Facile *N*-Arylation of Amines and Sulfonamides

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Supporting Information

General. The ^{1}H and ^{13}C NMR spectra were recorded at 300 and 75.5 MHz or 400 and 100 MHz respectively. All melting points are uncorrected. High resolution mass spectra were recorded on a Kratos MS50TC double focusing magnetic sector mass spectrometer using EI at 70 eV. All reagents were used directly as obtained commercially unless otherwise noted. All yields reported in the publication represent an average of at least two independent runs. CsF and acetonitrile were purchased from Sigma-aldrich Co.. The methoxy-substituted silylaryl triflate **1b** was prepared according to a previous literature procedure.[1]

General procedure for the *N*-arylation of amines and sulfonamides.

Procedure A: To a solution of MeCN (4 mL), amine or sulfonamide (0.25 mmol) and silylaryl triflate (0.225 mmol) was added CsF (0.45 mmol). The reaction mixture was allowed to stir at room temperature for 1 d and the resulting solution was washed by brine (20 mL) and extracted with diethyl ether (20 mL). The combined ether fractions were dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by flash chromatography on silica gel to afford the desired product.

Procedure B: To a solution of MeCN (4 mL), amine or sulfonamide (0.25 mmol) and silylaryl triflate (0.6 mmol) was added CsF (1.2 mmol). The reaction mixture was allowed to stir at room temperature for 3 d for *N*-arylated amines and 1 d for *N*-arylated sulfonamides. The resulting solution was washed with brine (20 mL) and extracted with diethyl ether (20 mL). The combined ether fractions were dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by flash chromatography on silica gel to afford the desired product.

Diphenylamine (entry 1, Table 1). Using procedure A, silylaryl triflate **1a** (0.225 mmol, 67 mg), aniline (0.25 mmol, 24 mg) and CsF (0.45 mmol, 69 mg) afforded the indicated compound (31 mg) in an 81 % yield as a white solid: mp 49-50 °C (lit.[2] mp 50-52 °C). The ^{1}H and ^{13}C NMR spectra match the literature data.[3] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 (td, J = 8.0, 1.2 Hz, 4H), 7.05 (dd, J = 8.0, 1.2 Hz, 4H), 6.93 (td, J = 8.0, 1.2 Hz, 2H), 5.61 (br s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.6 (2C), 129.8 (4C), 121.4 (2C), 118.3 (4C).

***N*-Phenyl-3-methoxyaniline (entry 2, Table 1).** Using procedure A, silylaryl triflate **1b** (0.225 mmol, 74 mg), aniline (0.25 mmol, 24 mg) and CsF (0.45 mmol, 69 mg) afforded the indicated compound (38 mg) in an 84 % yield as a colorless oil. The ^{1}H and ^{13}C NMR spectra match the literature data.[4] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.33-7.10 (m, 5H), 6.97 (t, J = 7.4 Hz, 1H), 6.69-6.67 (m, 2H), 6.51 (dd, J = 7.4, 1.9 Hz, 1H), 5.74 (s, 1H), 3.80 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.8 (1C), 144.7 (1C), 142.9 (1C), 130.2 (1C), 129.5 (1C), 121.4 (1C), 118.5 (1C), 110.4 (1C), 106.3 (1C), 103.4 (1C), 55.4 (1C).

Triphenylamine (entry 3, Table 1). Using procedure B, aniline (0.25 mmol, 24 mg), silylaryl triflate **1a** (0.6 mmol, 179 mg) and CsF (1.2 mmol, 182 mg) afforded the indicated compound (60 mg) in a 98 % yield as a white solid: mp 123-124 °C (lit.[5] mp 125-126 °C). The ^{1}H and ^{13}C NMR spectra match the literature data.[6] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.43-7.29 (m, 6H), 7.18-7.15 (m, 6H), 7.11-7.04 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 148.2 (3C), 129.5 (6C), 124.5 (6C), 123.0 (3C).

***N,N-bis*(3-Methoxyphenyl)aniline (entry 4, Table 1).** Using procedure B, aniline (0.25 mmol, 24 mg), silylaryl triflate **1b** (0.6 mmol, 196 mg) and CsF (1.2 mmol, 182 mg) afforded the indicated compound (72 mg) in a 94 % yield as a colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.26-7.21 (m, 2H), 7.16-7.08 (m, 4H), 7.03-6.98 (m, 1H), 6.68-6.62 (m, 4H), 6.57-6.54 (dd, J = 8.1, 0.9 Hz, 2H), 3.71 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.6 (2C), 149.2 (2C), 147.8 (1C), 129.9 (2C), 129.4 (2C), 124.8 (2C), 123.2 (1C), 116.9 (2C), 110.2 (2C), 108.4 (2C), 55.4 (2C). IR ($CDCl_3$) 3000, 2953 cm^{-1}; HRMS m/z 305.1428 (calcd $C_{20}H_{19}NO_2$, 305.1415).

***N*-Phenyl-2-iodoaniline (entry 5, Table 1).** Using procedure A, silylaryl triflate **1a** (0.225 mmol, 67 mg), *o*-iodoaniline (0.25 mmol, 55 mg) and CsF (0.45 mmol, 69 mg) afforded the indicated compound (55 mg) in an 83 % yield as a light yellow oil. ^{1}H NMR

(300 MHz, $CDCl_3$) δ 7.78-7.75 (m, 1H), 7.34-7.28 (m, 2H), 7.20-7.18 (m, 2H), 7.14-7.10 (m, 2H), 7.05-7.00 (m, 1H), 6.64-6.58 (m, 1H), 5.90 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 144.2 (1C), 142.2 (1C), 139.7 (1C), 129.7 (1C), 129.3 (1C), 122.8 (1C), 122.1 (1C), 120.2 (1C), 116.1 (1C), 89.0 (1C). IR ($CDCl_3$) 3379, 3041 cm^{-1}; HRMS m/z 294.9865 (calcd $C_{12}H_{10}IN$, 294.9858).

***N*-Propargylaniline (entry 6, Table 1).** Using procedure A, silylaryl triflate **1a** (0.225 mmol, 67 mg), propargyl amine (0.25 mmol, 14 mg) and CsF (0.45 mmol, 69 mg) afforded the indicated compound (19 mg) in a 62 % yield as a light yellow oil. The ^1H NMR spectrum matchs the literature data.[7] ^1H NMR (300 MHz, $CDCl_3$) δ 7.25-7.18 (m, 2H), 6.81-6.72 (m, 1H), 6.70-6.67 (m, 2H), 3.94 (m, 3H), 2.12 (t, J = 2.4 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 147.1 (1C), 129.4 (2C), 118.9 (1C), 113.7 (2C), 81.2 (1C), 71.5 (1C), 33.9 (1C).

***N*-Phenyl-*N*-propargylaniline (entry 7, Table 1).** Using procedure B, propargyl amine (0.25 mmol, 14 mg), silylaryl triflate **1a** (0.6 mmol, 179 mg) and CsF (1.2 mmol, 182 mg) afforded the indicated compound (40 mg) in a 78 % yield as a light yellow oil. ^1H NMR (300 MHz, $CDCl_3$) δ 7.32-7.23 (m, 4H), 7.10-6.98 (m, 6H), 4.41 (d, J = 2.4 Hz, 2H), 2.22 (t, J = 2.4 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 147.6 (2C), 129.5 (2C), 122.4 (2C), 121.4 (2C), 80.1 (1C), 72.5 (1C), 42.3 (1C). IR ($CDCl_3$) 3289, 3063, 2918 cm^{-1}; HRMS m/z 207.1051 (calcd $C_{15}H_{13}N$, 207.1048).

***N*-(2-Cyclohex-1-en-1-ylethyl)-*N*,*N*-diphenylamine (entry 8, Table 1).** Using procedure B, 2-(cyclohex-1-enyl)ethyl amine (0.25 mmol, 31 mg), silylaryl triflate **1a** (0.6 mmol, 179 mg) and CsF (1.2 mmol, 182 mg) afforded the indicated compound (69 mg) in a 99 % yield as a colorless oil. ^1H NMR (300 MHz, $CDCl_3$) δ 7.28-7.21 (m, 4H), 7.00-6.89 (m, 6H), 5.44 (s, 1H), 3.80-3.75 (m, 2H), 2.31-2.25 (t, J = 1.2 Hz, 2H), 1.97-1.92 (m, 4H), 1.60-1.51 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 148.1 (2C), 135.5 (1C), 129.4 (2C), 123.0 (1C), 121.2 (2C), 121.1 (2C), 51.4 (1C), 35.8 (1C), 28.8 (1C), 25.5 (1C), 23.1 (1C), 22.6 (1C). IR ($CDCl_3$) 3057, 3034, 2925 cm^{-1}; HRMS m/z 277.1835 (calcd $C_{20}H_{23}N$, 277.1830).

3-Diphenylamino-1-propanol (entry 9, Table 1). Using procedure B, 3-amino-1-propanol (0.25 mmol, 19 mg), silylaryl triflate **1a** (0.6 mmol, 179 mg) and CsF (1.2 mmol, 182 mg) afforded the indicated compound (47 mg) in an 83 % yield as a colorless

oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.27-7.20 (m, 4H), 7.02-6.89 (m, 6H), 3.31 (t, J = 7.2 Hz, 2H), 3.68 (t, J = 6.0 Hz, 2H), 1.92-1.83 (m, 2H), 1.73 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 148.3 (2C), 129.5 (2C), 121.6 (1C), 121.3 (2C), 60.8 (1C), 49.3 (1C), 30.6 (1C). IR ($CDCl_3$) 3351, 3041, 2943 cm^{-1}; HRMS m/z 227.1316 (calcd $C_{15}H_{17}NO$, 227.1310).

***N*-*tert*-Butyl-*N*-phenylaniline (entry 10, Table 1).** Using procedure B, *tert*-butylamine (0.25 mmol, 18.5 mg), silylaryl triflate **1a** (0.6 mmol, 179 mg) and CsF (1.2 mmol, 182 mg) afforded the indicated compound (55 mg) in a 97 % yield as a colorless oil. The ^{1}H and ^{13}C NMR spectra match the literature data.[8] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.20-6.94 (m, 10H), 1.40 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 148.9 (2C), 128.9 (2C), 127.1 (2C), 122.5 (2C), 56.0 (1C), 30.5 (3C).

***N*-Methyl-*N*-phenylaniline (entry 11, Table 1).** Using procedure B, *N*-methylaniline (0.25 mmol, 27 mg), silylaryl triflate **1a** (0.3 mmol, 90 mg) and CsF (0.6 mmol, 91 mg) afforded the indicated compound (66 mg) in a 93 % yield as a colorless oil. The ^{1}H and ^{13}C NMR spectra match the literature data.[9] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.33-7.23 (m, 4H), 7.08-6.94 (m, 6H), 3.34 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 149.0 (2C), 129.2 (2C), 121.2 (2C), 120.4 (2C), 40.2 (1C).

***N,N*-Diphenylmethanesulfonamide (entry 12, Table 1).** Using procedure B, methanesulfonamide (0.25 mmol, 24 mg), silylaryl triflate **1a** (0.6 mmol, 179 mg) and CsF (1.2 mmol, 182 mg) afforded the indicated compound (50 mg) in an 80 % yield as a white solid: mp 115-117 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41-7.35 (m, 8H), 7.31-7.25 (m, 2H), 3.16 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.5 (2C), 129.7 (2C), 127.8 (2C), 127.5 (2C), 40.3 (1C). IR ($CDCl_3$) 3071, 2983, 1343 cm^{-1}; HRMS m/z 247.0672 (calcd $C_{13}H_{13}NO_2S$, 247.0667).

***N,N*-Diphenylbenzenesulfonamide (entry 13, Table 1).** Using procedure B, benzenesulfonamide (0.25 mmol, 39 mg), silylaryl triflate **1a** (0.6 mmol, 179 mg) and CsF (1.2 mmol, 182 mg) afforded the indicated compound (77 mg) in a 99 % yield as a white solid: mp 120-121 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.73-7.70 (m, 2H), 7.62-7.45 (m, 3H), 7.34-7.23 (m, 10H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 141.6 (2C), 140.7 (1C), 133.0 (1C), 129.5 (4C), 129.1 (2C), 128.5 (4C), 127.9 (2C), 127.7 (2C). IR ($CDCl_3$) 3061, 1353 cm^{-1}; HRMS m/z 309.0829 (calcd $C_{18}H_{15}NO_2S$, 309.0823).

***N,N-bis*(3-Methoxyphenyl)-4-methylbenzenesulfonamide (entry 14, Table 1).** Using procedure B, *p*-toluenesulfonamide (0.25 mmol, 43 mg), silylaryl triflate **1b** (0.6 mmol, 197 mg) and CsF (1.2 mmol, 182 mg) afforded the indicated compound (88 mg) in a 92 % yield as a colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.62 (d, J = 8.4 Hz, 2H), 7.26 (d, J = 8.1 Hz, 2H), 7.22-7.16 (m, 2H), 6.84-6.77 (m, 6H), 3.73 (s, 6H), 2.41 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.3 (2C), 143.9 (1C), 142.7 (2C), 137.8 (1C), 129.9 (2C), 129.7 (2C), 128.0 (2C), 120.5 (2C), 114.4 (2C), 113.2 (2C), 55.6 (2C), 21.8 (1C). IR ($CDCl_3$) 3052, 2958, 1354 cm^{-1}; HRMS m/z 383.1199 (calcd $C_{21}H_{21}NO_4S$, 383.1191).

***N*-Methyl-*N*-phenyl-*p*-toluenesulfonamide (entry 15, Table 1).** Using procedure B, *N*-methyl-*p*-toluenesulfonamide (0.25 mmol, 47 mg), silylaryl triflate **1a** (0.3 mmol, 89 mg) and CsF (0.5 mmol, 76 mg) afforded the indicated compound (57 mg) in an 87 % yield as a white solid: mp 92-93 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.43-7.40 (m, 2H), 7.32-7.21 (m, 5H), 7.11-7.07 (m, 2H), 3.15 (s, 3H), 2.40 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.8 (1C), 141.8 (1C), 133.7 (1C), 129.5 (2C), 129.0 (2C), 128.1 (2C), 127.4 (1C), 126.8 (2C), 38.3 (1C), 21.7 (1C). IR ($CDCl_3$) 3018, 2918, 1344 cm^{-1}; HRMS m/z 261.0828 (calcd $C_{14}H_{15}NO_2S$, 261.0823).

References

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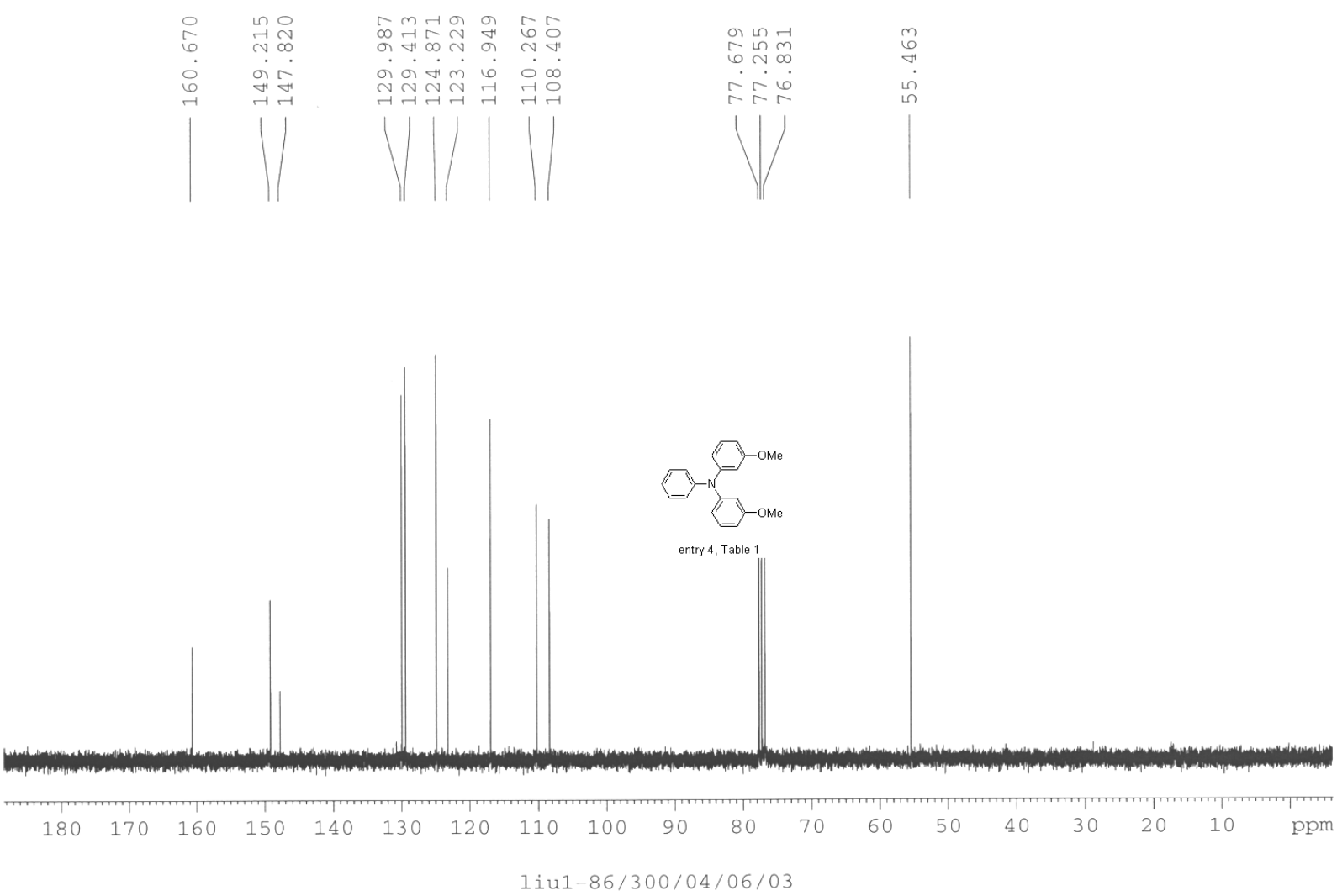
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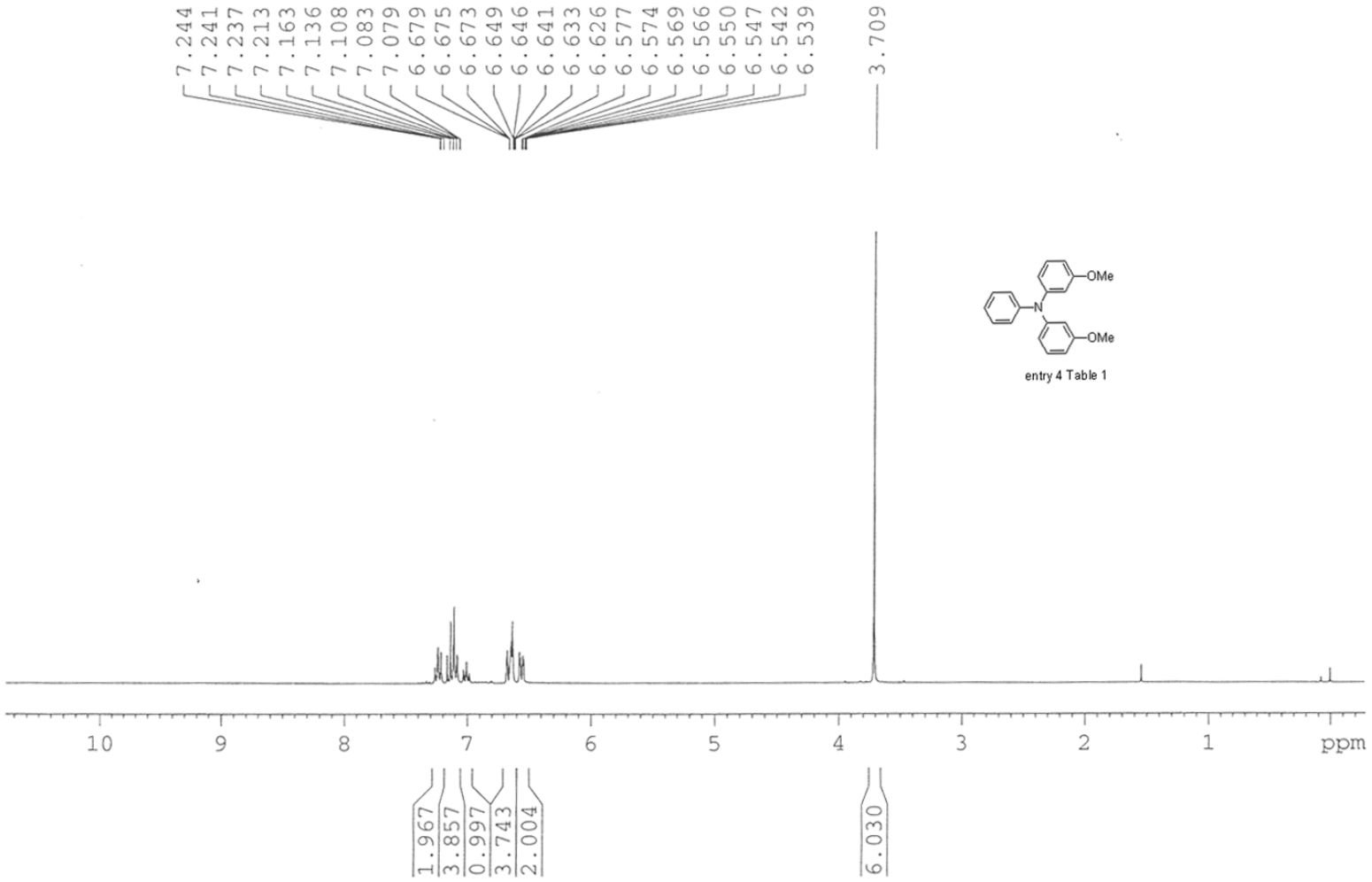
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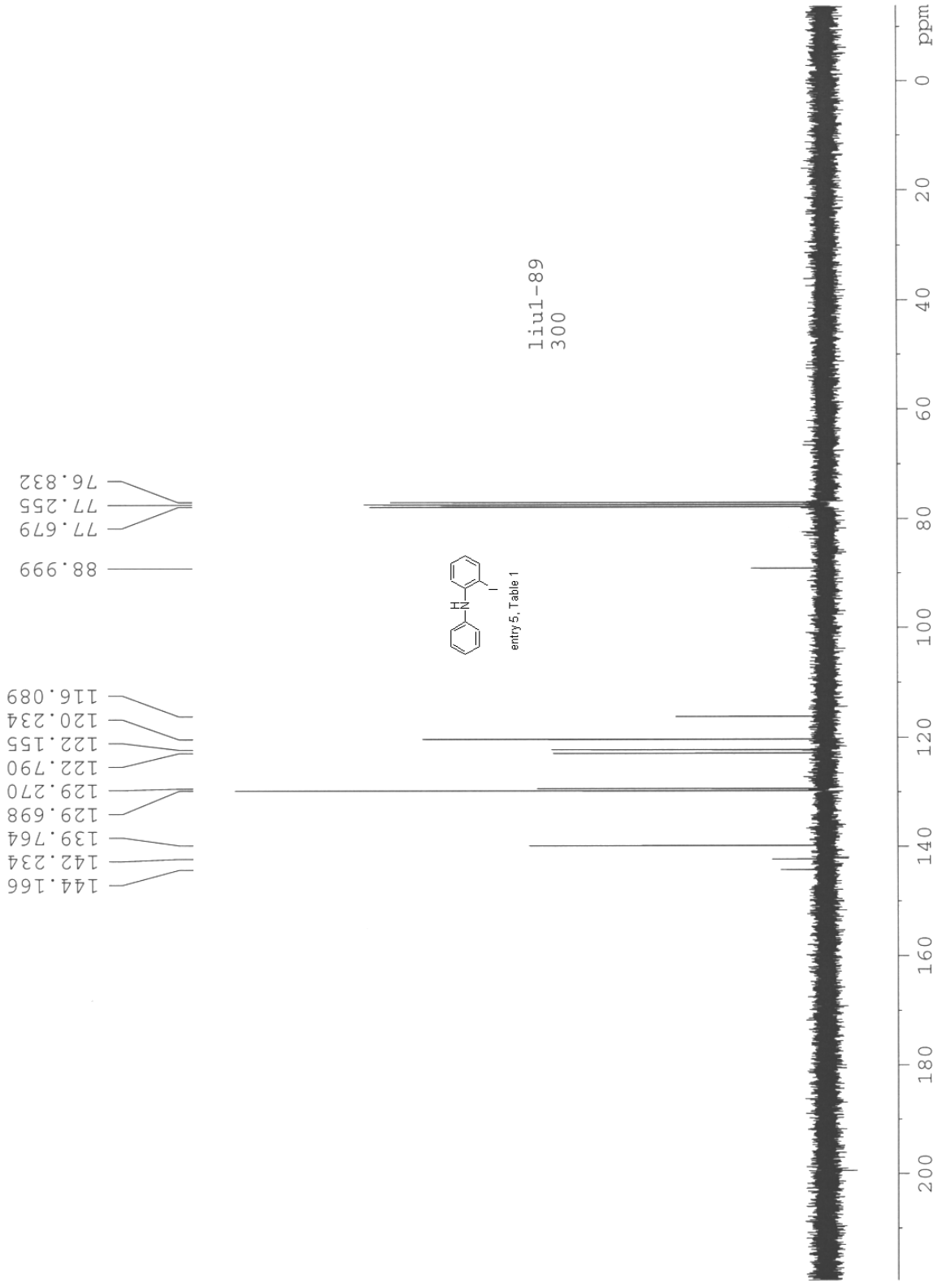
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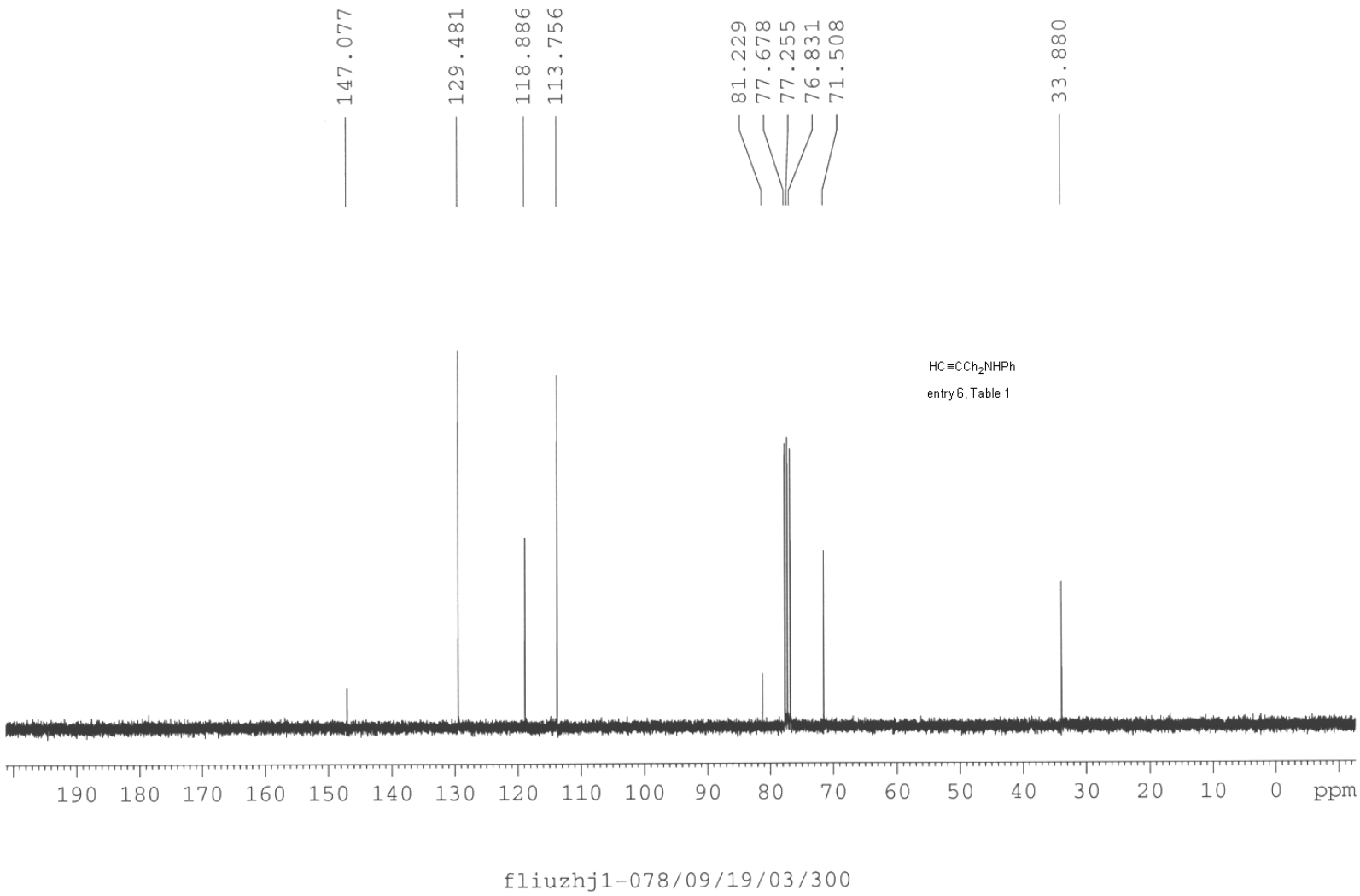
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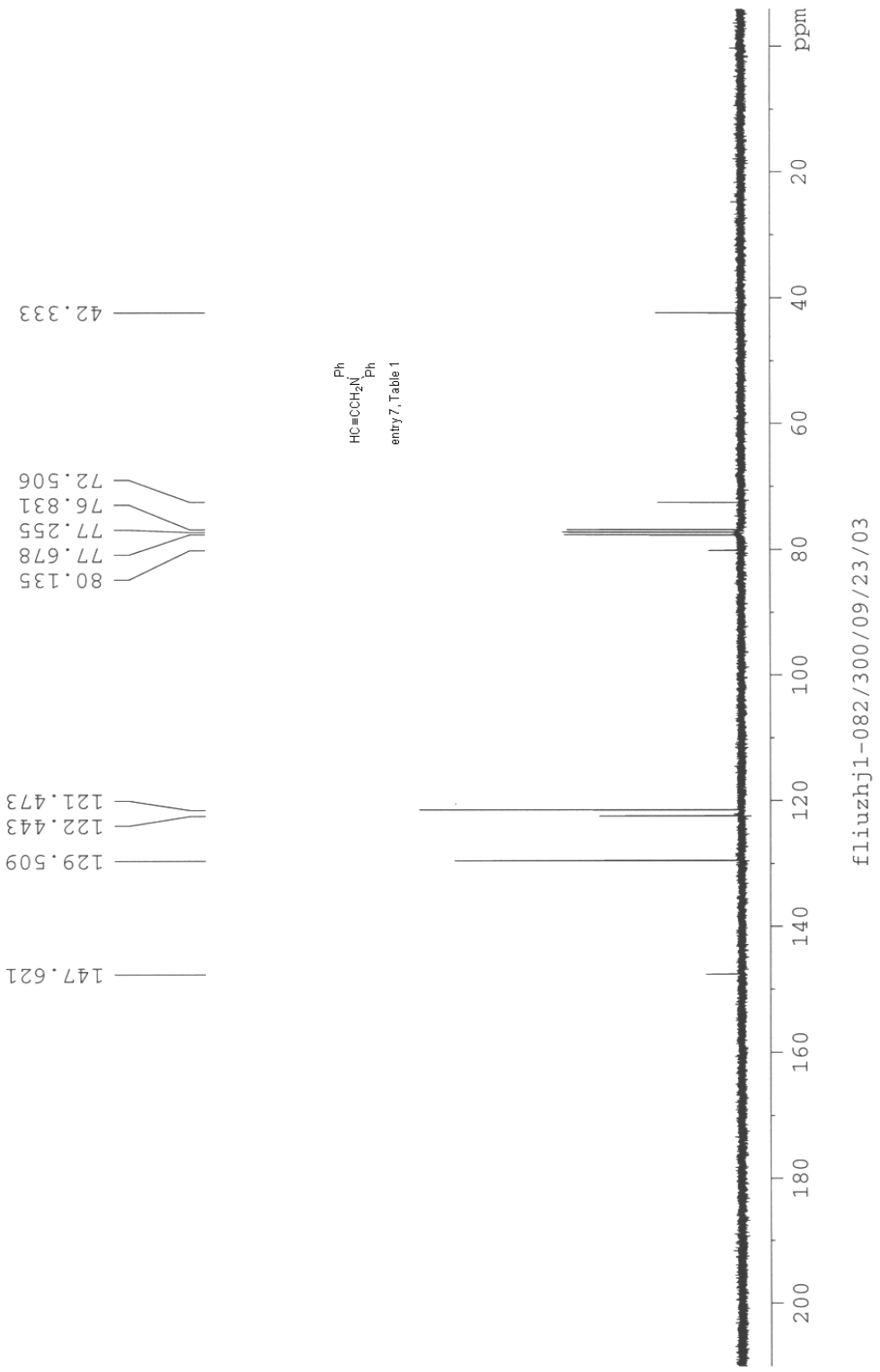
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Ph
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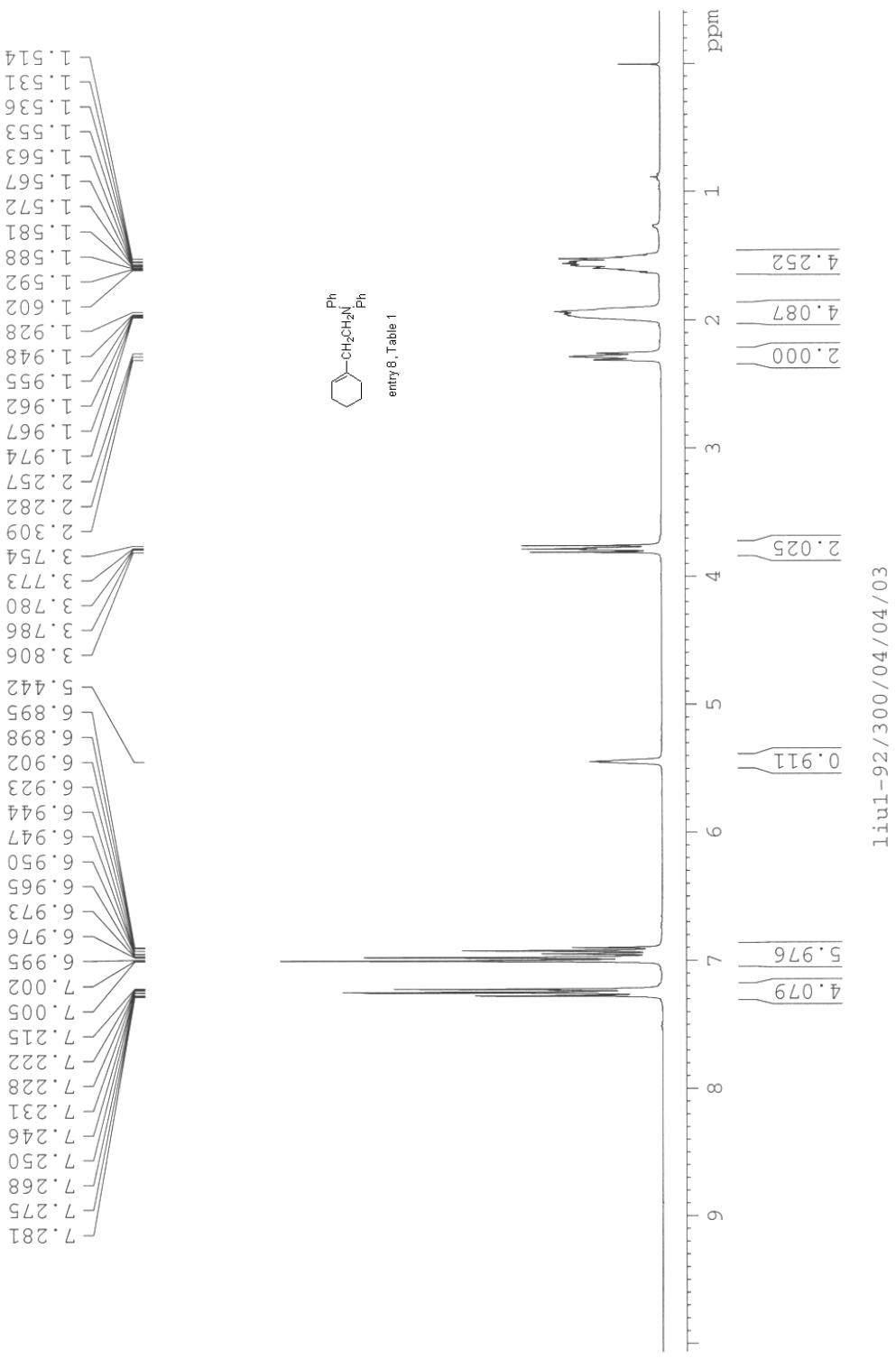
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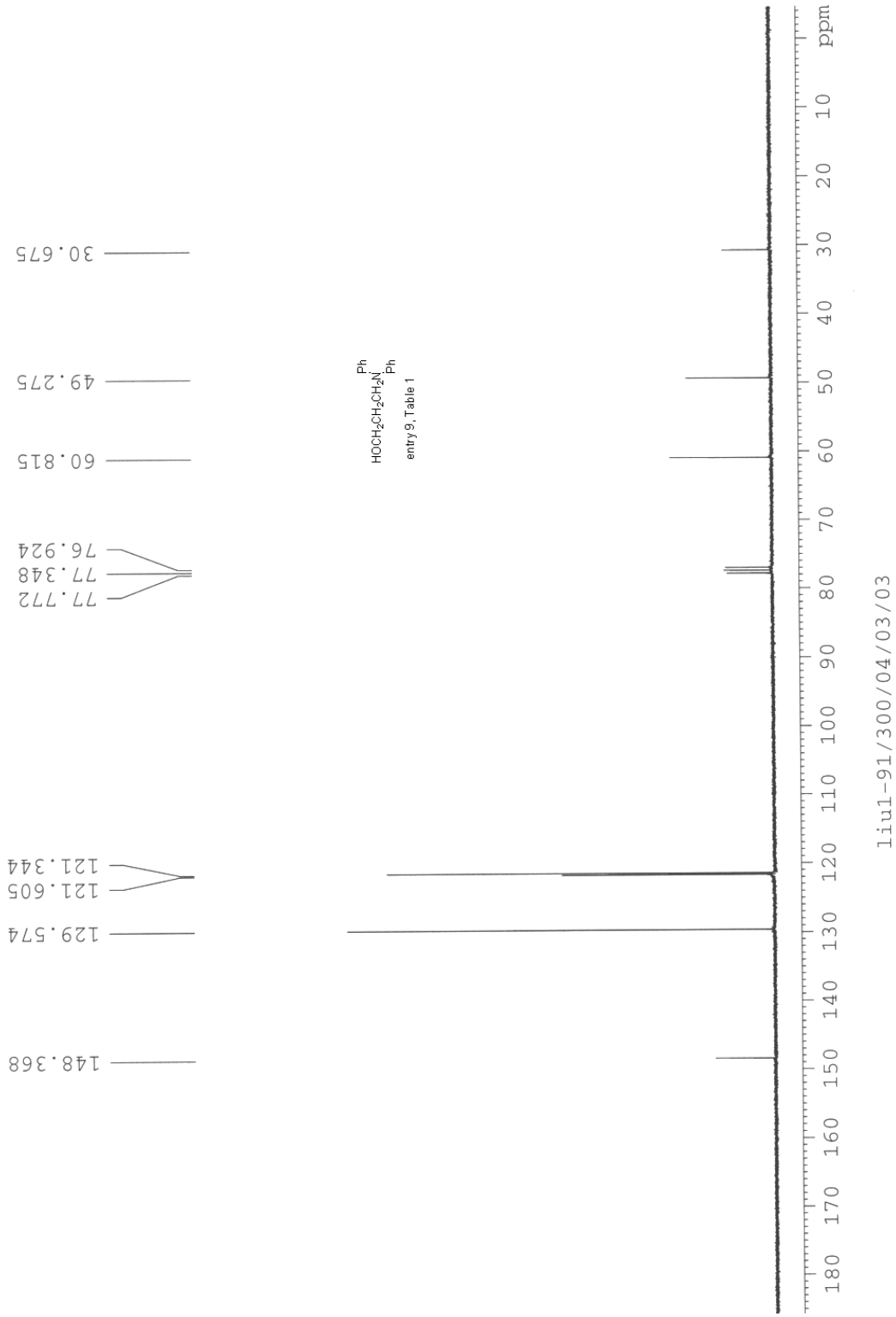
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Ph
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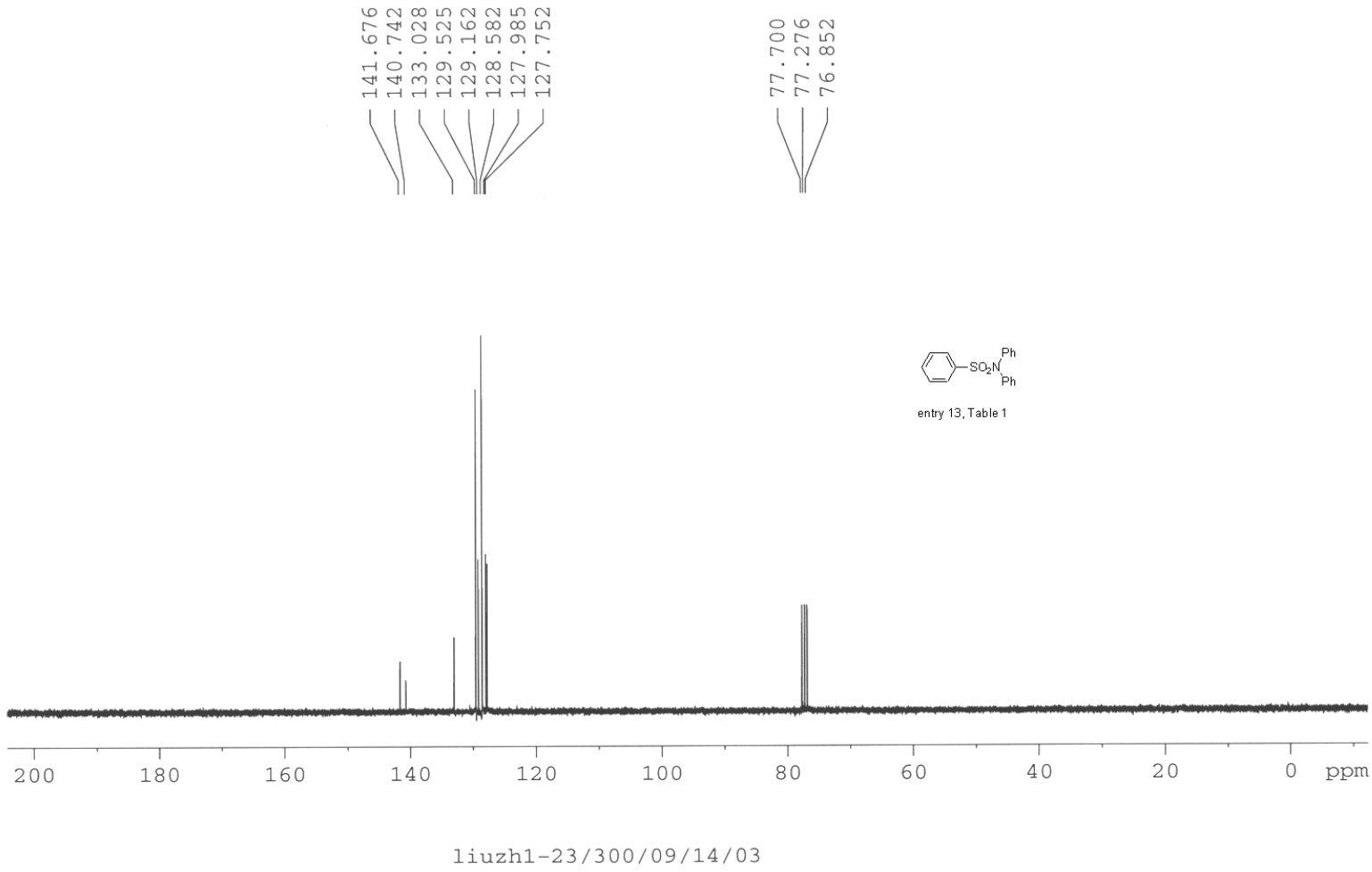
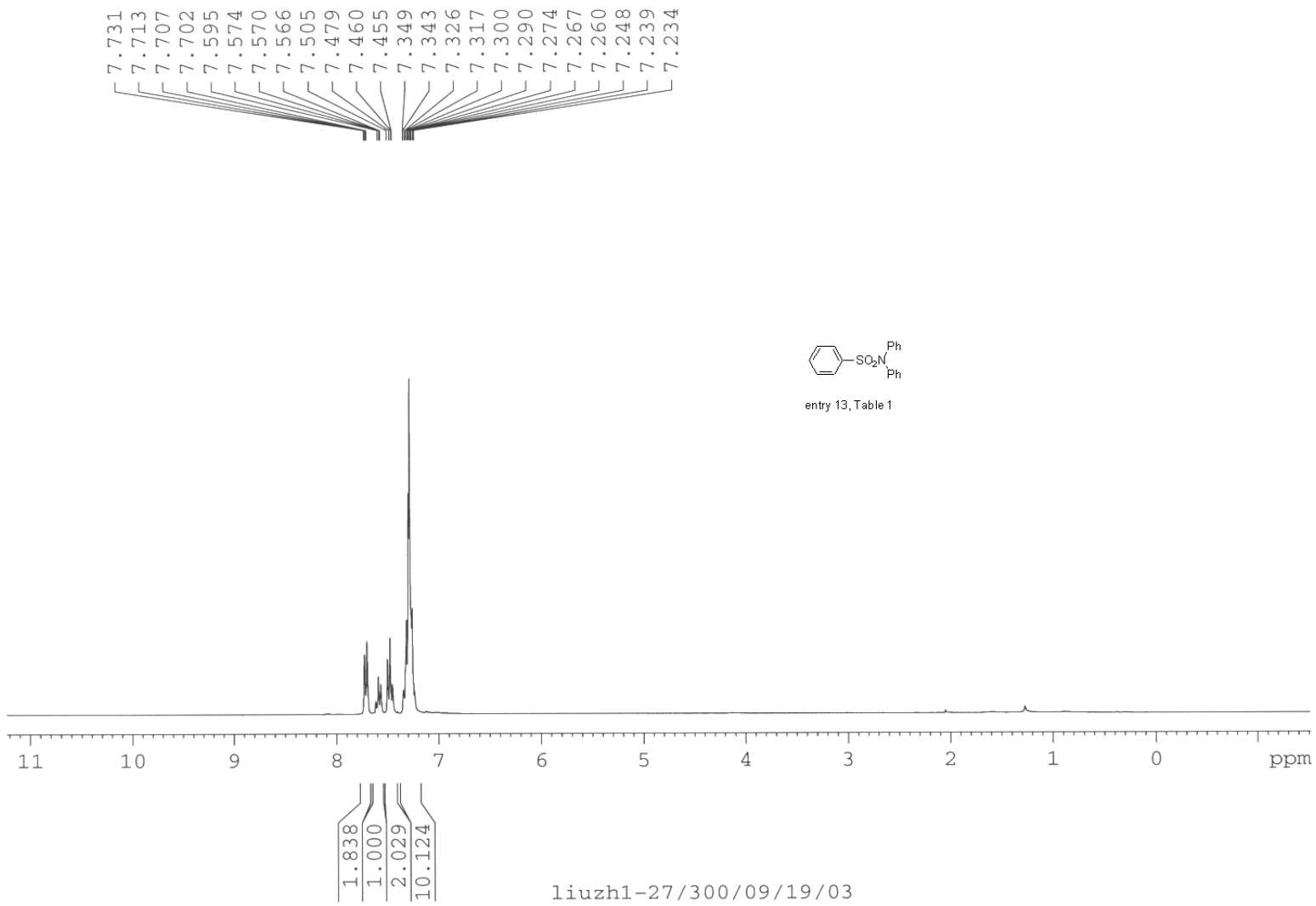
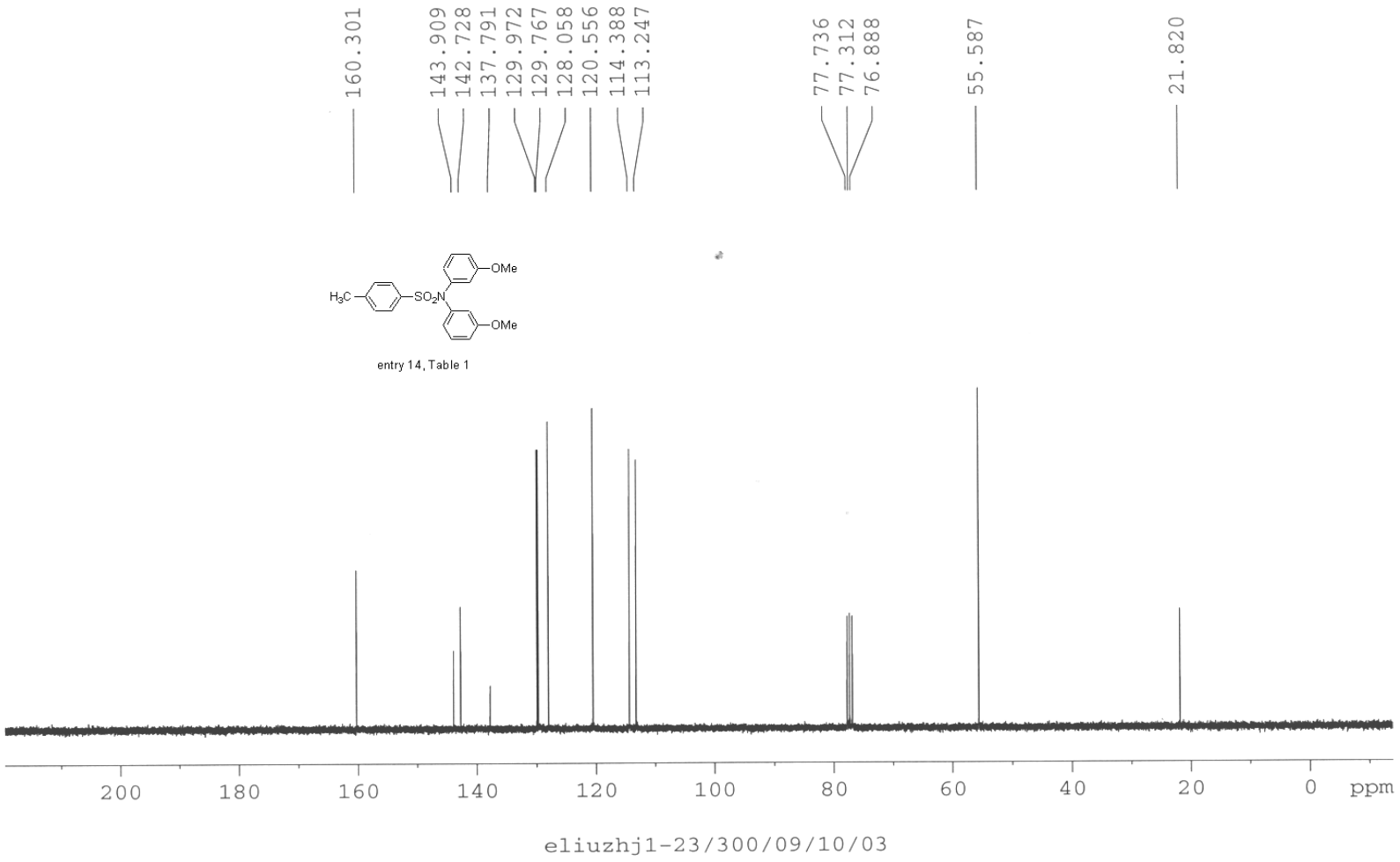
Ph
entry 9, Table 1
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
3.940
5.854
2.040
2.000
2.013
0.979
liu1-90/300/04/03/03

40.284
77.626
77.308
76.990
129.771
127.816
127.536
141.493
Ph
CH3SO2N
Ph
entry 12, Table 1
liuzhj1-30/400/09/19/03
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 ppm

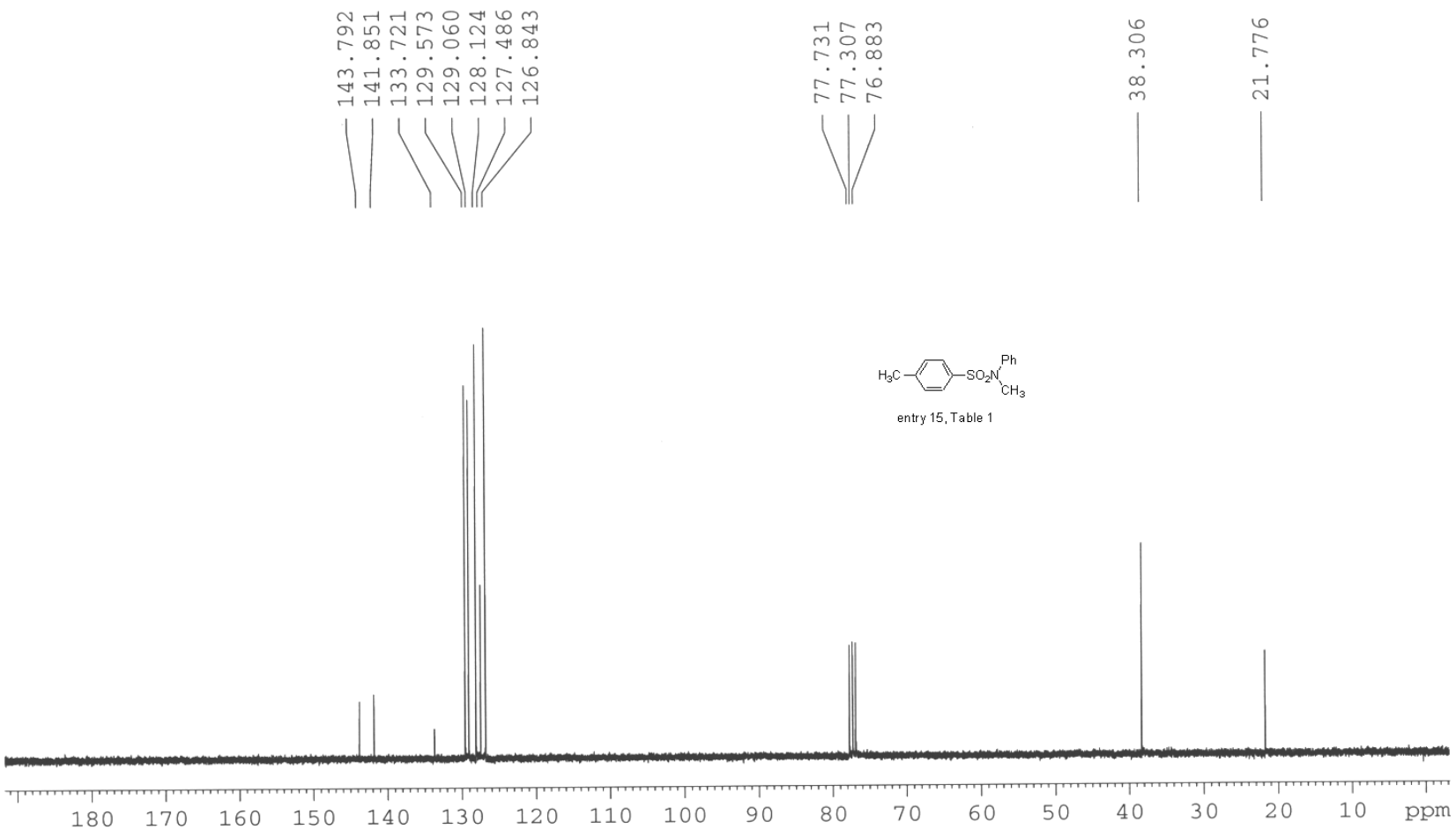
0.000
3.148
3.147
7.399
7.396
7.385
7.383
7.377
7.370
7.353
7.348
7.297
7.296
7.290
7.282
7.279
7.276
7.274
7.268
7.266
7.260
7.255
7.253
7.251
7.249
Ph
CH3SO2N
Ph
entry 12, Table 1
ppm
3.000
7.507
1.844
liuzhj1-29/400/09/14/03


141.676
140.742
133.028
129.525
129.162
128.582
127.985
127.752
77.700
77.276
76.852
Ph
SO2N
Ph
entry 13, Table 1
200
180
160
140
120
100
80
60
40
20
0
ppm
liuzh1-23/300/09/14/03


7.731
7.713
7.707
7.702
7.595
7.574
7.570
7.566
7.505
7.479
7.460
7.455
7.349
7.343
7.326
7.317
7.300
7.290
7.274
7.267
7.260
7.248
7.239
7.234
Ph
SO2N
Ph
entry 13, Table 1
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.838
1.000
2.029
10.124
liuzh1-27/300/09/19/03


160.301
143.909
142.728
137.791
129.972
129.767
128.058
120.556
114.388
113.247
77.736
77.312
76.888
55.587
21.820
OMe
OMe
entry 14, Table 1
200
180
160
140
120
100
80
60
40
20
0
ppm
eliuzhj1-23/300/09/10/03

7.626
7.598
7.274
7.247
7.217
7.216
7.190
7.166
7.162
6.840
6.835
6.812
6.807
6.784
6.782
6.776
3.733
2.415
OMe
OMe
SO2N
H3C
entry 14, Table 1
ppm
0
1
2
3
4
5
6
7
8
9
10
2.000
2.221
2.139
6.267
6.594
3.203
eliuzhj1-22/300/00/10/03


143.792
141.851
133.721
129.573
129.060
128.124
127.486
126.843
77.731
77.307
76.883
38.306
21.776
Ph
H3C
SO2N
CH3
entry 15, Table 1
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
liu1-39/300/09/14/03

0.000
2.409
3.156
7.432
7.410
7.404
7.324
7.314
7.308
7.294
7.289
7.283
7.268
7.261
7.256
7.244
7.217
7.108
7.101
7.095
7.087
7.080
7.075
entry 15, Table 1
ppm
3.215
3.230
2.020
5.293
2.000
liu1-38/300/09/14/03