Competition Studies in Alkyne Electrophilic Cyclization Reactions

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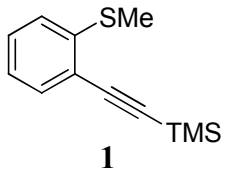
General. The ^{1}H and ^{13}C NMR spectra were recorded at 300 and 75.5 MHz or 400 and 100 MHz respectively. Thin layer chromatography was performed using commercially prepared 60-mesh silica gel plates and visualization was effected with short wavelength UV light (254 nm). DCM was distilled over CaH_2. Anhydrous MeCN was used as received. Purification of the compounds has been performed by column chromatography. All melting points are uncorrected.

Reagents. All reagents were used directly as obtained commercially unless otherwise noted.

Preparation of the non-commercial aryl iodides. 2-Iodophenyl methyl selenide was prepared using a modified literature procedure.[1] In a 100 ml round bottom flask, 8.0 mmol of the 2-iodoaniline was dissolved in 5 ml of HBF_4 (48% solution) and the mixture was stirred for 15 min and then allowed to cool to 0 ^{o}C. To this solution, an aqueous solution of $NaNO_2$ (8.0 mmol in 3 mL of water) was added dropwise to the reaction mixture. The mixture was allowed to warm to room temperature, filtered and washed with cold ethanol. The diazonium salt was dried under vacuum and used for the next step without purification. A suspension of 8.0 mmol of the crude diazonium salt in 25 mL of $CHCl_3$ containing 10 mol % of 18-crown-6 and 9.0 mmol of dimethyl diselenide was stirred at 0 ^{o}C. To this mixture, 16 mmol of KOAc was added in small portions over a period of 10 min and the resulting solution was allowed to stir for 4 h and then filtered. The solid residue was washed with chloroform and the resulting filtrate was washed with water (2 x 5 mL), dried over anhydrous $MgSO_4$, and concentrated under vacuum. The crude product obtained was then purified by flash chromatography on silica gel using ethyl acetate/hexanes as the eluent. The product was obtained as a yellow oil (yield =

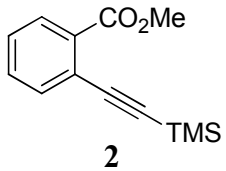
35%) with spectral properties identical to those previously reported.[1] *N*-Phenyl-2-iodobenzamide[2] and *N,N*-dimethyl-2-iodoaniline[3] were prepared according to literature procedures.

Preparation of the TMS-protected alkynes.



1

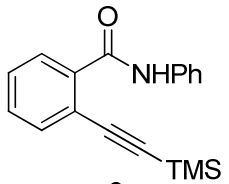
***o*-(Trimethylsilylethynyl)thioanisole (1).** This compound was prepared according to a modified literature procedure.[4] To a solution of *o*-iodothioanisole (5.0 mmol), $PdCl_2(PPh_3)_2$ (0.07 g, 2 mol %), and CuI (0.01 g, 1 mol %) in TEA (20 mL) (stirring for 5 min beforehand), 6.0 mmol of trimethylsilylacetylene (1.2 equiv) in 5 mL of TEA was added dropwise over 15 min. The reaction flask was flushed with argon and the mixture was stirred at room temperature for 20 h. The resulting solution was filtered, washed with brine, and extracted with diethyl ether (2 x 10 mL). The combined ether fractions were dried over Na_2SO_4 and concentrated under vacuum to yield the crude product. Purification by flash chromatography afforded the product (yield = 97%) as a yellow oil with spectral properties identical to those previously reported.[4]



2

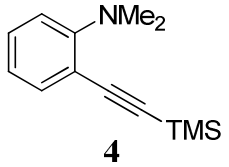
Methyl 2-(trimethylsilylethynyl)benzoate (2). This compound was prepared using the procedure (reaction time: 15 h) used for compound **1**. After flash chromatography, the

compound was isolated as a colorless oil (yield = 100%) with spectral properties identical to those previously reported.[5]



3

***N*-Phenyl 2-(trimethylsilylethynyl)benzamide (3).** To a solution of the iodobenzamide (5.0 mmol) in DMF (20 ml) were added $PdCl_2(PPh_3)_2$ (3 mol %), CuI (2 mol%) and DIPA (4 equiv). A solution of trimethylsilylacetylene (1.3 equiv) in 5 ml of DMF was added dropwise and the resulting mixture was then heated under an N_2 atm at 65 ^oC. After 4 h of stirring, the mixture was allowed to cool to room temperature and 25 ml of satd aq NH_4Cl and 25 ml of ethyl acetate were added. The organic layer was separated and the aqueous layer was back extracted with ethyl acetate. The combined organic layers were washed with brine and water and dried over anhydrous $MgSO_4$. The solvent was removed under reduced pressure and the residue was purified by column chromatography on silica gel to afford the product (80%) as a colorless solid with spectral properties identical to those previously reported:[6] mp 96-97 ºC (lit. mp[6] 95-96 ºC).

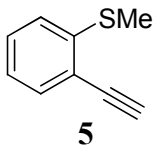


4

***N,N*-Dimethyl-2-(trimethylsilylethynyl)aniline (4).** This compound was prepared according to a literature procedure.[7] Purification by flash chromatography afforded the

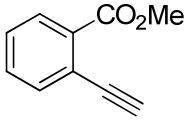
product as a yellow oil (yield = 74%) with spectral properties identical to those previously reported.[7]

Preparation of the terminal alkynes.



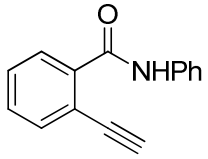
5

2-Ethynylthioanisole (5). A modified literature procedure[8] was used to prepare this compound. A solution of KOH (0.45 g, 8.0 mmol, 2 equiv) in 2 mL of water was added dropwise to *o*-(trimethylsilylethynyl)thioanisole (0.88 g, 4.0 mmol) in 20 mL of CH_3OH under an argon atmosphere at 25 ^oC. The mixture was stirred for another 0.5 h at 25 ^oC, and the CH_3OH was removed under vacuum. The residue was added to 20 mL of brine, and the mixture was extracted with EtOAc (3 x 20 mL), dried (Na_2SO_4), filtered, and the solvent removed under vacuum. Purification by flash chromatography afforded the product (yield = 86%) as a yellow oil: ^1H NMR (400 MHz, $CDCl_3$) δ 2.49 (s, 3H), 3.47 (s, 1H), 7.08 (td, J = 0.8, 7.2 Hz, 1H), 7.16 (d, J = 7.6 Hz, 1H), 7.31 (td, J = 1.2, 7.6 Hz, 1H), 7.46 (dd, J = 1.2, 7.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ15.3, 81.2, 83.7, 120.3, 124.38, 124.41, 129.5, 133.3, 142.0; IR (in CH_2Cl_2, cm^{-1}) 3288, 3049, 2922, 2102, 1436; HRMS Calcd for C_9H_8S: 148.0347. Found: 148.0334.



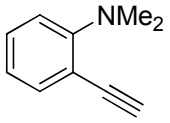
6

Methyl 2-ethynylbenzoate (6). A modified literature procedure was used.[5] To a solution of compound **2** (4.0 mmol) in methanol (20 mL), $KF{\cdot}2H_2O$ (2.26 g, 24 mmol, 6 equiv) was added and the reaction mixture was stirred for 36 h at 25 ^oC. After the reaction was over, methanol was removed under vacuum and the residue was extracted with EtOAc (3 x 20 mL), washed with 0.1M HCl and brine, dried (Na_2SO_4), filtered, and the solvent removed under vacuum. Purification by flash chromatography afforded the product (yield = 82%) as a colorless oil with spectral properties identical to those previously reported.[5]



7

***N*-Phenyl-2-ethynylbenzamide (7).** This compound was prepared according to the modified literature procedure used for compound **6** above, except that the reaction time was 0.5 h.[5] Purification by flash chromatography afforded the product (yield = 89%) as a yellow solid: mp 95-98 ^oC; ^1H NMR (300 MHz, $CDCl_3$) δ 3.57 (s, 1H), 7.11-7.16 (t, J = 7.2 Hz, 1H), 7.24-7.46 (m, 4H), 7.55-7.58 (m, 1H), 7.66 (d, J = 7.8 Hz, 2H), 7.96-7.99 (m, 1H), 9.02 (br s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 82.0, 84.4, 118.6, 120.1, 124.7, 129.2, 129.7, 130.0, 130.9, 134.2, 136.9, 138.1, 164.4; IR (in CH_2Cl_2, cm^{-1}) 3295, 3054, 2305, 1672, 1265; HRMS Calcd for $C_{15}H_{11}NO$: 221.08406. Found: 221.08437.

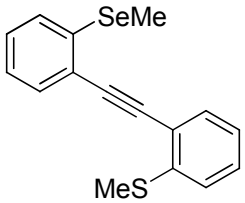


8

***N,N*-Dimethyl-2-ethynylaniline (8).** This compound was prepared according to a literature procedure.[7] Purification by flash chromatography afforded the product as a yellow oil (yield = 65%) with spectral properties identical to those previously reported.[7]

Preparation of the diarylalkynes.

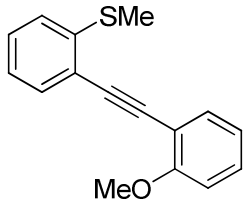
General procedure used for the Sonogashira Coupling. The following procedure has been used for the preparation of all of the diarylalkynes. Slight modifications were made wherever required (Table 2). To a solution of iodoarene (1.0 mmol), $PdCl_2(PPh_3)_2$ (0.014 g, 2 mol %), and CuI (0.002 g, 1 mol %) in TEA (4 mL) (stirring for 5 min beforehand), 1.2 mmol of trimethylsilylacetylene (1.2 equiv) in 1 mL of TEA was added dropwise over 10 min. The reaction flask was flushed with argon and the mixture was stirred at the indicated temperature for the indicated time (Table 2). After the reaction was over, the resulting solution was filtered, washed with brine, and extracted with EtOAc (2 x 10 mL). The combined extracts were dried over $MgSO_4$ and concentrated under vacuum to yield the crude product. Purification was performed by flash chromatography.



9

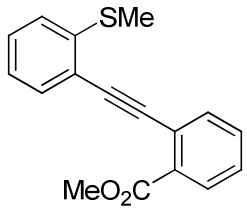
2-[(2-(Methylselenyl)phenyl)ethynyl]thioanisole (9). The product was obtained as a white solid: mp 105-107 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.37 (s, 3H), 2.52 (s, 3H), 7.10-7.19 (m, 3H), 7.24-7.33 (m, 3H), 7.54 (t, J = 6.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 6.5, 15.5, 92.6, 94.2, 121.4, 123.7, 124.3, 124.4, 125.3, 127.5, 129.10, 129.11,

132.77, 132.81, 136.5, 141.8; IR (in CH_2Cl_2, cm^{-1}) 3053, 2986, 2305, 1266; HRMS Calcd for $C_{16}H_{14}SSe$: 317.99814. Found: 317.99874.



10

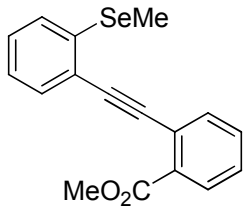
2-[(2-Methoxyphenyl)ethynyl]thioanisole (10). The product was obtained as a white solid: mp 117-119 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.52 (s, 3H), 3.93 (s, 3H), 6.89-6.96 (m, 2H), 7.11 (t, J = 7.5 Hz, 1H), 7.18 (d, J = 8.0 Hz, 1H), 7.26-7.33 (m, 2H), 7.51-7.56 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 15.4, 56.1, 91.1, 92.5, 110.9, 112.6, 120.6, 122.0, 124.41, 124.43, 128.7, 130.1, 132.5, 133.8, 141.7, 160.1; IR (in CH_2Cl_2, cm^{-1}) 3053, 2986, 2305, 1265; HRMS Calcd for $C_{16}H_{14}OS$: 254.07654. Found: 254.07689.



11

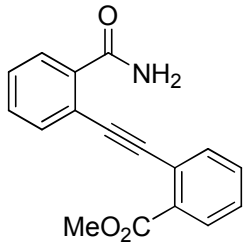
Methyl 2-[(2-(methylthio)phenyl)ethynyl]benzoate (11). The product was obtained as a colorless solid: mp 73-75 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.49 (s, 3H), 3.96 (s, 3H), 7.11 (t, J = 7.2 Hz, 1H), 7.16 (d, J = 7.6 Hz, 1H), 7.30 (t, J = 7.6 Hz, 1H), 7.37 (t, J = 7.6 Hz, 1H), 7.48 (t, J = 7.8 Hz, 1H), 7.54 (d, J = 7.8 Hz, 1H), 7.72 (d, J = 7.6 Hz, 1H), 7.97 (d, J = 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 15.2, 52.4, 91.9, 94.6, 121.4, 123.6, 124.1, 124.3, 128.1, 129.1, 130.5, 131.6, 131.8, 132.8, 134.4, 141.9, 166.8; IR (in

CH_2Cl_2, cm^{-1}) 3054, 2986, 2305, 1727, 1265; HRMS Calcd for $C_{17}H_{14}O_2S$: 282.07145. Found: 282.07178.



12

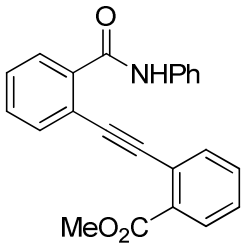
Methyl 2-[(2-(methylselenyl)phenyl)ethynyl]benzoate (12). The product was obtained as a yellow gel: ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.37 (s, 3H), 3.97 (s, 3H), 7.14-7.20 (m, 1H), 7.24-7.32 (m, 2H), 7.36-7.42 (m, 1H), 7.48-7.55 (m, 2H), 7.73 (dd, J = 0.9, 7.8 Hz, 1H), 7.98 (dd, J = 0.9, 7.8 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 6.3, 52.5, 92.9, 93.9, 123.7, 123.9, 125.4, 127.6, 128.3, 129.3, 130.6, 131.8, 131.9, 132.9, 134.5, 136.7, 166.9; IR (in CH_2Cl_2, cm^{-1}) 3053, 2928, 2305, 1729, 1262; HRMS Calcd for $C_{17}H_{14}O_2Se$: 330.01590. Found: 330.01629.



13

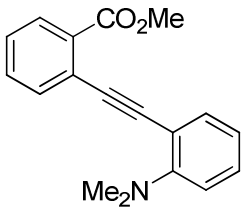
Methyl 2-[(2-carbamoylphenyl)ethynyl]benzoate (13). The product was obtained as a colorless solid: mp 122-124 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.93 (s, 3H), 6.48 (br s, 1H), 7.39-7.57 (m, 4H), 7.65-7.70 (m, 2H), 8.03 (dt, J = 0.9, 8.1 Hz, 1H), 8.21-8.24 (m, 1H), 8.37 (br s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 52.6, 92.7, 94.6, 120.3, 123.2, 128.8, 129.2, 130.75, 130.82, 130.9, 131.0, 132.3, 134.2, 134.3, 134.7, 166.3, 167.8; IR (in

CH_2Cl_2, cm^{-1}) 3486, 3370, 3054, 2987, 2305, 1722, 1670; HRMS Calcd for $C_{17}H_{13}NO_3$: 279.08954. Found: 279.08994.



14

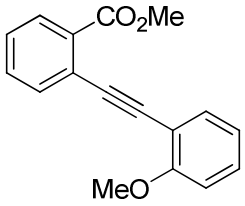
Methyl 2-[(2-(phenylcarbamoyl)phenyl)ethynyl]benzoate (14). The product was obtained as a yellow solid: mp 117-119 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.78 (s, 3H), 7.10-7.14 (m, 1H), 7.28-7.32 (m, 2H), 7.44 (td, J = 1.2, 7.6 Hz, 1H), 7.49-7.53 (m, 3H), 7.60-7.63 (m, 3H), 7.69-7.71 (m, 1H), 8.03-8.05 (m, 1H), 8.16-8.19 (m, 1H), 9.45 (br s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 52.5, 92.1, 95.2, 119.8, 121.4, 123.0, 124.7, 128.96, 129.03, 129.4, 130.8, 131.0, 131.1, 131.5, 132.4, 133.9, 134.3, 135.9, 138.2, 164.7, 166.2; IR (in CH_2Cl_2, cm^{-1}) 3350, 3053, 2985, 2306, 1719, 1665, 1265; HRMS Calcd for $C_{23}H_{17}NO_3$: 355.12084. Found: 355.12145.



15

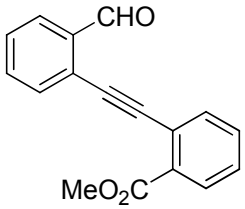
Methyl 2-[(2-(dimethylamino)phenyl)ethynyl]benzoate (15). The product was obtained as a yellow oil (strongly fluorescent under UV): ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.00 (s, 6H), 3.93 (s, 3H), 6.86-6.91 (m, 2H), 7.21-7.27 (m, 1H), 7.32 (td, J = 1.5, 7.8 Hz, 1H), 7.45 (td, J = 1.5, 7.5 Hz, 1H), 7.55 (dd, J = 1.8, 6.9 Hz, 1H), 7.64 (dt, J = 0.6, 7.8

Hz, 1H), 7.95 (dd, J = 1.5, 7.8 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 43.6, 52.2, 93.6, 94.1, 114.9, 116.8, 120.4, 124.4, 127.6, 129.6, 130.5, 131.5, 131.7, 133.7, 134.9, 154.7, 166.9; IR (in CH_2Cl_2, cm^{-1}) 2948, 2835, 2207, 1728, 1251; HRMS Calcd for $C_{18}H_{17}NO_2$: 279.12593. Found: 279.12658.



16

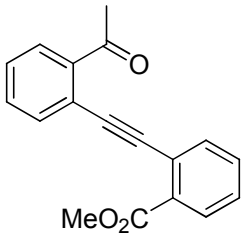
Methyl 2-[(2-methoxyphenyl)ethynyl]benzoate (16). The product was obtained as a light brown solid: mp 62-64 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.89 (s, 3H), 3.94 (s, 3H), 6.86-6.96 (m, 2H), 7.26-7.36 (m, 2H), 7.45 (t, J = 7.5 Hz, 1H), 7.55 (d, J = 7.5 Hz, 1H), 7.67 (d, J = 7.5 Hz, 1H), 7.96 (d, J = 7.8 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 52.3, 56.0, 91.2, 92.5, 111.0, 112.8, 120.7, 124.2, 128.0, 130.3, 130.6, 131.8, 131.9, 134.1, 134.3, 160.3, 167.1; IR (in CH_2Cl_2, cm^{-1}) 3054, 2951, 2838, 2306, 2216, 1716, 1270; HRMS Calcd for $C_{17}H_{14}O_3$: 266.09429. Found: 266.09468.



17

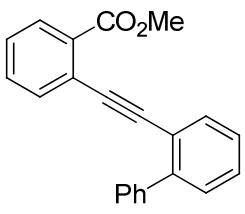
Methyl 2-[(2-formylphenyl)ethynyl]benzoate (17). The product was obtained as a white solid that turned gray upon exposure to air: mp 87-89 ^{o}C; ^{1}H NMR (300 MHz,

$CDCl_3$) δ 3.95 (s, 3H), 7.40-7.61 (m, 4H), 7.67-7.71 (m, 2H), 7.95-8.03 (m, 2H), 10.76 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 52.6, 90.1, 95.1, 123.1, 127.16, 127.24, 128.9, 129.0, 130.9, 131.9, 132.1, 133.6, 133.9, 134.5, 136.5, 166.4, 192.6; IR (in CH_2Cl_2, cm^{-1}) 3054, 2987, 2306, 1727, 1696, 1264; HRMS Calcd for $C_{17}H_{12}O_3$: 264.07864. Found: 264.07909.



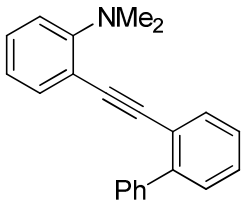
18

Methyl 2-[(2-acetylphenyl)ethynyl]benzoate (18). The product was obtained as an orange solid: mp 68-70 ^oC; ^1H NMR (400 MHz, $CDCl_3$) δ 2.79 (s, 3H), 3.93 (s, 3H), 7.36-7.40 (m, 2H), 7.46-7.49 (m, 2H), 7.66-7.76 (m, 3H), 7.96 (dd, J = 1.2, 7.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 29.8, 52.1, 93.2, 93.7, 121.6, 123.3, 128.2, 128.4, 128.6, 130.4, 131.2, 131.5, 131.7, 133.9, 134.1, 140.3, 166.2, 199.9; IR (in CH_2Cl_2, cm^{-1}) 3055, 2952, 2305, 1730, 1678, 1270; HRMS Calcd for $C_{18}H_{14}O_3$: 278.09429. Found: 278.09473.



19

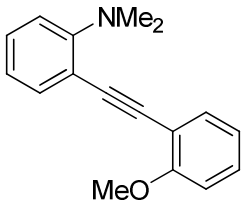
Methyl 2-(biphenyl-2-ylethynyl)benzoate (19). The product was obtained as a colorless solid: mp 67-69 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.82 (s, 3H), 7.29-7.47 (m, 9H), 7.68-7.74 (m, 3H), 7.92 (d, J = 7.8 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 52.3, 91.2, 94.5, 121.8, 124.1, 127.3, 127.7, 128.0, 128.2, 129.1, 129.67, 129.73, 130.6, 131.77, 131.82, 133.7, 134.1, 140.7, 144.0, 166.9; IR (in CH_2Cl_2, cm^{-1}) 3053, 2987, 2303, 2212, 1727, 1268; HRMS Calcd for $C_{22}H_{16}O_2$: 312.11503. Found: 312.11564.



20

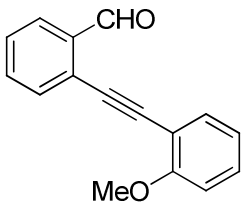
***N,N*-Dimethyl-2-(biphenyl-2-ylethynyl)aniline (20).** This compound was obtained in a 35% yield following the general procedure mentioned above. The following slightly modified literature procedure resulted in a significantly increased yield.[9] *N,N*-Dimethyl-2-iodoaniline (2.0 mmol), 42 mg of $PdCl_2(PPh_3)_2$ (0.06 mmol, 3.0 mol %), and 12 mg of CuI (0.06 mmol, 3.0 mol %) were placed in a dry reaction flask. Next, 5 mL of toluene and 0.6 mL of diisopropylamine were added to the flask, followed by 0.22 g of 2-methyl-3-butyn-2-ol (2.6 mmol, 1.3 equiv). The reaction mixture was stirred at 80 ^{o}C under argon for 1 h. As monitored by TLC, the first acetylene coupling was complete. The reaction temperature of the oil bath was increased to 110 ^{o}C, and 96 mg of NaH (120 mg of 80% dispersion, 4.0 mmol, 2.0 equiv) was added slowly to the mixture. After 5 min of stirring, 0.560 g (2.0 mmol, 1.0 equiv) of 2-iodobiphenyl was added to the reaction mixture, and the stirring was continued. After 25 min, 48 mg of NaH (another 60 mg portion of 80% dispersion) (2.0 mmol, 1.0 equiv) was added carefully, and the solution

was stirred at 110 °C for 1 h. After cooling to room temperature, the suspension was filtered, and the separated amine-hydrochloride was washed with toluene. Evaporation of the combined toluene solution gave a crude product, which was purified by column chromatography using hexane-ethyl acetate mixtures to afford the product as a yellow oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.79 (s, 6H), 6.78-6.85 (m, 2H), 7.16-7.45 (m, 9H), 7.66 (d, J = 6.9 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 43.4, 91.8, 94.6, 115.4, 116.9, 120.4, 122.4, 127.2, 127.5, 128.1, 128.4, 129.3, 129.6, 129.7, 132.9, 134.4, 140.9, 143.6, 154.6; IR (in CH_2Cl_2, cm^{-1}) 3051, 2984, 2304, 2207, 1269; HRMS Calcd for $C_{22}H_{19}N$: 297.15175. Found: 297.15210.



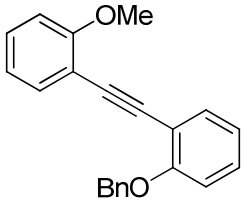
21

***N,N*-Dimethyl-2-[(2-methoxyphenyl)ethynyl]aniline (21).** The product was obtained as a green liquid (fluorescent under UV): ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.01 (s, 6H), 3.88 (s, 3H), 6.86-6.94 (m, 4H), 7.20-7.30 (m, 2H), 7.48-7.53 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 43.8, 55.9, 91.5, 93.1, 110.8, 113.4, 115.7, 117.1, 120.59, 120.64, 129.3, 129.6, 133.3, 134.6, 154.8, 160.2; IR (in CH_2Cl_2, cm^{-1}) 3054, 2986, 2305, 1265; HRMS Calcd for $C_{17}H_{17}NO$: 251.13101. Found: 251.13138.



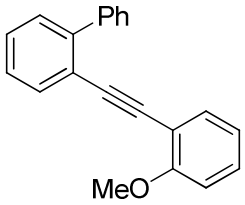
22

2-[(2-Methoxyphenyl)ethynyl]benzaldehyde (22). The product was obtained as a colorless solid: mp 82-84 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.91 (s, 3H), 6.90-6.98 (m, 2H), 7.31-7.44 (m, 2H), 7.49-7.58 (m, 2H), 7.62-7.65 (m, 1H), 7.94 (dd, J = 1.2, 7.8 Hz, 1H), 10.74 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 55.9, 89.2, 93.2, 110.8, 111.7, 120.7, 127.1, 127.5, 128.5, 130.7, 133.1, 133.4, 133.8, 136.0, 160.5, 192.7; IR (in CH_2Cl_2, cm^{-1}) 3053, 2987, 2305, 2215, 1694, 1271; HRMS Calcd for $C_{16}H_{12}O_2$: 236.08373. Found: 236.08409.



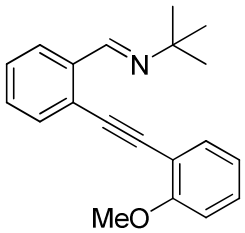
23

1-Benzyloxy-2-[(2-methoxyphenyl)ethynyl]benzene (23). The product was obtained as a white solid: mp 77-79 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.80 (s, 3H), 5.18 (s, 2H), 6.85-6.95 (m, 4H), 7.22-7.30 (m, 3H), 7.35 (t, J = 7.2 Hz, 2H), 7.49 (d, J = 7.2 Hz, 1H), 7.54-7.55 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 56.0, 70.6, 90.1, 90.3, 110.9, 113.0, 113.1, 113.9, 120.6, 121.0, 127.2, 127.8, 128.5, 129.6, 129.7, 133.6, 133.7, 137.3, 159.3, 160.0; IR (in CH_2Cl_2, cm^{-1}) 3052, 2984, 2306, 1266; HRMS Calcd for $C_{22}H_{18}O_2$: 314.13068. Found: 314.13110.



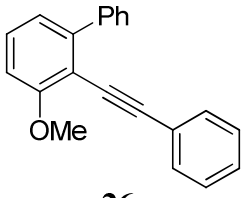
24

2-[(2-Methoxyphenyl)ethynyl]biphenyl (24). The product was obtained as a white solid: mp 69-71 ^{o}C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.78 (s, 3H), 6.78-6.86 (m, 2H), 7.18-7.45 (m, 8H), 7.66-7.72 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 56.0, 89.1, 93.6, 111.0, 113.0, 120.7, 122.2, 127.2, 127.6, 128.1, 128.6, 129.7, 129.8, 129.9, 133.4, 133.6, 140.8, 143.9, 160.2; IR (in CH_2Cl_2, cm^{-1}) 3052, 2965, 2304, 2212, 1263; HRMS Calcd for $C_{21}H_{16}O$: 284.12012. Found: 284.12065.



25

***N*-[2-((2-Methoxyphenyl)ethynyl)benzylidene]-2-methylpropan-2-amine (25).** The compound was prepared according to the literature procedure as a yellow oil and the spectral properties were found to be identical to those previously reported.[8]



26

3-Methoxy-2-(phenylethynyl)biphenyl (26). The product was obtained as yellow gel: ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.97 (s, 3H), 6.91 (d, J = 8.8 Hz, 1H), 7.03 (d, J = 7.6 Hz, 1H), 7.25-7.26 (m, 3H), 7.31-7.46 (m, 6H), 7.65 (d, J = 7.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 56.4, 85.6, 97.1, 109.4, 111.3, 122.1, 124.0, 127.7, 128.0, 128.1, 128.3, 129.3, 129.7, 131.5, 140.7, 146.1, 160.6; IR (in CH_2Cl_2, cm^{-1}) 3052, 2986, 2305, 1265; HRMS Calcd for $C_{21}H_{16}O$: 284.12012. Found: 284.12053.

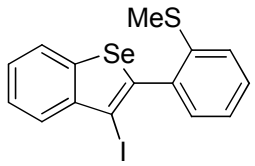
Electrophilic cyclization of the diarylalkynes.

General procedure for iodocyclization. To a solution of 0.25 mmol of the diarylalkyne in 3 mL of CH_2Cl_2 was added gradually the I_2/ICl (amounts as indicated in Table 3) in 2 mL of CH_2Cl_2. The reaction mixture was flushed with argon and allowed to stir at 25 °C for the indicated time (the reaction was monitored by TLC for completion). The excess I_2/ICl was removed by washing with satd aq $Na_2S_2O_3$. The mixture was then extracted by EtOAc or diethyl ether (2 x 10 mL). The combined organic layers were dried over anhydrous $MgSO_4$ and concentrated under vacuum to yield the crude product, which was purified by flash chromatography on silica gel with ethyl acetate/hexanes as the eluent.

General procedure for the PhSeCl cyclizations. To a solution of 0.25 mmol of the diarylalkyne in CH_2Cl_2 (3 mL) was added the PhSeCl (amount as indicated in Table 3) in 2 mL of CH_2Cl_2. The mixture was flushed with argon and allowed to stir at 25 °C for the indicated time. The reaction mixture was washed with 20 mL of water and extracted with EtOAc or diethyl ether. The combined organic layers were dried over anhydrous $MgSO_4$

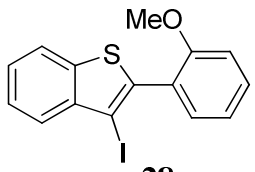
and concentrated under vacuum to yield the crude product, which was further purified by flash chromatography on silica gel with ethyl acetate/hexanes as the eluent.

General procedure for bromocyclization. To a solution of 0.25 mmol of the diarylalkyne in 3 mL of CH_2Cl_2 was added gradually 1.2 equiv of NBS dissolved in 2 mL of CH_2Cl_2. The reaction mixture was allowed to stir at 25 °C for 1-4 h. The excess NBS was removed by washing with satd aq $Na_2S_2O_3$. The mixture was then extracted by diethyl ether (2 x 10 mL). The combined ether layers were dried over anhydrous $MgSO_4$ and concentrated under vacuum to yield the crude product, which was purified by flash chromatography on silica gel using ethyl acetate/hexanes as the eluent.



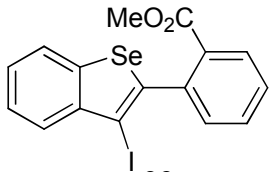
27

3-Iodo-2-(2-methylsulfanylphenyl)benzo[*b*]selenophene (27). The product was obtained as a white solid: mp 116-118 ^oC; ^1H NMR (400 MHz, $CDCl_3$) δ 2.43 (s, 3H), 7.24-7.38 (m, 4H), 7.42-7.50 (m, 2H), 7.85-7.90 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 16.0, 87.2, 124.7, 125.2, 125.6, 125.9, 126.1, 128.8, 129.8, 131.0, 135.6, 139.3, 141.4, 142.9, 143.1; IR (in CH_2Cl_2, cm^{-1}) 3054, 2986, 2925, 1264; HRMS Calcd for $C_{15}H_{11}ISSe$: 429.87914. Found: 429.88004.



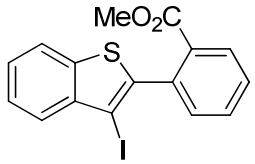
28

3-Iodo-2-(2-methoxyphenyl)benzo[*b*]thiophene (28). The product was obtained as a yellow solid: mp 104-106 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.83 (s, 3H), 7.00-7.08 (m, 2H), 7.35-7.40 (m, 2H), 7.45 (t, J = 8.0 Hz, 2H), 7.79 (t, J = 7.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ55.8, 82.9, 111.6, 120.6, 122.3, 123.7, 125.3, 125.4, 126.1, 130.9, 132.7, 139.6, 139.8, 141.4, 157.3; IR (in CH_2Cl_2, cm^{-1}) 3052, 2980, 1432, 1264; HRMS Calcd for $C_{15}H_{11}IOS$: 365.95764. Found: 365.95825.



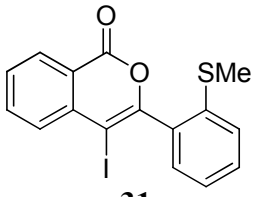
29

Methyl 2-(3-iodobenzo[*b*]selenophen-2-yl)benzoate (29). The product was obtained as a white solid: mp 113-115 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.69 (s, 3H), 7.32-7.35 (m, 1H), 7.42-7.55 (m, 3H), 7.60 (td, J = 1.2, 7.6 Hz, 1H), 7.85 (t, J = 7.2 Hz, 2H), 8.04 (dd, J = 1.2, 7.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ52.6, 85.3, 125.3, 125.8, 125.9, 128.6, 129.1, 130.7, 131.2, 131.9, 132.2, 138.2, 140.9, 142.8, 144.2, 167.1; IR (in CH_2Cl_2, cm^{-1}) 3054, 2984, 1727, 1266; HRMS Calcd for $C_{16}H_{11}IO_2Se$: 441.89690. Found: 441.89743.



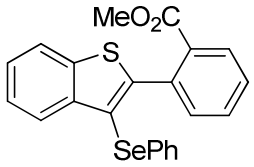
30

Methyl 2-(3-iodobenzo[*b*]thiophen-2-yl)benzoate (30). The product was obtained as a colorless solid: mp 111-115 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.68 (s, 3H), 7.38-7.39 (m, 3H), 7.53-7.64 (m, 2H), 7.76-7.81 (m, 2H), 8.05 (d, J = 7.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 52.6, 82.2, 122.3, 125.5, 125.6, 126.0, 129.4, 130.8, 131.7, 132.0, 132.6, 135.9, 139.4, 141.1, 142.2, 167.1; IR (in CH_2Cl_2, cm^{-1}) 3053, 2952, 1729, 1264; HRMS Calcd for $C_{16}H_{11}IO_2S$: 393.95245. Found: 393.95324.



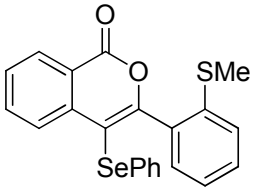
31

4-Iodo-3-[2-(methylthio)phenyl]isocoumarin (31). The product was obtained as a colorless solid: mp 128-130 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.48 (s, 3H), 7.28 (t, J = 7.6 Hz, 1H), 7.34-7.40 (m, 2H), 7.46 (t, J = 7.2 Hz, 1H), 7.60-7.63 (m, 1H), 7.82-7.85 (m, 2H), 7.33 (d, J = 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 16.7, 80.1, 120.7, 125.4, 127.1, 129.7, 130.1, 130.5, 130.8, 131.3, 135.5, 135.9, 137.8, 138.9, 154.6, 161.9; IR (in CH_2Cl_2, cm^{-1}) 3054, 2987, 1735, 1264; HRMS Calcd for $C_{16}H_{11}IO_2S$: 393.95245. Found: 393.95324.



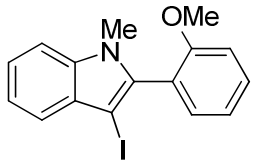
32

Methyl 2-[3-(phenylselenyl)benzo[*b*]thiophen-2-yl]benzoate (32). The product was obtained as a yellow gel: ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.59 (s, 3H), 7.07-7.12 (m, 5H), 7.36-7.39 (m, 3H), 7.48-7.53 (m, 2H), 7.80-7.87 (m, 2H), 7.97-8.00 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ52.3, 118.0, 122.3, 125.0, 125.16, 125.19, 126.2, 129.07, 129.11, 130.0, 130.05, 131.5, 131.8, 132.2, 132.8, 135.2, 139.7, 141.1, 147.9, 167.2; IR (in CH_2Cl_2, cm^{-1}) 3055, 2987, 1727, 1263; HRMS Calcd for $C_{22}H_{16}O_2SSe$: 424.00362. Found: 424.00442.



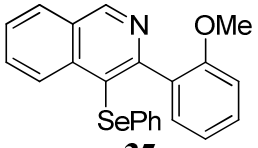
33

3-[2-(Methylthio)phenyl]-4-(phenylselenyl)isocoumarin (33). The product was obtained as a white solid: mp 111-113 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.43 (s, 3H), 7.13-7.23 (m, 6H), 7.29-7.35 (m, 2H), 7.39-7.43 (m, 1H), 7.52-7.56 (m, 1H), 7.68-7.72 (m, 1H), 8.00 (d, J = 8.0 Hz, 1H), 8.37 (dd, J = 0.8, 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 16.8, 108.0, 121.4, 125.1, 126.7, 126.9, 128.2, 129.1, 129.4, 129.9, 130.0, 130.2, 130.5, 131.5, 134.2, 135.5, 137.9, 139.1, 158.3, 162.1; IR (in CH_2Cl_2, cm^{-1}) 3056, 2987, 1743, 1262; HRMS Calcd for $C_{22}H_{16}O_2SSe$: 424.00362. Found: 424.00442.



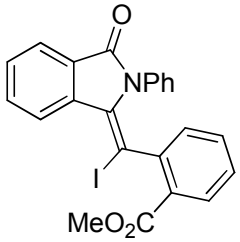
34

3-Iodo-2-(2-methoxyphenyl)-1-methylindole (34). The product was obtained as a white oil: ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.60 (s, 3H), 3.79 (s, 3H), 7.04 (d, J = 8.4 Hz, 1H), 7.10 (t, J = 7.6 Hz, 1H), 7.19-7.36 (m, 4H), 7.46-7.50 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ31.8, 55.8, 59.4, 109.8, 111.4, 120.4, 120.77, 120.79, 121.5, 122.7, 130.4, 131.1, 133.6, 137.6, 139.5, 158.0; IR (in CH_2Cl_2, cm^{-1}) 3053, 2939, 1465, 1265; HRMS Calcd for $C_{16}H_{14}INO$: 363.01202. Found: 363.01255.



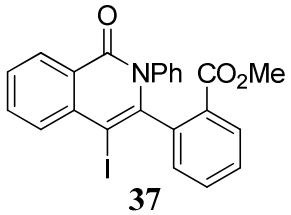
35

3-(2-Methoxyphenyl)-4-(phenylselenyl)isoquinoline (35). The product was obtained as a yellow gel: ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.66 (s, 3H), 6.94 (d, J = 8.0 Hz, 1H), 7.02-7.05 (m, 6H), 7.35-7.40 (m, 2H), 7.60 (t, J = 7.6 Hz, 1H), 7.67 (td, J = 1.2, 8.0 Hz, 1H), 8.00 (d, J = 8.0 Hz, 1H), 8.36 (d, J = 8.4 Hz, 1H), 9.36 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ55.4, 110.6, 120.6, 124.0, 126.1, 127.5, 128.3, 128.46, 128.49, 129.1, 129.8, 130.2, 130.8, 131.6, 131.7, 133.3, 138.1, 153.5, 156.1, 156.9; IR (in CH_2Cl_2, cm^{-1}) 3053, 2985, 1265; HRMS Calcd for $C_{22}H_{17}NOSe$: 391.04753. Found: 391.04826.



36

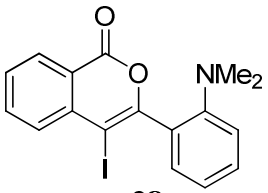
(*E*)-Methyl 2-[iodo(3-oxo-2-phenylisoindolin-1-ylidene)methyl]benzoate (36). The product was obtained as a yellow oil: ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.76 (s, 3H), 7.02 (t, J = 7.1 Hz, 1H), 7.10-7.18 (m, 4H), 7.37-7.43 (m, 2H), 7.54-7.62 (m, 2H), 7.70 (t, J = 7.5 Hz, 1H), 7.97-8.02 (m, 2H), 8.81 (d, J = 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ52.4, 71.2, 124.0, 124.7, 125.1, 125.2, 128.5, 128.6, 129.3, 130.7, 130.8, 131.0, 132.0, 132.2, 132.9, 135.1, 141.8, 144.7, 147.5, 151.9, 166.8; IR (in CH_2Cl_2, cm^{-1}) 3054, 2986, 1726, 1689, 1265; HRMS Calcd for $C_{23}H_{16}INO_3$: 481.01750. Found: 481.01876.



37

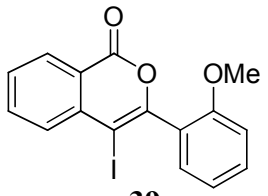
Methyl 2-(4-iodo-1-oxo-2-phenyl-1,2-dihydroisoquinolin-3-yl)benzoate (37). The product was obtained as a white solid: mp 112-114 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.79 (s, 3H), 6.98 (t, J = 7.3 Hz, 1H), 7.09-7.11 (m, 2H), 7.21 (t, J = 7.7 Hz, 2H), 7.46-7.52 (m, 3H), 7.57-7.68 (m, 3H), 8.04 (d, J = 7.7 Hz, 1H), 8.39 (d, J = 7.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ52.7, 76.6, 123.0, 123.9, 124.1, 127.8, 128.7, 129.2, 130.0, 130.1, 130.7, 130.9, 132.1, 132.4, 133.1, 134.5, 137.1, 145.9, 148.7, 153.8, 166.1; IR (in

CH_2Cl_2, cm^{-1}) 3052, 2985, 1725, 1664, 1263; HRMS Calcd for $C_{23}H_{16}INO_3$: 481.01750. Found: 481.01876.



38

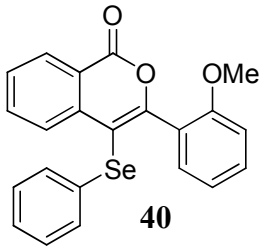
3-[2-(Dimethylamino)phenyl]-4-iodoisocoumarin (38). The product was obtained as a light green solid: mp 103-105 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.78 (s, 6H), 6.95-7.02 (m, 2H), 7.32-7.40 (m, 2H), 7.58 (t, J = 7.2 Hz, 1H), 7.79-7.87 (m, 2H), 8.31 (d, J = 7.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ43.4, 79.5, 117.8, 120.0, 120.2, 126.7, 129.2, 129.9, 131.16, 131.21, 132.2, 135.8, 138.6, 152.0, 156.2, 162.3; IR (in CH_2Cl_2, cm^{-1}) 3056, 2989, 1731, 1265; HRMS Calcd for $C_{17}H_{14}INO_2$: 391.00693. Found: 391.00772.



39

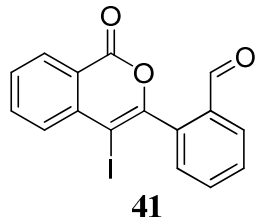
4-Iodo-3-(2-methoxyphenyl)isocoumarin (39). The product was obtained as a white solid: mp 166-168 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.85 (s, 3H), 6.96-6.98 (m, 1H), 7.05 (t, J = 7.6 Hz, 1H), 7.37 (d, J = 7.2 Hz, 1H), 7.46 (t, J = 7.2 Hz, 1H), 7.56-7.60 (m, 1H), 7.79-7.85 (m, 2H), 8.31 (d, J = 7.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ55.8, 79.8, 111.5, 120.5, 120.6, 125.1, 129.3, 129.9, 131.2, 131.4, 132.0, 135.7, 138.2, 153.8,

157.2, 162.2; IR (in CH_2Cl_2, cm^{-1}) 3054, 2928, 1735, 1265; HRMS Calcd for $C_{16}H_{11}IO_3$: 377.97530. Found: 377.97601.



40

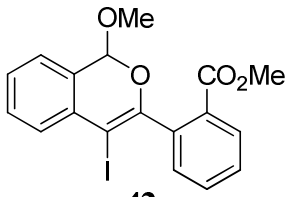
3-(2-Methoxyphenyl)-4-(phenylselenyl)isocoumarin (40). The product was obtained as a yellow solid: mp 113-116 ^oC; ^1H NMR (400 MHz, $CDCl_3$) δ 3.71 (s, 3H), 6.92 (d, J = 8.0 Hz, 1H), 6.99 (t, J = 7.6 Hz, 1H), 7.12-7.15 (m, 3H), 7.19-7.21 (m, 2H), 7.35-7.43 (m, 2H), 7.51 (t, J = 7.6 Hz, 1H), 7.67 (t, J = 7.6 Hz, 1H), 7.96 (d, J = 8.0 Hz, 1H), 8.35 (d, J = 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ55.5, 107.7, 111.0, 120.2, 121.2, 123.9, 126.5, 128.0, 128.7, 129.3, 129.6, 129.9, 131.0, 131.6, 131.8, 135.3, 138.2, 157.27, 157.35, 162.4; IR (in CH_2Cl_2, cm^{-1}) 3057, 2985, 1735, 1265; HRMS Calcd for $C_{22}H_{16}O_3Se$: 408.02647. Found: 408.02699.



41

3-(2-Formylphenyl)-4-iodoisocoumarin (41). The product was obtained as a white solid: mp 146-148 ^oC; ^1H NMR (400 MHz, $CDCl_3$) δ 7.57 (d, J = 7.2 Hz, 1H), 7.62-7.77 (m, 3H), 7.82-7.88 (m, 2H), 8.04 (dd, J = 1.2, 7.6 Hz, 1H), 8.34 (d, J = 7.6 Hz, 1H), 10.08 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ79.6, 120.7, 129.9, 130.2, 130.4, 131.0,

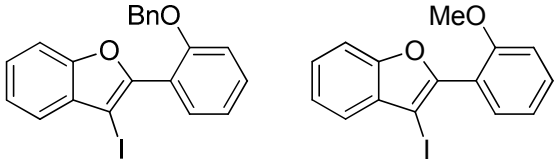
131.3, 131.6, 134.1, 134.2, 136.1, 137.3, 137.5, 152.9, 161.3, 190.2; IR (in CH_2Cl_2, cm^{-1}) 3054, 2985, 1740, 1706, 1265; HRMS Calcd for $C_{16}H_9IO_3$: 375.95965. Found: 375.96067.



42

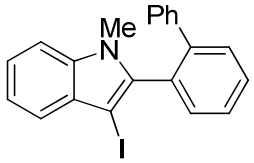
Methyl 2-(4-iodo-1-methoxy-1*H*-isochromen-3-yl)benzoate (42).

A modified literature procedure was used to prepare this compound.[10] To a solution of the diarylalkyne **15** (0.25 mmol), K_2CO_3 (0.25 mmol, 1.0 equiv) and methanol (0.3 mmol, 1.2 equiv) in CH_2Cl_2 (5.0 mL), iodine (0.3 mmol, 1.2 equiv) was added and the solution was stirred at room temperature for 10 h. The reaction mixture was then quenched with satd aq $Na_2S_2O_3$, extracted using EtOAc and washed with water. The combined organic extracts were dried over anhydrous $MgSO_4$ and concentrated under vacuum. Following flash column chromatography, compound **36** was isolated as the major product. The compound was unstable and decomposed during rotary evaporation (in order to remove the trace amounts of solvent). The NMR data showing the presence of the compound (note that the attached spectrum shows extra peaks because of the solvent and a slight impurity) is provided here: ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.56 (s, 3H), 3.80 (s, 3H), 6.06 (s, 1H), 7.21 (dt, J = 0.8, 7.2 Hz, 1H), 7.31 (td, J = 1.2, 7.2 Hz, 1H), 7.40-7.45 (m, 2H), 7.48-7.52 (m, 2H), 7.61 (td, J = 1.6, 7.6 Hz, 1H), 8.05 (dt, J = 0.8, 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ52.5, 56.0, 74.9, 100.5, 125.7, 127.1, 127.8, 129.0, 129.4, 130.0, 130.4, 130.5, 131.0, 131.3, 132.4, 138.8, 152.3, 166.7.



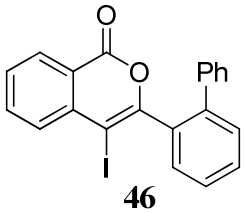
43 and **44**

2-[2-(Benzyloxy)phenyl]-3-iodobenzofuran (43) and 3-iodo-2-(2-methoxyphenyl)benzofuran (44). The reaction resulted in a complex inseparable mixture. However, these two products were observed by GC-MS analysis.



45

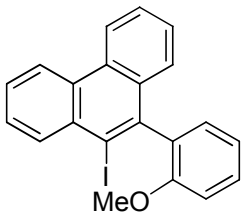
2-(Biphenyl-2-yl)-3-iodo-1-methylindole (45). The product was obtained as a yellow gel: ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.15 (s, 3H), 7.07-7.09 (m, 1H), 7.13-7.23 (m, 7H), 7.46-7.48 (m, 3H), 7.55-7.58 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ31.6, 60.4, 109.8, 120.6, 121.3, 122.6, 127.3, 127.5, 128.4, 128.8, 130.0, 130.2, 130.3, 130.4, 133.4, 137.1, 140.6, 141.7, 142.8; IR (in CH_2Cl_2, cm^{-1}) 3052, 2984, 1463, 1265; HRMS Calcd for $C_{21}H_{16}IN$: 409.03275. Found: 409.03326.



46

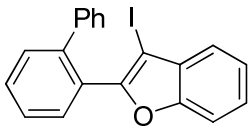
3-(Biphenyl-2-yl)-4-iodoisocoumarin (46). The product was obtained as a white solid: mp 132-134 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22-7.28 (m, 3H), 7.38-7.39 (m, 2H),

7.44-7.57 (m, 5H), 7.67-7.75 (m, 2H), 8.22 (d, J = 7.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ79.8, 120.2, 127.4, 127.6, 128.4, 128.8, 129.4, 129.9, 130.3, 130.5, 130.8, 131.0, 134.6, 135.8, 137.8, 140.2, 142.2, 155.7, 161.5; IR (in CH_2Cl_2, cm^{-1}) 3055, 2985, 1736, 1266; HRMS Calcd for $C_{21}H_{13}IO_2$: 423.99603. Found: 423.99687.



47

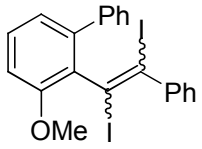
9-Iodo-10-(2-methoxyphenyl)phenanthrene (47). The product was obtained as a colorless solid: mp 135-137 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.69 (s, 3H), 7.09 (d, J = 8.4 Hz, 1H), 7.14-7.15 (m, 2H), 7.40-7.42 (m, 2H), 7.49-7.53 (m, 1H), 7.62-7.70 (m, 3H), 8.44-8.46 (m, 1H), 8.66-8.72 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ55.9, 107.5, 111.6, 121.0, 122.8, 122.9, 127.2, 127.3, 127.5, 128.0, 128.3, 129.8, 130.5, 130.8, 131.6, 132.4, 132.8, 134.3, 134.7, 142.8, 157.0; IR (in CH_2Cl_2, cm^{-1}) 3052, 2985, 1492, 1266; HRMS Calcd for $C_{21}H_{15}IO$: 410.01677. Found: 410.01757.



48

2-(Biphenyl-2-yl)-3-iodobenzofuran (48). The product was obtained as a yellow gel: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31-7.39 (m, 8H), 7.48-7.51 (m, 1H), 7.54-7.58 (m, 1H), 7.64-7.66 (m, 2H), 7.82 (d, J = 7.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ65.7, 111.4,

121.6, 123.4, 125.4, 127.17, 127.21, 128.3, 128.5, 128.8, 130.3, 130.6, 131.5, 132.0, 140.9, 142.7, 154.3, 156.0; IR (in CH_2Cl_2, cm^{-1}) 3053, 2984, 1449, 1263; HRMS Calcd for $C_{20}H_{13}IO$: 396.00112. Found: 396.00172.



49

2-(1,2-Diiodo-2-phenylvinyl)-3-methoxybiphenyl (49). The product was obtained as a dark yellow solid: mp 102-104 ^{o}C; ^{1}H NMR (CD_2Cl_2, 400 MHz) δ 4.00 (s, 3H), 6.98-7.01 (m, 2H), 7.10 (d, J = 7.2 Hz, 2H), 7.28-7.35 (m, 3H), 7.43-7.50 (m, 4H), 7.63 (d, J = 6.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 56.4, 95.5, 101.2, 110.7, 123.0, 127.7, 127.9, 128.0, 128.3, 128.5, 129.7, 130.1, 140.5, 141.6, 147.4, 155.6; IR (in CH_2Cl_2, cm^{-1}) 3053, 2986, 1422, 1265; HRMS Calcd for $C_{21}H_{16}I_2O$: 537.92907. Found: 537.92995.

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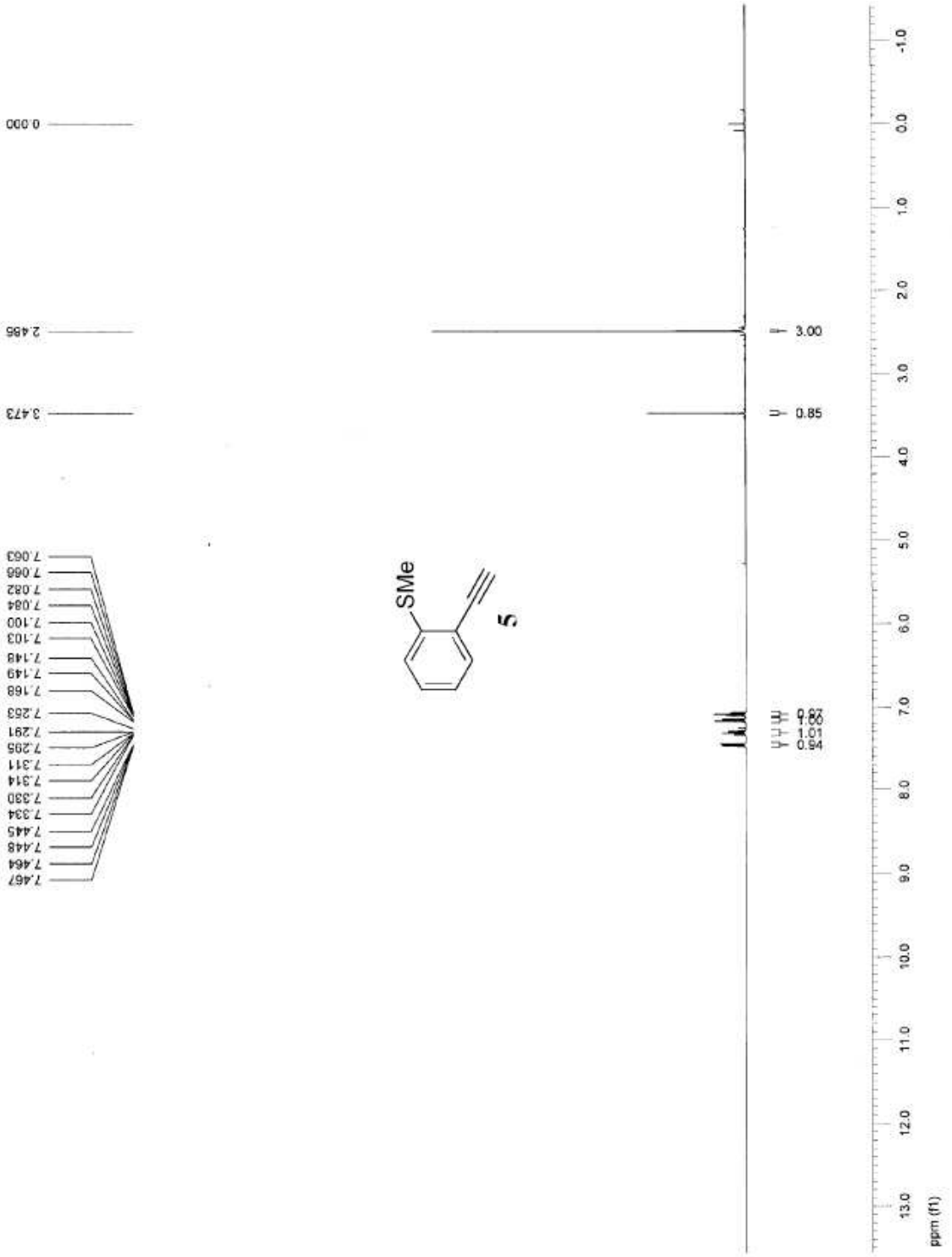
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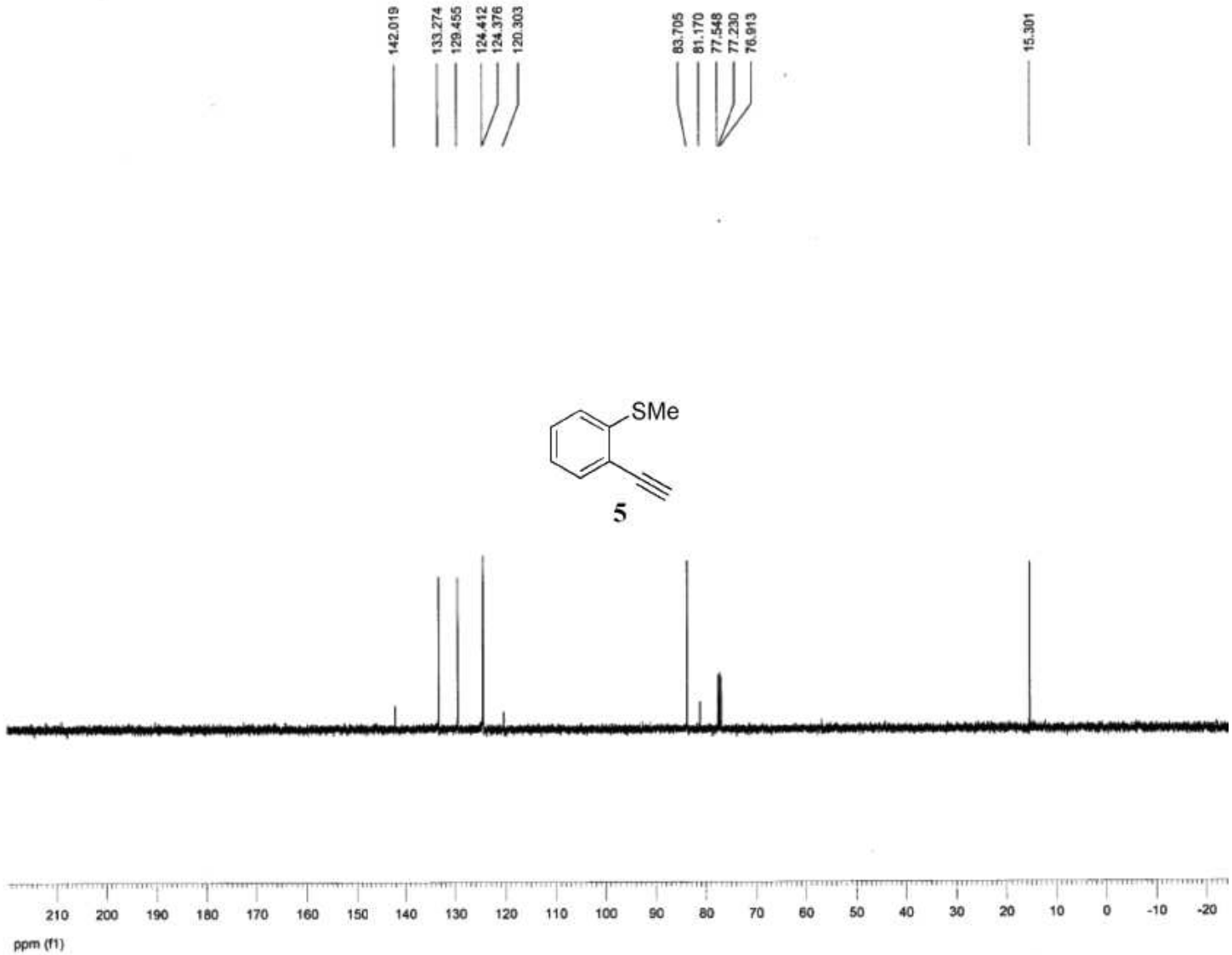
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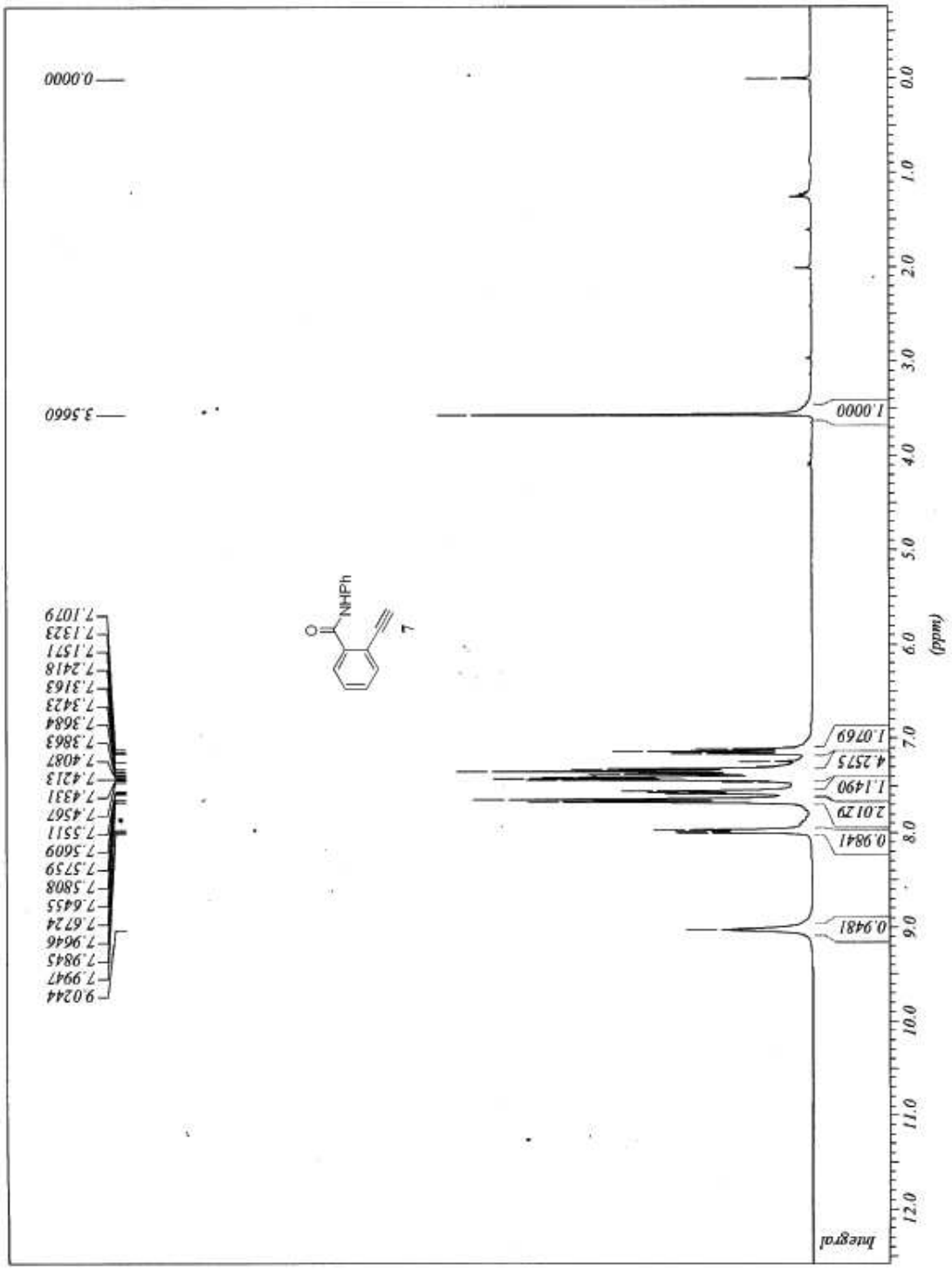
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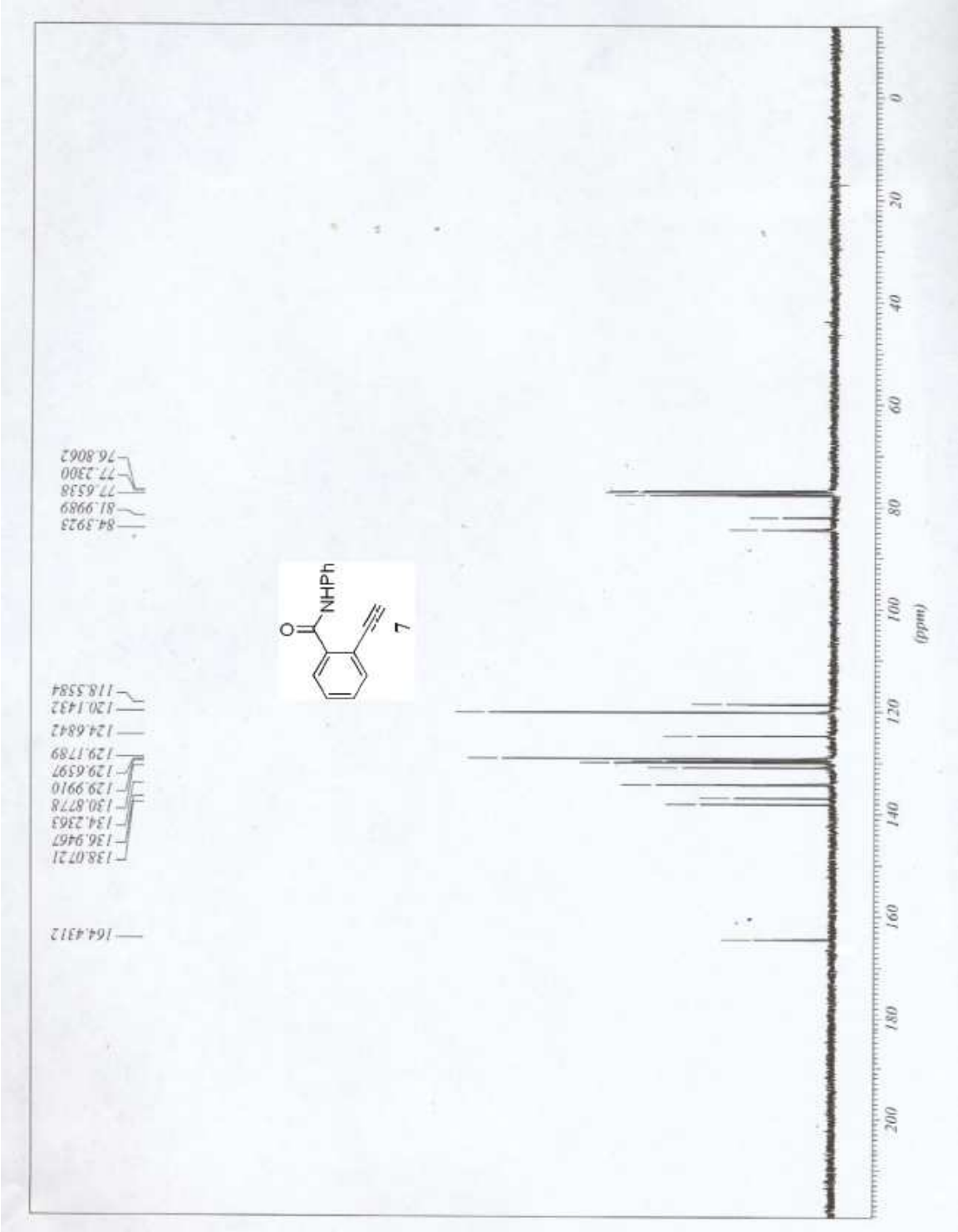
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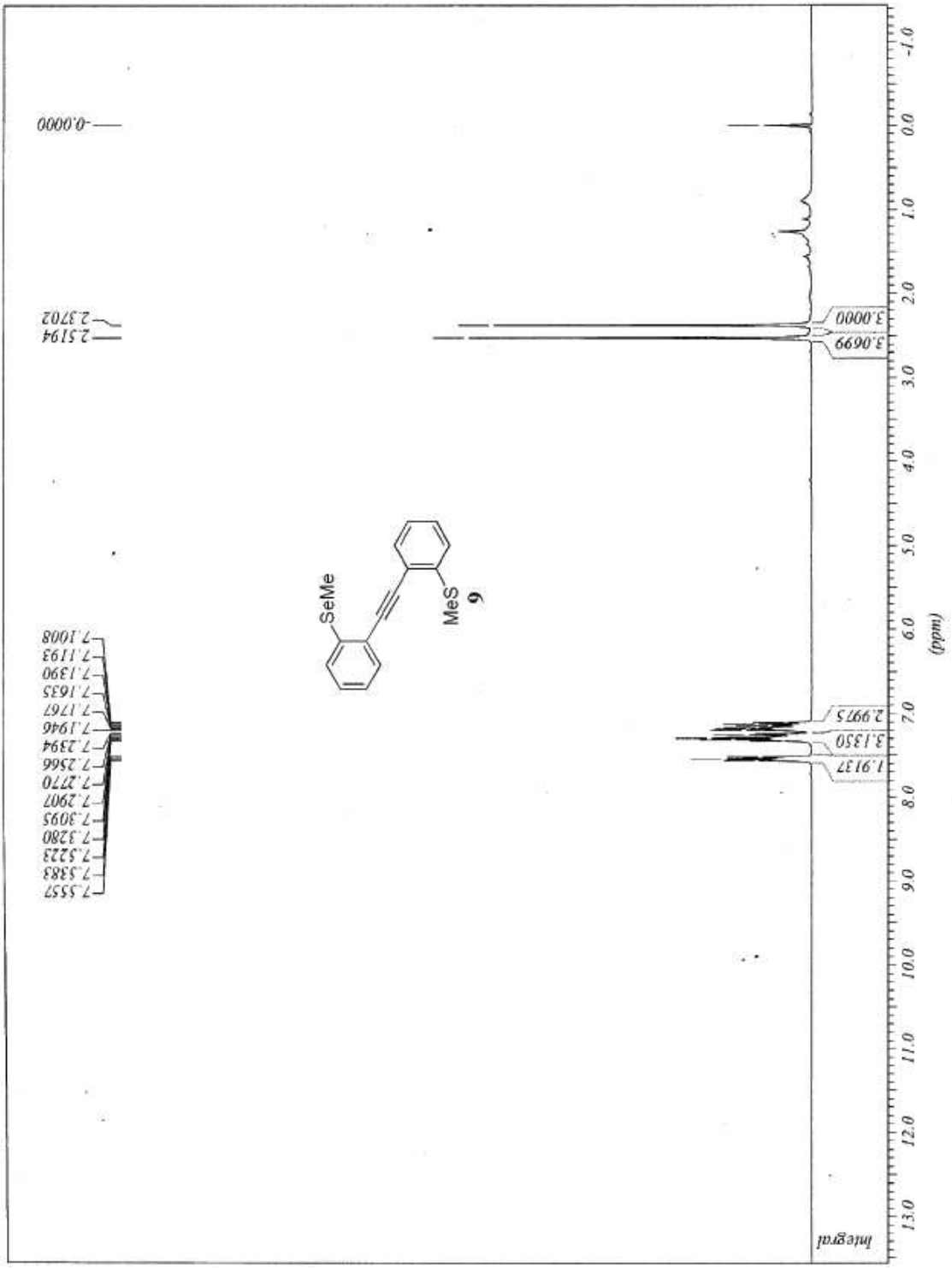
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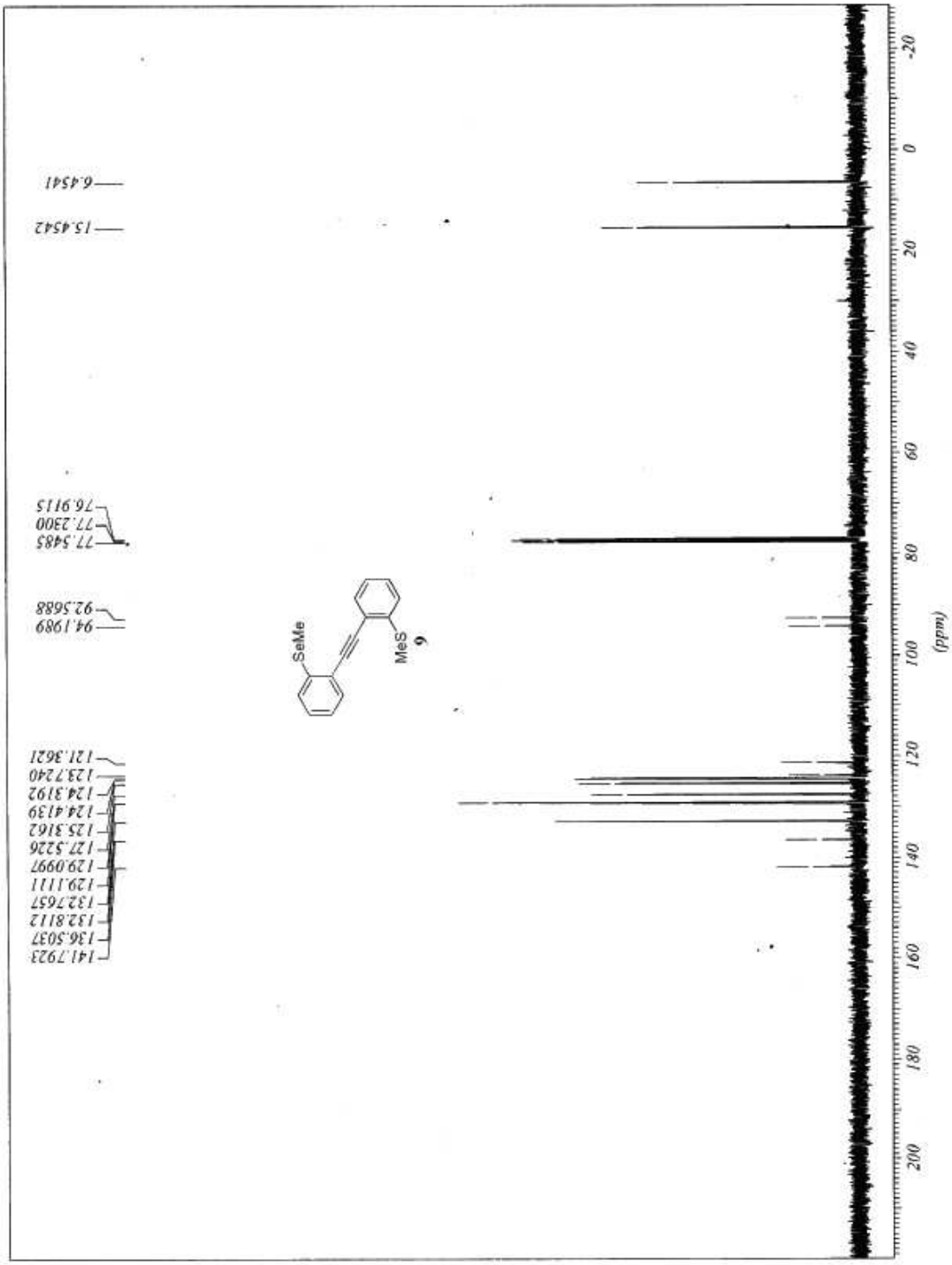
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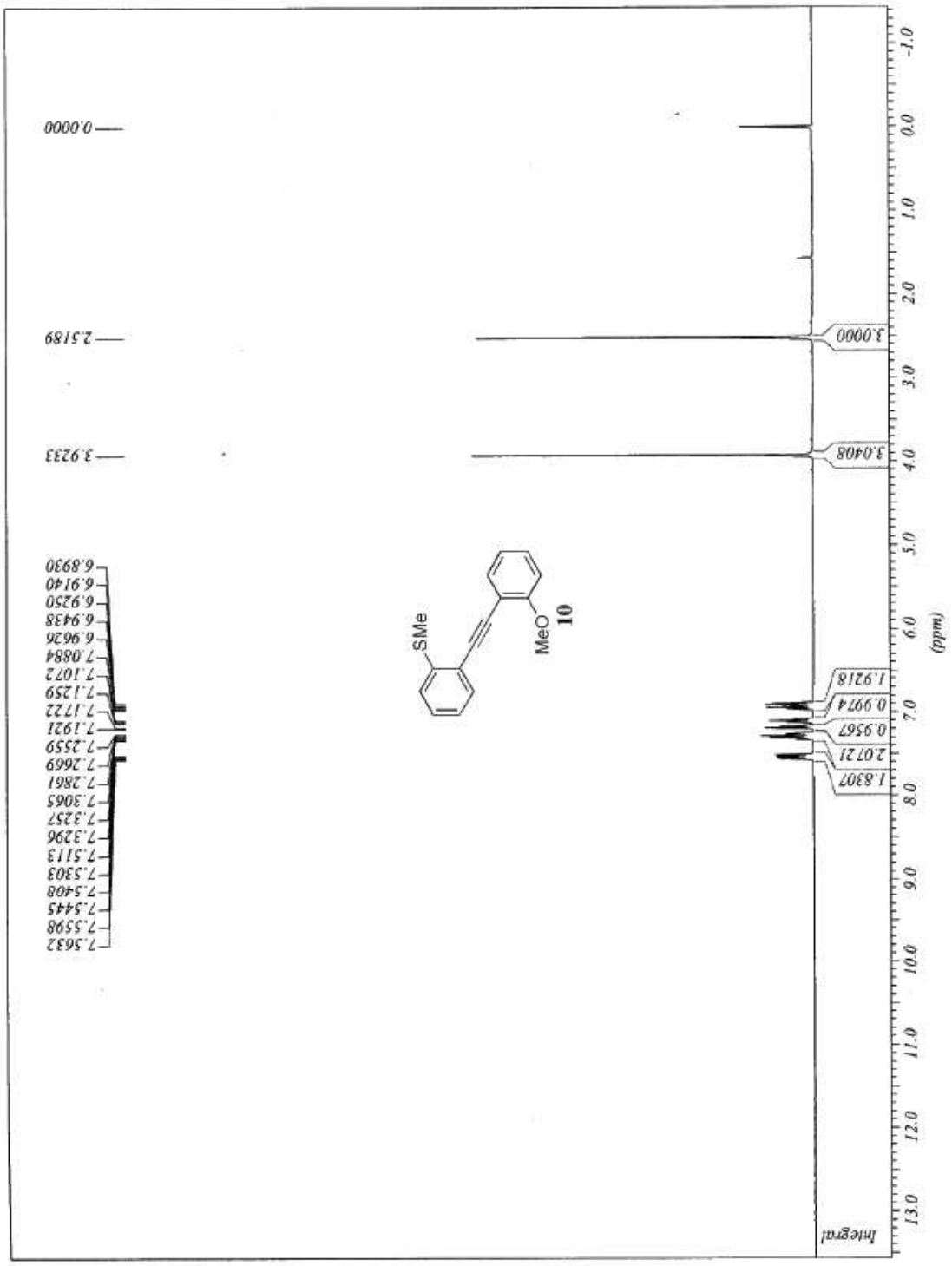
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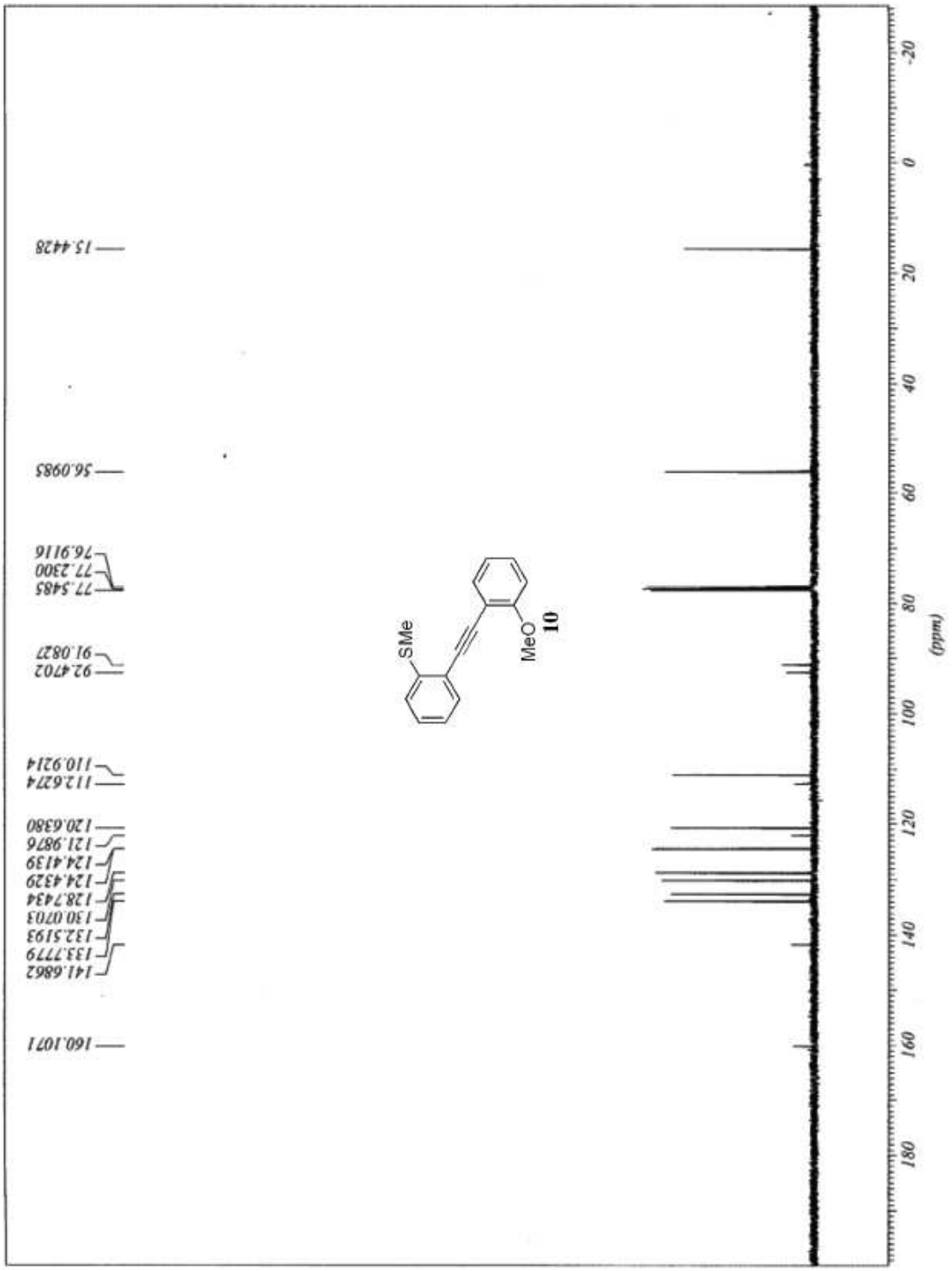
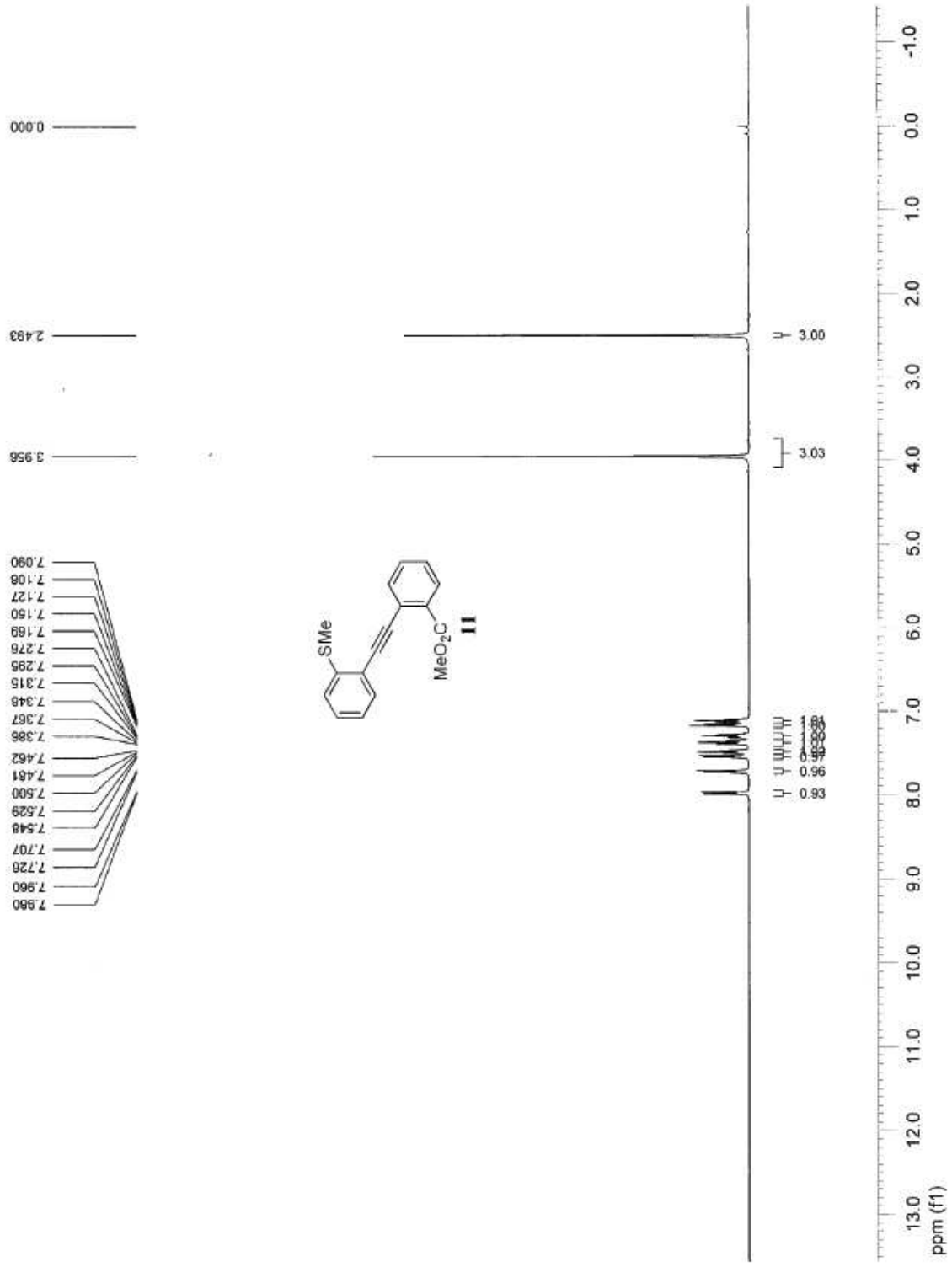
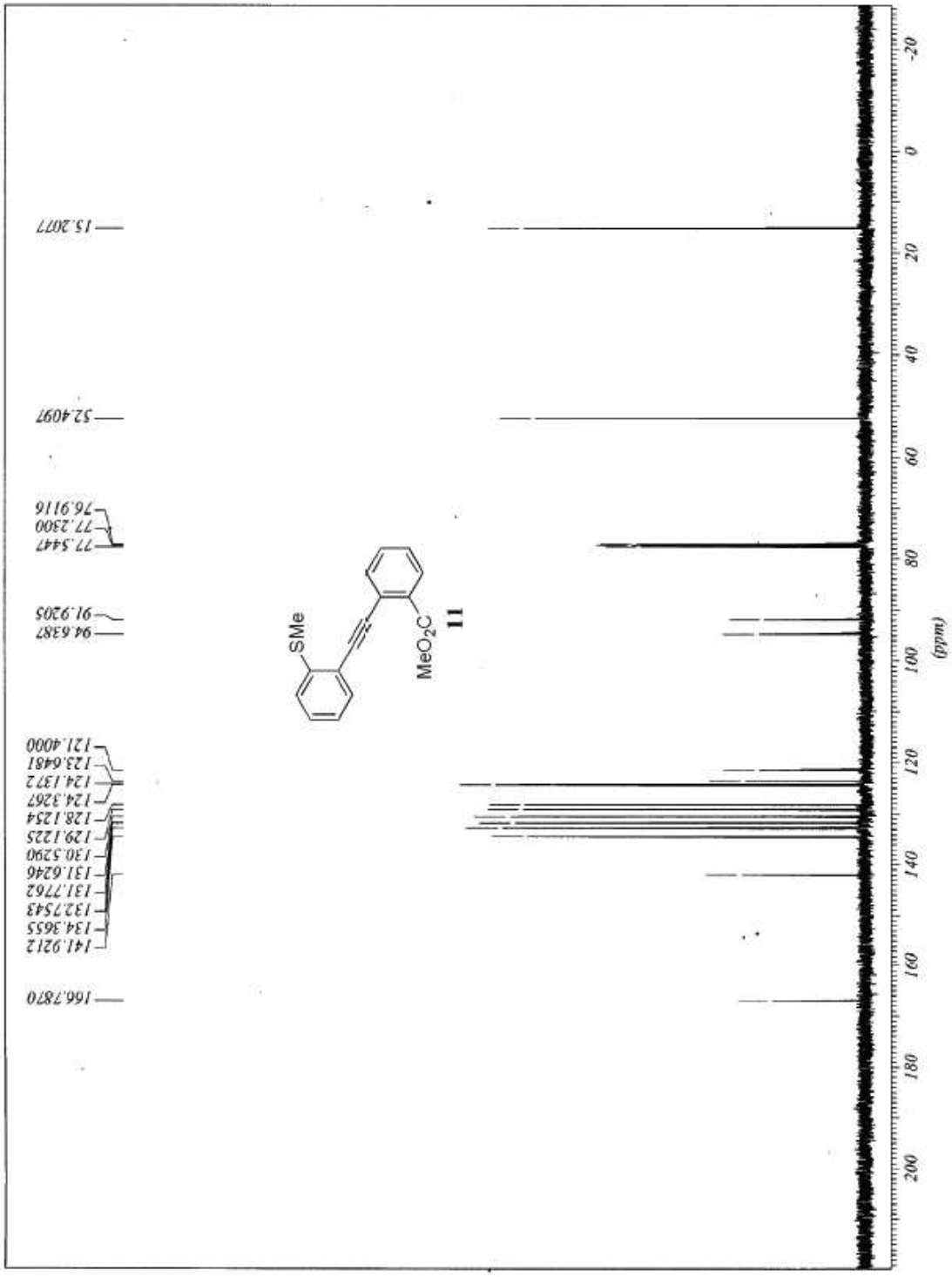
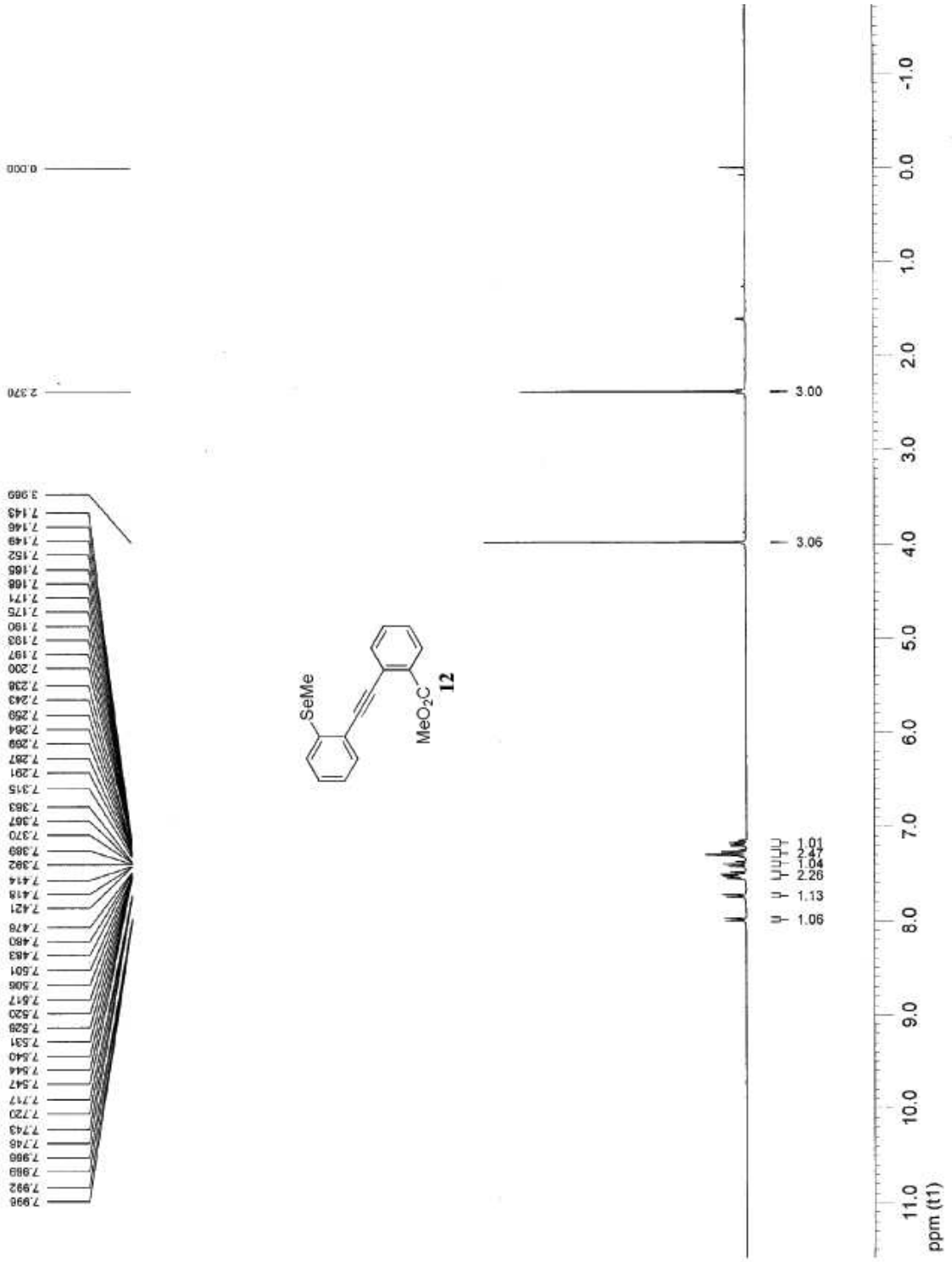
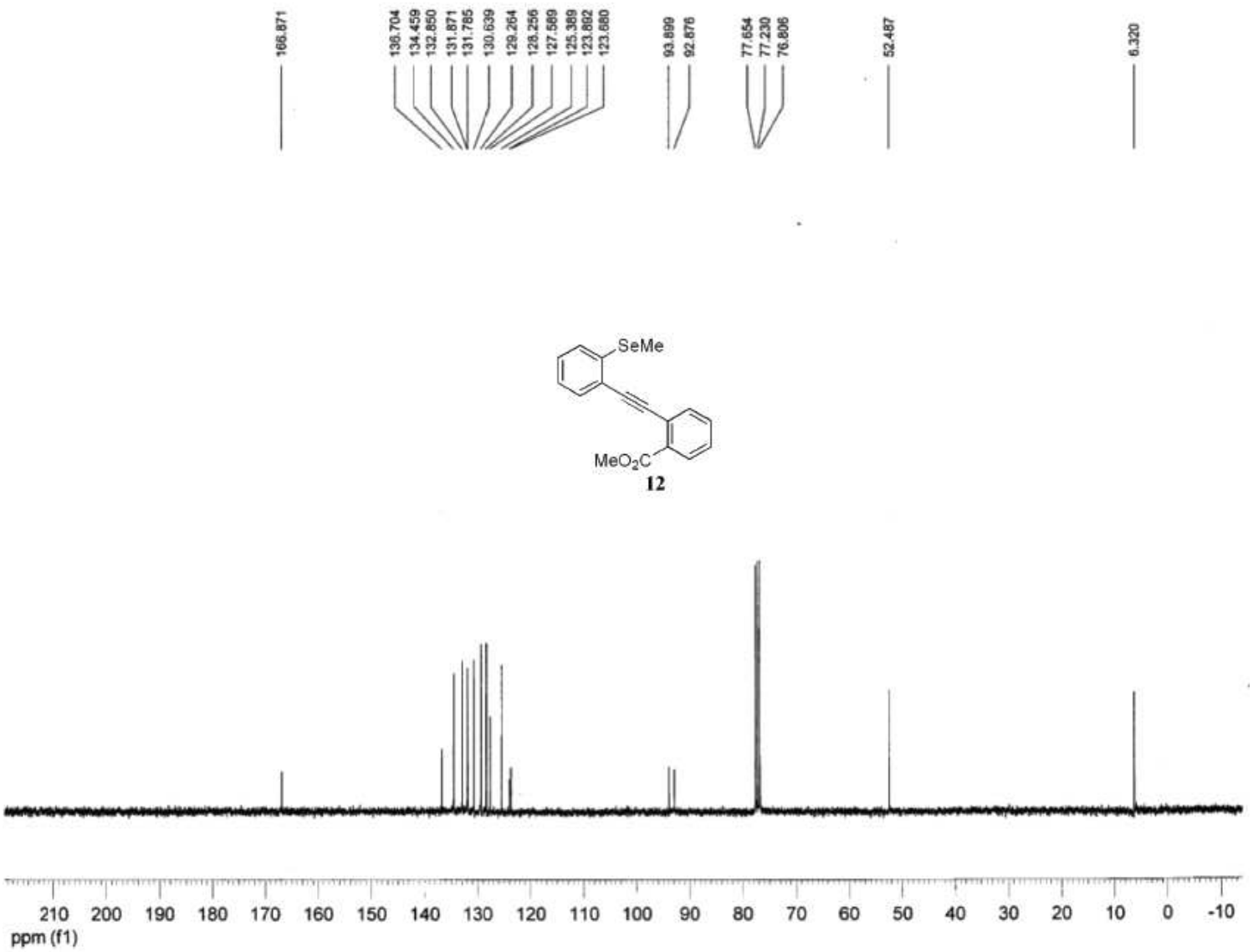
6.8930

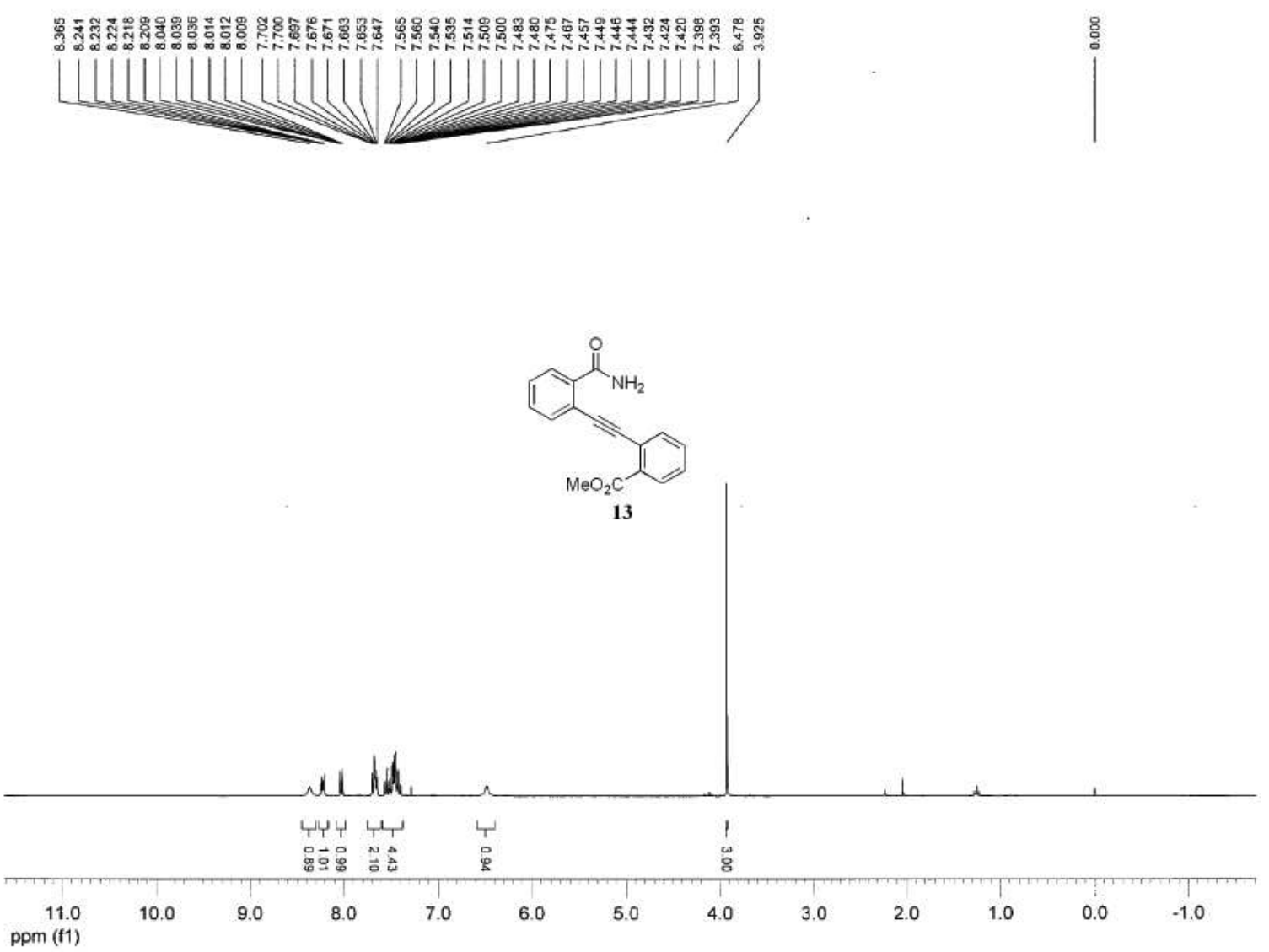

SMe
MeO
10
15.4428
56.0985
76.9116
77.2300
77.5485
91.0827
92.4702
110.9214
112.6274
120.6380
121.9876
124.4139
124.4329
128.7434
130.0703
132.5193
133.7779
141.6862
160.1071
(ppm)

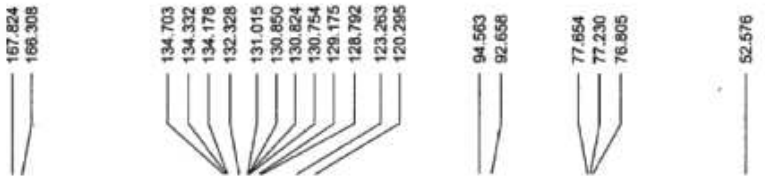

0.000
2.493
3.956
7.090
7.108
7.127
7.150
7.169
7.276
7.295
7.315
7.346
7.367
7.386
7.462
7.481
7.500
7.529
7.548
7.707
7.726
7.960
7.980
SMe
MeO2C
11
3.00
3.03
0.96
0.93
13.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
ppm (f1)

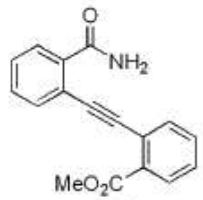

SMe
MeO2C
11
166.7870
141.9212
134.3655
132.7543
131.7762
131.6246
130.5290
129.1225
128.1254
124.3267
124.1372
123.6481
121.4000
94.6387
91.9205
77.5447
77.2300
76.9116
52.4097
15.2077
(ppm)


SeMe
MeO2C
12
3.00
3.06
1.01
2.47
1.04
2.26
1.13
1.06
ppm (t1)

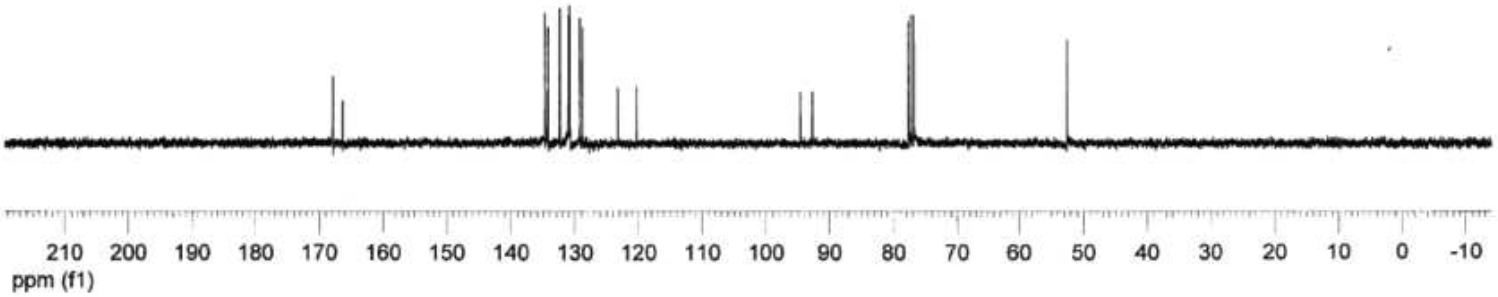

166.871
136.704
134.459
132.850
131.871
131.785
130.639
129.264
128.256
127.589
125.388
123.892
123.680
93.899
92.876
77.654
77.230
76.806
52.487
6.320
SeMe
MeO2C
12
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
ppm (f1)

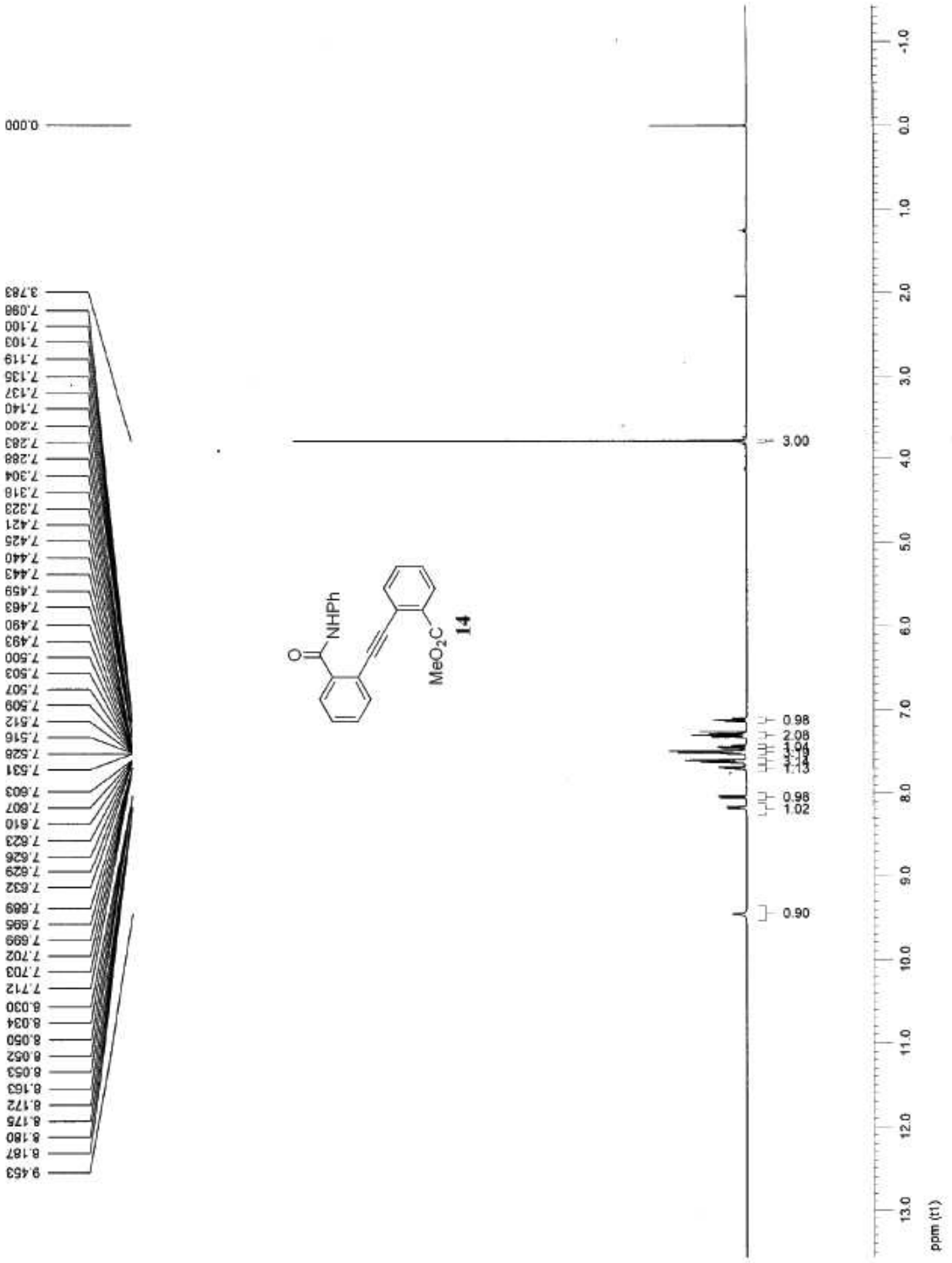

8.365
8.241
8.232
8.224
8.218
8.209
8.040
8.039
8.036
8.014
8.012
8.009
7.702
7.700
7.697
7.676
7.671
7.663
7.653
7.647
7.565
7.560
7.540
7.535
7.514
7.509
7.500
7.483
7.480
7.475
7.467
7.457
7.449
7.446
7.444
7.432
7.424
7.420
7.398
7.393
6.478
3.925
0.000
O
NH2
MeO2C
13
0.89
1.01
0.99
2.10
4.43
0.94
3.00
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
ppm (f1)

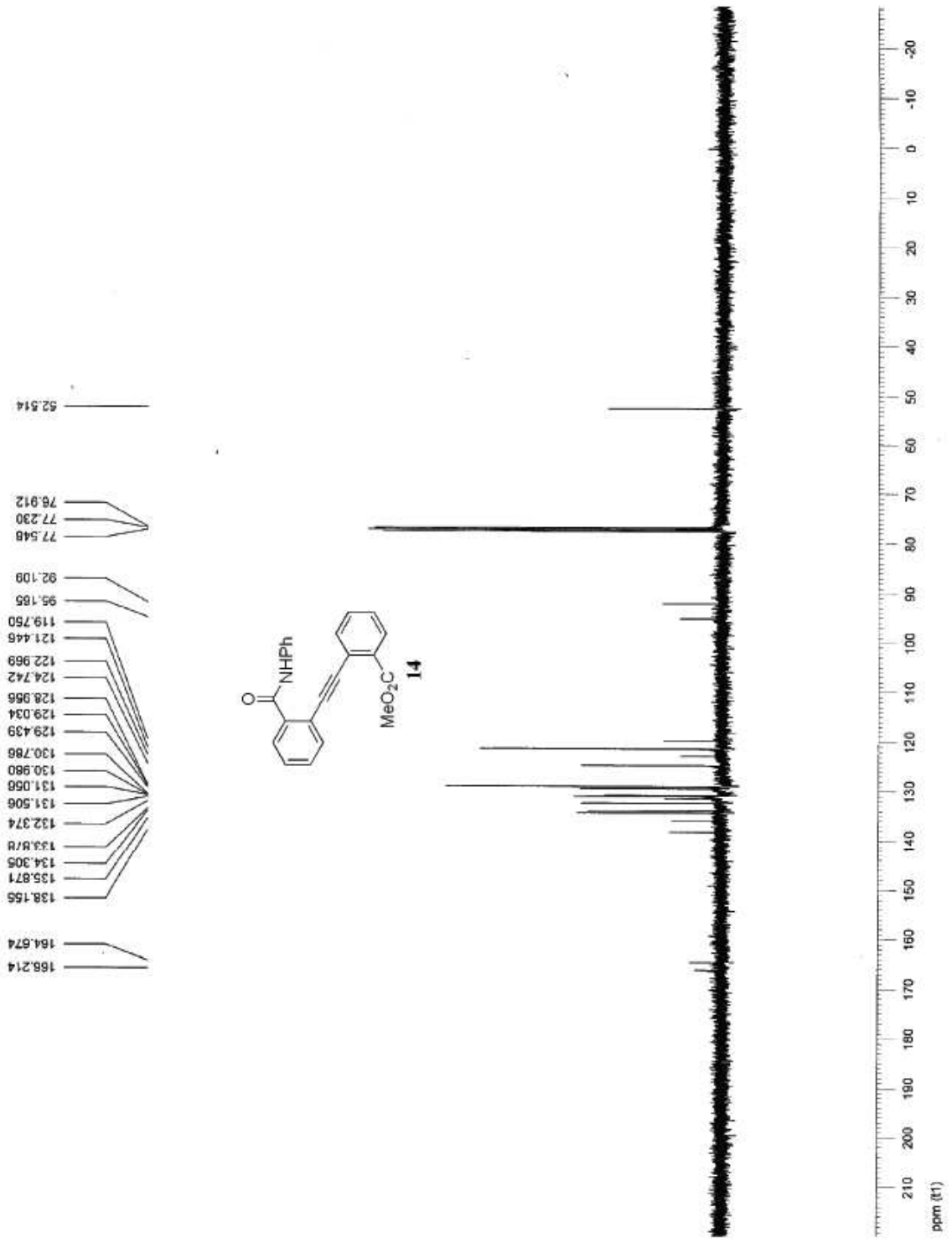

167.824
168.308
134.703
134.332
134.178
132.328
131.015
130.850
130.824
130.754
129.175
128.792
123.263
120.295
94.563
92.658
77.654
77.230
76.805
52.576


O
NH2
MeO2C

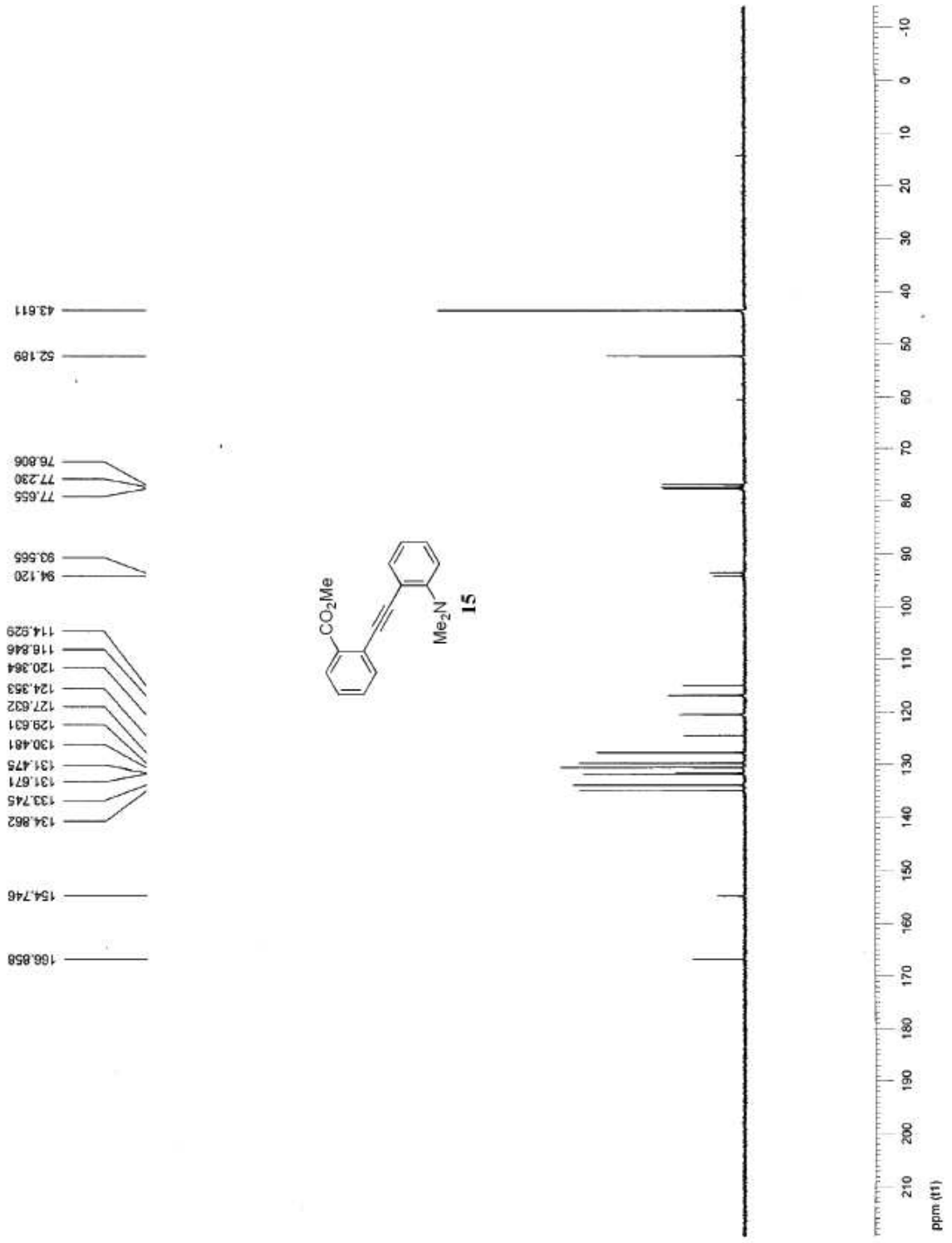
13

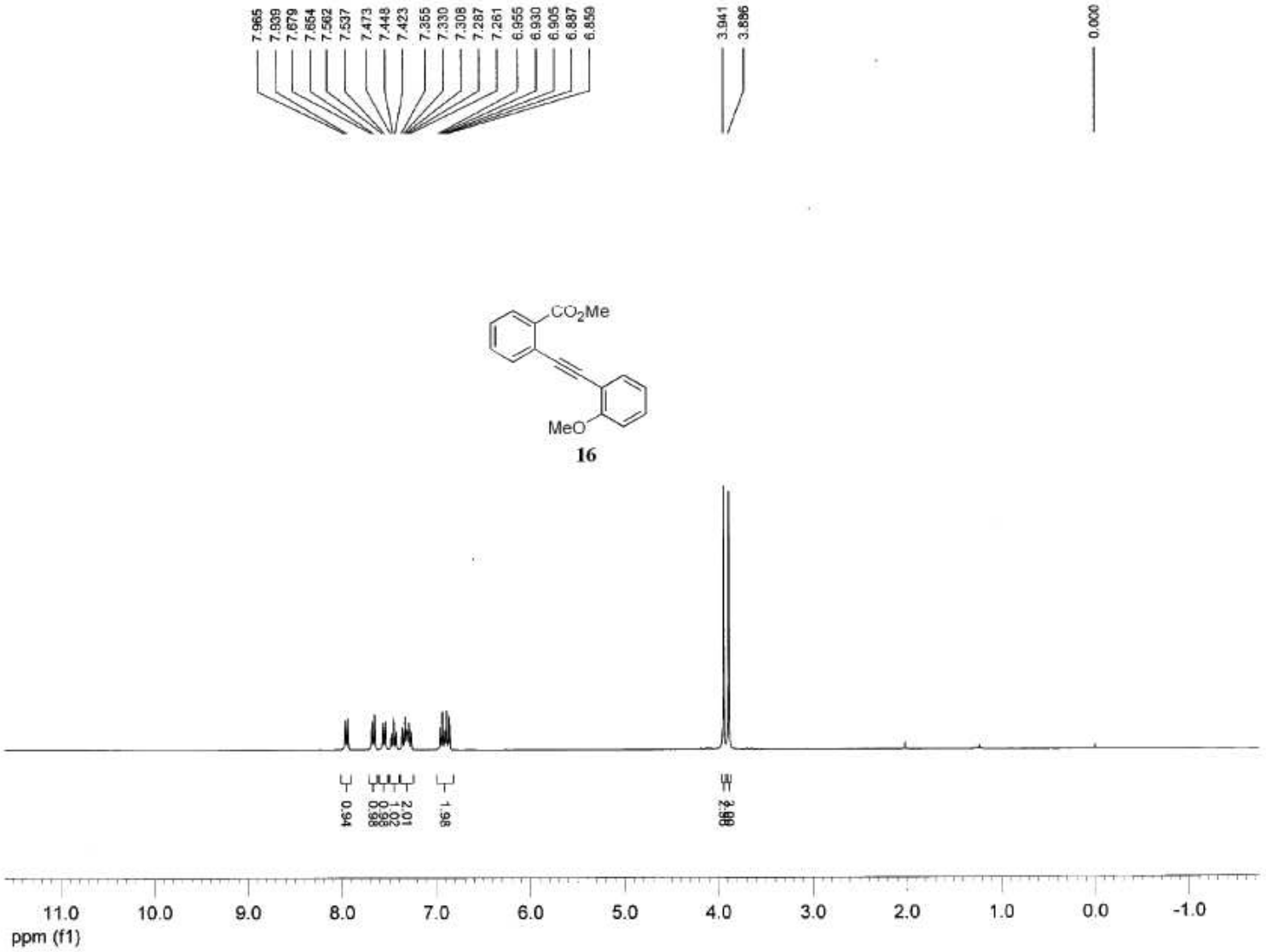

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
ppm (f1)

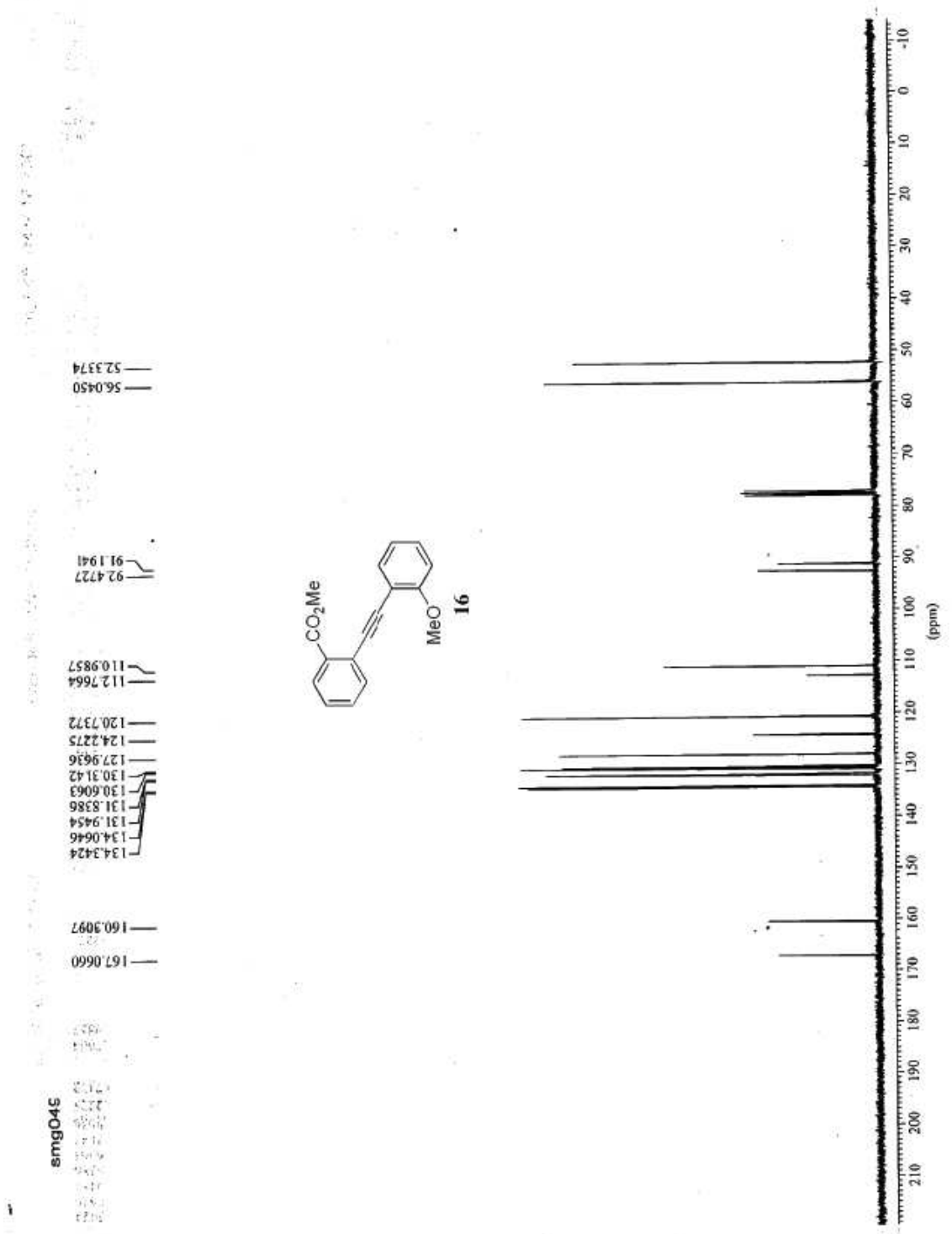

0.000
3.783
7.098
7.100
7.103
7.119
7.135
7.137
7.140
7.200
7.283
7.288
7.304
7.318
7.323
7.421
7.425
7.440
7.443
7.459
7.483
7.490
7.493
7.500
7.503
7.507
7.509
7.512
7.516
7.526
7.531
7.603
7.607
7.610
7.623
7.626
7.629
7.632
7.669
7.695
7.699
7.702
7.703
7.712
8.030
8.034
8.050
8.052
8.053
8.163
8.172
8.175
8.180
8.187
9.453
NHPh
O
MeO2C
14
3.00
0.98
2.08
0.98
1.02
0.90
13.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
ppm (t1)

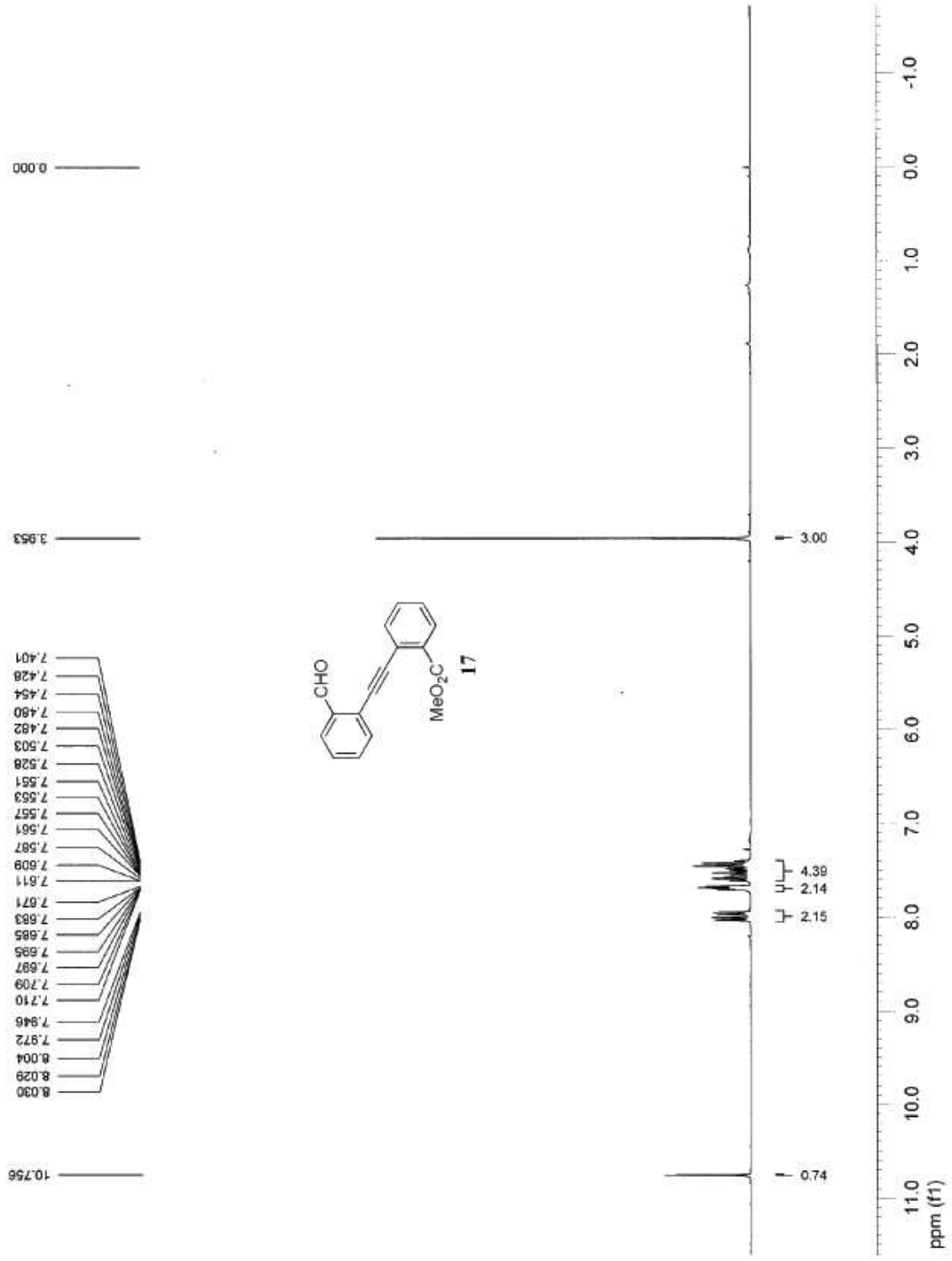

52.514
76.912
77.230
77.548
92.109
95.165
119.750
121.446
122.969
124.742
128.956
129.034
129.439
130.786
130.980
131.056
131.506
132.374
133.879
134.305
135.871
138.155
164.674
166.214
NHPh
O
MeO2C
14
ppm (t1)

0.000
3.003
3.929
6.858
6.882
6.908
7.210
7.215
7.240
7.320
7.325
7.346
7.351
7.421
7.426
7.446
7.451
7.471
7.476
7.537
7.543
7.563
7.568
7.621
7.622
7.625
7.646
7.648
7.651
7.932
7.937
7.958
7.963
CO2Me
Me2N
15
6.00
2.98
2.02
1.02
1.04
1.04
1.00
1.05
0.89
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
ppm (t1)


43.611
52.189
76.806
77.230
77.655
93.565
94.120
114.529
118.846
120.364
124.353
127.632
129.631
130.481
131.475
131.671
133.745
134.862
154.746
166.858
CO2Me
Me2N
15
ppm (t1)

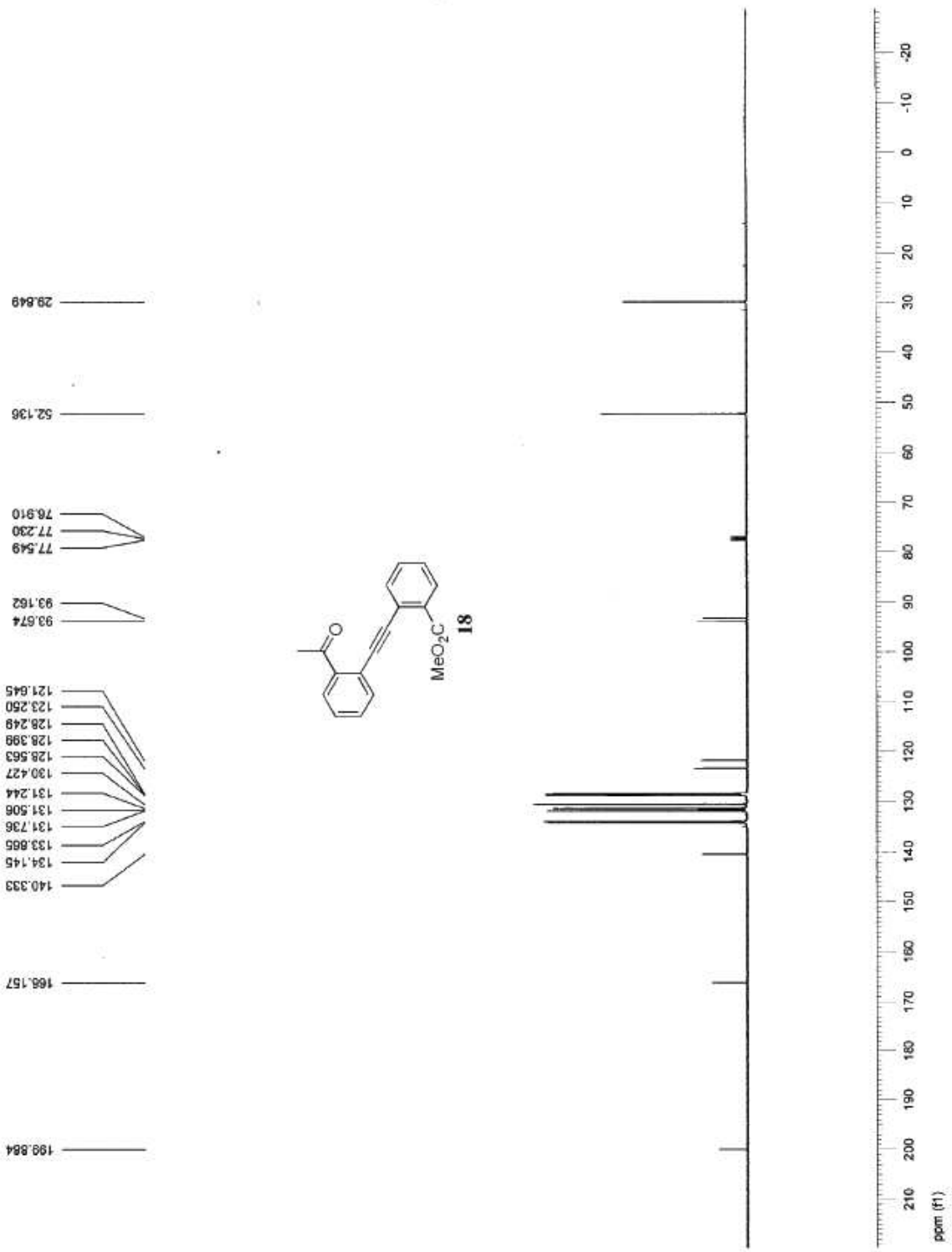

7.965
7.939
7.679
7.654
7.562
7.537
7.473
7.448
7.423
7.355
7.330
7.308
7.287
7.261
6.955
6.930
6.905
6.887
6.859
3.941
3.886
0.000
CO2Me
MeO
16
0.94
0.98
1.00
1.22
1.01
1.98
2.98
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
ppm (f1)

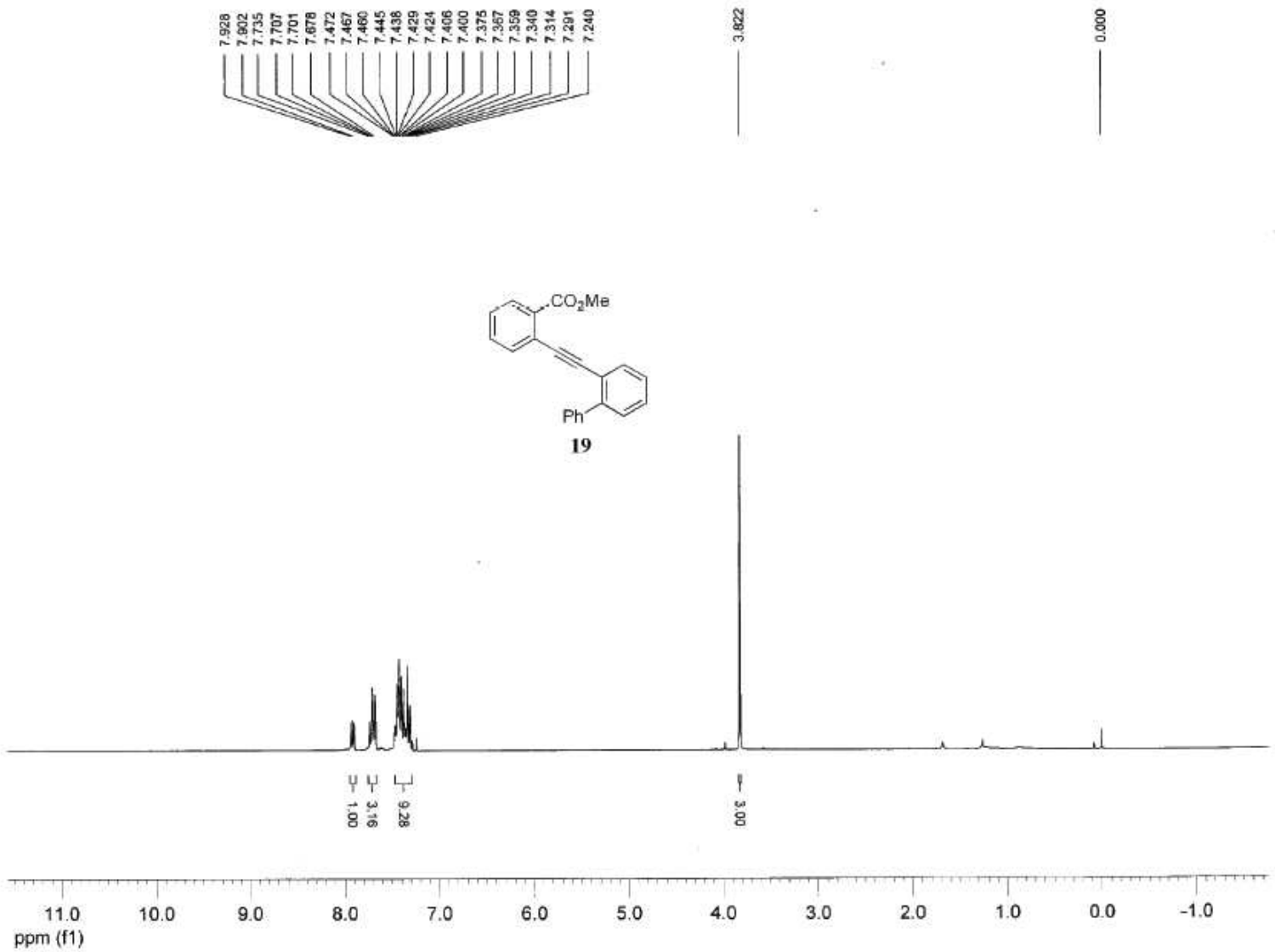

smg045
52.3374
56.0450
91.1941
92.4727
110.9857
112.7664
120.7372
124.2275
127.9636
130.3142
130.6063
131.8386
131.9454
134.0646
134.3424
160.3097
167.0660
CO2Me
MeO
16
(ppm)

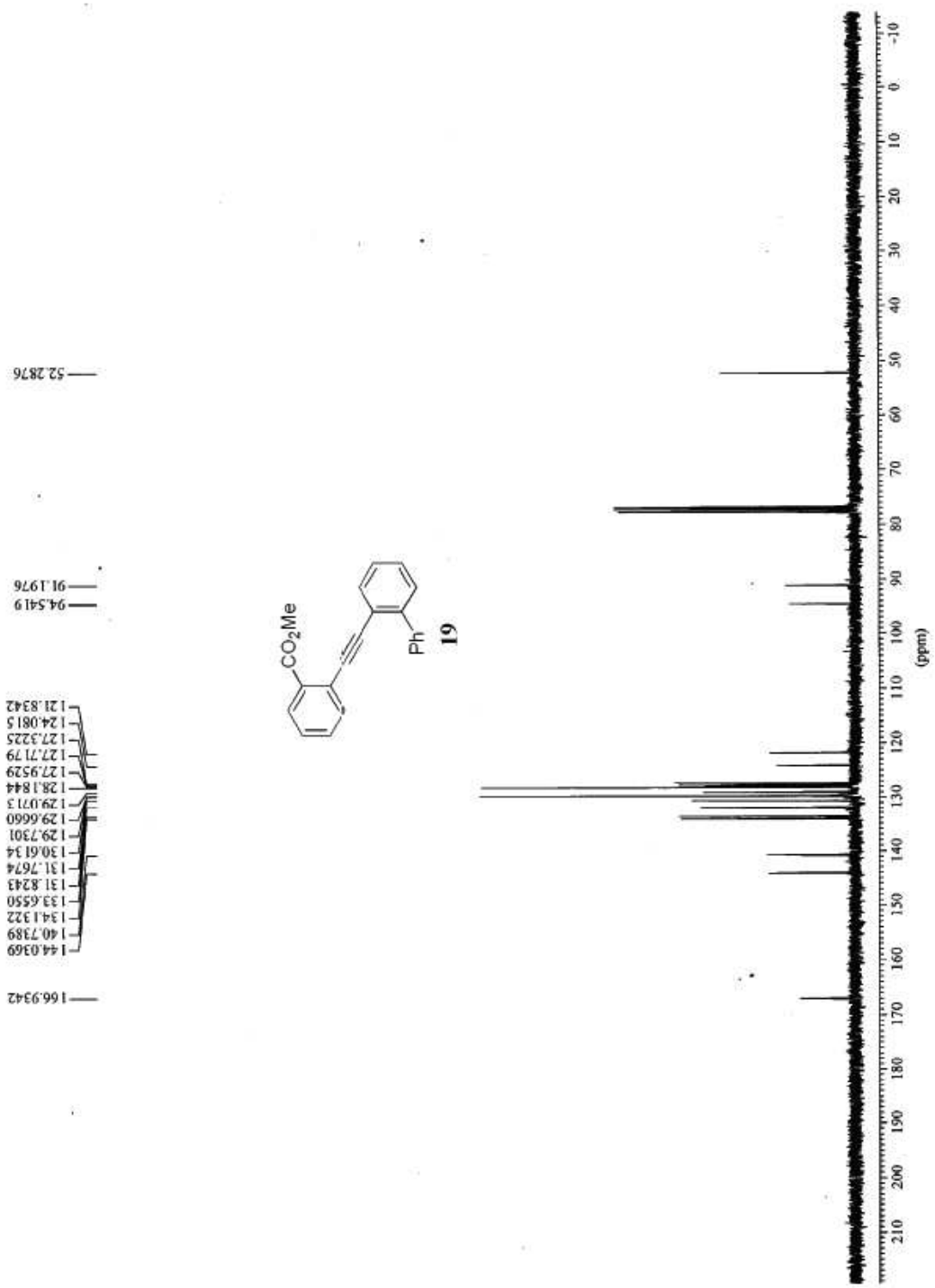

0.000
3.953
10.756
8.030
8.029
8.004
7.972
7.946
7.710
7.709
7.697
7.695
7.685
7.683
7.671
7.611
7.609
7.587
7.561
7.557
7.553
7.551
7.528
7.503
7.482
7.480
7.454
7.428
7.401
CHO
MeO2C
17
3.00
4.39
2.14
2.15
0.74
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
ppm (f1)

52.6188
90.0579
95.1153
123.1341
127.1587
127.2442
128.8825
129.0926
130.9126
131.9169
132.1342
133.6407
133.9470
134.4599
136.5042
166.4178
192.6487
CHO
MeO2C
17
(ppm)

7.9844
7.9821
7.9647
7.9624
7.7631
7.7608
7.7437
7.7411
7.7148
7.7128
7.6956
7.6759
7.6741
7.6567
7.4876
7.4839
7.4682
7.4647
7.4611
7.3984
7.3956
7.3764
7.3574
3.9273
2.7928
0.0000
MeO2C
18
Integral
0.9550
0.9788
1.9851
2.0971
2.0546
3.0000
2.8610
(ppm)

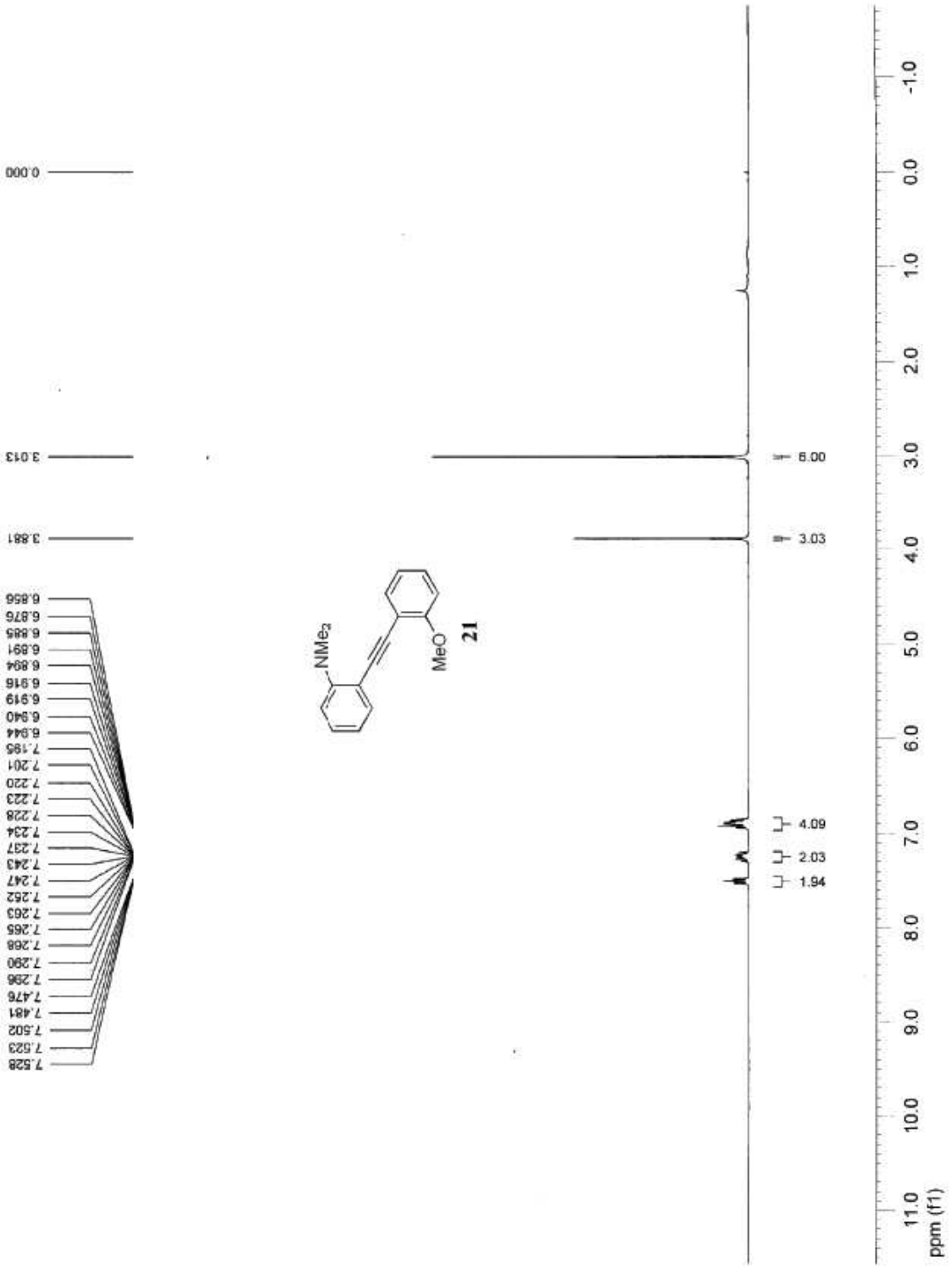

198.864
166.157
140.333
134.145
133.865
131.736
131.506
131.244
130.427
128.563
128.399
128.248
123.250
121.645
93.574
93.162
77.549
77.230
76.910
52.136
29.849
MeO2C
18
ppm (f1)

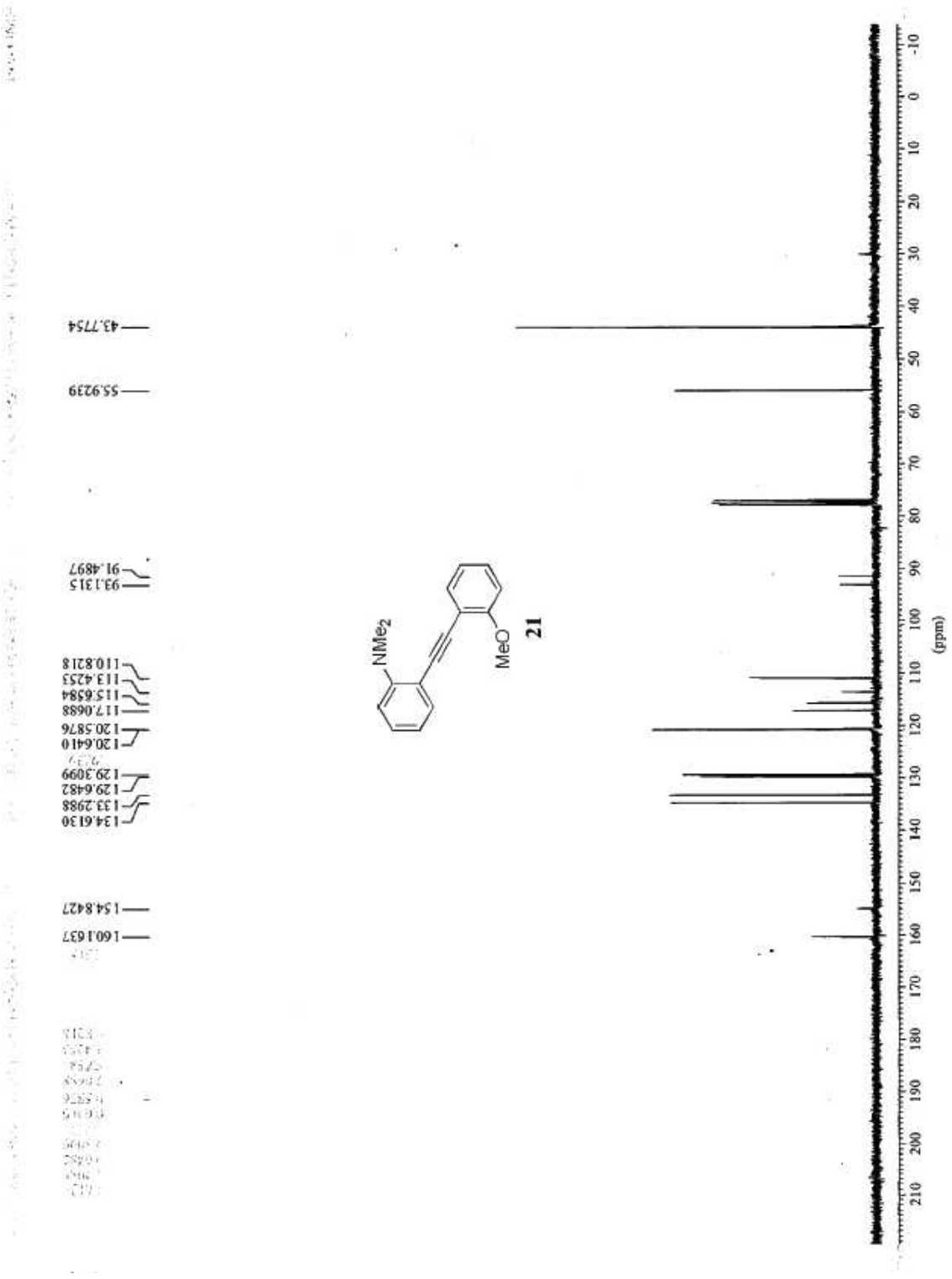

7.928
7.902
7.735
7.707
7.701
7.678
7.472
7.467
7.460
7.445
7.438
7.429
7.424
7.406
7.400
7.375
7.367
7.359
7.340
7.314
7.291
7.240
3.822
0.000
CO2Me
Ph
19
1.00
3.16
9.28
3.00
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
ppm (f1)

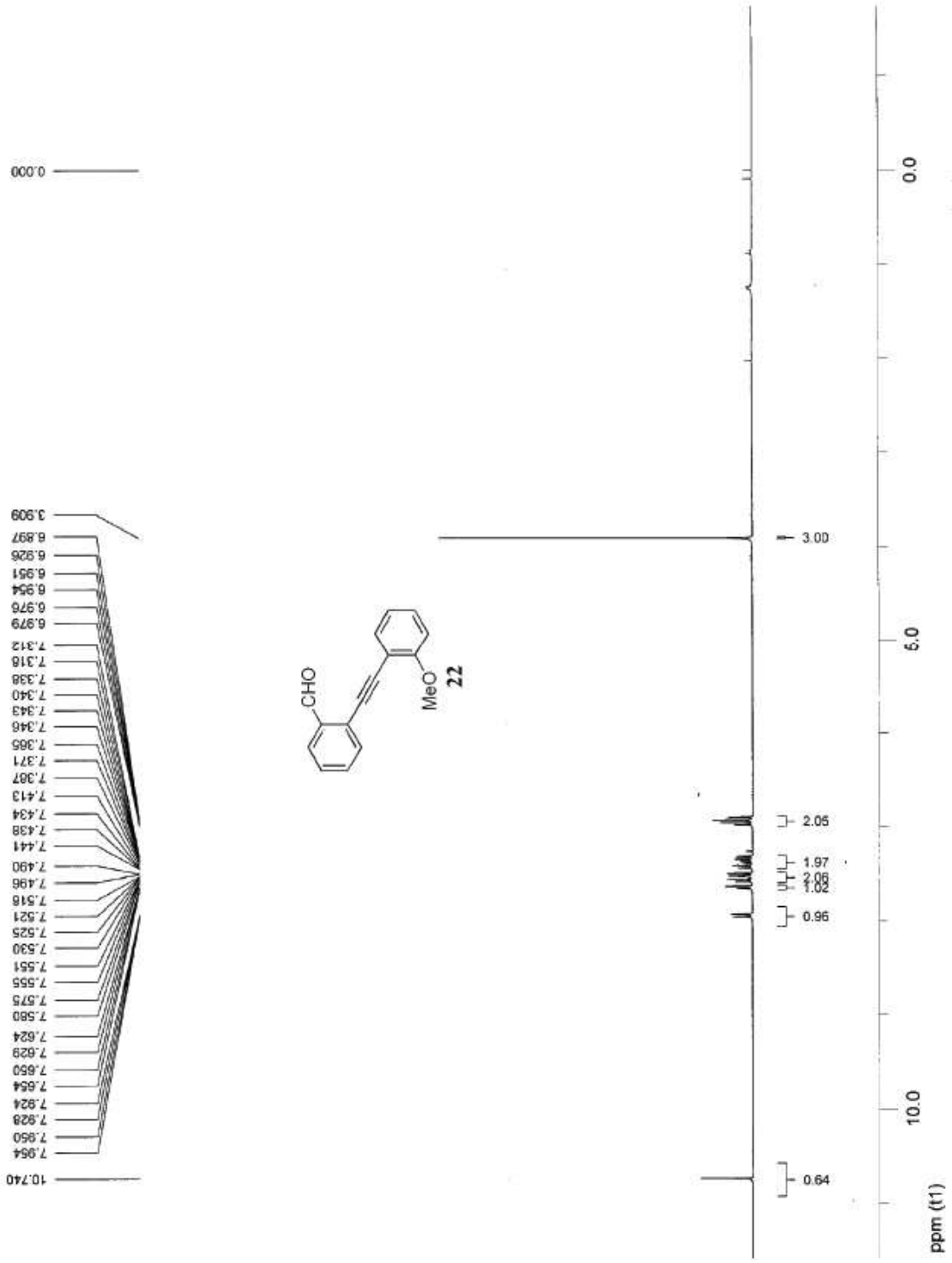

52.2876
91.1976
94.5419
121.8342
124.0815
127.3225
127.7179
127.9529
128.1844
129.0713
129.6660
129.7301
130.6134
131.7674
131.8243
133.6550
134.1322
140.7389
144.0369
166.9342
CO2Me
Ph
19
(ppm)

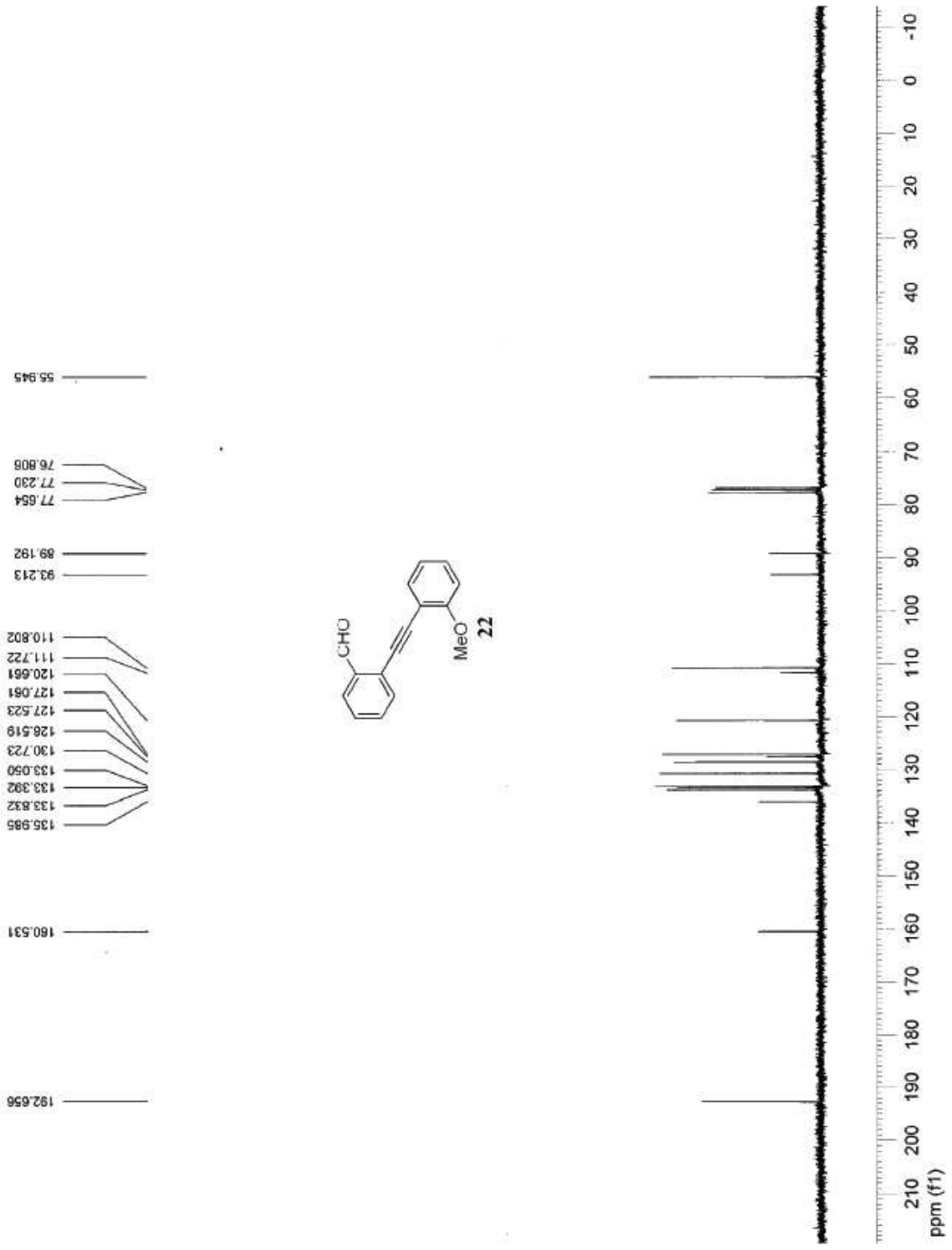
0.000
2.790
6.781
6.806
6.820
6.827
6.828
6.846
7.157
7.181
7.209
7.224
7.250
7.280
7.286
7.304
7.310
7.334
7.356
7.361
7.386
7.397
7.410
7.423
7.440
7.446
7.646
7.669
NMe2
Ph
20
6.00
2.18
9.45
2.34
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
ppm (f1)

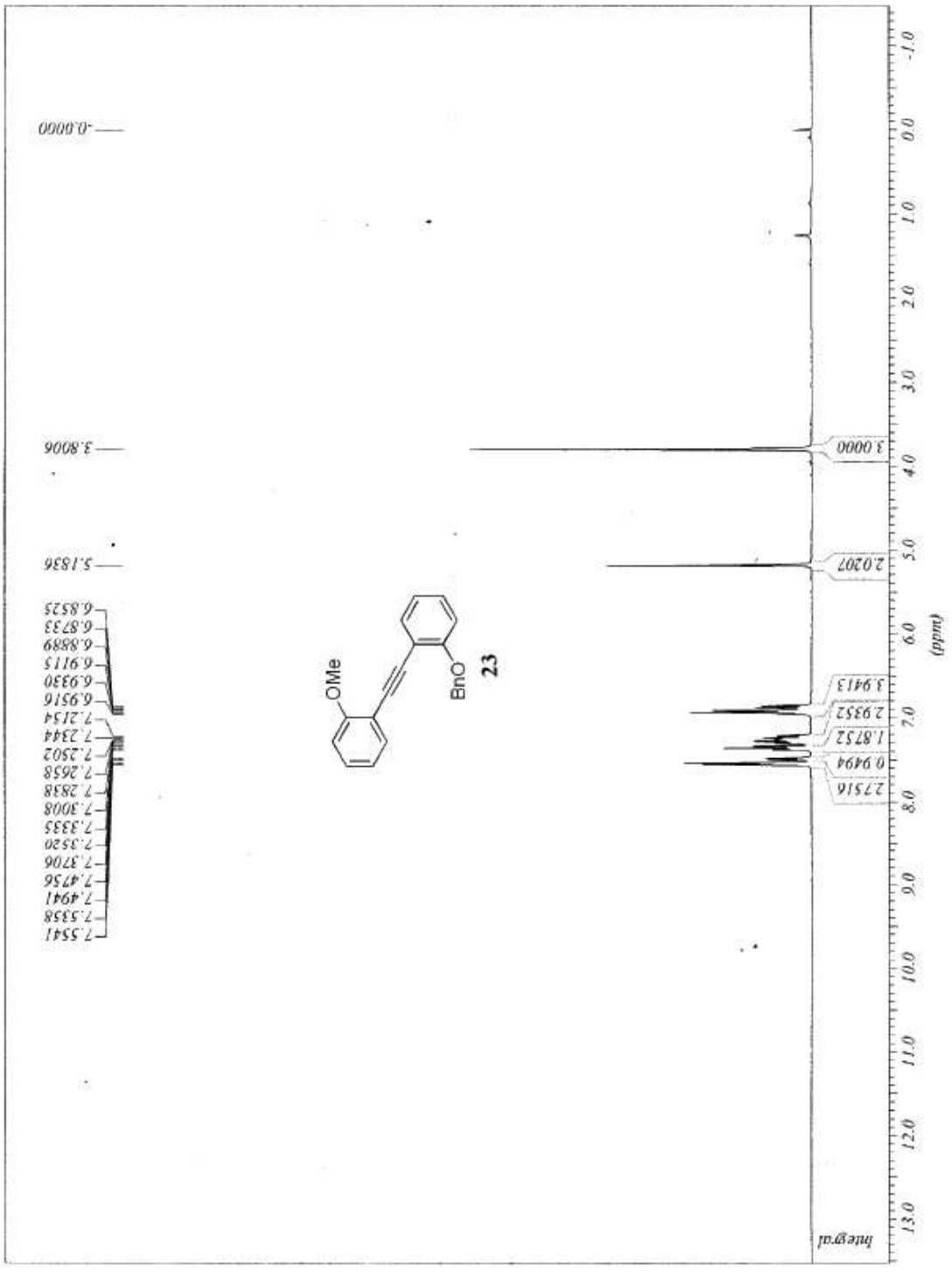
43.4481
76.9116
77.2300
77.5447
91.7800
94.6422
115.3716
116.9031
120.3909
122.4380
127.1655
127.4915
128.1018
128.3748
129.2619
129.6031
129.6675
132.8710
134.4442
140.9193
143.6337
154.6466
NMe2
Ph
20
-20
0
20
40
60
80
100
120
140
160
180
200
(ppm)

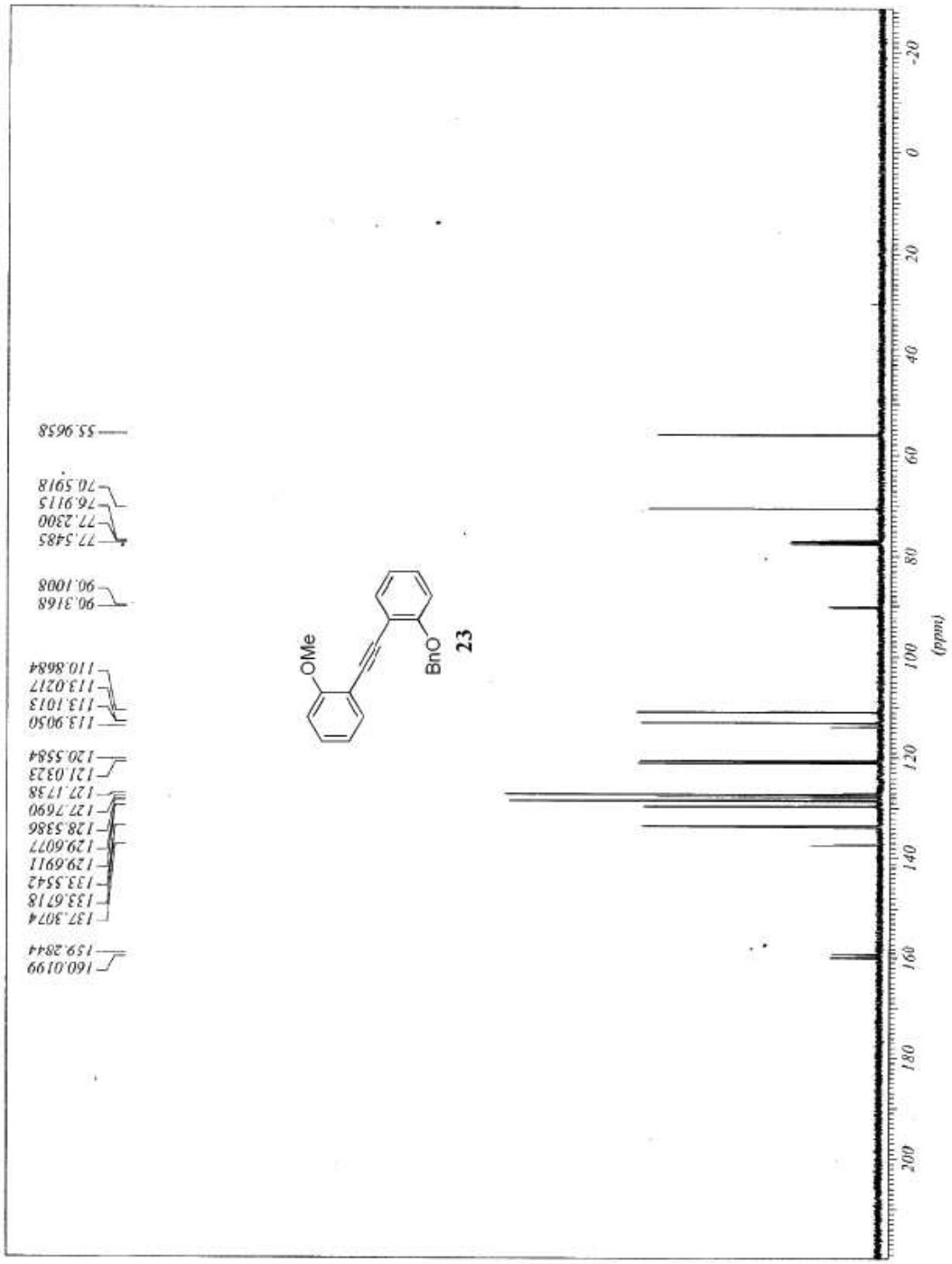

0.000
3.013
3.881
6.856
6.876
6.885
6.891
6.894
6.916
6.919
6.940
6.944
7.185
7.201
7.220
7.223
7.228
7.234
7.237
7.243
7.247
7.262
7.263
7.265
7.268
7.290
7.296
7.476
7.481
7.502
7.523
7.528
NMe2
MeO
21
6.00
3.03
4.09
2.03
1.94
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
ppm (f1)


43.7754
55.9239
91.4897
93.1315
110.8218
113.4253
115.6584
117.0688
120.5876
120.6410
129.3099
129.6482
133.2988
134.6130
154.8427
160.1637
NMe2
MeO
21
(ppm)

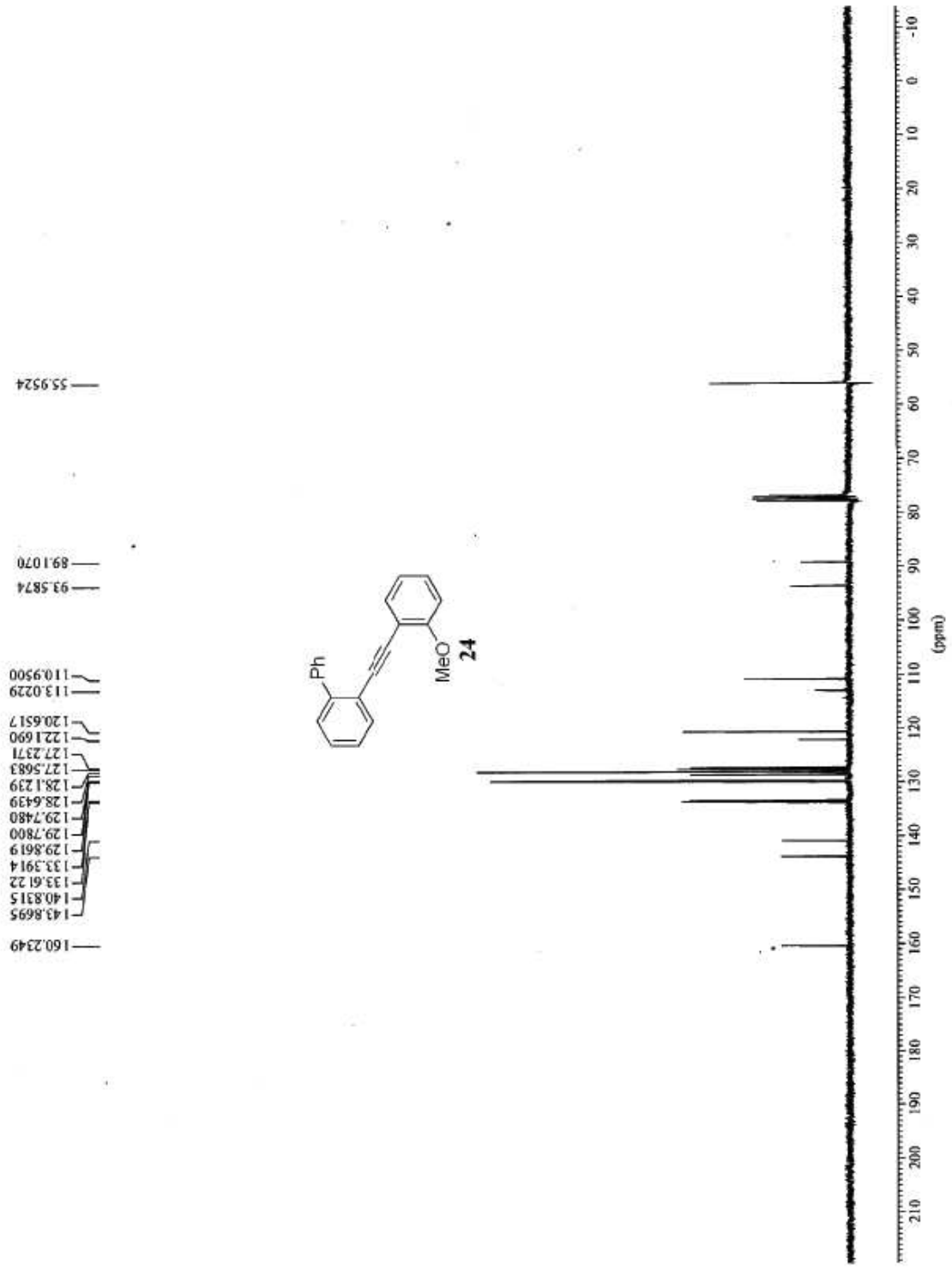

0.000
3.909
6.897
6.926
6.951
6.954
6.976
6.979
7.312
7.316
7.338
7.340
7.343
7.346
7.365
7.371
7.387
7.413
7.434
7.438
7.441
7.490
7.496
7.516
7.521
7.525
7.530
7.551
7.555
7.575
7.580
7.624
7.629
7.650
7.654
7.924
7.928
7.950
7.964
10.740
CHO
MeO
22
3.00
2.05
1.97
2.06
1.02
0.96
0.64
0.0
5.0
10.0
ppm (t1)


55.945
76.808
77.230
77.654
89.192
93.213
110.802
111.722
120.661
127.061
127.523
128.519
130.723
133.050
133.392
133.832
135.985
160.531
192.656
CHO
MeO
22
ppm (f1)


OMe
BnO
23
3.0000
2.0207
3.9413
2.9352
1.8752
0.9494
2.7516
(ppm)


OMe
BnO
23
55.9658
70.5918
76.9115
77.2300
77.5485
90.1008
90.3168
110.8684
113.0217
113.1013
113.9050
120.5584
121.0323
127.1738
127.7690
128.5386
129.6077
129.6911
133.5542
133.6718
137.3074
159.2844
160.0199
(ppm)

3.7764
7.7163
7.7119
7.6887
7.6655
7.6610
7.4445
7.4213
7.3948
7.3883
7.3708
7.3468
7.3248
7.3163
7.3094
7.2906
7.2853
7.2609
7.2560
7.2365
7.2316
7.2121
7.1860
6.8609
6.8360
6.8096
6.7807
Ph
MeO
24
Integral
3.0000
1.9714
8.1226
2.9246
(ppm)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93.5874
110.9500
113.0229
120.6517
122.1690
127.2371
127.5683
128.1239
128.6439
129.7480
129.7800
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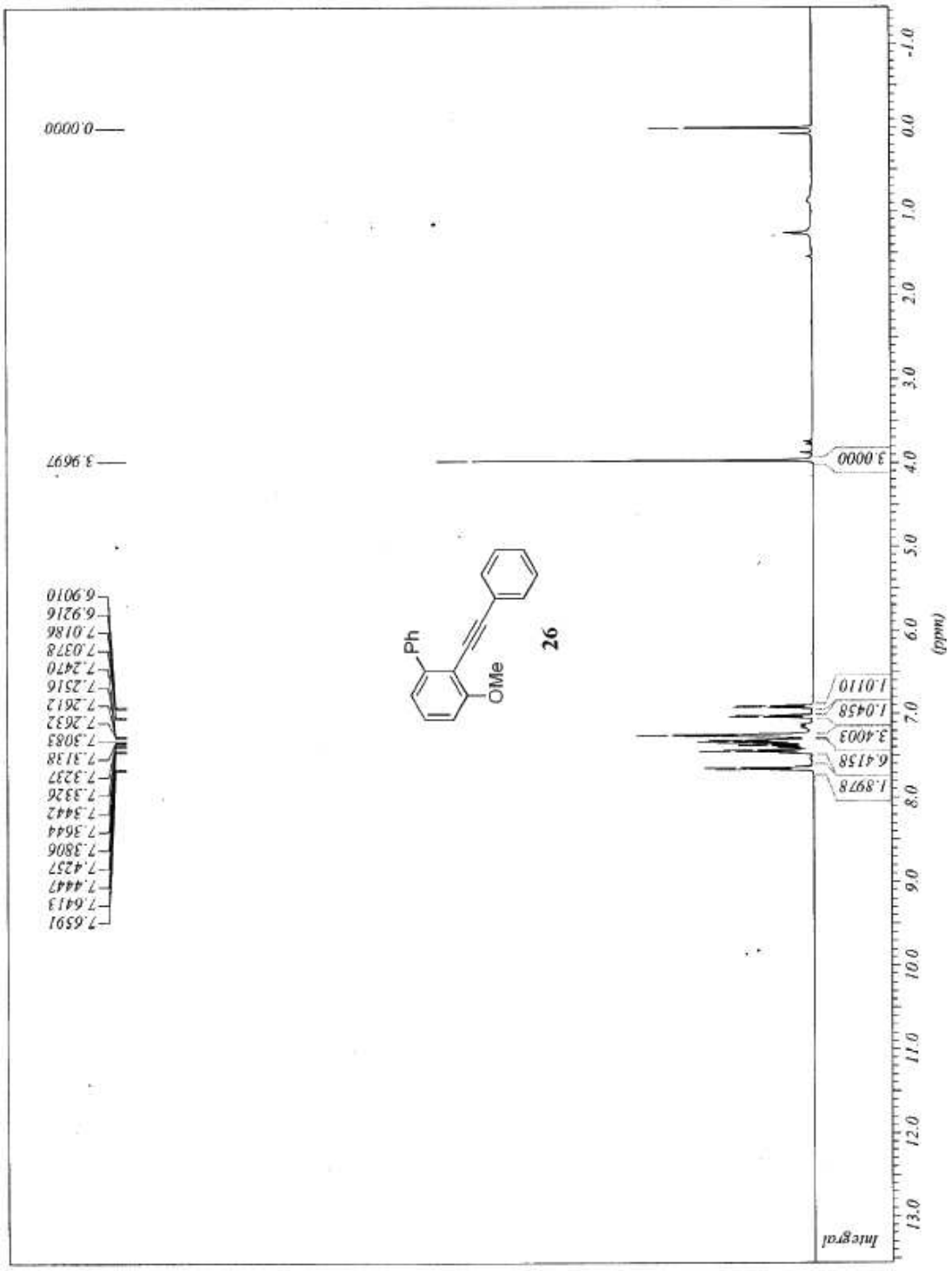
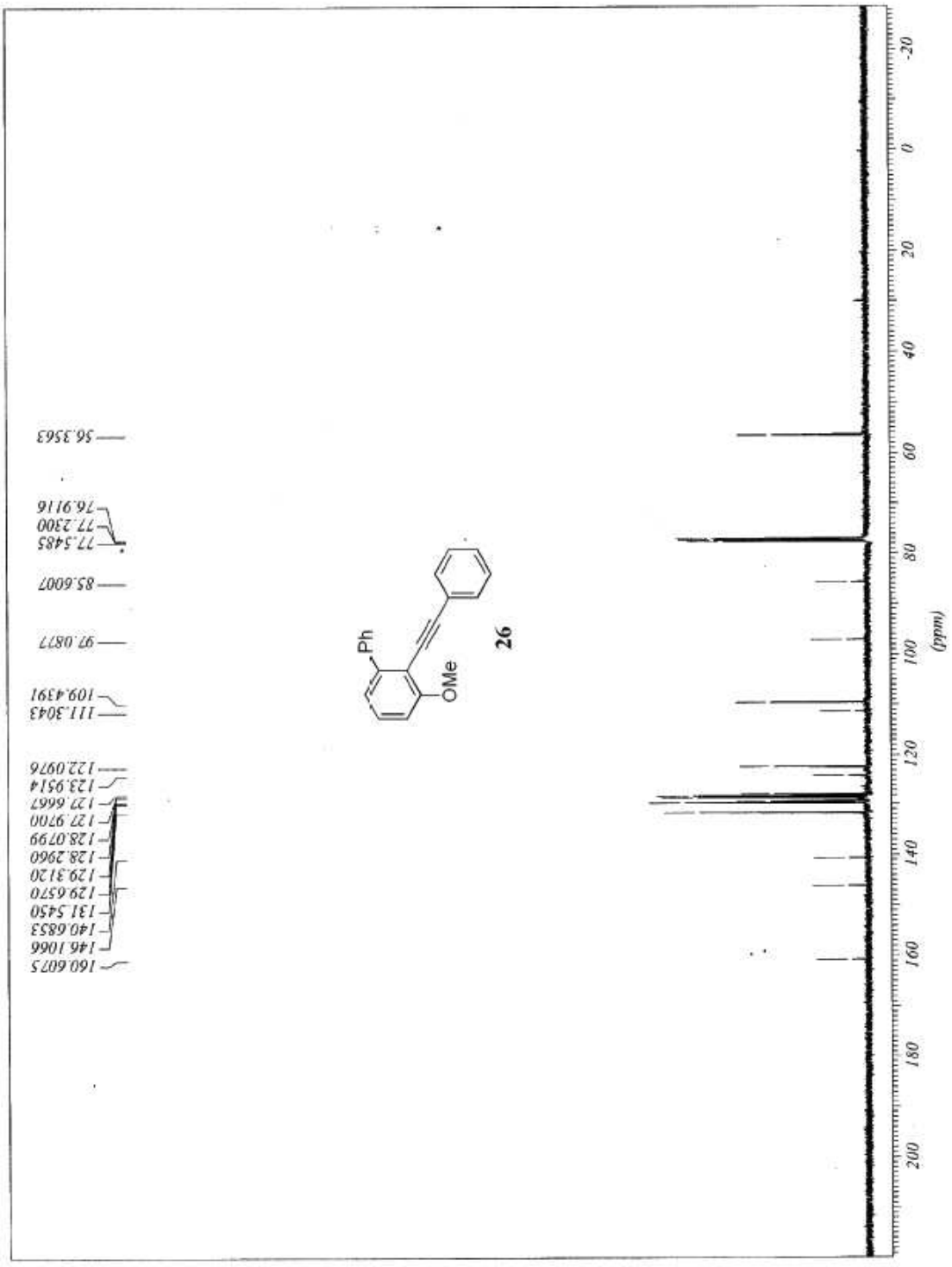
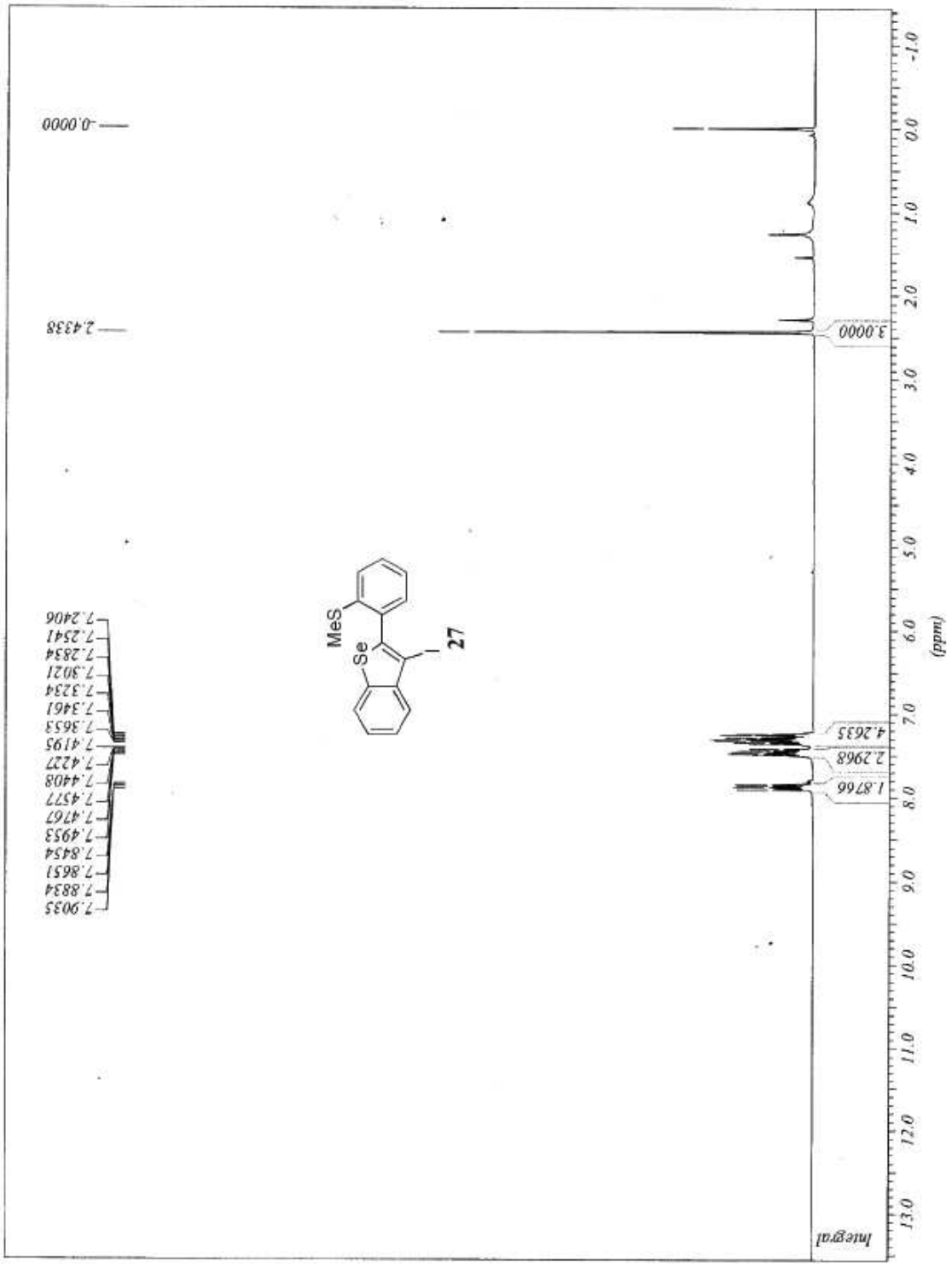
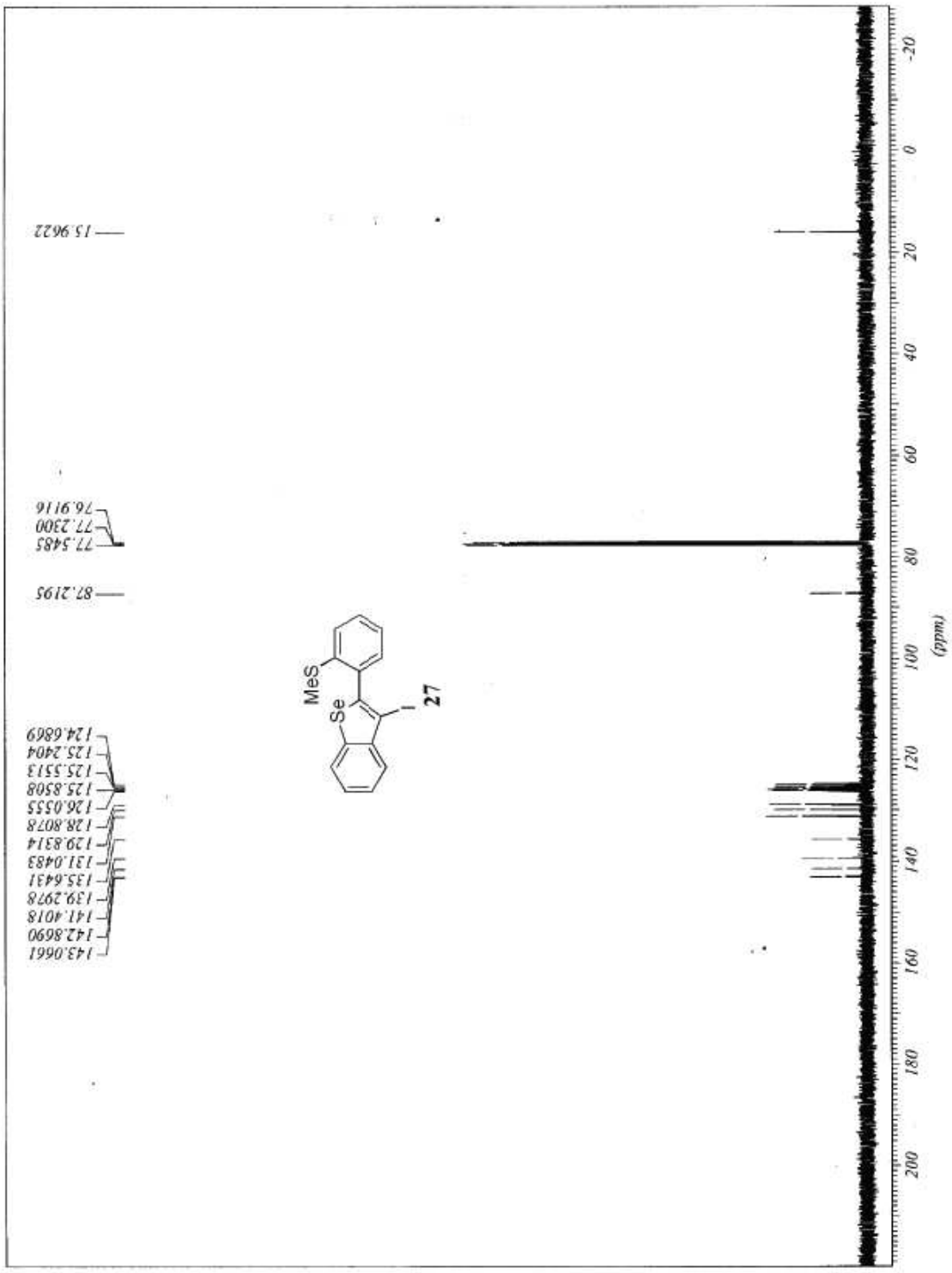
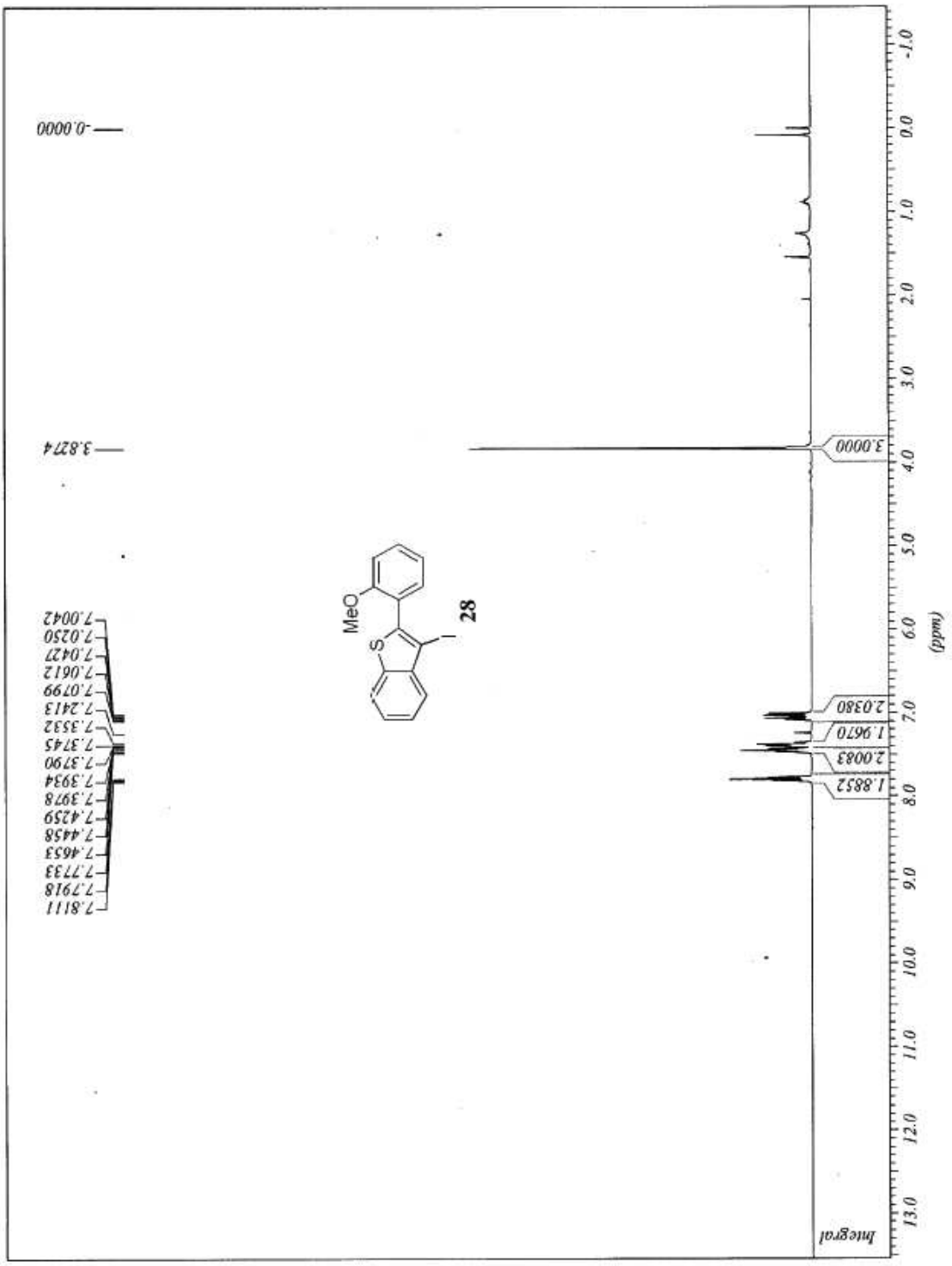
133.3914
133.6122
140.8315
143.8695
160.2349
Ph
MeO
24
(ppm)


Ph
OMe
26
integral
(ppm)
0.0000
3.9697
7.6591
7.6413
7.4447
7.4257
7.3806
7.3644
7.3442
7.3326
7.3237
7.3138
7.3083
7.2632
7.2612
7.2516
7.2470
7.0378
7.0186
6.9216
6.9010
3.0000
1.0110
1.0458
3.4003
6.4158
1.8978


Ph
OMe
26
56.3563
76.9116
77.2300
77.5485
85.6007
97.0877
109.4391
111.3043
122.0976
123.9514
127.6667
127.9700
128.0799
128.2960
129.3120
129.6570
131.5450
140.6853
146.1066
160.6075
(ppm)


MeS
Se
I
27
Integral
(ppm)
7.9035
7.8834
7.8651
7.8454
7.4953
7.4767
7.4577
7.4408
7.4227
7.4195
7.3653
7.3461
7.3234
7.3021
7.2834
7.2541
7.2406
2.4338
0.0000
3.0000
4.2635
2.2968
1.8766


15.9622
77.5485
77.2300
76.9116
87.2195
124.6869
125.2404
125.5513
125.8508
126.0555
128.8078
129.8314
131.0483
135.6431
139.2978
141.4018
142.8690
143.0661
MeS
Se
I
27
(ppm)


MeO
S
I
28
integral
(ppm)
1.8852
2.0083
1.9670
2.0380
3.0000
3.8274
0.0000
7.8111
7.7918
7.7733
7.4658
7.4453
7.4259
7.3978
7.3934
7.3790
7.3745
7.3532
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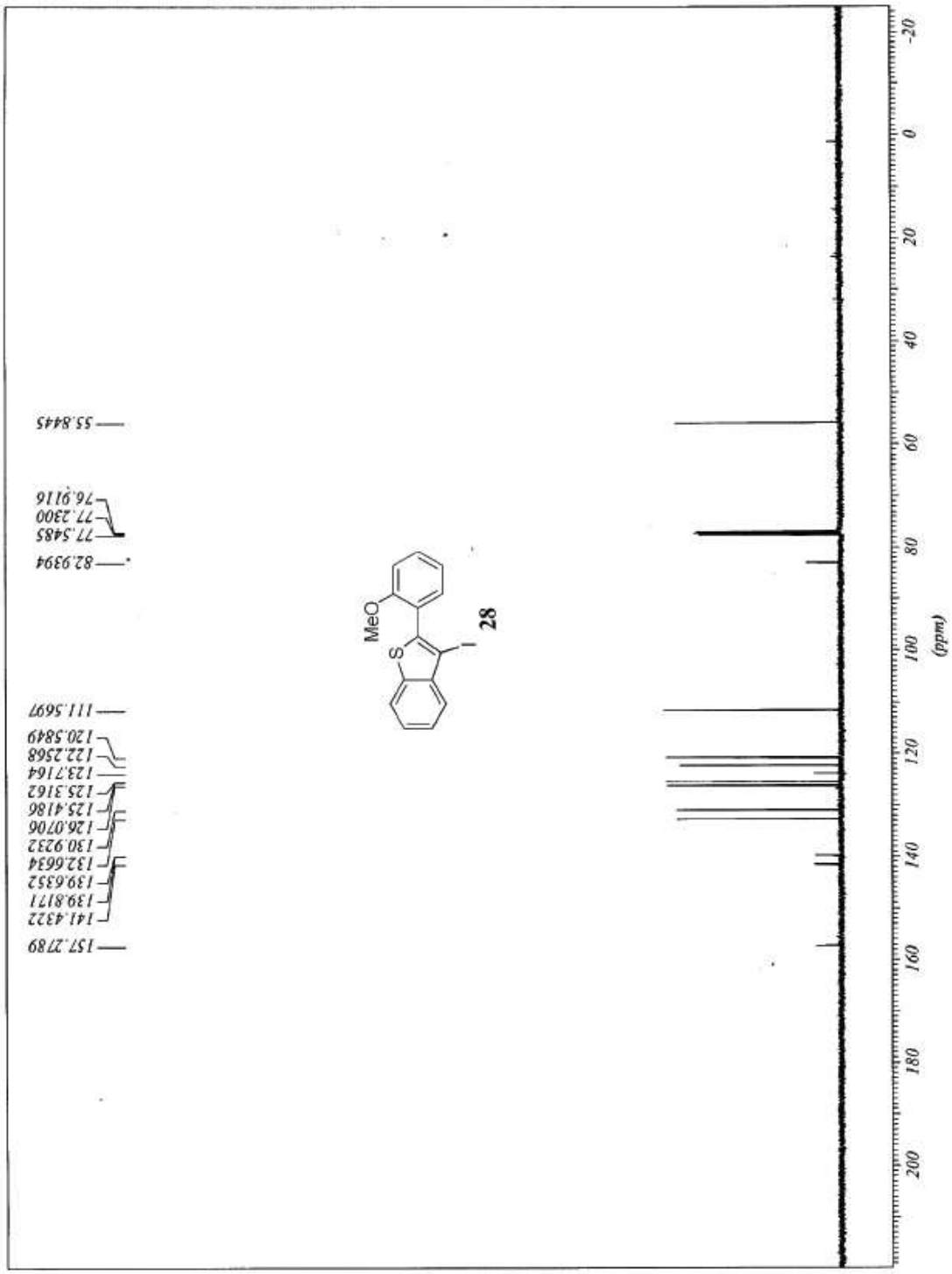
7.0799
7.0612
7.0427
7.0250
7.0042


MeO
S
I
28
55.8445
76.9116
77.2300
77.5485
82.3994
111.5697
120.5849
122.2568
123.7164
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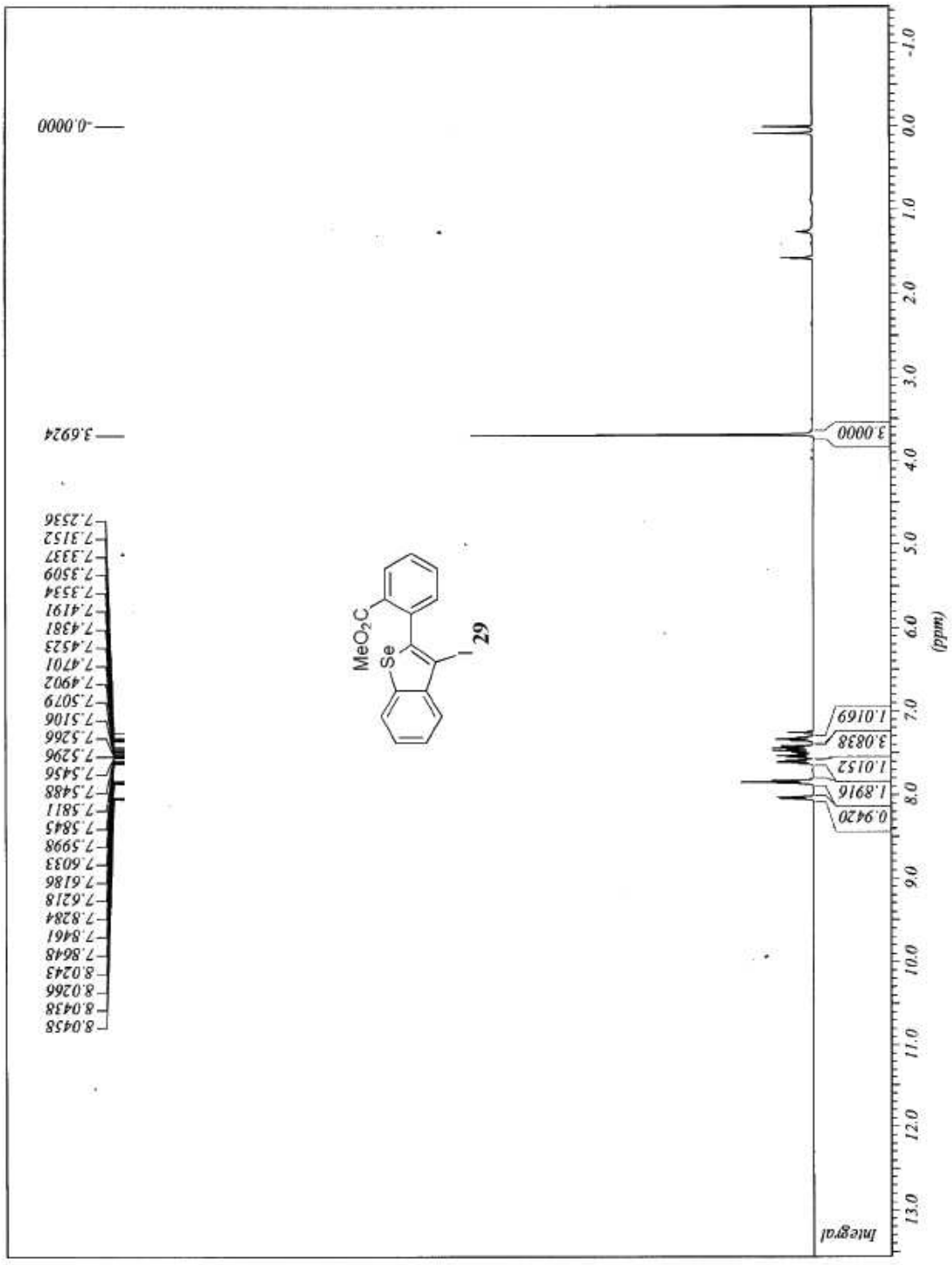
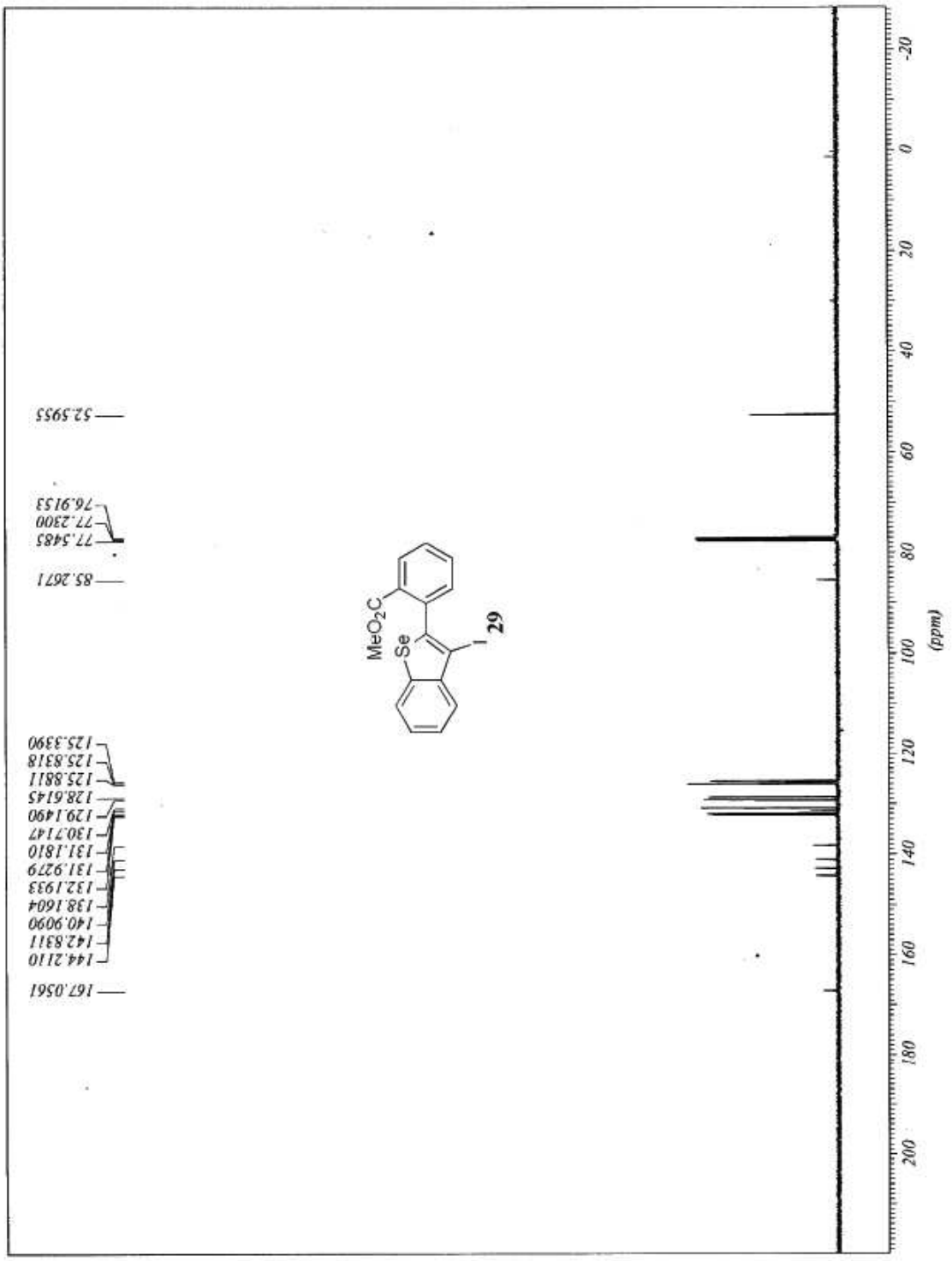
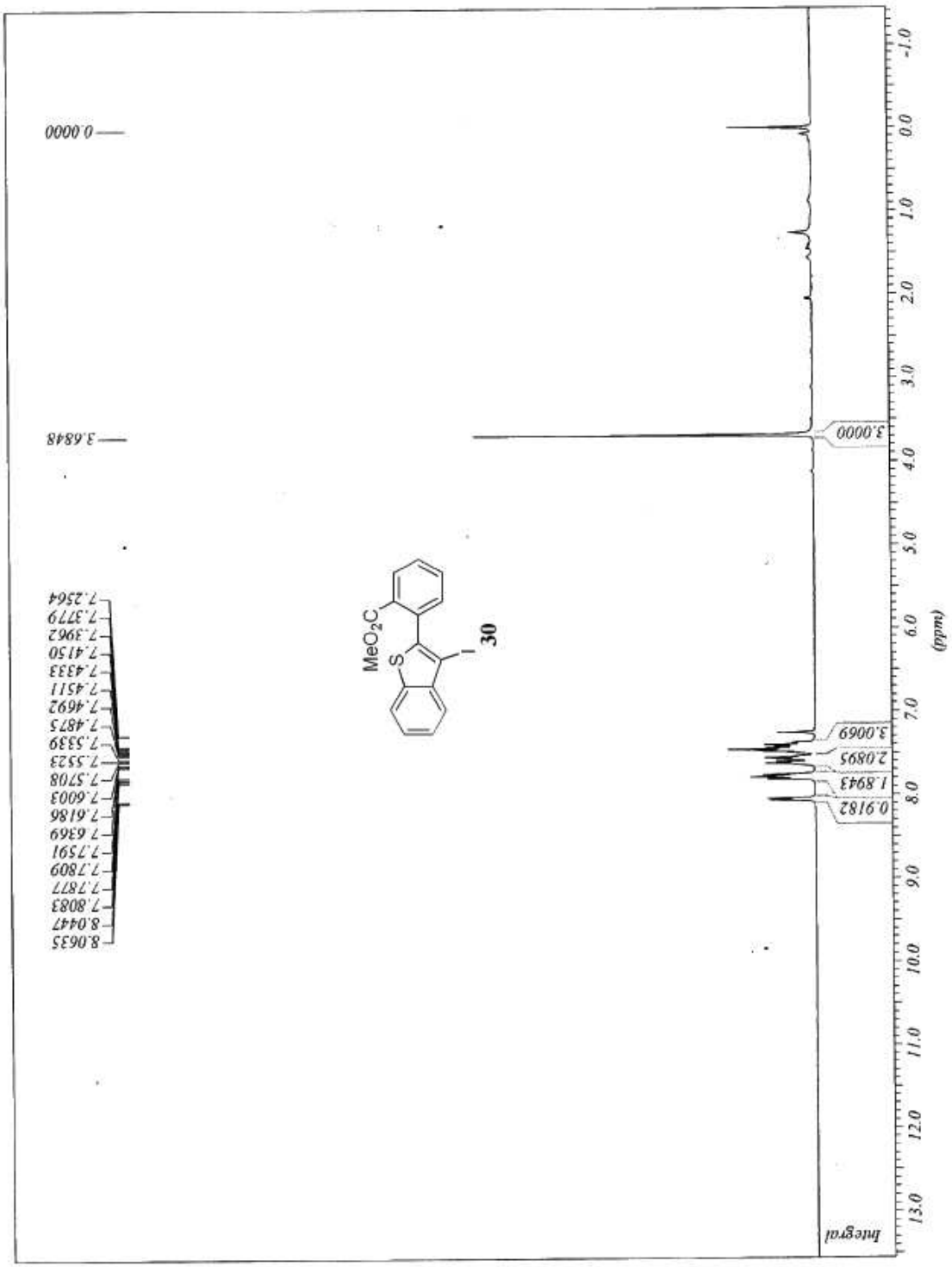
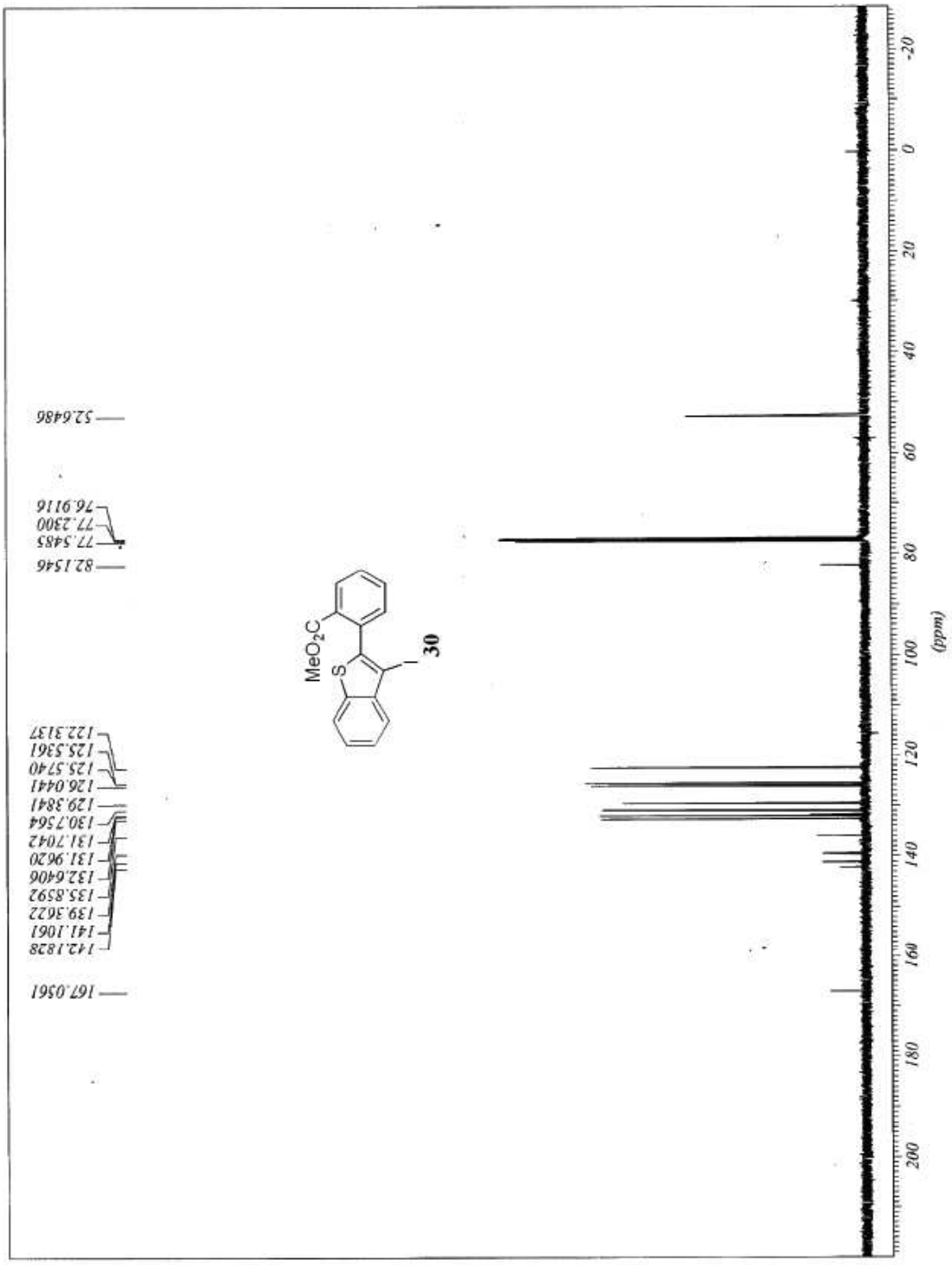
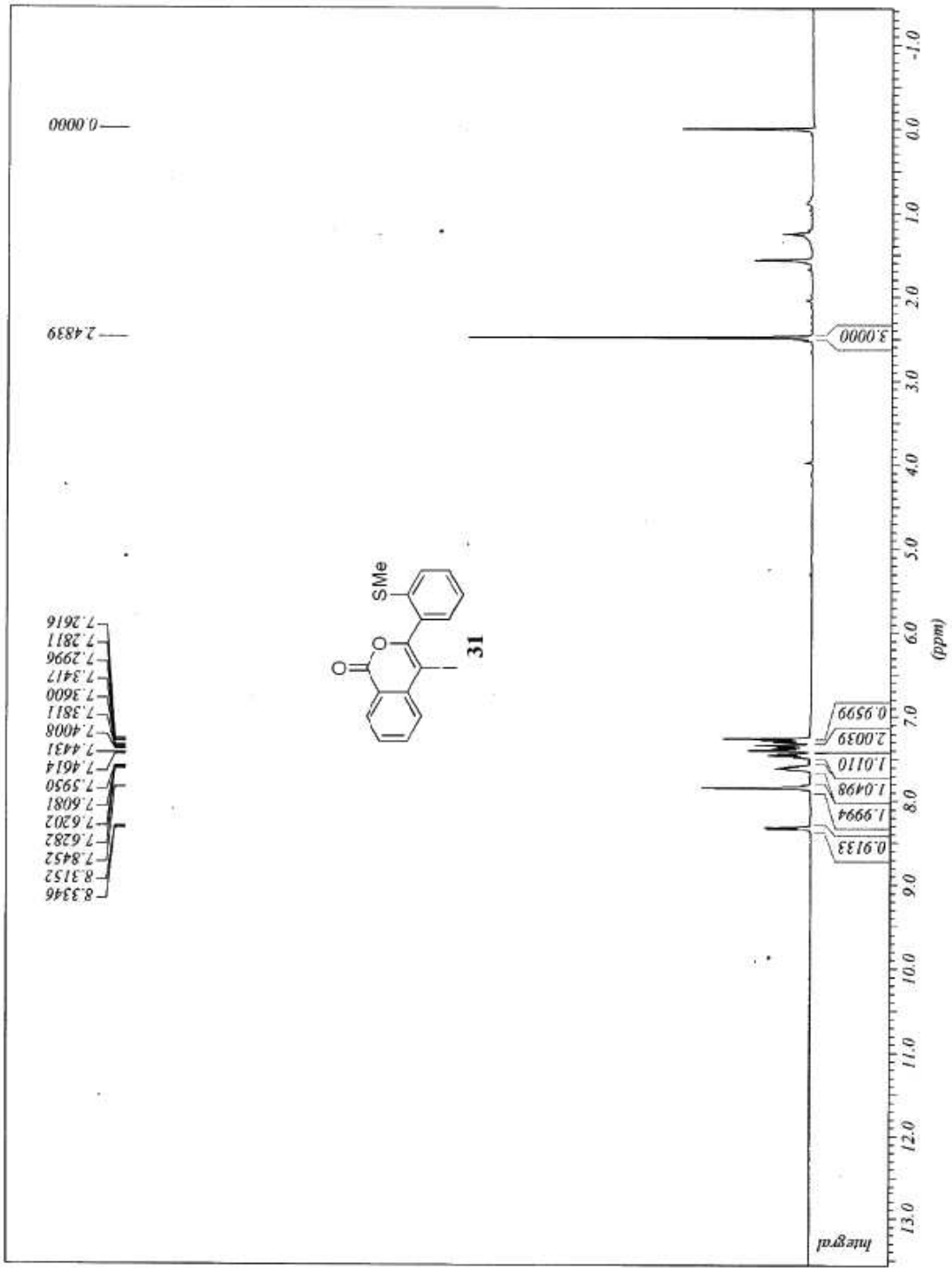
125.4186
126.0706
130.9232
132.6634
139.6352
139.8171
141.4322
157.2789
(ppm)


MeO2C
Se
I
29
integral
(ppm)
3.0000
1.0169
3.0838
1.0152
1.8916
0.9420
3.6924
0.0000
8.0458
8.0438
8.0266
8.0243
7.8648
7.8461
7.8284
7.6218
7.6186
7.6033
7.5998
7.5845
7.5811
7.5488
7.5456
7.5296
7.5266
7.5106
7.5079
7.4902
7.4701
7.4523
7.4381
7.4191
7.3534
7.3509
7.3337
7.3152
7.2536


MeO2C
Se
I
29
(ppm)
-20
0
20
40
60
80
100
120
140
160
180
200
52.5955
77.5485
77.2300
76.9153
85.2671
167.0561
144.2110
142.8311
140.9090
138.1604
132.1933
131.9279
131.1810
130.7147
129.1490
128.6145
125.8811
125.8318
125.3390


MeO2C
S
I
30
integral
(ppm)
3.0000
3.0069
2.0895
1.8943
0.9182
0.0000
3.6848
8.0635
8.0447
7.8083
7.7877
7.7809
7.7591
7.6369
7.6186
7.6003
7.5708
7.5523
7.5339
7.4875
7.4692
7.4511
7.4333
7.4150
7.3962
7.3779
7.2564


MeO2C
S
30
167.0561
142.1828
141.1061
139.3622
135.8592
132.6406
131.9620
131.7042
130.7564
129.3841
126.0441
125.5740
125.5361
122.3137
82.1546
77.5485
77.2300
76.9116
52.6486
(ppm)


SMe
O
O
31
integral
(ppm)
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0.0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
10.0
11.0
12.0
13.0
3.0000
2.0039
0.9599
1.0110
1.0498
1.9994
0.9133
0.0000
2.4839
8.3346
8.3152
7.8452
7.6282
7.6202
7.6081
7.5950
7.4614
7.4431
7.4008
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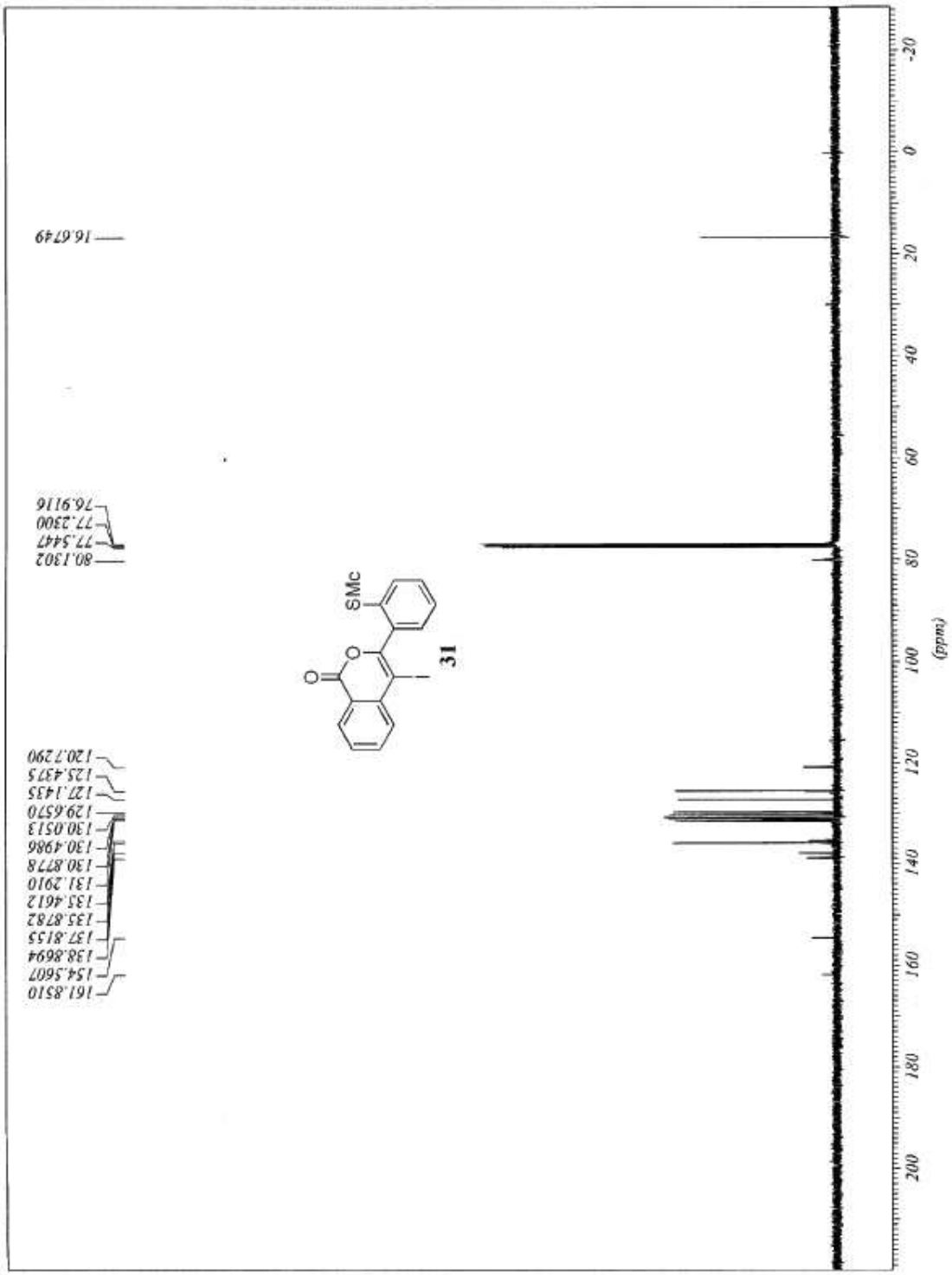
7.3600
7.3417
7.2996
7.2811
7.2616


16.6749
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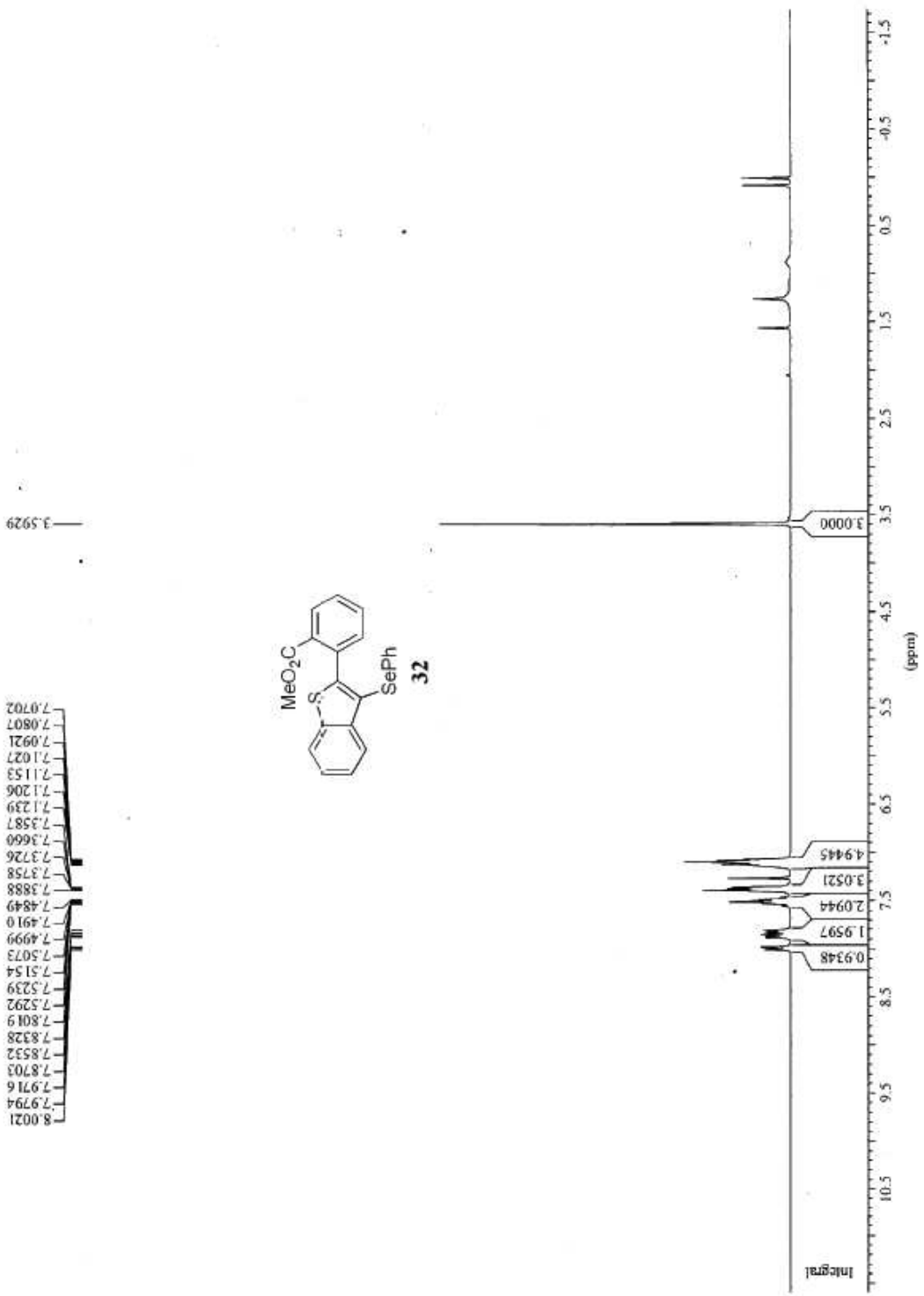
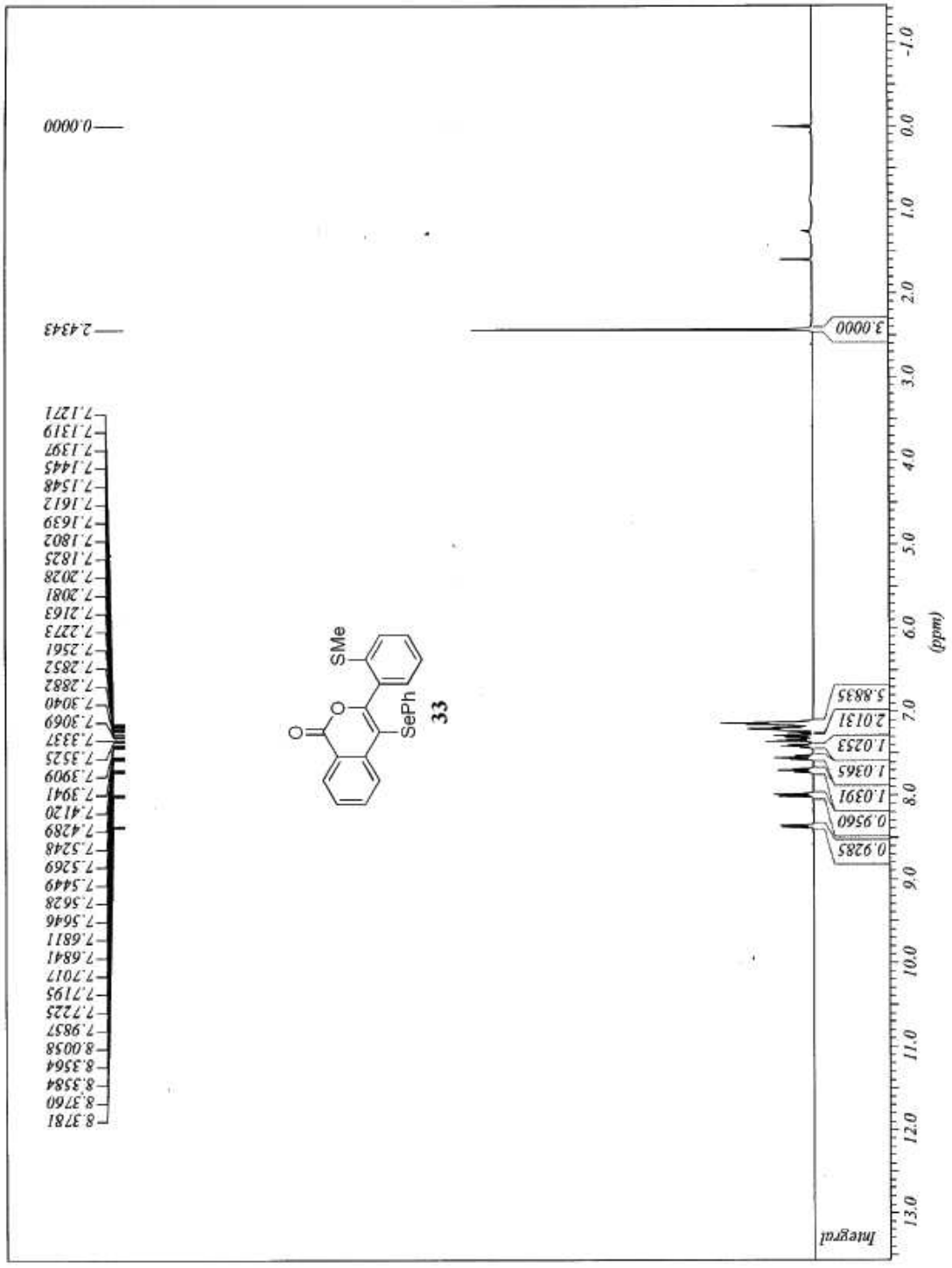
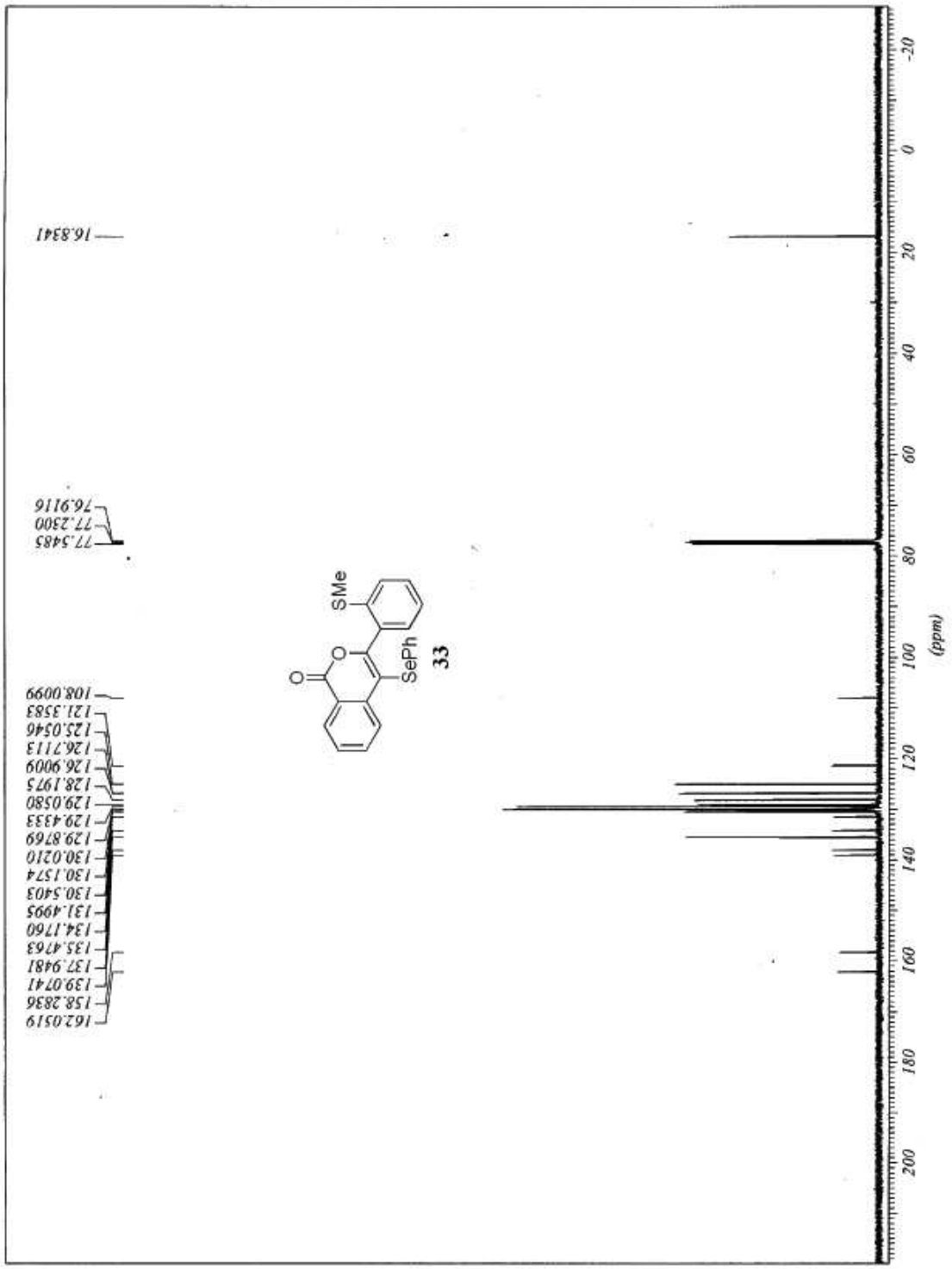
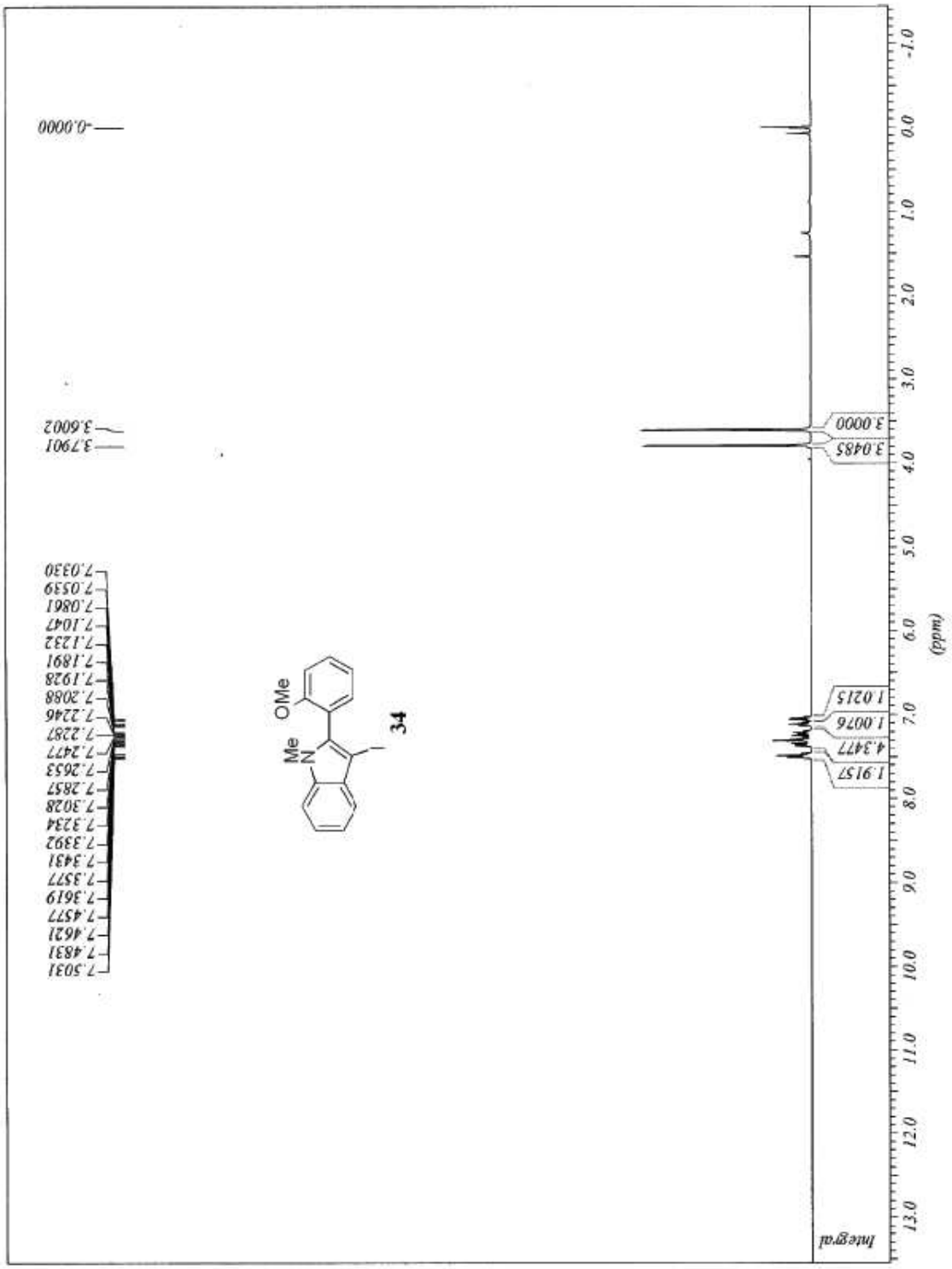
77.2300
77.5447
80.1302
120.7290
125.4375
127.1435
129.6570
130.0513
130.4986
130.8778
131.2910
135.4612
135.8782
137.8155
138.8694
154.5607
161.8510
SMe
O
O
31
-20
0
20
40
60
80
100
120
140
160
180
200
(ppm)

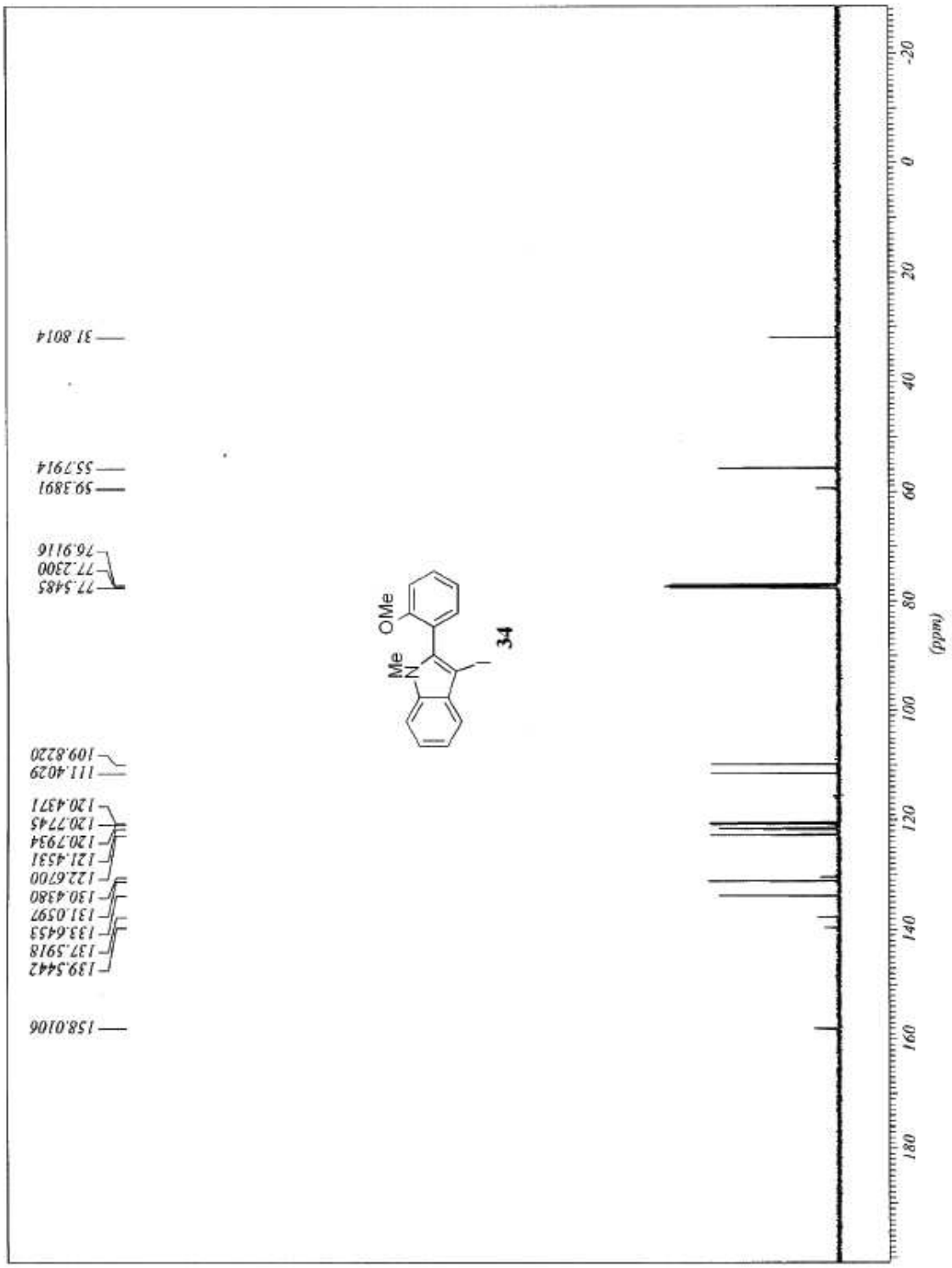

MeO2C
SePh
S
32
3.5929
8.0021
7.9794
7.9716
7.8703
7.8532
7.8328
7.8019
7.5292
7.5239
7.5154
7.5073
7.4999
7.4910
7.4849
7.3888
7.3758
7.3726
7.3660
7.3587
7.1239
7.1206
7.1153
7.1027
7.0921
7.0807
7.0702
3.0000
4.9445
3.0521
2.0944
1.9597
0.9348
Integral
(ppm)

MeO2C
S
SePh
32
(ppm)


SMe
SePh
O
O
33
0.0000
2.4343
7.1271
7.1319
7.1397
7.1445
7.1548
7.1612
7.1639
7.1802
7.1825
7.2028
7.2081
7.2163
7.2273
7.2561
7.2852
7.2882
7.3040
7.3069
7.3337
7.3525
7.3909
7.3941
7.4120
7.4289
7.5248
7.5269
7.5449
7.5628
7.5646
7.6811
7.6841
7.7017
7.7195
7.7225
7.9857
8.0058
8.3564
8.3584
8.3760
8.3781
3.0000
5.8835
2.0131
1.0253
1.0365
1.0391
0.9560
0.9285
Integral
(ppm)


16.8341
77.5485
77.2300
76.9116
108.0099
121.3583
125.0546
126.7113
126.9009
128.1975
129.0580
129.4333
129.8769
130.0210
130.1574
130.5403
131.4995
134.1760
135.4763
137.9481
139.0741
158.2836
162.0519
SMe
SePh
O
O
33
(ppm)


OMe
Me
N
34
integral
(ppm)


OMe
Me
N
I
34
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55.7914
59.3891
76.9116
77.2300
77.5485
109.8220
111.4029
120.4371
120.7745
120.7934
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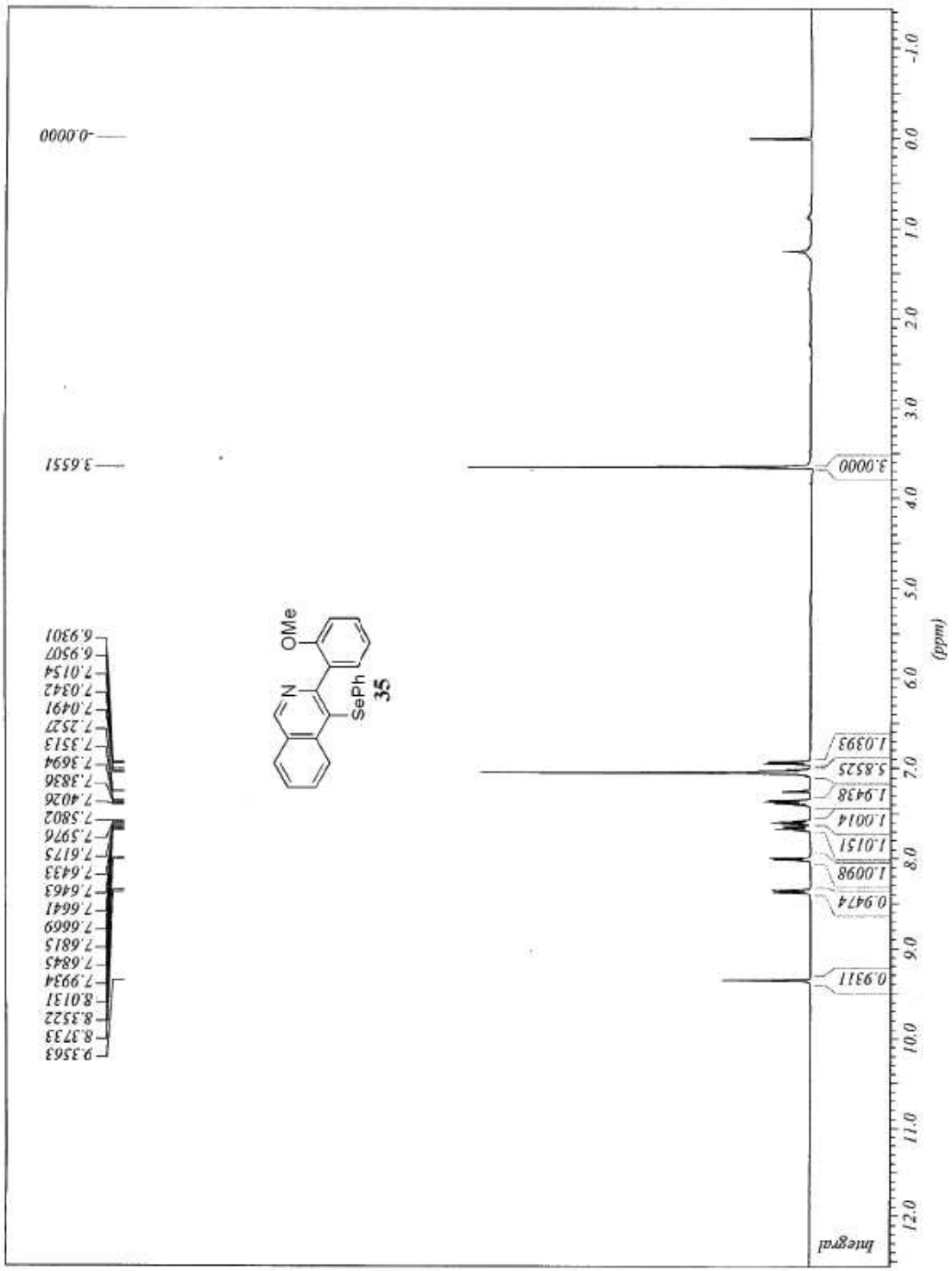
122.6700
130.4380
131.0597
133.6453
137.5918
139.5442
158.0106
(ppm)


OMe
N
SePh
35
integral
(ppm)
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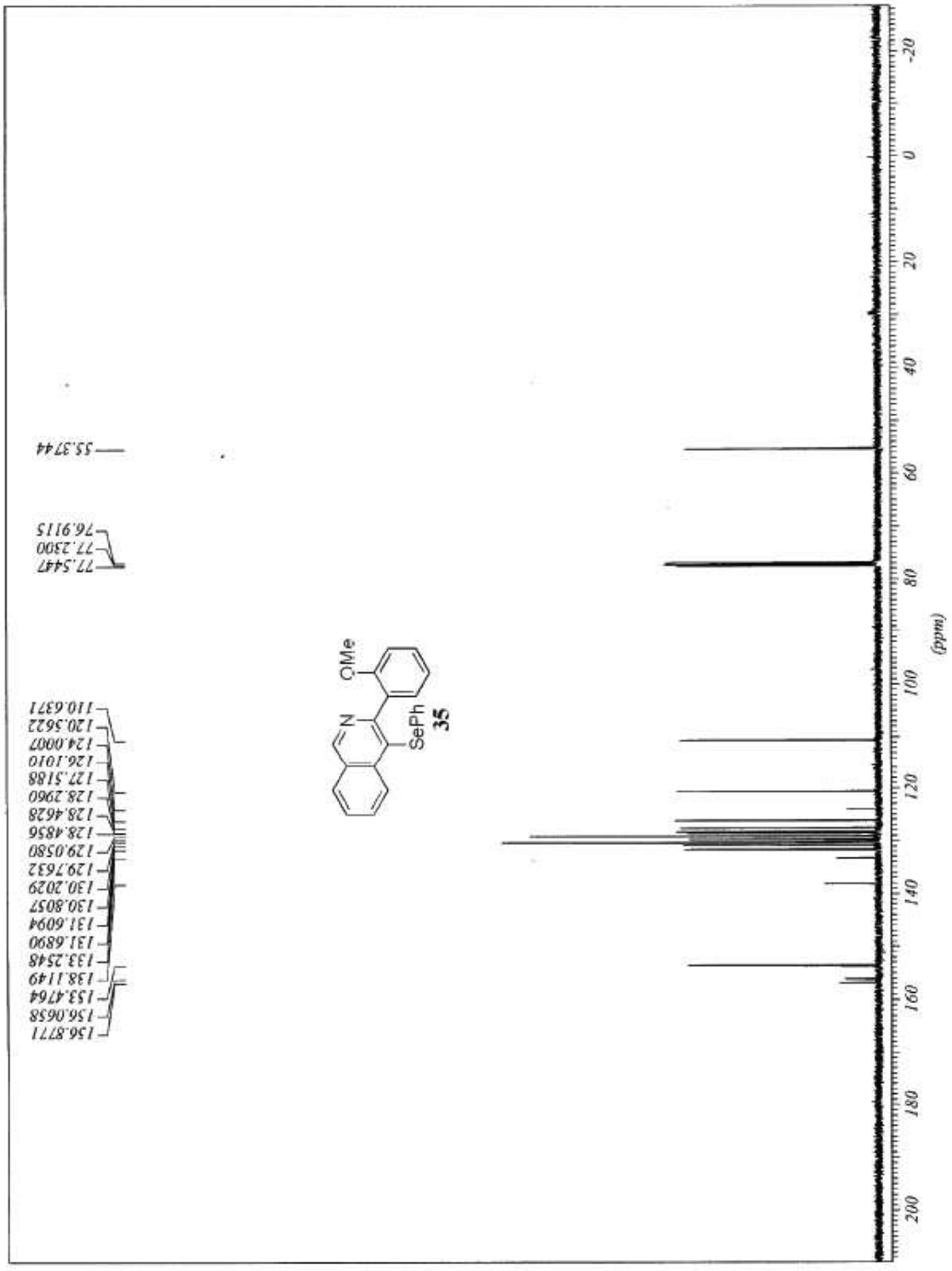
1.0393
5.8525
1.9438
1.0014
1.0151
1.0098
0.9474
0.9311
3.6551
0.0000
9.3563
8.3733
8.3522
8.0131
7.9934
7.6845
7.6815
7.6669
7.6641
7.6463
7.6433
7.6175
7.5976
7.5802
7.4026
7.3836
7.3694
7.3513
7.2527
7.0491
7.0342
7.0154
6.9507
6.9301


OMe
N
SePh
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55.3744
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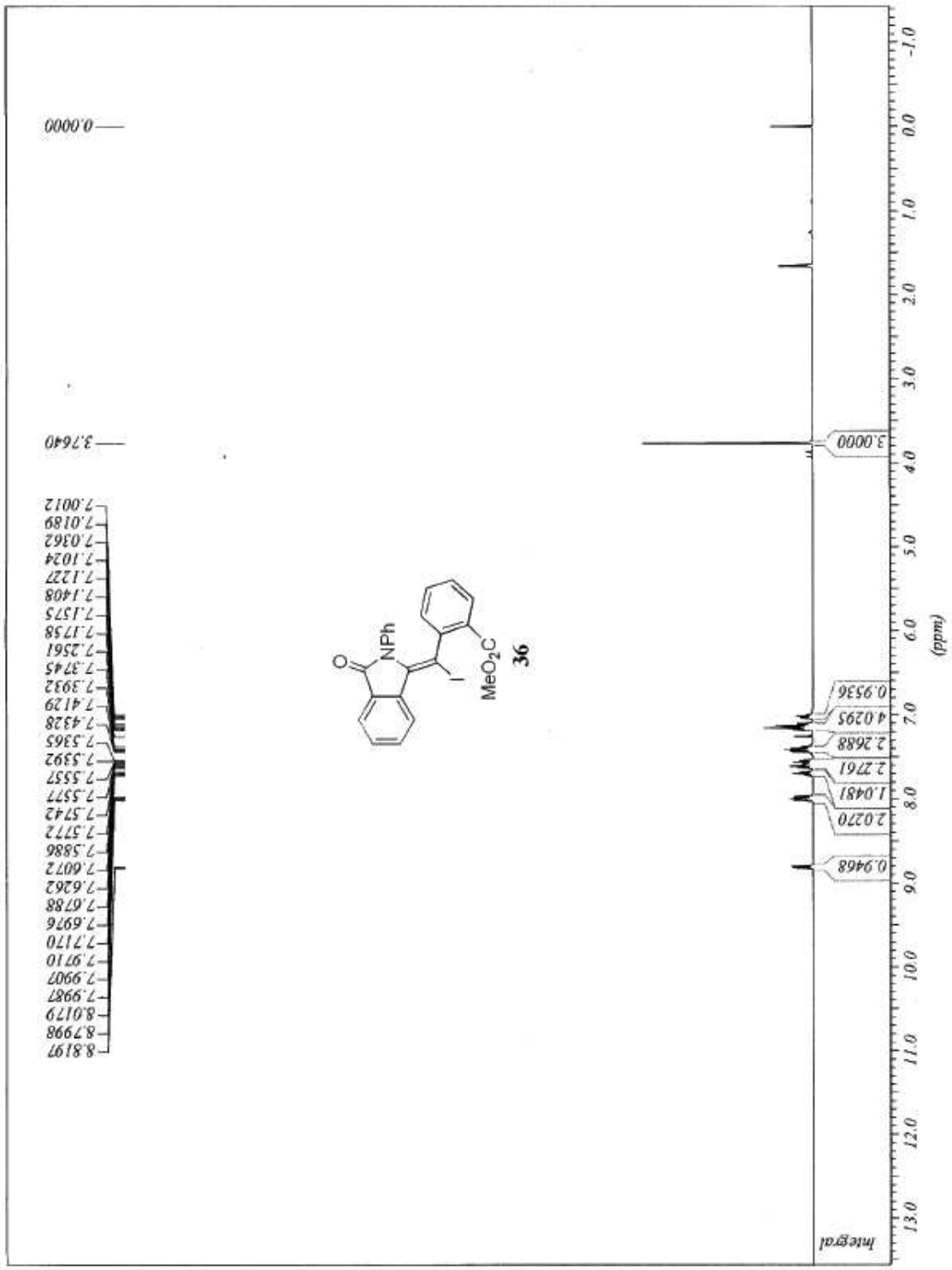
77.2500
76.9115
156.8771
156.0658
153.4764
138.1149
133.2548
131.6890
131.6094
130.8057
130.2029
129.7632
129.0580
128.4856
128.4628
128.2960
127.5188
126.1010
124.0007
120.5622
110.6371
(ppm)


integral
(ppm)
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2.0270
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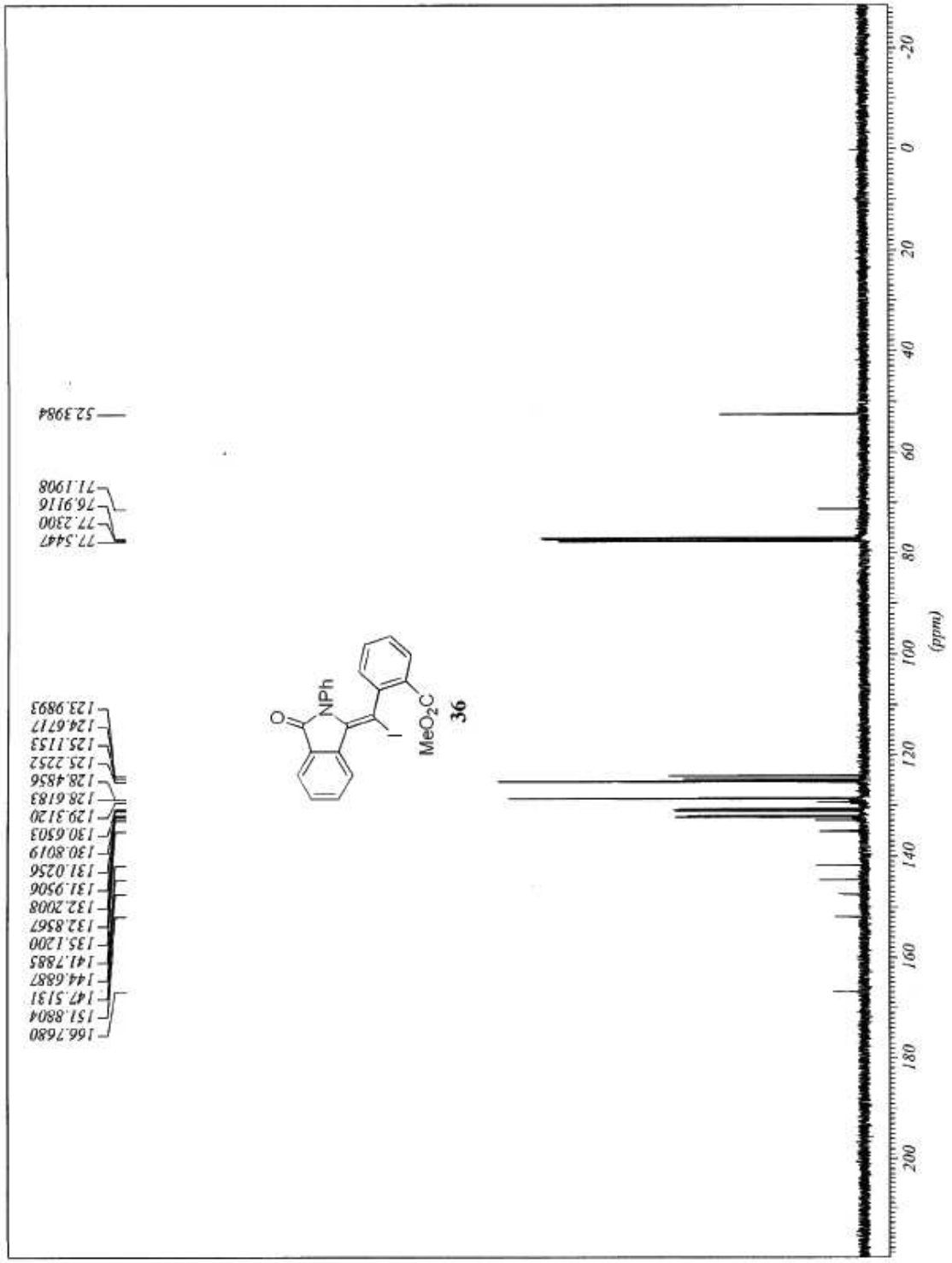
2.2688
4.0295
0.9536
0.9468
3.7640
0.0000
NPh
MeO2C
36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77.2300
77.5447
123.9893
124.6717
125.1153
125.2252
128.4856
128.6183
129.3120
130.6503
130.8019
131.0256
131.9506
132.2008
132.8567
135.1200
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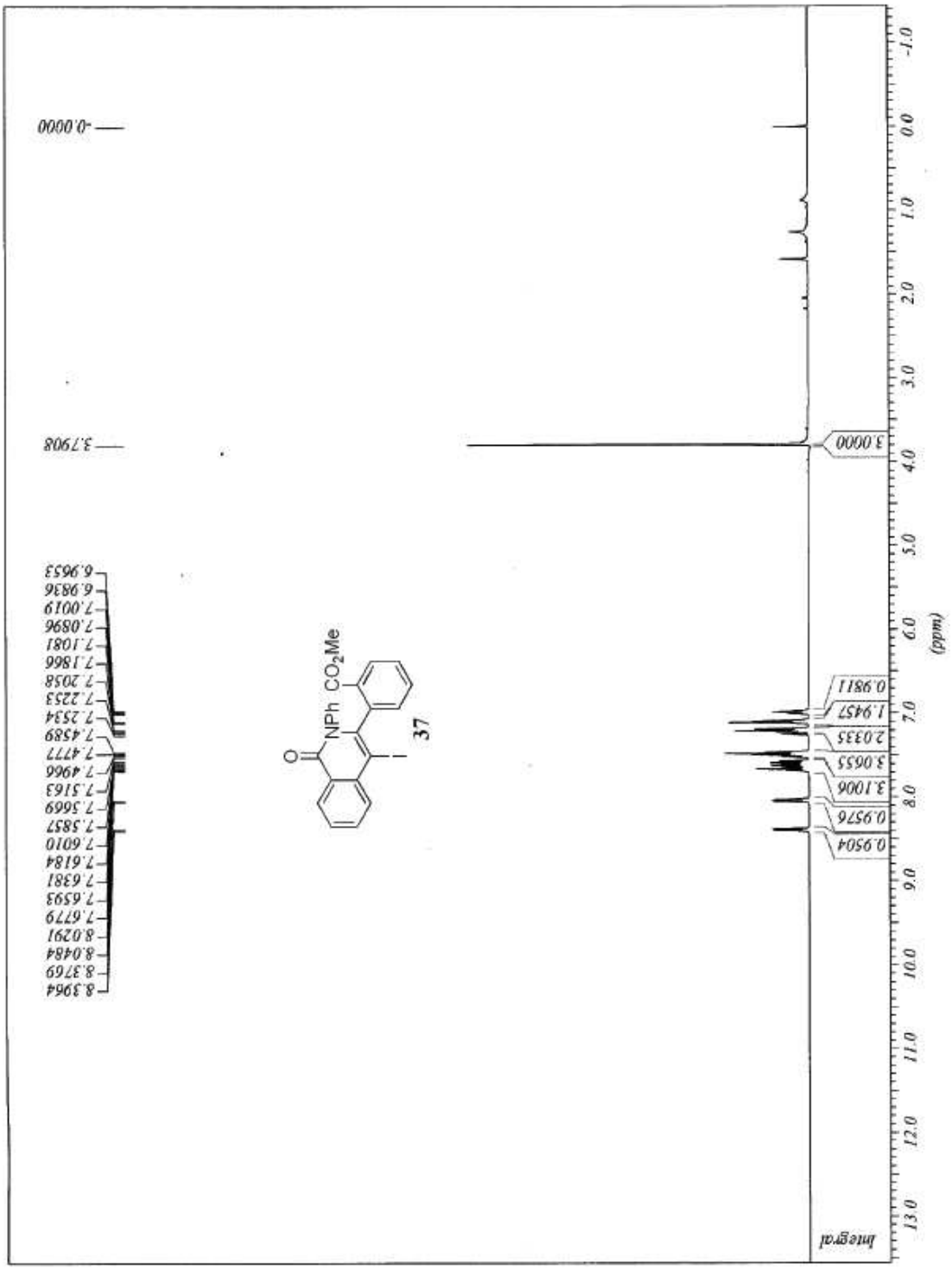
144.6887
147.5131
151.8804
166.7680
O
NPh
I
MeO2C
36
(ppm)


integral
(ppm)
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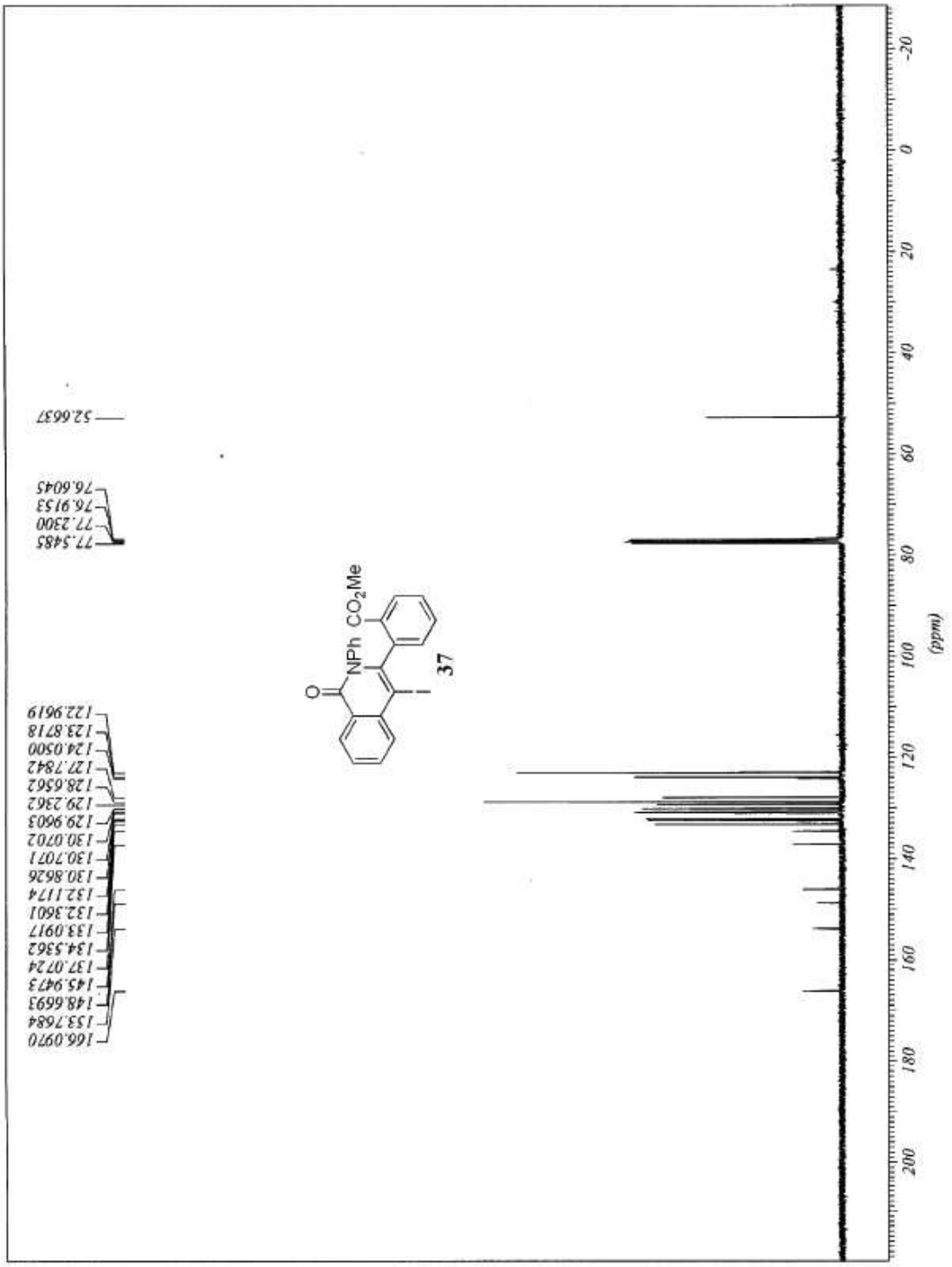
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
0.9504
0.9576
3.1006
3.0655
2.0335
1.9457
0.9811
3.0000
3.7908
-0.0000
8.3964
8.3769
8.0484
8.0291
7.6779
7.6593
7.6381
7.6184
7.6010
7.5857
7.5669
7.5163
7.4966
7.4777
7.4589
7.2534
7.2253
7.2058
7.1866
7.1081
7.0896
7.0019
6.9836
6.9653
NPh CO2Me
O
I
37


52.6637
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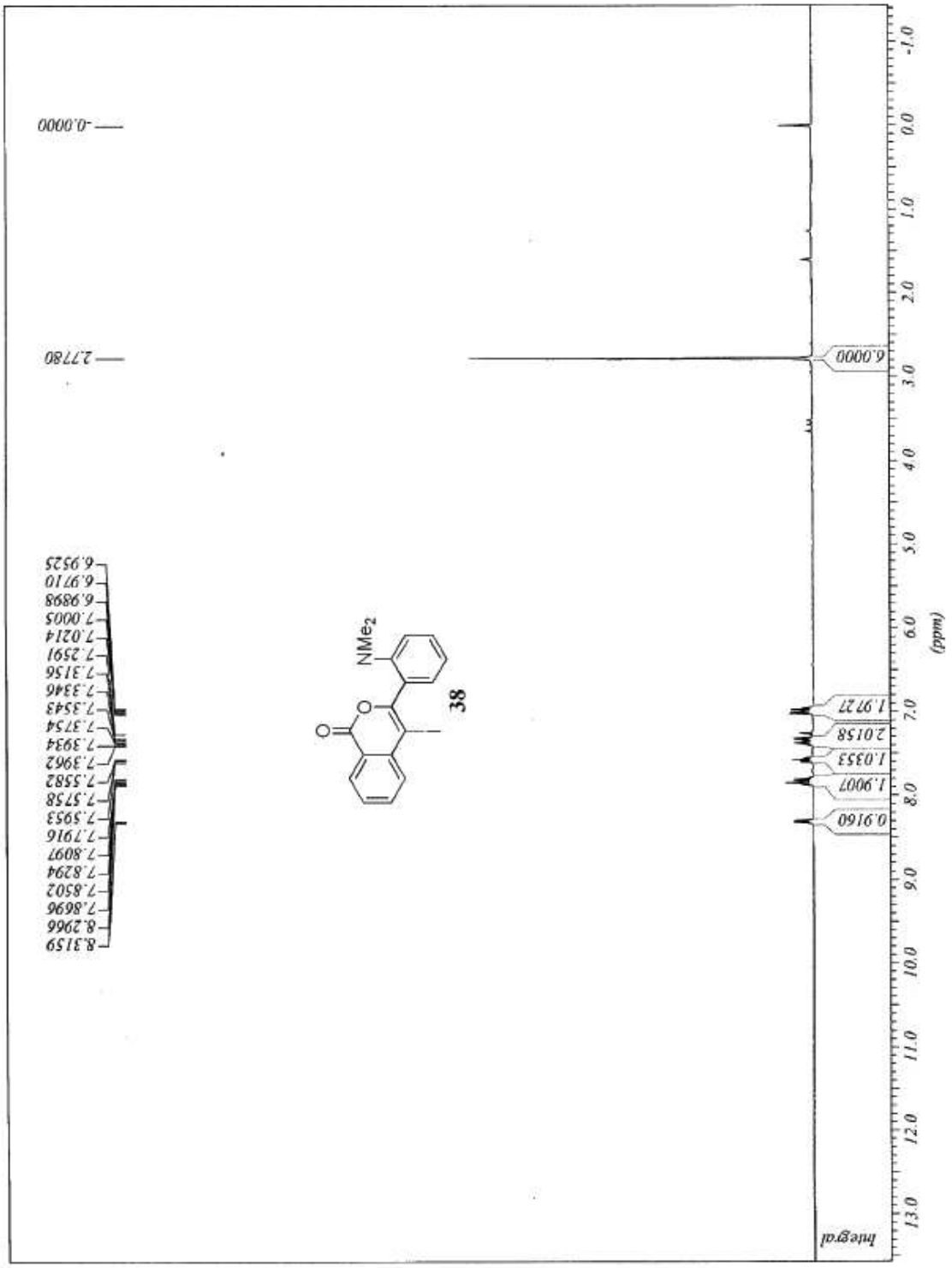
77.2300
76.9153
76.6045
166.0970
153.7684
148.6693
145.9473
137.0724
134.5362
133.0917
132.3601
132.1174
130.8626
130.7071
130.0702
129.9603
129.2362
128.6562
127.7842
124.0500
123.8718
122.9619
NPh
CO2Me
O
I
37
(ppm)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(ppm)
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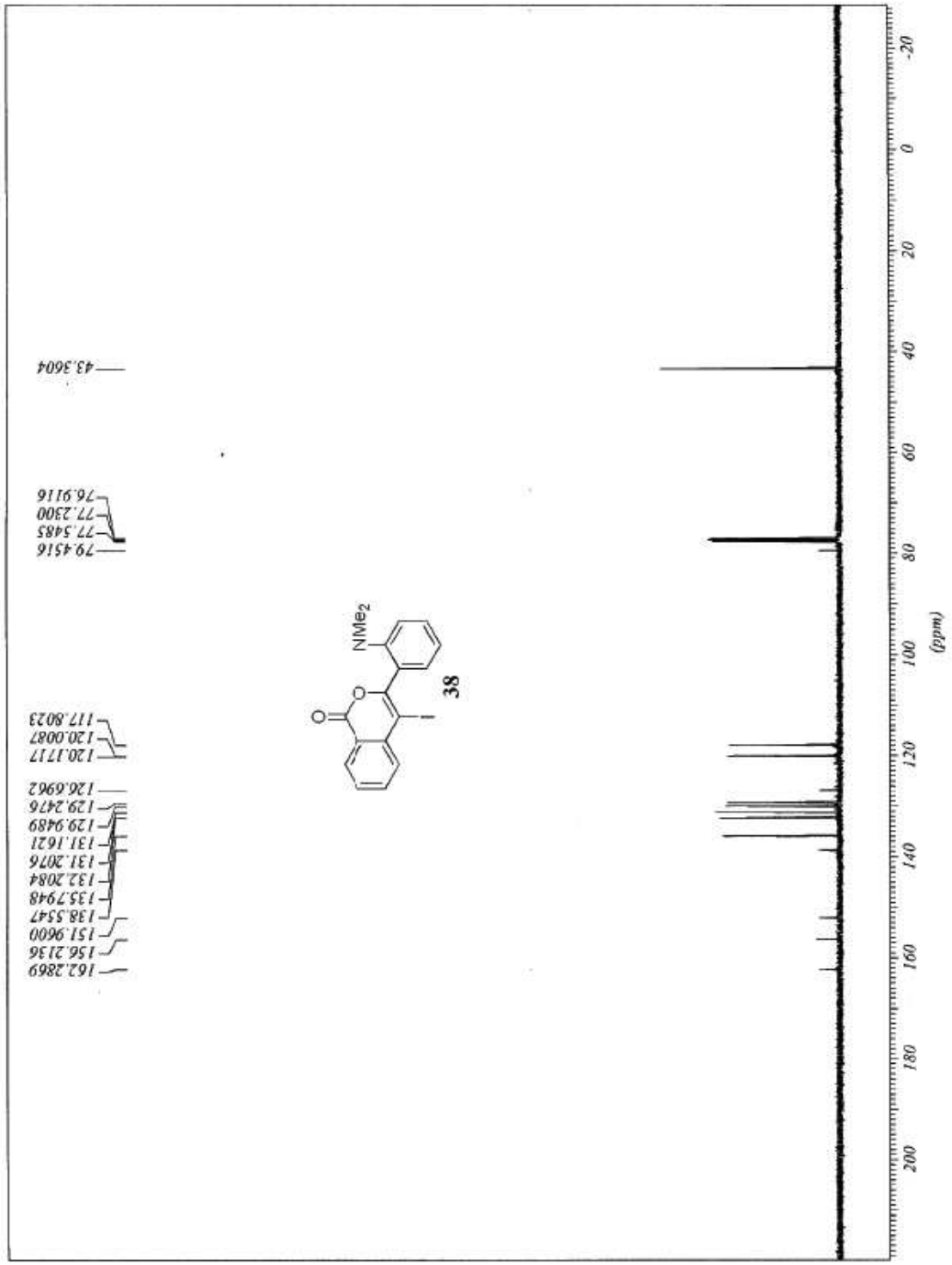
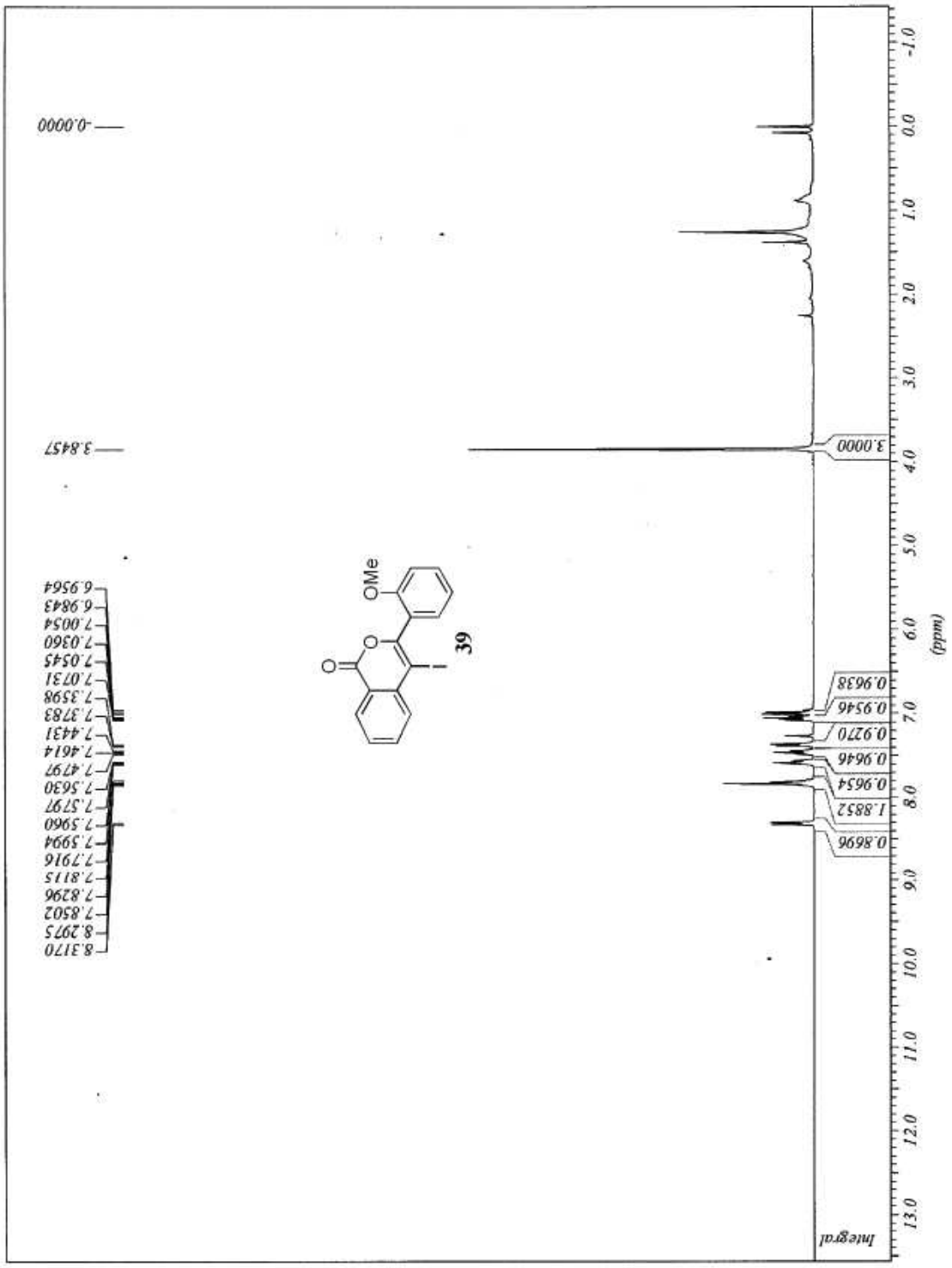
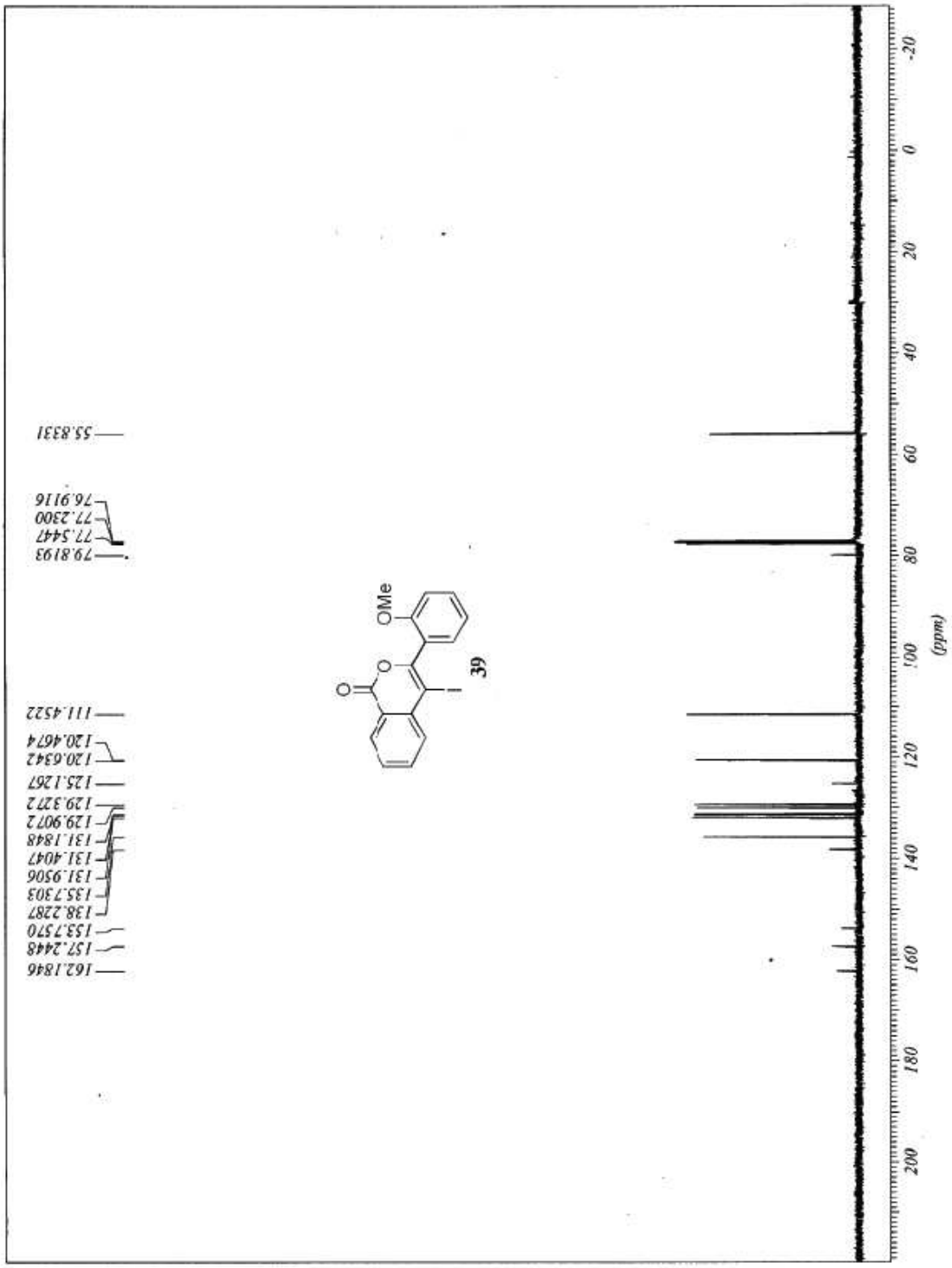
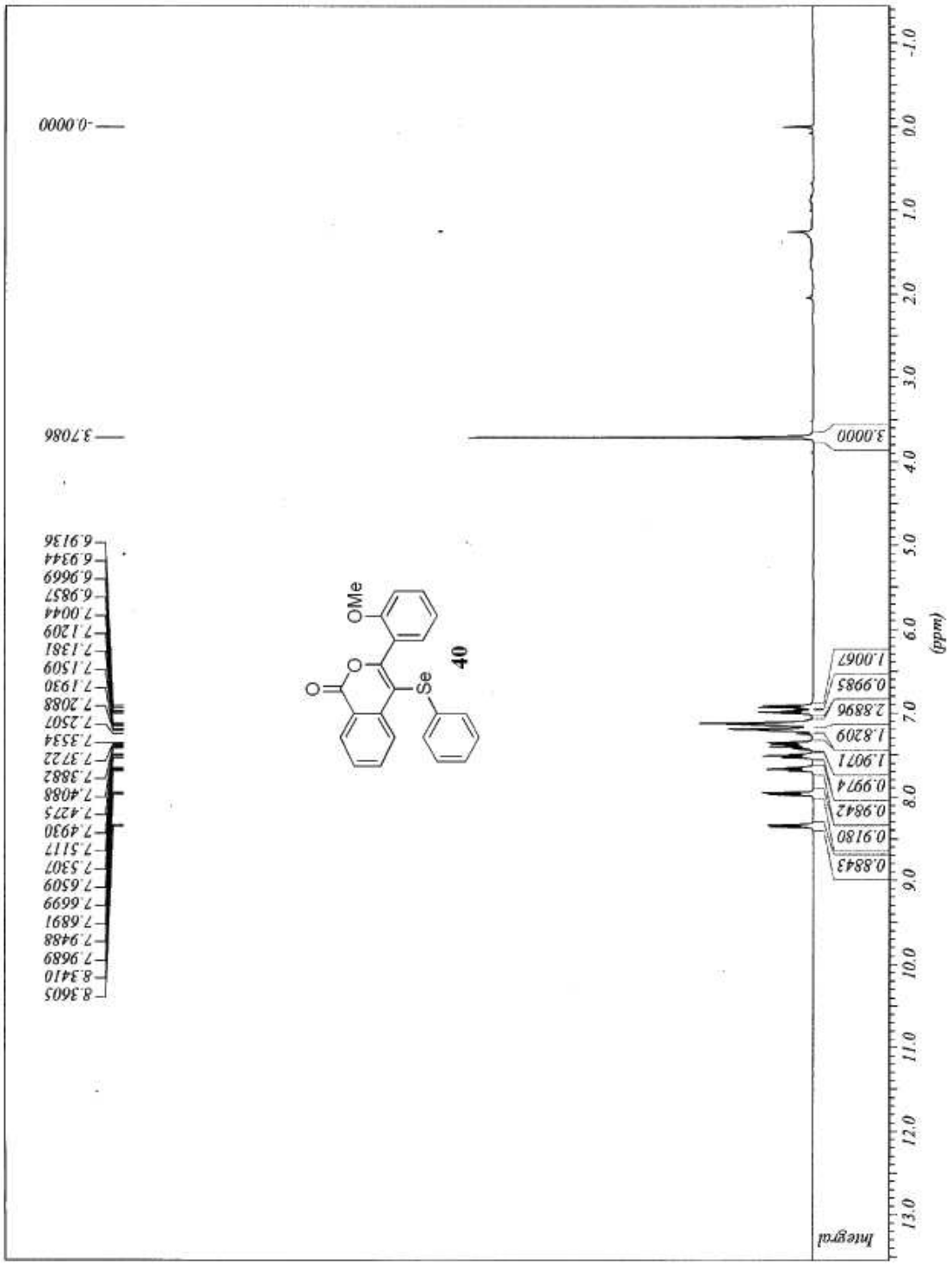
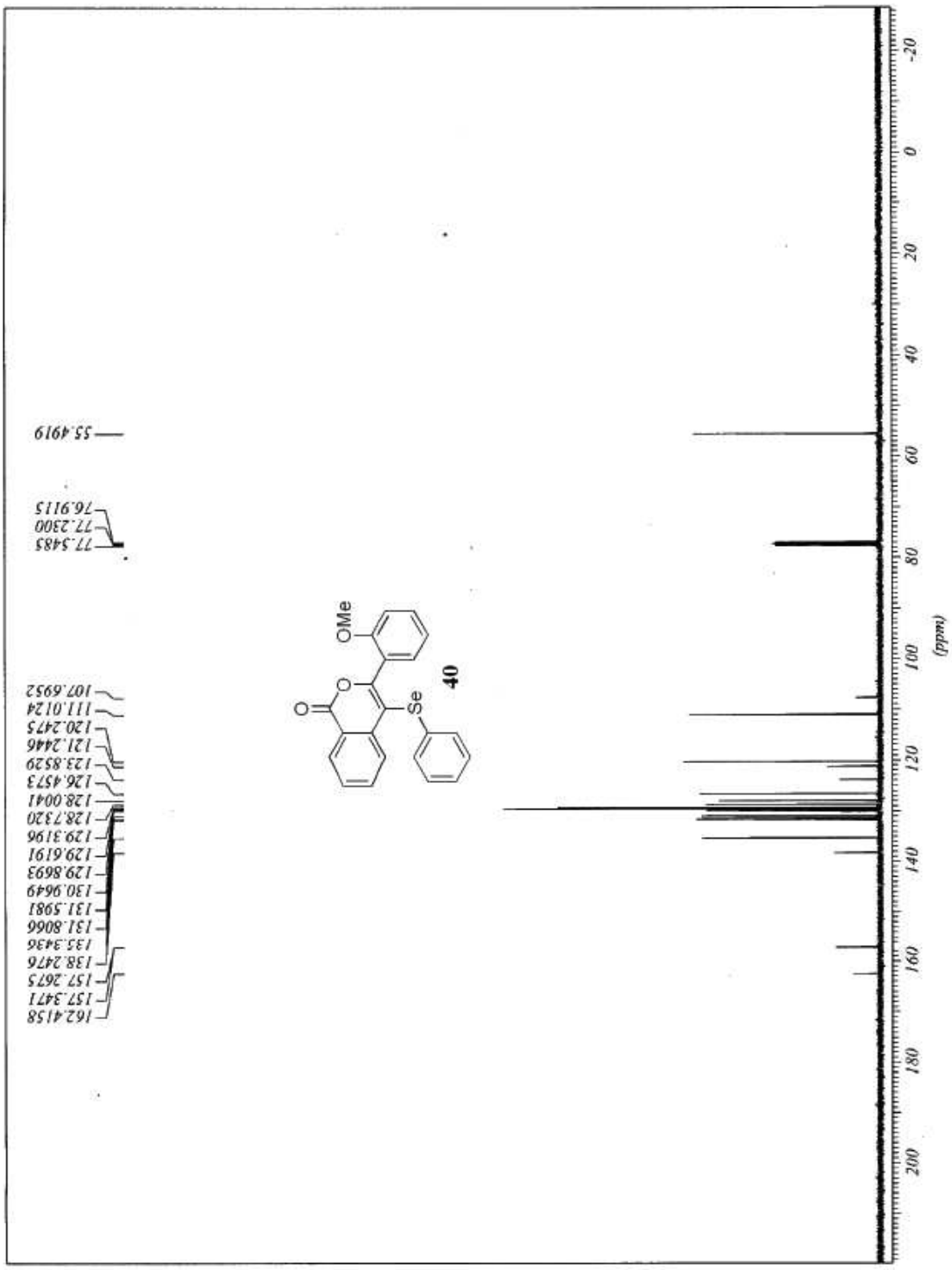
1.9727
2.0158
1.0353
1.9007
0.9160
38
NMe2
8.3159
8.2966
7.8696
7.8502
7.8294
7.8097
7.7916
7.5953
7.5758
7.5582
7.3962
7.3934
7.3754
7.3543
7.3346
7.3156
7.2591
7.0214
7.0005
6.9898
6.9710
6.9525
2.7780
0.0000

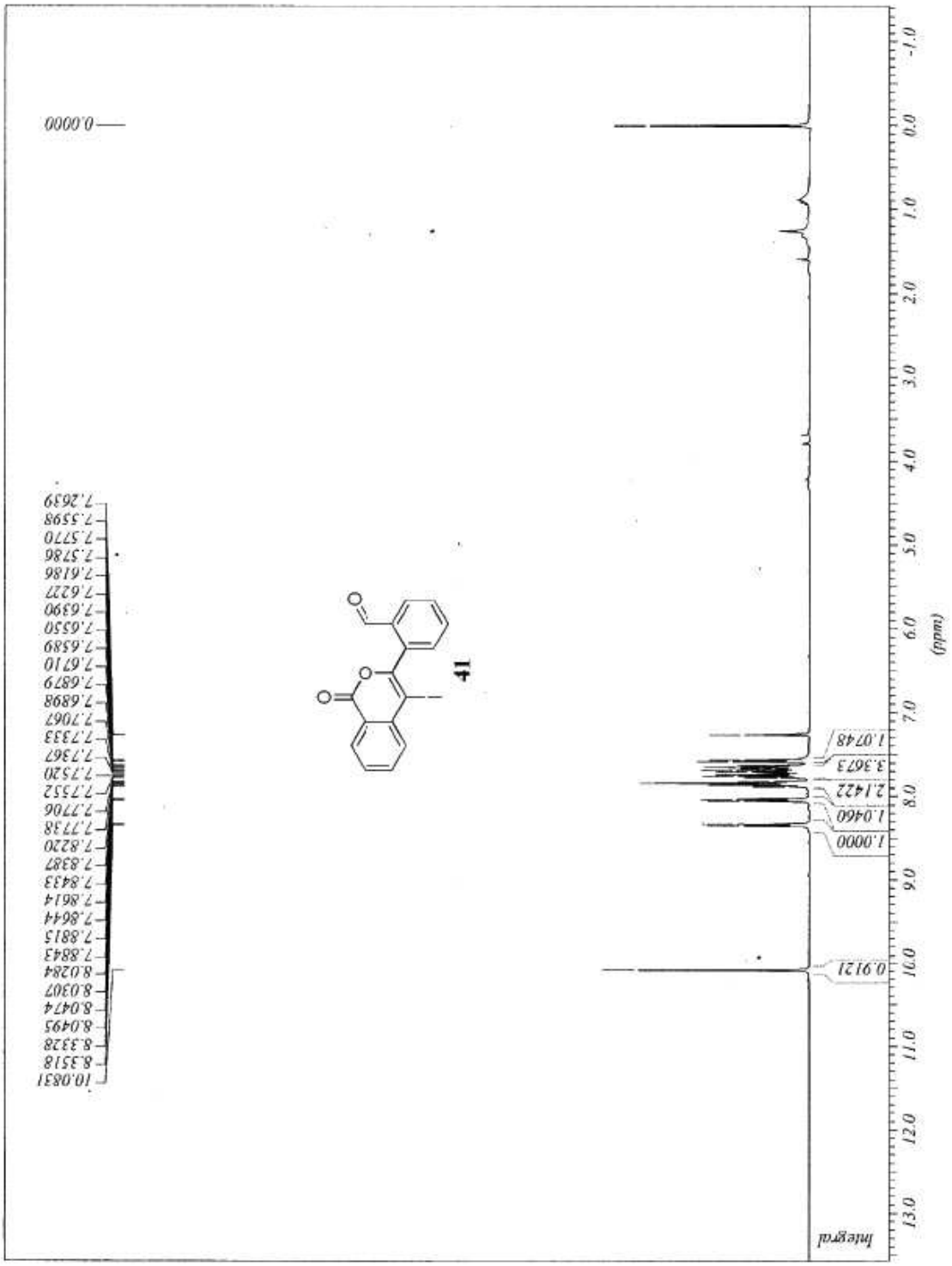

NMe2
O
O
I
38
162.2869
156.2136
151.9600
138.5547
135.7948
132.2084
131.3076
131.1621
129.9489
129.2476
126.6962
120.1717
120.0087
117.8023
79.4516
77.5485
77.2300
76.9116
43.3604
(ppm)

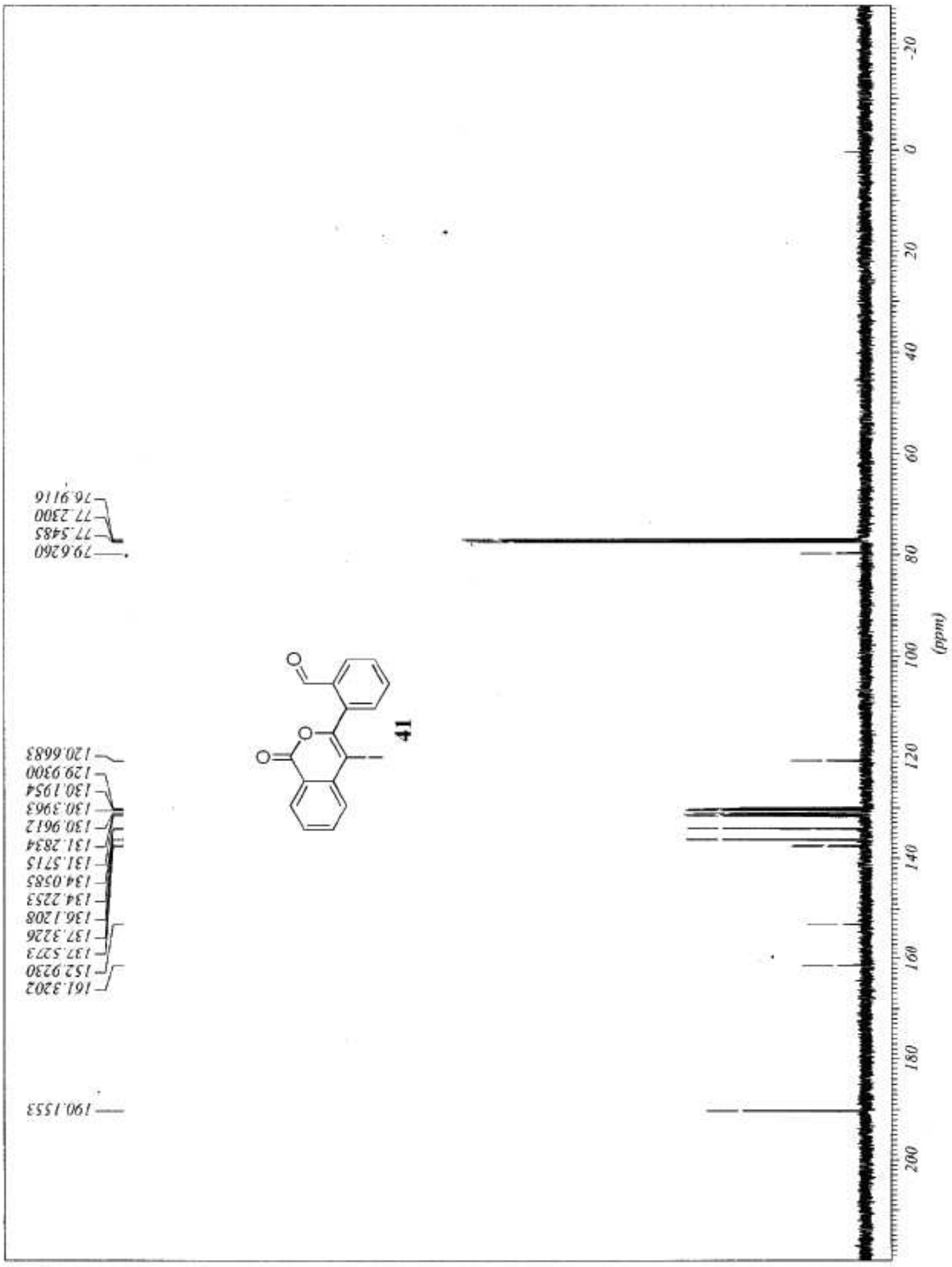

OMe
O
O
I
39
Integral
(ppm)
-1.0
0.0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
10.0
11.0
12.0
13.0
0.8696
1.8852
0.9654
0.9646
0.9270
0.9546
0.9638
3.0000
3.8457
-0.0000
8.3170
8.2975
7.8502
7.8296
7.8115
7.7916
7.5994
7.5960
7.5797
7.5630
7.4797
7.4614
7.4431
7.3783
7.3598
7.0731
7.0545
7.0360
7.0054
6.9843
6.9564


OMe
O
O
I
39
162.1846
157.2448
153.7570
138.2287
135.7303
131.9506
131.4047
131.1848
129.9072
129.3772
125.1267
120.6342
120.4674
111.4522
79.8193
77.5447
77.2300
76.9116
55.8331
200
180
160
140
120
100
80
60
40
20
0
-20
(ppm)

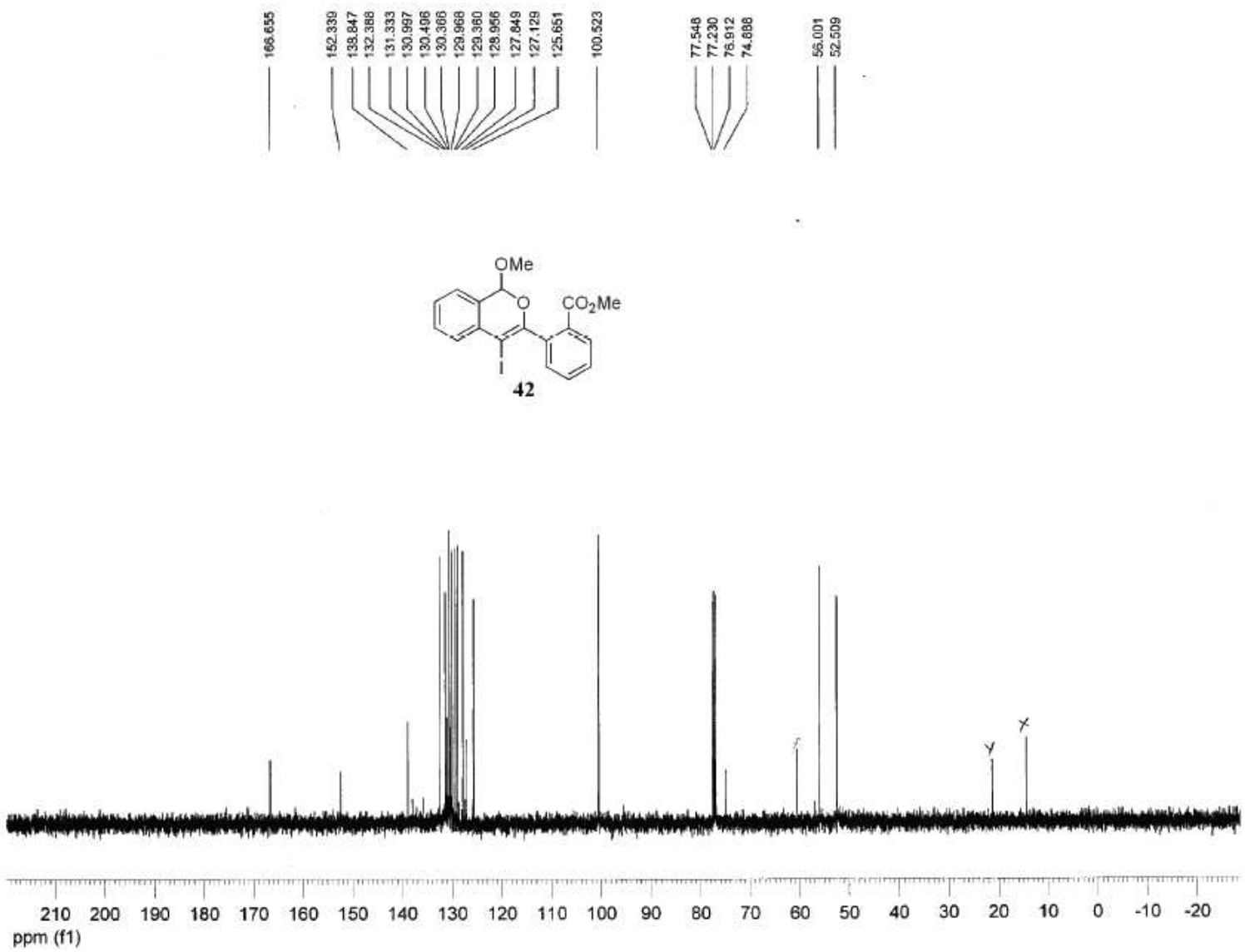

OMe
O
O
Se
40
integral
(ppm)

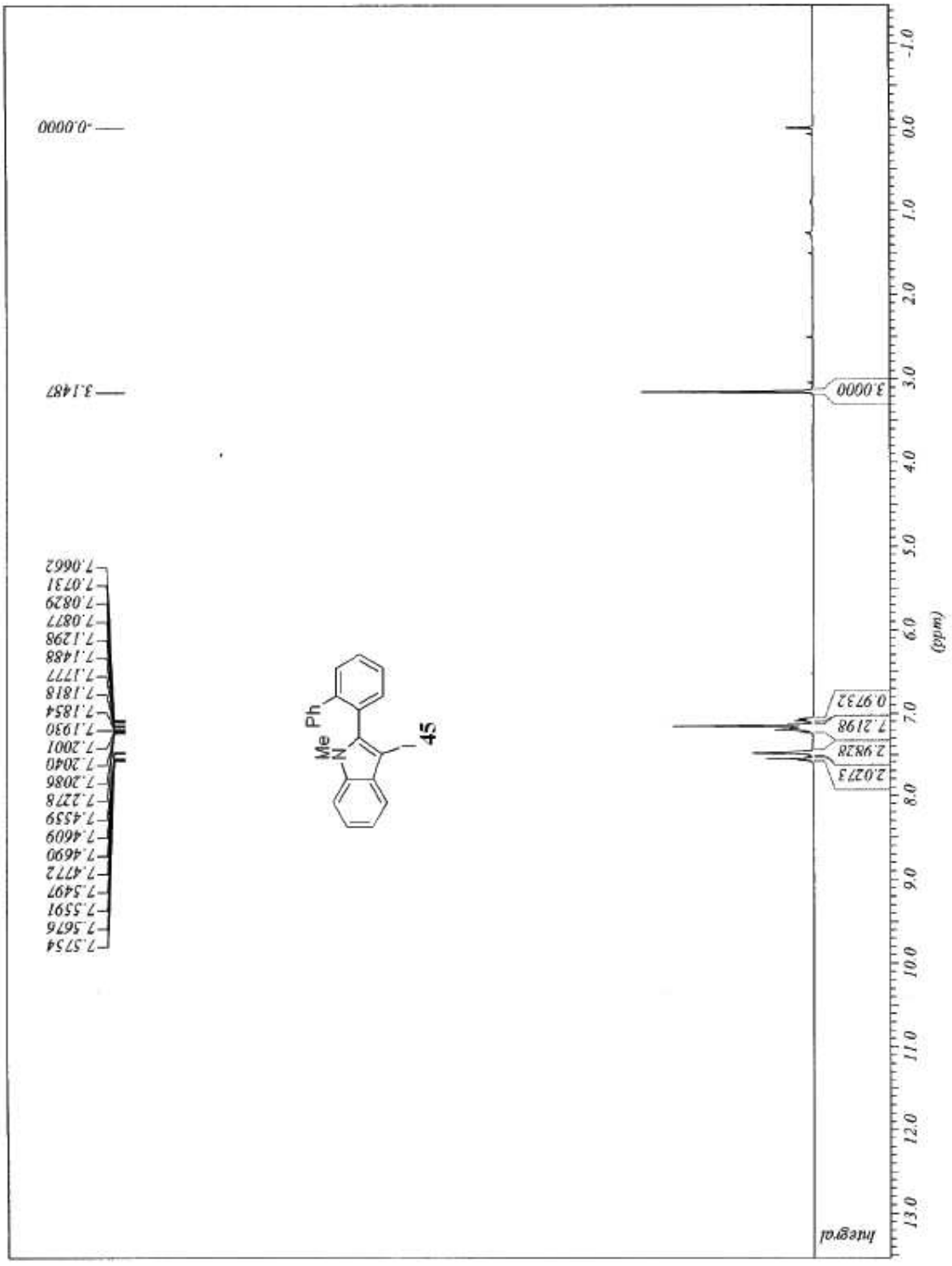

OMe
O
O
Se
40
55.4919
77.5485
77.2300
76.9115
162.4158
157.3471
157.2675
138.2476
135.3436
131.8066
131.5981
130.9649
129.8693
129.6191
129.3196
128.7320
128.0041
126.4573
123.8529
121.2446
120.2475
111.0124
107.6952
(ppm)

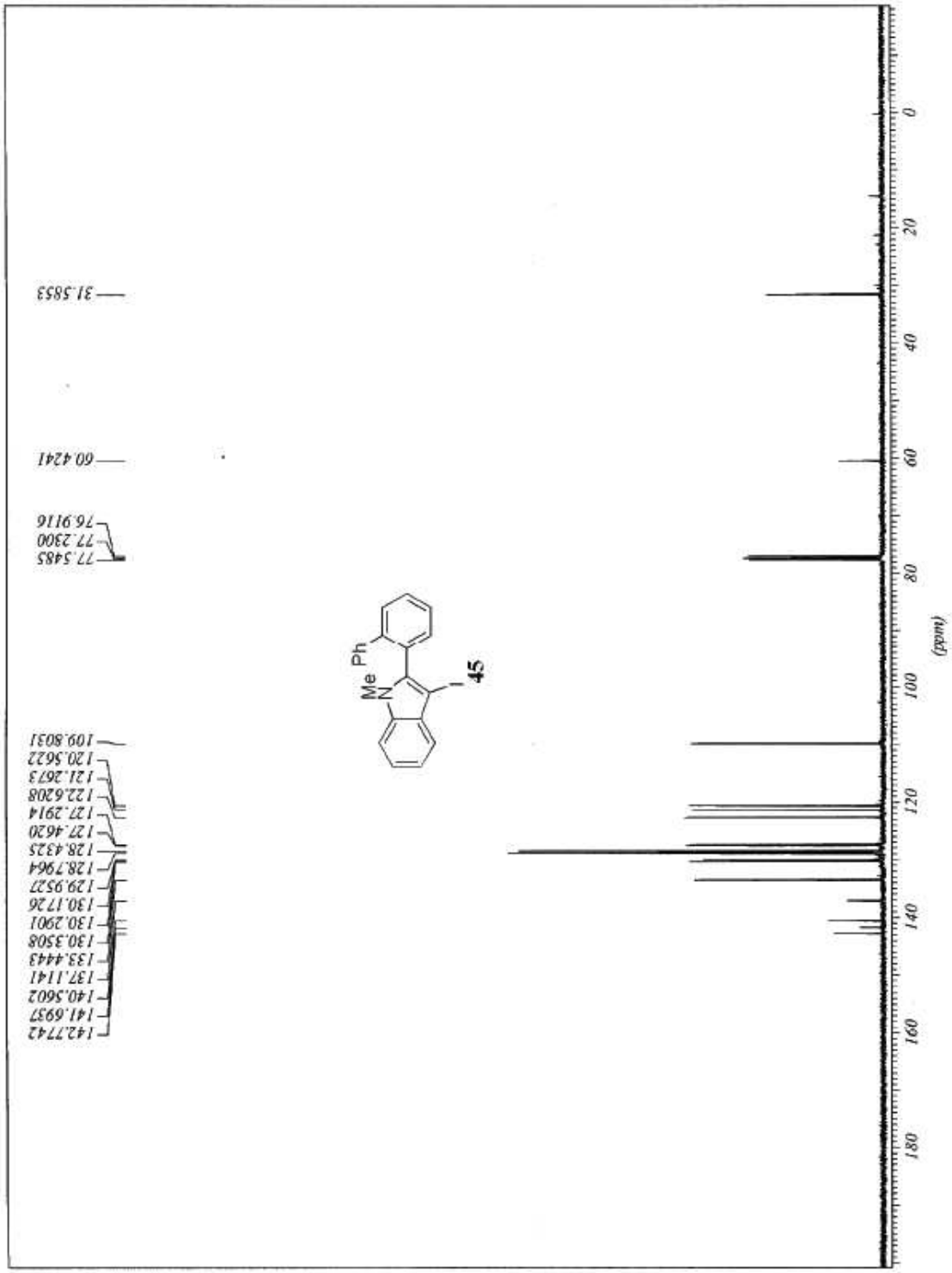

41
Integral
1.0748
3.3673
2.1422
1.0460
1.0000
0.9121
(ppm)
0.0000
10.0831
8.3518
8.3328
8.0495
8.0474
8.0307
8.0284
7.8843
7.8815
7.8644
7.8614
7.8433
7.8387
7.8220
7.7738
7.7706
7.7552
7.7520
7.7367
7.7333
7.7067
7.6898
7.6879
7.6710
7.6589
7.6550
7.6390
7.6227
7.6186
7.5786
7.5770
7.5598
7.2639

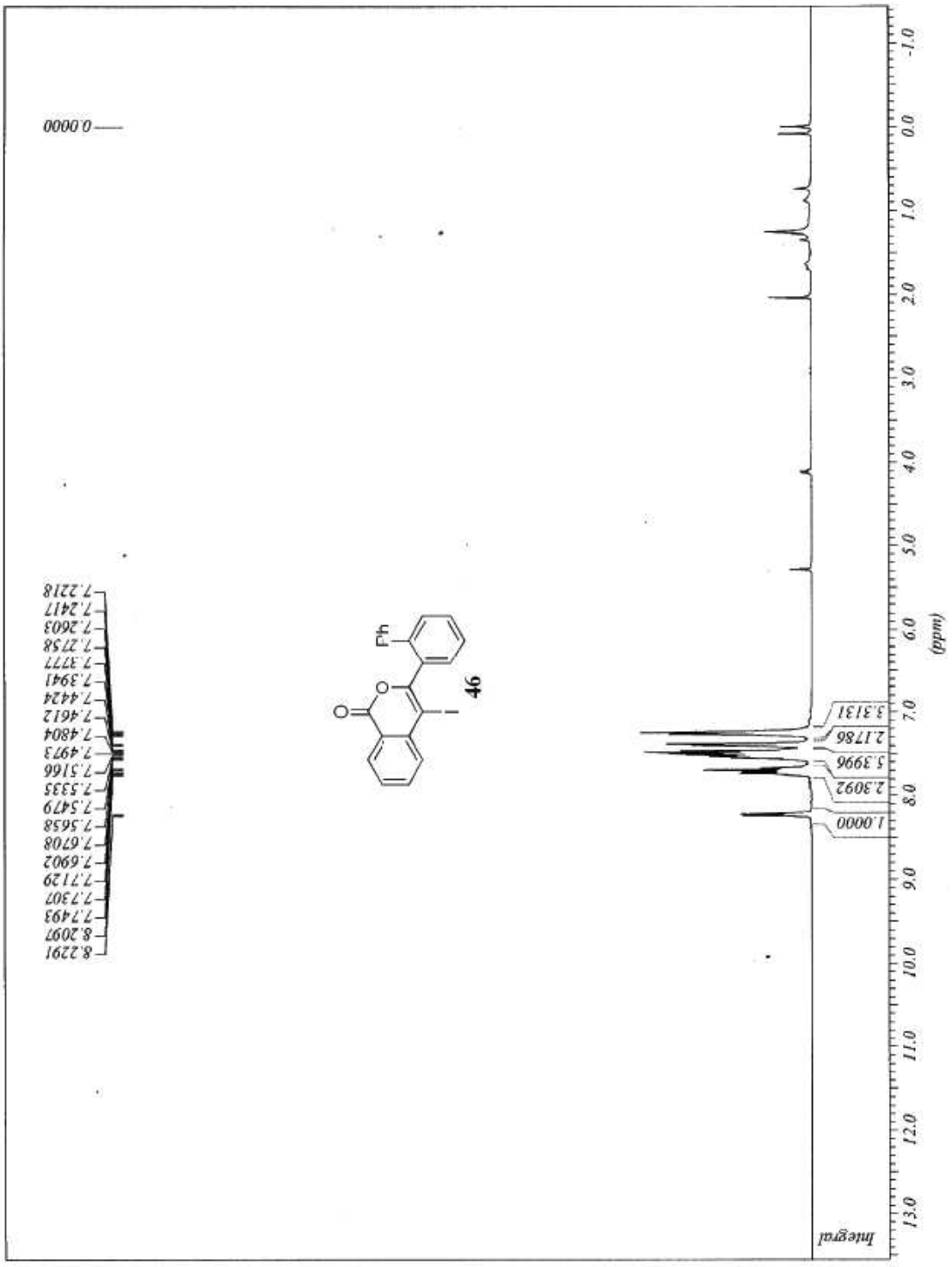

190.1553
161.3202
152.9230
137.5273
137.3236
136.1208
134.2253
134.0585
131.5715
131.2834
130.9612
130.3963
130.1954
129.9300
120.6683
79.6260
77.5485
77.2300
76.9116
41
(ppm)

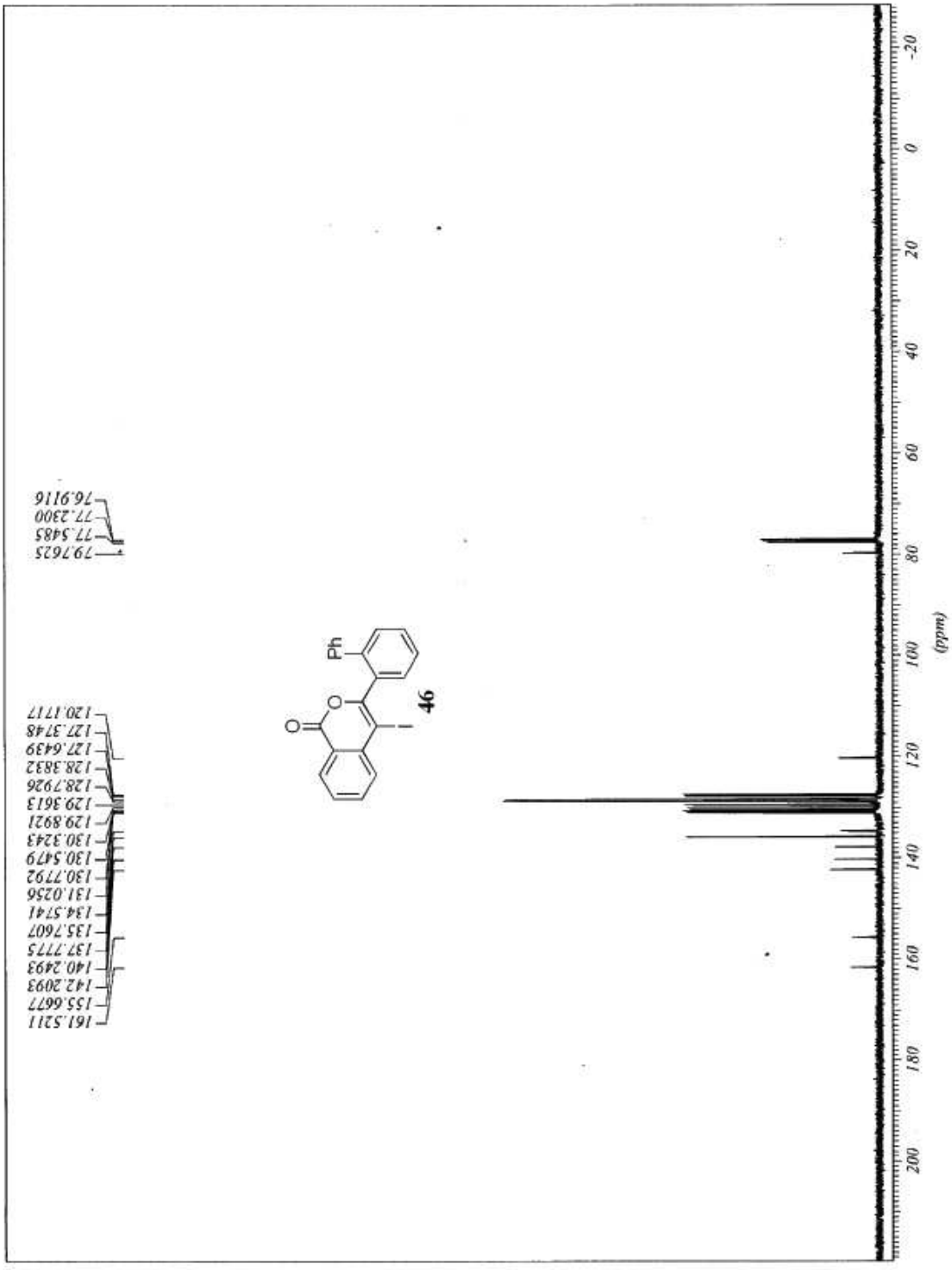
0.000
3.563
3.502
8.063
7.199
7.201
7.202
7.218
7.219
7.221
7.294
7.297
7.312
7.315
7.331
7.334
7.401
7.405
7.407
7.408
7.410
7.414
7.421
7.424
7.427
7.429
7.439
7.443
7.447
7.481
7.484
7.491
7.493
7.494
7.500
7.503
7.510
7.519
7.522
7.593
7.607
7.612
7.616
7.631
7.635
8.037
8.038
8.041
8.057
8.058
8.060
CO2Me
OMe
42
3.00
3.05
1.00
1.00
1.08
1.13
1.20
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (f1)

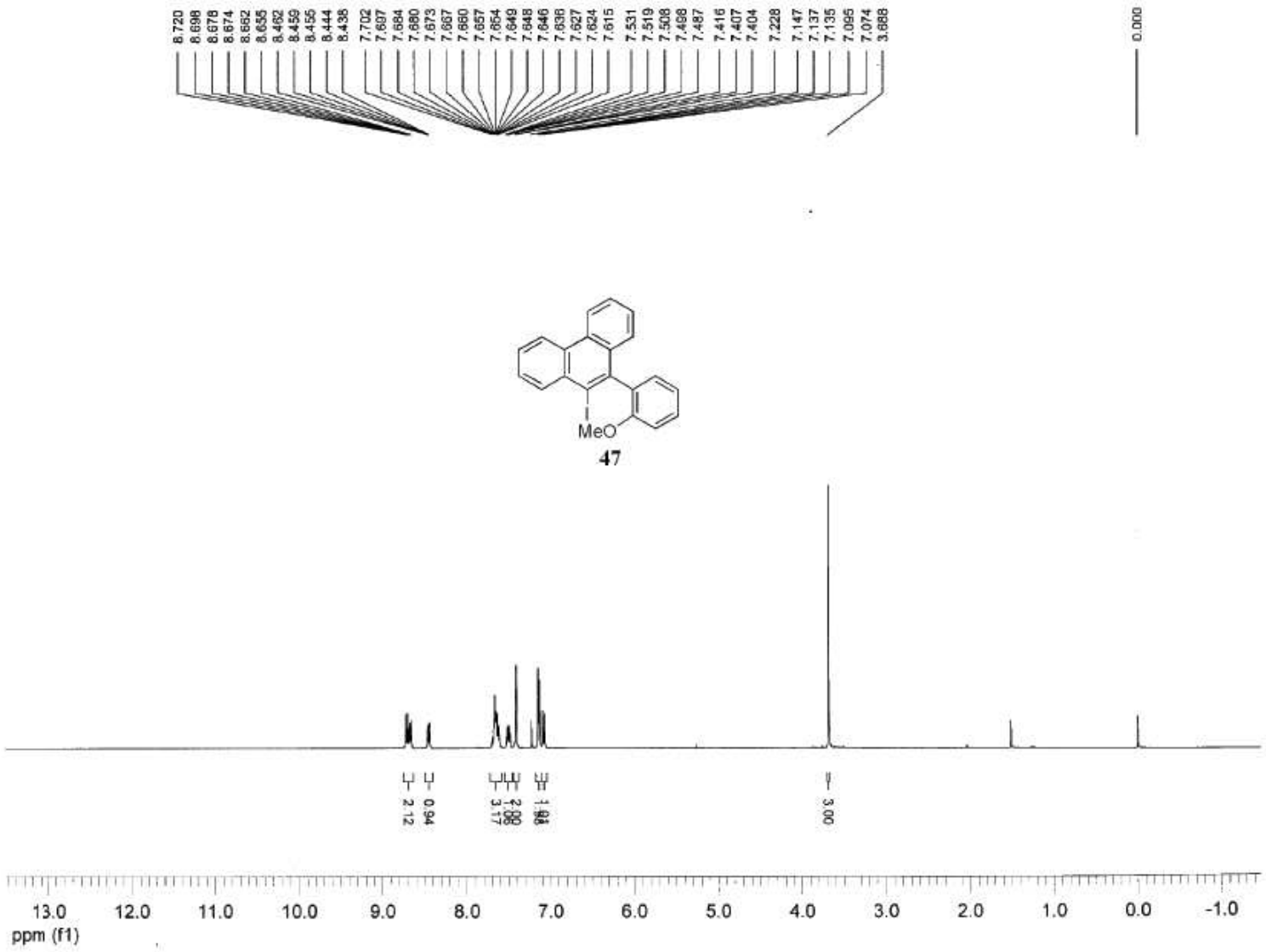

166.655
152.339
138.847
132.388
131.333
130.997
130.496
130.366
129.968
129.360
128.956
127.849
127.129
125.651
100.523
77.548
77.230
76.912
74.888
56.001
52.509
OMe
O
CO2Me
I
42
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
ppm (f1)

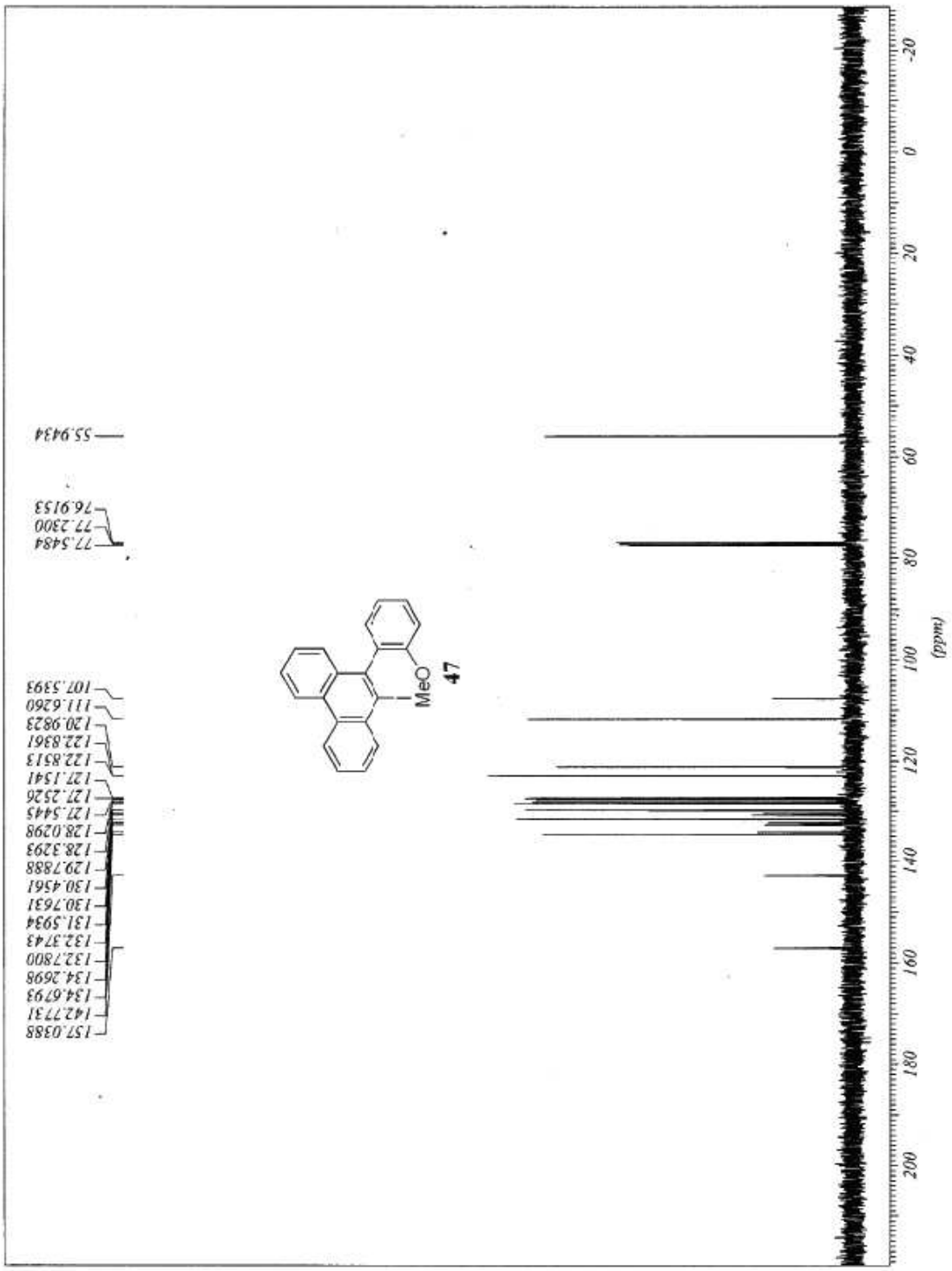

Integral
(ppm)
3.0000
2.0273
2.9828
7.2198
0.9732
45
Ph
Me
N
7.0662
7.0731
7.0829
7.0877
7.1298
7.1488
7.1777
7.1818
7.1854
7.1930
7.2001
7.2040
7.2086
7.2278
7.4559
7.4609
7.4690
7.4772
7.5497
7.5591
7.5676
7.5754
3.1487
-0.0000

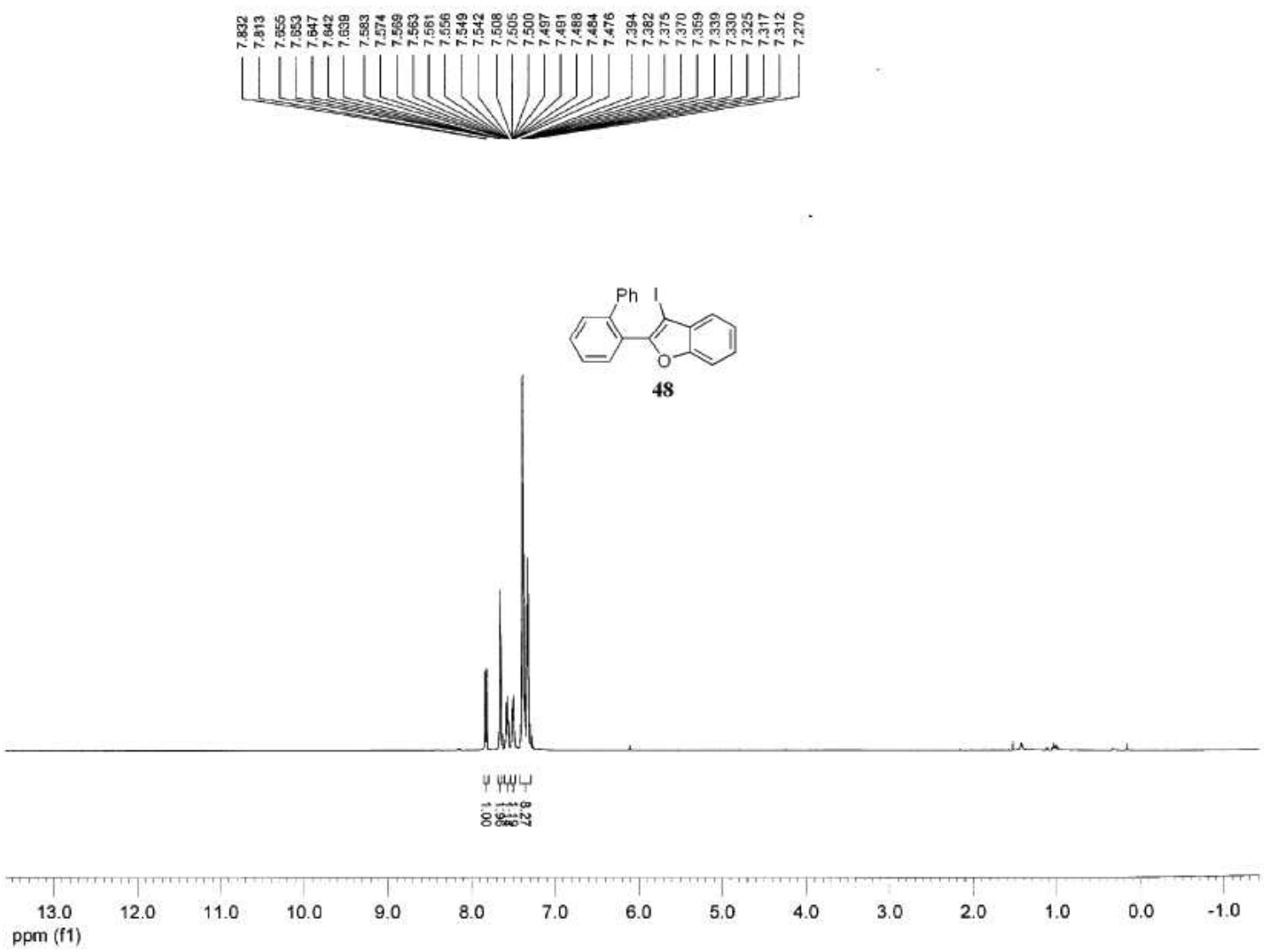

31.5853
60.4241
76.9116
77.2300
77.5485
109.8031
120.5622
121.2673
122.6208
127.2914
127.4620
128.4325
128.7964
129.9527
130.1726
130.2901
130.3508
133.4443
137.1141
140.5602
141.6937
142.7742
Me
Ph
N
45
0
20
40
60
80
100
120
140
160
180
(ppm)


0.0000
8.2291
8.2097
7.7493
7.7307
7.7129
7.6902
7.6708
7.5658
7.5479
7.5335
7.5166
7.4973
7.4804
7.4612
7.4424
7.3941
7.3777
7.2758
7.2603
7.2417
7.2218
Ph
O
O
I
46
Integral
3.3131
2.1786
5.3996
2.3092
1.0000
(ppm)
13.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0

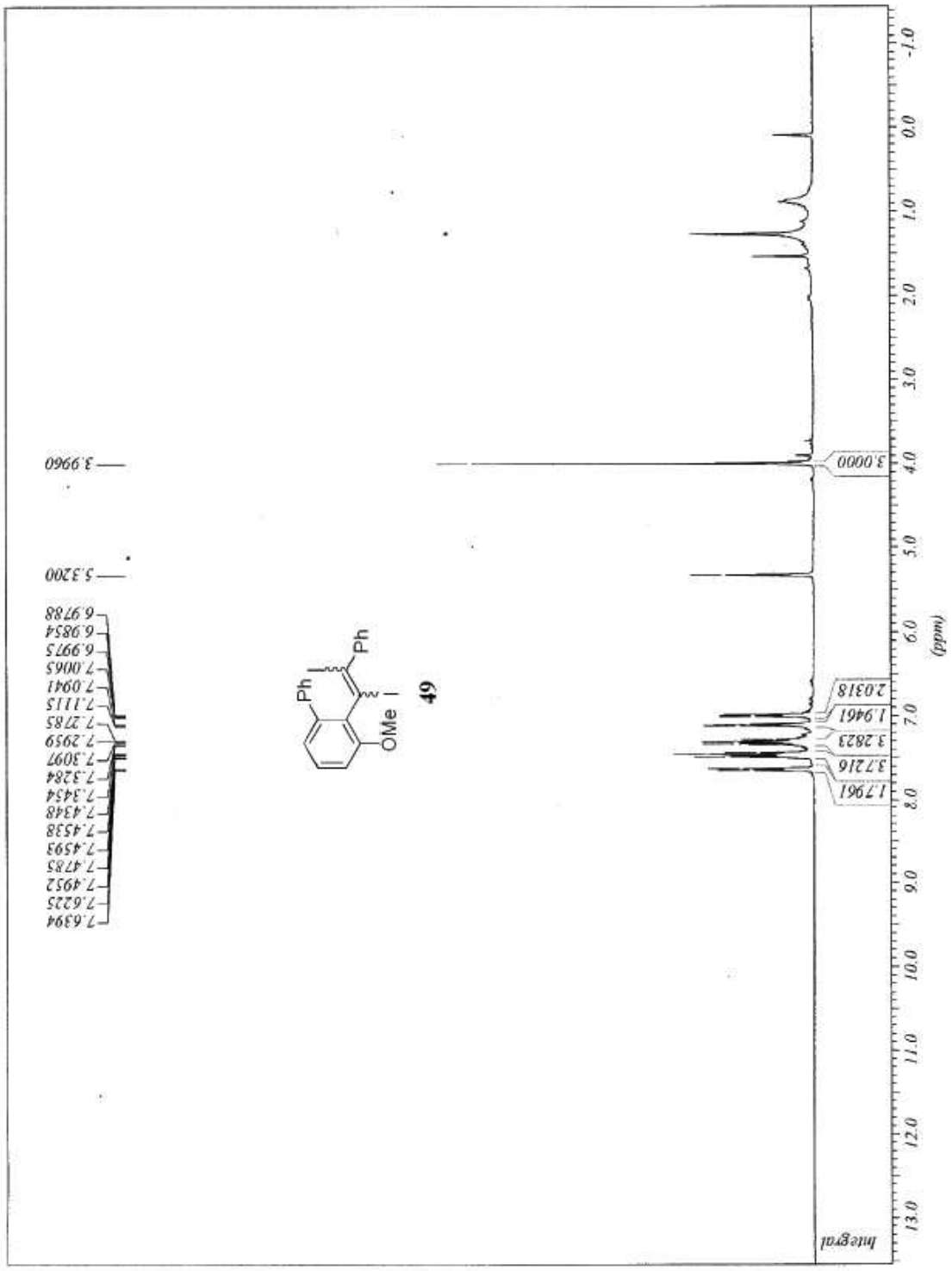

Ph
O
O
I
46
161.5211
155.6677
142.2093
140.2493
137.7775
135.7607
134.5741
131.0256
130.7792
130.5479
130.3243
129.8921
129.3613
128.7926
128.3832
127.6439
127.3748
120.1717
79.7625
77.5485
77.2300
76.9116
200
180
160
140
120
100
80
60
40
20
0
-20
(ppm)

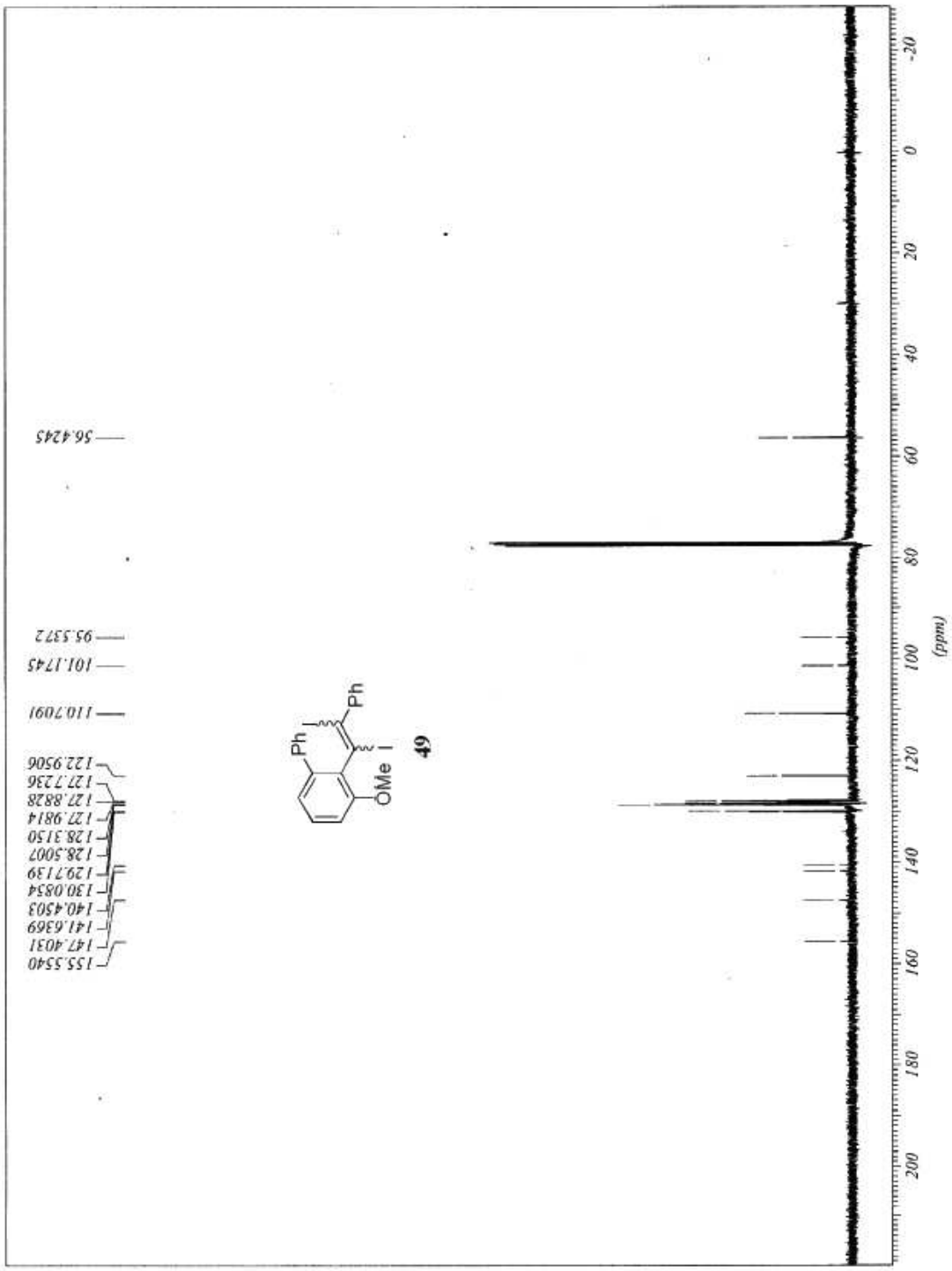

8.720
8.698
8.678
8.674
8.662
8.635
8.482
8.459
8.455
8.444
8.438
7.702
7.697
7.684
7.680
7.673
7.667
7.660
7.657
7.654
7.649
7.648
7.646
7.636
7.627
7.624
7.615
7.531
7.519
7.508
7.498
7.487
7.416
7.407
7.404
7.228
7.147
7.137
7.135
7.095
7.074
3.888
0.000
MeO
47
2.12
0.94
2.08
3.17
1.00
3.00
13.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
ppm (f1)


55.9434
77.5484
77.2300
76.9153
157.0388
142.7731
134.6793
134.2698
132.7800
132.3743
131.5934
130.7631
130.4561
129.7888
128.3293
128.0298
127.5445
127.2526
127.1541
122.8513
122.8361
120.9823
111.6260
107.5393
MeO
47
(ppm)


7.832
7.813
7.655
7.653
7.647
7.642
7.639
7.583
7.574
7.569
7.563
7.561
7.556
7.549
7.542
7.508
7.505
7.500
7.497
7.491
7.488
7.484
7.476
7.394
7.382
7.375
7.370
7.359
7.339
7.330
7.325
7.317
7.312
7.270
Ph
I
O
48
1.00
1.98
1.18
8.27
13.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
ppm (f1)

65.7166
76.9116
77.2300
77.5447
111.3644
121.5850
123.4464
125.4064
127.1692
127.2071
128.3065
128.4809
128.8221
130.2968
130.6456
131.4531
131.9687
140.8662
142.6973
154.3244
155.9735
Ph
O
48
(ppm)
0
20
40
60
80
100
120
140
160
180
200


Ph
Ph
OMe
I
49
7.6394
7.6225
7.4952
7.4785
7.4593
7.4538
7.4348
7.3454
7.3284
7.3097
7.2959
7.2785
7.1115
7.0941
7.0065
6.9975
6.9854
6.9788
5.3200
3.9960
3.0000
2.0318
1.9461
3.2823
3.7216
1.7961
bluegrnl
(ppm)


155.5540
147.4031
141.6369
140.4503
130.0834
129.7139
128.5007
128.3150
127.9814
127.8828
127.7236
122.9506
110.7091
101.1745
95.5372
56.4245
Ph
Ph
OMe I
49
(ppm)