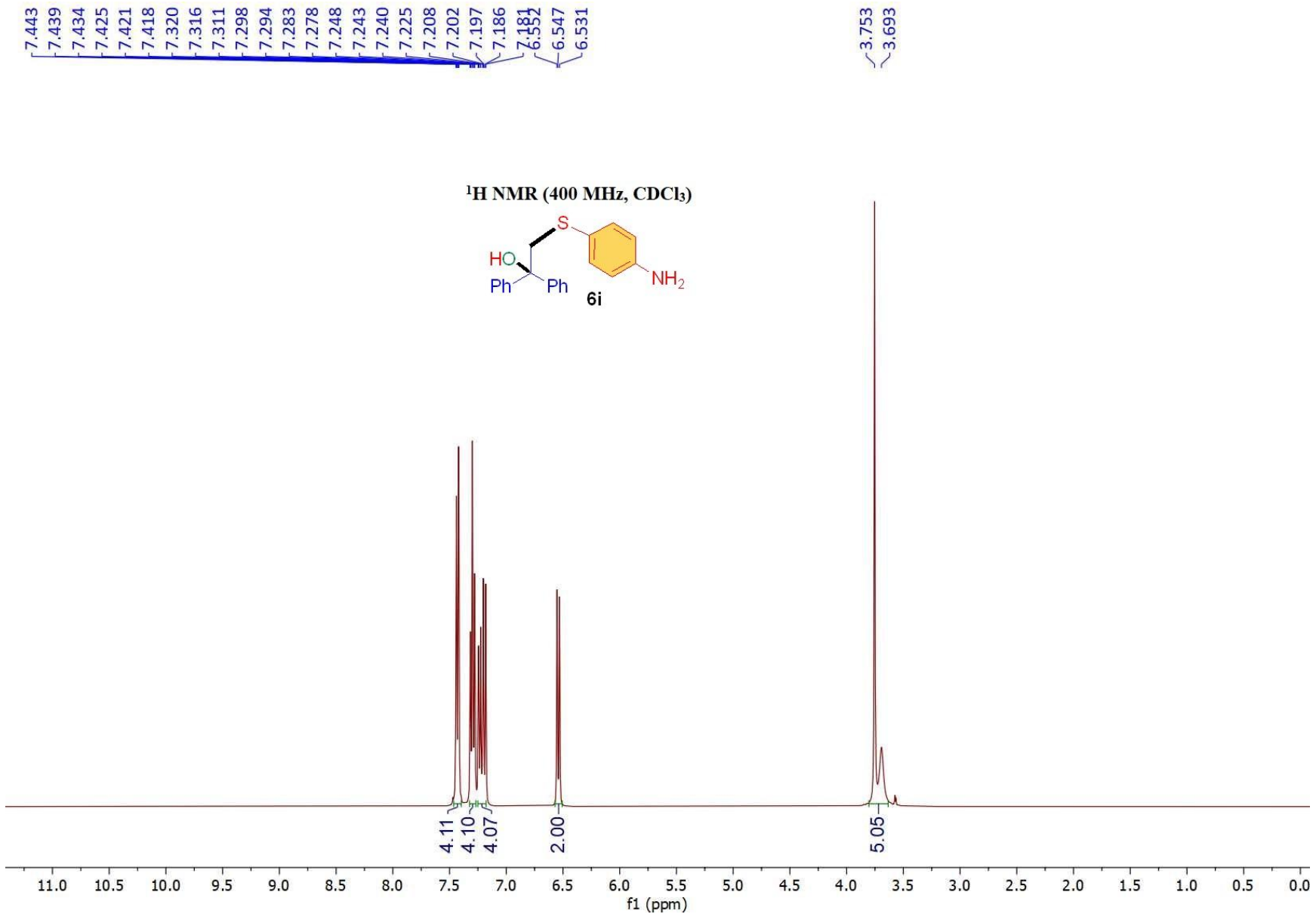
159.18
145.28
133.64
128.28
127.32
126.79
126.22
114.73
77.72
77.42
77.10
76.78
55.40
50.99

$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)



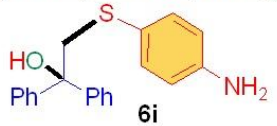
6h



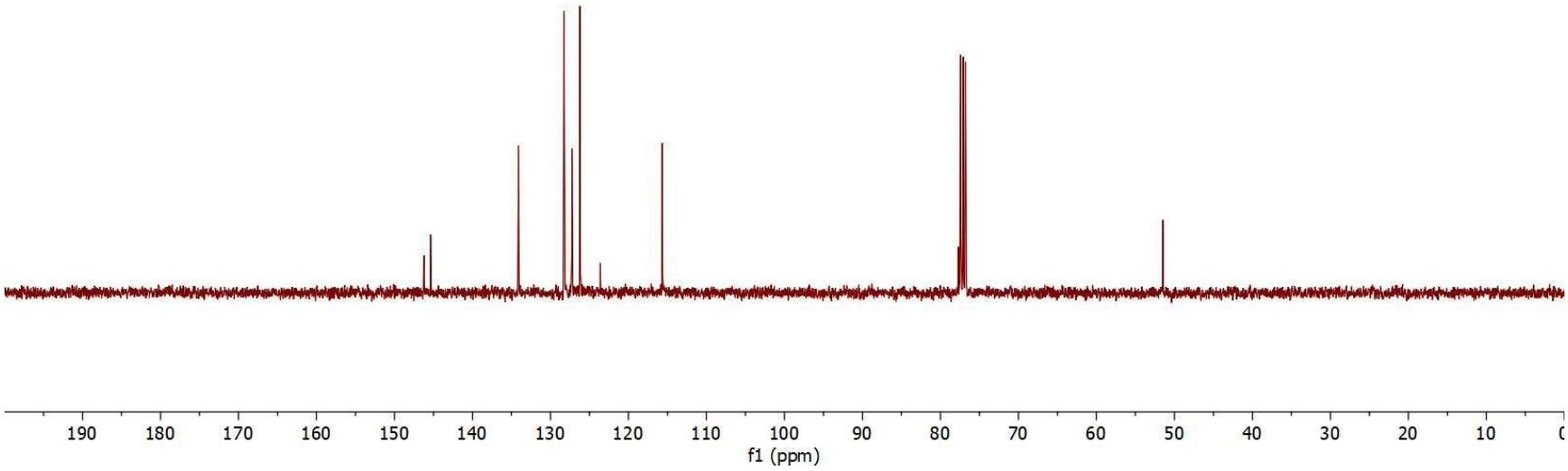

7.443
7.439
7.434
7.425
7.421
7.418
7.320
7.316
7.311
7.298
7.294
7.283
7.278
7.248
7.243
7.240
7.225
7.208
7.202
7.197
7.186
6.547
6.531
3.753
3.693
1H NMR (400 MHz, CDCl3)
S
HO
Ph
Ph
NH2
6i
4.11
4.10
4.07
2.00
5.05
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

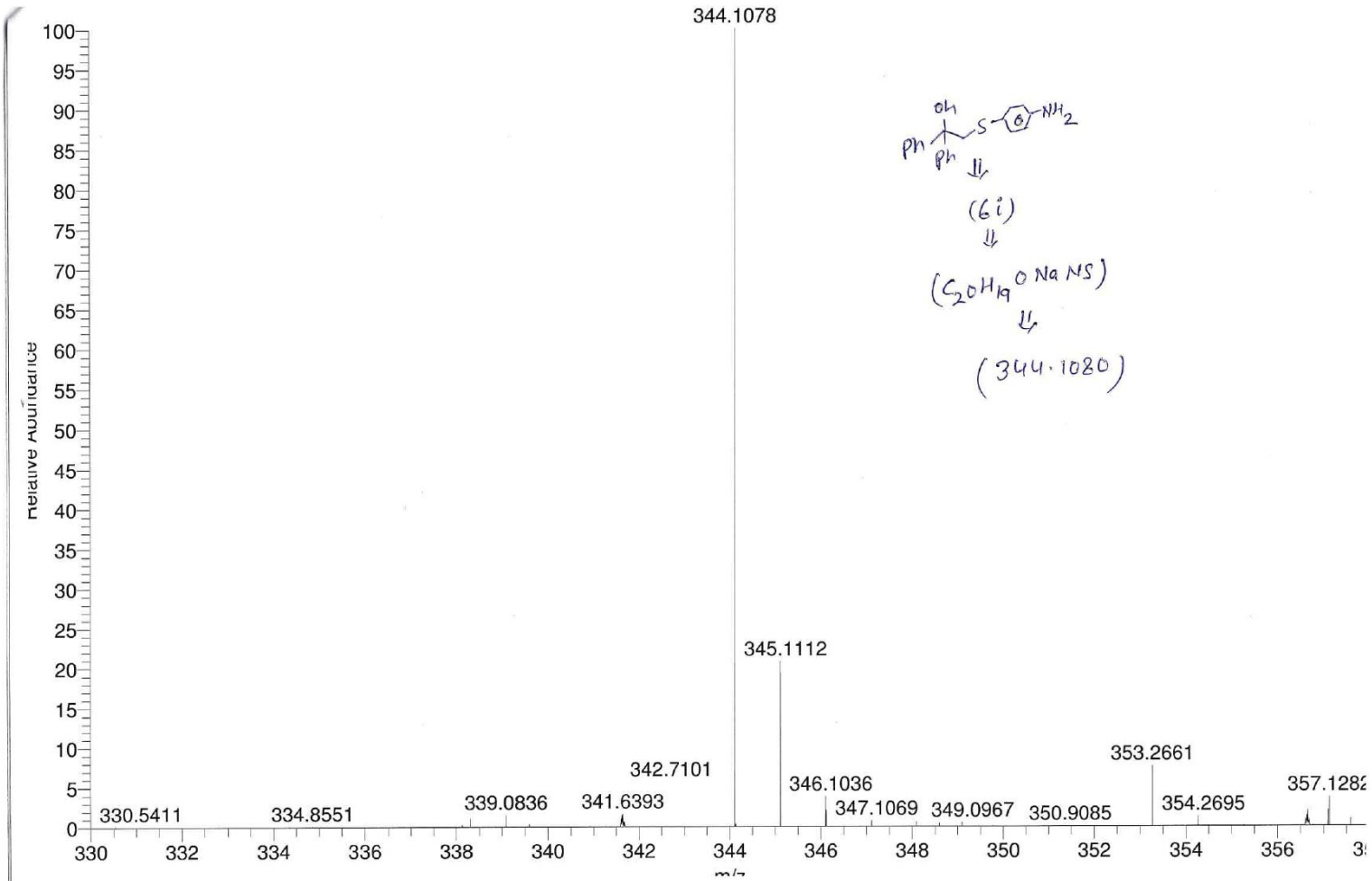
146.21
145.37
134.12
128.25
127.25
126.23
123.60
115.66
77.70
77.42
77.10
76.78
51.49

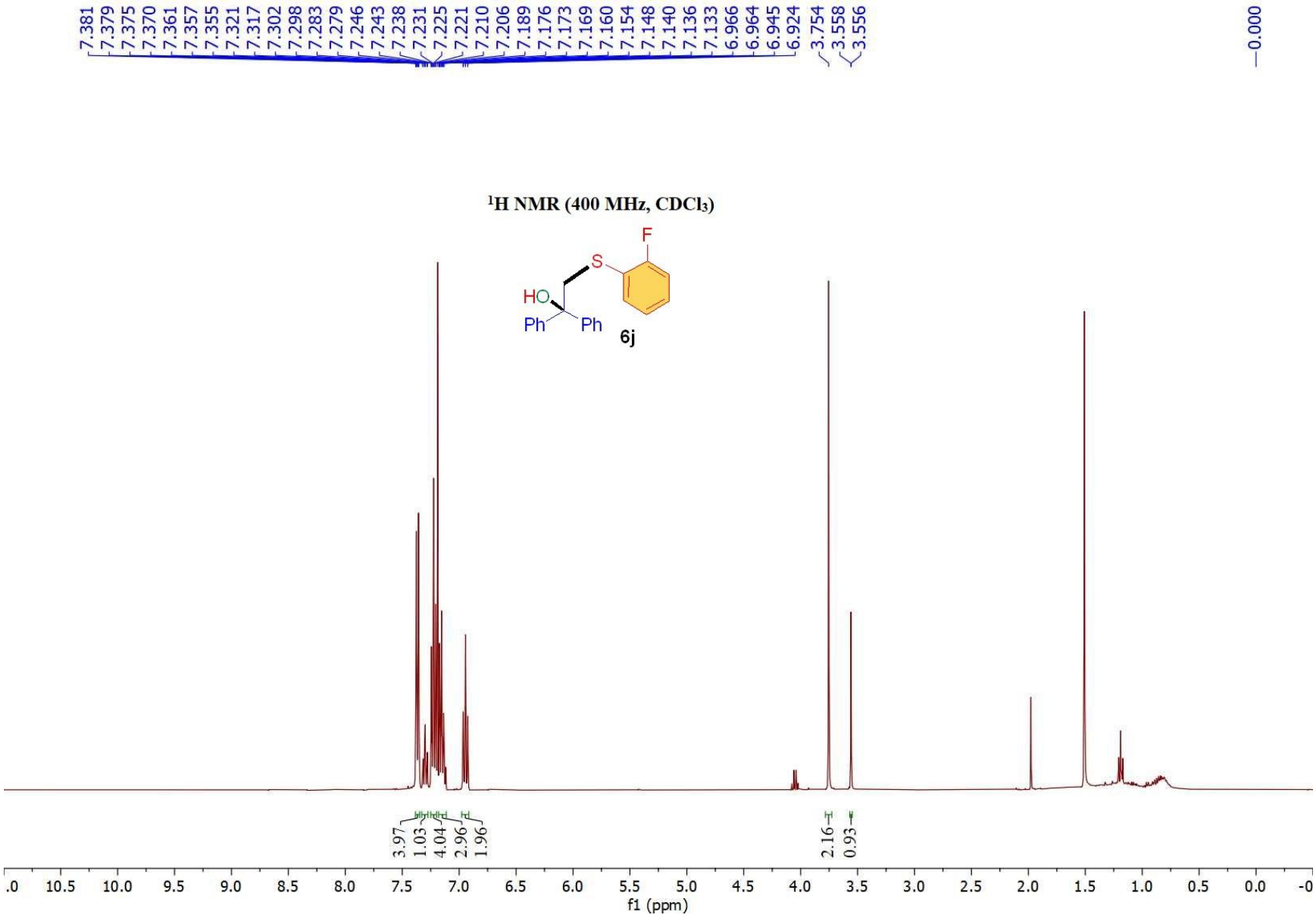
$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)

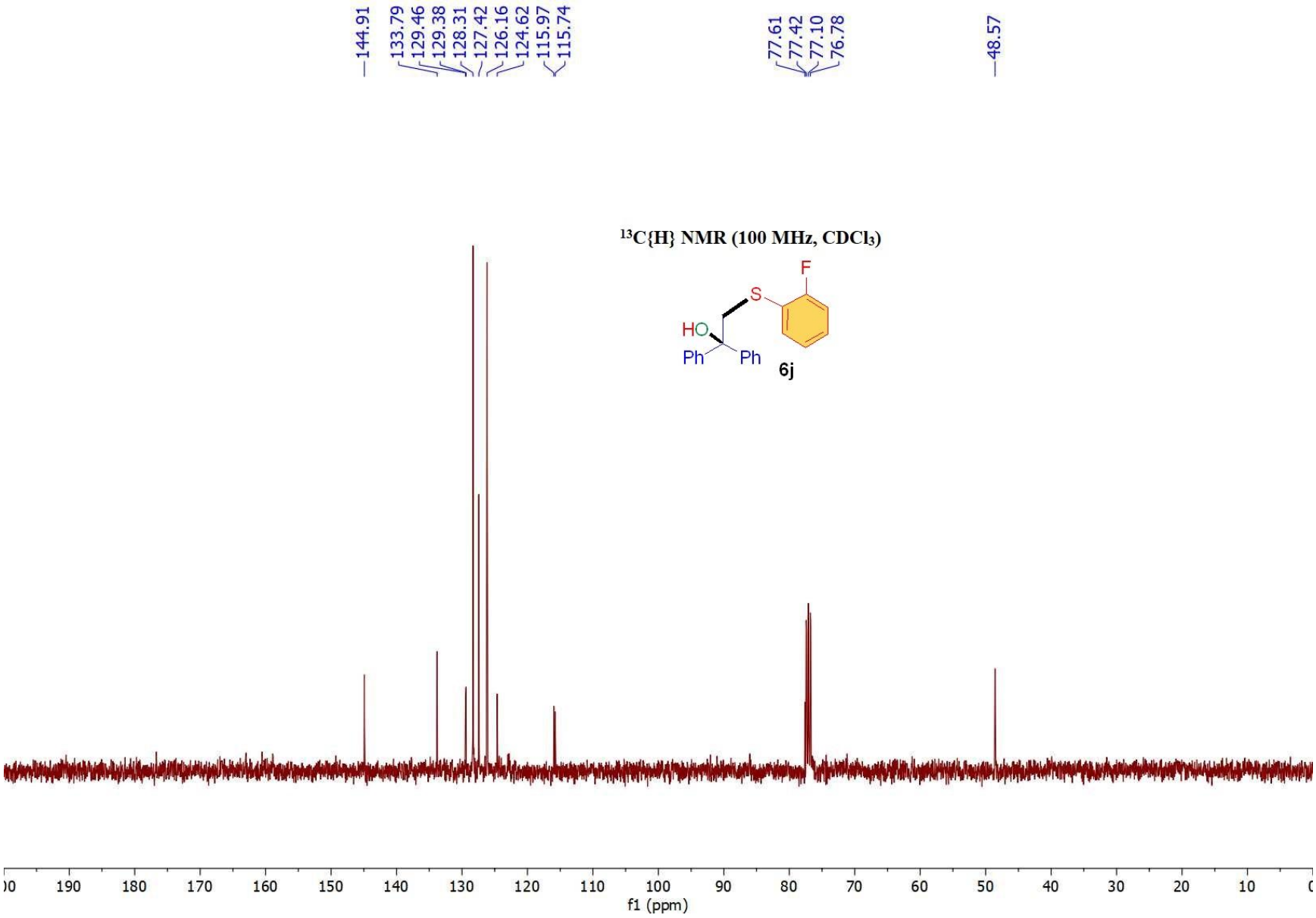


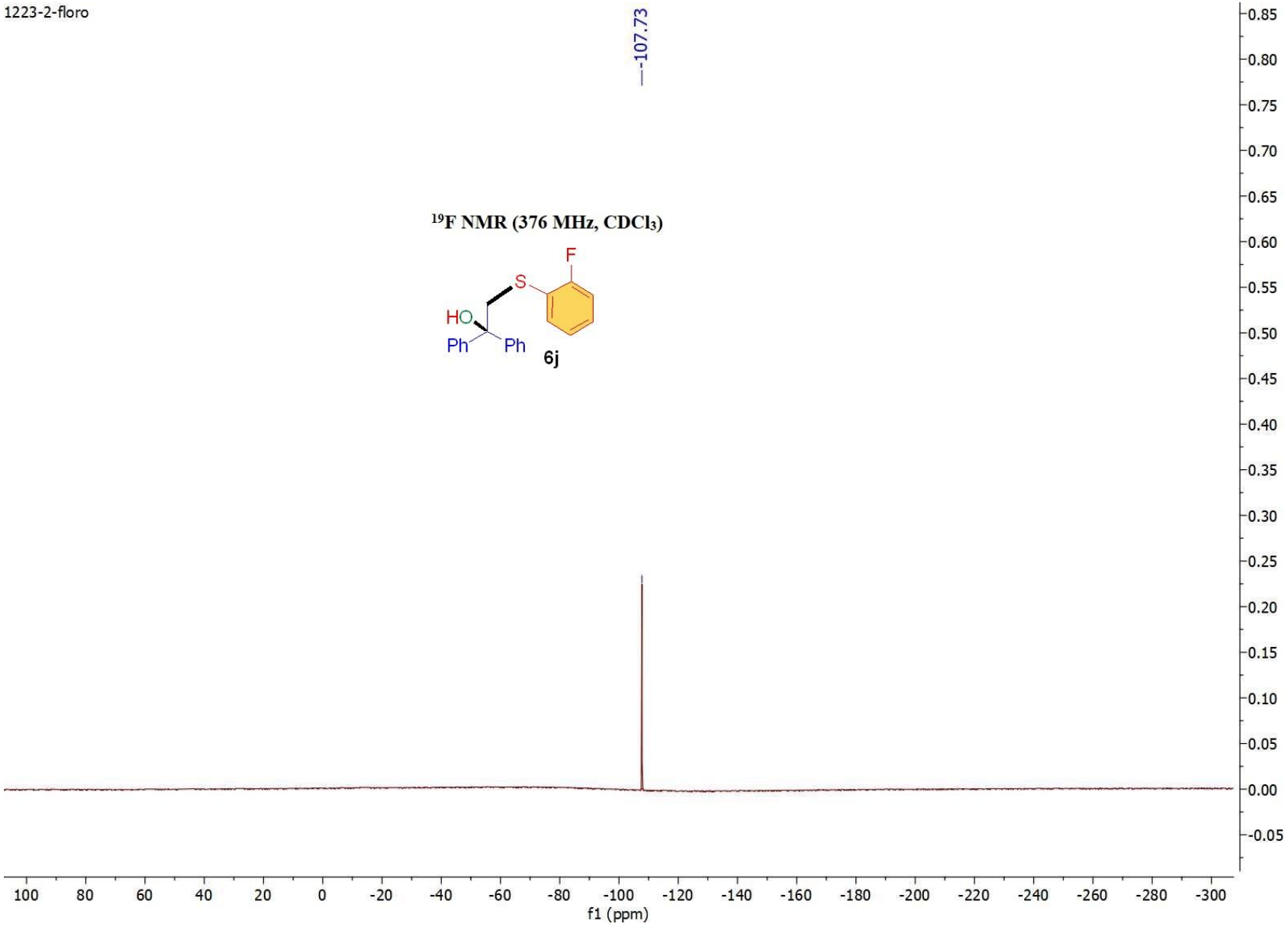
6i

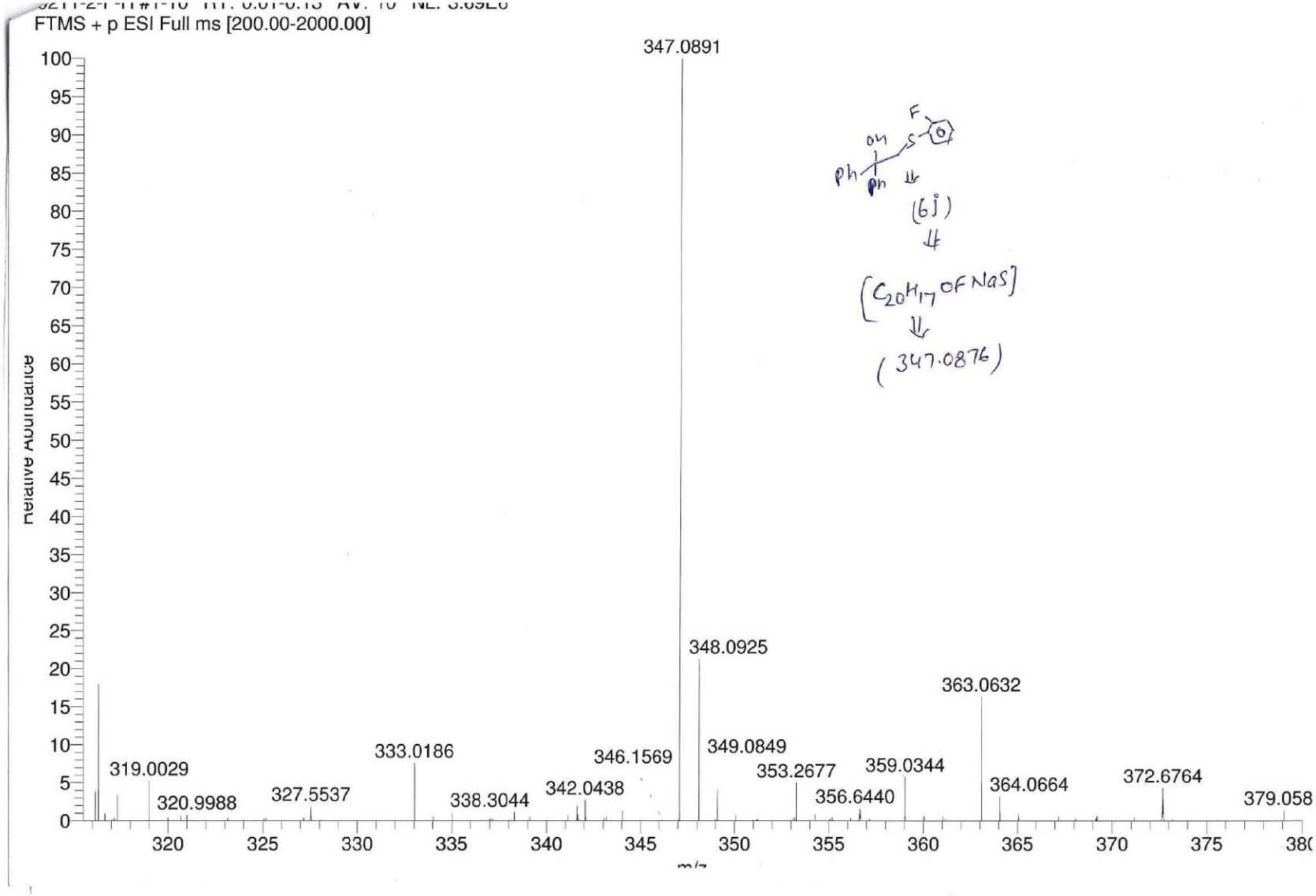



344.1078
345.1112
353.2661
342.7101
346.1036
357.1282
339.0836
341.6393
347.1069
349.0967
350.9085
354.2695
330.5411
334.8551
Relative Abundance
m/z
(6i)
(C20H19ONaNS)
(344.1080)

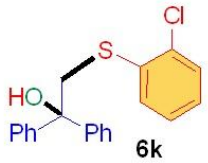

7.381
7.379
7.375
7.370
7.361
7.357
7.355
7.321
7.317
7.302
7.298
7.283
7.279
7.246
7.243
7.238
7.231
7.225
7.221
7.210
7.206
7.189
7.176
7.173
7.169
7.160
7.154
7.148
7.140
7.136
7.133
6.966
6.964
6.945
6.924
3.754
3.558
3.556
0.000
1H NMR (400 MHz, CDCl3)
F
S
HO
Ph
Ph
6j
3.97
1.03
4.04
2.96
1.96
2.16
0.93
f1 (ppm)


144.91
133.79
129.46
129.38
128.31
127.42
126.16
124.62
115.97
115.74
77.61
77.42
77.10
76.78
48.57
13C{H} NMR (100 MHz, CDCl3)
F
S
HO
Ph
Ph
6j
f1 (ppm)

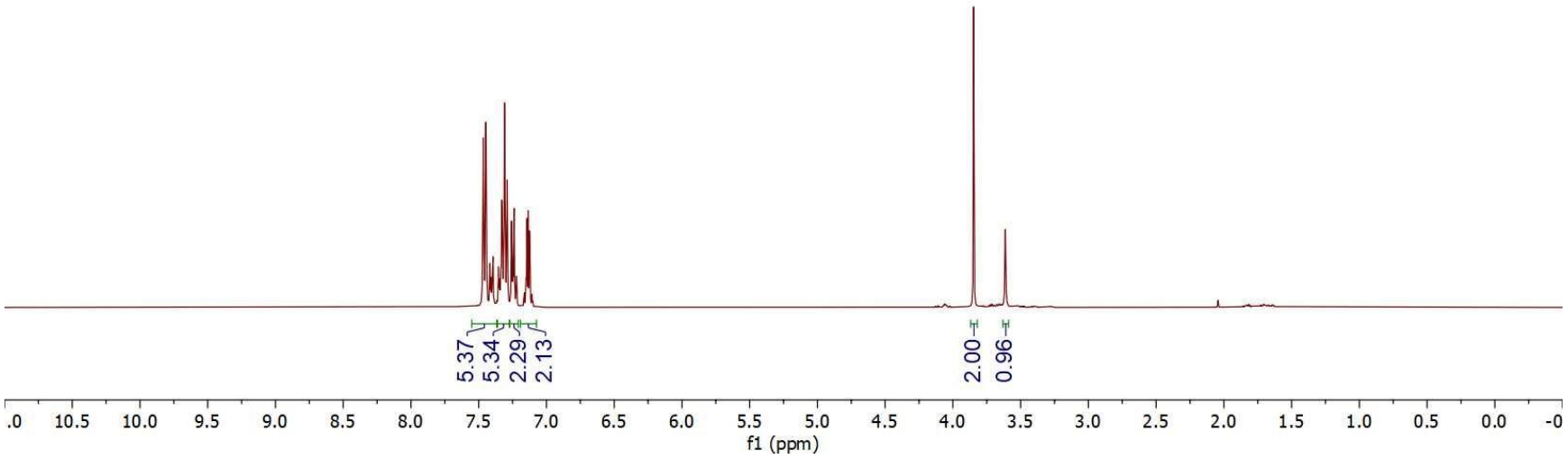

1223-2-floro
-107.73
19F NMR (376 MHz, CDCl3)
F
S
HO
Ph
Ph
6j
0.85
0.80
0.75
0.70
0.65
0.60
0.55
0.50
0.45
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00
-0.05
100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 -220 -240 -260 -280 -300
f1 (ppm)


FTMS + p ESI Full ms [200.00-2000.00]
Relative Abundance
m/z
347.0891
348.0925
363.0632
319.0029
320.9988
327.5537
333.0186
338.3044
342.0438
346.1569
349.0849
353.2677
356.6440
359.0344
364.0664
372.6764
379.058
(6j)
[C20H17OFNaS]
(347.0876)

^{1}H NMR (400 MHz, $CDCl_3$)

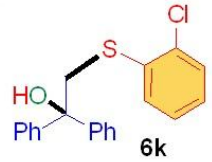


6k

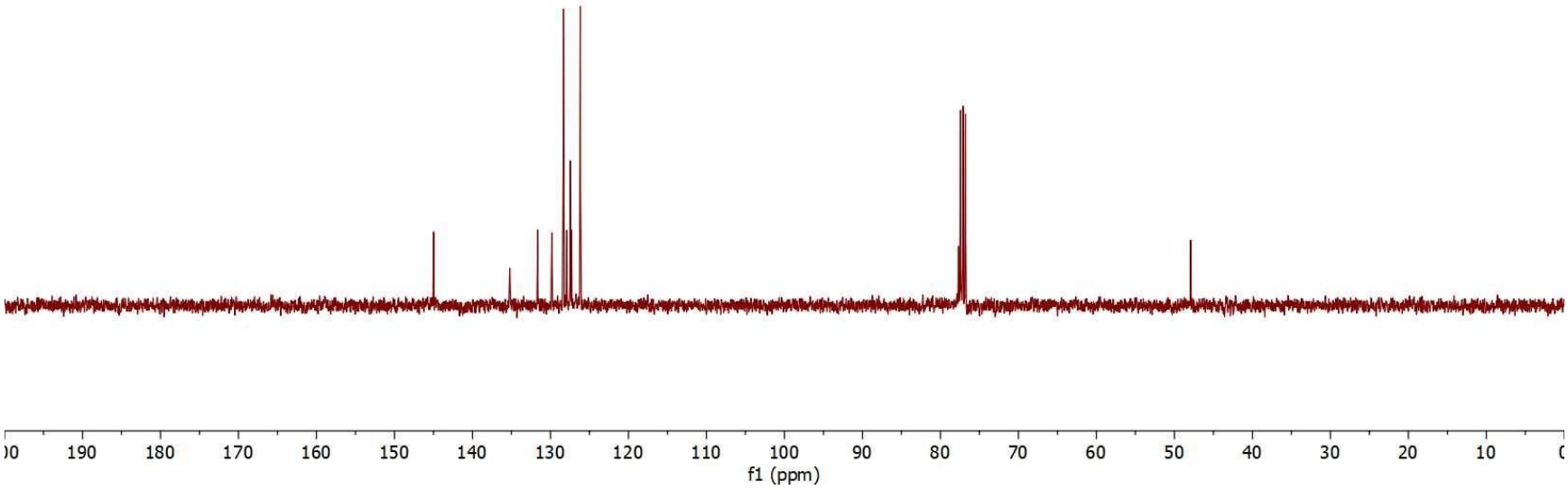


144.99
135.30
135.22
131.66
129.83
128.35
127.91
127.47
127.30
126.16
77.66
77.42
77.10
76.78
47.92

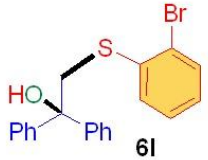
$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)



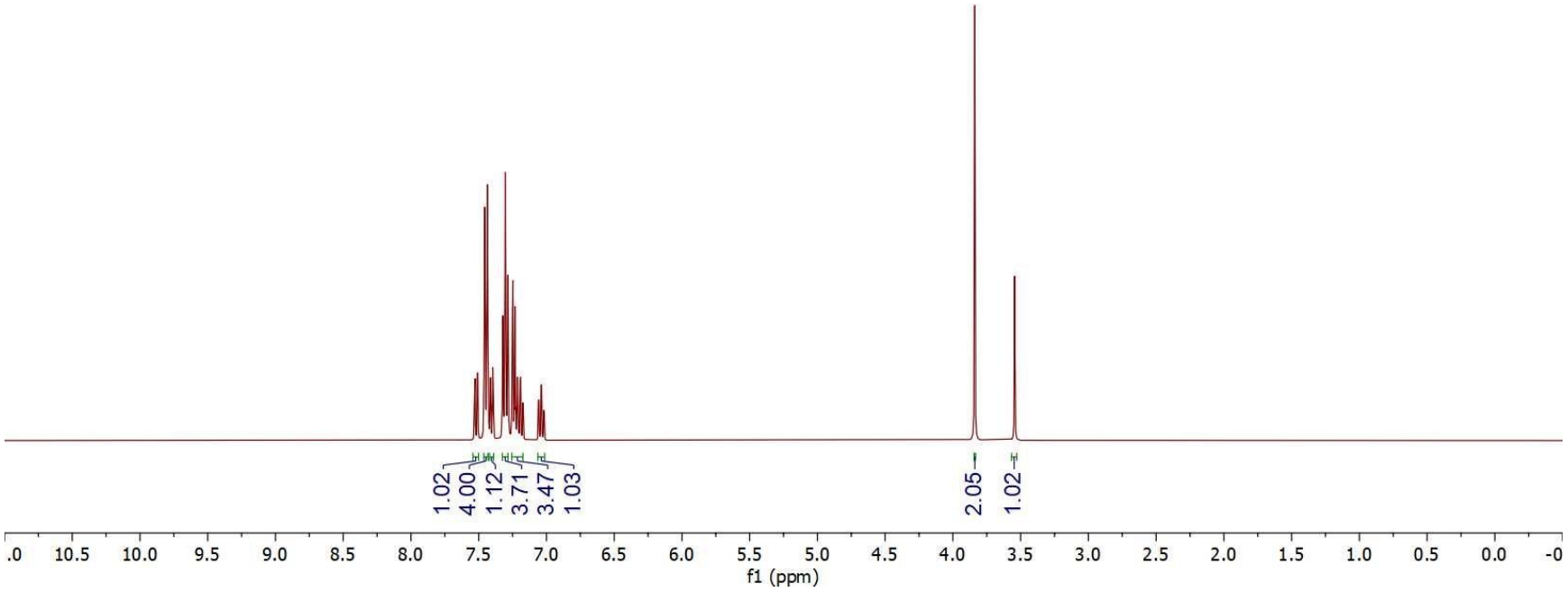
6k

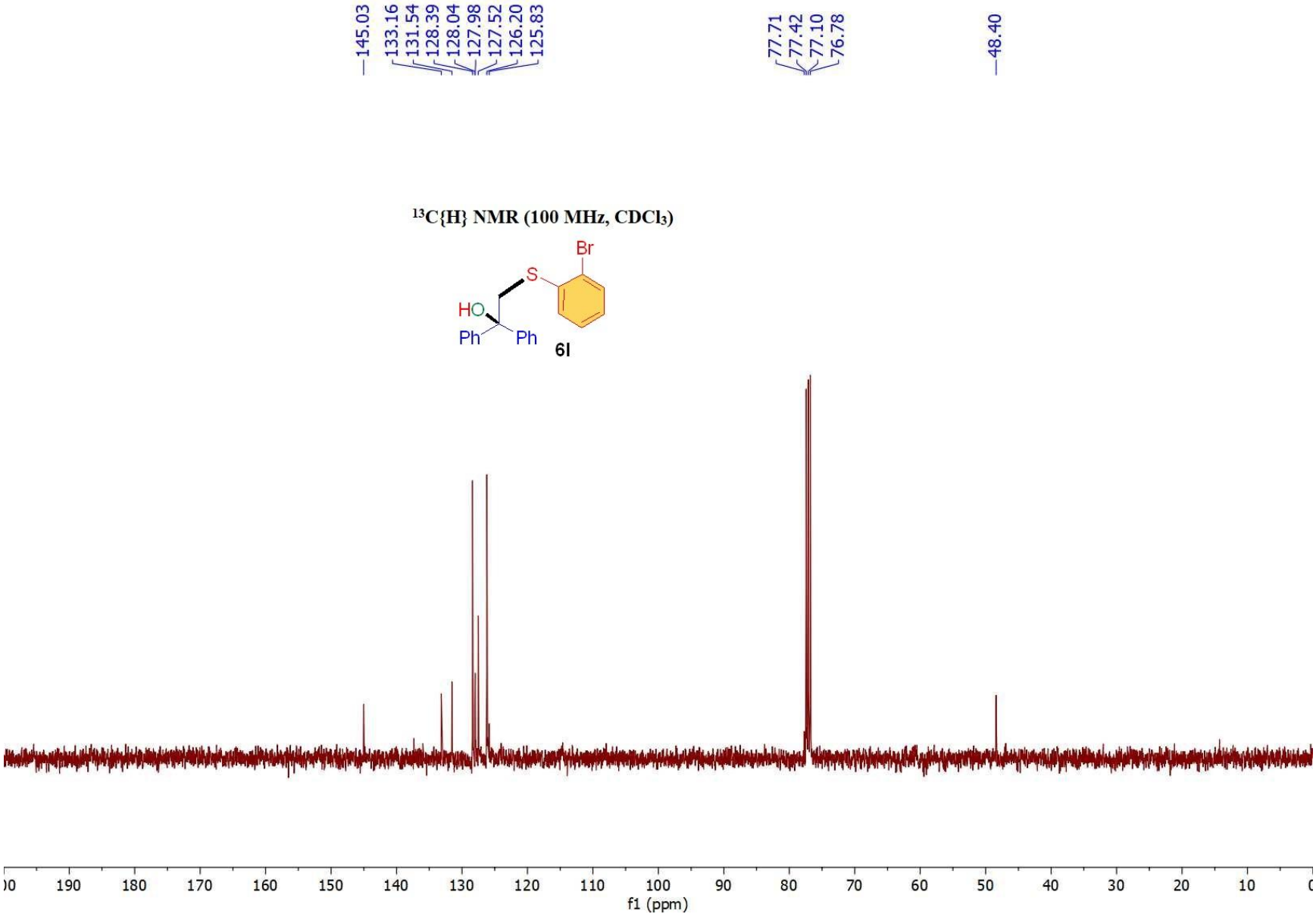


^{1}H NMR (400 MHz, $CDCl_3$)

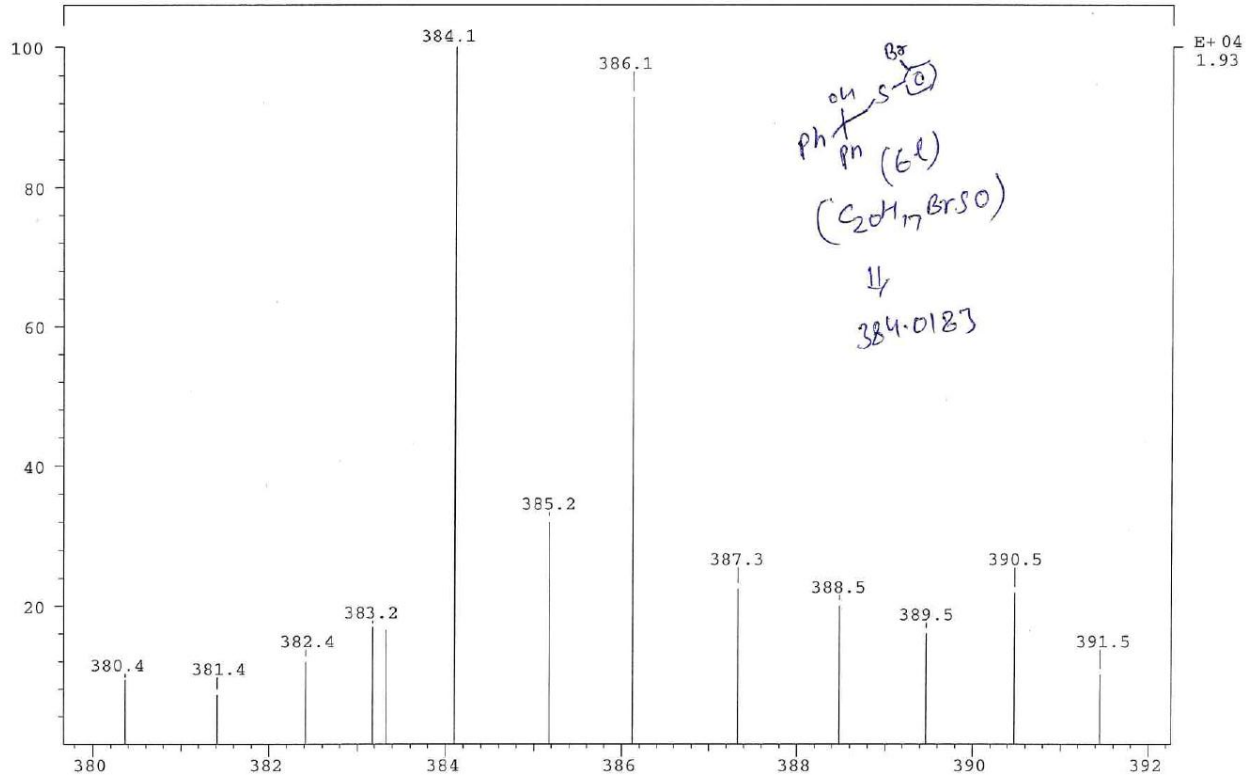


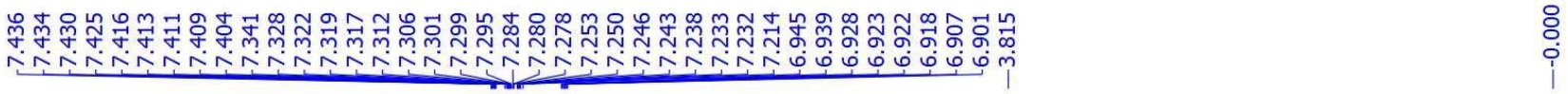
6l



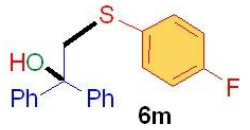

145.03
133.16
131.54
128.39
128.04
127.98
127.52
126.20
125.83
77.71
77.42
77.10
76.78
48.40
13C{H} NMR (100 MHz, CDCl3)
Br
S
HO
Ph
Ph
6l
f1 (ppm)

SPEC: lei1592_1208-2-br 10-Dec-24 Elapse: 03:31.3 99
Samp: Start : 15:32:13 520
Mode: EI +VE +LMR BSCAN (EXP) UP LR NRM
Oper: Inlet :
Base: 183.1 Inten : 52062976 Masses: 45 > 400
Norm: 384.1 RIC : 262255990 #peaks: 463
Peak: 5.00 mmu

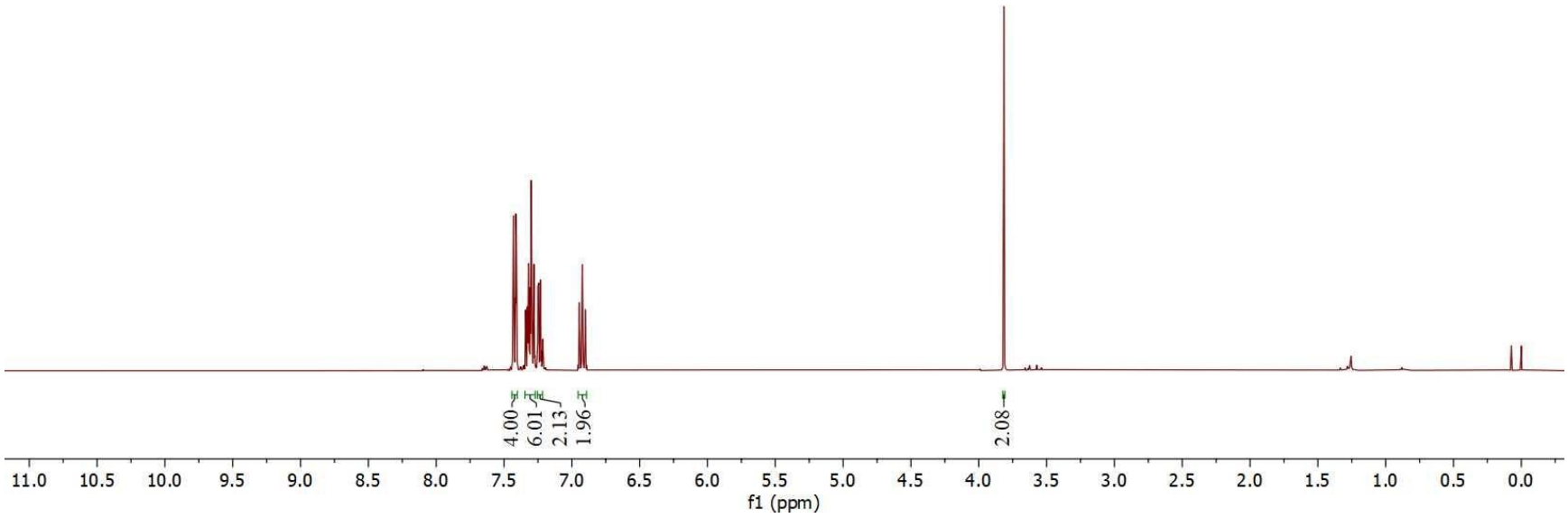


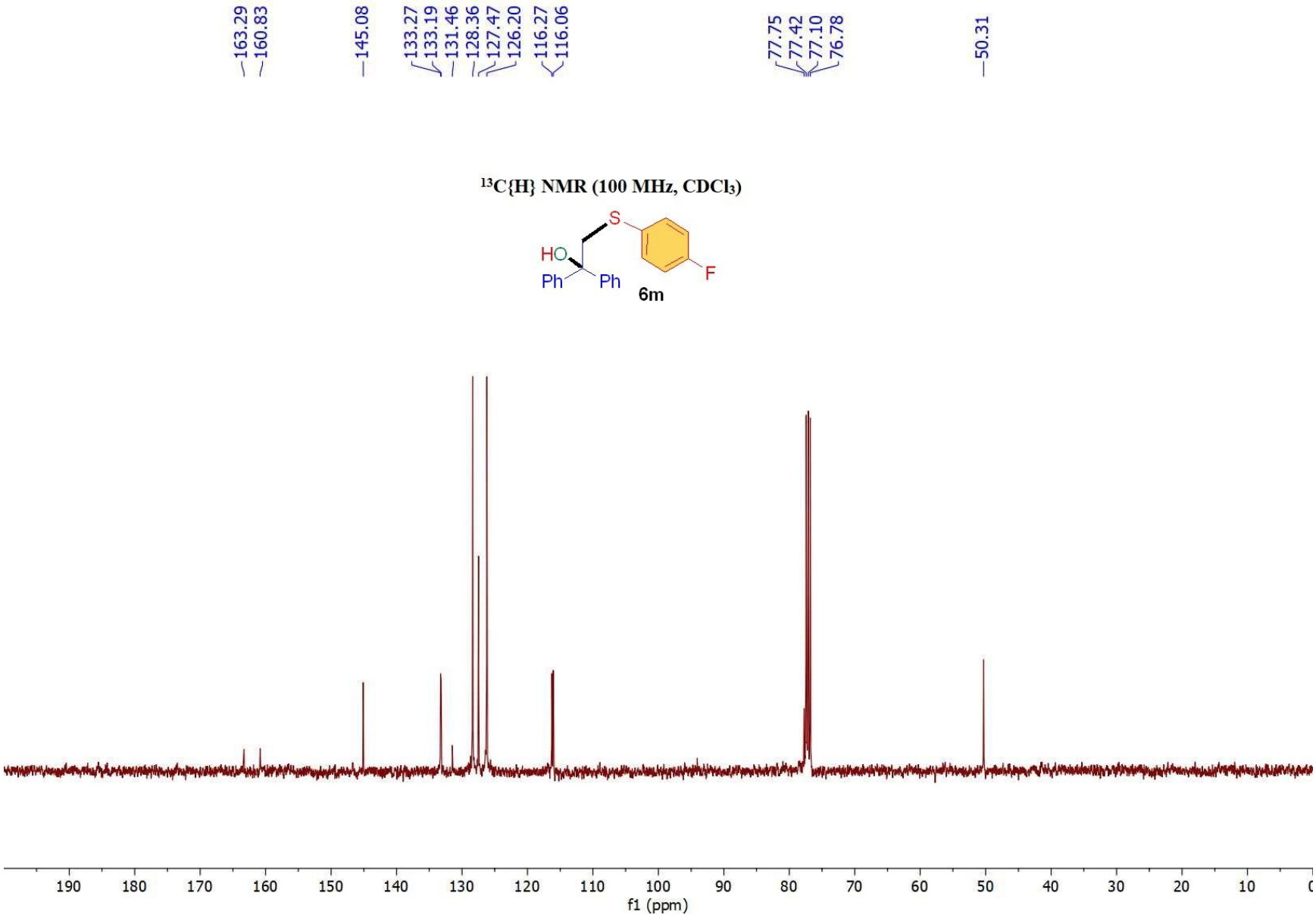


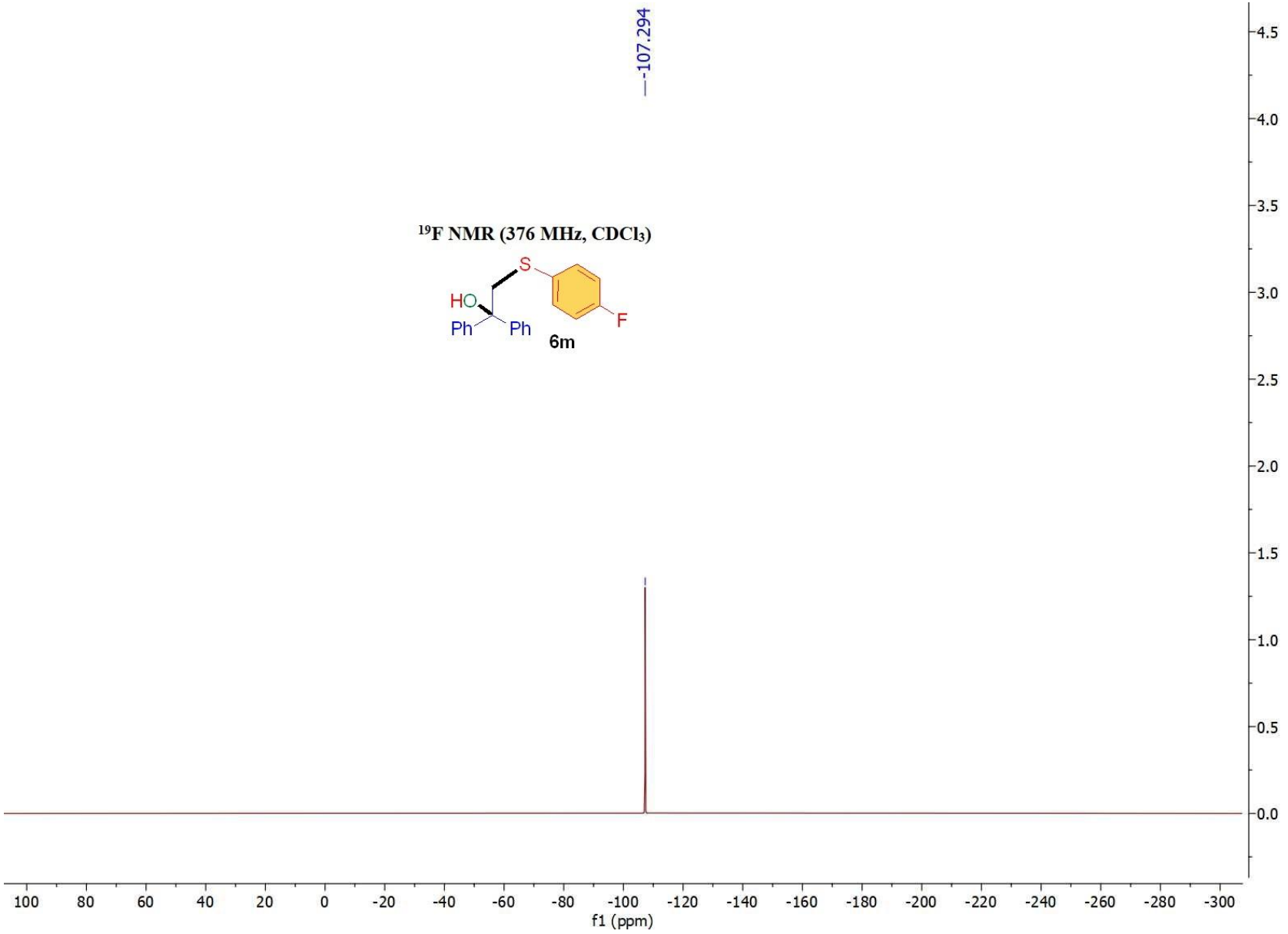
^{1}H NMR (400 MHz, $CDCl_3$)



6m



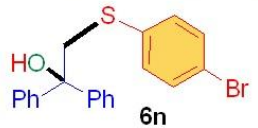

163.29
160.83
145.08
133.27
133.19
131.46
128.36
127.47
126.20
116.27
116.06
77.75
77.42
77.10
76.78
50.31
13C{H} NMR (100 MHz, CDCl3)
S
HO
Ph
Ph
F
6m
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)



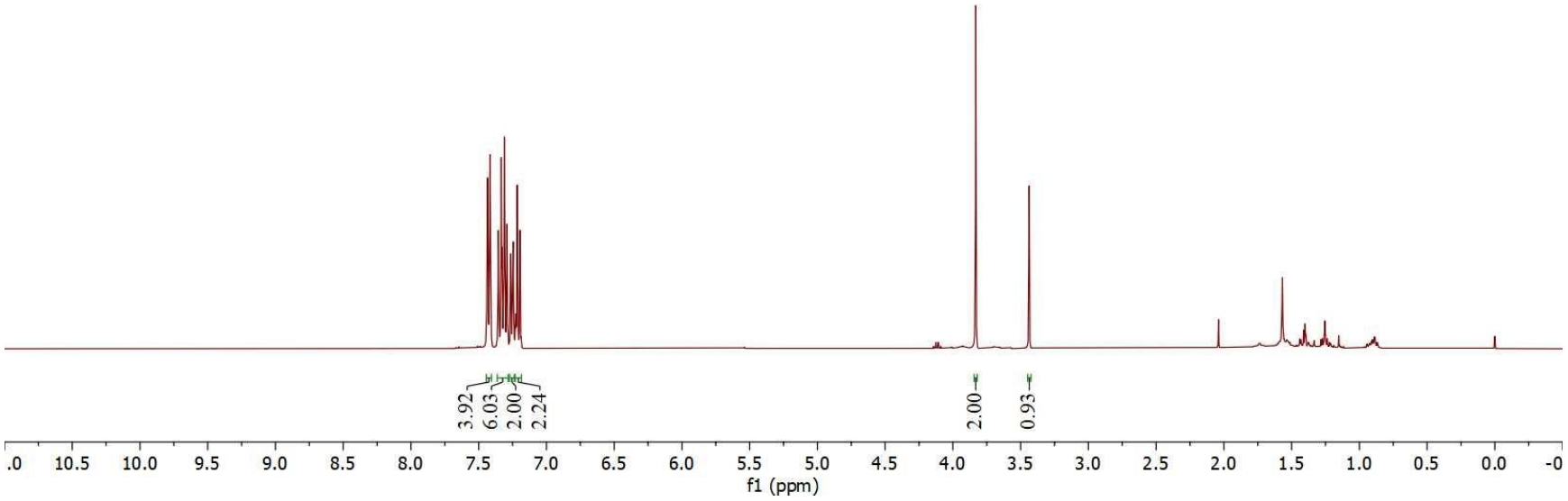
^{19}F NMR (376 MHz, $CDCl_3$)

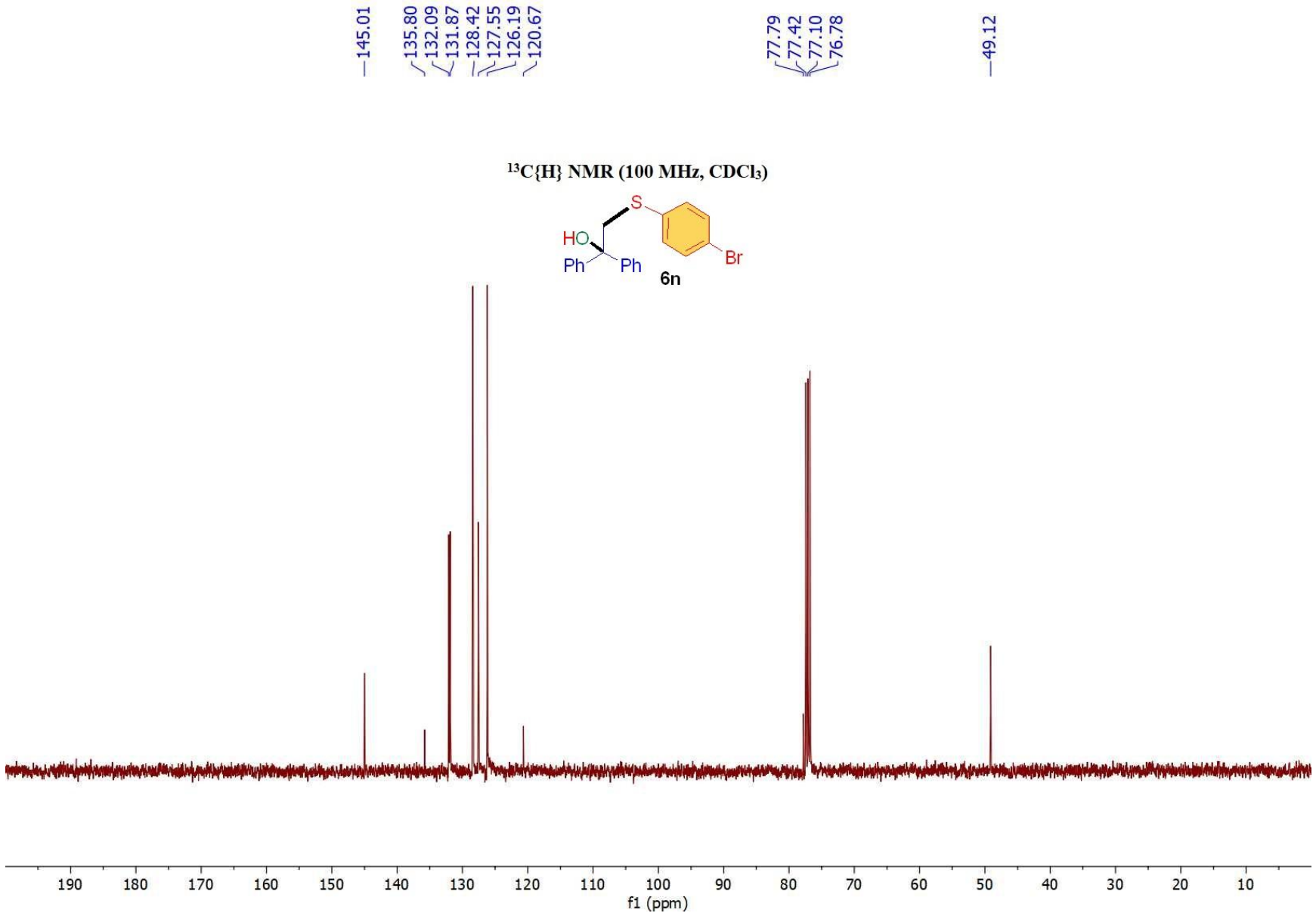
6m

^{1}H NMR (400 MHz, $CDCl_3$)



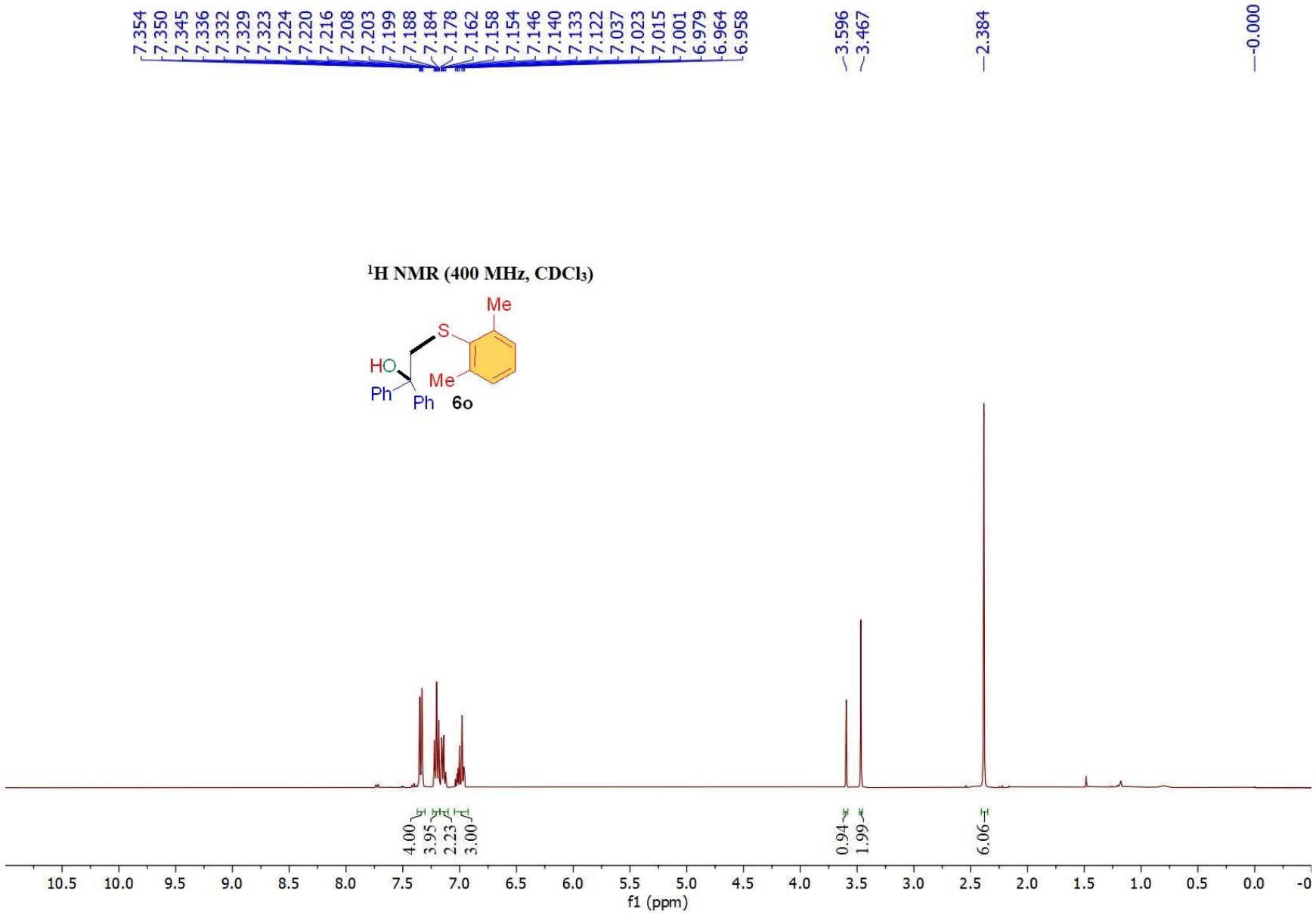
6n

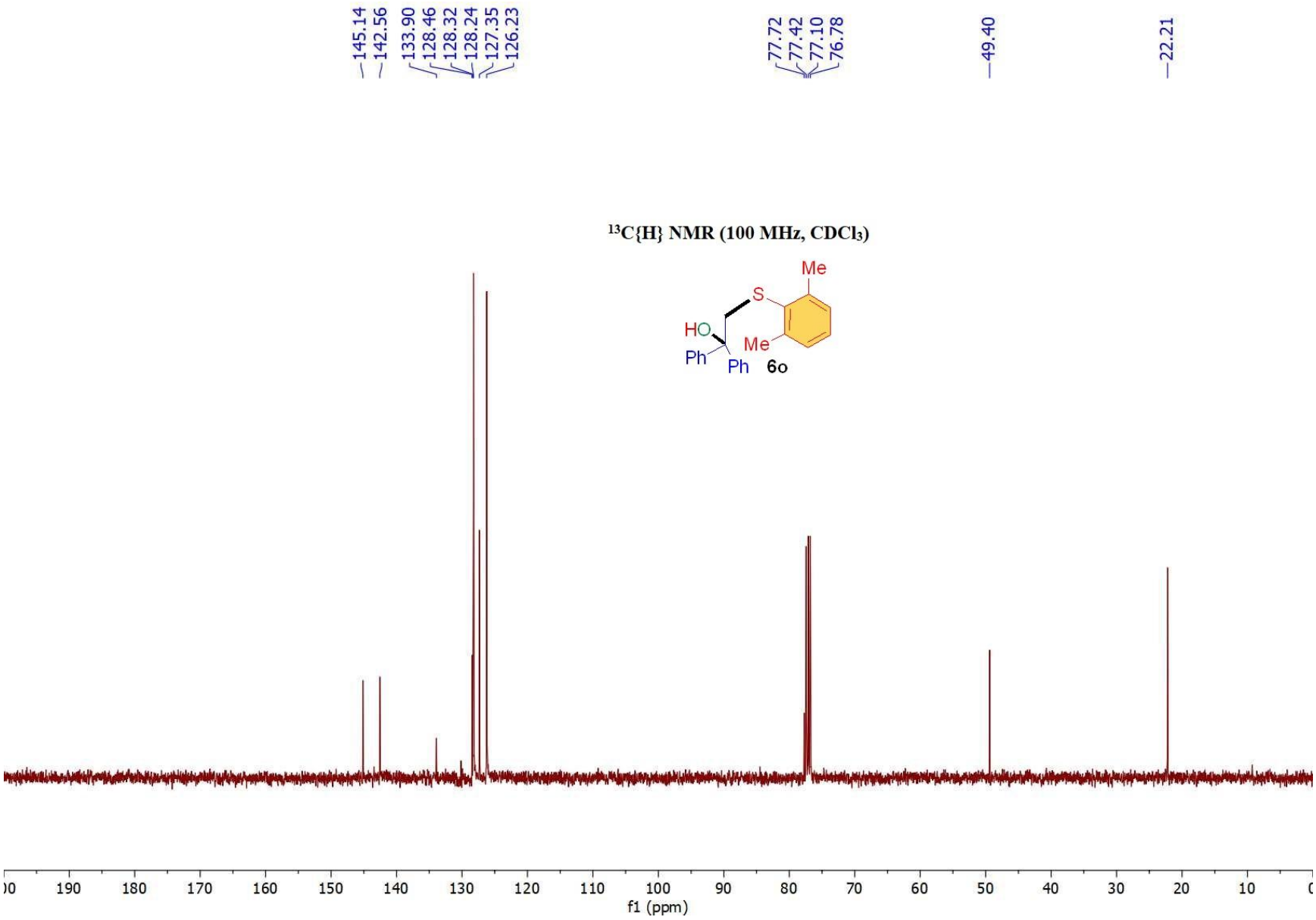




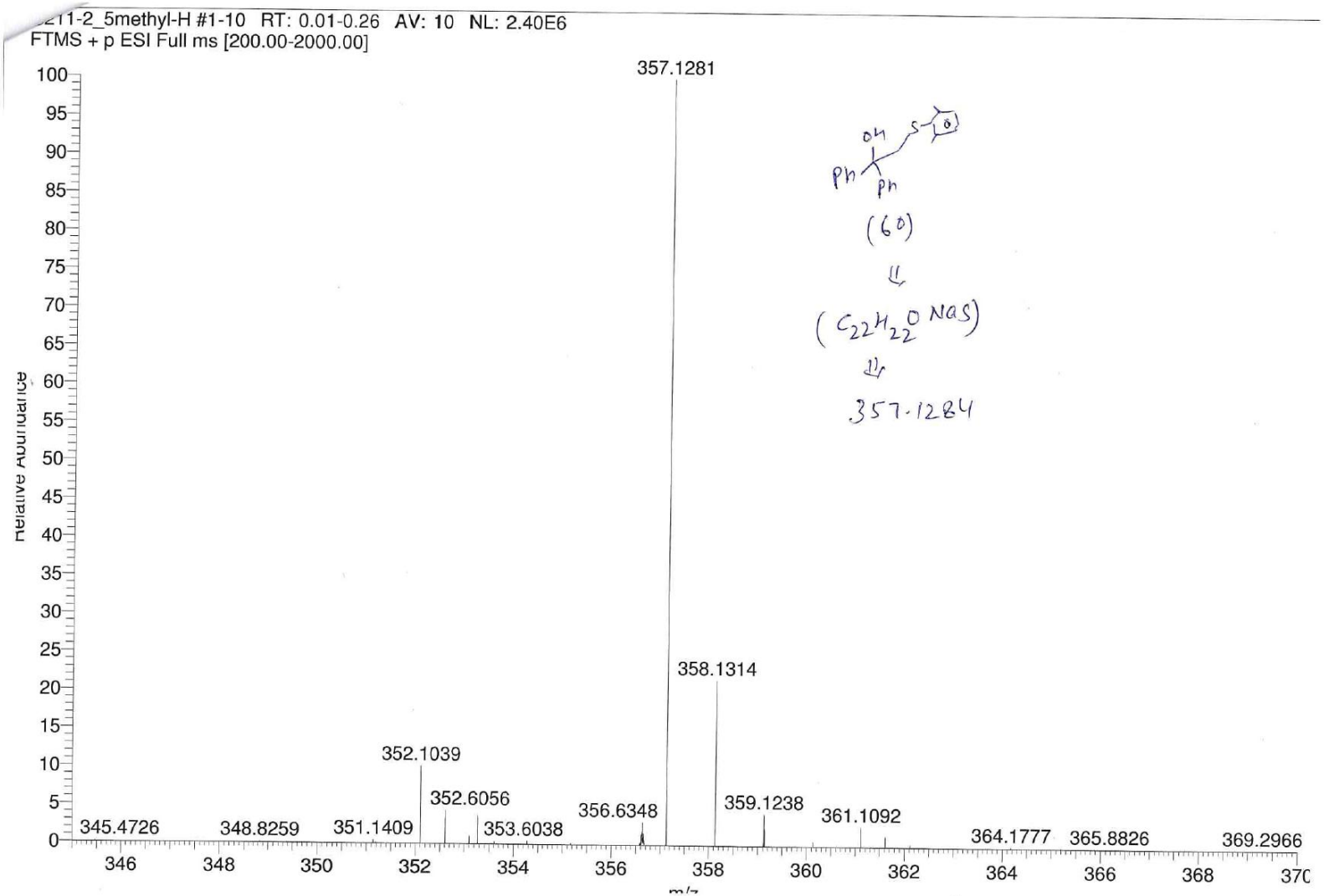
$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)

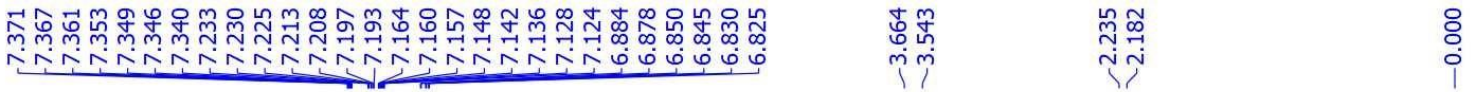
6n


7.354
7.350
7.345
7.336
7.332
7.329
7.323
7.224
7.220
7.216
7.208
7.203
7.199
7.188
7.184
7.178
7.162
7.158
7.154
7.146
7.140
7.133
7.122
7.037
7.023
7.015
7.001
6.979
6.964
6.958
3.596
3.467
2.384
-0.000
1H NMR (400 MHz, CDCl3)
Me
S
HO
Ph
Me
Ph
6o
4.00
3.95
2.23
3.00
0.94
1.99
6.06
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)

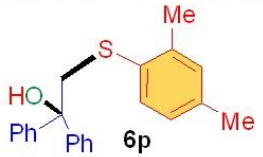


$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)

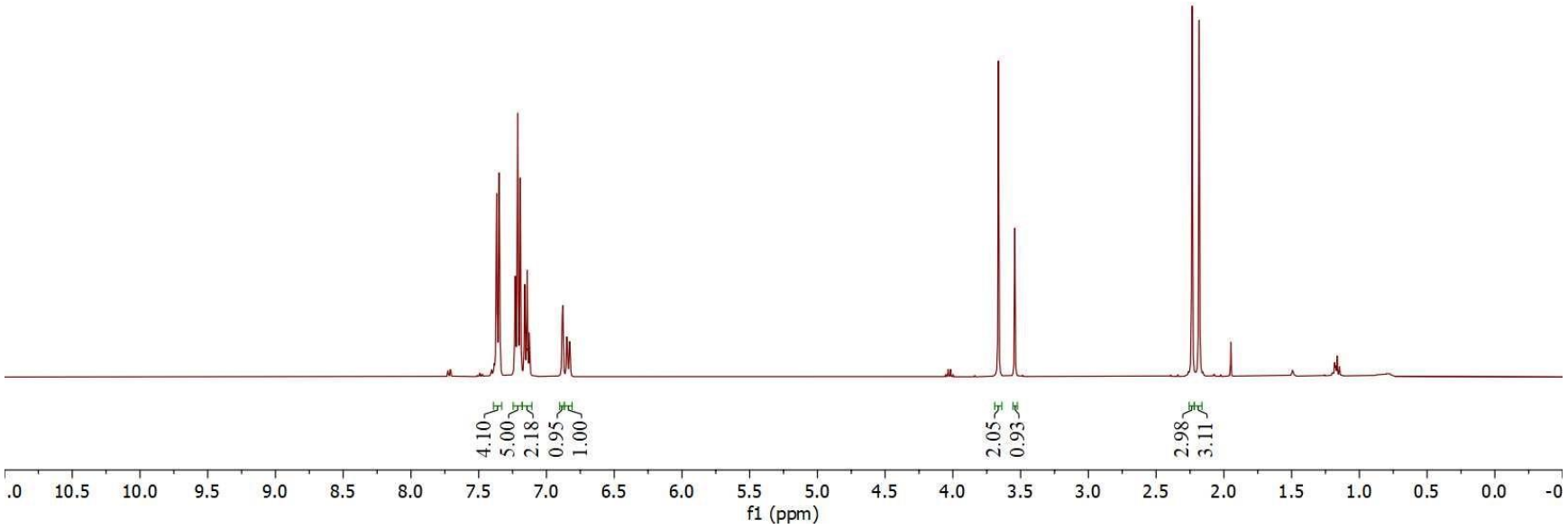

211-2_5methyl-H #1-10 RT: 0.01-0.26 AV: 10 NL: 2.40E6
FTMS + p ESI Full ms [200.00-2000.00]
Relative Abundance
m/z
357.1281
358.1314
352.1039
352.6056
356.6348
359.1238
361.1092
345.4726
348.8259
351.1409
353.6038
364.1777
365.8826
369.2966
OH
S
O
Ph
Ph
(60)
(C22H22ONaS)
357.1284

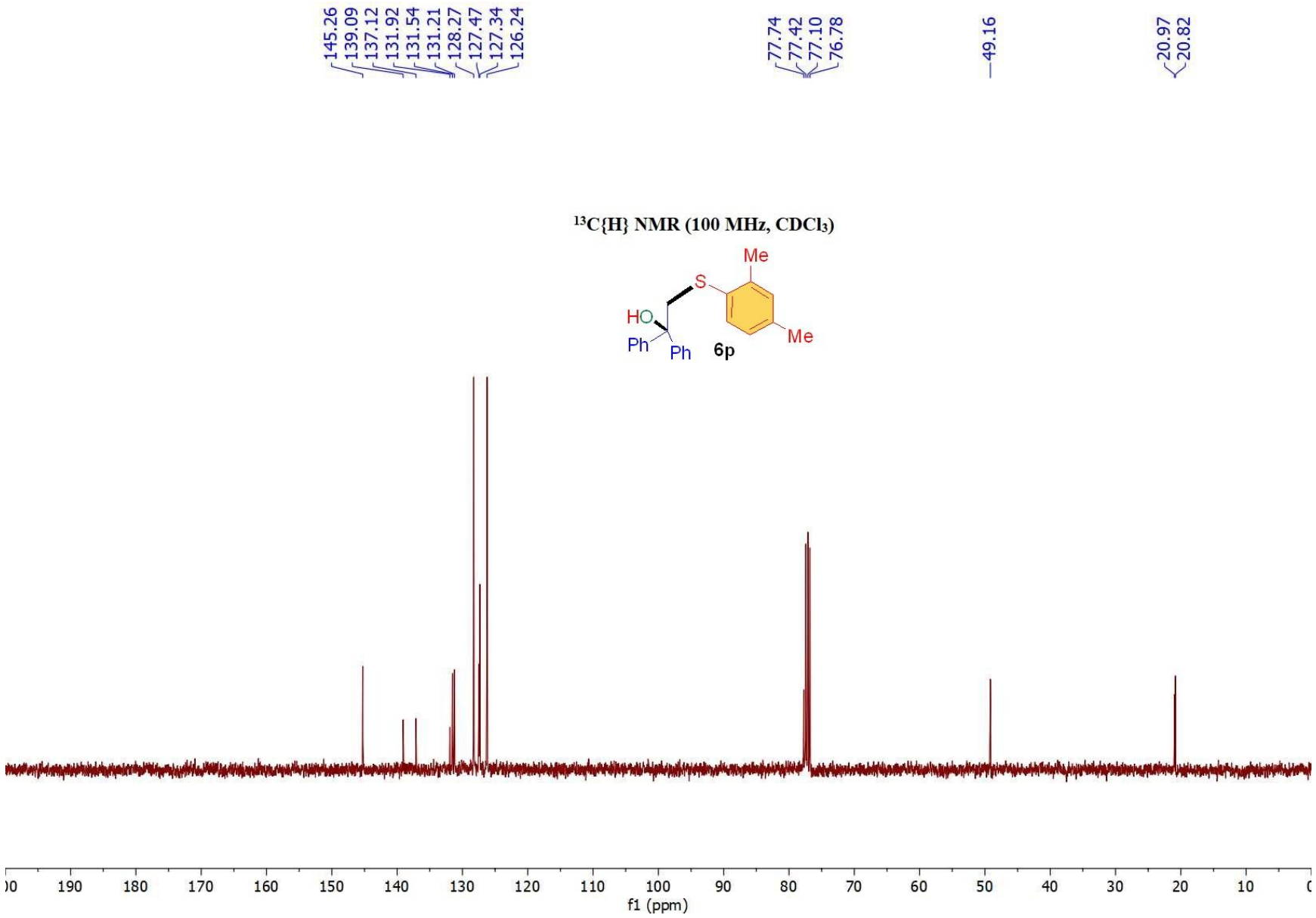


^{1}H NMR (400 MHz, $CDCl_3$)

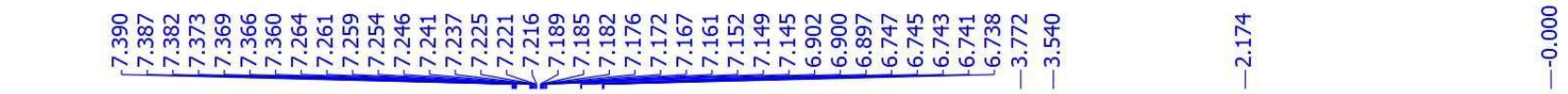


6p



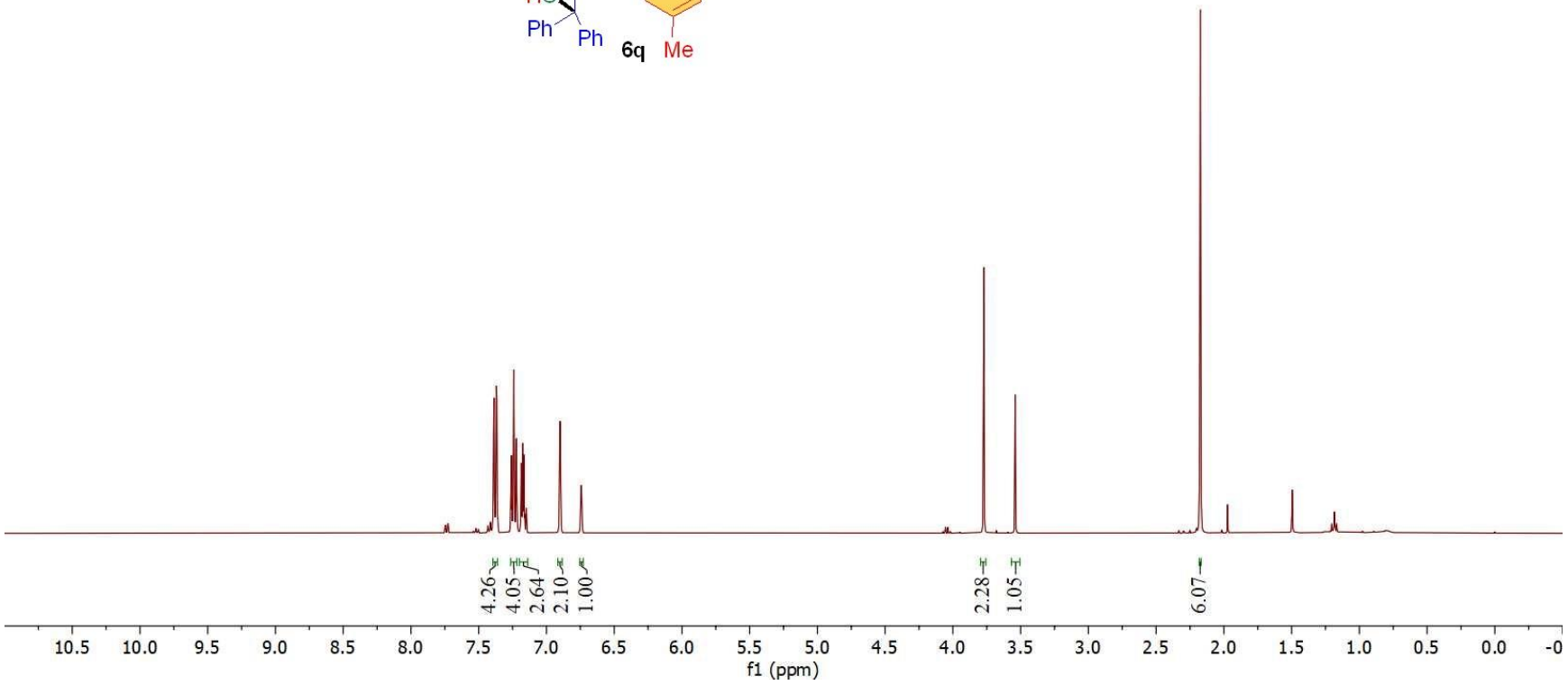


$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)



^{1}H NMR (400 MHz, $CDCl_3$)

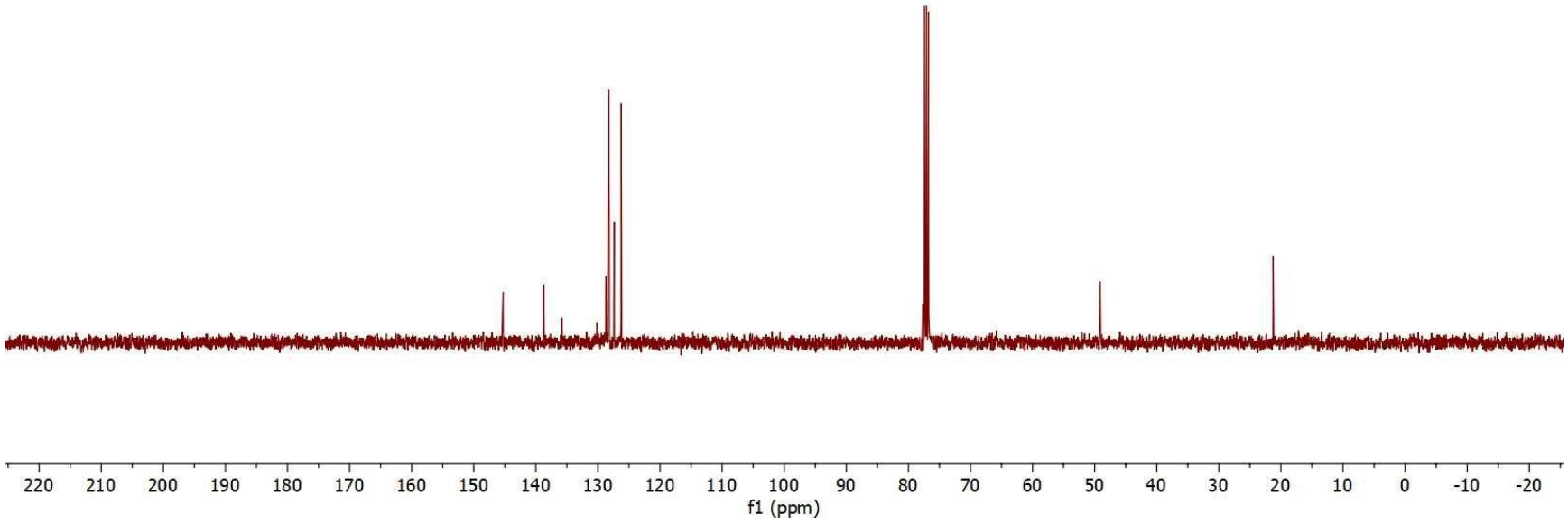
6q



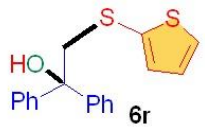
145.28
138.75
135.83
128.70
128.32
128.18
127.40
126.24
77.68
77.42
77.10
76.78
49.11
21.25

$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)

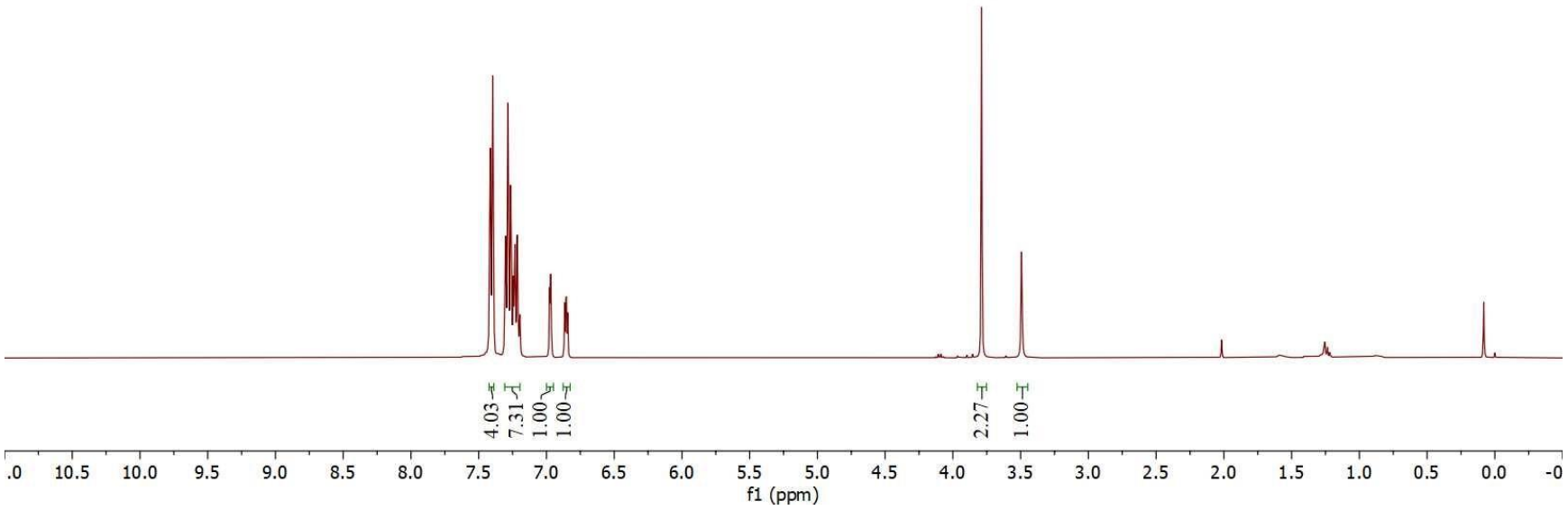
6q

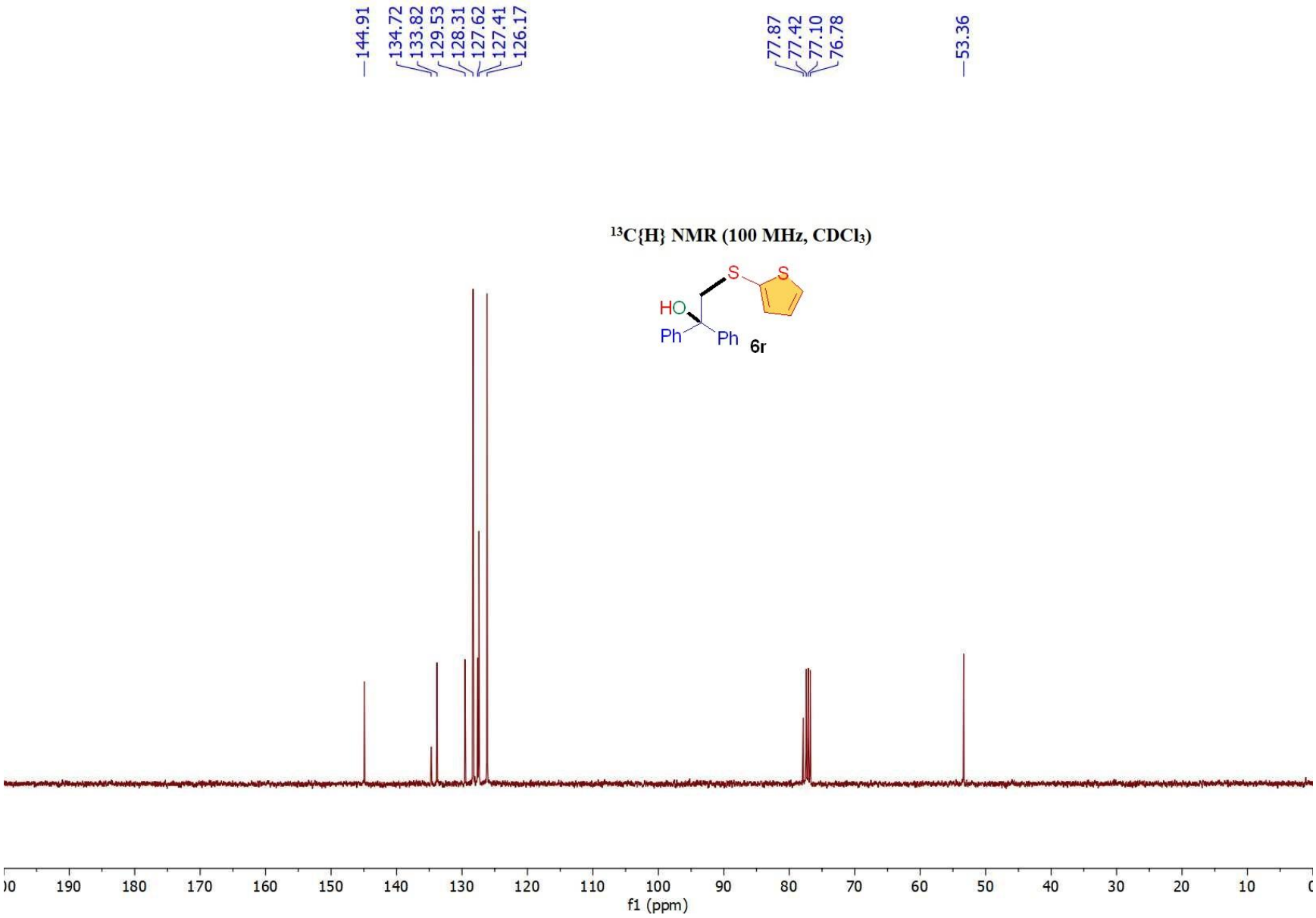


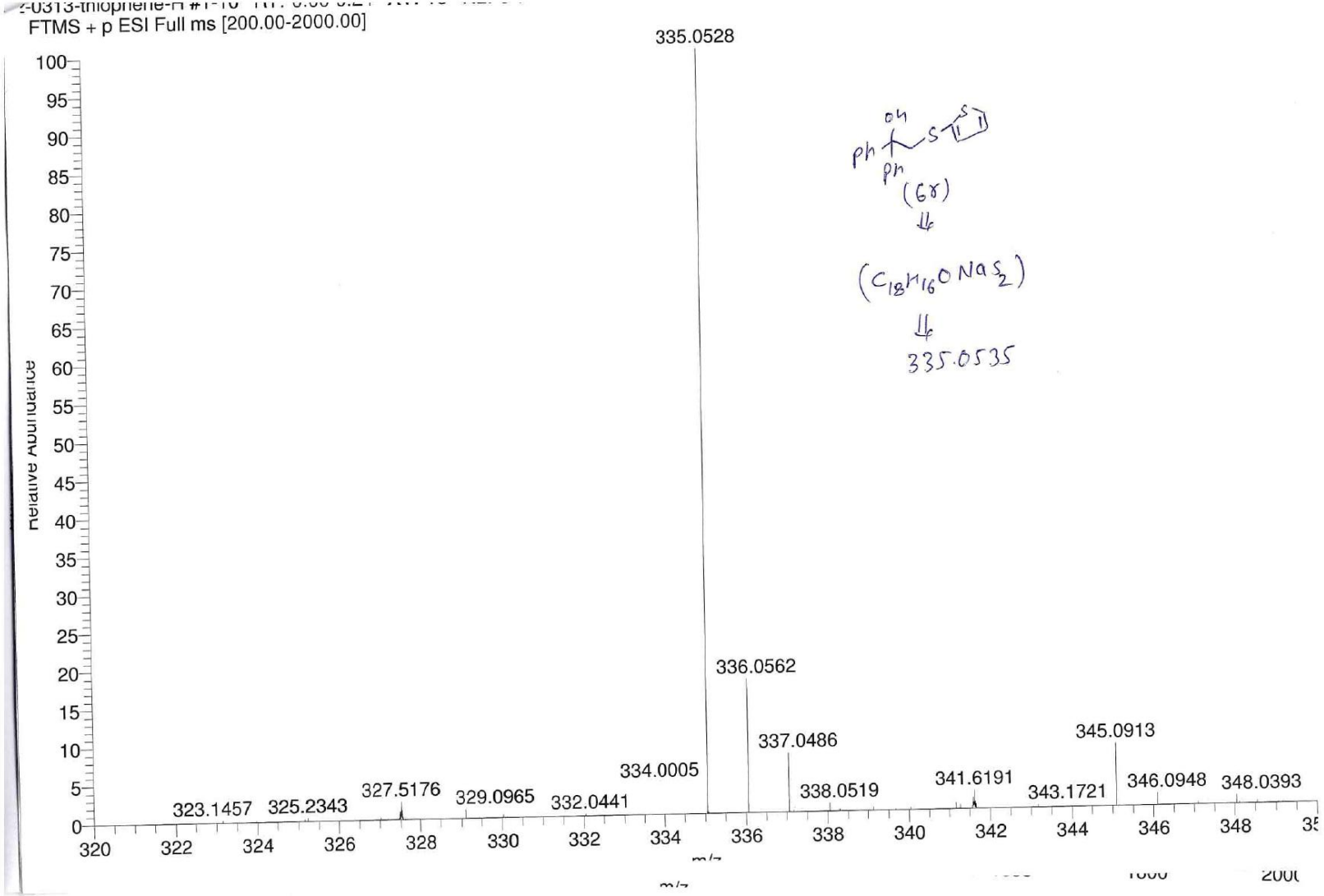
^{1}H NMR (400 MHz, $CDCl_3$)



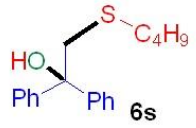
6r



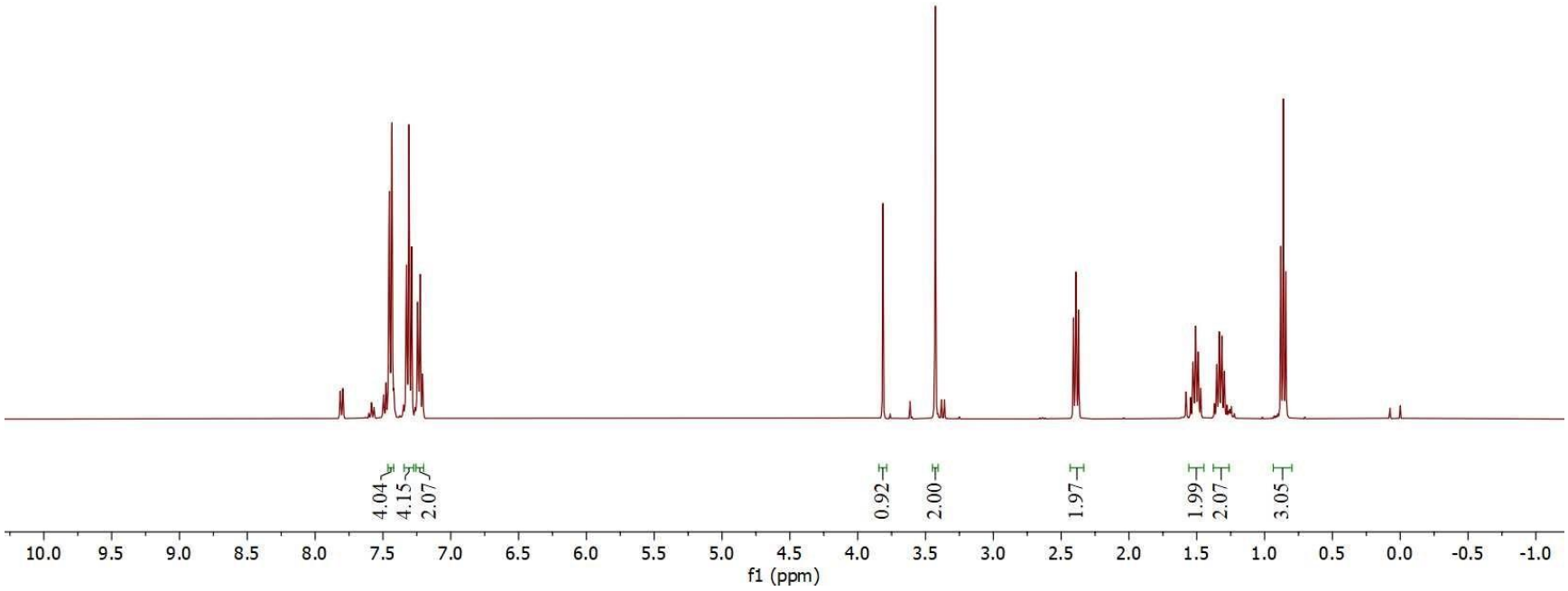

144.91
134.72
133.82
129.53
128.31
127.62
127.41
126.17
77.87
77.42
77.10
76.78
53.36
13C{H} NMR (100 MHz, CDCl3)
S
S
HO
Ph
Ph
6r
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


FTMS + p ESI Full ms [200.00-2000.00]
335.0528
336.0562
337.0486
345.0913
334.0005
338.0519
341.6191
343.1721
346.0948
348.0393
323.1457
325.2343
327.5176
329.0965
332.0441
Relative Abundance
m/z
(6r)
(C18H16ONaS2)
335.0535

^{1}H NMR (400 MHz, $CDCl_3$)

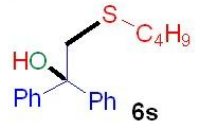


6s

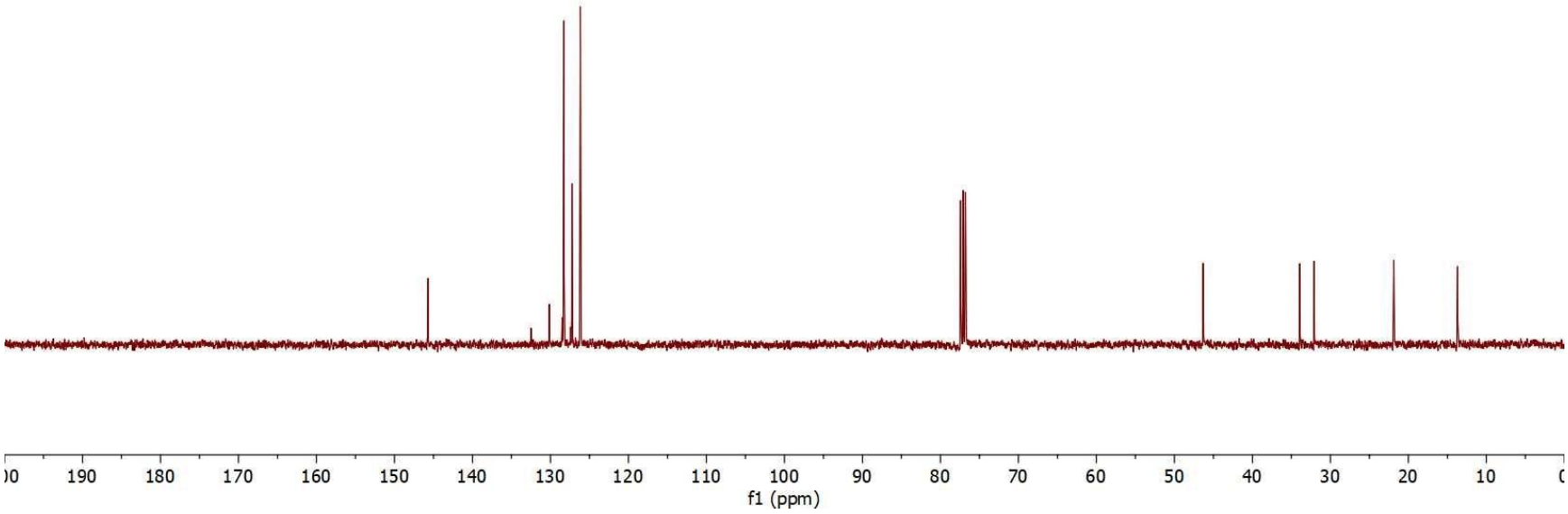


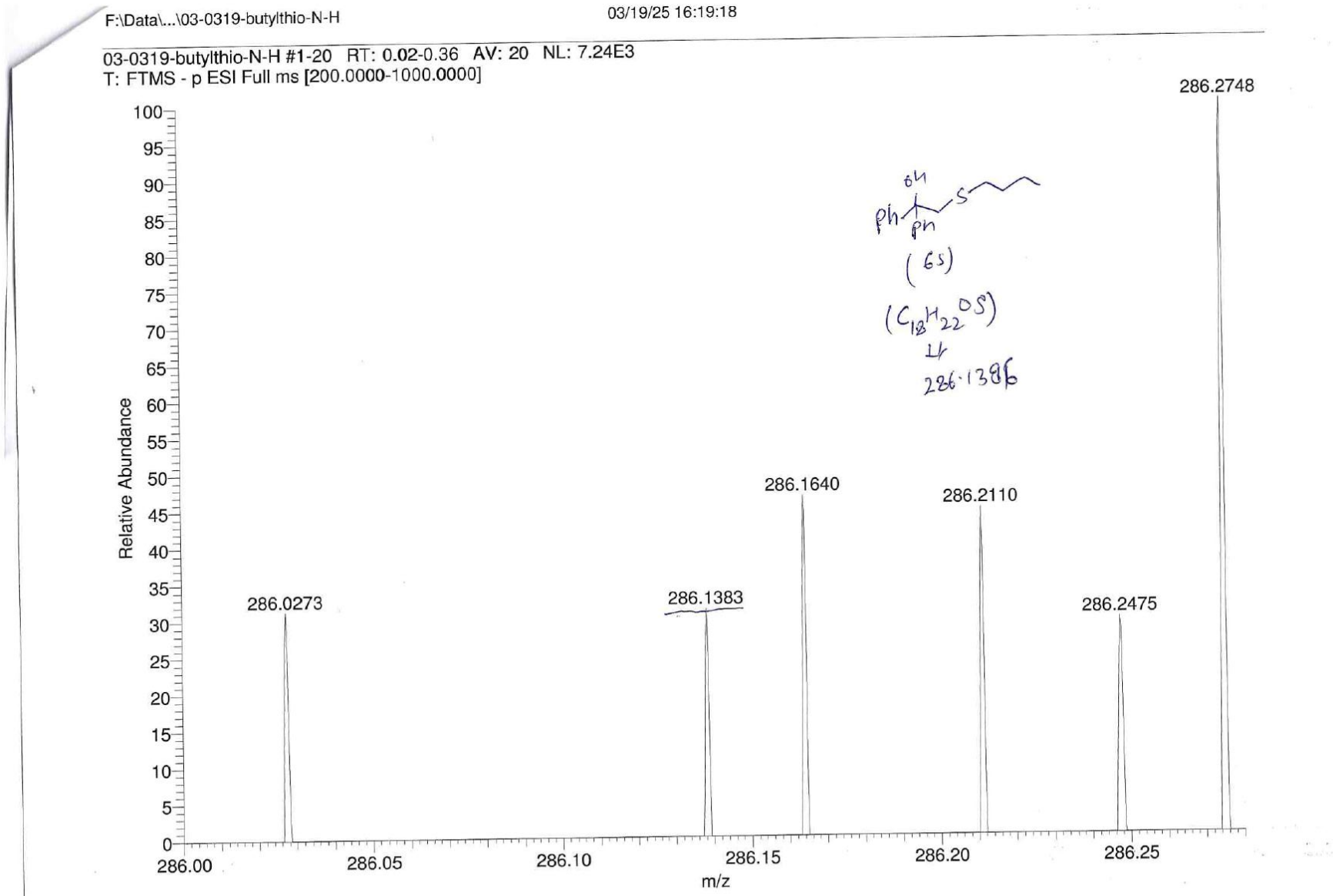
145.69
132.50
130.14
128.27
127.25
126.17
77.42
77.10
77.06
76.78
46.33
33.93
32.08
21.88
13.69

$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)

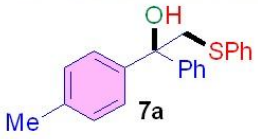


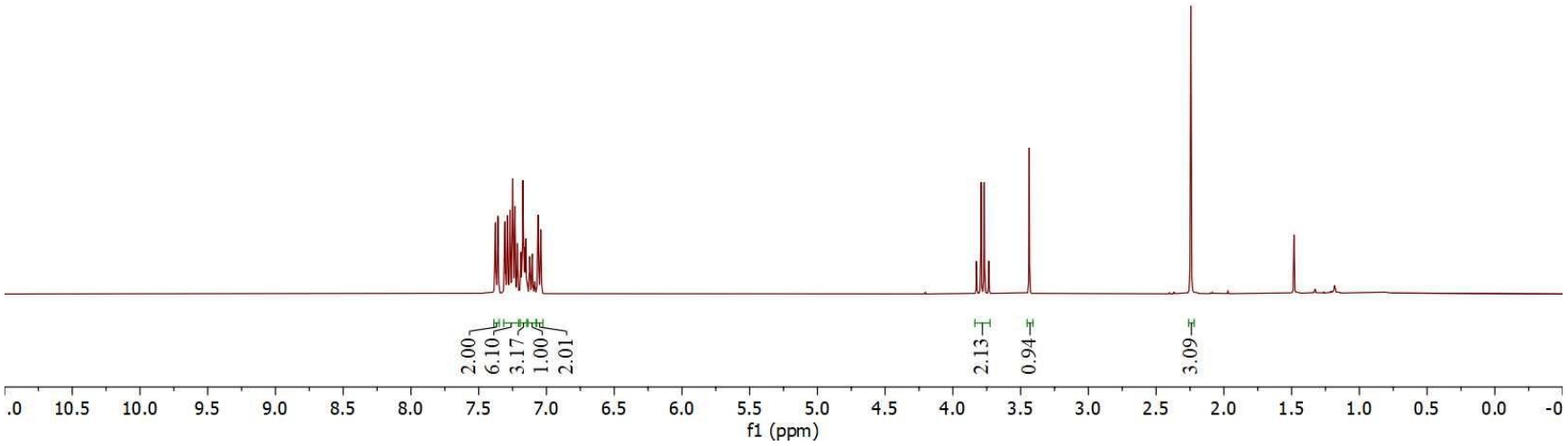
6s

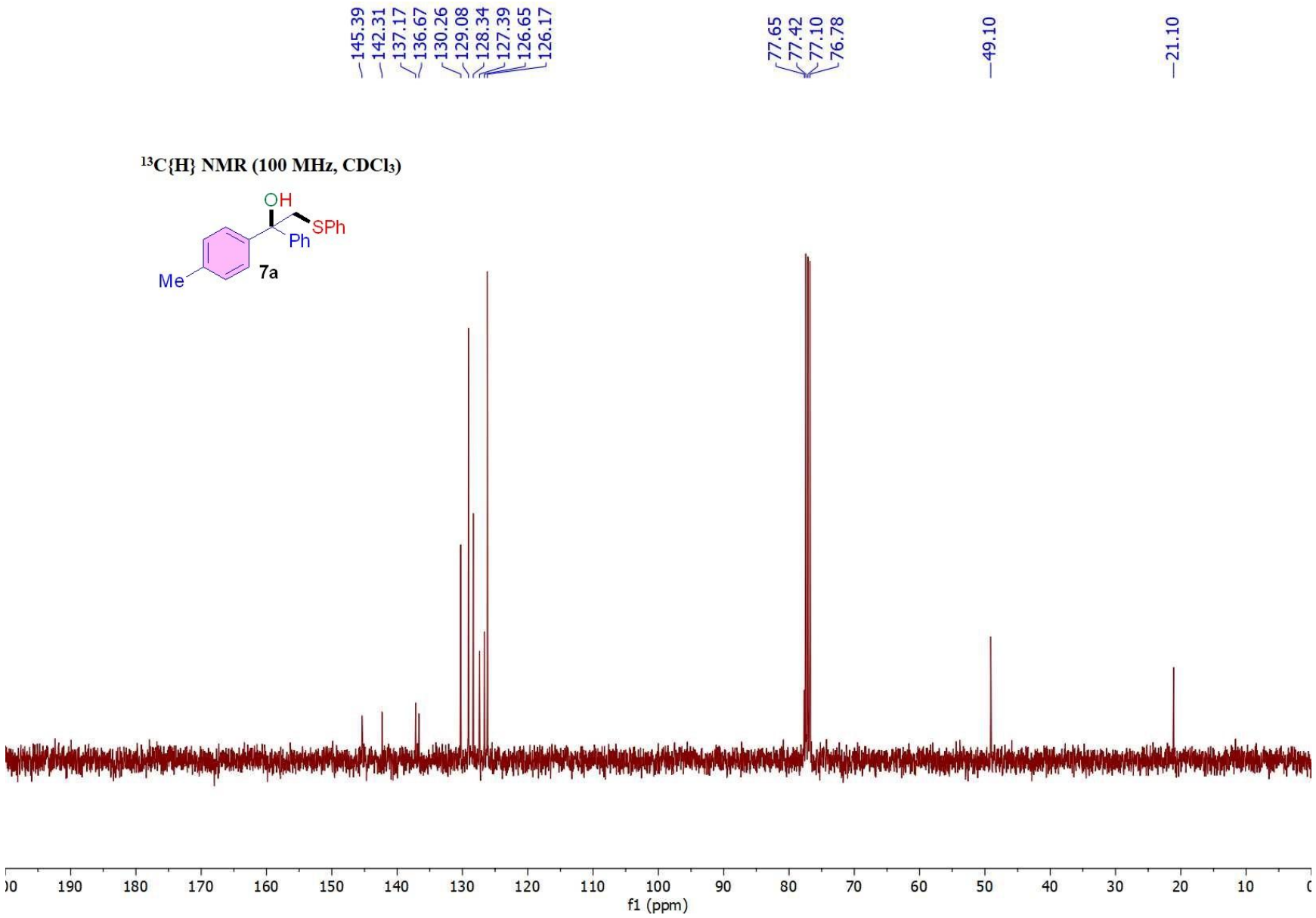



F:\Data\...\03-0319-butylthio-N-H
03/19/25 16:19:18
03-0319-butylthio-N-H #1-20 RT: 0.02-0.36 AV: 20 NL: 7.24E3
T: FTMS - p ESI Full ms [200.0000-1000.0000]
286.2748
286.1640
286.2110
286.0273
286.1383
286.2475
Relative Abundance
m/z
286.00
286.05
286.10
286.15
286.20
286.25
(6S)
(C18H22OS)
286.1386

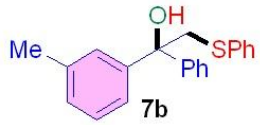
^{1}H NMR (400 MHz, $CDCl_3$)



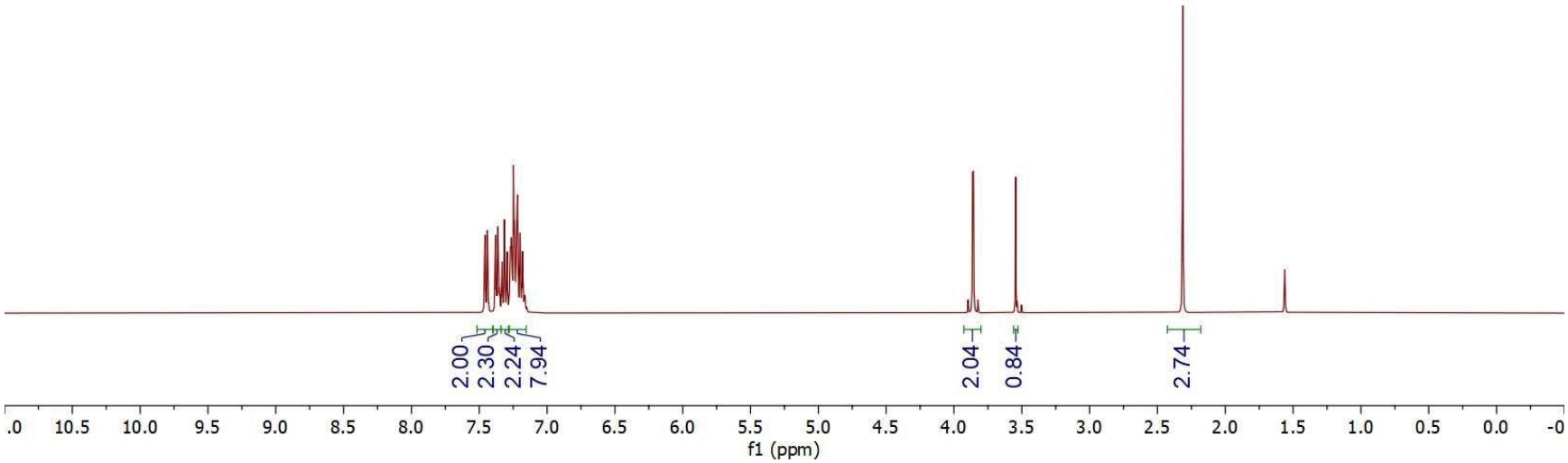


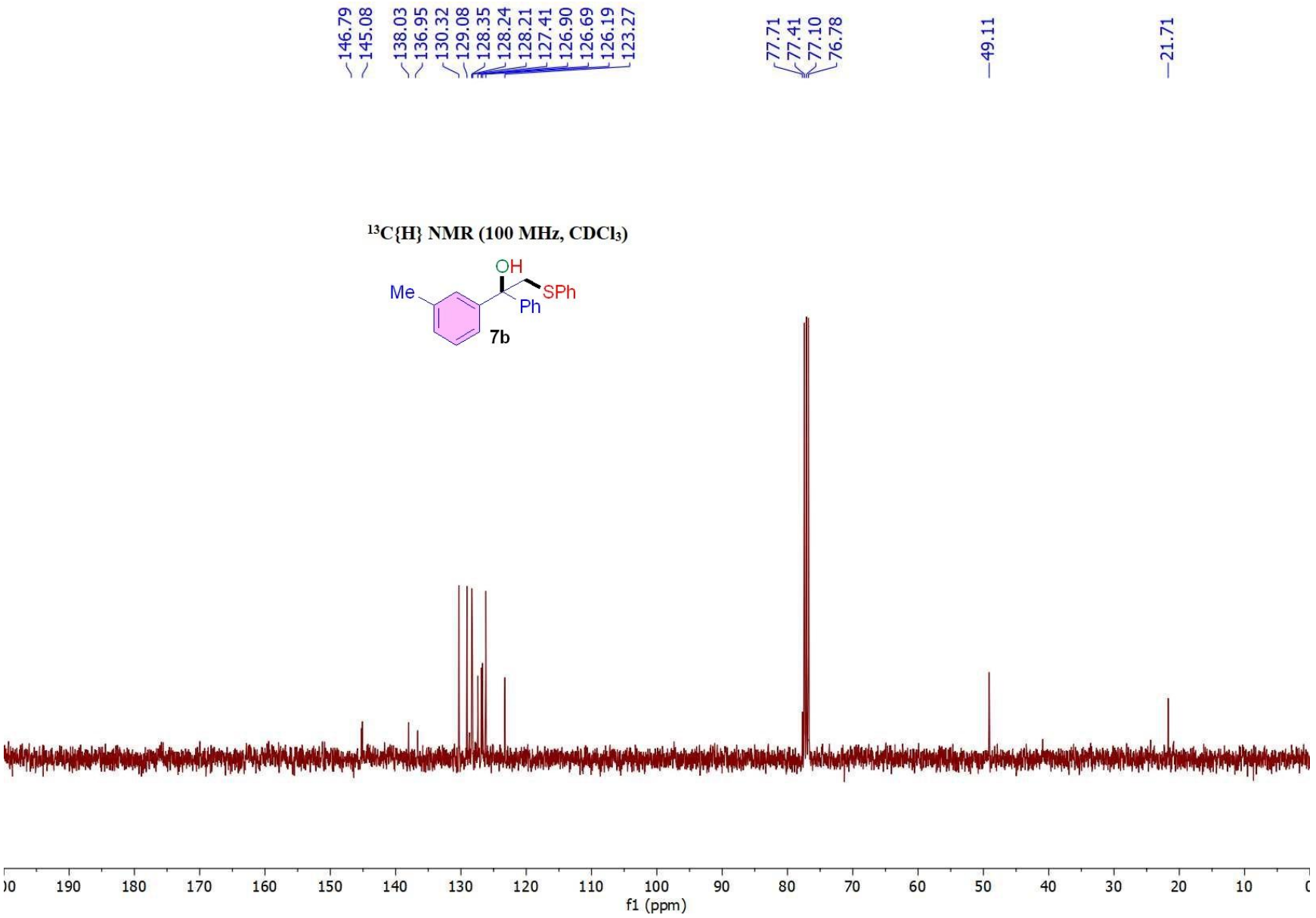

145.39
142.31
137.17
136.67
130.26
129.08
128.34
127.39
126.65
126.17
77.65
77.42
77.10
76.78
49.10
21.10
13C{H} NMR (100 MHz, CDCl3)
OH
SPh
Ph
Me
7a
f1 (ppm)

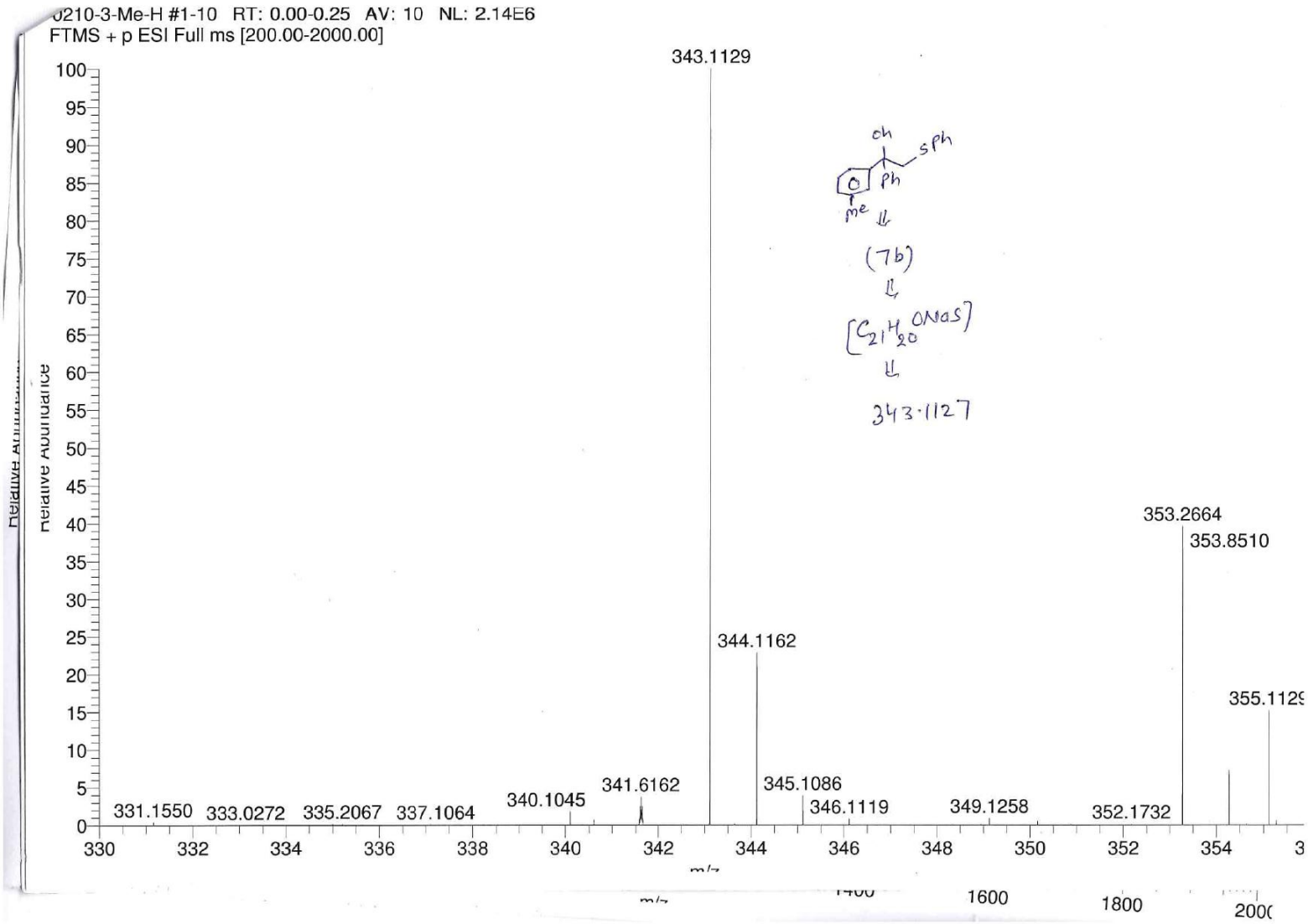
^{1}H NMR (400 MHz, $CDCl_3$)

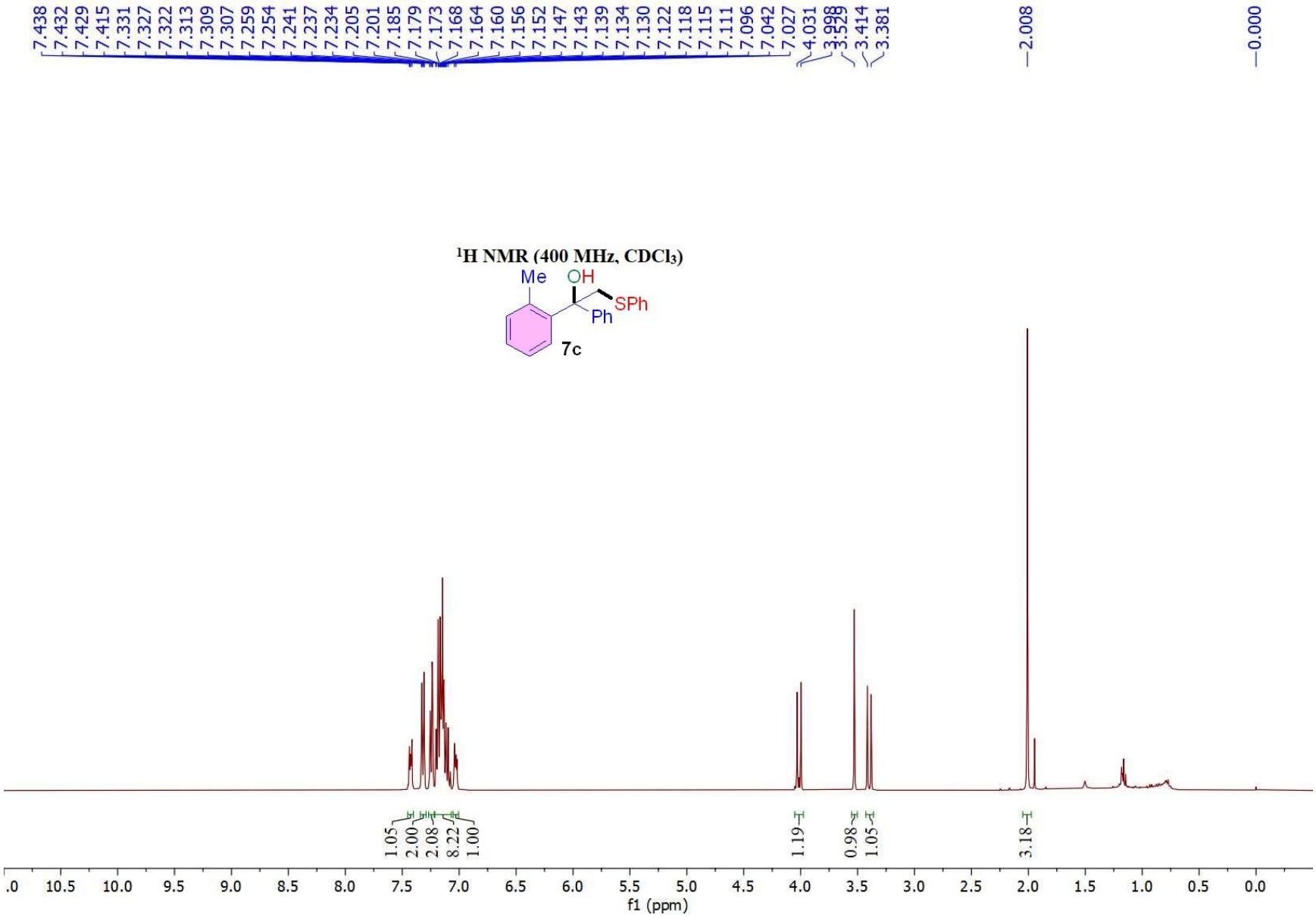


7b

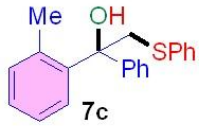



146.79
145.08
138.03
136.95
130.32
129.08
128.35
128.24
128.21
127.41
126.90
126.69
126.19
123.27
77.71
77.41
77.10
76.78
49.11
21.71
13C{H} NMR (100 MHz, CDCl3)
OH
Me
SPh
Ph
7b
f1 (ppm)

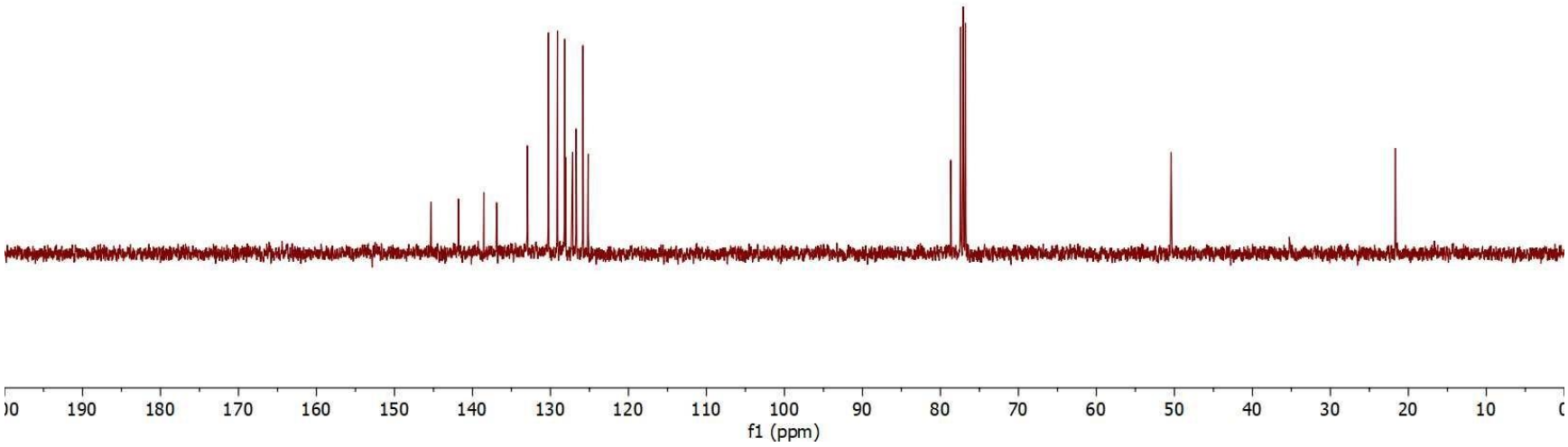

0210-3-Me-H #1-10 RT: 0.00-0.25 AV: 10 NL: 2.14E6
FTMS + p ESI Full ms [200.00-2000.00]
Relative Abundance
343.1129
353.2664
353.8510
344.1162
355.1129
345.1086
341.6162
340.1045
346.1119
349.1258
352.1732
331.1550
333.0272
335.2067
337.1064
m/z
(7b)
[C21H20ONaS]
343.1127

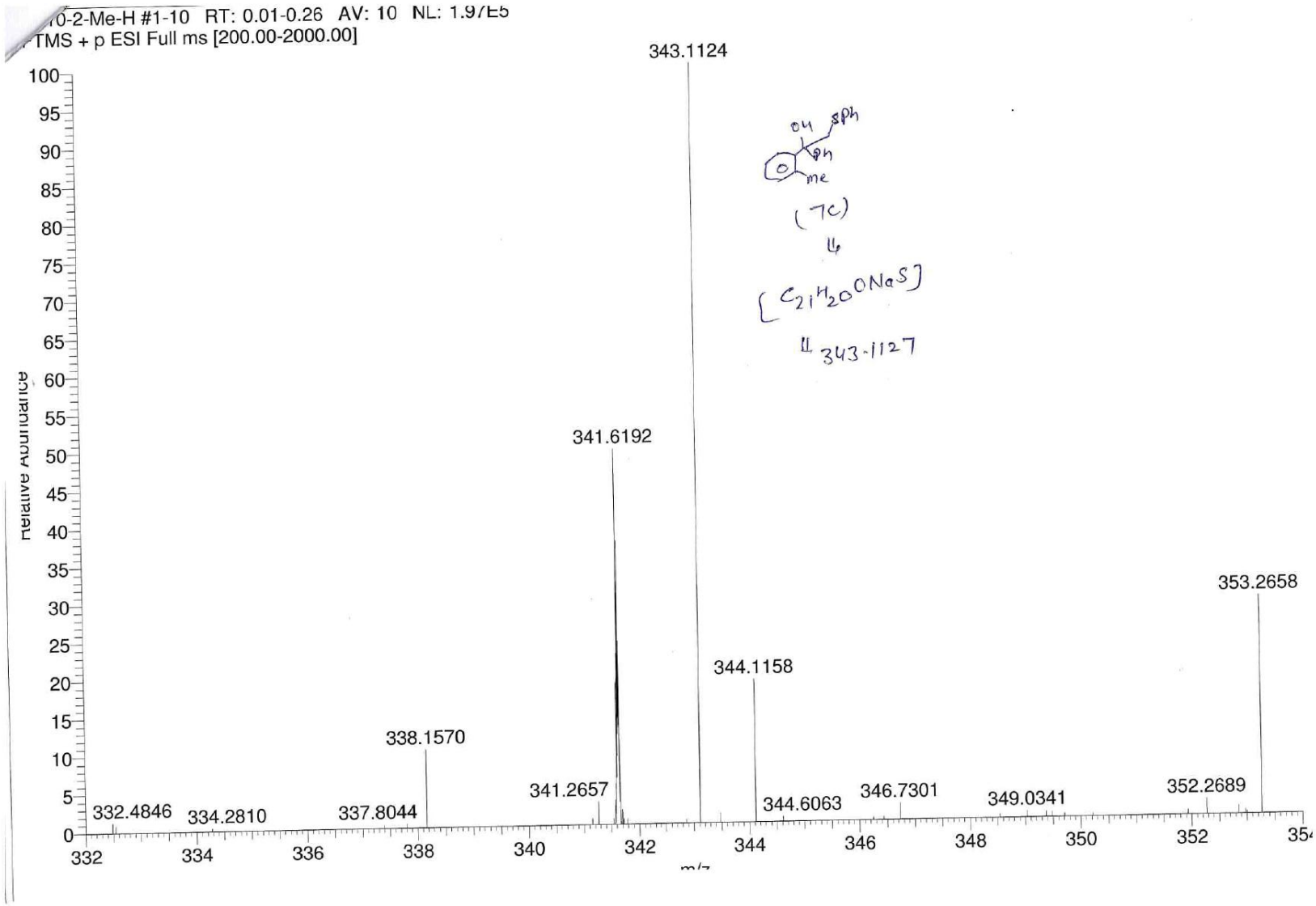

7.438
7.432
7.429
7.415
7.331
7.327
7.322
7.313
7.309
7.307
7.259
7.254
7.241
7.237
7.234
7.205
7.201
7.185
7.179
7.173
7.168
7.164
7.160
7.156
7.152
7.147
7.143
7.139
7.134
7.130
7.122
7.118
7.115
7.111
7.096
7.042
7.027
4.031
3.998
3.529
3.414
3.381
2.008
0.000
1H NMR (400 MHz, CDCl3)
Me
OH
Ph
SPh
7c
1.05
2.00
2.08
8.22
1.00
1.19
0.98
1.05
3.18
f1 (ppm)

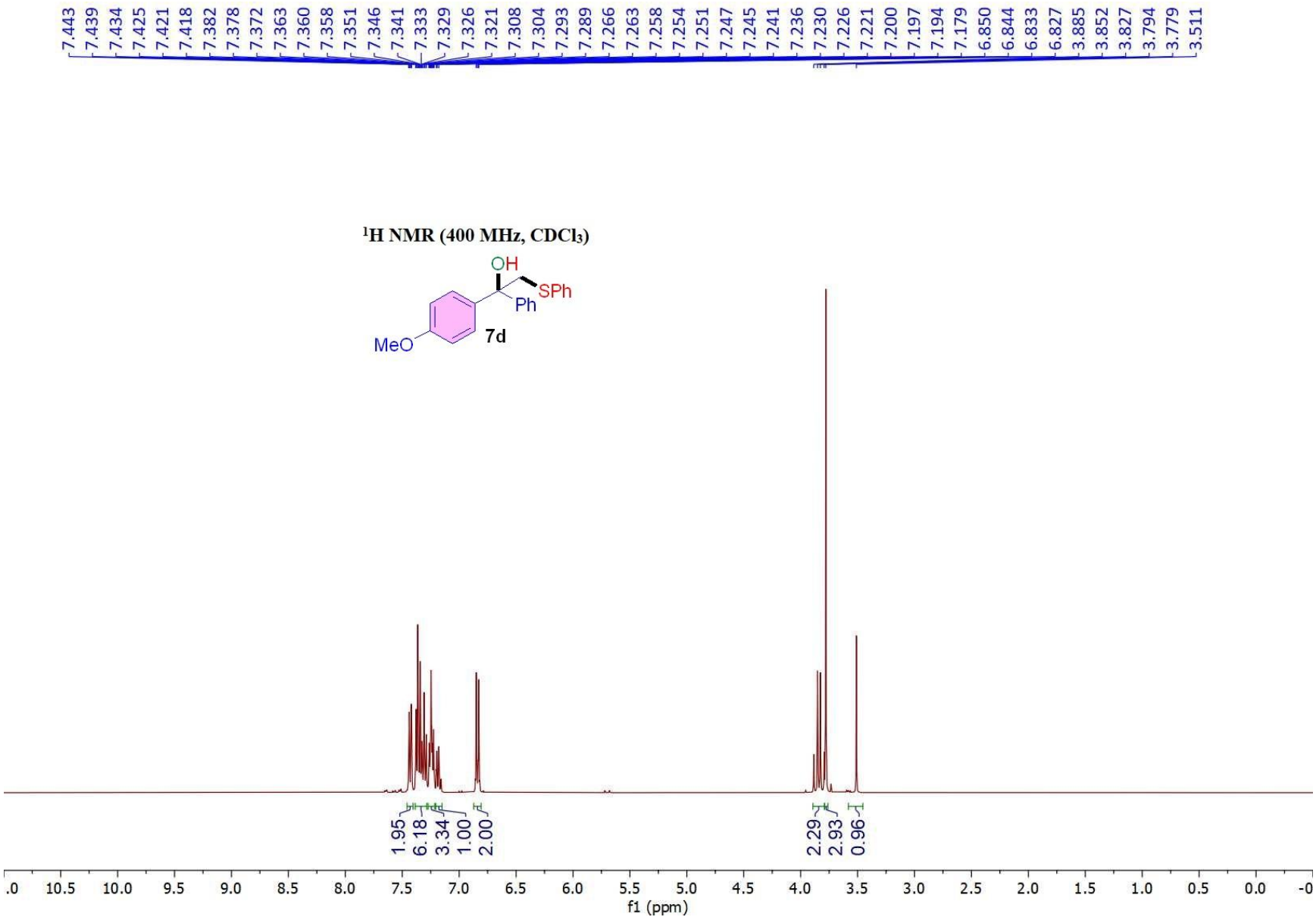
$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)



7c

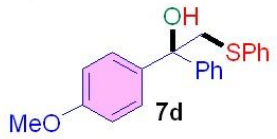



10-2-Me-H #1-10 RT: 0.01-0.26 AV: 10 NL: 1.97E5
TMS + p ESI Full ms [200.00-2000.00]
343.1124
341.6192
353.2658
344.1158
338.1570
341.2657
332.4846
334.2810
337.8044
344.6063
346.7301
349.0341
352.2689
Relative Abundance
m/z
(7c)
[C21H20ONaS]
343.1127

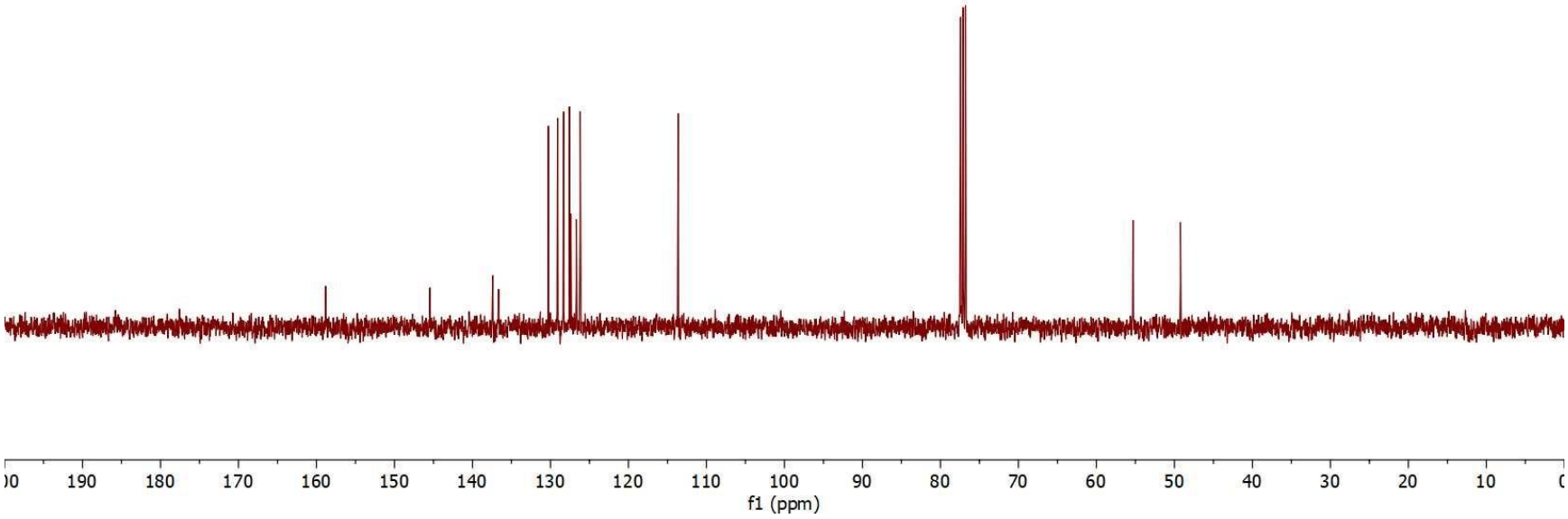

7.443
7.439
7.434
7.425
7.421
7.418
7.382
7.378
7.372
7.363
7.360
7.358
7.351
7.346
7.341
7.333
7.329
7.326
7.321
7.308
7.304
7.293
7.289
7.266
7.263
7.258
7.254
7.251
7.247
7.245
7.241
7.236
7.230
7.226
7.221
7.200
7.197
7.194
7.179
6.850
6.844
6.833
6.827
3.885
3.852
3.827
3.794
3.779
3.511
1H NMR (400 MHz, CDCl3)
OH
SPh
Ph
MeO
7d
1.95
6.18
3.34
1.00
2.00
2.29
2.93
0.96
f1 (ppm)

158.83
145.45
137.40
136.64
130.28
129.09
128.32
127.56
127.39
126.67
126.18
113.63
77.51
77.42
77.10
76.78
55.31
49.22

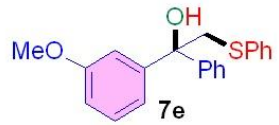
$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)



7d



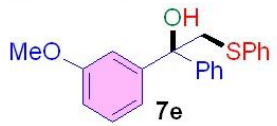
^{1}H NMR (400 MHz, $CDCl_3$)



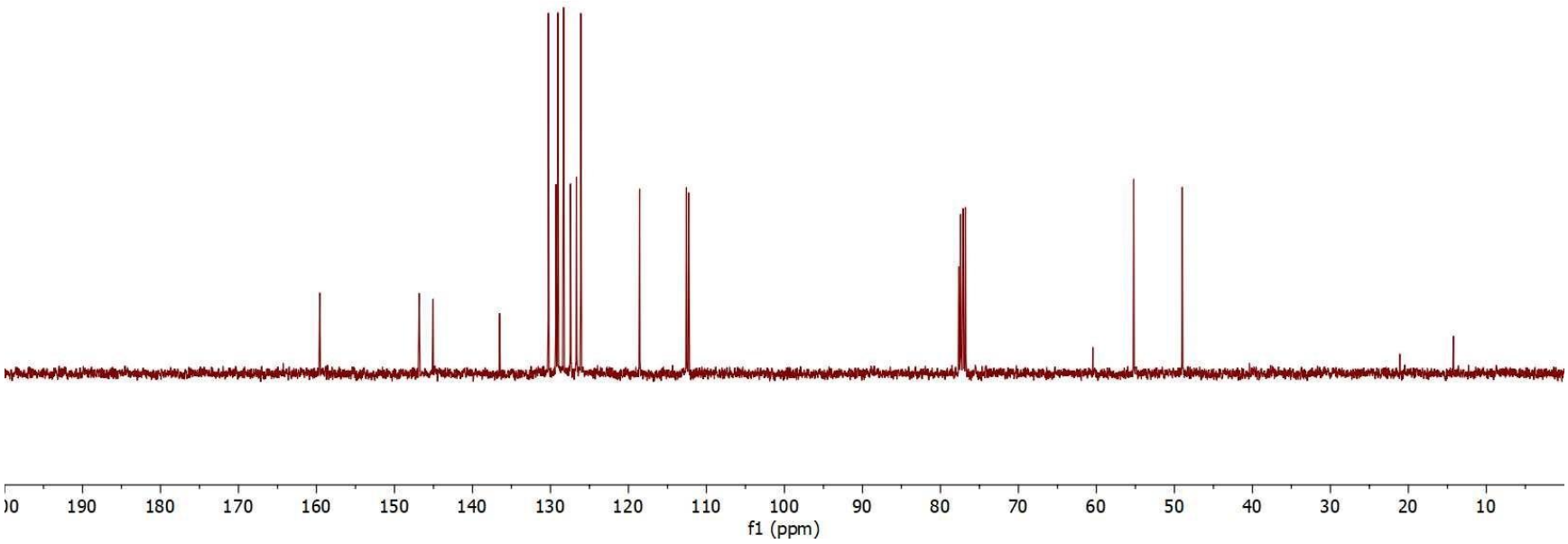
7e

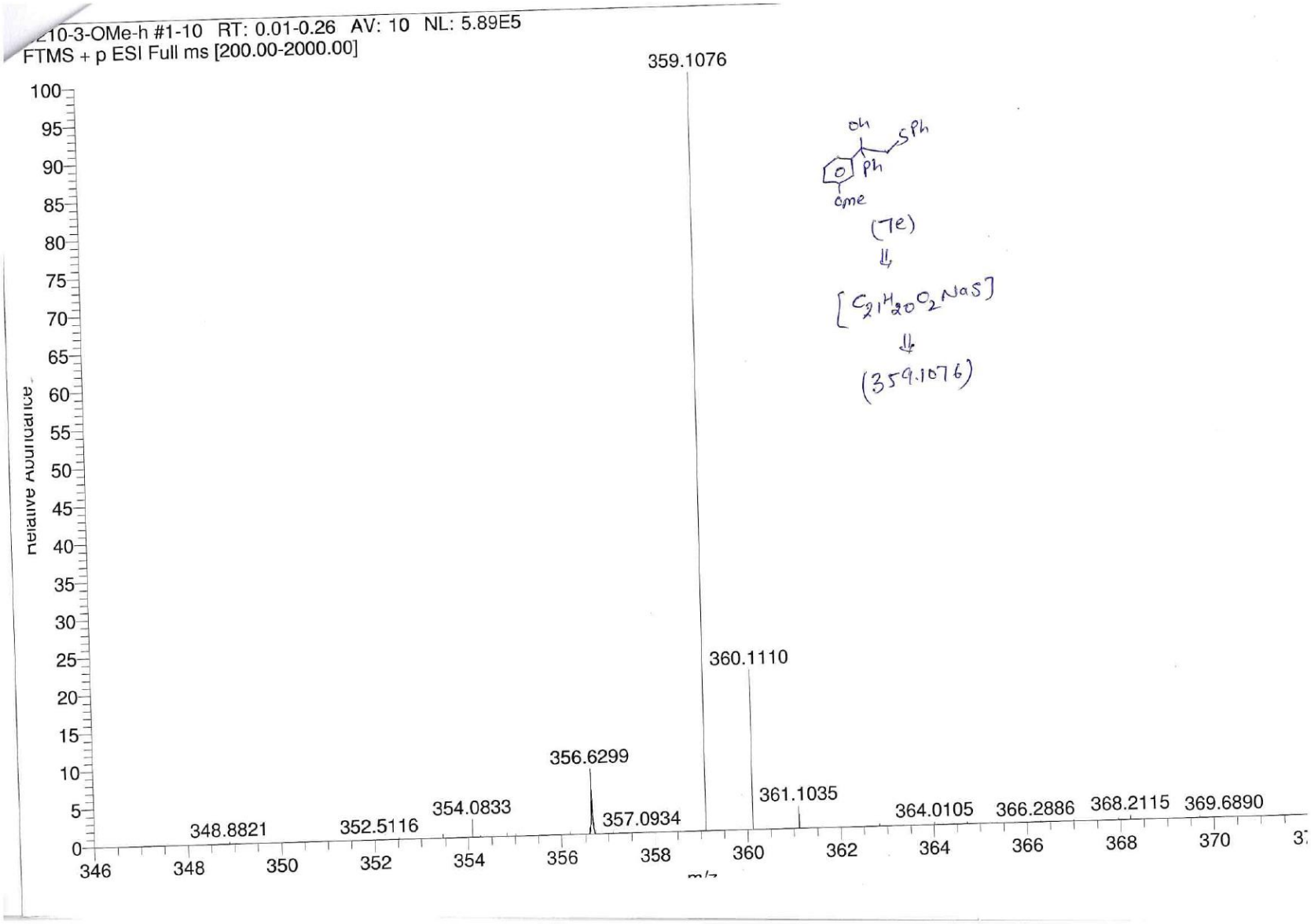
159.58
146.84
145.05
136.54
130.26
129.29
129.05
128.32
127.43
126.66
126.10
118.56
112.57
112.28
77.64
77.42
77.10
76.78
55.22
48.99

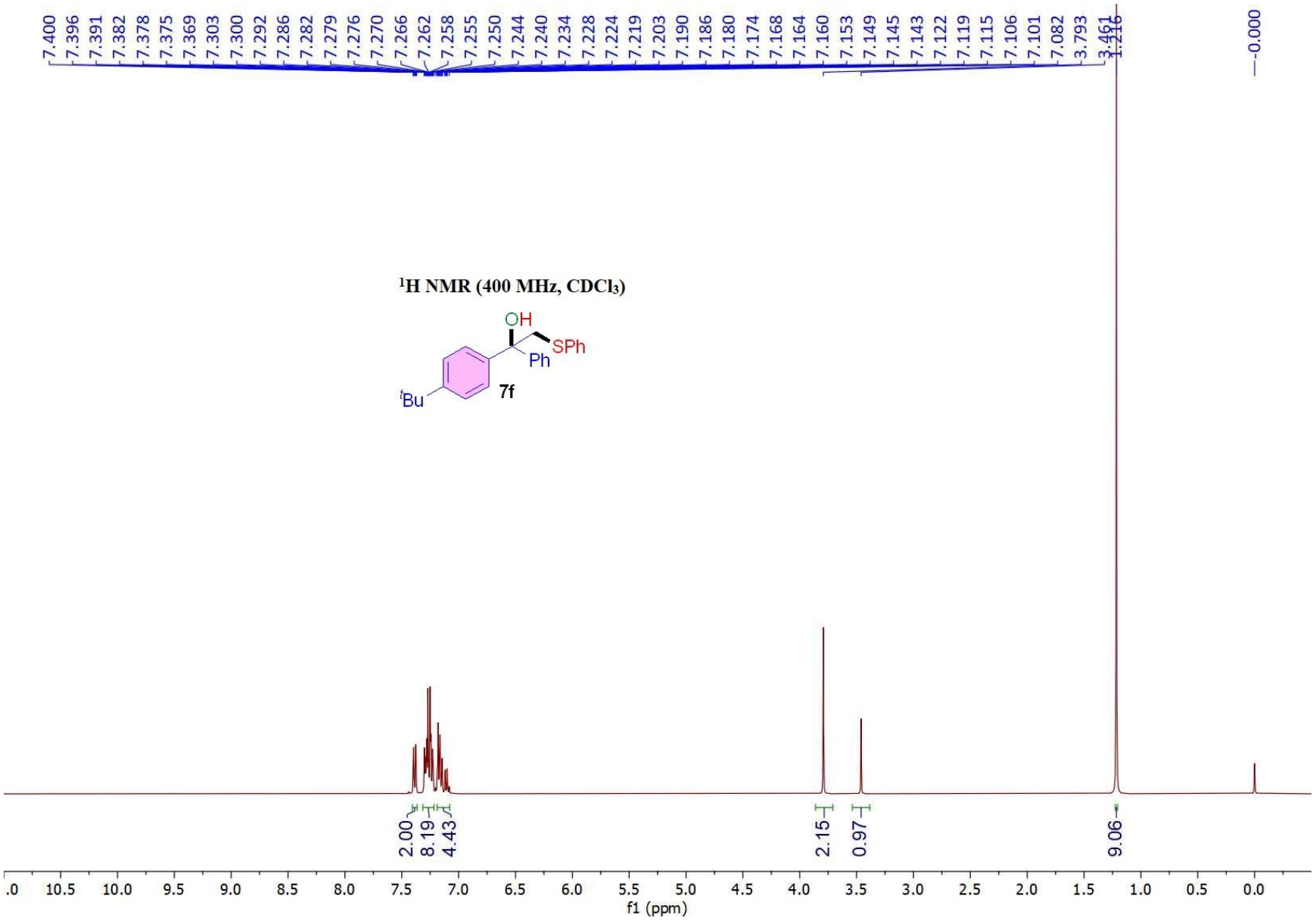
$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)

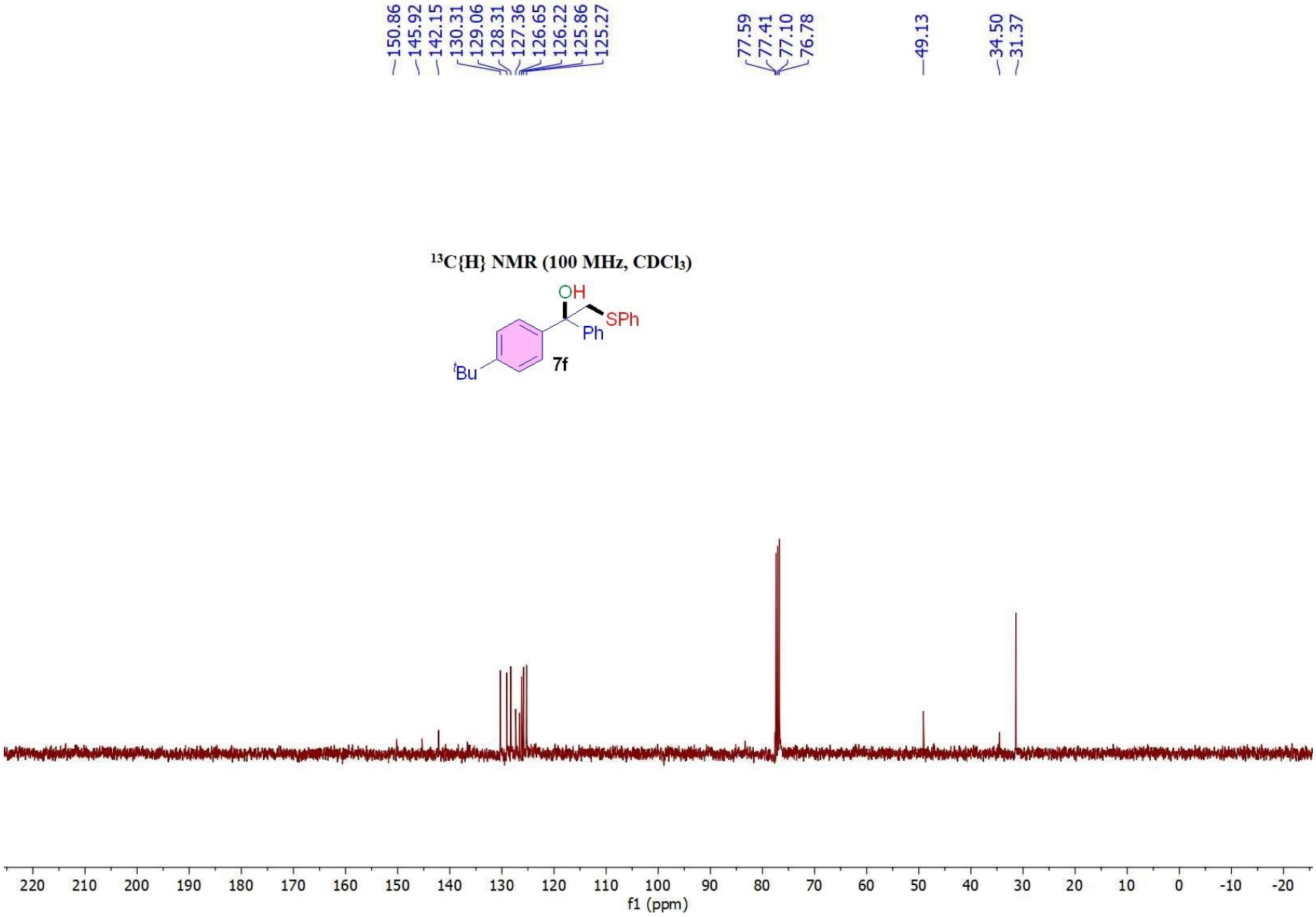


7e

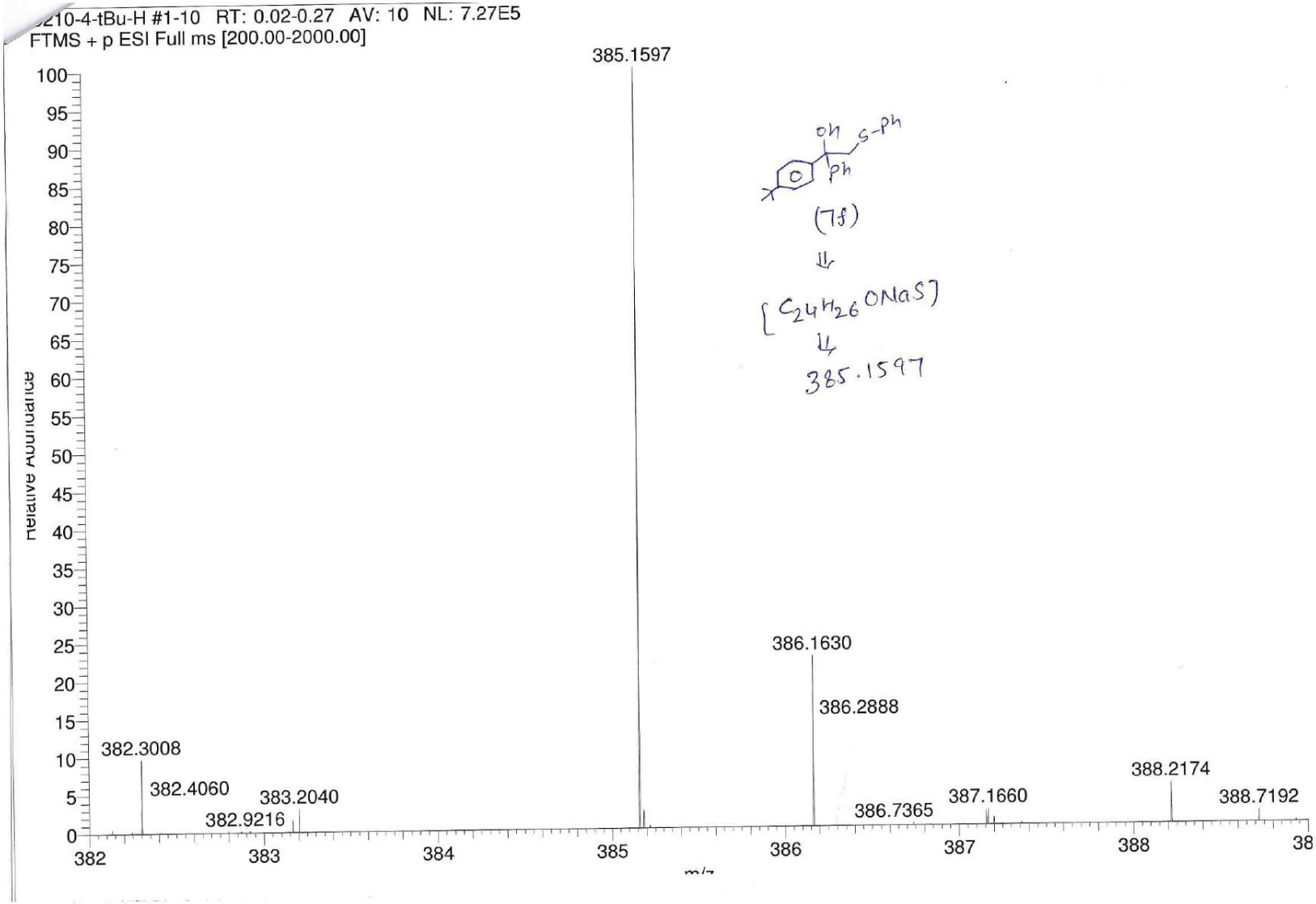


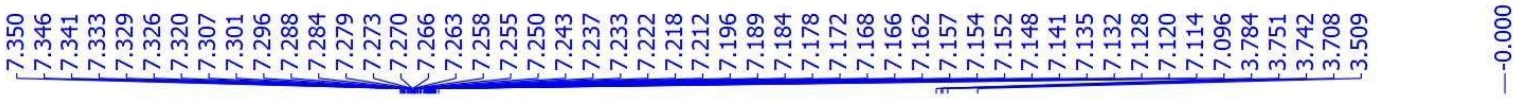

210-3-OMe-h #1-10 RT: 0.01-0.26 AV: 10 NL: 5.89E5
FTMS + p ESI Full ms [200.00-2000.00]
359.1076
360.1110
356.6299
361.1035
354.0833
357.0934
348.8821
352.5116
364.0105
366.2886
368.2115
369.6890
Relative Abundance
m/z
OH
SPh
Ph
OMe
(7e)
[C21H20O2NaS]
(359.1076)


7.400
7.396
7.391
7.382
7.378
7.375
7.369
7.303
7.300
7.292
7.286
7.282
7.279
7.276
7.270
7.266
7.262
7.258
7.255
7.250
7.244
7.240
7.234
7.228
7.224
7.219
7.203
7.190
7.186
7.180
7.174
7.168
7.164
7.160
7.153
7.149
7.145
7.143
7.122
7.119
7.115
7.106
7.101
7.082
3.793
3.461
1.216
-0.000
1H NMR (400 MHz, CDCl3)
OH
SPh
Ph
tBu
7f
2.00
8.19
4.43
2.15
0.97
9.06
f1 (ppm)

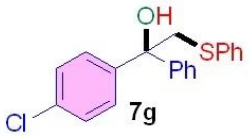


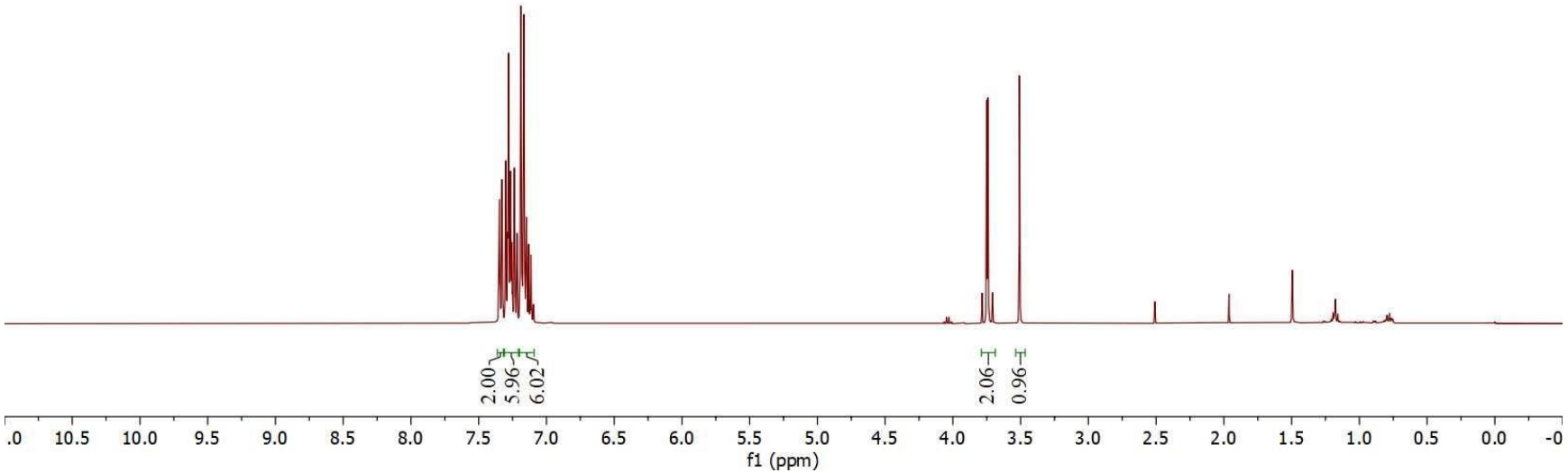
$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)


210-4-tBu-H #1-10 RT: 0.02-0.27 AV: 10 NL: 7.27E5
FTMS + p ESI Full ms [200.00-2000.00]
385.1597
386.1630
386.2888
382.3008
382.4060
383.2040
382.9216
386.7365
387.1660
388.2174
388.7192
Relative Abundance
m/z
(7f)
[C24H26ONaS]
385.1597



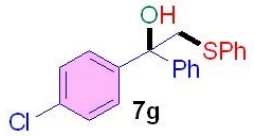
^{1}H NMR (400 MHz, $CDCl_3$)



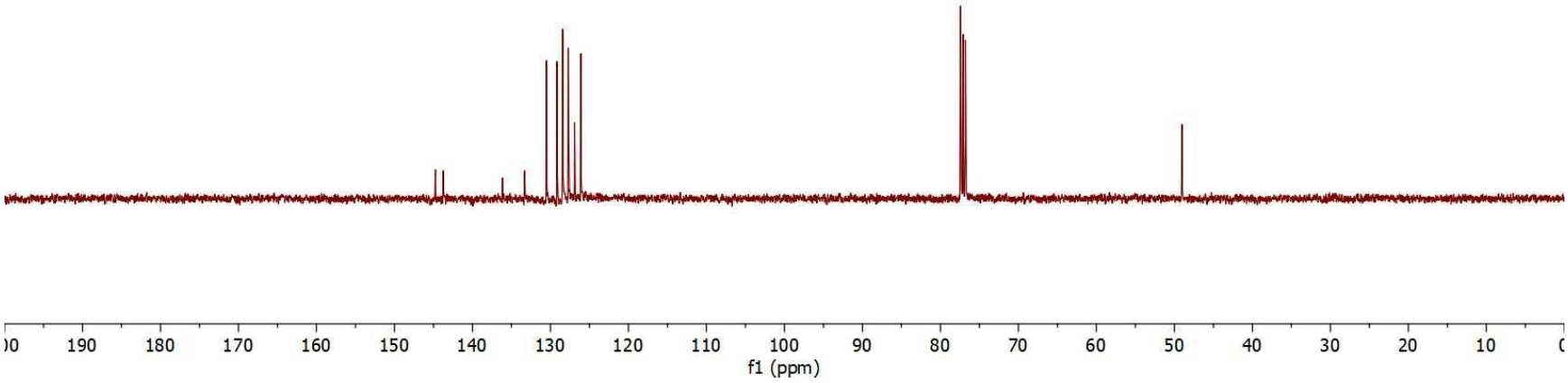


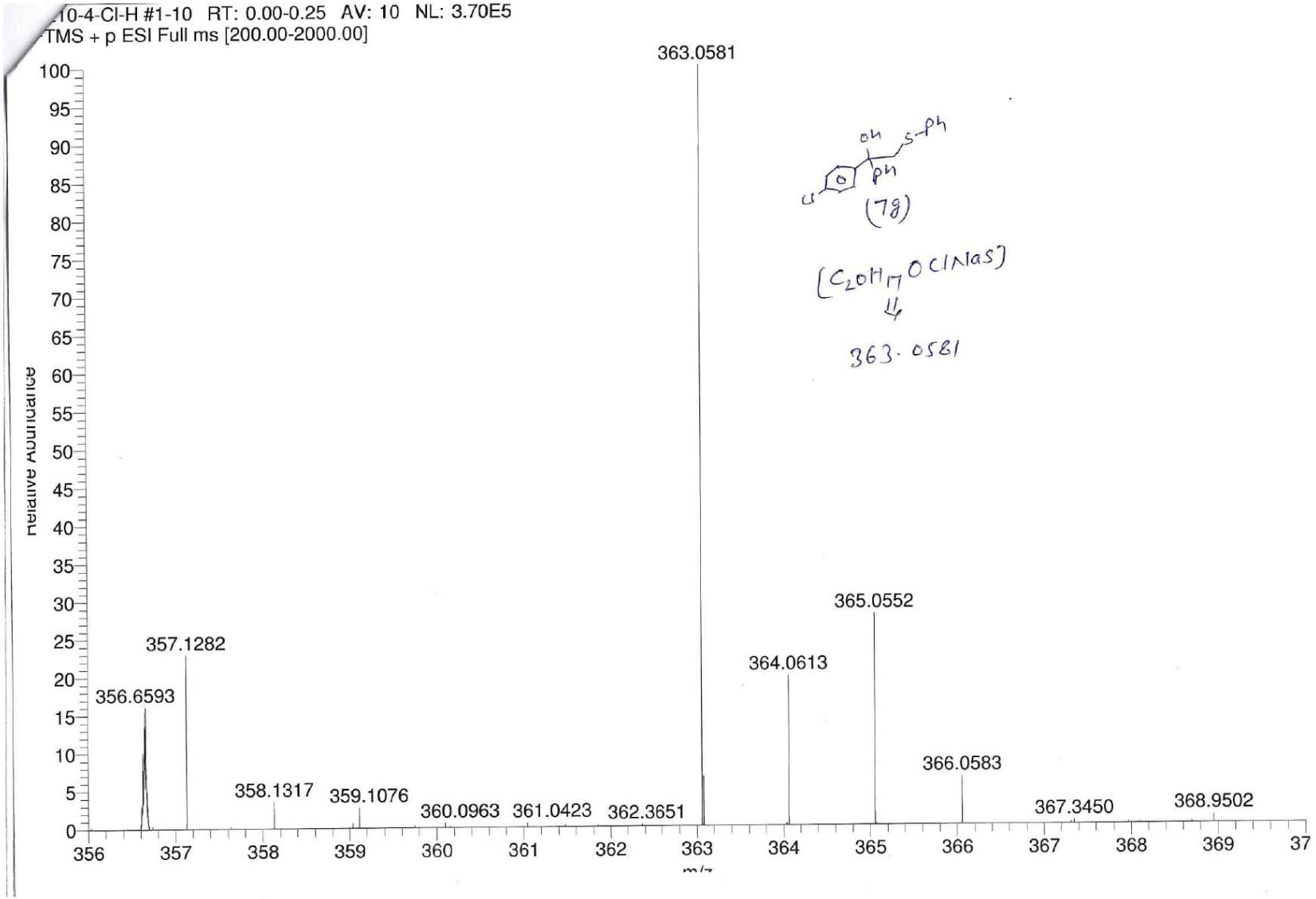
144.76
143.76
136.15
133.30
130.52
129.15
128.48
128.44
127.74
127.66
126.91
126.11
77.42
77.10
76.78
49.02

$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)

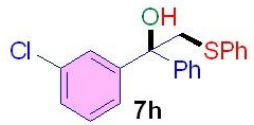


7g

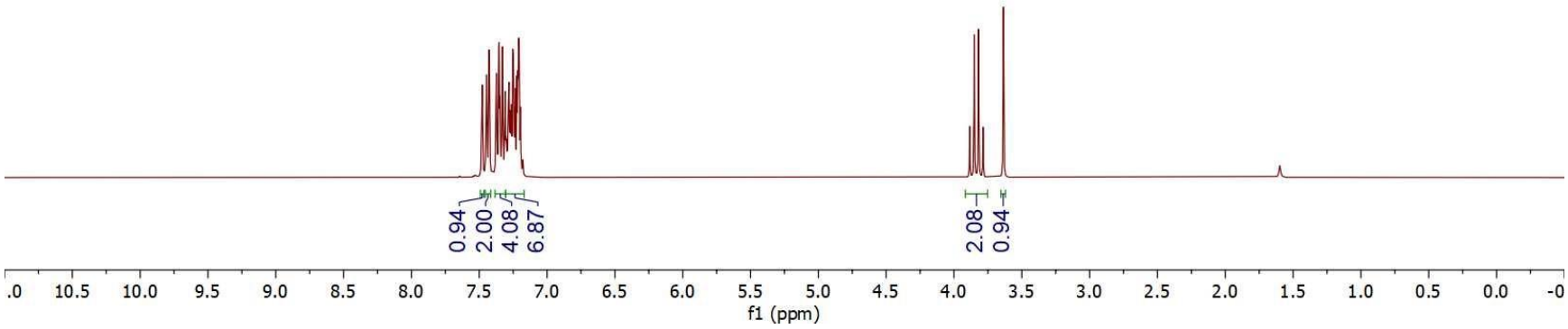



10-4-Cl-H #1-10 RT: 0.00-0.25 AV: 10 NL: 3.70E5
TMS + p ESI Full ms [200.00-2000.00]
363.0581
365.0552
357.1282
364.0613
356.6593
366.0583
358.1317
359.1076
360.0963
361.0423
362.3651
367.3450
368.9502
Relative Abundance
m/z
OH
S-Ph
Ph
Cl
(7g)
[C20H17OClNaS]+
363.0581

^{1}H NMR (400 MHz, $CDCl_3$)

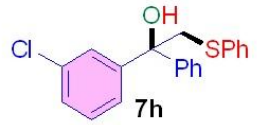


7h

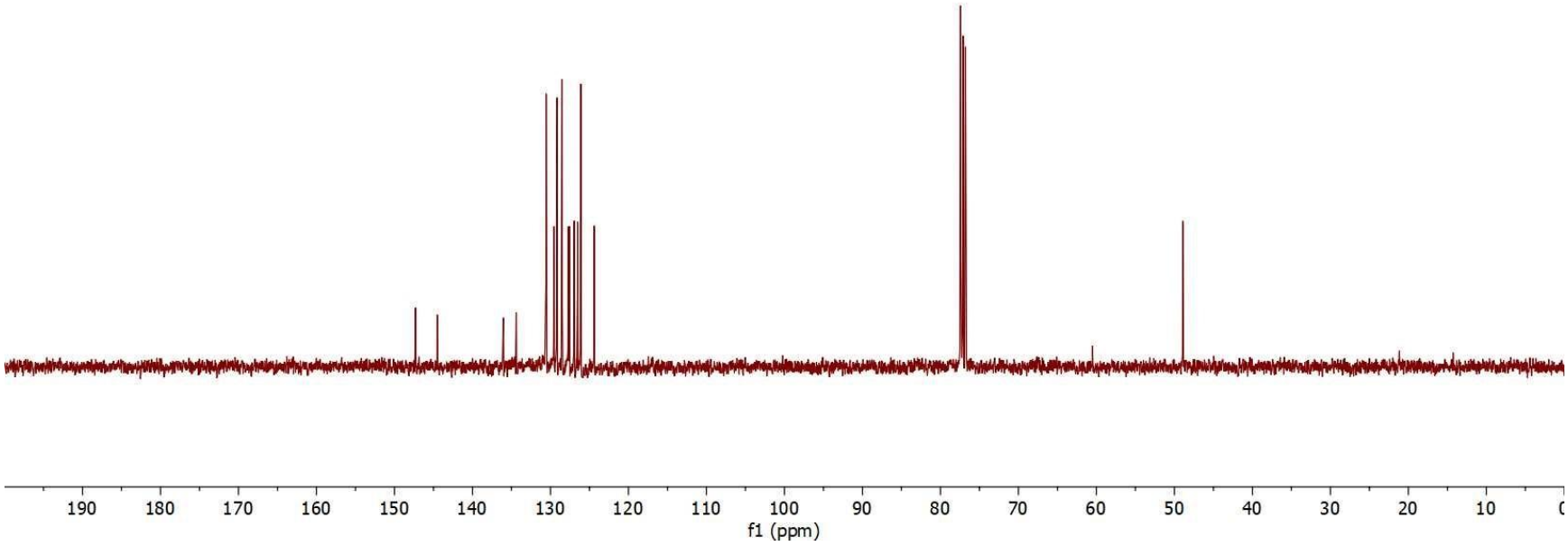


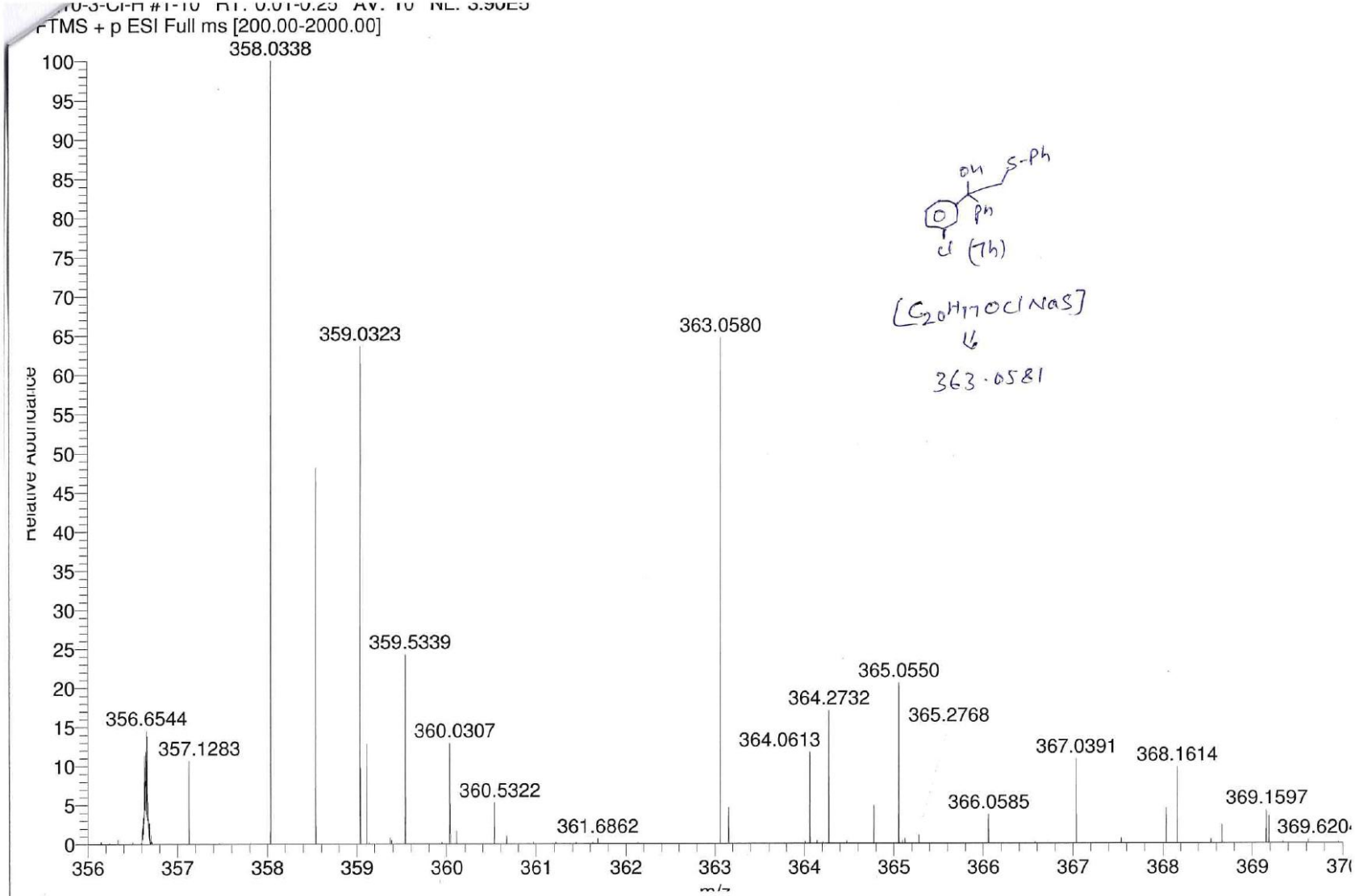
147.29
144.51
136.05
134.38
130.56
129.57
129.15
128.51
127.71
127.57
126.96
126.52
126.10
124.39
77.42
77.10
76.78
60.49
48.93

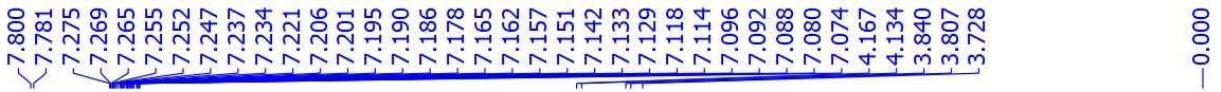
$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)



7h

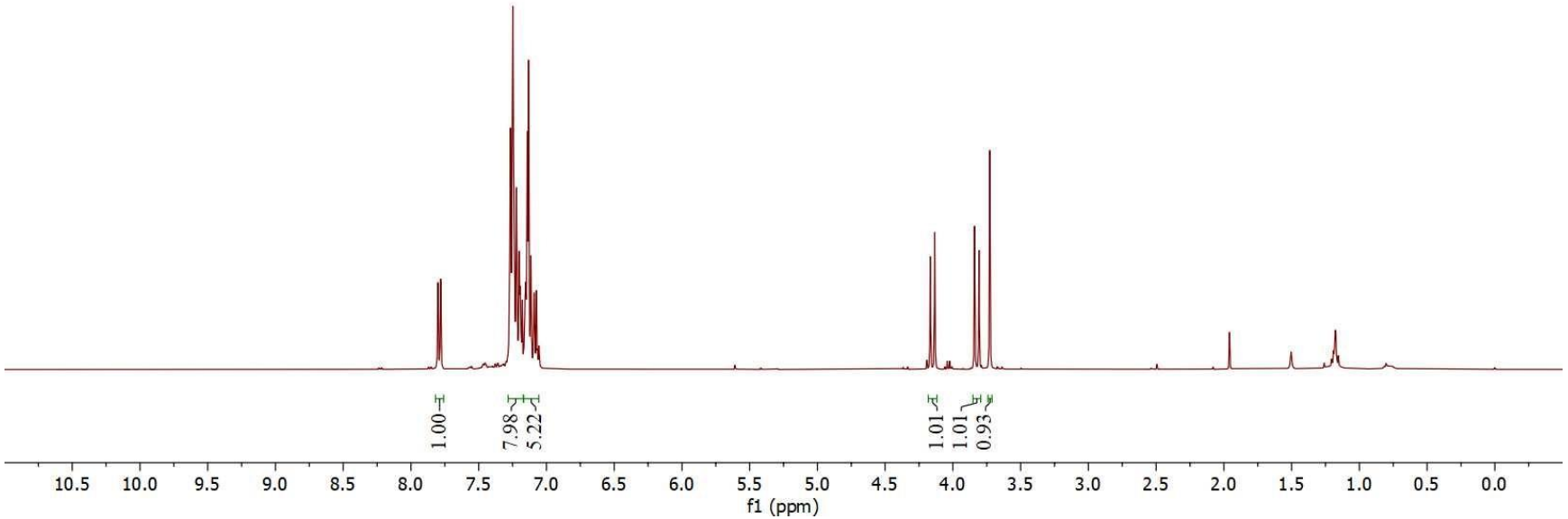



FTMS + p ESI Full ms [200.00-2000.00]
358.0338
359.0323
363.0580
359.5339
365.0550
364.2732
365.2768
356.6544
357.1283
360.0307
364.0613
367.0391
368.1614
360.5322
366.0585
369.1597
361.6862
Relative Abundance
m/z
OH
S-Ph
Ph
Cl (7h)
[C20H17OClNaS]
363.0581

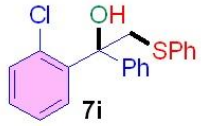


^{1}H NMR (400 MHz, $CDCl_3$)

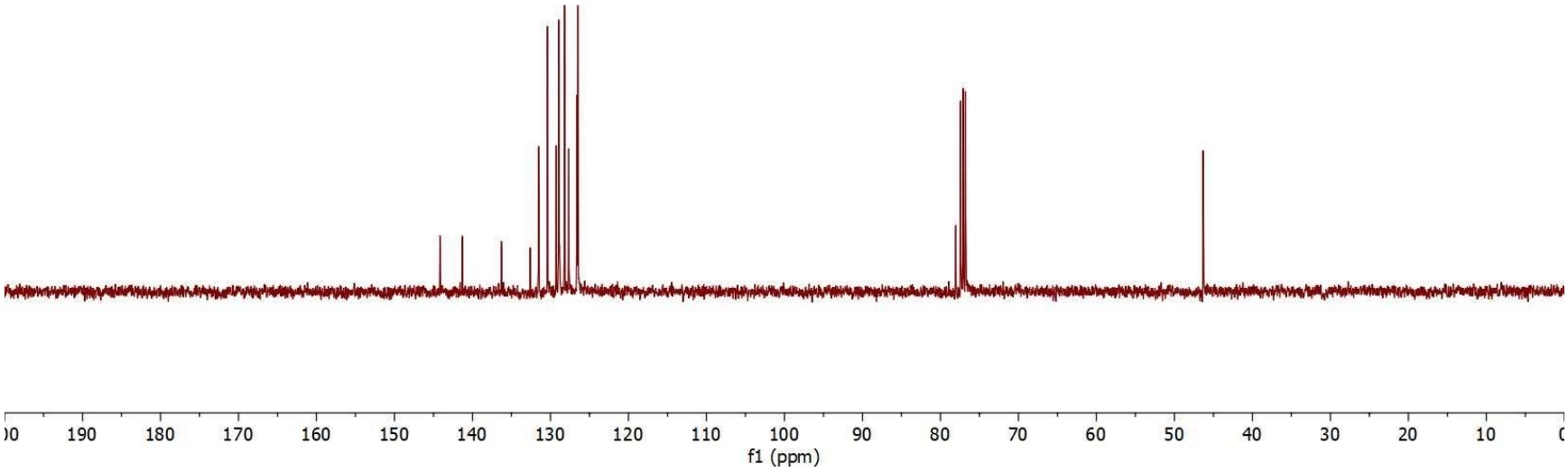
7i

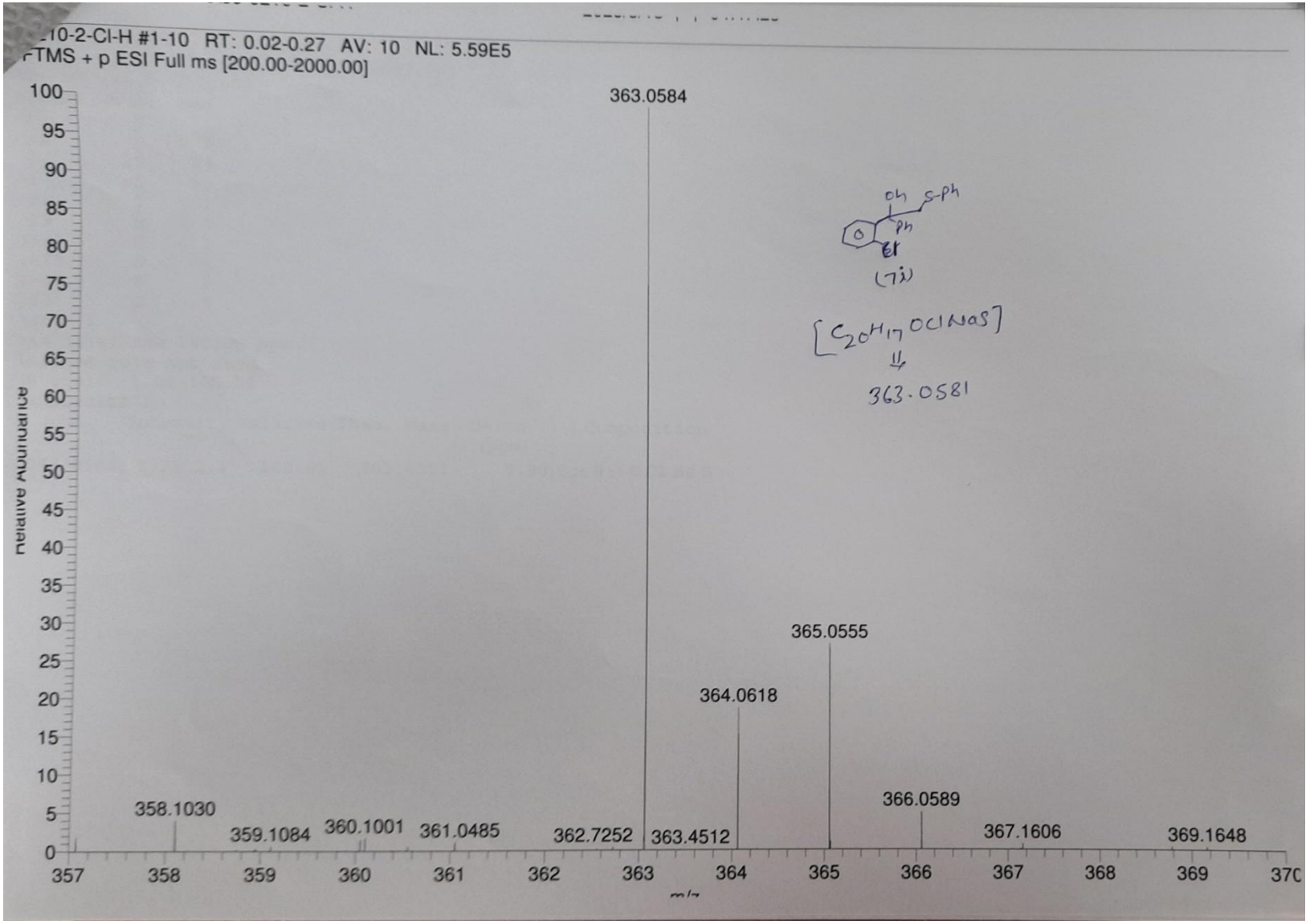


$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)

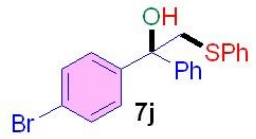


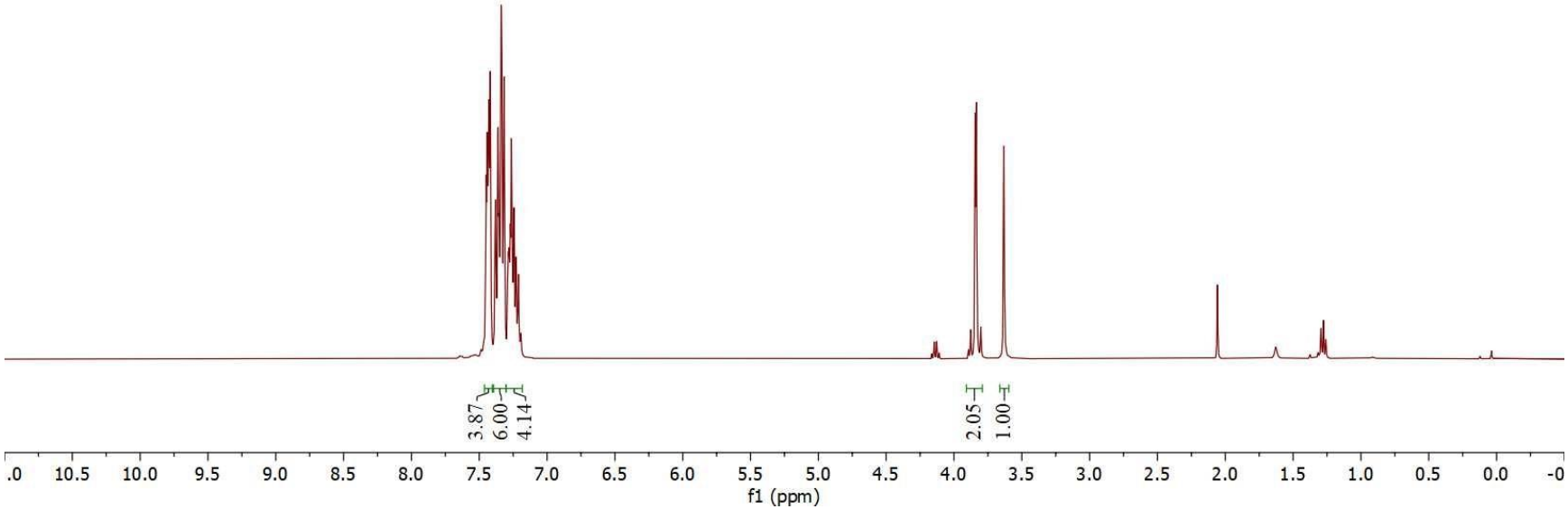
7i




10-2-Cl-H #1-10 RT: 0.02-0.27 AV: 10 NL: 5.59E5
TMS + p ESI Full ms [200.00-2000.00]
Relative Abundance
m/z
363.0584
365.0555
364.0618
366.0589
358.1030
359.1084
360.1001
361.0485
362.7252
363.4512
367.1606
369.1648
OH
S-Ph
Ph
Cl
(7i)
[C20H17OClNaS]
363.0581

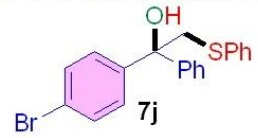
^{1}H NMR (400 MHz, $CDCl_3$)



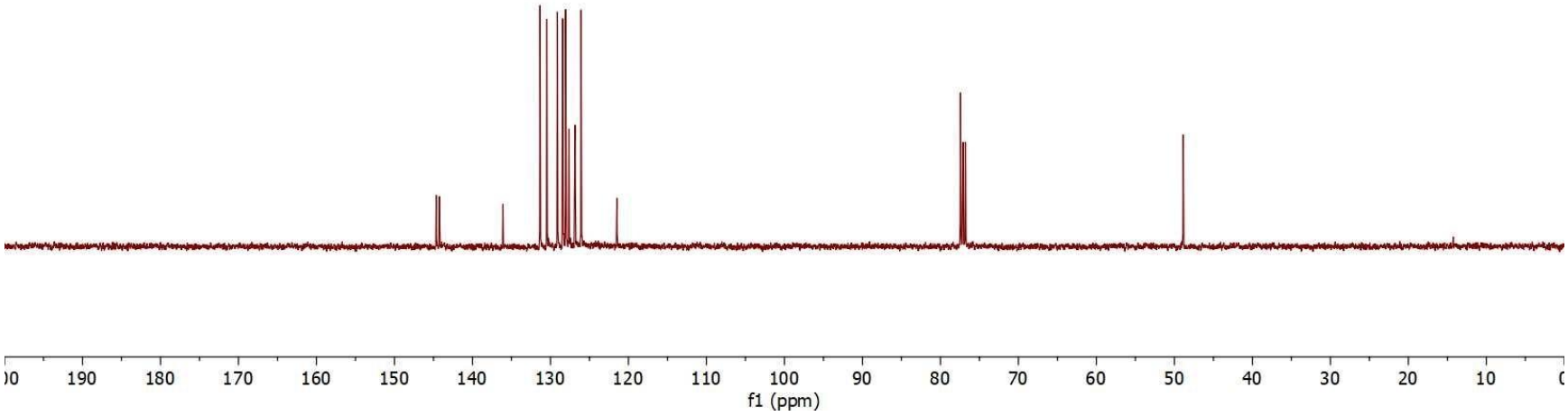


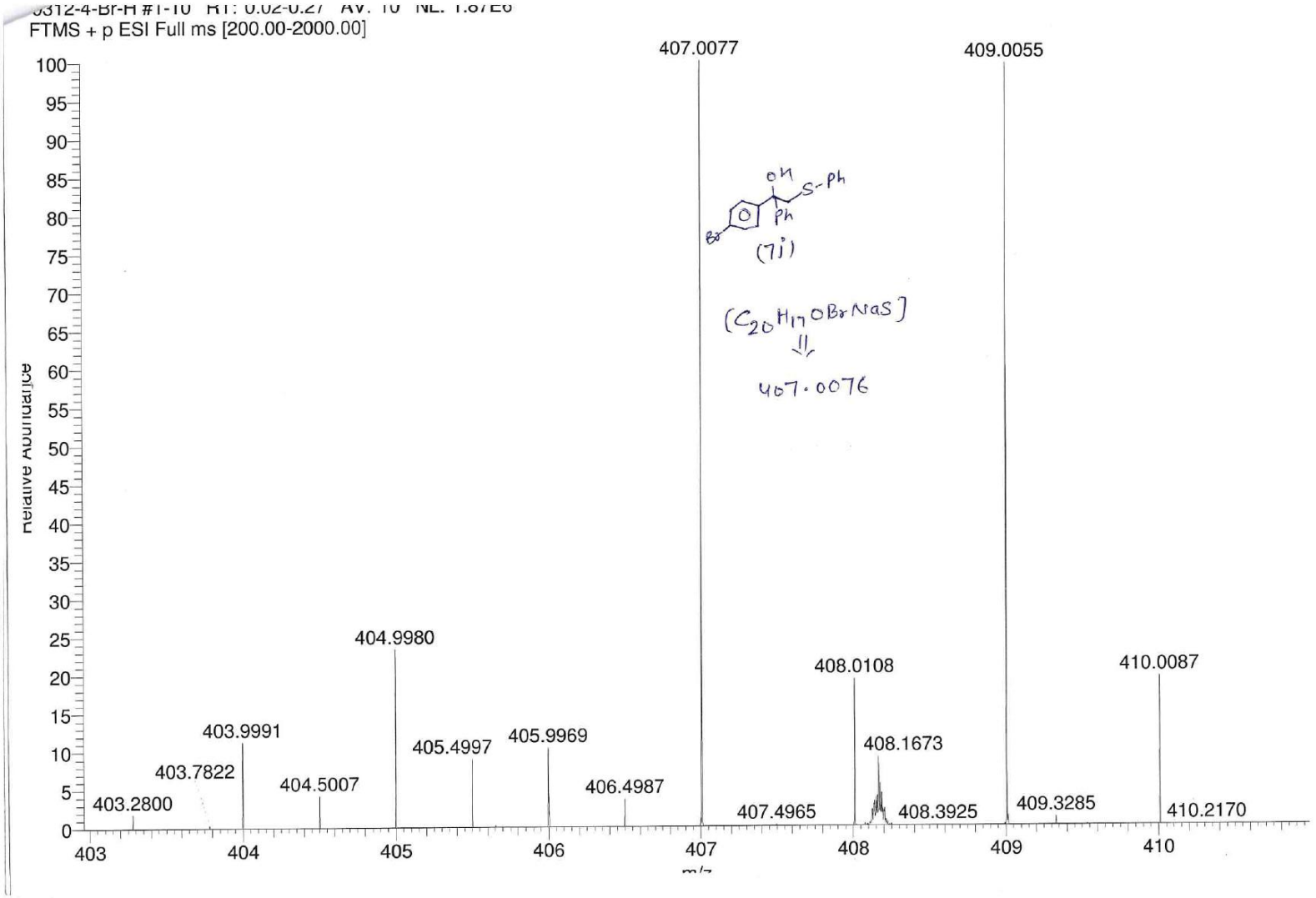
144.65
144.25
136.10
131.34
130.46
129.11
128.44
128.05
127.63
126.85
126.07
121.48
77.42
77.10
76.78
48.87

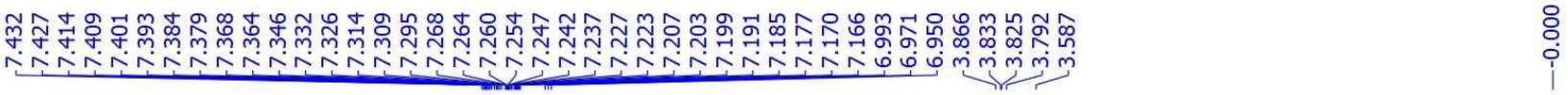
$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)



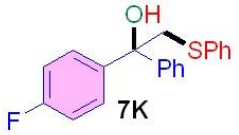
7j

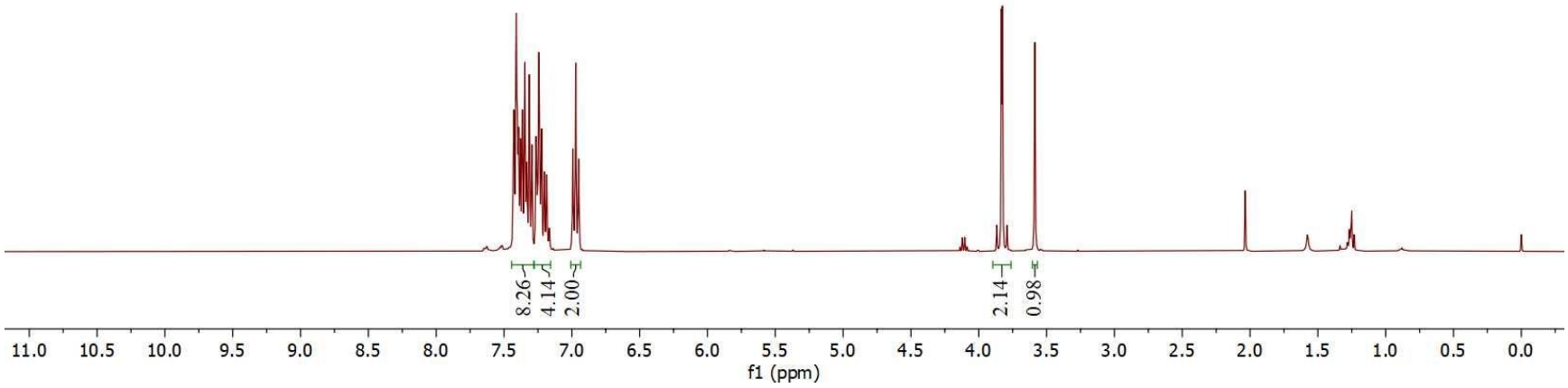


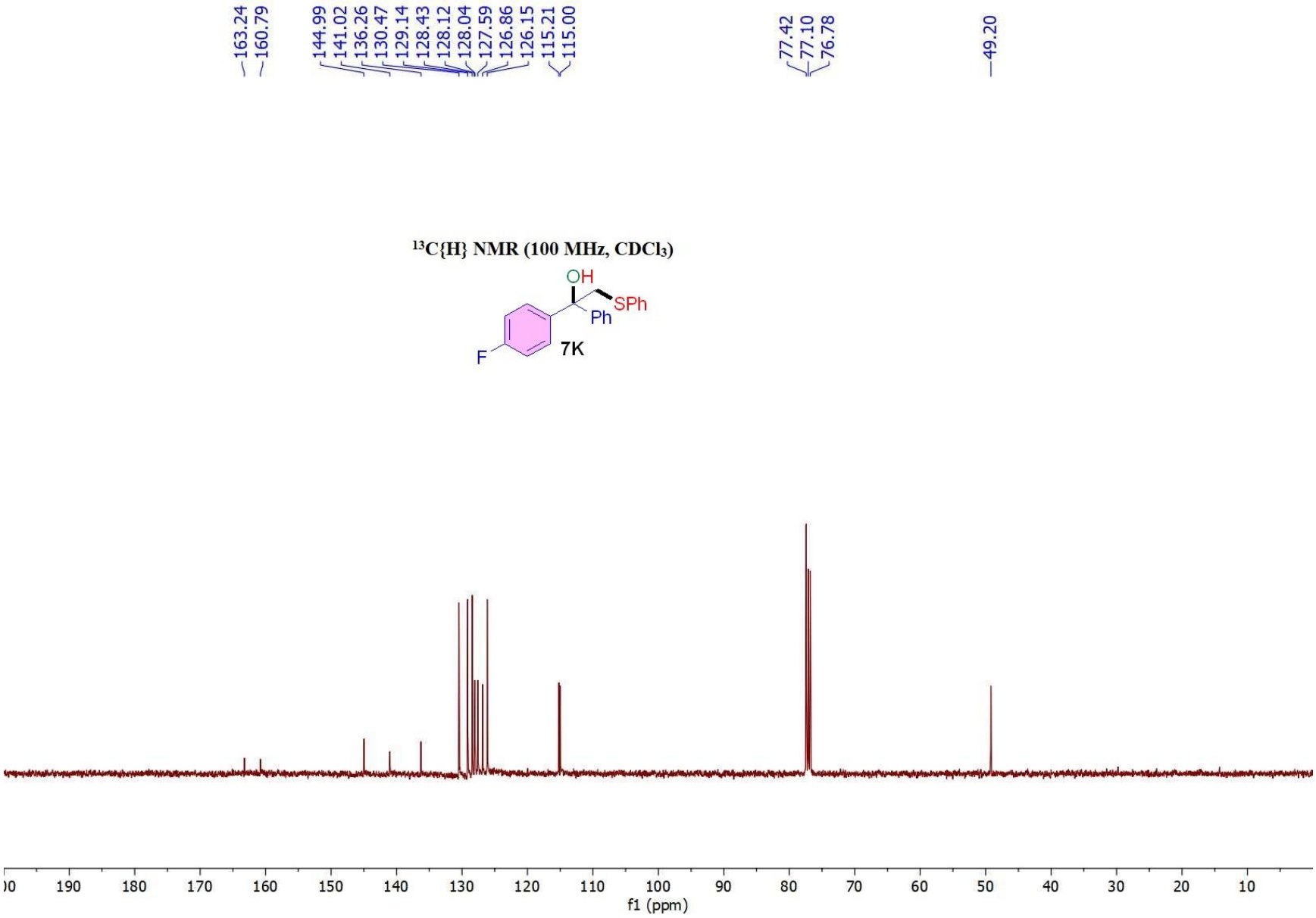

FTMS + p ESI Full ms [200.00-2000.00]
Relative Abundance
m/z
407.0077
409.0055
404.9980
408.0108
410.0087
403.9991
408.1673
405.4997
405.9969
403.7822
404.5007
406.4987
403.2800
407.4965
408.3925
409.3285
410.2170
OH
S-Ph
Ph
Br
(7j)
(C20H17OBrNaS]
407.0076



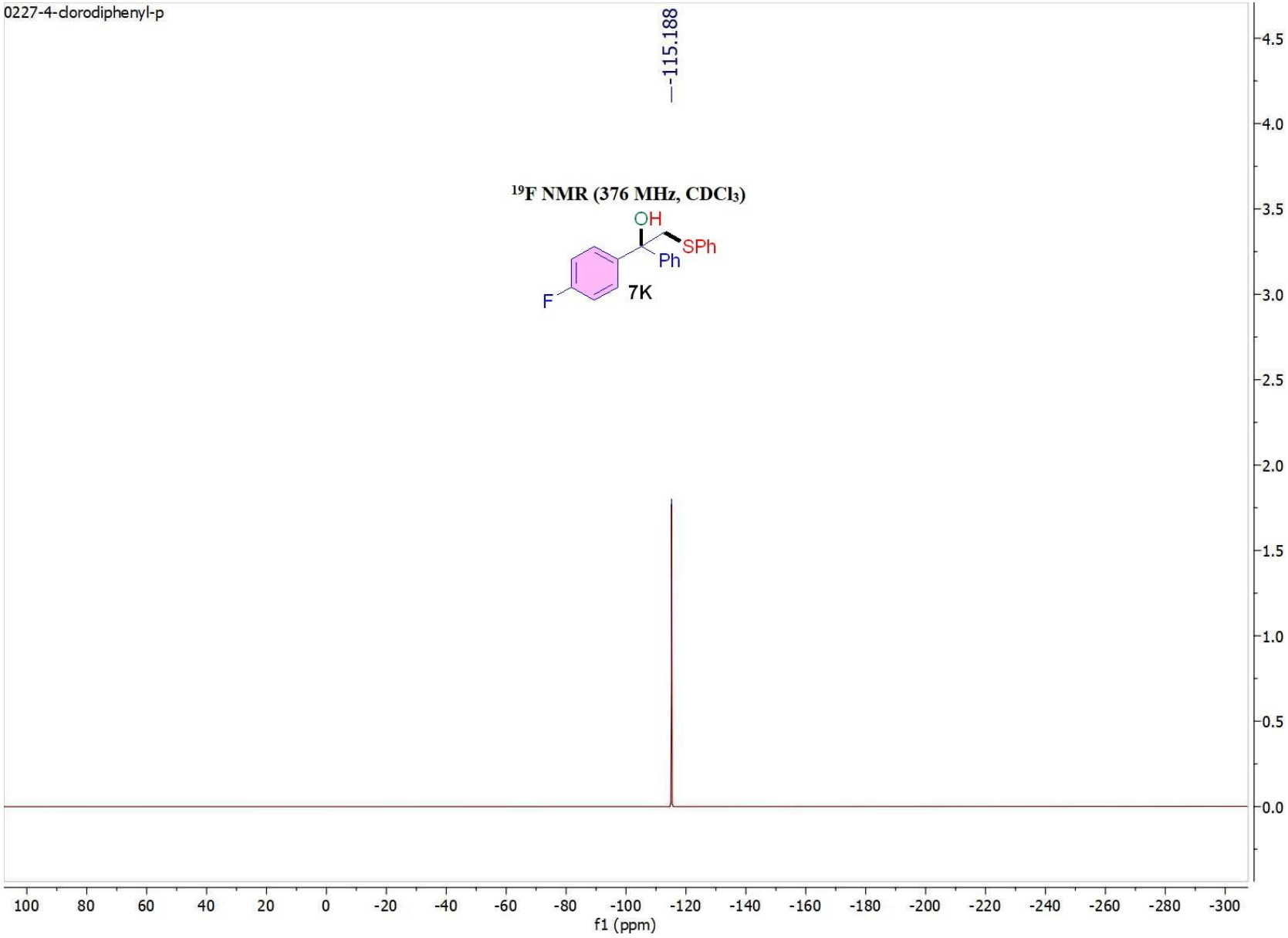
^{1}H NMR (400 MHz, $CDCl_3$)



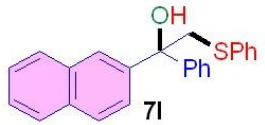




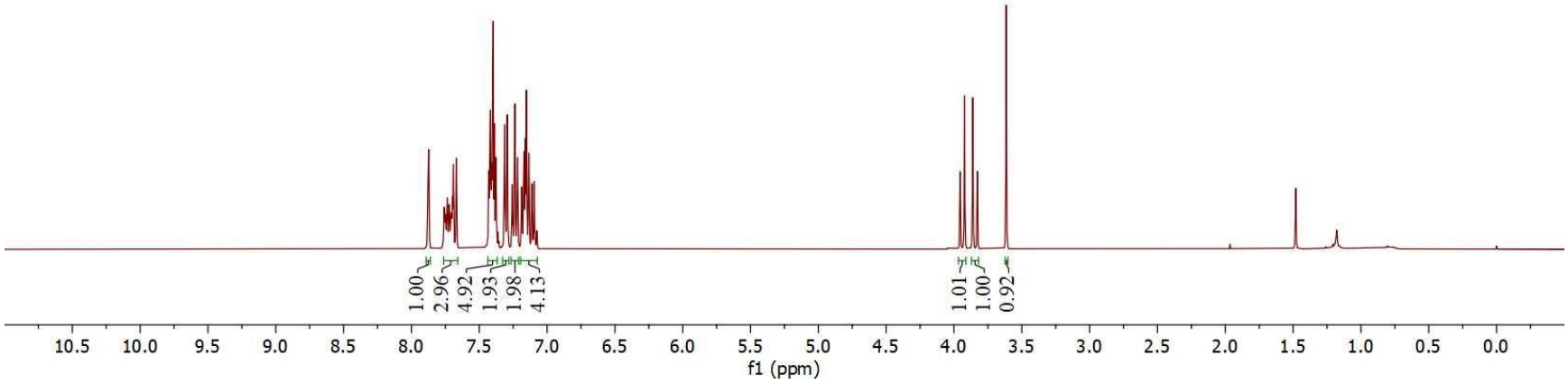
$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)

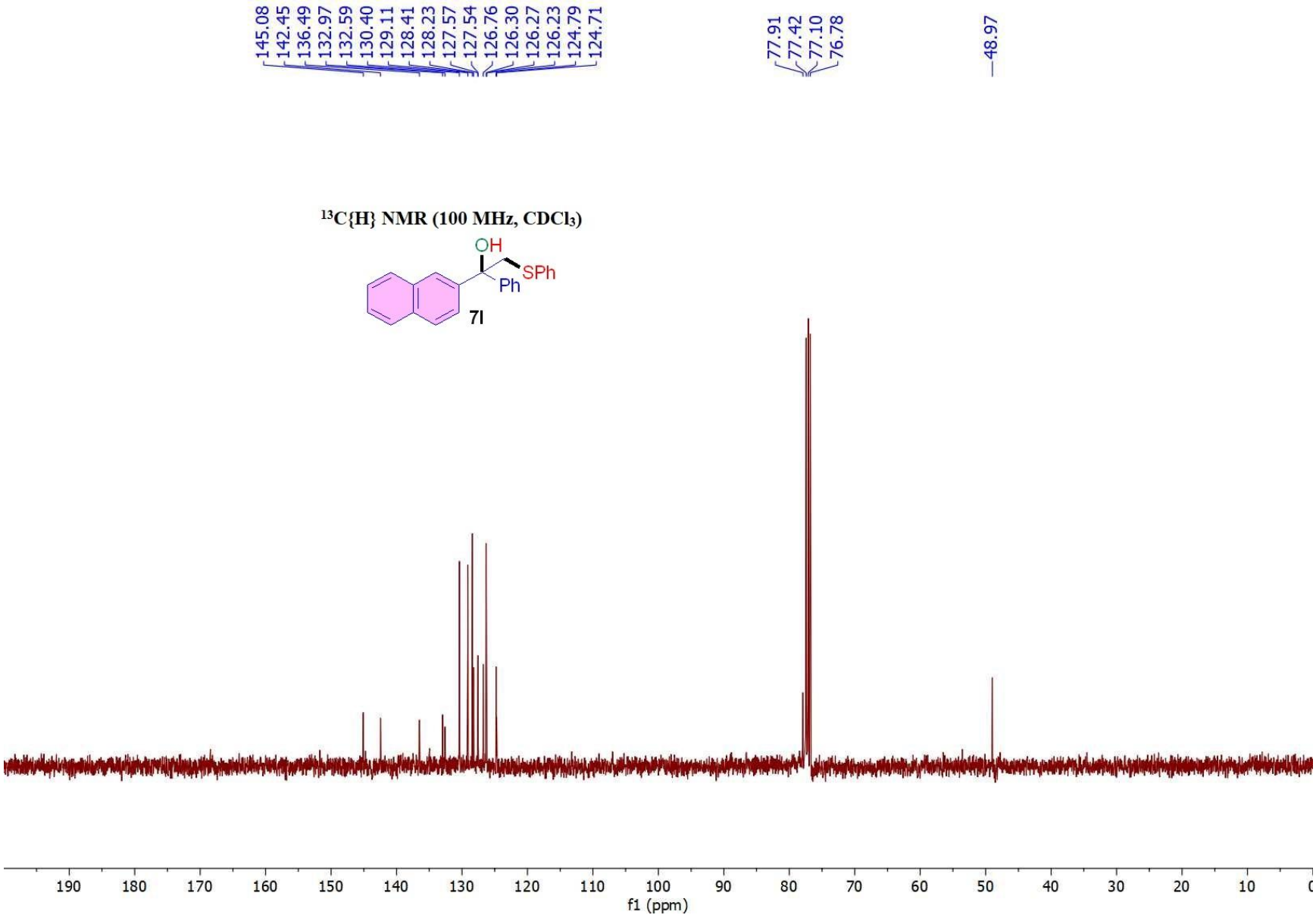

0227-4-clorodiphenyl-p
-115.188
19F NMR (376 MHz, CDCl3)
OH
SPh
Ph
F
7K
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 -220 -240 -260 -280 -300
f1 (ppm)

^{1}H NMR (400 MHz, $CDCl_3$)



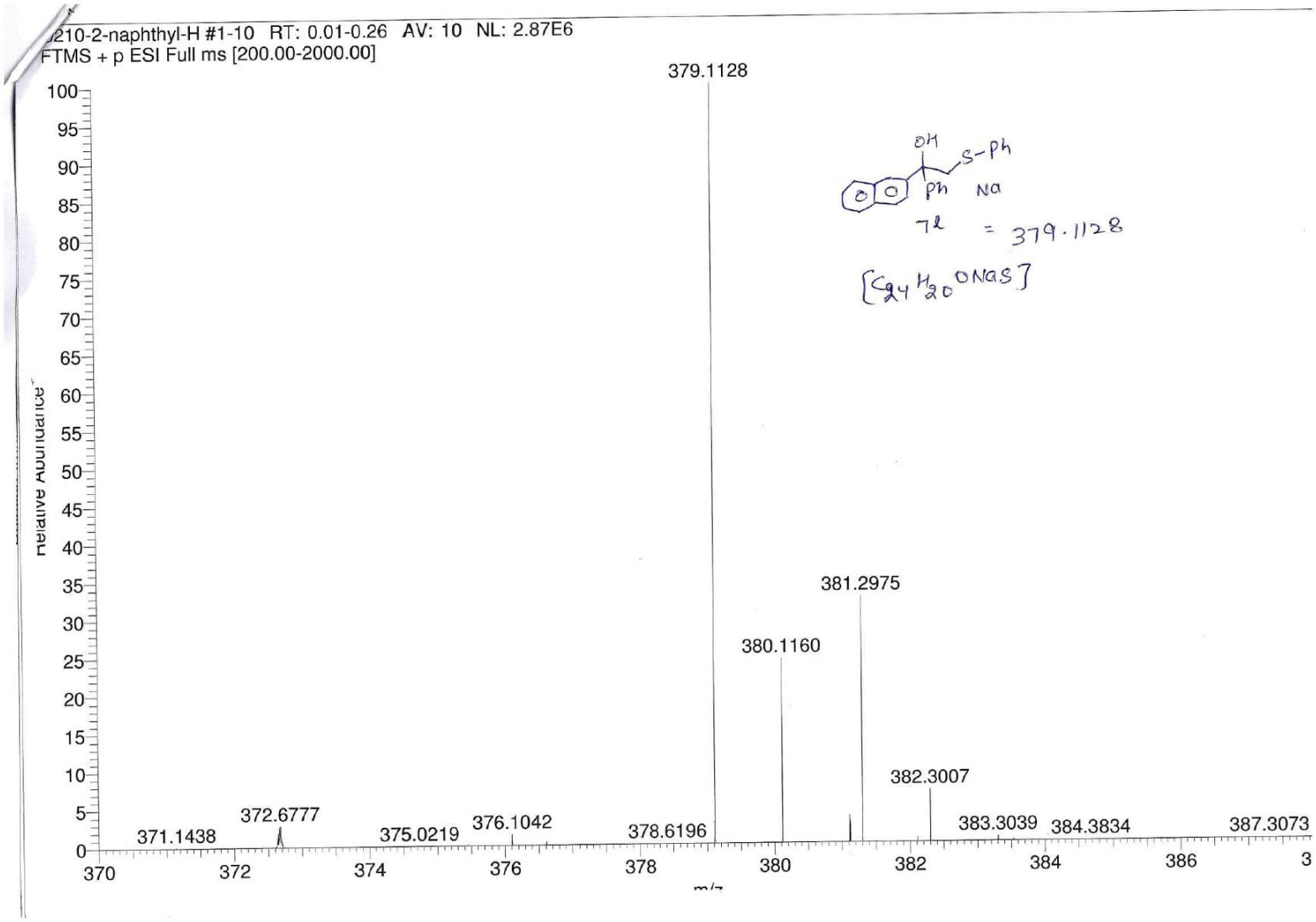
7l

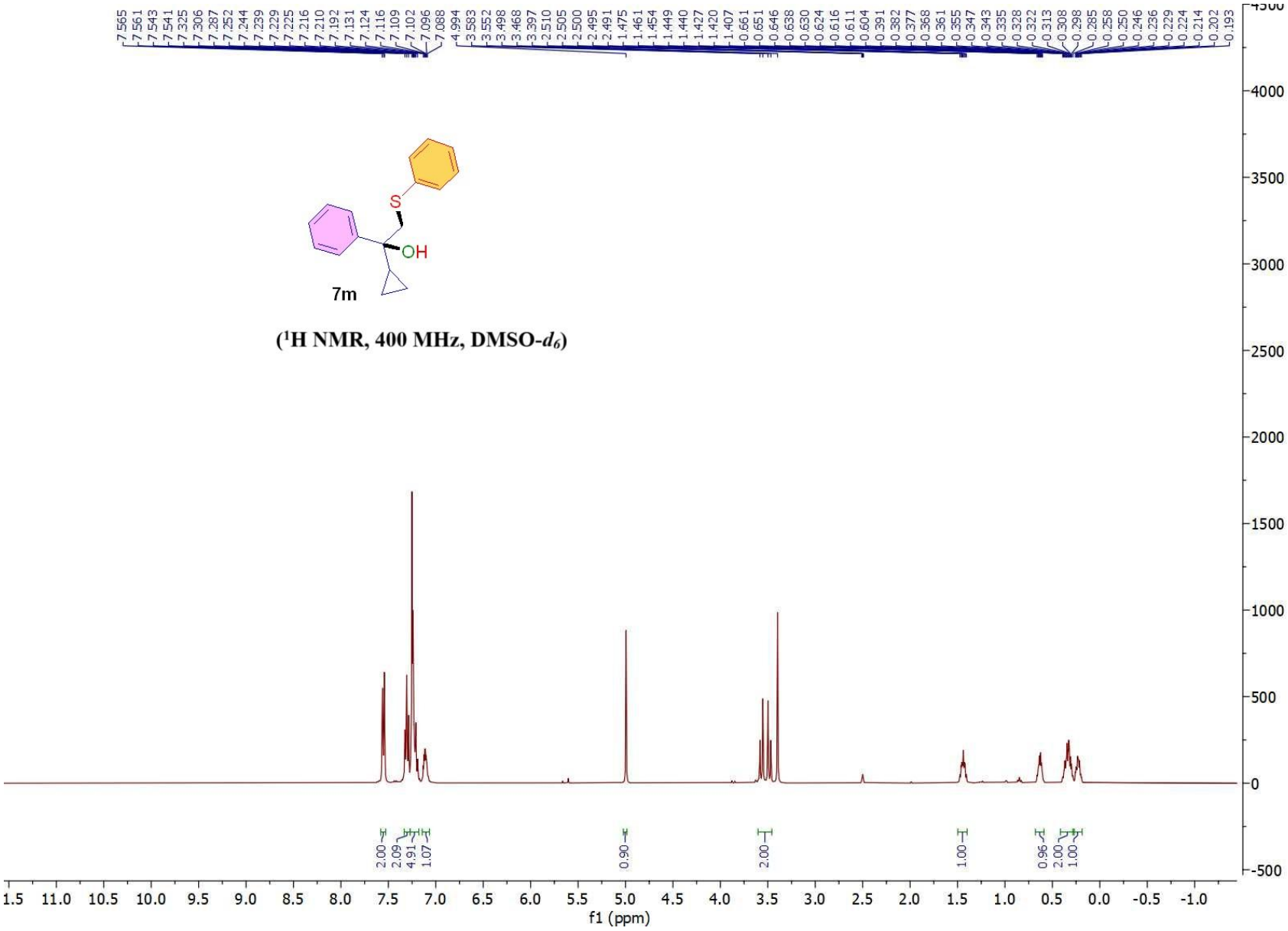


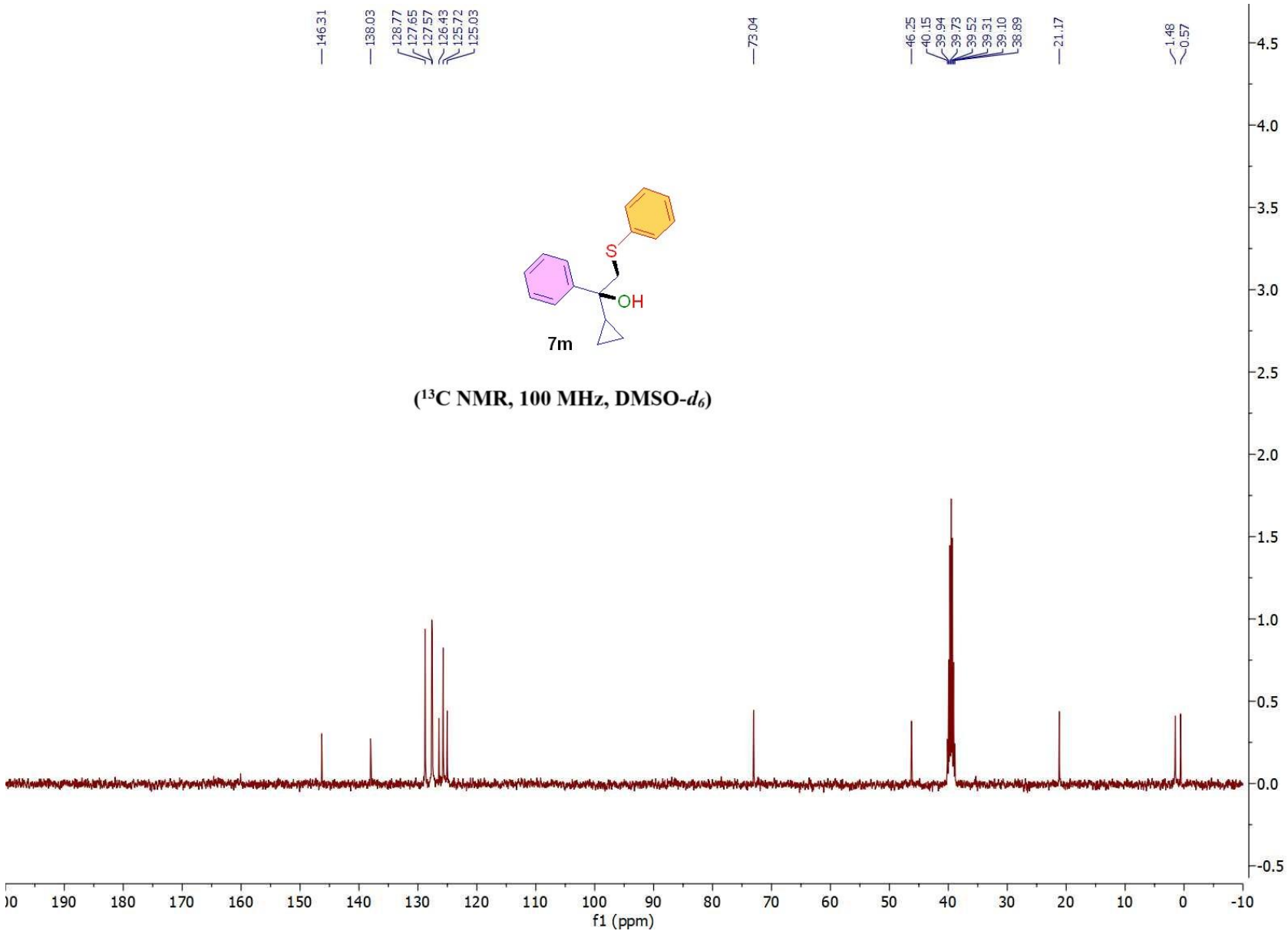


$^{13}C\{H\}$ NMR (100 MHz, $CDCl_3$)

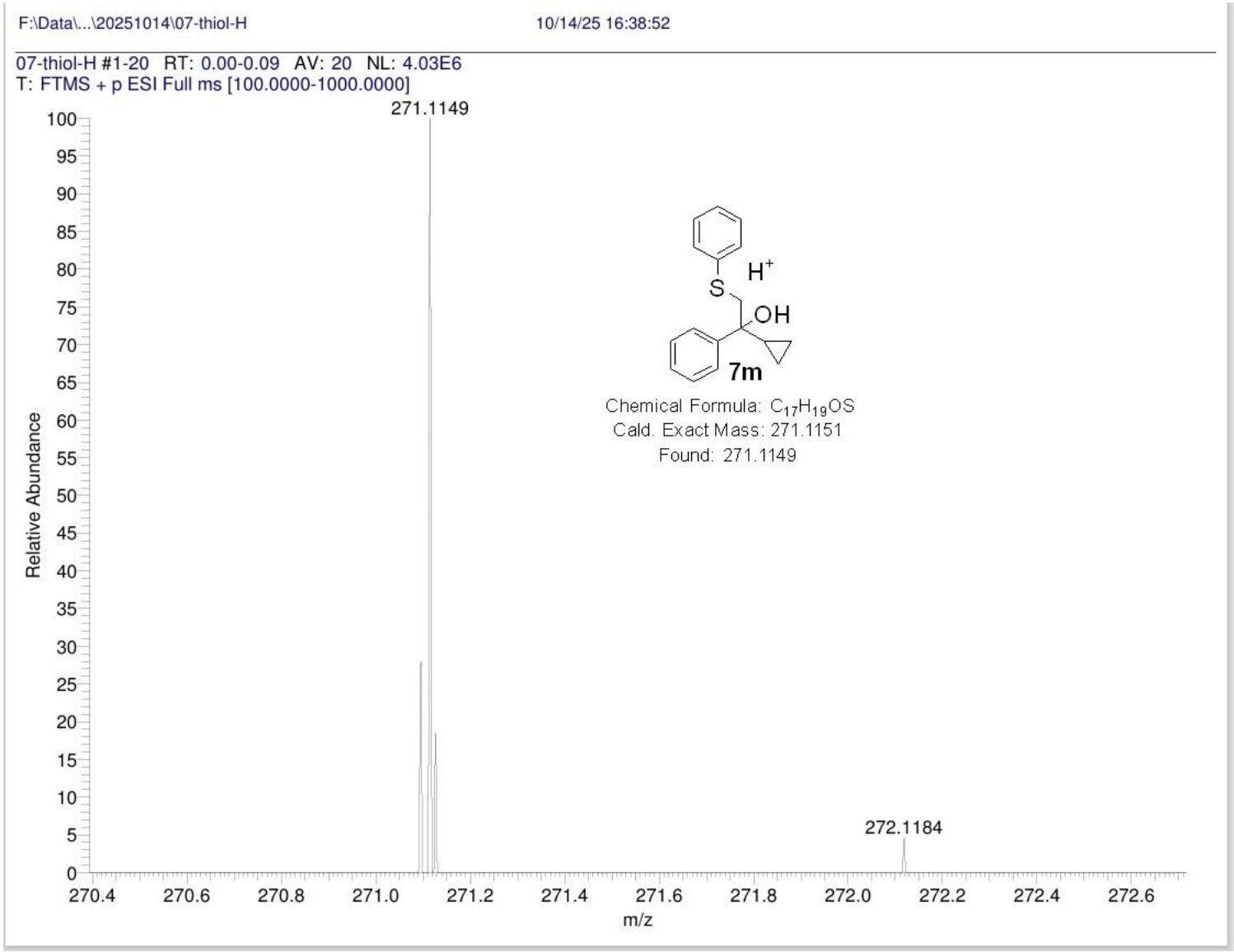
7l


0210-2-naphthyl-H #1-10 RT: 0.01-0.26 AV: 10 NL: 2.87E6
FTMS + p ESI Full ms [200.00-2000.00]
379.1128
381.2975
380.1160
382.3007
372.6777
371.1438
375.0219
376.1042
378.6196
383.3039
384.3834
387.3073
Relative Abundance
m/z
OH
S-Ph
Ph
Na
Tl = 379.1128
[C24H20ONaS]

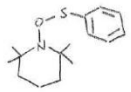

7.565
7.561
7.543
7.541
7.325
7.306
7.287
7.252
7.244
7.239
7.229
7.225
7.216
7.210
7.192
7.131
7.124
7.116
7.109
7.102
7.096
7.088
4.994
3.583
3.552
3.498
3.468
3.397
2.510
2.505
2.500
2.495
2.491
1.475
1.461
1.454
1.449
1.440
1.427
1.420
1.407
0.661
0.651
0.646
0.638
0.630
0.624
0.616
0.611
0.604
0.391
0.382
0.377
0.368
0.361
0.355
0.347
0.343
0.335
0.328
0.322
0.313
0.308
0.298
0.285
0.258
0.250
0.246
0.236
0.229
0.224
0.214
0.202
0.193
S
OH
7m
(1H NMR, 400 MHz, DMSO-d6)
2.00
2.09
4.91
1.07
0.90
2.00
1.00
0.96
2.00
1.00
f1 (ppm)



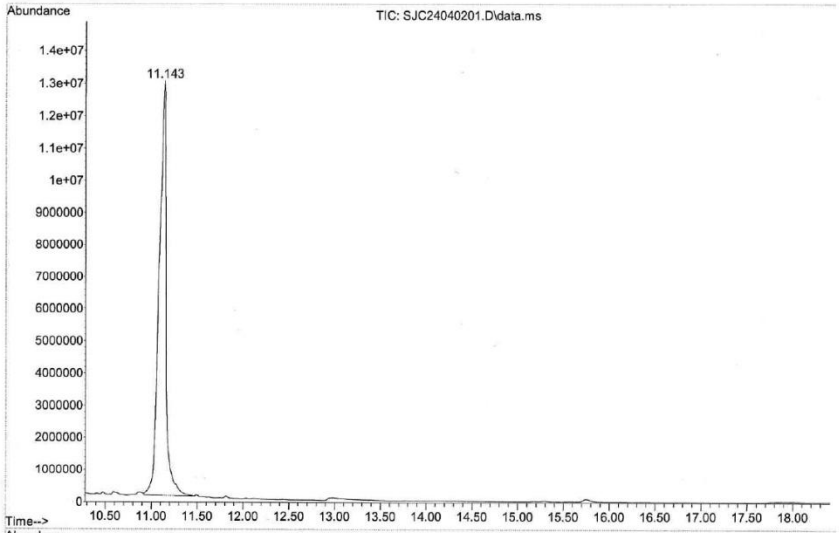
(¹³C NMR, 100 MHz, DMSO-d_6)

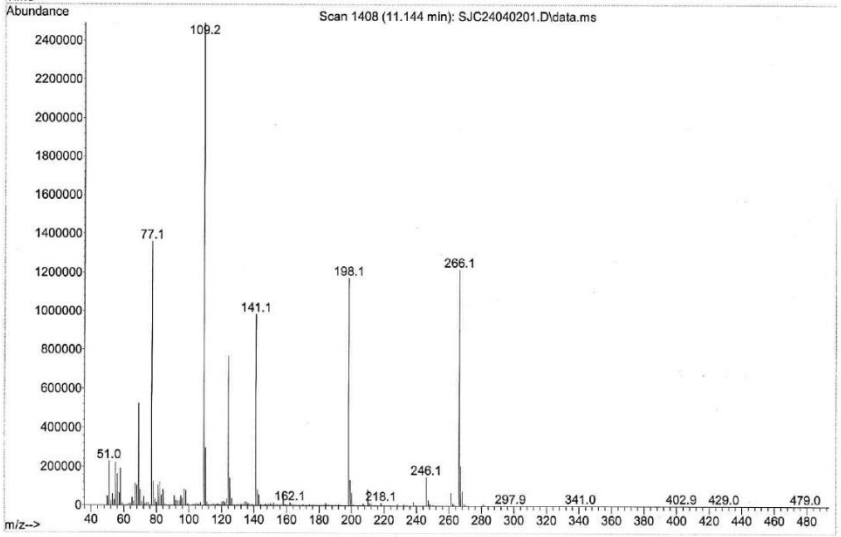

F:\Data\...\20251014\07-thiol-H
10/14/25 16:38:52
07-thiol-H #1-20 RT: 0.00-0.09 AV: 20 NL: 4.03E6
T: FTMS + p ESI Full ms [100.0000-1000.0000]
271.1149
272.1184
Relative Abundance
m/z
7m
Chemical Formula: C17H19OS
Cald. Exact Mass: 271.1151
Found: 271.1149

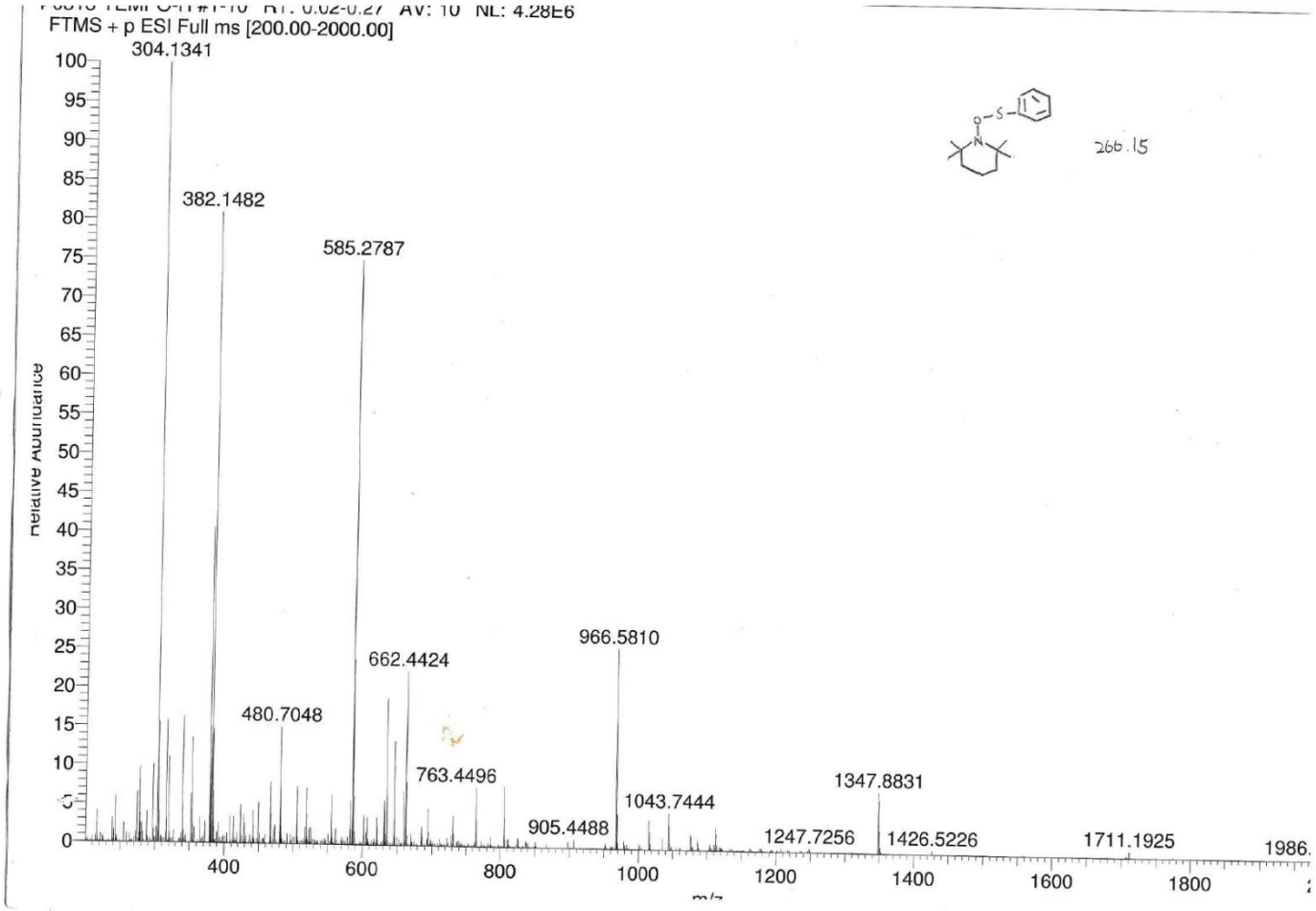
File :D:\MassHunter\GCMS\1\data\830\SJC24040201.D
Operator :
Acquired : 02 Apr 2024 14:31 using AcqMethod 20190906-1.M
Instrument : 830-GCMS
Sample Name: 1
Misc Info :
Vial Number: 13



Exact Mass : 266.






FTMS + p ESI Full ms [200.00-2000.00]
304.1341
382.1482
585.2787
662.4424
480.7048
763.4496
966.5810
905.4488
1043.7444
1247.7256
1347.8831
1426.5226
1711.1925
266.15
Relative Abundance
m/z