

Supporting Information for:

**RUTHENIUM PORPHYRIN CATALYZED FRIEDEL–CRAFTS TYPE
REACTION OF ARENES WITH IMINES**

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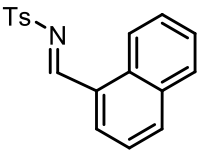
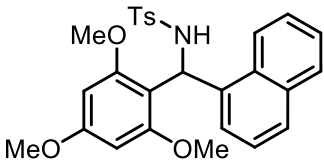
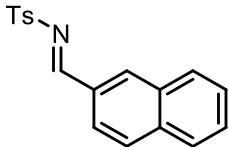
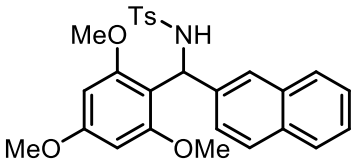
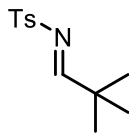
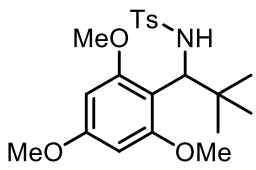
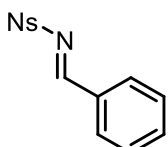
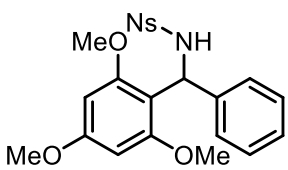
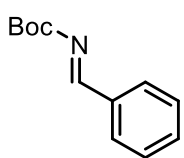
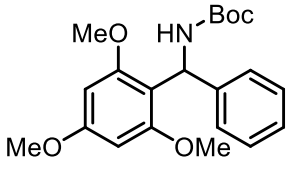
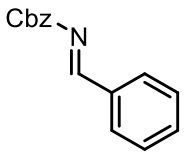
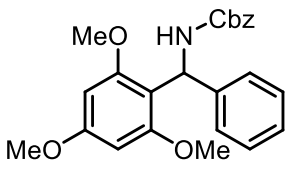
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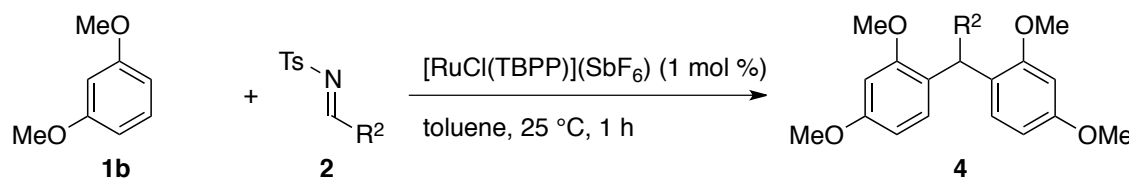
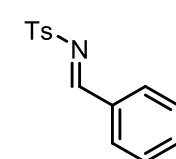
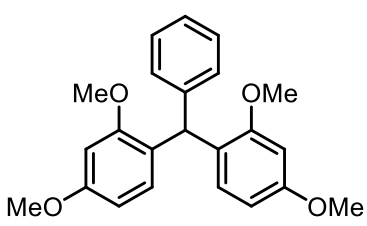
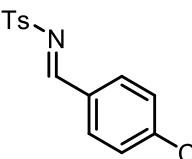
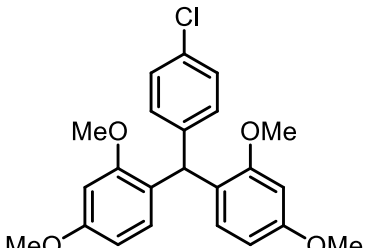
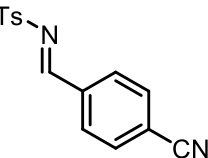
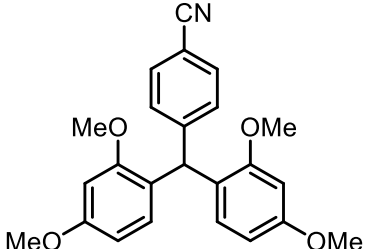
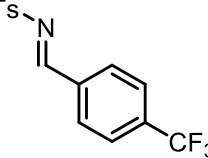
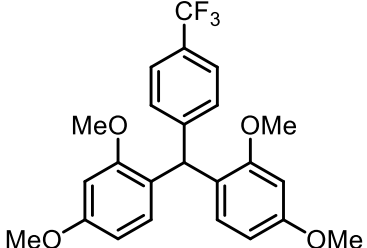
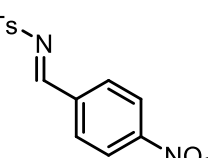
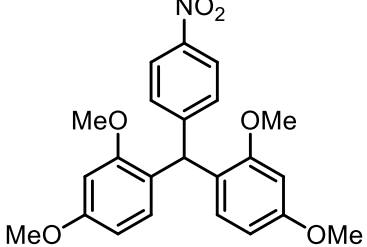
The Detailed Table 2

Entry	2	3	Yield (%)
1			86
2			85
3			83
4			94
5			72
6			71

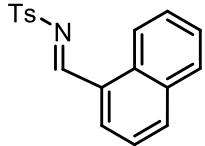
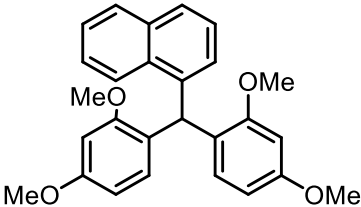
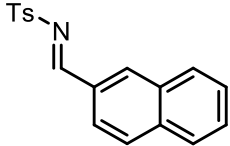
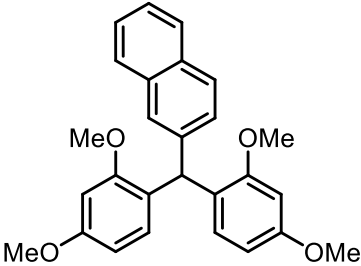
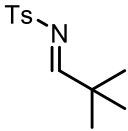
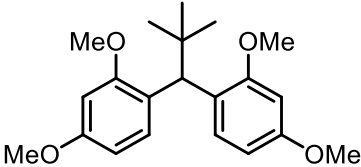
The Detailed Table 2 (continued)

entry	2	3	Yield (%)
7	 <p>2g</p>	 <p>3ag</p>	60
8	 <p>2h</p>	 <p>3ah</p>	70
9	 <p>2i</p>	 <p>3ai</p>	96
10	 <p>2j</p>	 <p>3aj</p>	67
11	 <p>2k</p>	 <p>3ak</p>	90
12	 <p>2l</p>	 <p>3al</p>	71

The Detailed Table 3

			
entry	2	4	Yield (%)
1	2a 	4ba 	69
2	2b 	4bb 	65
3	2d 	4bd 	54
4	2e 	4be 	52
5	2f 	4bf 	71

The Detailed Table 3 (continued)

entry	2	4	yield (%)
6	<p>2g</p> 	<p>4bg</p> 	47
7	<p>2h</p> 	<p>4bh</p> 	49
8	<p>2i</p> 	<p>4bi</p> 	44

Instrumentation and Chemicals

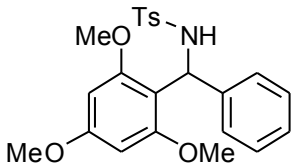
All manipulations of oxygen- and moisture-sensitive materials were conducted in a dry box or with a standard Schlenk technique under a purified argon atmosphere. Nuclear magnetic resonance spectra were taken on Varian UNITY INOVA 500 (^1H , 500 MHz; ^{13}C , 125.7 MHz) spectrometer using tetramethylsilane (^1H) as an internal standard. ^1H NMR data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, br = broad, m = multiplet), coupling constants (Hz), integration, and identification. High-resolution mass spectra were obtained with a Thermo Fisher SCIENTIFIC EXACTIVE spectrometer. Infrared spectra (IR) spectra were determined on a SHIMADZU IR Affinity-1 spectrometer. Melting points were determined using a YANAKO MP-500D. TLC analyses were performed by means of Merck Kieselgel 60 F₂₅₄ (0.25 mm) Plates. Visualization was accomplished with UV light (254 nm) and/or an aqueous alkaline KMnO_4 solution followed by heating. Flash column chromatography was carried out using Kanto Chemical silica gel (spherical, 40–50 μm). $[\text{Ru}(\text{TBPP})]\text{Cl}_2$ was prepared according to the reported procedure.¹ Unless otherwise noted, commercially available reagents were used without purification.

General Procedure: Ruthenium Catalyzed Reaction of Arene **1a** with Imine **2a**.

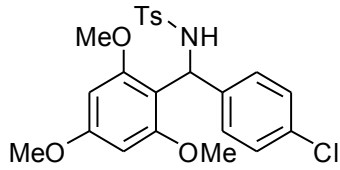
The reaction was performed in a 15 mL sealed tube equipped with a Teflon-coated magnetic stirrer bar. A solution of arene **1a** (34 mg, 0.2 mmol), imine **2a** (52 mg, 0.2 mmol) and [Ru(TBPP)](SbF₆)₂ (2.8 mg, 0.002 mmol) in 1.0 ml of toluene was stirred for 1 hour under argon atmosphere at 25 °C. The resulting reaction mixture was filtered through a silica gel pad, concentrated in vacuo. The residue was purified by flash silica gel column chromatography (20 g, 2x15 cm, hexane/ethyl acetate = 5:1) to give the corresponding product **3aa** (73 mg, 0.17 mmol) in 86% yield.

Characterization Data

4-Methyl-*N*-(phenyl(2,4,6-trimethoxyphenyl)methyl)benzenesulfonamide (**3aa**):

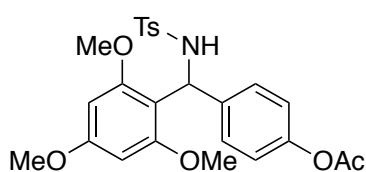
 Yield: 86%. ¹H NMR (CDCl₃) δ 7.53–7.50 (m, 2H), 7.28–7.15 (m, 5H), 7.01 (d, *J* = 8.0 Hz, 2H), 6.24 (d, *J* = 11.0 Hz, 1H), 6.12 (d, *J* = 10.5 Hz, 1H), 5.89 (s, 2H), 3.75 (s, 3H), 3.62 (s, 6H), 2.31 (s, 3H); ¹³C NMR (CDCl₃) δ 161.0, 158.0, 142.5, 141.5, 137.7, 128.8, 128.1, 127.0, 126.7, 126.6, 109.0, 90.7, 55.8, 55.5, 51.7, 21.5; IR (KBr): 3383, 3319, 1599, 1387, 1160, 1119, 814, 668, 544 cm⁻¹; HRMS (ESI) found 428.1530, Calcd for C₂₃H₂₆NO₅S: [M+H] (428.1526).

N-((4-chlorophenyl)(2,4,6-trimethoxyphenyl)methyl)-4-methylbenzenesulfonamide

 (**3ab**): Yield: 85%. ¹H NMR (CDCl₃) δ 7.50 (d, *J* = 8.5 Hz, 2H), 7.18 (s, 4H), 7.03–7.02 (m, 2H), 6.19 (d, *J* = 10.5 Hz, 1H), 6.06 (d, *J* = 10.5 Hz, 1H), 5.89 (s, 2H), 3.76 (s, 3H), 3.63 (s, 6H), 2.32 (s, 3H); ¹³C NMR (CDCl₃) δ 161.2, 157.9, 142.7, 140.1, 137.5, 132.5,

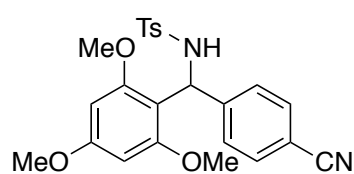
128.9, 128.2, 128.1, 127.0, 108.4, 90.7, 55.8, 55.5, 51.1, 21.6; IR (KBr): 3435, 3330, 2360, 1598, 1339, 1160, cm^{-1} ; HRMS (ESI) found 484.0939, Calcd for $\text{C}_{23}\text{H}_{24}\text{ClNO}_5\text{SNa}$: $[\text{M}+\text{Na}]$ (484.0956).

4-((4-Methylphenylsulfonamido)(2,4,6-trimethoxyphenyl)methyl)phenyl acetate



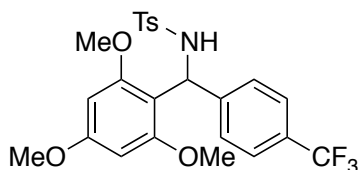
(3ac): Yield: 83%. ^1H NMR (CDCl_3) δ 7.49 (d, $J = 8.0$ Hz, 2H), 7.25–7.23 (m, 2H), 7.00 (d, $J = 8.0$ Hz, 2H), 6.92 (d, $J = 8.5$ Hz, 2H), 6.22 (d, $J = 10.5$ Hz, 1H), 6.07 (d, $J = 11$ Hz, 1H), 5.87 (s 2H), 3.74 (s 3H), 3.62 (s 6H), 2.30 (s 3H), 2.26 (s 3H); ^{13}C NMR (CDCl_3) δ 169.7, 161.1, 158.0, 149.5, 142.6, 139.0, 137.5, 128.8, 127.8, 127.0, 121.0, 108.5, 90.6, 55.7, 55.5, 51.3, 21.5, 21.3; IR (KBr): 3282, 2966, 1757, 1610, 1328, 1164, 809 cm^{-1} ; HRMS (ESI) found 486.1489, Calcd for $\text{C}_{25}\text{H}_{28}\text{NO}_7\text{S}$: $[\text{M}+\text{H}]$ (486.1581).

***N*-((4-Cyanophenyl)(2,4,6-trimethoxyphenyl)methyl)-4-methylbenzenesulfonamide**



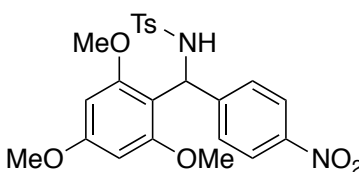
(3ad): Yield: 94%. ^1H NMR (CDCl_3) δ 7.53–7.50 (m 4H), 7.39–7.37 (m 2H), 7.05 (d $J = 8.0$ Hz, 2H), 6.23–6.20 (m, 1H), 6.11 (d, $J = 11.0$ Hz, 1H), 5.90 (s 2H), 3.76 (d, $J = 1.5$ Hz, 3H), 3.62 (s, 6H) 2.33 (s 3H); ^{13}C NMR (CDCl_3) δ 161.5, 157.8, 147.4, 142.9, 137.4, 132.0, 129.0, 127.2, 127.0, 119.2, 110.4, 107.9, 90.6, 55.8, 55.5, 51.3, 21.6; IR (KBr): 3271, 2227, 1610, 1164 cm^{-1} ; HRMS (ESI) found 453.1496, Calcd for $\text{C}_{24}\text{H}_{25}\text{N}_2\text{O}_5\text{S}$: $[\text{M}+\text{H}]$ (453.1479).

4-Methyl-*N*-((4-(trifluoromethyl)phenyl)(2,4,6-trimethoxyphenyl)methyl)benzenesulfonamide



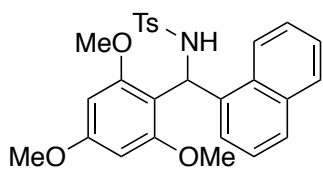
ulfonamide (3ae): Yield: 72%. ^1H NMR (CDCl_3) δ 7.52–7.50 (m 2H), 7.44 (d J = 8.0 Hz, 2H), 7.38–7.36 (m 2H), 7.01 (d J = 8.0 Hz, 2H), 6.34 (d, J = 10.5 Hz, 1H), 6.12 (d J = 10.5 Hz, 1H), 5.89 (s 2H), 3.74 (s 3H), 3.62 (s 6H), 2.23 (s 3H); ^{13}C NMR (CDCl_3) δ 161.3, 157.9, 145.8, 145.8, 142.8, 137.5, 128.8 (q, J = 32.0 Hz), 127.0, 126.8, 125.0 (q, J = 3.8 Hz), 124.4 (q, J = 270 Hz), 108.2, 90.7, 55.7, 55.5, 51.3, 21.5; ^{19}F NMR (CDCl_3) δ -62.7; IR (KBr): 3317, 2966, 1610, 1327, 1164, 1122 cm^{-1} ; HRMS (ESI) found 496.1339, Calcd for $\text{C}_{24}\text{H}_{25}\text{F}_3\text{NO}_5\text{S}$: $[\text{M}+\text{H}]$ (496.1400).

4-Methyl-*N*-((4-nitrophenyl)(2,4,6-trimethoxyphenyl)methyl)benzenesulfonamide



(3af): Yield: 71%. ^1H NMR (CDCl_3) δ 8.05–8.03 (m 2H), 7.53 (d, J = 8.0 Hz, 2H), 7.44–7.42 (m, 2H), 7.05–7.03 (m 2H), 6.38 (d, J = 10.5 Hz, 1H), 6.14 (d, J = 10.5 Hz, 1H), 5.89 (s 2H), 3.74 (s, 3H), 3.62 (s, 6H), 2.31 (s 3H); ^{13}C NMR (CDCl_3) δ 161.5, 157.7, 149.5, 146.7, 143.0, 137.3, 129.0, 127.3, 127.0, 123.3, 107.8, 90.6, 55.7, 55.5, 51.2, 21.5; IR (KBr): 3453, 3330, 1598, 1338, 1160, 668 cm^{-1} ; HRMS (ESI) found 473.1320, Calcd for $\text{C}_{23}\text{H}_{25}\text{N}_2\text{O}_7\text{S}$: $[\text{M}+\text{H}]$ (473.1377).

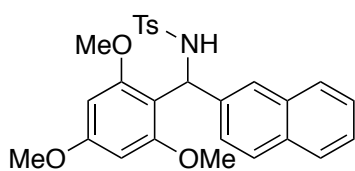
4-Methyl-*N*-(naphthalen-1-yl(2,4,6-trimethoxyphenyl)methyl)benzenesulfonamide



(3ag): Yield: 60%. ^1H NMR (CDCl_3) δ 8.75 (d, J = 8.0 Hz, 1H), 7.84 (d, J = 8.0 Hz, 1H), 7.74 (d, J = 8.0 Hz, 1H), 7.64 (t J = 7.8 Hz, 1H), 7.53–7.48 (m 3H), 7.25 (t, J = 7.8 Hz, 1H), 7.09 (d, J = 7.5 Hz, 1H), 6.98 (d, J = 8.0 Hz, 2H), 6.91 (d, J = 11 Hz, 1H), 6.13 (d, J =

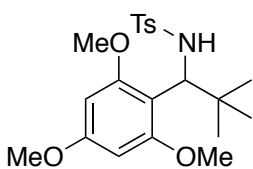
11.0 Hz, 1H), 5.86 (s 2H), 3.76 (s 3H), 3.66 (s 6H), 2.31 (s, 3H) ; ^{13}C NMR (CDCl_3) δ 161.1, 158.3, 143.0, 137.5, 134.9, 134.1, 131.8, 128.7, 128.5, 126.9, 126.6, 125.7, 125.7, 125.1, 124.8, 109.9, 107.1, 90.7, 55.7, 55.5, 50.6, 21.5; IR (KBr): 3282, 1594, 1457, 1327, 1160, 1128, 674 cm^{-1} ; HRMS (ESI) found 516.1228, Calcd for $\text{C}_{27}\text{H}_{27}\text{NO}_5\text{SK}$: [M+K] (516.1242).

4-Methyl-*N*-(naphthalen-2-yl(2,4,6-trimethoxyphenyl)methyl)benzenesulfonamide



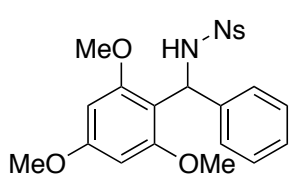
(3ah): Yield: 70%. ^1H NMR (CDCl_3) δ 7.78–7.76 (m 1H), 7.73–7.70 (m 2H), 7.64 (s 1H), 7.54 (d, $J = 8.0$ Hz, 2H), 7.44–7.41 (m 3H), 7.02 (d, $J = 8.0$ Hz, 2H), 6.33 (d, $J = 11.0$ Hz, 1H), 6.27 (d, $J = 11.0$ Hz, 1H), 5.92 (s 2H), 3.77 (s 3H), 3.63 (s 6H), 2.31 (s 3H); ^{13}C NMR (CDCl_3) δ 161.1, 158.1, 142.6, 138.8, 137.7, 133.3, 132.6, 128.9, 128.2, 127.8, 127.6, 127.0, 125.9, 125.7, 125.5, 124.8, 108.8, 90.7, 55.8, 55.5, 51.8, 21.6; IR (KBr): 3364, 2938, 1594, 1418, 1333, 1155, 815 cm^{-1} ; HRMS (ESI) found 516.1232, Calcd for $\text{C}_{27}\text{H}_{27}\text{NO}_5\text{SK}$: [M+K] (516.1242).

N-(2,2-Dimethyl-1-(2,4,6-trimethoxyphenyl)propyl)-4-methylbenzenesulfonamide



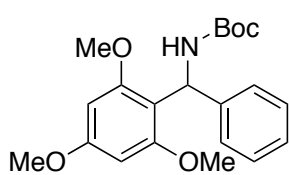
(3ai): Yield: 96%. ^1H NMR (CDCl_3) δ 7.39 (d, $J = 8.5$ Hz, 2H), 6.91 (s 2H), 6.21 (d, $J = 11.5$ Hz, 1H), 5.79 (d, $J = 3.5$ Hz, 2H), 4.57 (d, $J = 11.0$ Hz, 1H), 3.72 (s, 3H), 3.69 (s, 3H), 3.63 (s, 3H), 2.24 (s 3H), 0.90 (s, 9H); ^{13}C NMR (CDCl_3) δ 160.3, 158.4, 158.2, 142.2, 137.3, 128.5, 126.7, 107.0, 90.6, 90.1, 58.2, 55.4, 55.4, 55.3, 37.2, 27.2, 21.4; IR (KBr): 3321, 2971, 1588, 1428, 1334, 1166 cm^{-1} ; HRMS (ESI) found 408.1828, Calcd for $\text{C}_{21}\text{H}_{30}\text{NO}_5\text{S}$: [M+H] (408.1839).

2-Nitro-*N*-(phenyl(2,4,6-trimethoxyphenyl)methyl)benzenesulfonamide (3aj):



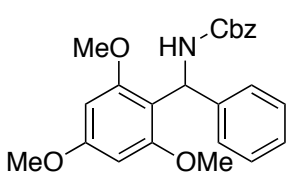
Yield: 67%. ^1H NMR (CDCl_3) δ 7.91 (m 1H), 7.85 (d, $J = 8.0$ Hz, 1H), 7.51–7.49 (m 1H), 7.46–7.45 (m 1H), 7.34 (d, $J = 10.5$ Hz, 1H), 7.29–7.31 (m, 2H), 7.20–7.19 (m 2H), 7.15–7.12 (m 1H), 6.31 (d, $J = 10.5$ Hz, 1H), 5.85 (s 2H), 3.70 (s 3H), 3.69 (s 6H); ^{13}C NMR (CDCl_3) δ 161.1, 158.0, 147.3, 140.8, 134.7, 132.8, 132.0, 131.3, 128.1, 126.9, 126.6, 124.8, 108.1, 90.3, 55.7, 55.5, 52.5; IR (KBr): 3315, 1594, 1358, 1150, 1040 cm^{-1} ; HRMS (ESI) found 459.1210, Calcd for $\text{C}_{22}\text{H}_{23}\text{N}_2\text{O}_7\text{S}$: $[\text{M}+\text{H}]$ (459.1220).

***tert*-Butyl (phenyl(2,4,6-trimethoxyphenyl)methyl)carbamate (3ak):** Yield: 90%. ^1H



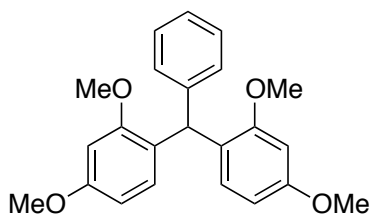
NMR (CDCl_3) δ 7.27–7.25 (m 4H), 7.18–7.15 (m 1H), 6.61 (d, $J = 10.0$ Hz, 1H), 6.28 (d, $J = 10.5$ Hz, 1H), 6.17 (s, 2H), 3.82 (s, 3H), 3.78 (s 6H), 1.49 (s 9H); ^{13}C NMR (CDCl_3) δ 160.6, 158.6, 155.8, 143.6, 128.0, 126.2, 126.1, 111.1, 91.2, 79.1, 56.0, 55.4, 48.2, 28.7, 28.4; IR (KBr): 3457, 2975, 1701, 1496, 1120 cm^{-1} ; HRMS (ESI) found 374.1951, Calcd for $\text{C}_{21}\text{H}_{28}\text{NO}_5$: $[\text{M}+\text{H}]$ (374.1962).

Benzyl (phenyl(2,4,6-trimethoxyphenyl)methyl)carbamate (3al): Yield: 71%. ^1H



NMR (CDCl_3) δ 7.41–7.18(m, 8H), 6.71–6.68 (m 1H), 6.54–6.51 (m 1H), 6.17–6.15 (m 2H), 5.20–5.11 (m 2H), 3.81 (s 3H), 3.77–3.72 (m, 6H); ^{13}C NMR (CDCl_3) δ 160.8, 158.5, 156.3, 143.0, 136.8, 128.6, 128.4, 128.2, 128.1, 128.4, 126.1, 125.9, 110.7, 91.2, 66.8, 56.0, 55.8, 55.4, 48.9; IR (neat): 3443, 2940, 1716, 1610, 1454, 1230, 1126 cm^{-1} ; HRMS (ESI) found 408.1790, Calcd for $\text{C}_{24}\text{H}_{26}\text{NO}_5$: $[\text{M}+\text{H}]$ (408.1805).

4,4'-(phenylmethylene)bis(1,3-dimethoxybenzene) (4ba): Yield: 69%. ¹H NMR

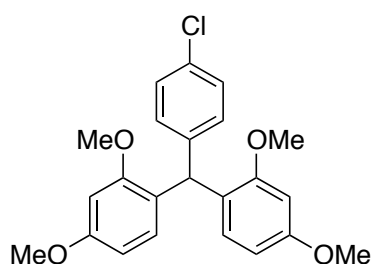


(CDCl₃) δ 7.27–7.23 (m 2H), 7.19–7.16 (m 1H), 7.05 (d, *J* = 7.0 Hz, 2H), 6.69 (d, *J* = 7.0 Hz, 2H), 6.47 (s 2H), 6.39–6.37 (m 2H), 6.03 (s 1H), 3.80 (s 6H), 3.70 (s, 6H);

¹³C NMR (CDCl₃) δ 159.3, 158.2, 144.7, 130.6, 129.3,

128.0, 125.7, 125.5, 103.6, 98.9, 55.9, 55.4, 42.3; IR (KBr): 2962, 1587, 1503, 1210 cm⁻¹; HRMS (ESI) found 365.1738, Calcd for C₂₃H₂₅O₄: [M+H] (365.1747).

4,4'-((4-chlorophenyl)methylene)bis(1,3-dimethoxybenzene) (4bb): Yield: 65%. ¹H



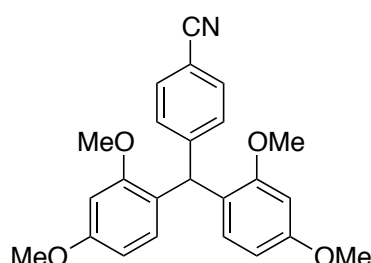
NMR (CDCl₃) δ 7.21–7.19 (m 2H), 6.98–6.97 (m 2H), 6.65 (d, *J* = 8.5 Hz, 2H), 6.47–6.46 (m 2H) 6.39–6.37 (m 2H), 5.97 (s 1H), 3.80 (s 6H), 3.69 (s 6H); ¹³C NMR

(CDCl₃) δ 159.5, 158.1, 143.5, 131.4, 130.6, 130.5,

128.1, 124.9, 103.6, 98.9, 55.8, 55.4, 41.8; IR (KBr):

2834, 1587, 1208, 1113, 840 cm⁻¹; HRMS (ESI) found 399.1351, Calcd for C₂₃H₂₄ClO₄: [M+H] (399.1358).

4-(bis(2,4-dimethoxyphenyl)methyl)benzonitrile (4bc): Yield: 54%. ¹H NMR

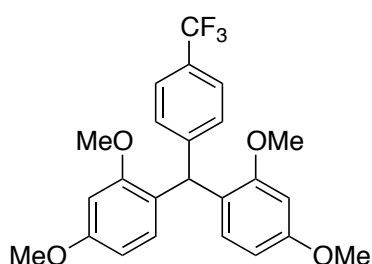


(CDCl₃) δ 7.51 (d, *J* = 8.5 Hz, 2H), 7.14–7.12 (m 2H), 6.62–6.60 (m 2H), 6.46 (s 2H), 6.38–6.36 (m 2H), 6.01 (s 1H), 3.79 (s 6H), 3.67 (s 6H); ¹³C NMR (CDCl₃) δ

159.8, 158.1, 151.1, 131.9, 130.5, 129.9, 123.7, 119.6,

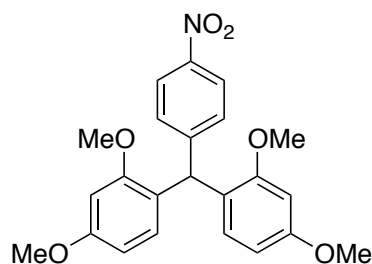
109.5, 103.8, 98.9, 55.8, 55.5, 42.7; IR (KBr): 3475, 2226, 1609, 1506, 1297, 1209, 1036, 825 cm^{-1} ; HRMS (ESI) found 390.1691, Calcd for $\text{C}_{24}\text{H}_{24}\text{NO}_4$: [M+H] (390.1700).

4,4'-((4-(trifluoromethyl)phenyl)methylene)bis(1,3-dimethoxybenzene) (4be):



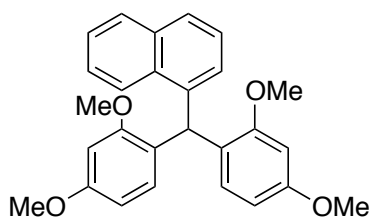
Yield: 52%. ^1H NMR (CDCl_3) δ 7.46 (d, $J = 8.0$ Hz, 2H), 7.15–7.13 (m 2H), 6.64 (d, $J = 8.5$ Hz, 2H), 6.47–6.46 (m 2H), 6.38–6.36 (m 2H), 6.04 (s 1H), 3.79 (s 6H), 3.68 (s, 6H); ^{13}C NMR (CDCl_3) δ 159.7, 158.2, 149.3, 130.5, 129.5, 128.0 (q, $J = 32.0$ Hz), 125.0 (q, $J = 3.7$ Hz), 124.7 (q, $J = 270.4$ Hz), 124.7 (q, $J = 90$ Hz), 103.8, 98.9, 55.8, 55.4, 42.4; ^{19}F NMR (CDCl_3) δ -62.5; IR (neat): 2917, 1452, 1103, 698 cm^{-1} ; HRMS (ESI) found 433.1618, Calcd for $\text{C}_{24}\text{H}_{24}\text{F}_3\text{O}_4$: [M+H] (433.1621).

4,4'-((4-nitrophenyl)methylene)bis(1,3-dimethoxybenzene) (4bf): Yield: 71%. ^1H



NMR (CDCl_3) δ 6.09 (d, $J = 8.5$ Hz, 2H), 7.20 (d, $J = 8.5$ Hz, 2H), 6.65 (d, $J = 8.0$ Hz, 2H), 6.49 (s 2H), 6.41–6.39 (m 2H), 6.08 (s 1H), 3.81 (s 6H), 3.69 (s 6H); ^{13}C NMR (CDCl_3) δ 159.6, 158.1, 153.4, 146.2, 130.5, 129.8, 123.5, 123.3, 103.8, 98.9, 55.7, 55.4, 42.6; IR (KBr): 2962, 1607, 1517, 1344, 1294, 1209, 1031, 824 cm^{-1} ; HRMS (ESI) found 410.1618, Calcd for $\text{C}_{23}\text{H}_{24}\text{NO}_6$: [M-H] (410.1598).

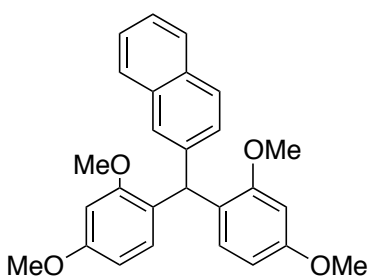
1-(bis(2,4-dimethoxyphenyl)methyl)naphthalene (4bg): Yield: 47%. ¹H NMR



(CDCl₃) δ 7.95 (d, *J* = 8.0 Hz, 1H), 7.82 (d, *J* = 8.0 Hz, 1H), 7.69 (d, *J* = 8.0 Hz, 1H), 7.42–7.34 (m 2H), 7.31 (t, *J* = 7.5 Hz, 1H), 6.92 (d, *J* = 7.5 Hz, 1H), 6.71 (s 1H), 6.66 (d, *J* = 8.5 Hz, 2H), 6.50 (s, 2H), 6.32 (dd, *J* =

8.8, 2.3 Hz, 2H), 3.77 (s, 6H), 3.70 (s, 6H); ¹³C NMR (CDCl₃) δ 159.4, 158.0, 141.2, 134.1, 132.2, 130.8, 128.6, 126.8, 125.9, 125.8, 125.3, 125.3, 125.0, 124.8, 103.6, 98.9, 55.9, 55.4, 38.4; IR (KBr): 3443, 2937, 1609, 1503, 1292, 1207, 1035 cm⁻¹; HRMS (ESI) found 437.1708, Calcd for C₂₇H₂₆O₄Na: [M+Na] (437.1723).

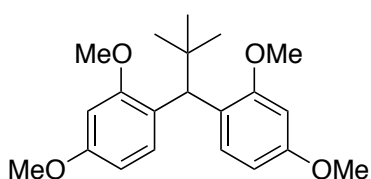
2-(bis(2,4-dimethoxyphenyl)methyl)naphthalene (4ah): Yield: 49%. ¹H NMR



(CDCl₃) d 7.81–7.79 (m 1H), 7.72 (d, *J* = 8.5 Hz, 1H), 7.70–7.68 (m 1H), 7.43–7.40 (m 2H), 7.36 (s 1H), 7.29–7.27 (m, 1H), 6.72 (d, 8.5 Hz, 2H), 6.49 (d, *J* = 2.5 Hz, 2H), 6.38, (dd, *J* = 8.5, 2.5 Hz, 2H), 6.18 (s 1H), 3.81 (s 6H), 3.70 (s, 6H); ¹³C NMR (CDCl₃) d 159.4,

158.3, 142.6, 133.6, 132.2, 130.8, 128.8, 128.0, 127.7, 127.5, 127.0, 125.7, 125.2, 125.2, 103.6, 98.9, 55.9, 55.4, 42.4; IR (KBr): 3453, 2956, 1587, 1502, 1210, cm⁻¹; HRMS (ESI) found 437.1711, Calcd for C₂₇H₂₆O₄Na: [M+Na] (437.1723).

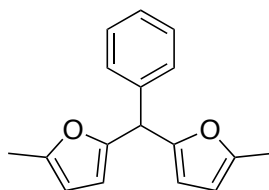
4,4'-(2,2-dimethylpropane-1,1-diyl)bis(1,3-dimethoxybenzene) (4bi): Yield: 44%. ¹H



NMR (CDCl₃) d 7.43 (d, *J* = 8.5 Hz, 2H), 6.43–6.40 (m 4H), 4.97 (s 1H), 3.76 (s 12H), 1.00 (s 9H); ¹³C NMR (CDCl₃) d 159.0, 158.5, 130.6, 125.6, 103.8, 99.0, 56.1,

55.3, 40.9, 35.7, 29.4; IR (KBr): 2971, 1586, 1209, 1154, 839 cm^{-1} ; HRMS (ESI) found 345.2051, Calcd for $\text{C}_{21}\text{H}_{29}\text{O}_4$: [M+H] (345.2060).

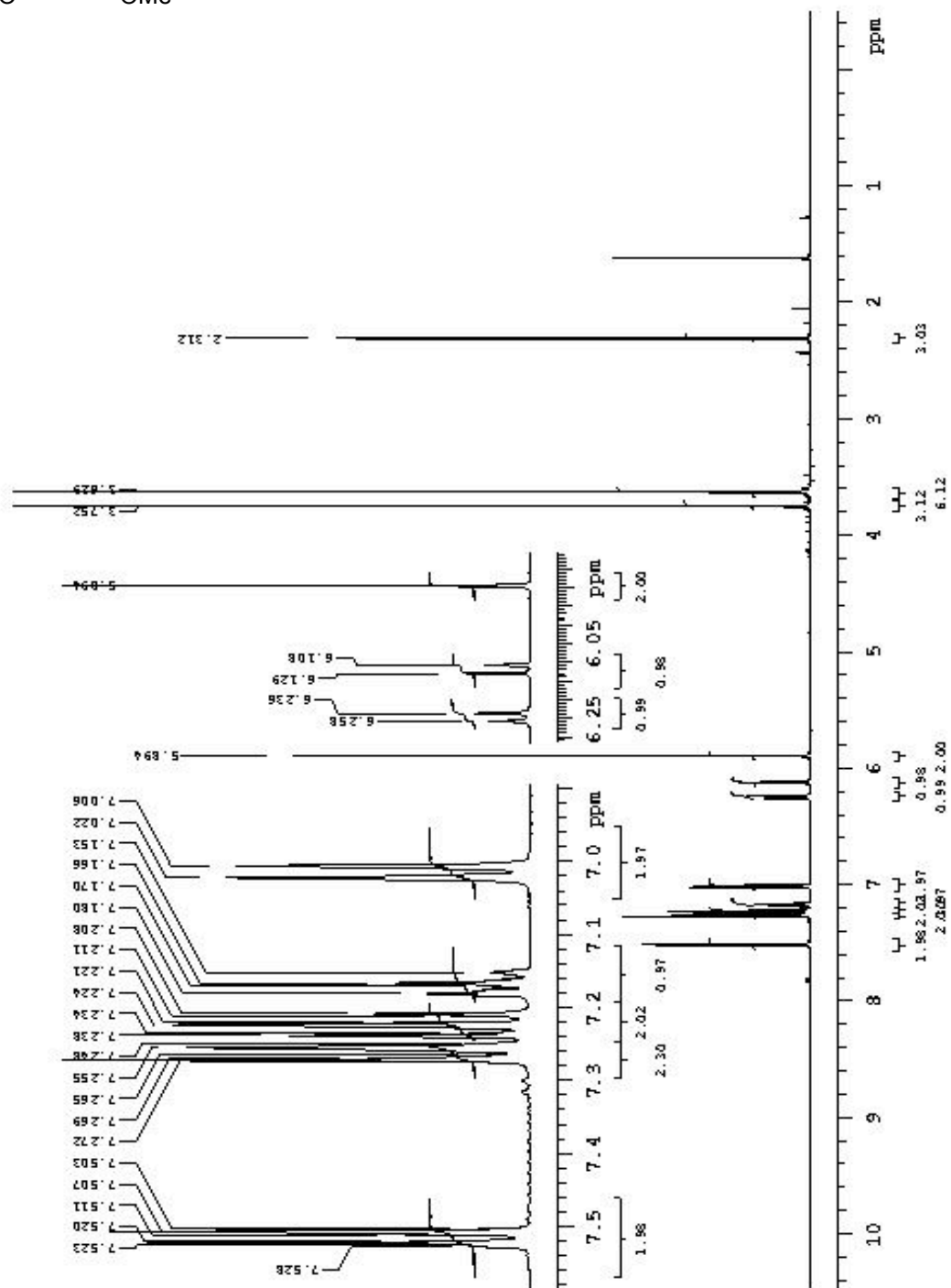
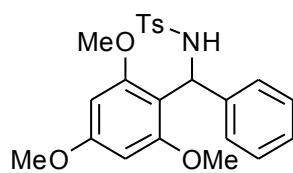
5,5'-(phenylmethylene)bis(2-methylfuran) (5ca): Yield: 77%. ^1H NMR (CDCl_3) d

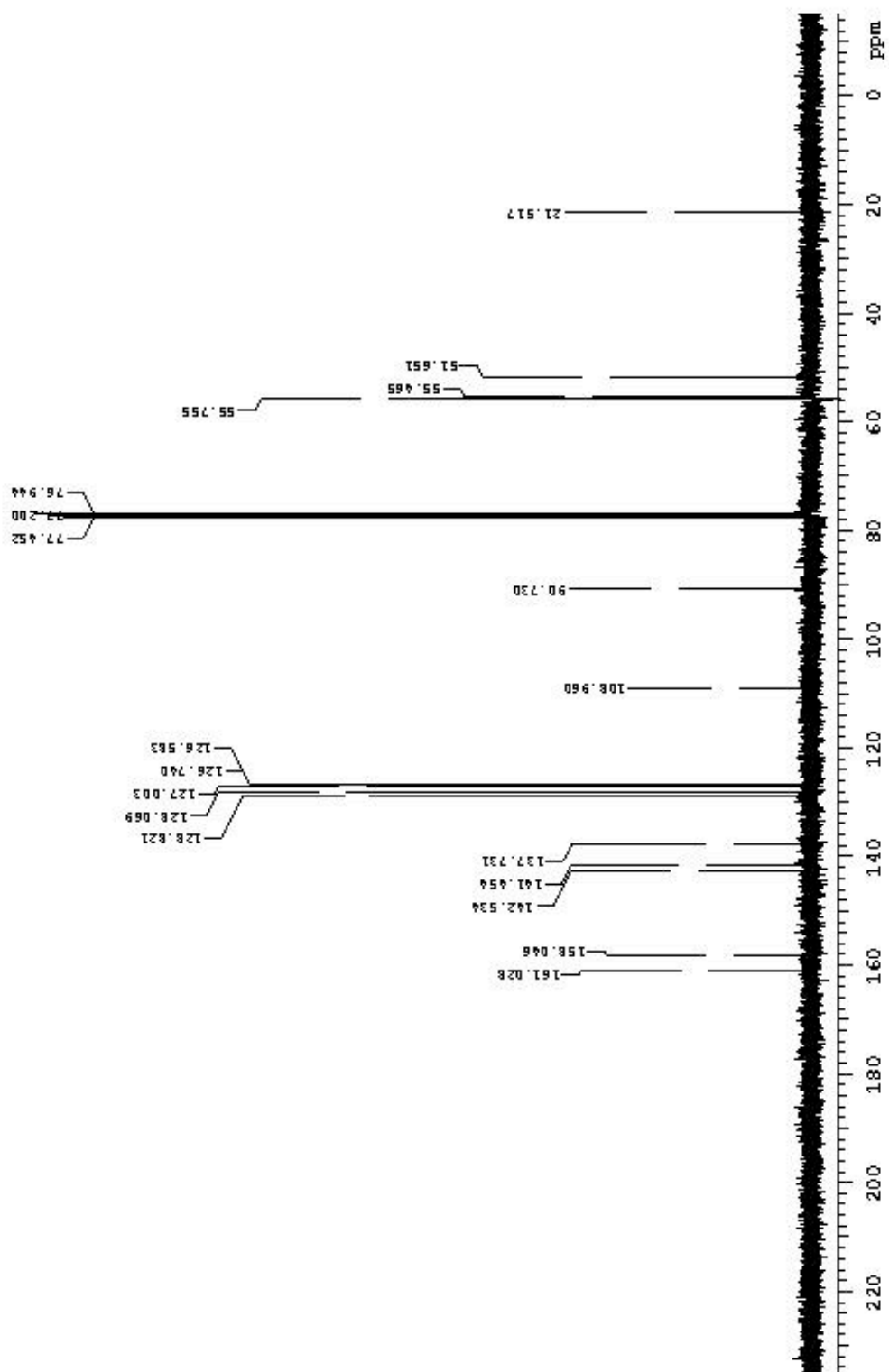


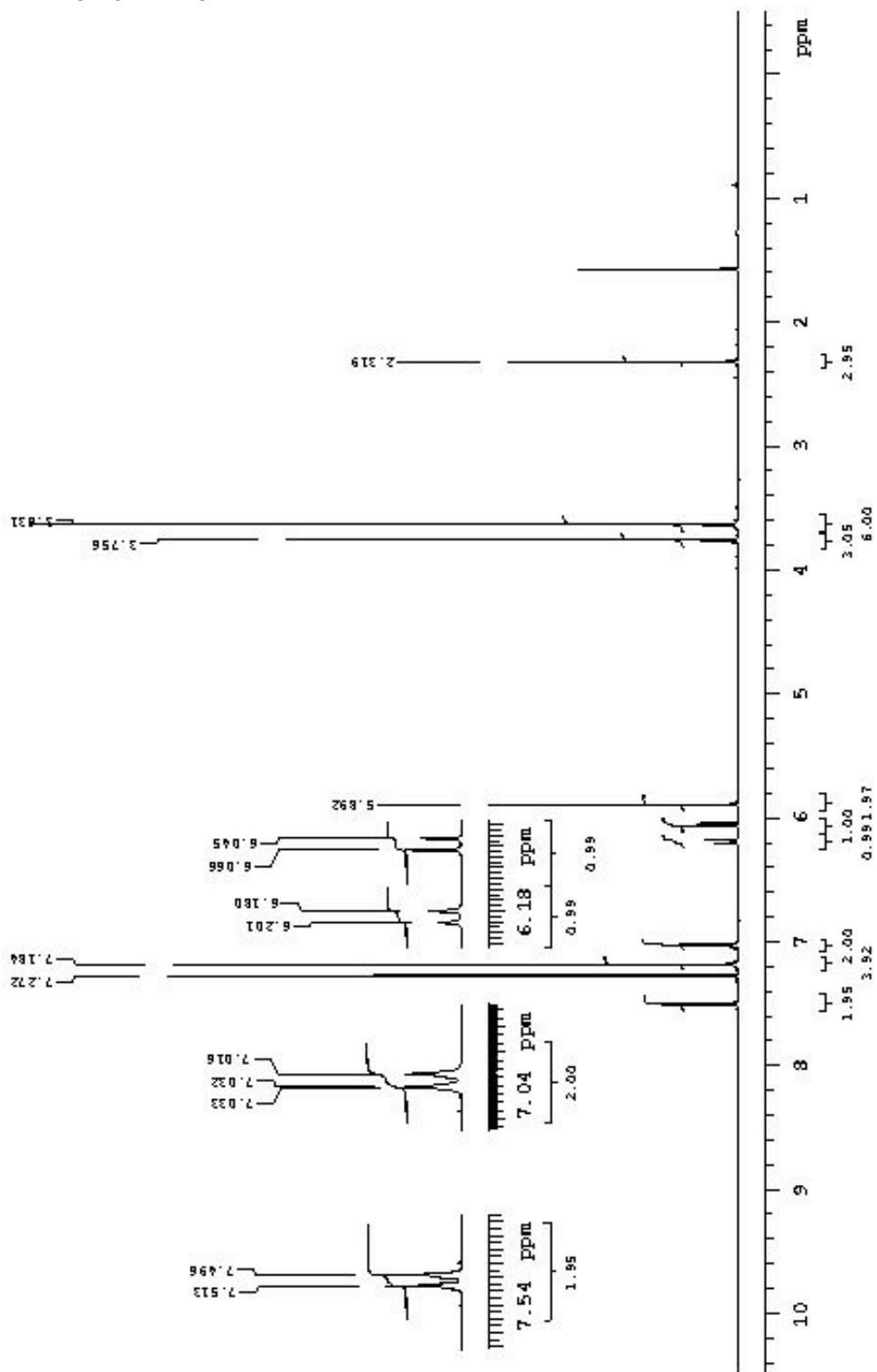
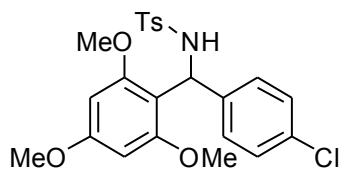
7.33–7.23 (m 5H), 5.88–5.86 (m 4H), 5.34 (s 1H); ^{13}C NMR (CDCl_3) d 153.0, 151.6, 140.1, 128.6, 128.5, 127.1, 108.3, 106.2, 45.2, 13.8; IR (neat): 3029, 2922, 1558, 1496, 1452, 1218, 1022, 778, 732, 699 cm^{-1} ; HRMS (ESI) found 253.1216, Calcd for

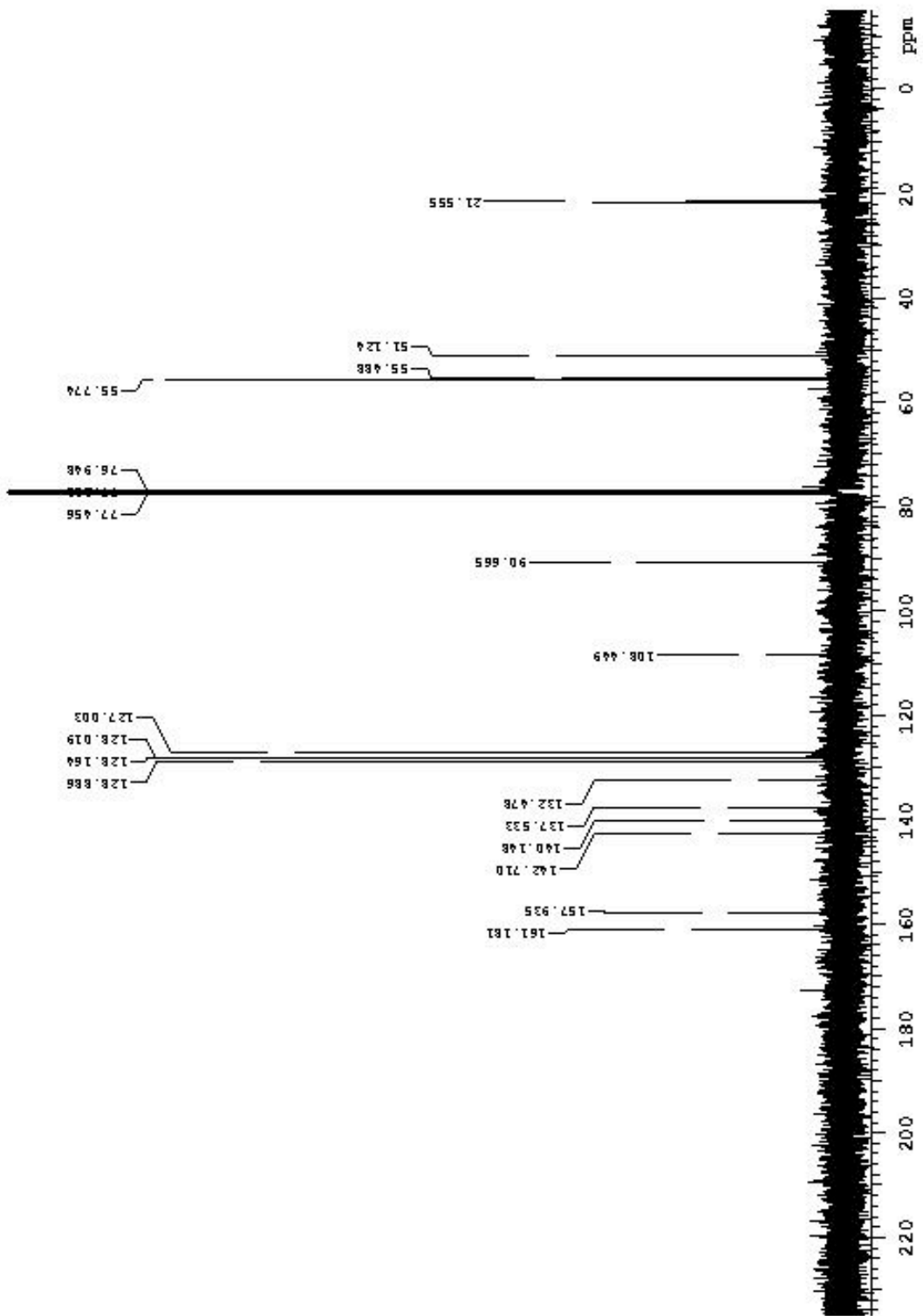
$\text{C}_{17}\text{H}_{17}\text{O}_2$: [M+H] (253.1223).

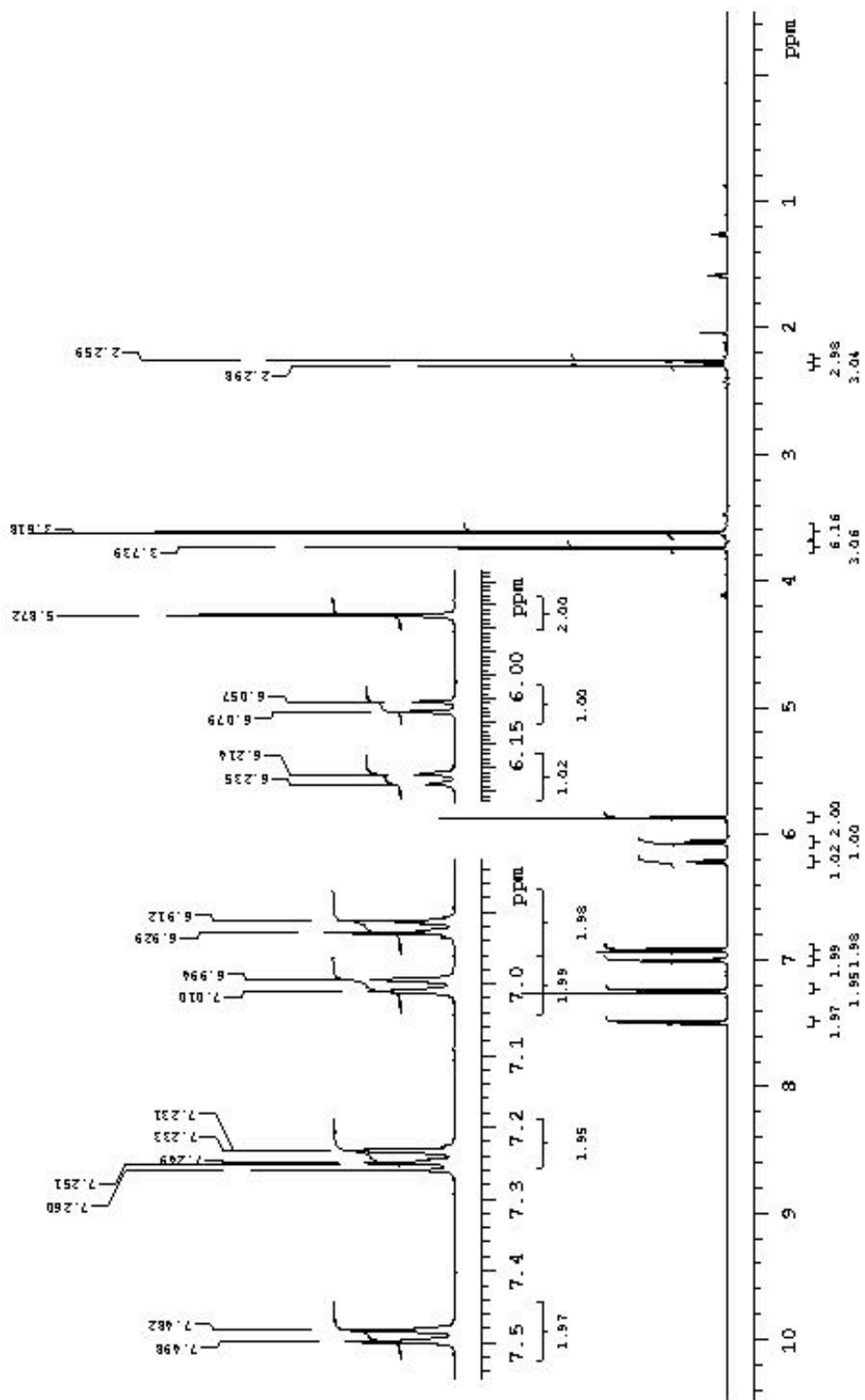
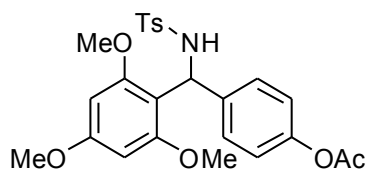
¹H NMR and ¹³C NMR Spectra of Products

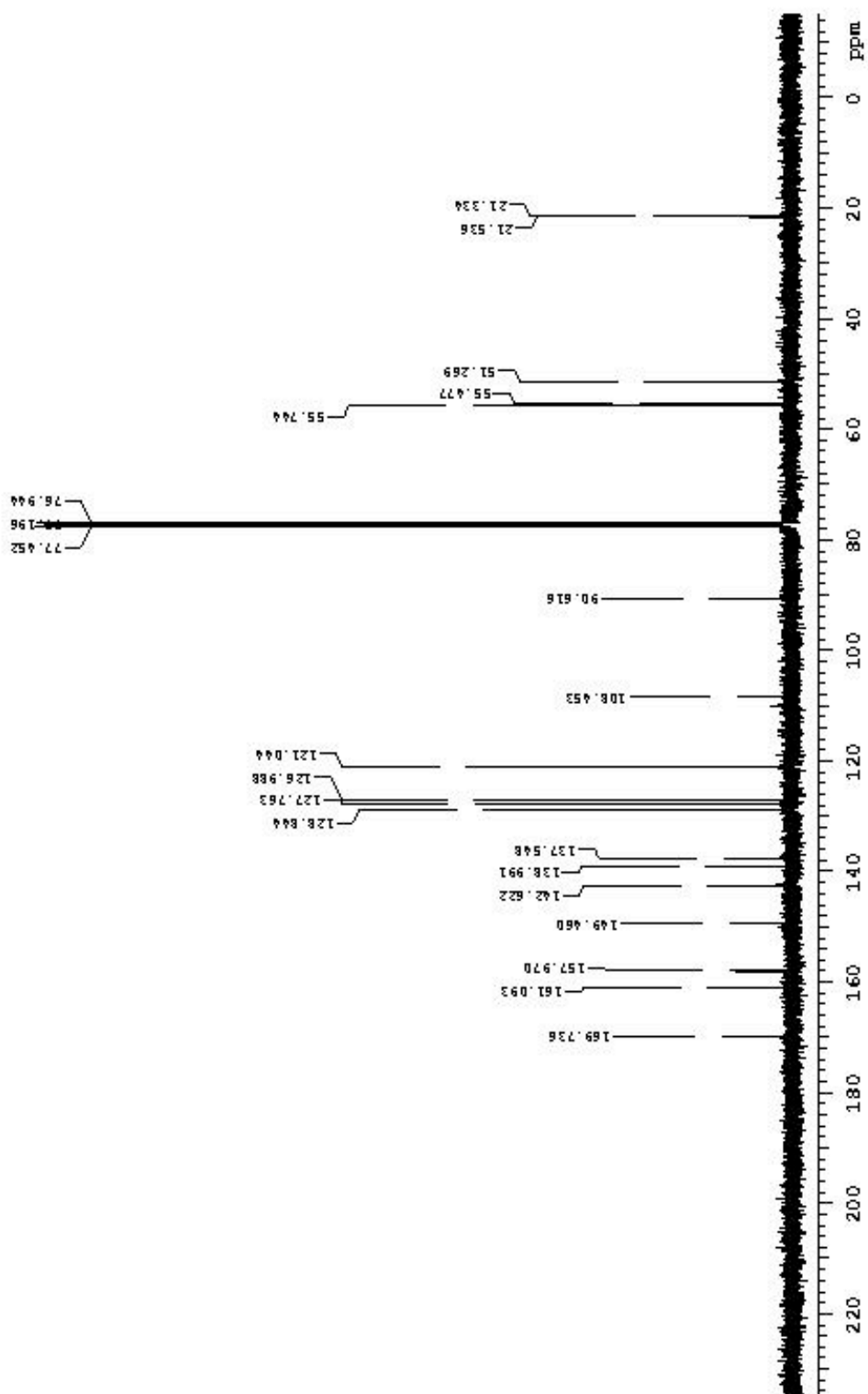


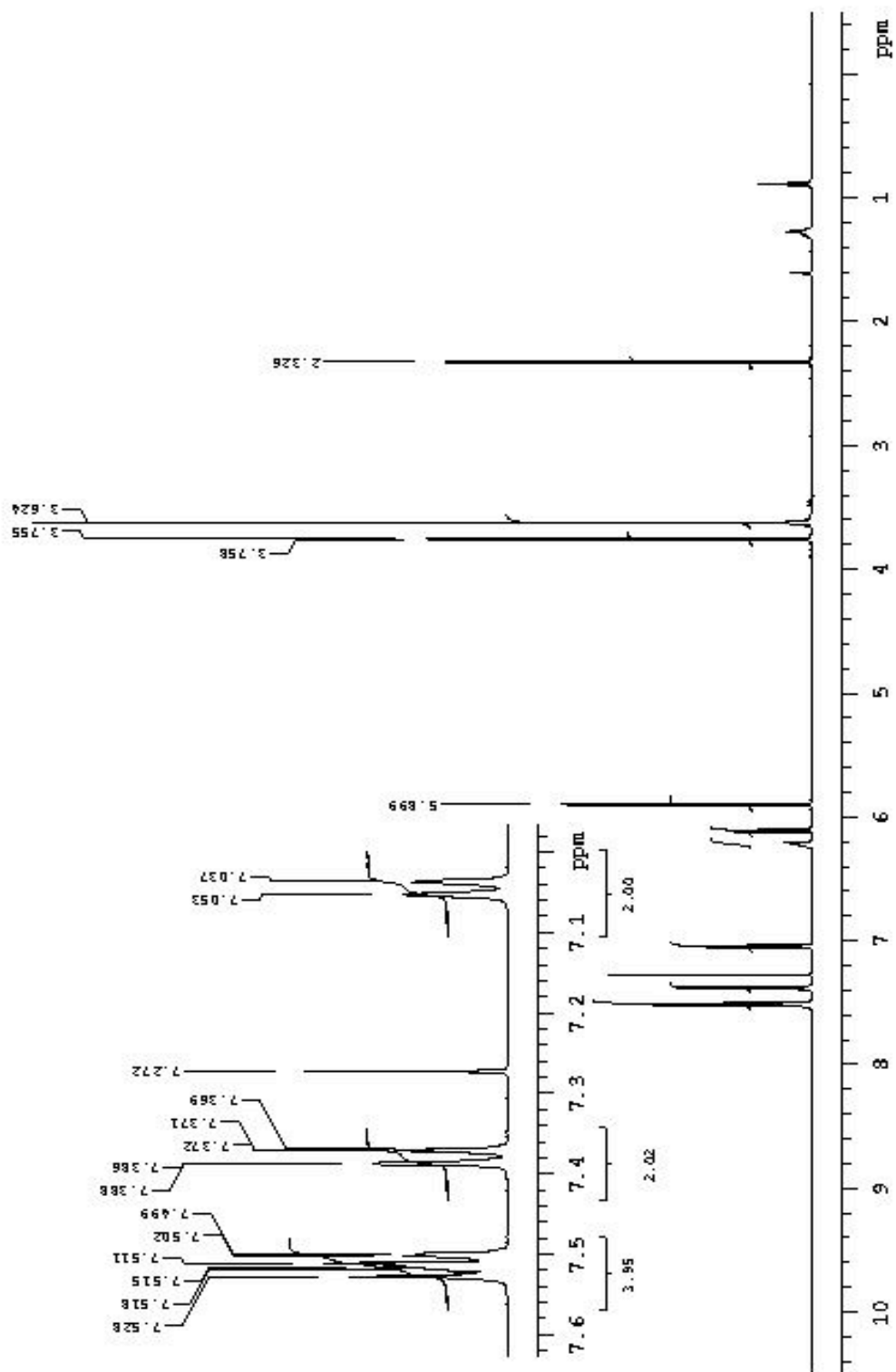
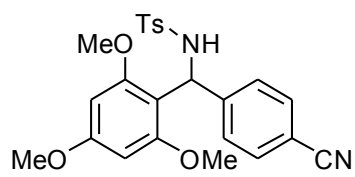


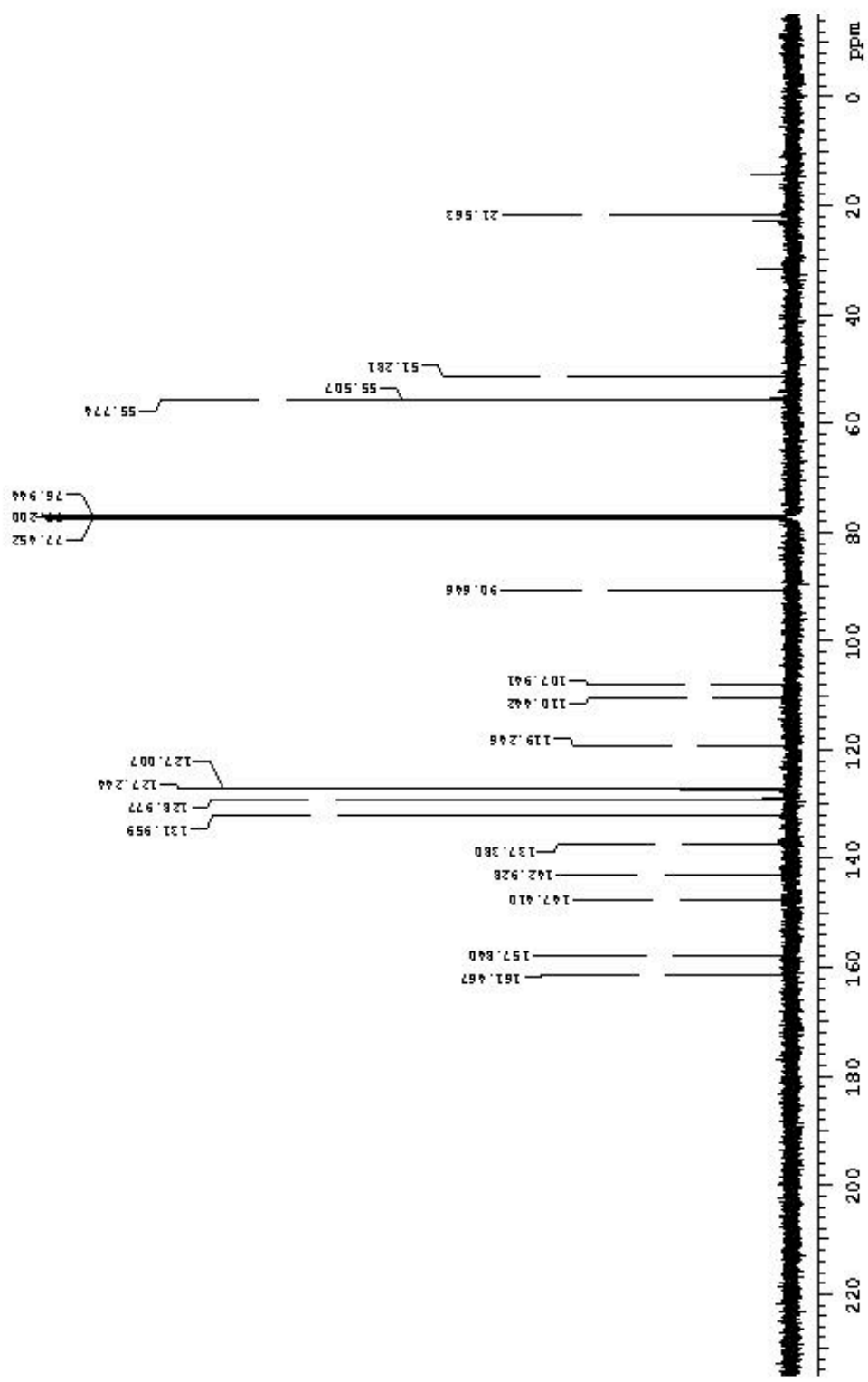


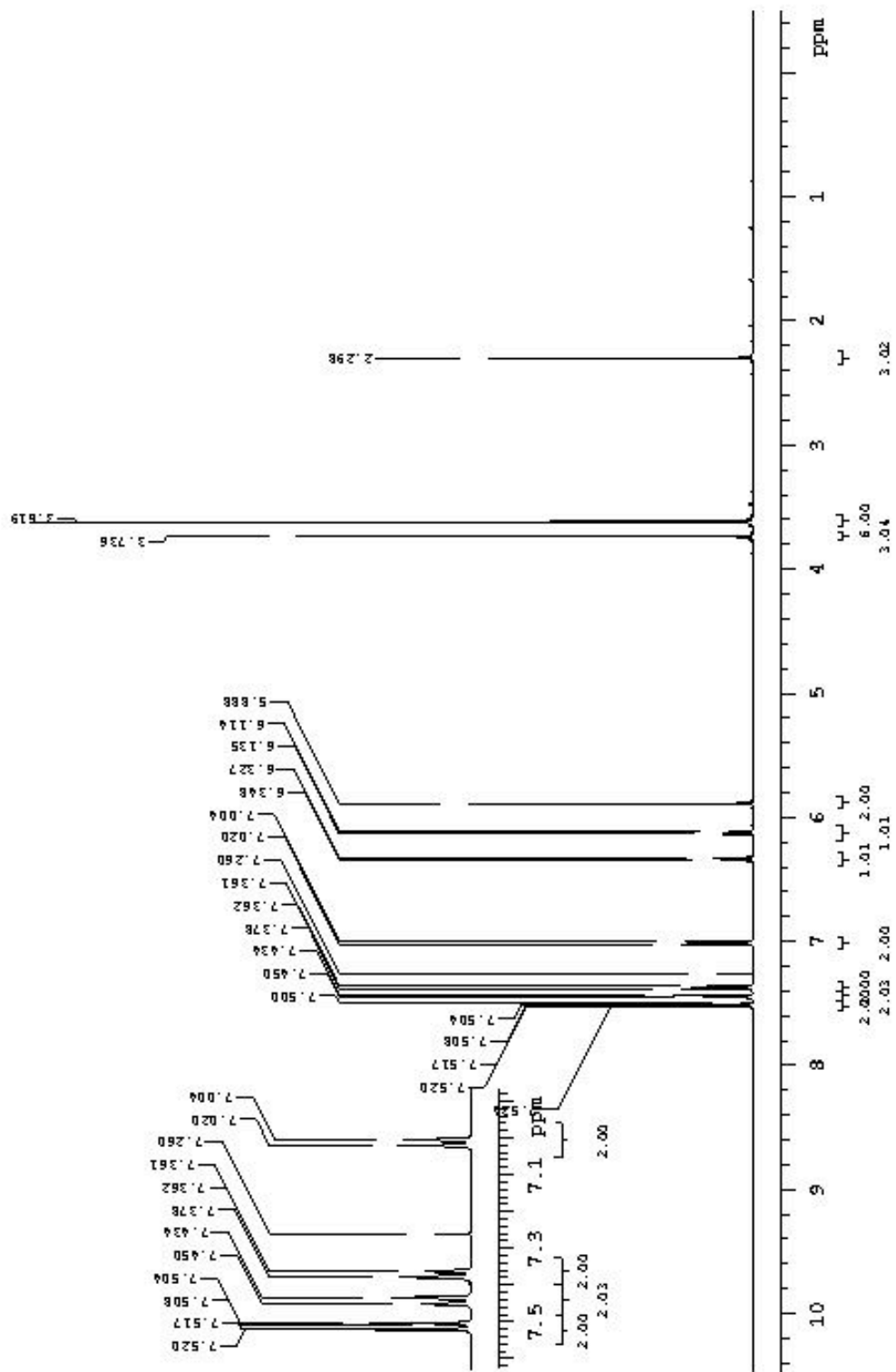
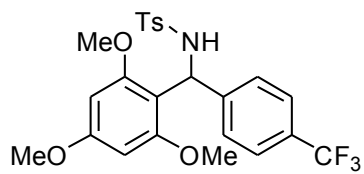


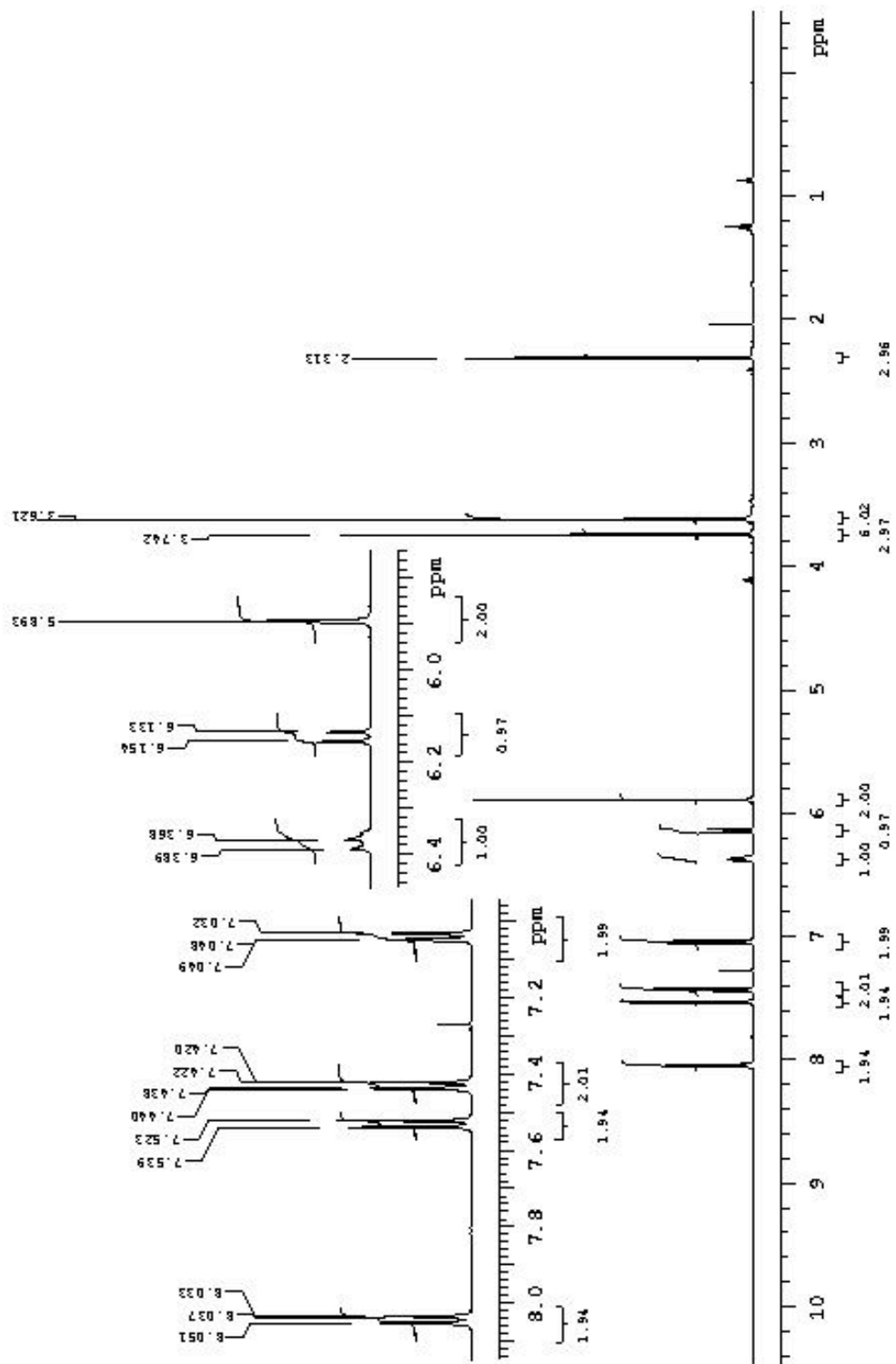
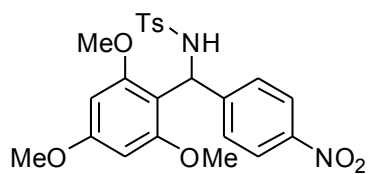


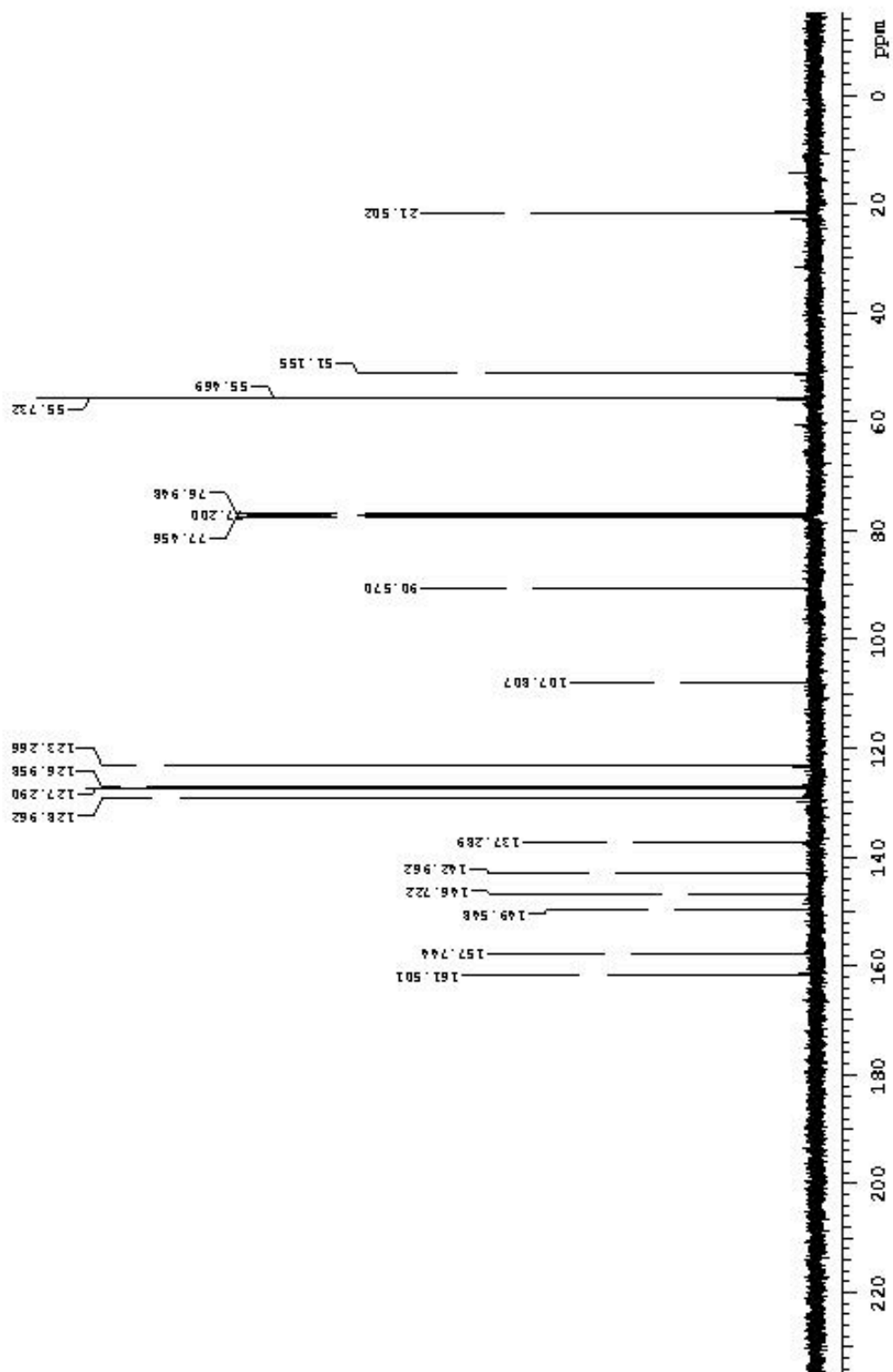


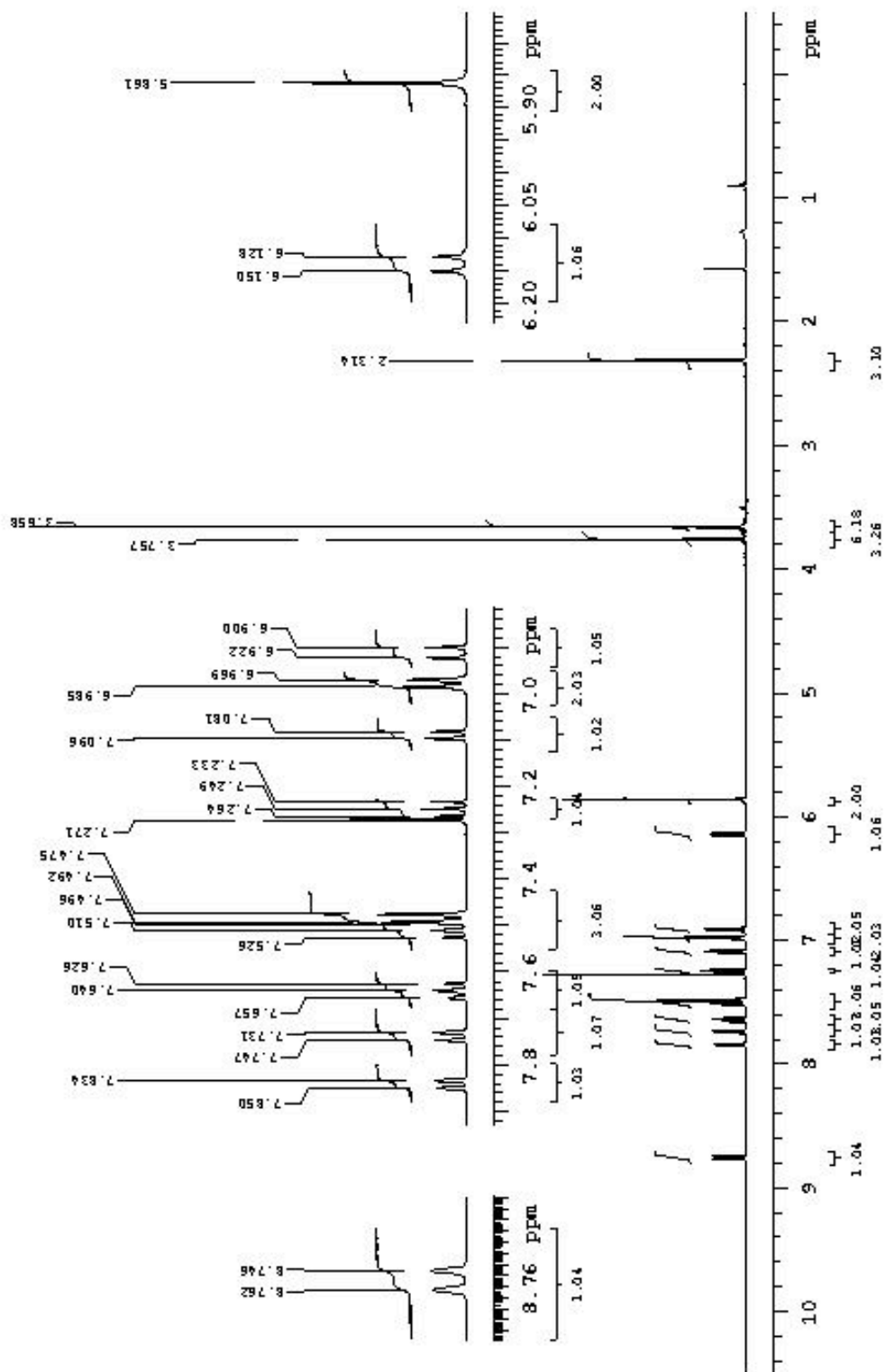
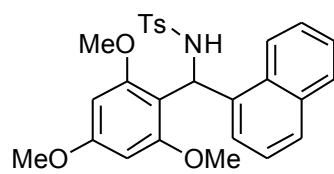


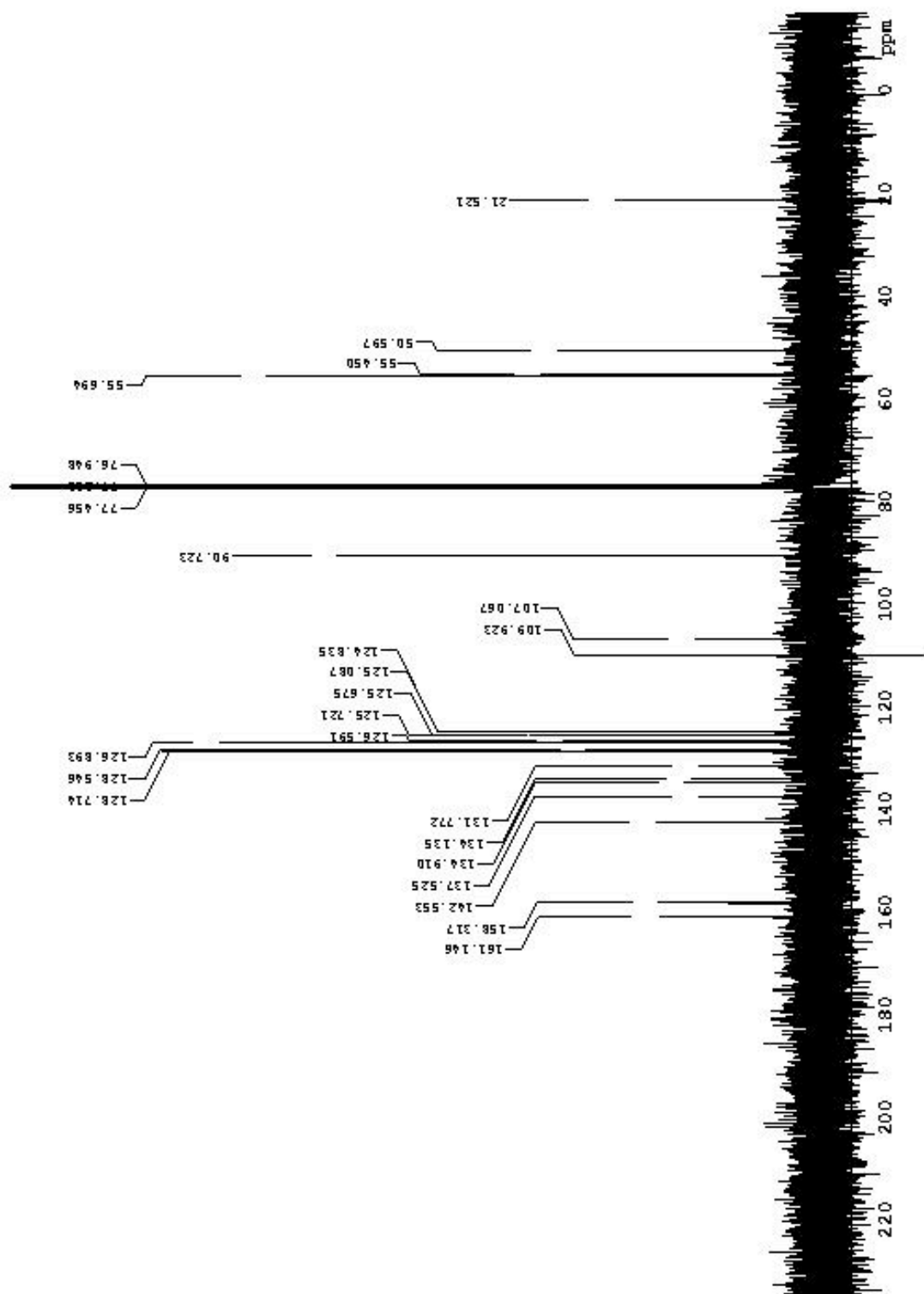


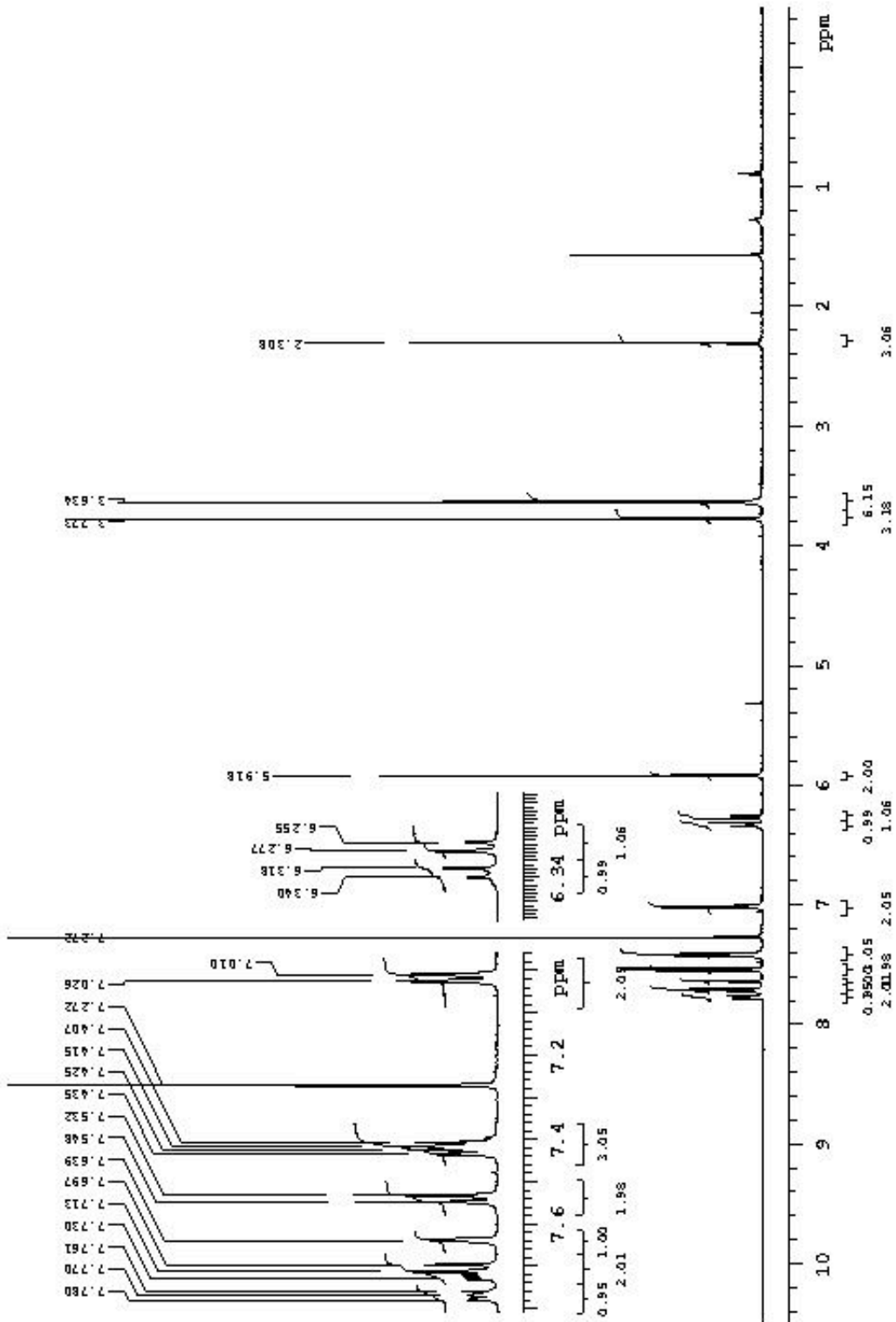
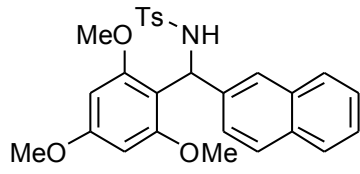


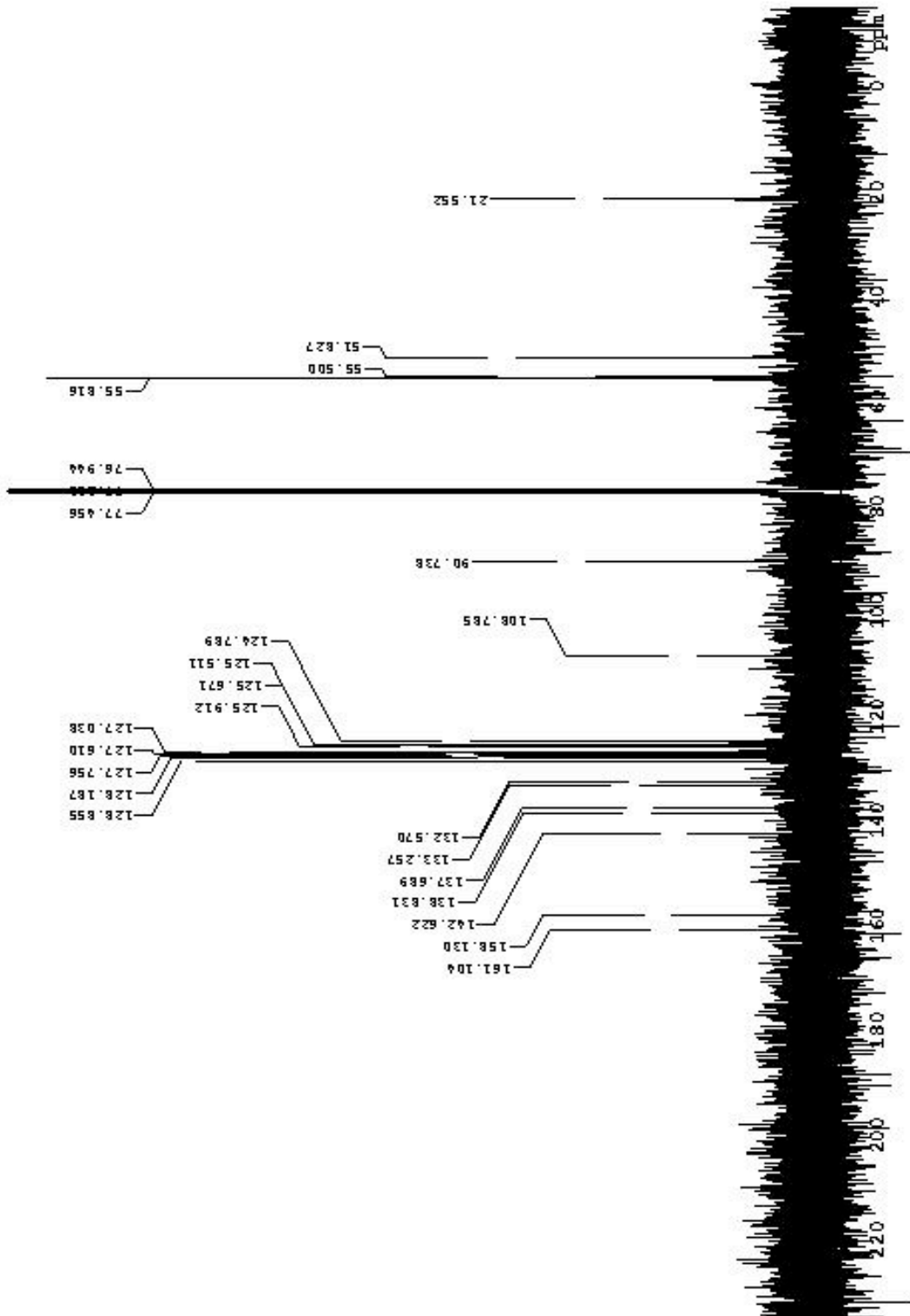


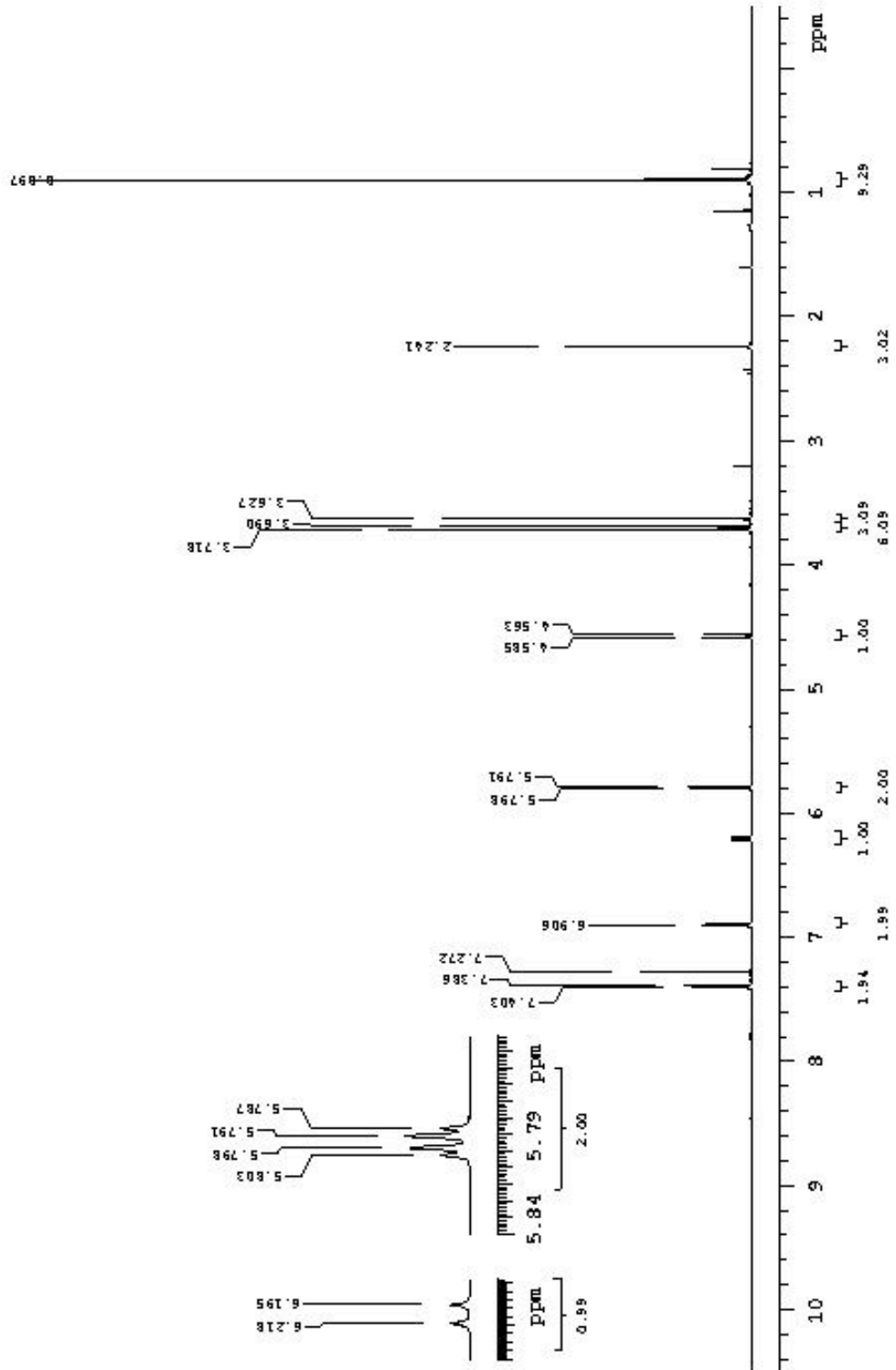
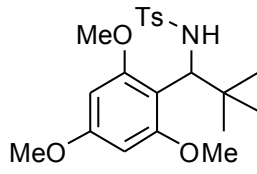


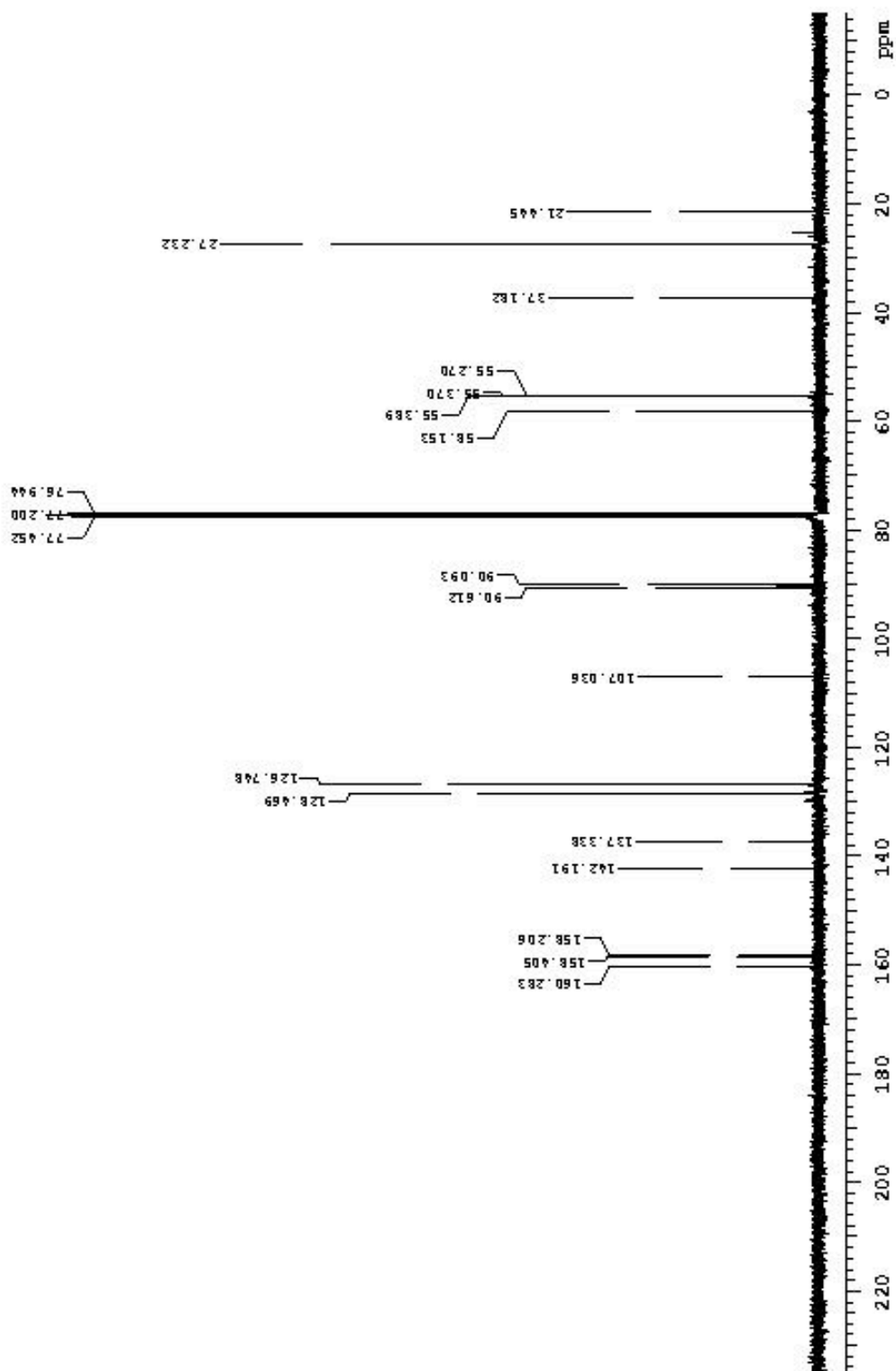


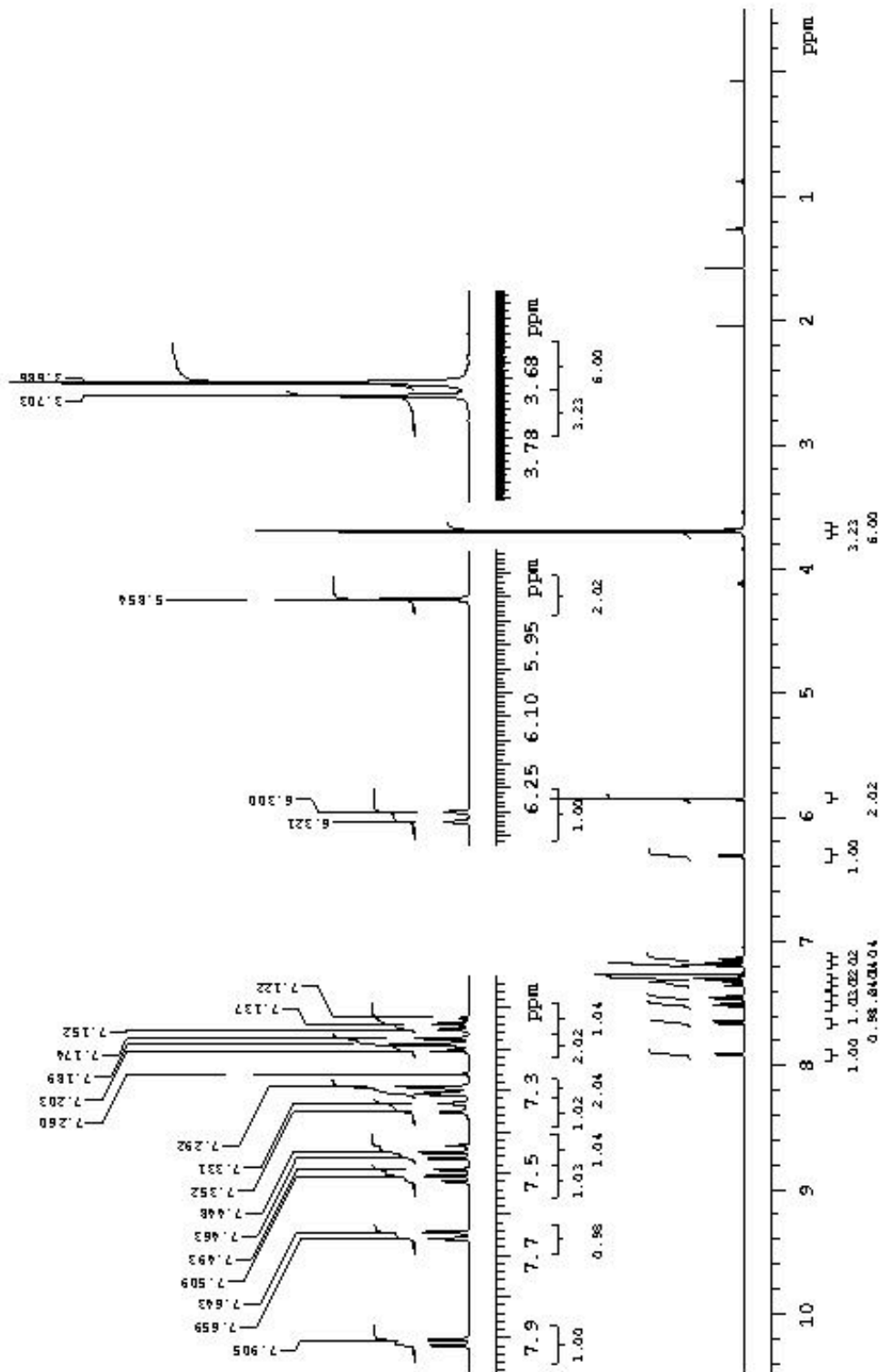
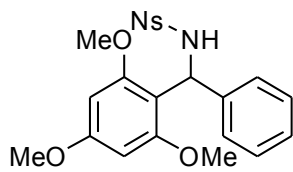


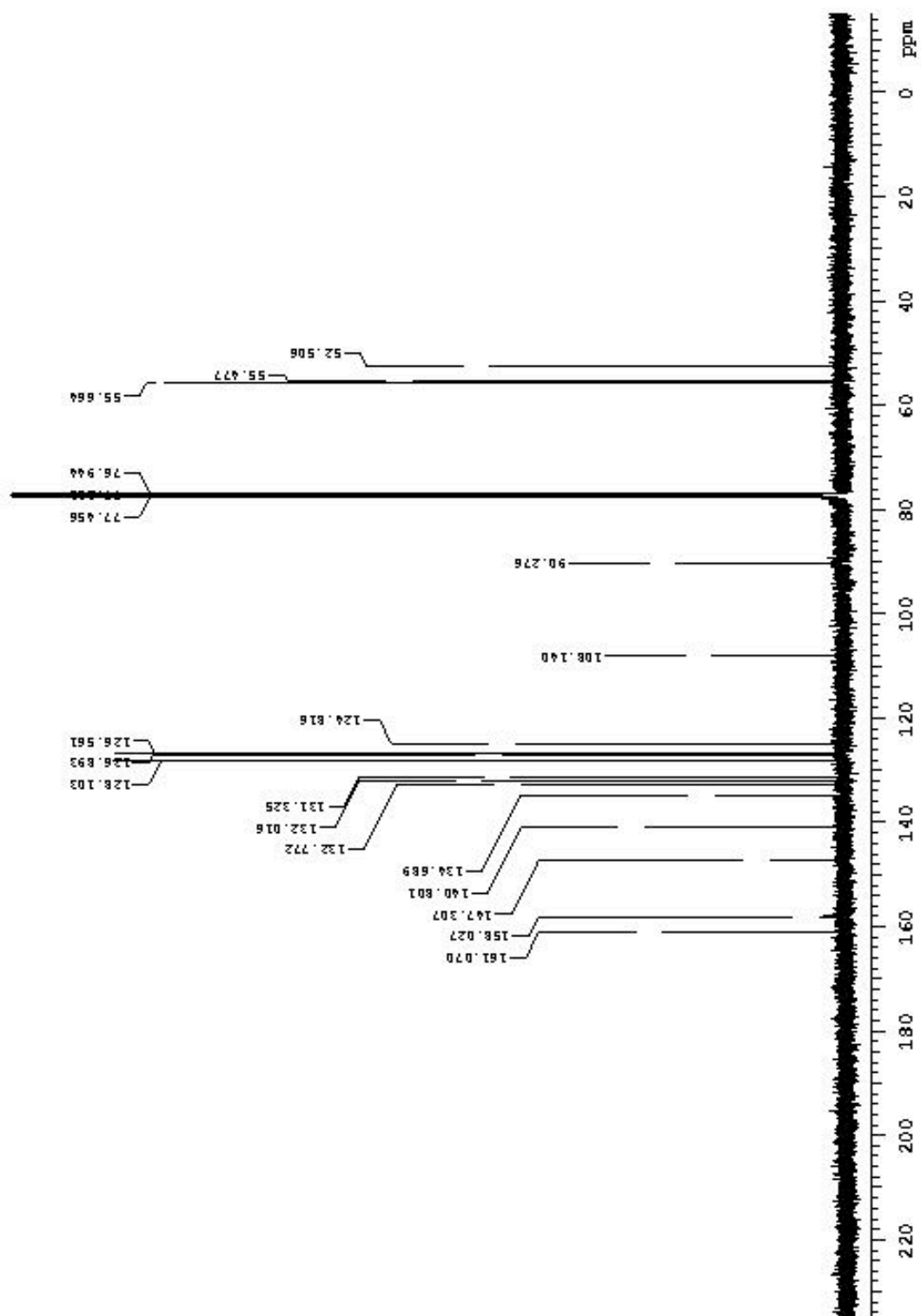


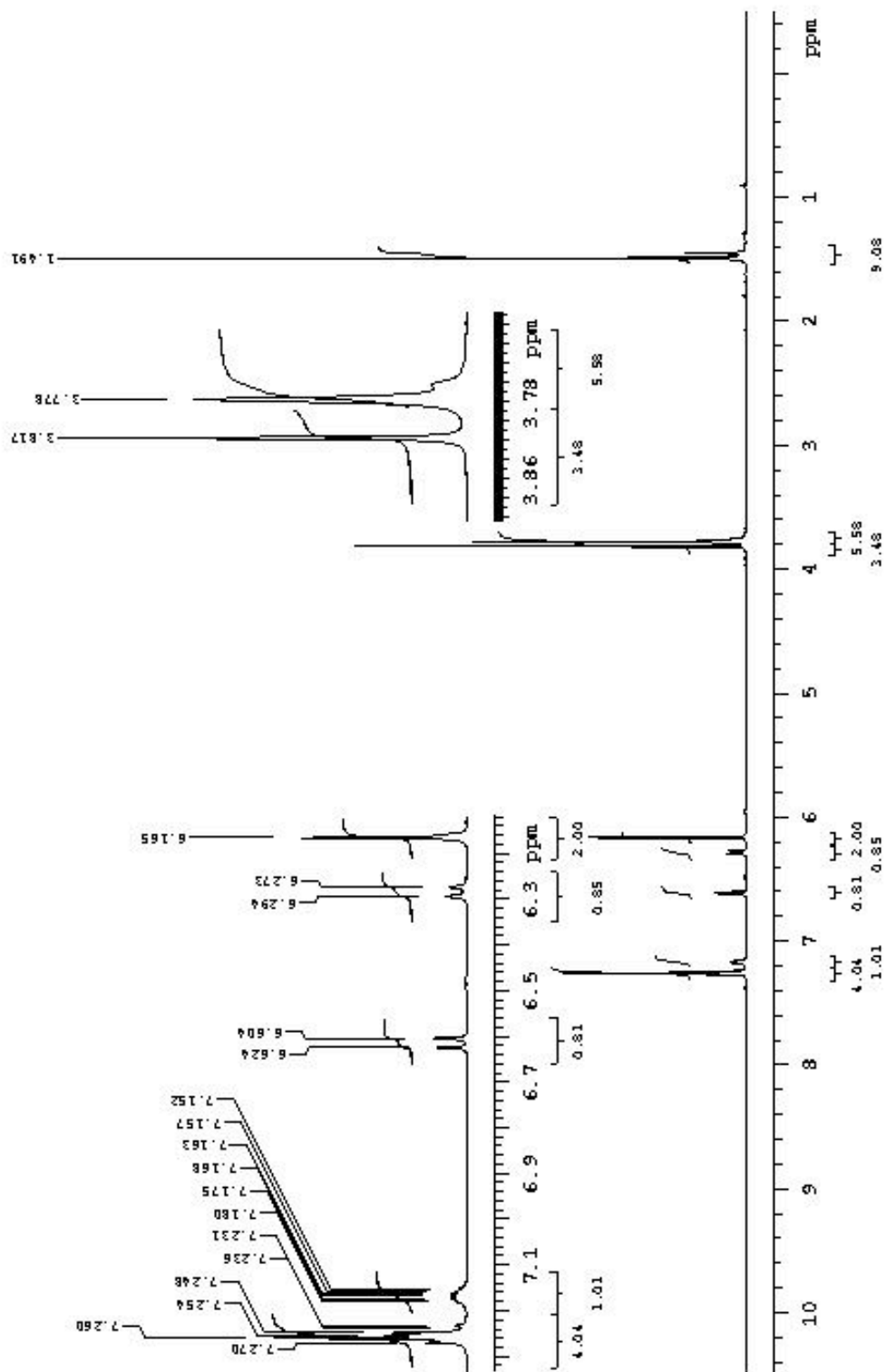
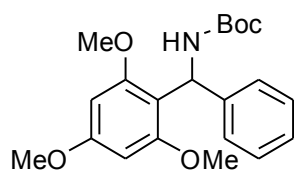


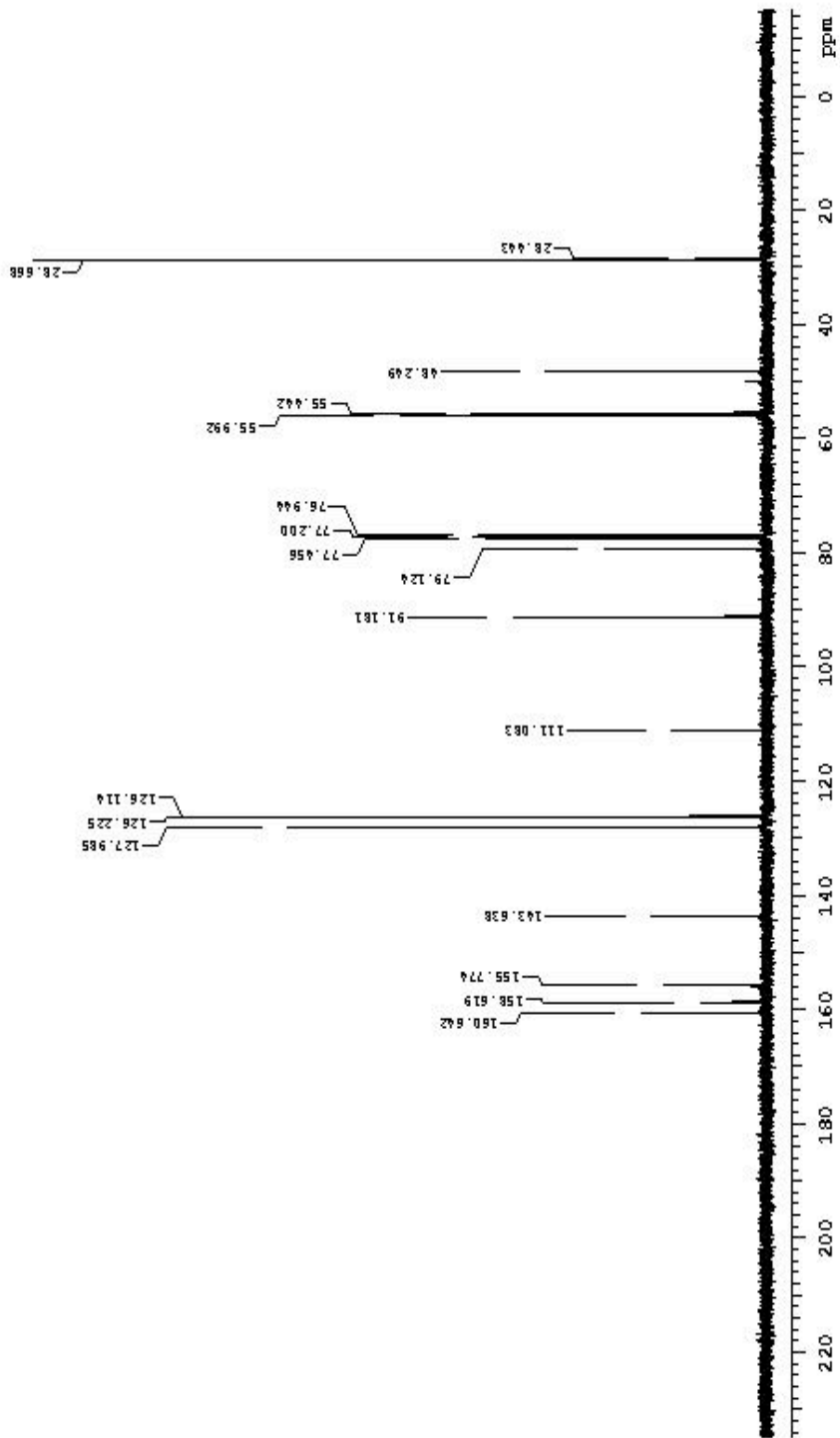


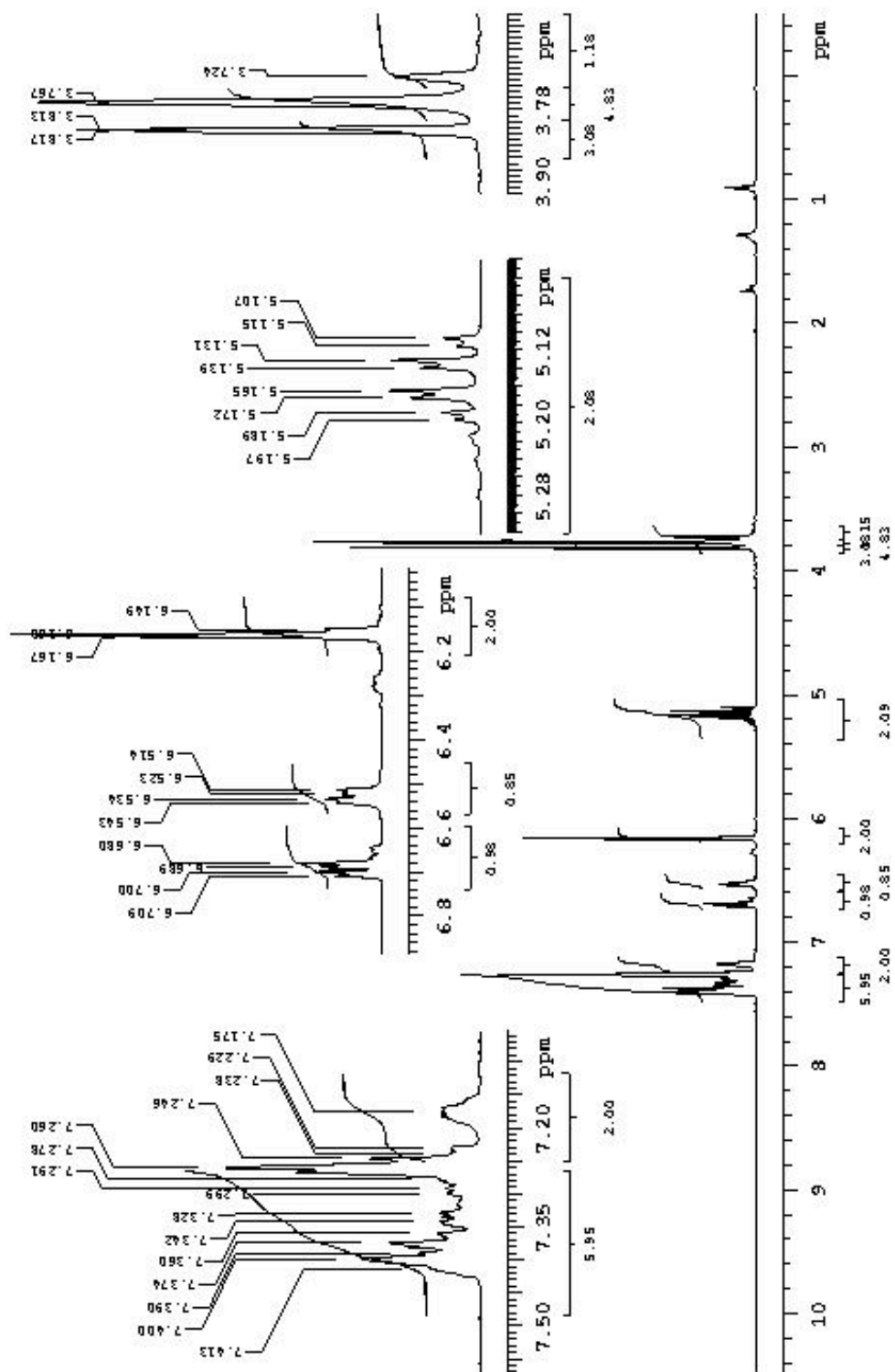
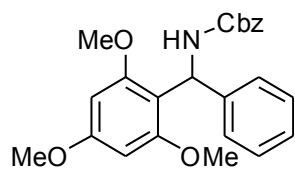


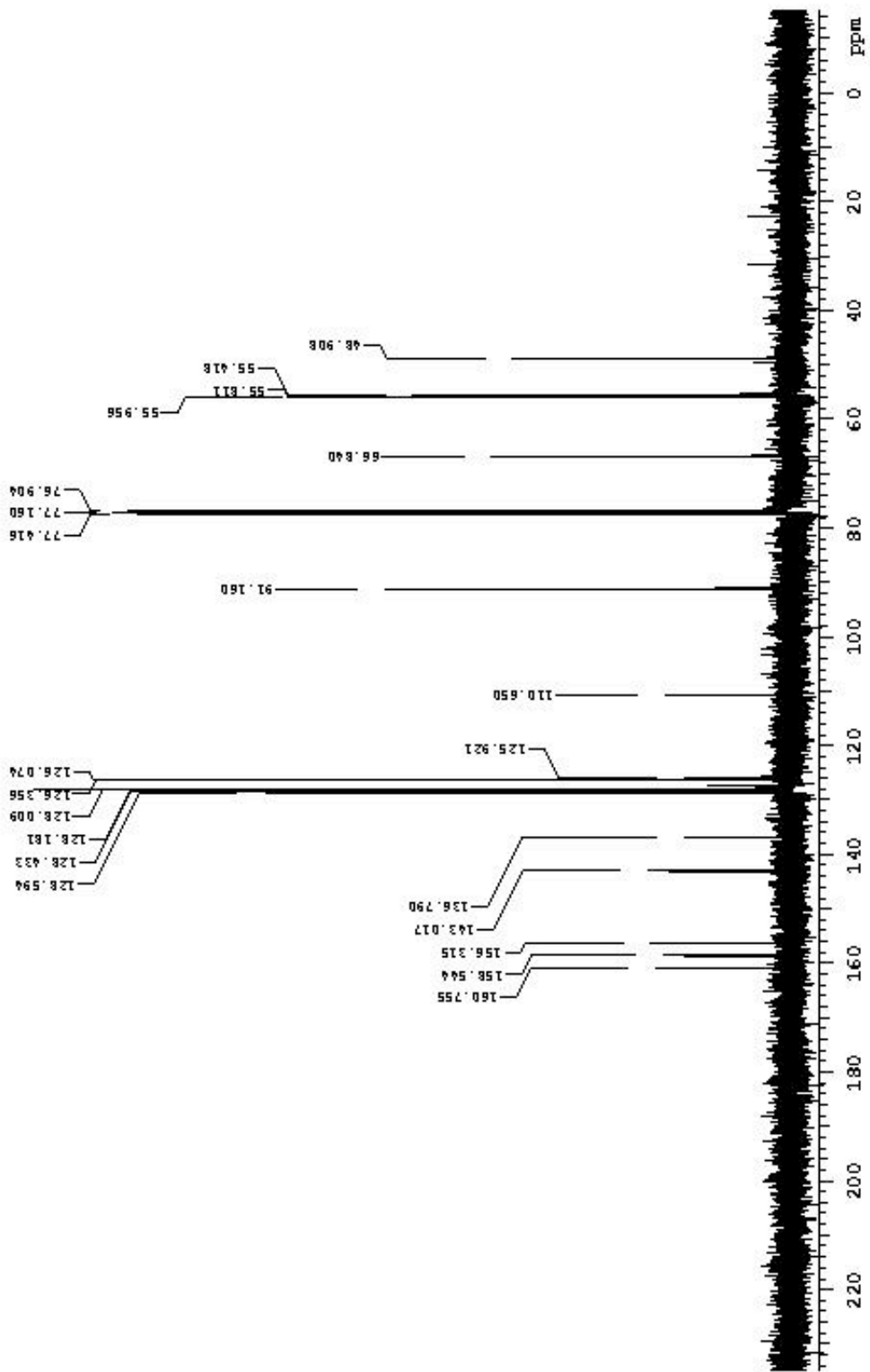


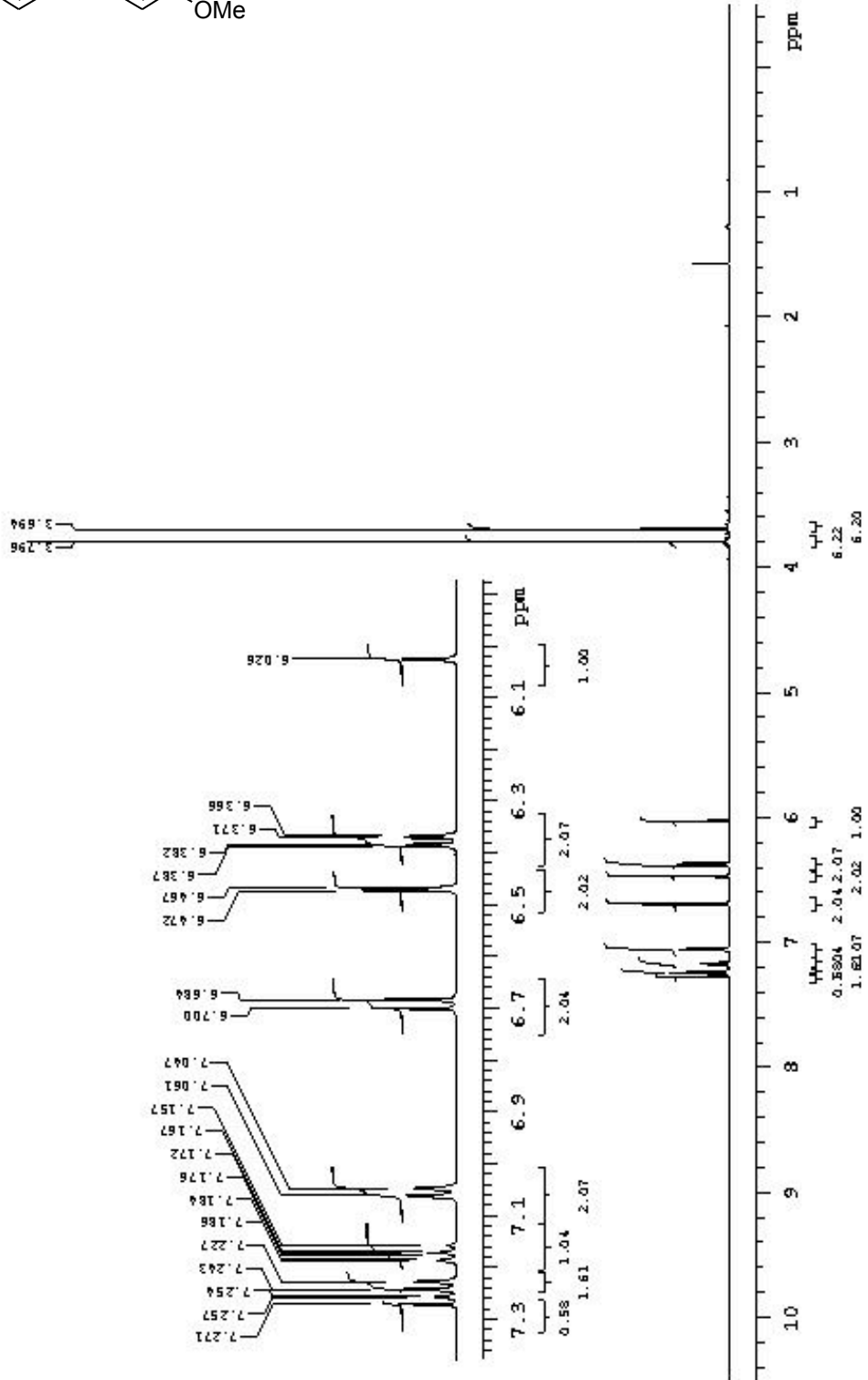
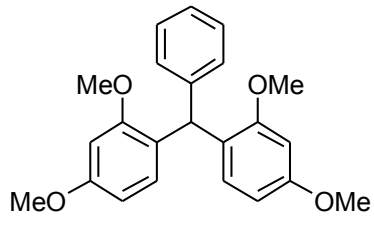


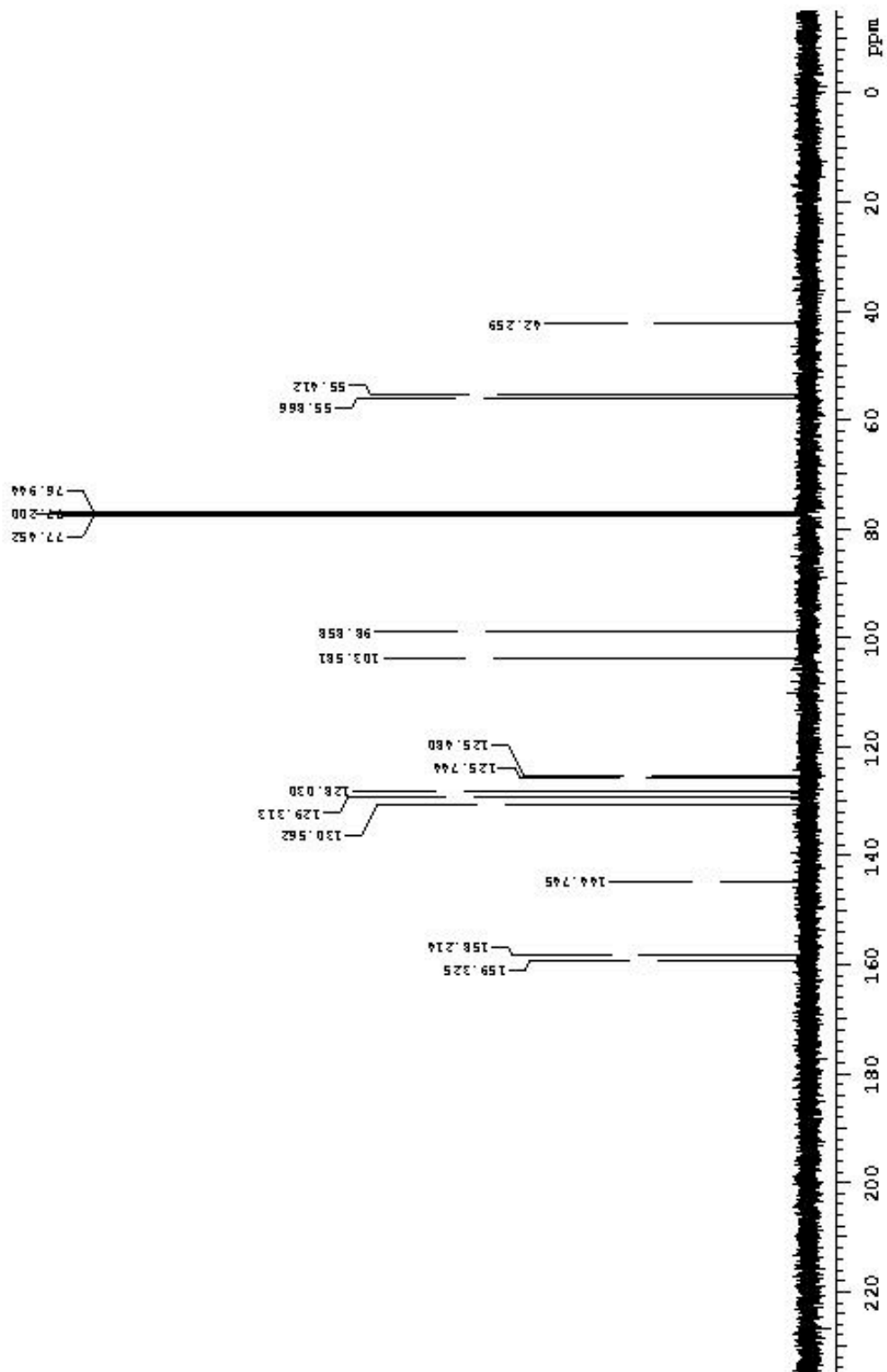


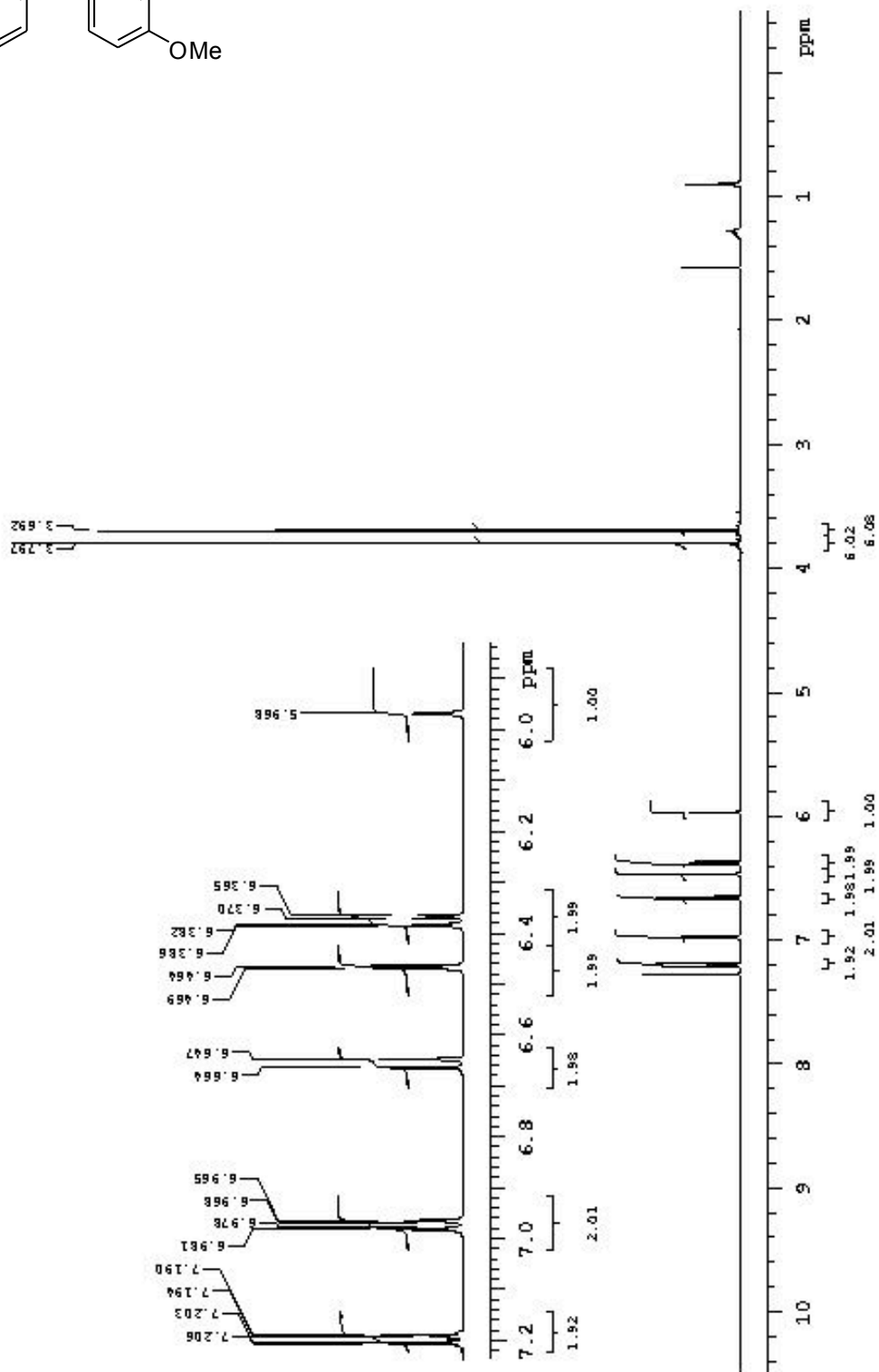
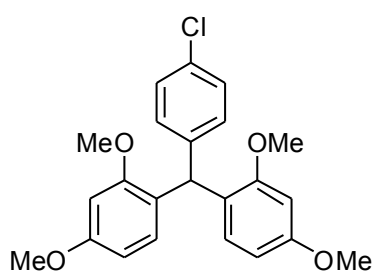


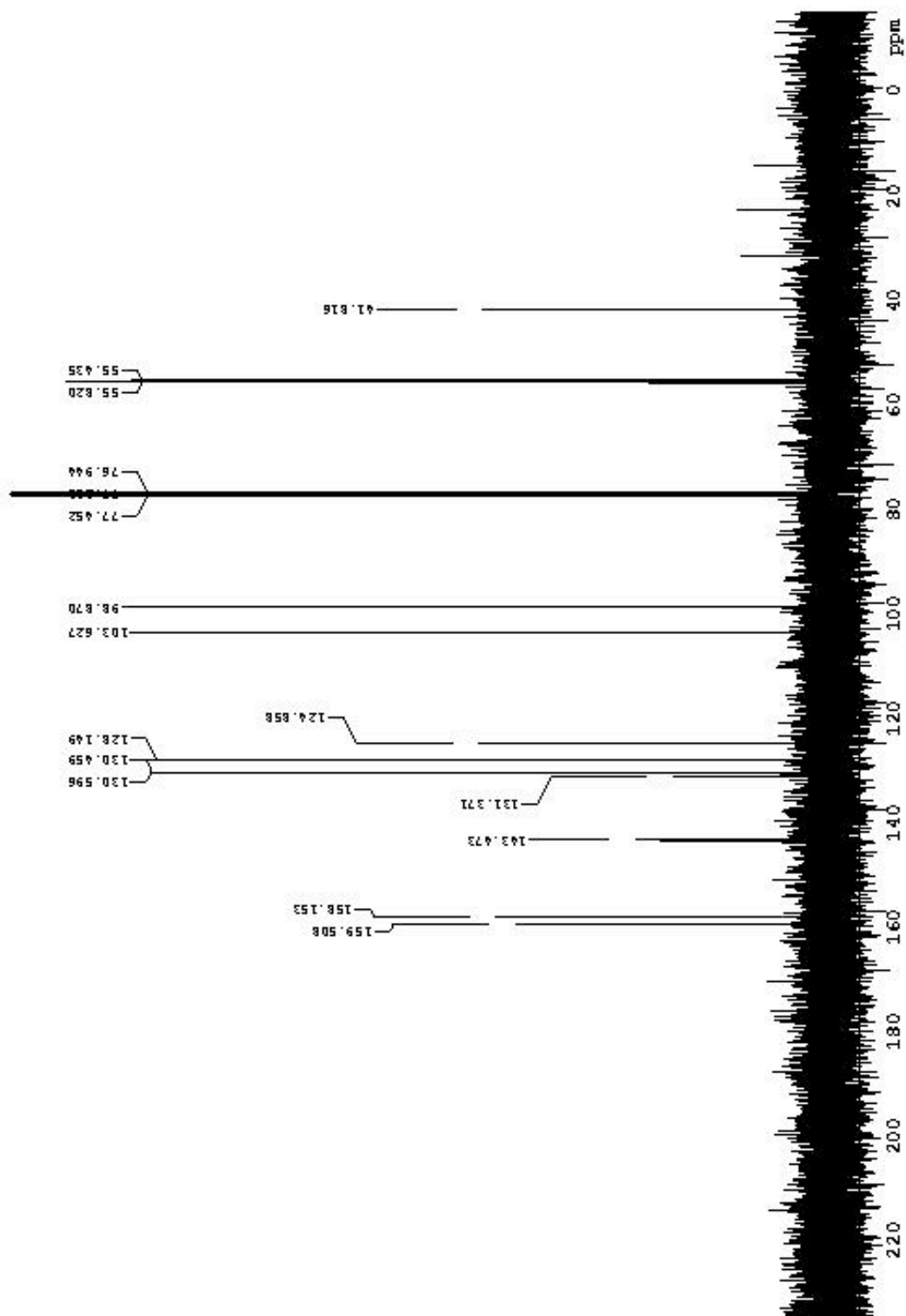


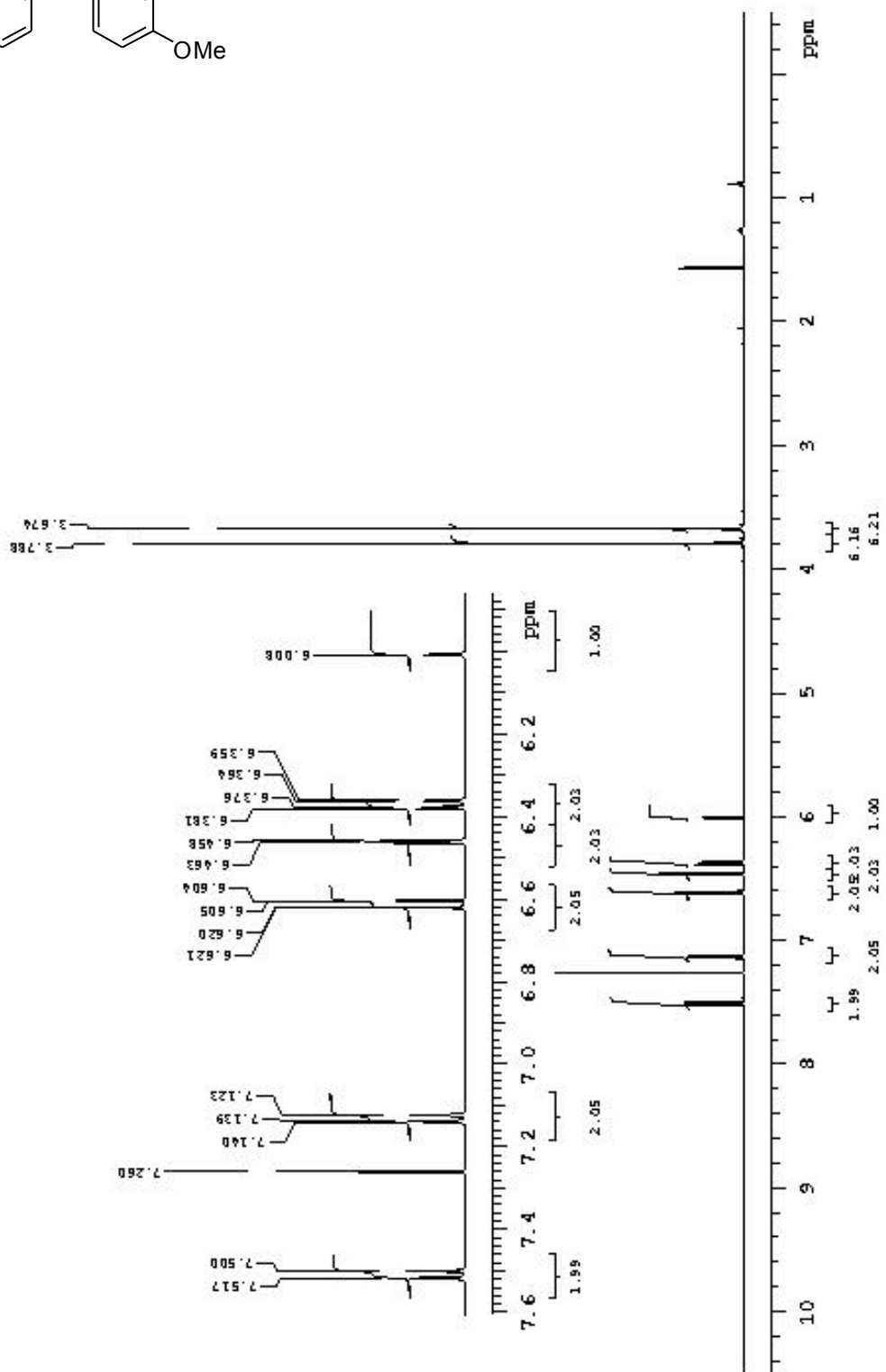
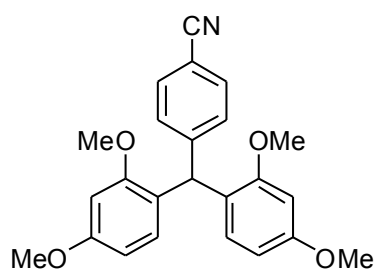


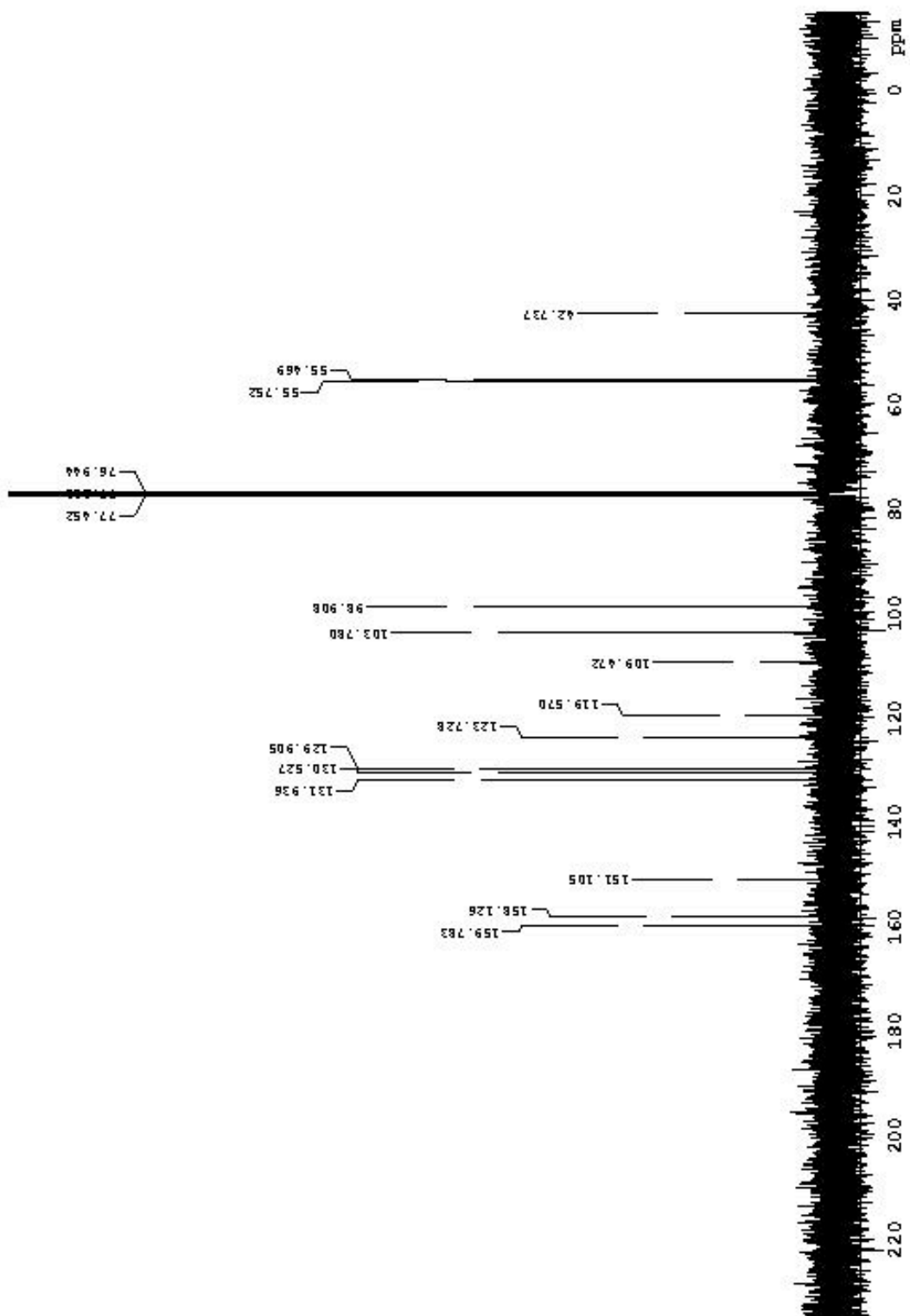


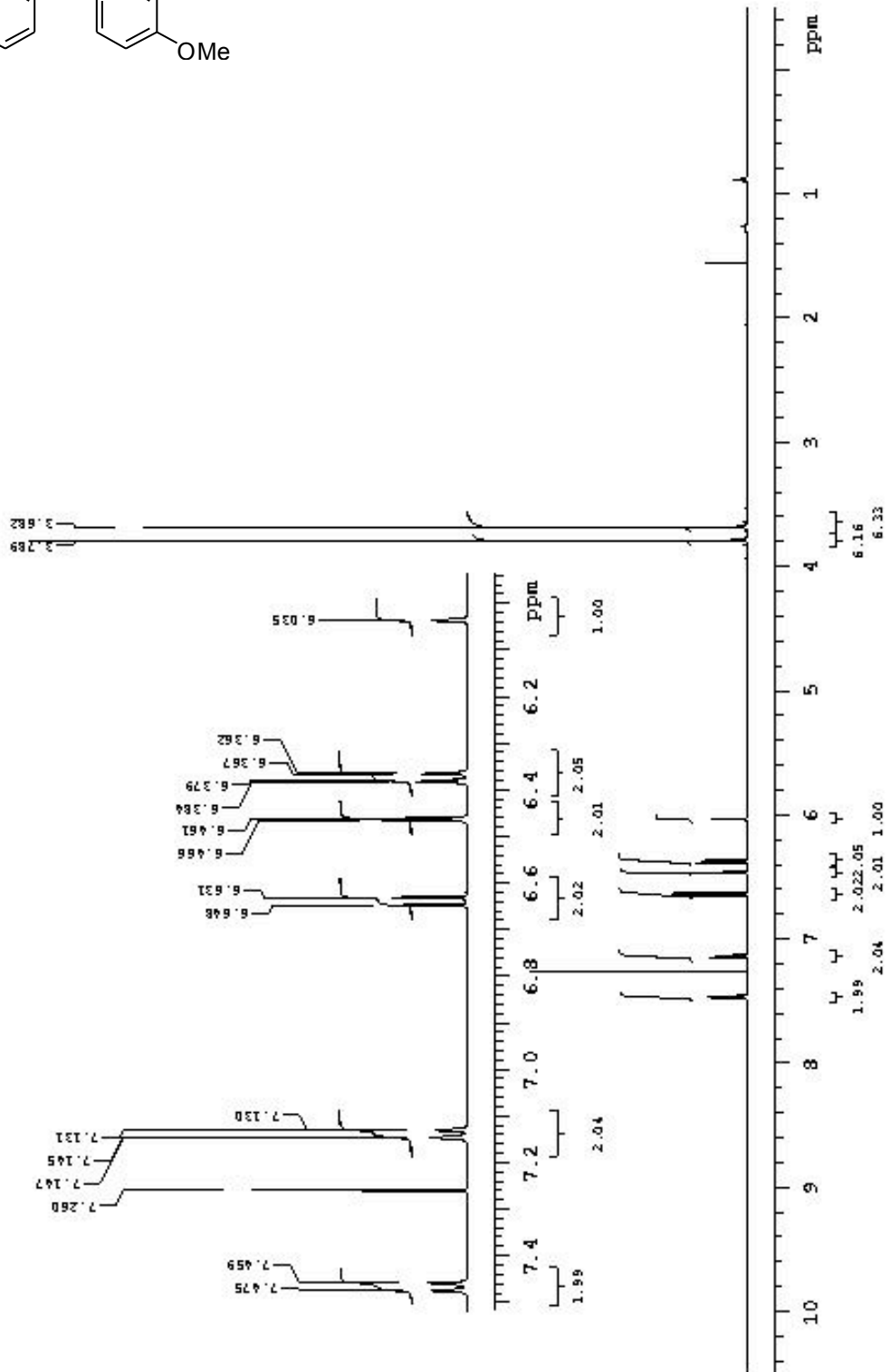
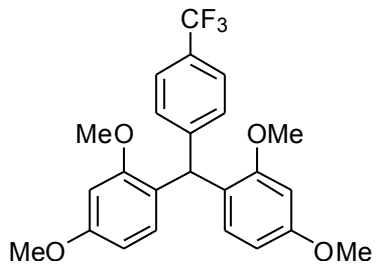


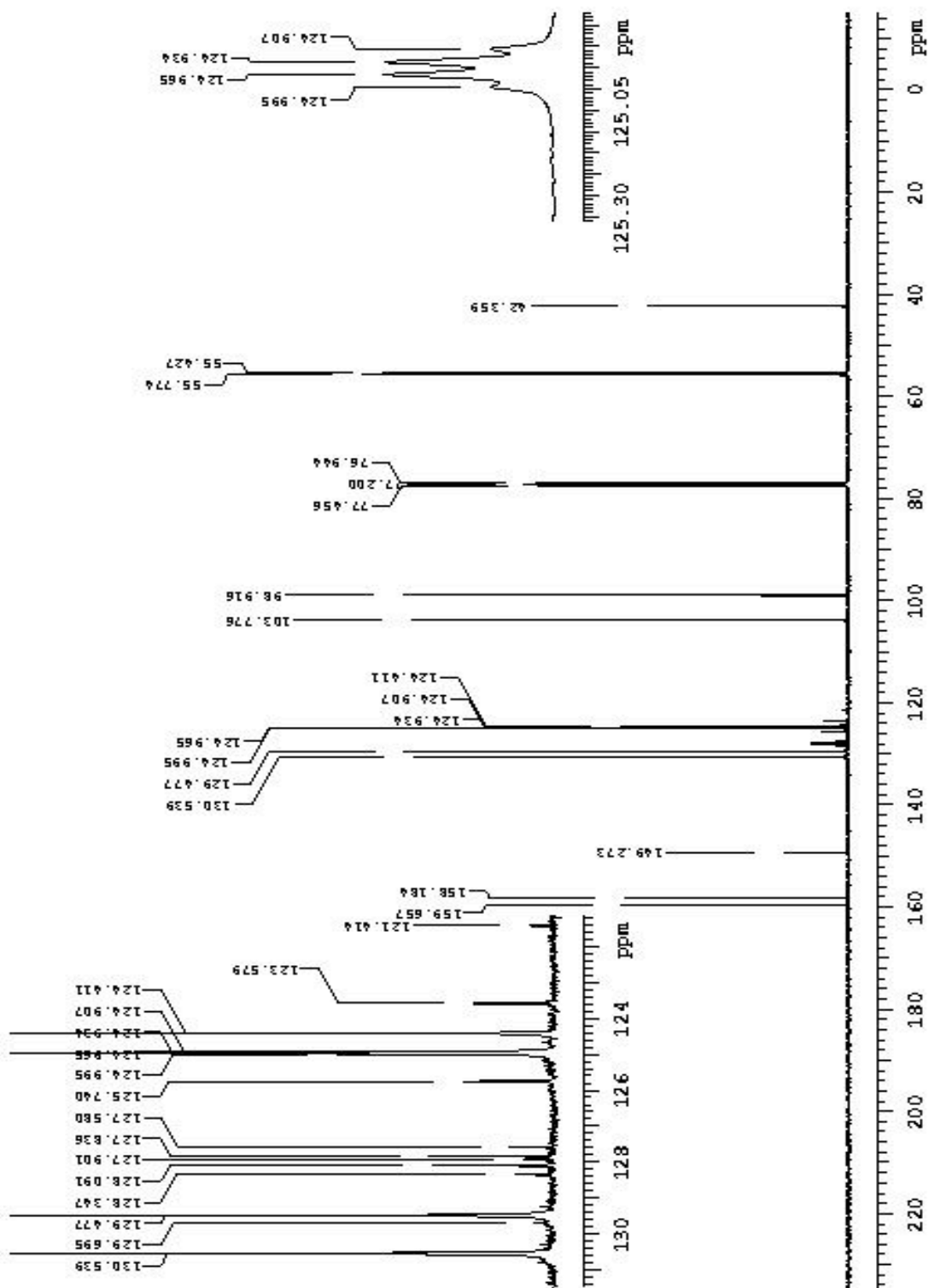


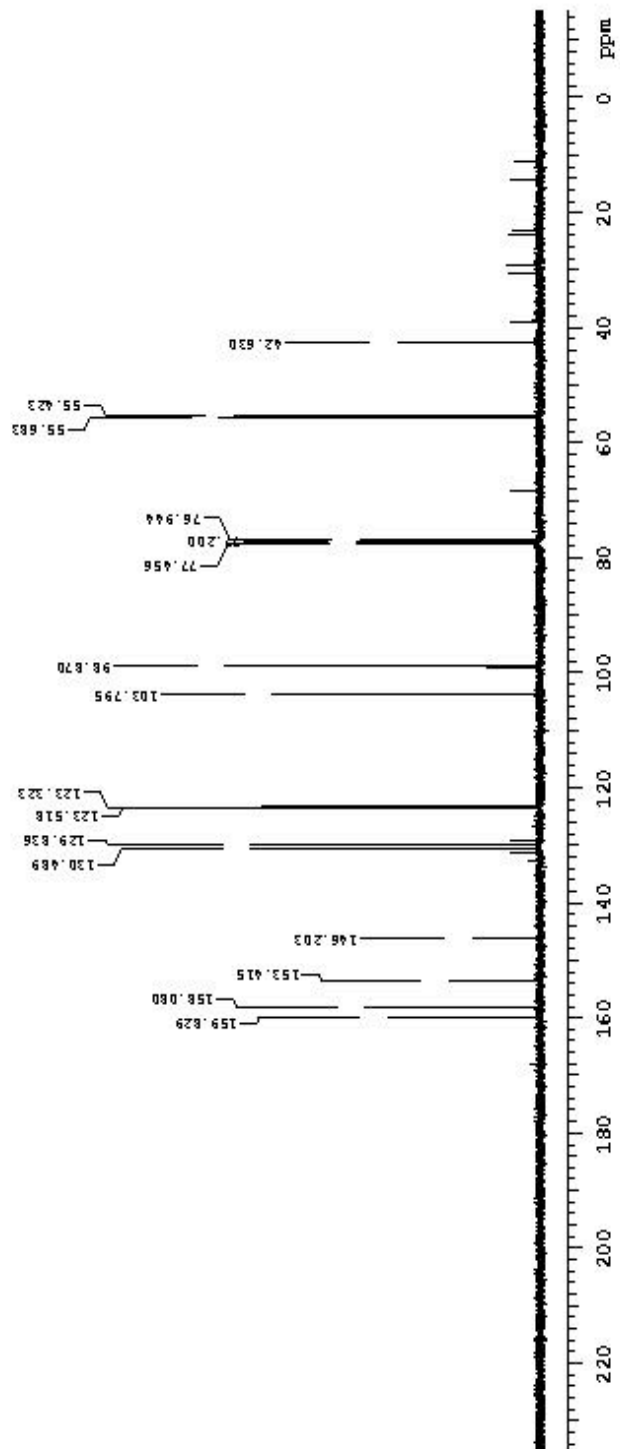


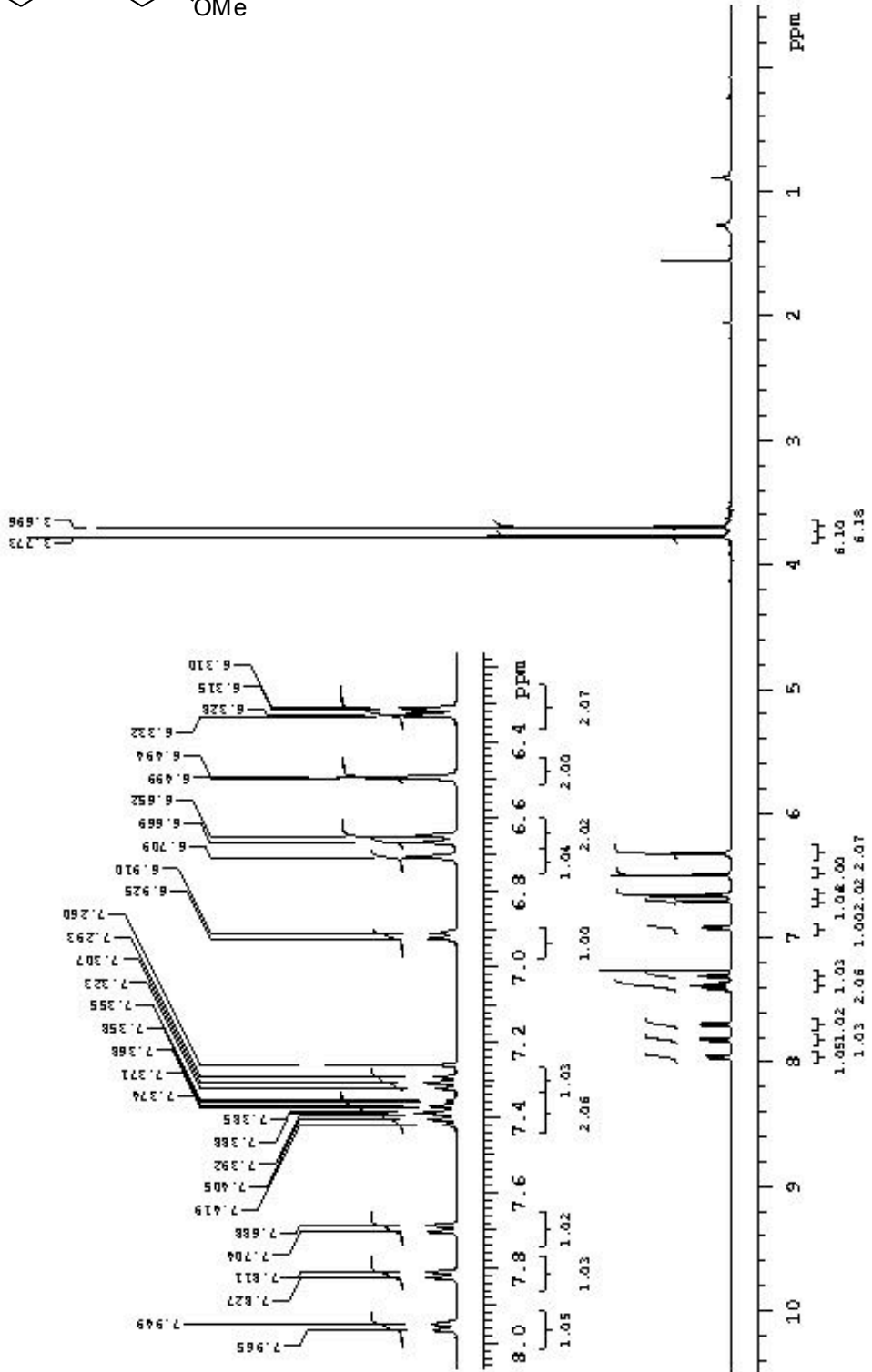
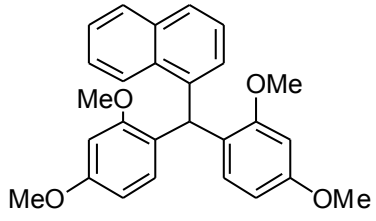


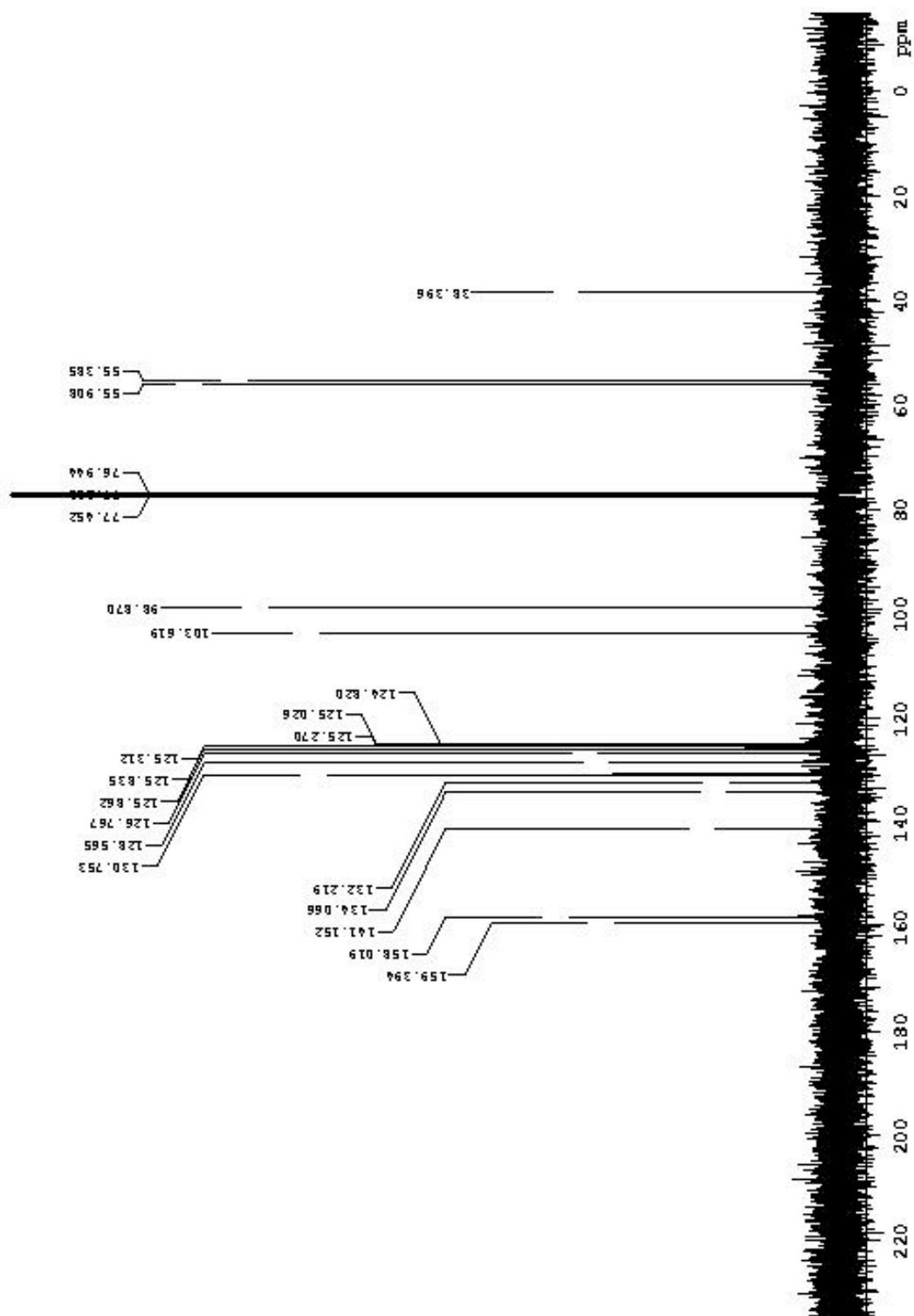


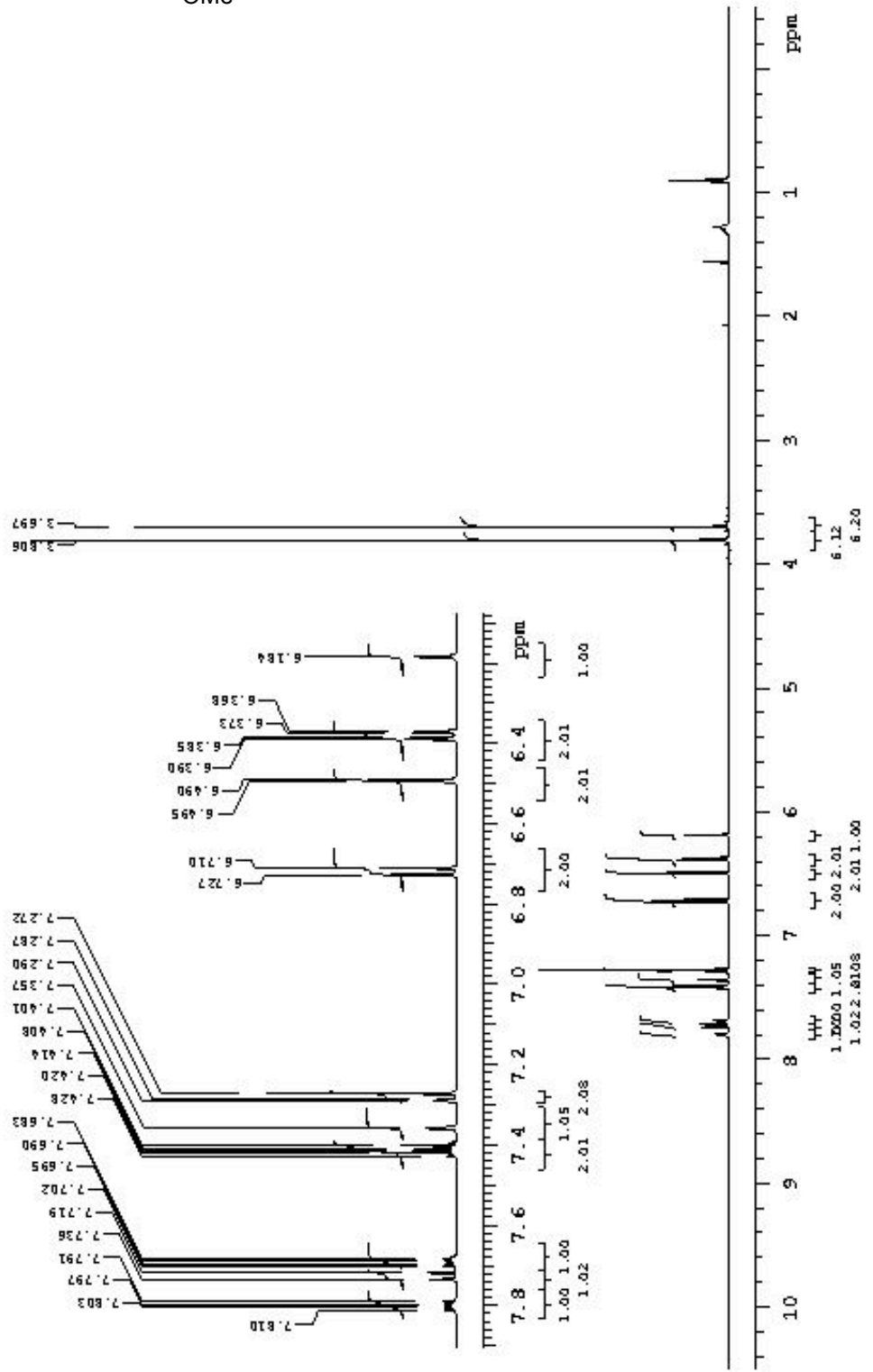
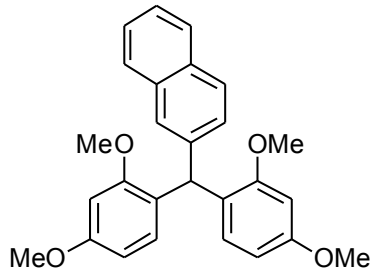


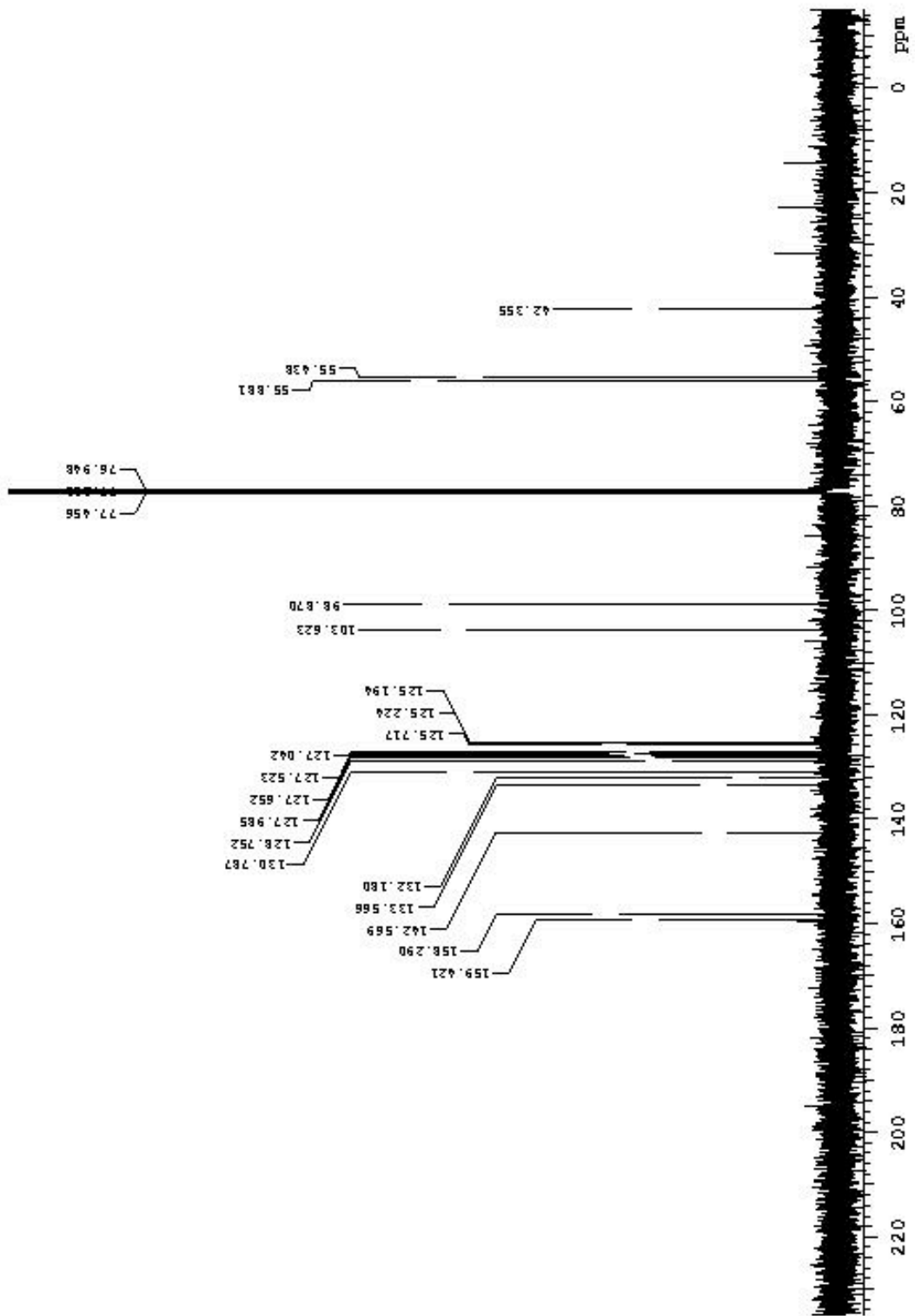


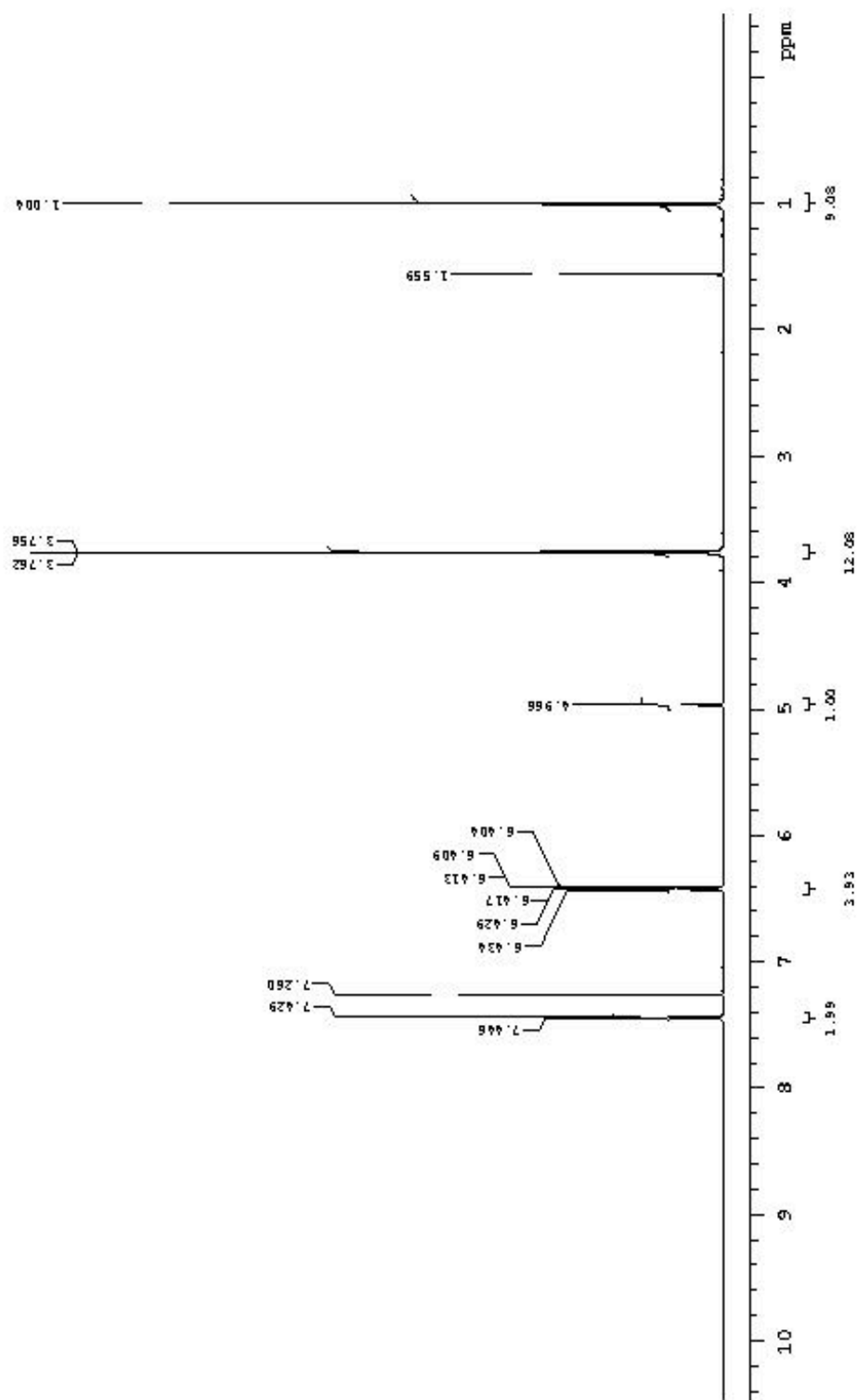
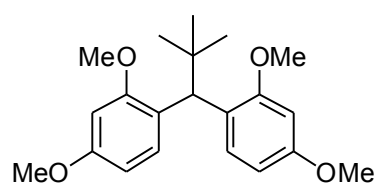


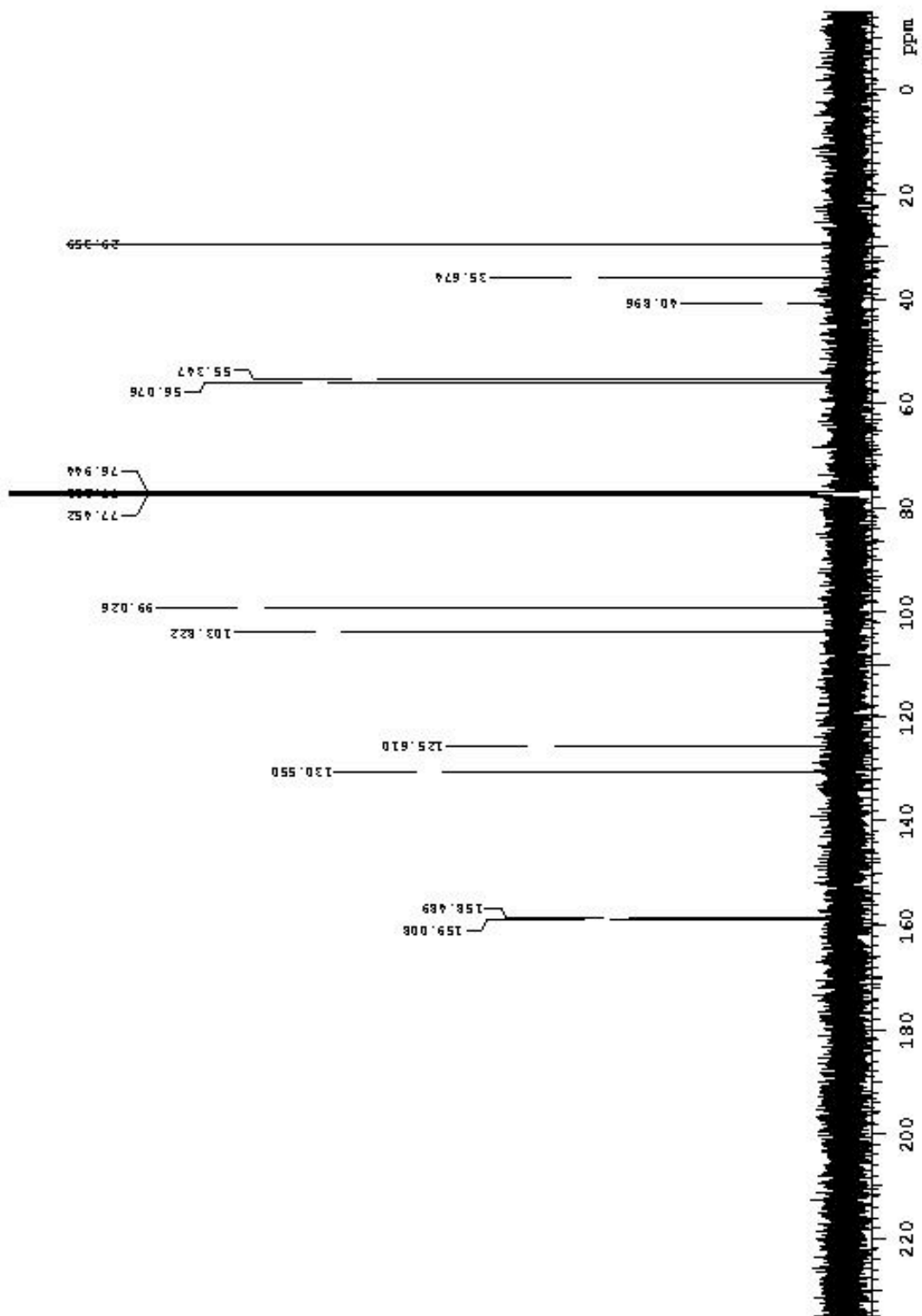


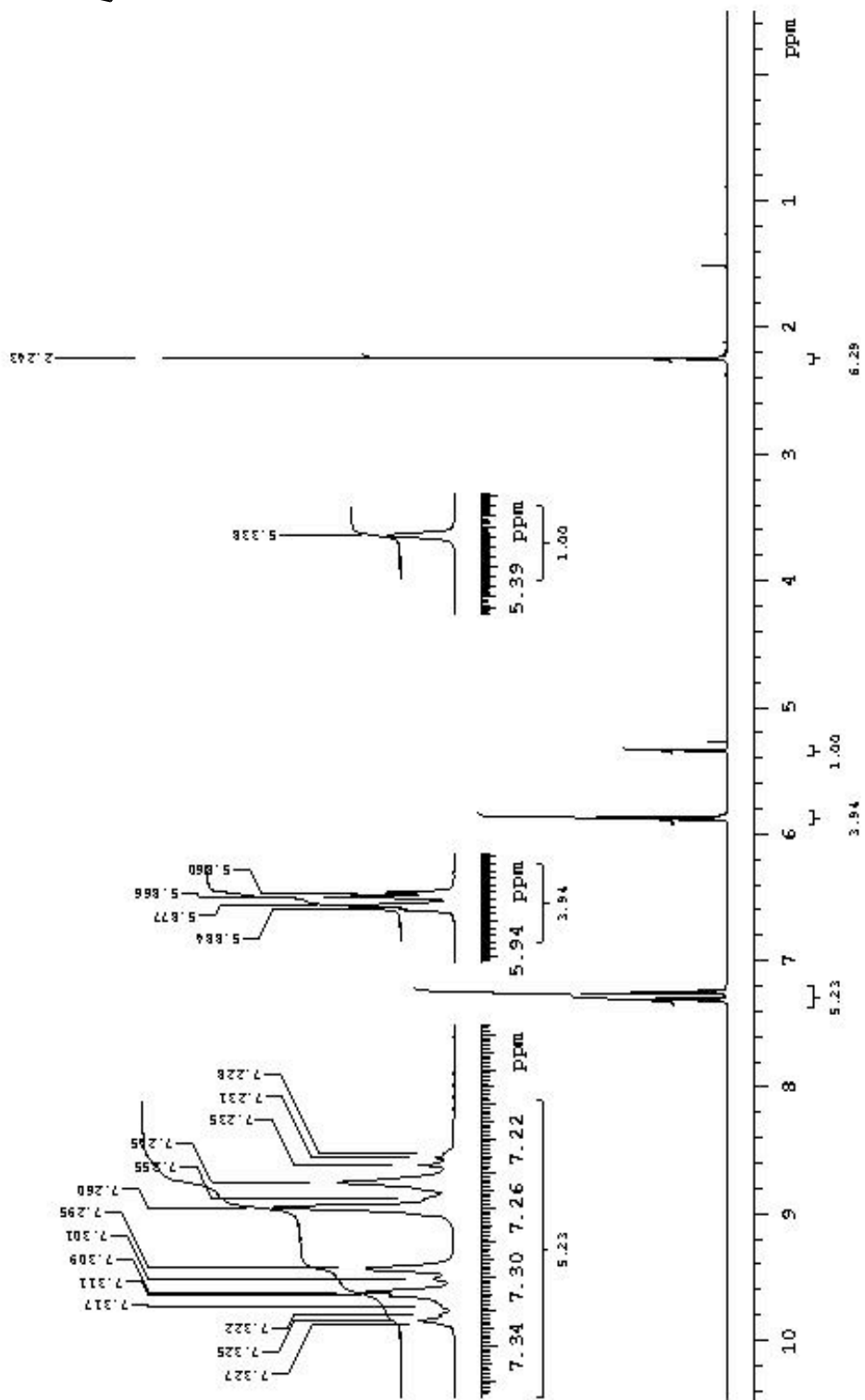
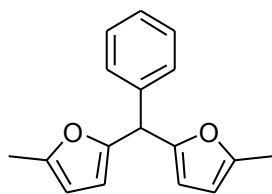


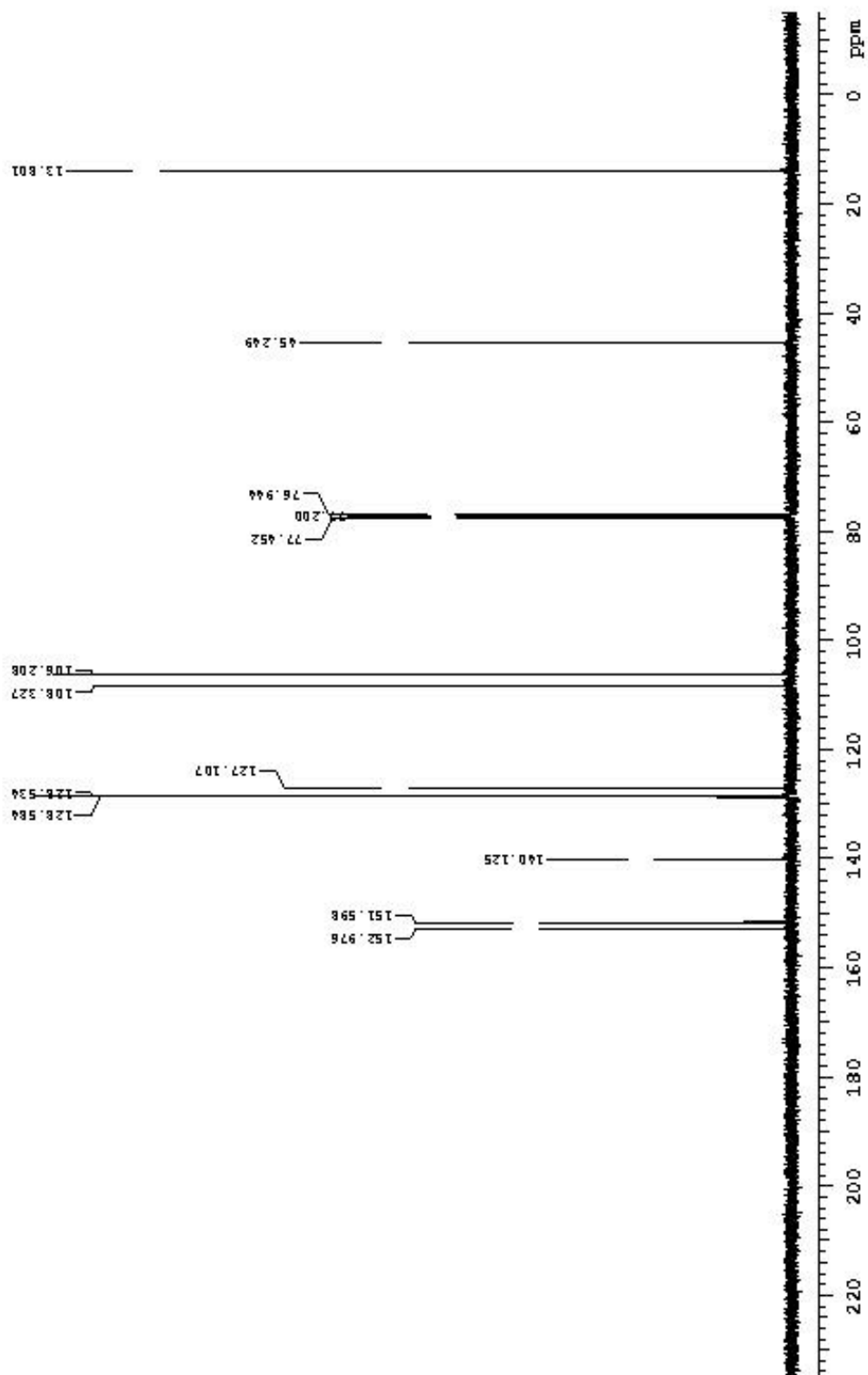












References

1. W.-H. Leung, T. S. M. Hun, H.-w. Hou, and K.-Y. Wu, *J. Chem. Soc., Dalton Trans.*, 1997, 237.