

## SUPPORTING INFORMATION

### Synthesis of Naphthalenes and 2-Naphthols by the Electrophilic Cyclization of Alkynes

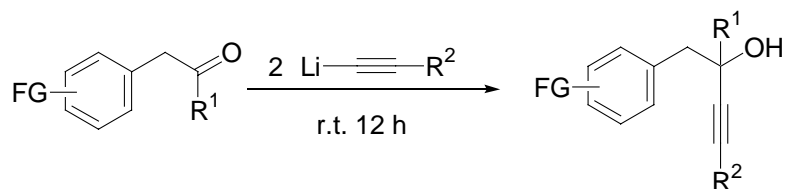
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**General.** The  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were recorded at 300 and 75.5 MHz or 400 and 100 MHz respectively. Thin layer chromatography was performed using commercially prepared 60-mesh silica gel plates, and visualization was effected with short wavelength UV light (254 nm). All melting points are uncorrected. High resolution mass spectra were recorded on a MS50TC double focusing magnetic sector mass spectrometer using EI at 70 eV. All reagents were used directly as obtained commercially unless otherwise noted.

**Synthesis of Starting Materials.** Compounds **1**, **13**, **15**, **17**, **19**, **22**, **24**, **26**, **28**, **30**, **34**, **36**, **38**, **41** and **47** were prepared by the condensation of an  $\alpha$ -arylacetaldehyde or an  $\alpha$ -arylaceton with the corresponding lithium acetylides. A typical reaction is



described below. To a solution of the acetylene in anhydrous THF was added 1 equiv of *n*-BuLi at 0 °C under an Ar atmosphere. The resulting solution was stirred at that temperature for 1 h. Then 0.5 equiv of the  $\alpha$ -arylacetaldehyde or  $\alpha$ -arylaceton in THF was added by syringe. The reaction mixture was kept under the inert atmosphere and stirred for 12 h while it warmed up to ambient temperature. The mixture was then quenched by adding satd aq  $\text{NH}_4\text{Cl}$  and extracted twice with diethyl ether. The combined ether fractions were dried over  $\text{MgSO}_4$  and concentrated under vacuum to yield the crude product, which was purified by flash chromatography on silica gel.

**1,4-Diphenylbut-3-yn-2-ol (1).** The indicated compound was prepared as a light yellow oil in a 75% yield from the coupling of phenylacetylene with phenylacetaldehyde.

The reaction mixture was chromatographed using 5:1 hexane/EtOAc. The spectral properties were identical with those previously reported.<sup>1</sup>

**4-(Cyclohex-1-enyl)-1-phenylbut-3-yn-2-ol (13).** The indicated compound was prepared as a light yellow oil in a 65% yield from the reaction of 1-ethynylcyclohexene and phenylacetaldehyde. The reaction mixture was chromatographed using 5:1 hexane/EtOAc. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz) δ 1.52-1.59 (m, 4H), 2.03-2.05 (m, 4H), 2.57-2.59 (m, 1H), 2.95-2.98 (d, *J* = 6.3 Hz, 2H), 4.61-4.63 (m, 1H), 6.04-6.05 (m, 1H), 7.20-7.27 (m, 5H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz) δ 21.8, 22.6, 25.9, 29.4, 44.6, 63.9, 87.3, 87.8, 120.4, 127.0, 128.5, 130.2, 135.4, 137.2; IR (CHCl<sub>3</sub>) 3420, 2941, 2372, 1653 cm<sup>-1</sup>; HRMS *m/z* 226.1360 (calcd for C<sub>16</sub>H<sub>18</sub>O, 226.1358).

**1-Phenyloct-3-yn-2-ol (15).** The indicated compound was prepared as a colorless oil in a 50% yield from the reaction of 1-hexyne and phenylacetaldehyde. The reaction mixture was chromatographed using 10:1 hexane/EtOAc. The spectral properties were identical with those previously reported.<sup>2</sup>

**4-Cyclohexyl-1-phenylbut-3-yn-2-ol (17).** The indicated compound was prepared as a colorless oil in a 52% yield from the reaction of cyclohexylacetylene and phenylacetaldehyde. The reaction mixture was chromatographed using 10:1 hexane/EtOAc. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz) δ 1.29-1.81 (m, 10H), 2.41 (m, 1H), 2.82 (m, 1H), 3.00-3.02 (d, *J* = 6.3 Hz, 2H), 4.56-4.61 (m, 1H), 7.26-7.38 (m, 5H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz) δ 25.1, 26.2, 29.3, 32.9, 44.8, 63.6, 81.0, 90.7, 126.9, 128.5, 130.2, 137.4; IR (CHCl<sub>3</sub>) 3395, 3031, 2927, 2857, 2237 cm<sup>-1</sup>; HRMS *m/z* 228.1516 (calcd for C<sub>16</sub>H<sub>20</sub>O, 228.1514).

**1-Phenyl-4-(trimethylsilyl)but-3-yn-2-ol (19).** The indicated compound was prepared as a light yellow oil in a 70% yield from the reaction of (trimethylsilyl)acetylene and phenylacetaldehyde. The reaction mixture was chromatographed using 10:1 hexane/EtOAc.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz)  $\delta$  0.20 (s, 9H), 2.40-2.60 (br s, 1H), 3.00-3.02 (d,  $J = 6.3$  Hz, 2H), 4.54-4.58 (t,  $J = 6.3$  Hz, 1H), 7.26-7.36 (m, 5H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz)  $\delta$  0, 44.2, 63.7, 90.6, 106.4, 127.0, 128.4, 130.1, 136.8; IR ( $\text{CHCl}_3$ ) 3430, 2966, 2367, 1948  $\text{cm}^{-1}$ ; HRMS  $m/z$  218.1131 (calcd for  $\text{C}_{13}\text{H}_{18}\text{OSi}$ , 218.1127).

**4-Ethoxy-1-phenylbut-3-yn-2-ol (22).** The indicated compound was prepared as an orange oil from the reaction of ethoxyethyne and phenylacetaldehyde in a 71% yield. The reaction mixture was chromatographed using 5:1 hexane/EtOAc.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz)  $\delta$  1.20-1.27 (t,  $J = 7.2$  Hz, 3H), 2.50 (br s, 1H), 2.89-2.91 (d,  $J = 7.8$  Hz, 2H), 3.93-4.00 (q,  $J = 7.8$  Hz, 2H), 4.52-4.53 (m, 1H), 7.18-7.28 (m, 5H), 7.34-7.37 (m, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz)  $\delta$  14.6, 39.5, 45.3, 63.4, 74.8, 94.8, 126.9, 128.5, 130.1, 137.7; IR ( $\text{CHCl}_3$ ) 3394, 3030, 2930, 2262, 1495  $\text{cm}^{-1}$ ; HRMS  $m/z$  190.0996 (calcd for  $\text{C}_{12}\text{H}_{14}\text{O}_2$ , 190.0994).

**2-Methyl-1-(4-methylphenyl)-4-phenylbut-3-yn-2-ol (26).** The indicated compound was prepared as a light yellow oil from the reaction of phenylacetylene and (4-methylphenyl)acetone in a 100% yield. The reaction mixture was chromatographed using 4:1 hexane/EtOAc.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz)  $\delta$  1.58 (s, 3H), 2.28 (s, 1H), 2.58 (s, 1H), 2.96-2.98 (d,  $J = 4.8$  Hz, 2H), 7.02-7.10 (d,  $J = 7.8$  Hz, 2H), 7.21-7.26 (m, 5H), 7.34-7.37 (m, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz)  $\delta$  21.5, 29.8, 49.8, 68.8, 84.8, 93.2, 123.2, 128.6, 128.6, 129.2, 131.1, 131.9, 133.7, 136.8; IR ( $\text{CHCl}_3$ ) 3553, 3401, 2927, 2867, 1517, 1443  $\text{cm}^{-1}$ ; HRMS  $m/z$  250.1363 (calcd for  $\text{C}_{18}\text{H}_{18}\text{O}$ , 250.1358).

**1,4-Diphenylpent-1-yn-3-ol (28).** The indicated compound was prepared as a light yellow oil from the reaction of phenylacetylene and 2-phenylpropanal in an 85% yield (diastereomeric mixture). The reaction mixture was chromatographed using 5:1 hexane/EtOAc.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz)  $\delta$  1.42-1.46 (m, 3H), 2.29-2.32 (m, 1H), 2.09-3.13 (m, 1H), 4.62-4.64 (m, 1H), 7.20-7.41 (m, 10H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz)  $\delta$  16.7, 17.1, 46.3, 46.8, 68.0, 68.3, 86.7, 89.1, 89.1, 123.0, 127.3, 127.3, 128.5, 128.5, 128.6, 128.6, 128.6, 128.7, 128.7, 128.8, 128.9, 131.9, 132.0, 132.0, 142.1, 142.4; IR ( $\text{CHCl}_3$ ) 3546, 3364, 3064, 3030, 2975, 2912, 1495, 1454  $\text{cm}^{-1}$ ; HRMS  $m/z$  236.1205 (calcd for  $\text{C}_{17}\text{H}_{16}\text{O}$ , 236.1201).

**1-(4-Methoxyphenyl)-4-phenylbut-3-yn-2-ol (30).** To a cooled suspension (ice bath) of PCC (4.31 g, 20 mmol) in  $\text{CH}_2\text{Cl}_2$  (200 mL) was slowly added a solution of 2-(4-methoxyphenyl)ethanol (15 mmol) in  $\text{CH}_2\text{Cl}_2$  (10 mL). The mixture was stirred at that temperature for 2 h, then warmed up to rt for another 2 h. The mixture was filtered through celite and the solvents were removed under vacuum. The reaction mixture was chromatographed using 5:1 hexane/EtOAc to give the (4-methoxyphenyl)acetaldehyde as a colorless oil in a 75% yield. To a solution of phenylacetylene (10 mmol) in 30 mL of anhydrous THF at 0 °C was added *n*-BuLi (10 mmol, 2.5M in hexane) under  $\text{N}_2$ , and the resulting solution was stirred at that temperature for 1 h. Then (4-methoxyphenyl)acetaldehyde (5 mmol) in 10 mL of THF was added to the solution under an  $\text{N}_2$  atmosphere. The resulting solution was stirred at rt for 2 h. Brine (30 mL) was then added to quench the reaction and the solution was extracted twice using diethyl ether (2 x 30 mL). The solvents were removed under vacuum, and the reaction mixture was chromatographed using 5:1 hexane/EtOAc to obtain alkynol **30** as a light orange oil in an

80% yield.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz)  $\delta$  2.91 (s, 1H), 2.99-3.01 (d,  $J = 6.3$  Hz, 2H), 3.71 (s, 3H), 4.71 (m, 1H), 6.80-6.83 (d,  $J = 8.7$  Hz, 2H), 7.16-7.26 (m, 5H), 7.35-7.38 (m, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz)  $\delta$  43.8, 55.5, 64.1, 85.9, 90.2, 114.1, 122.9, 128.6, 128.7, 129.1, 131.2, 131.9, 158.8; IR ( $\text{CH}_2\text{Cl}_2$ ) 3310, 1609  $\text{cm}^{-1}$ ; HRMS  $m/z$  252.1154 (calcd for  $\text{C}_{17}\text{H}_{16}\text{O}_2$ , 252.1150).

**1-(4-Fluorophenyl)-2-methyl-4-phenylbut-3-yn-2-ol (34).** The indicated compound was prepared as a light yellow oil in a 92% yield from the reaction of phenylacetylene and (4-fluorophenyl)acetone. The reaction mixture was chromatographed using 5:1 hexane/EtOAc.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  1.62 (s, 3H), 2.04 (br s, 1H), 2.97-3.06 (m, 2H), 7.00-7.04 (m, 2H), 7.28-7.37 (m, 7H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  29.6, 48.9, 68.5, 84.9, 92.2, 114.9, 115.1, 122.6, 128.4, 128.5, 131.0, 131.6, 132.2, 132.2, 132.3, 161.0, 163.4 (the extra peaks are due to F splitting); IR ( $\text{CHCl}_3$ ) 3416, 3056, 2991, 2937, 1713, 1608, 1508  $\text{cm}^{-1}$ ; HRMS  $m/z$  254.1111 (calcd for  $\text{C}_{17}\text{H}_{15}\text{FO}$ , 254.1107).

**1-(1-Naphthyl)-4-phenylbut-3-yn-2-ol (36).** The indicated compound was prepared as a light yellow oil in a 90% yield from the reaction of phenylacetylene and 1-naphthylacetaldehyde, which was prepared by the Swern oxidation of 2-(1-naphthyl)ethanol.<sup>3</sup> The reaction mixture was chromatographed using 5:1 hexane/EtOAc.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz)  $\delta$  2.06-2.08 (m, 2H), 3.58-3.62 (m, 2H), 4.96-5.02 (m, 1H), 7.25-7.56 (m, 9H), 7.78-7.89 (m, 2H), 8.14-8.17 (d,  $J = 8.04$ , 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz)  $\delta$  41.4, 63.4, 86.0, 89.8, 122.6, 124.0, 125.6, 125.8, 126.3, 128.0, 128.4, 128.5, 128.6, 129.0, 131.8, 132.4, 132.9, 134.1; IR ( $\text{CHCl}_3$ ) 3361, 3054, 2933, 1489  $\text{cm}^{-1}$ ; HRMS  $m/z$  272.1204 (calcd for  $\text{C}_{20}\text{H}_{16}\text{O}$ , 272.1201).

**1-(3-Methoxyphenyl)-2-methyl-4-phenylbut-3-yn-2-ol (38).** The indicated compound was prepared as a light yellow oil in a 95% yield from the reaction of phenylacetylene and (3-methoxyphenyl)acetone. The reaction mixture was chromatographed using 5:1 hexane/EtOAc. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz) δ 1.64 (s, 3H), 2.00-2.10 (br s, 1H), 2.96-3.09 (q, *J* = 12.9 Hz, 2H), 3.77 (s, 3H), 6.82-6.86 (m, 1H), 6.95-6.98 (m, 2H), 7.23-7.32 (m, 4H), 7.36-7.40 (m, 2H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz) δ 29.6, 49.8, 55.2, 68.4, 84.6, 92.5, 112.7, 116.3, 122.7, 123.1, 128.3, 128.3, 129.1, 131.6, 137.8, 159.4; IR (CHCl<sub>3</sub>) 3476, 3061, 2986, 2942, 2842, 1608 cm<sup>-1</sup>; HRMS *m/z* 266.1310 (calcd for C<sub>18</sub>H<sub>18</sub>O<sub>2</sub>, 266.1307).

**1-(2-Naphthyl)-4-phenylbut-3-yn-2-ol (41).** To the solution of 2-(2-naphthyl)ethanol (2 mmol) in 10 mL of CH<sub>2</sub>Cl<sub>2</sub> was added Dess-Martin periodinane (2 mmol). The mixture was stirred for 2 h. The reaction was diluted with another 20 mL of CH<sub>2</sub>Cl<sub>2</sub>. The organic layer was washed with 20 mL of aq satd Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>, 20 mL of aq satd NaHCO<sub>3</sub> and brine, and then dried over MgSO<sub>4</sub>, and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel using 10:1 hexane/EtOAc to afford 2-naphthylacetaldehyde in a 98% yield. Compound **41** was prepared by the reaction of phenylacetylene and 1-naphthylacetaldehyde as a light yellow oil. The reaction mixture was chromatographed using 5:1 hexane/EtOAc. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz) δ 2.11-2.13 (d, *J* = 5.9 Hz, 1H), 3.28-3.30 (m, 2H), 4.89-4.95 (m, 1H), 7.27-7.52 (m, 8H), 7.81-7.86 (m, 4H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz) δ 44.5, 63.9, 86.2, 89.6, 122.7, 125.9, 126.3, 127.9, 127.9, 128.2, 128.4, 128.5, 128.7, 128.8, 131.9, 132.7, 133.7, 134.3; IR (CHCl<sub>3</sub>) 3349, 3056, 2927, 2867, 1602, 1491 cm<sup>-1</sup>; HRMS *m/z* 272.1204 (calcd for C<sub>20</sub>H<sub>16</sub>O, 272.1201).

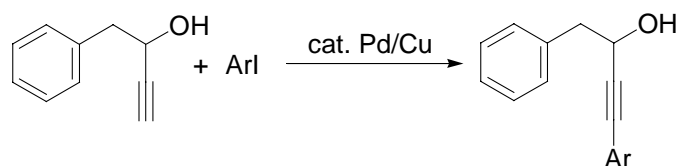
**1-(Benzothien-3-yl)-4-phenylbut-3-yn-2-ol (43).** A solution of 3-benzothienylacetic acid (6.6 mmol) in 10 mL of anhydrous diethyl ether was slowly added to a stirred solution of LAH (11.2 mmol) in 50 mL of diethyl ether at 0 °C. The ice bath was removed and the solution was stirred for 4 h, while it was allowed to warm to room temperature. Water was slowly added to decompose the excess LAH (caution - gas evolution and an exotherm). The organic layer was subsequently washed with 10% H<sub>2</sub>SO<sub>4</sub>, aq satd NaHCO<sub>3</sub> and brine. The organic layer was dried over Na<sub>2</sub>SO<sub>4</sub> and the solvent was removed under vacuum to give 3-(2-hydroxyethyl)benzothiophene, which was used directly in the next step. To the solution of 3-(2-hydroxyethyl)benzothiophene in 30 mL of CH<sub>2</sub>Cl<sub>2</sub> was added PCC (8.6 mmol) at 0 °C. The mixture was stirred for 4 h at 0 °C until all of the alcohol was consumed. The light brown solution was filtered and the filtrate was collected. The solvent was removed and the residue was purified by chromatography on silica gel using 10:1 hexane/EtOAc to give (3-benzothienyl)acetaldehyde in a 35% overall yield. To a solution of phenylacetylene (2.0 mmol) in 20 mL of anhydrous THF was added *n*-BuLi (2.0 mmol, 2.5M in hexane) at 0 °C under an Ar atmosphere. The resulting solution was stirred at that temperature for 1 h. Then (3-benzothienyl)acetaldehyde (1.0 mmol) in 5 mL of anhydrous THF was added by syringe. The reaction mixture was kept under an inert atmosphere and stirred for 2 h. The mixture was then quenched by adding satd aq NH<sub>4</sub>Cl and extracted twice with diethyl ether. The combined ether fractions were dried over MgSO<sub>4</sub> and concentrated under vacuum to yield the crude product, which was purified by flash chromatography on silica gel using 10:1 hexane/EtOAc to give 1-(benzothien-3-yl)-4-phenylbut-3-yn-2-ol (139 mg, 50%) as an orange oil. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz) δ 2.24 (br s, 1H), 3.34-3.36

(d,  $J = 6.3$  Hz, 2H), 4.90-4.94 (t,  $J = 6.3$  Hz, 1H), 7.22-7.40 (m, 8H), 7.84-7.87 (m, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz)  $\delta$  37.1, 62.7, 86.0, 89.7, 122.2, 122.6, 123.1, 124.3, 124.6, 124.7, 128.5, 128.8, 131.5, 131.9, 139.4, 140.6; IR ( $\text{CHCl}_3$ ) 3430, 3060, 1615  $\text{cm}^{-1}$ ; HRMS  $m/z$  278.0772 (calcd for  $\text{C}_{18}\text{H}_{14}\text{OS}$ , 278.0765).

**1-(Benzothien-3-yl)-4-(cyclohexen-1-yl)but-3-yn-2-ol (45).** This compound was prepared using the same method as compound **43**, but coupling (3-benzothienyl)acetaldehyde and 1-ethynylcyclohexene. The crude product was purified by flash chromatography on silica gel using 5:1 hexane/EtOAc to give compound **45** (142 mg, 51%) as an orange oil.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz)  $\delta$  1.51-1.60 (m, 4H), 1.99-2.13 (m, 5H), 3.25-3.27 (d,  $J = 6.3$  Hz, 2H), 4.78-7.82 (t,  $J = 6.3$  Hz, 1H), 6.01-6.03 (m, 1H), 7.22-7.39 (m, 3H), 7.79-7.85 (m, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz)  $\delta$  21.7, 22.6, 25.9, 29.2, 37.3, 62.6, 87.1, 87.8, 120.2, 122.2, 123.1, 124.2, 124.5, 124.5, 131.7, 135.8, 139.4, 140.5; IR ( $\text{CHCl}_3$ ) 3386, 3064, 2930, 2860, 2185, 1669, 1435  $\text{cm}^{-1}$ ; HRMS  $m/z$  282.1085 (calcd for  $\text{C}_{18}\text{H}_{18}\text{OS}$ , 282.1078).

**1-(1H-Indol-3-yl)-2-methyl-4-phenylbut-3-yn-2-ol (47).** The indicated compound was prepared as a sticky light red oil from the reaction of phenylacetylene and 3-(1H-indolyl)acetone in a 55% yield. The reaction mixture was chromatographed using 3:1 hexane/EtOAc.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz)  $\delta$  1.68 (s, 3H), 2.45 (br s, 1H), 3.14-3.19 (d,  $J = 14.4$  Hz, 1H), 3.28-3.33 (d,  $J = 14.4$  Hz, 1H), 7.11-7.35 (m, 9H), 7.75-7.78 (d,  $J = 7.8$  Hz, 1H), 8.15 (s, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz)  $\delta$  29.7, 39.9, 69.0, 83.7, 93.7, 110.8, 111.4, 119.9, 120.0, 122.3, 123.1, 124.3, 128.5, 128.6, 131.9, 136.3; IR ( $\text{CHCl}_3$ ) 3412, 3060, 2986, 2927, 1458  $\text{cm}^{-1}$ ; HRMS  $m/z$  275.1314 (calcd for  $\text{C}_{19}\text{H}_{17}\text{NO}$ , 275.1310).

Compounds **5**, **7**, and **9** were prepared by the Sonogashira reaction of 1-phenylbut-3-yn-2-ol with the corresponding aryl halide.



**1-Phenylbut-3-yn-2-ol.** To a solution of 1-phenyl-4-(trimethylsilyl)but-3-yn-2-ol (**19**, 1.20 g, 5.5 mmol) in 20 mL of MeOH was added KF (0.41 g, 7.0 mmol). After being stirred for 12 h at room temperature, the reaction was quenched by adding brine. The reaction mixture was extracted with Et<sub>2</sub>O (2 x 30 mL). The extracts were dried over MgSO<sub>4</sub> and the solvent was removed under reduced pressure. The residue was purified by flash chromatography (10:1 hexane/EtOAc) on silica gel to afford 642 mg of the product (80% yield) as a light yellow oil. The spectra properties were identical with those previously reported.<sup>26</sup>

**General Procedure for the Coupling Reaction of 1-Phenylbut-3-yn-2-ol with Aryl Halides.** To a solution of Et<sub>3</sub>N (30 mL), PdCl<sub>2</sub>(PPh<sub>3</sub>)<sub>2</sub> (2 mol %), 3.0 mmol of 1-phenylbut-3-yn-2-ol, and 1.2 equiv of aryl iodide (stirring for 3 min beforehand) was added CuI (1 mol %). The reaction mixture was flushed with Ar and the flask was then sealed. The mixture was stirred at room temperature and monitored by TLC for completion (2-12 h). The resulting solution was filtered, washed with a satd aq NaCl solution, and extracted with diethyl ether. The combined ether fractions were dried over MgSO<sub>4</sub> and concentrated under vacuum to yield the crude product, which was purified by flash chromatography on silica gel.

**4-(4-Methoxyphenyl)-1-phenylbut-3-yn-2-ol (5).** The indicated compound was prepared as a yellow oil from *p*-iodoanisole in a 75% yield. The reaction mixture was

chromatographed using 3:1 hexane/EtOAc.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  2.21 (s, 1H), 3.06-3.08 (m, 2H), 3.77 (s, 3H), 4.77 (m, 1H), 6.79-6.81 (d,  $J = 8.8$  Hz, 3H), 7.24-7.33 (m, 7H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  44.3, 55.3, 63.8, 85.8, 88.2, 114.0, 114.7, 127.0, 128.5, 130.0, 133.2, 136.8, 159.8; IR ( $\text{CHCl}_3$ ) 3384, 3060, 2929, 2837, 1606  $\text{cm}^{-1}$ ; HRMS  $m/z$  252.1154 (calcd for  $\text{C}_{17}\text{H}_{16}\text{O}_2$ , 252.1150).

**1-[4-(3-Hydroxy-4-phenylbut-1-ynyl)phenyl]ethanone (7).** The indicated compound was prepared as a light orange oil from 4-iodoacetophenone in a 72% yield. The reaction mixture was chromatographed using 3:1 hexane/EtOAc.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  2.56 (s, 3H), 2.66 (s, 1H), 3.10-3.12 (d,  $J = 6.4$  Hz, 2H), 4.80-4.83 (t,  $J = 6.4$  Hz, 1H), 7.25-7.33 (m, 5H), 7.41-7.43 (d,  $J = 8.0$  Hz, 2H), 7.84-7.86 (d,  $J = 8.0$  Hz, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  26.7, 44.1, 63.7, 85.0, 93.1, 127.1, 127.6, 128.3, 128.5, 129.9, 131.8, 136.4, 136.5, 197.7; IR ( $\text{CHCl}_3$ ) 3411, 3022, 2930, 2868, 1670, 1593  $\text{cm}^{-1}$ ; HRMS 264.1156  $m/z$  (calcd for  $\text{C}_{18}\text{H}_{16}\text{O}_2$ , 264.1150).

**1-Phenyl-4-(thien-2-yl)but-3-yn-2-ol (9).** The indicated compound was prepared as a light brown oil from 2-iodothiophene in a 78% yield. The reaction mixture was chromatographed using 5:1 hexane/EtOAc.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  2.67 (s, 1H), 3.02-3.04 (d,  $J = 6.4$  Hz, 2H), 4.72-4.75 (t,  $J = 6.4$  Hz, 2H), 6.87-6.90 (m, 1H), 7.12-7.16 (m, 2H), 7.21-7.28 (m, 5H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  44.1, 63.9, 79.3, 93.7, 122.6, 127.1, 127.1, 127.5, 128.6, 130.0, 132.4, 136.7; IR ( $\text{CHCl}_3$ ) 3361, 3106, 3026, 2927, 2223, 1613  $\text{cm}^{-1}$ ; HRMS  $m/z$  228.0613 (calcd for  $\text{C}_{14}\text{H}_{12}\text{OS}$ , 228.0609).

**1,4-Diphenyl-1-butyne (49).** To a solution of  $\text{Et}_3\text{N}$  (30 mL),  $\text{PdCl}_2(\text{PPh}_3)_2$  (2 mol %), 4-phenyl-1-butyne (3.0 mmol), and 1.2 equiv of phenyl iodide (stirring for 3 min beforehand) was added  $\text{CuI}$  (1 mol %). The reaction mixture was flushed with Ar and the

flask was then sealed. The mixture was stirred at room temperature for 12 h. The resulting solution was filtered, washed with a satd aq NaCl solution, and extracted with diethyl ether. The combined ether fractions were dried over MgSO<sub>4</sub> and concentrated under vacuum to yield the crude product, which was purified by flash chromatography on silica gel using hexane to give 1,4-diphenyl-1-butyne (84%) as a light yellow oil. The spectral properties were identical with those previously reported.<sup>4</sup>

**1,3-Diphenyl-1-propyne (51).** To a solution of Et<sub>3</sub>N (20 mL), PdCl<sub>2</sub>(PPh<sub>3</sub>)<sub>2</sub> (2 mol %), 5.0 mmol of 3-phenyl-1-propyne, and 7.5 mmol of PhI (stirring for 3 min beforehand) was added CuI (1 mol %). The reaction mixture was flushed with Ar and the flask was then sealed. The mixture was stirred at room temperature for 2 h. The resulting solution was filtered, washed with a satd aq NaCl solution, and extracted with diethyl ether. The combined ether fractions were dried over MgSO<sub>4</sub> and concentrated under vacuum to yield the crude product, which was purified by flash chromatography using hexane on silica gel. Compound **51** was obtained as a colorless oil in an 85% yield. The spectral properties were identical with those previously reported.<sup>5</sup>

Compound **53** was prepared according to the literature procedure.<sup>6</sup>

**1-Phenyl-4-[4-(2-hydroxy-4-phenyl-3-butynyl)phenyl]but-3-yn-2-ol (55).** 1,4-Benzenediacetic acid (6.5 mmol) in 50 mL of ethanol was mixed with 5 mL of 36N concentrated sulfuric acid. The reaction mixture was refluxed for 18 h. The ethanol was removed by vacuum and the residue was worked-up using ether and brine. The organic layer was separated, washed using a satd aq NaHCO<sub>3</sub> solution and brine. The organic layer was dried over Na<sub>2</sub>SO<sub>4</sub> and the solvent was removed under vacuum to give the crude product, which was purified by flash chromatography using 3:1 hexane/EtOAc to

give 1,4-benzenediacetic acid diethyl ester in a 77% yield. A solution of 1,4-benzenediacetic acid diethyl ester (5.0 mmol) in 10 mL of anhydrous diethyl ether was slowly added to a stirred solution of LAH (10.0 mmol) in 30 mL of diethyl ether. The resulting mixture was refluxed for 3 h. Water was added to decompose the excess LAH. Concentrated HCl was added to dissolve the precipitate. The organic layer was extracted with ether and washed with aq satd NaHCO<sub>3</sub> and brine. The organic layer was dried over Na<sub>2</sub>SO<sub>4</sub> and the solvent was removed under vacuum to give the crude product, which was purified by flash chromatography using EtOAc to give 1,4-benzenediethanol in an 84% yield. To a solution of 1,4-benzenediethanol (1 mmol) in 10 mL of CH<sub>2</sub>Cl<sub>2</sub> was added Dess-Martin periodinane (3 mmol) at r.t. The mixture was stirred 4 h at room temperature until all of the alcohol was consumed. The solution was filtered and the filtrate was collected. The solvent was removed and the residue was purified by chromatography on silica gel using 3:1 hexane/EtOAc to give 1,4-benzenediacetaldehyde in a 62% overall yield. To a solution of phenylacetylene (10.8 mmol) in 20 mL of anhydrous THF was added *n*-BuLi (10.8 mmol) at 0 °C under an Ar atmosphere. The resulting solution was stirred at that temperature for 1 h. Then 1,4-benzenediacetaldehyde (2.7 mmol) in 5 mL of anhydrous THF was added by syringe. The reaction mixture was kept under an inert atmosphere and stirred for 12 h. The mixture was then quenched by the addition of satd aq NH<sub>4</sub>Cl and extracted twice with diethyl ether. The combined ether fractions were dried over MgSO<sub>4</sub> and concentrated under vacuum to yield the crude product, which was purified by flash chromatography on silica gel using 3:1 hexane/EtOAc to give compound **55** (21%) as light yellow solid: mp 144-145 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 2.15 (s, 2H), 3.07-3.12 (m, 4H), 4.77-4.80

(m, 2H), 7.25-7.32 (m, 9H), 7.38-7.40 (m, 4H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  43.9, 63.7, 85.8, 89.5, 122.6, 128.4, 128.5, 130.0, 131.7, 135.3; IR ( $\text{CHCl}_3$ ) 3368, 3056, 2927, 2863, 2199, 1606, 1487  $\text{cm}^{-1}$ ; HRMS  $m/z$  366.1616 (calcd for  $\text{C}_{26}\text{H}_{22}\text{O}_2$ , 366.1620).

**1-Benzyl-3-phenylprop-2-ynyl acetate (58).** To a solution of 1,4-diphenylbut-3-yn-2-ol (2 mmol) in 10 mL of  $\text{CH}_3\text{CN}$  was added acetic anhydride (2.2 mmol) and 4-(dimethylamino)pyridine (2.2 mmol) at room temperature. The mixture was stirred at room temperature for 12 h. The mixture was quenched by the addition of satd aq  $\text{NH}_4\text{Cl}$  and extracted twice with diethyl ether. The combined ether fractions were dried over  $\text{MgSO}_4$  and concentrated under vacuum to yield the crude product, which was purified by flash chromatography on silica gel using 20:1 hexane/EtOAc to give compound **58** (97%) as light yellow oil:  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  1.98 (s, 3H), 3.11-3.13 (d,  $J = 6.8$  Hz, 2H), 7.20-7.37 (m, 10H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  21.1, 41.4, 65.2, 86.4, 122.3, 127.2, 128.5, 128.5, 128.8, 129.9, 132.0, 136.1, 169.9; IR ( $\text{CHCl}_3$ ) 2930, 1721  $\text{cm}^{-1}$ ; HRMS  $m/z$  294.1145 (calcd for  $\text{C}_{18}\text{H}_{16}\text{O}_2$ , 294.1150).

**[(1*R*, 2*R*) and (1*S*, 2*R*)]-2-Phenyl-1-(phenylethynyl)cyclohexanol, (59) and (61).** To a stirred solution of phenylacetylene (1.94 g, 19 mmol) and TMEDA (2.87 mL, 19 mmol) in THF was added dropwise a 2.5 M solution of *n*-BuLi (7.6 mL, 19 mmol) in hexane at  $-78$   $^\circ\text{C}$ . After stirring was continued for 1 h at  $-78$   $^\circ\text{C}$ , a solution of 2-phenylcyclohexanone (1.74 g, 10 mmol) in 10 mL of THF was added dropwise to this reaction mixture, and stirring was continued for an additional hour at the same temperature. The reaction mixture was diluted with water and extracted with  $\text{Et}_2\text{O}$ . The residue upon work-up was chromatographed on silica gel using 20:1 hexane/EtOAc to afford compound **59** in a 38% yield as a sticky light yellow oil and compound **61** in a

32% yield as a yellow oil. Their structures were determined by NOESY NMR spectroscopy (see the spectra that follow) of the corresponding methyl ethers. **59**:  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz)  $\delta$  1.35-1.45 (m, 1H), 1.58-1.96 (m, 6H), 2.05-2.20 (q,  $J = 13.0$ , 3.3 Hz, 1H), 2.25-2.30 (d,  $J = 13.0$  Hz, 1H), 2.82-2.88 (dd,  $J = 13.0$ , 3.6 Hz, 1H), 7.17-7.32 (m, 8H), 7.37-7.39 (m, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz)  $\delta$  21.2, 26.4, 27.0, 40.1, 53.1, 69.6, 85.0, 93.8, 123.2, 127.3, 128.2, 128.5, 128.5, 129.8, 131.9, 142.1; IR ( $\text{CHCl}_3$ ) 3560, 3449, 3030, 2934  $\text{cm}^{-1}$ ; HRMS  $m/z$  276.1519 (calcd for  $\text{C}_{20}\text{H}_{20}\text{O}$ , 276.1514). **61**:  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz)  $\delta$  1.26-1.42 (m, 1H), 1.66-1.83 (m, 6H), 2.00-2.09 (m, 1H), 2.23-2.31 (m, 2H), 2.68-2.73 (dd,  $J = 13.0$ , 2.4 Hz, 1H), 7.21-7.33 (m, 7H), 7.37-7.43 (m, 4H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz)  $\delta$  24.6, 26.4, 30.4, 40.7, 55.5, 73.1, 87.7, 90.7, 123.3, 127.3, 128.3, 128.6, 128.6, 129.9, 131.9, 141.0; IR ( $\text{CHCl}_3$ ) 3542, 2431, 3030, 2934, 2860  $\text{cm}^{-1}$ ; HRMS  $m/z$  276.1519 (calcd for  $\text{C}_{20}\text{H}_{20}\text{O}$ , 276.1514).

**[(1*R*, 2*R*) and (1*S*, 2*R*)]-2-Phenyl-1-(phenylethynyl)cyclohexyl methyl ether, (62) and (63).** To a stirred solution of [(1*R*, 2*R*) or (1*S*, 2*R*)]-2-phenyl-1-(phenylethynyl)cyclohexanol (2 mmol) in THF was added NaH (60 % in mineral oil) (10 mmol). After stirring was continued for 10 min at room temperature, MeI (20 mmol) was added dropwise to this reaction mixture, and stirring was continued for an additional hour. The reaction mixture was diluted with water and extracted with  $\text{Et}_2\text{O}$ . The residue upon work-up was chromatographed on silica gel using 20:1 hexane/EtOAc to afford compound **62** in a 100% yield as a light yellow solid and compound **63** in an 80% yield as a colorless oil. **62**: mp 82-83  $^\circ\text{C}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  1.35-1.45 (m, 1H), 1.50-1.68 (m, 4H), 1.82-1.85 (m, 1H), 2.13-2.20 (m, 1H), 2.41-2.44 (d,  $J = 12.4$  Hz, 1H), 2.81-2.85 (m, 1H), 3.30 (s, 3H), 7.19-7.29 (m, 8H), 7.40-7.42 (m, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ ,

100 MHz)  $\delta$  20.7, 26.3, 27.8, 34.8, 51.1, 53.8, 75.1, 86.5, 90.8, 123.1, 126.4, 127.4, 128.1, 128.2, 129.9, 131.8, 142.9; IR (CHCl<sub>3</sub>) 3034, 2938, 2856, 1487 cm<sup>-1</sup>; HRMS  $m/z$  290.1674 (calcd for C<sub>22</sub>H<sub>22</sub>O, 290.1671). **63**: 1.30-1.60 (m, 2H), 1.75-1.84 (m, 4H), 2.05-2.14 (m, 1H), 2.38-2.41 (d,  $J$  = 12.4 Hz, 1H), 2.76-2.80 (m, 1H), 3.23 (s, 3H), 7.17-7.30 (m, 6H), 7.42-7.45 (m, 4H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$  24.1, 26.3, 31.0, 37.8, 51.7, 54.0, 78.7, 87.9, 89.7, 123.1, 126.5, 127.5, 128.3, 128.4, 129.5, 131.7, 142.4; IR (CHCl<sub>3</sub>) 3033, 2935, 2855 cm<sup>-1</sup>; HRMS  $m/z$  290.1674 (calcd for C<sub>22</sub>H<sub>22</sub>O, 290.1671).

Compound **61** was prepared according to the literature procedure.<sup>7</sup> The spectral properties were identical with those previously reported.<sup>8</sup>

**1,4-Diphenyl-3-butyn-2-one (65)**. To the solution of 1,4-diphenylbut-3-yn-2-ol (2.0 mmol) in 10 mL of CH<sub>2</sub>Cl<sub>2</sub> was added Dess-Martin periodinane (2.4 mmol). The mixture was stirred for 2 h. The reaction was diluted with another 20 mL of CH<sub>2</sub>Cl<sub>2</sub>. The organic layer was washed with 20 mL of satd aq Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>, 20 mL of satd aq NaHCO<sub>3</sub> and brine. The organic layer was dried over MgSO<sub>4</sub> and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel using 10:1 hexane/EtOAc to afford compound **65** in an 80% yield as a light yellow oil. The spectral properties were identical with those previously reported.<sup>9</sup>

**4-(Cyclohexen-1-yl)-1-phenylbut-3-yn-2-one (68)**. Compound **68** was prepared by the method used to prepare **65**, except that 4-(cyclohexen-1-yl)-1-phenylbut-3-yn-2-ol was used as the starting material. The residue was purified by column chromatography on silica gel using 10:1 hexane/EtOAc to afford compound **68** in a 58% yield as a light yellow oil. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) 1.55-1.63 (m, 4H), 2.05-2.13 (m, 4H), 3.82 (s,

2H), 6.35-6.36 (m, 1H), 7.25-7.35 (m, 5H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  21.1, 21.9, 26.2, 28.2, 52.2, 86.2, 95.6, 119.0, 127.3, 128.7, 129.9, 133.6, 143.1, 185.4; IR ( $\text{CHCl}_3$ ) 3031, 2937, 2865, 2186, 1712, 1671  $\text{cm}^{-1}$ ; HRMS  $m/z$  224.1204 (calcd for  $\text{C}_{16}\text{H}_{16}\text{O}$ , 224.1201).

**4-Cyclohexyl-1-phenylbut-3-yn-2-one (70).** Compound **70** was prepared by the method used to prepare **65**, except that 4-cyclohexyl-1-phenylbut-3-yn-2-ol was used as the starting material. The reaction mixture was purified by column chromatography on silica gel using 10:1 hexane/EtOAc to afford compound **70** in an 83% yield as a light yellow oil.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz) 1.37-1.46 (m, 6H), 1.57-1.74 (m, 4H), 2.46-2.50 (m, 1H), 3.78 (s, 2H), 7.22-7.33 (m, 5H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  24.5, 25.6, 29.1, 31.4, 52.4, 80.7, 100.2, 127.3, 128.6, 129.9, 133.5, 185.6; IR ( $\text{CHCl}_3$ ) 2938, 2860, 2233, 1695  $\text{cm}^{-1}$ ; HRMS  $m/z$  226.1360 (calcd for  $\text{C}_{16}\text{H}_{18}\text{O}$ , 226.1358).

**General Procedure for the Electrophilic Cyclization of Alkynols by  $\text{I}_2$ .** 0.3 Mmol of the alkynol, 3 equiv of  $\text{I}_2$ , 2 equiv of  $\text{NaHCO}_3$ , and 3 mL of  $\text{CH}_3\text{CN}$  were placed in a vial. The reaction mixture was stirred at room temperature, and the reaction was monitored by TLC to establish completion. The reaction mixture was diluted with 25 mL of ether, and washed with 20 mL of satd aq  $\text{Na}_2\text{S}_2\text{O}_3$ . The organic layer was separated and the aqueous layer was extracted with another 25 mL of ether. The combined organic layers were dried over  $\text{MgSO}_4$  and filtered. The solvent was evaporated under reduced pressure and the product was isolated by chromatography on a silica gel column.

**General Procedure for the Electrophilic Cyclization of Alkynols by  $\text{ICl}$ ,  $\text{Br}_2$ , and  $\text{PhSeBr}$ .** 0.30 Mmol of the alkynol, 2 equiv of  $\text{NaHCO}_3$  and 2 mL of  $\text{CH}_3\text{CN}$  were placed in a vial. 2 Equiv of  $\text{ICl}$ ,  $\text{Br}_2$  or  $\text{PhSeBr}$  in 1 mL of  $\text{CH}_3\text{CN}$  were added dropwise

to the vial. The reaction mixture was stirred at room temperature for 5 min. The reaction mixture was then diluted with 25 mL of ether, and washed with 20 mL of satd aq  $\text{Na}_2\text{S}_2\text{O}_3$ . The organic layer was separated and the aqueous layer was extracted with another 25 mL of ether. The combined organic layers were dried over  $\text{MgSO}_4$  and filtered. The solvent was evaporated under reduced pressure and the product was isolated by chromatography on a silica gel column.

**General Procedure for the Electrophilic Cyclization of Alkynols by NBS.**

0.30 Mmol of the alkynol, 1.5 equiv of NBS and 6 mL of  $\text{CH}_3\text{CN}$  were placed in a vial. The reaction mixture was refluxed for 20 min under Ar, diluted with 25 mL of ether, and washed with 20 mL of satd aq  $\text{Na}_2\text{S}_2\text{O}_3$ . The organic layer was separated and the aqueous layer was extracted with another 25 mL of ether. The combined organic layers were dried over  $\text{MgSO}_4$  and filtered. The solvent was evaporated under reduced pressure and the product was isolated by chromatography on a silica gel column.

**2-Iodo-1-phenylnaphthalene (2).** The reaction mixture was chromatographed using hexane to afford 74 mg (75%) or 92 mg (93%) of the product as a colorless oil from  $\text{I}_2$  or  $\text{ICl}$ , respectively. The spectral properties were identical with those previously reported.<sup>10</sup>

**2-Bromo-1-phenylnaphthalene (3).** The reaction mixture was chromatographed using hexane to afford 76 mg (89%) of the product as a colorless oil. The spectral properties were identical with those previously reported.<sup>10</sup>

**1-Phenyl-2-(phenylseleno)naphthalene (4).** The reaction mixture was chromatographed using 5:1 hexane/EtOAc to afford 39 mg (36%) of the product as a white solid: mp 93-94 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz)  $\delta$  7.26-7.54 (m, 14H), 7.66-7.68

(d,  $J = 8.7$  Hz, 1H), 7.80-7.83 (m, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz)  $\delta$  125.8, 126.4, 126.7, 128.1, 128.1, 128.2, 128.4, 128.4, 128.8, 129.7, 130.4, 130.7, 131.3, 132.4, 133.3, 135.3, 139.9, 139.9; IR ( $\text{CHCl}_3$ ) 3054, 1648, 1572  $\text{cm}^{-1}$ ; HRMS  $m/z$  360.0422 (calcd for  $\text{C}_{22}\text{H}_{16}\text{Se}$ , 360.0417).

**2-Iodo-1-(4-methoxyphenyl)naphthalene (6).** The reaction mixture was chromatographed using 40:1 hexane/EtOAc to afford 81 mg (75%) of the product as a white solid: mp 110-111  $^\circ\text{C}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz)  $\delta$  3.90 (s, 3H), 7.04-7.06 (m, 2H), 7.16-7.19 (m, 2H), 7.31-7.36 (m, 1H), 7.44-7.49 (m, 2H), 7.53-7.56 (d,  $J = 8.7$  Hz, 1H), 7.81-7.84 (d,  $J = 8.4$  Hz, 1H), 7.94-7.97 (d,  $J = 8.4$  Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz)  $\delta$  55.5, 99.5, 114.0, 126.4, 126.9, 127.5, 128.1, 129.1, 131.4, 133.1, 133.9, 135.7, 135.9, 144.5, 159.4; IR ( $\text{CHCl}_3$ ) 3056, 2918, 2366, 1613  $\text{cm}^{-1}$ ; HRMS  $m/z$  360.0017 (calcd for  $\text{C}_{17}\text{H}_{13}\text{IO}$ , 360.0011).

**1-[4-(2-Iodo-1-naphthyl)phenyl]ethanone (8).** The reaction mixture was chromatographed using 3:1 hexane/EtOAc to afford 64 mg (54%) or 109 mg (92%) of the product from  $\text{I}_2$  or  $\text{ICl}$  respectively as a light yellow solid: mp 135-136  $^\circ\text{C}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz)  $\delta$  2.69 (s, 3H), 7.32-7.37 (m, 4H), 7.45-7.51 (m, 1H), 7.56-7.59 (d,  $J = 8.7$  Hz, 1H), 7.82-7.85 (d,  $J = 8.1$  Hz, 1H), 7.93-7.96 (d,  $J = 8.7$  Hz, 1H), 8.10-8.13 (d,  $J = 8.1$  Hz, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 75 MHz)  $\delta$  27.0, 97.8, 126.7, 126.9, 127.3, 128.3, 128.8, 129.7, 130.7, 133.0, 133.2, 135.7, 136.8, 143.5, 148.3, 198.0; IR ( $\text{CHCl}_3$ ) 3054, 2924, 2847, 1693  $\text{cm}^{-1}$ ; HRMS  $m/z$  372.0017 (calcd for  $\text{C}_{18}\text{H}_{13}\text{IO}$ , 372.0011).

**2-(2-Iodo-1-naphthyl)thiophene (10).** The reaction mixture was chromatographed using hexane to afford 95 mg (94%) of the product as a colorless sticky oil:  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  7.01-7.02 (m, 1H), 7.19-7.22 (m, 1H), 7.36-7.38 (m,

1H), 7.40-7.51 (m, 2H), 7.56-7.61 (m, 2H), 7.80-7.82 (d,  $J = 8.4$  Hz, 1H), 7.93-7.95 (d,  $J = 8.4$  Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  101.6, 126.5, 126.5, 127.1, 127.2, 127.9, 128.6, 130.1, 132.7, 134.6, 135.5, 137.3, 143.9 (one carbon is missing due to overlap); IR ( $\text{CHCl}_3$ ) 3106, 3066, 1643  $\text{cm}^{-1}$ ; HRMS  $m/z$  335.9476 (calcd for  $\text{C}_{14}\text{H}_9\text{IS}$ , 335.9470).

**2-(2-Bromo-1-naphthyl)thiophene (11).** The reaction mixture was chromatographed using 5:1 hexane/EtOAc to afford 39 mg (45%) of the product as a colorless oil:  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  7.05-7.06 (m, 1H), 7.21-7.24 (m, 1H), 7.42-7.53 (m, 3H), 7.61-7.63 (d,  $J = 8.4$  Hz, 1H), 7.70-7.76 (m, 2H), 7.83-7.85 (d,  $J = 7.6$  Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  124.1, 126.3, 126.5, 126.6, 127.1, 127.3, 127.9, 128.7, 129.9, 130.0, 132.2, 132.6, 135.0, 139.8; IR ( $\text{CHCl}_3$ ) 3062, 2925, 2853, 1584  $\text{cm}^{-1}$ ; HRMS  $m/z$  287.9612 (calcd for  $\text{C}_{14}\text{H}_9\text{BrS}$ , 287.9608).

**5-Bromo-2-(2-bromo-1-naphthyl)thiophene (12).** The reaction mixture was chromatographed using 5:1 hexane/EtOAc to afford 39 mg (36%) of the product as a colorless oil:  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  6.80-6.81 (d,  $J = 4.0$  Hz, 1H), 7.15-7.16 (d,  $J = 3.6$  Hz, 1H), 7.44-7.50 (m, 2H), 7.66-7.75 (m, 3H), 7.82-7.84 (d,  $J = 7.6$  Hz, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  113.1, 124.1, 126.2, 126.4, 127.5, 128.0, 129.2, 129.8, 129.9, 130.5, 131.5, 132.2, 134.8, 141.4; IR ( $\text{CHCl}_3$ ) 3053, 2924, 2853, 1582, 1503, 1442  $\text{cm}^{-1}$ ; HRMS  $m/z$  367.8700 (calcd for  $\text{C}_{14}\text{H}_8\text{Br}_2\text{S}$ , 367.8693).

**1-(Cyclohex-1-enyl)-2-iodonaphthalene (14).** The reaction mixture was chromatographed using hexane to afford 69 mg (69%) of the product as a colorless sticky oil:  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  1.76-1.92 (m, 4H), 2.11-2.16 (m, 1H), 2.28-2.31 (m, 1H), 2.36-2.37 (m, 3H), 5.63-5.64 (m, 1H), 7.40-7.47 (m, 3H), 7.75-7.77 (m, 1H), 7.83-7.85 (d,  $J = 8.4$  Hz, 1H), 7.91-7.93 (m, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  22.2, 23.1,

25.5, 29.5, 97.5, 126.1, 126.6, 126.7, 128.1, 128.2, 128.8, 132.4, 133.1, 135.6, 140.4, 146.5; IR (CHCl<sub>3</sub>) 3056, 2927, 2857, 2837, 1643 cm<sup>-1</sup>; HRMS *m/z* 334.0222 (calcd for C<sub>16</sub>H<sub>15</sub>I, 334.0219).

**1-Butyl-2-iodonaphthalene (16).** The reaction mixture was chromatographed using 10:1 hexane/EtOAc to afford 70 mg (75%) of the product as a colorless oil: <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 1.00-1.04 (t, *J* = 7.2 Hz, 3H), 1.53-1.66 (m, 2H), 3.23-3.27 (t, *J* = 7.2 Hz, 3H), 7.37-7.39 (d, *J* = 8.8 Hz, 3H), 7.47-7.51 (m, 2H), 7.78-7.80 (m, 1H), 7.83-7.85 (d, *J* = 8.4 Hz, 1H), 8.03-8.05 (m, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ 14.1, 23.1, 31.9, 37.6, 99.3, 124.6, 125.9, 126.7, 127.8, 128.7, 132.2, 133.5, 136.3, 141.8; IR (CHCl<sub>3</sub>) 3051, 2962, 2927, 2877, 1653 cm<sup>-1</sup>; HRMS *m/z* 310.0220 (calcd for C<sub>14</sub>H<sub>15</sub>I, 310.0219).

**1-Cyclohexyl-2-iodonaphthalene (18).** The reaction mixture was chromatographed using 10:1 hexane/EtOAc to afford 91 mg (90%) of the product as a colorless oil: <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 1.45-1.57 (m, 3H), 1.84-1.95 (m, 5H), 2.23-2.33 (m, 2H), 3.60-3.66 (m, 1H), 7.30-7.32 (d, *J* = 8.4 Hz, 1H), 7.42-7.46 (m, 2H), 7.76-7.78 (m, 2H), 7.88-7.90 (d, *J* = 8.8 Hz, 1H), 8.42-8.44 (m, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ 26.3, 27.5, 31.1, 53.5, 101.4, 125.2, 125.6, 125.7, 128.3, 129.1, 131.9, 134.7, 137.0, 144.7; IR (CHCl<sub>3</sub>) 3056, 2937, 2857, 1583 cm<sup>-1</sup>; HRMS *m/z* 336.0380 (calcd for C<sub>16</sub>H<sub>17</sub>I, 336.0375).

**1,2-Diiodonaphthalene (21).** The reaction mixture was chromatographed using 10:1 hexane/EtOAc to afford 57 mg (50%) of the product as a white solid: mp 81-82 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 7.49-7.57 (m, 3H), 7.70-7.72 (m, 1H), 7.89-7.92 (d, *J* = 8.8 Hz, 1H), 8.23-8.25 (m, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ 109.4, 113.7, 126.9, 128.5,

129.0, 130.0, 132.2, 135.4, 135.8, 136.9; IR (CHCl<sub>3</sub>) 3086, 3039 cm<sup>-1</sup>; HRMS *m/z* 379.8565 (calcd for C<sub>10</sub>H<sub>6</sub>I<sub>2</sub>, 379.8559).

**1-Ethoxy-2-iodonaphthalene (23).** The reaction mixture was chromatographed using 20:1 hexane/EtOAc to afford 46 mg (54%) of the product as a light yellow oil: <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 1.57-1.61 (t, *J* = 7.2 Hz, 3H), 4.09-4.14 (q, *J* = 7.2 Hz, 2H), 7.34-7.36 (d, *J* = 8.8 Hz, 1H), 7.48-7.50 (m, 2H), 7.74-7.76 (d, *J* = 8.4 Hz, 1H), 7.79-7.81 (m, 1H), 8.06-8.08 (m, 2H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ 15.8, 70.3, 87.6, 122.3, 125.6, 126.5, 126.7, 128.0, 128.7, 134.8, 135.3, 155.7; IR (CHCl<sub>3</sub>) 3057, 2978, 2928, 2885 cm<sup>-1</sup>; HRMS *m/z* 297.9858 (calcd for C<sub>12</sub>H<sub>11</sub>IO, 297.9855).

**2-Iodo-3,7-dimethyl-1-phenylnaphthalene (27).** The reaction mixture was chromatographed using 5:1 hexane/EtOAc to afford 82 mg (78%) of the product as a light yellow solid: mp 78-79 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz) δ 2.33 (s, 3H), 2.65 (s, 3H), 7.04 (s, 1H), 7.19-7.22 (m, 2H), 7.25-7.29 (d, *J* = 8.4 Hz, 1H), 7.48-7.52 (m, 3H), 7.63-7.66 (m, 2H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz) δ 22.1, 30.4, 107.3, 126.4, 127.2, 127.2, 127.9, 128.6, 128.7, 128.9, 130.3, 131.5, 132.0, 135.8, 137.4, 145.0; IR (CHCl<sub>3</sub>) 3050, 2947, 2919, 2855 cm<sup>-1</sup>; HRMS *m/z* 358.0225 (calcd for C<sub>18</sub>H<sub>15</sub>I, 358.0219).

**2-Iodo-4-methyl-1-phenylnaphthalene (29).** The reaction mixture was chromatographed using hexane to afford 78 mg (76%) of the product as a white solid: mp 103-104 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz) δ 2.68 (s, 3H), 7.22-7.25 (m, 2H), 7.33-7.35 (m, 1H), 7.40-7.54 (m, 5H), 7.85 (s, 1H), 7.96-7.99 (d, *J* = 8.4 Hz, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz) δ 19.2, 98.6, 124.5, 126.2, 126.6, 128.0, 128.1, 128.6, 130.4, 132.3, 133.6, 135.9, 136.1, 143.1, 143.7; IR (CHCl<sub>3</sub>) 3062, 3029, 2943, 2920, 2856, 1580 cm<sup>-1</sup>; HRMS *m/z* 344.0068 (calcd for C<sub>17</sub>H<sub>13</sub>I, 344.0062).

**2-Iodo-7-methoxy-1-phenylnaphthalene (31).** The reaction mixture was chromatographed using 10:1 hexane/EtOAc to afford 97 mg (90%) of the product as a colorless oil:  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  3.64 (s, 3H), 6.67 (s, 1H), 7.11-7.15 (d,  $J = 8.8$  Hz, 2H), 7.25-7.27 (m, 2H), 7.47-7.52 (m, 4H), 7.72-7.74 (d,  $J = 9.2$  Hz, 1H), 7.82-7.85 (d,  $J = 8.8$  Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  55.1, 99.2, 106.0, 118.6, 127.9, 128.4, 128.6, 128.7, 129.5, 129.9, 133.3, 134.6, 143.2, 143.5, 158.1; IR ( $\text{CHCl}_3$ ) 3058, 2956, 2933, 2849, 1622, 1500  $\text{cm}^{-1}$ ; HRMS  $m/z$  360.0017 (calcd for  $\text{C}_{17}\text{H}_{13}\text{IO}$ , 360.0011).

**2-Bromo-7-methoxy-1-phenylnaphthalene (32).** The reaction mixture was chromatographed using 10:1 hexane/EtOAc to afford 86 mg (55%) of the product as a colorless oil:  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  3.64 (s, 3H), 6.69-6.70 (d,  $J = 2.4$  Hz, 1H), 7.11-7.14 (dd,  $J = 2.4, 8.8$  Hz, 1H), 7.29-7.31 (m, 2H), 7.45-7.53 (m, 3H), 7.56-7.64 (m, 2H), 7.72-7.74 (d,  $J = 9.2$  Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  55.2, 105.5, 118.4, 122.2, 127.6, 127.8, 127.9, 128.5, 128.7, 129.5, 130.1, 135.2, 138.6, 139.9, 158.3; IR ( $\text{CHCl}_3$ ) 3058, 3001, 2959, 2937, 2838  $\text{cm}^{-1}$ ; HRMS  $m/z$  312.0155 (calcd for  $\text{C}_{17}\text{H}_{13}\text{BrO}$ , 312.0150).

**3-Hydroxy-2-iodo-1-phenylspiro[4.5]deca-1,6,9-trien-8-one (33).** The reaction mixture was chromatographed using EtOAc to afford 27.3 mg (25%) of the product as a yellow solid: mp 168-170  $^\circ\text{C}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  2.32-2.36 (m, 1H), 2.61-2.67 (m, 2H), 5.02-5.04 (m, 1H), 6.21-6.26 (m, 2H), 6.76-6.79 (m, 1H), 6.95-6.98 (m, 1H), 7.12-7.13 (m, 2H), 7.26-7.30 (m, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  44.0, 56.9, 81.1, 106.2, 127.7, 128.3, 128.8, 129.2, 129.6, 134.9, 150.5, 151.3, 152.3, 185.4; IR ( $\text{CH}_2\text{Cl}_2$ ) 3369, 1667, 1613  $\text{cm}^{-1}$ ; HRMS  $m/z$  363.9968 (calcd for  $\text{C}_{16}\text{H}_{13}\text{IO}_2$ , 363.9960).

**7-Fluoro-2-iodo-3-methyl-1-phenylnaphthalene (35).** The reaction mixture was chromatographed using 10:1 hexane/EtOAc to afford 86 mg (79%) of the product as a light yellow solid: mp 112-114 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz) δ 2.66 (s, 3H), 6.89-6.94 (m, 1H), 7.19-7.24 (m, 3H), 7.48-7.55 (m, 3H), 7.72-7.77 (m, 2H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz) δ 30.4, 108.4, 110.9, 111.2, 116.8, 117.2, 127.1, 127.1, 128.2, 128.9, 129.6, 129.8, 130.1, 130.2, 130.2, 132.3, 132.5, 137.8, 137.8, 144.3, 145.1, 159.1, 162.3 (the extra peaks are due to F splitting); IR (CHCl<sub>3</sub>) 3056, 2921, 1628 cm<sup>-1</sup>; HRMS *m/z* 361.9975 (calcd for C<sub>17</sub>H<sub>12</sub>FI, 361.9968); Anal. Calcd for C<sub>17</sub>H<sub>12</sub>FI: C, 56.4; H, 3.4; Found: C, 56.2; H, 3.9.

**2-Iodo-1-phenanthrene (37).** The reaction mixture was chromatographed using 5:1 hexane/EtOAc to afford 83 mg (70%) of product as a white solid: mp 162-163 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz) δ 7.27-7.40 (m, 3H), 7.52-7.70 (m, 6H), 7.85-7.88 (d, *J* = 8.1 Hz, 1H), 8.16-8.19 (d, *J* = 8.8 Hz, 1H), 8.43-8.46 (d, *J* = 8.9 Hz, 1H), 8.70-8.73 (d, *J* = 8.2 Hz, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz) δ 99.4, 122.9, 124.0, 125.8, 127.2, 127.3, 127.9, 128.1, 128.7, 128.8, 130.1, 130.2, 131.6, 132.0, 136.3, 143.8, 145.1 (one carbon was missing due to overlap); IR (CHCl<sub>3</sub>) 3082, 3050, 2921, 2860 cm<sup>-1</sup>; HRMS *m/z* 380.0068 (calcd for C<sub>20</sub>H<sub>13</sub>I, 380.0062).

**2-Iodo-6-methoxy-3-methyl-1-phenylnaphthalene (39).** The reaction mixture was chromatographed using 40:1 hexane/EtOAc to afford 93 mg (83%) of product as a sticky oil: <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz) δ 2.66 (s, 3H), 3.91 (s, 3H), 6.90-6.91 (dd, *J* = 2.7, 9.3 Hz, 1H), 7.06-7.07 (d, *J* = 2.4 Hz, 1H), 7.19-7.23 (m, 3H), 7.47-7.52 (m, 3H), 7.67 (s, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz) δ 30.3, 55.4, 103.5, 105.0, 118.5, 126.3, 127.2,

127.7, 128.4, 129.0, 130.0, 134.3, 138.8, 144.6, 145.3, 158.0; IR (CHCl<sub>3</sub>) 2961, 2916, 2852, 1628 cm<sup>-1</sup>; HRMS *m/z* 374.0174 (calcd for C<sub>18</sub>H<sub>15</sub>IO, 374.0168).

**2-Iodo-8-methoxy-3-methyl-1-phenylnaphthalene (40).** The reaction mixture was chromatographed using 40:1 hexane/EtOAc to afford 13.4 mg (12%) of product as a sticky oil: <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz) δ 2.66 (s, 3H), 3.91 (s, 3H), 6.90-6.91 (dd, *J* = 2.7, 9.3 Hz, 1H), 7.06-7.07 (d, *J* = 2.4 Hz, 1H), 7.19-7.23 (m, 3H), 7.47-7.52 (m, 3H), 7.67 (s, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz) δ 16.1, 56.4, 91.8, 104.0, 120.0, 124.5, 126.8, 127.2, 127.4, 129.0, 134.0, 136.6, 138.4, 145.0, 154.2; IR (CHCl<sub>3</sub>) 2971, 2918, 2852, 1642 cm<sup>-1</sup>; HRMS *m/z* 374.0174 (calcd C<sub>18</sub>H<sub>15</sub>IO, 374.0168).

**3-Iodo-4-phenylphenanthrene (42).** The reaction mixture was chromatographed using 5:1 hexane/EtOAc to afford 85 mg (75%) of product as a white solid: mp 155-156 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz) δ 7.10-7.13 (m, 1H), 7.28-7.31 (m, 2H), 7.40-7.45 (m, 2H), 7.57-7.60 (m, 4H), 7.70-7.83 (m, 3H), 8.19-8.22 (d, *J* = 8.4 Hz, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz) δ 103.9, 125.7, 126.5, 127.2, 128.2, 128.4, 128.7, 128.9, 129.6, 130.0, 130.2, 130.2, 130.4, 133.6, 133.7, 137.0, 143.9, 148.1; IR (CHCl<sub>3</sub>) 3067, 3052, 2923 cm<sup>-1</sup>; HRMS *m/z* 380.0068 (calcd for C<sub>20</sub>H<sub>13</sub>I, 380.0062).

**3-Iodo-4-phenyldibenzo[*b,d*]thiophene (44).** The reaction mixture was chromatographed using hexane to afford 96 mg (83%) of the product as a white solid: mp 116-117 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz) δ 7.37-7.52 (m, 7H), 7.70-7.77 (m, 2H), 7.96-7.99 (d, *J* = 8.4 Hz, 1H), 8.08-8.11 (m, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz) δ 96.8, 122.0, 122.1, 122.8, 124.9, 127.5, 128.9, 129.0, 129.4, 135.6, 135.6, 135.8, 140.3, 141.2, 141.4, 143.1; IR (CHCl<sub>3</sub>) 3056, 2918, 2360, 2849, 1564 cm<sup>-1</sup>; HRMS *m/z* 385.9632 (calcd for C<sub>18</sub>H<sub>11</sub>IS, 385.9626).

**4-(Cyclohexen-1-yl)-3-iododibenzo[*b,d*]thiophene (46).** The reaction mixture was chromatographed using 5:1 hexane/EtOAc to afford 86 mg (74%) of product as a white solid: mp 105-106 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz) δ 1.75-1.92 (m, 4H), 2.25-2.28 (m, 3H), 2.39-2.44 (m, 1H), 5.78-5.81 (m, 1H), 7.38-7.47 (m, 2H), 7.65-7.67 (d, *J* = 8.1 Hz, 1H), 7.77-7.80 (m, 1H), 7.84-7.87 (d, *J* = 8.1 Hz, 1H), 8.05-8.08 (m, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz) δ 22.2, 23.1, 25.5, 27.8, 96.1, 121.2, 122.0, 122.8, 124.7, 127.3, 129.3, 135.3, 135.6, 135.9, 140.2, 140.5, 141.2, 143.3; IR (CHCl<sub>3</sub>) 3062, 2933, 2834, 2853, 1432 cm<sup>-1</sup>; HRMS *m/z* 389.9946 (calcd for C<sub>18</sub>H<sub>15</sub>IS, 389.9939); Anal. Calcd for C<sub>18</sub>H<sub>15</sub>IS: C, 55.4; H, 3.9; Found: C, 55.9; H, 4.4.

**2-Iodo-3-methyl-1-phenyl-9*H*-carbazole (48).** The reaction mixture was chromatographed using 3:1 hexane/EtOAc to afford 57 mg (50%) of the product as a white solid: mp 135-136 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 2.70 (s, 3H), 7.21-7.23 (d, *J* = 9.2 Hz, 1H), 7.26-7.28 (d, *J* = 8.0 Hz, 1H), 7.36-7.40 (m, 3H), 7.50-7.58 (m, 4H), 7.96 (s, 1H), 8.04-8.06 (d, *J* = 7.6 Hz, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ 30.1, 103.0, 110.8, 119.6, 119.7, 120.6, 122.8, 123.0, 126.4, 128.3, 129.1, 130.0, 130.2, 132.4, 136.6, 139.6, 141.6; IR (CHCl<sub>3</sub>) 3433, 3058, 2921, 2849 cm<sup>-1</sup>; HRMS *m/z* 383.0177 (calcd for C<sub>19</sub>H<sub>14</sub>IN, 383.0171).

**2-Iodo-1-phenyl-3,4-dihydronaphthalene (50).** The reaction mixture was chromatographed using hexane to afford 76 mg (76%) of the product as a white solid: mp 80-81 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 2.95-2.99 (m, 2H), 3.07-3.11 (m, 2H), 6.61-6.63 (d, *J* = 7.6 Hz, 1H), 6.99-7.03 (m, 1H), 7.13-7.19 (m, 4H), 7.38-7.46 (m, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ 30.1, 39.8, 101.1, 126.4, 126.6, 127.4, 127.4, 127.6, 128.5, 129.7,

134.7, 135.3, 143.3, 144.6; IR (CHCl<sub>3</sub>) 3062, 3017, 2940, 2889, 2832, 2361 cm<sup>-1</sup>; HRMS *m/z* 332.0079 (calcd for C<sub>16</sub>H<sub>13</sub>I, 332.0062).

**2,6-Diiodo-1,5-diphenylanthracene (56).** The reaction mixture was chromatographed using hexane to afford 56 mg (32%) of the product as a white solid: mp 212-213 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 6.18 (m, 2H), 6.70 (m, 1H), 6.97-7.05 (m, 2H), 7.18-7.24 (m, 1H), 7.32-7.34 (d, *J* = 8.4 Hz, 1H), 7.55 (s, 1H), 8.08-8.10 (d, *J* = 8.4 Hz, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz) δ 101.0, 126.9, 127.3, 127.6, 130.8, 139.0, 139.6, 142.4, 144.7; IR (CHCl<sub>3</sub>) 3080, 3052, 3031, 2919, 2850 cm<sup>-1</sup>; HRMS *m/z* 581.9344 (calcd for C<sub>26</sub>H<sub>16</sub>I<sub>2</sub>, 581.9341); Anal. Calcd for C<sub>26</sub>H<sub>16</sub>I<sub>2</sub>: C, 53.6; H, 2.8; Found: C, 54.0; H, 3.1.

**3,6-Diiodo-4,5-diphenylphenanthrene (57).** The reaction mixture was chromatographed using hexane to afford 35 mg (20%) of the product as a white solid: decomposes at 195 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 7.20-7.24 (m, 2H), 7.26-7.30 (m, 3H), 7.39-7.41 (d, *J* = 9.2 Hz, 1H), 7.50-7.59 (m, 9H), 8.20-8.22 (d, *J* = 8.4 Hz, 1H), 7.55 (s, 1H), 8.08-8.10 (d, *J* = 8.4 Hz, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz) δ 99.6, 104.3, 126.9, 127.8, 127.9, 128.2, 128.5, 129.2, 129.6, 129.8, 129.9, 130.0, 130.1, 132.8, 133.3, 134.7, 137.4, 143.8, 143.9, 144.3, 147.8; IR (CHCl<sub>3</sub>) 3058, 2925, 2849 cm<sup>-1</sup>; HRMS *m/z* 581.9344 (calcd for C<sub>26</sub>H<sub>16</sub>I<sub>2</sub>, 581.9341).

**10-Iodo-9-phenyl-1,2,3,4-tetrahydrophenanthrene (60).** The reaction mixture was chromatographed using 20:1 hexane/EtOAc to afford the product as a light yellow solid (72% from **59**, 99% from **61**): mp 134-135 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 1.92-1.93 (m, 4H), 2.91-2.94 (m, 2H), 3.18-3.21 (m, 2H), 7.21-7.33 (m, 4H), 7.46-7.53 (m, 4H), 8.01-8.03 (d, *J* = 8.4 Hz, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ 22.9, 24.3, 27.0, 38.6,

110.0, 122.9, 125.6, 126.2, 127.6, 128.1, 128.4, 130.3, 131.9, 132.0, 133.6, 135.6, 143.7  
145.3; IR (CHCl<sub>3</sub>) 3067, 2930, 2856 cm<sup>-1</sup>; HRMS *m/z* 384.0380 (calcd for C<sub>20</sub>H<sub>17</sub>I,  
384.0375).

**3-Iodo-4-phenyl-2-naphthol (66).** The reaction mixture was chromatographed  
using 10:1 hexane/EtOAc to afford the product as a light yellow solid: mp 65-68 °C; <sup>1</sup>H  
NMR (CDCl<sub>3</sub>, 400 MHz) δ 5.68 (s, 1H), 7.17-7.19 (m, 1H), 7.22-7.25 (m, 2H), 7.28-7.30  
(d, *J* = 8.8 Hz, 1H), 7.41-7.53 (m, 5H), 7.72-7.74 (d, *J* = 8.4 Hz, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>,  
100 MHz) δ 96.8, 109.0, 124.3, 126.6, 127.1, 127.3, 128.1, 128.6, 128.7, 129.8, 134.6,  
143.4, 146.4, 151.1; IR (CHCl<sub>3</sub>) 3435, 3061, 1648 cm<sup>-1</sup>; HRMS *m/z* 345.9860 (calcd for  
C<sub>16</sub>H<sub>11</sub>IO, 345.9855).

**4-(Cyclohexen-1-yl)-3-iodo-2-naphthol (69).** The reaction mixture was  
chromatographed using 10:1 hexane/EtOAc to afford 68 mg (65%) of the product as a  
light orange oil: <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 1.77-1.93 (m, 4H), 2.15-2.18 (m, 1H),  
2.29-2.32 (m, 3H), 5.63-5.65 (m, 2H), 7.26-7.30 (m, 2H), 7.40-7.44 (m, 1H), 7.66-7.68  
(d, *J* = 8.0 Hz, 1H), 7.81-7.83 (d, *J* = 8.4 Hz, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ 22.1,  
23.0, 25.4, 29.3, 95.6, 108.1, 124.2, 126.7, 126.8, 127.0, 127.5, 129.4, 134.8, 140.6,  
148.5, 151.1; IR (CHCl<sub>3</sub>) 3470, 3058, 2928, 2856, 2834, 1563 cm<sup>-1</sup>; HRMS *m/z* 350.0172  
(calcd for C<sub>16</sub>H<sub>15</sub>IO, 350.0168).

**4-Cyclohexyl-3-iodo-2-naphthol (71).** The reaction mixture was  
chromatographed using 10:1 hexane/EtOAc to afford 83 mg (79%) of the product as a  
light yellow solid: mp 103-104 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 1.44-1.56 (m, 3H),  
1.84-1.97 (m, 5H), 2.26-2.35 (m, 3H), 3.66-3.72 (m, 1H), 5.72 (s, 1H), 7.24-7.31 (m,  
2H), 7.37-7.41 (m, 1H), 7.66-7.68 (d, *J* = 8.0 Hz, 1H), 8.37-8.39 (d, *J* = 8.8 Hz, 1H); <sup>13</sup>C

NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$  26.2, 27.5, 31.1, 54.5, 99.9, 108.7, 122.9, 125.5, 126.4, 126.8, 127.7, 135.4, 146.8, 151.0; IR (CHCl<sub>3</sub>) 3463, 2941, 2925, 2853 cm<sup>-1</sup>; HRMS *m/z* 352.0329 (calcd for C<sub>16</sub>H<sub>17</sub>IO, 352.0324).

**8-Methoxyfluoranthene (72).** To a solution of 2-iodo-1-(4-methoxyphenyl)naphthalene (0.20 mmol) and cesium pivalate (0.40 mmol) in 3.5 mL of DMF were added Pd(OAc)<sub>2</sub> (2.5 mg, 5 mol %) and DPPM (3.84 mg, 10 mol %). The resulting mixture was heated under an N<sub>2</sub> atmosphere at 120 °C for 24 h. The mixture was cooled to room temperature and diluted with 25 ml of ether, washed with 25 mL of satd aq NaCl, dried over MgSO<sub>4</sub> and filtered. The solvent was evaporated under reduced pressure. The reaction mixture was chromatographed using 40:1 hexane/EtOAc to afford 30.2 mg (65%) of the product as a light yellow solid: mp 120-121 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz)  $\delta$  3.93 (s, 3H), 6.89-6.92 (m, 1H), 7.45-7.46 (m, 1H), 7.56-7.63 (m, 2H), 7.74-7.84 (m, 4H), 7.89-7.91 (m, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz) 55.9, 107.9, 113.1, 119.2, 120.2, 122.5, 125.7, 127.2, 128.0, 128.3, 130.2, 132.7, 133.1, 137.1, 137.2, 141.5, 160.2; IR (CHCl<sub>3</sub>) 3053, 3012, 2971, 2940, 2832, 2361, 1603 cm<sup>-1</sup>; HRMS *m/z* 232.0891 (calcd for C<sub>17</sub>H<sub>12</sub>O, 232.0888).

**1-(Fluoranthen-8-yl)ethanone (73).** To a solution of 1-[4-(2-iodo-1-naphthyl)phenyl]ethanone (0.20 mmol) and cesium pivalate (0.40 mmol, 2.0 equiv) in 3.5 mL of DMF were added Pd(OAc)<sub>2</sub> (2.5 mg, 5 mol %) and DPPM (3.84 mg, 10 mol %). The resulting mixture was heated under an N<sub>2</sub> atmosphere at 120 °C for 3.5 d. The mixture was cooled to room temperature and diluted with 25 ml of ether, washed with 25 mL of satd aq NaCl, dried over MgSO<sub>4</sub> and filtered. The solvent was evaporated under reduced pressure. The reaction mixture was chromatographed using 20:1 hexane/EtOAc

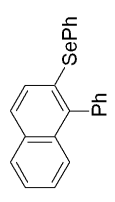
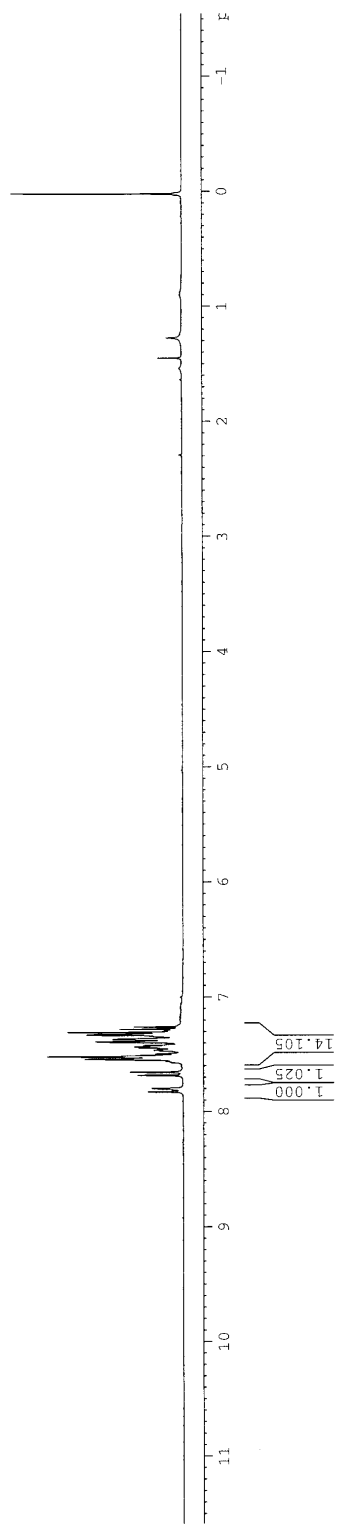
to afford 31.2 mg (64%) of the product as a light green yellow solid: mp 95-96 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz) δ 2.70 (s, 3H), 7.64-7.69 (m, 2H), 7.87-8.01 (m, 6H), 8.47-8.48 (m, 1H), 7.74-7.84 (m, 4H), 7.89-7.91 (m, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz) 27.1, 121.0, 121.4, 121.7, 127.4, 128.1, 128.3, 128.5, 128.6, 130.2, 133.4, 135.9, 136.2, 136.5, 139.8, 143.9, 198.2; IR (CHCl<sub>3</sub>) 3050, 3005, 2925, 1678, 1614, 1432 cm<sup>-1</sup>; HRMS *m/z* 244.0892 (calcd for C<sub>18</sub>H<sub>12</sub>O, 244.0888).

**2,4-Diphenylnaphtho[2,3-*b*]furan (74).** To a solution of 3-iodo-4-phenyl-2-naphthol (0.42 mmol) in 4 mL of NEt<sub>3</sub>/DMF (V/V = 1/1) in a vial was added PdCl<sub>2</sub>(PPh<sub>3</sub>)<sub>2</sub> (15 mg, 2.5 mol %), CuI (2.1 mg, 1.25 mol %), and phenylacetylene (0.50 mmol, 1.2 equiv). The vial was flushed with N<sub>2</sub> and sealed. The reaction mixture was heated to 100 °C for 12 h, cooled to room temperature, filtered, diluted with 25 ml of ether, washed with 25 mL of satd aq NaCl, dried over MgSO<sub>4</sub>, and filtered. The solvent was evaporated under reduced pressure. The reaction mixture was chromatographed using hexane to afford 67 mg (51%) of the product as a white solid: mp 183-184 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz) δ 6.94 (s, 1H), 7.23-7.37 (m, 2H), 7.42-7.45 (m, 3H), 7.49-7.52 (m, 1H), 7.55-7.58 (m, 1H), 7.87-7.98 (m, 5H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 75 MHz) 100.8, 106.4, 124.2, 124.8, 125.3, 125.9, 127.6, 128.3, 128.6, 128.6, 128.9, 129.1, 129.9, 130.2, 130.7, 131.2, 132.0, 138.3, 153.2, 158.2; IR (CHCl<sub>3</sub>) 2924, 2852 cm<sup>-1</sup>; HRMS *m/z* 320.1205 (calcd for C<sub>24</sub>H<sub>16</sub>O, 320.1201).

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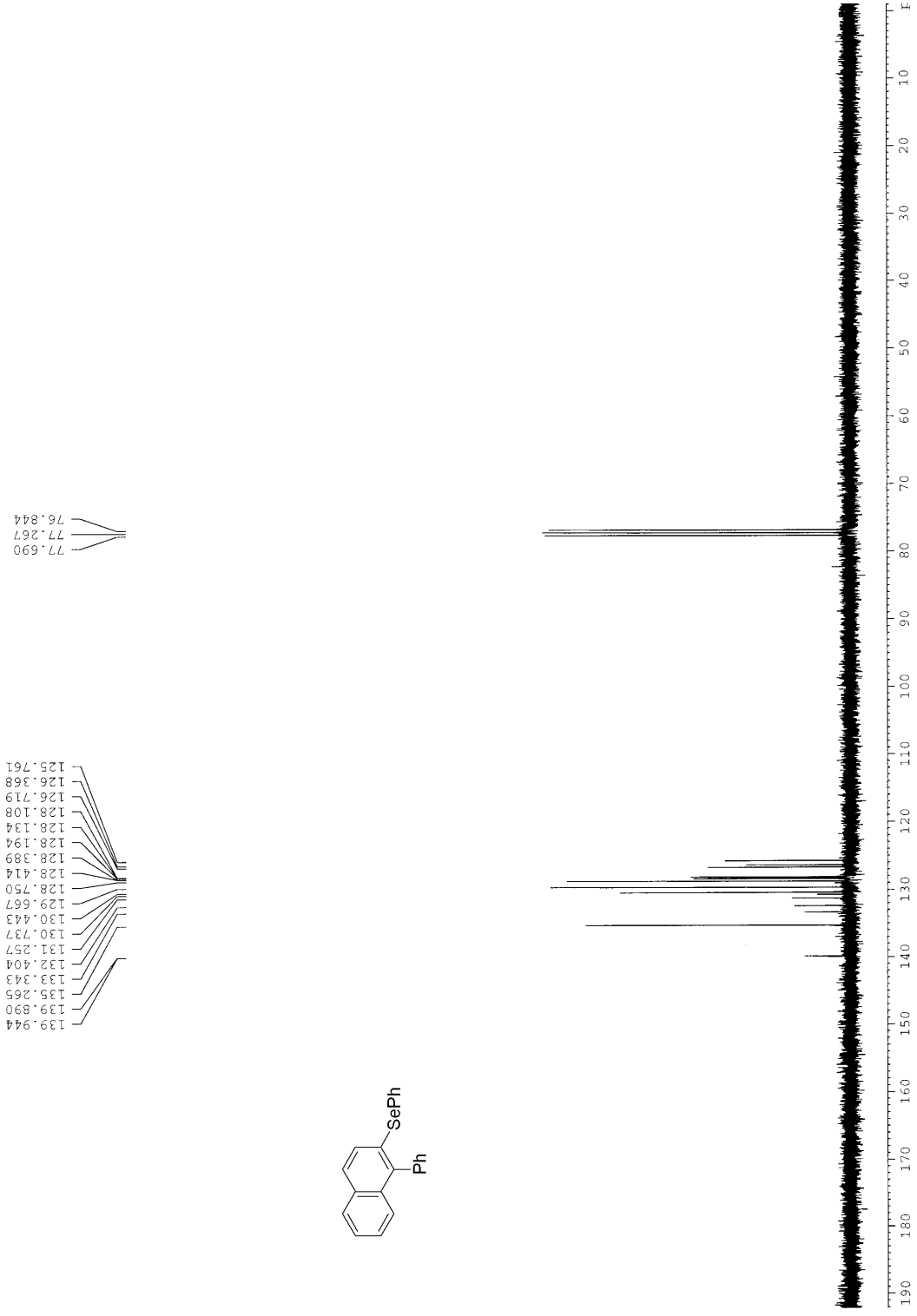
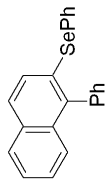
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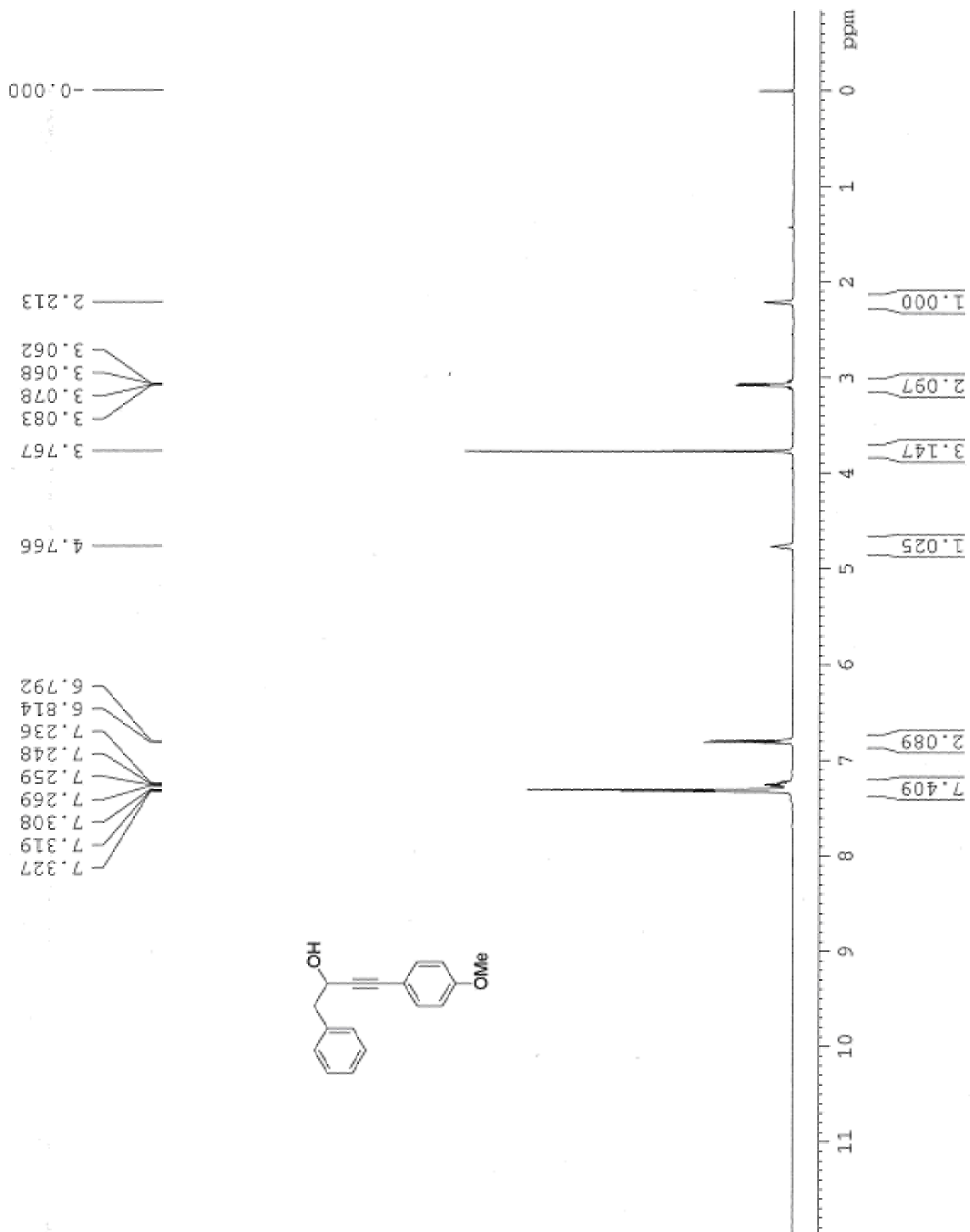
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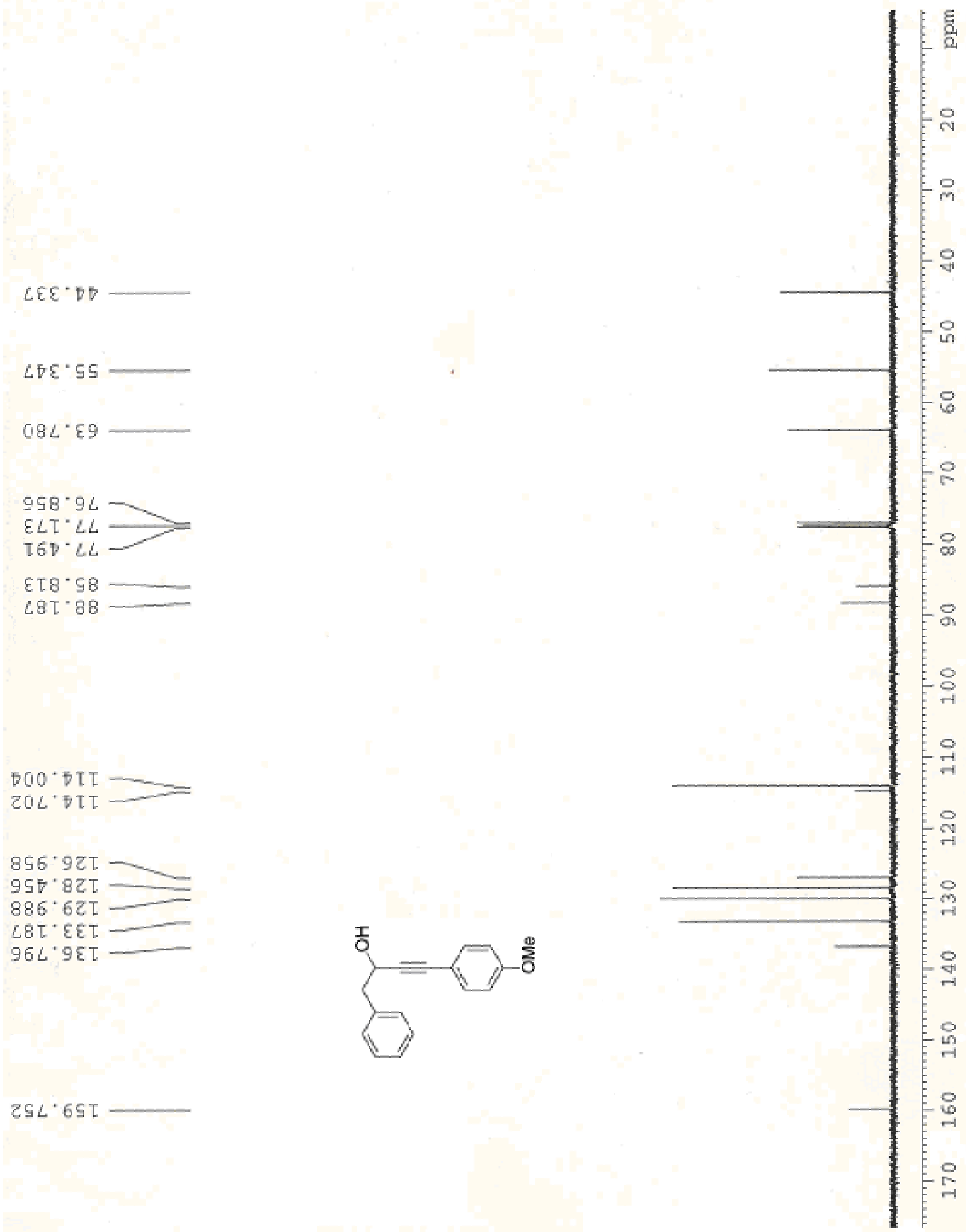


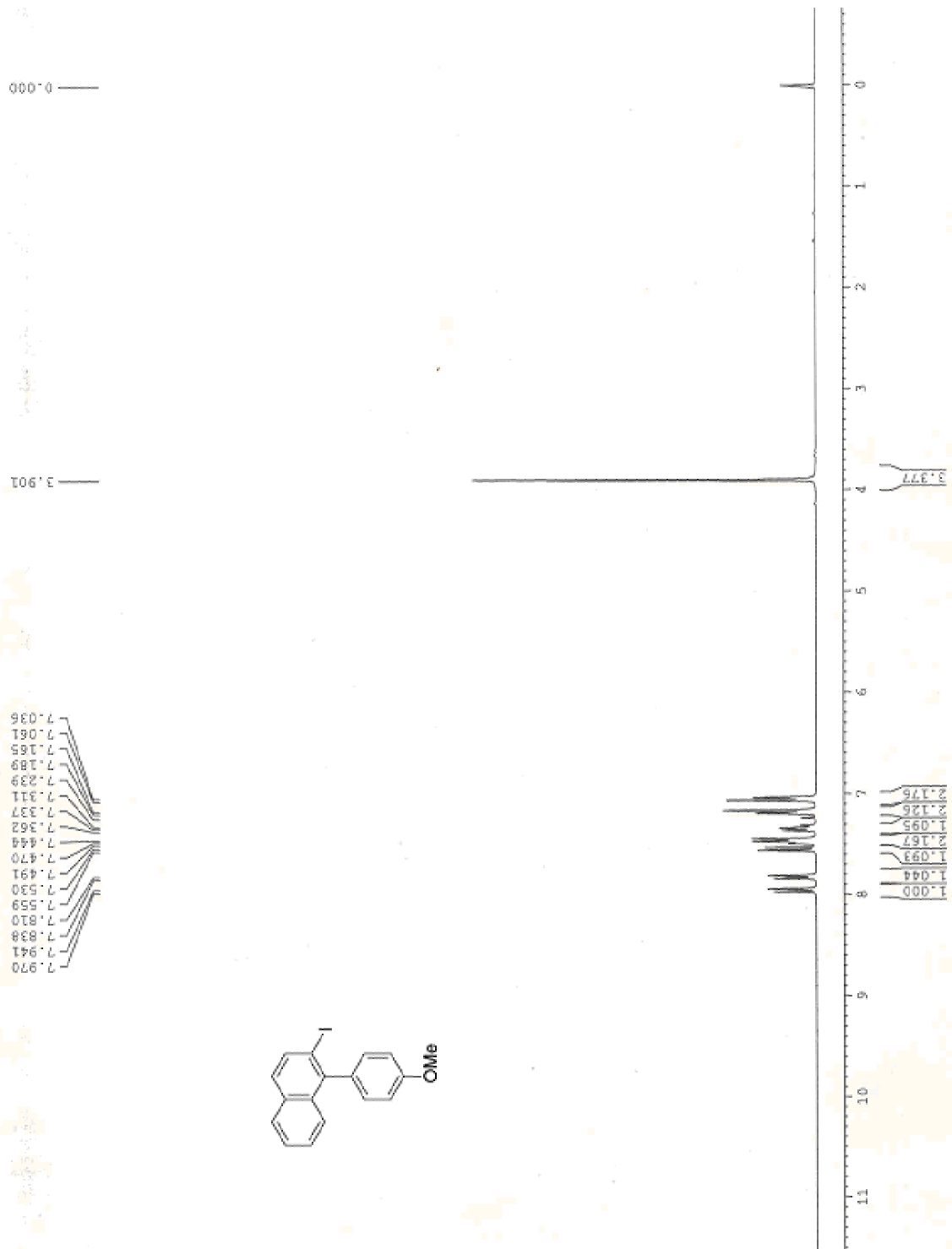
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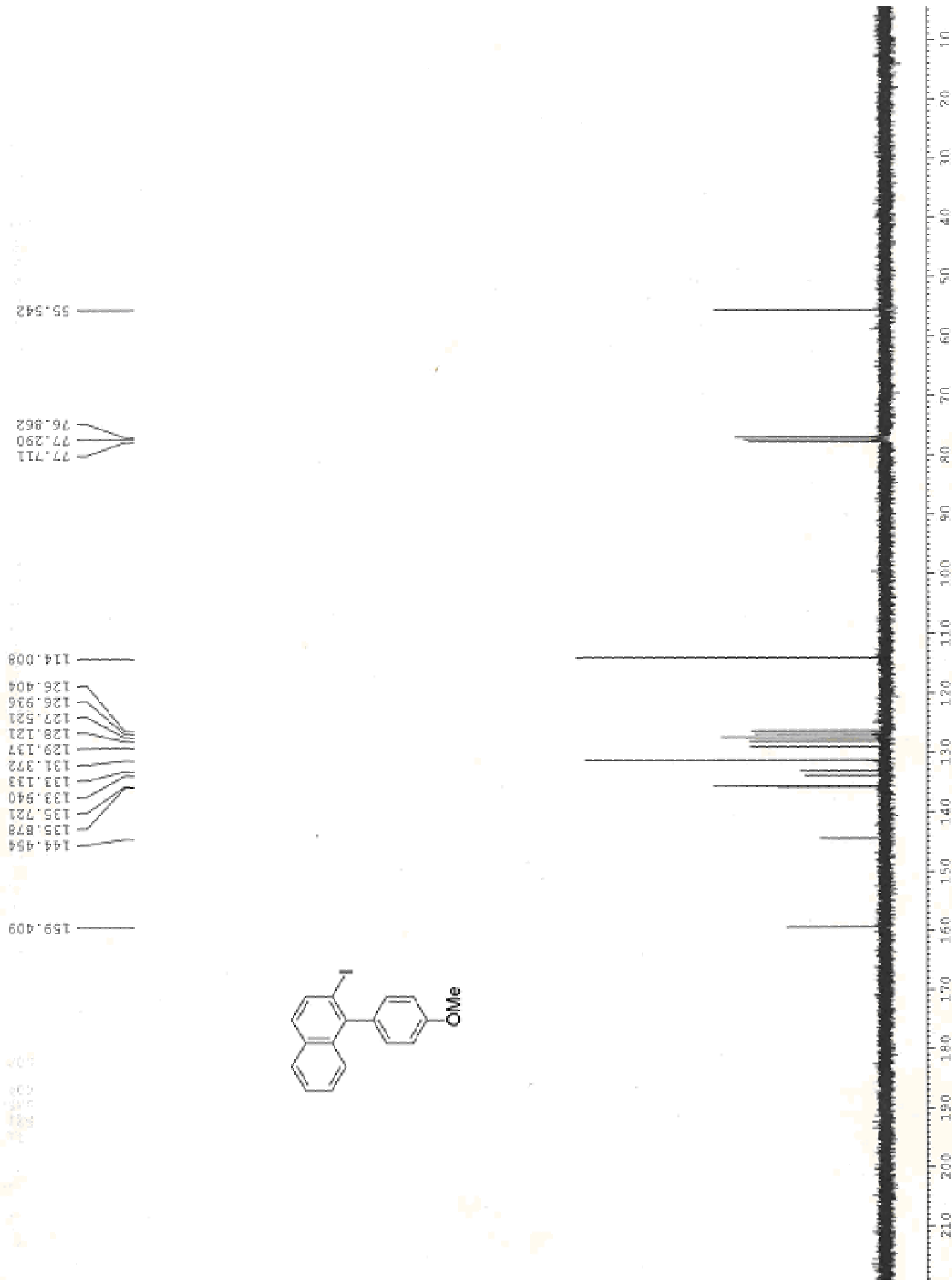
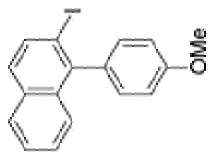
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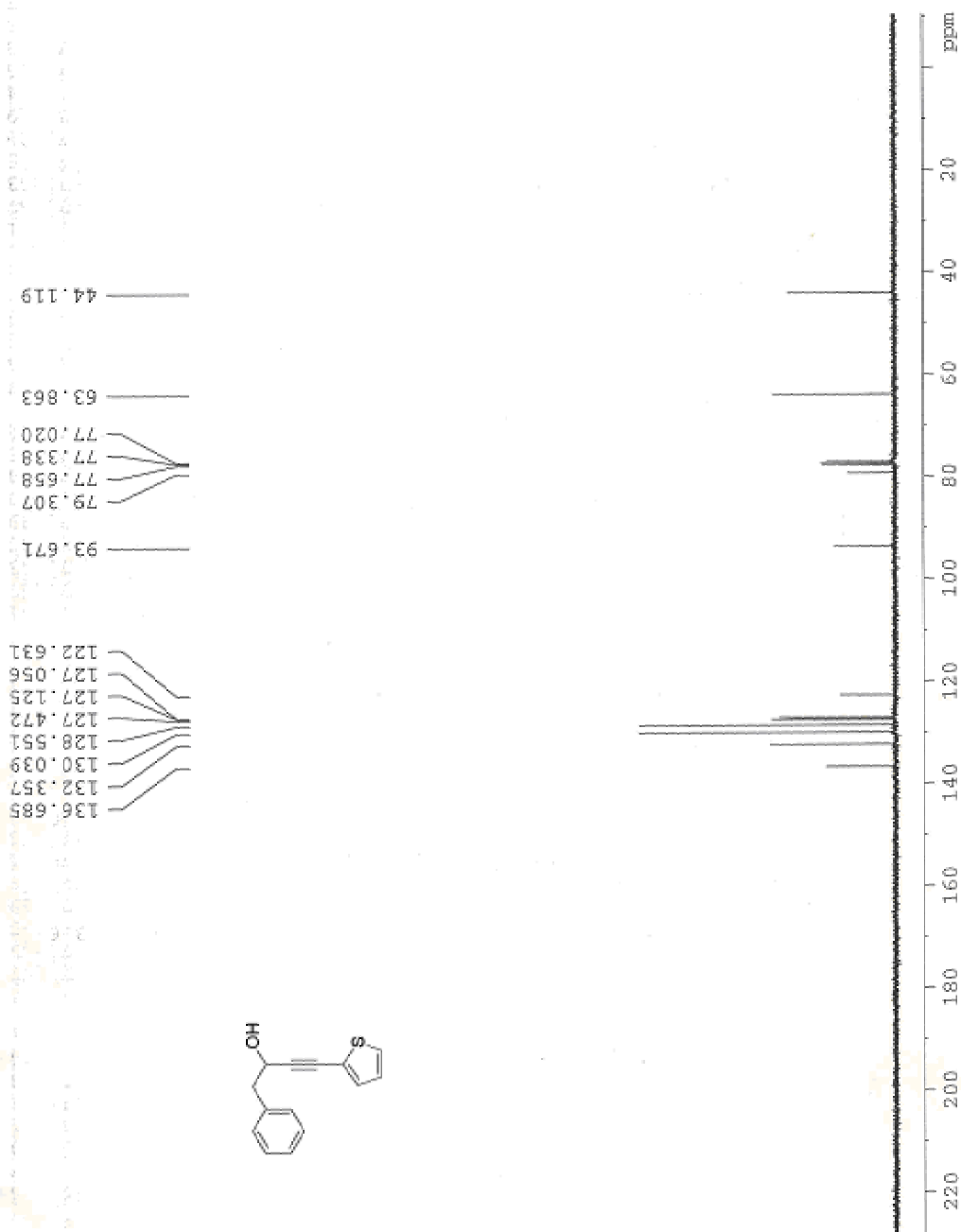


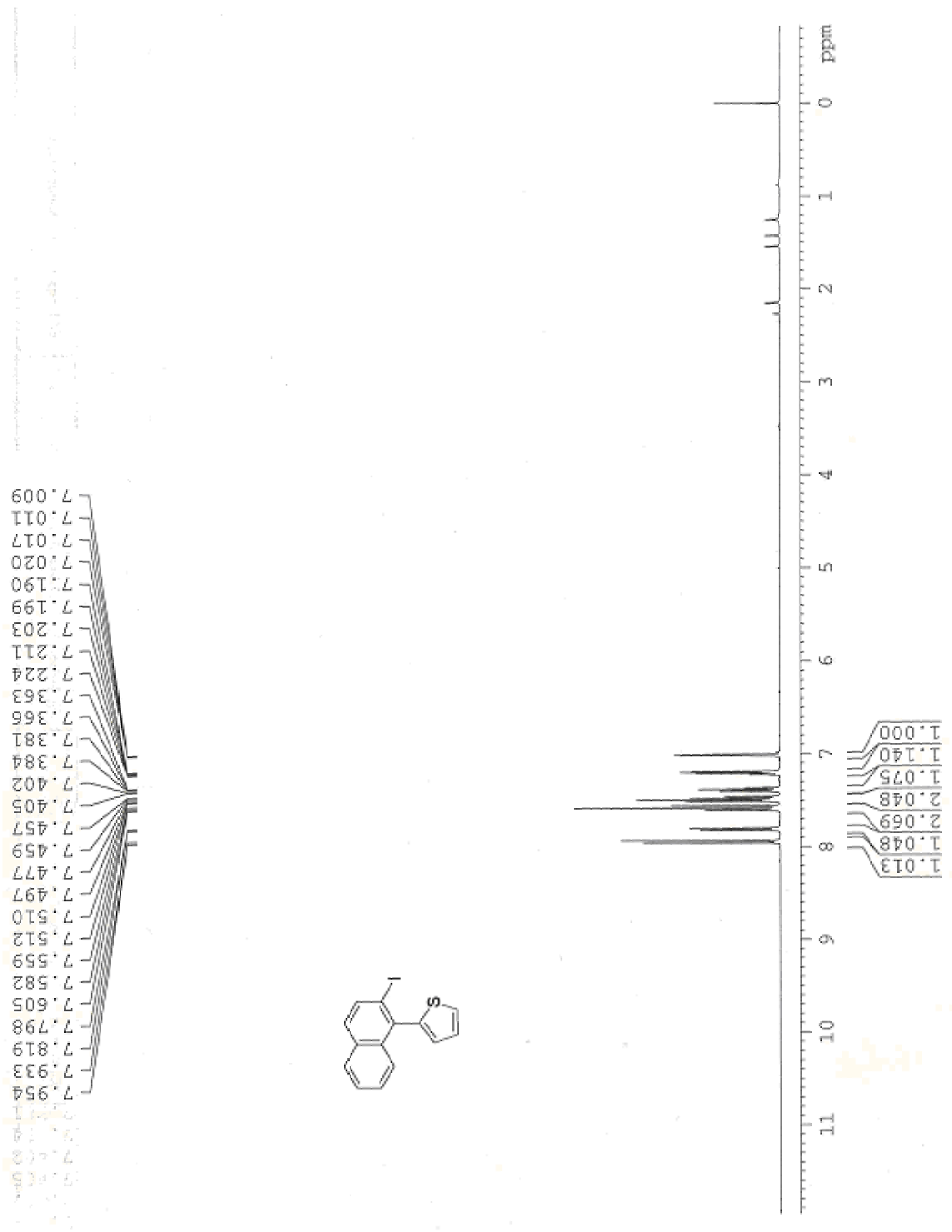


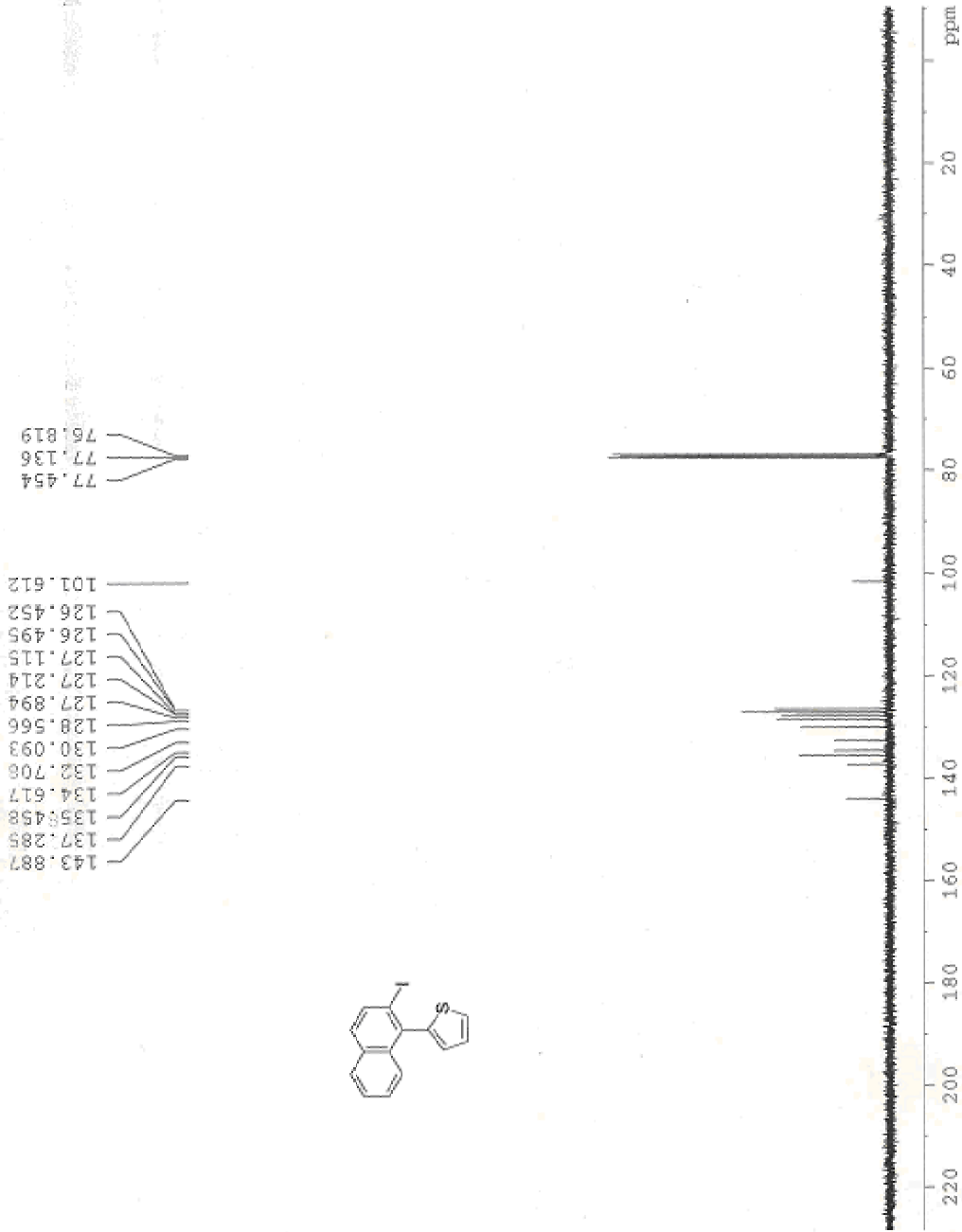
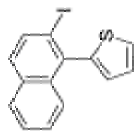


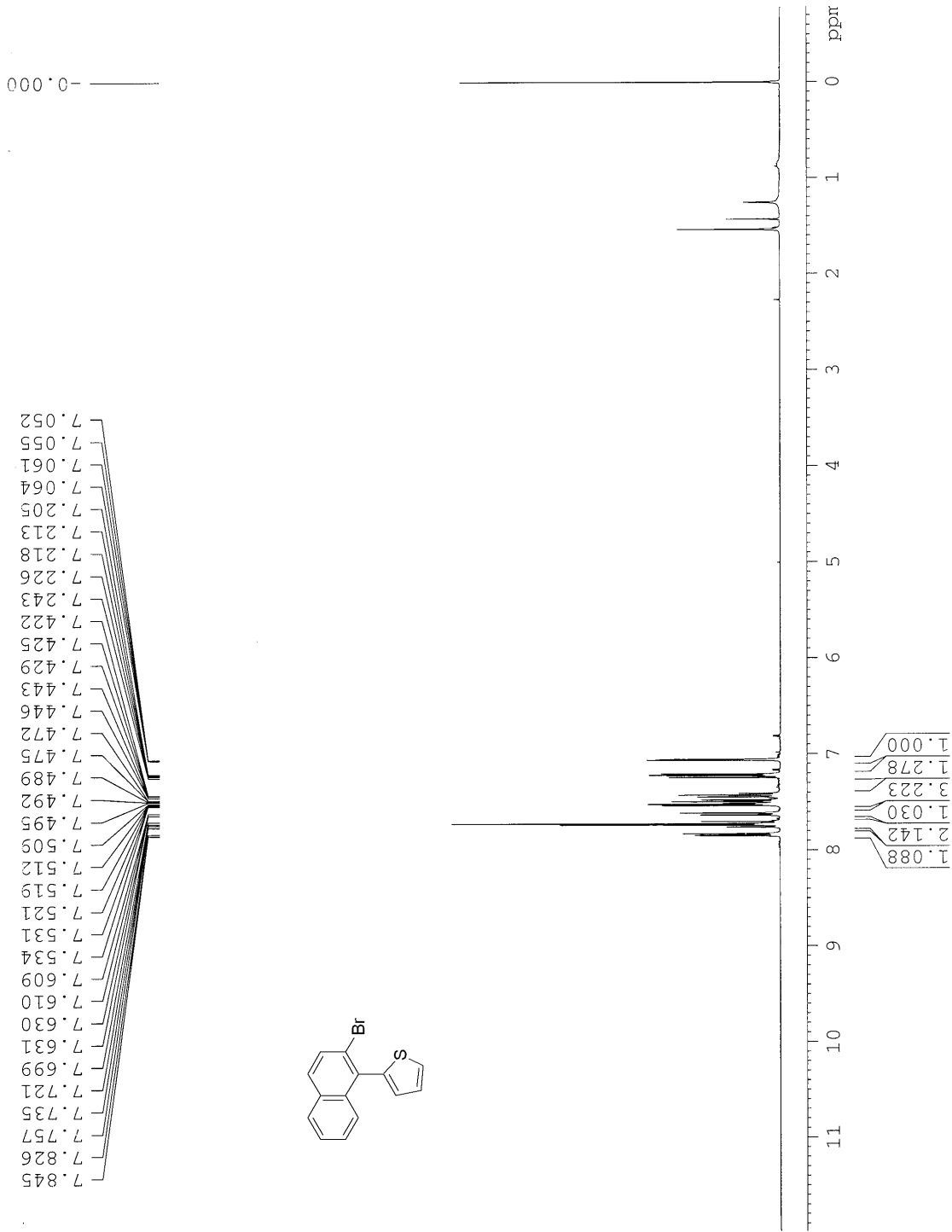


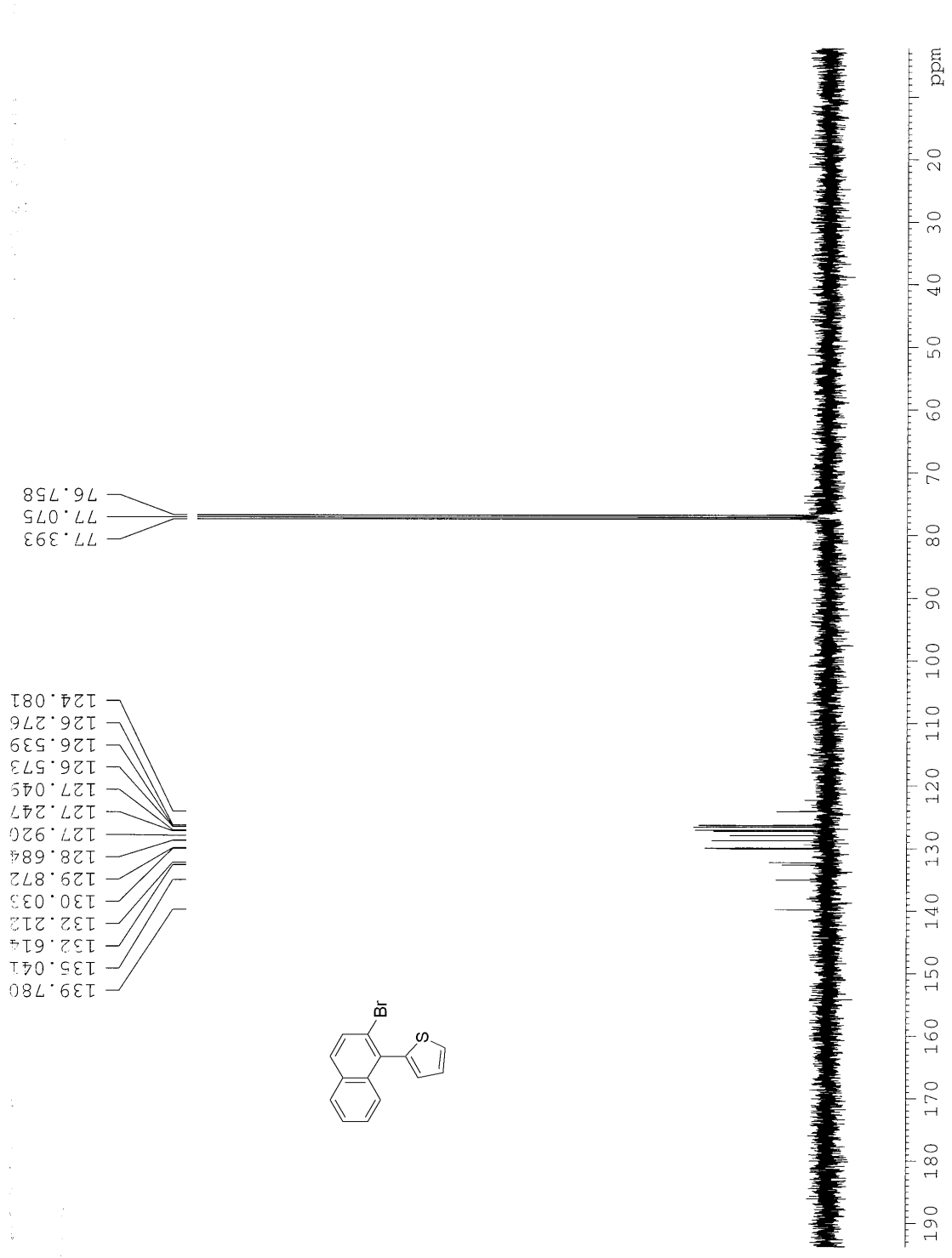


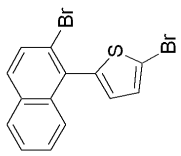




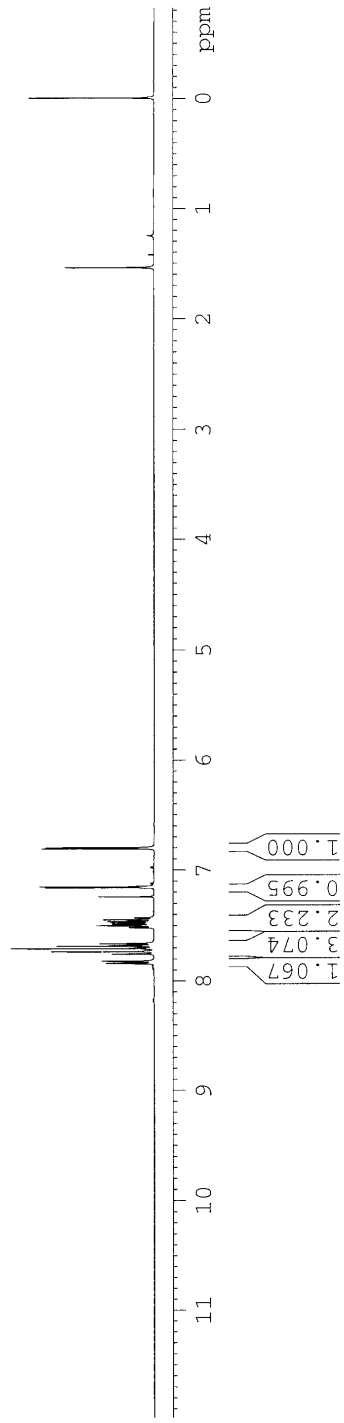


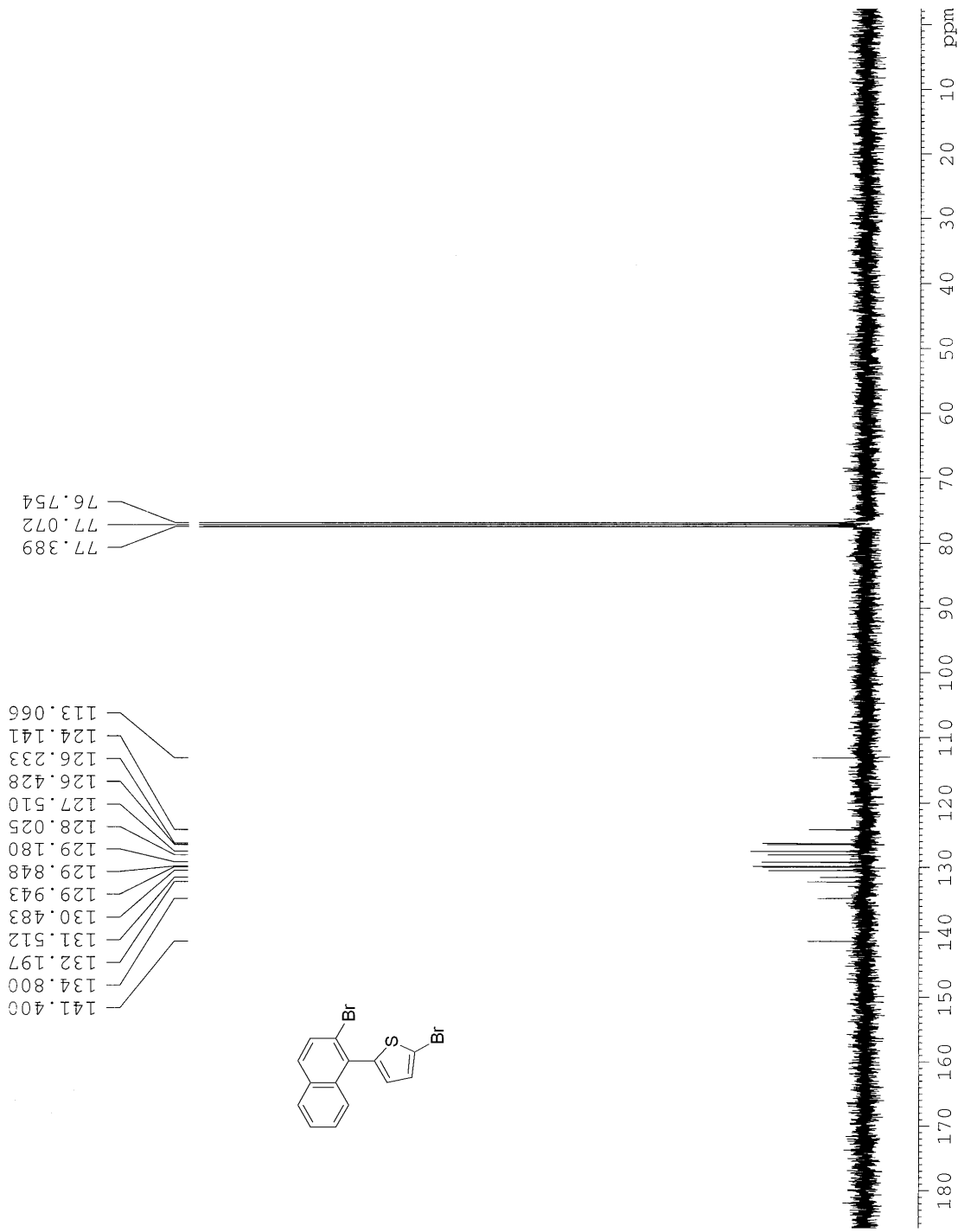


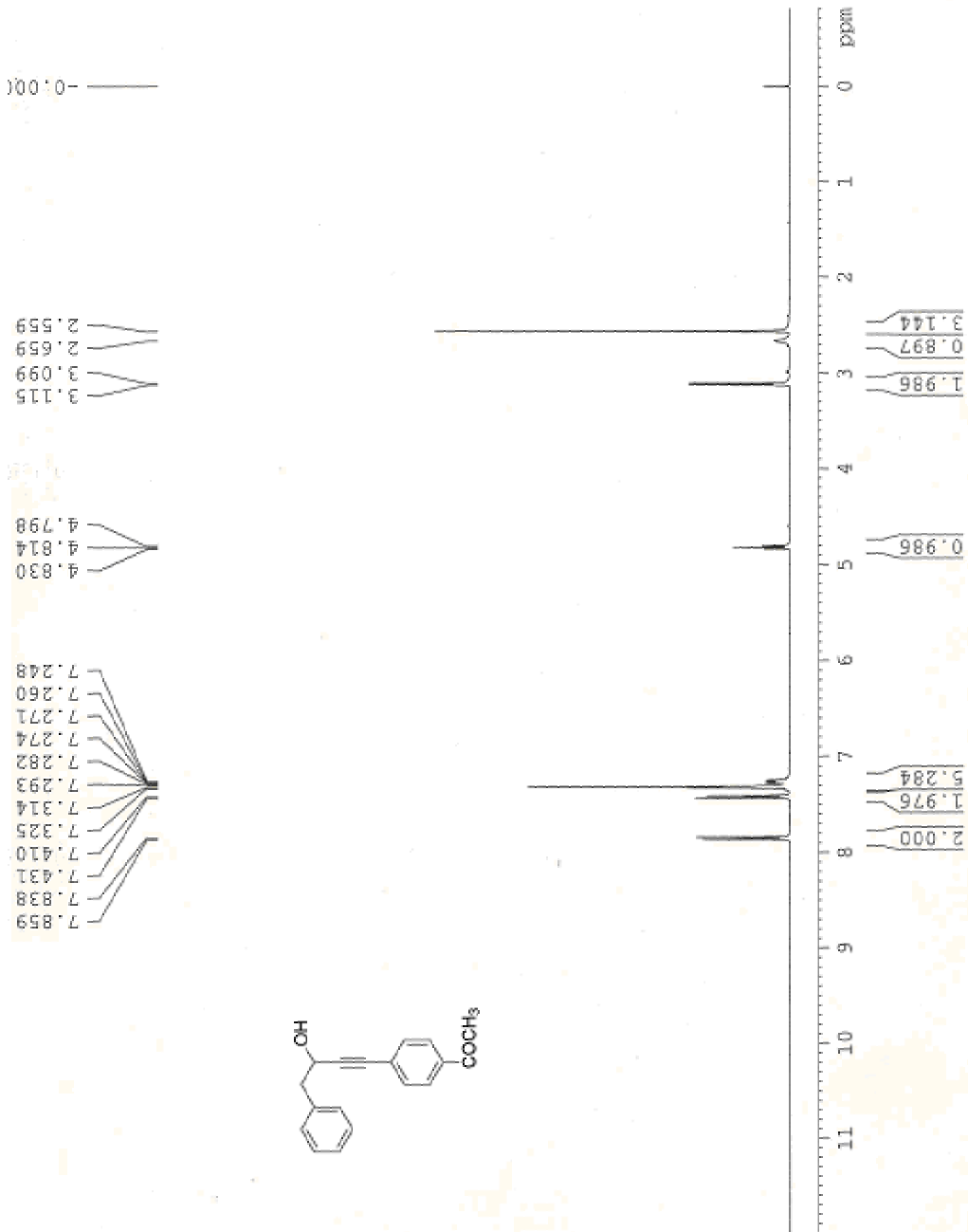


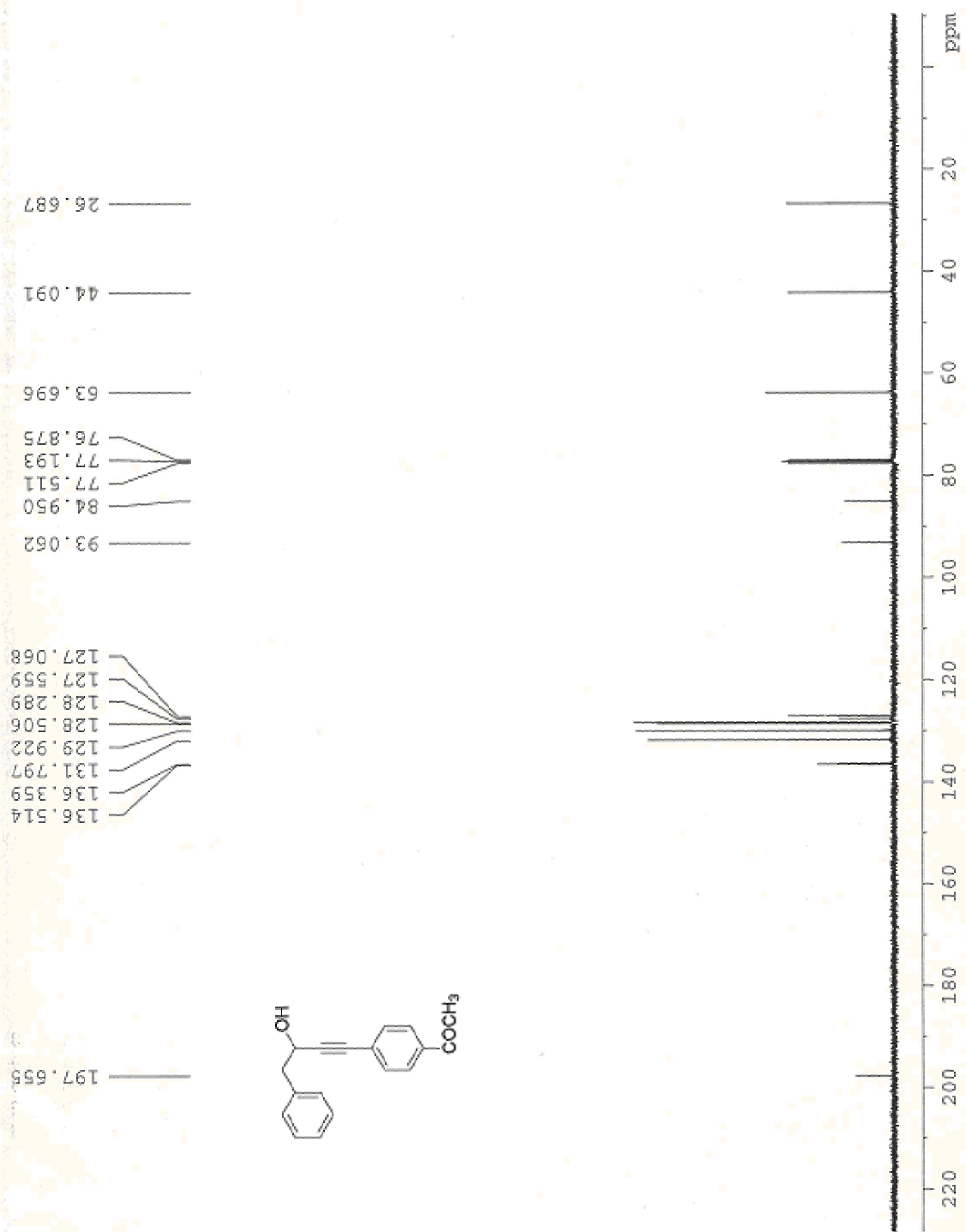


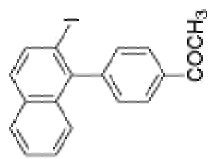
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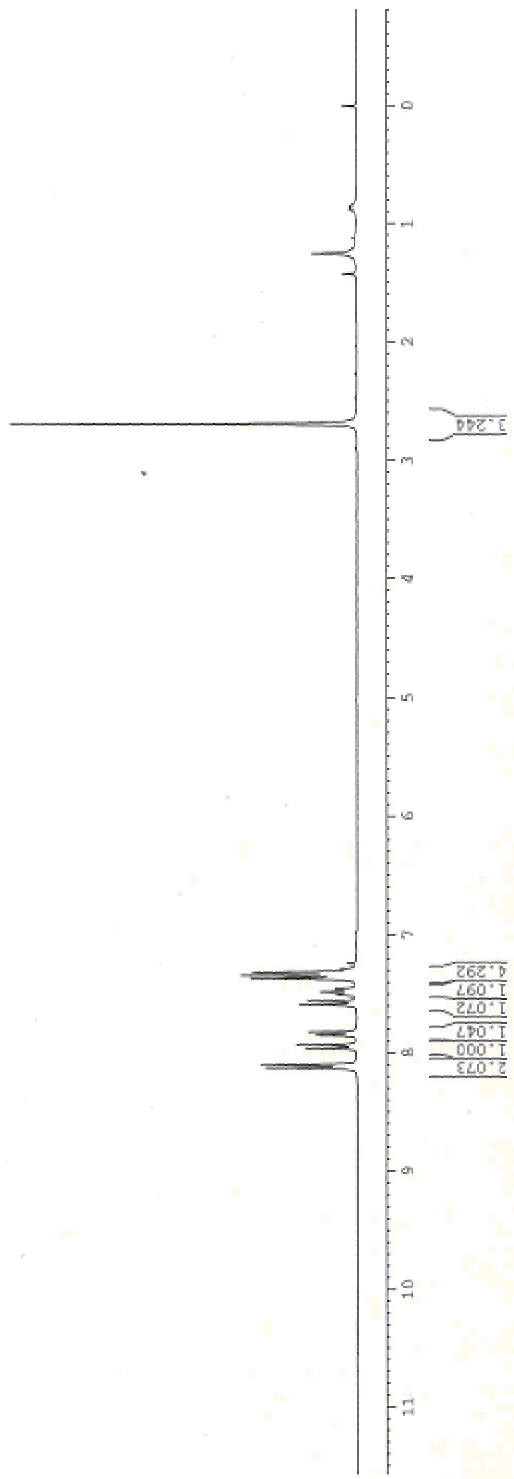


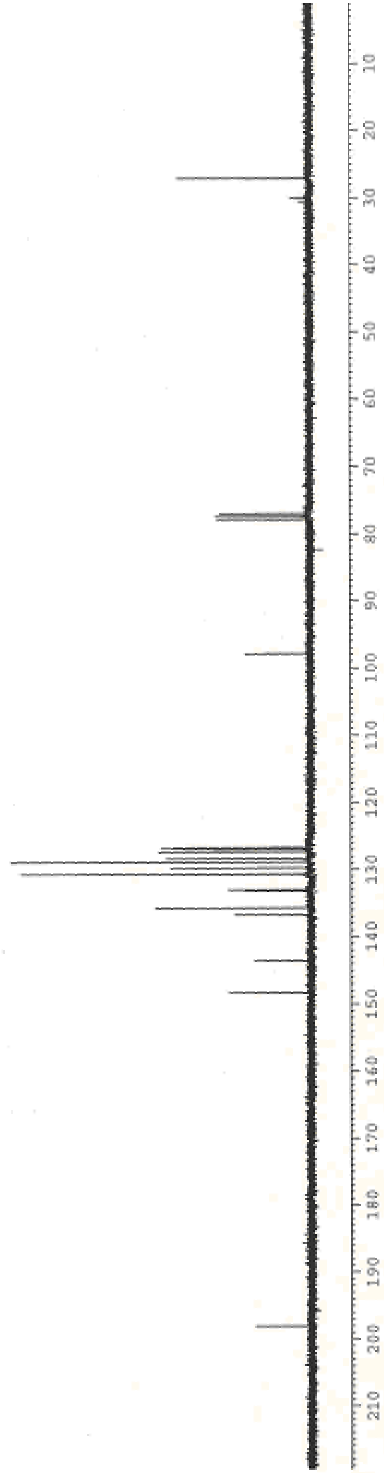
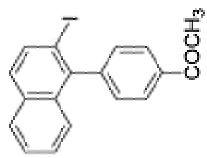


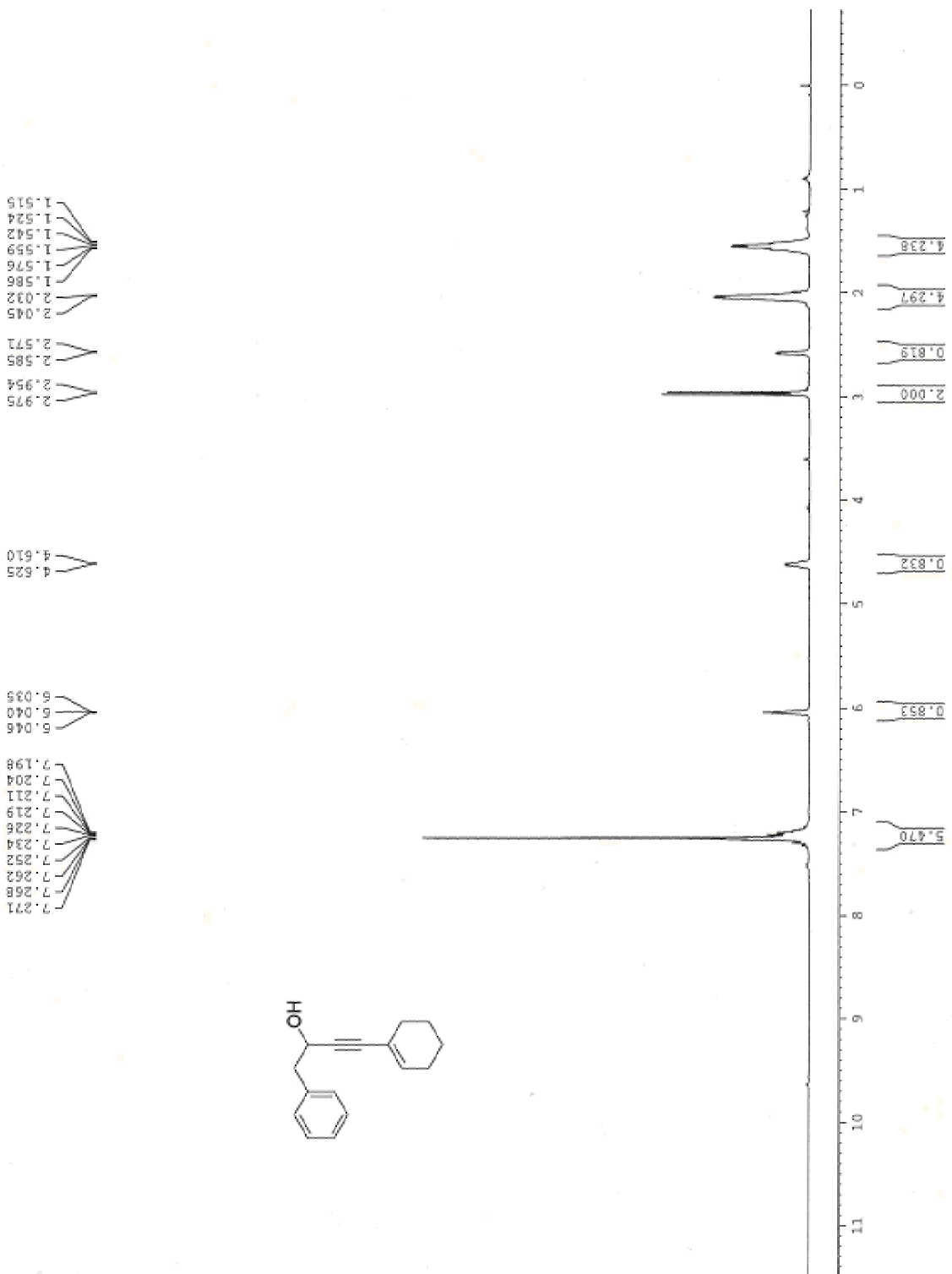


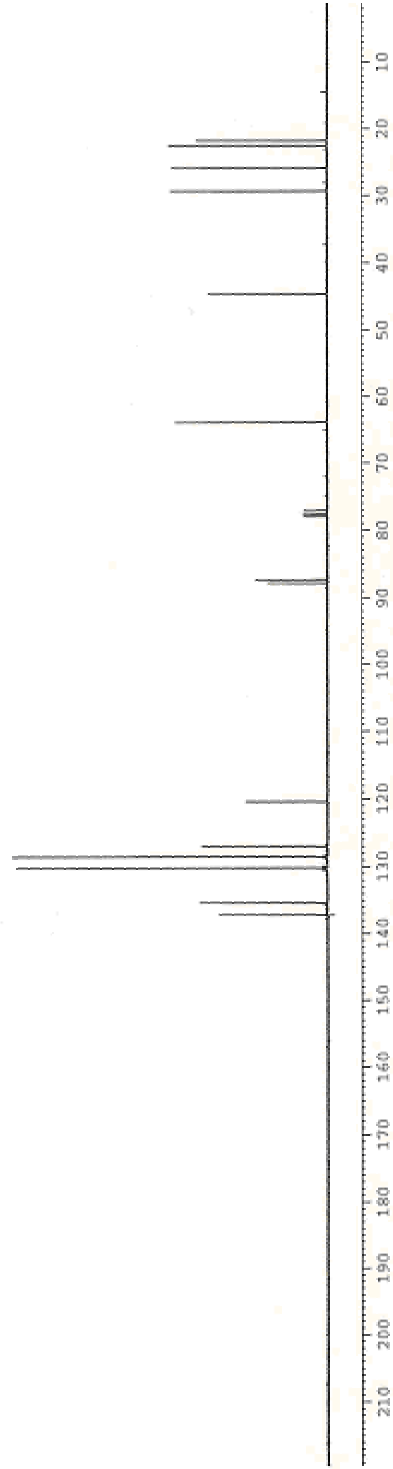
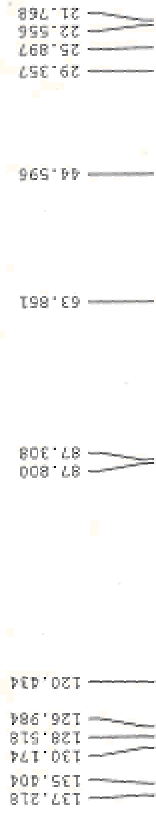
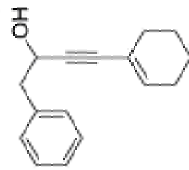
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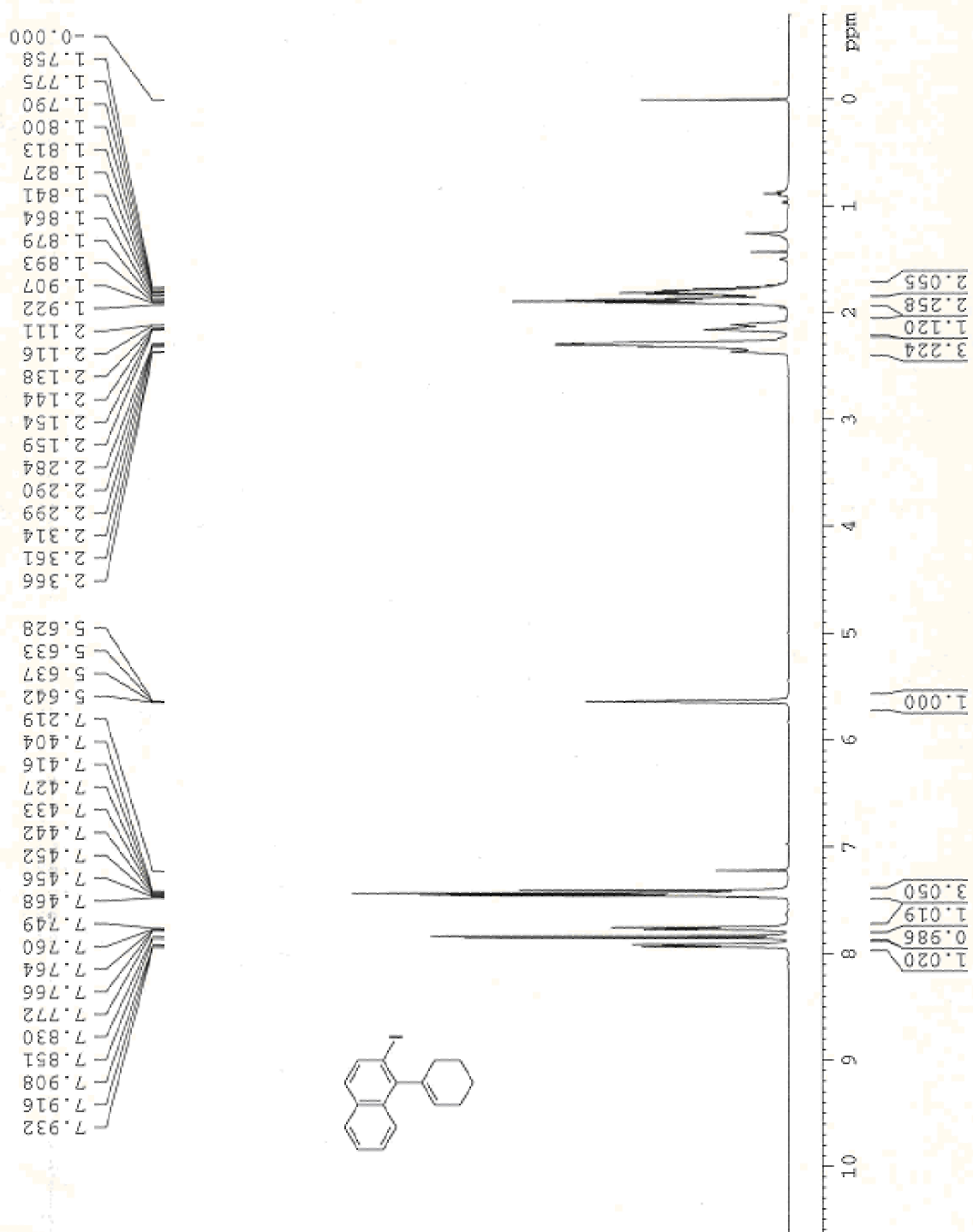
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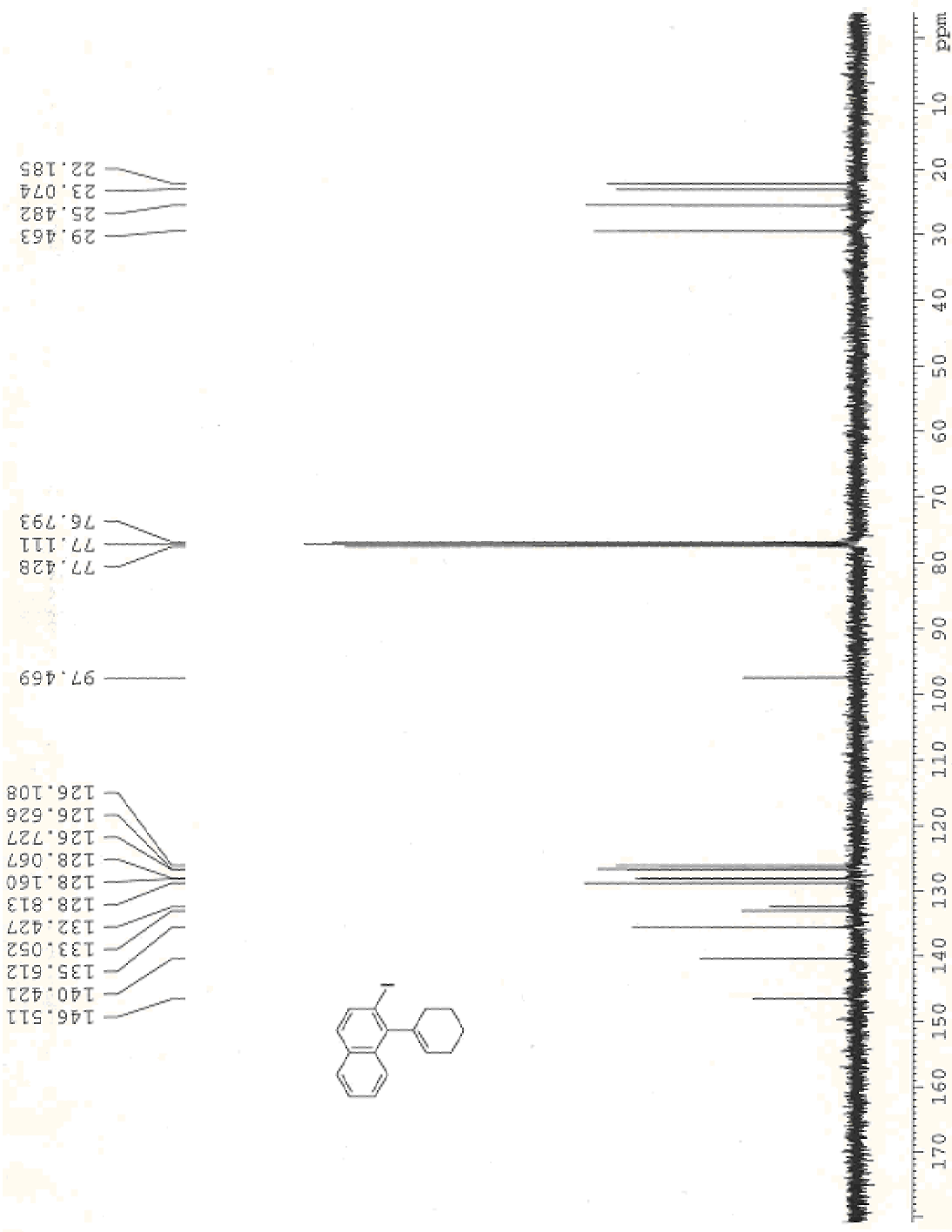


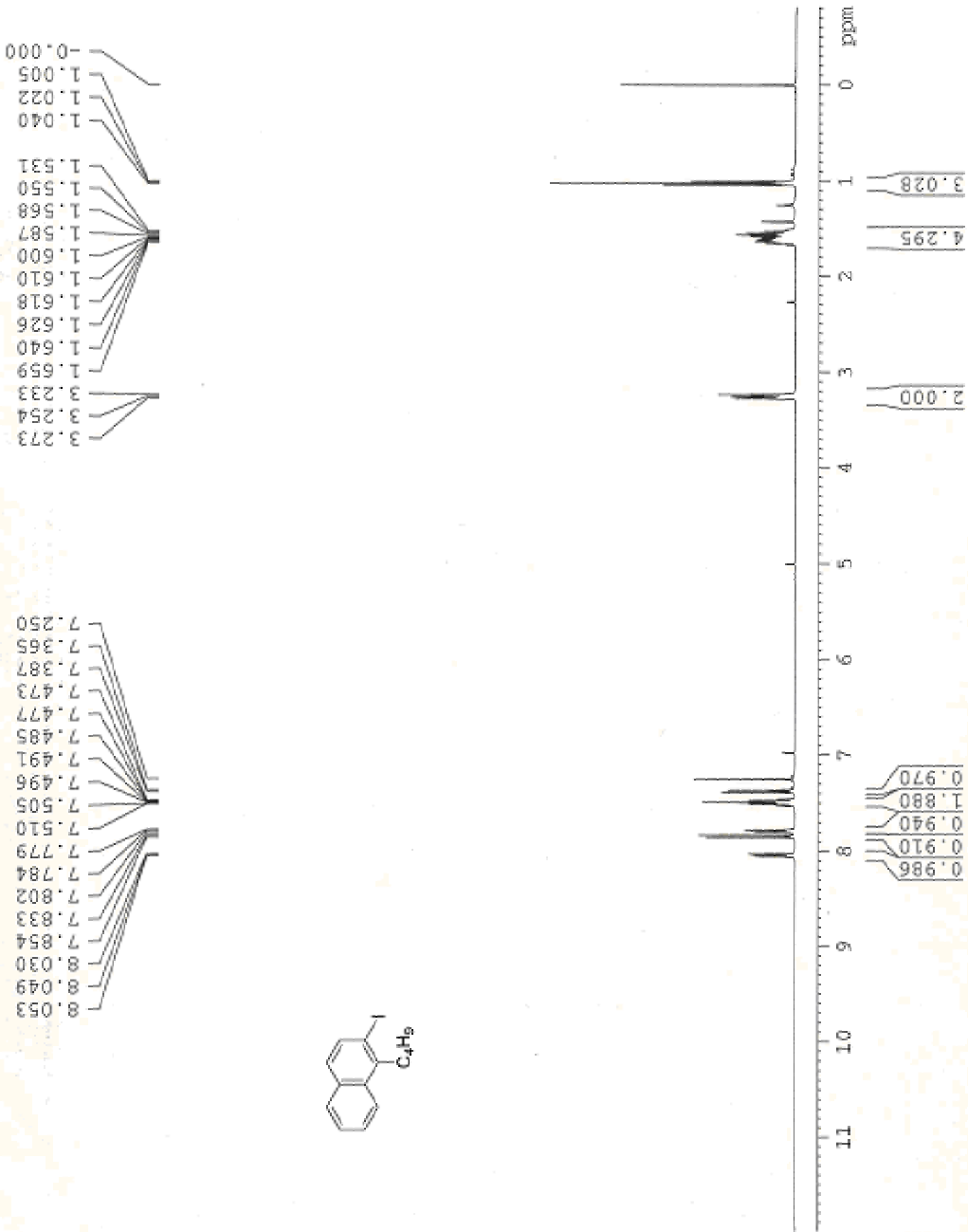


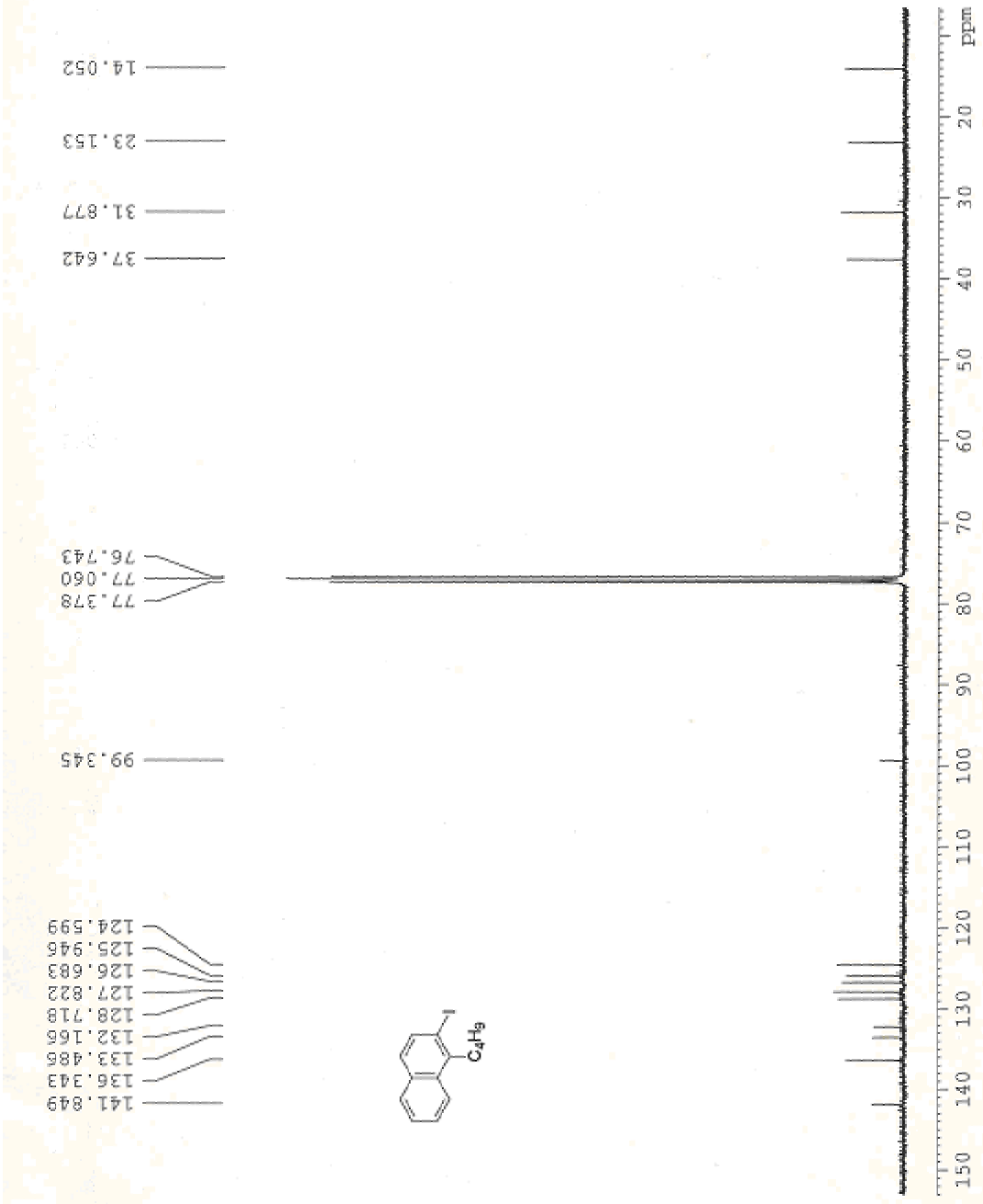


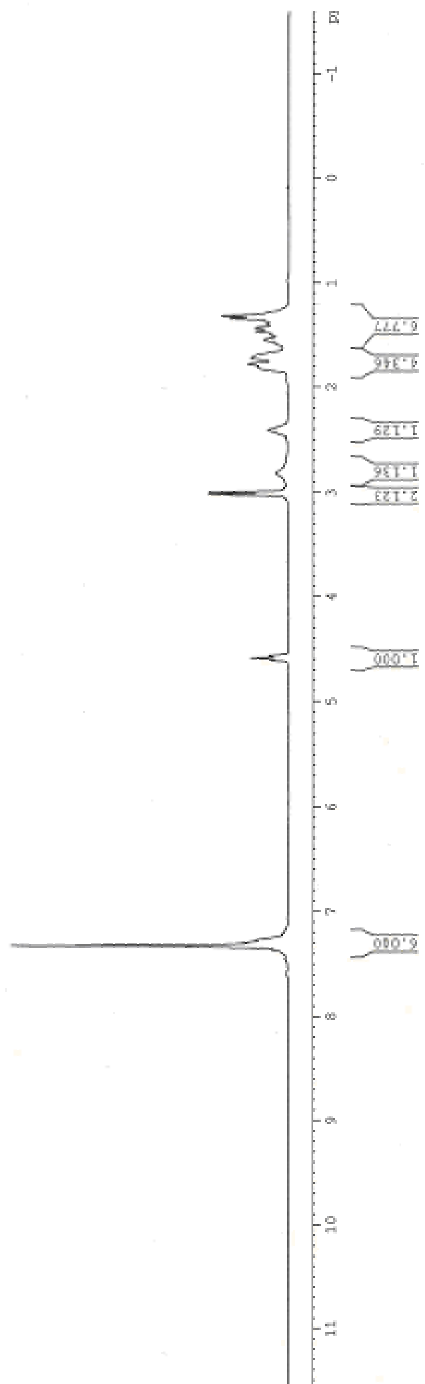
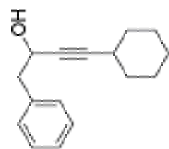




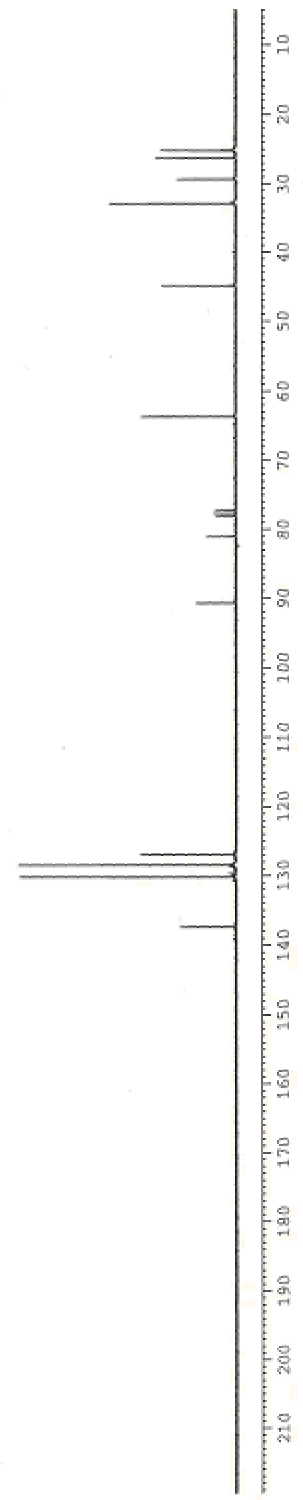
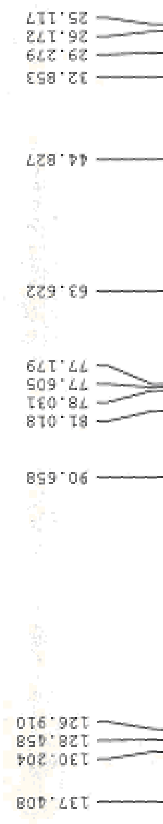
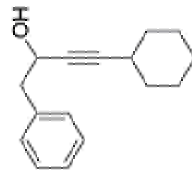


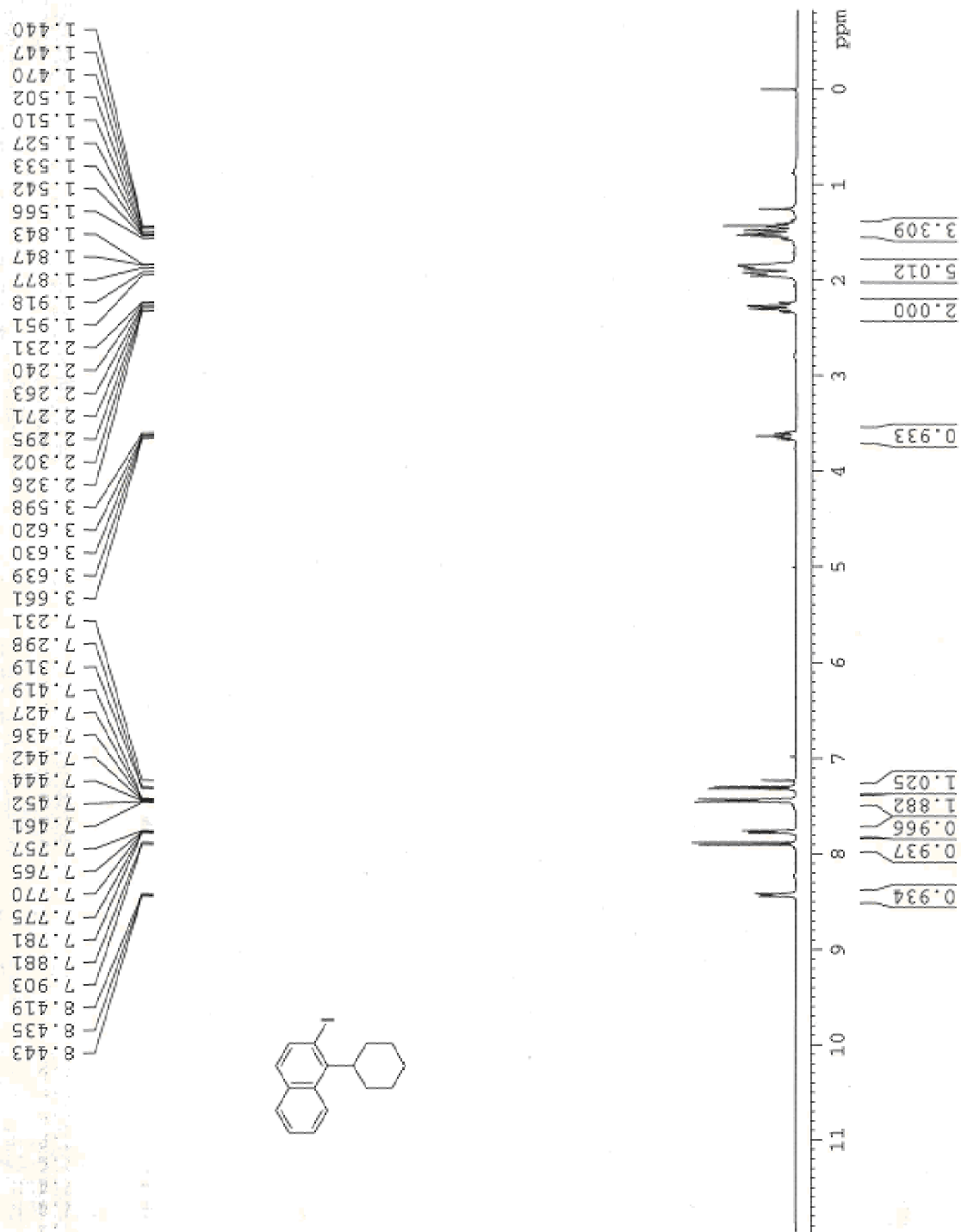


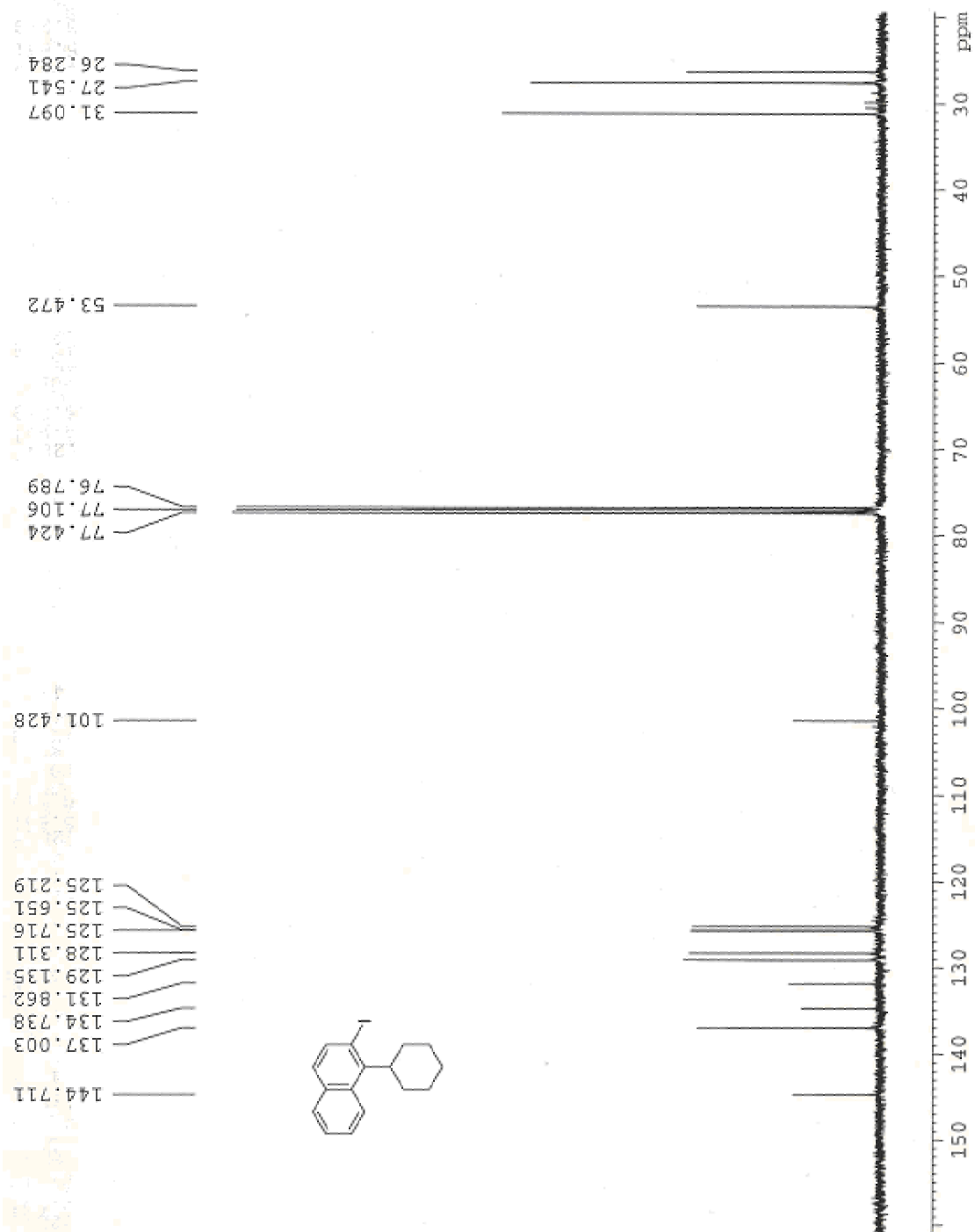


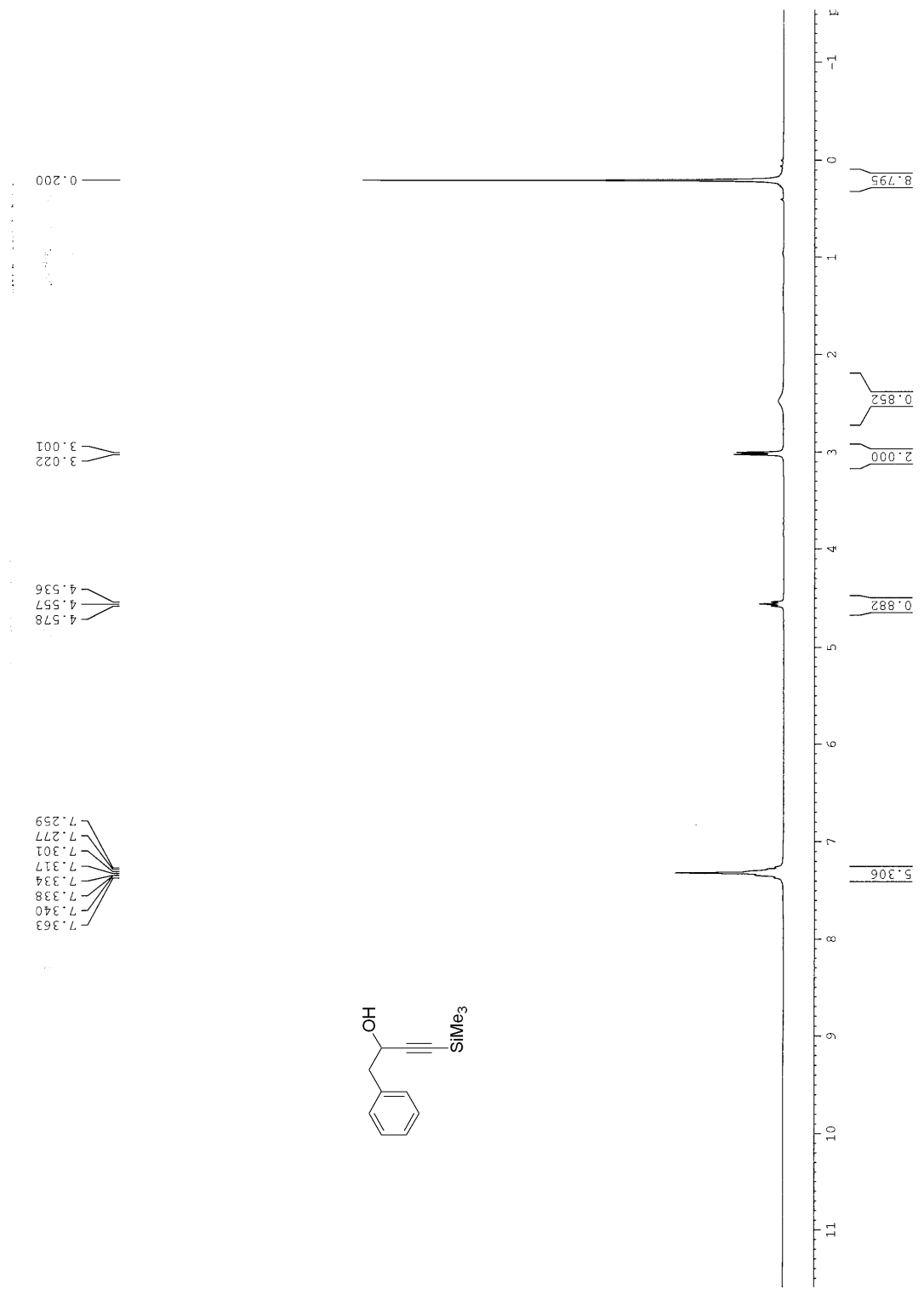


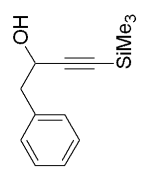
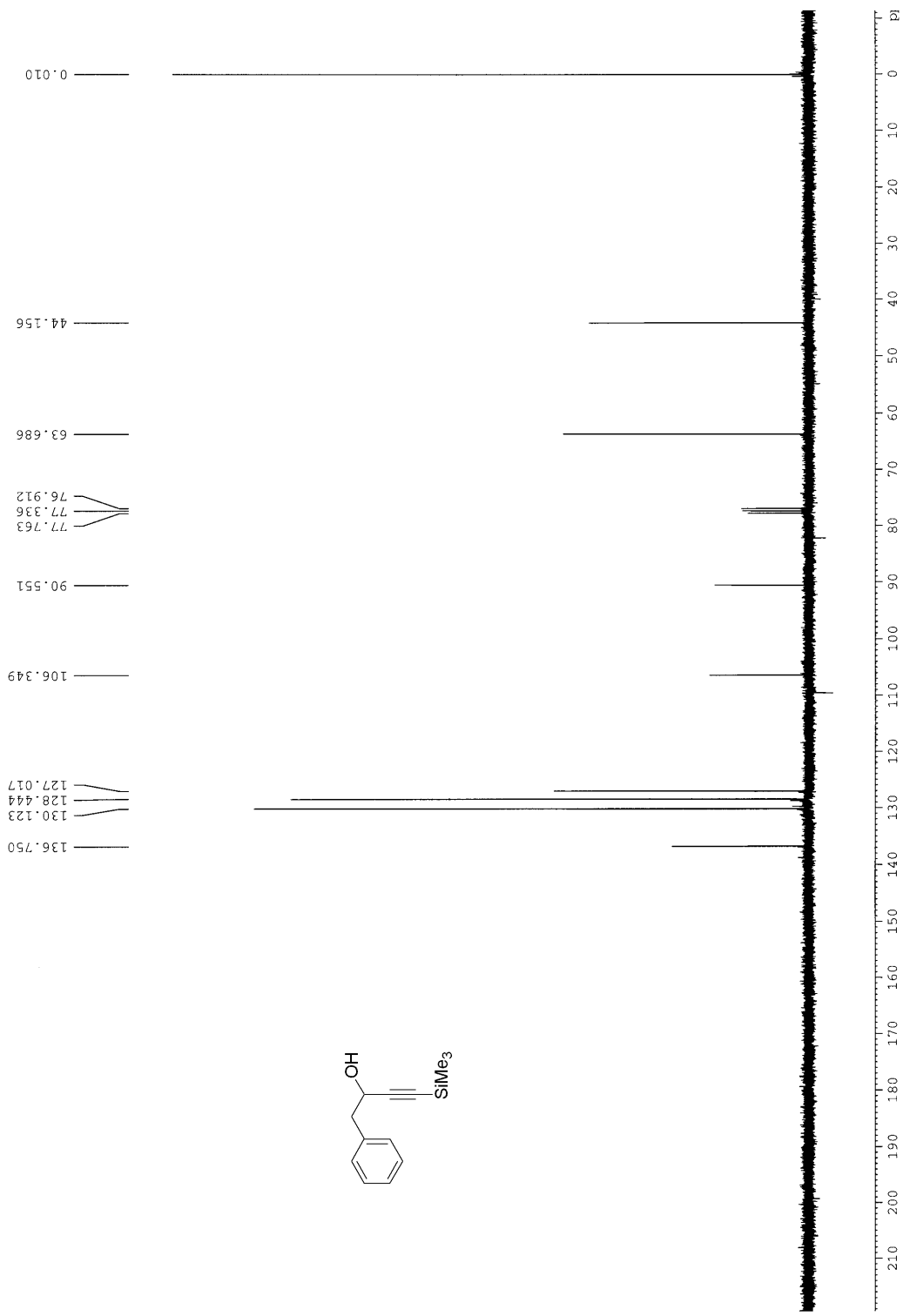
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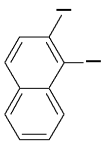
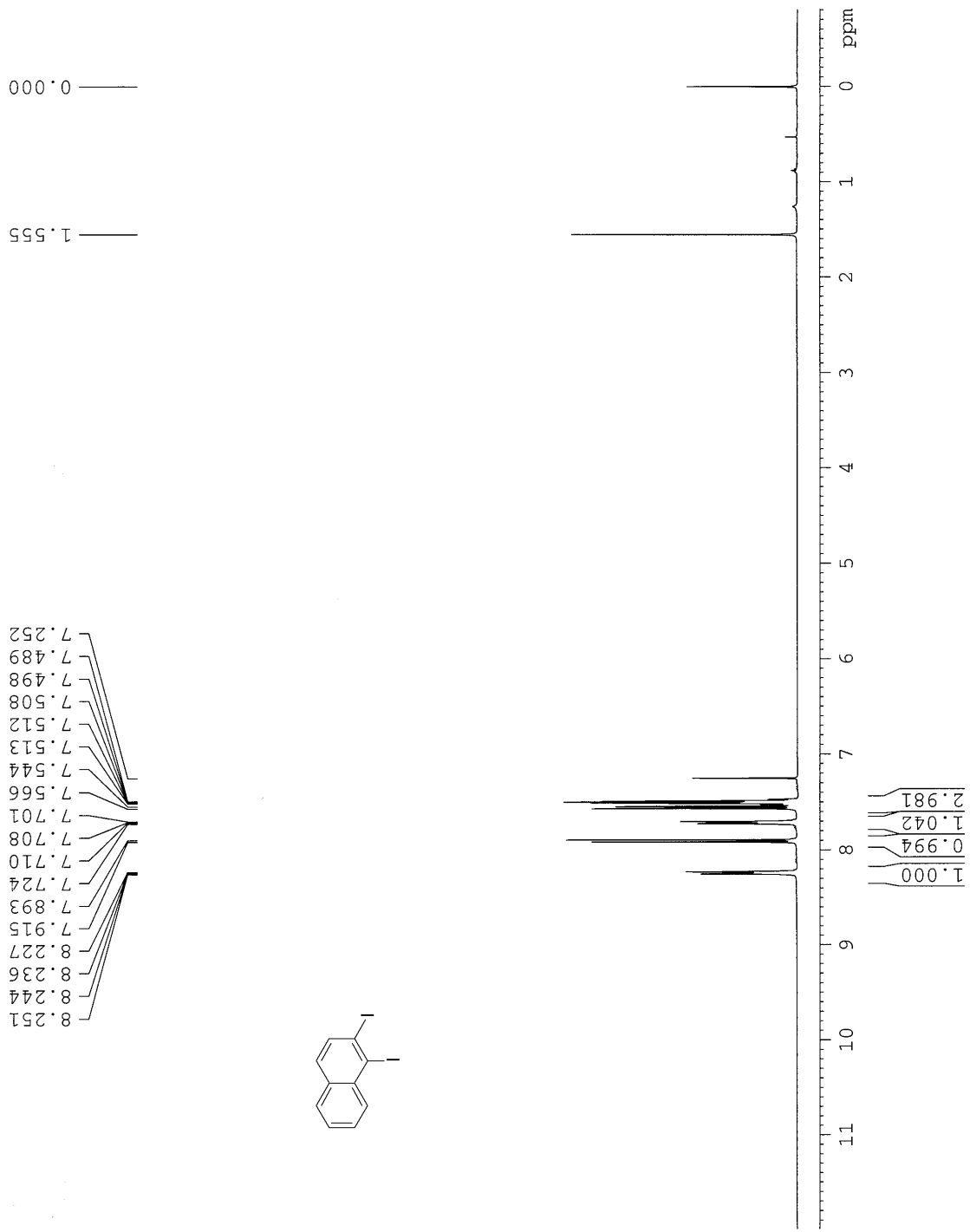


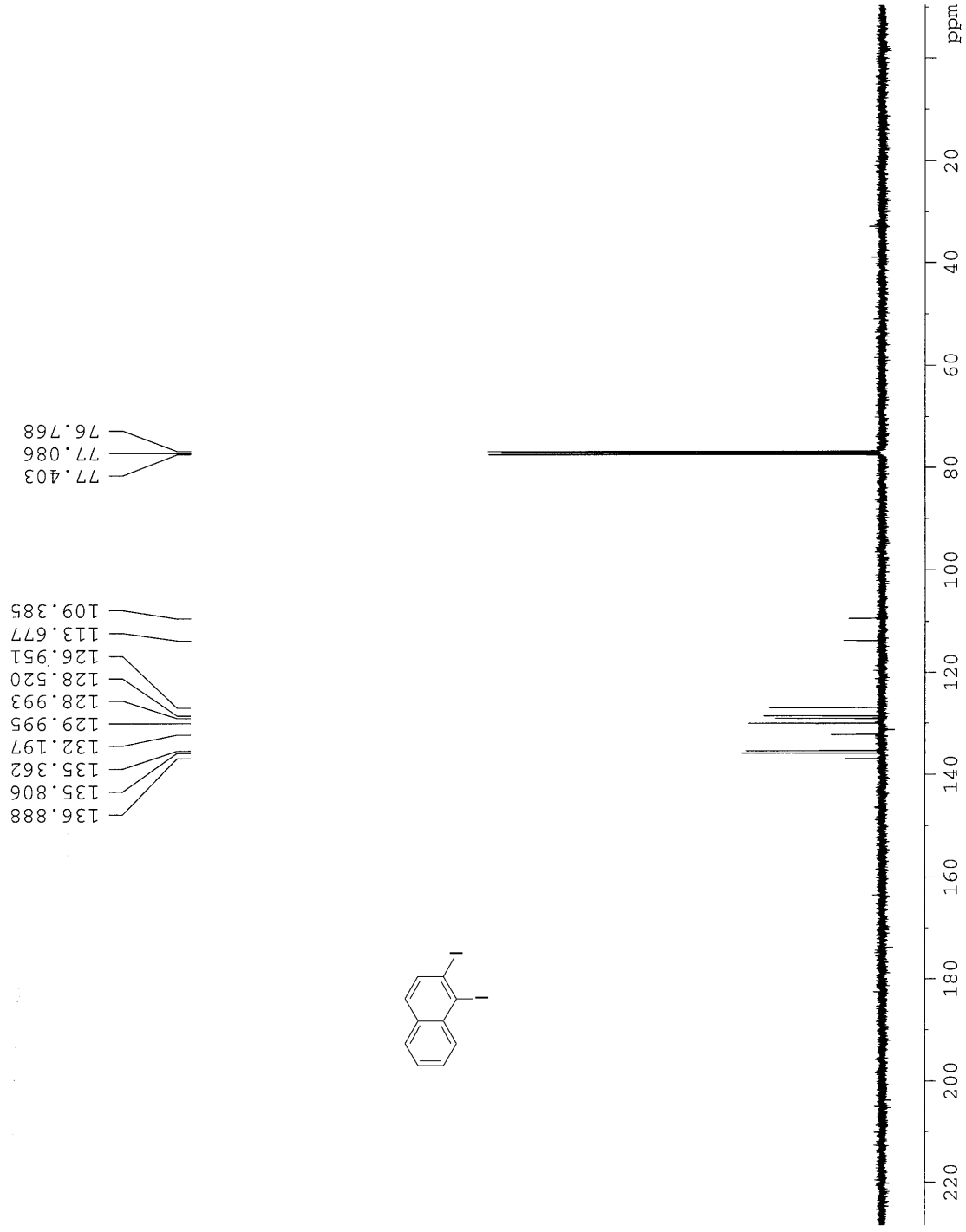
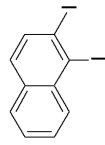


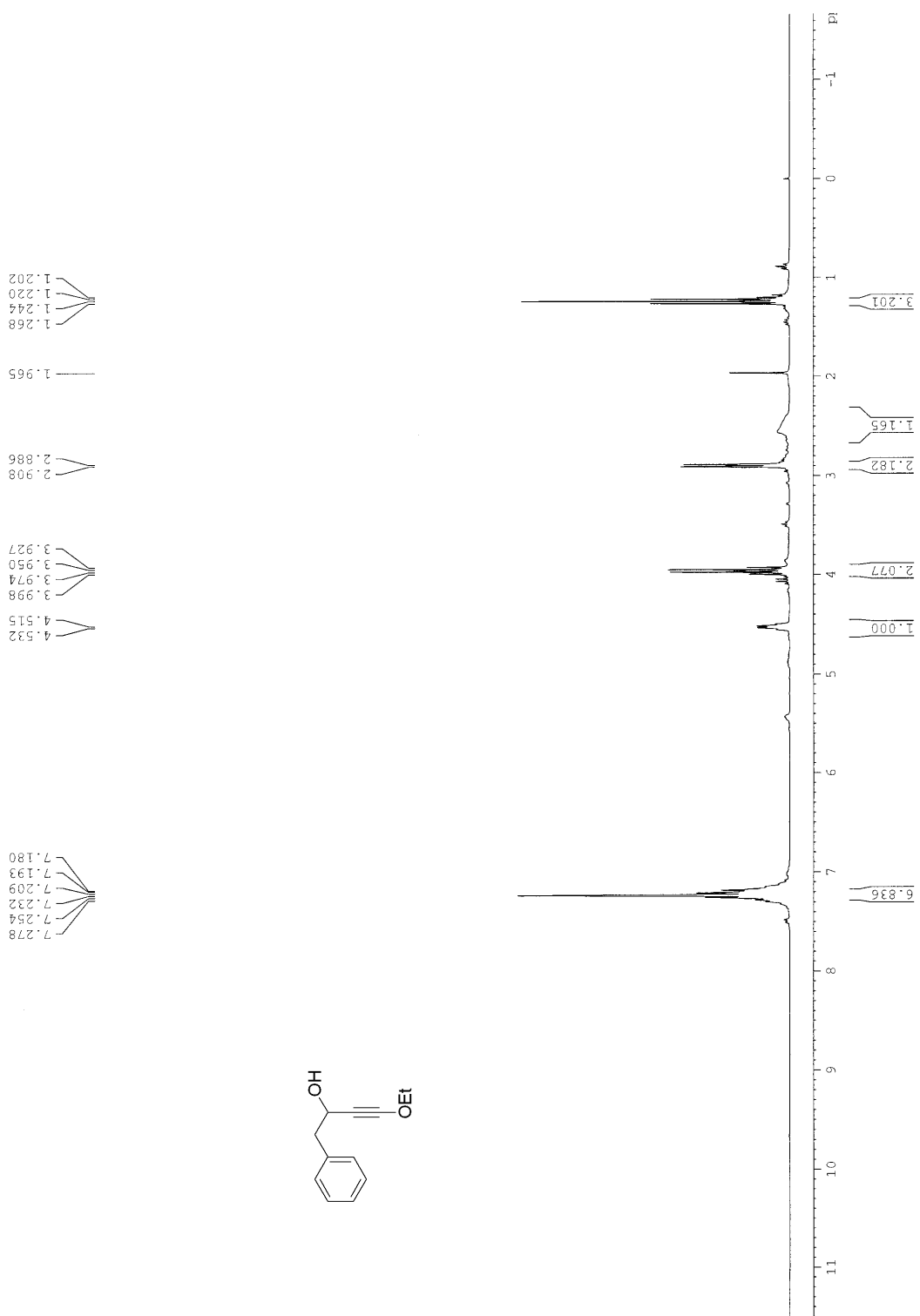


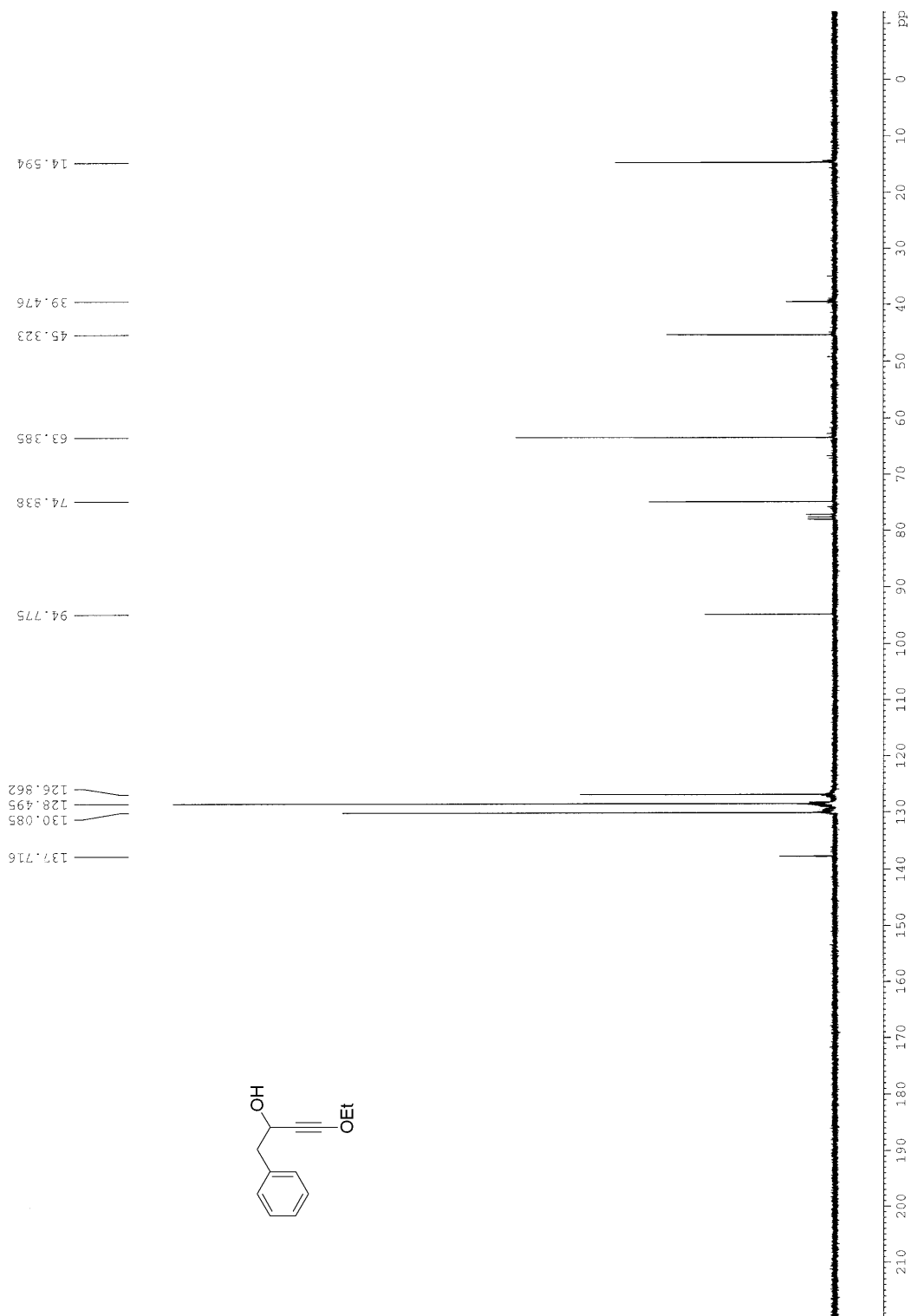


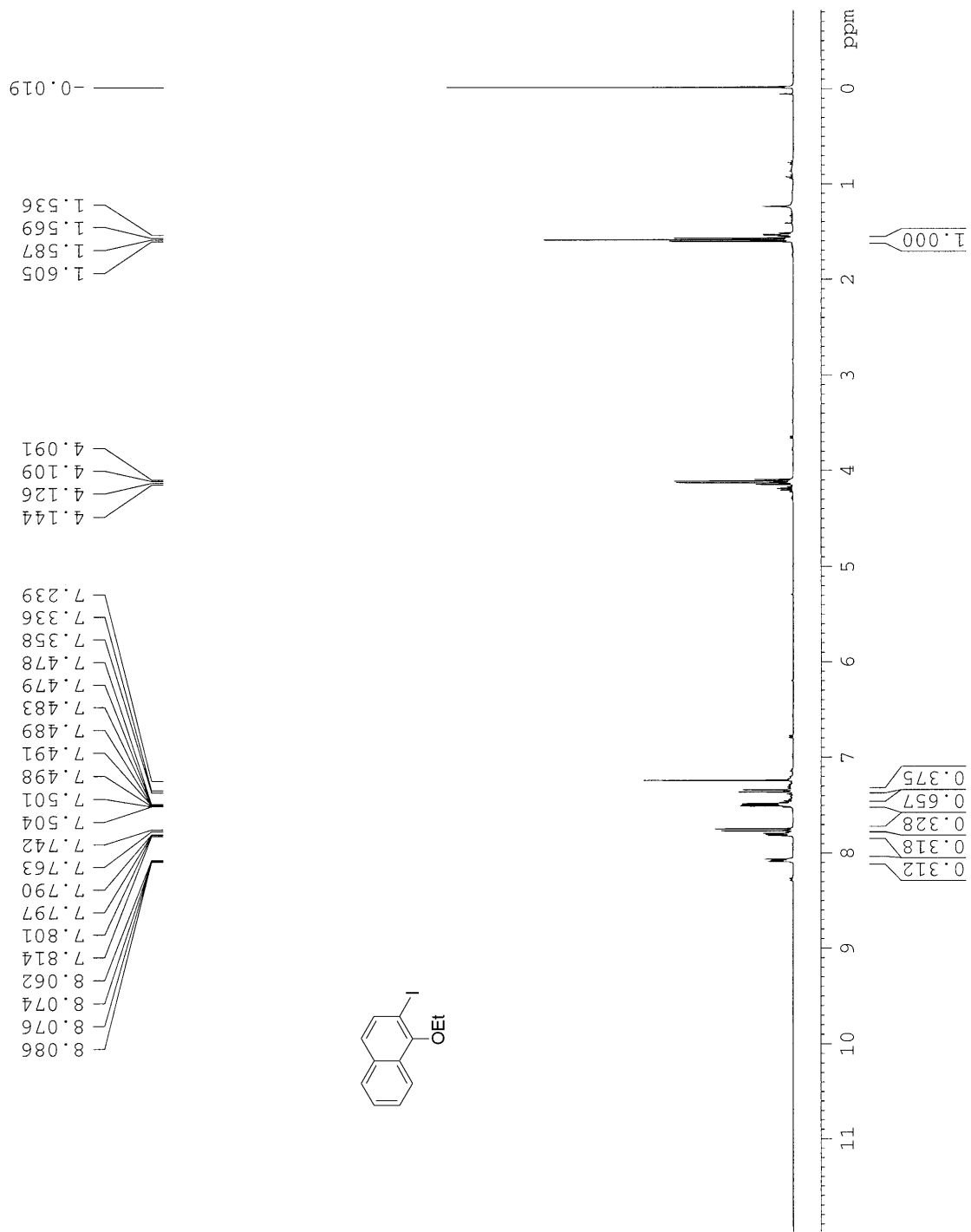


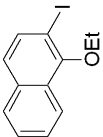
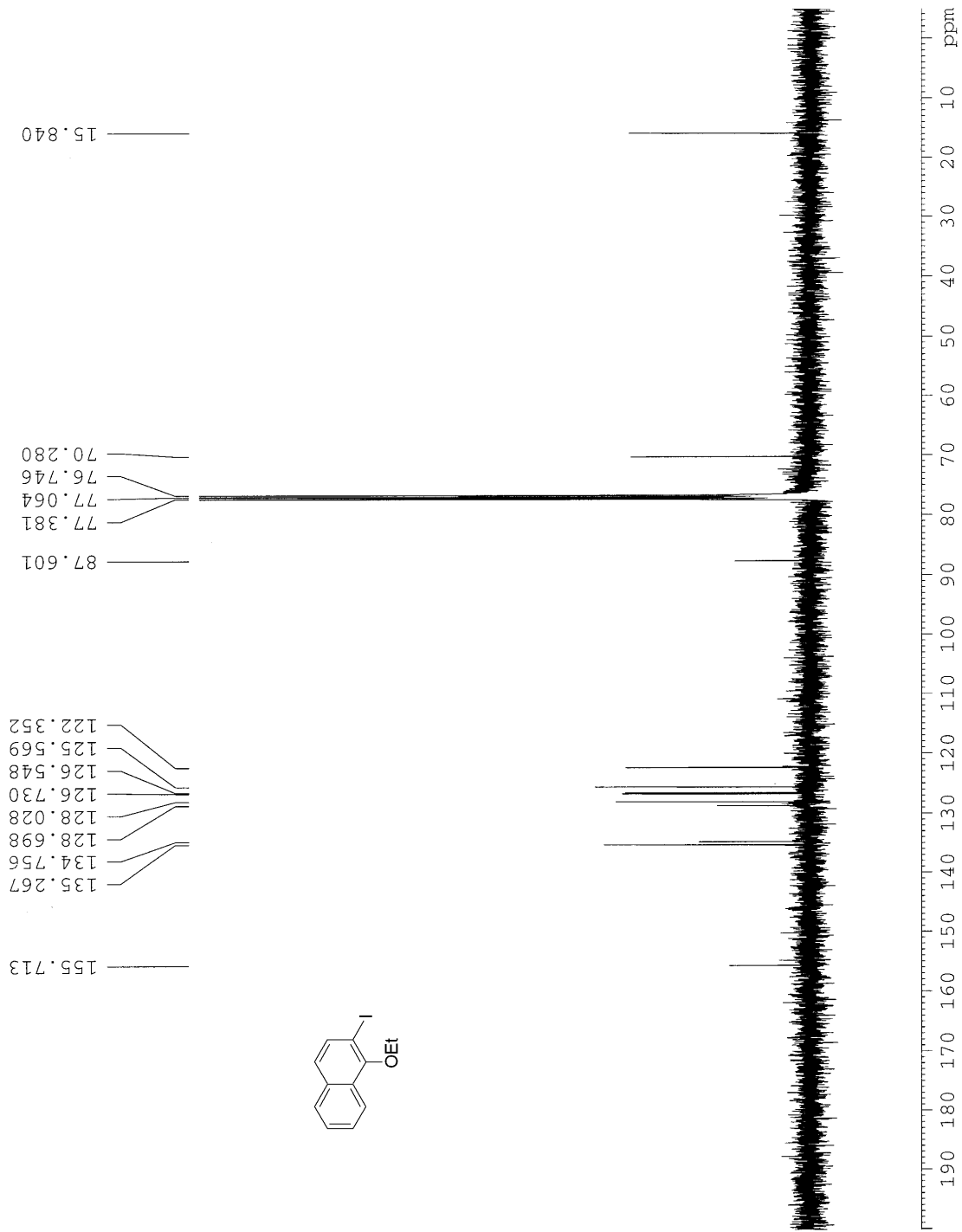


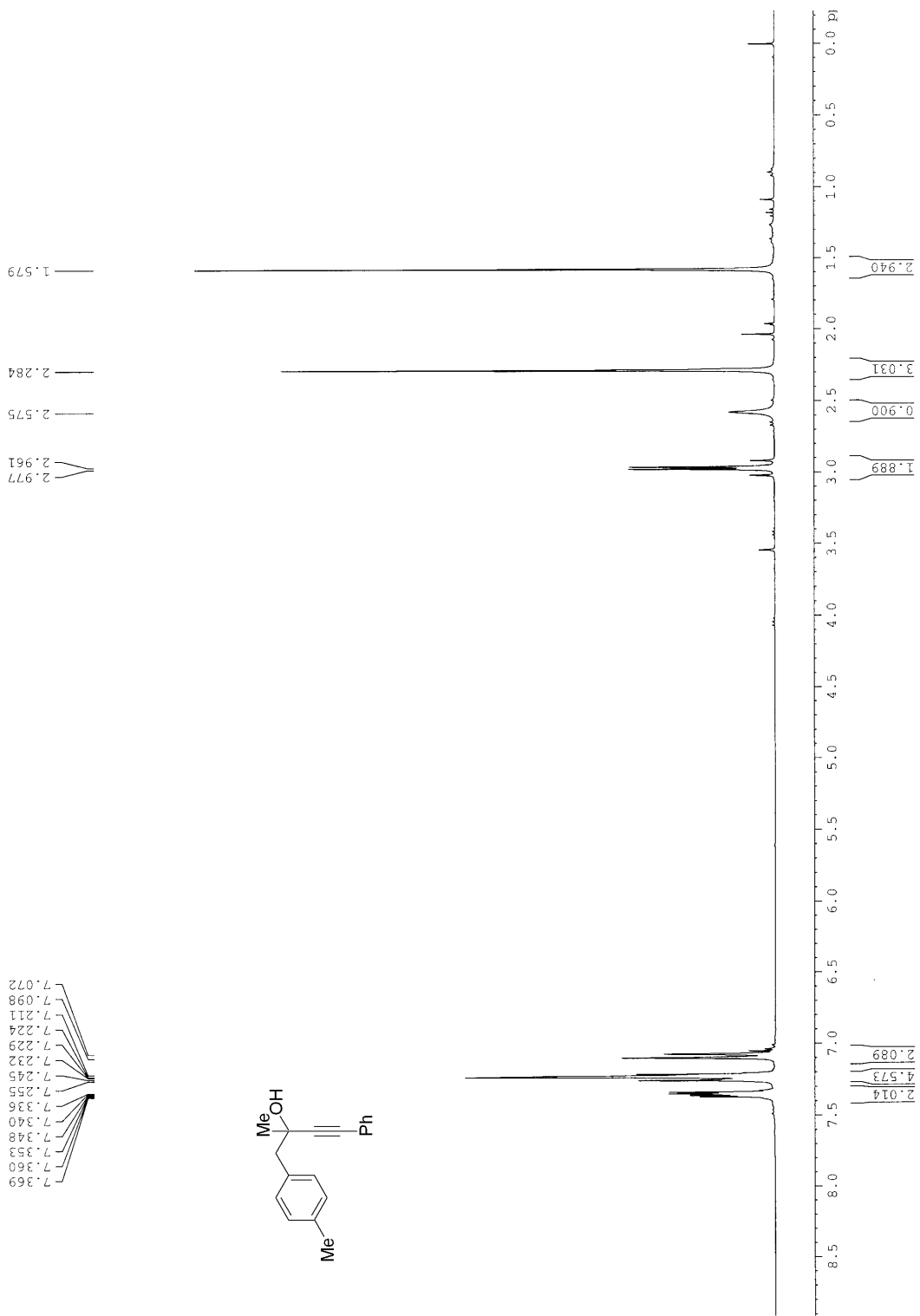


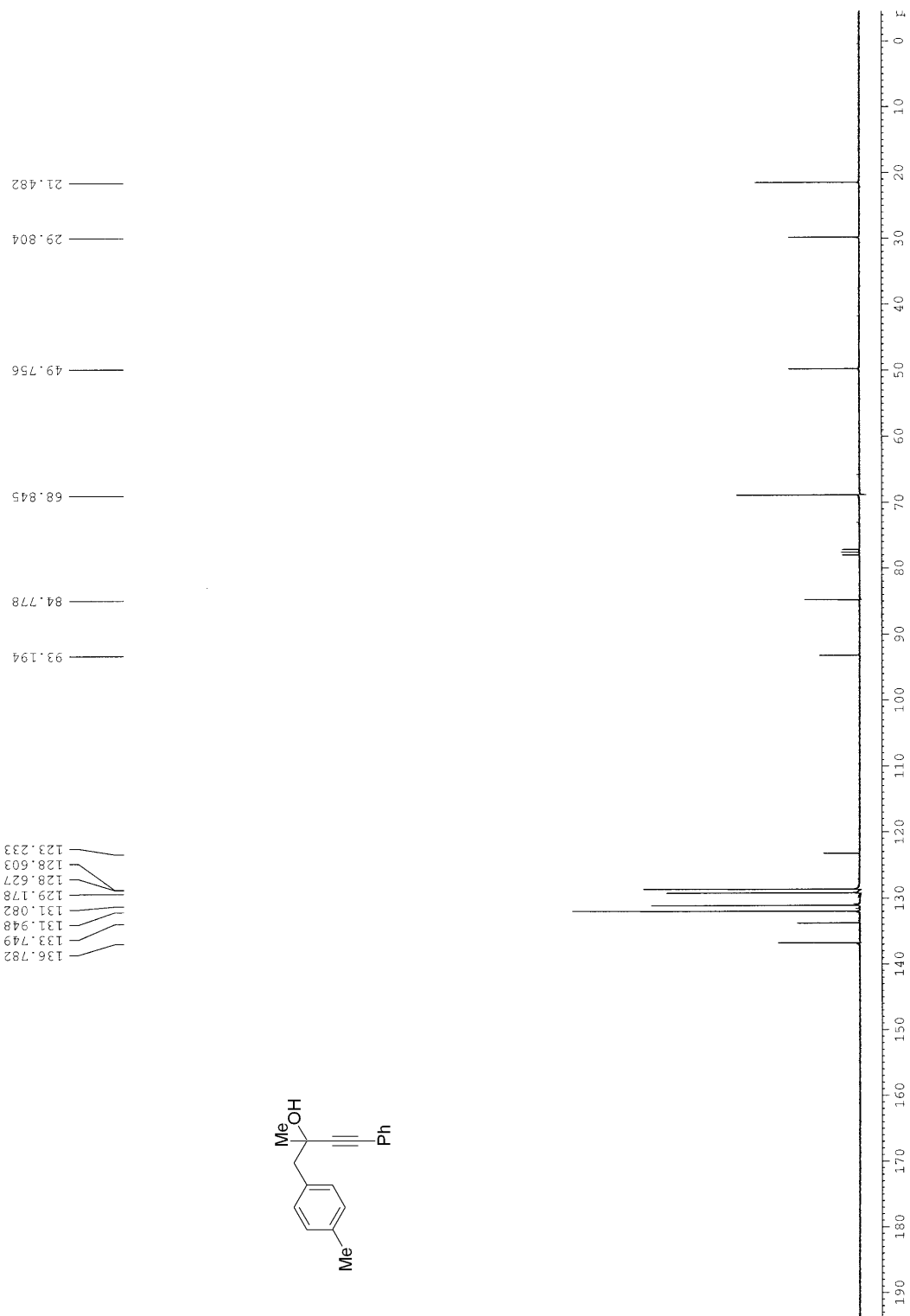
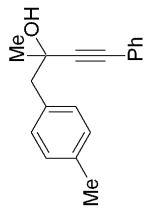


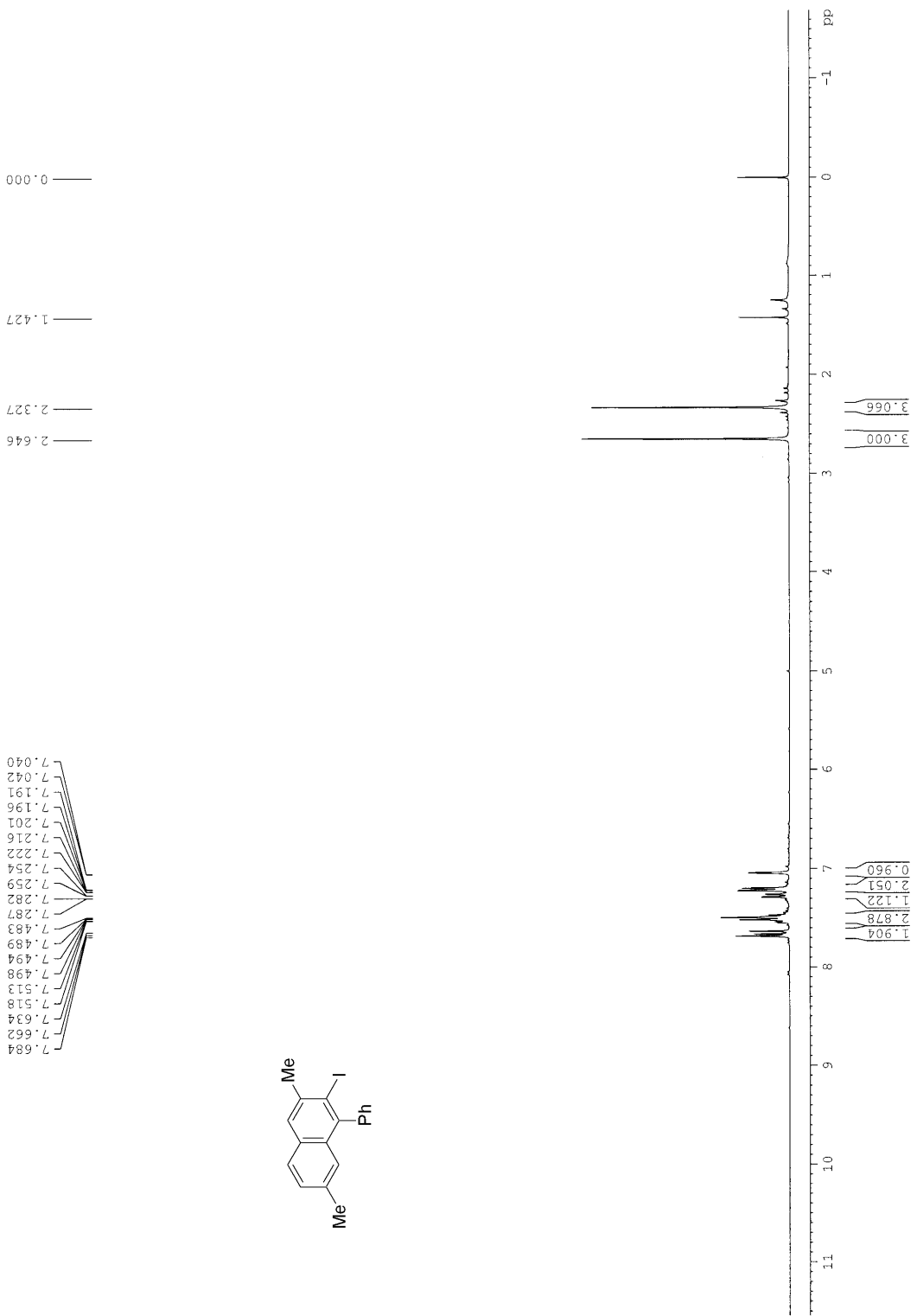


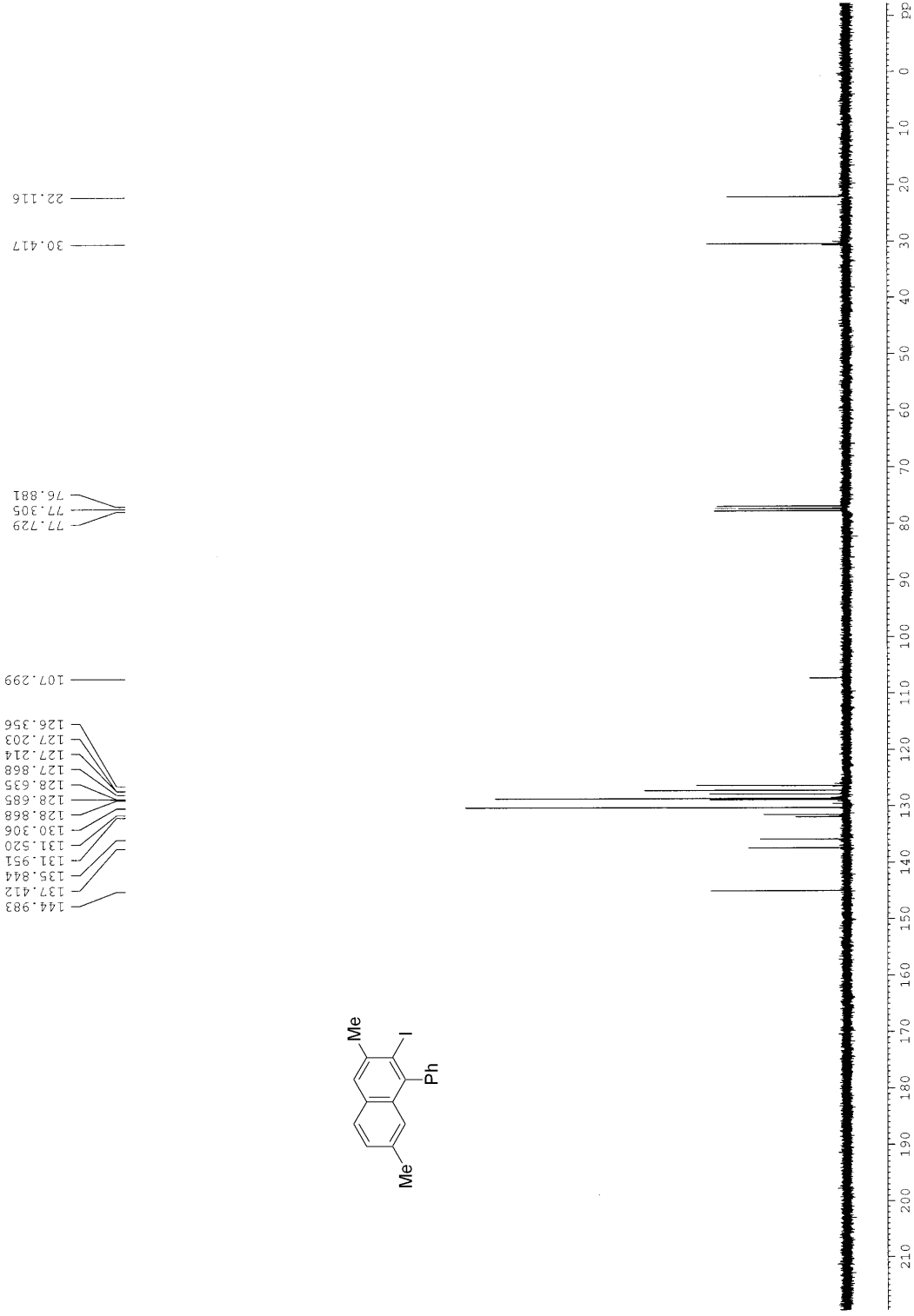
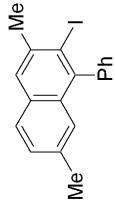


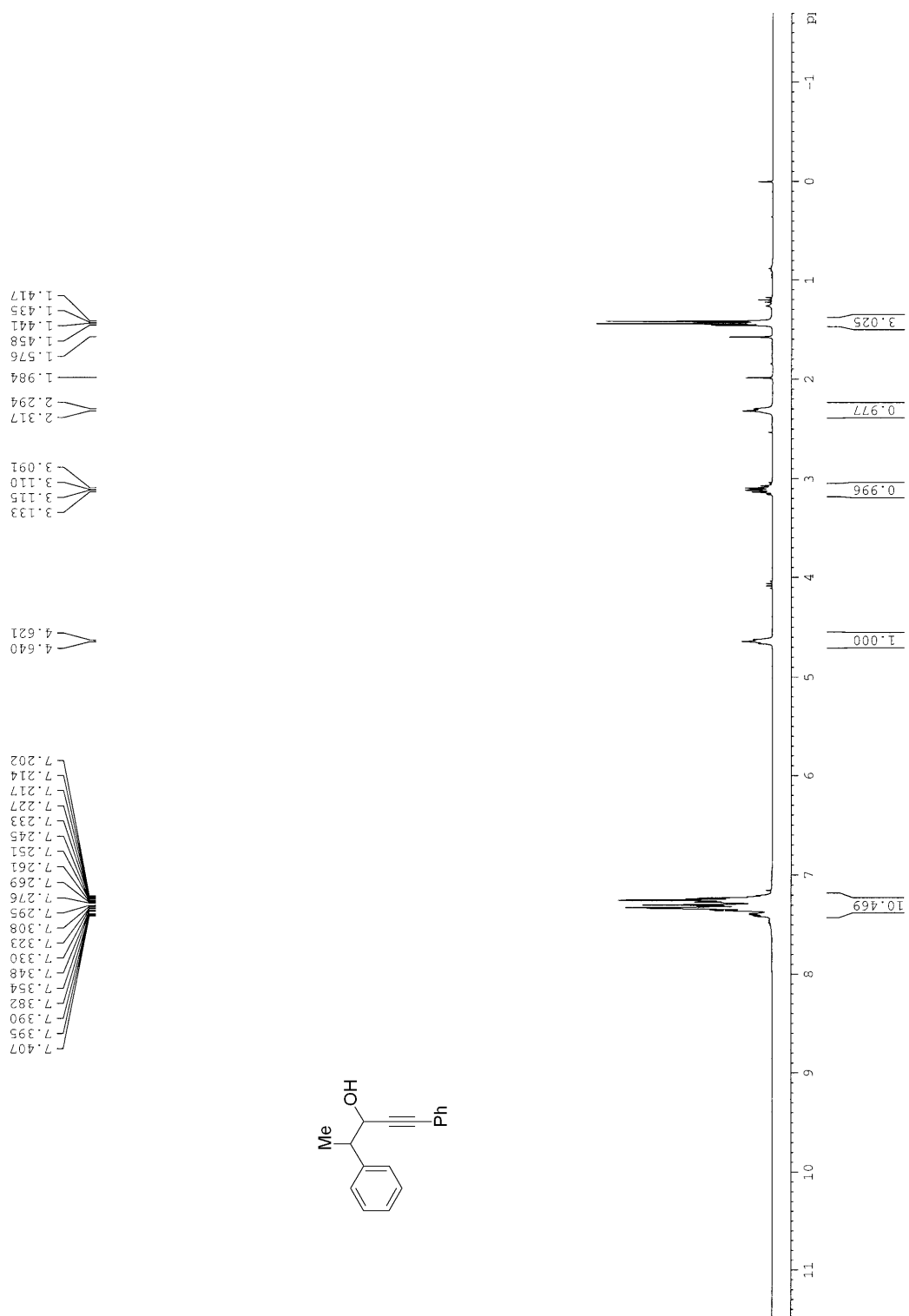


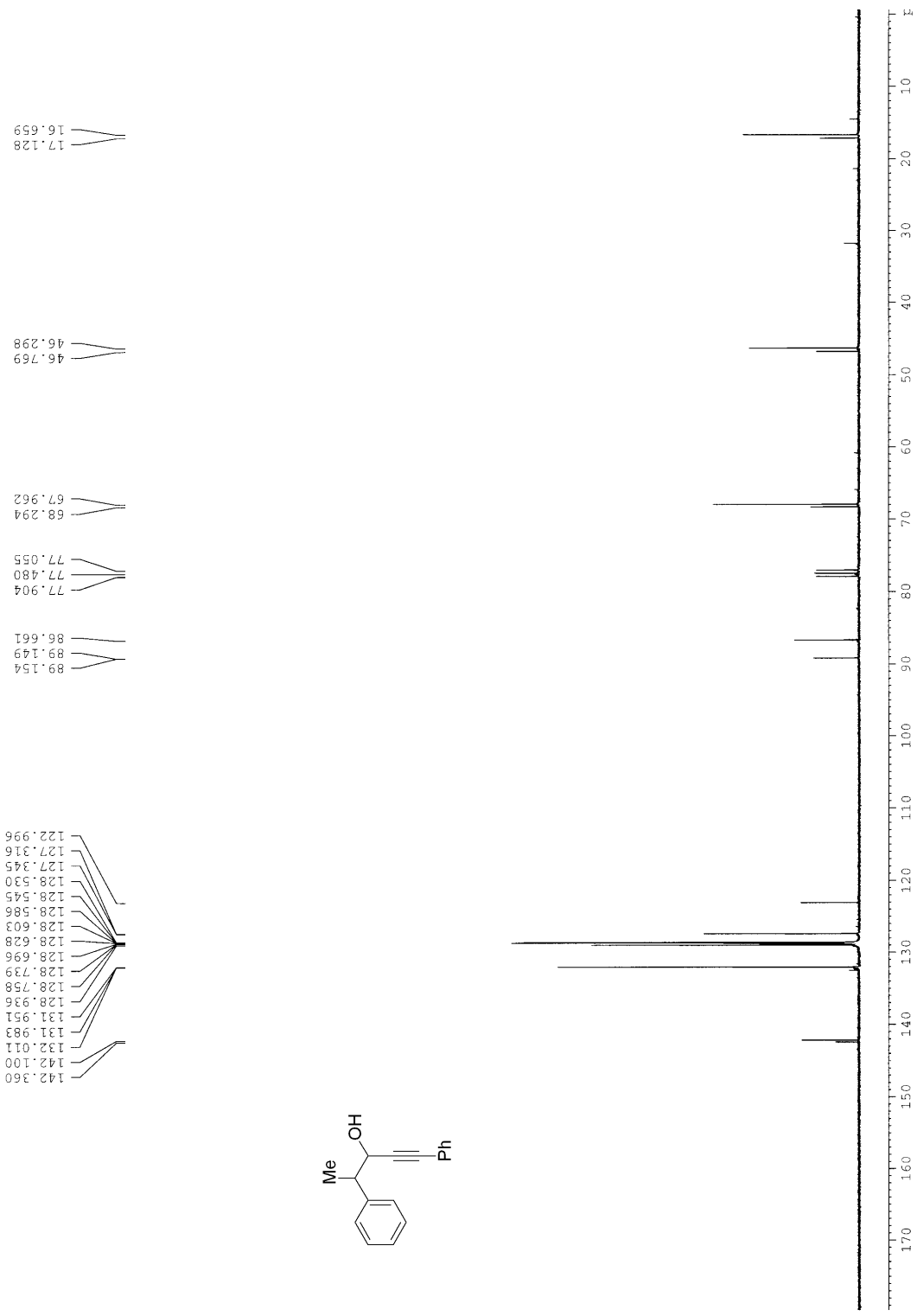












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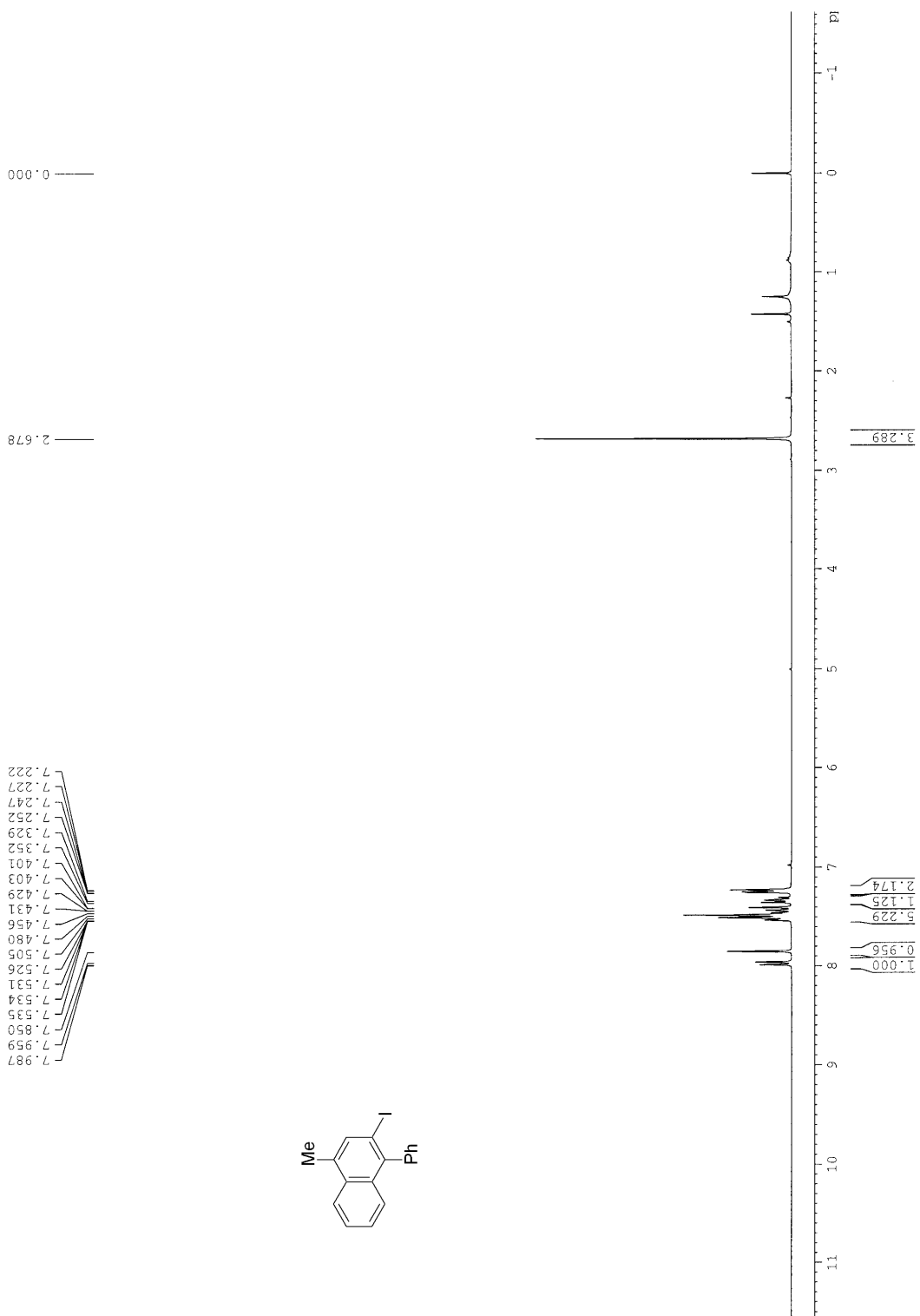
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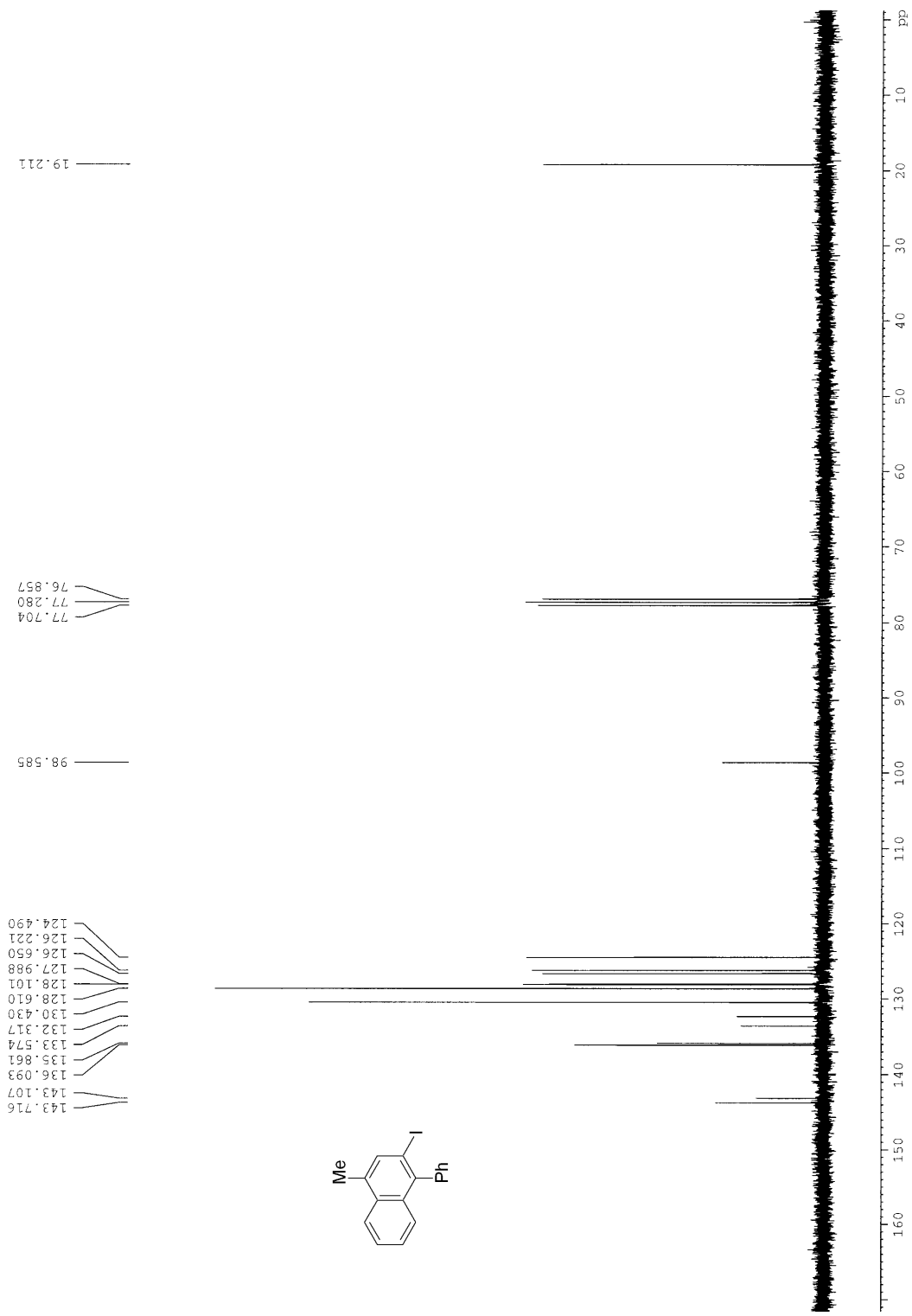
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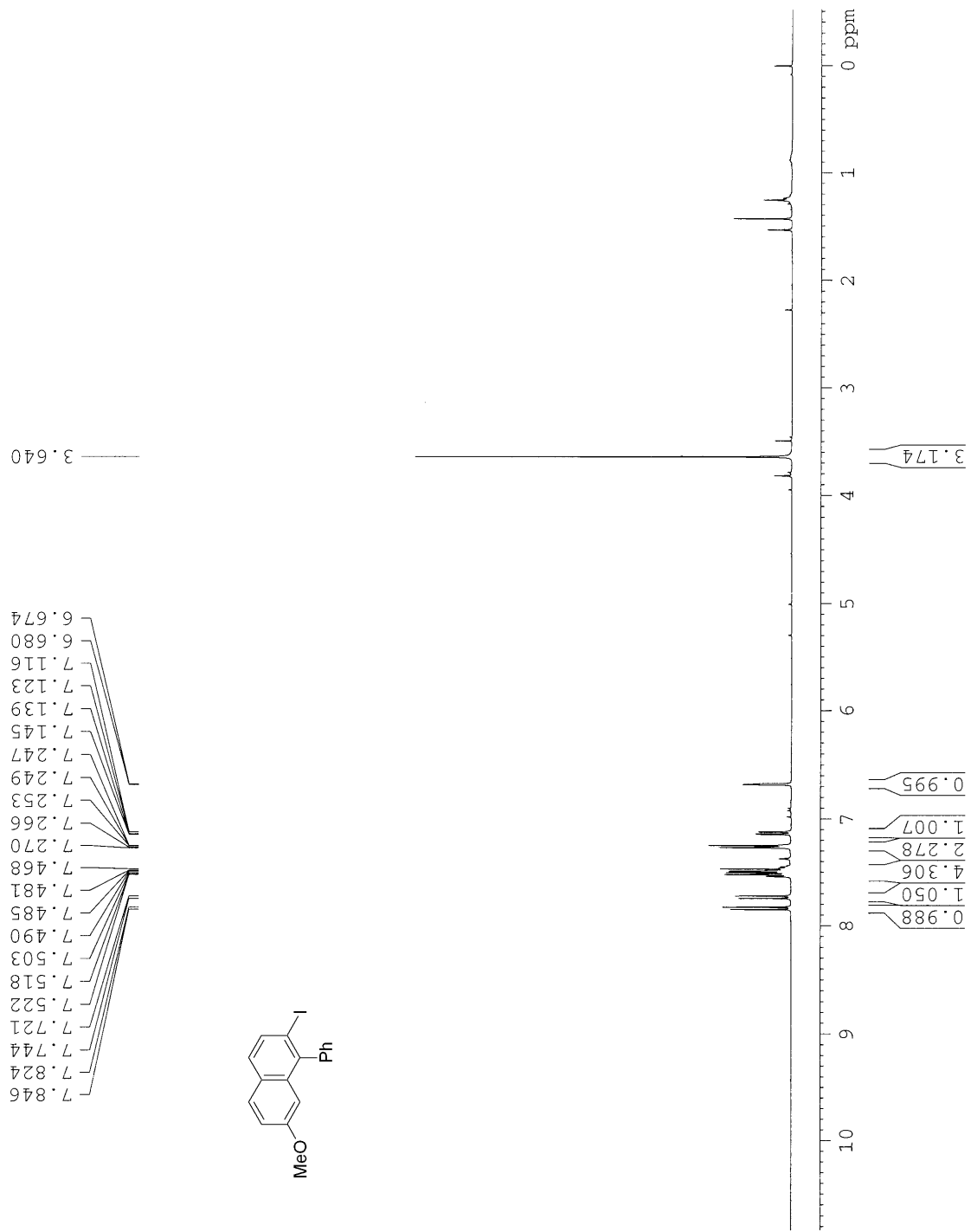
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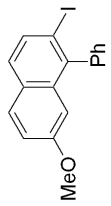
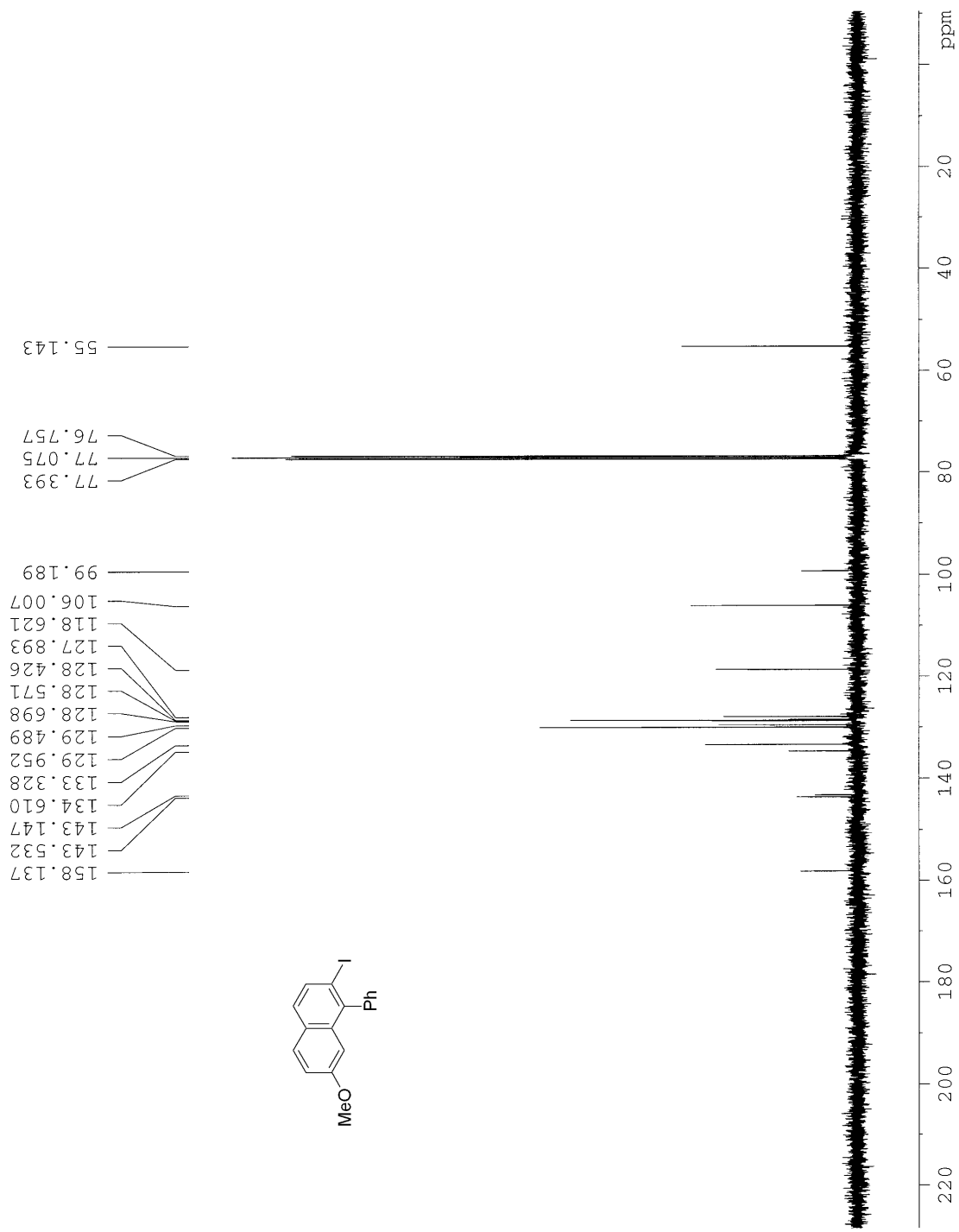
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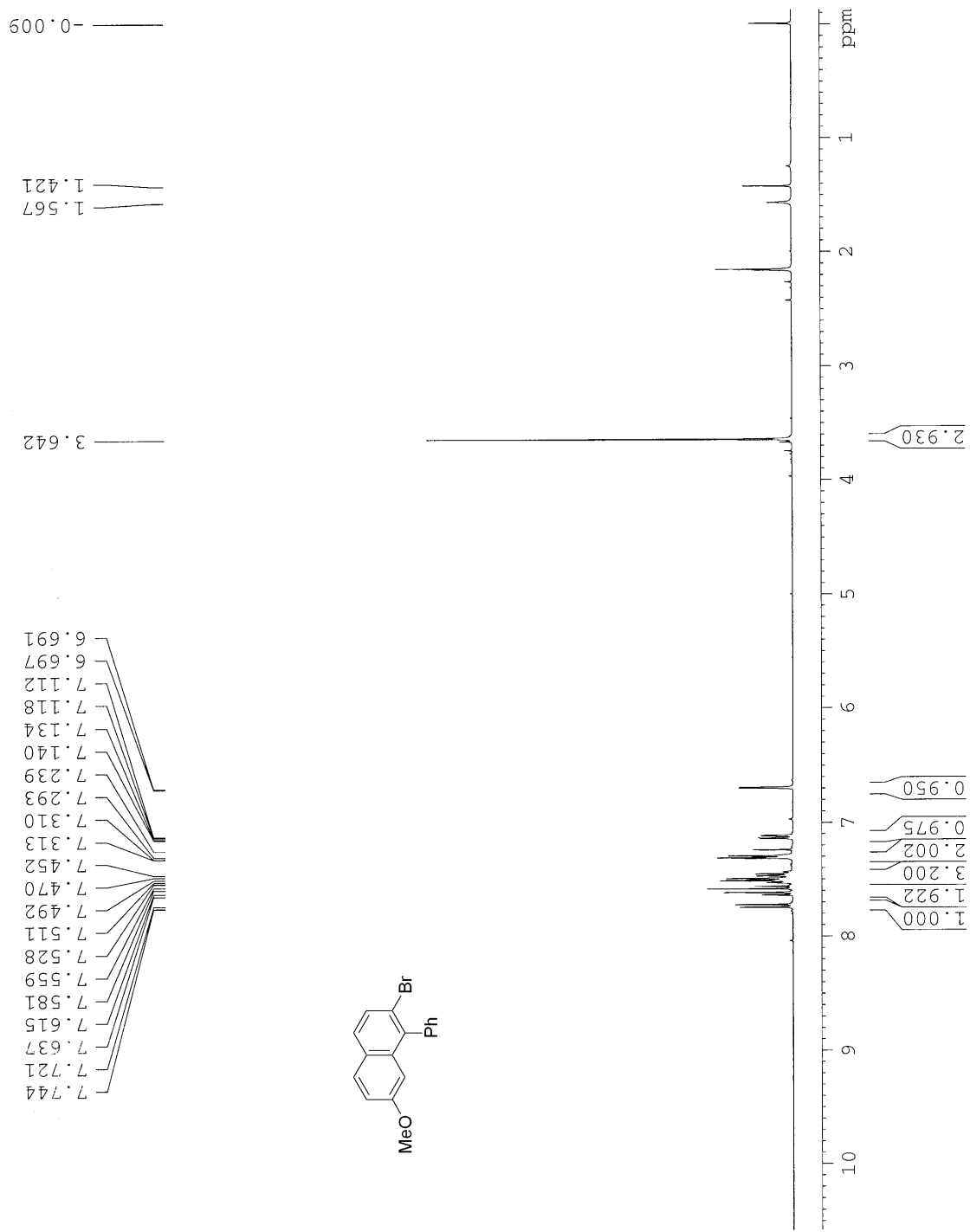
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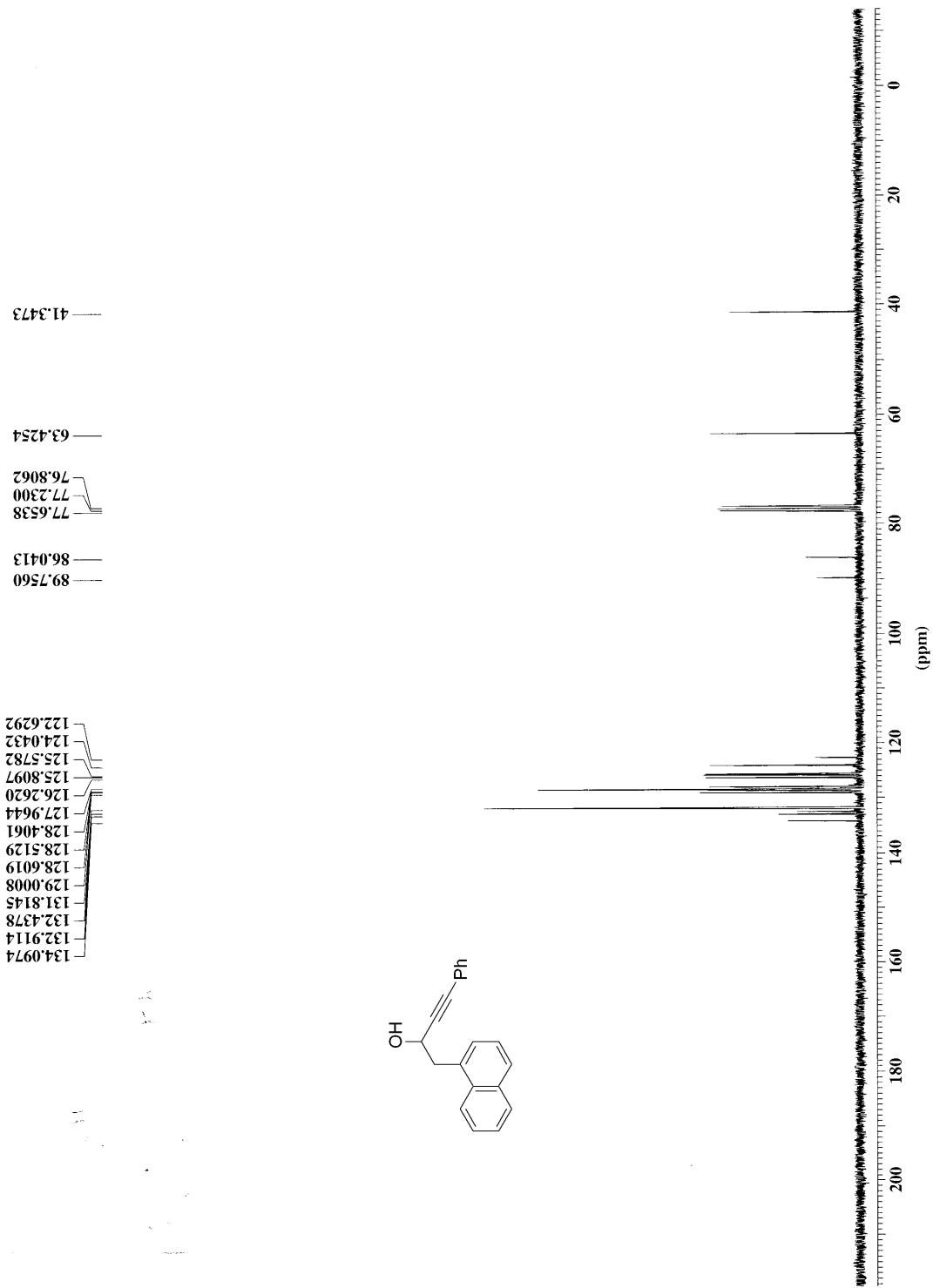


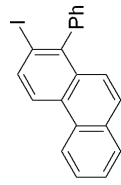




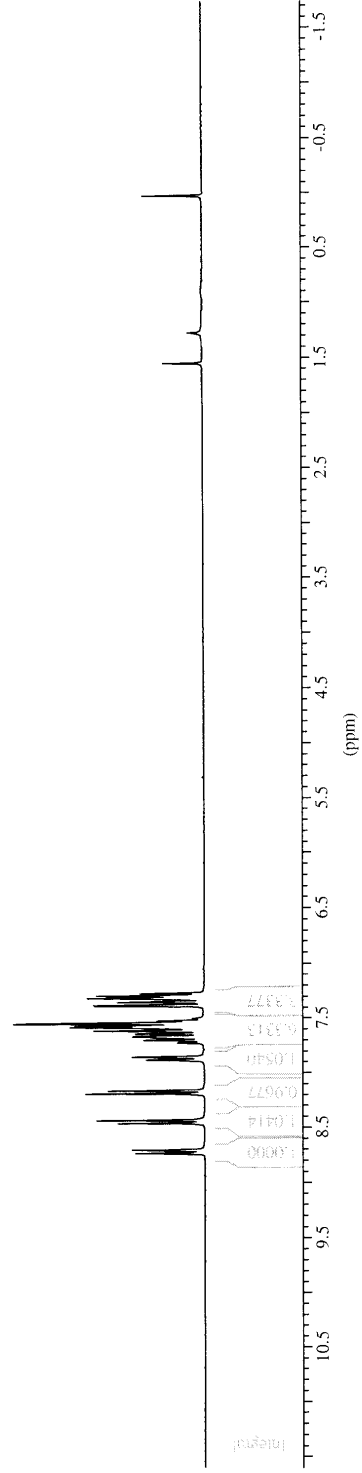


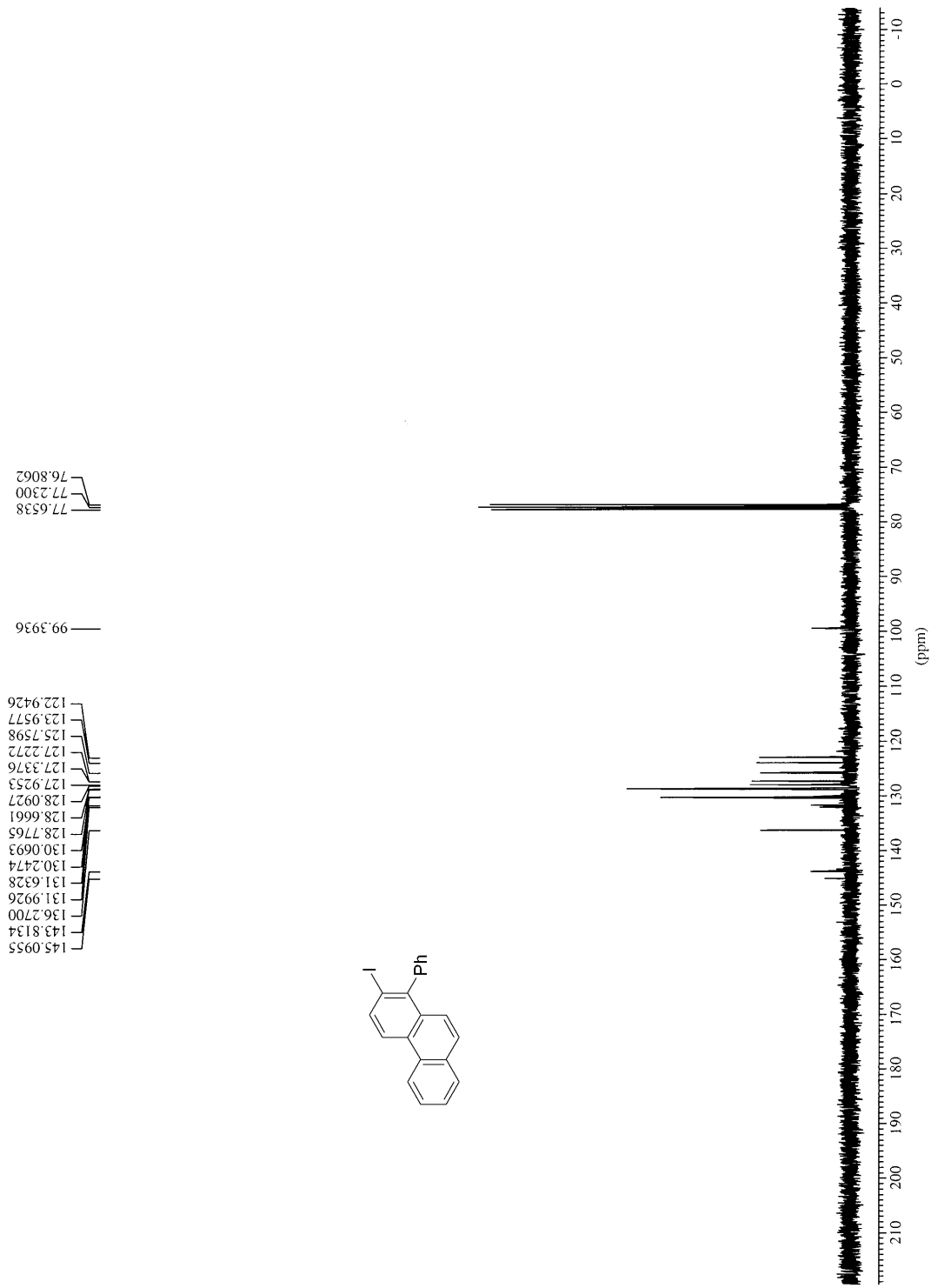


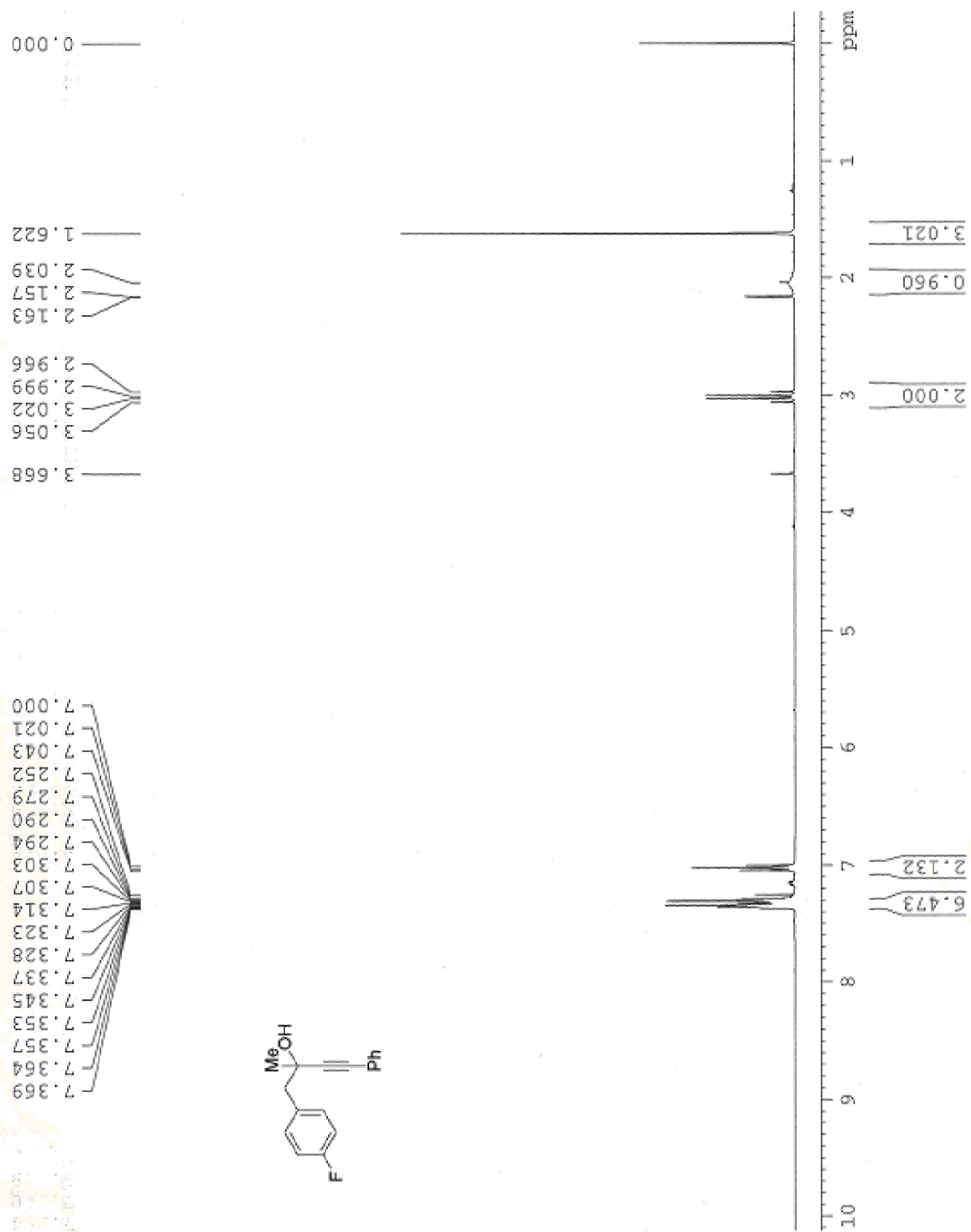


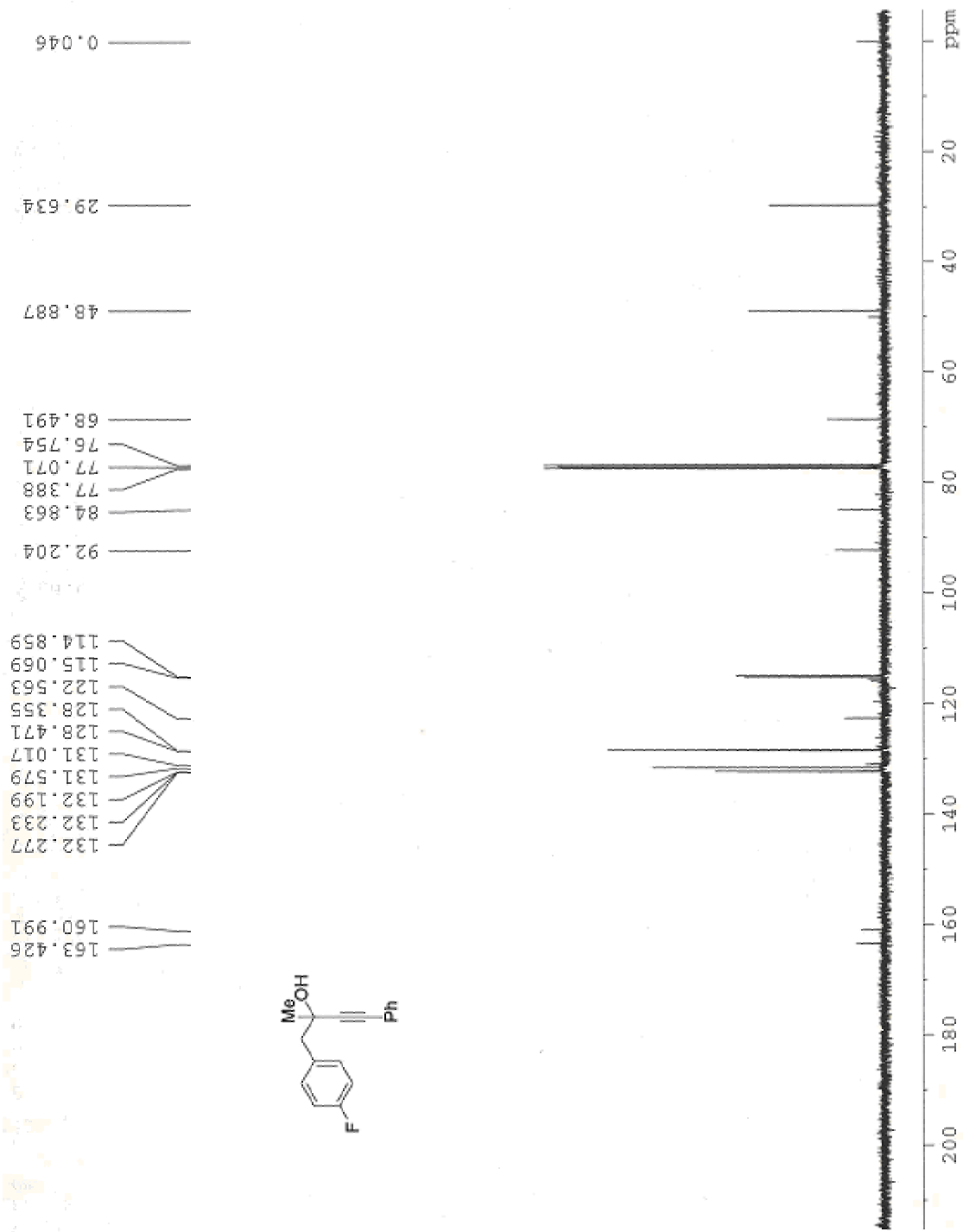
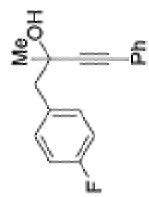


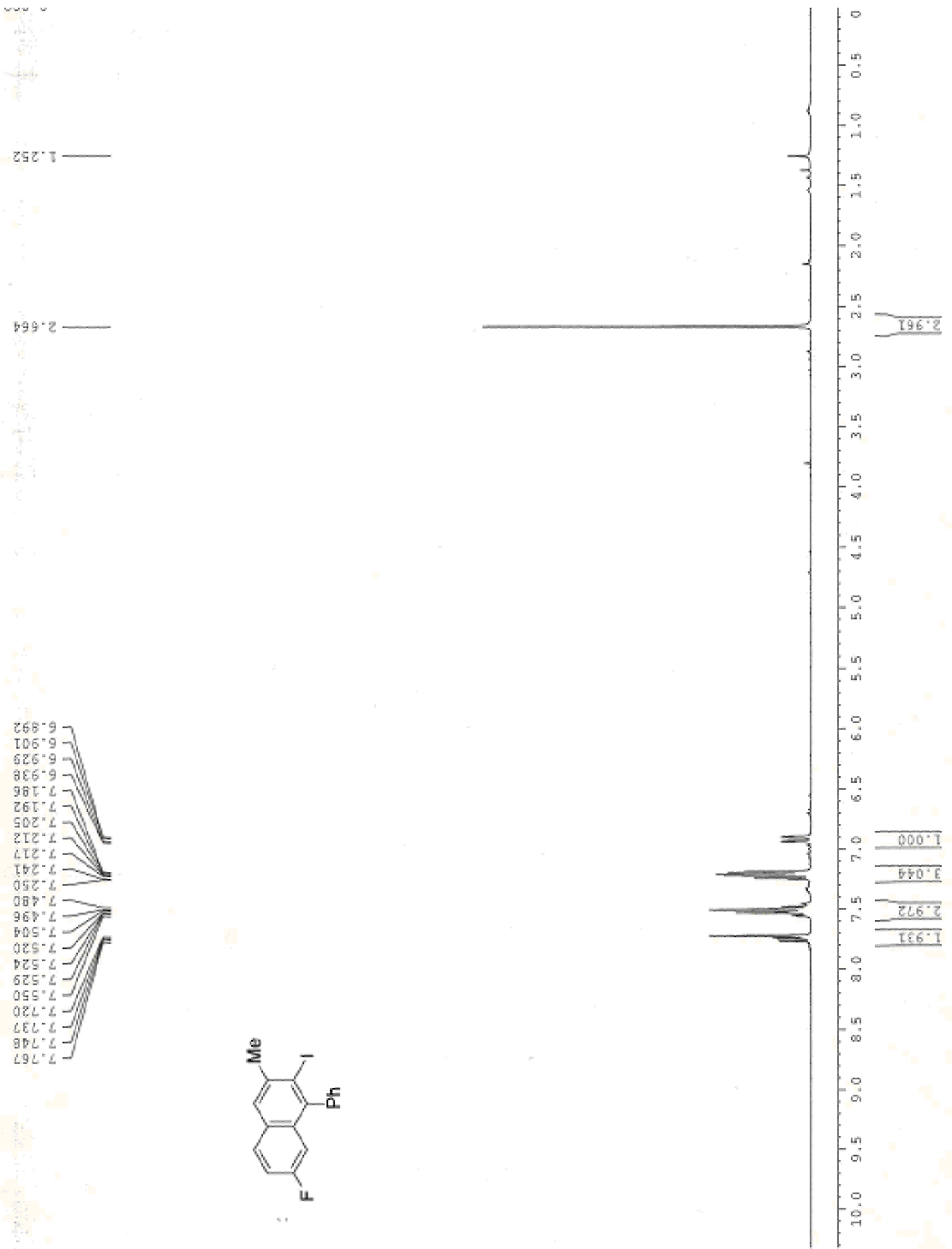
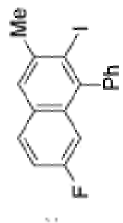
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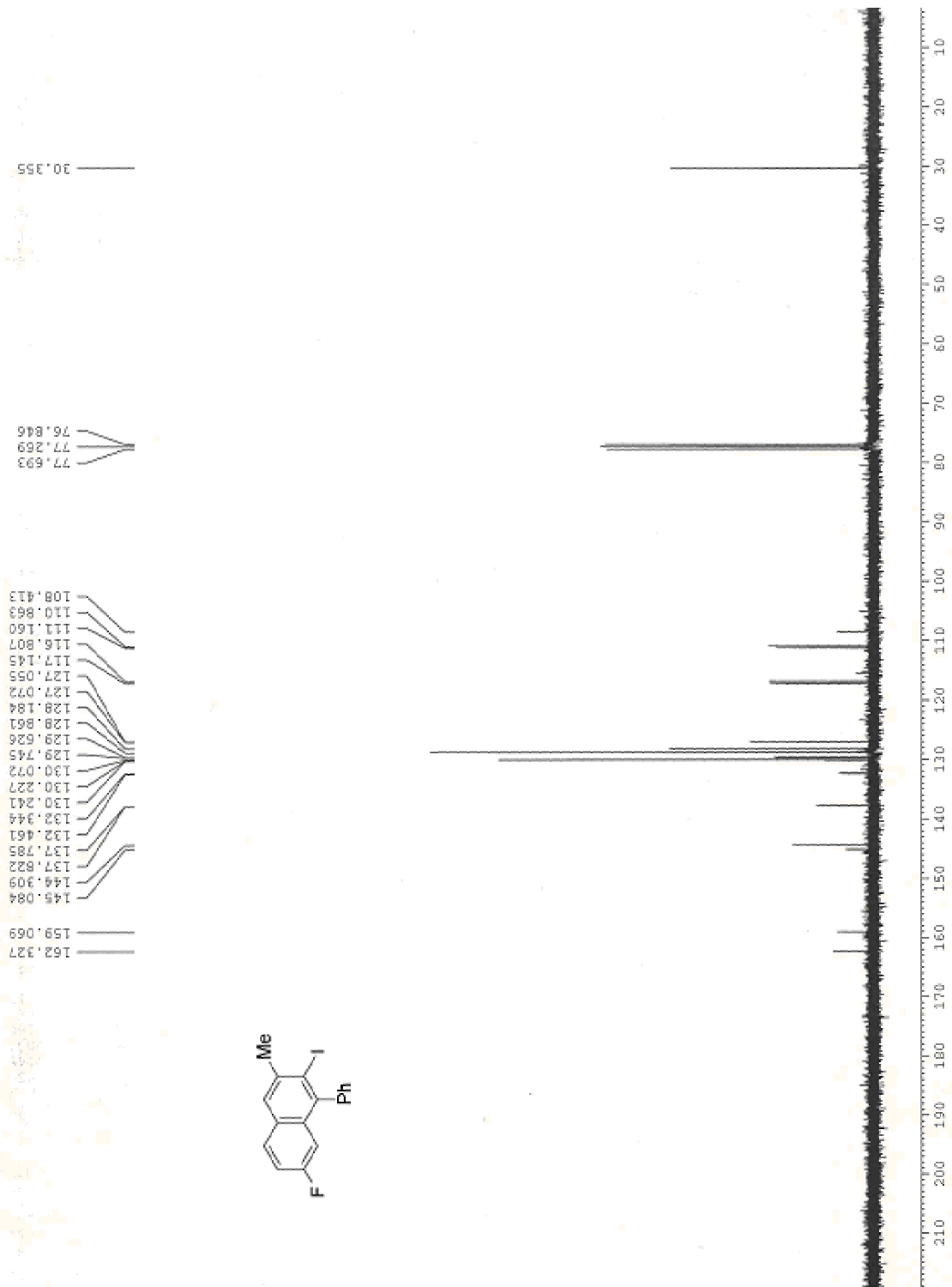
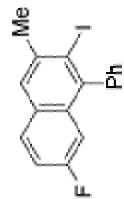


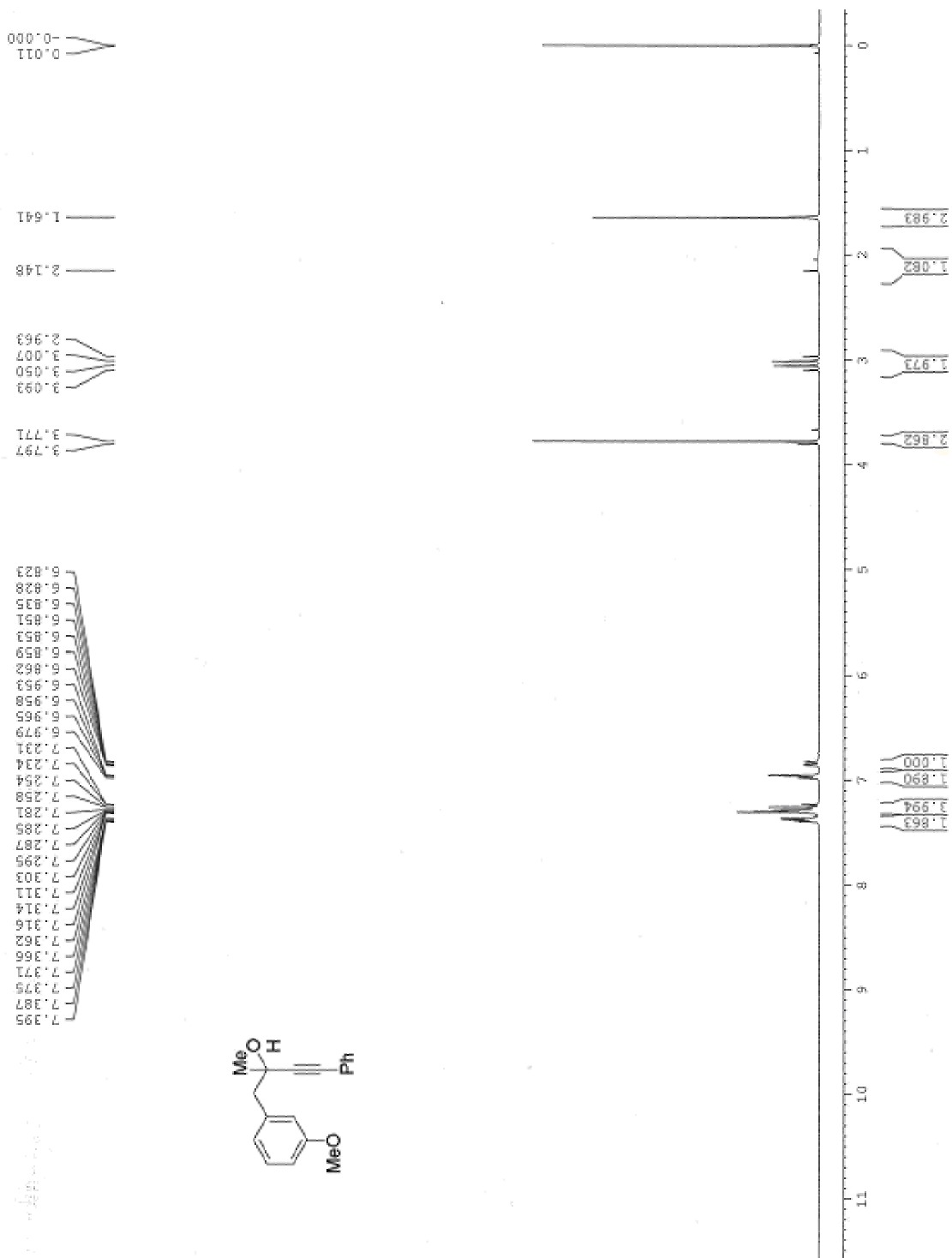


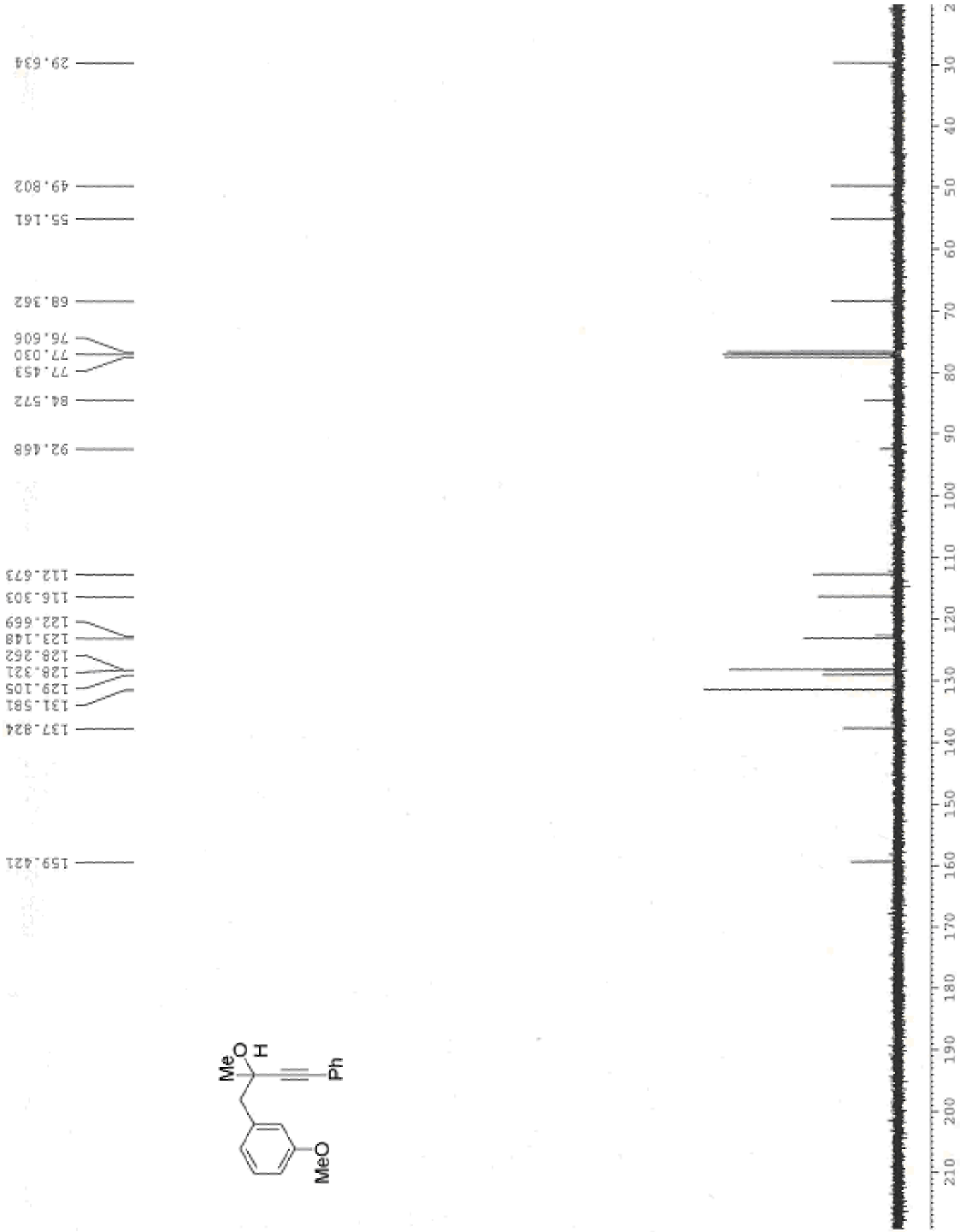
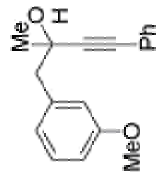


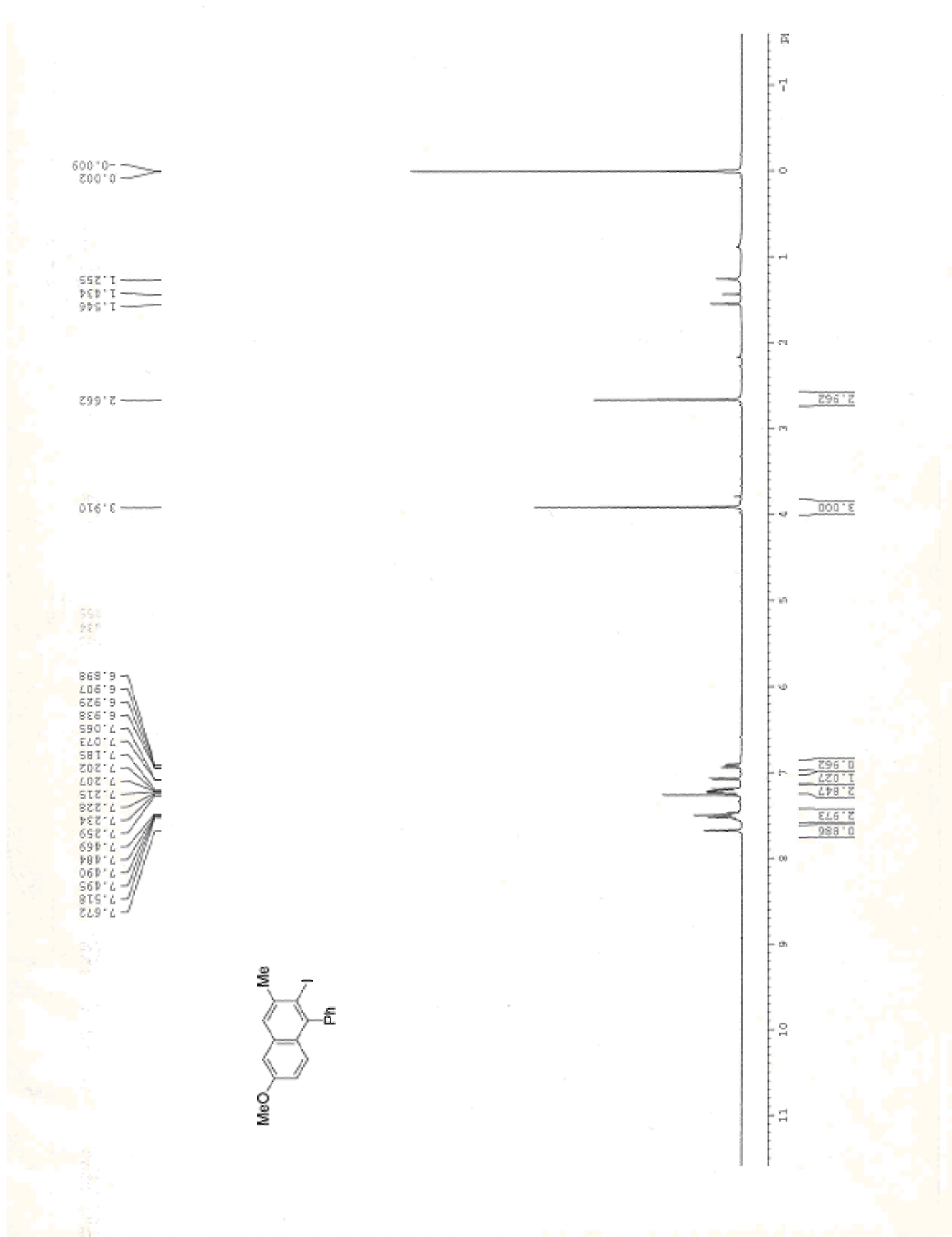


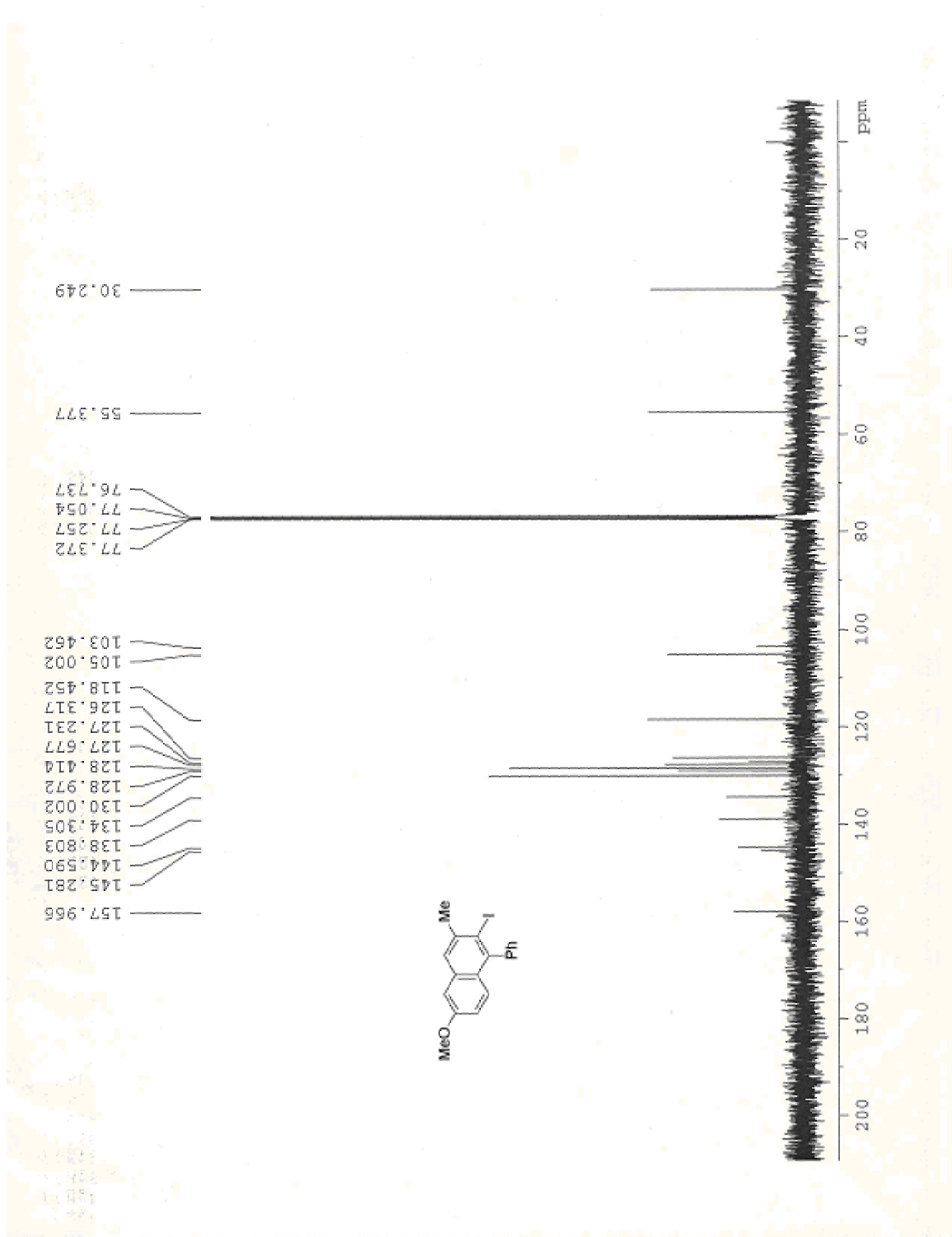


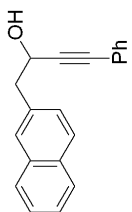
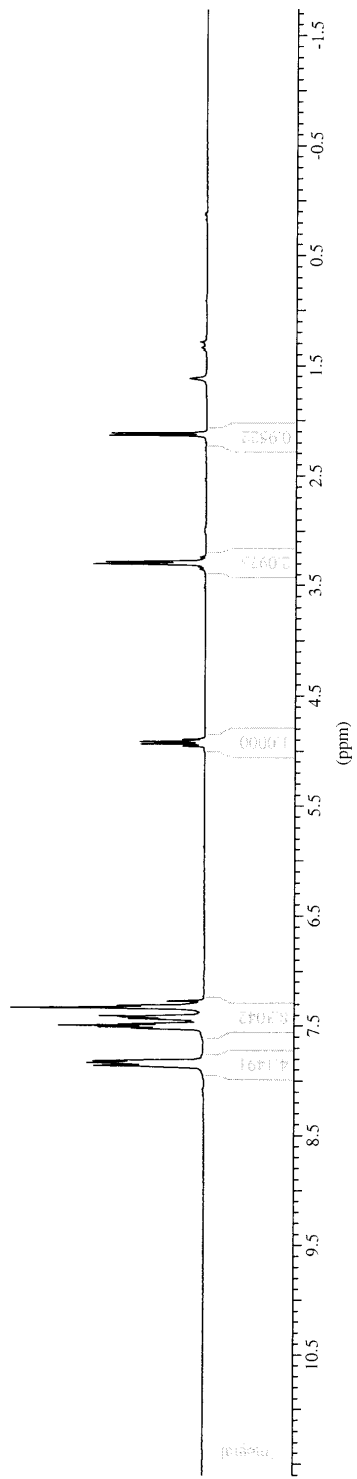


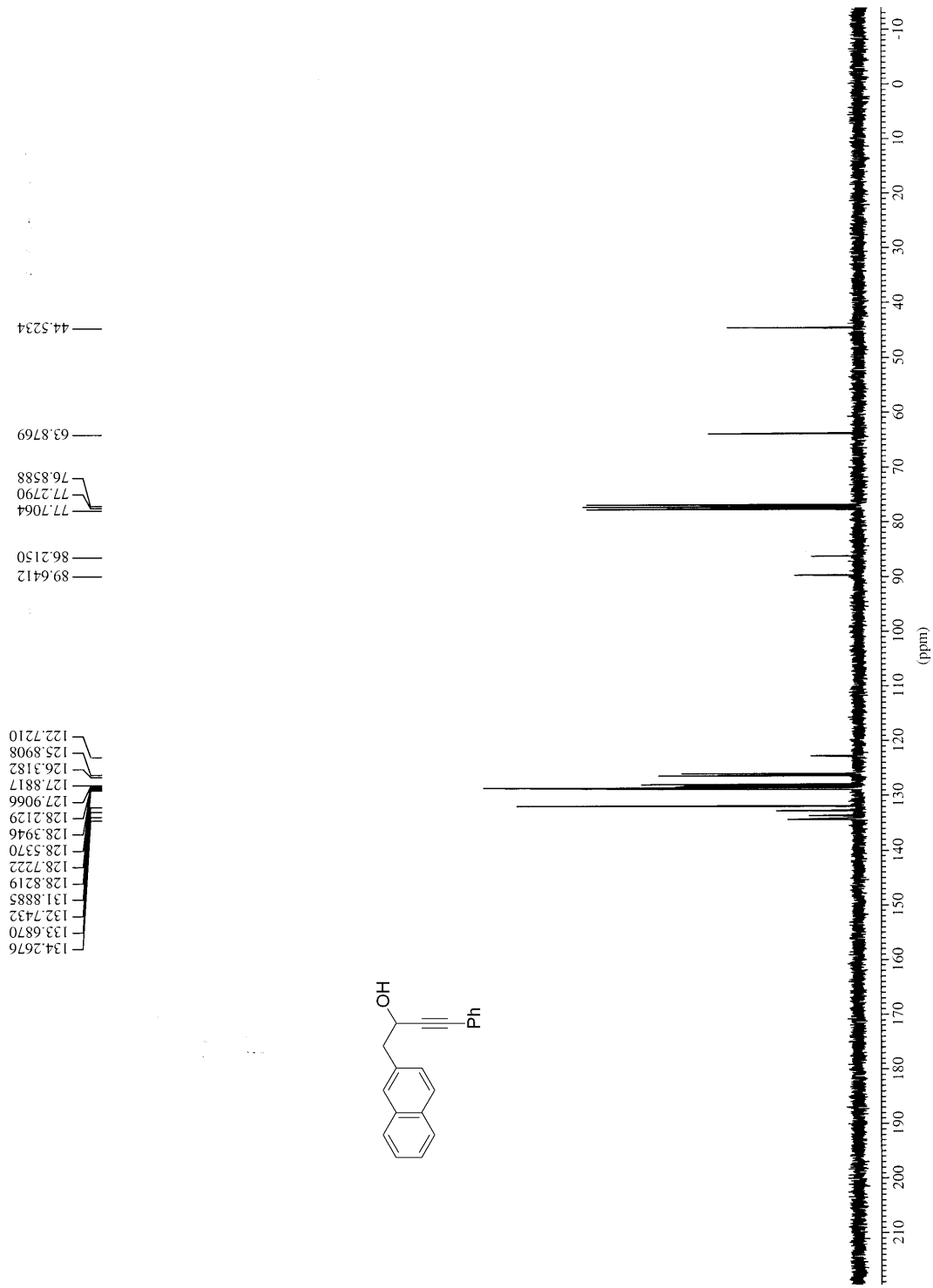


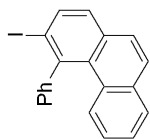




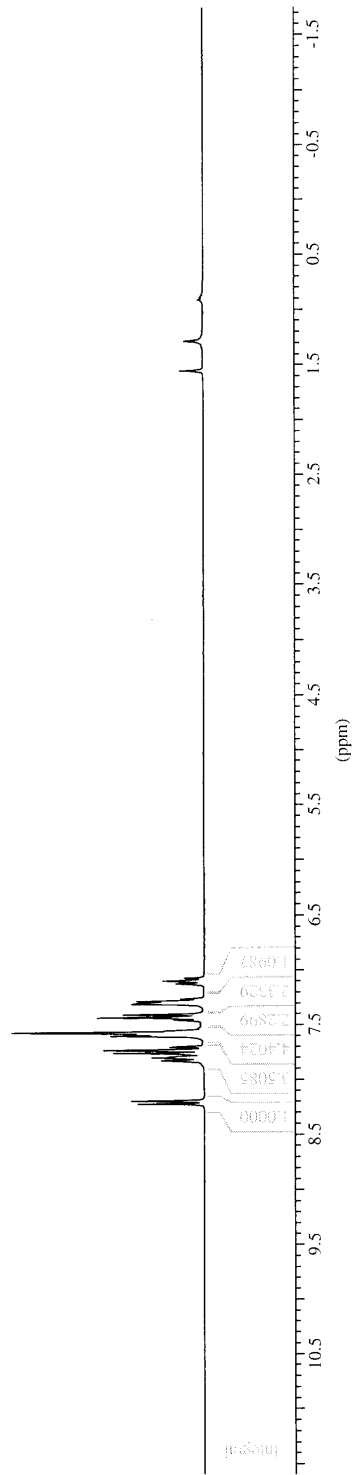


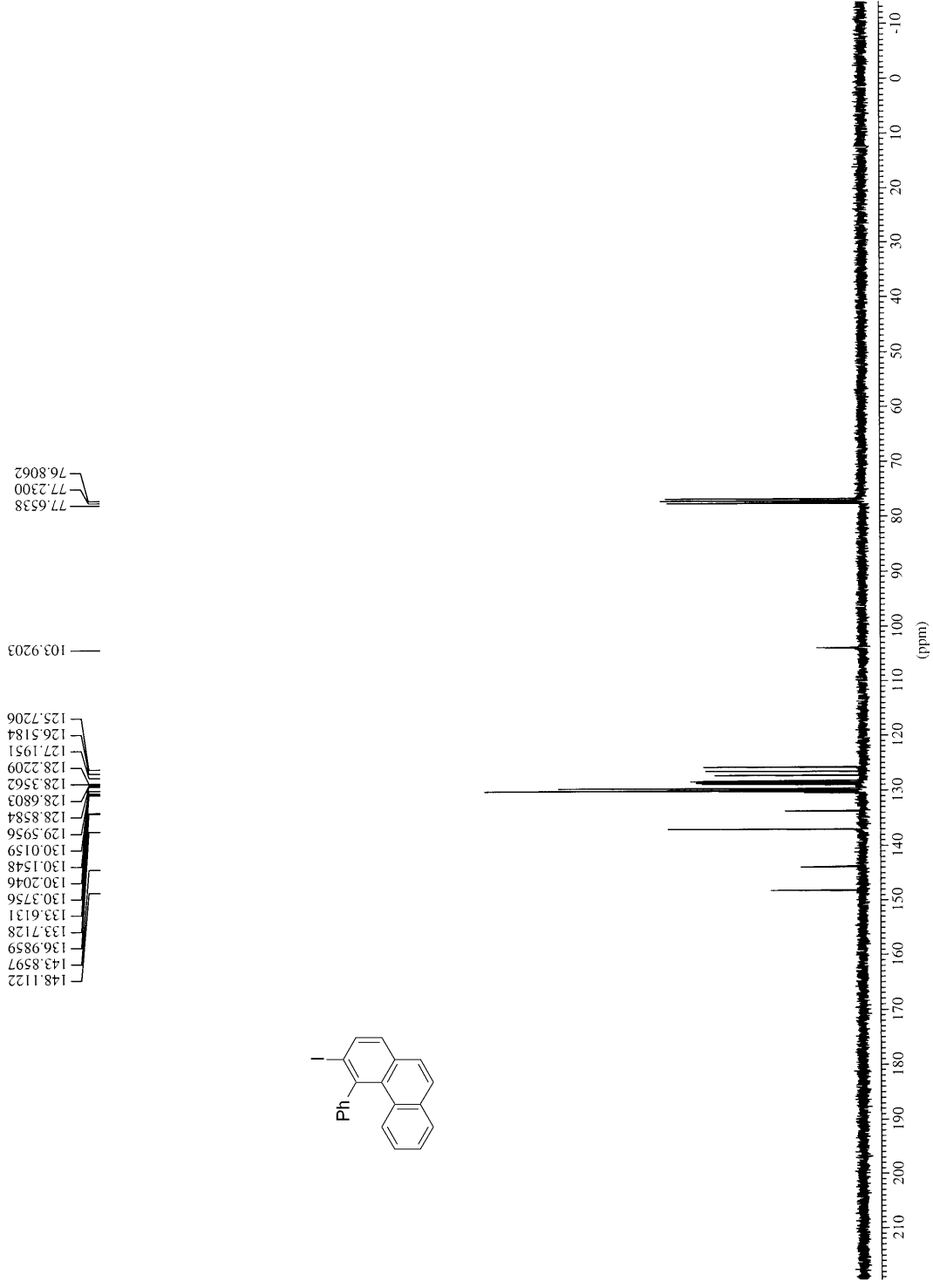
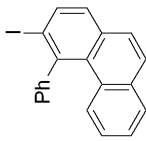


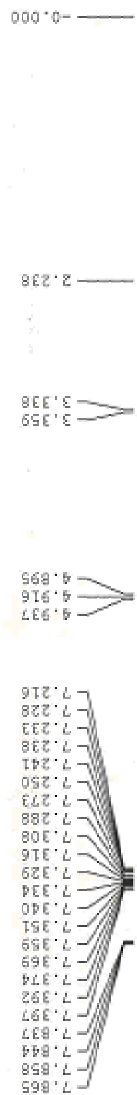
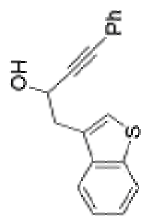
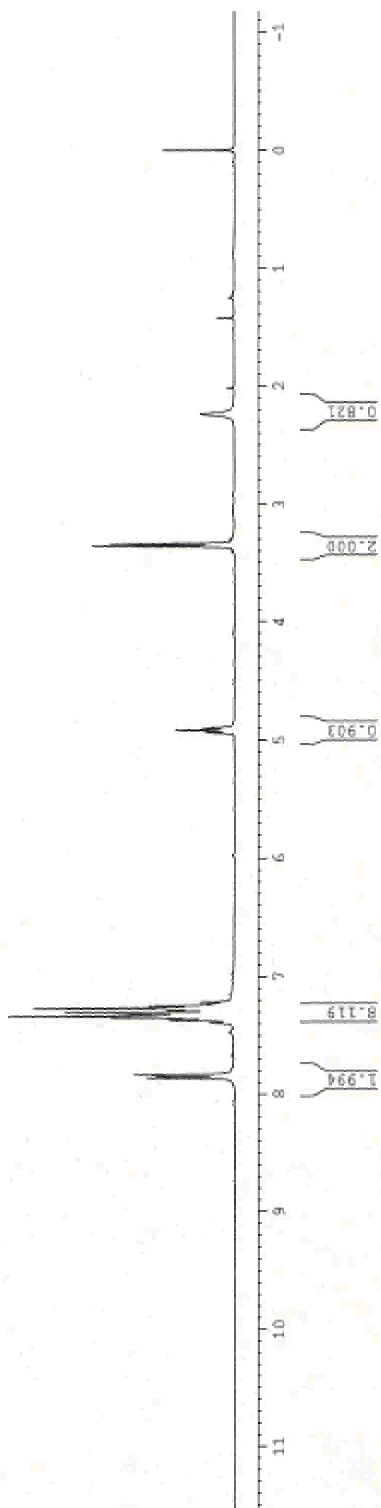


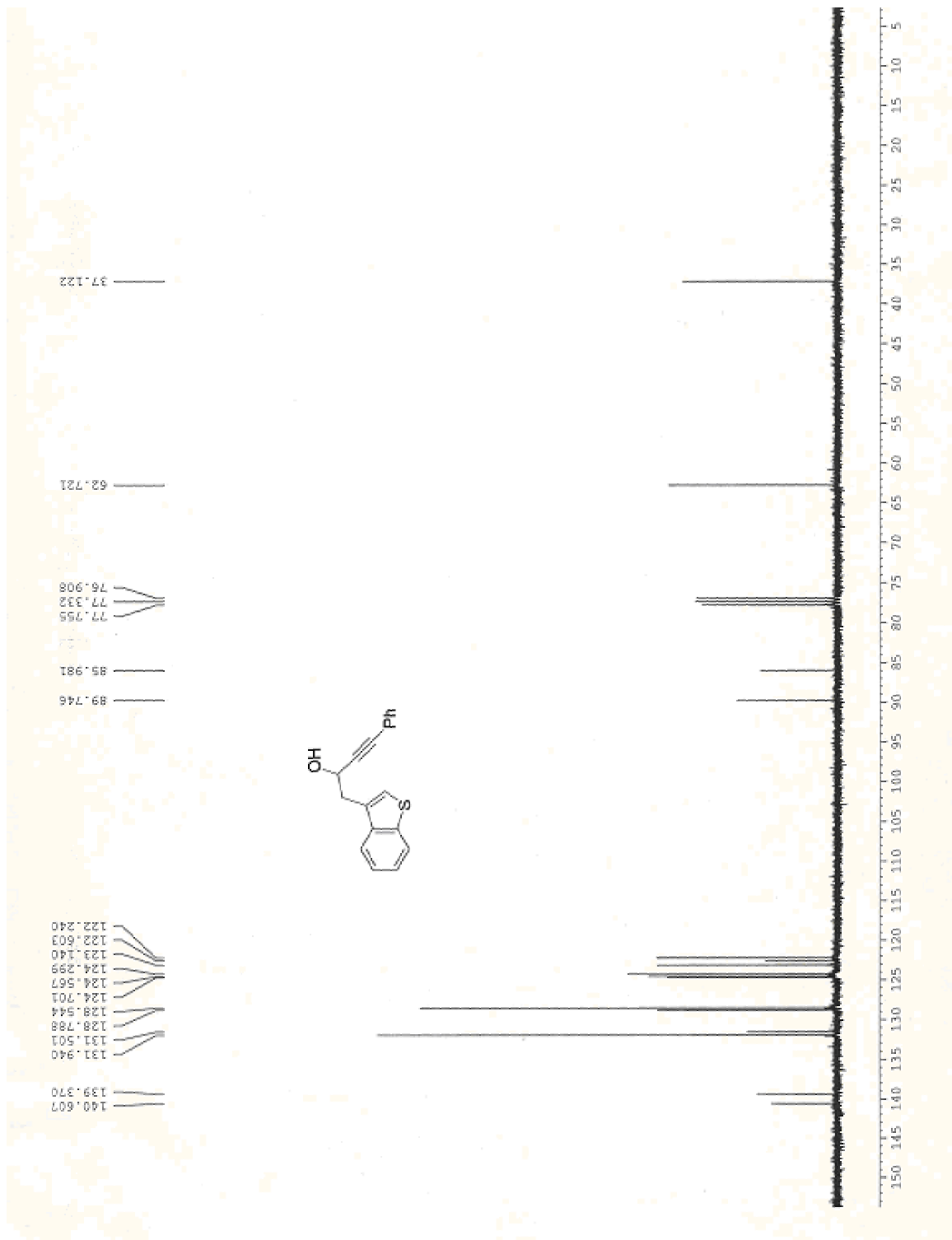


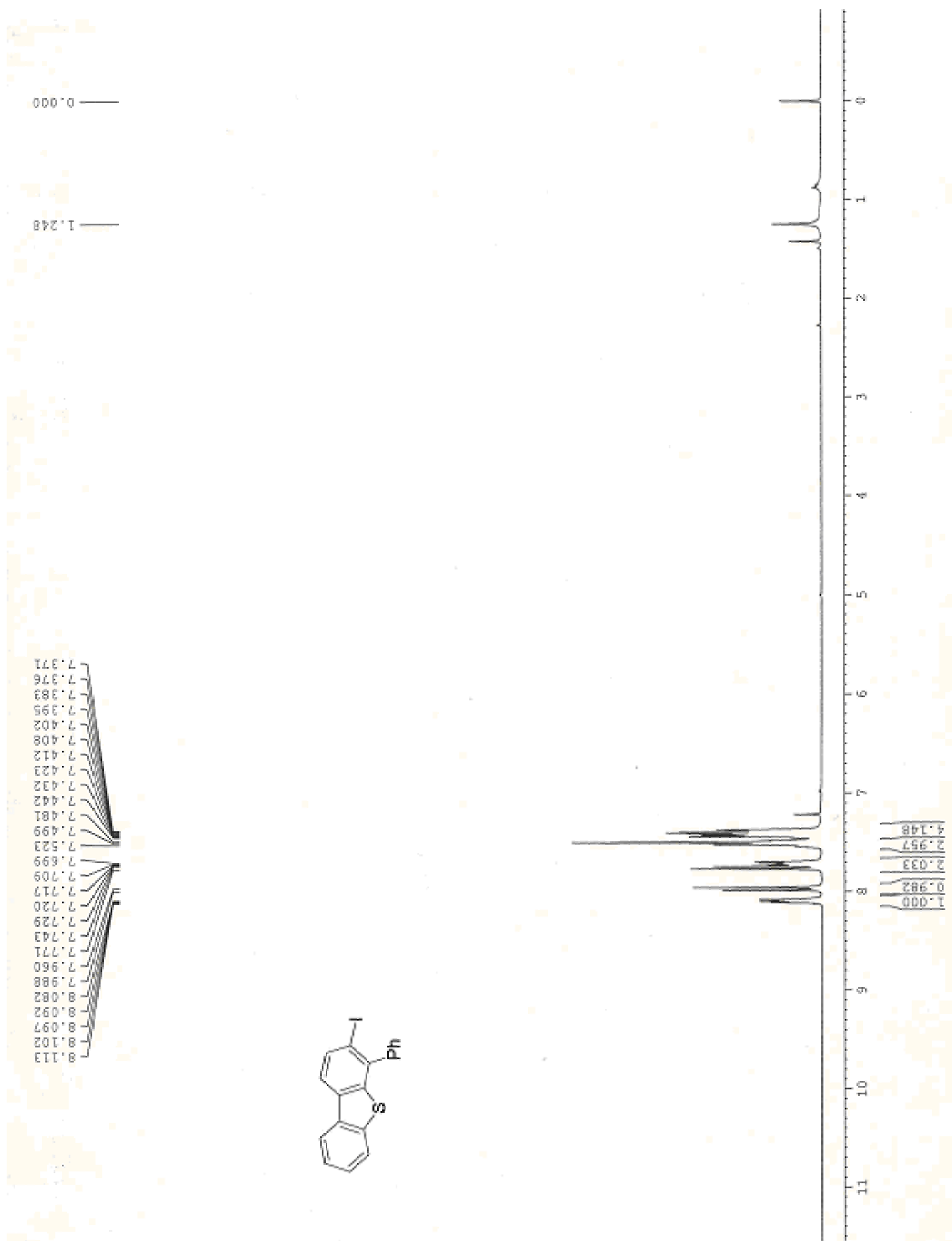
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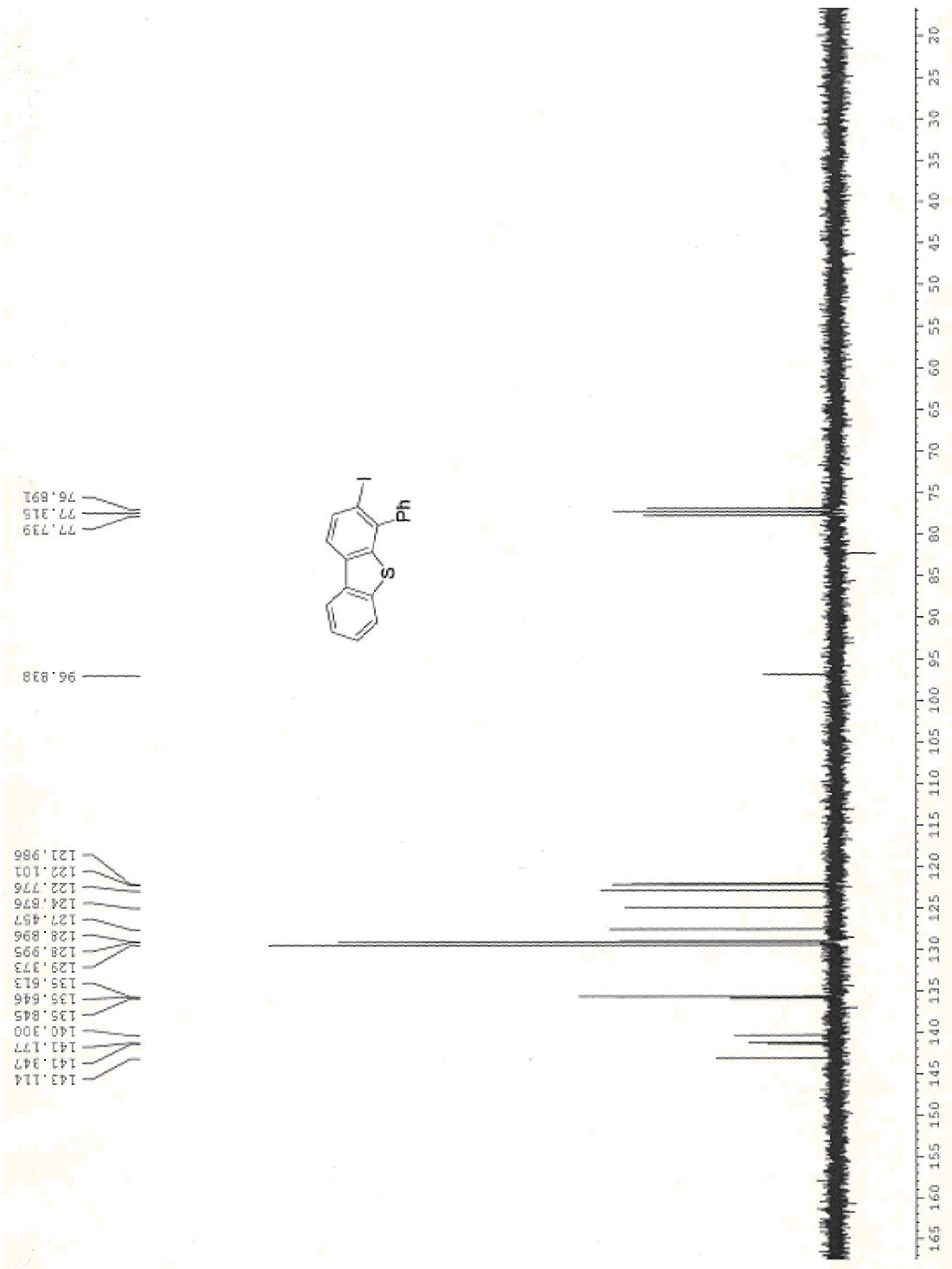


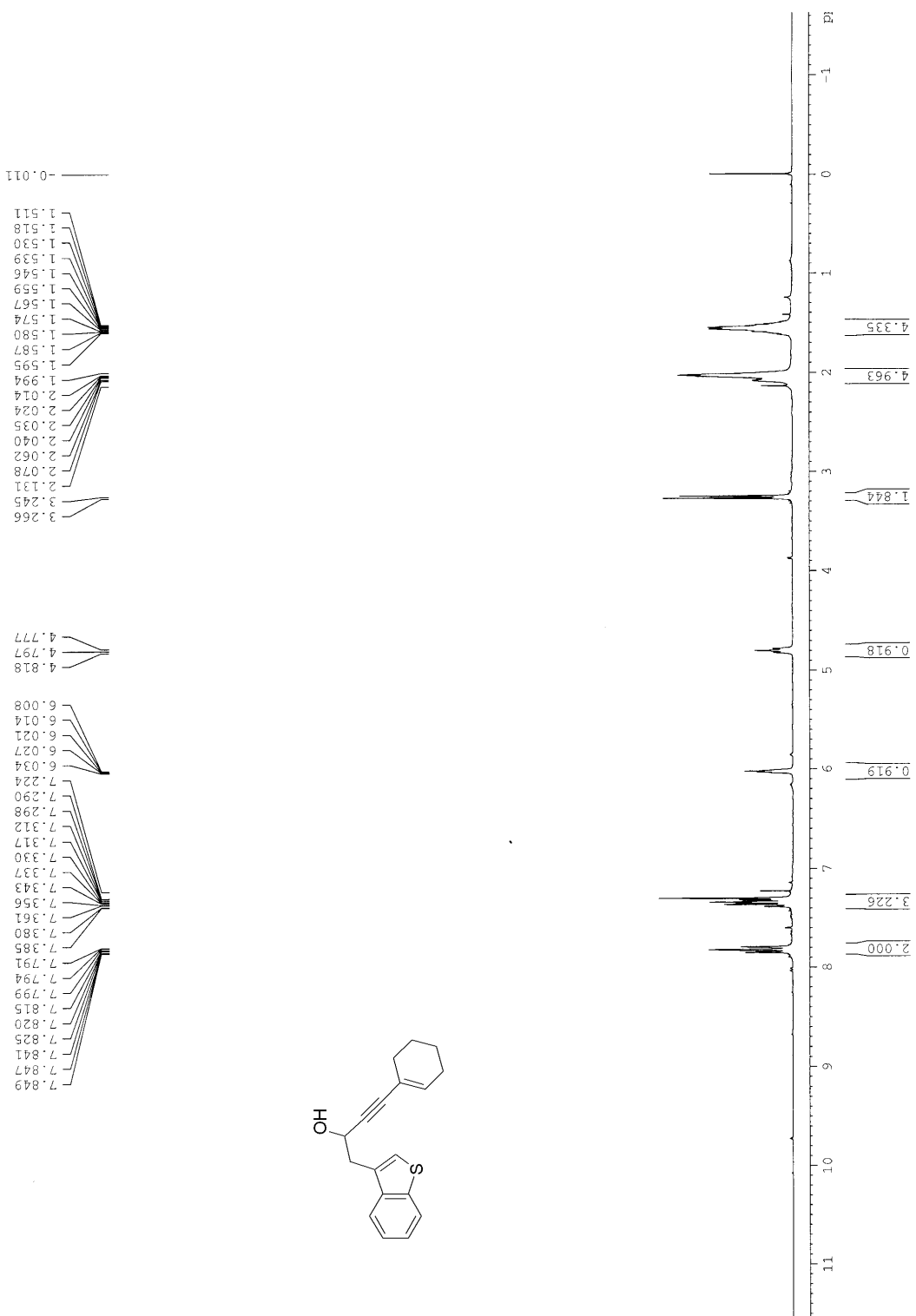


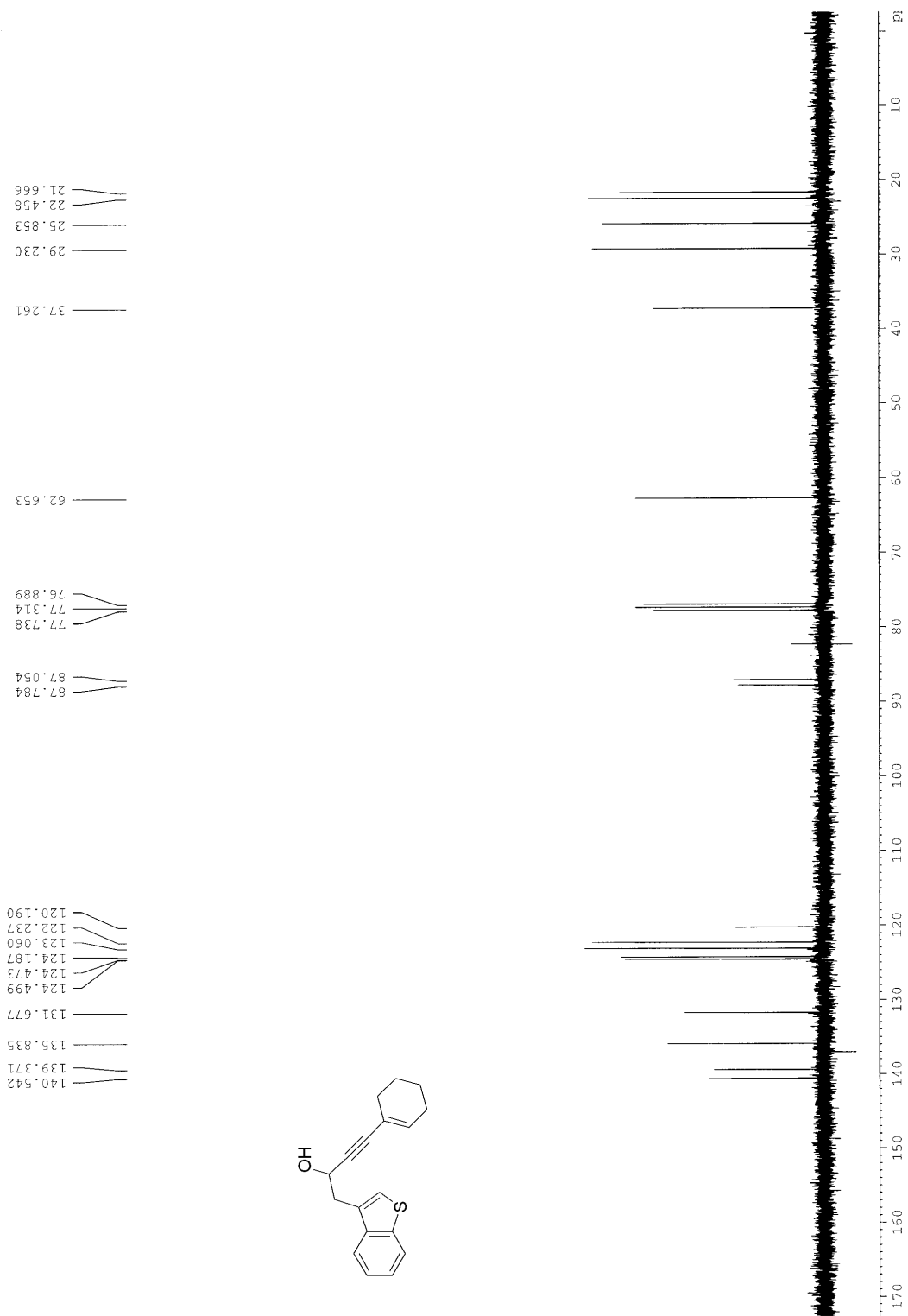


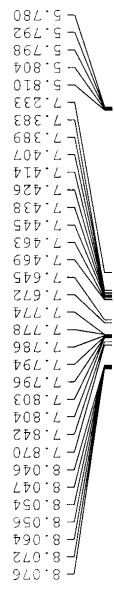
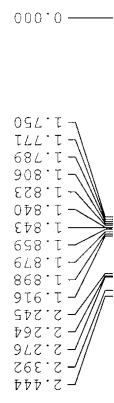
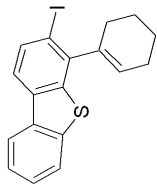
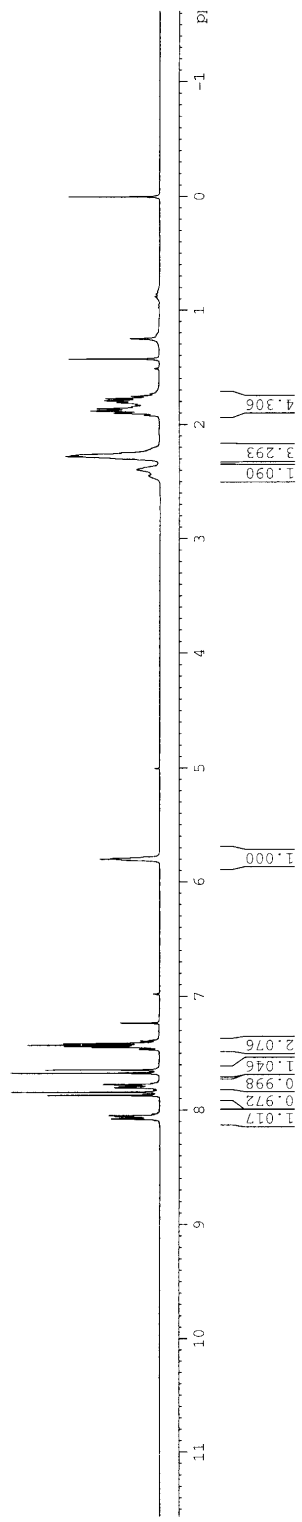


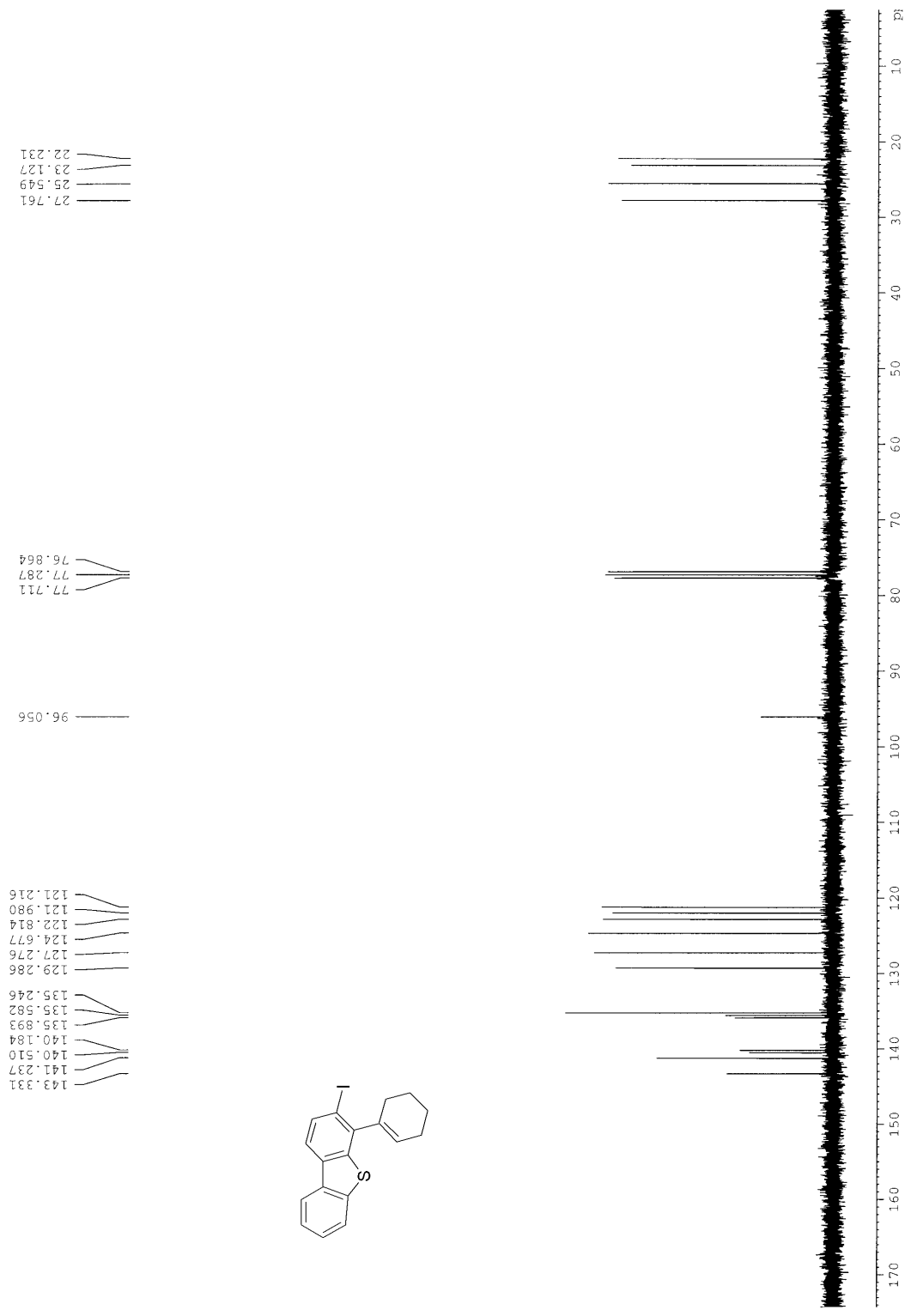
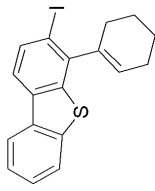


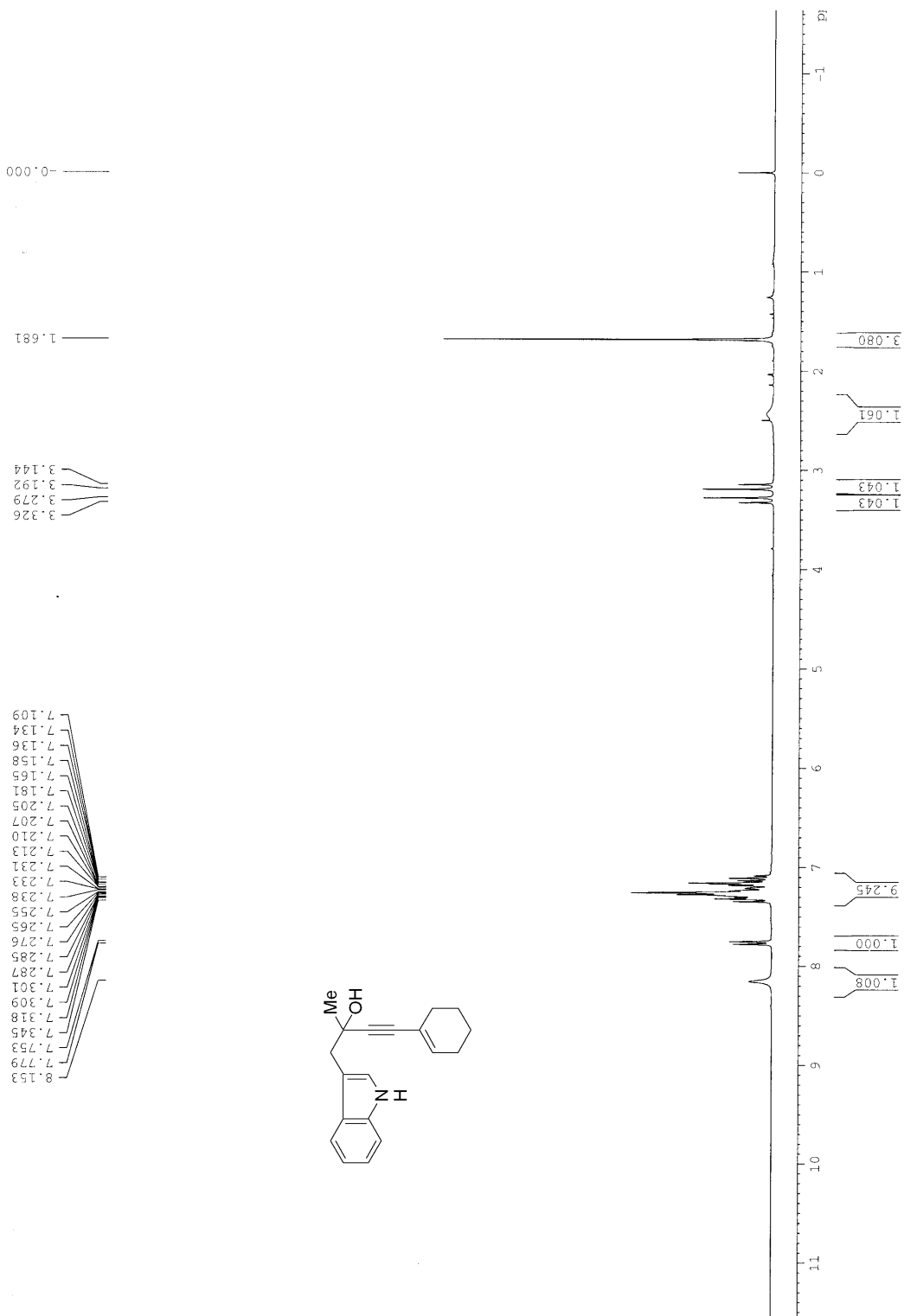


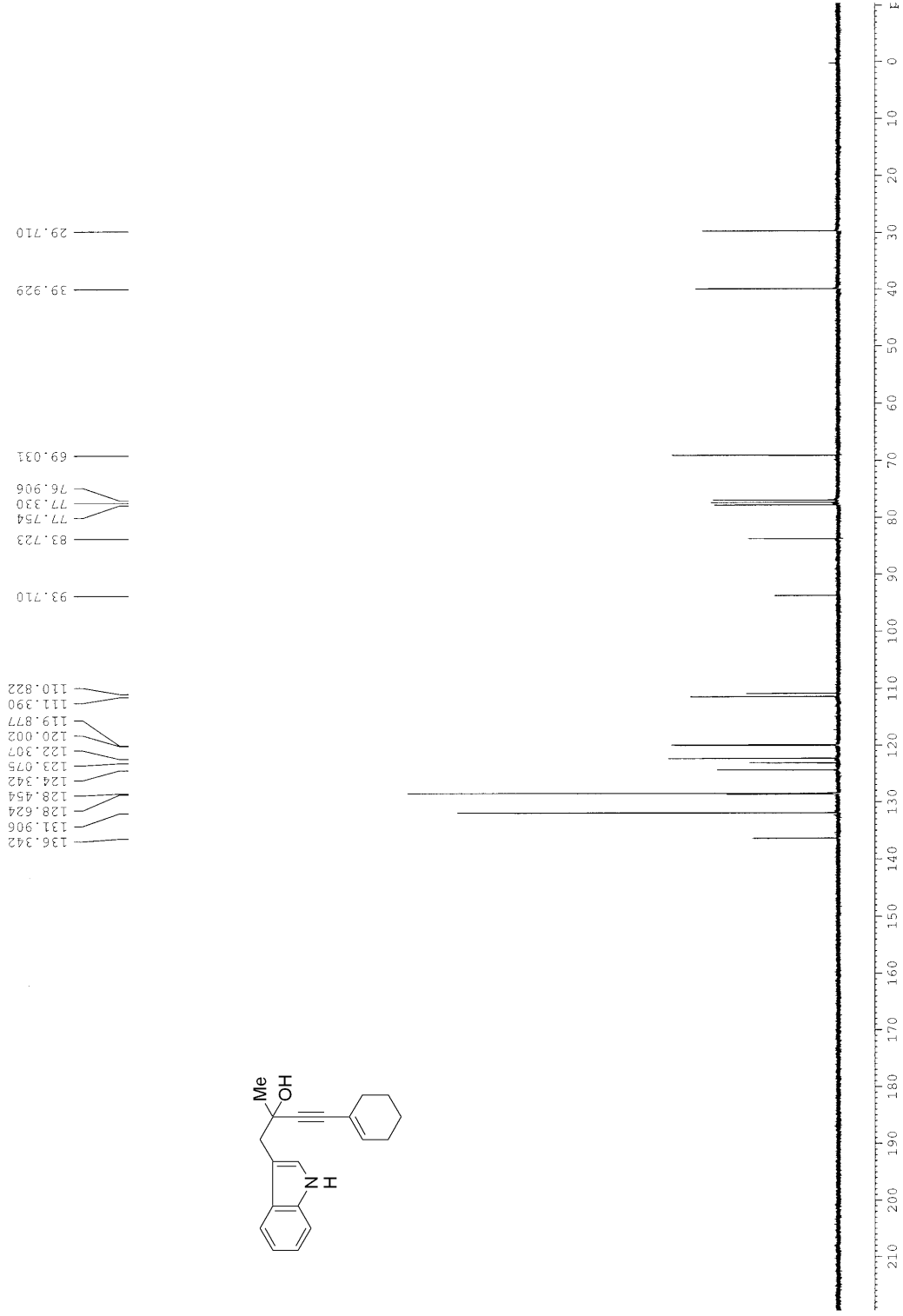
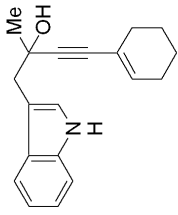


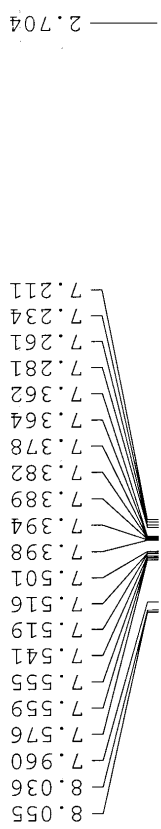
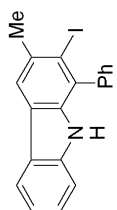
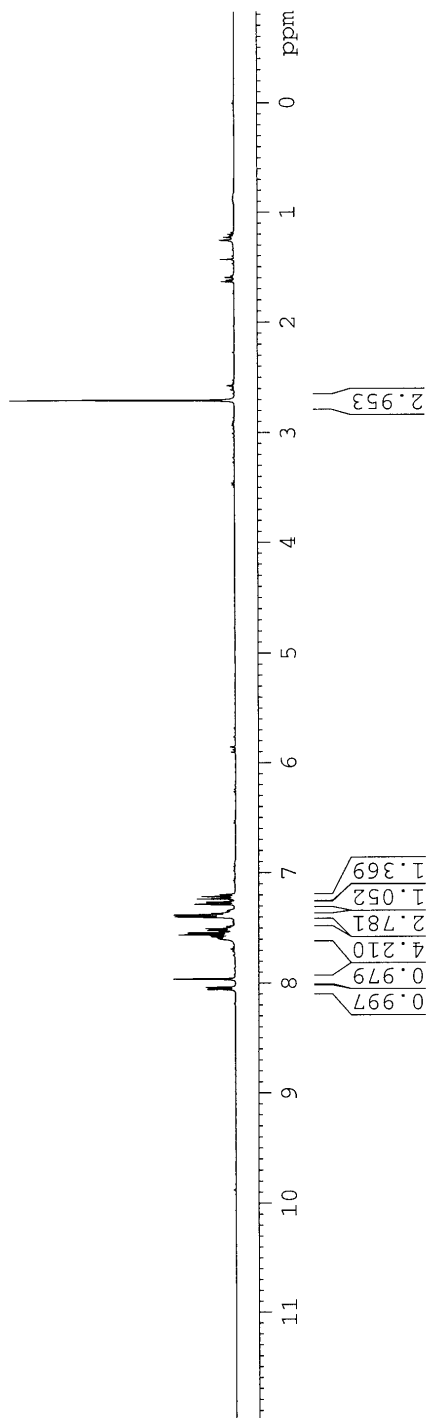


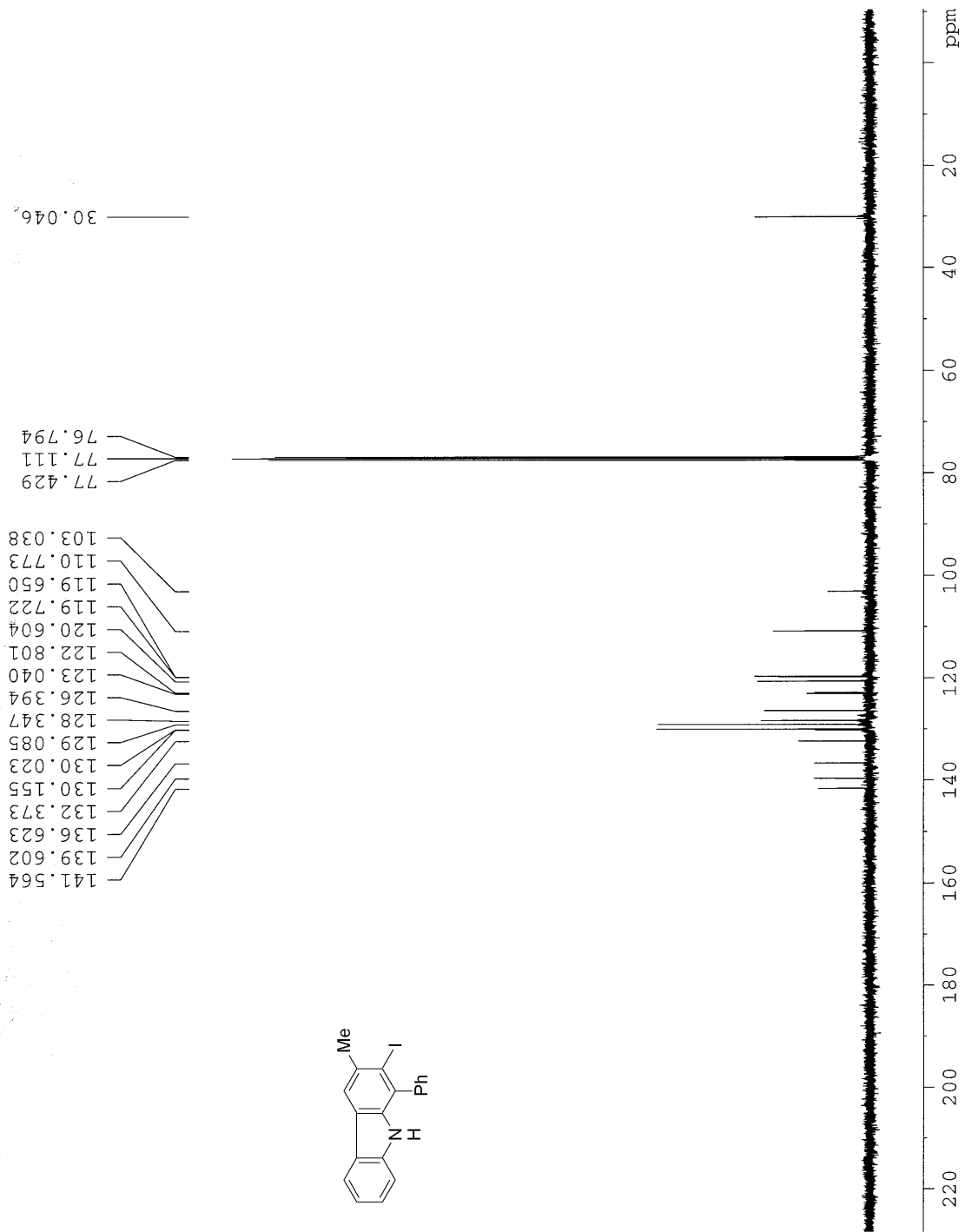


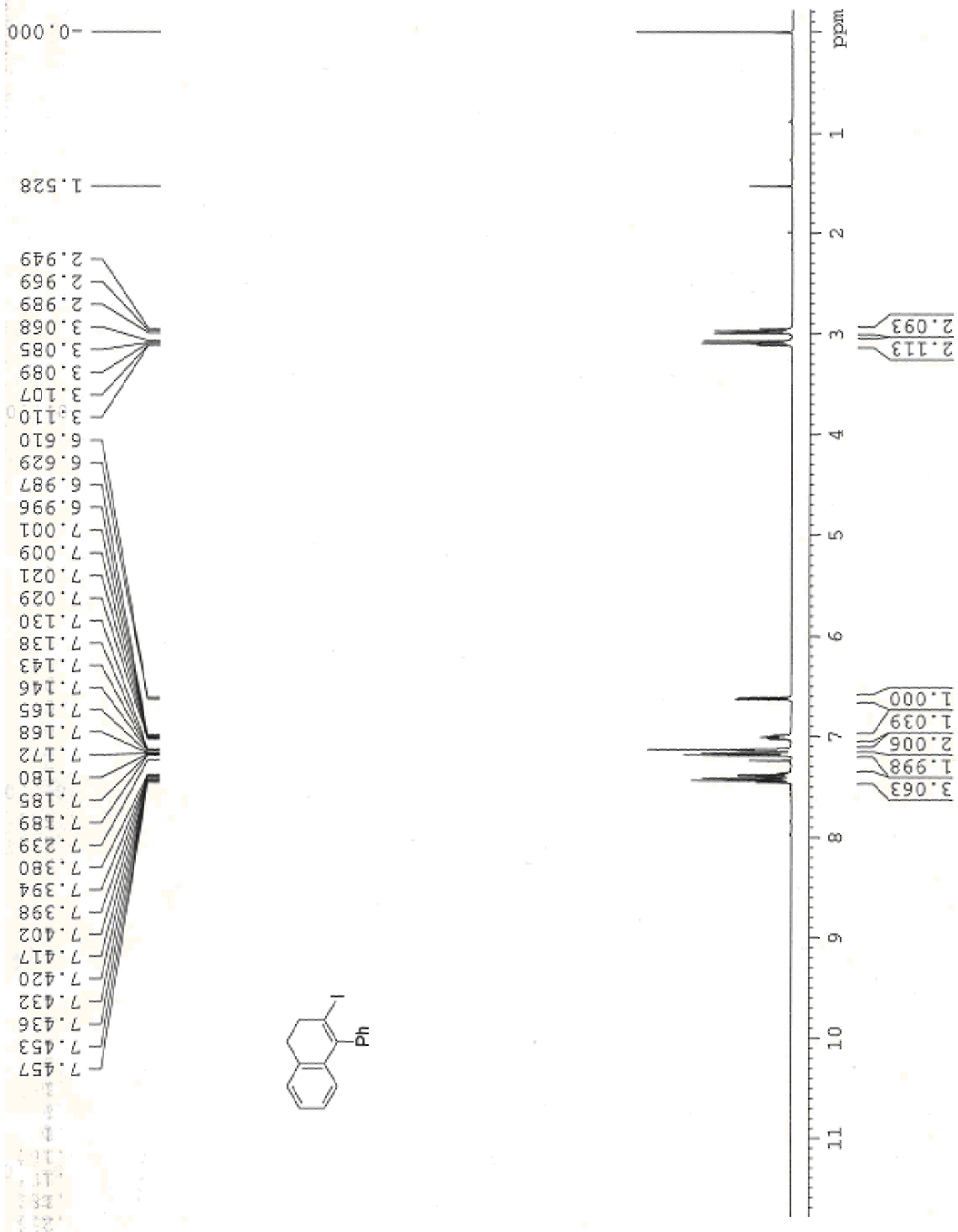




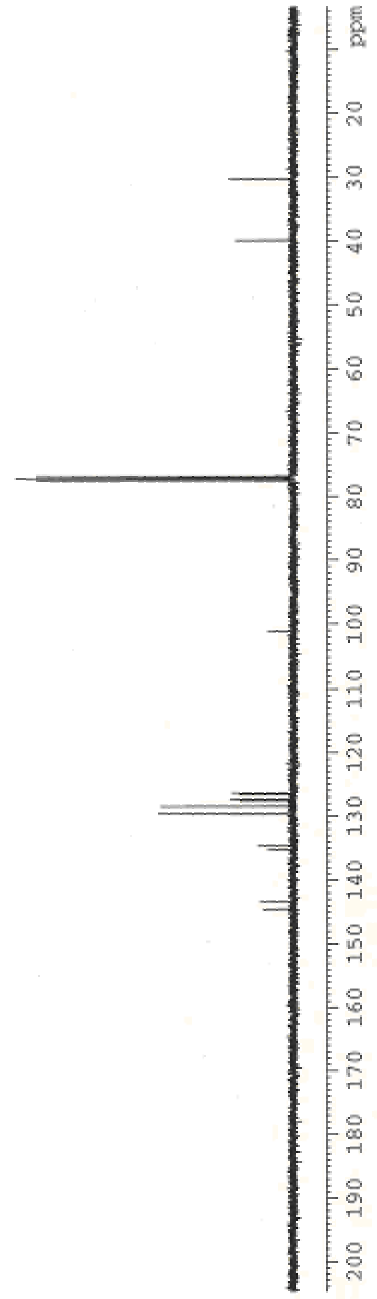
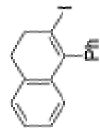


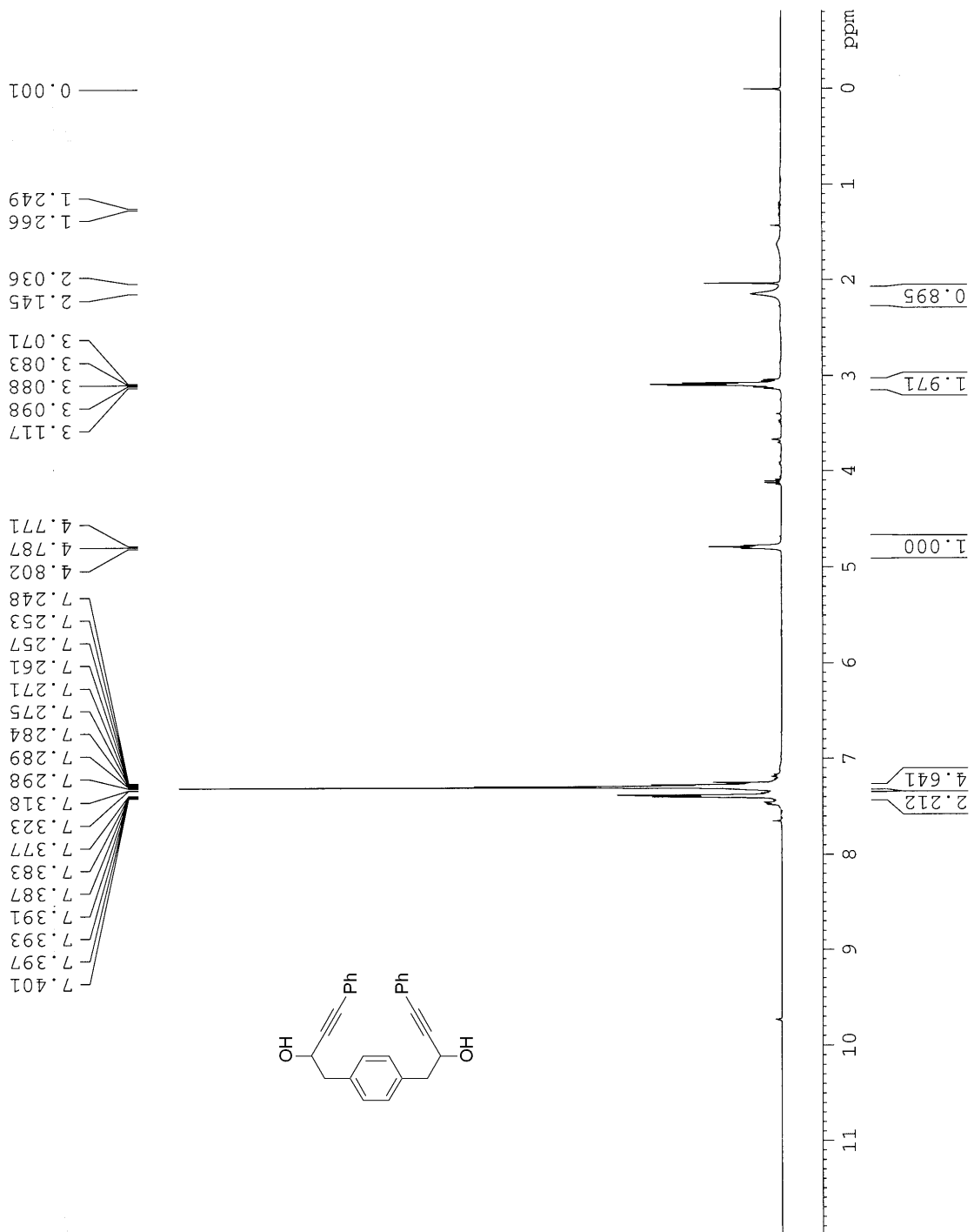


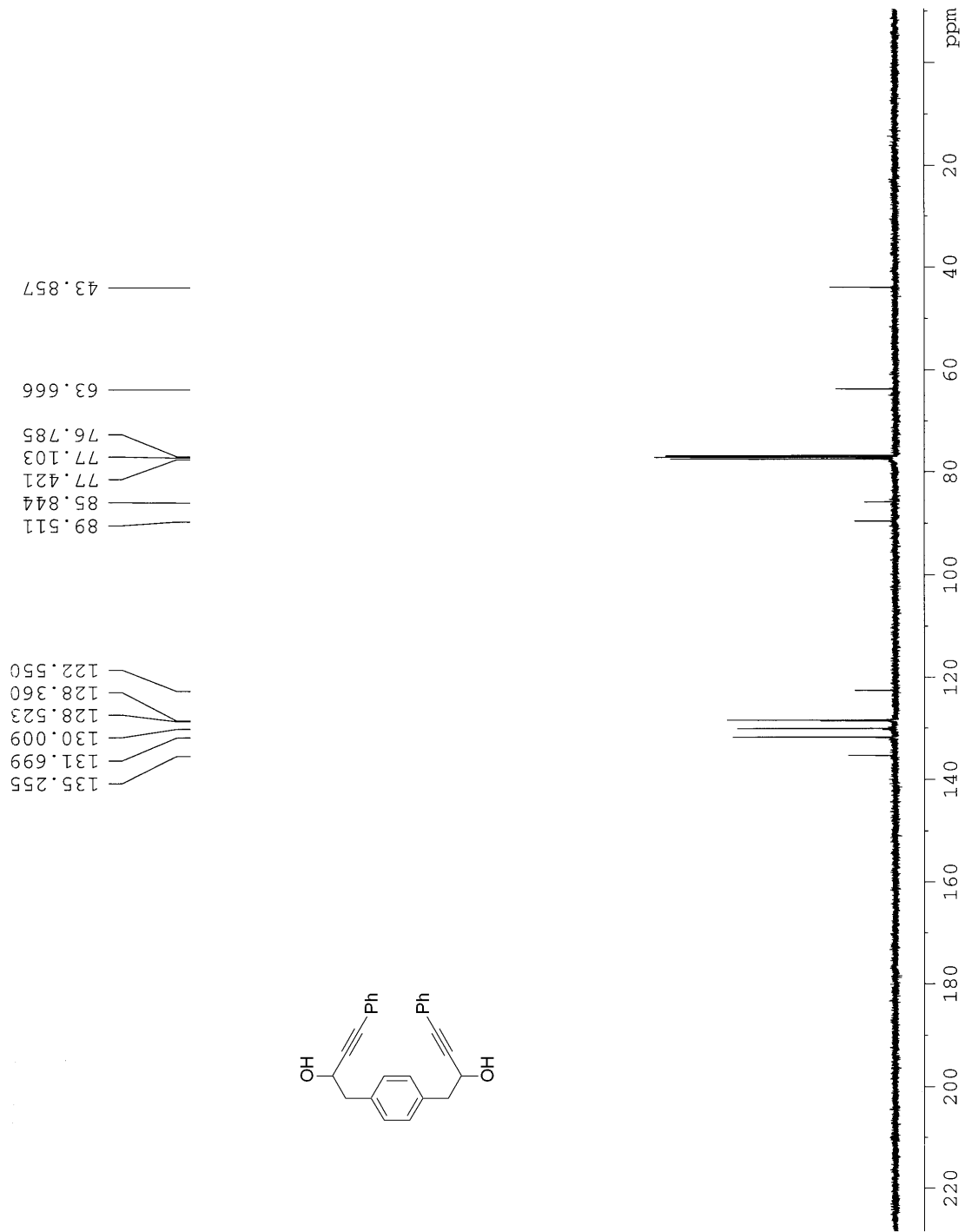


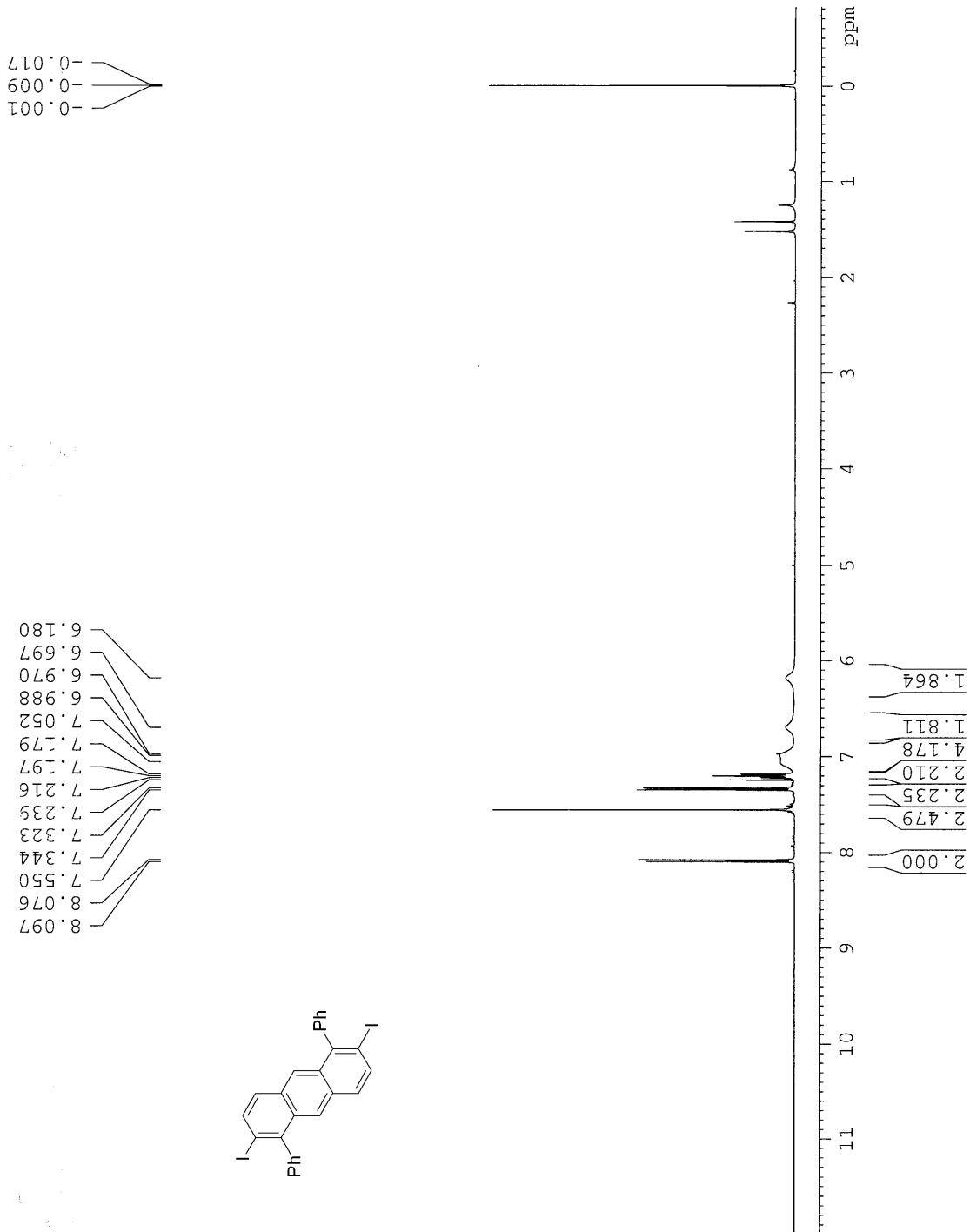


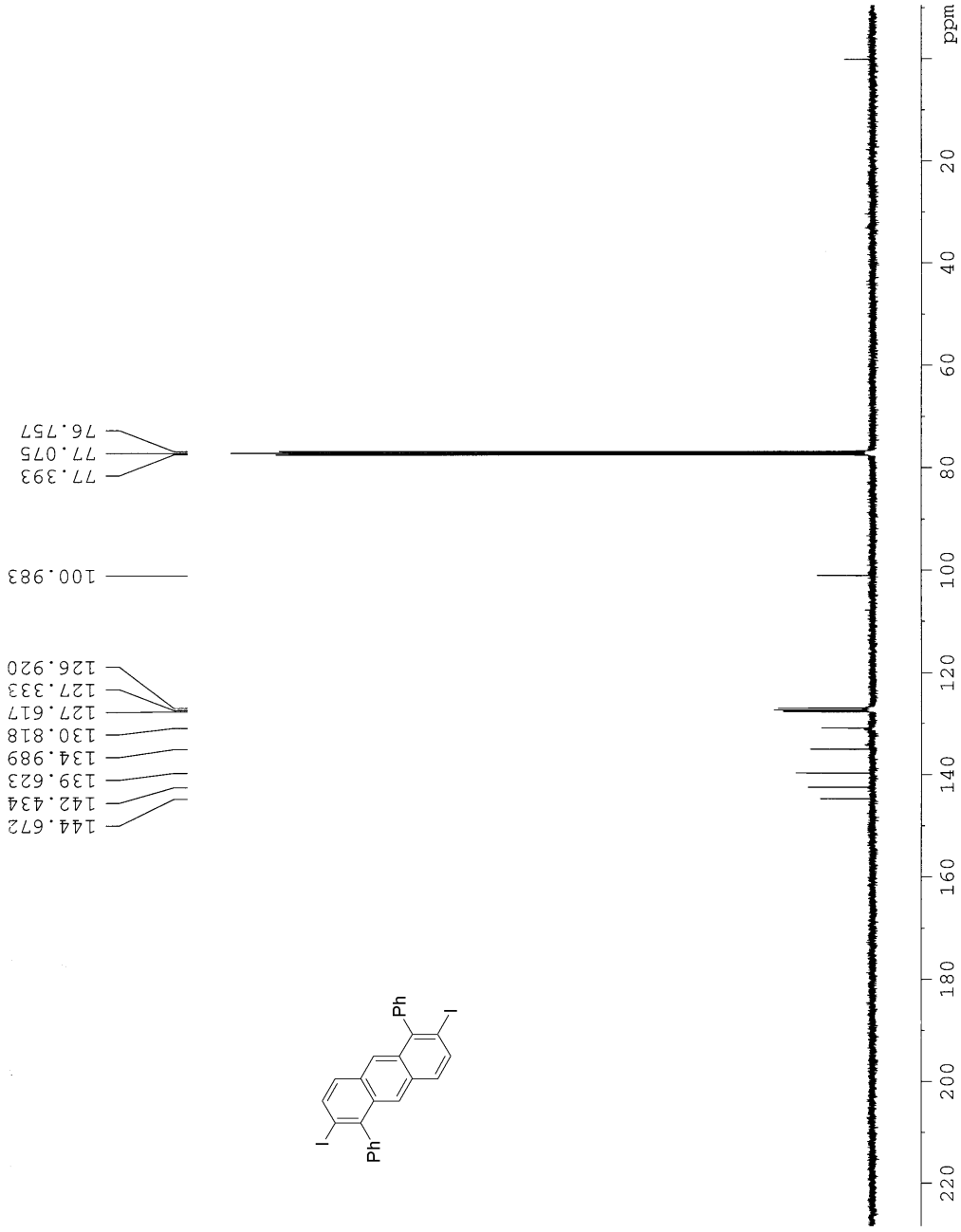
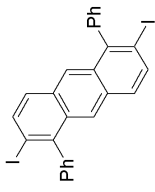
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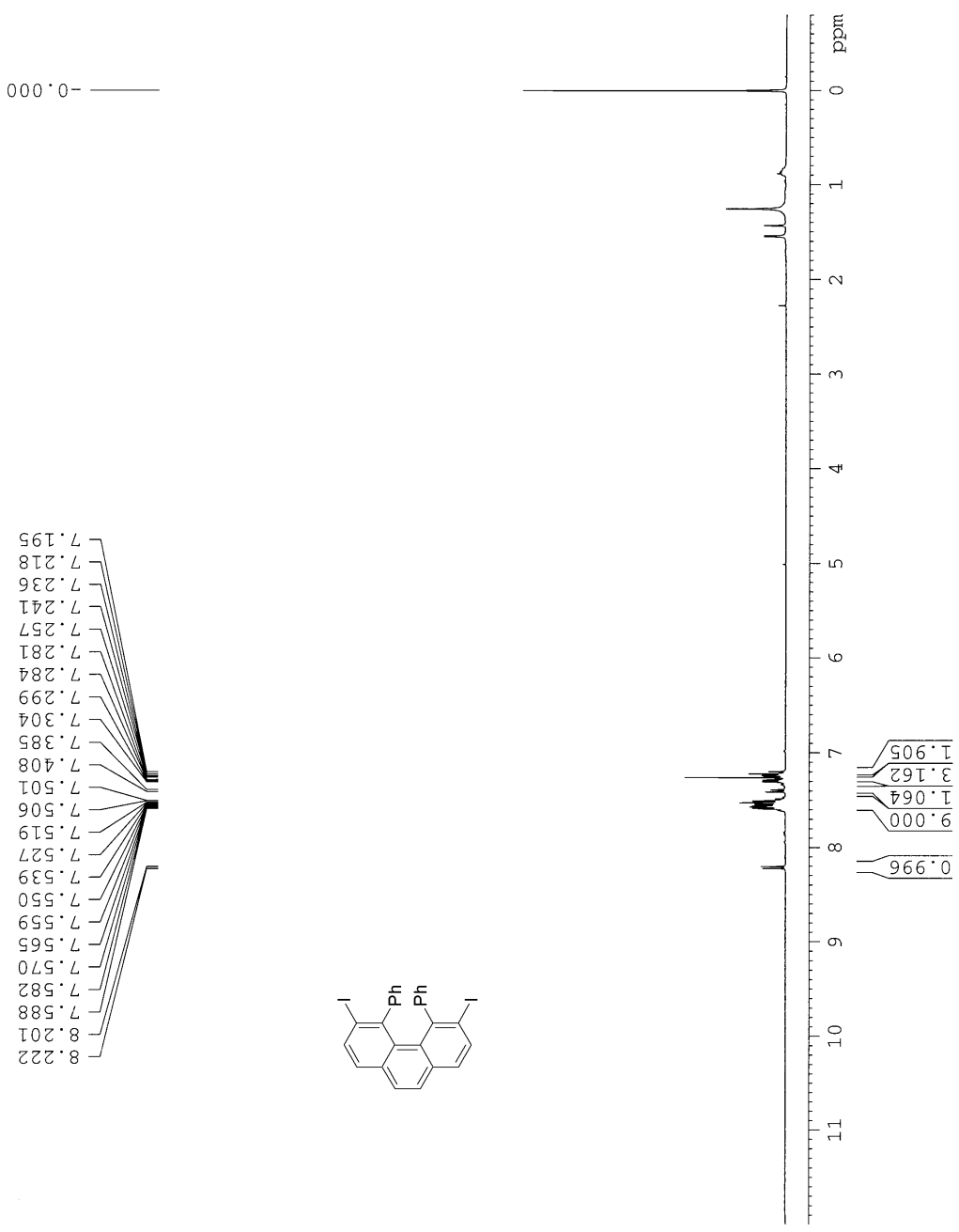


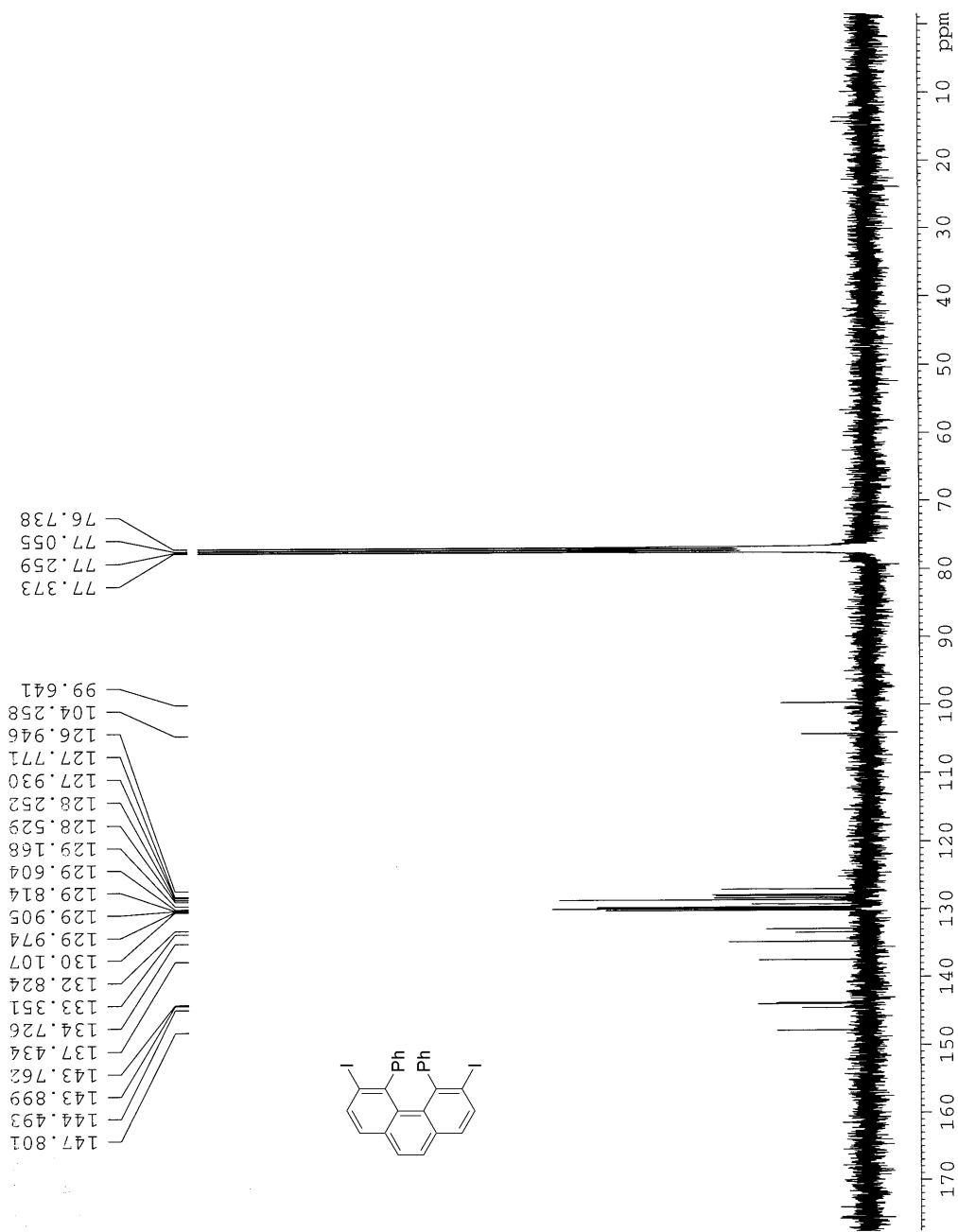


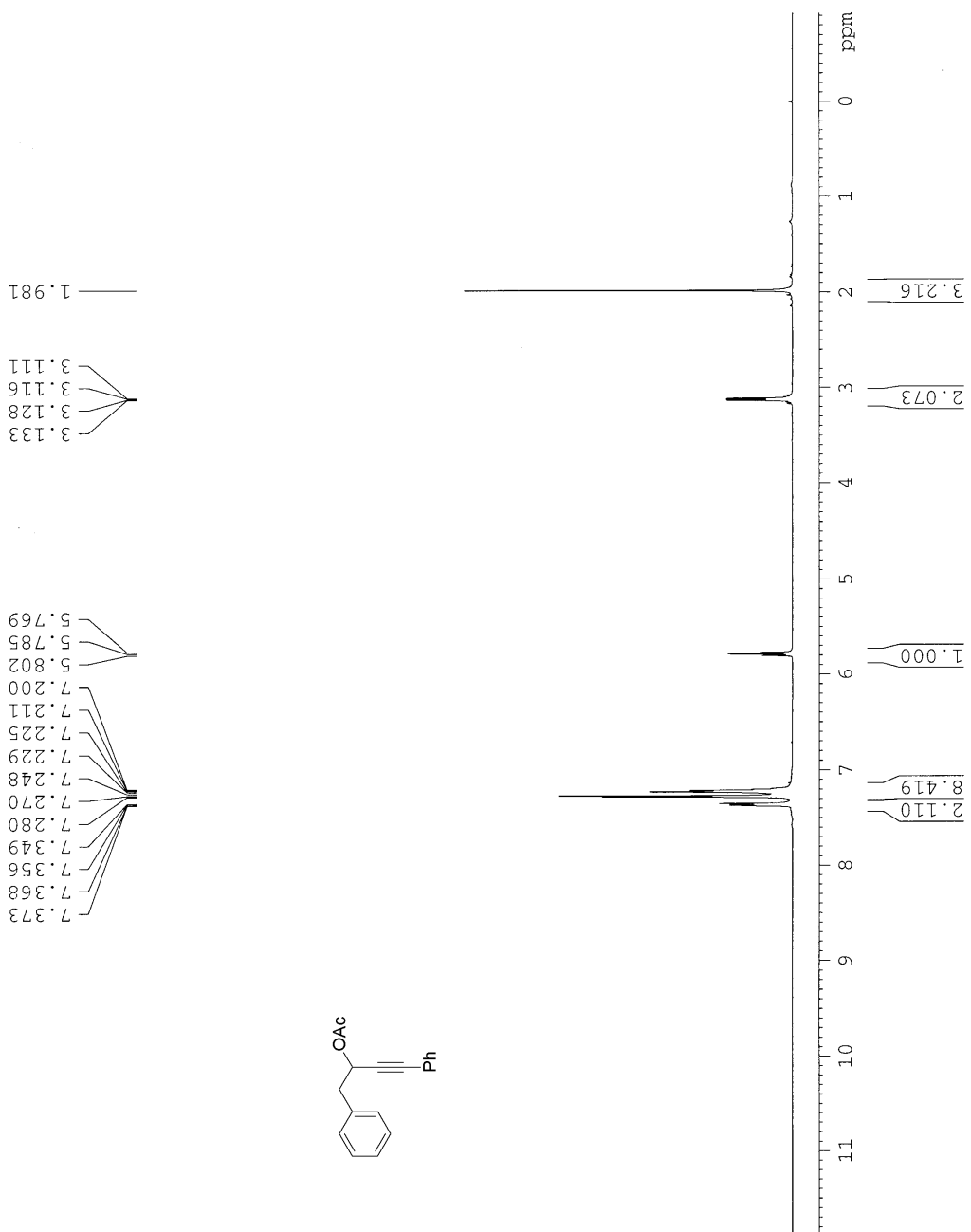


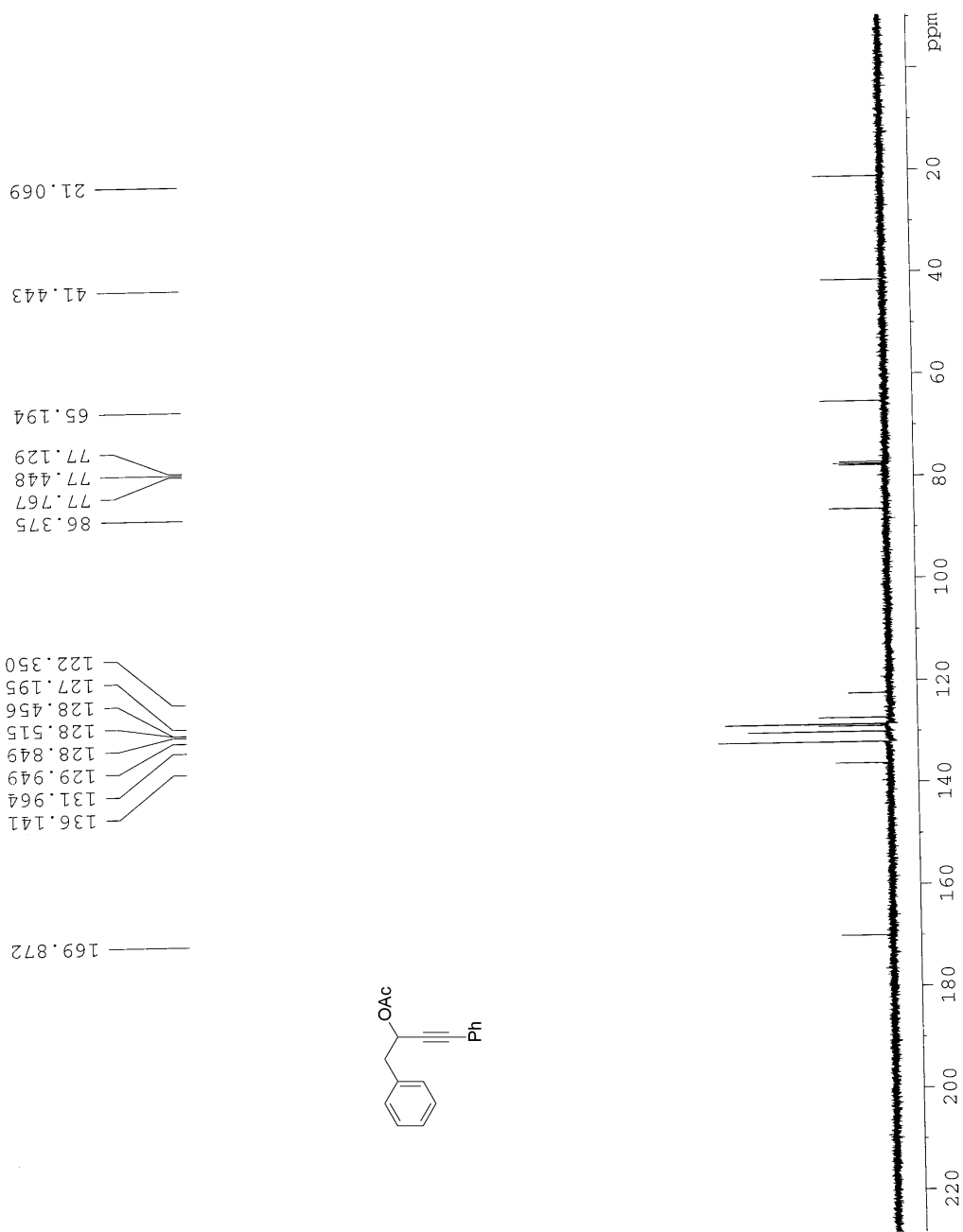


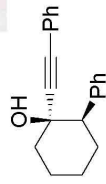




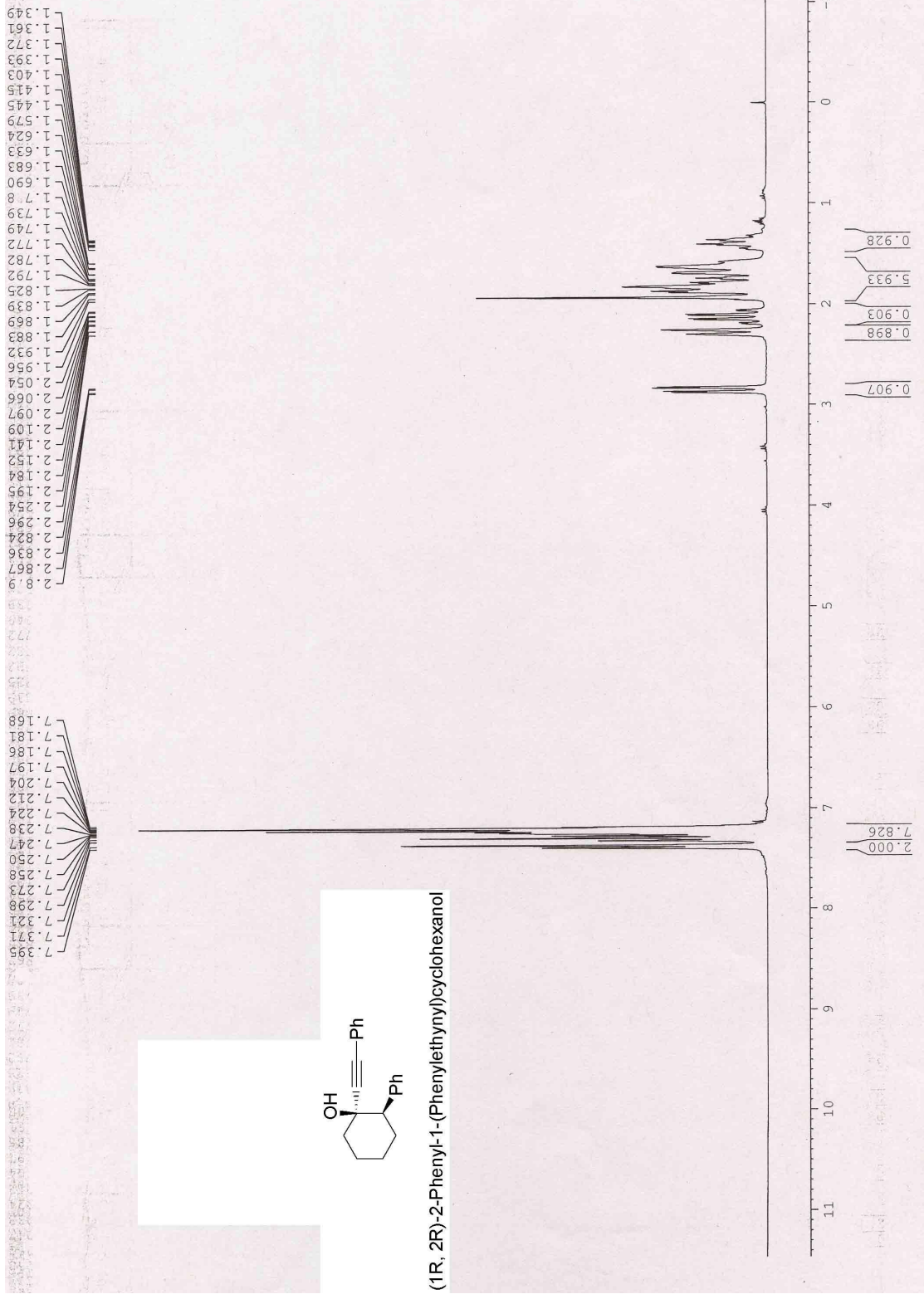


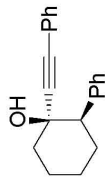




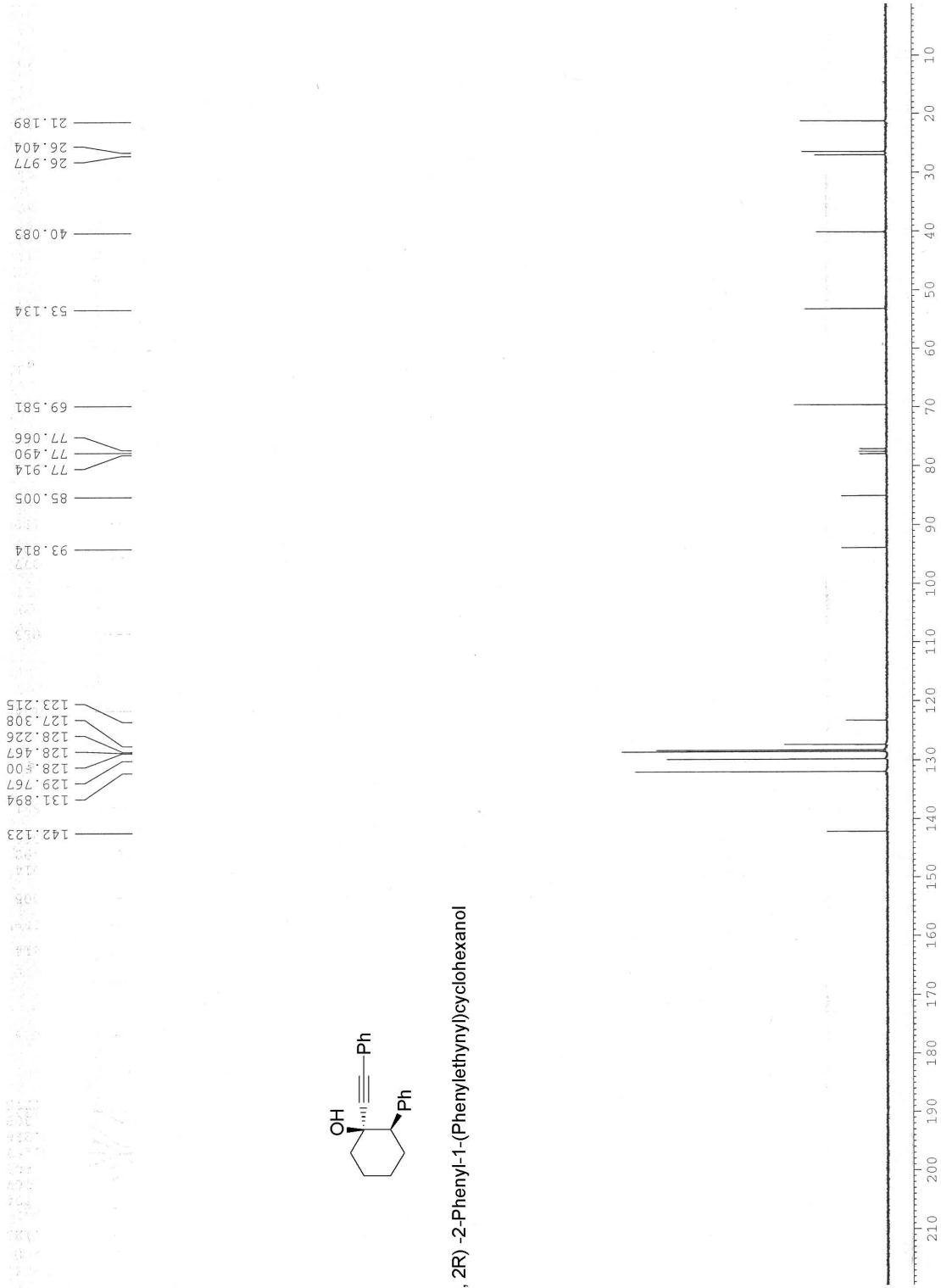


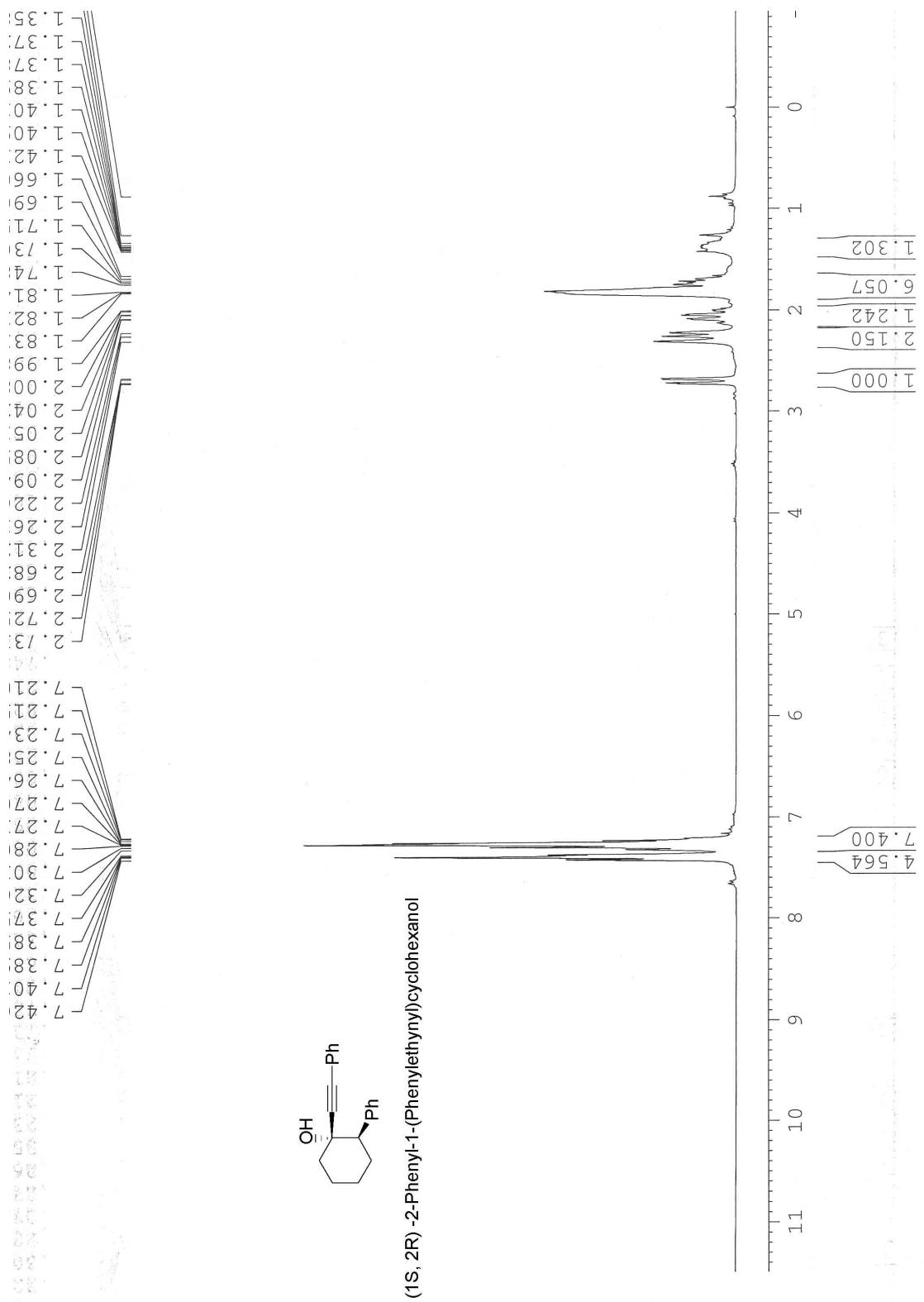
(1R, 2R)-2-Phenyl-1-(Phenylethynyl)cyclohexanol

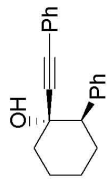




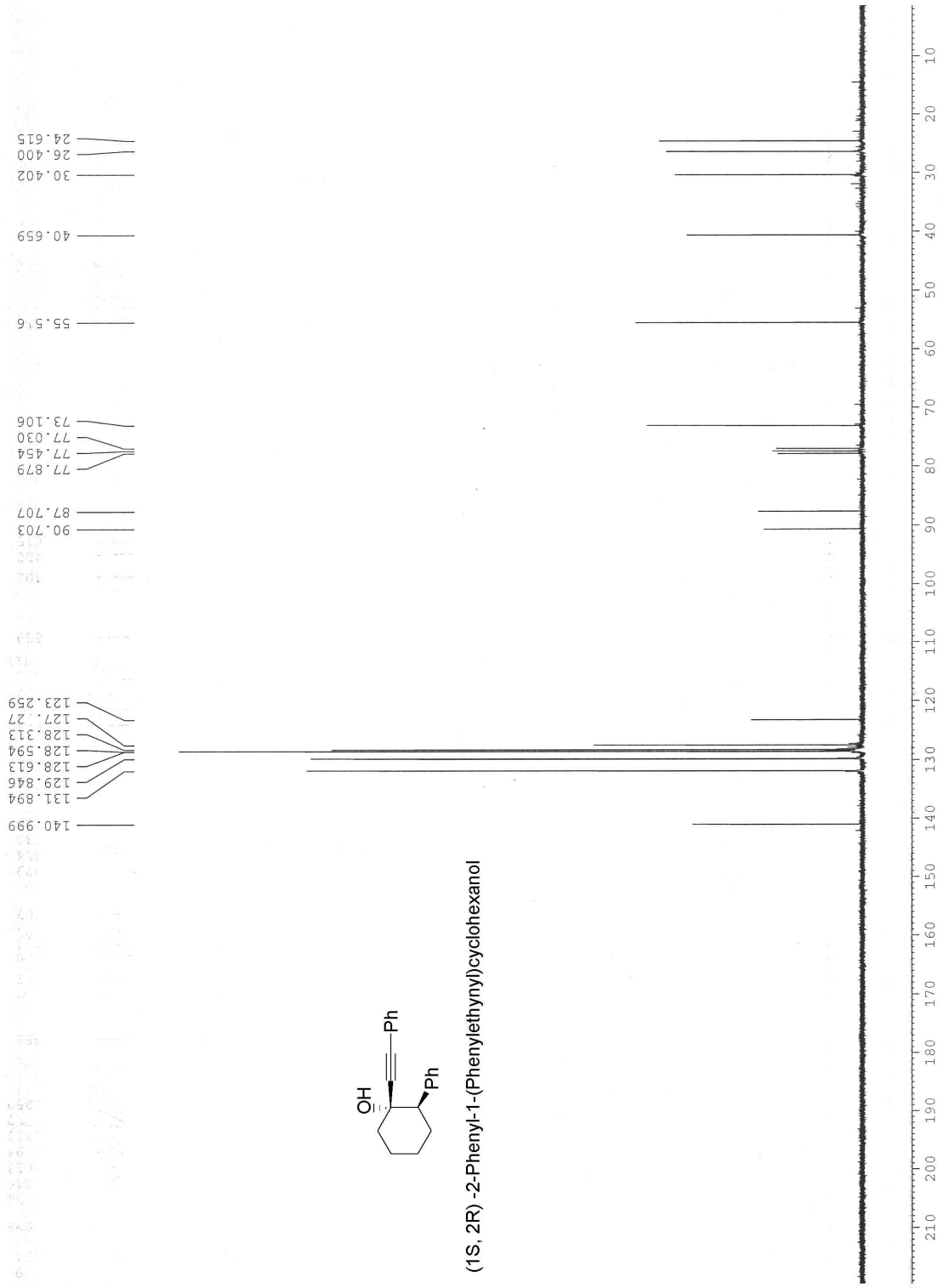
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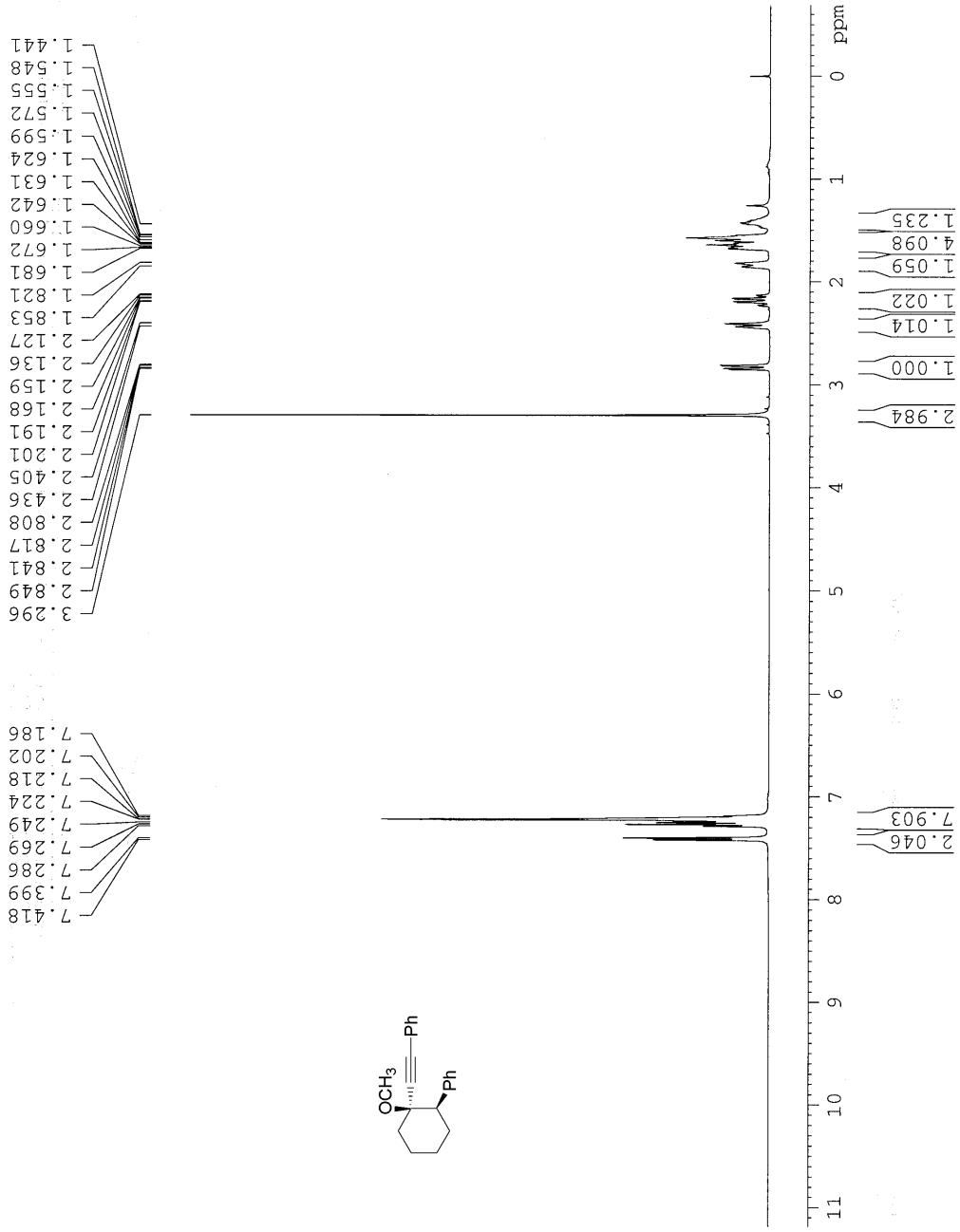
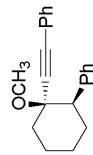


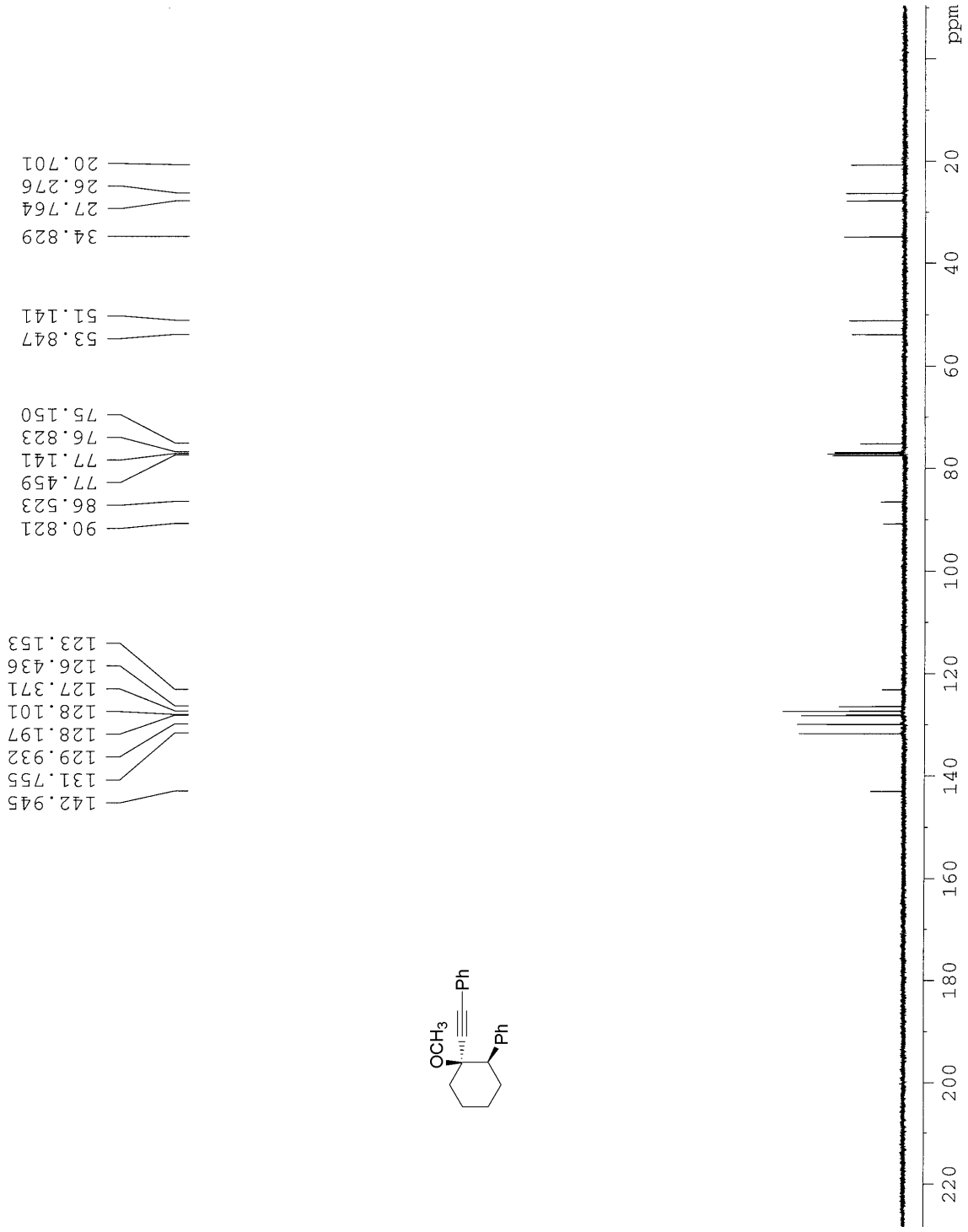
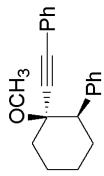


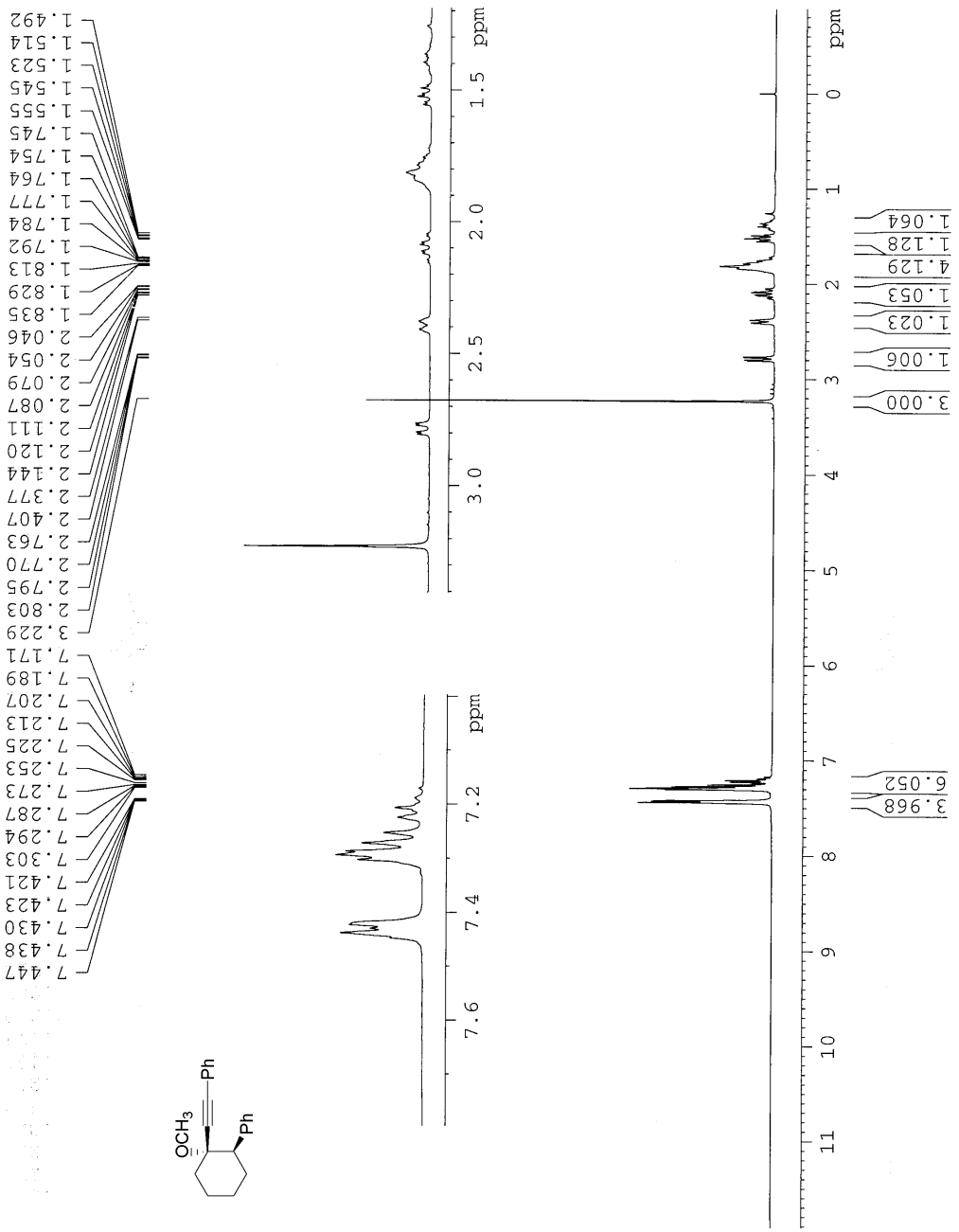


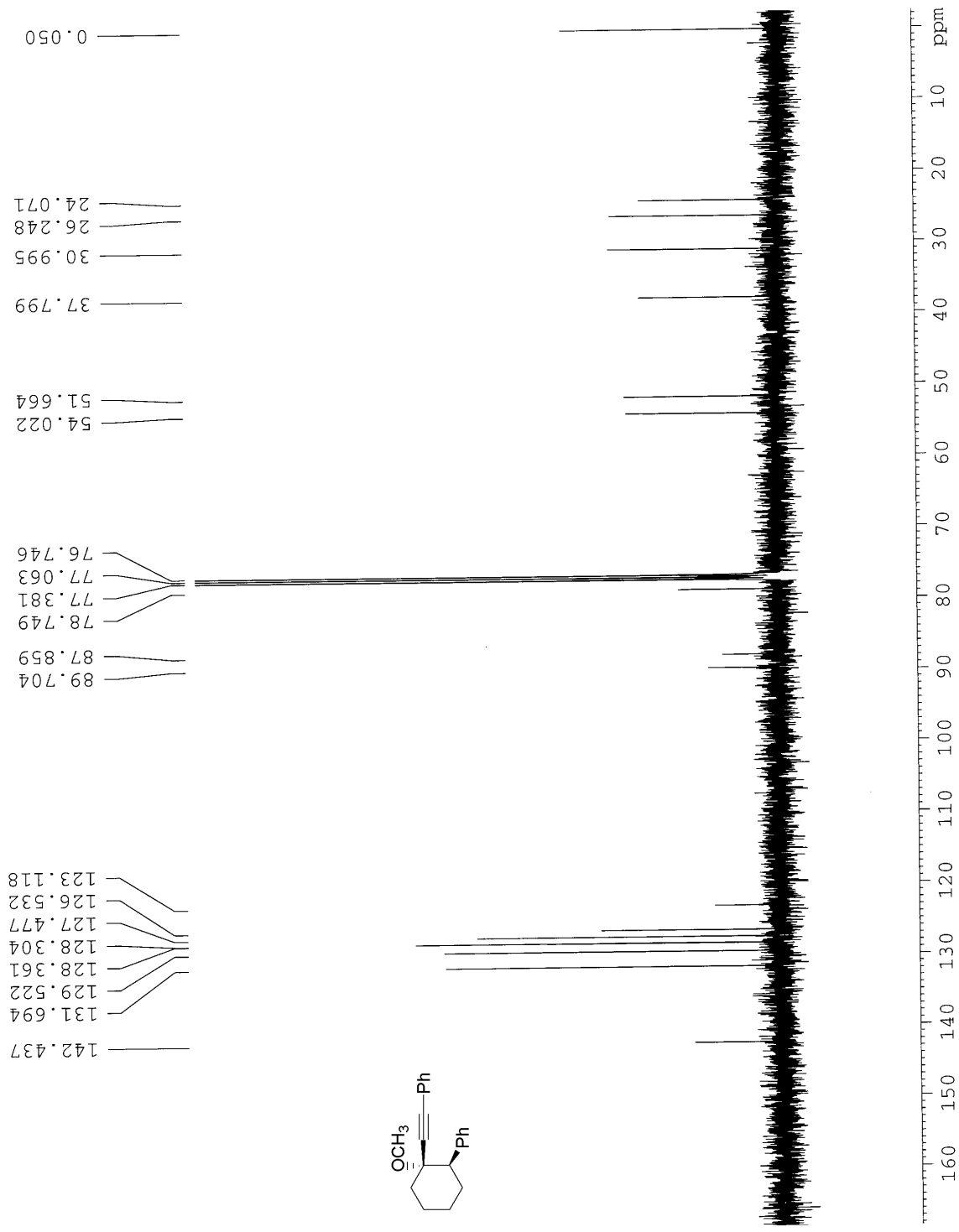
(1S, 2R) -2-Phenyl-1-(Phenylethynyl)cyclohexanol

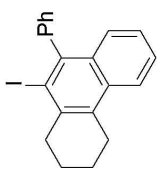
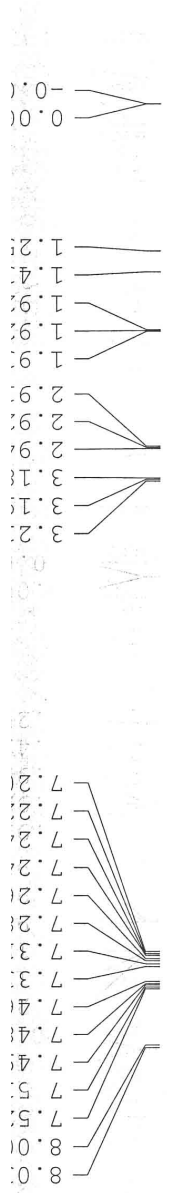




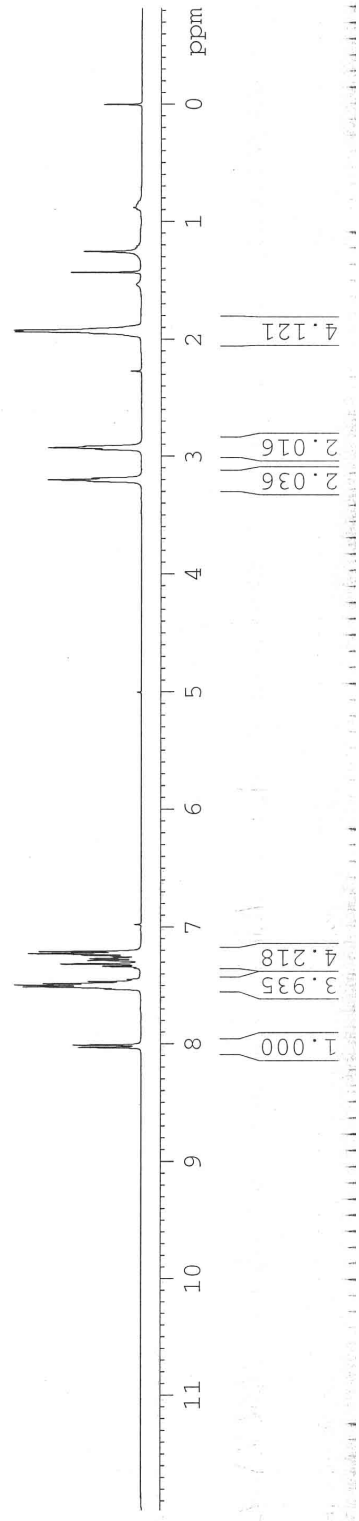


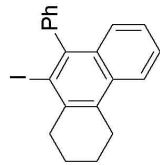




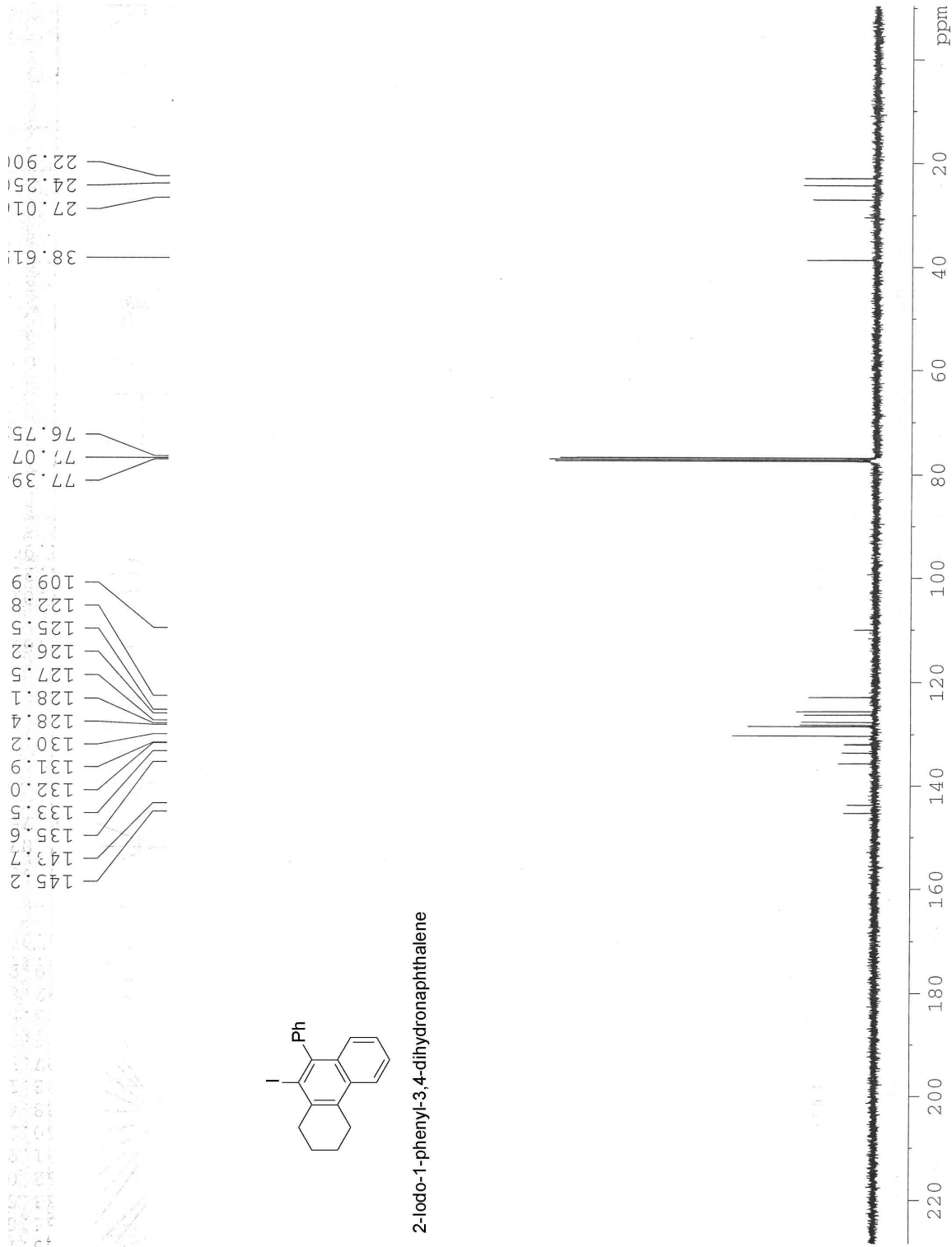


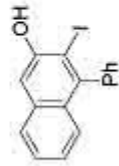
2-iodo-1-phenyl-3,4-dihydronaphthalene



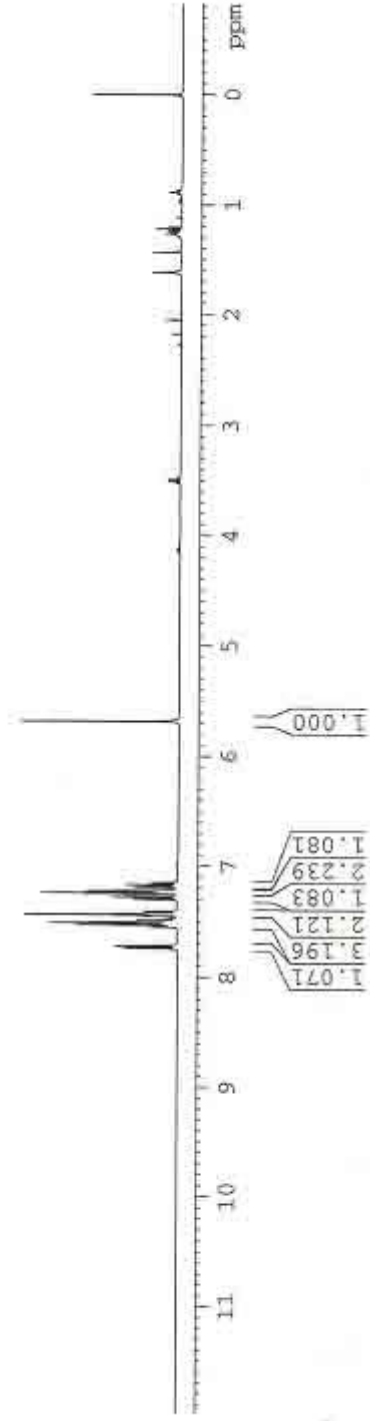
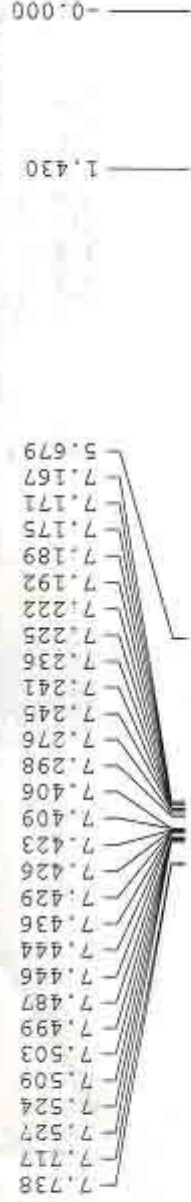


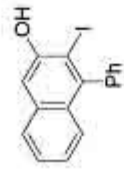
2-iodo-1-phenyl-3,4-dihydronaphthalene



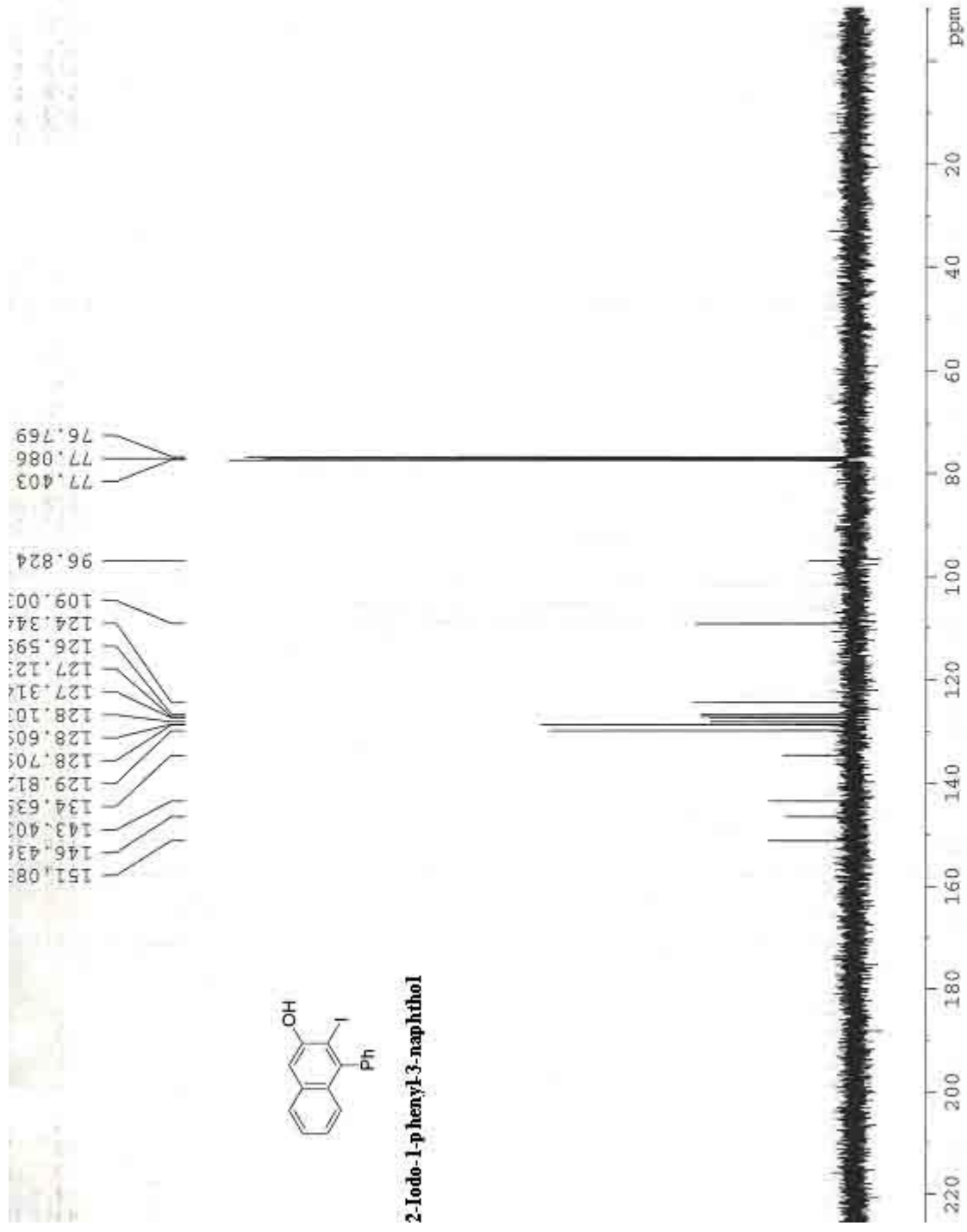


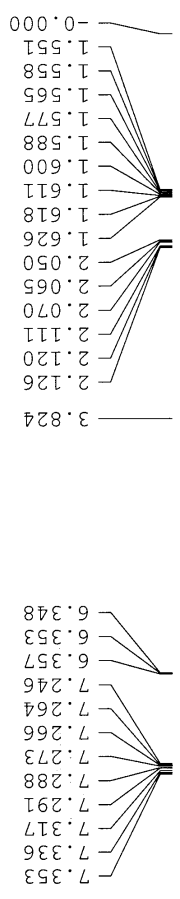
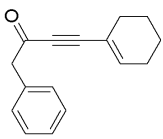
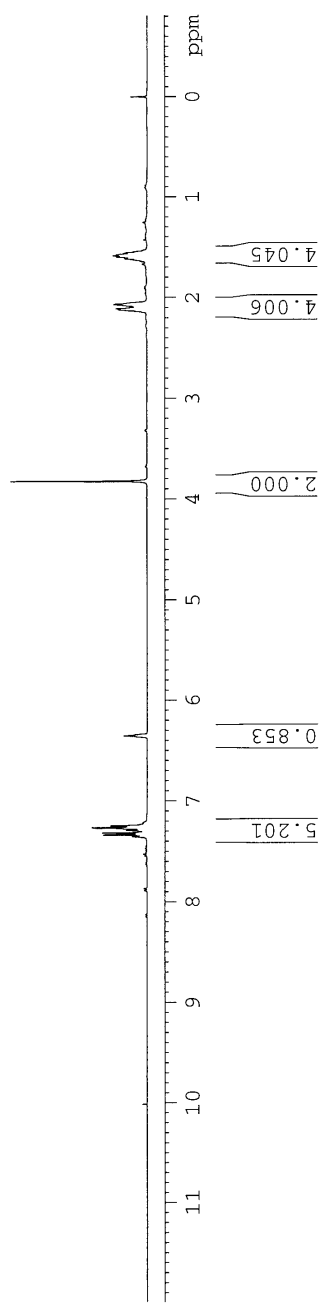
**2-Iodo-1-p-phenyl-3-naphthol**

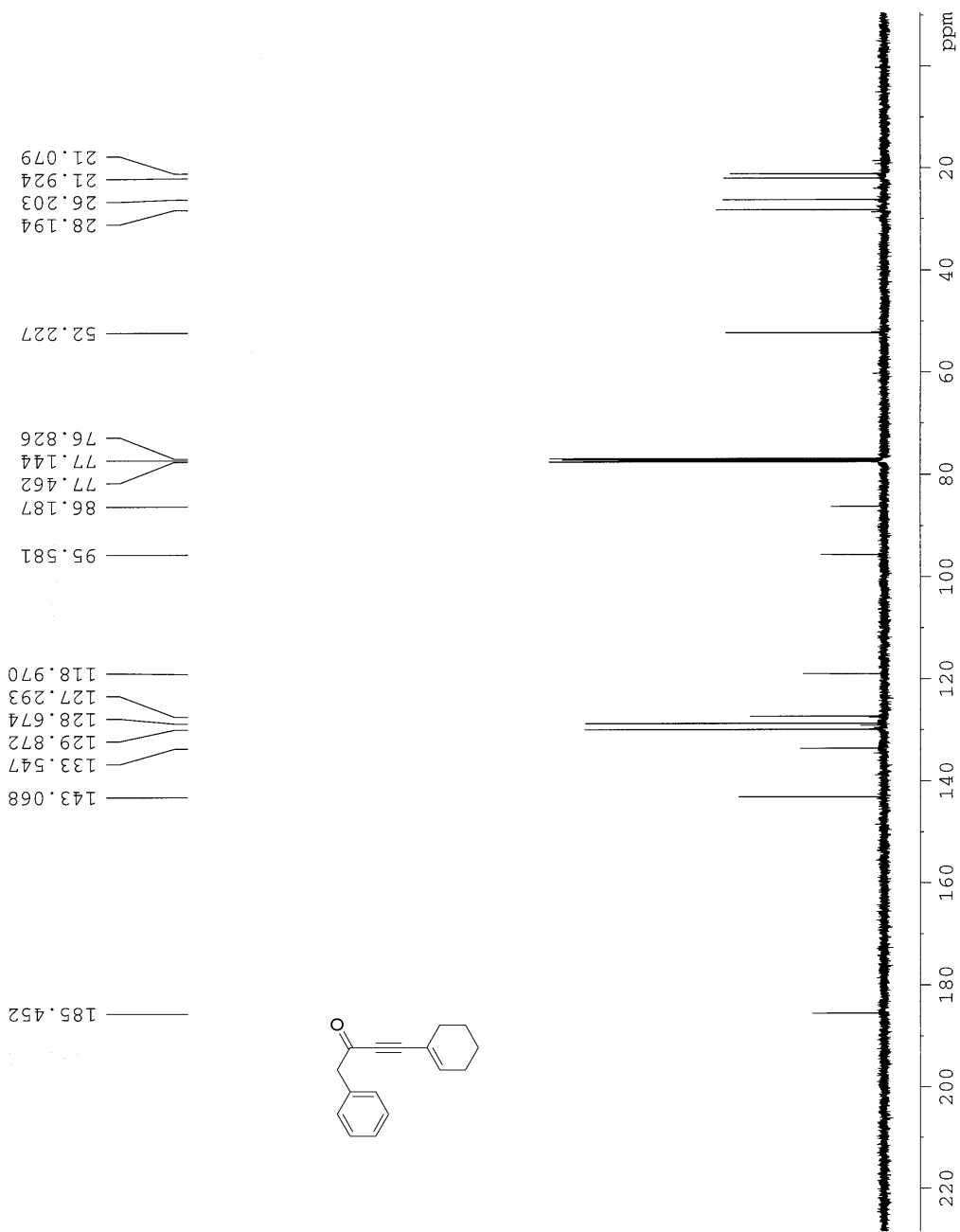


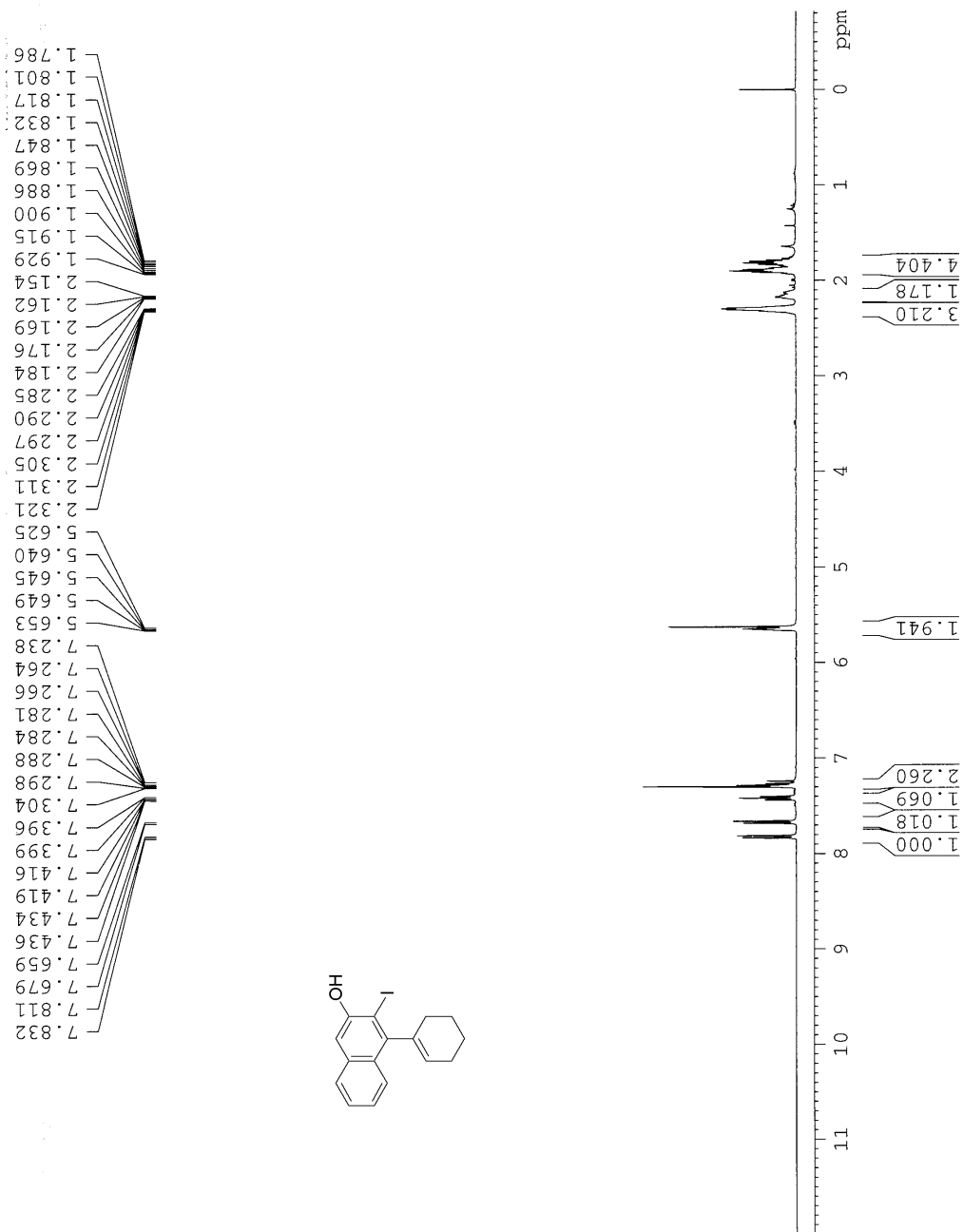


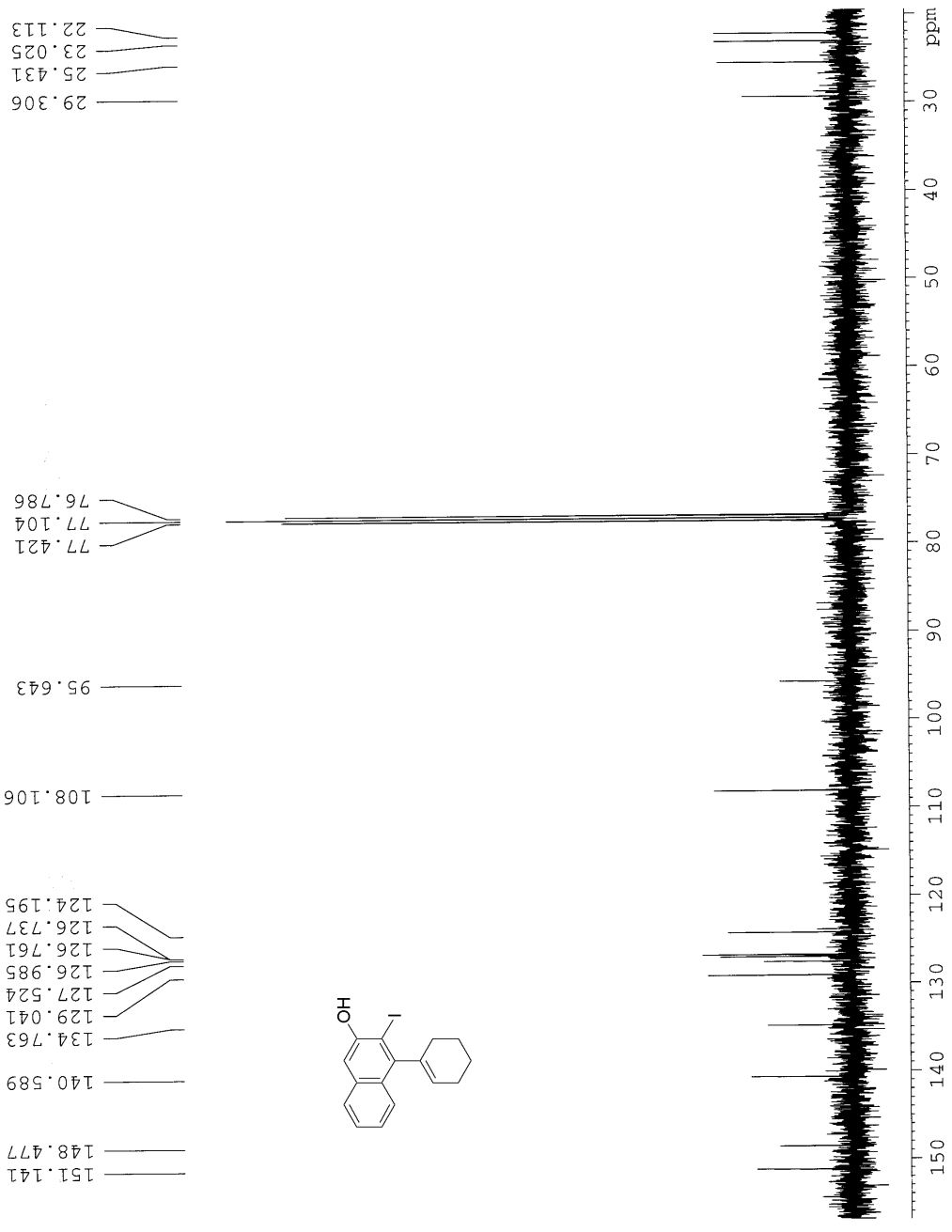
**2-Iodo-1-p henyl-3-naphthol**

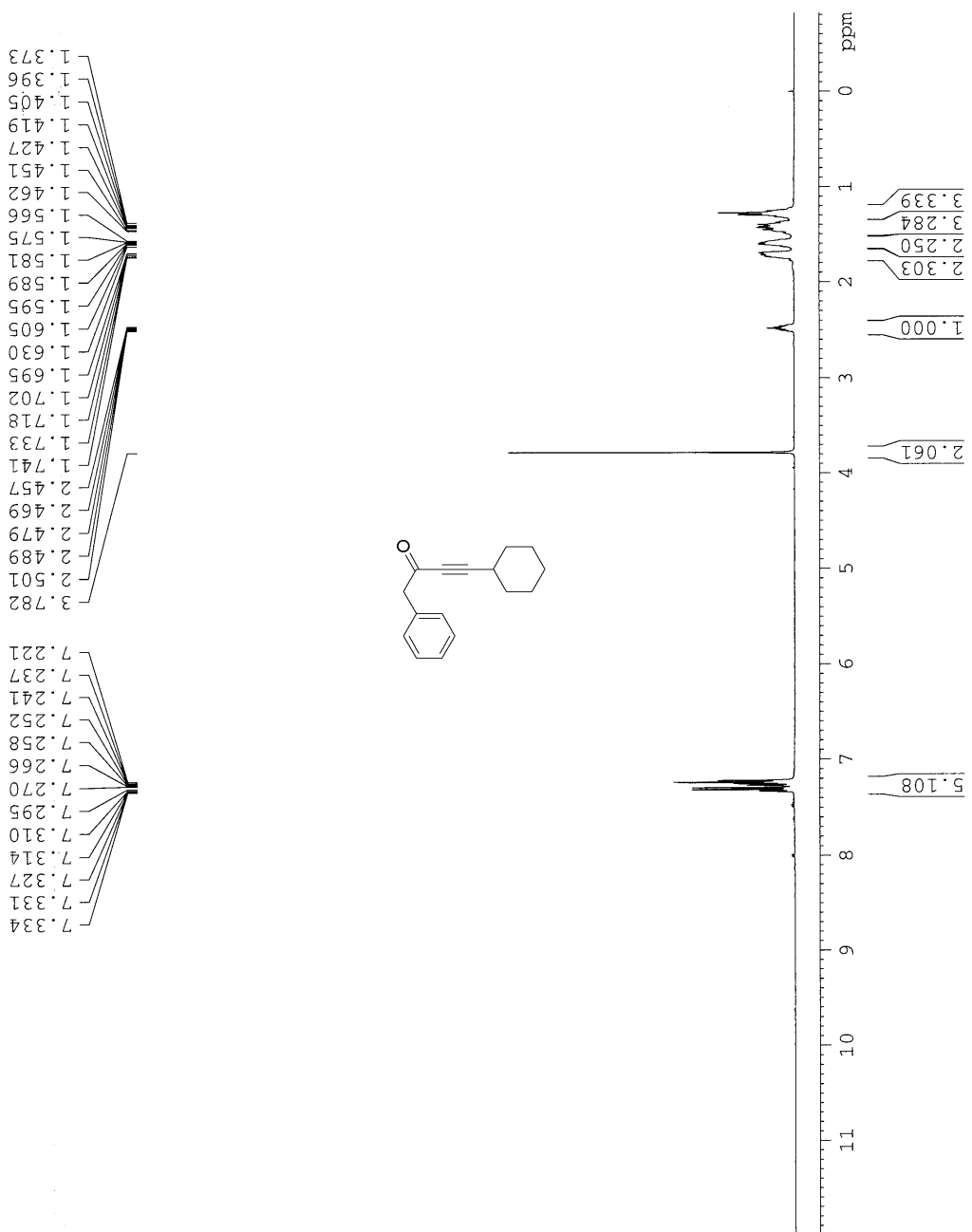


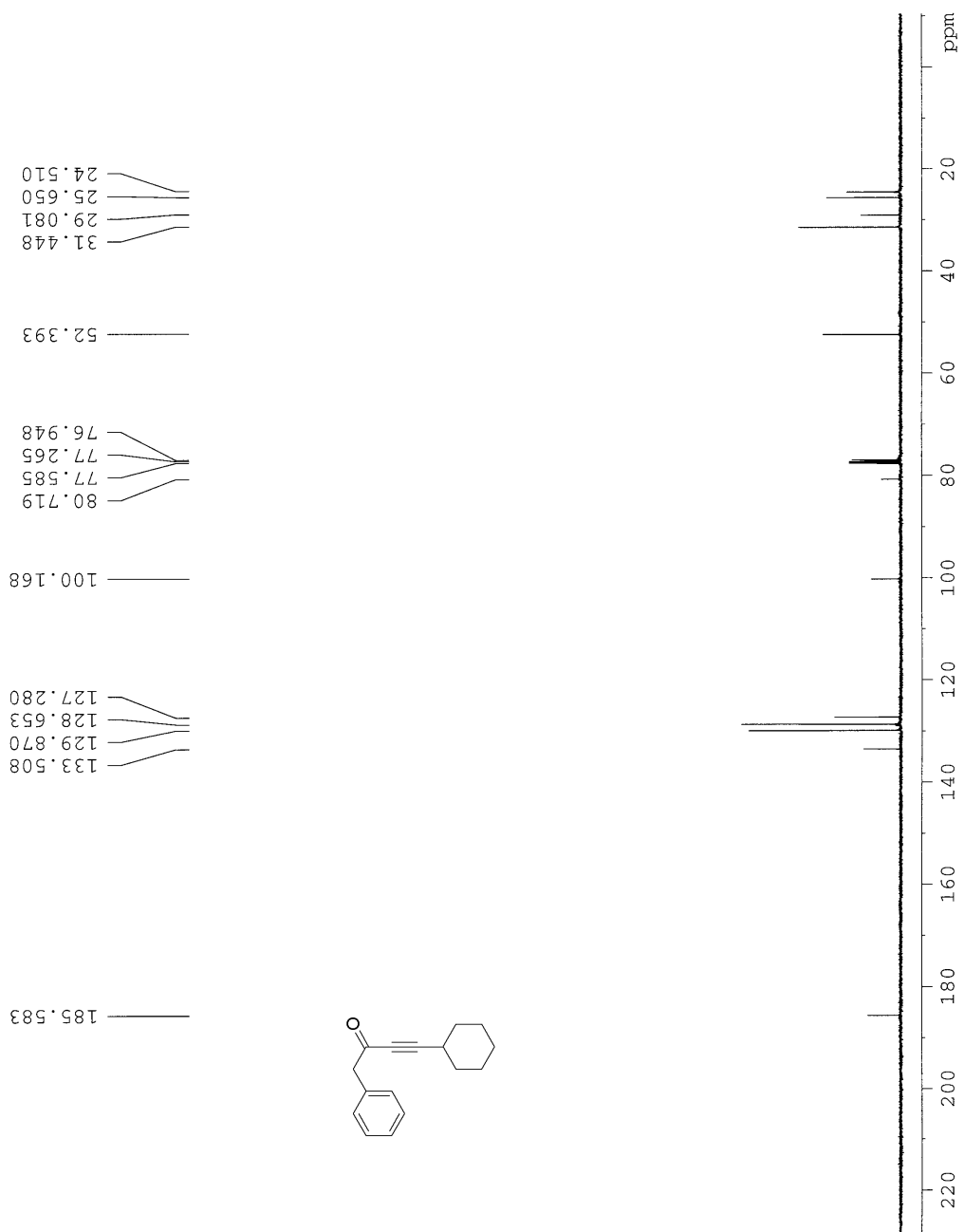


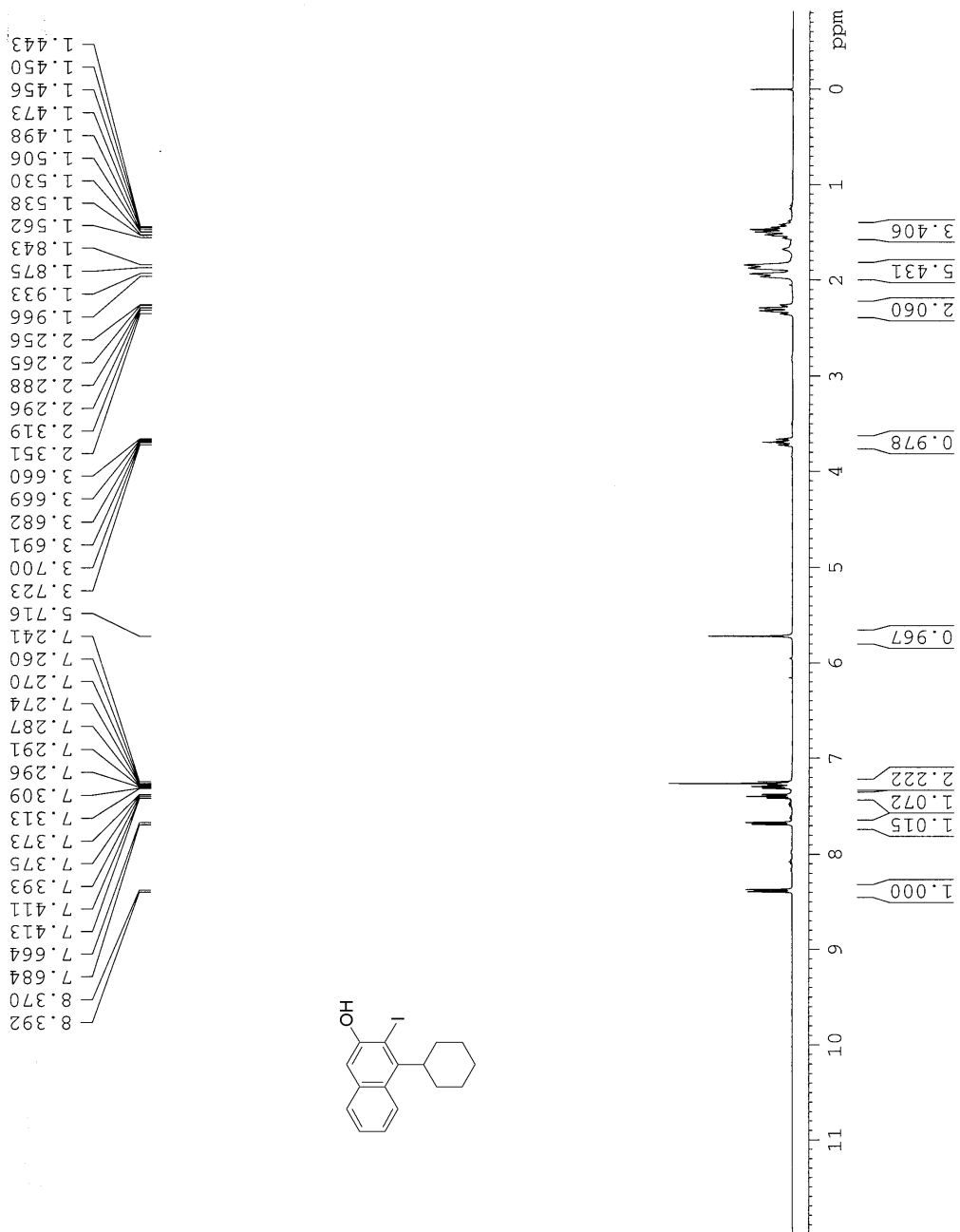


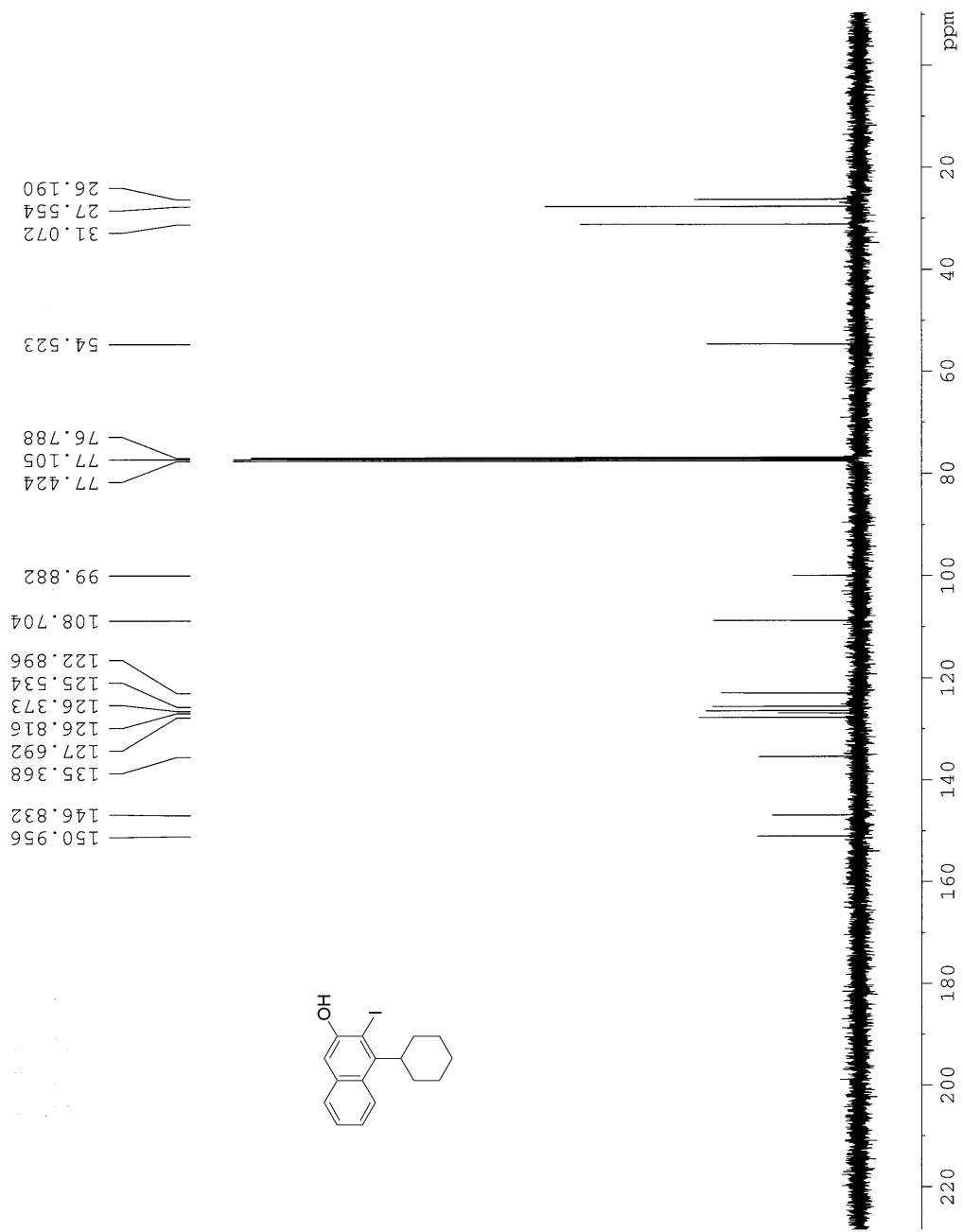


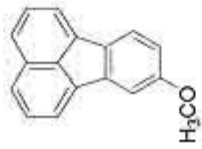




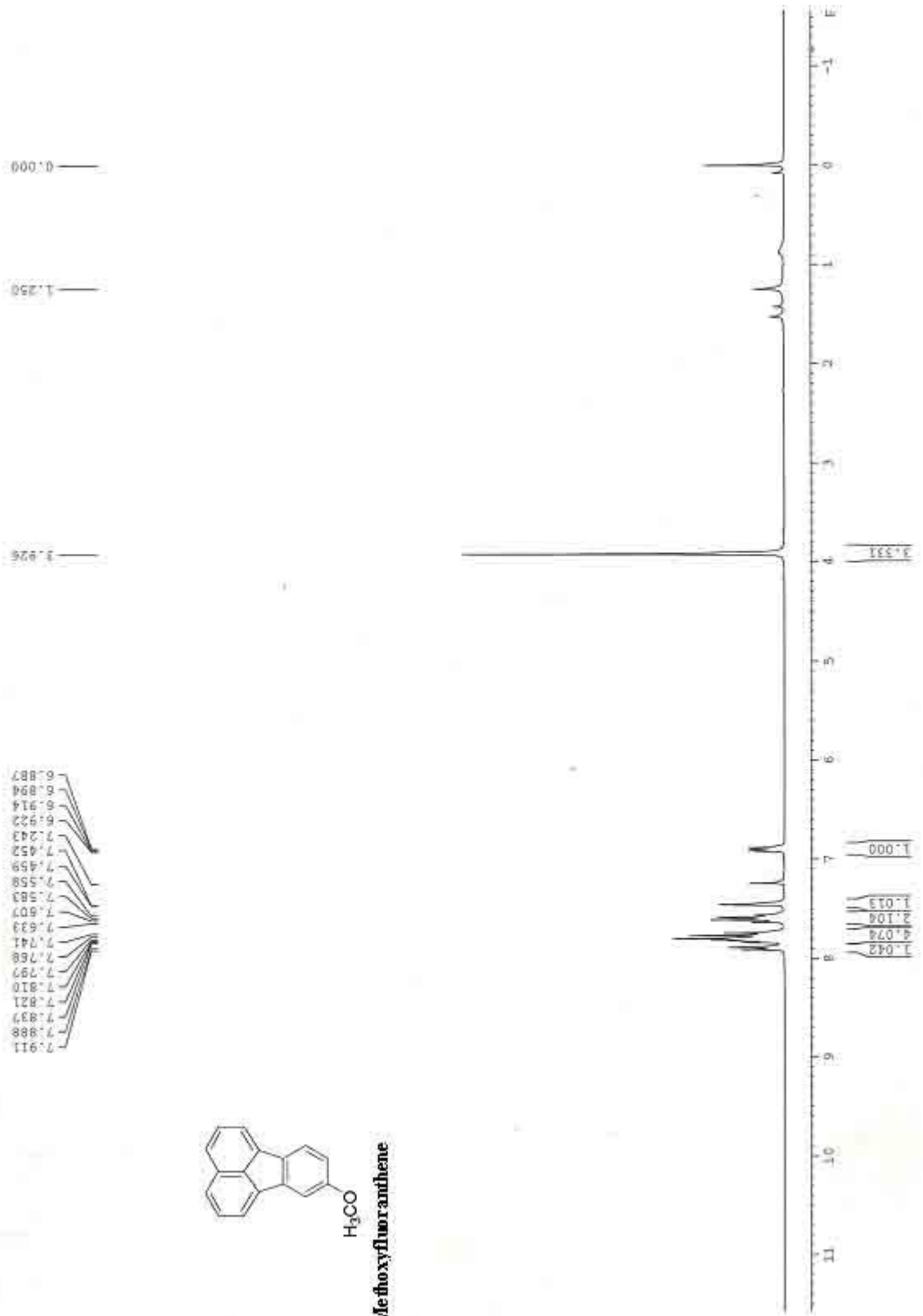


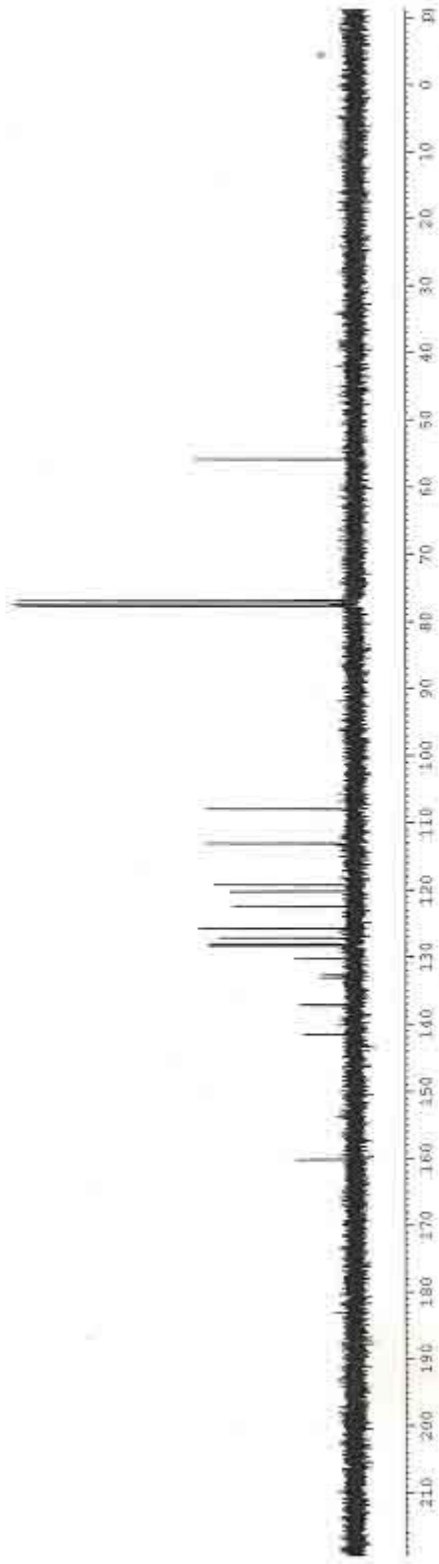
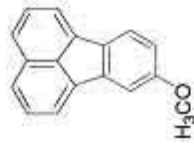


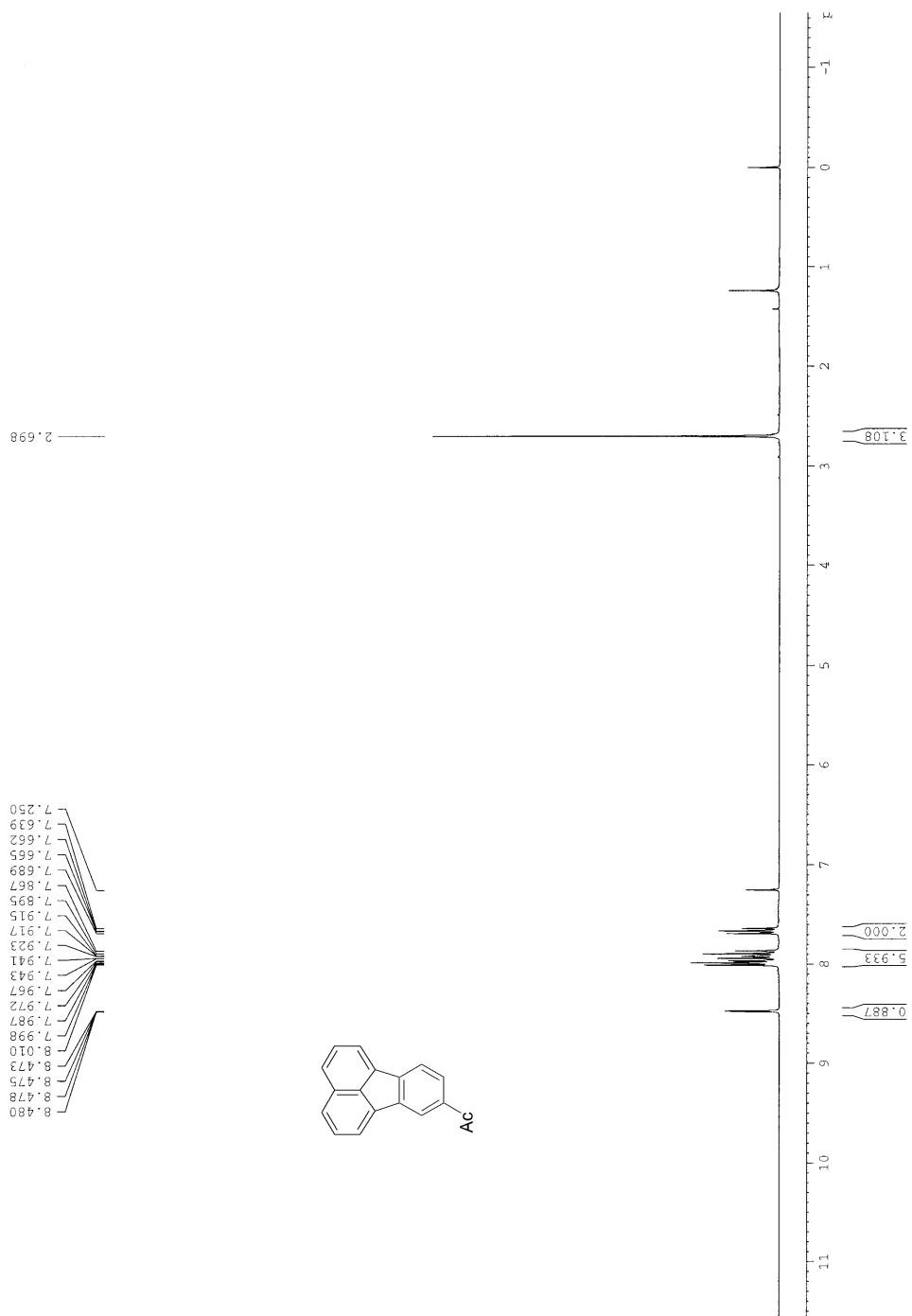


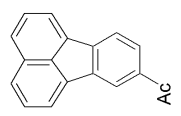
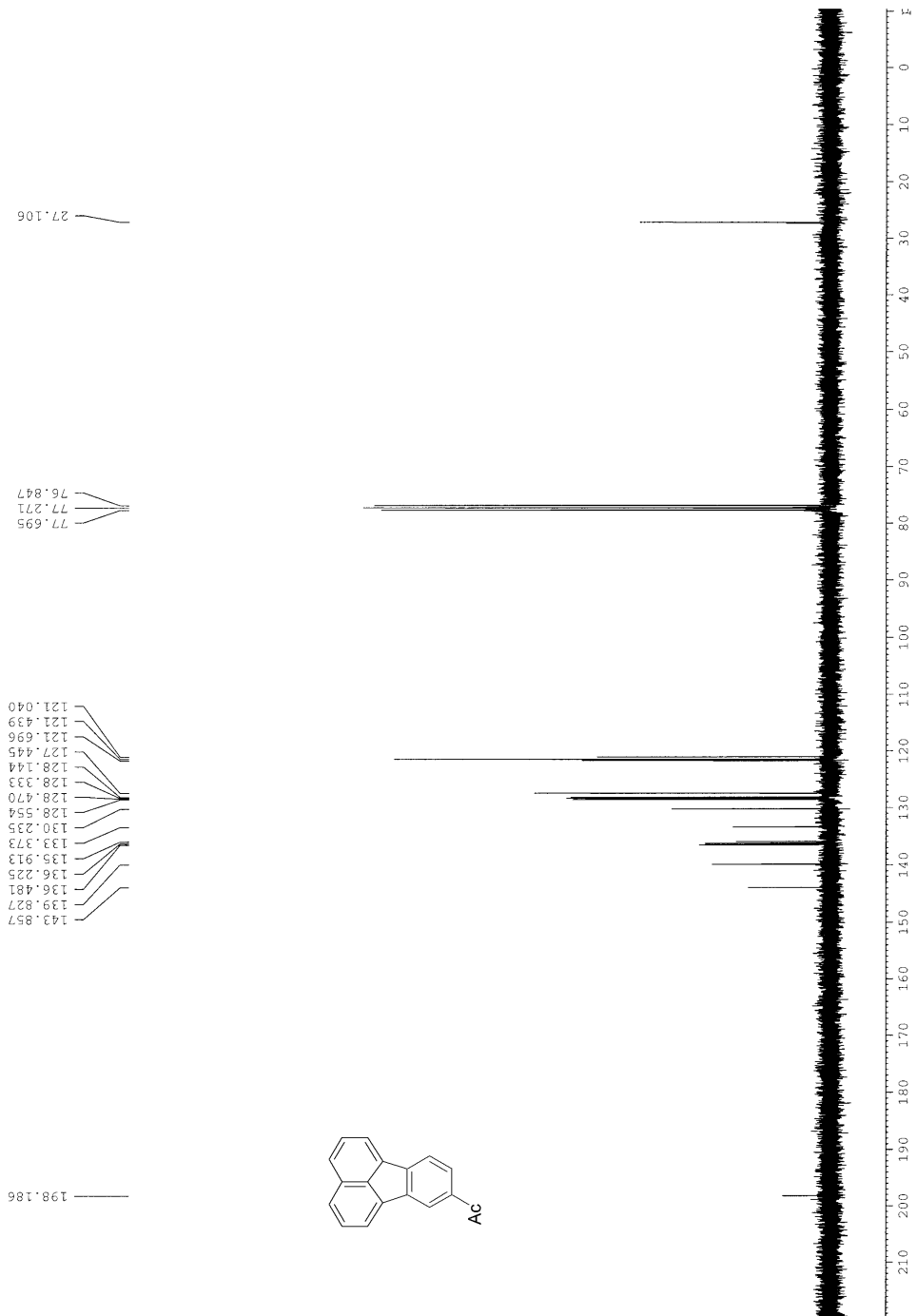


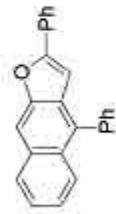
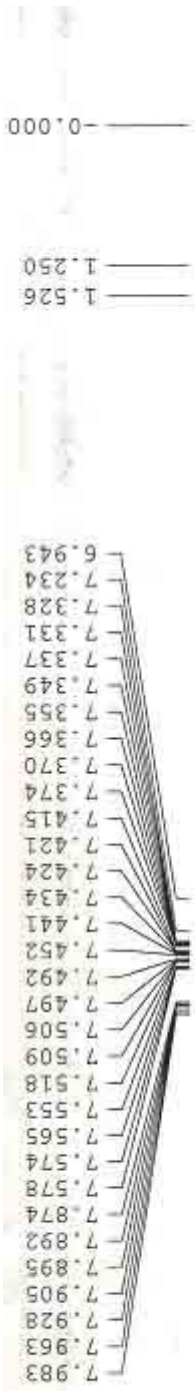
8-Methoxyfluoranthene



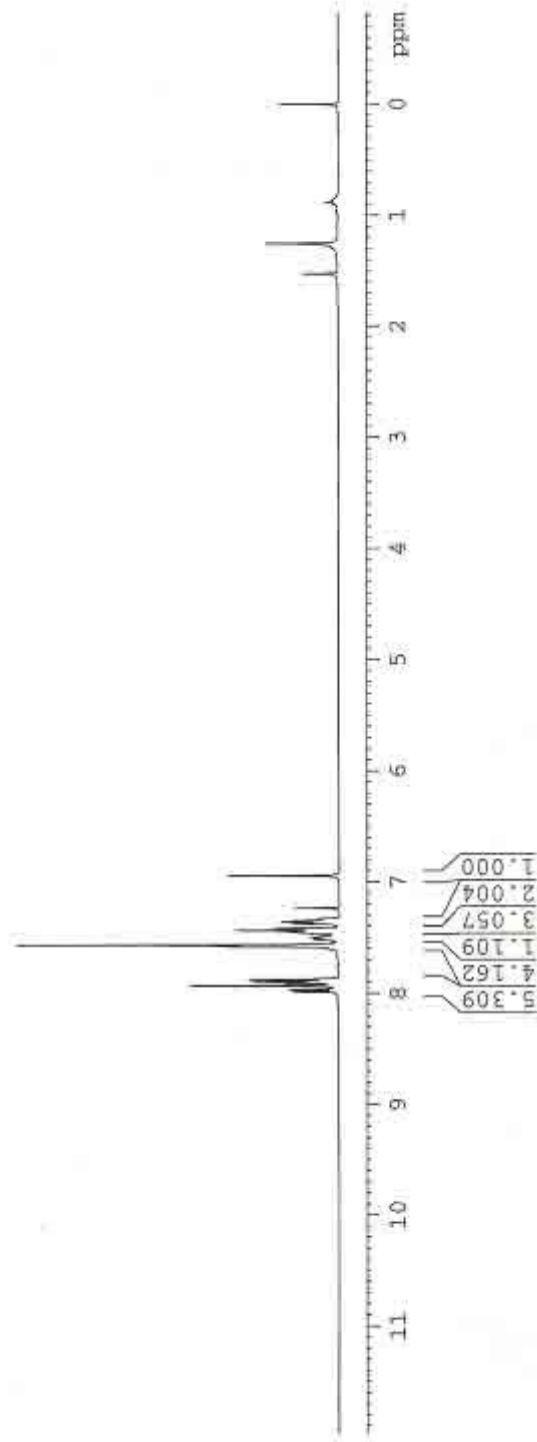


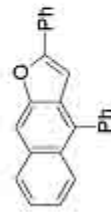






2,4-Diphenylbenzo[2,3-b]furan





2,4-Diphenylbenzo [2,3-b]furan

