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$AlAr_3(THF)$: Highly Efficient Reagents for Cross-couplings with Aryl Bromides and Chlorides Catalyzed by the Economic Palladium Complex of PCy_3

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Table of Contents

I. General Procedures of Couplings of Aryl Halides with $AlAr_3$(THF) S3

II. Blank Reaction of $Al(2,4,6\text{-}Me_3C_6H_2)_3$(THF) in the Presence of $Pd(OAc)_2$ and PCy_3 S3

III. ^{1}H and ^{13}C NMR Spectroscopic Data of Coupling Products S3

IV. References S8

V. ^{1}H and ^{13}C NMR Spectra of Coupling Products S8

1. **4-Methoxybipheny** S9
2. **4-phenylthioanisol**e S11
3. **4-Methybiphenyl** S13
4. **4-*tert*-Butylbiphenyl** S15
5. **4-Fluorobiphenyl** S17
6. **4-Nitrobiphenyl** S19
7. **3,5-Dimethylbiphenyl** S21
8. **2-Methoxybiphenyl** S23
9. **2,4-Dimethylbiphenyl** S25
10. **2,6-Dimethylbiphenyl** S27
11. **2-Methybiphenyl** S29
12. **4-Cyanobiphenyl** S31
13. **4-Phenyl-methylbenzoate** S33
14. **4-Methoxy-2',4',6'-trimethylbiphenyl** S35
15. **4-Fluoro-2',4',6'-trimethylbiphenyl** S37
16. **1-Naphthyl-2',4',6'-trimethylbenzene** S39
17. **1-Methoxy-2',4',6'-trimethylbiphenyl** S41
18. **2,4-Dimethyl-2',4',6'-trimethylbiphenyl** S43
19. **2-*p*-Tolylnaphthalene** S45
20. **4-trimethylsilyl-4'-methylbiphenyl** S47

I. General Procedures of Couplings of Aryl Halides with $AlAr_3(THF)$

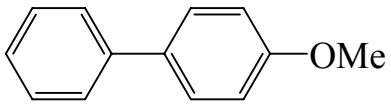
To a solution of $Pd(OAc)_2$ (2.2 mg, 0.010 mmol) and a phosphine ligand (0.020 mmol) in 2 mL solvent at a suitable temperature, $AlAr_3(THF)$ (1.3 mmol) and an aryl halide (1.0 mmol) in 2 mL solvent were added. The mixture was reacted for a reaction time as indicated. The solvent was then removed under reduced pressures. The residue was dissolved in 5 mL CH_2Cl_2 and the resulting solution was passed through a short column of silica gel. The solution was dried and the coupling product was purified by column chromatography with an eluent of EtOAc/hexane. For couplings with the $Al(2,4,6\text{-}Me_3C_6H_2)_3(THF)$ reagent, half quantities of substrates, $Pd(OAc)_2$, PCy_3, and the aluminium reagent were used.

II. Blank Reaction of $Al(2,4,6\text{-}Me_3C_6H_2)_3(THF)$ in the Presence of $Pd(OAc)_2$ and PCy_3

To a solution of $Pd(OAc)_2$ (0.0561 g, 0.250 mmol) and PCy_3 (0.139 g, 0.500 mmol) in 2 mL toluene, $Al(2,4,6\text{-}Me_3C_6H_2)_3(THF)$ (0.286 g, 0.625 mmol) in 2 mL toluene was added under a nitrogen atmosphere at room temperature. The resulting solution was heated at 100 ^oC for 1 h and then the solvent was removed to give a yellow solid. The solid was dissolved in 5 mL CH_2Cl_2 and the mixture was passed through a short column of silica gel. The solution was dried completely and the resulted white solid was purified by column chromatography with an eluent of AcOEt/hexane (1/50) to afford a white solid of coupling product (0.0561 g, 94.3%). ^1H NMR (400 MHz, $CDCl_3$): δ 6.93 (s, 4H, Ar), 2.32 (s, 6H, CH_3), 1.86 (s, 12H, CH_3) ppm.

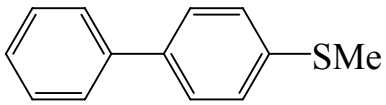
III. ^1H and ^{13}C NMR Spectroscopic Data of Coupling Products

1. **4-Methoxybiphenyl**[1]



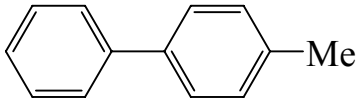
White solid (0.167 g, 91.0%). ^1H NMR (400 MHz, $CDCl_3$): δ 7.54-7.49 (m, 4H, Ar), 7.40-7.36 (m, 2H, Ar), 7.28 (d, 1H, J = 7.6 Hz, Ar), 6.94 (d, 2H, J = 6.8 Hz, Ar), 3.80 (s, 3H, OCH_3) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 159.1, 140.8, 133.7, 128.6, 128.1, 126.7, 126.1, 114.2, 55.2 ppm

2. **4-phenylthioanisole**[2]



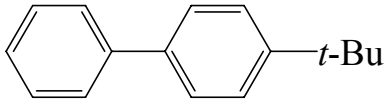
Pale yellow solid (0.186 g, 93.0%). ^1H NMR (400 MHz, $CDCl_3$): δ 7.57-7.50 (m, 4H, Ar), 7.44-7.40 (m, 2H, Ar), 7.34-7.31 (m, 3H, Ar), 2.51 (s, 3H, SCH_3) ppm. $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 140.5, 138.0, 137.6, 128.8, 127.5, 127.2, 127.0, 126.8, 15.9 ppm

3. **4-Methybiphenyl**[3]



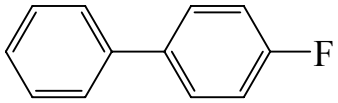
White solid (0.139 g, 83.0%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.55 (d, 2H, *J* = 8.0 Hz, Ar), 7.47 (d, 2H, *J* = 8.0 Hz, Ar), 7.38 (t, 2H, *J* = 7.6 Hz, Ar), 7.29 (t, 1H, *J* = 7.6 Hz, Ar), 7.21 (d, 2H, *J* = 7.6 Hz, Ar), 2.36 (s, 3H, CH_3) ppm. ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 141.1, 138.3, 136.9, 129.4, 128.7, 127.2, 127.1, 126.9, 21.0 ppm.

4. **4-*tert*-Butylbiphenyl**[4]



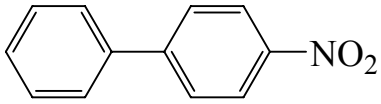
White solid (0.199 g, 95%); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.57 (d, 2H, *J* = 7.6 Hz, Ar), 7.52 (d, *J* = 8.4 Hz, Ar), 7.44 (d, 2H, *J* = 8.4 Hz, Ar), 7.39 (t, 2H, *J* = 8.0 Hz, Ar), 7.29 (t, 1H, *J* = 6.8 Hz, Ar), 1.34 (s, 9H, $C(CH_3)_3$) ppm. ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 150.1, 141.1, 138.3, 128.7, 127.1, 127.0, 126.8, 125.7, 34.5, 31.4 ppm.

5. **4-Fluorobiphenyl**[5]



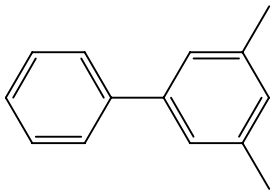
White solid (0.156 g, 91%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.56-7.52 (m, 4H, Ar), 7.46-7.41 (m, 2H, Ar), 7.36-7.32 (m, 1H, Ar), 7.14-7.05 (m, 2H, Ar) ppm. ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 163.7, 161.2, 140.2, 137.3, 128.8, 128.7, 127.2, 127.1, 127.0, 115.5 (d, *J* = 21.3 Hz) ppm.

6. **4-Nitrobiphenyl**[1]



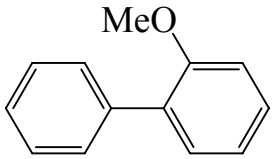
Pale yellow solid (0.129 g, 65%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.28 (d, 2H, *J* = 8.0 Hz, Ar), 7.73 (d, 2H, *J* = 8.0 Hz, Ar), 7.62 (d, 2H, *J* = 7.6 Hz, Ar), 7.52-7.42 (m, 3H, Ar) ppm ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 147.6, 147.1, 138.7, 129.1, 128.9, 127.7, 127,3, 124.0 ppm.

7. **3,5-Dimethylbiphenyl**[6]



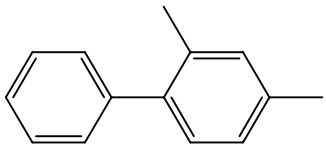
Colorless liquid (0.172 g, 95%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.53 (d, 2H, *J* = 7.2 Hz, Ar), 7.36 (t, 2H, *J* = 7.2 Hz, Ar), 7.32-7.24 (m, 1H, Ar), 7.18 (s, 2H, Ar), 6.94 (s, 1H, Ar), 2.33 (s, 6H, CH_3) ppm. ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 141.4, 141.2, 138.1, 128.8, 128.7, 127.1, 127.0, 125.1, 21.3 ppm.

8. **2-Methoxybiphenyl**[7]



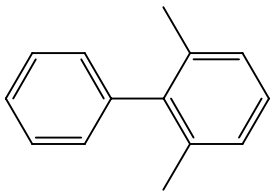
White solid (0.169 g, 92.0%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.52 (d, 2H, *J* = 6.8 Hz, Ar), 7.39 (t, 2H, *J* = 7.6 Hz, Ar), 7.30 (t, 3H, *J* = 7.6 Hz, Ar), 7.10 (t, 1H, *J* = 7.6 Hz, Ar), 6.95 (d, 1H, *J* = 7.6 Hz, Ar), 3.77 (s, 3H, OCH_3) ppm. ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 156.4, 138.5, 130.8, 130.7, 129.5, 128.6, 127.9, 126.8, 120.8, 111.3, 55.5 ppm.

9. **2,4-Dimethylbiphenyl**



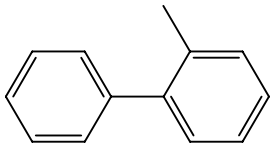
Colorless liquid (0.171 g, 94.0%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.42-7.25 (m, 5H, Ar), 7.13-7.00 (m, 3H, Ar), 2.33 (s, 3H, CH_3), 2.22 (s, 3H, CH_3) ppm. ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 141.9, 139.1, 136.8, 135.0, 131.0, 129.7, 129.2, 128.7, 128.0, 127.1, 126.5, 20.9, 20.3 ppm.

10. **2,6-Dimethylbiphenyl**[8]



Colorless liquid (0.127 g, 70.0%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.40 (t, 2H, *J* = 7.6 Hz, Ar), 7.34-7.28 (m, 1H, Ar), 7.15-7.08 (m, 5H, Ar), 2.02 (s, 6H, CH_3) ppm. ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 141.9, 141.1, 136.0, 129.0, 128.7, 128.4, 127.2, 126.9, 20.7 ppm.

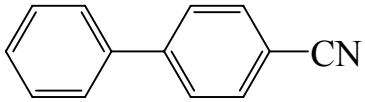
11. **2-Methybiphenyl**[3]



Colorless liquid (0.158 g, 94.0%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.64-7.56 (m, 2H, Ar), 7.45-7.38 (m, 3H, Ar), 7.38-7.30 (m, 2H, Ar), 7.27-7.22 (m, 2H, Ar), 2.27 (s, 3H, CH_3) ppm.

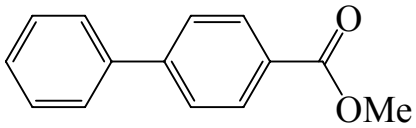
$^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$): δ 142.0, 141.3, 135.3, 130.3, 129.8, 129.1, 128.8, 127.2, 126.7, 125.7, 20.4 ppm

12. **4-Cyanobiphenyl**[1]



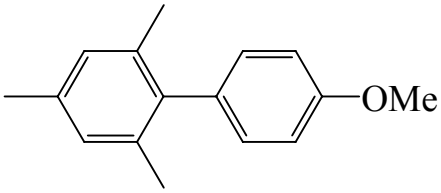
White solid (0.166 g, 93.0%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.70 (dd, 4H, J = 18.0, 8.0 Hz, Ar), 7.63-7.57 (m, 2H, Ar), 7.50-7.40 (m, 3H, Ar) ppm. $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$): δ 145.7, 139.2, 132.6, 129.1, 128.6, 127.7, 127.2, 118.9, 110.9 ppm.

13. **4-Phenyl-methylbenzoate**[1]



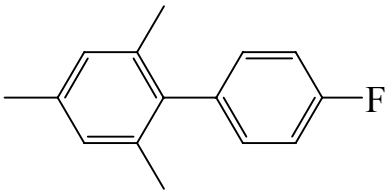
White solid (0.197 g, 93.0%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.10 (d, 2H, *J* = 8.4 Hz, Ar), 7.68-7.48 (m, 4H, Ar), 7.45 (t, 2H, *J* = 8.4 Hz, Ar), 7.38 (t, 1H, *J* = 7.6 Hz, Ar), 3.92 (s, 3H, CH_3) ppm. $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$): δ 166.9, 145.6, 140.0, 130.1, 128.9, 128.1, 127.2, 127.0, 52.0 ppm.

14. **4-Methoxy-2',4',6'-trimethylbiphenyl**[9]



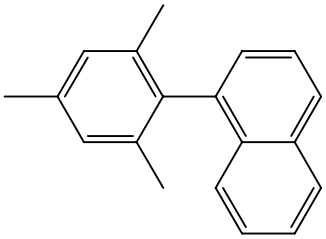
White solid (0.107 g, 95.0 %). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.06-7.03 (m, 2H, Ar), 6.96-6.93 (m, 4H, Ar), 3.85 (s, 3H, OCH_3), 2.32 (s, 3H, CH_3), 2.01 (s, 6H, CH_3) ppm. $^{13}C\{^{1}H\}$ NMR (100 MHz): δ 156.7, 136.5, 135.2, 130.9, 129.5, 128.3, 127.9, 120.6, 110.8, 55.4, 21.1, 20.3 ppm.

15. **4-Fluoro-2',4',6'-trimethylbiphenyl**



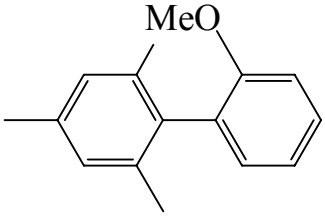
White solid (0.101 g, 94.0%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.10-7.08 (m, 4H, Ar), 6.93 (s, 2H, Ar), 2.32 (s, 3H, CH_3), 1.98 (s, 6H, CH_3) ppm. $^{13}C\{^{1}H\}$ NMR (100 MHz): δ 162.9, 160.5, 138.0, 136.9, 136.7, 136.1, 130.8, 130.7, 128.1, 115.4, 115.2, 20.9, 20.6 ppm.

16. **1-Naphthyl-2',4',6'-trimethylbenzene**[1]



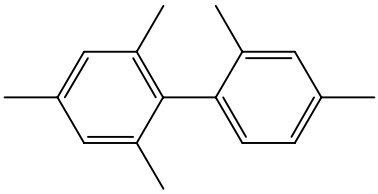
White solid (0.124 g, 92.0%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.86 (dd, 2H, 19.6, 4.0 Hz, Ar), 7.52 (dd, 1H, *J* = 8.4, 7.2 Hz, Ar), 7.47-7.43 (m, 1H, Ar), 7.36-7.31 (m, 2H, Ar), 7.26-7.23 (m, 1H, Ar), 7.00 (s, 2H, Ar), 2.38 (s, 3H, CH_3), 1.87 (s, 6H, CH_3) ppm. ^{13}C{^{1}H} NMR (100 MHz): δ 138.8, 136.9, 136.8, 133.8, 132.0, 128.3, 128.1, 127.0, 126.7, 126.0, 125.7, 125.6, 125.5, 21.1, 20.3 ppm.

17. **1-Methoxy-2',4',6'-trimethylbiphenyl**[10]



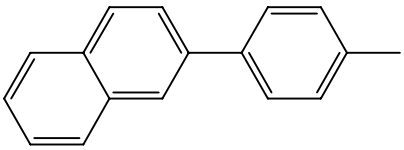
White solid (0.108 g, 96.0%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34-7.30 (m, 1H, Ar), 7.03-6.93 (m, 5H, Ar), 3.73 (s, 3H, OCH_3), 2.32 (s, 3H, CH_3), 1.98 (s, 6H, CH_3) ppm. ^{13}C{^{1}H} NMR (100 MHz): δ 156.7, 136.6, 136.4, 135.3, 130.9, 129.5, 128.3, 127.9, 120.6, 110.8, 55.3, 21.1, 20.3 ppm.

18. **2,4-Dimethyl-2',4',6'-trimethylbiphenyl**



White solid (0.103 g, 92.0%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.09 (s, 1H, Ar), 7.04 (d, 1H, *J* = 7.6 Hz, Ar), 6.92 (s, 2H, Ar), 6.88 (d, 1H, *J* = 7.6 Hz, Ar), 2.36 (s, 3H, CH_3), 2.33 (s, 3H, CH_3), 1.93 (s, 3H, CH_3), 1.91 (s, 6H, CH_3) ppm. ^{13}C{^{1}H} NMR (100 MHz): δ 138.2, 137.5, 136.3, 136.2, 135.9, 135.6, 130.6, 129.0, 128.2, 127.9, 126.7, 21.1, 21.0, 20.3, 19.4 ppm.

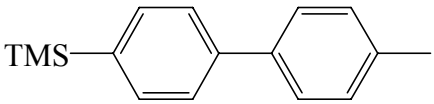
19. **2-*p*-Tolylnaphthalene**[11]



White solid (0.101 g, 93.0%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.02 (s, 1H, Ar), 7.91-7.84

(m, 3H, Ar), 7.75-7.73 (m, 1H, Ar), 7.64-7.62 (m, 2H, Ar), 7.52-7.44 (m, 2H, Ar), 7.30 (d, 2H, J = 8.4 Hz, Ar), 2.42 (s, 3H, CH_3) ppm. $^{13}C\{^1H\}$ NMR (100 MHz): δ 138.50, 138.24, 137.14, 133.74, 132.52, 129.57, 128.33, 128.13, 127.62, 127.24, 126.20, 125.74, 125.54, 125.41, 21.10 ppm.

20. **4-trimethylsilyl-4'-methylbiphenyl**[12]



White solid (0.102 g, 85.0%). ^1H NMR (400 MHz, $CDCl_3$): δ 7.70-7.68 (m, 4H, Ar), 7.60 (d, 2H, J = 8.0 Hz, Ar), 7.34 (d, 2H, J = 8.0 Hz, Ar), 2.49 (s, 3H, CH_3), 0.41 (s, 9H, CH_3) ppm. $^{13}C\{^1H\}$ NMR (100 MHz): δ 141.55, 138.81, 138.29, 137.05, 133.78, 129.48, 126.99, 126.31, 21.08, -1.08 ppm.

IV. References

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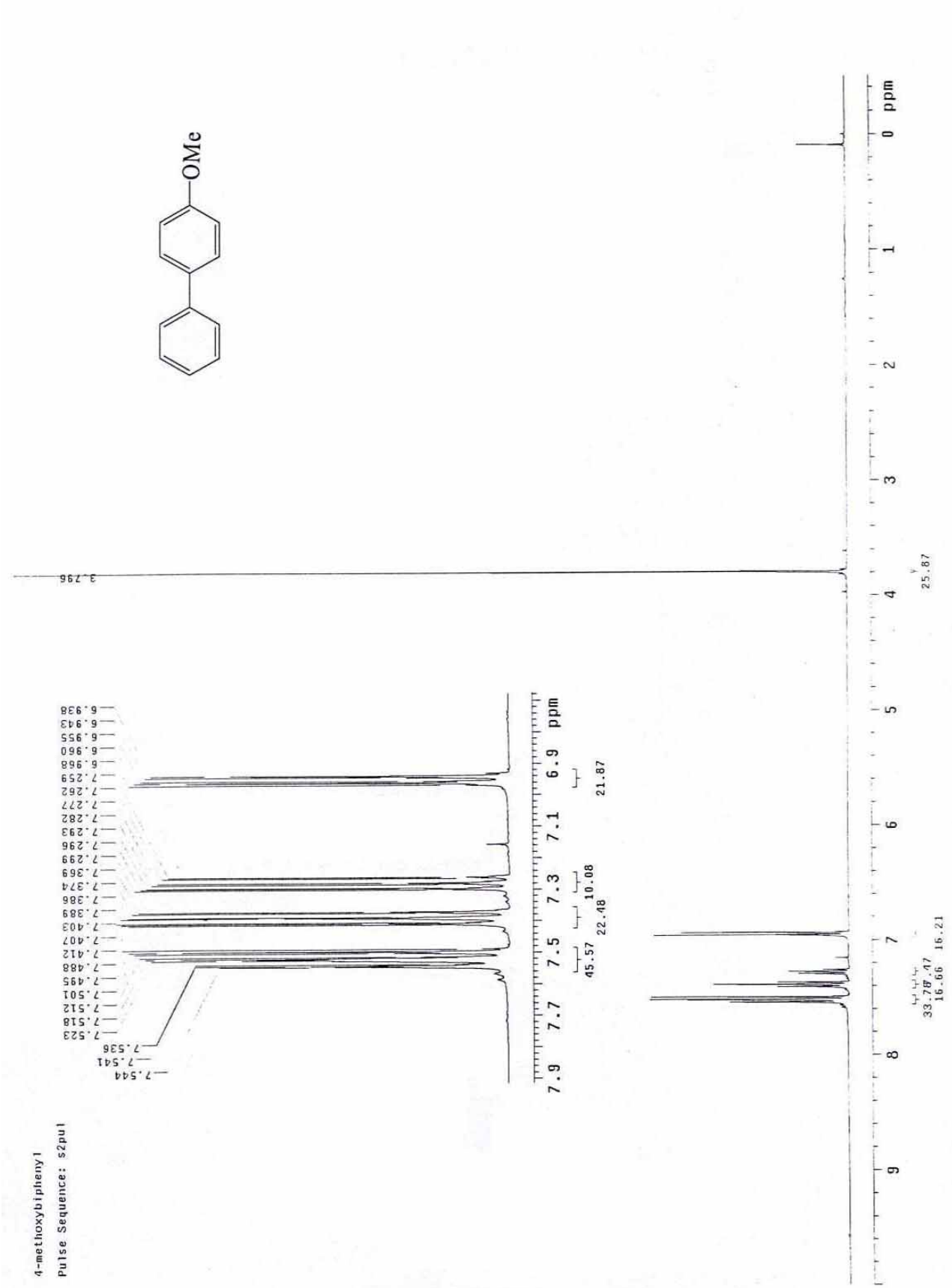
V. ^1H and ^{13}C NMR Spectra of Coupling Products


OMe
4-methoxybiphenyl
Pulse Sequence: s2pul
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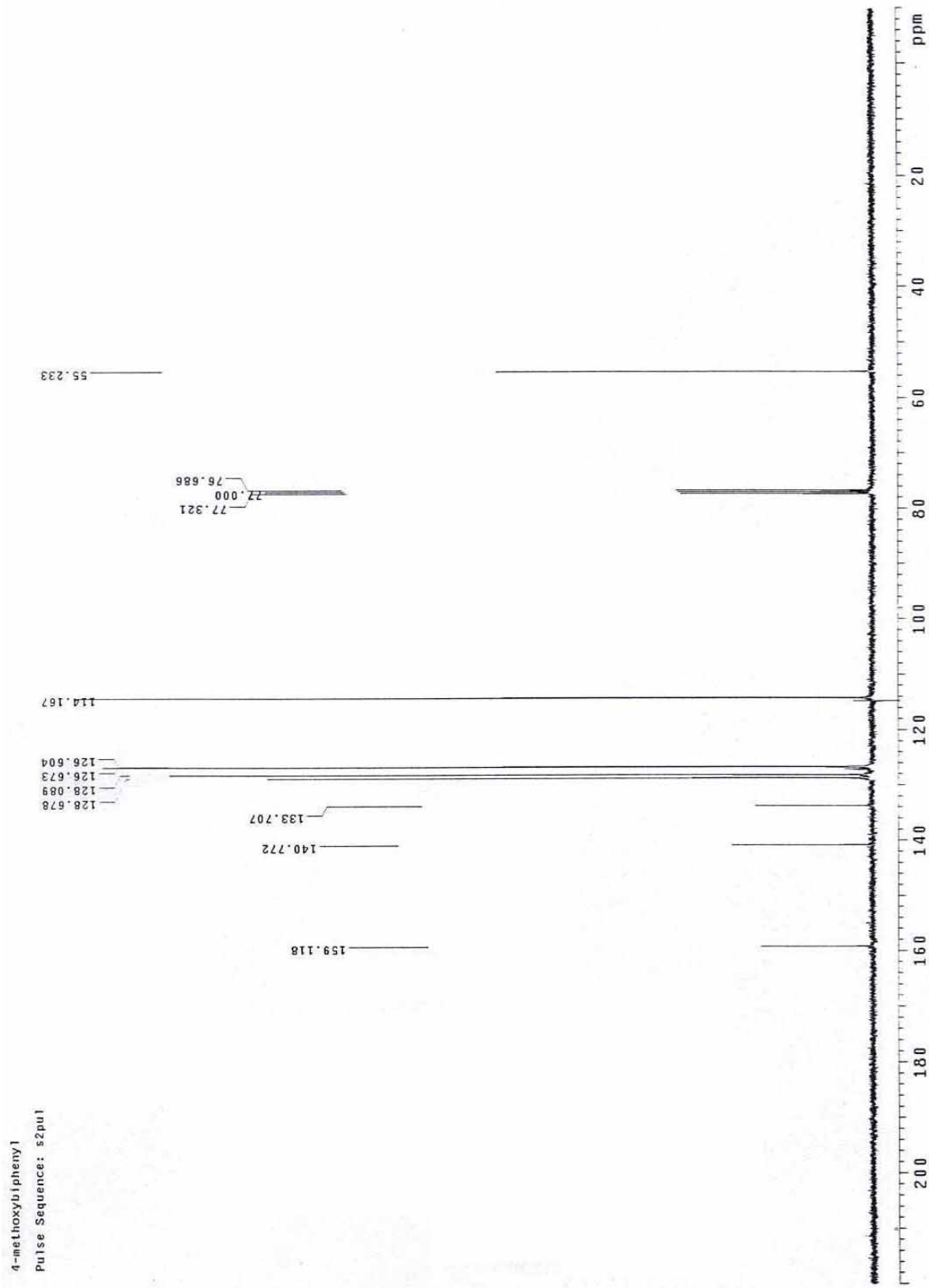
7.541
7.538
7.523
7.518
7.512
7.501
7.495
7.488
7.412
7.407
7.403
7.389
7.386
7.374
7.369
7.299
7.296
7.293
7.282
7.277
7.262
7.259
6.968
6.960
6.955
6.943
6.938
3.796
7.9
7.7
7.5
7.3
7.1
6.9
ppm
45.57
22.48
10.08
21.87
25.87
33.78
16.66
16.21
0
1
2
3
4
5
6
7
8
9
ppm


4-methoxybiphenyl
Pulse Sequence: s2pul
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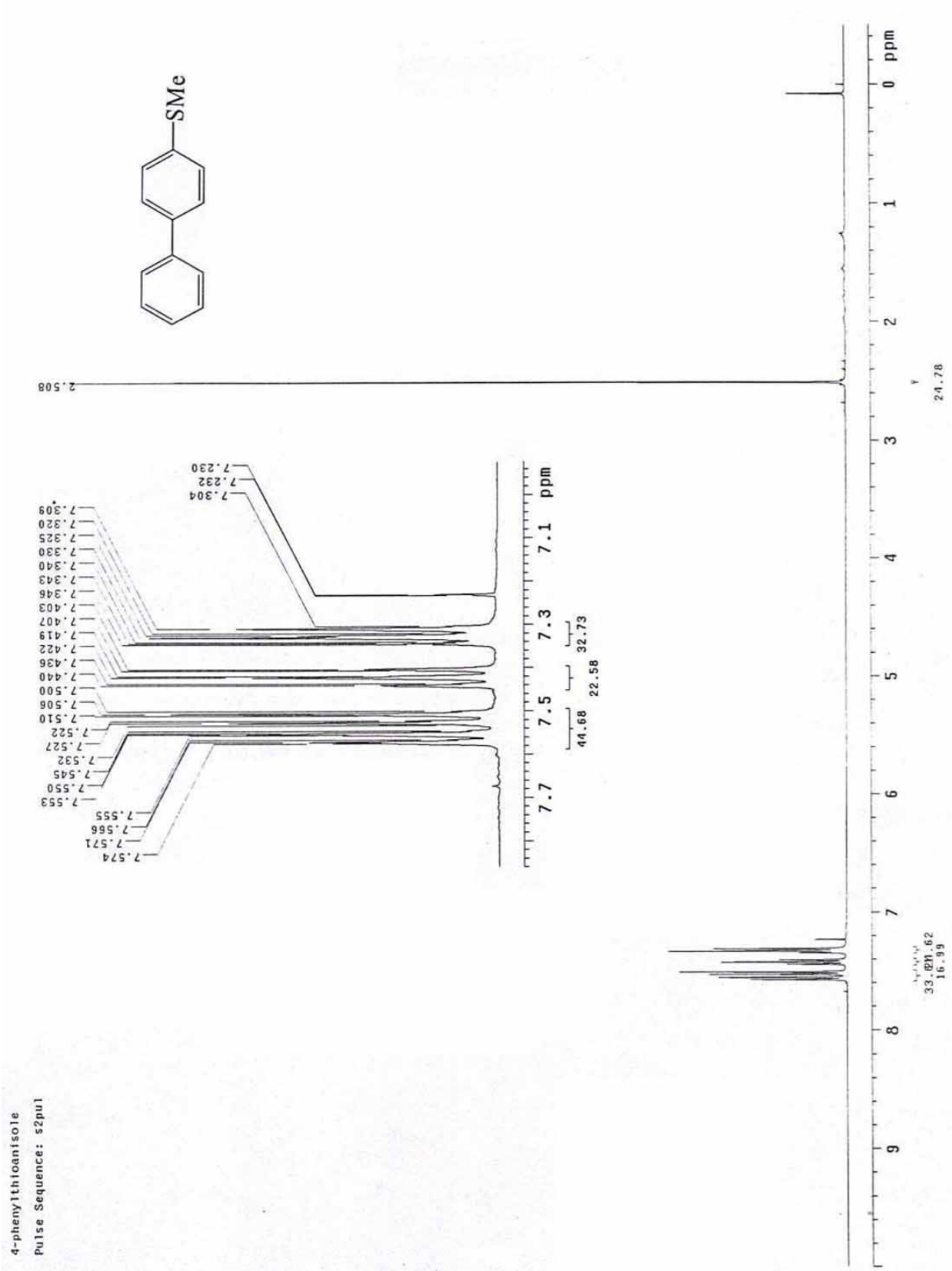
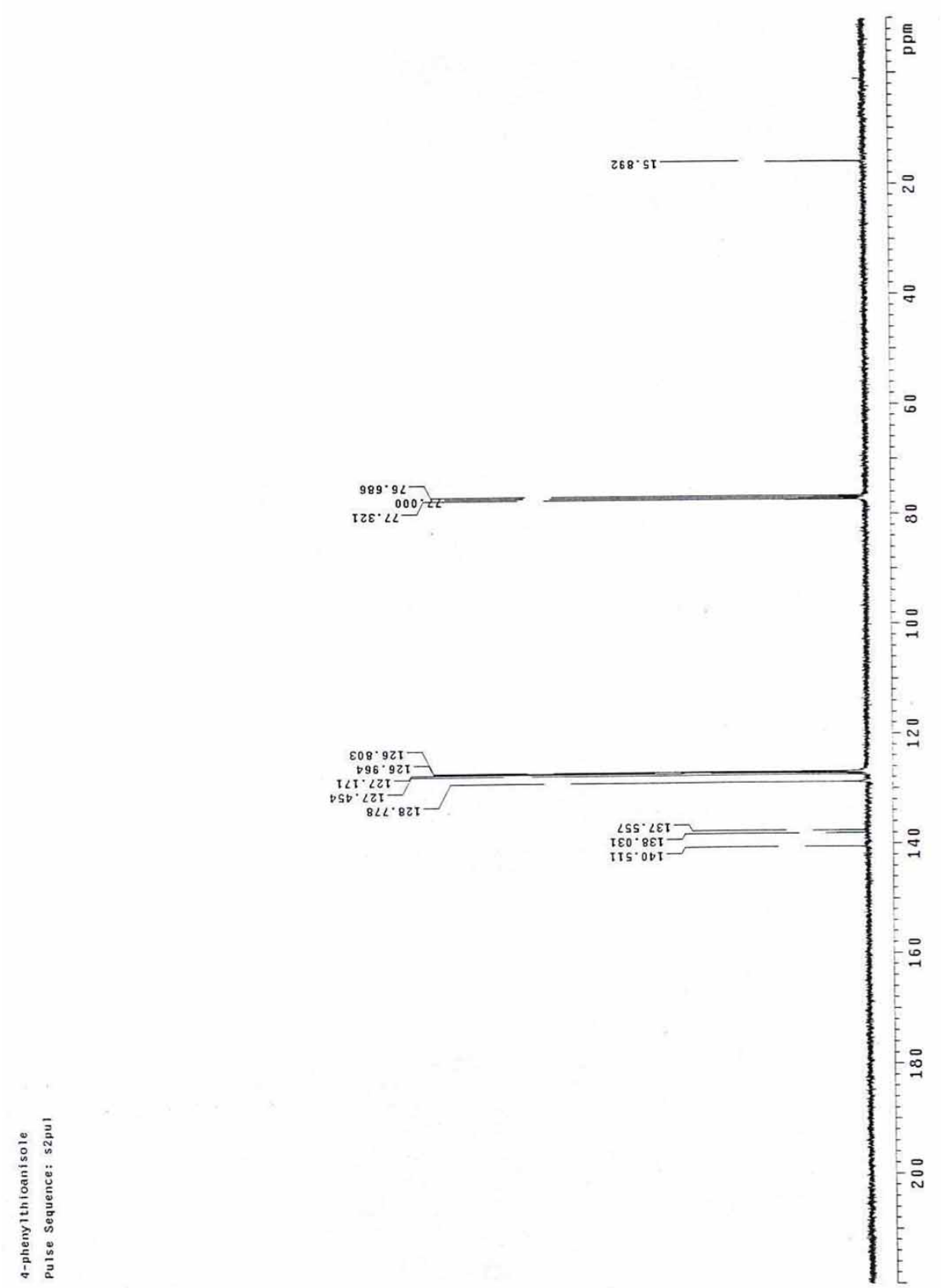
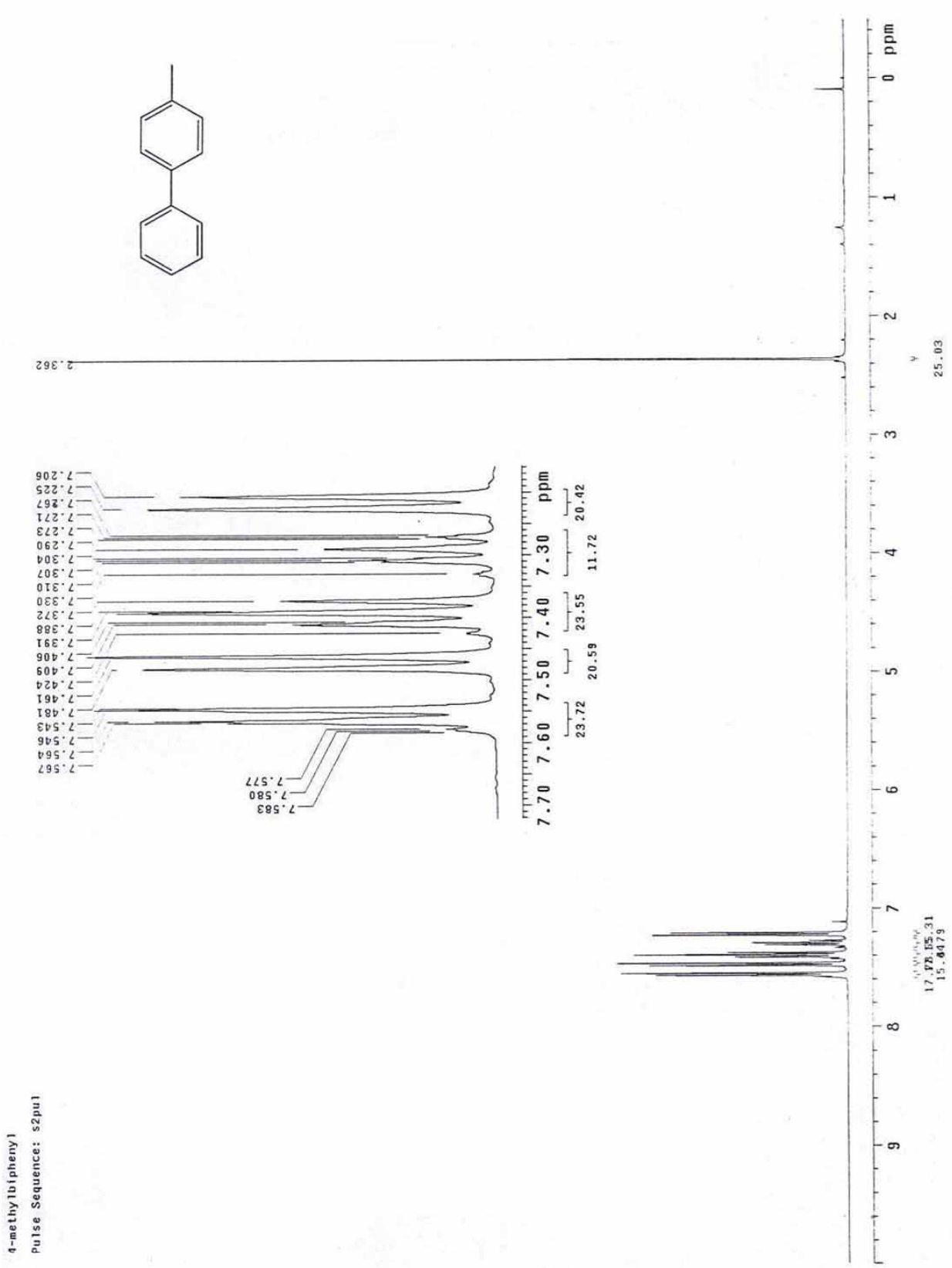
140.772
133.707
128.678
128.089
126.673
126.604
114.167
77.321
77.000
76.686
55.233
ppm
200
180
160
140
120
100
80
60
40
20


4-phenylthioanisole
Pulse Sequence: s2pul
SMe
2.508
7.574
7.571
7.566
7.555
7.553
7.550
7.545
7.532
7.527
7.522
7.510
7.506
7.500
7.440
7.436
7.422
7.419
7.407
7.403
7.346
7.343
7.340
7.330
7.325
7.320
7.309
7.304
7.232
7.230
7.7
7.5
7.3
7.1
ppm
44.68
22.58
32.73
33.80
16.99
24.78
0
1
2
3
4
5
6
7
8
9


4-phenylthioanisole
Pulse Sequence: s2pul
ppm
200
180
160
140
120
100
80
60
40
20
15.892
77.321
77.000
76.686
128.778
127.454
127.171
126.964
126.803
137.557
138.031
140.511


4-methylbiphenyl
Pulse Sequence: s2pul
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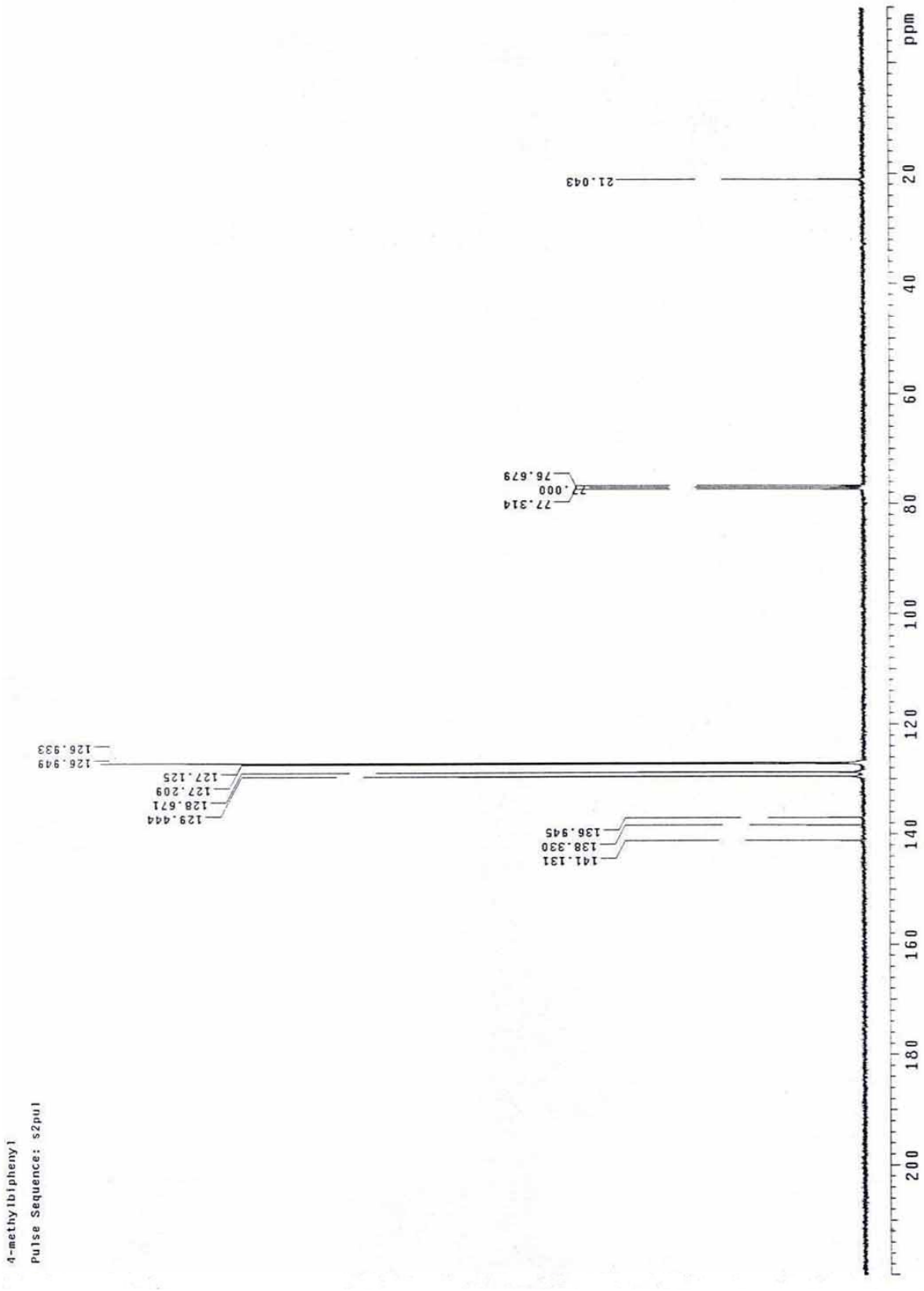
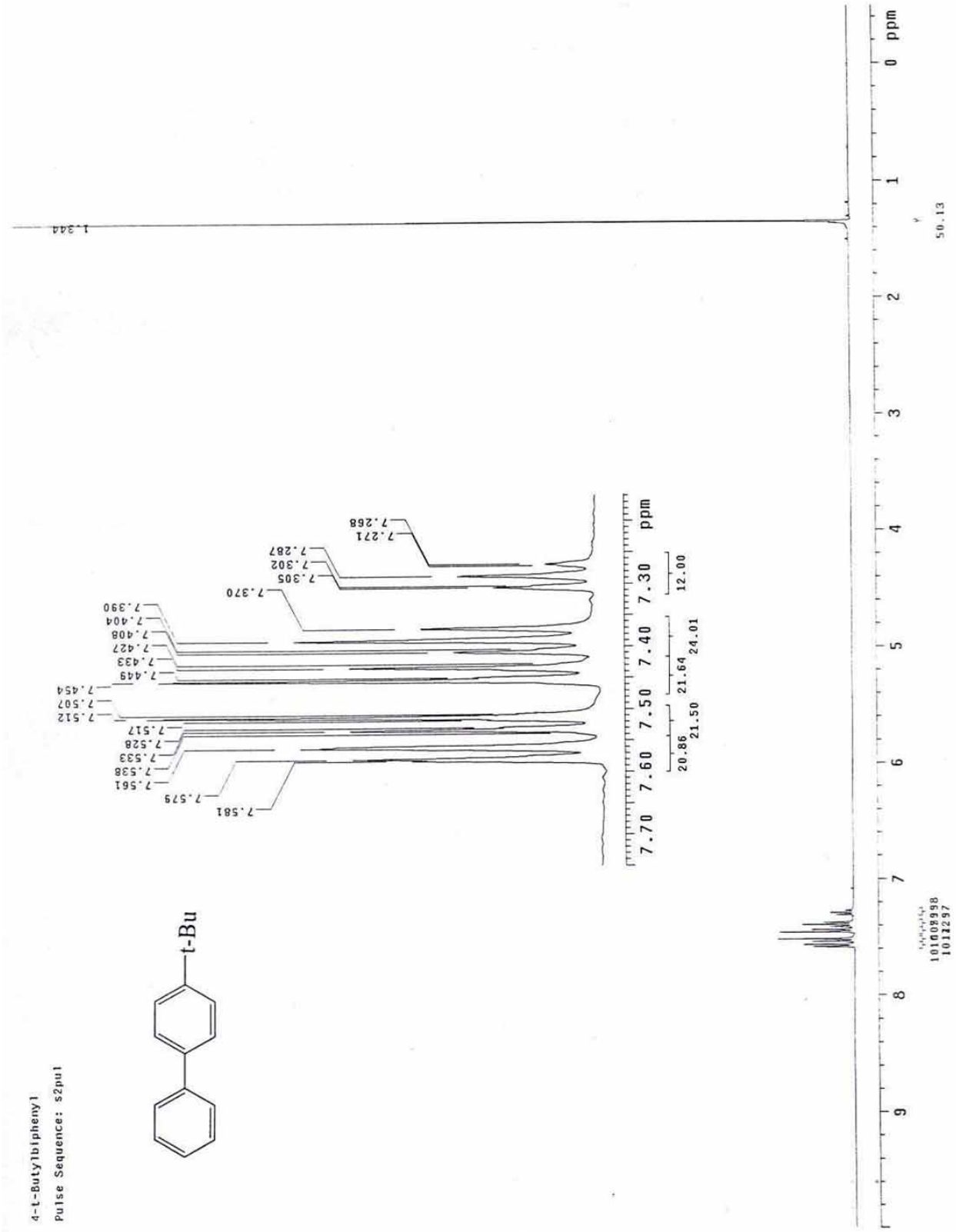
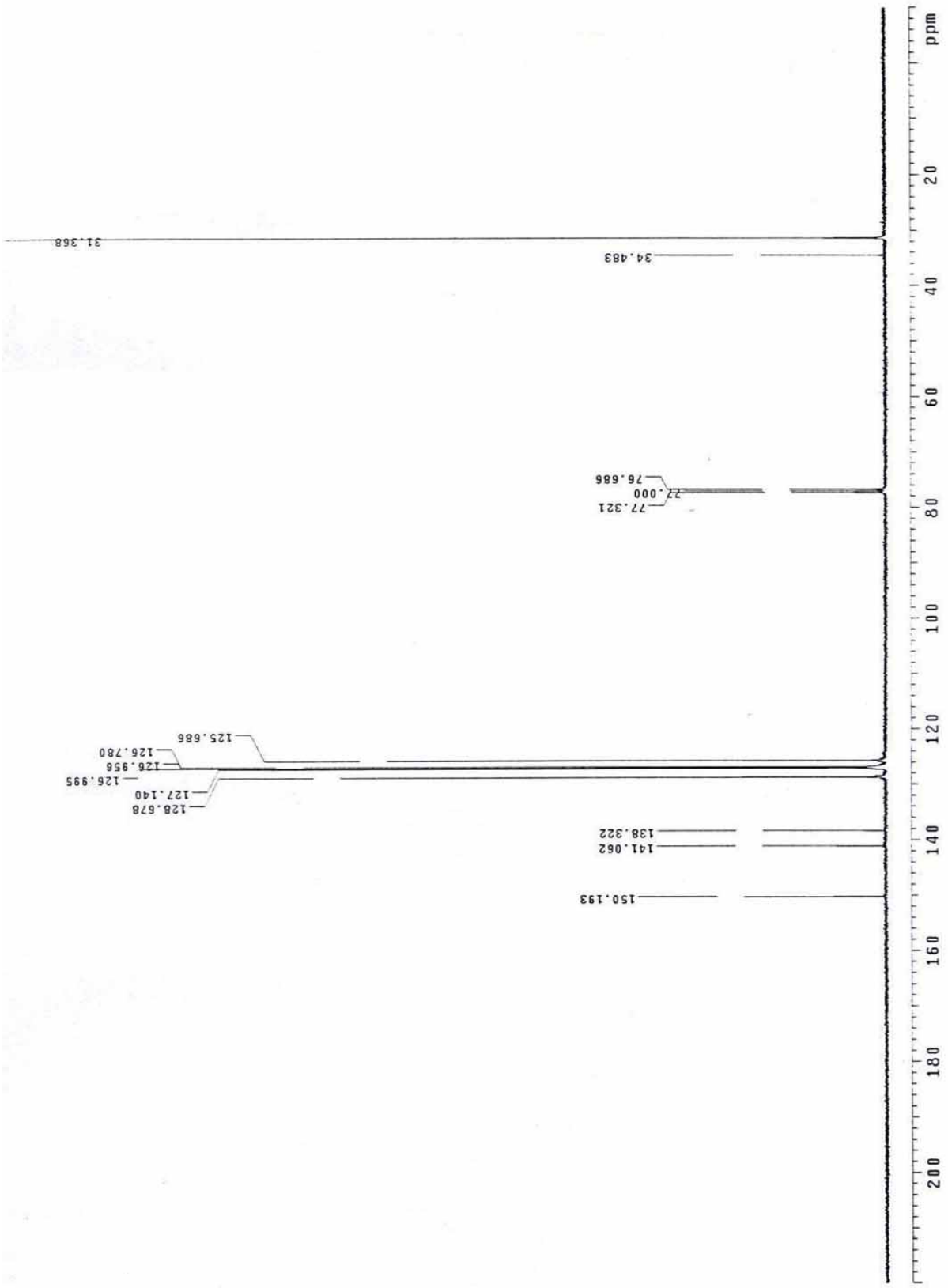
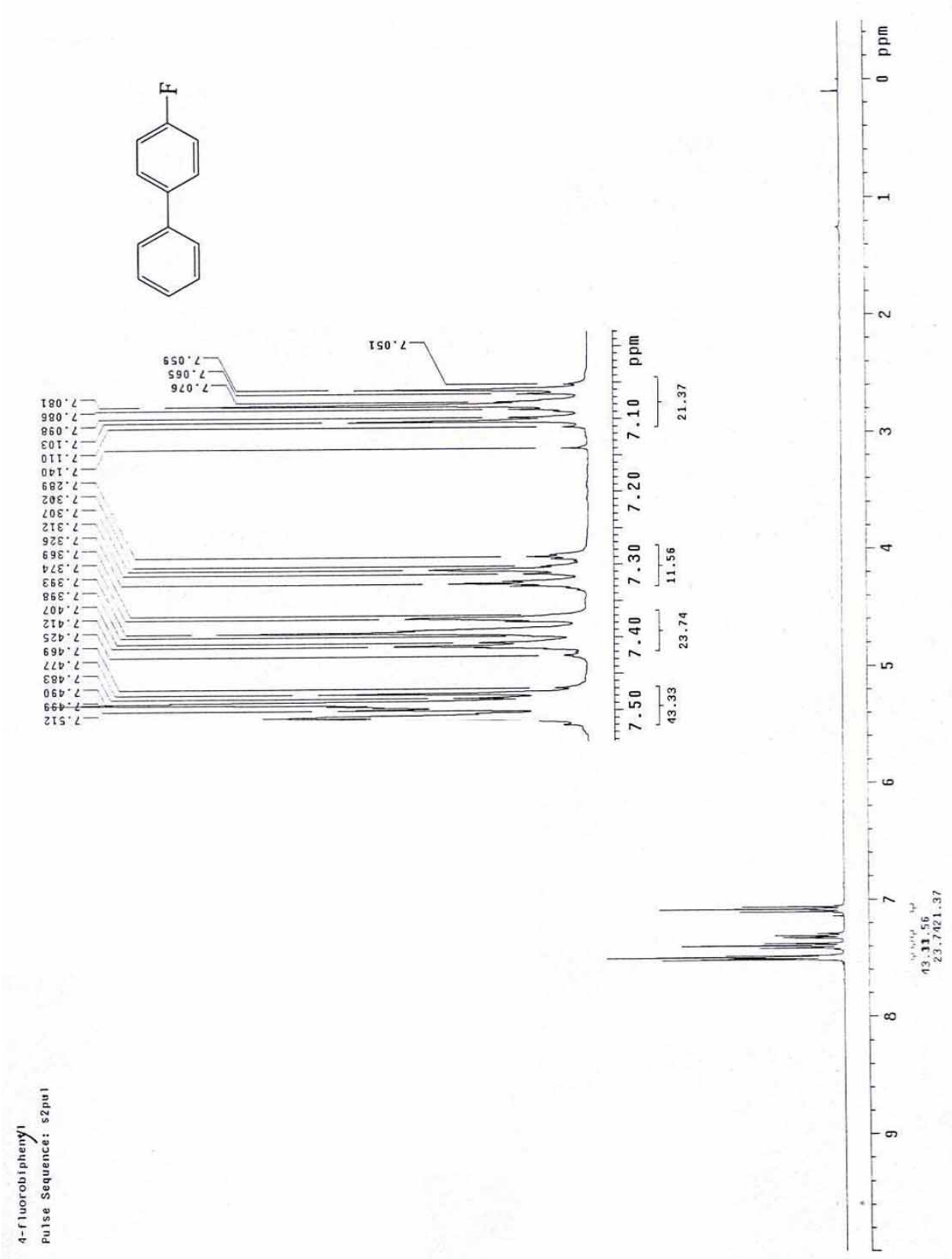
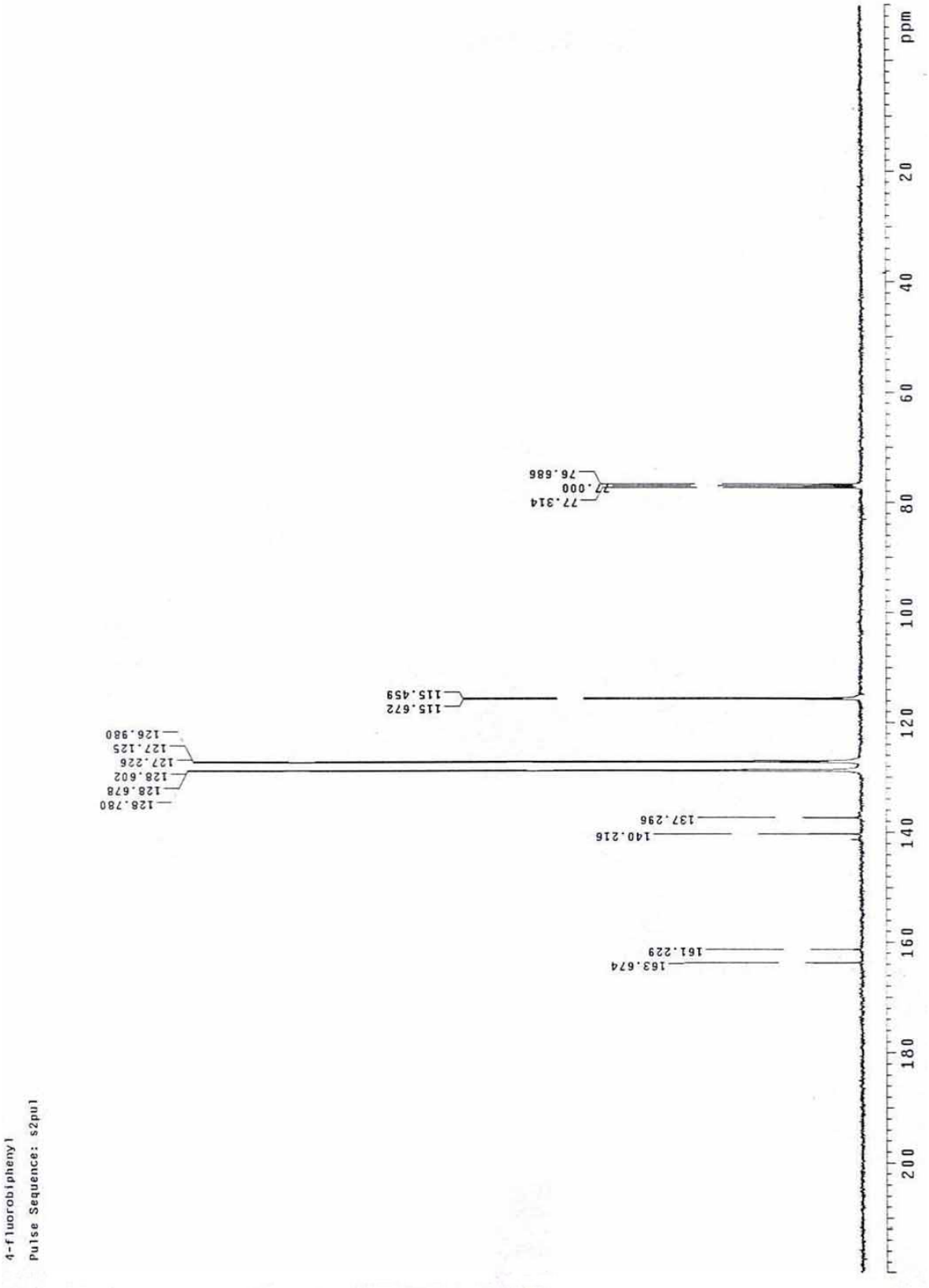
7.583
7.580
7.577
7.567
7.564
7.546
7.543
7.481
7.461
7.424
7.409
7.406
7.391
7.388
7.372
7.330
7.310
7.307
7.304
7.290
7.273
7.271
7.267
7.225
7.206
7.70 7.60 7.50 7.40 7.30 ppm
23.72
20.59
23.55
11.72
20.42
0 ppm
25.03
17.97
15.0479

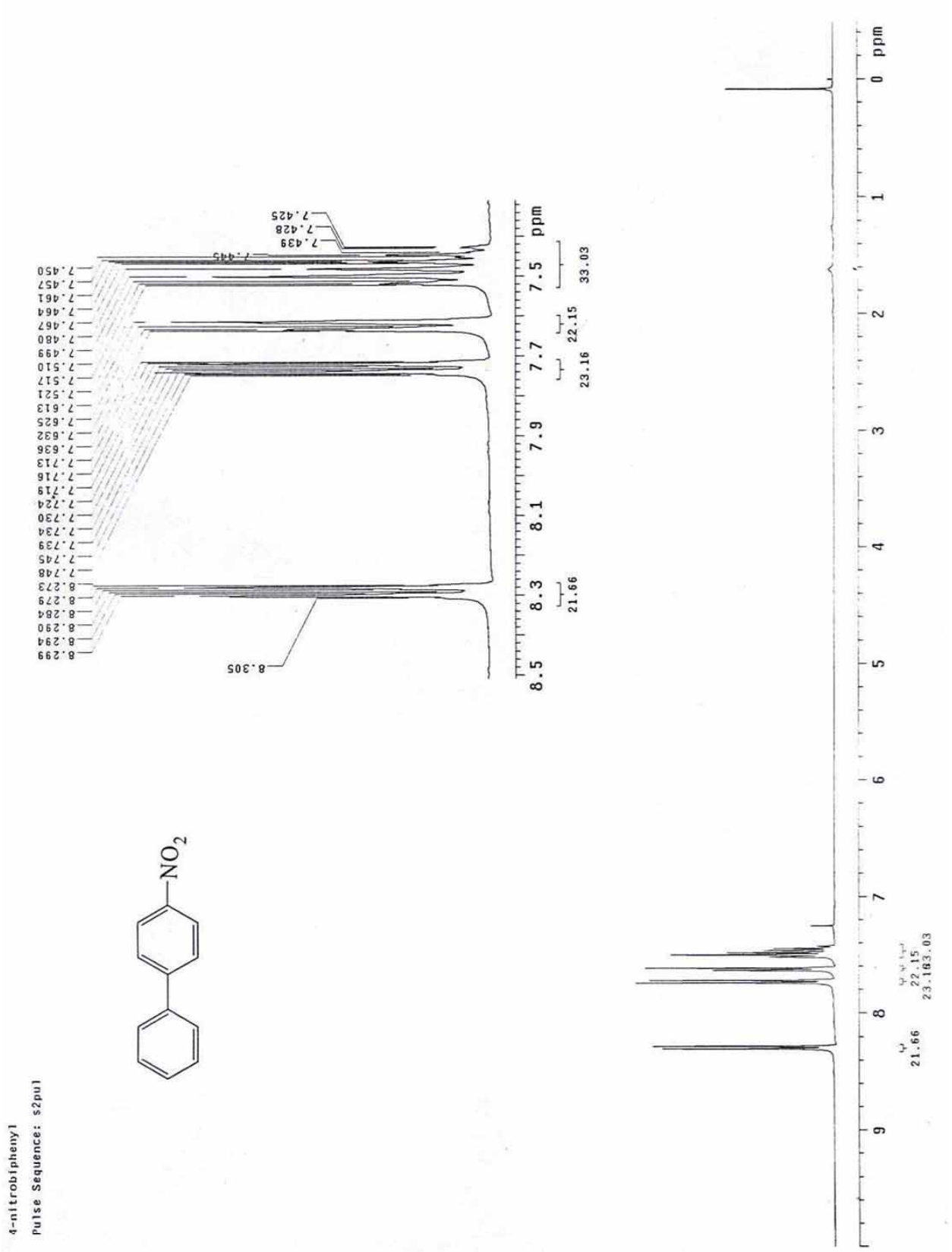

4-methylbiphenyl
Pulse Sequence: s2pul
ppm
20
40
60
80
100
120
140
160
180
200
21.043
77.314
77.000
76.679
126.933
126.949
127.125
127.209
128.671
129.444
136.945
138.330
141.131

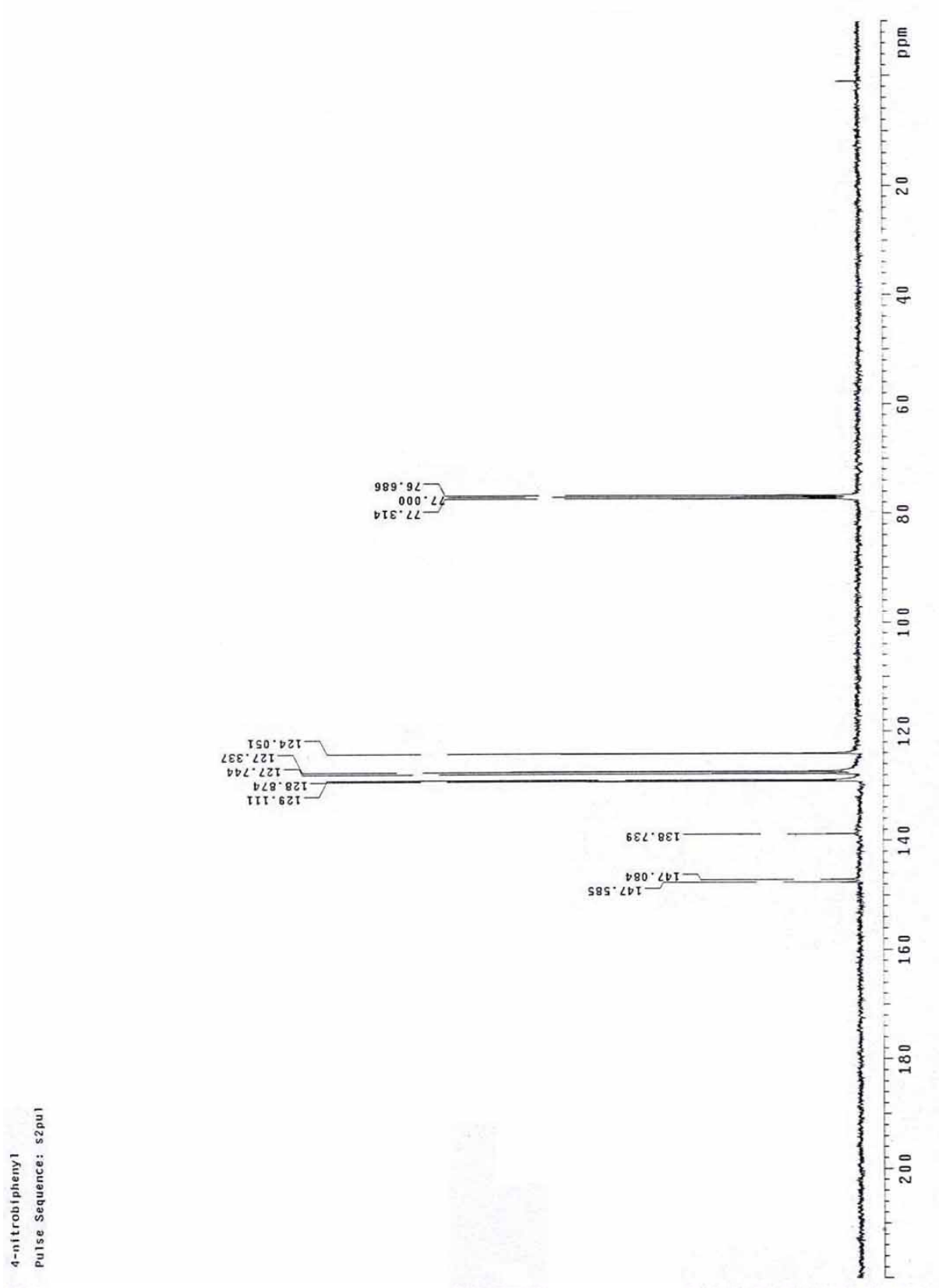

4-t-Butylbiphenyl
Pulse Sequence: s2pul
t-Bu
7.581
7.579
7.561
7.538
7.533
7.528
7.517
7.512
7.507
7.454
7.449
7.433
7.427
7.408
7.404
7.390
7.370
7.305
7.302
7.287
7.271
7.268
7.70 7.60 7.50 7.40 7.30 ppm
20.86
21.50
21.64
24.01
12.00
1.344
50.13
0 ppm

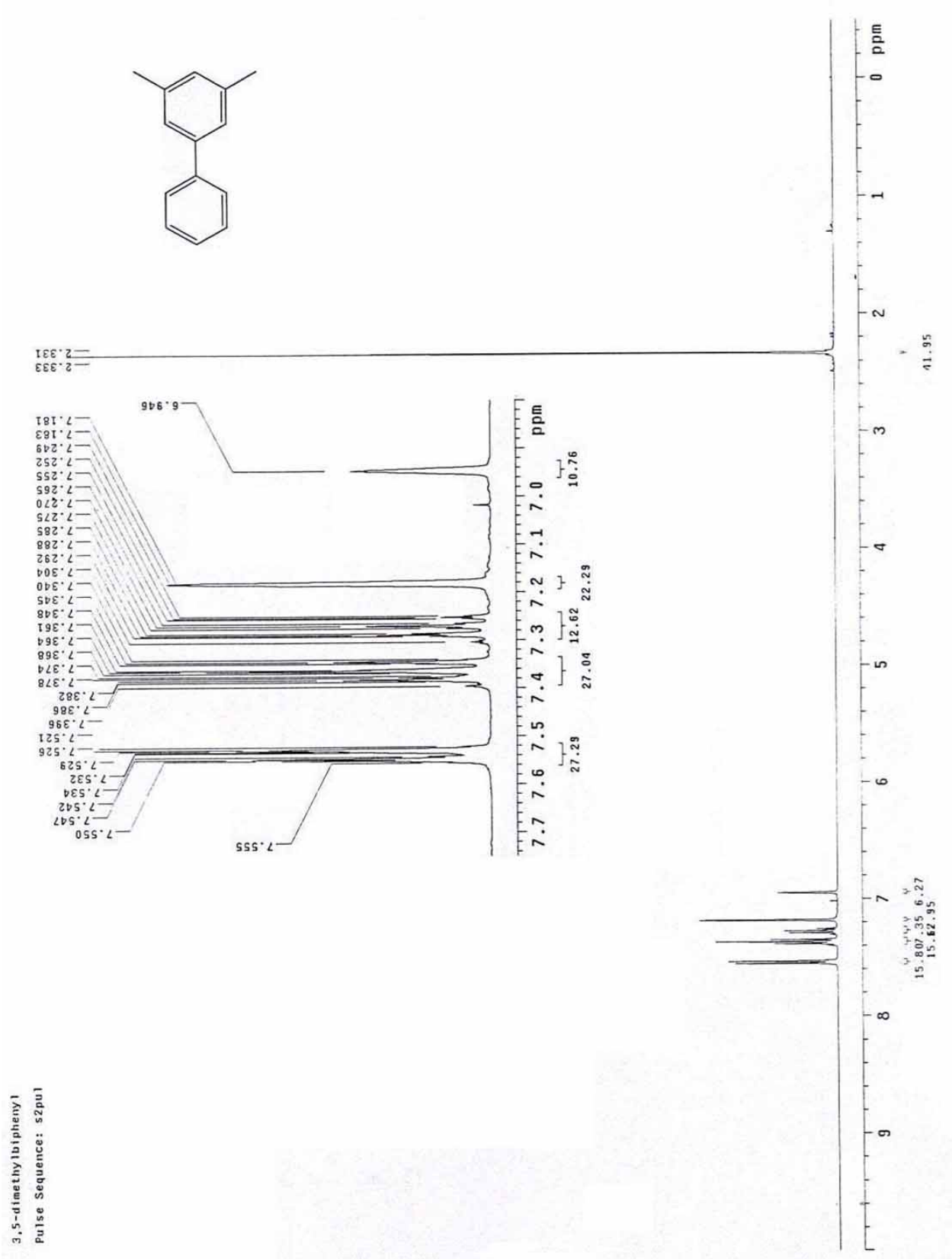

150.193
141.062
138.322
128.678
127.140
126.995
126.956
126.780
125.686
77.321
77.000
76.686
34.483
31.368
ppm
200
180
160
140
120
100
80
60
40
20

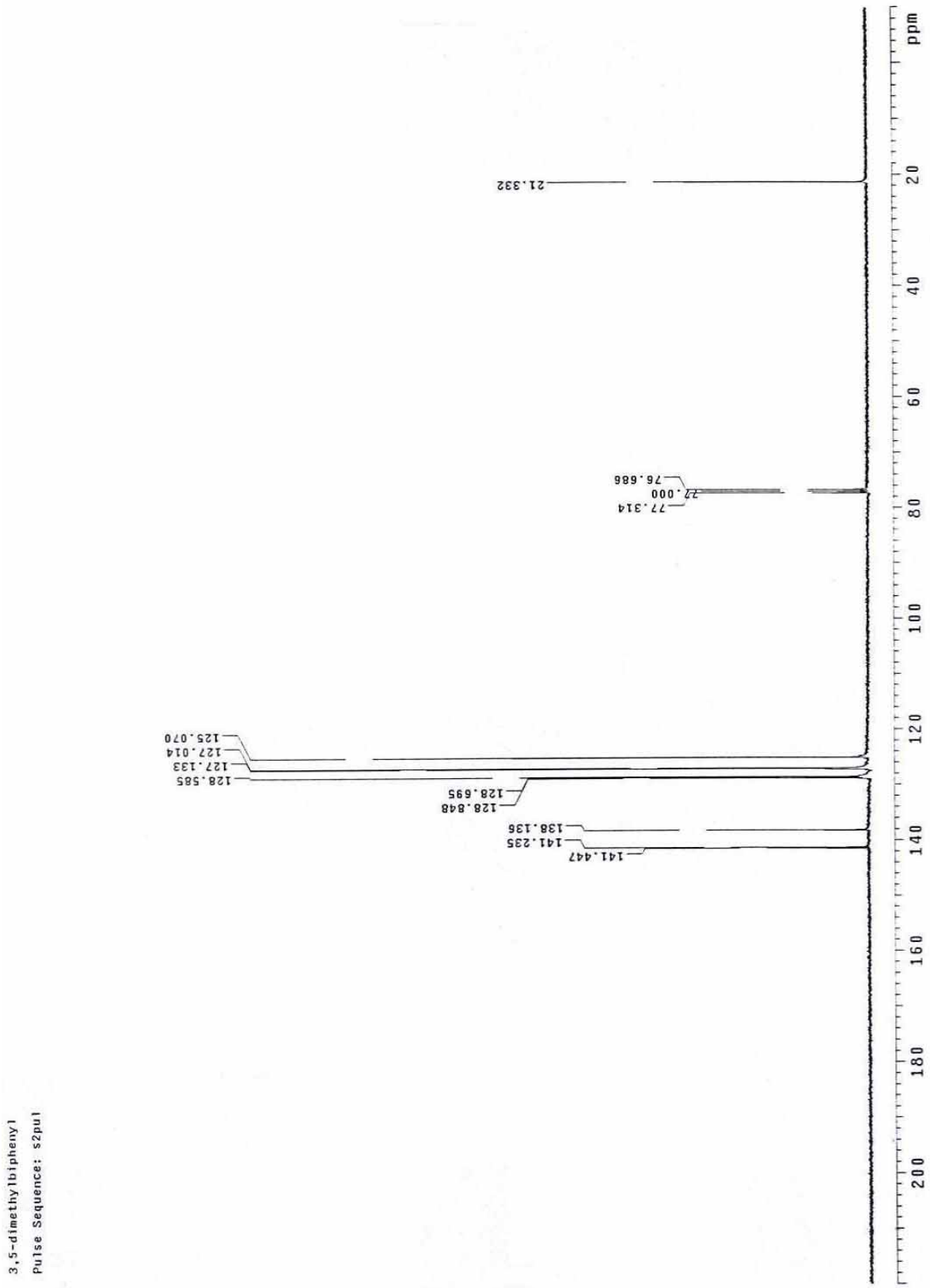

4-fluorobiphenyl
Pulse Sequence: s2pul
F
7.512
7.499
7.490
7.483
7.477
7.469
7.425
7.412
7.407
7.398
7.393
7.374
7.369
7.326
7.312
7.307
7.302
7.289
7.140
7.110
7.103
7.098
7.086
7.081
7.076
7.065
7.059
7.051
7.50
7.40
7.30
7.20
7.10
ppm
43.33
23.74
11.56
21.37
0
1
2
3
4
5
6
7
8
9
ppm
43.56
23.7421.37

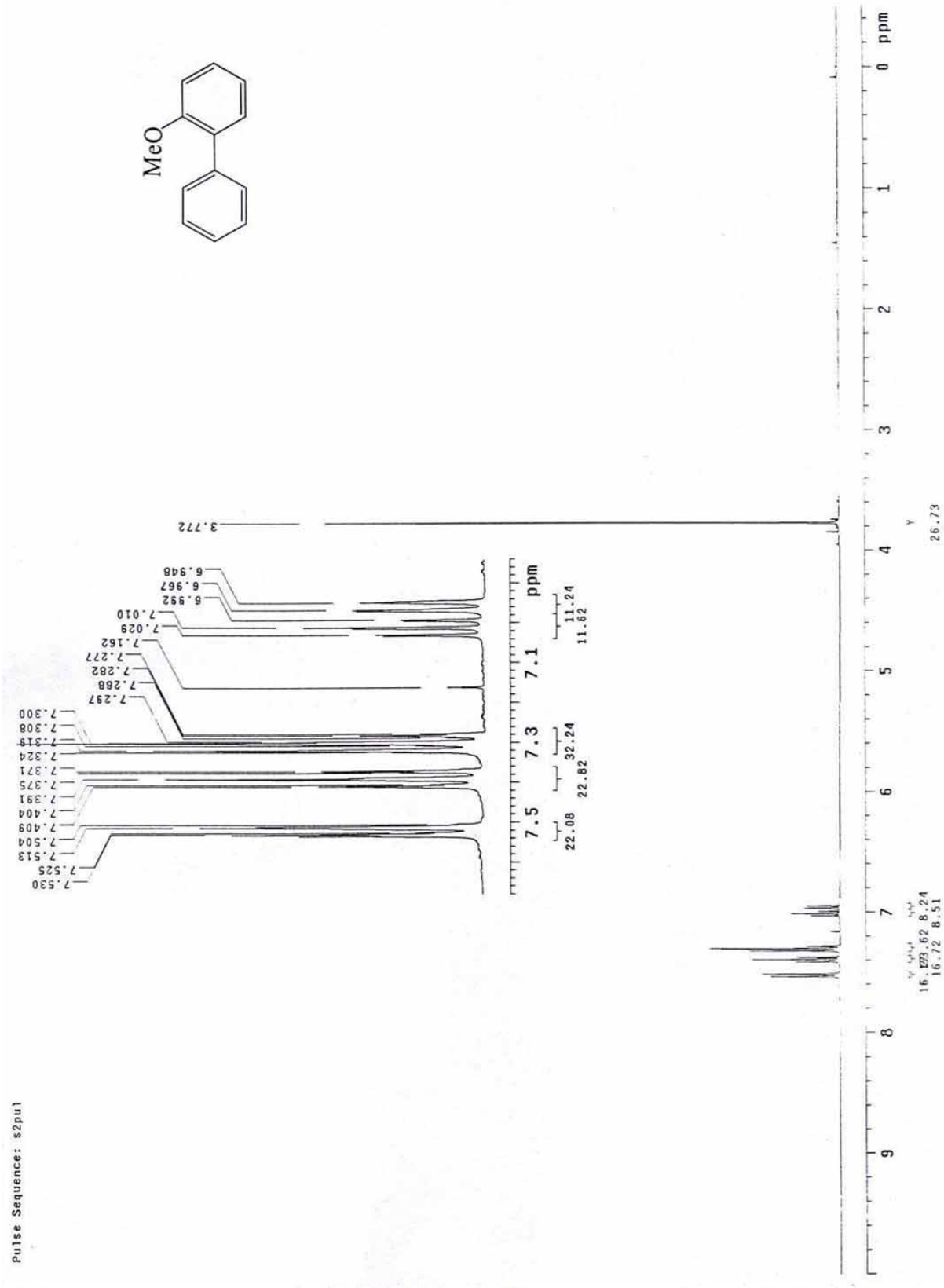

4-fluorobiphenyl
Pulse Sequence: s2pul
ppm
200
180
160
140
120
100
80
60
40
20
163.674
161.229
140.216
137.296
128.780
128.678
128.602
127.226
127.125
126.980
115.672
115.459
77.314
77.000
76.686

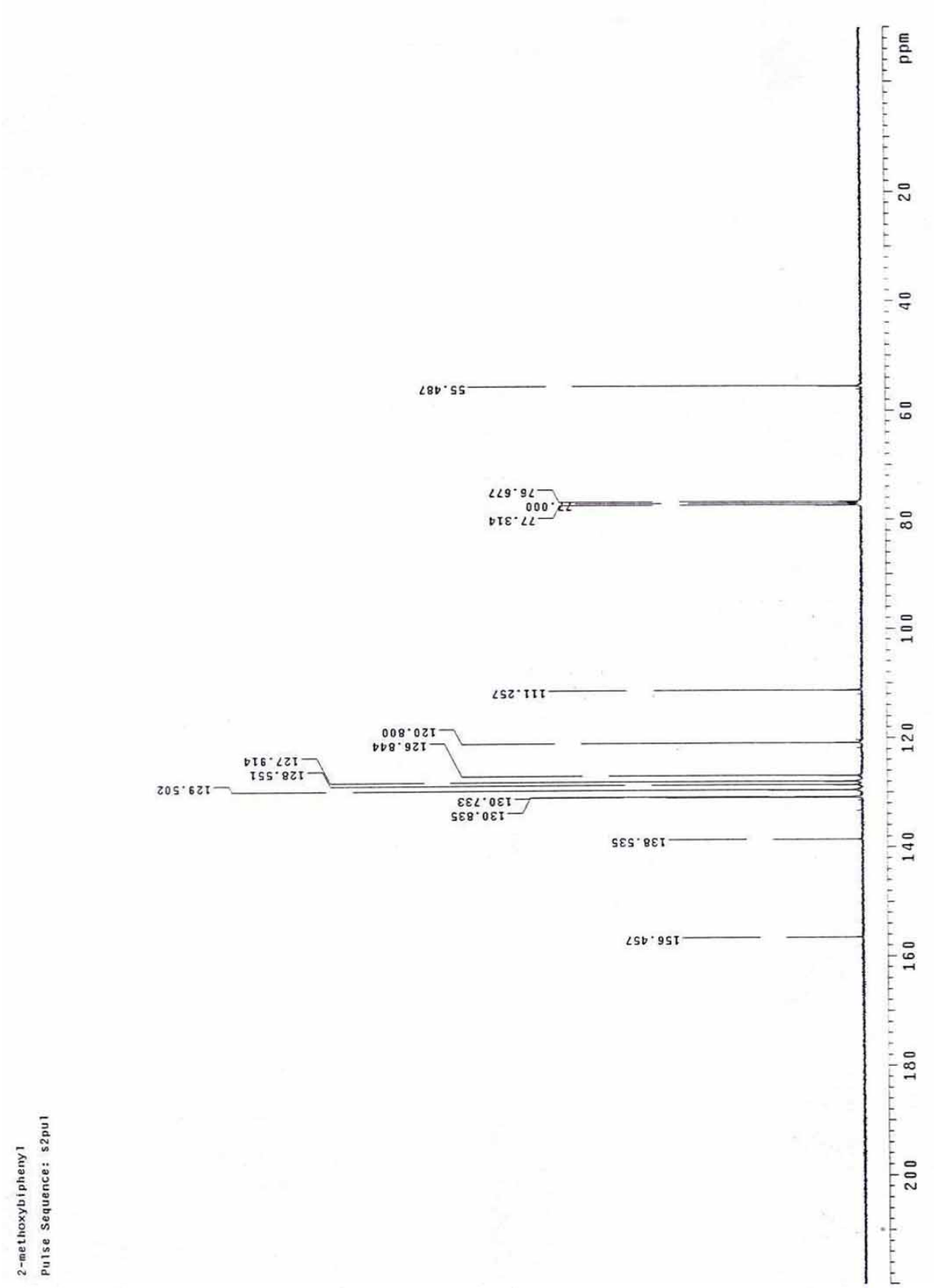

4-nitrobiphenyl
Pulse Sequence: s2pul
NO2
8.305
8.299
8.294
8.290
8.284
8.279
8.273
7.748
7.745
7.739
7.734
7.730
7.724
7.719
7.716
7.713
7.636
7.632
7.625
7.613
7.521
7.517
7.510
7.499
7.480
7.467
7.464
7.461
7.457
7.450
7.445
7.439
7.428
7.425
8.5
8.3
8.1
7.9
7.7
7.5
ppm
21.66
23.16
22.15
33.03
0
1
2
3
4
5
6
7
8
9
21.66
22.15
23.183.03

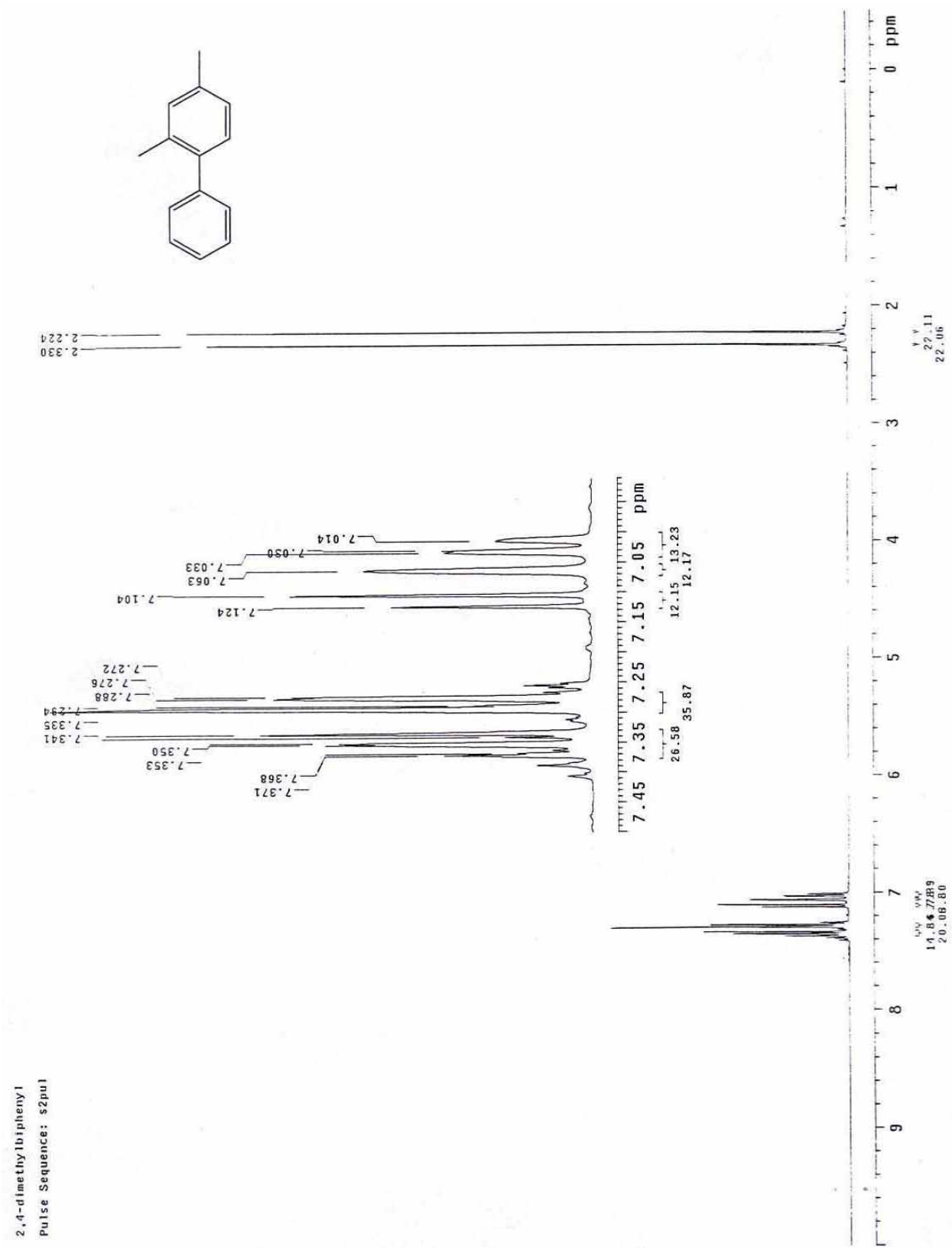

4-nitrobiphenyl
Pulse Sequence: s2pul
147.585
147.084
138.739
129.111
128.874
127.744
127.337
124.051
77.314
77.000
76.686
ppm
200
180
160
140
120
100
80
60
40
20

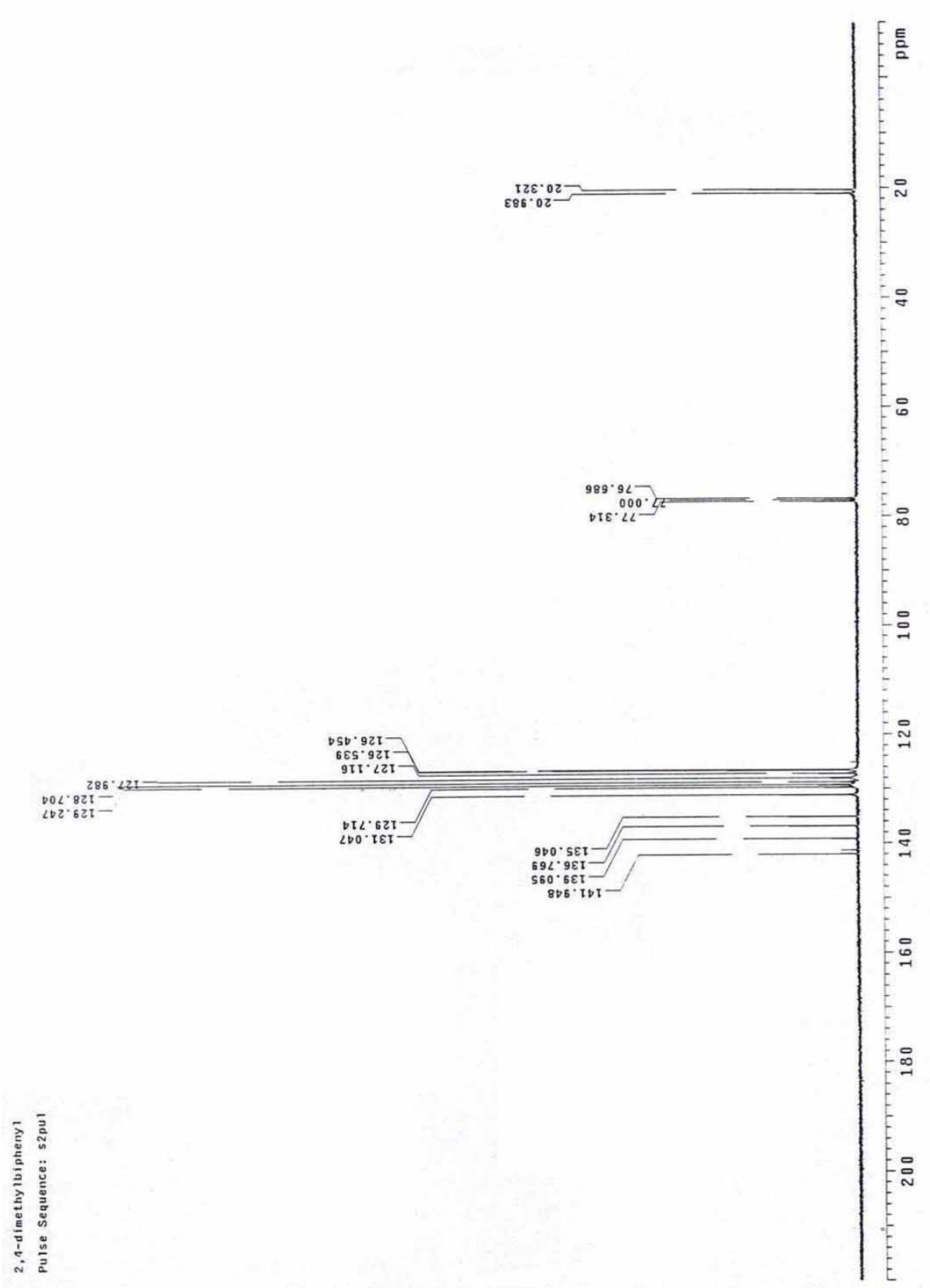

3,5-dimethylbiphenyl
Pulse Sequence: s2pul
2.331
2.333
6.946
7.181
7.183
7.249
7.252
7.255
7.265
7.270
7.275
7.285
7.288
7.292
7.304
7.340
7.345
7.348
7.361
7.364
7.368
7.374
7.378
7.382
7.386
7.396
7.521
7.526
7.529
7.532
7.534
7.542
7.547
7.550
7.555
ppm
10.76
22.29
12.62
27.04
27.29
41.95
15.807.35 6.27
15.62.95

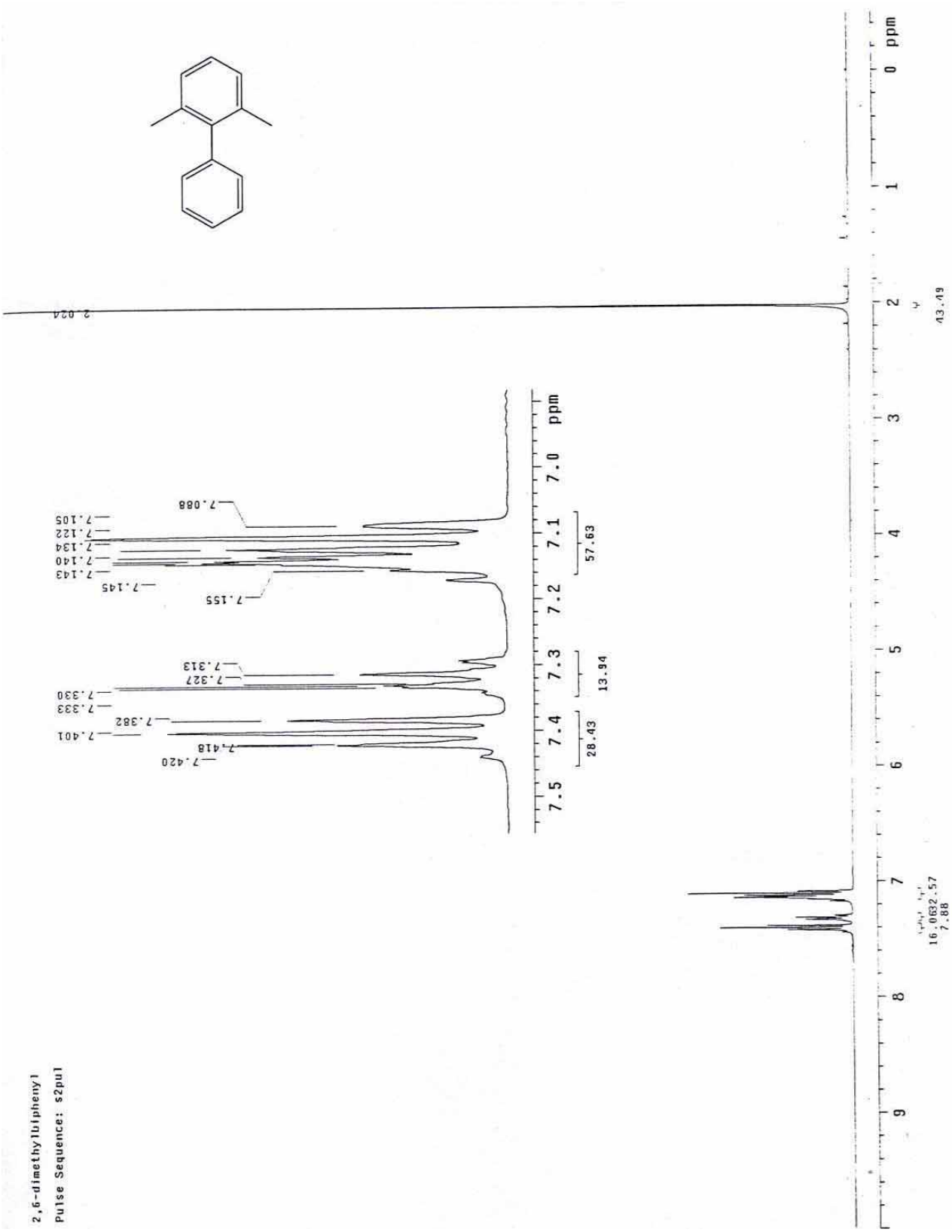

3,5-dimethylbiphenyl
Pulse Sequence: s2pul
ppm
20
40
60
80
100
120
140
160
180
200
21.332
76.686
77.000
77.314
125.070
127.014
127.133
128.585
128.695
128.848
138.136
141.235
141.447


MeO
Pulse Sequence: s2pul
7.530
7.525
7.513
7.504
7.409
7.404
7.391
7.375
7.371
7.324
7.319
7.308
7.300
7.297
7.288
7.282
7.277
7.162
7.029
7.010
6.992
6.967
6.948
3.772
7.5
7.3
7.1
ppm
22.08
22.82
32.24
11.24
11.62
26.73
16.72 8.51
0 ppm


2-methoxybiphenyl
Pulse Sequence: s2pul
ppm
20
40
60
80
100
120
140
160
180
200
55.487
76.677
77.000
77.314
111.257
120.800
126.844
127.914
128.551
129.502
130.733
130.835
138.535
156.457


2,4-dimethylbiphenyl
Pulse Sequence: s2pul
2.330
2.224
7.014
7.050
7.063
7.033
7.104
7.124
7.272
7.276
7.288
7.294
7.335
7.341
7.350
7.353
7.368
7.371
7.45 7.35 7.25 7.15 7.05 ppm
26.58
35.87
12.15
13.23
12.17
22.11
22.06
14.86
7.89
20.08
80
0 ppm
1
2
3
4
5
6
7
8
9


2,4-dimethylbiphenyl
Pulse Sequence: s2pul
ppm
20.983
20.321
77.314
77.000
76.686
141.948
139.095
136.769
135.046
131.047
129.714
129.247
128.704
127.982
127.116
126.539
126.454


2,6-dimethylbiphenyl
Pulse Sequence: s2pul
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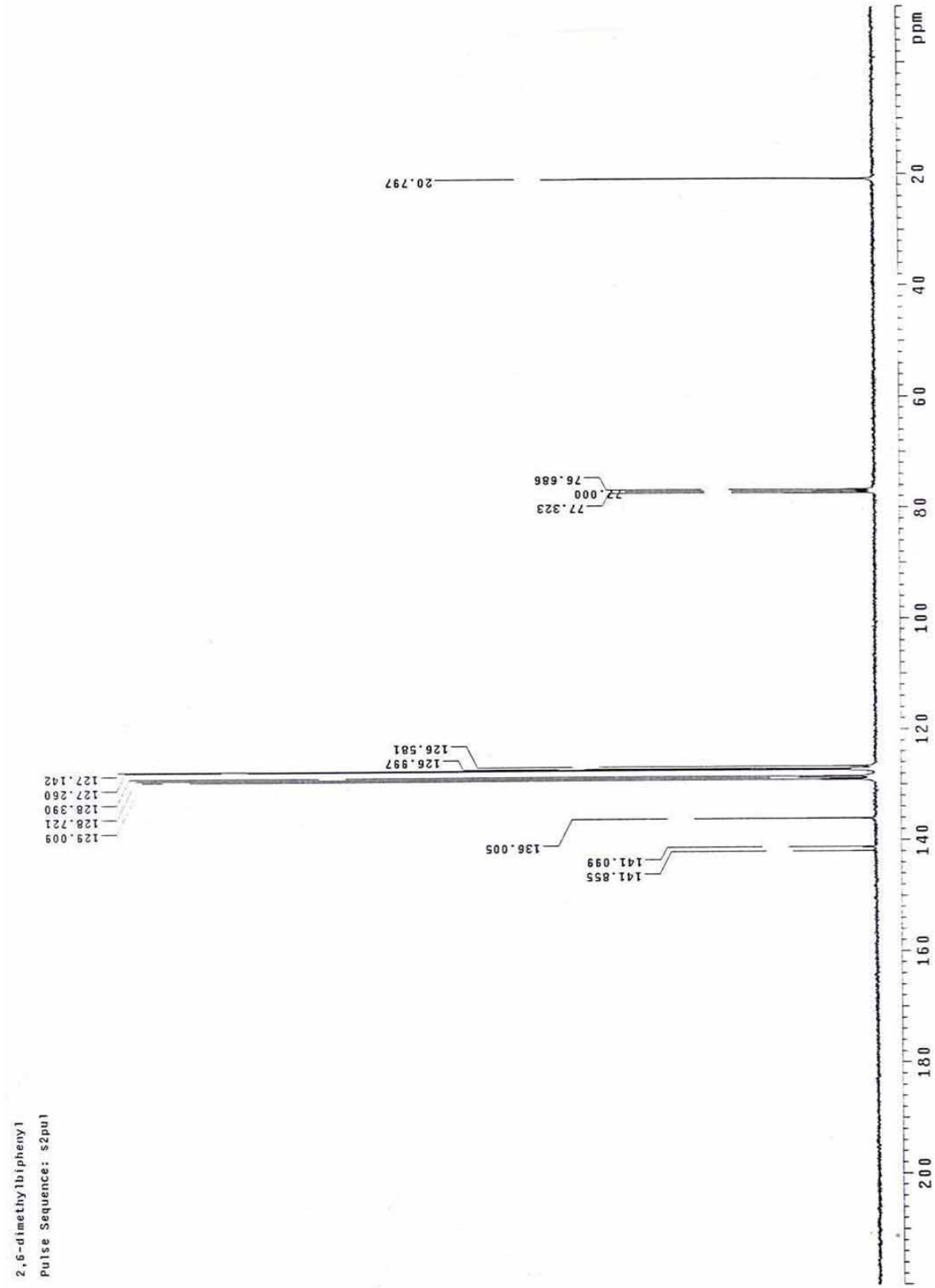
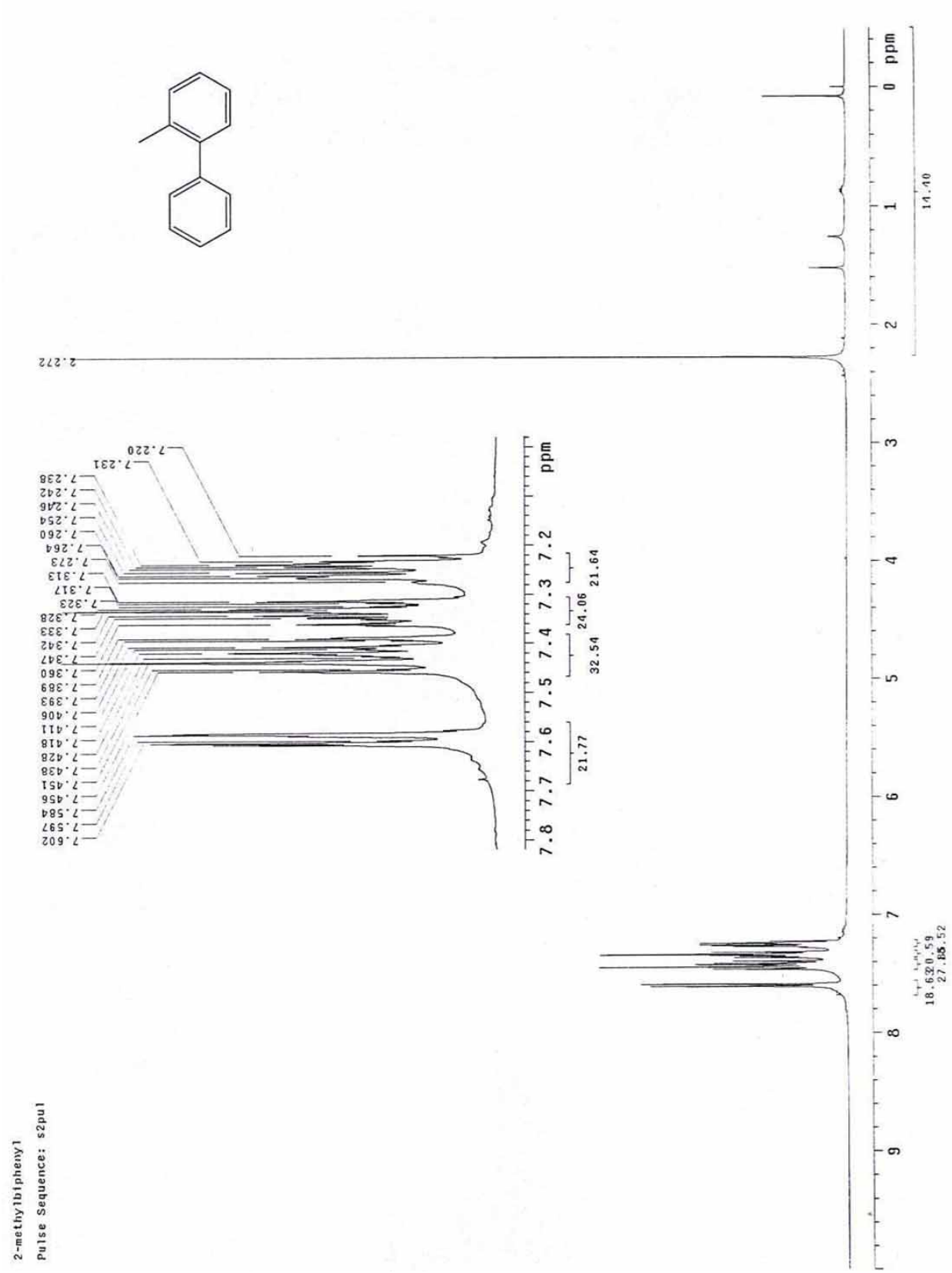
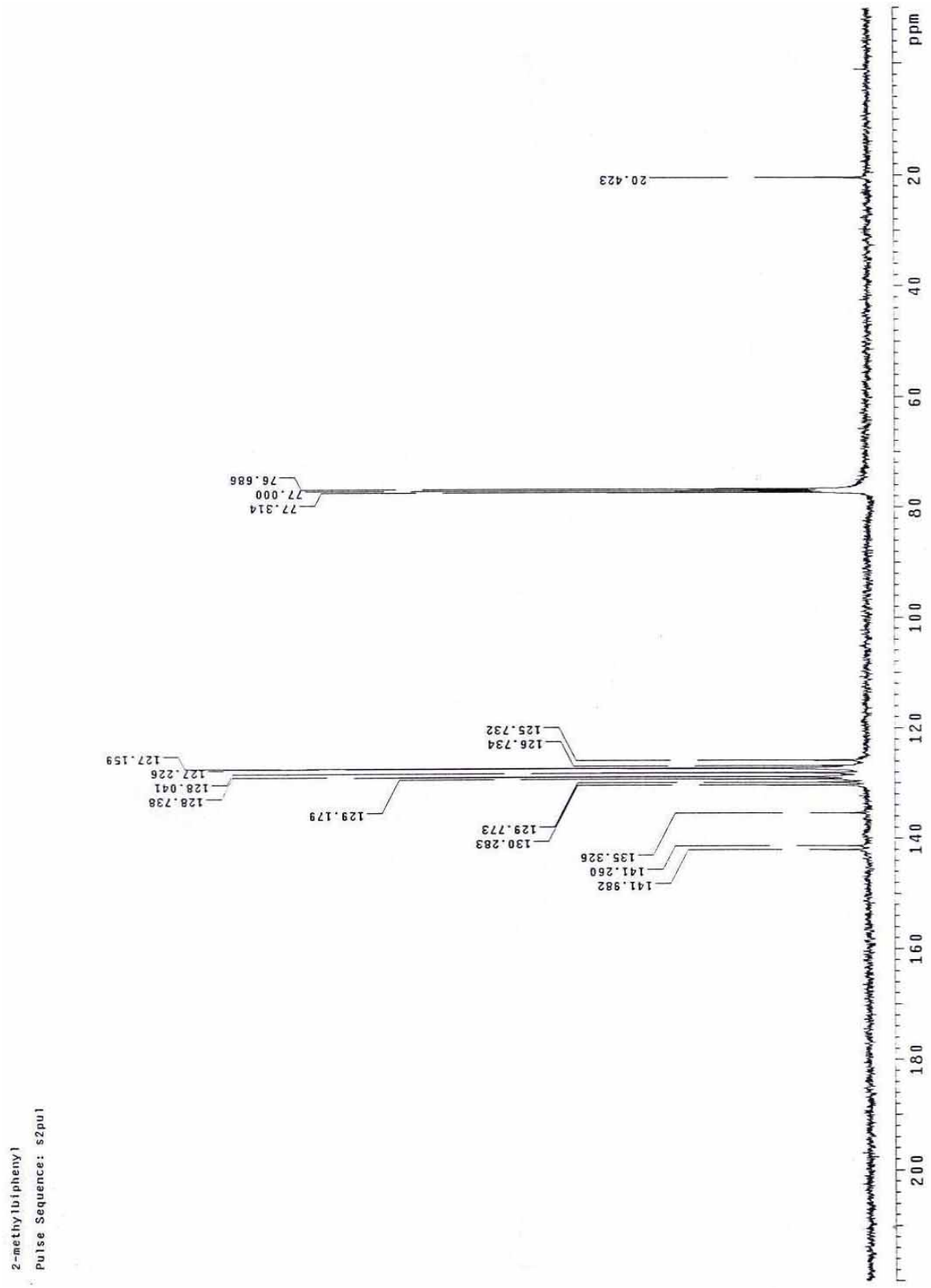
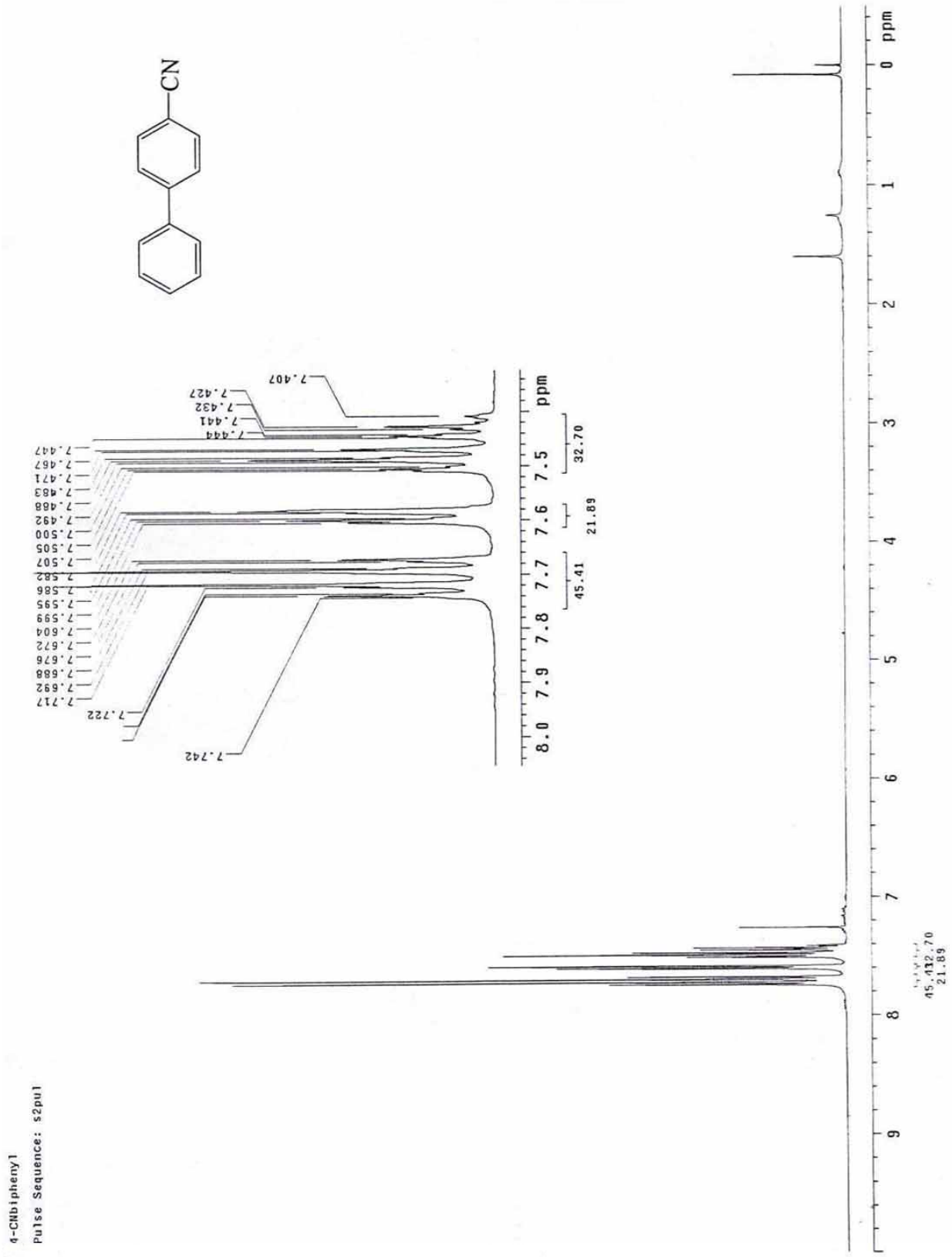
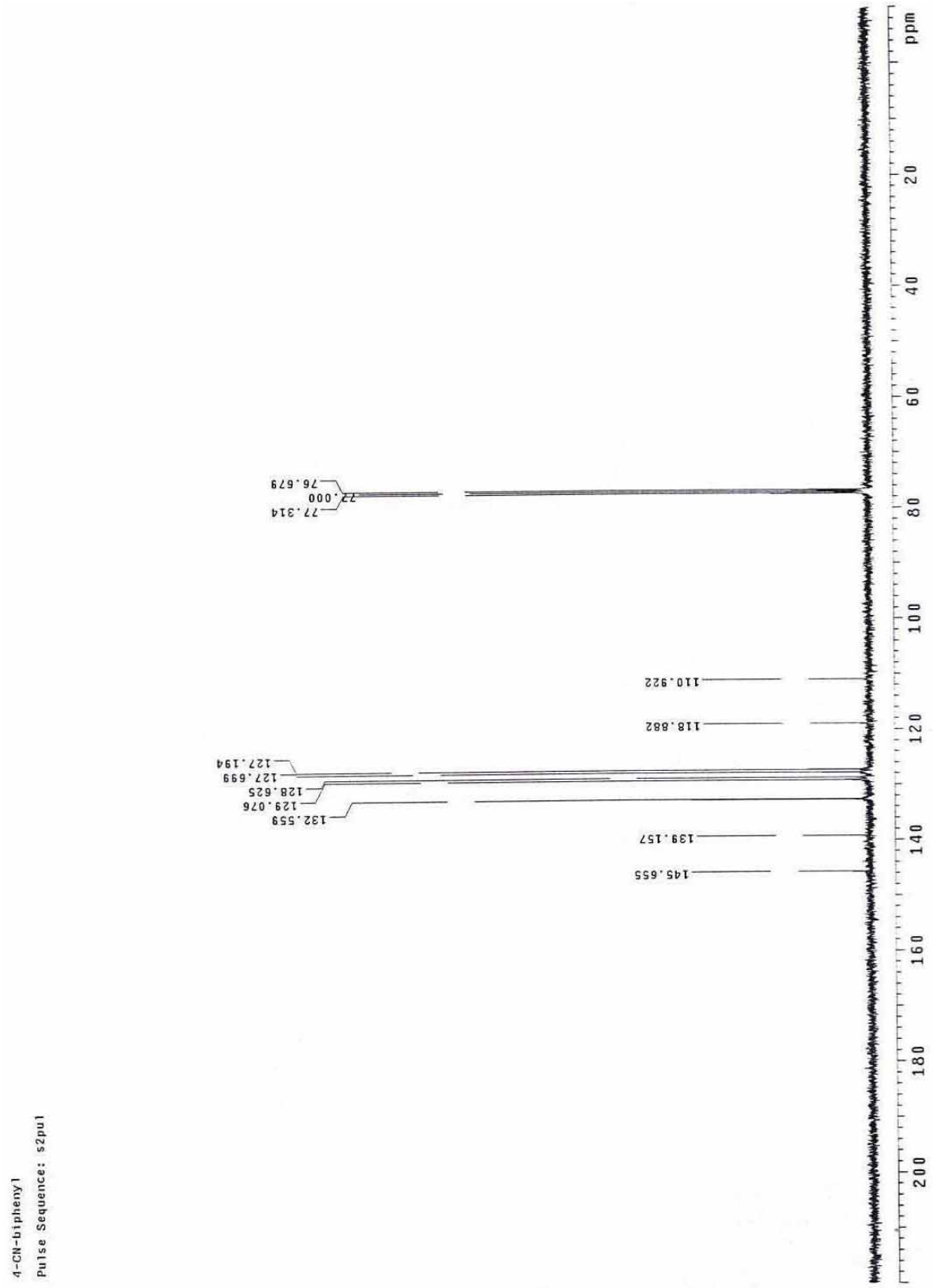
7.418
7.401
7.382
7.333
7.330
7.327
7.313
7.155
7.145
7.143
7.140
7.134
7.122
7.105
7.088
28.43
13.94
57.63
16.0632.57
7.88
13.49
2.024
ppm

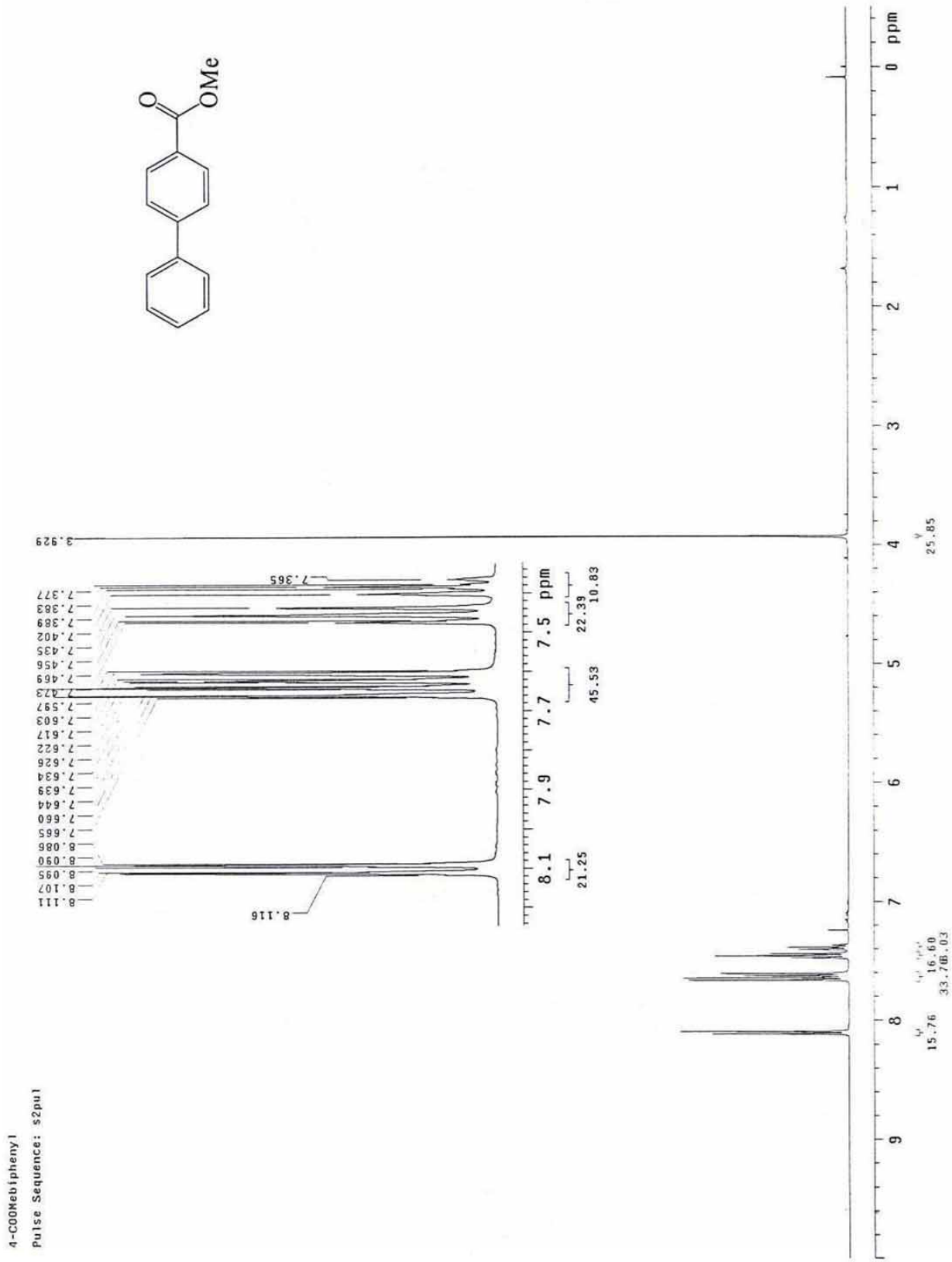

2,6-dimethylbiphenyl
Pulse Sequence: s2pul
ppm
20.797
76.686
77.000
77.323
126.581
126.997
127.142
127.260
128.390
128.721
129.009
136.005
141.099
141.855

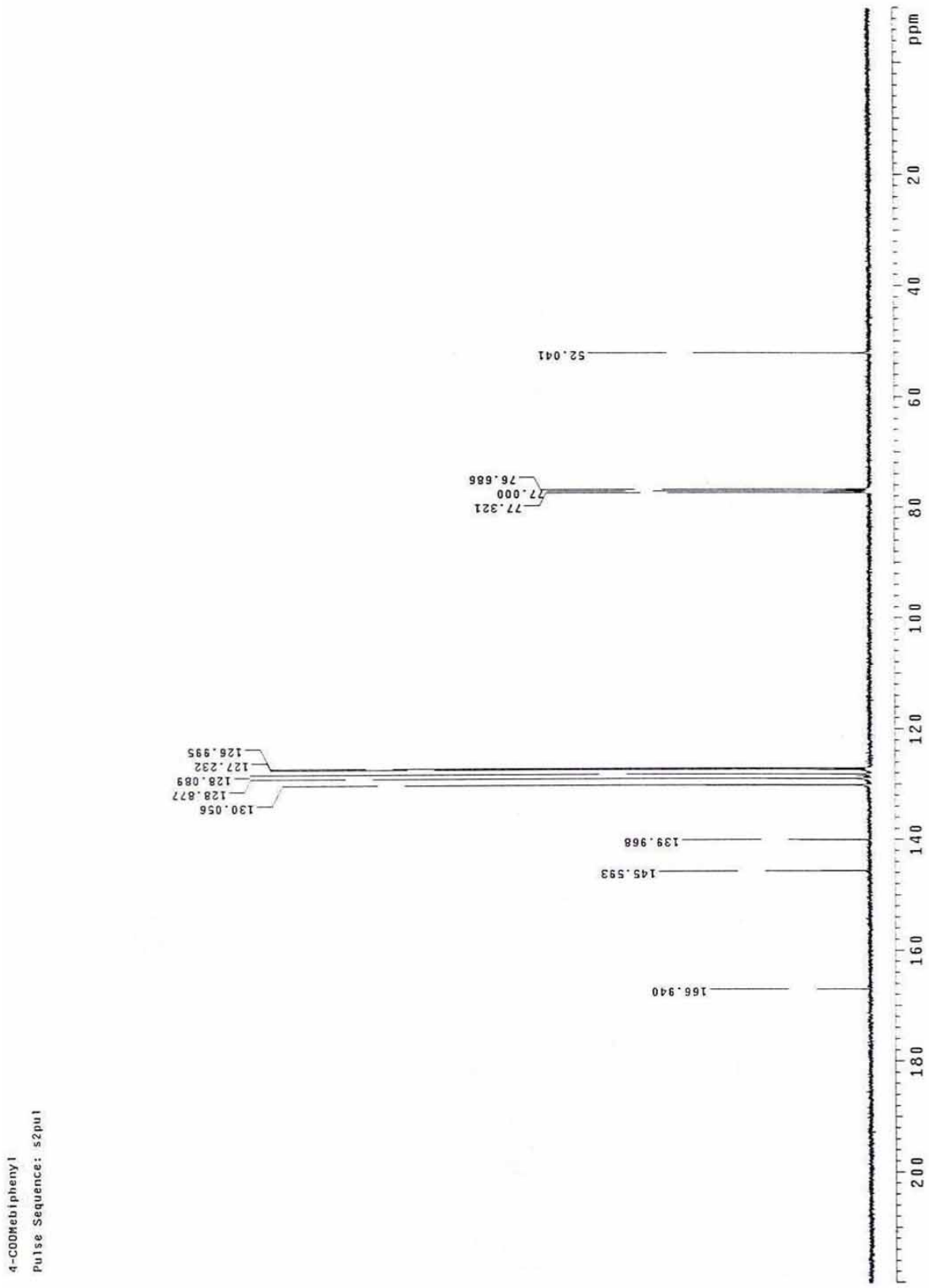

2-methylbiphenyl
Pulse Sequence: s2pul
7.602
7.597
7.584
7.456
7.451
7.438
7.428
7.418
7.411
7.406
7.393
7.389
7.360
7.347
7.342
7.333
7.328
7.323
7.317
7.313
7.273
7.264
7.260
7.254
7.246
7.242
7.238
7.231
7.220
2.272
7.8 7.7 7.6 7.5 7.4 7.3 7.2 ppm
21.77
32.54
24.06
21.64
ppm
14.10
18.620.59
27.185.52

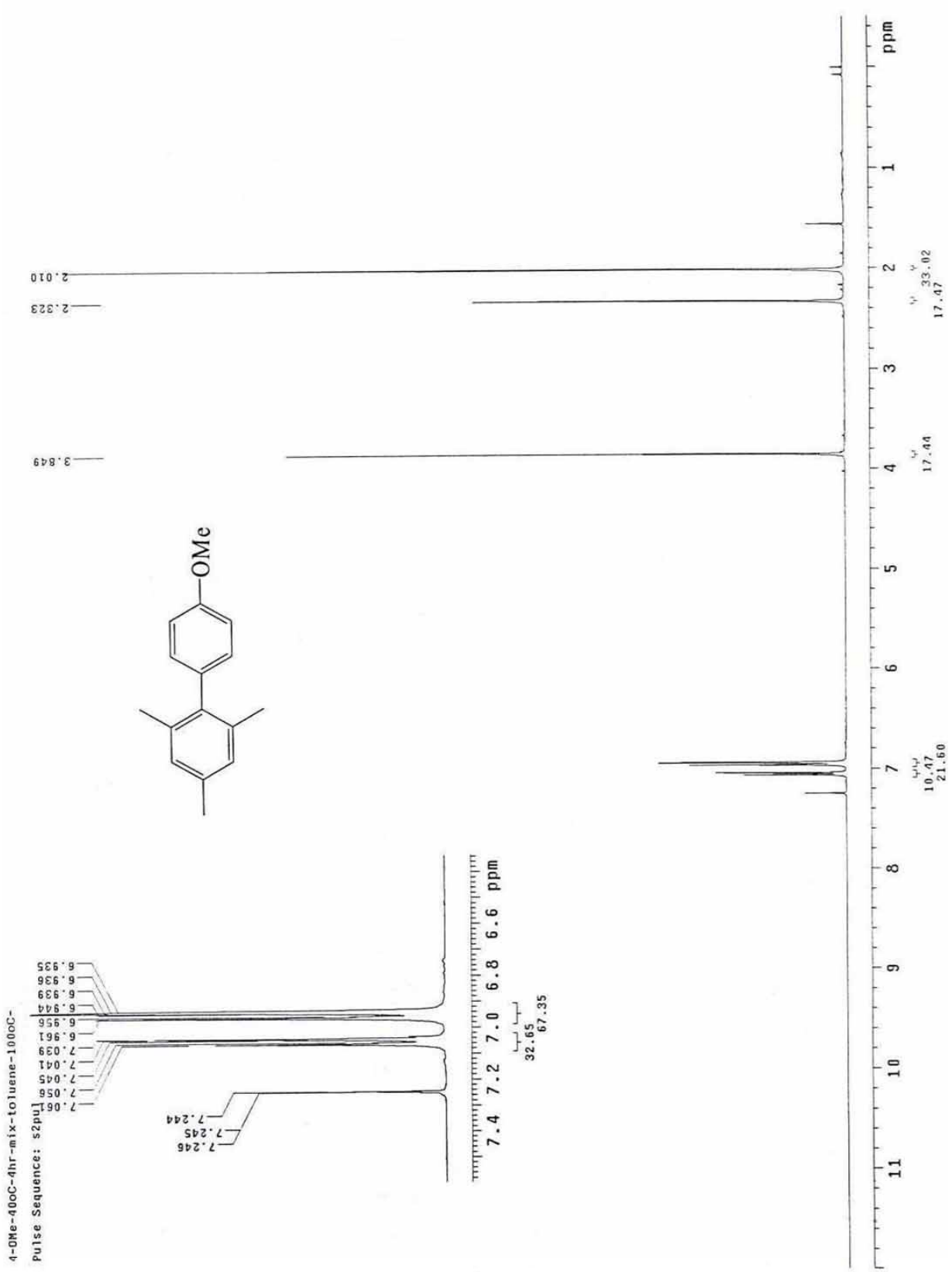

2-methylbiphenyl
Pulse Sequence: s2pul
ppm
20.423
76.686
77.000
77.314
125.732
126.734
127.159
127.226
128.041
128.738
129.179
129.773
130.283
135.326
141.260
141.982

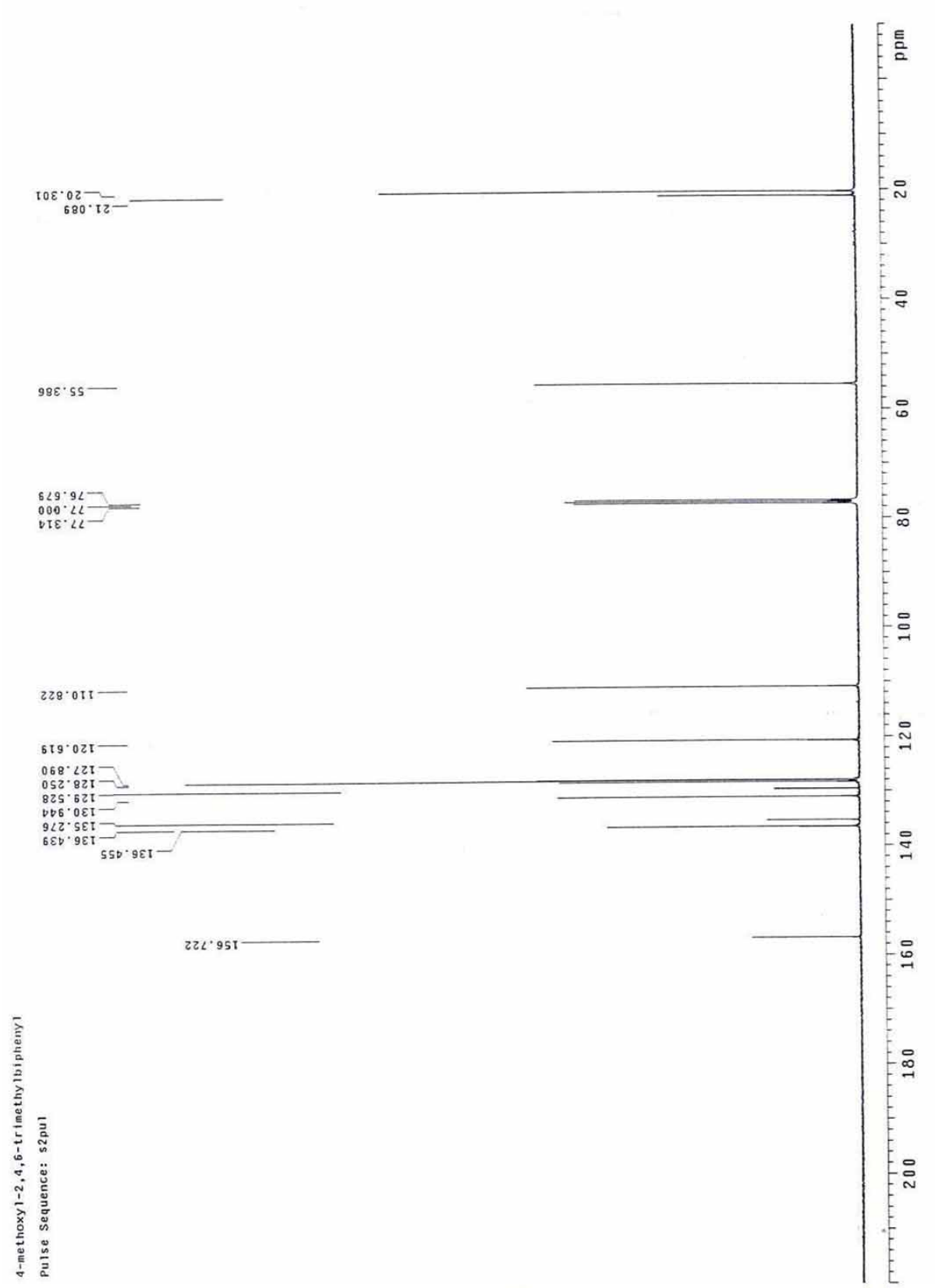

4-CNbiphenyl
Pulse Sequence: s2pul
CN
7.407
7.427
7.432
7.441
7.444
7.447
7.467
7.471
7.483
7.488
7.492
7.500
7.505
7.507
7.582
7.586
7.595
7.599
7.604
7.672
7.676
7.688
7.692
7.717
7.722
7.742
8.0 7.9 7.8 7.7 7.6 7.5 ppm
45.41
21.89
32.70
0 ppm 1 2 3 4 5 6 7 8 9
45.41
32.70
21.89

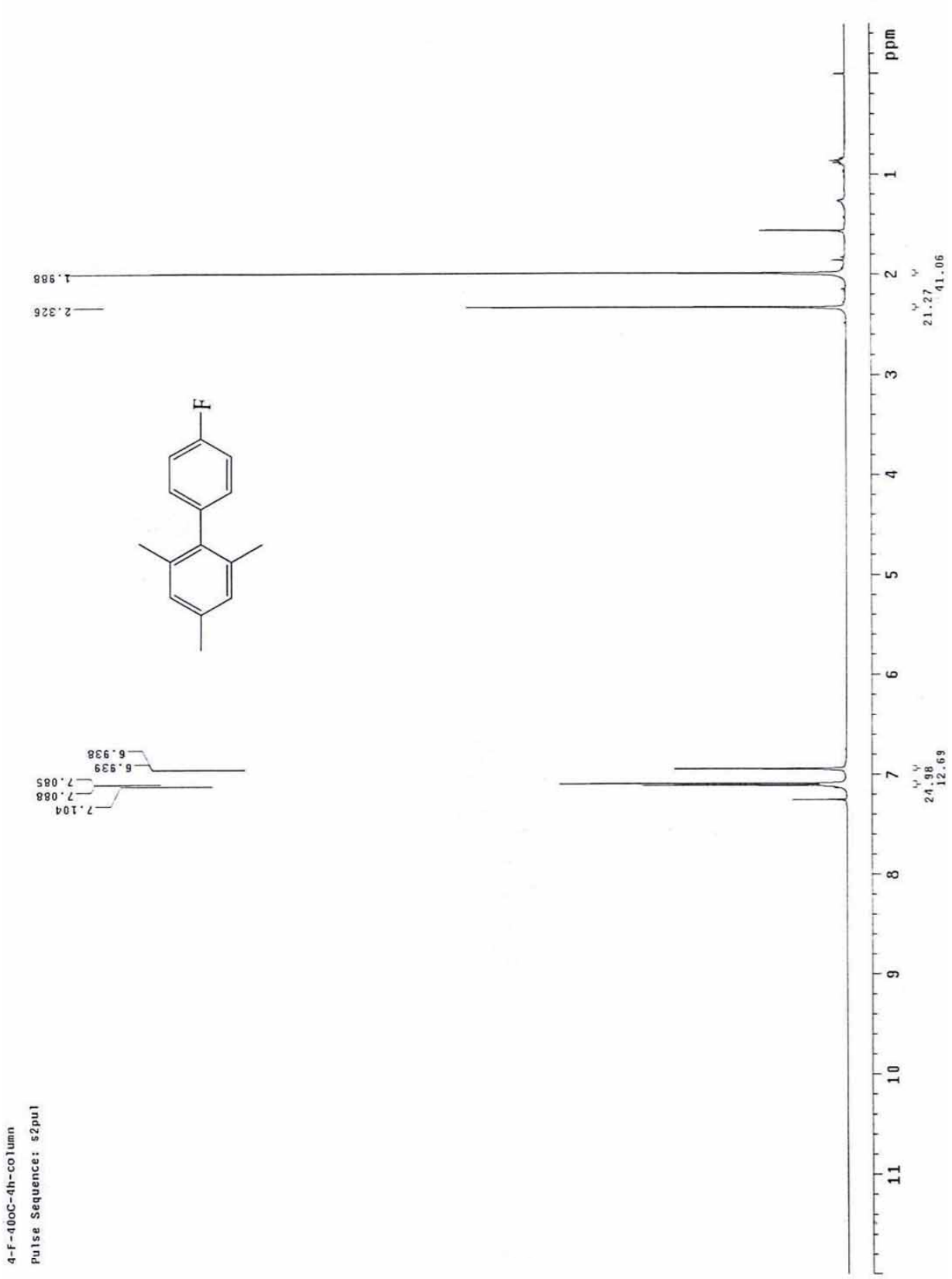

4-CN-biphenyl
Pulse Sequence: s2pul
ppm
20
40
60
80
100
120
140
160
180
200
76.679
77.000
77.314
110.922
118.882
127.194
127.699
128.625
129.076
132.559
139.157
145.655

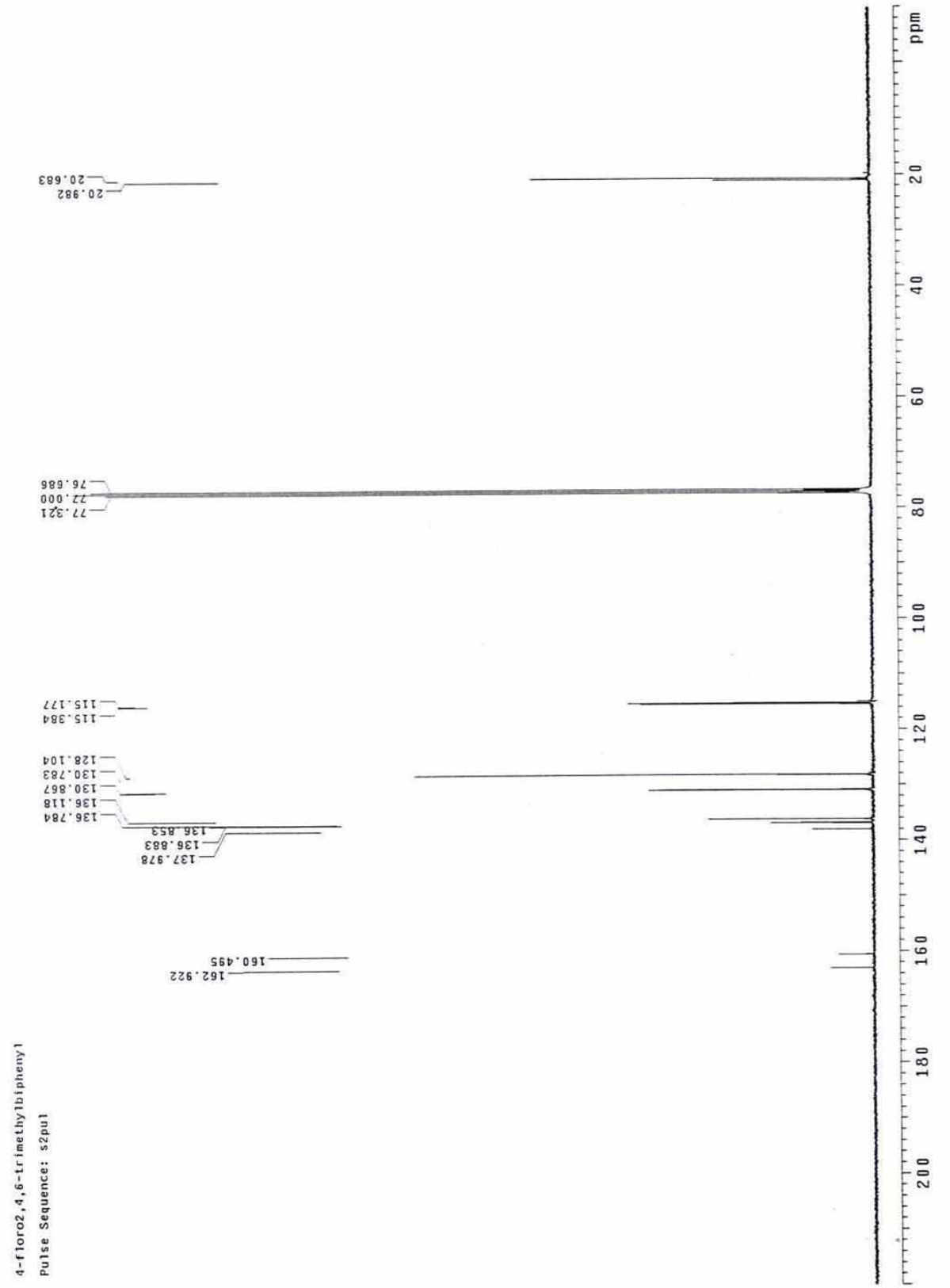

4-COOMebiphenyl
Pulse Sequence: s2pul
OMe
O
8.929
7.365
7.377
7.383
7.389
7.402
7.435
7.456
7.469
7.473
7.597
7.603
7.617
7.622
7.626
7.634
7.639
7.644
7.660
7.665
8.086
8.090
8.095
8.107
8.111
8.116
8.1
7.9
7.7
7.5 ppm
21.25
45.53
22.39
10.83
25.85
15.76
16.60
33.70
8.03
ppm

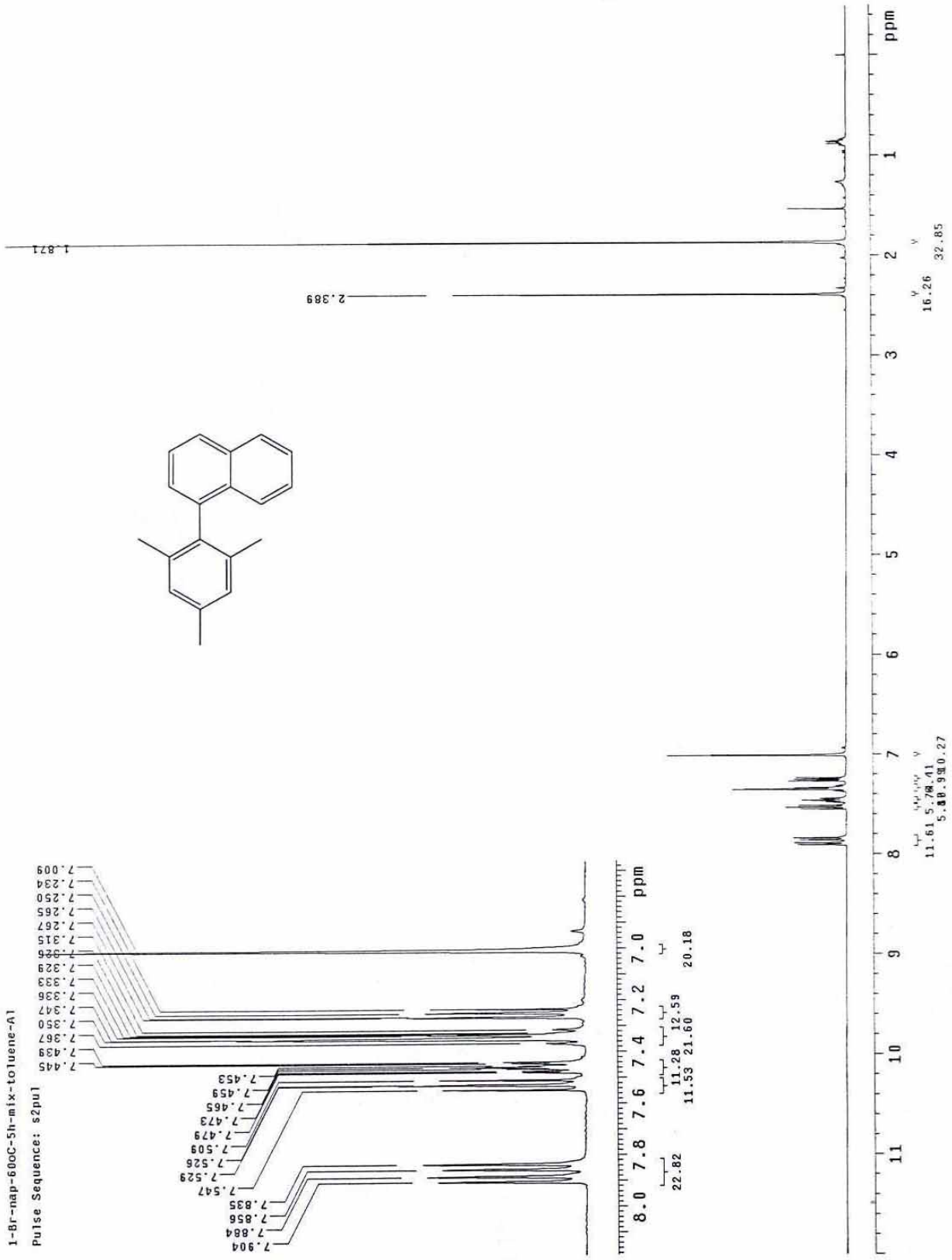

4-COOMebiphenyl
Pulse Sequence: s2pul
166.940
145.593
139.968
130.056
128.877
128.089
127.232
126.995
77.321
77.000
76.686
52.041
ppm
200
180
160
140
120
100
80
60
40
20

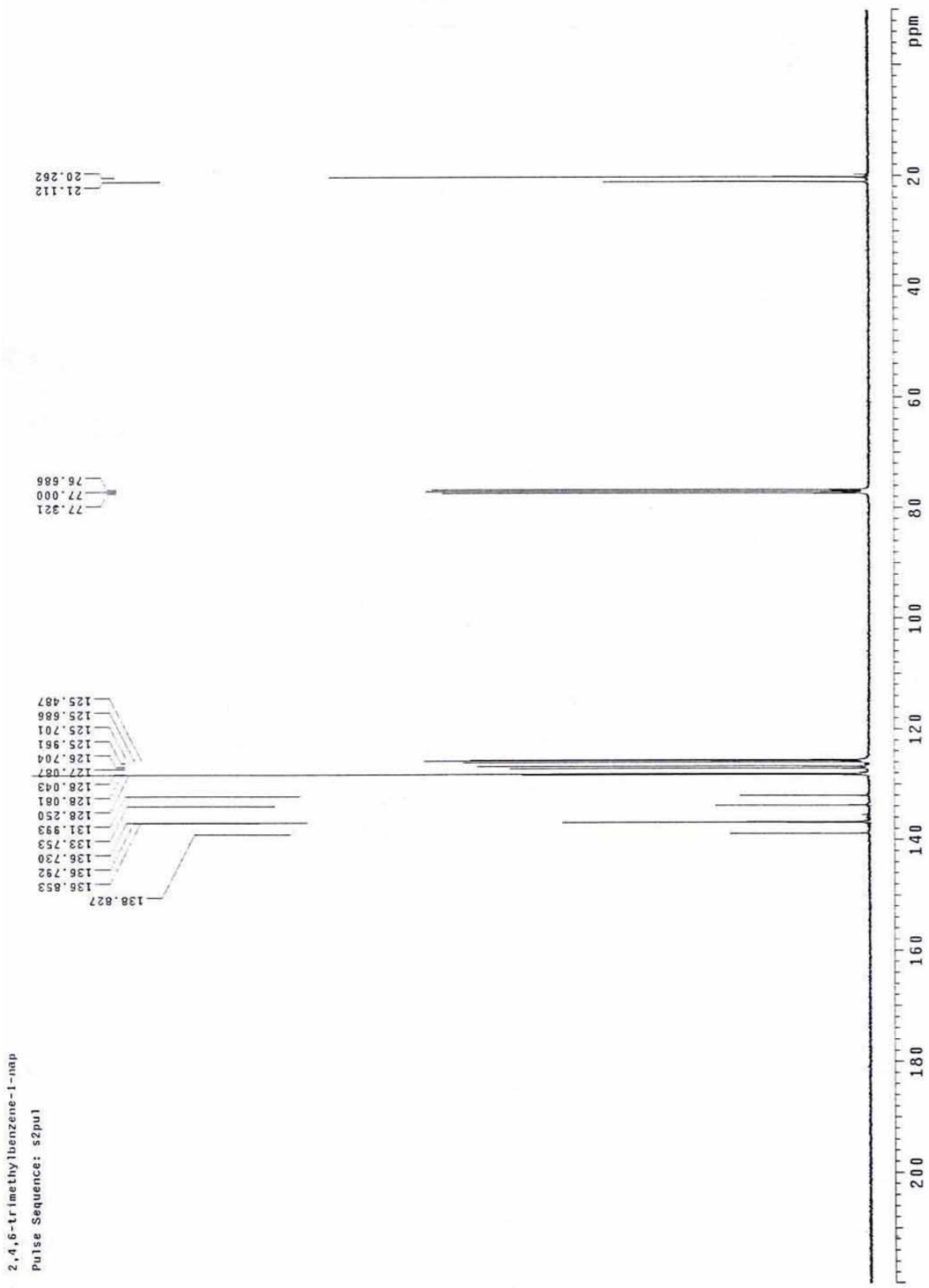

4-OMe-40oC-4hr-mix-toluene-100oC-
Pulse Sequence: s2pul
OMe
2.010
2.323
3.849
7.061
7.056
7.045
7.041
7.039
6.961
6.956
6.944
6.939
6.936
6.935
7.246
7.245
7.244
ppm
32.65
67.35
33.02
17.47
17.44
10.47
21.60

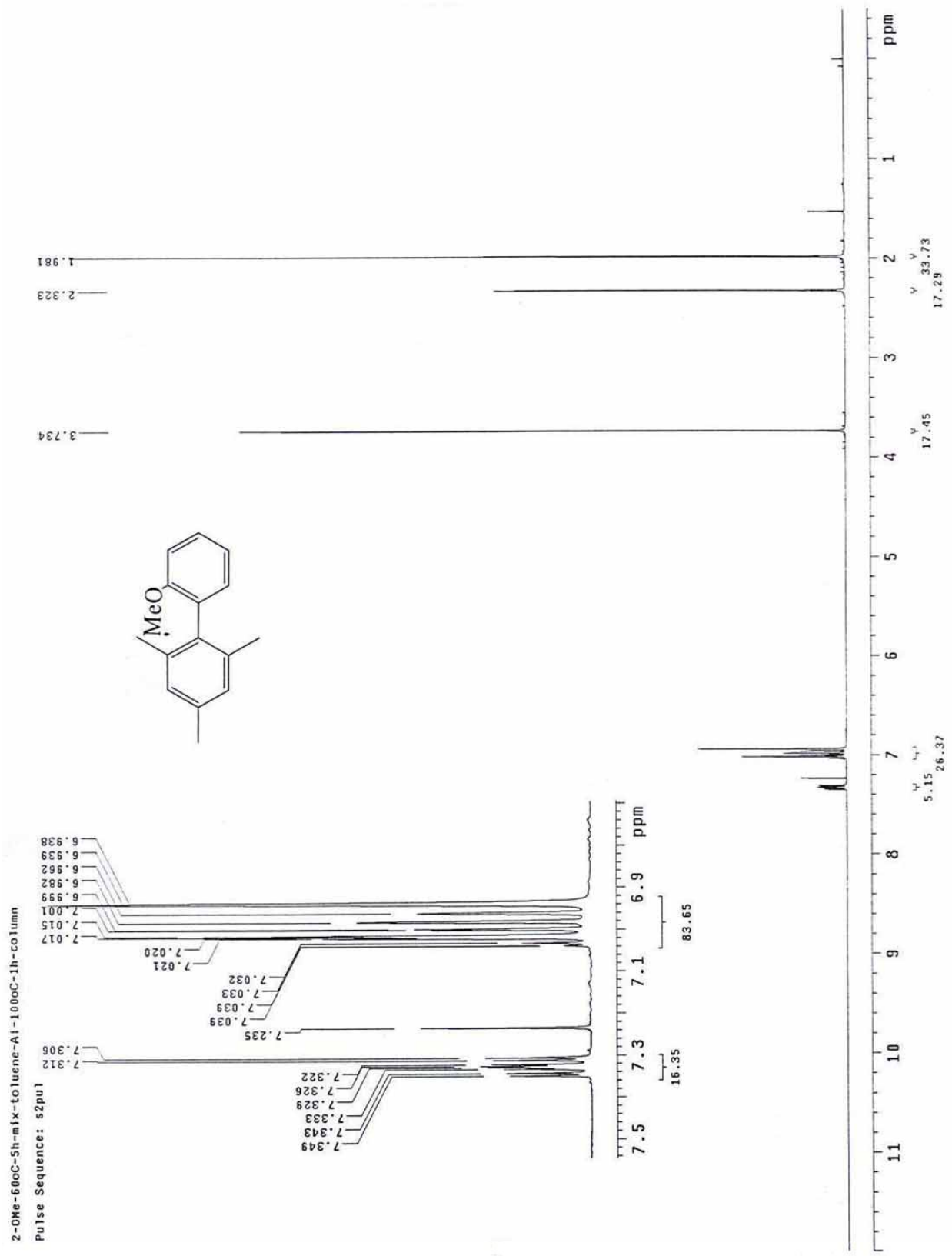

4-methoxyl-2,4,6-trimethylbiphenyl
Pulse Sequence: s2pul
ppm
20.301
21.089
55.386
76.679
77.000
77.314
110.822
120.619
127.890
128.250
129.528
130.944
135.276
136.439
136.455
156.722
20
40
60
80
100
120
140
160
180
200

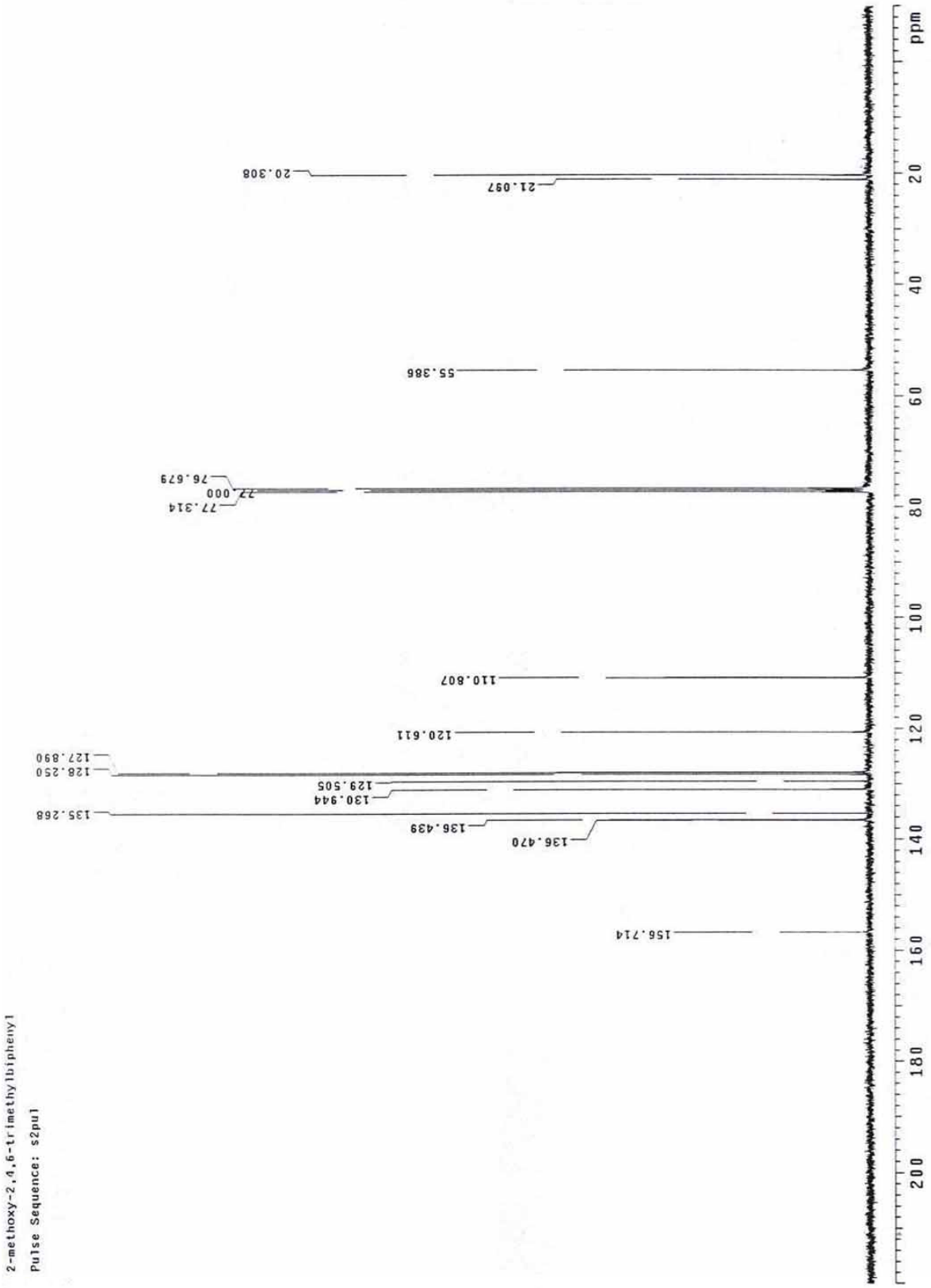

4-F-40oC-4h-column
Pulse Sequence: s2pul
1.988
2.326
6.938
6.939
7.085
7.088
7.104
F
ppm
1
2
3
4
5
6
7
8
9
10
11
21.27
41.06
24.98
12.69

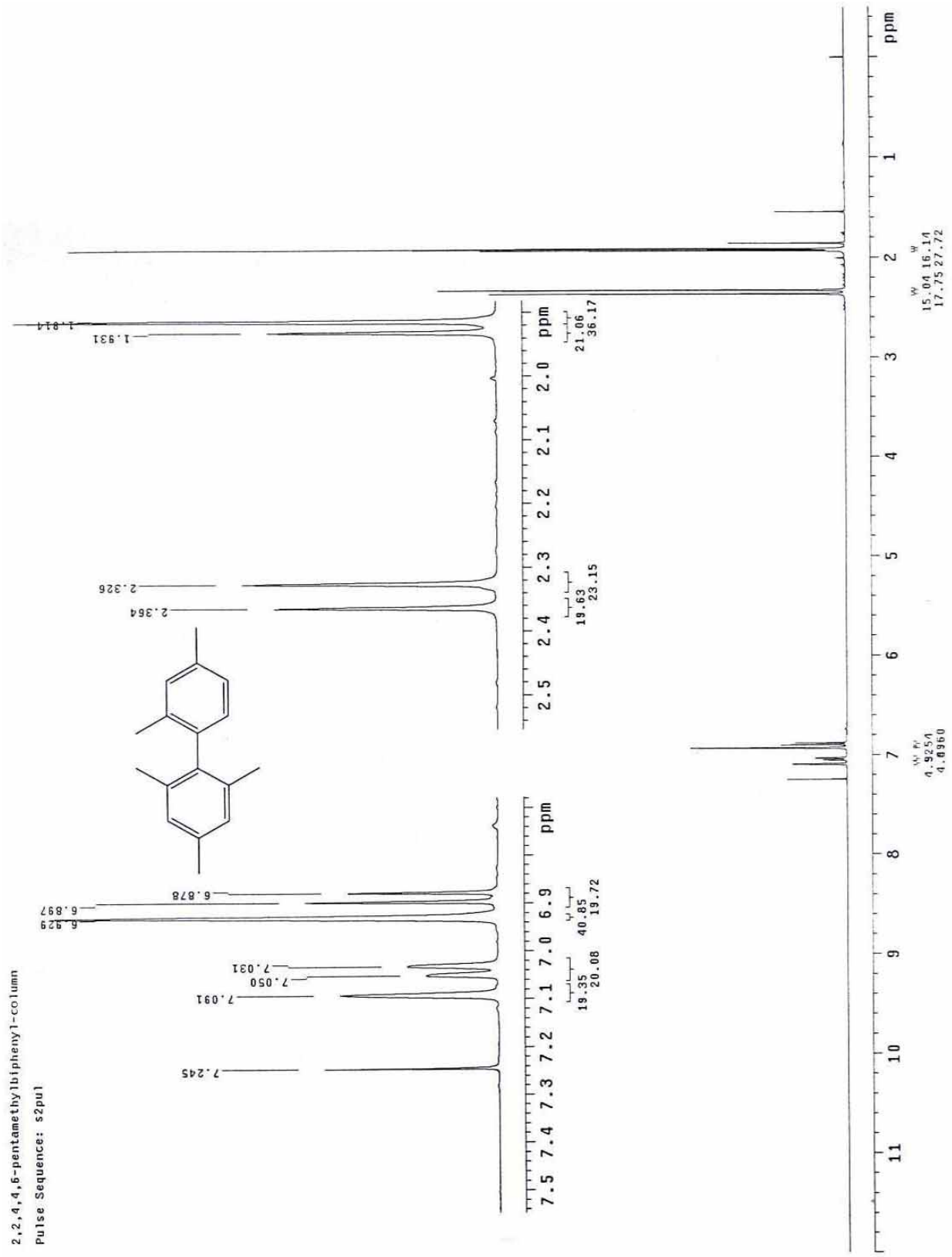

4-floro2,4,6-trimethylbiphenyl
Pulse Sequence: s2pul
ppm
20
40
60
80
100
120
140
160
180
200
20.982
20.683
77.321
77.000
76.686
115.384
115.177
128.104
130.783
130.867
136.118
136.784
136.853
136.883
137.978
160.495
162.922

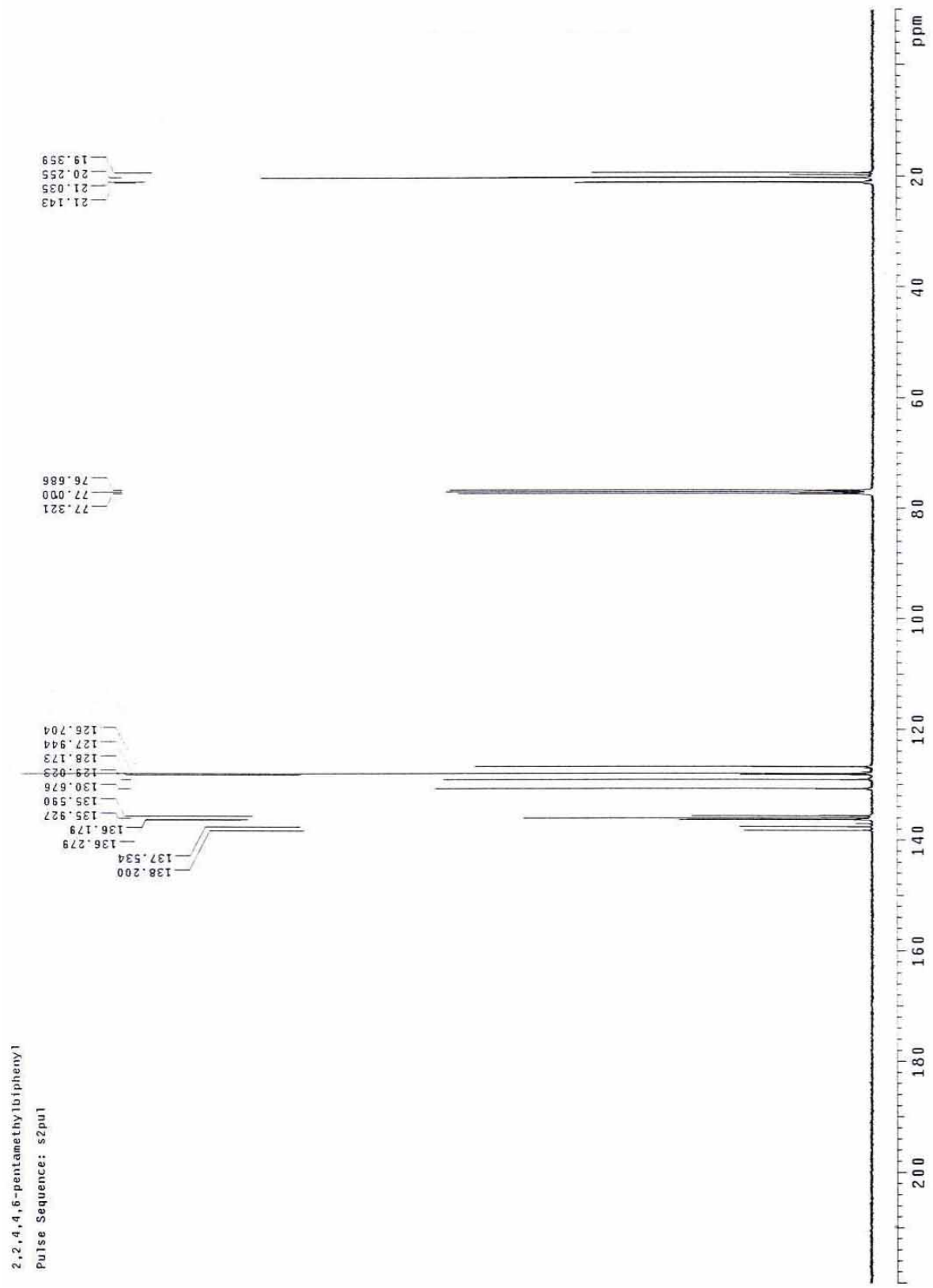

1-Br-nap-60oC-5h-mix-toluene-Al
Pulse Sequence: s2pul
ppm
2.389
1.871
7.904
7.884
7.856
7.835
7.547
7.529
7.526
7.509
7.479
7.473
7.465
7.459
7.453
7.445
7.439
7.367
7.350
7.347
7.336
7.333
7.329
7.326
7.315
7.267
7.265
7.250
7.234
7.009
22.82
11.53
11.28
21.60
12.59
20.18
16.26
32.85
11.61
5.78
5.88
5.99
10.27

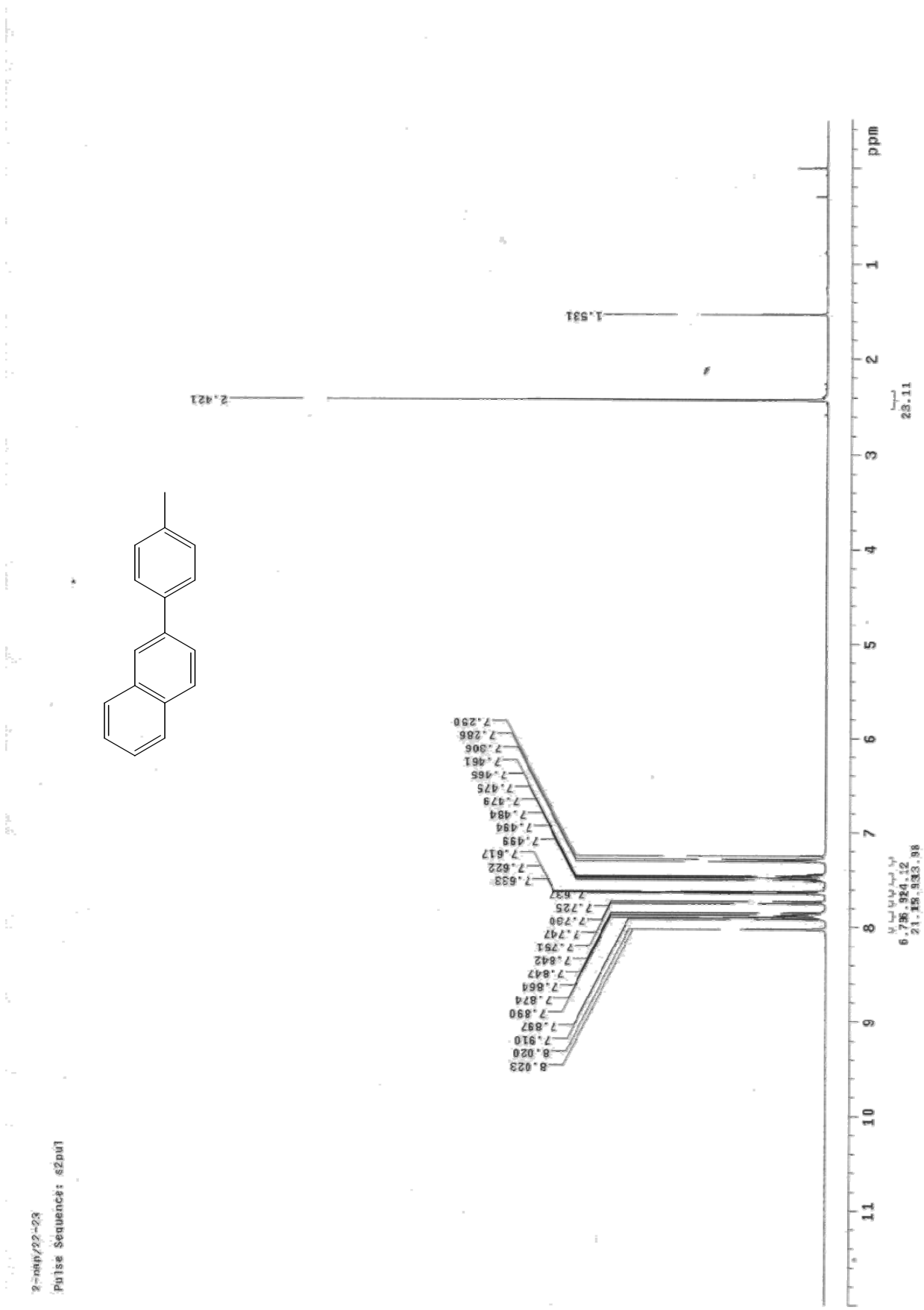

20.262
21.112
76.686
77.000
77.321
125.487
125.686
125.701
125.961
126.704
127.087
128.043
128.081
128.250
131.993
133.753
136.730
136.792
136.853
138.827
2,4,6-trimethylbenzene-1-nap
Pulse Sequence: s2pul
ppm
20
40
60
80
100
120
140
160
180
200

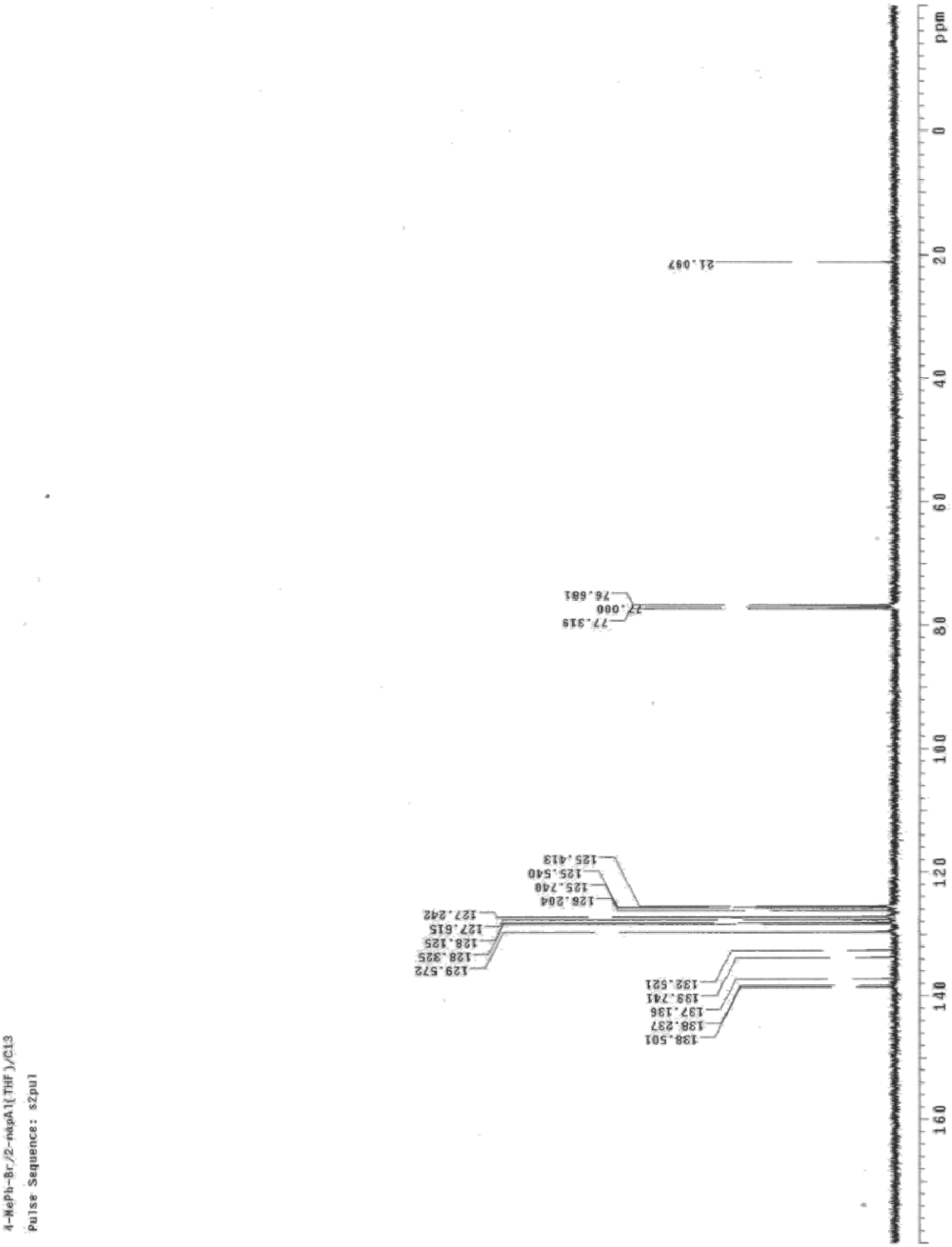

2-OMe-60oC-5h-mix-toluene-Al-100oC-1h-column
Pulse Sequence: s2pul
MeO
1.981
2.323
3.734
7.312
7.306
7.235
7.039
7.039
7.033
7.032
7.021
7.020
7.017
7.015
7.001
6.999
6.982
6.962
6.939
6.938
7.349
7.343
7.333
7.329
7.326
7.322
83.65
16.35
ppm
33.73
17.29
17.45
5.15
26.37

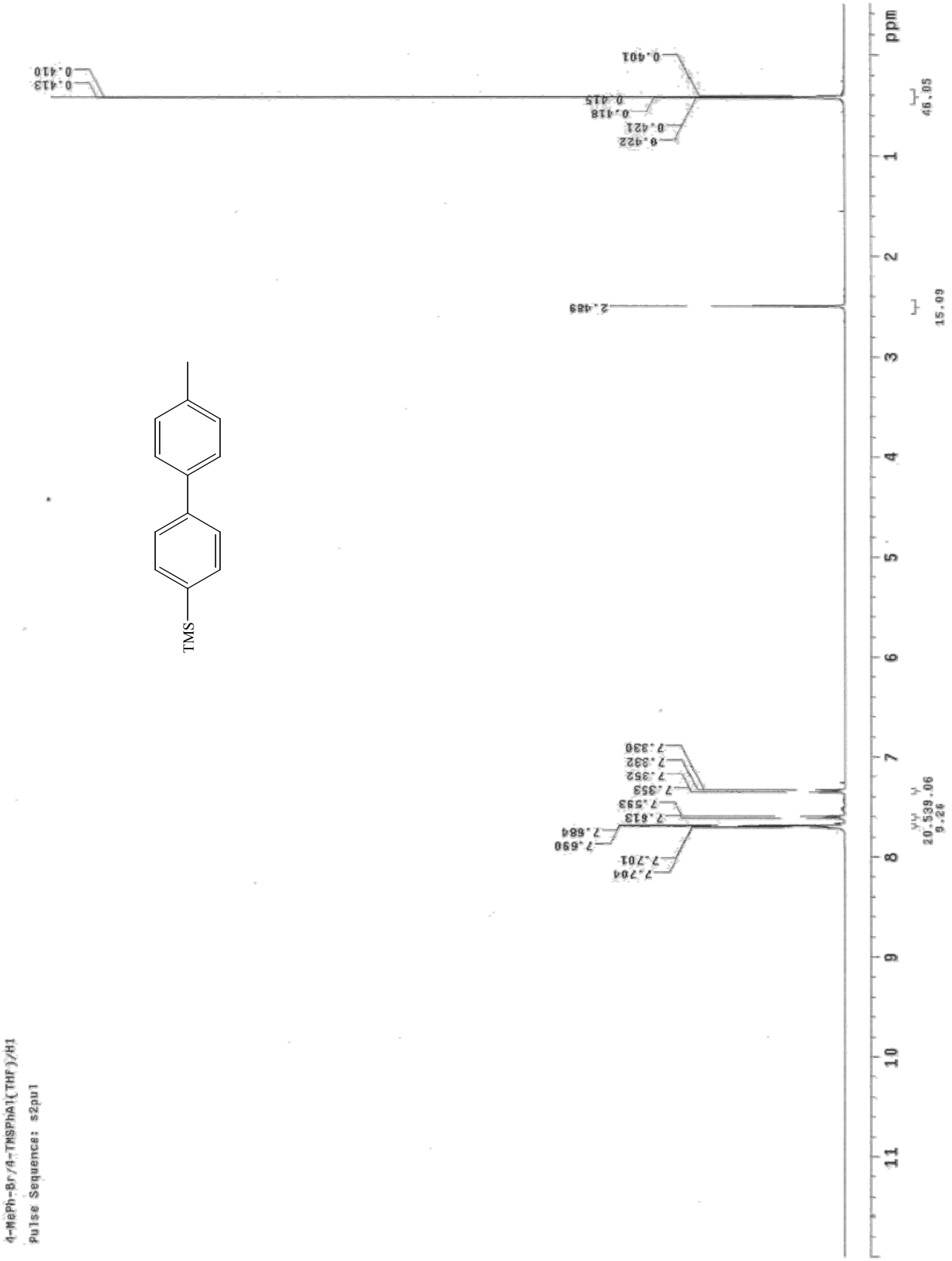

2-methoxy-2,4,6-trimethylbiphenyl
Pulse Sequence: s2pul
ppm
20
40
60
80
100
120
140
160
180
200
20.308
21.097
55.386
76.679
77.000
77.314
110.807
120.611
127.890
128.250
129.505
130.944
135.268
136.439
136.470
156.714

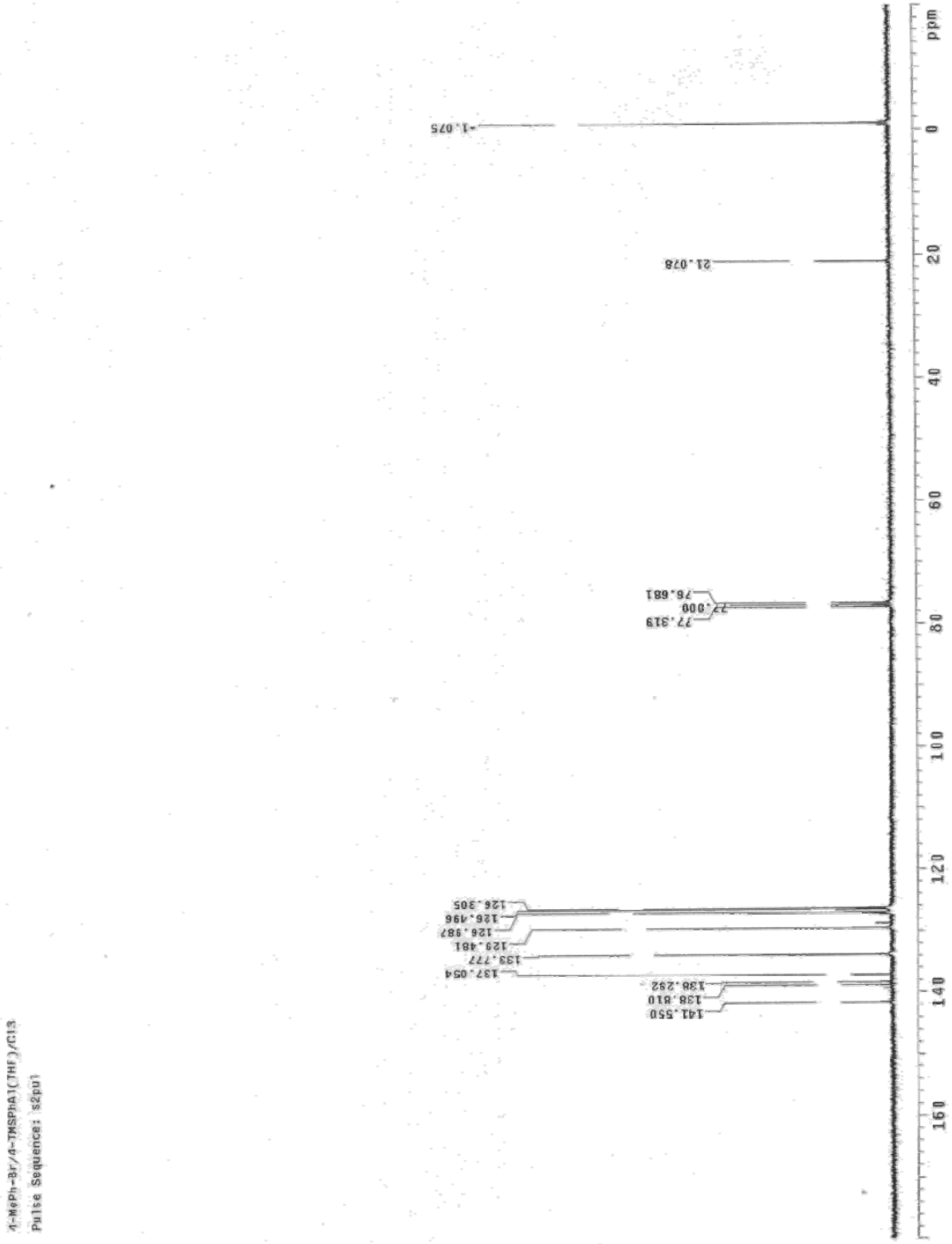

2,2,4,4,6-pentamethylbiphenyl-column
Pulse Sequence: s2pul
7.245
7.091
7.050
7.031
6.929
6.897
6.878
2.364
2.326
1.931
1.814
ppm


2,2,4,4,6-pentamethylbiphenyl
Pulse Sequence: s2pul
ppm
20
40
60
80
100
120
140
160
180
200
19.359
20.255
21.035
21.143
76.686
77.000
77.321
126.704
127.944
128.173
129.023
130.676
135.590
135.927
136.179
136.279
137.534
138.200


2-nap/22-23
Pulse Sequence: s2pul
8.023
8.020
7.910
7.897
7.890
7.874
7.864
7.847
7.842
7.751
7.747
7.730
7.725
7.637
7.633
7.622
7.617
7.499
7.494
7.484
7.479
7.475
7.465
7.461
7.306
7.286
7.250
2.421
1.531
23.11
6.736,924.12
21.15,933.98
ppm


4-MePh-Br/2-napAl(THF)/C13
Pulse Sequence: s2pul
ppm
0
20
40
60
80
100
120
140
160
21.087
77.319
77.000
76.681
125.413
125.540
125.740
126.204
127.242
127.615
128.125
128.325
129.572
132.521
138.501
138.237
137.136
139.741


4-MePh-Br/4-TMSPhAl(THF)/H1
Pulse Sequence: s2pul
TMS
7.704
7.701
7.690
7.684
7.613
7.593
7.353
7.352
7.332
7.330
2.489
0.422
0.421
0.418
0.415
0.401
0.413
0.410
ppm
20.539.06
9.26
15.09
46.05


4-MeOPh-Br/4-TMSPhA1(THF)/C13
Pulse Sequence: s2pul
ppm
160
140
120
100
80
60
40
20
0
141.550
138.810
138.292
137.054
133.777
129.481
126.987
126.196
126.305
77.319
77.000
76.681
21.078
-1.075