Facile *O*-Arylation of Phenols and Carboxylic Acids

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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, acetonitrile and silyaryl triflate **1a** were purchased from Sigma-Aldrich Co. The methoxy-substituted silylaryl triflate **1b**[1] and 4,5-dimethyl-substituted silylaryl triflate **1c**[2] was prepared according to a previous literature procedure.

General procedure for the *O*-arylation of phenols and carboxylic acids.

Procedure A: To a solution of MeCN (4 mL), the phenol (0.25 mmol) and the silylaryl triflate (0.375 mmol) was added CsF (0.75 mmol). The reaction mixture was allowed to stir at room temperature for 1 d and 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.

Procedure B: To a solution of MeCN (4 mL), the carboxylic acid (0.25 mmol) and the silylaryl triflate (0.5 mmol) was added CsF (1.0 mmol). The reaction mixture was allowed to stir at room temperature for 1 d and 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.

Diphenyl ether (entry 1, Table 1). Using procedure A, phenol (0.25 mmol, 24 mg), silylaryl triflate **1a** (0.375 mmol, 112 mg) and CsF (0.75 mmol, 114 mg) afforded the indicated compound (39 mg) in a 92 % yield as a colorless oil. The ^{1}H and ^{13}C NMR spectra match the literature data:[3] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.37-7.32 (m, 4H), 7.14-7.08 (m, 2H), 7.05-7.01 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 157.4 (2C), 129.9 (2C), 123.4 (2C), 119.1 (2C).

4-Methoxyphenyl phenyl ether (entry 2, Table 1). Using procedure A, 4-methoxyphenol (0.25 mmol, 31 mg), silylaryl triflate **1a** (0.375 mmol, 112 mg) and CsF (0.75 mmol, 114 mg) afforded the indicated compound (49 mg) in a 98 % 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 7.34-7.29 (m, 2H), 7.08-6.88 (m, 7H), 3.82 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.7 (1C), 156.1 (1C), 150.3 (1C), 129.8 (2C), 122.6 (1C), 121.1 (2C), 117.8 (2C), 115.1 (2C), 55.8 (1C); IR ($CDCl_3$) 3040, 2952, 1589, 1225 cm^{-1}.

3,4-Dimethyphenyl 4-methoxyphenyl ether (entry 3, Table 1). Using procedure A, 4-methoxyphenol (0.25 mmol, 31 mg), silylaryl triflate **1c** (0.375 mmol, 122 mg) and CsF (0.75 mmol, 114 mg) afforded the indicated compound (54 mg) in a 96 % yield as a colorless oil. The ^{1}H and ^{13}C NMR spectra match the literature data:[5] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.04 (d, J = 8.1 Hz, 1H), 6.95-6.81(m, 4H), 6.77-6.65 (m, 2H), 3.76 (s, 3H), 2.20 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.1 (1C), 155.4 (1C), 150.6 (1C), 137.9 (1C), 130.5 (1C), 130.1 (1C), 120.1 (2C), 119.1 (1C), 115.0 (1C), 114.7 (2C), 55.3 (1C), 19.7 (1C), 18.7 (1C); IR ($CDCl_3$) 2931, 2837, 1506, 1497, 1284, 1178 cm^{-1}.

4-Phenoxybenzaldehyde (entry 4, Table 1). Using procedure A, 4-hydroxybenzaldehyde (0.25 mmol, 31 mg), silylaryl triflate **1a** (0.375 mmol, 112 mg) and CsF (0.75 mmol, 114 mg) afforded the indicated compound (45 mg) in a 91 % yield as a colorless oil. The ^{1}H and ^{13}C NMR spectra match the literature data:[6] ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.92 (s, 1H), 7.86-7.82 (m, 2H), 7.44-7.38 (m, 2H), 7.25-7.20 (m, 1H), 7.10-7.04 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 190.9 (1C), 163.4 (1C), 155.3 (1C), 132.1 (2C), 131.5 (1C), 130.3 (2C), 125.1 (2C), 120.6 (2C), 117.8 (2C); IR ($CDCl_3$) 3061, 2827, 1694, 1584, 1488 cm^{-1}; HRMS m/z 198.0683 (calcd $C_{13}H_{10}O_2$, 198.0680).

4-Nitrophenyl phenyl ether (entry 5, Table 1). Using procedure A, 4-nitrophenol (0.25 mmol, 35 mg), silylaryl triflate **1a** (0.375 mmol, 112 mg) and CsF (0.75 mmol, 114 mg)

afforded the indicated compound (52 mg) in a 96 % yield as a yellow solid: mp 58-59 °C (lit.[7] mp 58-59 °C). The ^{1}H NMR spectrum matchs the literature data:[8] ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.21-8.18 (m, 2H), 7.46-7.41 (m, 2H), 7.28-7.25 (m, 1H), 7.11-7.07 (m, 2H), 7.02-6.99 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 163.6 (1C), 154.9 (1C), 142.8 (1C), 130.5 (2C), 126.2 (2C), 125.6 (1C), 120.7 (2C), 117.3 (2C); IR ($CDCl_3$) 3110, 3076, 1582, 1486, 1247 cm^{-1}; HRMS m/z 215.2042 (calcd $C_{12}H_9NO_3$, 215.2047).

3-Methoxyphenyl 4-nitrophenyl ether (entry 6, Table 1). Using procedure A, 4-nitrophenol (0.25 mmol, 35 mg), silylaryl triflate **1b** (0.375 mmol, 123 mg) and CsF (0.75 mmol, 114 mg) afforded the indicated compound (59 mg) in a 96 % yield as a white solid: mp 86-87 °C (lit.[9] mp 85 °C); ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.21-8.17 (m, 2H), 7.32 (t, J = 8.1 Hz, 1H), 7.05-7.00 (m, 2H), 6.82-6.78 (m, 1H), 6.68-6.65 (m, 2H), 3.81 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 163.4 (1C), 161.5 (1C), 156.0 (1C), 142.9 (1C), 130.9 (1C), 126.1 (2C), 117.4 (2C), 112.7 (1C), 111.2 (1C), 106.7 (1C), 55.7 (1C); IR ($CDCl_3$) 3077, 2941, 1592, 1486, 1345 cm^{-1}; HRMS m/z 245.0691 (calcd $C_{13}H_{11}NO_4$, 245.0688).

4-Iodophenyl phenyl ether (entry 7, Table 1). Using procedure A, 4-iodophenol (0.25 mmol, 55 mg), silylaryl triflate **1a** (0.375 mmol, 112 mg) and CsF (0.75 mmol, 114 mg) afforded the indicated compound (70 mg) in a 95 % yield as a white solid: mp 47-48 °C (lit.[10] mp 48 °C); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.62-7.58 (m, 2H), 7.37-7.31 (m, 2H), 7.14-7.09 (m, 1H), 7.01-6.98 (m, 2H), 6.78-6.74 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 157.7 (1C), 156.7 (1C), 138.8 (2C), 130.1 (2C), 124.0 (1C), 121.1 (2C), 119.4 (2C), 86.1 (1C); IR ($CDCl_3$) 3040, 1571, 1477, 1239 cm^{-1}; HRMS m/z 295.9705 (calcd $C_{12}H_9IO$, 295.9698).

4-Iodophenyl 3-methoxyphenyl ether (entry 8, Table 1). Using procedure A, 4-iodophenol (0.25 mmol, 55 mg), silylaryl triflate **1b** (0.375 mmol, 123 mg) and CsF (0.75 mmol, 114 mg) afforded the indicated compound (76 mg) in a 94 % yield as a colorless oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.62-7.59 (m, 2H), 7.25-7.20 (m, 1H), 6.79-6.76 (m, 2H), 6.69-6.65 (m, 1H), 6.59-6.55 (m, 2H), 3.77 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 161.2 (1C), 158.0 (1C), 157.4 (1C), 138.8 (2C), 130.5 (1C), 121.2 (2C), 111.4 (1C), 109.6 (1C), 105.3 (1C), 86.3 (1C), 55.6 (1C); IR ($CDCl_3$) 3062, 2956, 2833, 1580, 1479, 1272 cm^{-1}; HRMS m/z 325.9792 (calcd $C_{13}H_{11}IO_2$, 325.9803).

***o*-Diphenoxybenzene (entry 9, Table 1).** Using procedure A, 1,2-benzenediol (0.25 mmol, 28 mg), silylaryl triflate **1a** (0.75 mmol, 223 mg) and CsF (1.5 mmol, 228 mg) afforded the indicated compound (47 mg) in a 72 % yield as a white solid: mp 88-90 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.29-7.23 (m, 4H), 7.13-7.00 (m, 6H), 6.92-6.88 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 157.7 (2C), 148.0 (2C), 129.7 (4C), 124.9 (2C), 123.0 (2C), 121.8 (2C), 117.8 (4C); IR ($CDCl_3$) 3063, 3038, 1583, 1487, 1262 cm^{-1}; HRMS m/z 262.0999 (calcd $C_{18}H_{14}O_2$, 262.0994).

Phenyl 2,4,6-trimethylphenyl ether (entry 10, Table 1). Using procedure A, 2,4,6-trimethylphenol (0.25 mmol, 34 mg), silylaryl triflate **1a** (0.375 mmol, 112 mg) and CsF (0.75 mmol, 114 mg) afforded the indicated compound (36 mg) in a 68 % yield as a colorless oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.26-7.21 (m, 2H), 6.97-6.89 (m, 3H), 6.77-6.74 (m, 2H), 2.30 (s, 3H), 2.08 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.2 (1C), 149.0 (1C), 134.6 (1C), 131.3 (2C), 129.8 (2C), 129.7 (2C), 121.3 (1C), 114.8 (2C), 21.0 (1C), 16.4 (2C); IR ($CDCl_3$) 3025, 2919, 1594, 1482, 1223 cm^{-1}; HRMS m/z 212.1204 (calcd $C_{15}H_{16}O$, 212.1201).

Phenyl benzoate (entry 11, Table 1). Using procedure B, benzoic acid (0.25 mmol, 31 mg), silylaryl triflate **1a** (0.5 mmol, 149 mg) and CsF (1.0 mmol, 152 mg) afforded the indicated compound (40 mg) in an 81 % yield as a white solid: mp 69-70 ^{o}C (lit.[11] mp 68-70 ^{o}C). The ^{1}H and ^{13}C NMR spectra match the literature data:[12] ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.23-8.19 (m, 2H), 7.63 (t, J = 7.2 Hz, 1H), 7.53-7.41 (m, 4H), 7.29-7.20 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.4 (1C), 151.2 (1C), 133.8 (1C), 130.4 (2C), 129.8 (1C), 129.7 (2C), 128.8 (2C), 126.1 (1C), 121.9 (2C); IR ($CDCl_3$) 3068, 1730 cm^{-1}.

Phenyl 4-nitrobenzoate (entry 12, Table 1). Using procedure B, 4-nitrobenzoic acid (0.25 mmol, 42 mg), silylaryl triflate **1a** (0.5 mmol, 149 mg) and CsF (1.0 mmol, 152 mg) afforded the indicated compound (40 mg) in a 66 % yield as a white solid: mp 127-128 ^{o}C (lit.[13] mp 128-129 ^{o}C); ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.40-8.33 (m, 4H), 7.48-7.43 (m, 2H), 7.34-7.22 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 163.5 (1C), 151.1 (1C), 150.7 (1C), 135.2 (1C), 131.5 (2C), 129.9 (2C), 126.6 (1C), 123.9 (2C), 121.6 (2C); IR ($CDCl_3$) 3111, 1740 cm^{-1}; HRMS m/z 243.0536 (calcd $C_{13}H_9NO_4$, 243.05316).

Phenyl 4-methoxybenzoate (entry 13, Table 1). Using procedure B, 4-methoxybenzoic acid (0.25 mmol, 38 mg), silylaryl triflate **1a** (0.5 mmol, 149 mg) and CsF (1.0 mmol,

152 mg) afforded the indicated compound (55 mg) in a 96 % yield as a white solid: mp 67-69 °C (lit.[14] mp 67-69 °C). The ^{1}H and ^{13}C NMR spectra match the literature data:[15] ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17-8.13 (m, 2H), 7.43-7.39 (m, 2H), 7.27-7.18 (m, 3H), 6.99-6.95 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.1 (1C), 164.1 (1C), 151.3 (1C), 132.5 (2C), 129.6 (2C), 125.9 (1C), 122.1 (1C), 122.0 (2C), 114.0 (2C), 55.7 (1C); IR ($CDCl_3$) 3058, 2967, 2839, 1727, 1278 cm^{-1}; HRMS m/z 228.0789 (calcd $C_{14}H_{12}O_3$, 228.0786).

3-Methoxyphenyl 4-methoxybenzoate (entry 14, Table 1). Using procedure B, 4-methoxybenzoic acid (0.25 mmol, 38 mg), silylaryl triflate **1b** (0.5 mmol, 164 mg) and CsF (1.0 mmol, 152 mg) afforded the indicated compound (63 mg) in a 98 % yield as a white solid: mp 102-103 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.16-8.12 (m, 2H), 7.30 (t, J = 8.1 Hz, 1H), 6.98-6.95 (m, 2H), 6.82-6.75 (m, 3H), 3.88 (s, 3H), 3.80 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.0 (1C), 164.1 (1C), 160.7 (1C), 152.3 (1C), 132.5 (2C), 130.0 (1C), 122.0 (1C), 114.2 (1C), 114.0 (2C), 111.9 (1C), 107.9 (1C), 55.7 (1C), 55.6 (1C); IR ($CDCl_3$) 3006, 2937, 2838, 1726, 1605, 1255 cm^{-1}; HRMS m/z 258.0895 (calcd $C_{15}H_{14}O_4$, 258.0892).

3-Methoxyphenyl 4-bromobenzoate (entry 15, Table 1). Using procedure B, 4-bromobenzoic acid (0.25 mmol, 50 mg), silylaryl triflate **1b** (0.5 mmol, 164 mg) and CsF (1.0 mmol, 152 mg) afforded the indicated compound (68 mg) in an 89 % yield as a white solid: mp 59-61 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.07-8.03 (m, 2H), 7.67-7.63 (m, 2H), 7.32 (t, J = 8.1 Hz, 1H), 6.85-6.76 (m, 3H), 3.23 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 164.6 (1C), 160.8 (1C), 151.9 (1C), 132.2 (2C), 131.8 (2C), 130.1 (1C), 129.0 (1C), 128.7 (1C), 114.0 (1C), 112.2 (1C), 107.8 (1C), 55.7 (1C); IR ($CDCl_3$) 2933, 2834, 1729, 1585, 1254 cm^{-1}; HRMS m/z 305.9897 (calcd $C_{14}H_{11}BrO_3$, 305.9891).

Phenyl *o*-iodobenzoate (entry 16, Table 1). Using procedure B, 2-iodobenzoic acid (0.25 mmol, 62 mg), silylaryl triflate **1a** (0.5 mmol, 149 mg) and CsF (1.0 mmol, 152 mg) afforded the indicated compound (63 mg) in a 78 % yield as a colorless oil. The ^{1}H and ^{13}C NMR spectra match the literature data:[16] ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.08-8.02 (m, 2H), 7.51-7.41 (m, 3H), 7.31-7.19 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.1 (1C), 150.9 (1C), 141.9 (1C), 134.4 (1C), 133.4 (1C), 131.7 (1C), 129.7 (2C), 128.3 (1C),

126.3 (1C), 121.8 (2C), 94.8 (1C); IR ($CDCl_3$) 3063, 1743 cm^{-1}; HRMS m/z 323.9653 (calcd $C_{13}H_9IO_2$, 323.9647).

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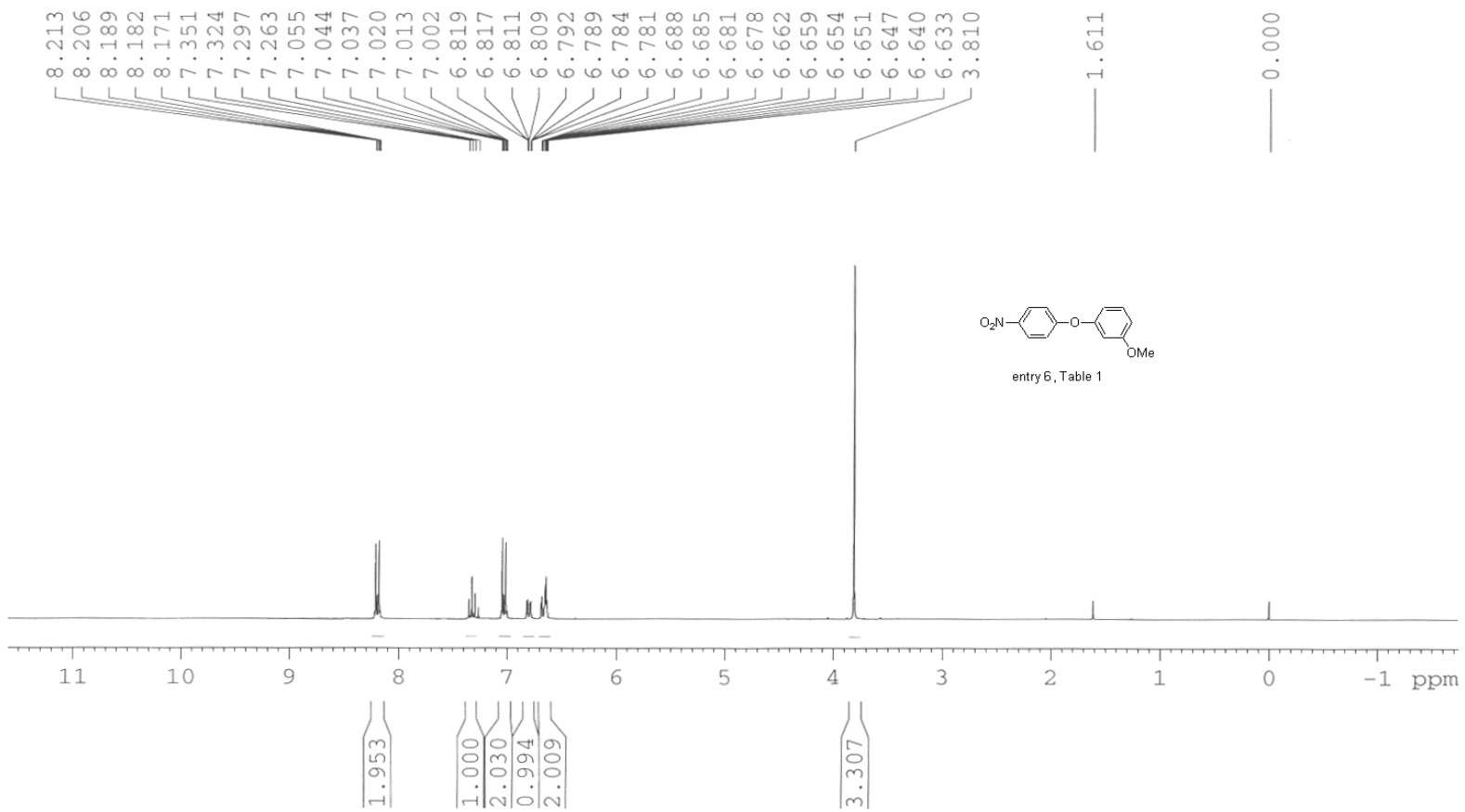

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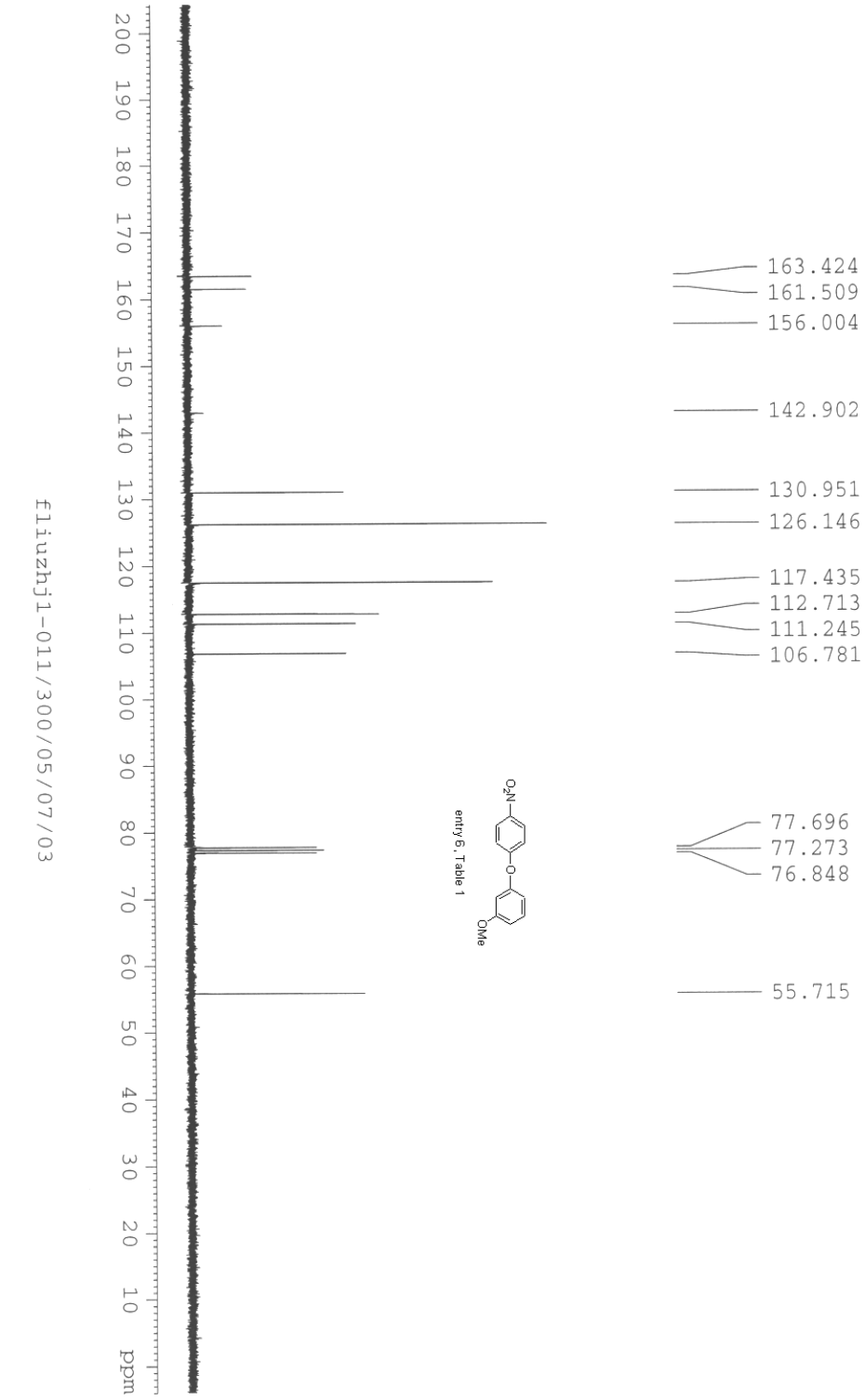
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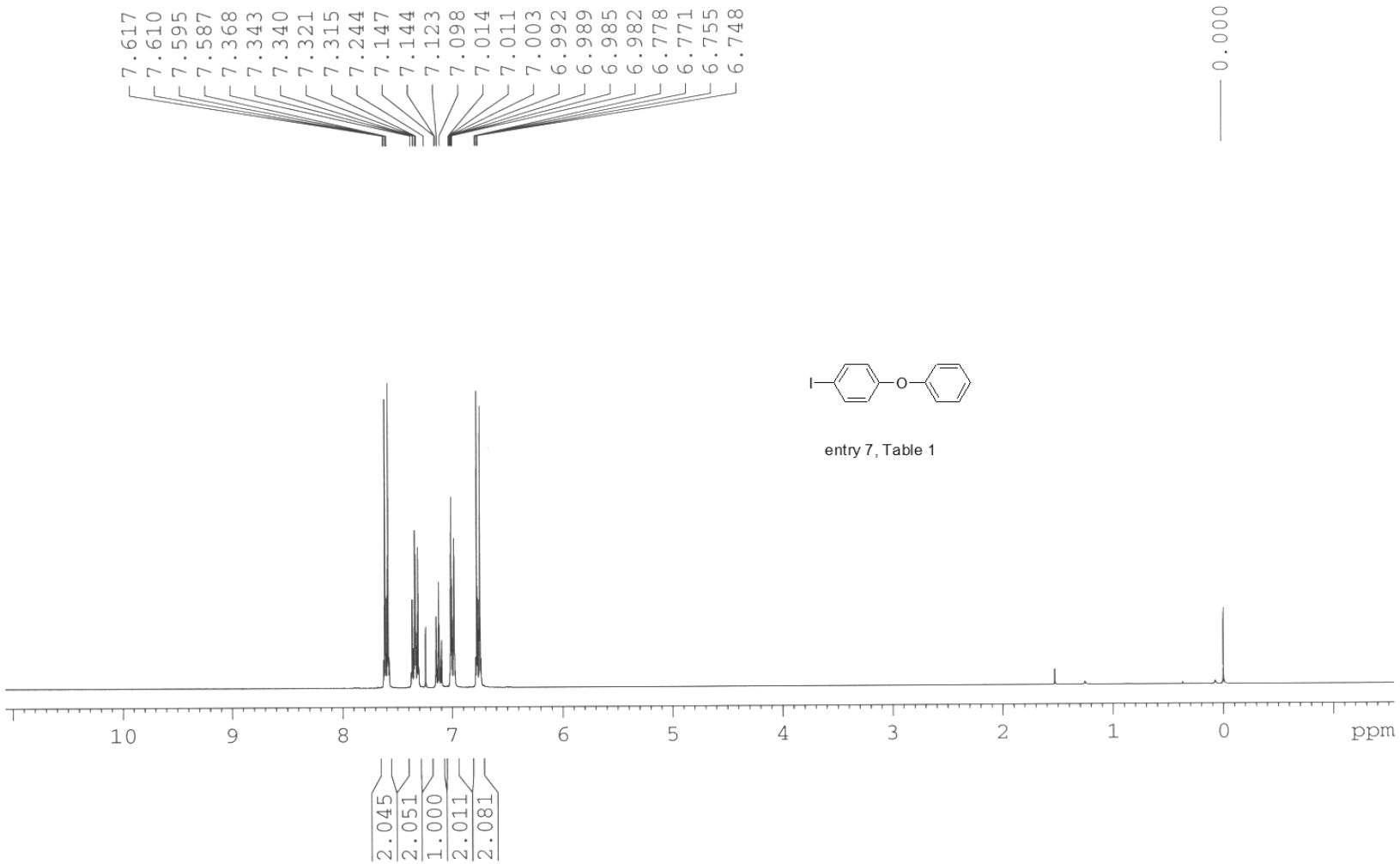
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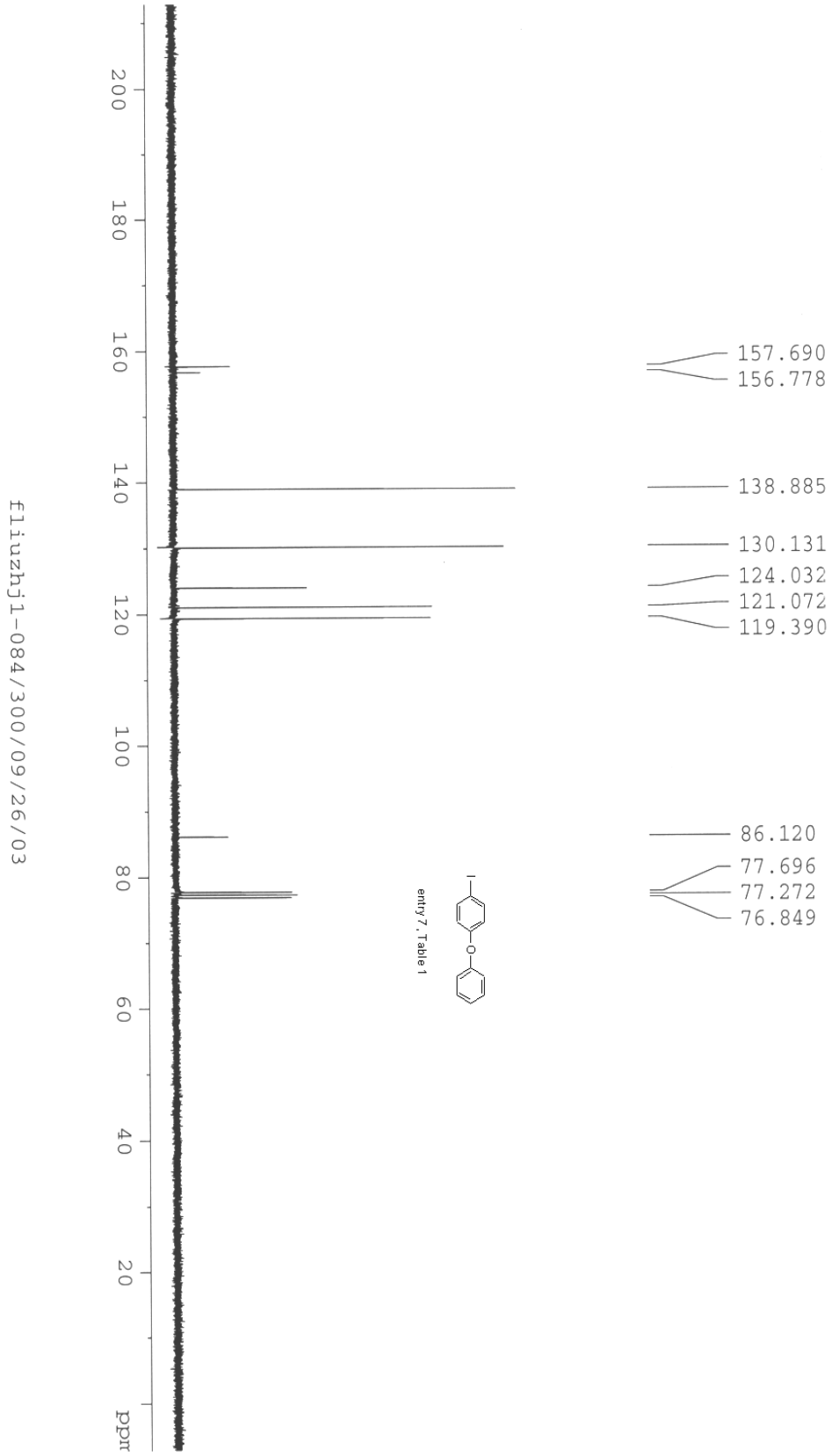
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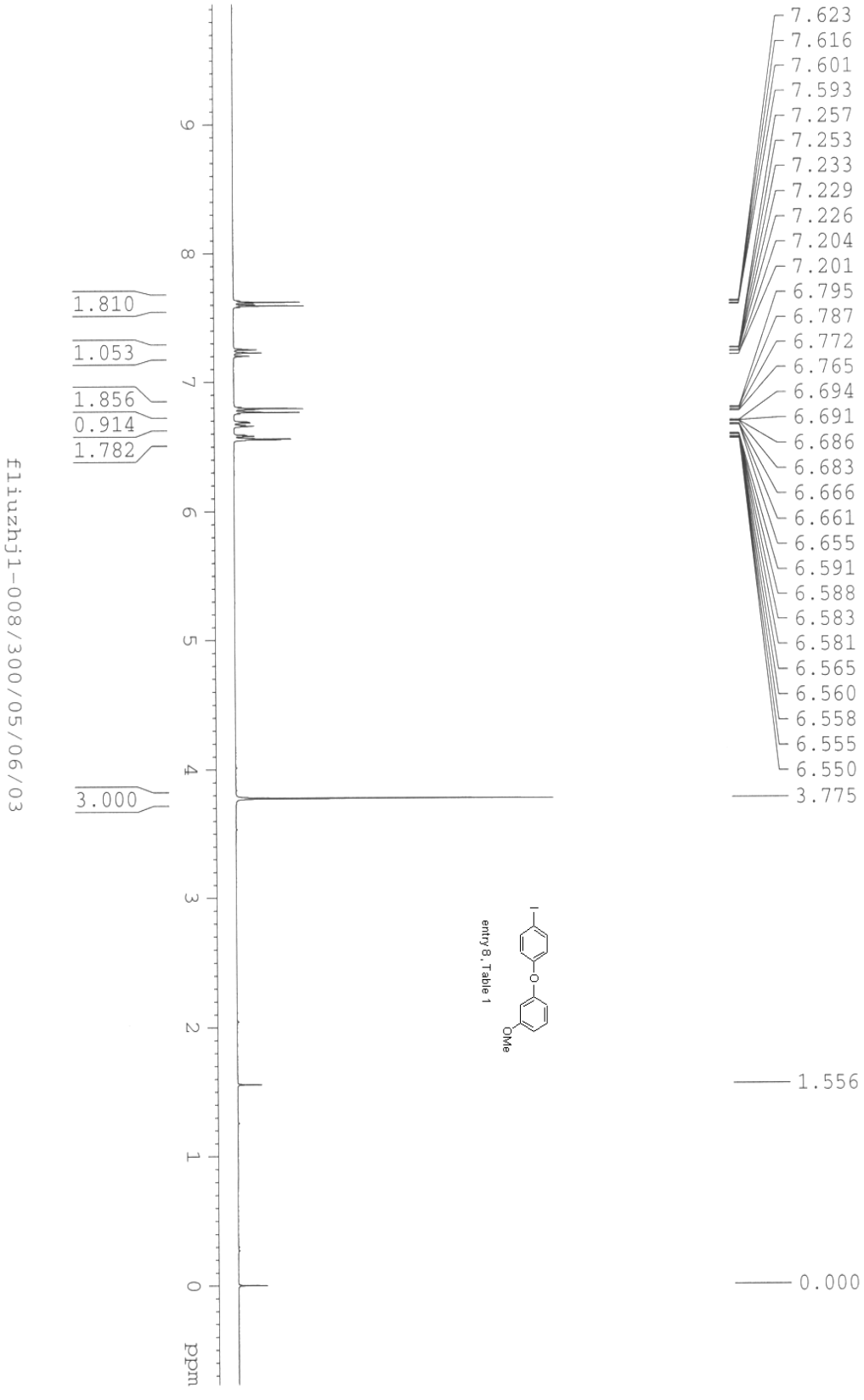
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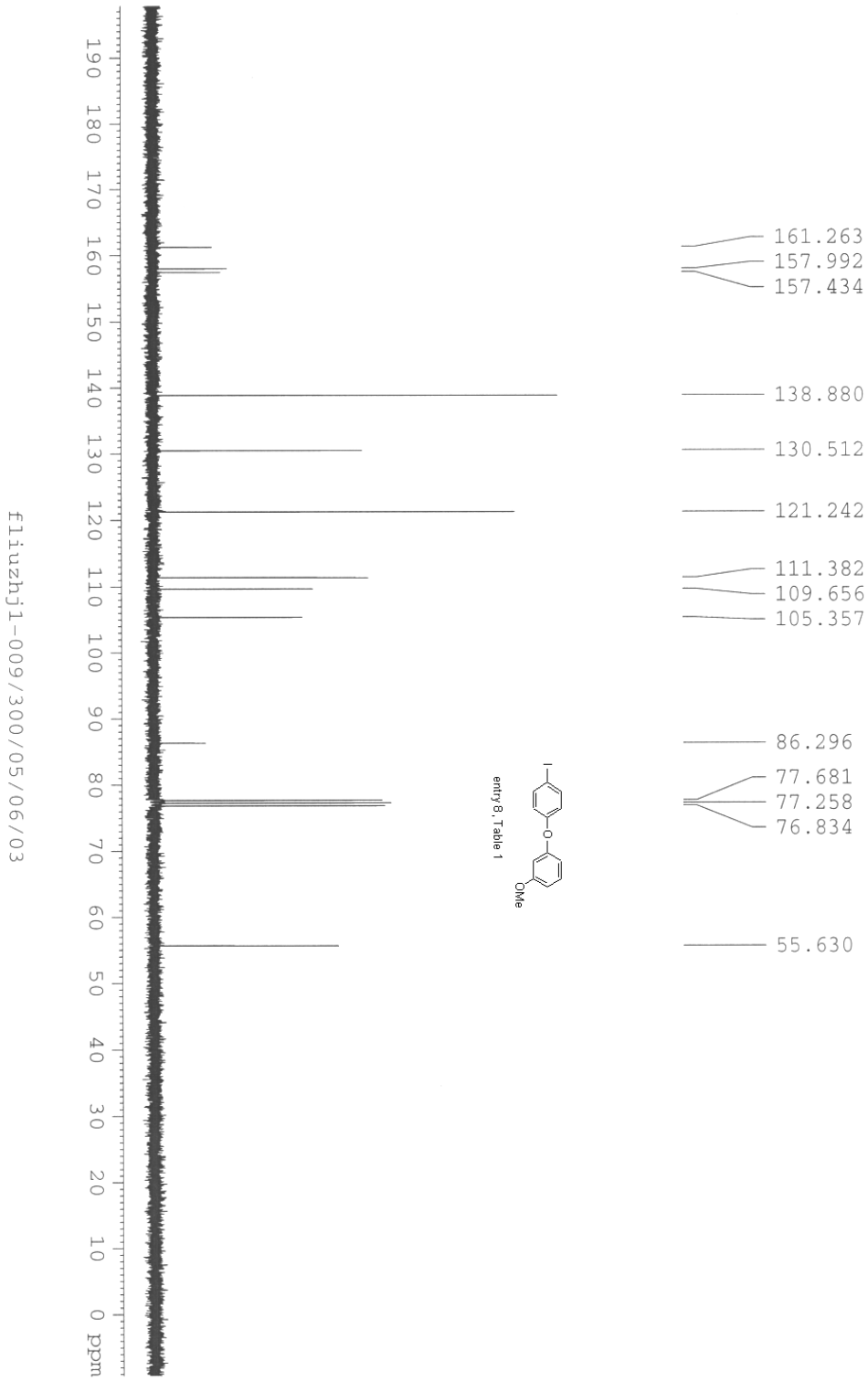
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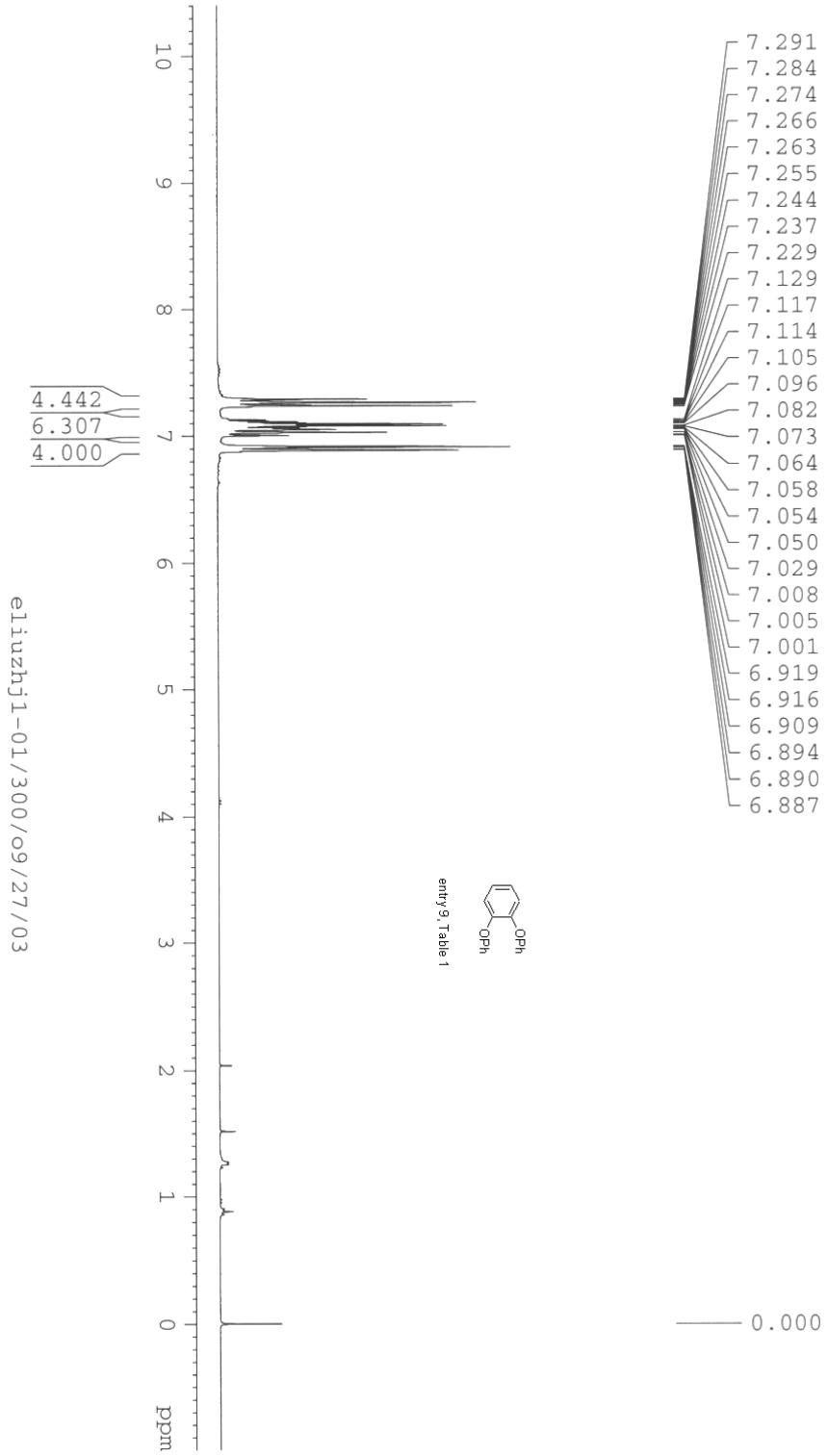
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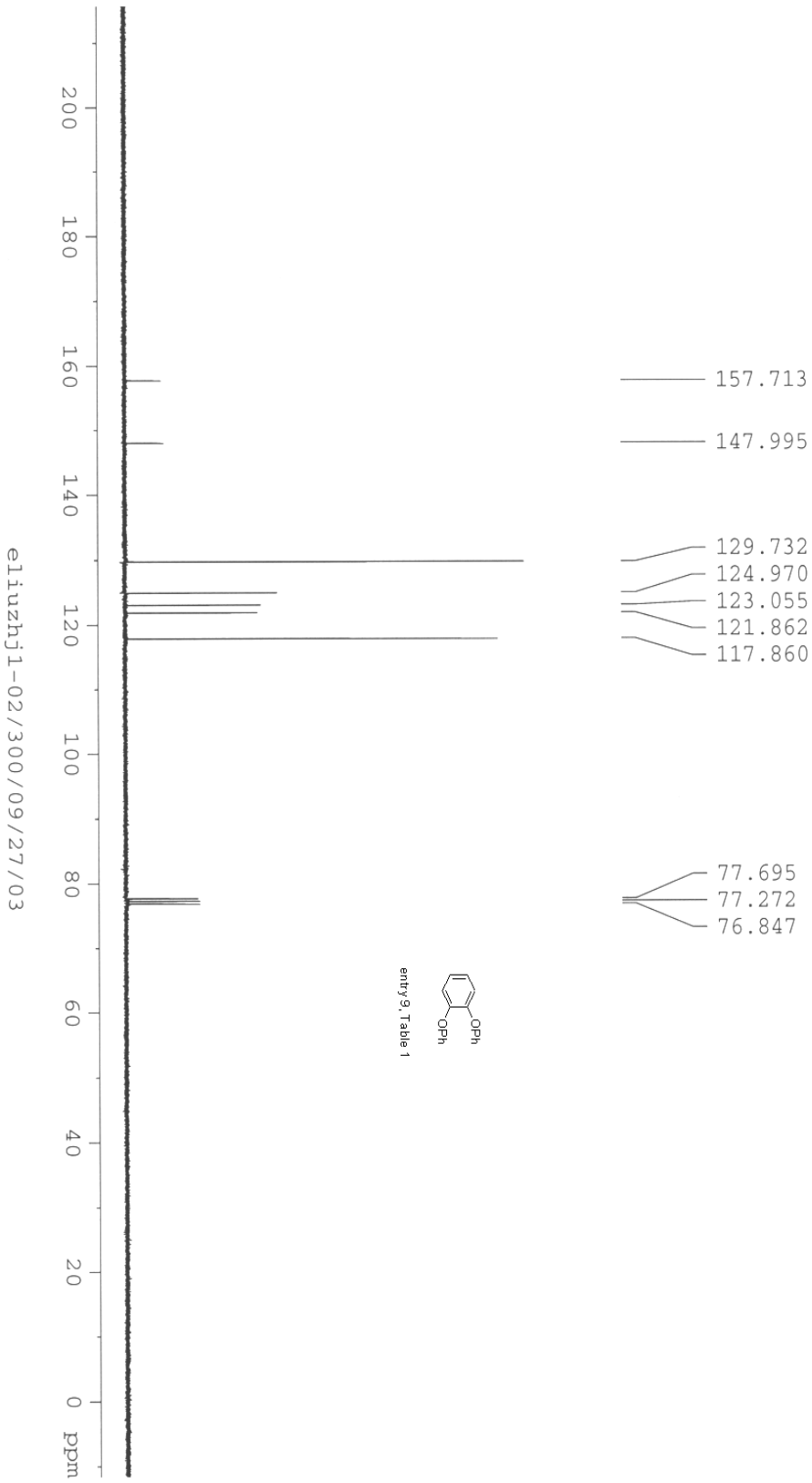
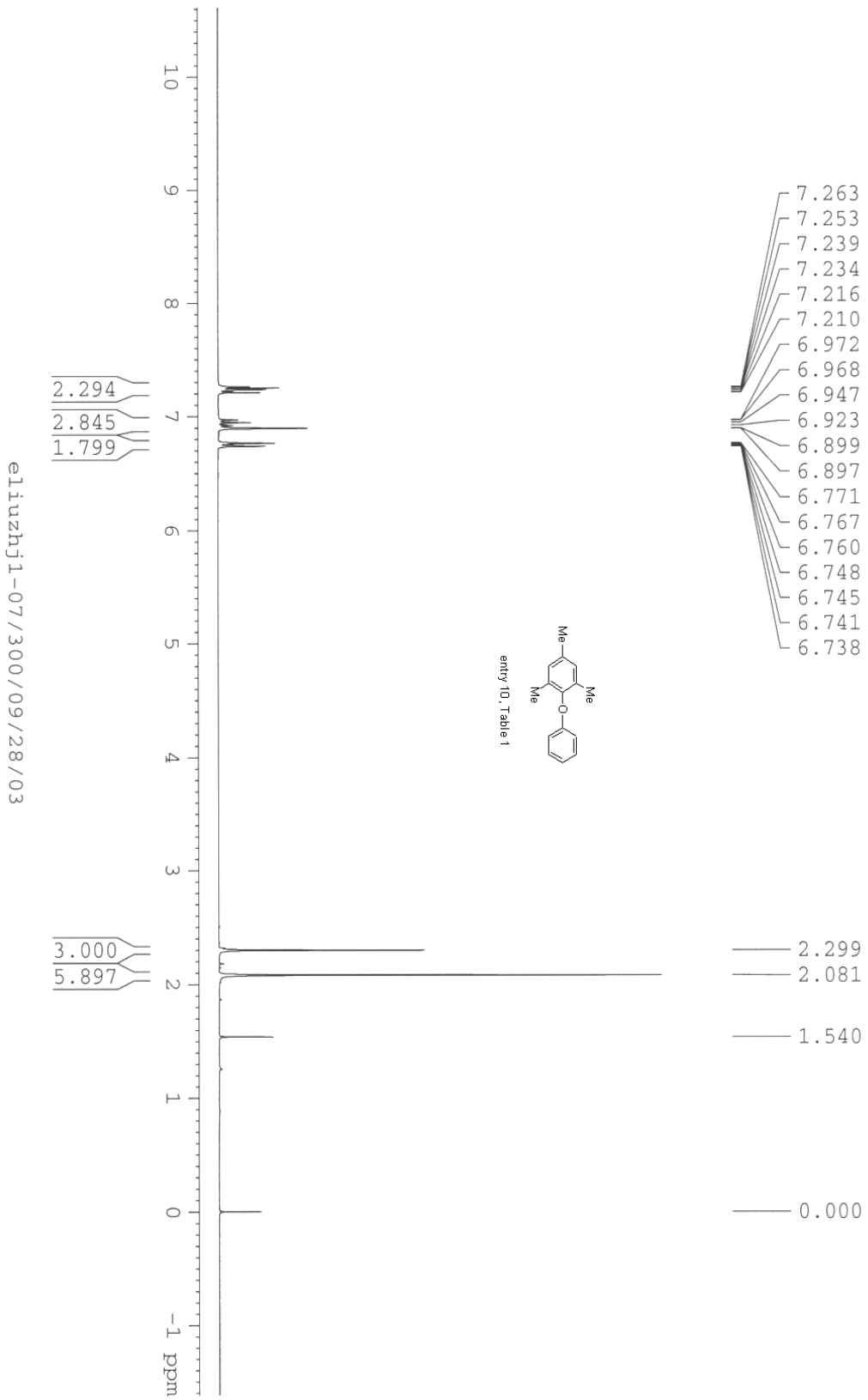
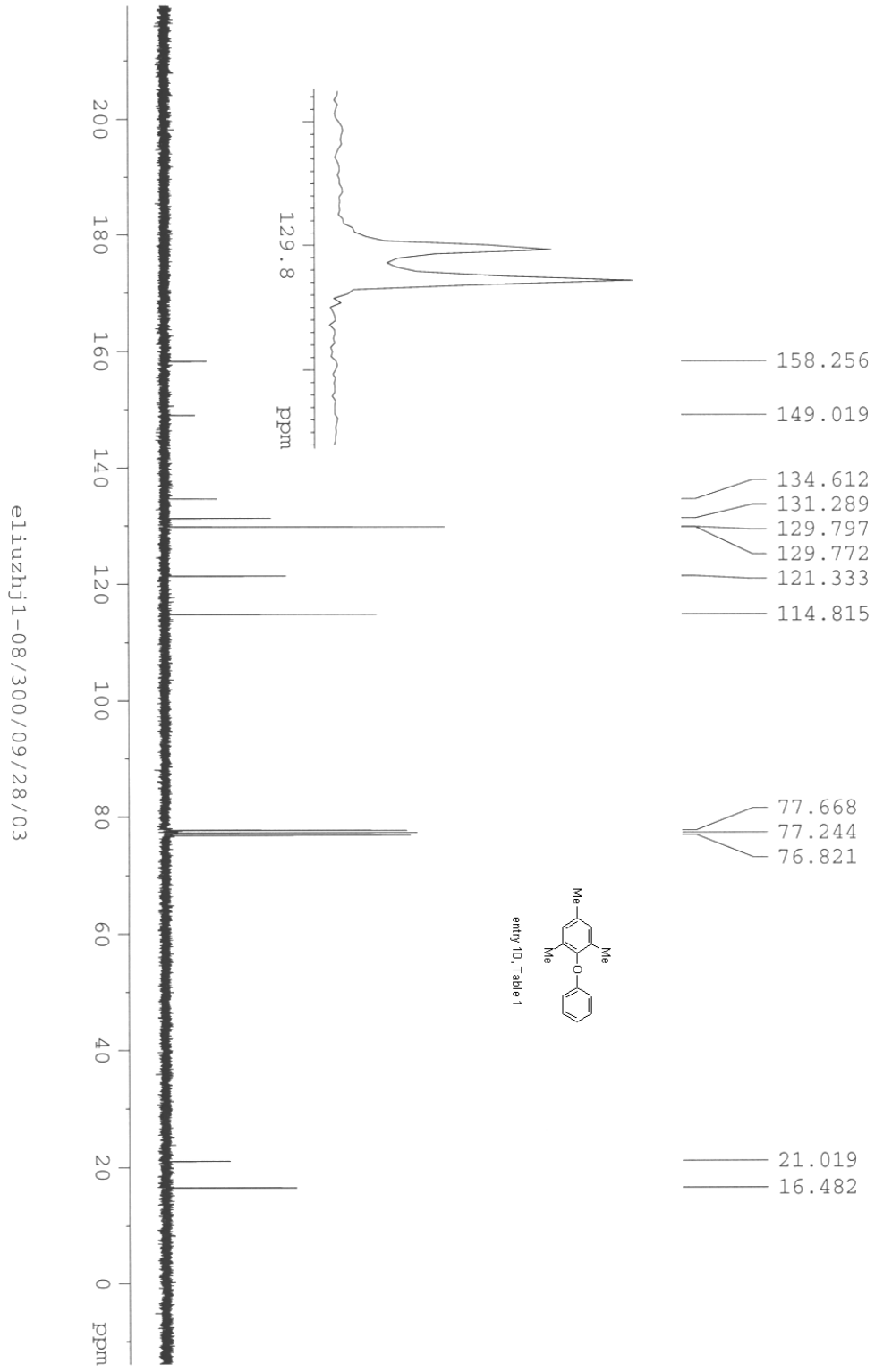
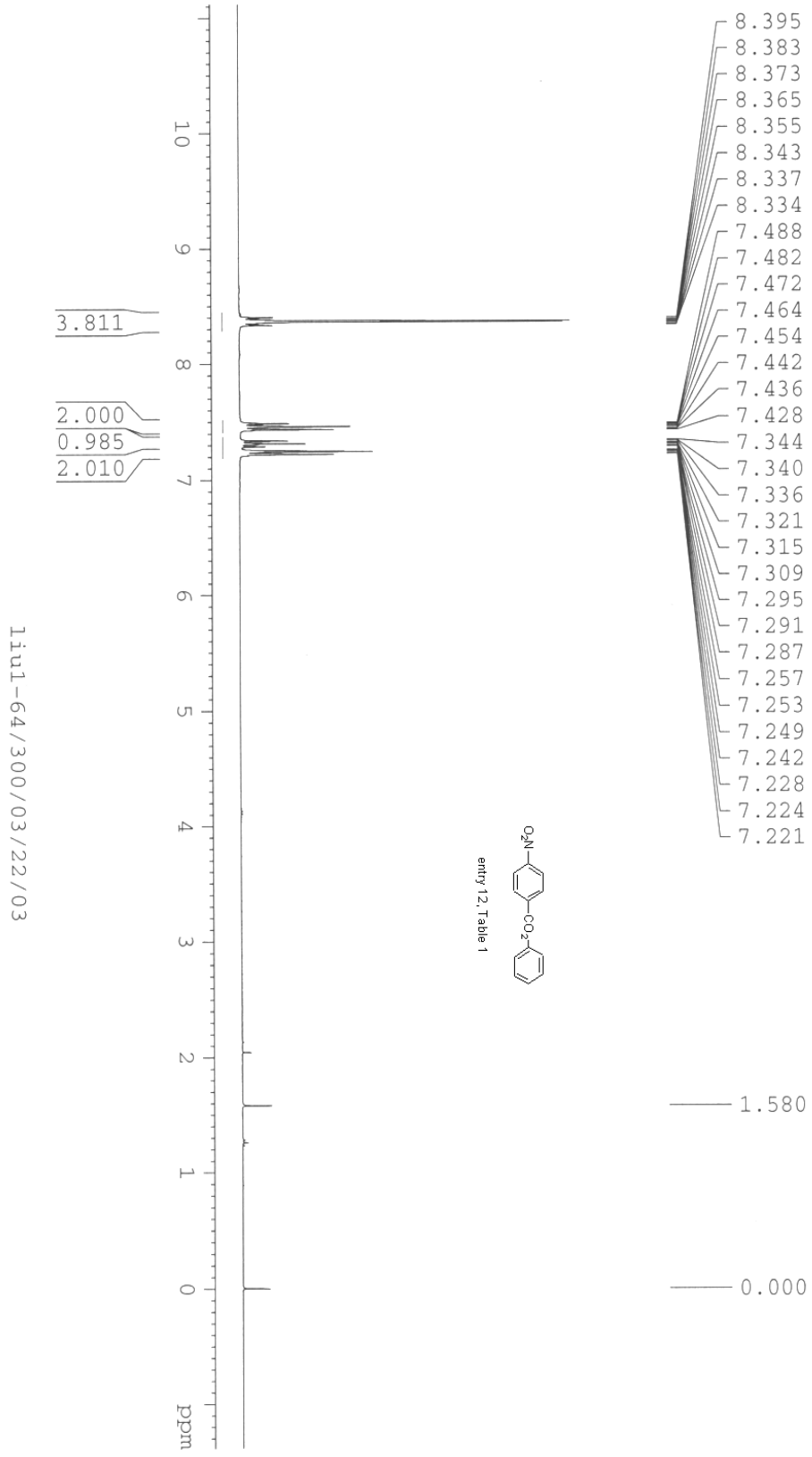
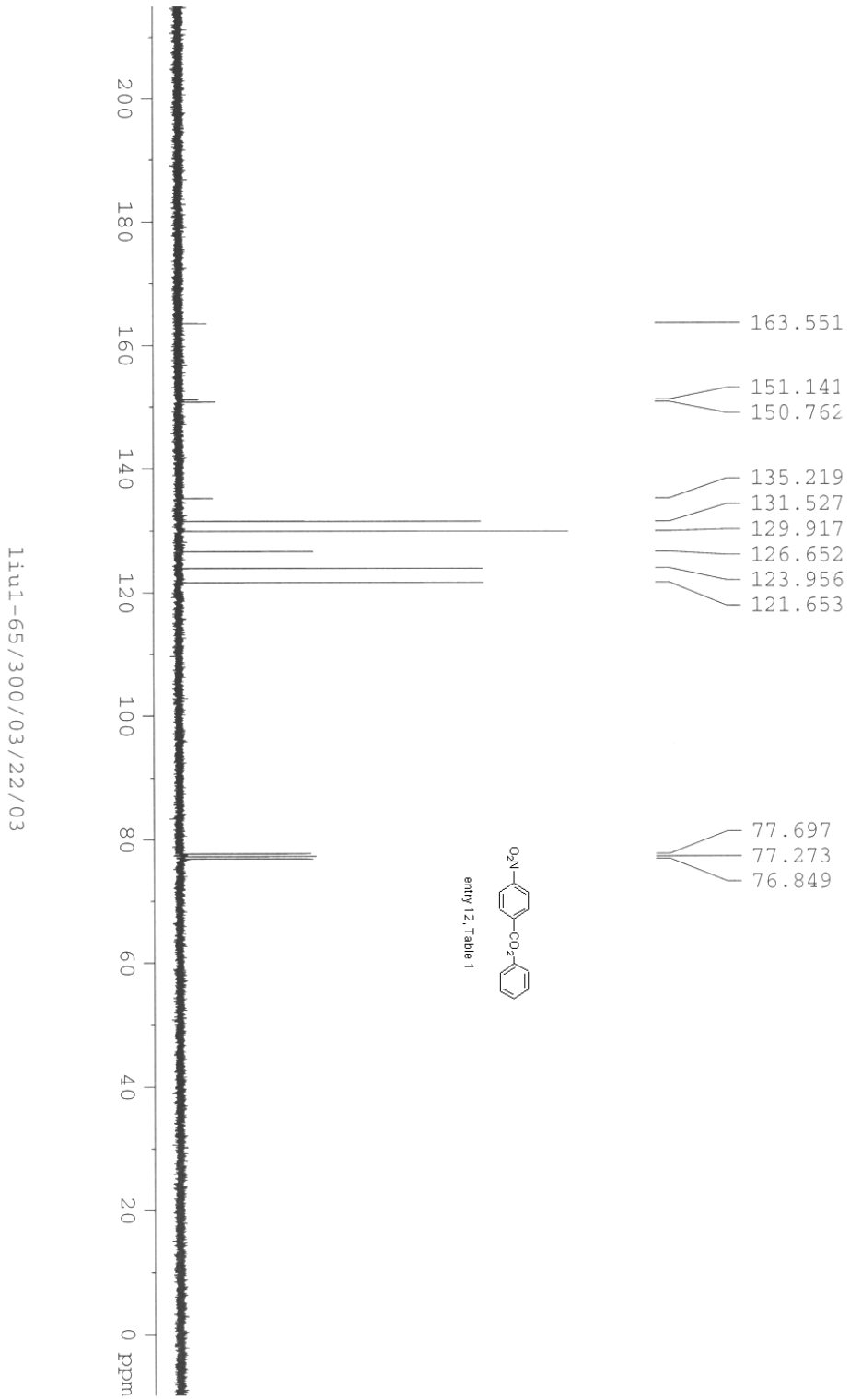
0
ppm

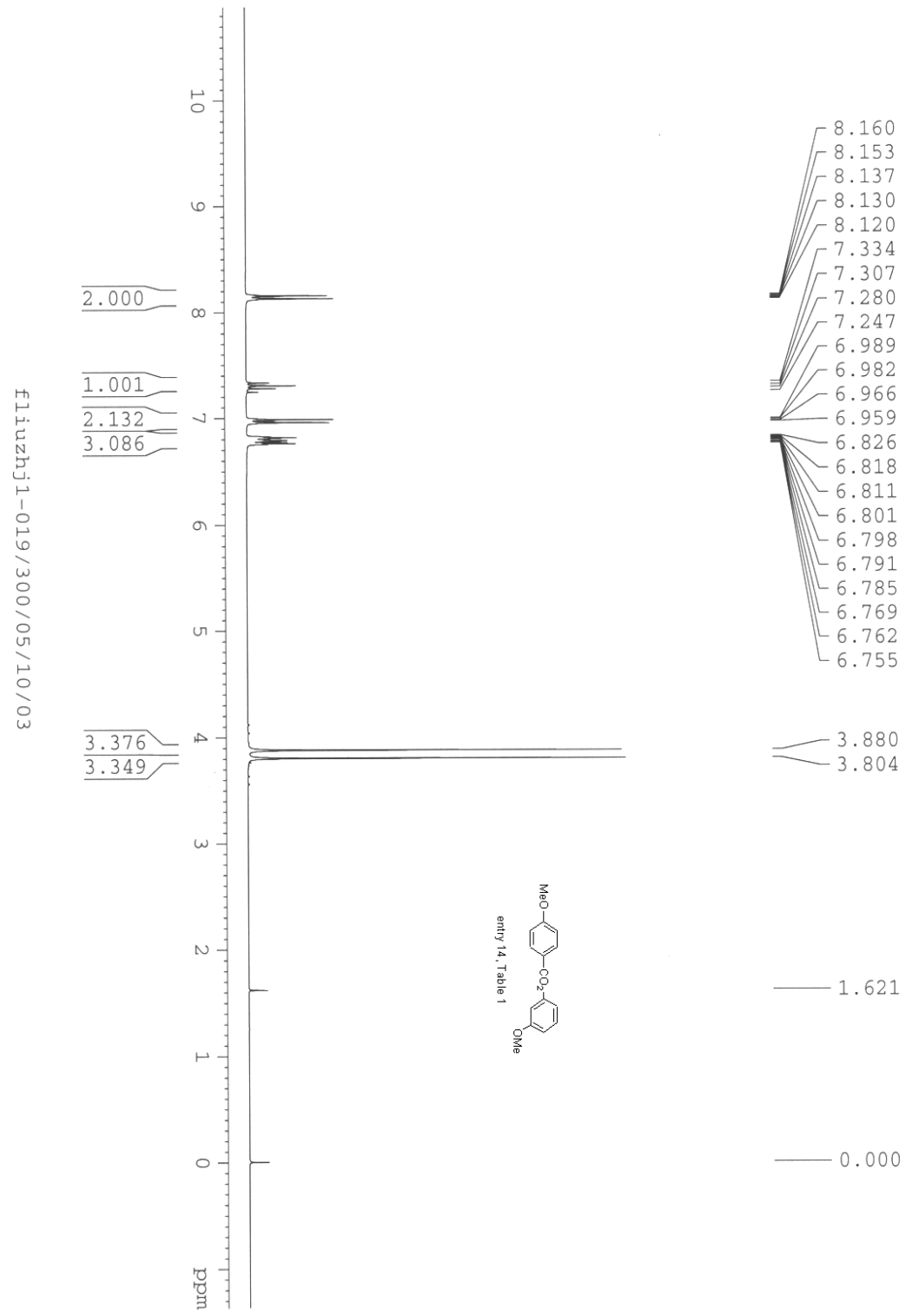

157.713
147.995
129.732
124.970
123.055
121.862
117.860
77.695
77.272
76.847
OPh
OPh
entry 9, Table 1
eliuzhj1-02/300/09/27/03
200
180
160
140
120
100
80
60
40
20
0
ppm

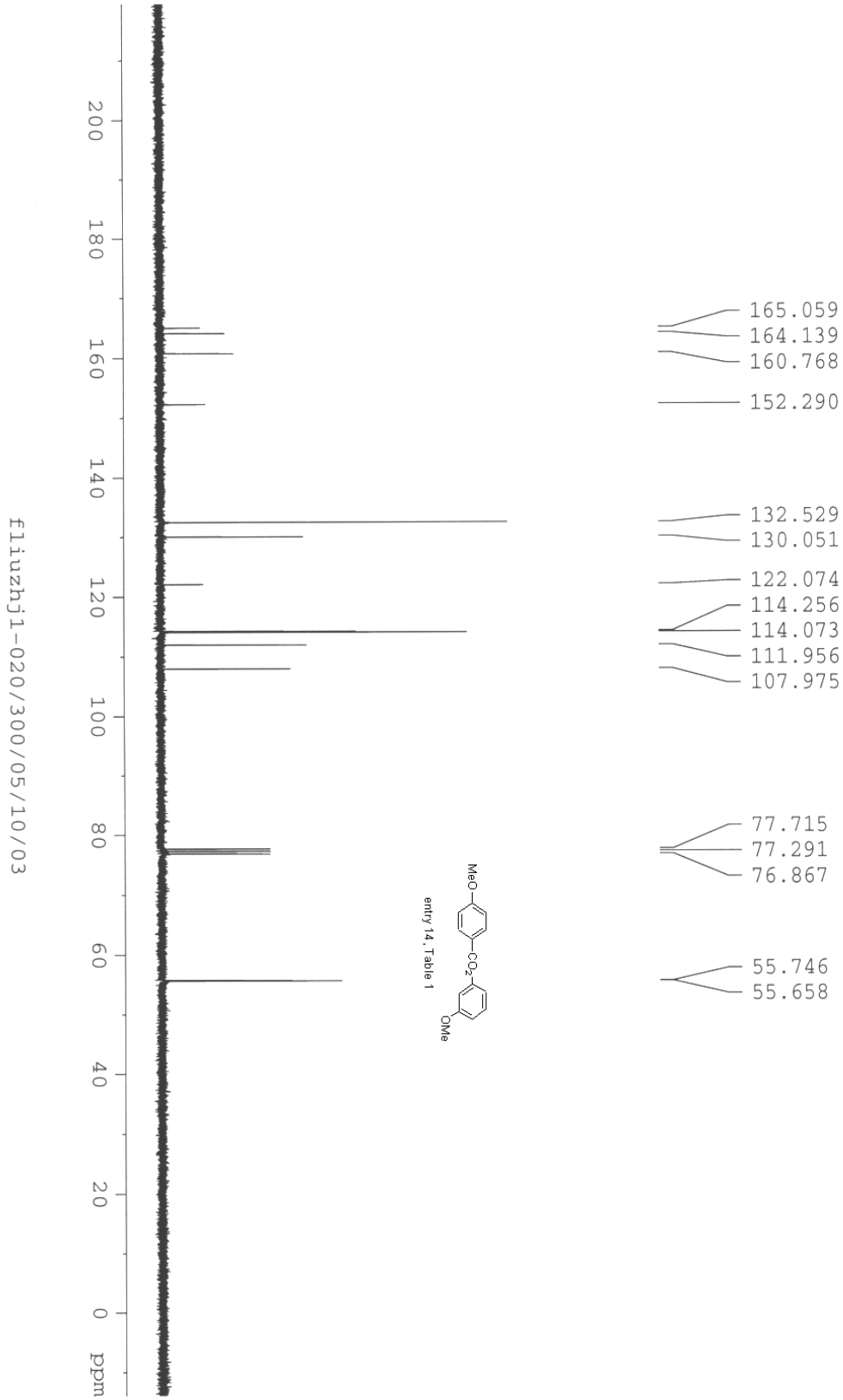

eliuzhj1-07/300/09/28/03
entry 10, Table 1
Me
Me
Me
O
7.263
7.253
7.239
7.234
7.216
7.210
6.972
6.968
6.947
6.923
6.899
6.897
6.771
6.767
6.760
6.748
6.745
6.741
6.738
2.299
2.081
1.540
0.000
2.294
2.845
1.799
3.000
5.897
10
9
8
7
6
5
4
3
2
1
0
-1 ppm

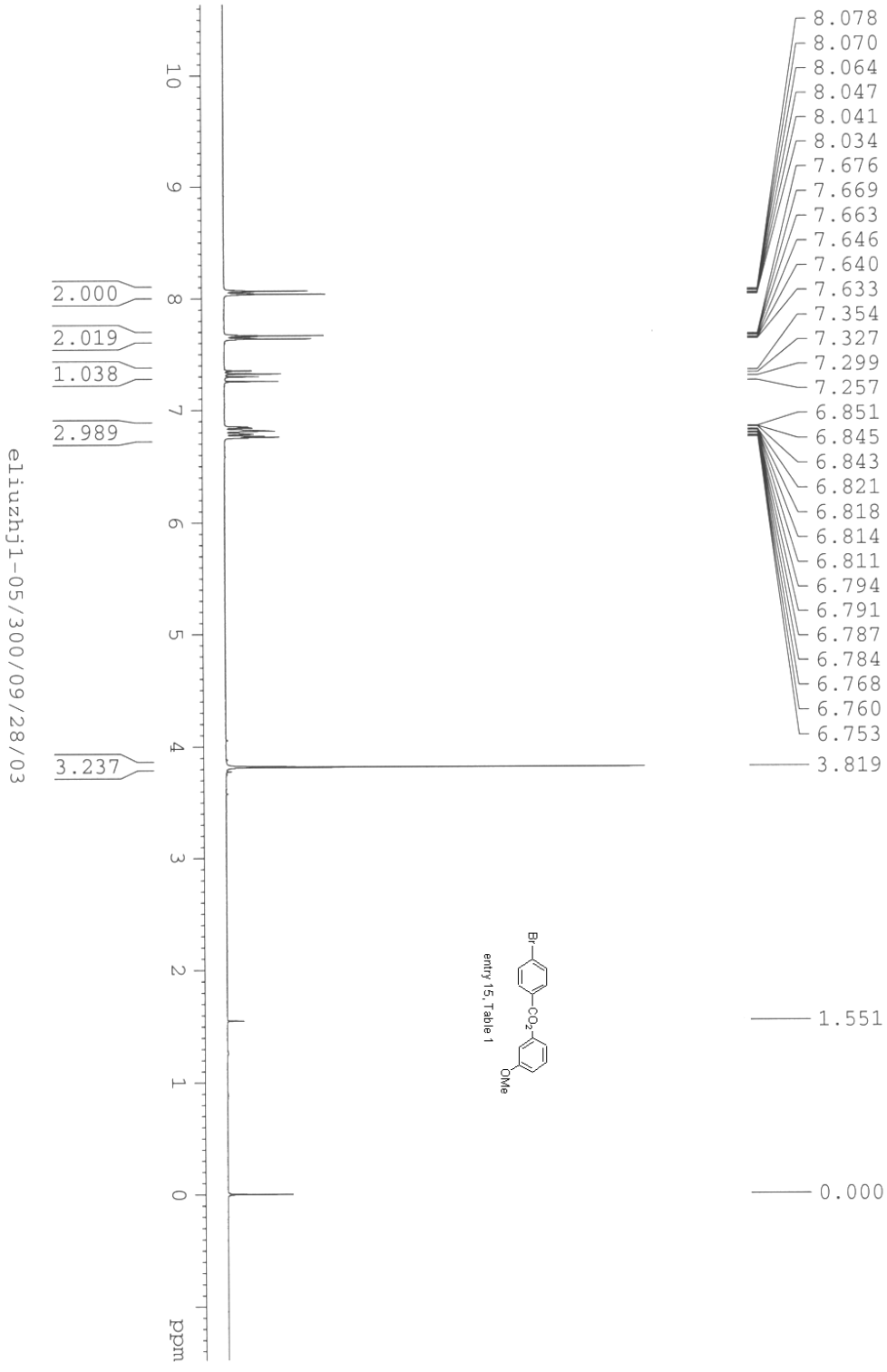

158.256
149.019
134.612
131.289
129.797
129.772
121.333
114.815
77.668
77.244
76.821
21.019
16.482
129.8
ppm
200
180
160
140
120
100
80
60
40
20
0
ppm
Me
Me
Me
O
entry 1D, Table 1
eliuzhj1-08/300/09/28/03

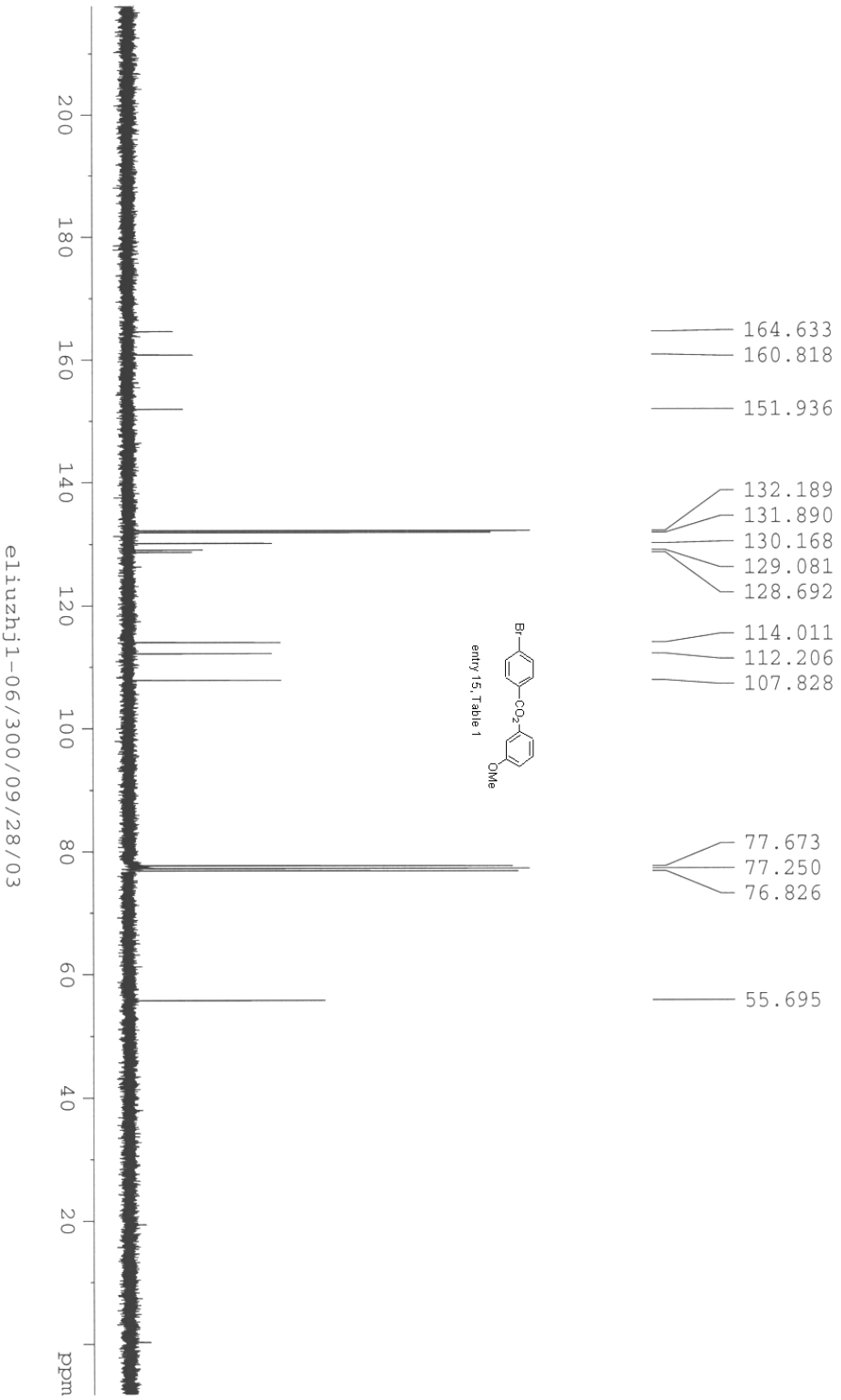

8.395
8.383
8.373
8.365
8.355
8.343
8.337
8.334
7.488
7.482
7.472
7.464
7.454
7.442
7.436
7.428
7.344
7.340
7.336
7.321
7.315
7.309
7.295
7.291
7.287
7.257
7.253
7.249
7.242
7.228
7.224
7.221
3.811
2.000
0.985
2.010
1.580
0.000
entry 12, Table 1
liu1-64/300/03/22/03
ppm


163.551
151.141
150.762
135.219
131.527
129.917
126.652
123.956
121.653
77.697
77.273
76.849
entry 12, Table 1
liu1-65/300/03/22/03
200
180
160
140
120
100
80
60
40
20
0 ppm


fliuzhj1-019/300/05/10/03
2.000
1.001
2.132
3.086
3.376
3.349
ppm
entry 14, Table 1
MeO
CO2
OMe
8.160
8.153
8.137
8.130
8.120
7.334
7.307
7.280
7.247
6.989
6.982
6.966
6.959
6.826
6.818
6.811
6.801
6.798
6.791
6.785
6.769
6.762
6.755
3.880
3.804
1.621
0.000


fliuzhj1-020/300/05/10/03
200
180
160
140
120
100
80
60
40
20
0
ppm
MeO
CO2
OMe
entry 14, Table 1
165.059
164.139
160.768
152.290
132.529
130.051
122.074
114.256
114.073
111.956
107.975
77.715
77.291
76.867
55.746
55.658


8.078
8.070
8.064
8.047
8.041
8.034
7.676
7.669
7.663
7.646
7.640
7.633
7.354
7.327
7.299
7.257
6.851
6.845
6.843
6.821
6.818
6.814
6.811
6.794
6.791
6.787
6.784
6.768
6.760
6.753
3.819
1.551
0.000
2.000
2.019
1.038
2.989
3.237
Br
CO2
OMe
entry 15, Table 1
eliuzhj1-05/300/09/28/03
ppm


164.633
160.818
151.936
132.189
131.890
130.168
129.081
128.692
114.011
112.206
107.828
77.673
77.250
76.826
55.695
Br
CO2
OMe
entry 15, Table 1
eliuzhj1-06/300/09/28/03
200
180
160
140
120
100
80
60
40
20
ppm