SUPPORTING INFORMATION

Synthesis of Fluoren-9-ones via Palladium-Catalyzed Cyclocarbonylation of *o*-Halobiaryls

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Reagents. 2-Iodobiphenyl and 2-iodothioanisole were obtained from Lancaster Synthesis Ltd. 2-Bromobiphenyl, 1-bromo-2-iodobenzene, 4-bromotoluene, 3-bromotoluene, 2-fluoroanisole, 4-bromoanisole, phenyllithium, phenylacetylene, cesium carbonate, pivalic acid and triethylamine were obtained from Aldrich Chemical Co., Inc. Bis(tricyclohexylphosphine)palladium(0) was purchased from Strem Chemicals, Inc.

Synthesis of *o*-Halobiaryls

2-Iodo-4'-methoxybiphenyl (3). 2-Iodo-4'-methoxybiphenyl (**3**) was prepared by a procedure reported by Hart.[1] A solution of 2-bromoiodobenzene (1.415 g, 5.0 mmol) in THF (10 mL) was added slowly (90 min) to a solution of 4-methoxyphenylmagnesium bromide [prepared from 4-bromoanisole (1.87 g, 10 mmol) and Mg (0.246 g, 10 mmol) in THF (30 mL)], and the mixture was stirred under Ar for an additional 14 h at room temperature. The reaction was quenched by adding 3.8 g (15 mmol) of iodine, and the

mixture was stirred for an additional 30 min at room temperature. The excess I_2 was destroyed by adding 10% aq $NaHSO_3$ (35 mL); the organic layer was separated and rewashed with brine (20 mL). Finally, the organic layer was dried ($MgSO_4$), filtered and the solvent removed under reduced pressure. The residue was chromatographed using 20:1 hexanes/ethyl acetate to afford 0.610 g (39%) of the desired compound **3** as a yellow solid. This compound was further purified by recrystallization from hexanes to yield a white solid: mp 58-60 ^{0}C; ^{1}H NMR ($CDCl_3$) δ 3.86 (s, 3H), 6.94-7.02 (m, 3H), 7.26-7.30 (m, 3H), 7.36 (td, *J* = 7.2, 0.9 Hz, 1H), 7.93 (dd, *J* = 8.0, 0.9 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 55.5, 99.4, 113.5, 128.4, 128.8, 130.4, 130.7, 137.0, 139.7, 146.5, 159.3; HRMS *m/z* 309.98594 (calcd for $C_{13}H_{11}IO$, 309.98547).

2-Iodo-4'-methylbiphenyl (5). This biphenyl was prepared by the same method used to prepare **3**, but 4-bromotoluene (1.71 g, 10 mmol) was employed. It was obtained as a colorless liquid (0.88 g, 60%) with spectral properties identical to those previously reported.[2]

2-Iodo-3'-methylbiphenyl (10). This biphenyl was prepared by the same method used to prepare **3**, but 3-bromotoluene (1.71 g, 10 mmol) was employed. It was obtained as a colorless liquid (0.84 g, 57%): ^{1}H NMR ($CDCl_3$) δ 2.44 (s, 3H), 7.04 (td, *J* = 7.6, 2 Hz, 1H), 7.16-7.18 (m, 2H), 7.22-7.24 (m, 1H), 7.31-7.41 (m, 3H), 7.97 (dd, *J* = 8.0, 0.8 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 21.6, 98.8, 126.5, 127.9, 128.1, 128.4, 128.8, 130.1, 130.2, 137.6, 139.5, 144.2, 146.8; HRMS *m/z* 293.99093 (calcd for $C_{13}H_{11}I$, 293.99055).

4-(2-Iodophenyl)benzaldehyde (8). This aldehyde was prepared in two steps from 2-iodo-4'-methylbiphenyl (**5**). Compound 5 was brominated by the procedure of Ponchant[2] to afford 4'-bromomethyl-2-iodobiphenyl (**26**). Compound **26** was then

oxidized to aldehyde **8** using the following procedure: a solution of $AgClO_4$ (0.29 g, 1.4 mmol) in DMSO (5 mL) was added quickly with stirring to 4'-bromomethyl-2-iodo-biphenyl (0.50 g, 1.3 mmol) in DMSO (2 mL). The resulting mixture was allowed to stand for 30 min at room temperature in the dark. At this point, triethylamine (0.81 g, 8 mmol) was added and the mixture stirred for an additional 20 min. The reaction mixture was quenched with brine (25 mL) and extracted with diethyl ether (60 mL). The aqueous layer was reextracted with diethyl ether (20 mL), and the organic layers were combined, dried ($MgSO_4$) and filtered. The solvent was removed under reduced pressure, and the resulting yellow oil was purified by column chromatography using 5:1 hexanes/ethyl acetate to afford 0.308 g (77%) of 4-(2-iodophenyl)benzaldehyde (**8**) as a white solid: mp 79-80 ^{o}C (lit.[3] mp 80-81 ^{o}C). The spectral properties were identical to those previously reported.[3]

3-(2-Iodophenyl)benzaldehyde (13). This aldehyde was prepared in two steps from 2-iodo-3'-methylbiphenyl (**10**) by the same method used to prepare 4-(2-iodo-phenyl)benzaldehyde (**8**). Bromination[2] of **10** produced 3'-bromomethyl-2-iodo-biphenyl (**27**) in 70% yield: ^{1}H NMR ($CDCl_3$) δ 4.54 (s, 2H), 7.04 (td, *J* = 8.55, 1.8 Hz, 1H), 7.26-7.42 (m, 6H), 7.95 (dd, *J* = 7.8, 1.2 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 33.4, 98.6, 128.3, 128.4, 128.5, 129.1, 129.5, 130.1, 130.2, 137.5, 139.7, 144.7, 146.0. Oxidation of **27** (0.485 g, 1.3 mmol) using DMSO (7 mL) and $AgClO_4$ (0.29 g, 1.4 mmol) gave 0.300 g (75%) of the desired product **13** as a clear oil: ^{1}H NMR ($CDCl_3$) δ 7.06 (td, *J* = 7.8, 1.8 Hz, 1 H), 7.30 (dd, *J* = 7.8, 1.8 Hz, 1 H), 7.41 (td, *J* = 7.4, 1.2 Hz, 1 H), 7.56-7.64 (m, 2 H), 7.85-7.86 (m, 1 H), 7.91 (dt, *J* = 6.9, 1.8 Hz, 1 H), 7.96 (dd, *J* = 7.8, 1.2 Hz, 1 H), 10.06 (s, 1 H); ^{13}C NMR ($CDCl_3$) δ 98.3, 128.5, 128.8, 128.9, 129.5, 130.1, 130.8, 135.5,

136.3, 139.8, 145.0, 145.2, 192.1; IR ($CHCl_3$) 1698 cm^{-1}; HRMS *m/z* 307.97049 (calcd for $C_{13}H_9IO$, 307.96982).

2-Iodo-3-methoxybiphenyl (16). 1-Lithio-2-methoxybiphenyl (**28**) was prepared in situ from 2-fluoroanisole (1.134 g, 9.0 mmol) and phenyllithium (10 mL of 1.8 M solution in hexanes) in diethyl ether (20 ml) by the procedure of Huisgen.[4] The reaction mixture was cooled to 0 ^oC using an ice bath and I_2 (3.5 g, 13.8 mmol) was added slowly with constant stirring. The mixture was stirred for an additional 30 min, then the excess I_2 was destroyed by adding 10% aq $NaHSO_3$ (35 mL). The organic layer was rewashed with brine (20 mL), dried ($MgSO_4$) and filtered. Removal of solvent under reduced pressure gave a yellow oil that was purified by chromatography using 20:1 hexanes/ethyl acetate to afford 1.02 g (36%) of the desired compound **16** as a yellow solid. Recrystallization from hexanes/ethyl acetate gave the desired product as a white solid: mp 83-84 ^oC; ^1H NMR ($CDCl_3$) δ 3.94 (s, 3H), 6.82 (dd, *J* = 8.2, 1.2 Hz, 1H), 6.92 (dd, *J* = 7.5, 1.2 Hz, 1H), 7.30-7.43 (m, 6H); ^{13}C NMR ($CDCl_3$) δ 56.7, 91.4, 109.5, 122.8, 127.6, 127.9, 129.0, 129.4, 144.6, 148.9, 158.4; HRMS *m/z* 309.98594 (calcd for $C_{13}H_{11}IO$, 309.98547).

9-Iodo-10-phenylphenanthrene (18). This iodobiaryl was prepared from 9-phenyl-10-(trimethylsilyl)phenanthrene[5] using an iodination procedure from the literature.[6] To a solution of 9-phenyl-10-(trimethylsilyl)phenanthrene (0.133 g, 0.4 mmol) in CH_2Cl_2 (2 mL) was added ICl (0.078 g, 0.48 mmol) in CH_2Cl_2 (2 mL) at -78 ^oC. The reaction mixture was stirred at -78 ^oC for 1 h, then the excess ICl was destroyed by adding 10% aq $Na_2S_2O_3$ (10 mL). The organic layer was dried ($MgSO_4$), filtered and the solvent removed under reduced pressure. The remaining solid was recrystallized from

methanol/CH_2Cl_2 to afford 0.13 g (85%) of the desired compound **18** as a white solid: mp 118-120 ^oC; ^1H NMR ($CDCl_3$) δ 7.28 (dd, *J* = 6.0, 1.8 Hz, 2H), 7.38 (d, *J* = 3.9 Hz, 2H), 7.50-7.56 (m, 3H), 7.60-7.70 (m, 3H), 8.44-8.47 (m, 1H), 8.63-8.70 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 106.6, 122.6, 122.7, 127.0, 127.1, 127.5, 127.8, 128.1, 128.5, 128.7, 130.0, 130.3, 130.6, 132.3, 132.4, 134.7, 145.3, 145.4; IR ($CDCl_3$) 3064, 3025, 1481 cm^{-1}; HRMS *m/z* 380.0062 (calcd for $C_{20}H_{13}I$, 380.0062).

2-Bromo-1-phenylnaphthalene (20). This starting material was prepared by the procedure of Wittig.[7] ^1H NMR ($CDCl_3$) δ 7.27-7.31 (m, 2H), 7.32-7.36 (m, 1H), 7.41-7.52 (m, 5H), 7.65-7.72 (m, 2H), 7.79-7.82 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 121.8, 126.3, 127.0, 127.1, 128.1, 128.2, 128.6, 129.2, 130.2, 130.4, 132.6, 134.2, 139.9, 140.1.

4-Iodo-3-phenylisoquinoline (22). This aryl iodide was prepared by the procedure of Larock.[8] ^1H NMR ($CDCl_3$) δ 7.45-7.53 (m, 3H), 7.61-7.71 (m, 3H), 7.83 (ddd, *J* = 8.4, 6.9, 1.5 Hz, 1H), 7.96 (d, *J* = 8.1 Hz, 1H), 8.24 (d, *J* = 8.4 Hz, 1H), 9.17 (s, 1H); ^{13}C NMR ($CDCl_3$) δ 98.2, 128.0, 128.1, 128.1, 128.4, 129.9, 132.4, 132.5, 138.7, 143.7, 152.1, 157.1 (one sp^2 carbon missing due to overlap); IR (neat) 3055, 1630, 1549 cm^{-1}; HRMS *m/z* 330.9852 (calcd for $C_{15}H_{10}IN$, 330.9858).

3-Iodo-2-phenylbenzothiophene (24). This starting material was prepared in two steps from commercially available 2-iodothioanisole. To a solution of 2-iodo-thioanisole (1.53 g, 6.1 mmol) and phenylacetylene (0.75 g, 7.3 mmol) in Et_3N (25 mL) was added $PdCl_2(PPh_3)_2$ (86 mg, 2 mol %). The mixture was stirred for 5 min under Ar and CuI (11 mg, 1 mol %) was added. The resulting mixture was then heated under an Ar atmosphere at 60 ^oC for 2 h. The reaction mixture was allowed to cool to room temperature, and the ammonium iodide salt was removed by filtration. The solvent was

removed under reduced pressure and the residue was purified by column chromatography using 20:1 hexanes/ethyl acetate to afford 1.26 g (92%) of 2-(2-phenylethynyl)thioanisole (**29**) as a yellow oil: ^{1}H NMR ($CDCl_3$) δ 2.51 (s, 3H), 7.10 (td, *J* = 7.5, 1.2 Hz, 1H), 7.17 (dd, *J* = 7.8, 0.9 Hz, 1H), 7.27-7.37 (m, 4H), 7.48 (ddd, *J* = 7.5, 1.2, 0.6 Hz, 1H), 7.56-7.60 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 15.3, 87.1, 96.1, 123.4, 124.4, 124.4, 124.5, 128.6, 128.6, 129.0, 131.8, 132.5, 141.9; IR (CH_2Cl_2) 1599, 1491 cm^{-1}; HRMS *m/z* 224.06627 (calcd for $C_{15}H_{12}S$, 224.06597). 3-Chloromercurio-2-phenyl-benzothiophene was prepared in situ from **29** by the procedure of Larock.[9] To a suspension of $Hg(OAc)_2$ (0.318 g, 1 mmol) in glacial HOAc (3 mL) at room temperature was added 2-(2-phenylethynyl)thioanisole (0.224 g, 1 mol), and the resulting solution was stirred at room temperature for 30 min. The reaction mixture was quenched by adding I_2 (0.38 g, 1.5 mmol), and the mixture was stirred vigorously for an additional 30 min. Excess I_2 was destroyed by adding 10% aq $Na_2S_2O_3$ (30 mL), and the aqueous layer was extracted with diethyl ether (50 mL). The organic layer was dried ($MgSO_4$), filtered and the solvent removed under reduced pressure. The residue was purified by column chromatography using 30:1 hexanes/ethyl acetate to afford 0.304 g (90%) of 3-iodo-2-phenylbenzothiophene as a yellow oil: ^{1}H NMR ($CDCl_3$) δ 7.34-7.40 (td, *J* = 7.5, 1.5 Hz, 1H), 7.42-7.50 (m, 4H), 7.66-7.69 (m, 2H), 7.75-7.78 (m, 1H), 7.81-7.84 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 79.7, 122.4, 125.7, 125.8, 126.6, 128.8, 129.2, 130.3, 134.9, 139.2, 142.2, 142.4; IR (CH_2Cl_2) 3065, 1601, 1477, 1433 cm^{-1}; HRMS *m/z* 335.94745 (calcd for $C_{14}H_9IS$, 335.94697).

General Procedure for the Pd-Catalyzed Cyclocarbonylation of *o*-Halobiaryls. DMF (6 mL), $Pd(PCy_3)_2$ (8.4 mg, 0.0125 mmol), anhydrous cesium pivalate (0.117 g, 0.5 mmol), and the *o*-halobiaryl (0.25 mmol) were stirred under an Ar atmosphere at room temperature for 5 min. The mixture was flushed with CO and fitted with a CO filled balloon. The reaction mixture was heated to 110 ^{o}C with vigorous stirring for 7 h. The reaction mixture was then cooled to room temperature, diluted with diethyl ether (35 mL) and washed with brine (30 mL). The aqueous layer was reextracted with diethyl ether (15 mL). The organic layers were combined, dried ($MgSO_4$), filtered and the solvent removed under reduced pressure. The residue was purified by column chromatography on a silica gel column.

Fluoren-9-one (2). The reaction mixture was chromatographed using 7:1 hexanes/ethyl acetate to afford 45.1 mg (100%) of the indicated compound as a yellow solid: mp 82-83 ^{o}C. This compound was identified by comparing the ^{1}H NMR and ^{13}C NMR spectra and melting point with an authentic sample obtained from Aldrich Chemical Co., Inc.

2-Methylfluoren-9-one (5). The reaction mixture was chromatographed using 7:1 hexanes/ethyl acetate to afford 47.1 mg (97%) of the indicated compound as a yellow solid: mp 90-91 ^{o}C (lit.[10] mp 92 ^{o}C); ^{1}H NMR ($CDCl_3$) δ 2.33 (s, 3H), 7.19-7.24 (m, 2H), 7.33 (d, *J* = 7.6 Hz, 1H), 7.04- 7.41 (m, 3H), 7.58 (d, *J* = 7.2 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 21.4, 120.0, 120.2, 124.2, 125.0, 128.6, 134.3, 134.4, 134.7, 135.1, 139.3, 141.8, 144.7, 194.2. The spectral properties were identical to those previously reported.[10]

2-Methoxyfluoren-9-one (7). The reaction mixture was chromatographed using 6:1 hexanes/ethyl acetate to afford 52.6 mg (100%) of the indicated compound as a

yellow solid: mp 78-79 ^{o}C (lit.[11] mp 78 ^{o}C); ^{1}H NMR ($CDCl_3$) δ 3.82 (s, 3H), 6.94 (dd, *J* = 8.4, 2.8 Hz, 1H), 7.14-7.18 (m, 2H), 7.34-7.41 (m, 3H), 7.56 (d, *J* = 7.6 Hz, 1H); IR (CH_2Cl_2) 1717 cm^{-1}. The spectral properties were identical to those previously reported.[11]

9-Oxofluorene-2-carbaldehyde (9). The reaction mixture was chromatographed using 2:1 hexanes/ethyl acetate to afford 52.0 mg (100%) of the indicated compound as a yellow solid: mp 204-205 ^{o}C (lit.[12] mp 203-204 ^{o}C); ^{13}C NMR ($CDCl_3$) δ 121.0, 121.7, 125.0, 130.8, 135.0, 135.1, 135.4, 136.4, 137.5, 143.3, 149.9, 190.8, 192.4. The spectral properties were identical to those previously reported.[12]

3-Methylfluoren-9-one (11). The reaction mixture was chromatographed using 9:1 hexanes/ethyl acetate to afford 43.7 mg (90%) of the indicated compound as a yellow solid: mp 66-67 ^{o}C (lit.[13] mp 65 ^{o}C); ^{13}C NMR ($CDCl_3$) δ 22.2, 120.1, 121.3, 124.2, 124.3, 129.0, 129.6, 131.9, 134.5, 134.7, 144.3, 144.8, 145.9, 193.3. The spectral properties were identical to those previously reported.[14]

1-Methylfluoren-9-one (12). The reaction mixture was chromatographed using 9:1 hexanes/ethyl acetate to afford 4.9 mg (10%) of the indicated compound as a yellow solid: mp 98-99 ^{o}C (lit.[15] mp 98-99 ^{o}C). The spectral properties were identical to those previously reported.[15]

9-Oxofluorene-3-carbaldehyde (14) and 9-oxofluorene-1-carbaldehyde (15). The reaction mixture was chromatographed using 3:1 hexanes/ethyl acetate to afford 48.9 mg (94%) of the indicated compounds as a 9:1 inseparable mixture of isomers (the ratio was determined by ^{1}H NMR spectroscopic analysis). **9-Oxo-fluorene-1-carbaldehyde** (minor isomer): ^{1}H NMR ($CDCl_3$) δ 11.06 (s, 1H) (as a characteristic peak); ^{13}C NMR

($CDCl_3$) δ 120.7, 124.9, 125.2, 126.3, 130.0, 133.6, 134.0, 134.7, 135.6, 143.8, 144.5, 145.0, 190.5, 194.4; IR (CH_2Cl_2) 1695, 1711 cm^{-1}; HRMS *m/z* 208.05281 (calcd for $C_{14}H_8O_2$, 208.05243). **9-Oxofluorene-3-carbaldehyde** (major isomer): this isomer was purified by recrystallization from hexanes/ethyl acetate to afford 29.2 mg (56%) of the desired compound as a yellow solid: mp 148-149 ^oC (lit.[16] mp 149-150 ^oC); ^1H NMR ($CDCl_3$) δ 7.37 (td, *J* = 7.5, 1.2 Hz, 1H), 7.57 (td, *J* = 7.5, 1.2 Hz, 1H), 7.62-7.64 (m, 1H), 7.71 (d, *J* = 7.5 Hz, 1H), 7.82-7.82 (m, 2H), 8.03 (t, *J* = 0.9, 1H), 10.10 (s, 1H); ^{13}C NMR ($CDCl_3$) δ 119.7, 121.2, 124.8, 125.0, 130.1, 132.7, 134.4, 135.7, 138.8, 141.2, 143.6, 145.2, 191.7, 193.0; IR ($CHCl_3$) 1719, 1701 cm^{-1}; HRMS *m/z* 208.05281 (calcd for $C_{14}H_8O_2$, 208.05243).

1-Methoxyfluoren-9-one (17). The reaction mixture was chromatographed using 3:1 hexanes/ethyl acetate to afford 52.0 mg (99%) of the indicated compound as a yellow solid: mp 142-143 ^oC (lit.[17] mp 141-142 ^oC); ^1H NMR ($CDCl_3$) δ 3.97 (s, 3H), 6.81 (d, *J* = 8.1 Hz, 1H), 7.11 (dd, *J* = 7.2, 0.6 Hz, 1H), 7.27 (td, *J* = 6.9, 1.2 Hz, 1H), 7.40-7.49 (m, 3H), 7.63 (dt, *J* = 7.2, 1.0 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 56.2, 113.1, 113.3, 120.3, 120.4, 124.1, 129.4, 134.1, 134.7, 137.0, 143.4, 146.7, 158.6, 187.5. The spectral properties were identical to those previously reported.[17]

Indene[1,2-*l*]phenanthrene-13-one (19). The reaction mixture was chromatographed using 5:1 hexanes/ethyl acetate to afford 68.7 mg (98%) of the indicated compound as an orange solid: mp 183-184 ^oC (ethanol/acetone) (lit.[18] mp 185-187 ^oC); ^1H NMR ($CDCl_3$) δ 7.25-7.34 (m, 1H), 7.48 (td, *J* = 8.2, 1.2 Hz, 1H), 7.62-7.79 (m, 5H), 8.02 (d, *J* = 7.5 Hz, 1H), 8.57-8.63 (m, 2H), 8.70 (d, *J* = 8.1 Hz, 1H), 9.21-9.24 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 122.8, 123.5, 123.7, 124.0, 125.5, 125.9, 126.2, 127.4,

127.5, 127.7, 127.8, 128.6, 129.0, 129.4, 131.2, 134.1, 134.5, 134.8, 144.0, 144.8, 196.0. The spectral properties were identical to those previously reported.[18]

Benzo[*c*]fluoren-7-one (21). The reaction mixture was chromatographed using 7:1 hexanes/ethyl acetate to afford 55.3 mg (96%) of the indicated compound as an orange solid: mp 160-161 ^{o}C (lit.[19] mp 161-162 ^{o}C). The spectral properties were identical to those previously reported.[19]

11-Oxoindeno[1,2-*c*]isoquinoline (23). The reaction mixture was chromatographed using 1:1 hexanes/ethyl acetate to afford 54.9 mg (95%) of the indicated compound as a yellow solid: mp 198-200 ^{o}C (lit.[20] mp 199-200 ^{o}C); ^{13}C NMR ($CDCl_3$) δ 119.8, 120.7, 123.6, 123.9, 127.7, 128.8, 129.3, 130.6, 132.6, 133.6, 134.7, 135.0, 143.9, 158.4, 162.5, 194.2. The spectral properties were identical to those previously reported.[20]

10-Oxo-10-*H*-benz[*b*]indeno[1,2-*d*]thiophene (25). The reaction mixture was chromatographed using 7:1 hexanes/ethyl acetate to afford 39.6 mg (67%) of the indicated compound as an orange solid: mp 203-204 ^{o}C (lit.[21] mp 205-206 ^{o}C); ^{1}H NMR ($CDCl_3$) δ 7.17-7.49 (m, 6H), 7.76-7.80 (m, 1H), 8.10-8.14 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 120.4, 123.1, 123.3, 123.7, 125.5, 126.8, 129.7, 132.6, 133.8, 135.0, 137.1, 138.8, 144.2, 162.4, 187.4; IR (CH_2Cl_2) 1718, 1697, 1608 cm^{-1}; HRMS *m/z* 236.03008 (calcd for $C_{15}H_8OS$, 236.02959).

Cesium pivalate. Pivalic acid (3.164 g, 31 mmol) and cesium carbonate (5.00 g, 15.3 mmol) in water (3 mL) were heated at 50 ^{o}C for 10 min with stirring. The resulting solution was heated at 100 ^{o}C under reduced pressure until a white solid was obtained.

The solid cesium pivalate was crushed into a powder then carefully dried by heating at 100 ^{o}C under reduced pressure (200 millitorr.) for 2 h.

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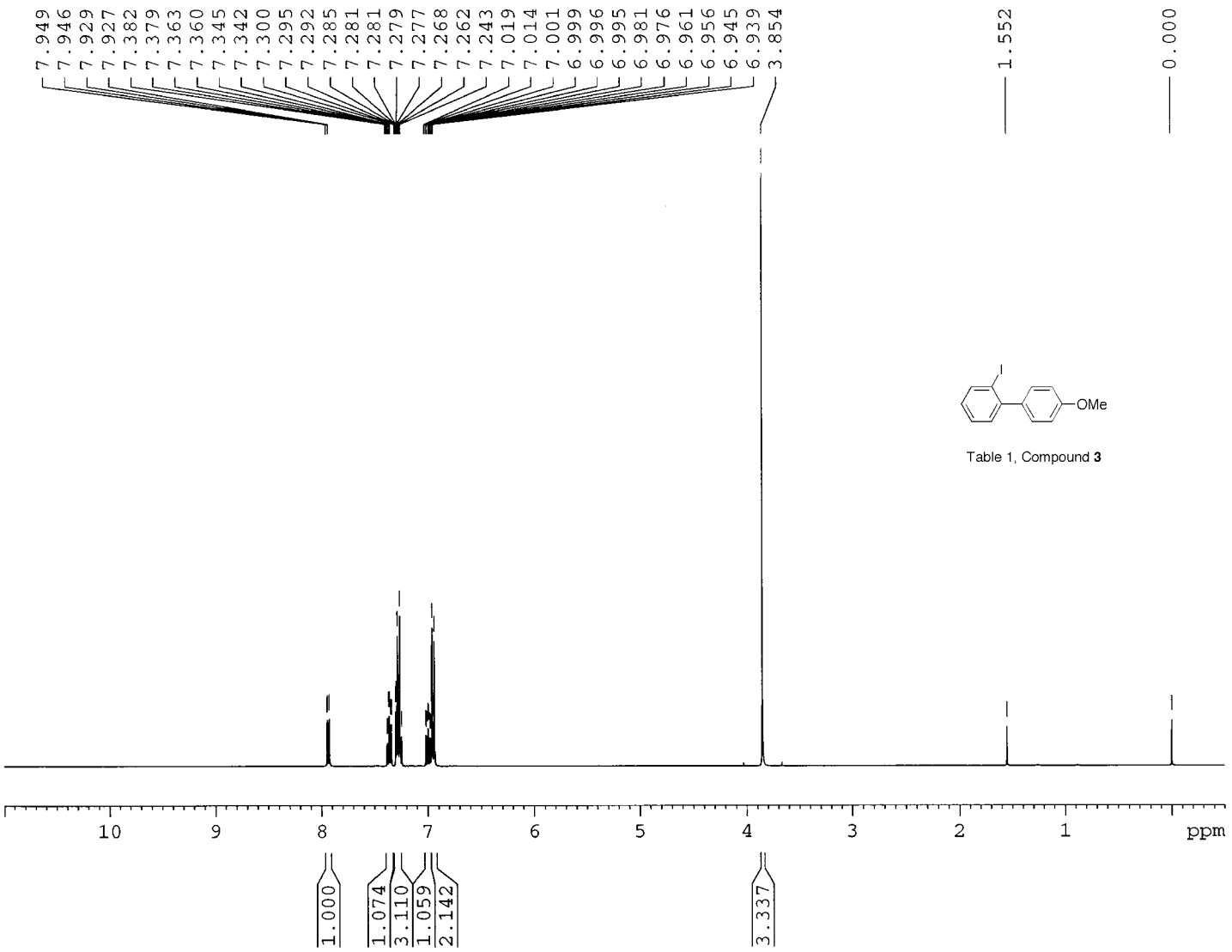
The ^{1}H and ^{13}C spectra for compounds 3, 10, 13, 16, 18, 19, 22, 24 and 29 follow (18 pages).



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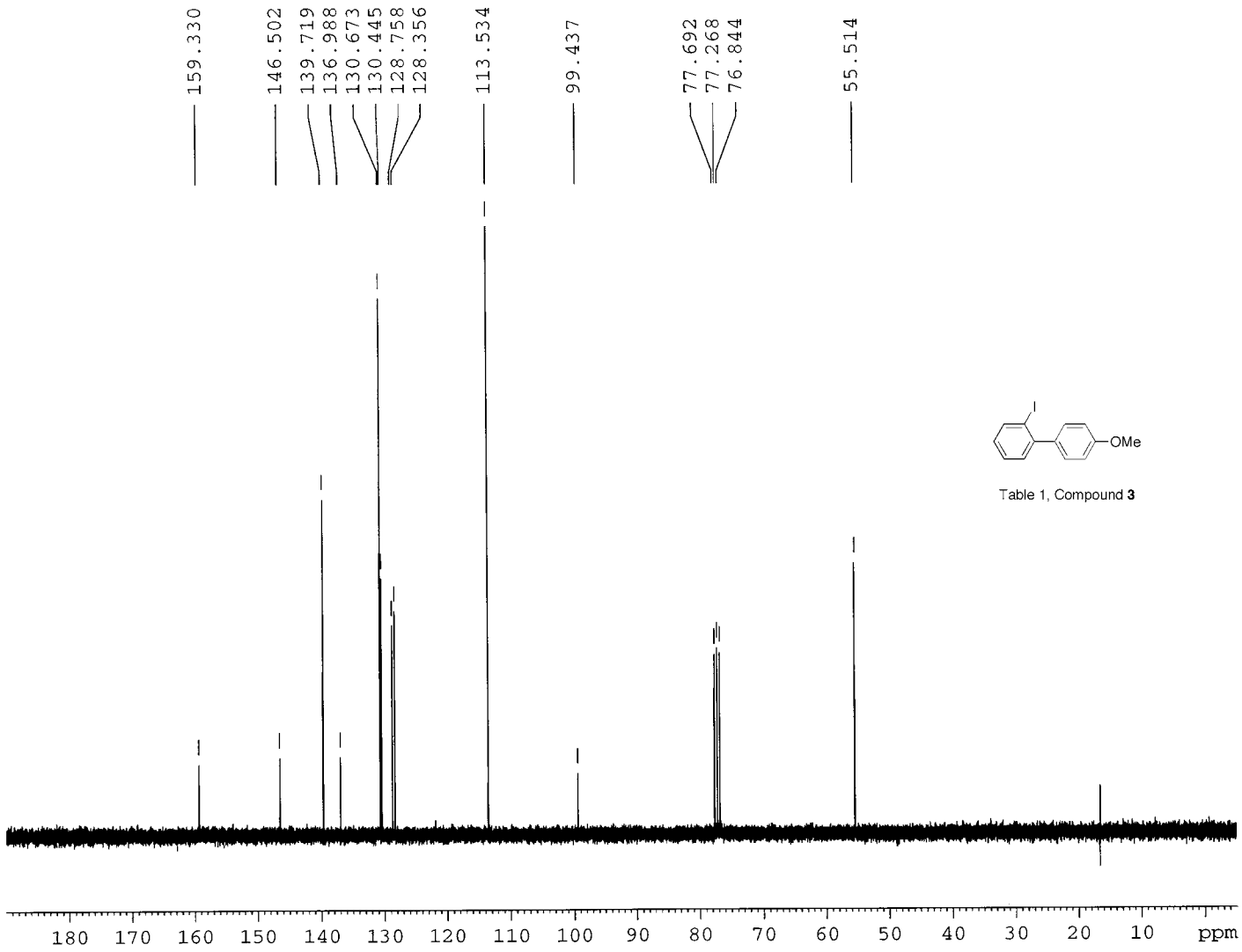

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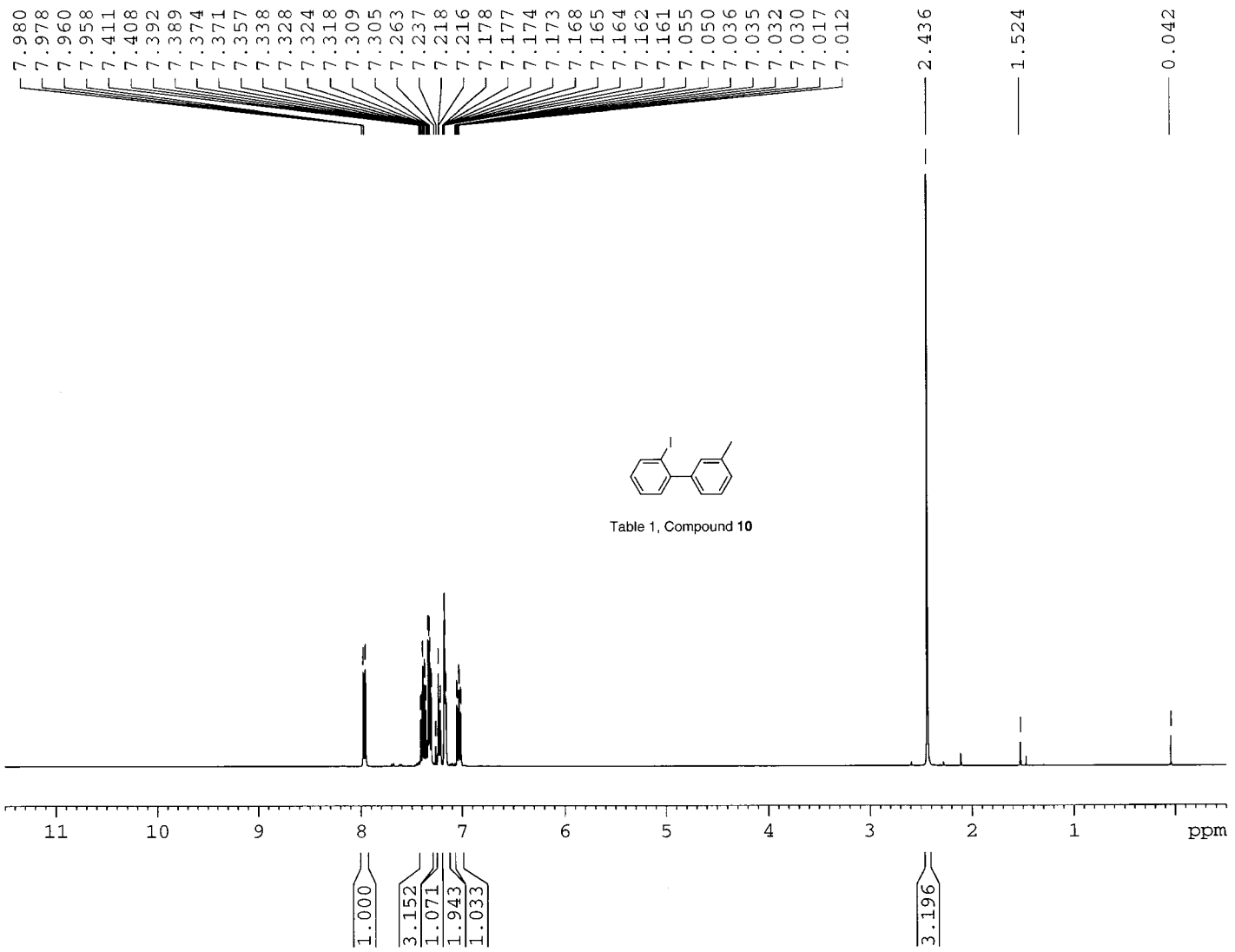

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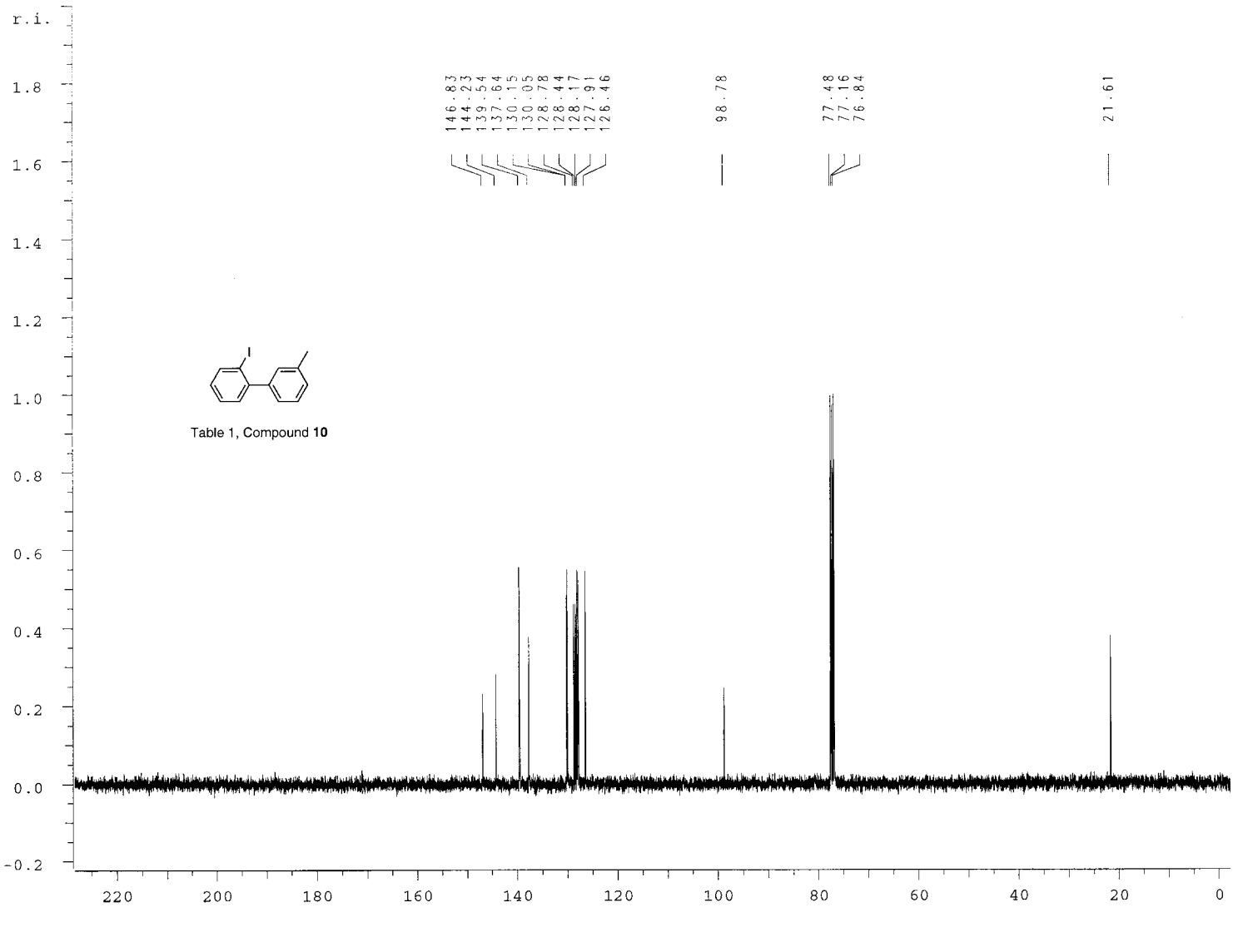

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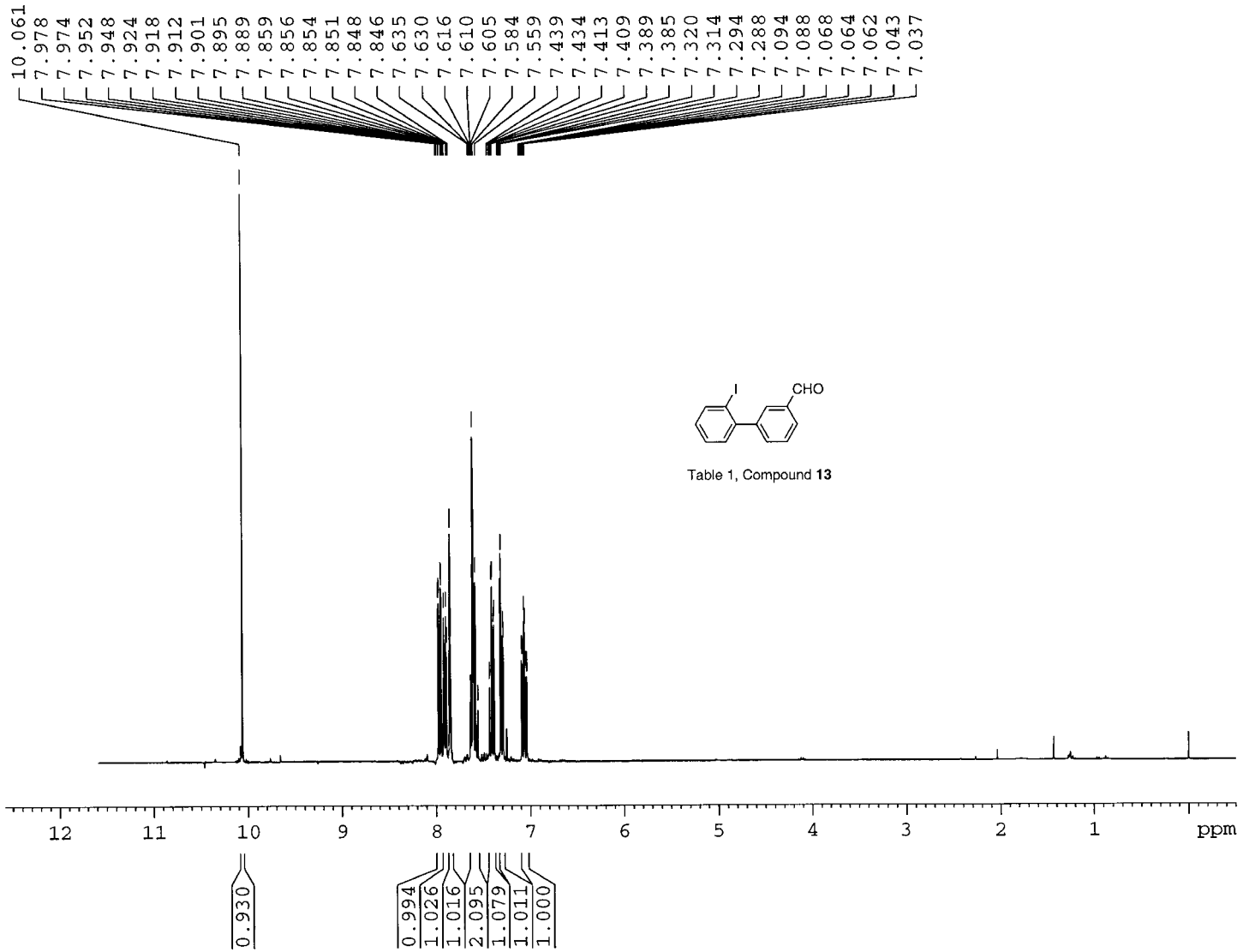

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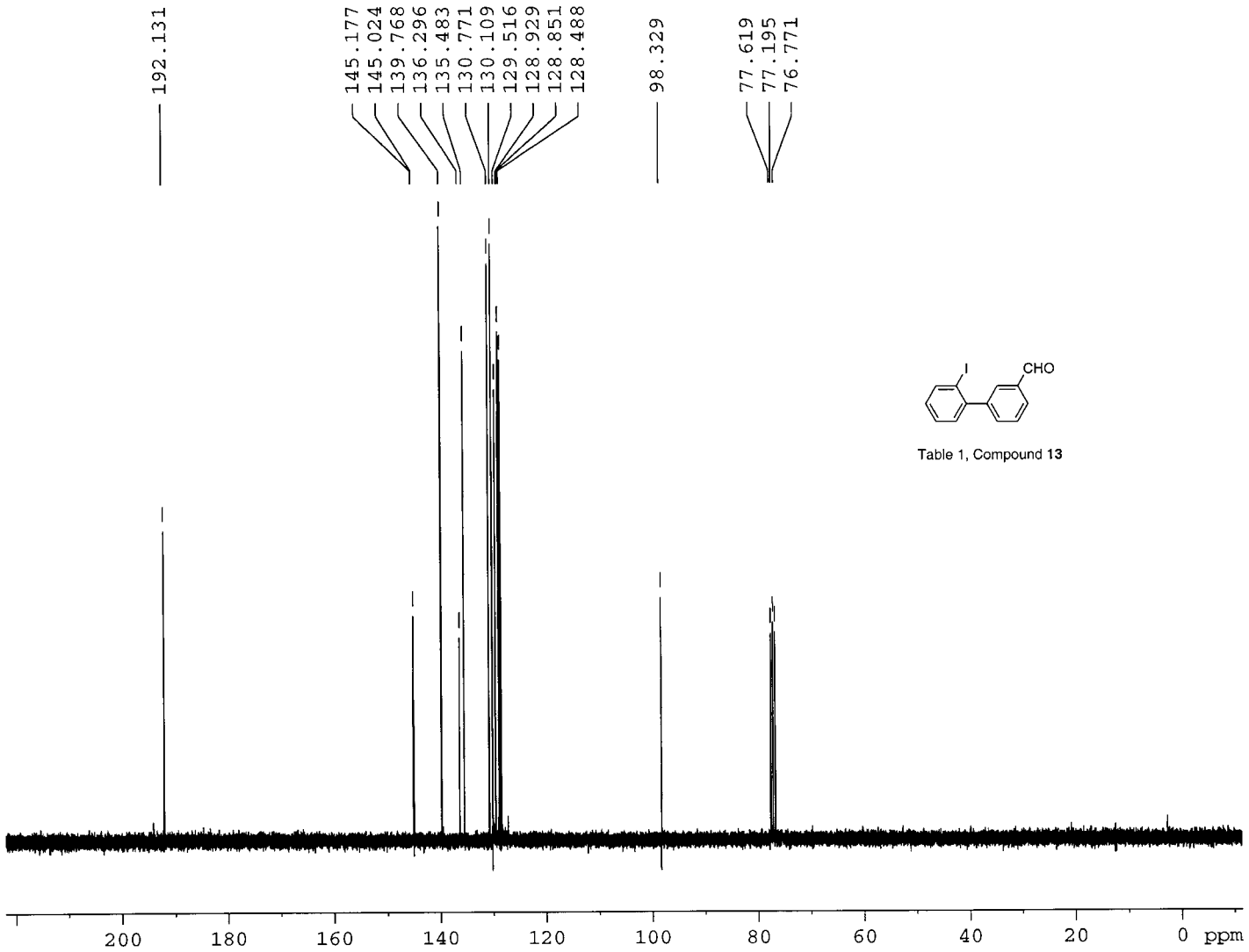

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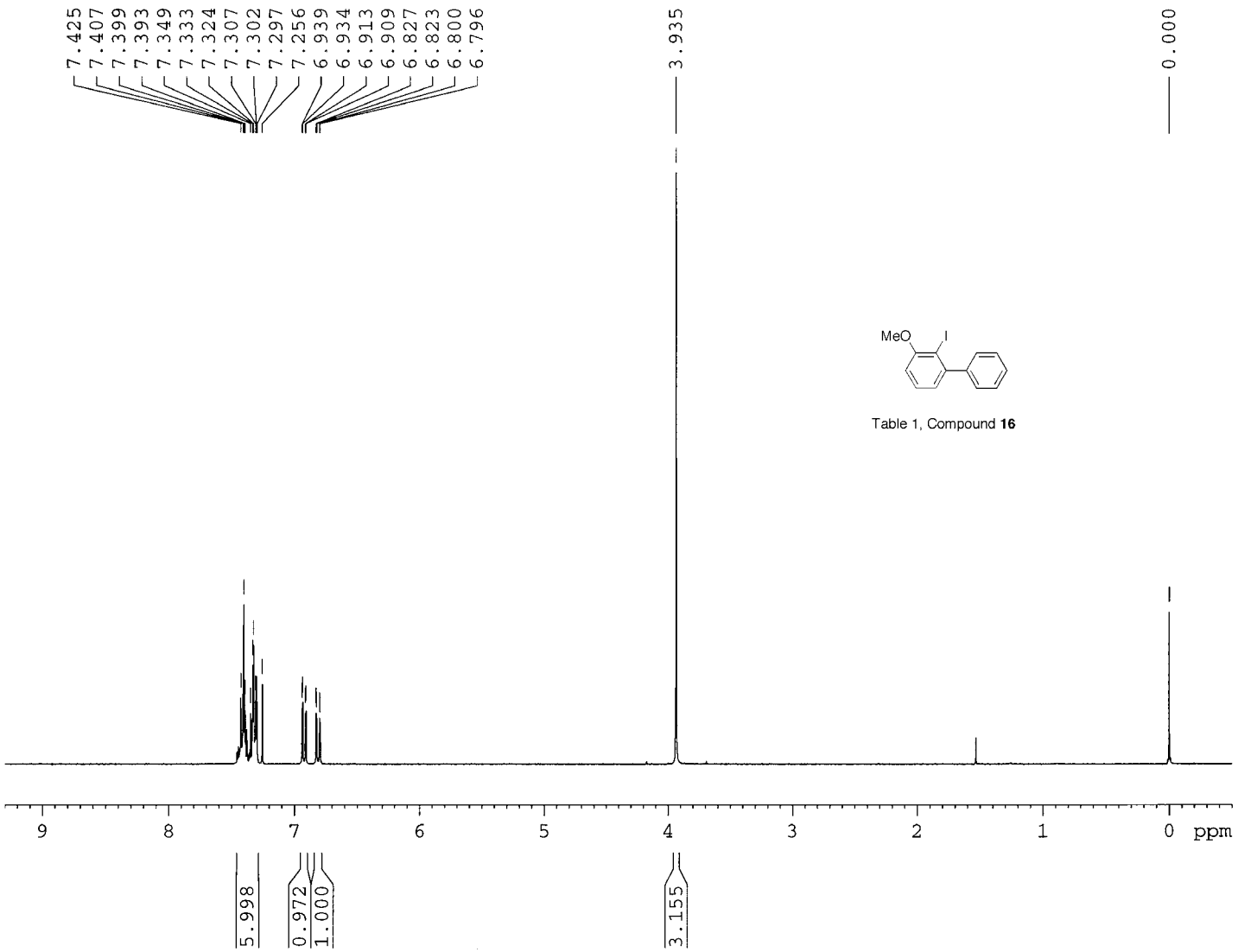

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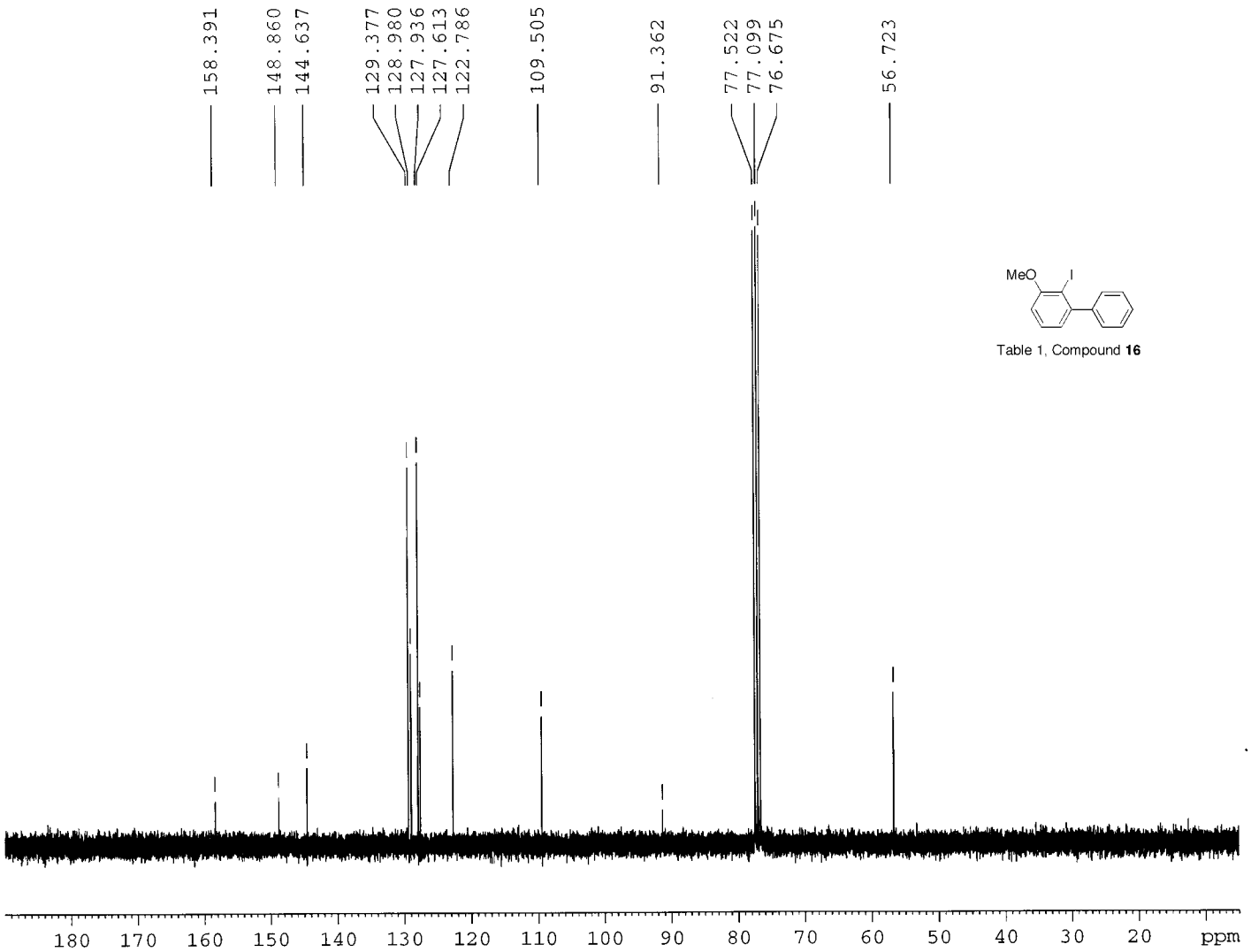

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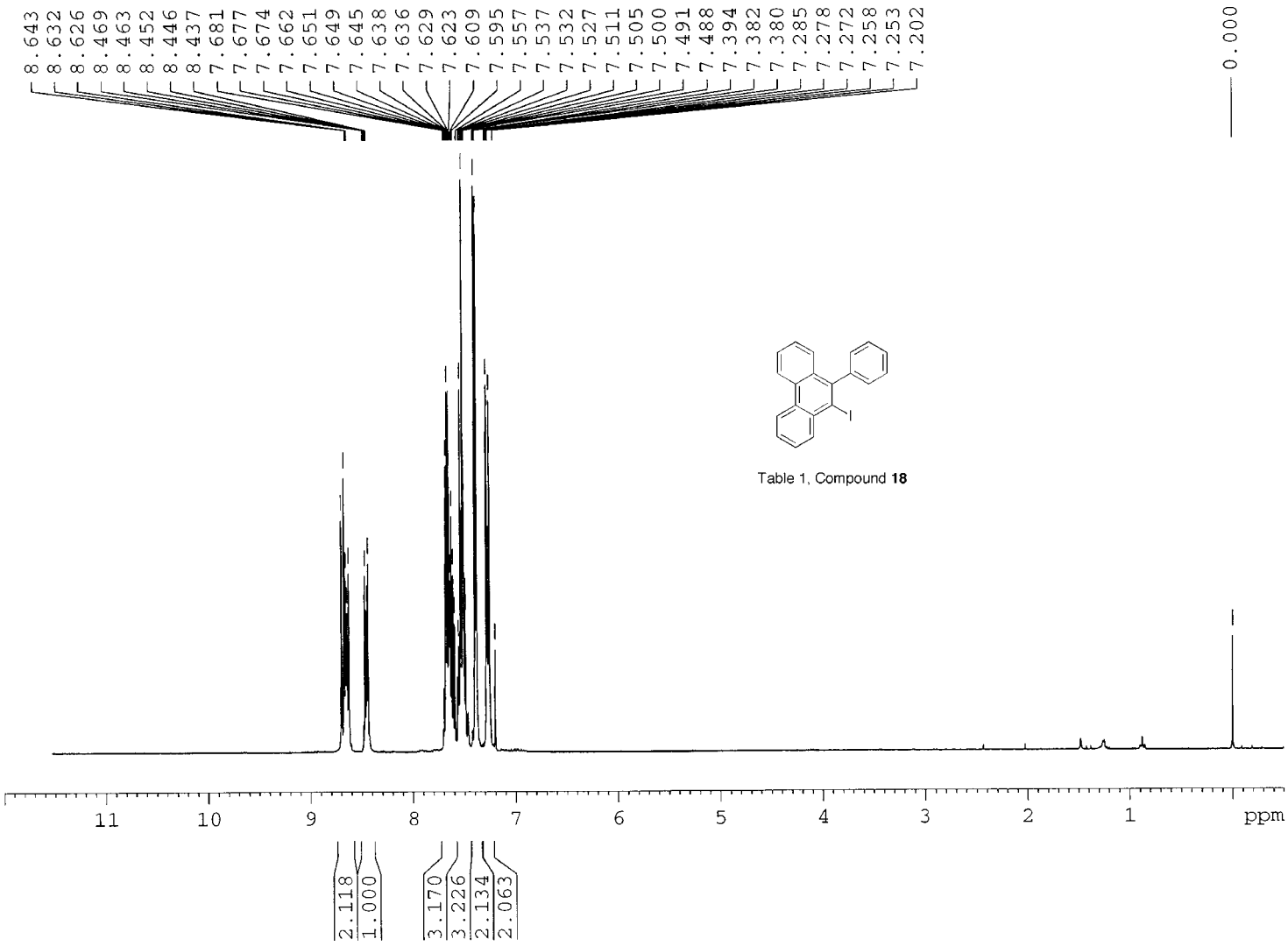

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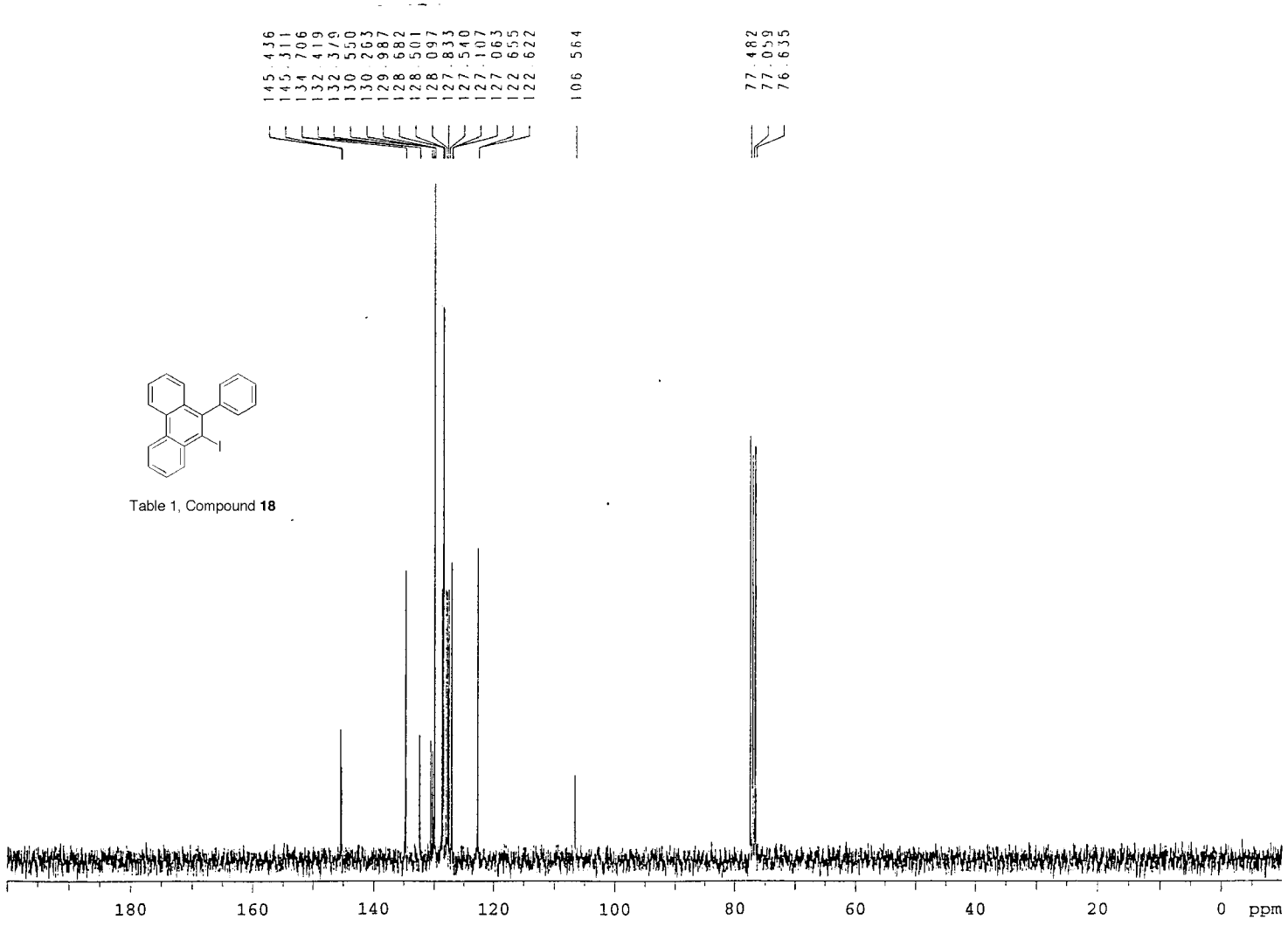

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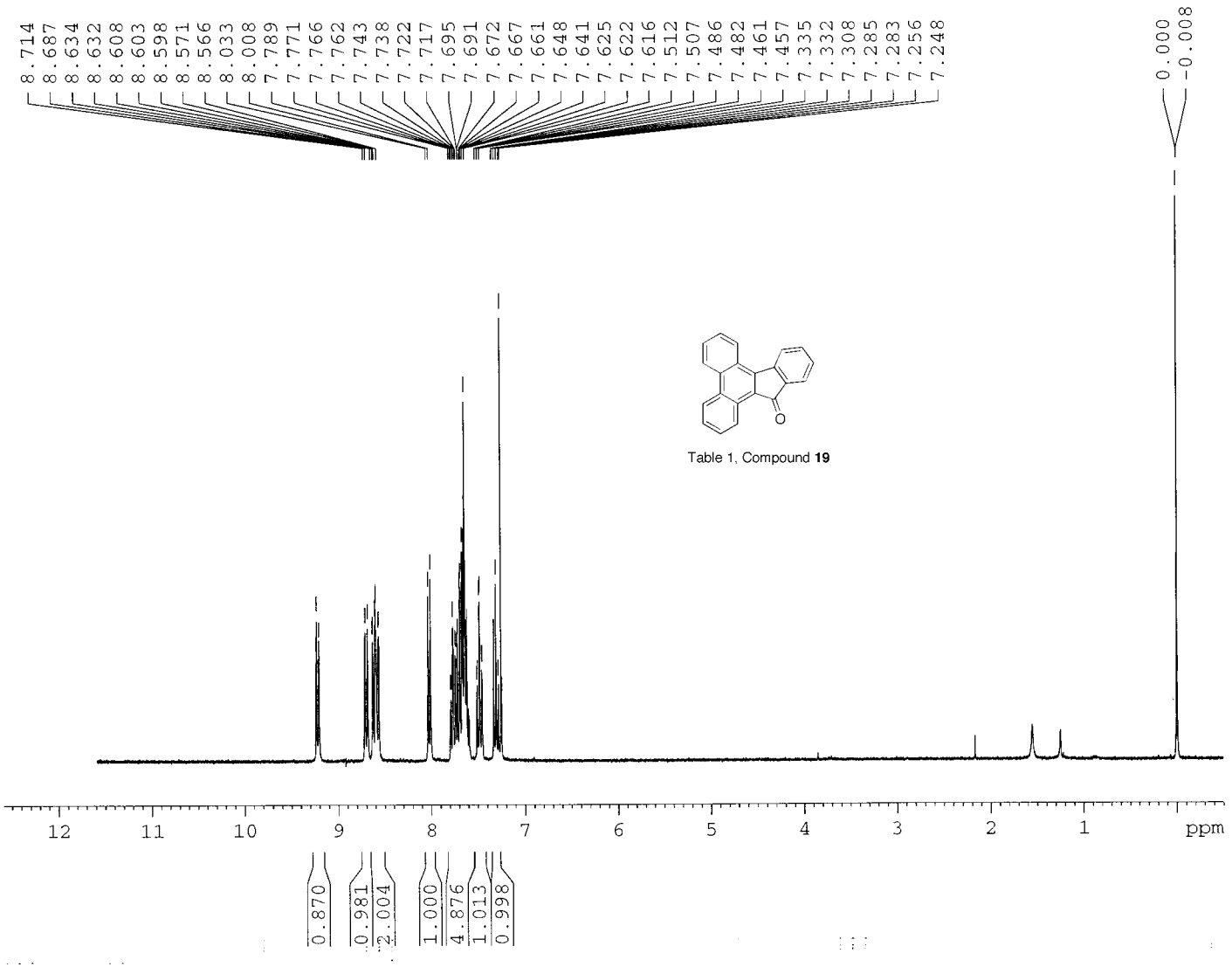

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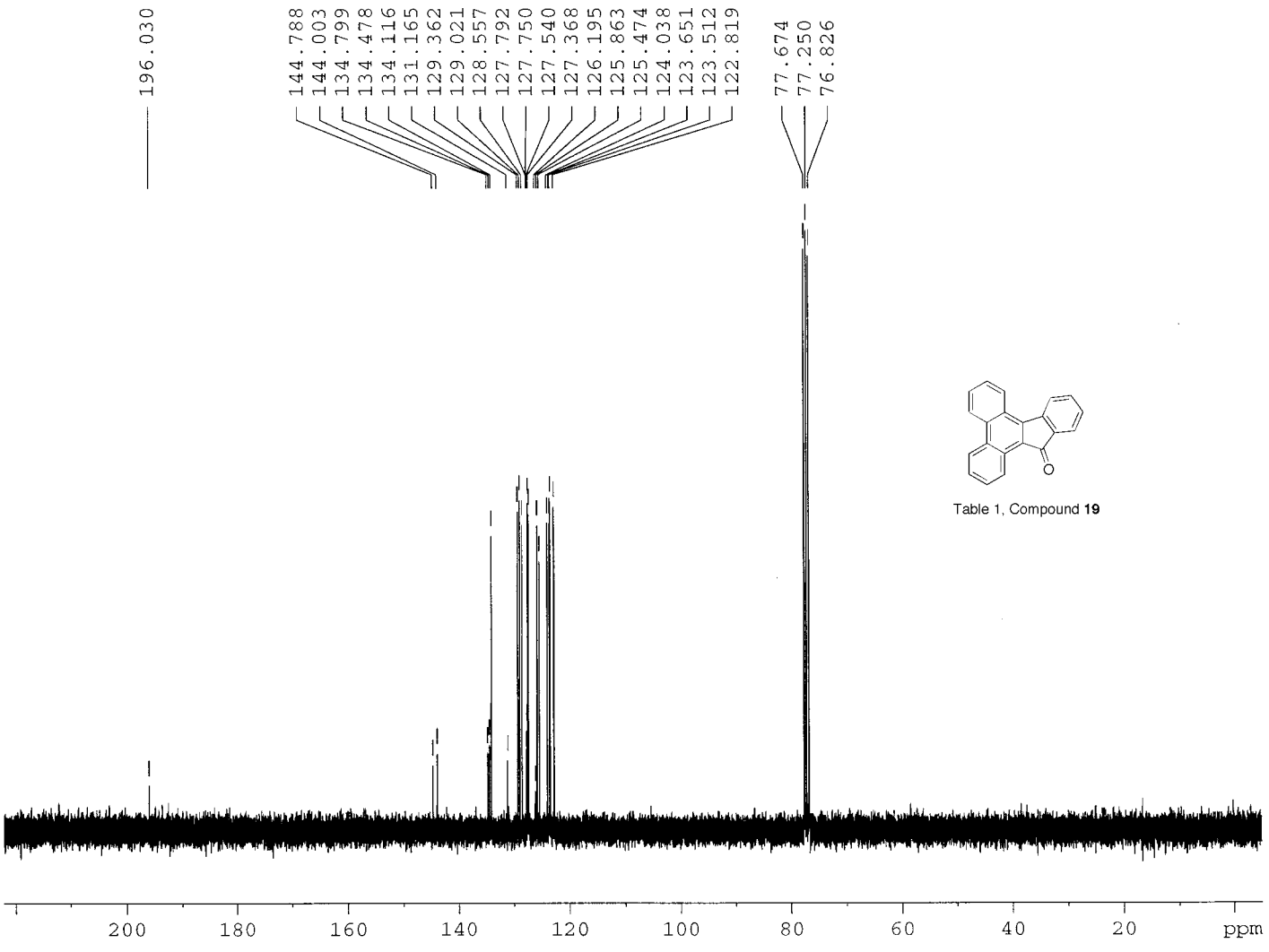

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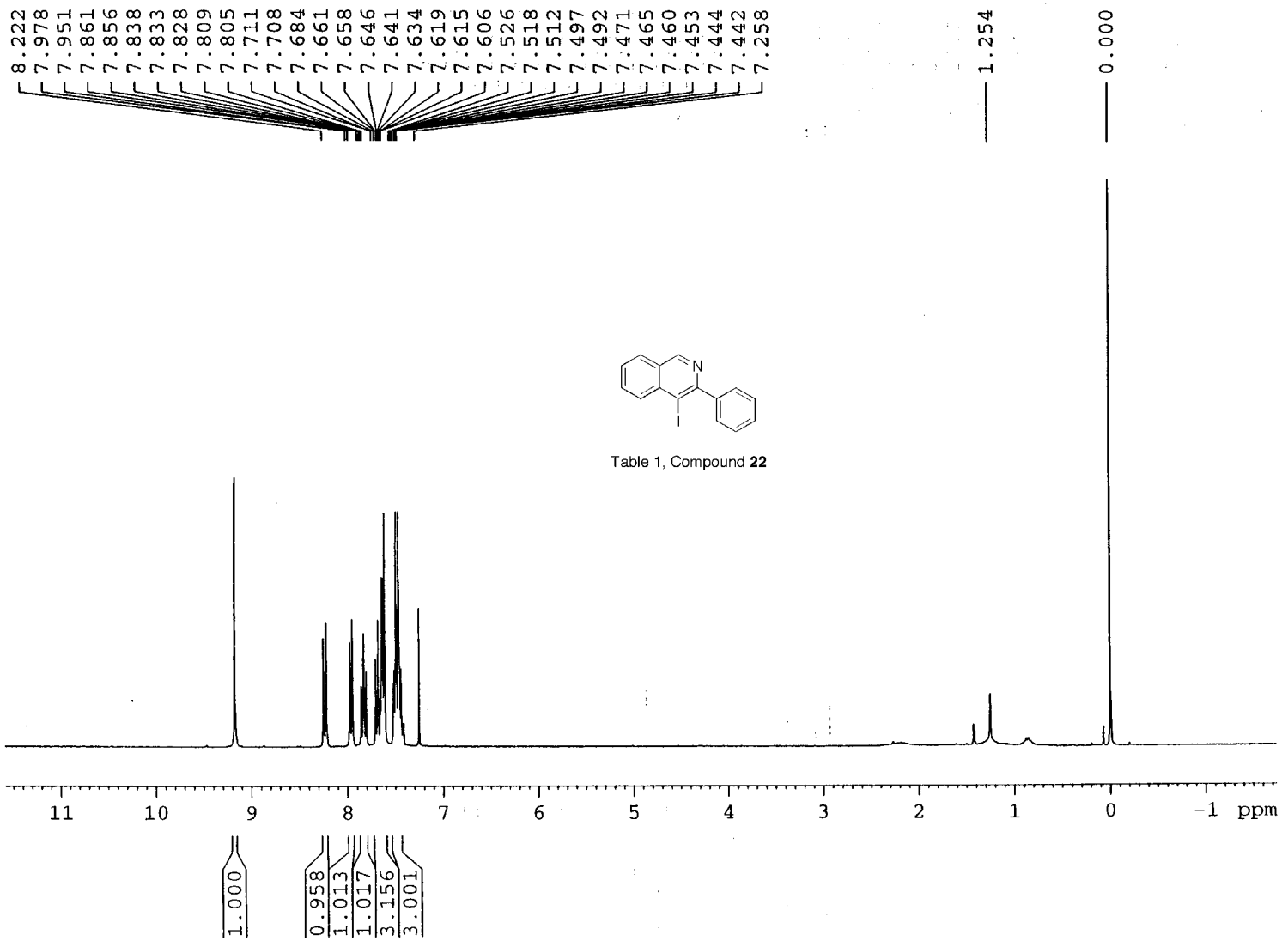

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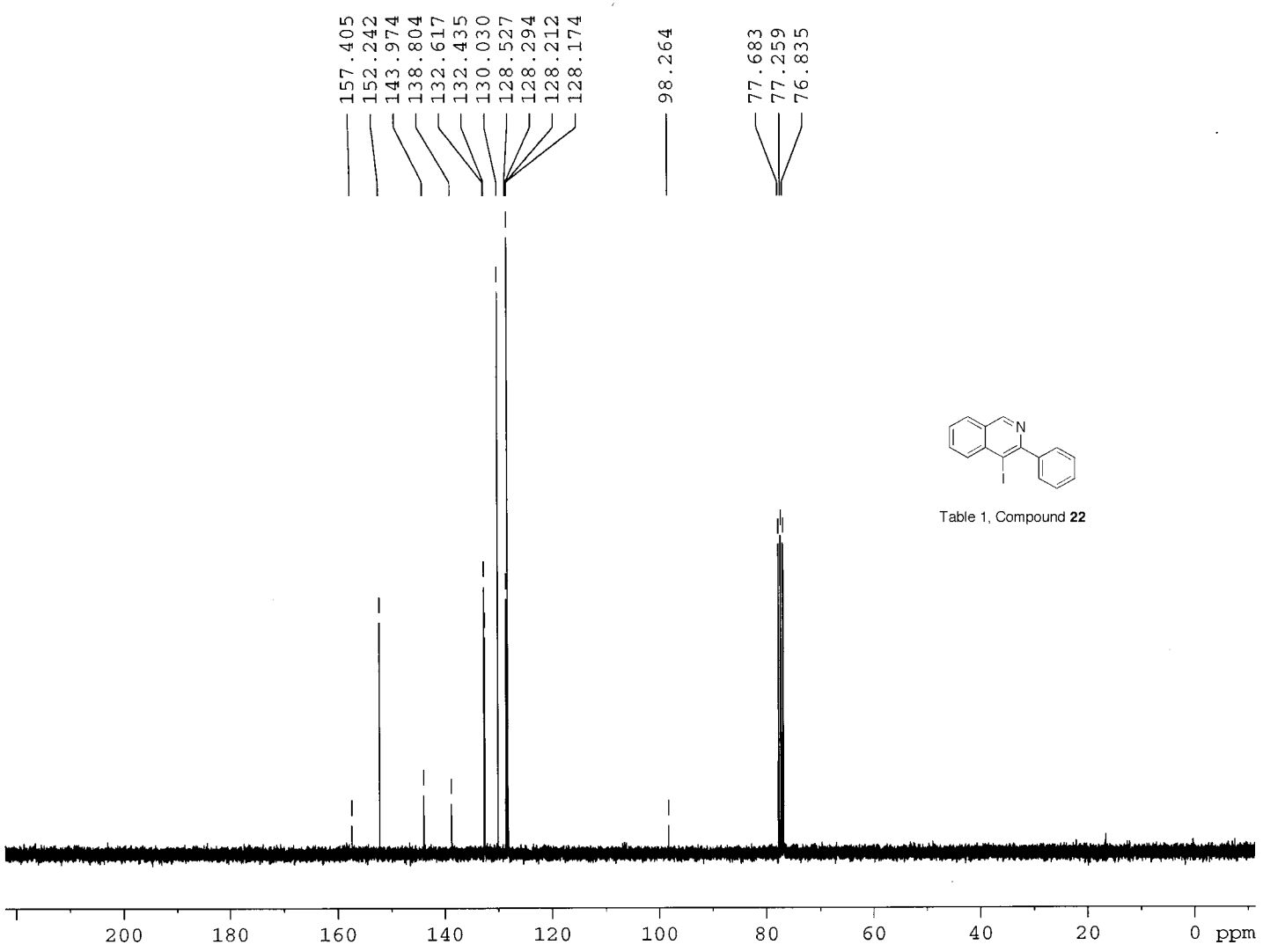

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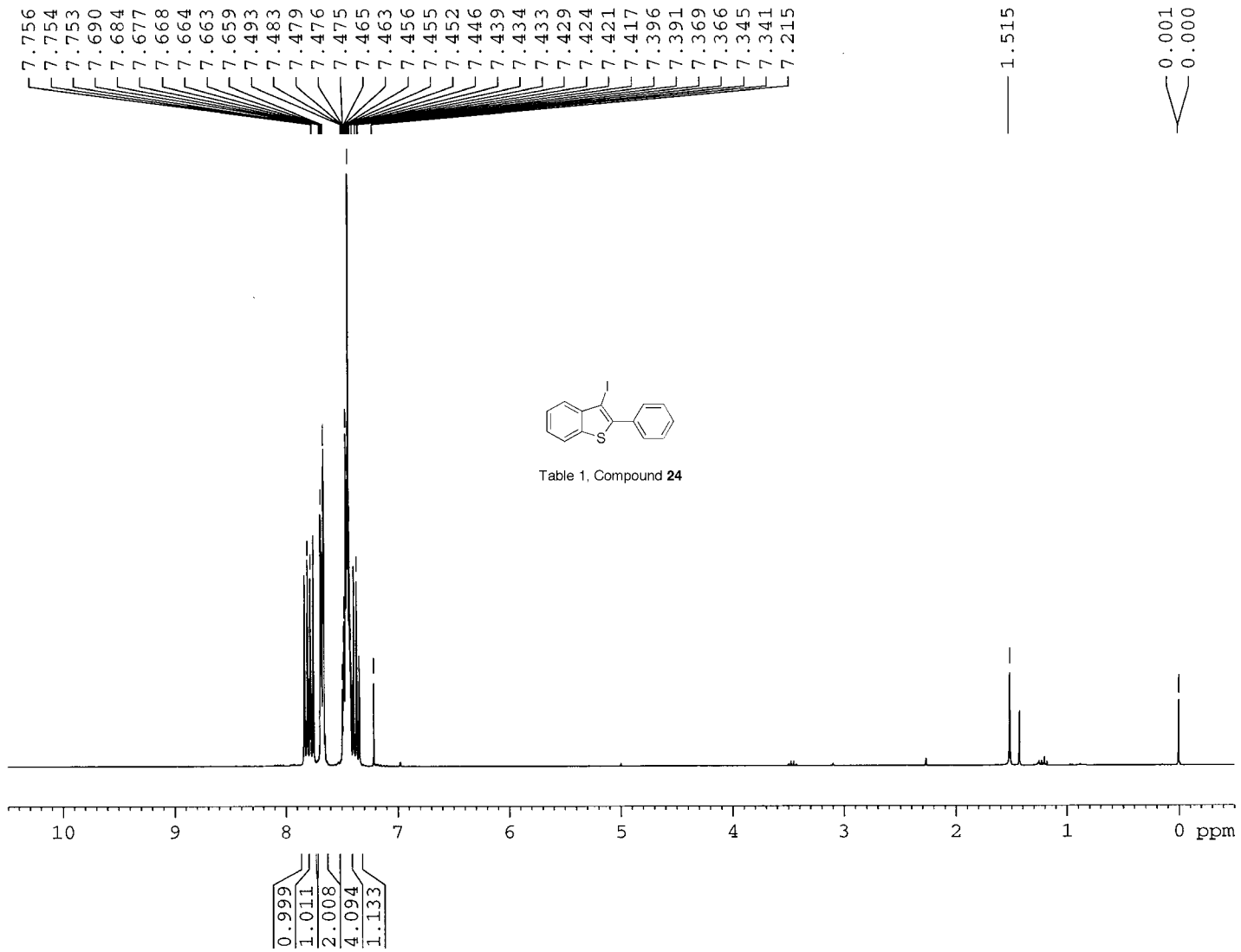

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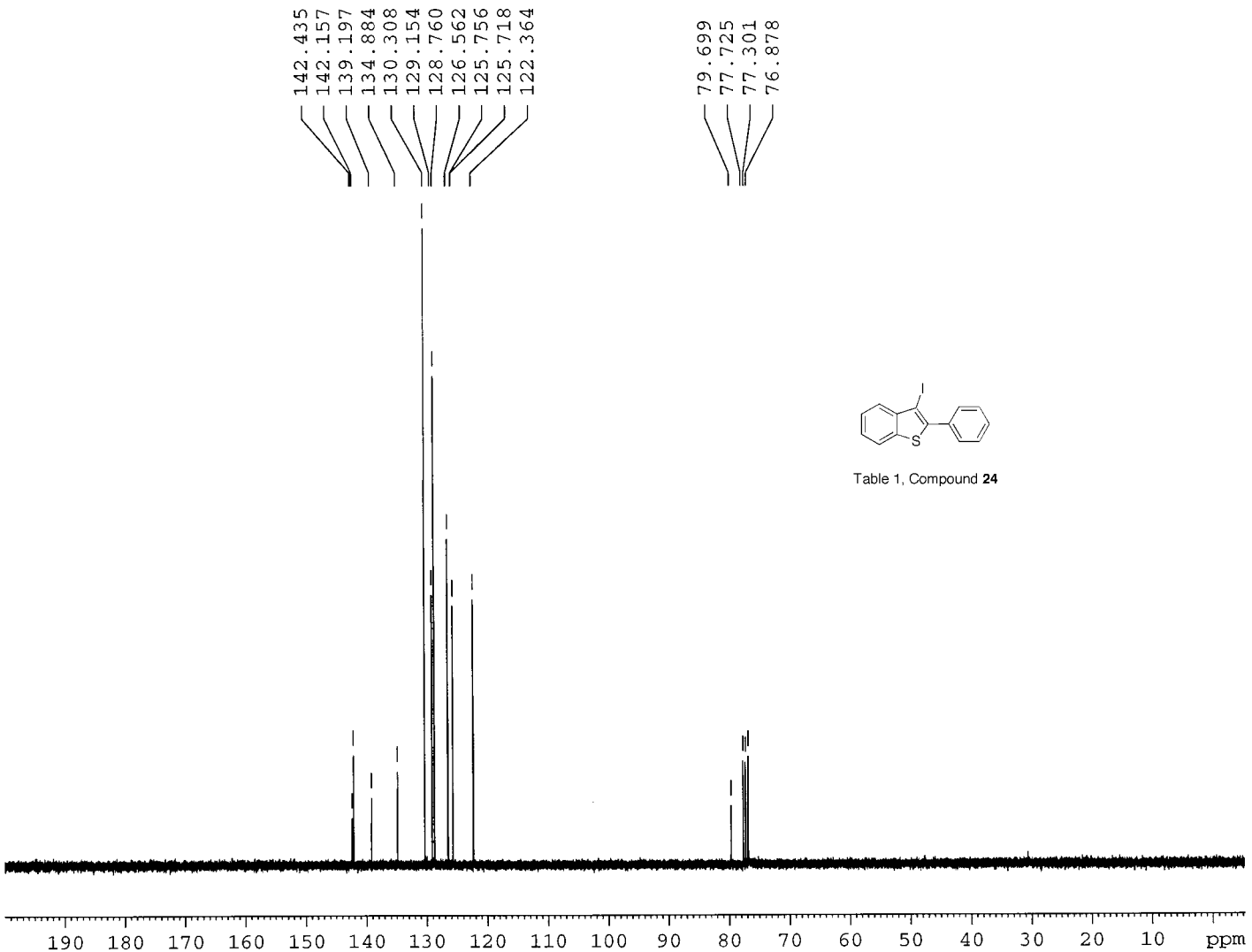

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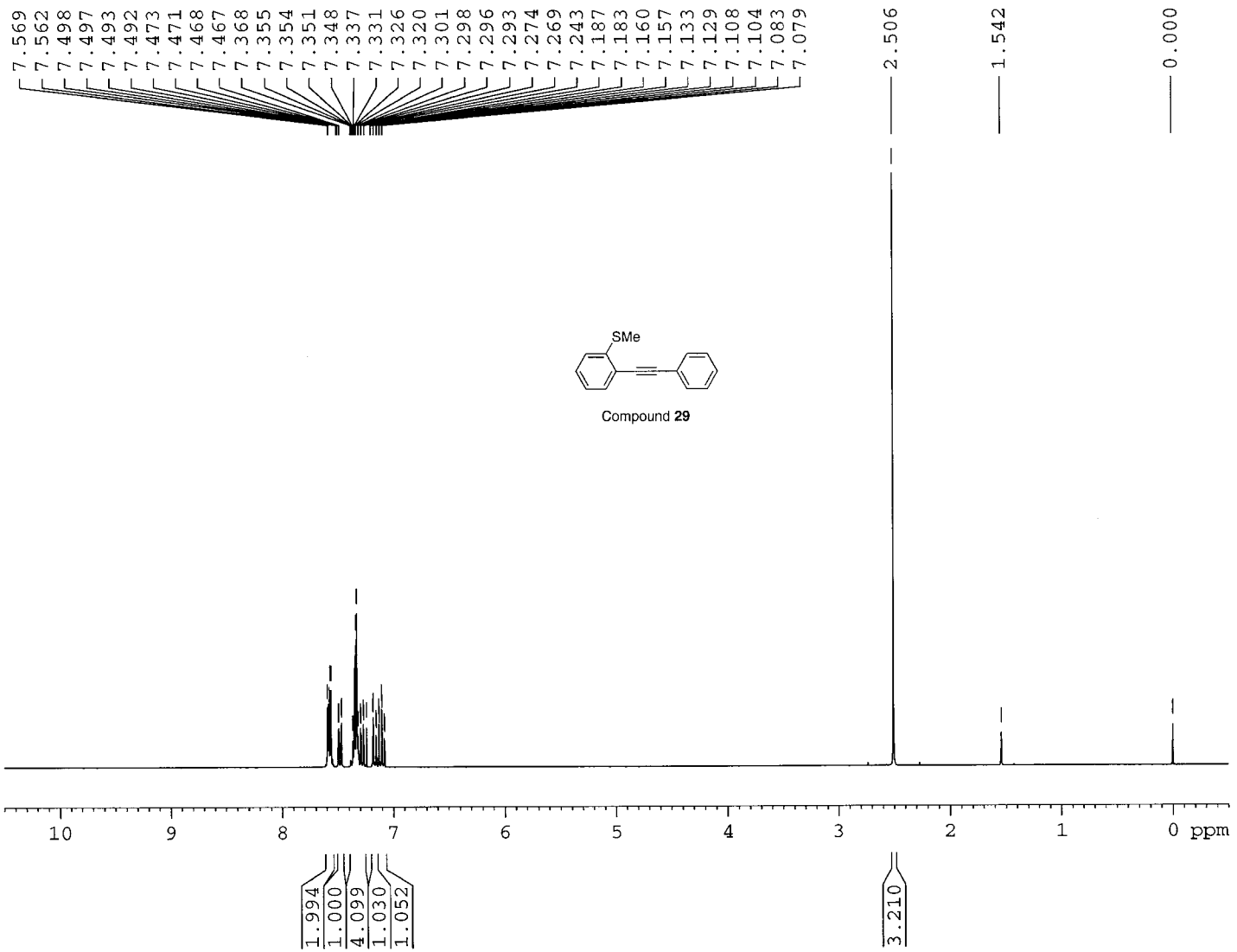
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7.562
7.498
7.497
7.493
7.492
7.473
7.471
7.468
7.467
7.368
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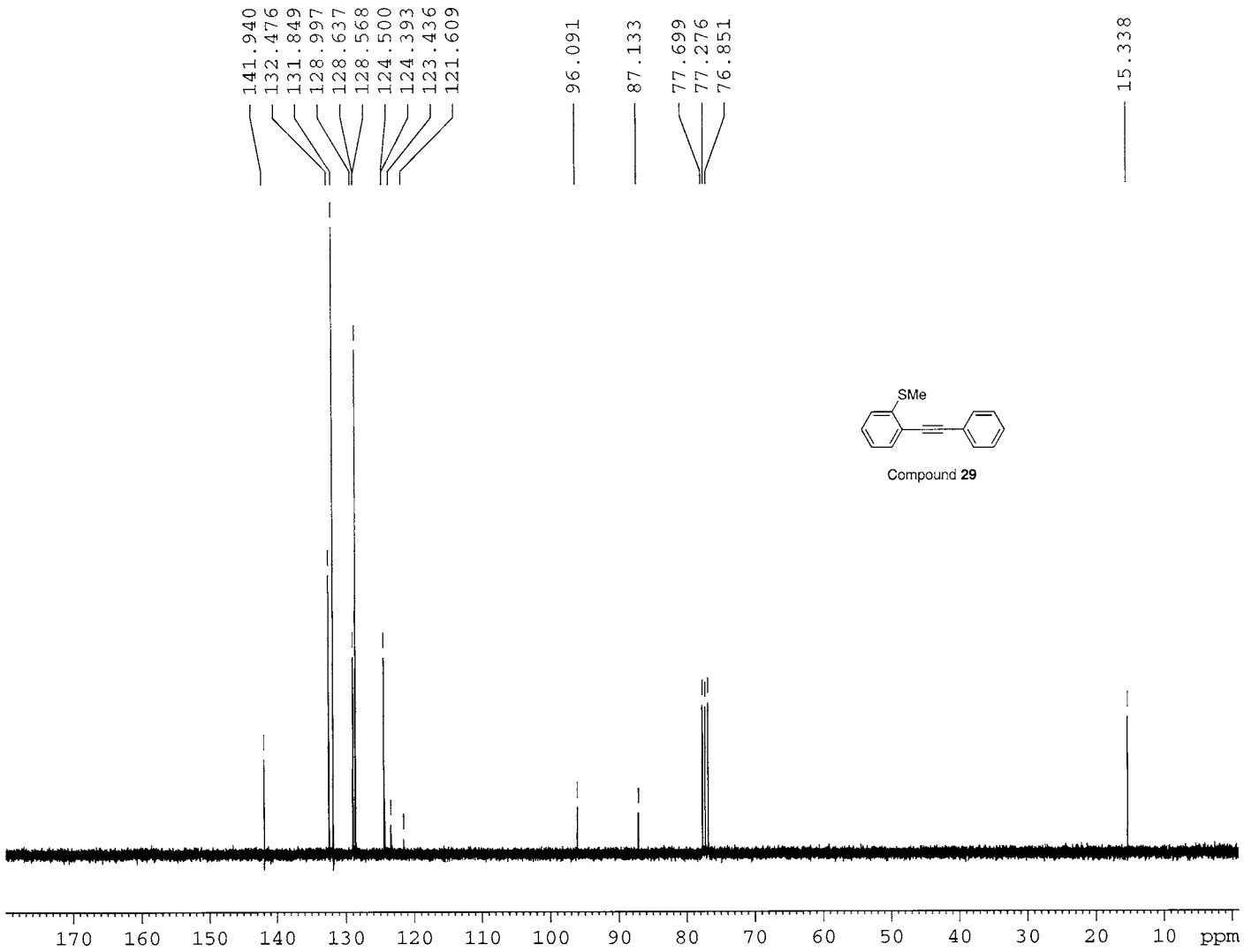
7.354
7.351
7.348
7.337
7.331
7.326
7.320
7.301
7.298
7.296
7.293
7.274
7.269
7.243
7.187
7.183
7.160
7.157
7.133
7.129
7.108
7.104
7.083
7.079
2.506
1.542
0.000
SMe
10
9
8
7
6
5
4
3
2
1
0 ppm
1.994
1.000
4.099
1.030
1.052
3.210

Compound **29**


141.940
132.476
131.849
128.997
128.637
128.568
124.500
124.393
123.436
121.609
96.091
87.133
77.699
77.276
76.851
15.338
SMe
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

Compound **29**