Supporting Information

For

Catalytic Three-Component One-Pot Reaction of Hydrazones, Dihaloarenes and Amines

Maxime Roche, Abdallah Hamze,* Jean-Daniel Brion, and Mouad Alami*

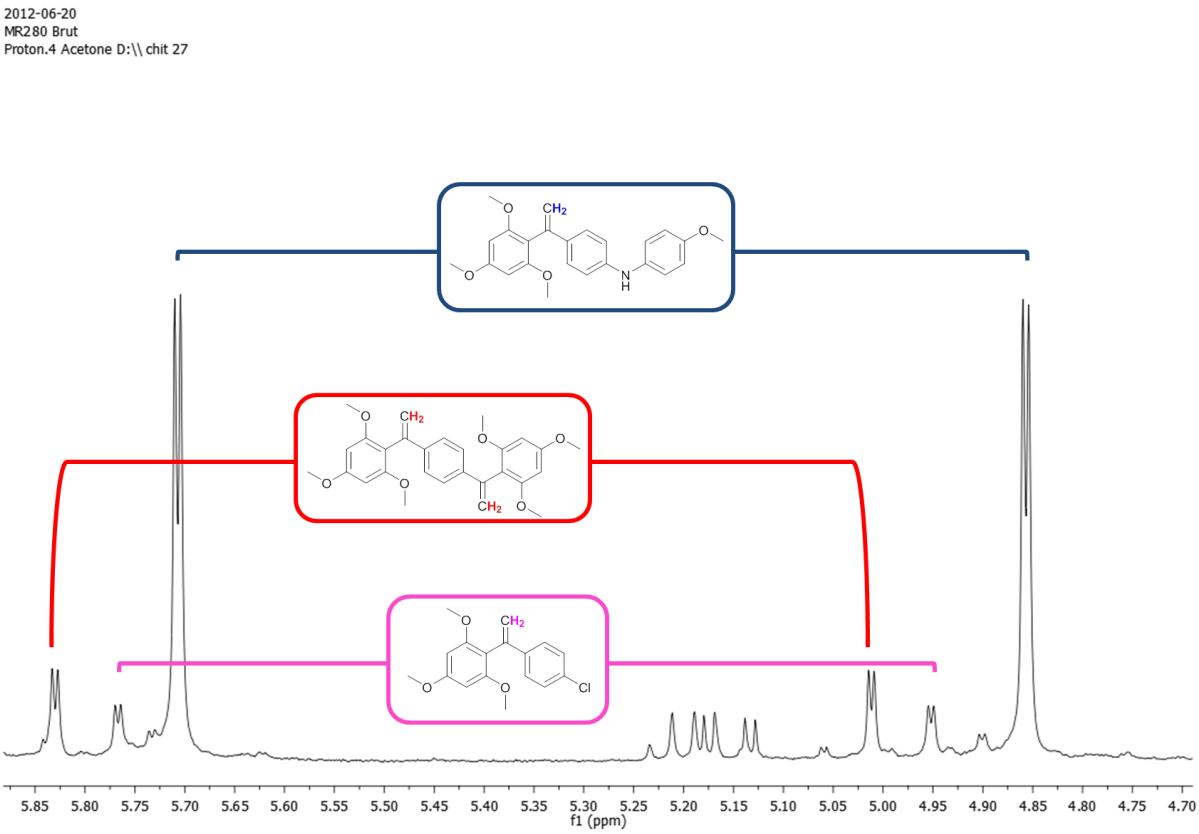
Univ Paris-Sud, CNRS, BioCIS UMR 8076, LabEx LERMIT, Laboratoire de Chimie Thérapeutique, Faculté de Pharmacie, 5 Rue J. B. Clément, Châtenay-Malabry, F-92296, France.

abdallah.hamze@u-psud.fr; mouad.alami@u-psud.fr

List of Contents

I. Monitoring the Reaction

In each experiment, the ratio of the desired molecule **4a** and the byproducts (**5a**, **6a)** formed was determined by ^{1}HNMR in the crude reaction mixture, according to scheme 1. The two olefinc protons of **4a** (blue color) show two doublets at 5.71 ppm (d, *J* = 1.7 Hz) and 4.86 ppm (d, *J* = 1.7 Hz). The dimeric coumpound **6a** (red color) has two doublets at 5.83 ppm (d, *J* = 1.7 Hz) and 5.01 ppm (d, *J* = 1.7 Hz). The undesired product **5a** (pink color) has two doublets at 5.77 ppm (d, *J* = 1.6 Hz) and 4.95 ppm (d, *J* = 1.6 Hz).



Scheme 1

II. General Experimental Methods

Melting points (mp) were recorded on a Büchi B-450 apparatus and were uncorrected. NMR spectra were performed on a Bruker AMX 200 (^{1}H, 200 MHz; ^{13}C, 50 MHz), Bruker AVANCE 300 or Bruker AVANCE 400 (^{1}H, 400 MHz; ^{13}C, 100 MHz). Solvent peaks were used as reference values, with $CDCl_3$ at 7.26 ppm for ^{1}H NMR and 77.16 ppm for ^{13}C NMR, with CD_3COCD_3 at 2.05 ppm for ^{1}H NMR and 29.84 ppm for ^{13}C NMR. Chemical shifts δ are given in ppm, and the following abbreviations are used: singlet (s), doublet (d), doublet of doublet (dd), triplet (t), multiplet (m) and broad singlet (bs). Infrared spectra (IR) were measured on

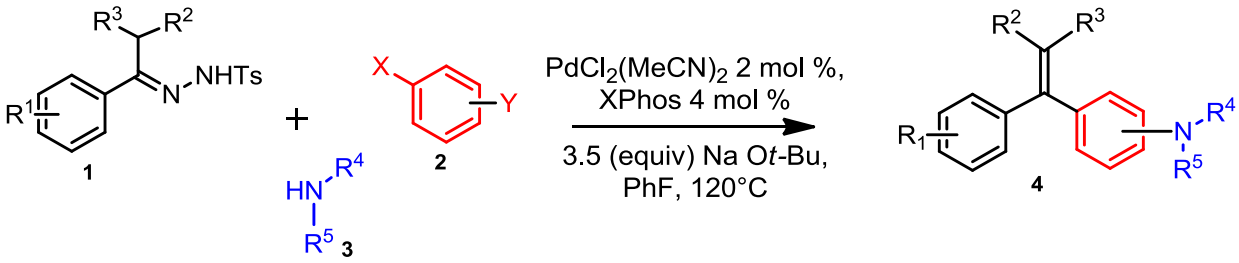
a Bruker Vector 22 spectrophotometer and were recorded neat (neat, cm^{-1}). Low resolution mass spectra (*m*/*z*) were recorded on a Bruker Esquire electrospray ionization apparatus. High resolution mass spectra were recorded on a MicrotofQ Bruker Daltonics. Reaction courses and product mixtures were routinely monitored by TLC on silica gel (precoated F254 Merck plates), and compounds were visualized under a UVP Mineralight UVGL-58 lamp (254 nm) and with phosphomolybdic acid/Δ, anisaldehyde/Δ, or vanillin/Δ. Flash chromatography was performed using silica gel 60 (40–63 mm, 230–400 mesh ASTM) at medium pressure (200 mbar). Fluorobenzene was used as received, dioxane, dichloromethane, cyclohexane and tetrahydrofuran were dried using the procedures described in D. Perrin Purification of Laboratory Chemicals, Pergamon Press Ltd. 1980, 2nd Ed. Organic extracts were, in general, dried over $MgSO_4$ or Na_2SO_4. All products reported showed ^{1}H and ^{13}C NMR spectra in agreement with the assigned structures.

III. General Procedure for Preparation of Hydrazones[1]

To a rapidly stirred suspension of *p*-toluenesulphonohydrazide (5 mmol) in dry methanol (10 mL) at 60 °C, the ketone (5 mmol) was added dropwise. Within 5-60 min the *N*-tosylhydrazone began to precipitate. The mixture was cooled to 0 °C and the product was collected on a Büchner funnel, washed by petroleum ether then was dried *in vacuo* to afford the pure product.

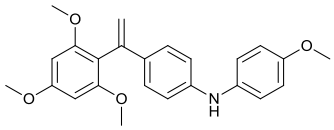
(1) Creary, X.; Tam, W. W.; Albizati, K. F.; Stevens, R. V., *Org. Synth.* **1986**, *64*, 207.

IV. Typical Procedure for Pd-Catalyzed One-Pot 3CR

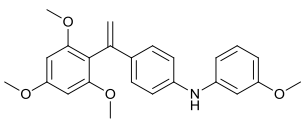


The reactions were carried out in a sealed tube with *N*-tosylhydrazone **1** (1.2 mmol), aryl halides **2** (1 mmol), amine **3** (1.2 mmol), $PdCl_2(MeCN)_2$ (2 mol %), Xphos (4 mol %), NaOtBu (3.5 equiv) in 4.0 mL PhF. The mixture was stirred at 120 °C for 6.0 h. The crude reaction mixture was allowed to cool to room temperature. EtOAc was added to the mixture, which was filtered through *celite*. The solvents were evaporated under reduced pressure and the crude residue was purified by flash chromatography on silica gel.

V. Product Characterization

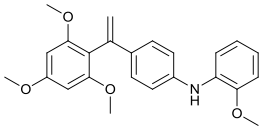


4-Methoxy-*N*-(4-(1-(2,4,6-trimethoxyphenyl)vinyl)phenyl)aniline (4a). White solid (360 mg, 92% yield); mp: 134-136 °C; TLC : R_f = 0.30 (EtOAc/Cyclohexane, 2/8, SiO_2); IR (neat, cm^{-1}) 3476, 3458, 1700, 1602, 1582, 1508, 1466, 1453, 1412, 1334, 1319, 1245, 1224, 1203, 1182, 1159, 1122, 1034, 949; ^{1}H NMR (300 MHz, CD_3COCD_3) δ (ppm) 7.16 – 7.05 (m, 4H), 6.90 – 6.80 (m, 4H), 6.29 (s, 2H), 5.71 (d, *J* = 1.7 Hz, 1H), 4.86 (d, *J* = 1.7 Hz, 1H), 3.84 (s, 3H), 3.75 (s, 3H), 3.66 (s, 6H); ^{13}C NMR (75 MHz, CD_3COCD_3) δ (ppm) 161.7 (C), 159.7 (2C), 155.7 (C), 145.5 (C), 141.8 (C), 137.3 (C), 132.7 (C), 127.5 (2CH), 121.9 (2CH), 115.6 (2CH), 115.4 (2CH), 113.5 (C), 112.8 (CH_2), 91.8 (2CH), 56.1 ($2CH_3$), 55.7 (CH_3), 55.6 (CH_3); HRMS (ESI) $(M + H)^+$: *m/z* calcd for $C_{24}H_{26}NO_4$ 392.1856; found 392.1850.

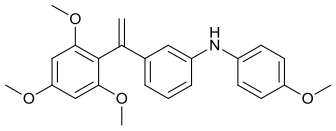


3-Methoxy-*N*-(4-(1-(2,4,6-trimethoxyphenyl)vinyl)phenyl)aniline (4b). Yellow oil (296 mg, 76% yield); TLC : R_f = 0.29 (EtOAc/Cyclohexane, 2/8, SiO_2); IR (neat, cm^{-1}) 3420, 3371, 3320, 3305, 3222, 1609, 1587, 1513, 1454, 1428, 1304, 1245; 1204, 1151 1124, 976; ^{1}H NMR (300 MHz, CD_3COCD_3) δ (ppm) 7.39 (bs, 1H), 7.21 (d, *J* = 8.6 Hz, 2H), 7.13 (t, *J* = 8.3 Hz, 1H), 7.03 (d, *J* = 8.7 Hz, 2H), 6.74 – 6.65 (m, 2H), 6.47 – 6.40 (m, 1H), 6.31 (s, 2H), 5.77 (d, *J* = 1.6 Hz, 1H), 4.93 (d, *J* = 1.6 Hz, 1H), 3.86 (s, 3H), 3.75 (s, 3H), 3.68 (s, 6H); ^{13}C NMR (75 MHz, CD_3COCD_3) δ 161.8 (C), 159.7 (2C), 146.0 (C), 143.4 (C), 141.7 (C), 134. (C),

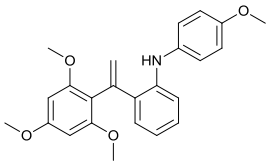
130.7 (CH), 127.5 (2CH), 117.9 (2CH), 113.7 (CH_2), 113.3 (C), 110.4 (CH), 106.3 (CH), 103.5 (CH), 91.8 (2CH), 56.1 ($2CH_3$), 55.6 (CH_3), 55.3 (CH_3); HRMS (ESI) $(M + H)^+$: *m*/*z* calcd for $C_{24}H_{26}NO_4$ 392.1856; found 392.1853.



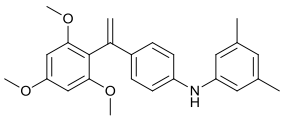
2-Methoxy-*N*-(4-(1-(2,4,6-trimethoxyphenyl)vinyl)phenyl)aniline (4c). White solid (300 mg, 77% yield); mp: 145-147 °C; TLC : R_f = 0.53 (EtOAc/Cyclohexane, 2/8, SiO_2); IR (neat, cm^{-1}) 1596, 1519, 1458, 1413, 1335, 1296, 1244, 1225, 1204, 1160, 1124, 1051, 1029, 949; ^1H NMR (300 MHz, CD_3COCD_3) δ (ppm) 7.31 – 7.25 (m, 1H), 7.20 (d, *J* = 8.7 Hz, 2H), 7.05 (d, *J* = 8.7 Hz, 2H), 6.98 – 6.91 (m, 1H), 6.86 – 6.80 (m, 2H), 6.76 (s, 1H), 6.30 (s, 2H), 5.77 (d, *J* = 1.7 Hz, 1H), 4.92 (d, *J* = 1.7 Hz, 1H), 3.84 (s, 6H), 3.67 (s, 6H); ^{13}C NMR (75 MHz, CD_3COCD_3) δ (ppm) 161.7 (C), 159.64 (2C), 149.8 (C), 143.3 (C), 141.7 (C), 134.2 (C), 133.7 (C), 127.5 (2CH), 121.4 (CH), 120.9 (CH), 118.0 (2CH), 116.1 (CH), 113.6 (CH_2), 113.2 (C), 111.8 (CH), 91.8 (2CH), 56.1 ($2CH_3$), 56.0 (CH_3), 55.6 (CH_3); HRMS (ESI) $(M + H)^+$: *m*/*z* calcd for $C_{24}H_{26}NO_4$ 392.1856; found 392.1848.



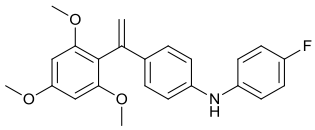
***N*-(4-Methoxyphenyl)-3-(1-(2,4,6-trimethoxyphenyl)vinyl)aniline (4d).** Pale pink oil (320 mg, 82% yield); TLC : R_f = 0.40 (EtOAc/Cyclohexane, 2/8, SiO_2); IR (neat, cm^{-1}) 1601, 1581, 1507, 1413, 1334, 1243, 1225, 1203, 1158, 1125, 1035, 949; ^1H NMR (300 MHz, CD_3COCD_3) δ (ppm) 7.11 – 6.95 (m, 4H), 6.93 – 6.90 (m, 1H), 6.87 – 6.76 (m, 4H), 6.27 (s, 2H), 5.79 (d, *J* = 1.8 Hz, 1H), 5.01 (d, *J* = 1.8 Hz, 1H), 3.84 (s, 3H), 3.74 (s, 3H), 3.67 (s, 6H); ^{13}C NMR (75 MHz, CD_3COCD_3) δ (ppm) 161.8 (C), 159.7 (2C), 155.4 (C), 145.9 (C), 143.1 (C), 142.6 (C), 137.7 (C), 129.4 (CH), 121.6 (2CH), 117.6 (CH), 115.9 (CH_2), 115.3 (2CH), 115.1 (CH), 114.6 (CH), 113.2 (C), 91.8 (2CH), 56.1 ($2CH_3$), 55.7 (CH_3), 55.6 (CH_3); HRMS (ESI) $(M + H)^+$: *m*/*z* calcd for $C_{24}H_{26}NO_4$ 392.1856; found 392.1850.



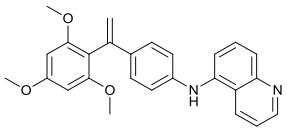
***N*-(4-Methoxyphenyl)-2-(1-(2,4,6-trimethoxyphenyl)vinyl)aniline (4e).** Colorless oil (286 mg, 73% yield); TLC : R_f = 0.51 (EtOAc/Cyclohexane, 2/8, SiO_2); IR (neat, cm^{-1}) 1701, 1604, 1580, 1508, 1451, 1412, 1334, 1292, 1245, 1224, 1203, 1160, 1122, 1034; 949, 917; ^1H NMR (300 MHz, CD_3COCD_3) δ (ppm) 7.26 – 7.20 (m, 1H), 7.05 – 6.95 (m, 4H), 6.87 – 6.80 (m, 2H), 6.75 – 6.67 (m, 1H), 6.54 (bs, 1H), 6.24 (s, 2H), 5.48 (d, *J* = 2.5 Hz, 1H), 5.30 (d, *J* = 2.5 Hz, 1H), 3.78 (s, 3H), 3.75 (s, 6H), 3.74 (s, 3H); ^{13}C NMR (75 MHz, CD_3COCD_3) δ (ppm) 161.8 (C), 159.1 (2C), 155.7 (C), 142.9 (C), 141.3 (C), 137.9 (C), 132.8 (C), 131.2 (CH), 128.0 (CH), 122.0 (CH_2), 121.9 (2CH), 119.3 (CH), 115.4 (2CH), 115.2 (CH), 113.6 (C), 92.0 (2CH), 56.3 ($2CH_3$) 55.7 (CH_3), 55.6 (CH_3); HRMS (ESI) $(M + Na)^+$: *m*/*z* calcd for $C_{24}H_{25}NNaO_4$ 414.1676; found 414.1661.



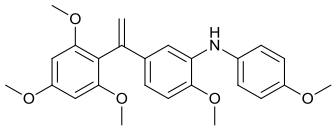
3,5-Dimethyl-*N*-(4-(1-(2,4,6-trimethoxyphenyl)vinyl)phenyl)aniline (4f). Pink solid (276 mg, 71% yield); mp: 135-137 °C; TLC : R_f = 0.39 (EtOAc/Cyclohexane, 2/8, SiO_2); IR (neat, cm^{-1}) 1605, 1583, 1512, 1454, 1414, 1317, 1225, 1204, 1160, 1124, 950; ^1H NMR (300 MHz, CD_3COCD_3) δ (ppm) 7.14 (d, *J* = 8.7 Hz, 2H), 7.05 (m, 2H), 6.82 (d, *J* = 8.7 Hz, 2H), 6.71 (d, *J* = 7.5 Hz, 1H), 6.63 (s, 1H), 6.29 (s, 2H), 5.73 (d, *J* = 1.7 Hz, 1H), 4.88 (d, *J* = 1.7 Hz, 1H), 3.84 (s, 3H), 3.67 (s, 6H), 2.23 (s, 3H), 2.18 (s, 3H); ^{13}C NMR (75 MHz, CD_3COCD_3) δ (ppm) 161.7 (C), 159.7 (2C), 145.1 (C), 142.3 (C), 141.8 (2C), 136.7 (C), 133.2 (C), 131.6 (CH), 127.5 (2CH), 127.0 (C), 123.6 (CH), 121.2 (CH), 116.9 (2CH), 113.1 (CH_2), 91.8 (2CH), 56.1 ($2CH_3$), 55.6 (CH_3), 21.2 ($2CH_3$); HRMS (ESI) $(M + Na)^+$: *m/z* calcd for $C_{25}H_{27}NNaO_3$ 412.1883; found 412.1873.



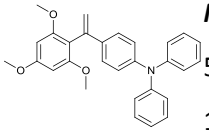
4-Fluoro-*N*-(4-(1-(2,4,6-trimethoxyphenyl)vinyl)phenyl)aniline (4g). White solid (254 mg, 67% yield); mp: 131-133 °C; TLC : R_f = 0.44 (EtOAc/Cyclohexane, 2/8, SiO_2); IR (neat, cm^{-1}) 1602, 1583, 1506, 1467, 1413, 1320, 1226, 1204, 1160, 1124, 949; ^1H NMR (300 MHz, CD_3COCD_3) δ (ppm) 7.32 (bs, 1H), 7.21 – 7.08 (m, 4H), 7.07 – 6.89 (m, 4H), 6.29 (s, 2H), 5.74 (d, *J* = 1.7 Hz, 1H), 4.90 (d, *J* = 1.7 Hz, 1H), 3.84 (s, 3H), 3.67 (s, 6H); ^{13}C NMR (75 MHz, CD_3COCD_3) δ (ppm) 161.8 (C), 159.7 (2C), 158.1 (d, *J* = 236 Hz, C), 144.2 (C), 141.8 (C), 140.9 (d, *J* = 2 Hz, C), 133.9 (C), 127.6 (2CH), 120.3 (d, *J* = 8 Hz, 2CH), 116.8 (2CH), 116.4 (d, *J* = 23 Hz, 2CH), 113.5 (CH_2), 113.3 (C), 91.8 (2CH), 56.1 ($2CH_3$), 55.6 (CH_3); ^{19}F NMR (188 MHz, CD_3COCD_3) δ (ppm) 54.2; HRMS (ESI) $(M + H)^+$: *m/z* calcd for $C_{23}H_{23}FNO_3$ 380.1656; found 380.1647.



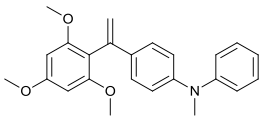
***N*-(4-(1-(2,4,6-Trimethoxyphenyl)vinyl)phenyl)quinolin-5-amine (4h).** Yellow oil (268 mg, 65% yield); TLC : R_f = 0.60 (MeOH/ CH_2Cl_2, 2/98, SiO_2); IR (neat, cm^{-1}) 1771, 1606, 1586, 1572, 1512, 1467, 1410, 1325, 1248, 1224, 1203, 1160, 1122, 1035, 949; ^1H NMR (300 MHz, CD_3COCD_3) δ (ppm) 8.87 (dd, *J* = 4.1, 1.7 Hz, 1H), 8.57 (m, 1H), 7.72 – 7.56 (m, 3H), 7.41 (m, 2H), 7.21 (d, *J* = 8.8 Hz, 2H), 7.00 (d, *J* = 8.8 Hz, 2H), 6.30 (s, 2H), 5.77 (d, *J* = 1.7 Hz, 1H), 4.93 (d, *J* = 1.7 Hz, 1H), 3.84 (s, 3H), 3.68 (s, 6H); ^{13}C NMR (75 MHz, CD_3COCD_3) δ (ppm) 161.8 (C), 159.7 (2C), 151.2 (CH), 150.6 (C), 144.6 (C), 141.8 (C), 141.0 (C), 134.6 (C), 131.5 (CH), 130.3 (CH), 127.6 (2CH), 123.7 (CH), 123.2 (C), 121.0 (CH), 118.2 (2CH), 114.9 (CH), 113.8 (CH_2), 113.3 (C), 91.8 (2CH), 56.1 ($2CH_3$), 55.6 (CH_3); HRMS (ESI) $(M + H)^+$: *m/z* calcd for $C_{26}H_{25}N_2O_3$ 413.1860; found 413.1849.



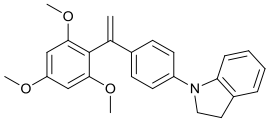
2-Methoxy-*N*-(4-methoxyphenyl)-5-(1-(2,4,6-trimethoxyphenyl)vinyl)-aniline (4i). Colorless oil (295 mg, 70% yield); TLC : R_f = 0.13 (EtOAc/Cyclohexane, 2/8, SiO_2); IR (neat, cm^{-1}) 1605, 1583, 1512, 1494, 1465, 1413, 1334, 1226, 1203, 1181, 1159, 1127, 1034; ^1H NMR (300 MHz, CD_3COCD_3) δ (ppm) 6.92 – 6.71 (m, 7H), 6.68 (bs, 1H), 6.24 (s, 2H), 6.02 (d, *J* = 2.7 Hz, 1H), 5.21 (d, *J* = 2.7 Hz, 1H), 3.81 (s, 3H), 3.72 (s, 3H), 3.71 (s, 3H), 3.66 (s, 6H); ^{13}C NMR (75 MHz, CD_3COCD_3) δ (ppm) 161.5 (C), 159.6 (2C), 154.4 (C), 153.1 (C), 139.5 (C), 139.0 (C), 138.8 (C), 132.6 (C), 120.8 (CH_2), 120.7 (CH), 118.9 (2CH), 117.8 (CH), 115.3 (2CH), 115.1 (CH), 106.2 (C), 91.9 (2CH), 57.0 (CH_3), 56.2 ($2CH_3$), 55.8 (CH_3), 55.6 (CH_3); HRMS (ESI) $(M + H)^+$: *m*/*z* calcd for $C_{25}H_{28}NO_5$ 422.1962; found 422.1963.



***N*,*N*-Diphenyl-4-(1-(2,4,6-trimethoxyphenyl)vinyl)aniline (4j).** Colorless oil (240 mg, 55% yield); TLC : R_f = 0.56 (EtOAc/Cyclohexane, 2/8, SiO_2); IR (neat, cm^{-1}) 1584, 1490, 1413, 1333, 1274, 1224, 1204, 1159, 1124, 950; ^1H NMR (300 MHz, CD_3COCD_3) δ (ppm) 7.33 – 7.17 (m, 6H), 7.03 (m, 6H), 6.90 (d, *J* = 8.7 Hz, 2H), 6.29 (s, 2H), 5.80 (d, *J* = 1.6 Hz, 1H), 4.99 (d, *J* = 1.6 Hz, 1H), 3.83 (s, 3H), 3.68 (s, 6H); ^{13}C NMR (75 MHz, CD_3COCD_3) δ (ppm) 161.8 (C), 159.6 (2C), 148.7 (2C), 147.6 (C), 141.6 (C), 136.6 (C), 130.2 (4CH), 127.6 (2CH), 124.9 (4CH), 124.1 (2CH), 123.7 (2CH), 115.1 (CH_2), 112.9 (C), 91.8 (2CH), 56.1 ($2CH_3$), 55.6 (CH_3); HRMS (ESI) $(M + H)^+$: *m*/*z* calcd for $C_{29}H_{28}NO_3$ 438.2064; found 438.2046.

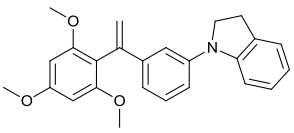


***N*-Methyl-*N*-phenyl-4-(1-(2,4,6-trimethoxyphenyl)vinyl)aniline (4k).** Yellow solid (270 mg, 72% yield); mp: 126-128 °C; TLC : R_f = 0.55 (EtOAc/Cyclohexane, 2/8, SiO_2); IR (neat, cm^{-1}) 3480, 3359, 1589, 1582, 1494, 1467, 1453, 1412, 1334, 1224, 1203, 1160, 1123, 950; ^1H NMR (300 MHz, CD_3COCD_3) δ (ppm) 7.32 – 7.18 (m, 4H), 7.07 – 7.00 (m, 2H), 6.98 – 6.87 (m, 3H), 6.31 (s, 2H), 5.79 (d, *J* = 1.7 Hz, 1H), 4.95 (d, *J* = 1.7 Hz, 1H), 3.85 (s, 3H), 3.69 (s, 6H), 3.30 (s, 3H); ^{13}C NMR (75 MHz, CD_3COCD_3) δ (ppm) 161.8 (C), 159.7 (2C), 150.0 (C), 149.0 (C), 141.7 (C), 134.9 (C), 130.0 (2CH), 127.5 (2CH), 122.1 (CH), 121.4 (2CH), 120.4 (2CH), 114.2 (CH_2), 113.2 (C), 91.8 (2CH), 56.1 ($2CH_3$), 55.6 (CH_3), 40.5 (CH_3); HRMS (ESI) $(M + H)^+$: *m*/*z* calcd for $C_{24}H_{26}NO_3$ 376.1907; found 376.1911.

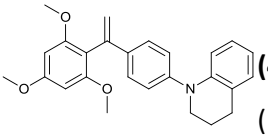


1-(4-(1-(2,4,6-Trimethoxyphenyl)vinyl)phenyl)indoline (4l). White solid (330 mg, 85% yield); mp: 109-110 °C; TLC : R_f = 0.51 (EtOAc/Cyclohexane, 2/8, SiO_2); IR (neat, cm^{-1}) 1597, 1581, 1514, 1484, 1455, 1413, 1385, 1334, 1224, 1203, 1159, 1122, 949; ^1H NMR (300 MHz, CD_3COCD_3) δ (ppm) 7.32 – 7.24 (m, 2H), 7.17 – 7.08 (m, 4H), 7.06 – 6.99 (m, 1H), 6.69 (td, *J* = 7.3, 1.0 Hz, 1H), 6.31 (s, 2H), 5.80 (d, *J* = 1.6 Hz, 1H), 4.95 (d, *J* = 1.6 Hz, 1H), 3.93 (t, *J* = 8.4 Hz, 2H), 3.85 (s, 3H), 3.68 (s, 6H), 3.09 (t, *J* = 8.4 Hz, 2H); ^{13}C NMR (75 MHz,

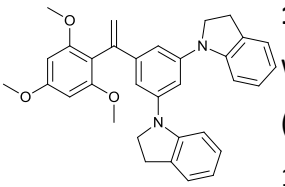
CD_3COCD_3) δ (ppm) 161.8 (C), 159.7 (2C), 144.0 (C), 141.7 (C), 134.5 (C), 132.3 (C), 130.1 (C), 127.8 (CH), 127.4 (2CH), 125.8 (CH), 119.7 (CH), 117.6 (2CH), 114.1 (CH_2), 113.2 (C), 108.9 (CH), 91.8 (2CH), 56.1 ($2CH_3$), 55.7 (CH_3), 52.6 (CH_2), 28.6 (CH_2); HRMS (ESI) $(M + H)^+$: *m/z* calcd for $C_{25}H_{26}NO_3$ 388.1907; found 388.1905.



1-(3-(1-(2,4,6-Trimethoxyphenyl)vinyl)phenyl)indoline (4m). White solid (314 mg, 81% yield); mp: 133-135 °C; TLC : R_f = 0.50 (EtOAc/Cyclohexane, 3/7, SiO_2); IR (neat, cm^{-1}) 1601, 1586, 1489, 1460, 1413, 1336, 1226, 1204, 1159, 1125; ^1H NMR (300 MHz, CD_3COCD_3) δ (ppm) 7.24 (t, *J* = 7.9 Hz, 1H), 7.17 (m, 1H), 7.11 (d, *J* = 7.1 Hz, 1H), 7.06 – 6.91 (m, 4H), 6.66 (td, *J* = 7.1, 1.5 Hz, 1H), 6.31 (s, 2H), 5.90 (d, *J* = 1.6 Hz, 1H), 5.09 (d, *J* = 1.6 Hz, 1H), 3.88 (t, *J* = 8.4 Hz, 2H), 3.84 (s, 3H), 3.70 (s, 6H), 3.06 (t, *J* = 8.4 Hz, 2H); ^{13}C NMR (75 MHz, CD_3COCD_3) δ (ppm) 162.0 (C), 159.6 (2C), 148.0 (C), 144.8 (C), 142.9 (C), 142.3 (C), 132.2 (C), 129.6 (CH), 127.7 (CH), 125.8 (CH), 119.5 (CH), 119.3 (CH), 116.7 (2CH), 116.4 (CH_2), 112.9 (C), 108.5 (CH), 91.7 (2CH), 56.1 ($2CH_3$), 55.7 (CH_3), 52.5 (CH_2), 28.6 (CH_2); HRMS (ESI) $(M + H)^+$: *m/z* calcd for $C_{25}H_{26}NO_3$ 388.1907; found 388.1902.

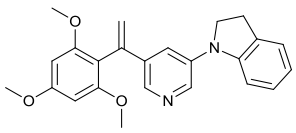


1-(4-(1-(2,4,6-Trimethoxyphenyl)vinyl)phenyl)-1,2,3,4-tetrahydroquinoline (4n). Pink solid (313 mg, 78% yield); mp: 147-149 °C; TLC : R_f = 0.56 (EtOAc/Cyclohexane, 2/8, SiO_2); IR (neat, cm^{-1}) 1599, 1580,1510, 1491, 1453, 1412, 1334, 1224, 1203, 1159, 1124, 950; ^1H NMR (300 MHz, CD_3COCD_3) δ (ppm) 7.27 (d, *J* = 8.7 Hz, 2H), 7.10 (d, *J* = 8.7 Hz, 2H), 6.99 (d, *J* = 7.4 Hz, 1H), 6.88 (t, *J* = 7.7 Hz, 1H), 6.70 (d, *J* = 8.1 Hz, 1H), 6.64 (td, *J* = 7.3, 1.0 Hz, 1H), 6.30 (s, 2H), 5.83 (d, *J* = 1.6 Hz, 1H), 5.00 (d, *J* = 1.6 Hz, 1H), 3.85 (s, 3H), 3.69 (s, 6H), 3.64 – 3.57 (m, 2H), 2.84 – 2.74 (m, 2H), 2.02 – 1.94 (m, 2H); ^{13}C NMR (75 MHz, CD_3COCD_3) δ (ppm) 161.9 (C), 159.7 (2C), 148.2 (C), 145.2 (C), 141.7 (C), 137.2 (C), 130.1 (CH), 127.6 (2CH), 127.0 (CH), 125.8 (C), 124.3 (2CH), 119.2 (CH), 117.4 (C), 116.8 (CH), 115.1 (CH_2), 91.9 (2CH), 56.1 ($2CH_3$), 55.7 (CH_3), 51.4 (CH_2), 28.4 (CH_2), 23.5 (CH_2); HRMS (ESI) $(M + H)^+$: *m/z* calcd for $C_{26}H_{28}NO_3$ 402.2064; found 402.2055.

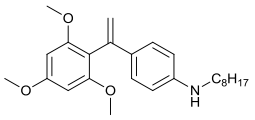


1,1'-(5-(1-(2,4,6-Trimethoxyphenyl)vinyl)-1,3-phenylene)diindoline (4o). White solid (362 mg, 72% yield); mp: 168-170 °C; TLC : R_f = 0.50 (EtOAc/Cyclohexane, 2/8, SiO_2); IR (neat, cm^{-1}) 1711, 1579, 1483, 1414, 1358, 1222, 1158, 1126, 1092; ^1H NMR (300 MHz, $CDCl_3$) δ (ppm) 7.15 (d, *J* = 7.0 Hz, 2H), 7.08 – 6.98 (m, 4H), 6.93 – 6.88 (m, 3H), 6.72 (td, *J* = 7.0, 1.6 Hz, 2H), 6.21 (s, 2H), 5.98 (d, *J* = 1.4 Hz, 1H), 5.23 (d, *J* = 1.4 Hz, 1H), 3.94 (t, *J* = 8.4 Hz, 4H), 3.85 (s, 3H), 3.75 (s, 6H), 3.10 (t, *J* = 8.4 Hz, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm) 160.8 (C), 158.8 (2C), 147.5 (2C), 144.7 (2C), 142.4 (C), 141.6

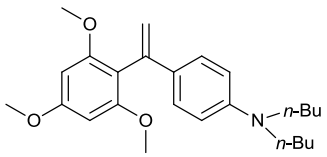
(C), 131.3 (2C), 127.1 (2CH), 125.1 (2CH), 118.7 (2CH), 116.6 (CH_2), 112.5 (C), 109.4 (2CH), 108.5 (2CH), 106.4 (CH), 91.0 (2CH), 56.2 ($2CH_3$), 55.5 (CH_3), 52.3 ($2CH_2$), 28.3 ($2CH_2$); HRMS (ESI) $(M + H)^+$: *m*/*z* calcd for $C_{33}H_{33}N_2O_3$ 505.2486; found 505.2465.



1-(5-(1-(2,4,6-Trimethoxyphenyl)vinyl)pyridin-3-yl)indoline (4p). Colorless oil (200 mg, 51% yield); TLC : R_f = 0.12 (EtOAc/Cyclohexane, 2/8, SiO_2); IR (neat, cm^{-1}) 1604, 1581, 1486, 1463, 1414, 1380, 1337, 1263, 1227, 1204, 1159, 1125, 1052, 949; ^1H NMR (300 MHz, CD_3COCD_3) δ (ppm) 8.33 (d, *J* = 2.6 Hz, 1H), 8.19 (d, *J* = 1.8 Hz, 1H), 7.49 – 7.41 (m, 1H), 7.17 (d, *J* = 7.2 Hz, 1H), 7.03 (t, *J* = 7.7 Hz, 1H), 6.95 (d, *J* = 7.7 Hz, 1H), 6.73 (td, *J* = 7.3, 1.0 Hz, 1H), 6.32 (s, 2H), 5.98 (d, *J* = 1.3 Hz, 1H), 5.20 (d, *J* = 1.3 Hz, 1H), 3.96 (t, *J* = 8.4 Hz, 2H), 3.85 (s, 3H), 3.72 (s, 6H), 3.12 (t, *J* = 8.4 Hz, 2H); ^{13}C NMR (75 MHz, CD_3COCD_3) δ (ppm) 162.2 (C), 159.6 (2C), 147.2 (C), 140.9 (C), 140.4 (CH), 139.7 (C), 138.4 (CH), 137.6 (C), 132.4 (C), 127.8 (CH), 126.1 (CH), 121.8 (CH), 120.4 (CH), 118.3 (CH_2), 111.8 (C), 108.7 (CH), 91.7 (2CH), 56.2 ($2CH_3$), 55.7 (CH_3), 52.2 (CH_2), 28.7 (CH_2); HRMS (ESI) $(M + H)^+$: *m*/*z* calcd for $C_{24}H_{25}N_2O_3$ 389.1860; found; 389.1872.

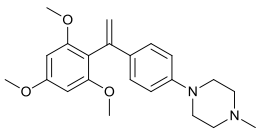


***N*-Octyl-4-(1-(2,4,6-trimethoxyphenyl)vinyl)aniline (4q).** White solid (200 mg, 50% yield); mp: 90-92 °C; TLC : R_f = 0.60 (EtOAc/Cyclohexane, 2/8, SiO_2); IR (neat, cm^{-1}) 1611, 1584, 1520, 1467, 1454, 1412, 1225, 1204, 1160, 1125, 950; ^1H NMR (300 MHz, CD_3COCD_3) δ (ppm) 7.05 (d, *J* = 8.7 Hz, 2H), 6.48 (d, *J* = 8.7 Hz, 2H), 6.27 (s, 2H), 5.63 (d, *J* = 1.8 Hz, 1H), 4.81 (bs, 1H), 4.76 (d, *J* = 1.8 Hz, 1H), 3.84 (s, 3H), 3.65 (s, 6H), 3.08 (t, *J* = 6.1 Hz, 2H), 1.61 (m, 2H), 1.34 (m, 10H), 0.86 (m, 3H); ^{13}C NMR (75 MHz, CD_3COCD_3) δ (ppm) 161.6 (C), 159.7 (2C), 149.4 (C), 142.0 (C), 129.9 (C), 127.5 (2CH), 112.5 (2CH), 112.1 (C), 111.4 (CH_2), 91.8 (2CH), 56.1 ($2CH_3$), 55.6 (CH_3), 44.3 (CH_2), 32.6 (CH_2), 30.2 ($3CH_2$), 27.9 (CH_2), 23.3 (CH_2), 14.4 (CH_3); HRMS (ESI) $(M + H)^+$: *m*/*z* calcd for $C_{25}H_{36}NO_3$ 398.2690; found 398.2712.



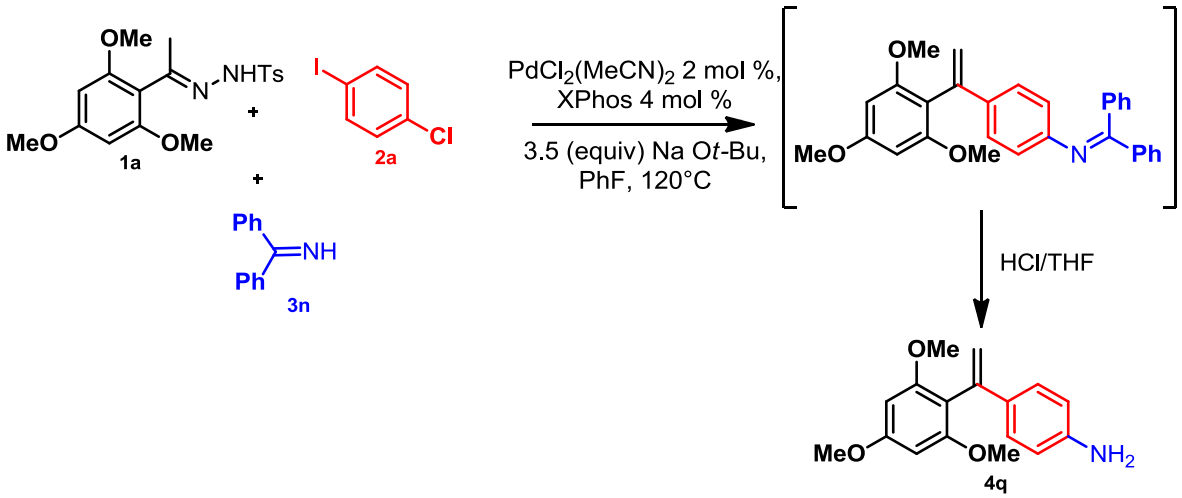
***N*,*N*-dibutyl-4-(1-(2,4,6-trimethoxyphenyl)vinyl)aniline (4r).** Colorless oil (226 mg, 57 % yield); TLC : R_f = 0.67 (EtOAc/Cyclohexane, 2/8, SiO_2); IR (neat, cm^{-1}) 2956, 1604, 1582, 1518, 1466, 1453, 1411, 1287, 1223, 1203, 1182, 1160, 1123, 1037, 949; ^1H NMR (300 MHz, CD_3COCD_3) δ (ppm) 7.10 (d, *J* = 9.0 Hz, 2H), 6.54 (d, *J* = 9.0 Hz, 2H), 6.28 (s, 2H), 5.65 (d, *J* = 1.8 Hz, 1H), 4.77 (d, *J* = 1.8 Hz, 1H), 3.84 (s, 3H), 3.66 (s, 6H), 3.34 – 3.24 (m, 4H), 1.61 – 1.51 (m, 4H), 1.42 – 1.29 (m, 4H), 0.94 (t, *J* = 7.3 Hz, 6H); ^{13}C NMR (75 MHz, CD_3COCD_3) δ 161.6 (C), 159.7 (2C), 148.3 (C), 141.9 (C), 128.8 (C), 127.6 (2CH), 116.3 (C), 112.0 (2CH), 111.3 (CH_2), 91.8 (2CH), 56.1 ($2CH_3$), 55.6 (CH_3), 51.3

($2CH_2$), 30.3 ($2CH_2$), 20.9 ($2CH_2$), 14.3 ($2CH_3$); HRMS (ESI) $(M + H)^+$: *m/z* calcd for $C_{25}H_{36}NO_3$ 398.2695; found 398.2710.



1-Methyl-4-(4-(1-(2,4,6-trimethoxyphenyl)vinyl)phenyl)piperazine (4s). Colorless oil (266 mg, 72% yield); TLC : R_f = 0.26 ($MeOH/CH_2Cl_2$, 5/95, SiO_2); IR (neat, cm^{-1}) 1604, 1583, 1515, 1496, 1453, 1413, 1378, 1335, 1293, 1239, 1224, 1204, 1160, 1124, 1052, 1009, 949; ^1H NMR (300 MHz, CD_3COCD_3) δ (ppm) 7.15 (d, *J* = 8.8 Hz, 2H), 6.81 (d, *J* = 8.8 Hz, 2H), 6.28 (s, 2H), 5.72 (d, *J* = 1.6 Hz, 1H), 4.87 (d, *J* = 1.6 Hz, 1H), 3.84 (s, 3H), 3.65 (s, 6H), 3.21 – 3.07 (m, 4H), 2.51 – 2.41 (m, 4H), 2.24 (s, 3H); ^{13}C NMR (75 MHz, CD_3COCD_3) δ (ppm) 161.7 (C), 159.7 (2C), 151.5 (C), 141.8 (C), 132.7 (C), 127.3 (2CH), 115.8 (2CH), 113.2 (CH_2), 112.3 (C), 91.8 (2CH), 56.1 ($2CH_3$), 55.9 ($2CH_2$), 55.6 (CH_3), 49.5 ($2CH_2$), 46.4 (CH_3); HRMS (ESI) $(M + H)^+$: *m/z* calcd for $C_{22}H_{29}N_2O_3$ 369.2178; found 369.2175.

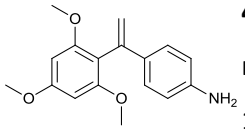
Preparation of aniline derivative 4q



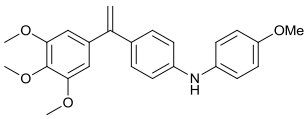
After coupling of *N*-tosylhydrazone **1a**, 1-chloro-4-iodobenzene **2a** and benzophenone imine **3n**, the imine adduct was then hydrolyzed to the desired aniline derivative **4q**, using the protocol developed by Buchwald et al.[2]

To a solution of the imine adduct in THF (0.3 M) was added aqueous 2.0 M HCl (added 5% by volume of THF). After 20 minutes hydrolysis was complete and the reaction mixture was partitioned between 0.5 M HCl and 2:1 hexane/EtOAc. The aqueous layer was separated and made alkaline. The product aniline was extracted with CH_2Cl_2, dried over anhydrous Na_2SO_4 and concentrated *in vacuo*.

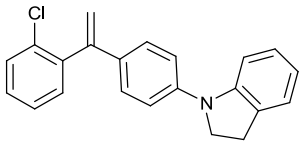
(2) Wolfe, J. P.; Åhman, J.; Sadighi, J. P.; Singer, R. A.; Buchwald, S. L., *Tetrahedron Lett.* **1997**, *38*, 6367.



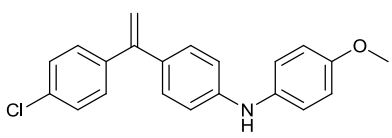
4-(1-(2,4,6-trimethoxyphenyl)vinyl)aniline (4t). White solid (128 mg, 45 % yield); mp: 138-140 °C; TLC : R_f = 0.54 (EtOAc/Cyclohexane, 5/5, SiO_2); IR (neat, cm^{-1}) 1602, 1581, 1514, 1453, 1411, 1334, 1225, 1203, 1183, 1159, 1119, 1050, 1034, 949; ^{1}H NMR (300 MHz, CD_3COCD_3) δ (ppm) 7.01 (d, *J* = 8.5 Hz, 2H), 6.53 (d, *J* = 8.5 Hz, 2H), 6.27 (s, 2H), 5.63 (d, *J* = 1.8 Hz, 1H), 4.78 (d, *J* = 1.8 Hz, 1H), 4.55 (brs, 2H), 3.83 (s, 3H), 3.65 (s, 6H); ^{13}C NMR (75 MHz, CD_3COCD_3) δ (ppm) 161.6 (C), 159.7 (2C), 148.5 (C), 142.1 (C), 130.7 (C), 127.5 (2CH), 114.6 (2CH), 113.8 (C), 111.7 (CH_2), 91.8 (2CH), 56.1 ($2CH_3$), 55.6 (CH_3); HRMS (ESI) $(M + H)^+$: *m/z* calcd for $C_{17}H_{20}NO_3$ 286.1438; found 286.1421.



4-Methoxy-*N*-(4-(1-(3,4,5-trimethoxyphenyl)vinyl)phenyl)aniline (4u). Pale pink oil (344 mg, 88% yield); TLC : R_f = 0.38 (EtOAc/Cyclohexane, 2/8, SiO_2); IR (neat, cm^{-1}) 1700, 1604, 1578, 1501, 1463, 1410, 1346, 1295, 1230, 1179, 1123, 1034, 1004, 944; ^{1}H NMR (300 MHz, CD_3COCD_3) δ (ppm) 7.19 (d, *J* = 8.8 Hz, 2H), 7.14 (d, *J* = 8.8 Hz, 2H), 6.95 (d, *J* = 8.9 Hz, 2H), 6.89 (d, *J* = 8.9 Hz, 2H), 6.62 (s, 2H), 5.33 (d, *J* = 1.4 Hz, 1H), 5.25 (d, *J* = 1.4 Hz, 1H), 3.78 (s, 6H), 3.77 (s, 3H), 3.75 (s, 3H); ^{13}C NMR (75 MHz, CD_3COCD_3) δ (ppm) 156.0 (C), 154.0 (2C), 151.0 (C), 146.5 (C), 138.5 (C), 136.8 (C), 132.2 (C), 129.9 (2CH), 122.5 (2CH), 115.4 (2CH), 115.2 (2CH), 114.3 (C), 111.4 (CH_2), 106.9 (2CH), 60.6 (CH_3), 56.5 ($2CH_3$), 55.7 (CH_3); HRMS (ESI) $(M + Na)^+$: *m/z* calcd for $C_{24}H_{25}NNaO_4$ 414.1676; found 414.1663.

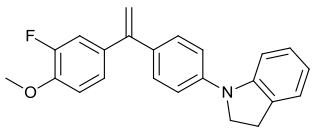


1-(4-(1-(2-chlorophenyl)vinyl)phenyl)indoline (4v). Colorless oil (185 mg, 56% yield); TLC : R_f = 0.87 (Pentane, SiO_2); IR (neat) 1702, 1595, 1512, 1482, 1458, 1384, 1335, 1309, 1264, 1156, 1126, 1046, 895, 831, 768; ^{1}H NMR (300 MHz, CD_3COCD_3) δ (ppm) 7.55 – 7.30 (m, 4H), 7.27 – 7.10 (m, 6H), 7.04 (t, *J* = 7.7 Hz, 1H), 6.72 (t, *J* = 7.4 Hz, 1H), 5.82 (d, *J* = 1.0 Hz, 1H), 5.12 (d, *J* = 1.0 Hz, 1H), 3.94 (t, *J* = 8.5 Hz, 2H), 3.10 (t, *J* = 8.5 Hz, 2H); ^{13}C NMR (75 MHz, CD_3COCD_3) δ 148.0 (C), 147.3 (C), 144.7 (C), 143.8 (C), 141.9 (C), 133.8 (C), 132.6 (C), 132.5 (CH), 130.5 (CH), 130.0 (CH), 128.0 (2CH), 127.8 (CH), 125.9 (CH), 120.0 (CH), 117.6 (2CH), 114.0 (CH_2), 109.2 (CH), 52.5 (CH_2), 28.5 (CH_2); HRMS (ESI) $(M + H)^+$: *m/z* calcd for $C_{22}H_{19}ClN$ 332.1201; found 332.1190.

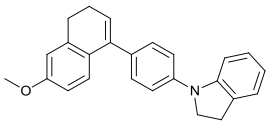


4-(1-(4-chlorophenyl)vinyl)-N-(4-methoxyphenyl)aniline (4w). White solid (119 mg, 35 % yield); mp: 116-118 °C; TLC : R_f = 0.55 (AcOEt/Cyclohexane, 2/8, SiO_2); IR (neat) 1605, 1510, 1327, 1246, 1180, 1091, 1035, 1013; ^{1}H NMR (300 MHz, CD_3COCD_3) δ (ppm) 7.40 (d, *J* = 8.9 Hz, 2H), 7.35 (d, *J* = 8.9 Hz, 2H), 7.28 (bs, 1H), 7.18-7.10 (m, 4H), 6.94 (d, *J* = 8.8 Hz, 2H), 6.90 (d, *J* = 9.0 Hz, 2H), 5.40

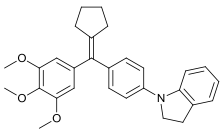
(d, *J* = 1.2 Hz, 1H), 5.28 (d, *J* = 1.2 Hz, 1H), 3.77 (s, 3H); ^{13}C NMR (75 MHz, CD_3COCD_3) δ (ppm) 149.8 (C), 146.8 (C), 141.8 (C), 141.6 (C), 136.7 (C), 133.8 (C), 131.7 (C), 130.8 (2CH), 129.8 (2CH), 129.1 (2CH), 122.6 (2CH), 115.4 (2CH), 115.3 (2CH), 112.4 (CH_2), 55.7 (CH_3); HRMS (ESI) $(M + H)^+$: *m*/*z* calcd for $C_{21}H_{19}ClNO$ 336.1155; found 336.1145.



1-(4-(1-(3-Fluoro-4-methoxyphenyl)vinyl)phenyl)indoline (4x). White solid (248 mg, 72% yield); mp: 139-140 °C; TLC : R_f = 0.74 (EtOAc/Cyclohexane, 2/8, SiO_2); IR (neat, cm^{-1}) 1598, 1515, 1484, 1459, 1386, 1336, 1307, 1269, 1119, 1026; ^{1}H NMR (300 MHz, CD_3COCD_3) δ (ppm) 7.33 (d, *J* = 9.0 Hz, 2H), 7.26 (d, *J* = 9.0 Hz, 2H), 7.22 – 7.00 (m, 6H), 6.74 (t, *J* = 7.4 Hz, 1H), 5.38 (d, *J* = 1.1 Hz, 1H), 5.34 (d, *J* = 1.2 Hz, 1H), 3.99 (t, *J* = 8.5 Hz, 2H), 3.92 (s, 3H), 3.13 (t, *J* = 8.4 Hz, 2H); ^{13}C NMR (75 MHz, CD_3COCD_3) δ (ppm) 154.4 (C), 151.2 (C), 149.4 (C), 147.4 (C), 144.9 (C), 135.7 (d, *J* = 7 Hz, C), 134.0 (C), 132.5 (C), 129.8 (2CH), 127.8 (CH), 126.0 (CH), 125.2 (d, *J* = 3 Hz, CH), 120.0 (CH), 117.6 (2CH), 116.4 (d, *J* = 19 Hz, CH), 114.2 (CH), 112.6 (CH_2), 109.2 (CH), 56.6 (CH_3), 52.6 (CH_2), 28.6 (CH_2); ^{19}F NMR (188 MHz, CD_3COCD_3) δ (ppm) 42.5; HRMS (ESI) $(M + H)^+$: *m*/*z* calcd for $C_{23}H_{21}FNO$ 346.1602; found 346.1577.

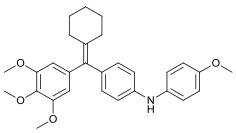


1-(4-(6-Methoxy-3,4-dihydronaphthalen-1-yl)phenyl)indoline (4y). White solid (329 mg, 93% yield); mp: 123-125 °C; TLC : R_f = 0.73 (EtOAc/Cyclohexane, 2/8, SiO_2); IR (neat, cm^{-1}) 2931, 2831, 1598, 1566, 1513, 1483, 1459, 1425, 1384, 1335, 1308, 1248, 1166, 1120, 1038; ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm) 7.34 (d, *J* = 8.7 Hz, 2H), 7.24 – 7.16 (m, 4H), 7.15 – 7.09 (m, 1H), 7.06 (d, *J* = 8.6 Hz, 1H), 6.82 – 6.74 (m, 2H), 6.68 (dd, *J* = 8.5, 2.7 Hz, 1H), 5.98 (t, *J* = 4.7 Hz, 1H), 4.00 (t, *J* = 8.5 Hz, 2H), 3.83 (s, 3H), 3.16 (t, *J* = 8.4 Hz, 2H), 2.84 (t, *J* = 7.8 Hz, 2H), 2.45 – 2.36 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm) 158. 7 (C), 147.2 (C), 143.2 (C), 139.2 (C), 139.0 (C), 133.6 (C), 131.4 (C), 129.5 (2CH), 128.5 (C), 127.2 (CH), 126.8 (CH), 125.2 (CH), 124.5 (CH), 119.0 (CH), 117.4 (2CH), 113.9 (CH), 110.9 (CH), 108.4 (CH), 55.4 (CH_3), 52.3 (CH_2), 29.1 (CH_2), 28.3 (CH_2), 23.7 (CH_2); HRMS (ESI) $(M + H)^+$: *m*/*z* calcd for $C_{25}H_{24}NO$ 354.1852; found 354.1836.

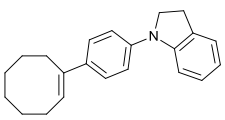


1-(4-(Cyclopentylidene(3,4,5-trimethoxyphenyl)methyl)phenyl)indoline (4z). White solid (400 mg, 90% yield); mp: 113-115 °C; TLC : R_f = 0.58 (EtOAc/Cyclohexane, 2/8, SiO_2); IR (neat, cm^{-1}) 1712, 1598, 1579, 1512, 1484, 1459, 1409, 1385, 1337, 1233, 1126, 1009; ^{1}H NMR (300 MHz, CD_3COCD_3) δ (ppm) 7.20 (s, 4H), 7.14 (t, *J* = 7.1 Hz, 2H), 7.03 (t, *J* = 8.0 Hz, 1H), 6.70 (td, *J* = 7.3, 0.9 Hz, 1H), 6.48 (s, 2H), 3.95 (t, *J* = 8.5 Hz, 2H), 3.77 (s, 6H), 3.73 (s, 3H), 3.10 (t, *J* = 8.4 Hz, 2H), 2.51 – 2.34 (m, 4H), 1.74 – 1.59 (m, 4H); ^{13}C NMR (75 MHz, CD_3COCD_3) δ (ppm) 154.1 (2C), 143.1 (C), 142.8 (2C), 140.3 (C), 136.5 (C), 134.0 (C), 132.3

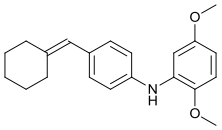
(C), 130.5 (2CH), 127.8 (CH), 125.8 (CH), 119.7 (CH), 118.4 (C), 117.6 (2CH), 108.8 (CH), 107.7 (2CH), 60.5 ($2CH_3$), 56.5 (CH_3), 52.6 (CH_2), 33.9 (CH_2), 33.8 (CH_2), 28.6 (CH_2), 27.6 (CH_2), 27.4 (CH_2); HRMS (ESI) $(M + H)^+$: *m/z* calcd for $C_{29}H_{32}NO_3$ 442.2377; found 442.2363.



4-(Cyclohexylidene(3,4,5-trimethoxyphenyl)methyl)-*N*-(4-methoxyphenyl)-aniline (4aa). White solid (346 mg, 75% yield); mp: 98-100 °C; TLC : R_f = 0.41 (EtOAc/Cyclohexane, 2/8, SiO_2); IR (neat, cm^{-1}) 1752, 1701, 1606, 1578, 1504, 1464, 1408, 1342, 1229, 1179, 1124, 1038, 1005; ^1H NMR (300 MHz, CD_3COCD_3) δ (ppm) 7.15 – 7.02 (m, 3H), 7.01 – 6.94 (m, 2H), 6.92 – 6.81 (m, 4H), 6.41 (s, 2H), 3.76 (m, 9H), 3.71 (s, 3H), 2.35 – 2.15 (m, 4H), 1.61 (bs, 6H); ^{13}C NMR (75 MHz, CD_3COCD_3) δ (ppm) 155.7 (C), 154.0 (2C), 144.8 (C), 140.1 (C), 138.4 (C), 137.8 (C), 137.3 (C), 135.8 (C), 134.1 (C), 131.1 (2CH), 122.0 (2CH), 115.5 (2CH), 115.4 (2CH), 108.0 (2CH), 60.5 (CH_3), 56.4 ($2CH_3$), 55.7 (CH_3), 33.4 (CH_2), 33.0 (CH_2), 29.3 ($2CH_2$), 27.5 (CH_2); HRMS (ESI) $(M + H)^+$: *m/z* calcd for $C_{29}H_{34}NO_4$ 460.2482; found 460.2468.

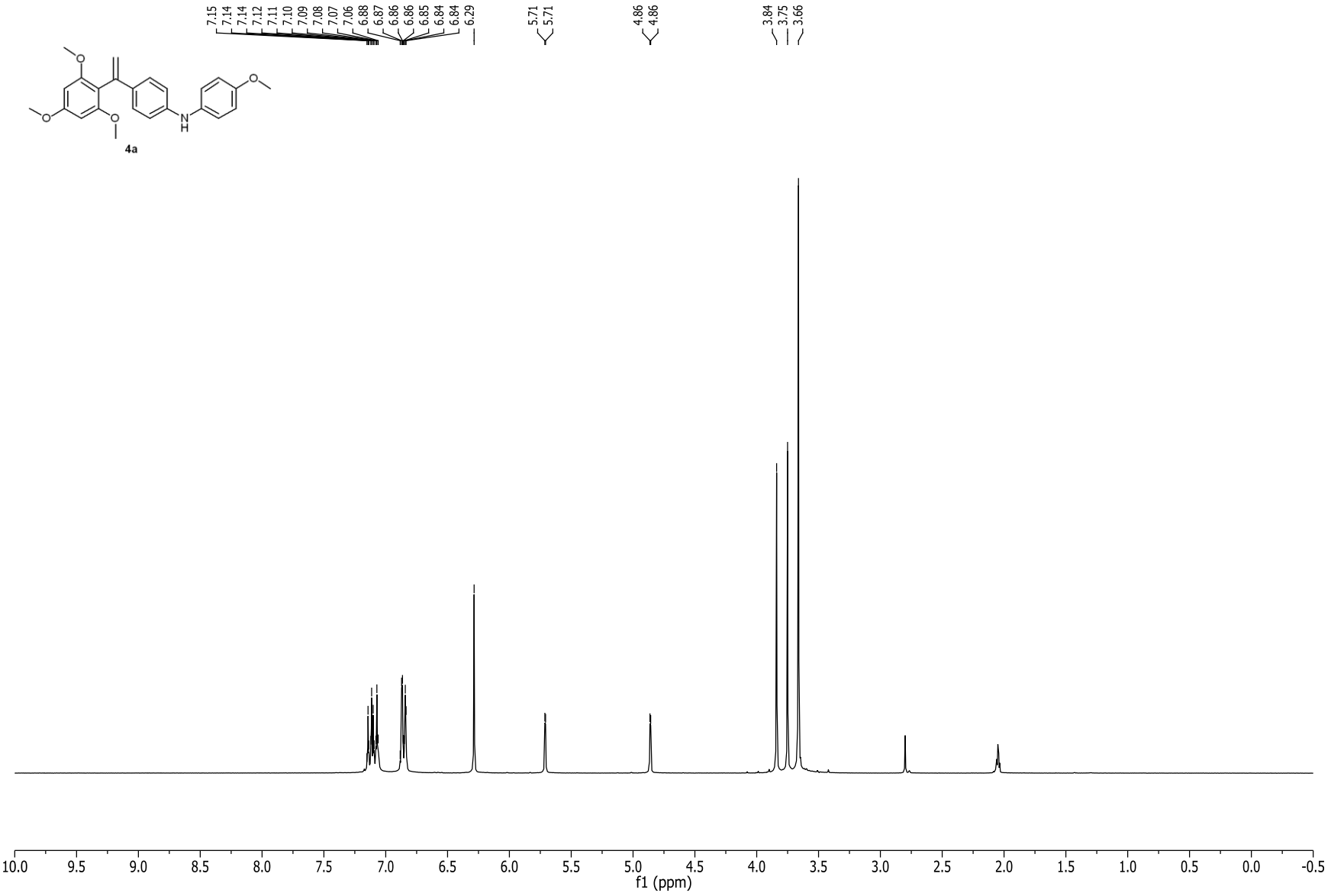


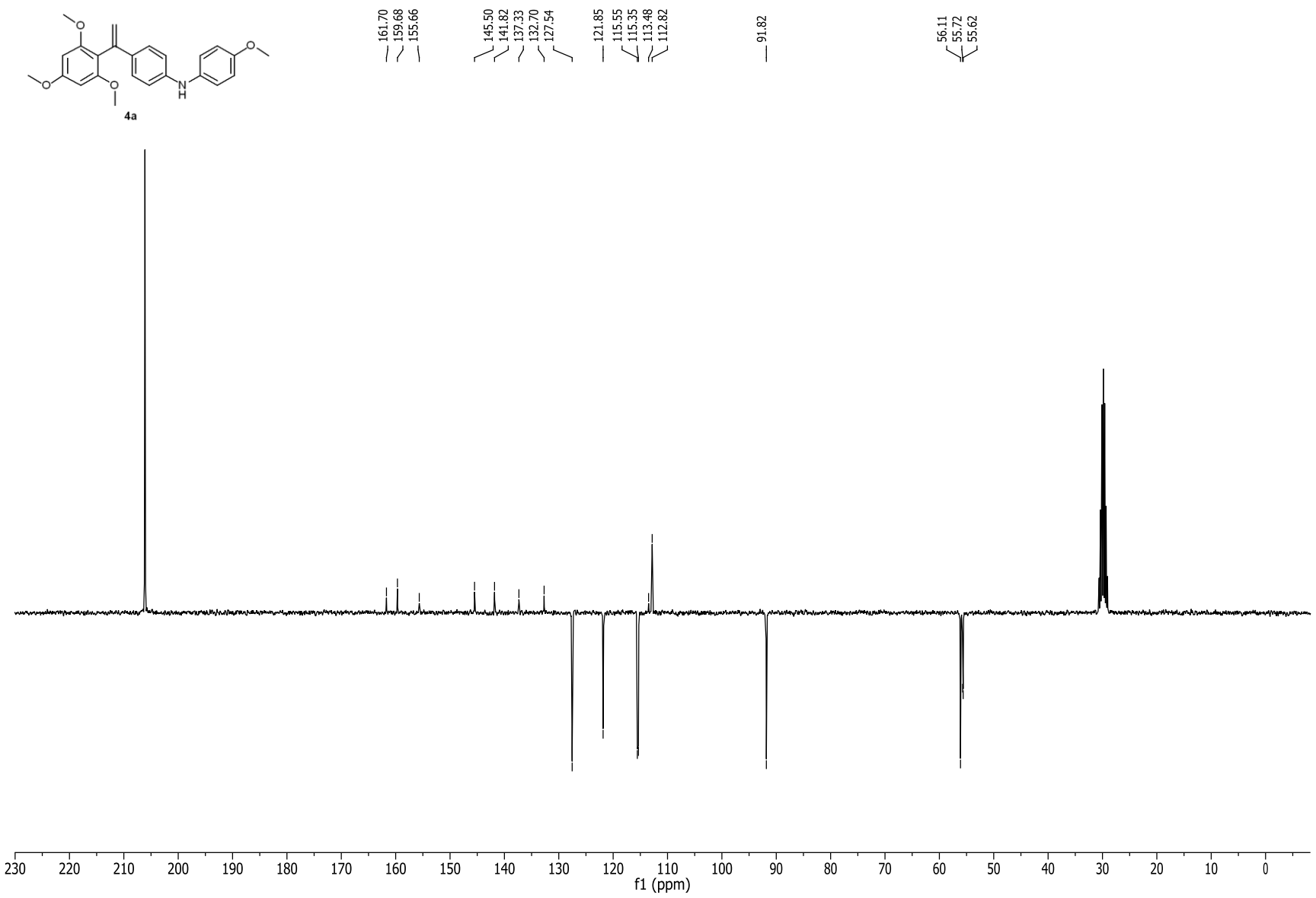
(*E*)-1-(4-Cyclooctenylphenyl)indoline (4ab). White solid (206 mg, 68% yield); mp: 85-87 °C; TLC : R_f = 0.39 (Heptane, SiO_2); IR (neat, cm^{-1}) 2921, 2847, 1597, 1512, 1483, 1458, 1384, 1335, 1313, 1263, 1227; ^1H NMR (300 MHz, CD_3COCD_3) δ (ppm) 7.43 (d, *J* = 8.8 Hz, 2H), 7.19 (d, *J* = 8.8 Hz, 2H), 7.17 – 6.99 (m, 3H), 6.70 (td, *J* = 7.3, 1.0 Hz, 1H), 6.01 (t, *J* = 8.3 Hz, 1H), 3.93 (t, *J* = 8.5 Hz, 2H), 3.09 (t, *J* = 8.5 Hz, 2H), 2.68 – 2.59 (m, 2H), 2.37 – 2.24 (m, 2H), 1.70 – 1.48 (m, 8H); ^{13}C NMR (75 MHz, CD_3COCD_3) δ (ppm) 147.9 (C), 143.7 (C), 140.5 (C), 136.0 (C), 132.3 (C), 127.8 (CH), 127.1 (2CH), 126.5 (CH), 125.8 (CH), 119.6 (CH), 118.1 (2CH) , 108.8 (CH), 52.7 (CH_2), 30.9 (CH_2), 30.1 (CH_2), 28.6 (CH_2), 28.6 (CH_2), 28.0 (CH_2), 27.7 (CH_2), 26.8 (CH_2); HRMS (ESI) $(M + H)^+$: *m/z* calcd for $C_{22}H_{26}N$ 304.2060; found 304.2055.

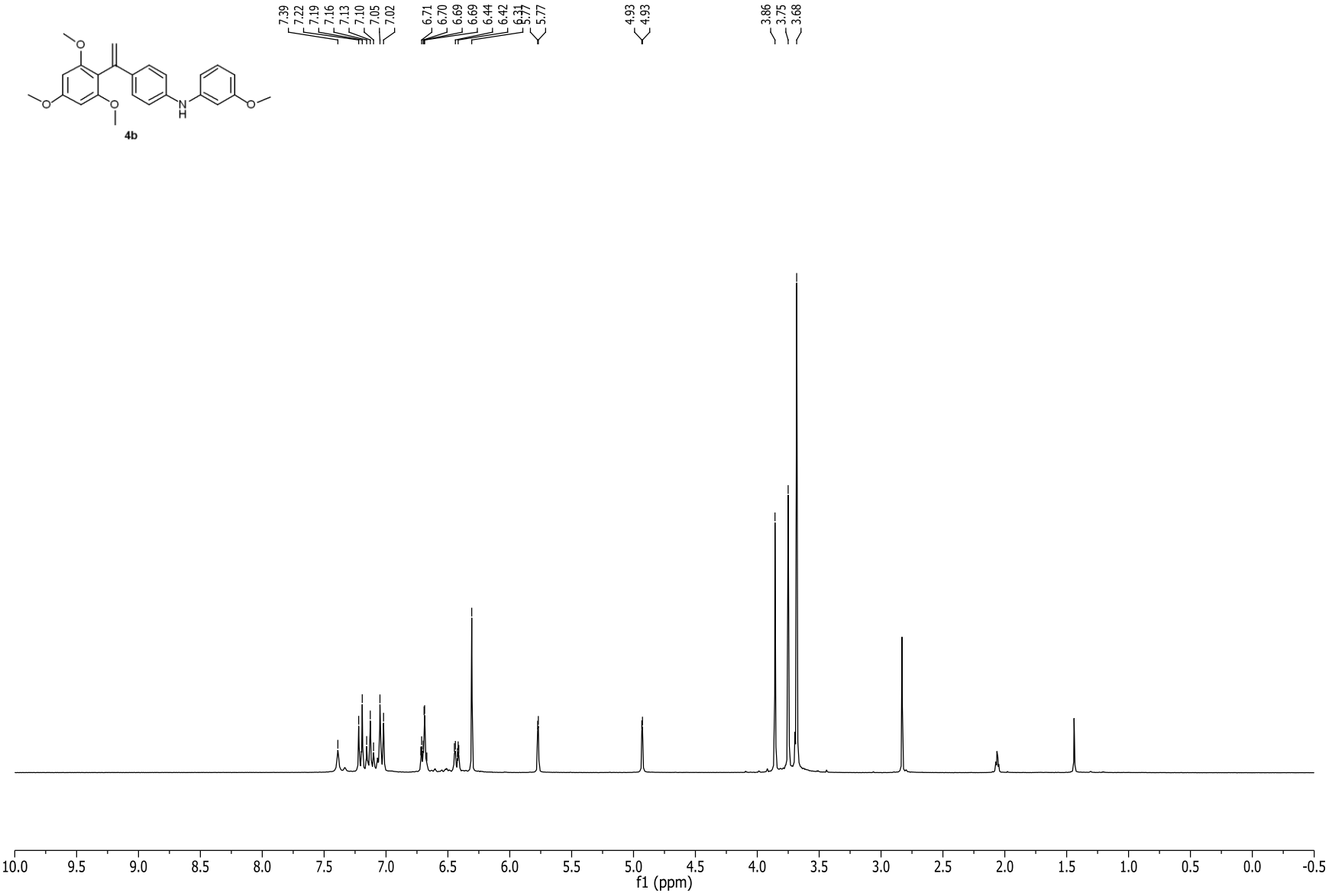


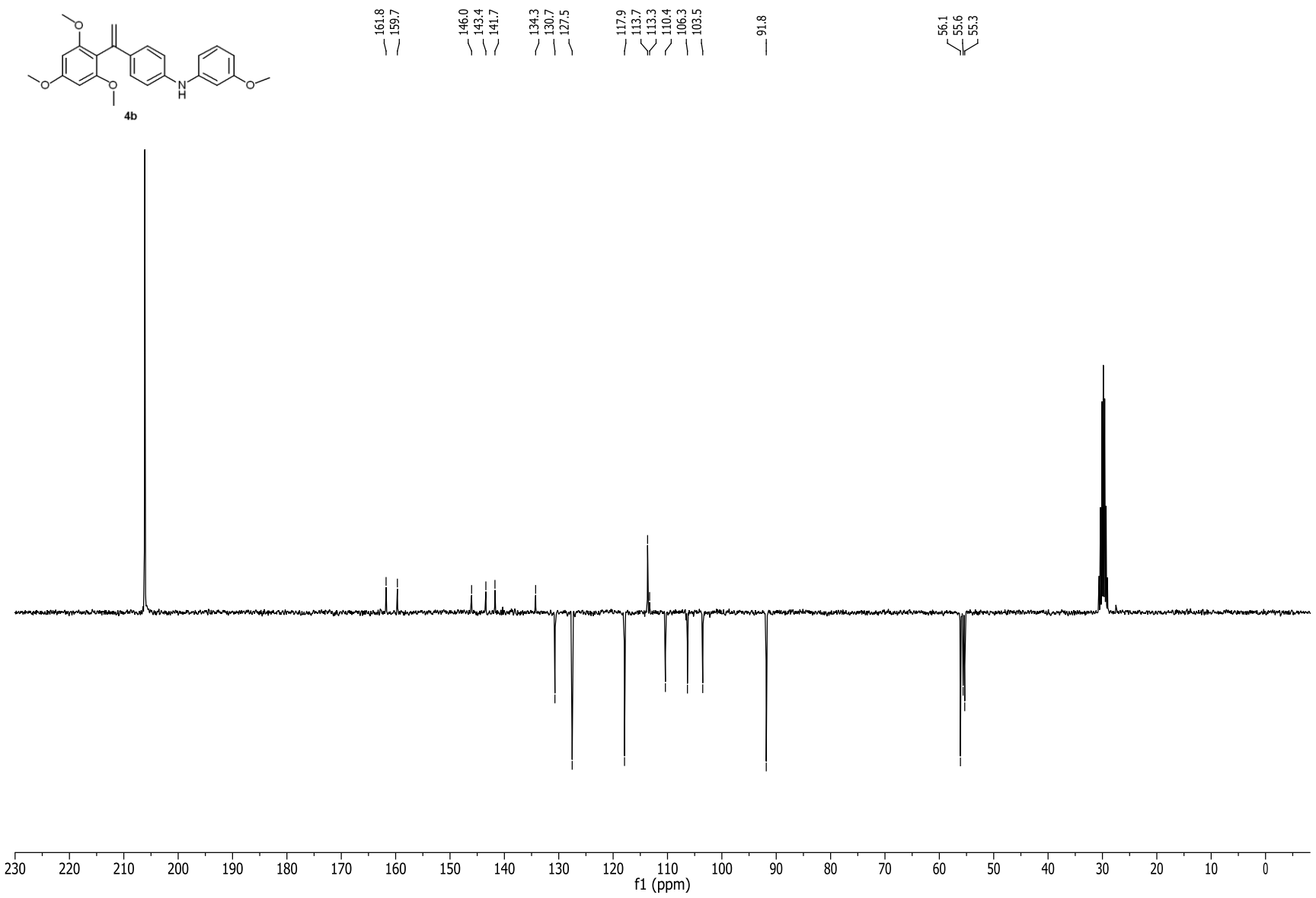
***N*-(4-(Cyclohexylidenemethyl)phenyl)-2,5-dimethoxyaniline (4ac).** Colorless oil (230 mg, 70% yield); TLC : R_f = 0.67 (EtOAc/Cyclohexane, 2/8, SiO_2); IR (neat) 3418, 2927, 1600, 1524, 1508, 1464, 1400, 1286, 1240, 1214, 1179, 1163, 1130, 1050, 1026; ^1H NMR (300 MHz, CD_3COCD_3) δ (ppm) 7.24 – 7.06 (m, 4H), 6.91 – 6.84 (m, 2H), 6.77 (bs, 1H), 6.36 (dd, *J* = 8.8, 2.9 Hz, 1H), 6.18 (s, 1H), 3.80 (s, 3H), 3.70 (s, 3H), 2.45 – 2.37 (m, 2H), 2.30 – 2.19 (m, 2H), 1.66 – 1.49 (m, 6H); ^{13}C NMR (75 MHz, CD_3COCD_3) δ (ppm) 155.2 (C), 144.0 (C), 142.3 (C), 141.8 (C), 135.0 (C), 131.9 (C), 130.5 (2CH), 122.7 (CH), 119.0 (2CH), 112.6 (CH), 103.9 (CH), 102.7 (CH), 56.6 (CH_3), 55.7 (CH_3), 38.3 (CH_2), 30.1 (CH_2), 29.5 (CH_2), 28.6 (CH_2), 27.4 (CH_2). HRMS (ESI) $(M + H)^+$: *m/z* calcd for $C_{21}H_{26}NO_2$ 324.1964; found 324.1958.

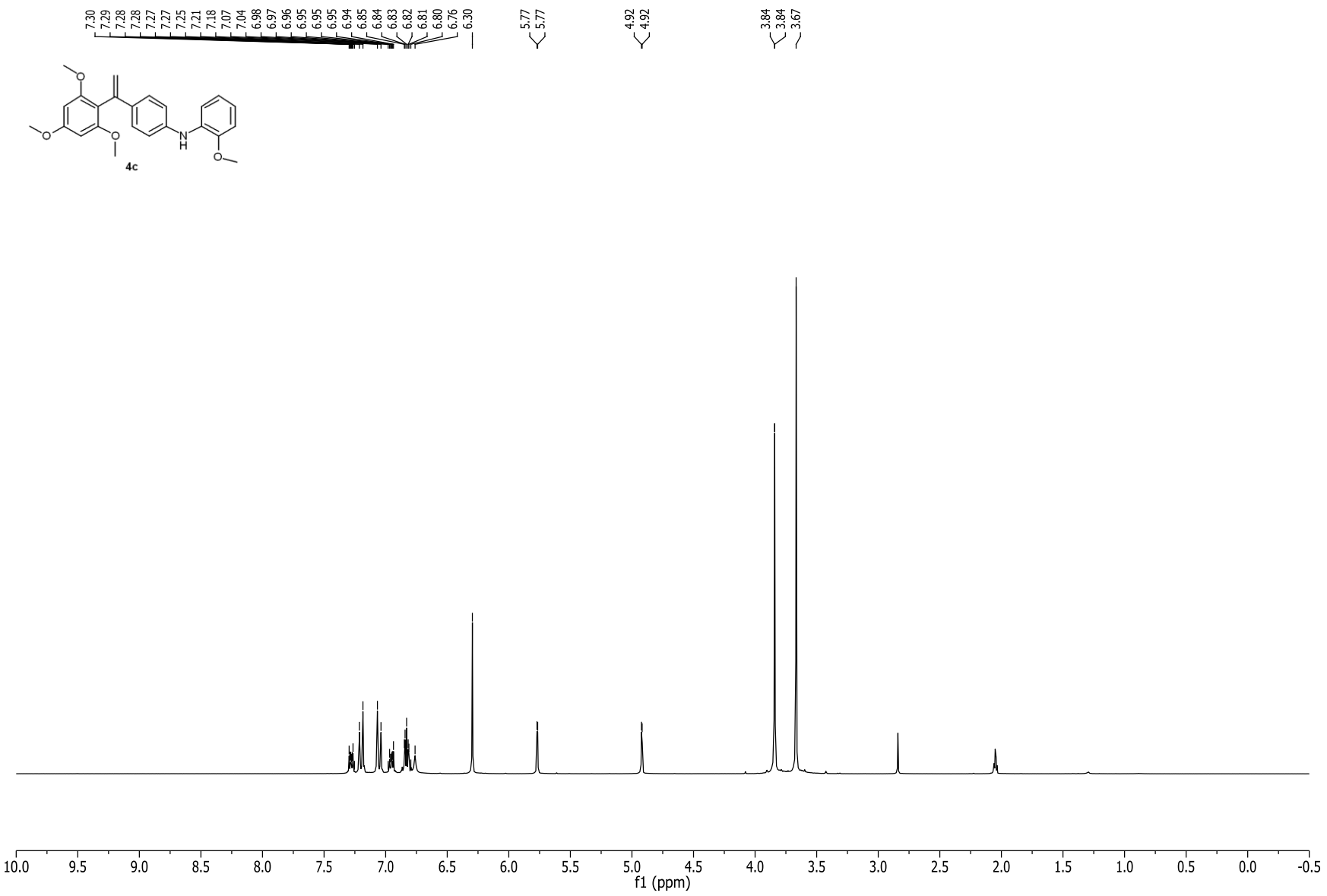
IV ^{1}H and ^{13}C NMR Spectra for compounds 4a – 4ac

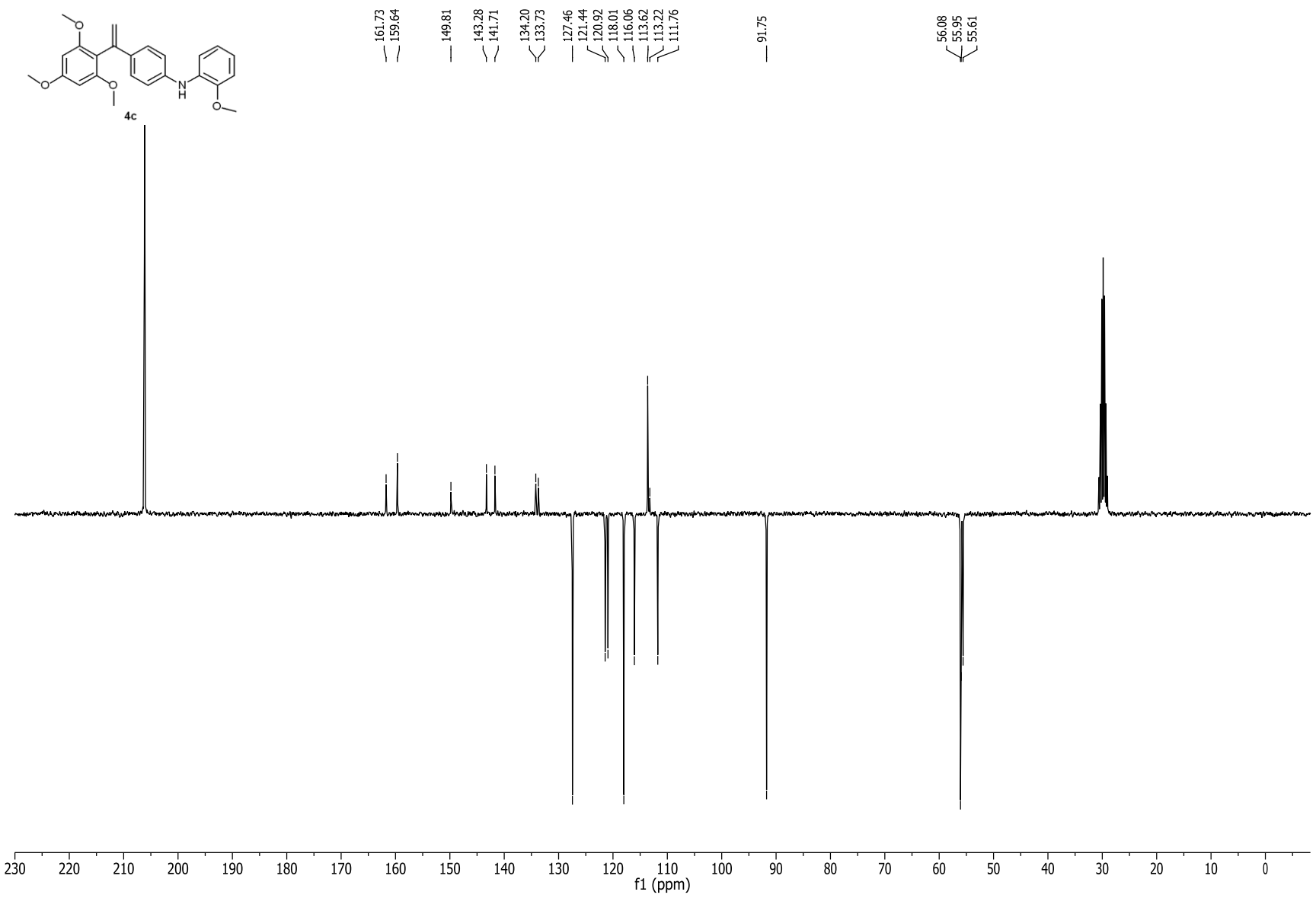

7.15
7.14
7.14
7.12
7.11
7.10
7.09
7.08
7.07
7.06
6.88
6.87
6.86
6.86
6.85
6.84
6.84
6.29
5.71
5.71
4.86
4.86
3.84
3.75
3.66
O
O
O
O
N
H
4a
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

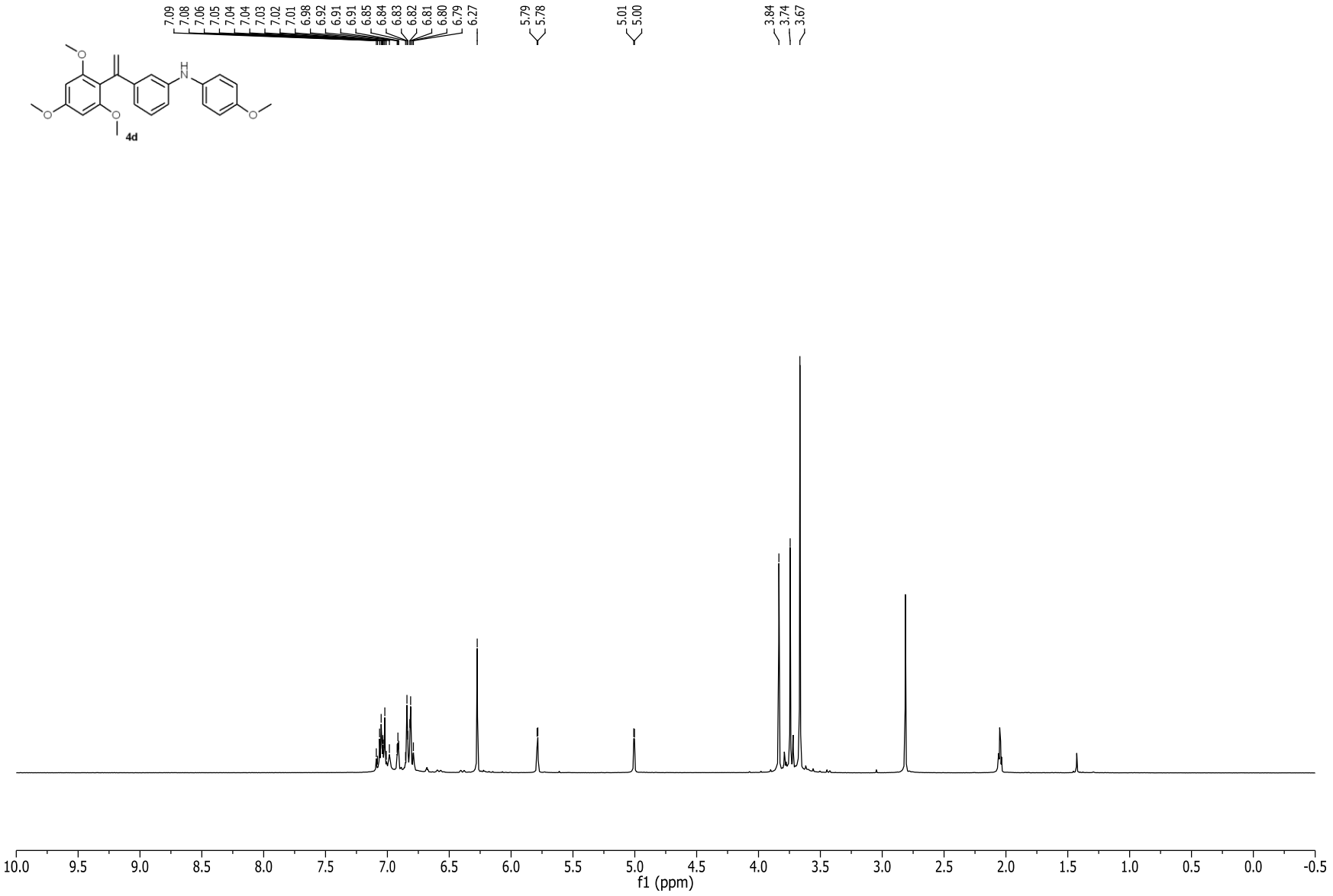

4a
161.70
159.68
155.66
145.50
141.82
137.33
132.70
127.54
121.85
115.55
115.35
113.48
112.82
91.82
56.11
55.72
55.62
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

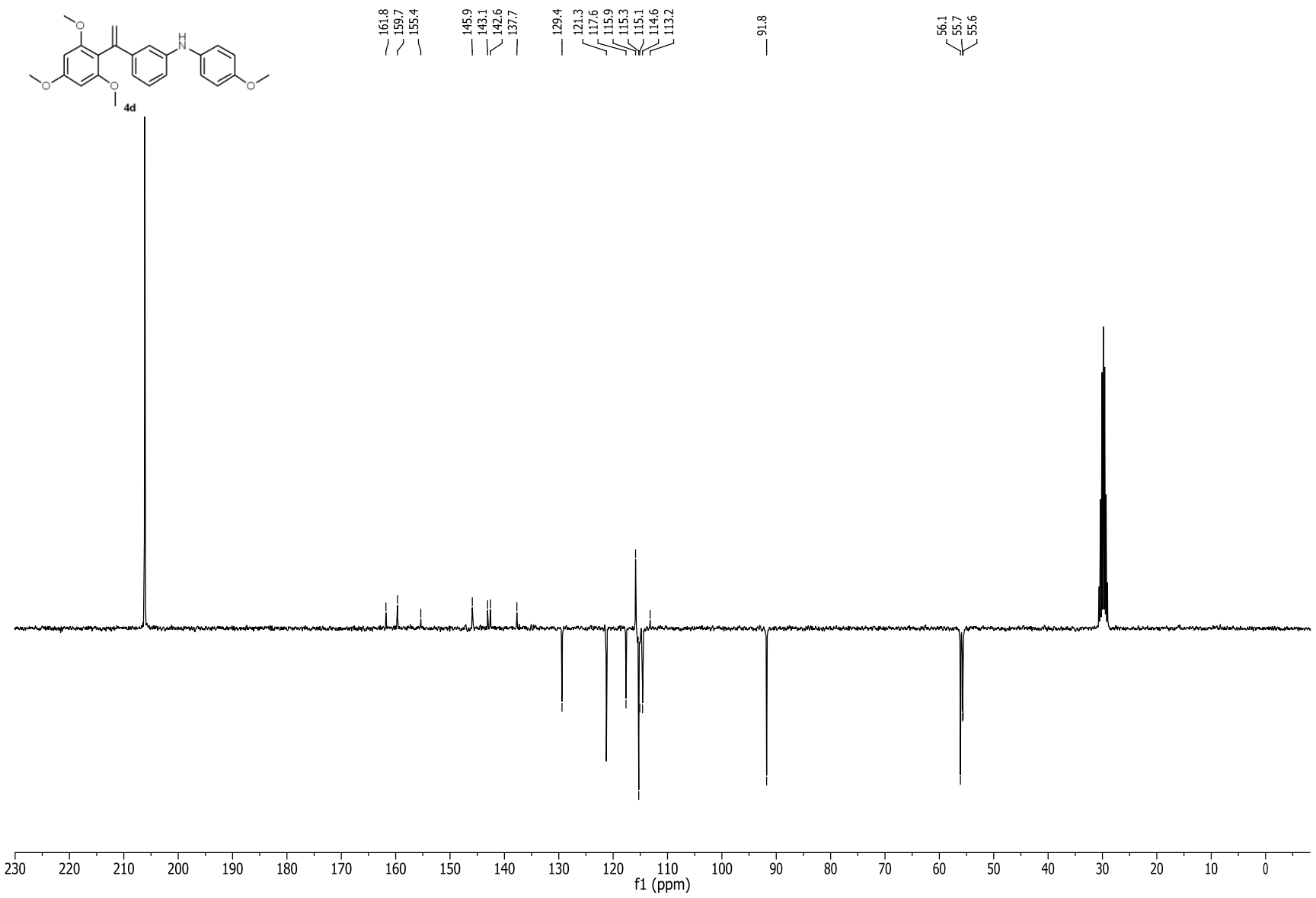

7.39
7.22
7.19
7.16
7.13
7.10
7.05
7.02
6.71
6.70
6.69
6.69
6.44
6.42
6.31
5.77
5.77
4.93
4.93
3.86
3.75
3.68
O
O
O
O
N
H
4b
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

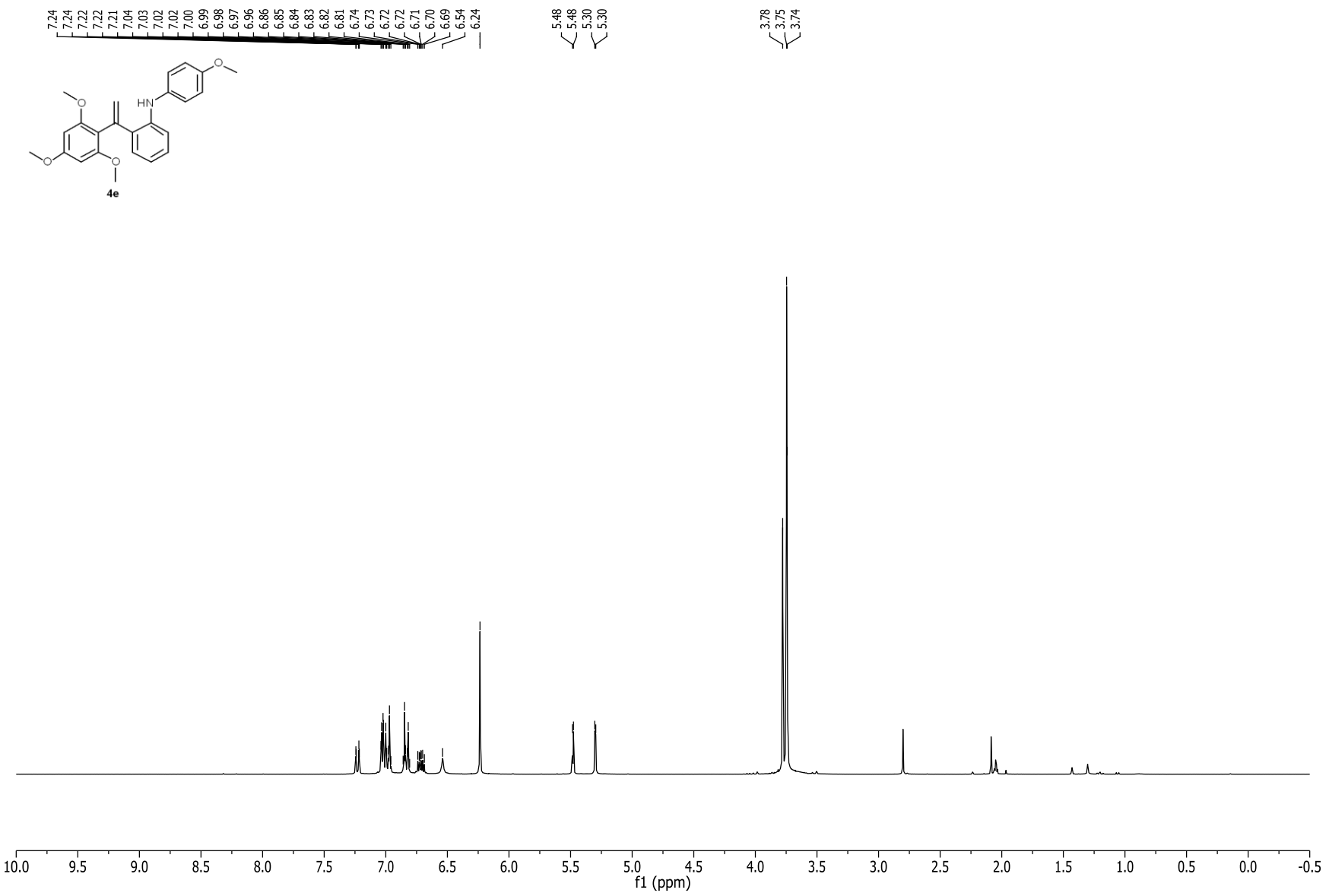

4b
161.8
159.7
146.0
143.4
141.7
134.3
130.7
127.5
117.9
113.7
113.3
110.4
106.3
103.5
91.8
56.1
55.6
55.3
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

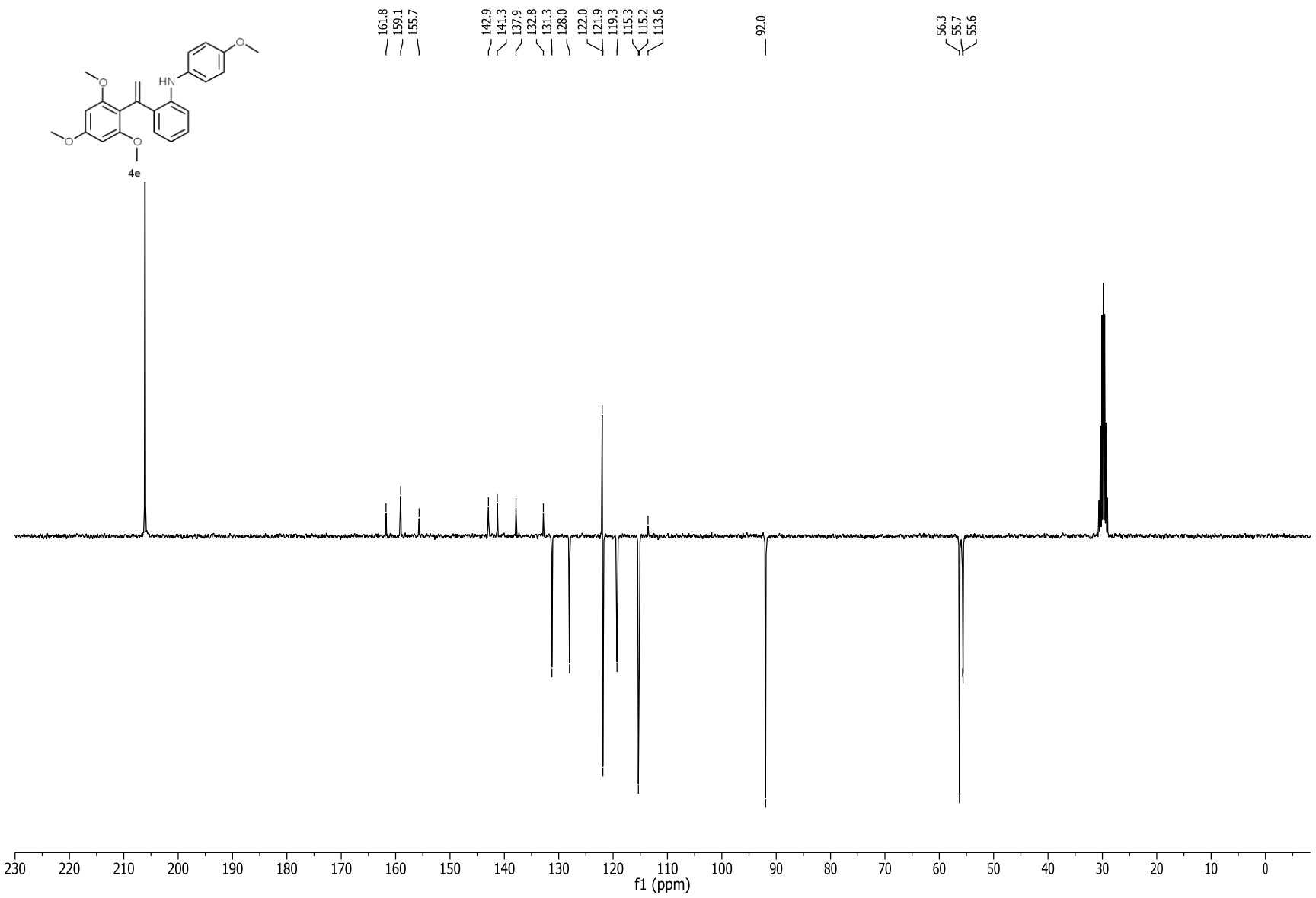

7.30
7.29
7.28
7.28
7.27
7.27
7.25
7.21
7.18
7.07
7.04
6.98
6.97
6.96
6.95
6.95
6.95
6.94
6.85
6.84
6.83
6.82
6.81
6.80
6.76
6.30
5.77
5.77
4.92
4.92
3.84
3.84
3.67
O
O
O
N
H
O
4c
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

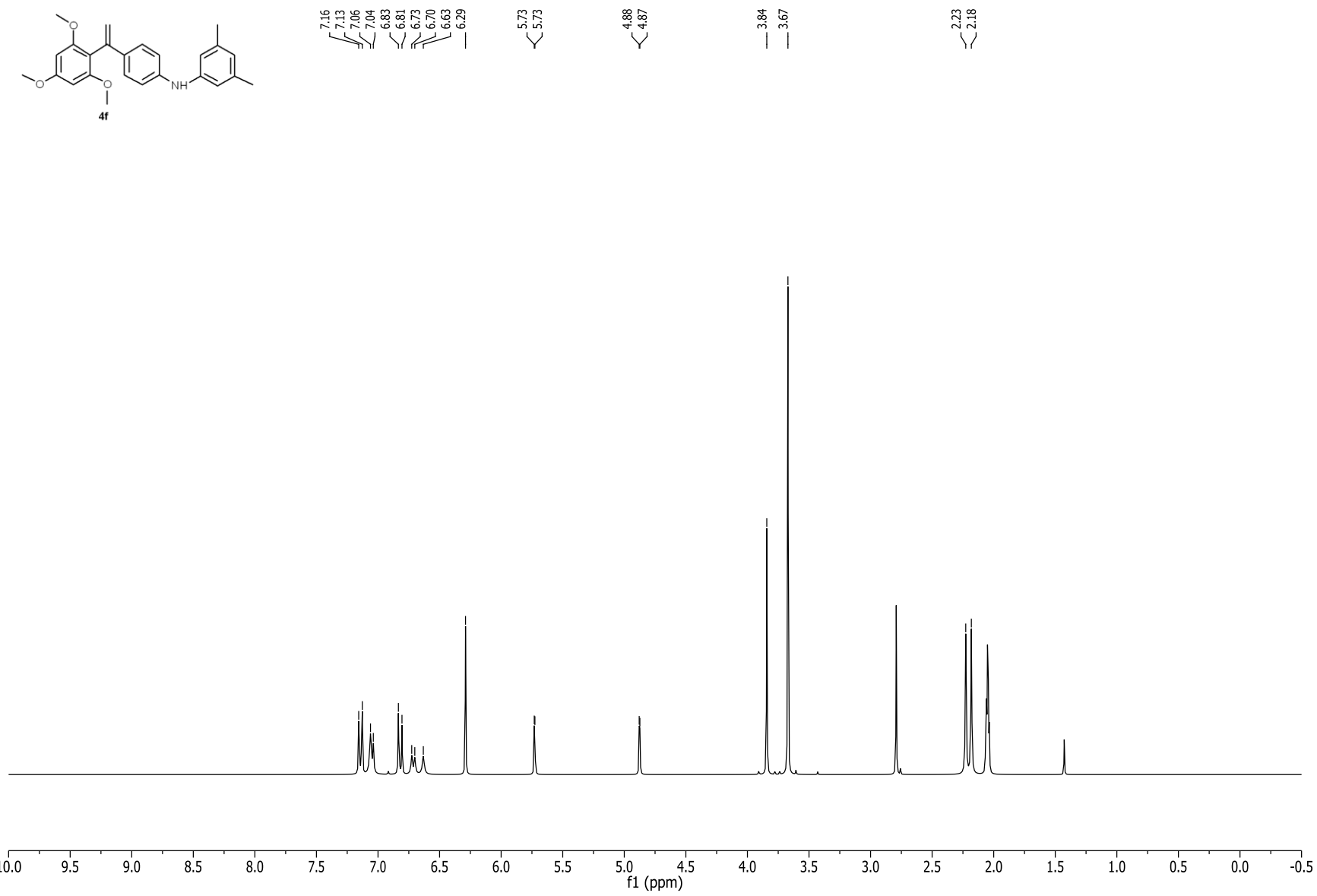

4c
161.73
159.64
149.81
143.28
141.71
134.20
133.73
127.46
121.44
120.92
118.01
116.06
113.62
113.22
111.76
91.75
56.08
55.95
55.61
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

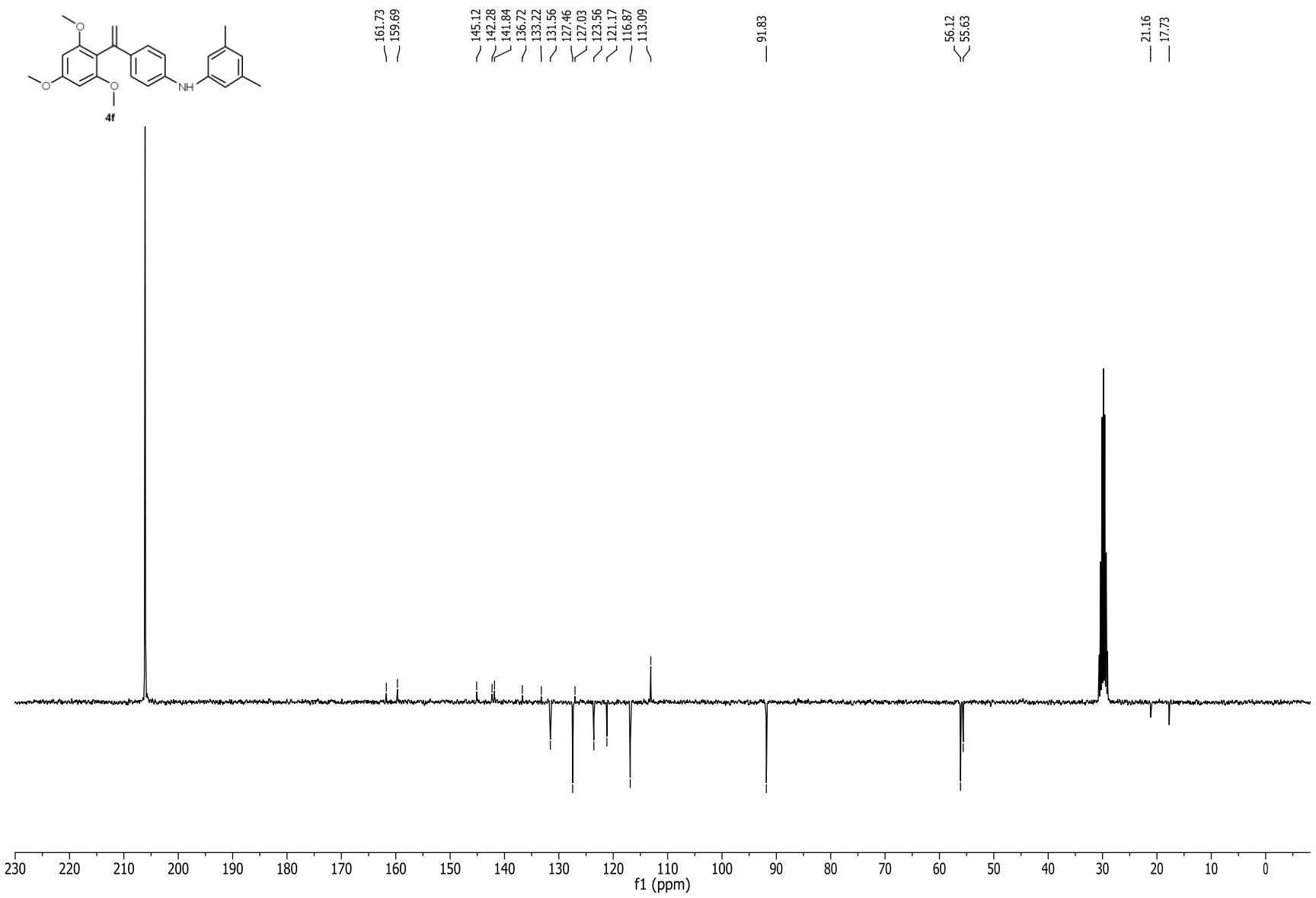

7.09
7.08
7.06
7.05
7.04
7.04
7.03
7.02
7.01
6.98
6.92
6.91
6.91
6.85
6.84
6.83
6.82
6.81
6.80
6.79
6.27
5.79
5.78
5.01
5.00
3.84
3.74
3.67
4d
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

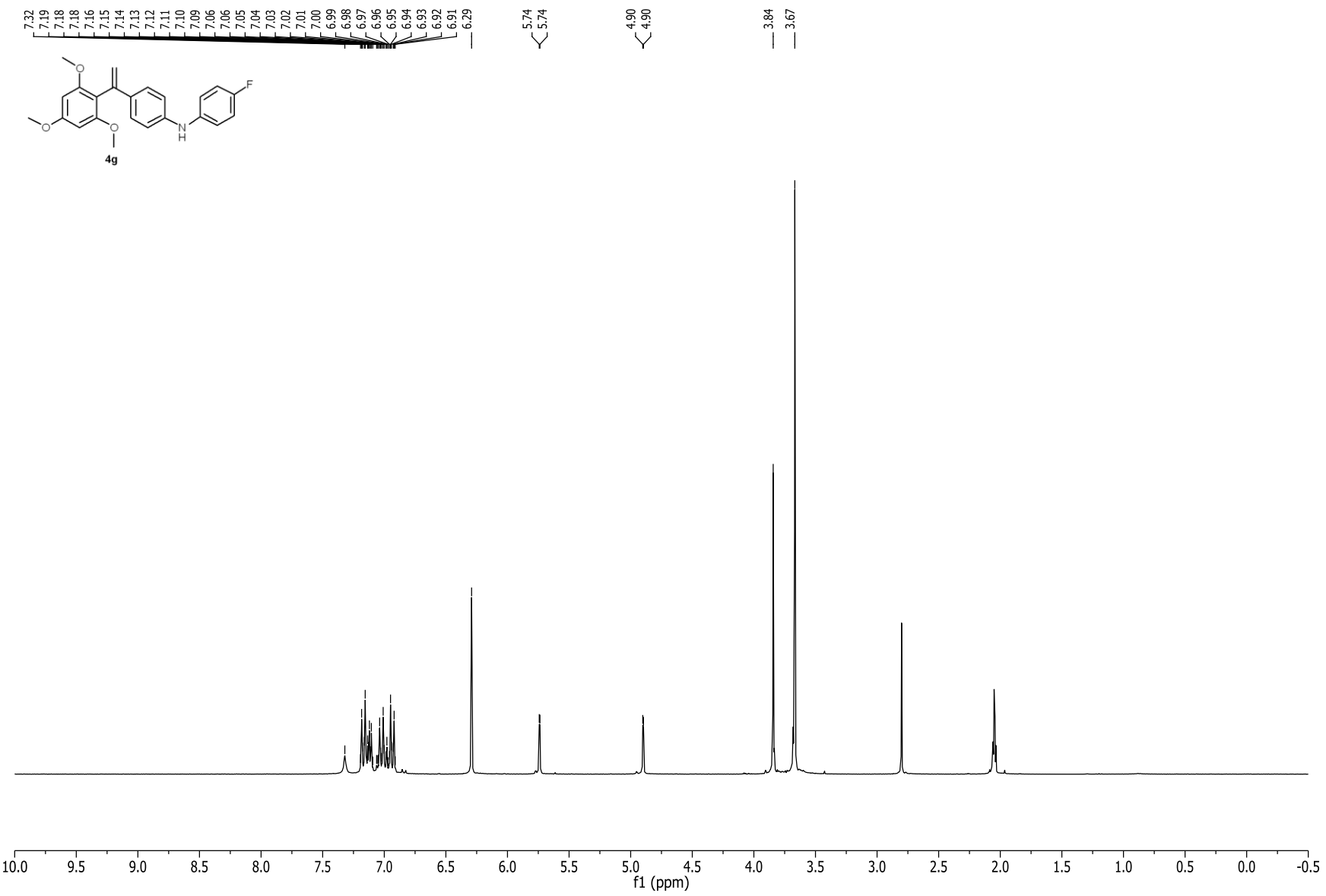

4d
161.8
159.7
155.4
145.9
143.1
142.6
137.7
129.4
121.3
117.6
115.9
115.3
115.1
114.6
113.2
91.8
56.1
55.7
55.6
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

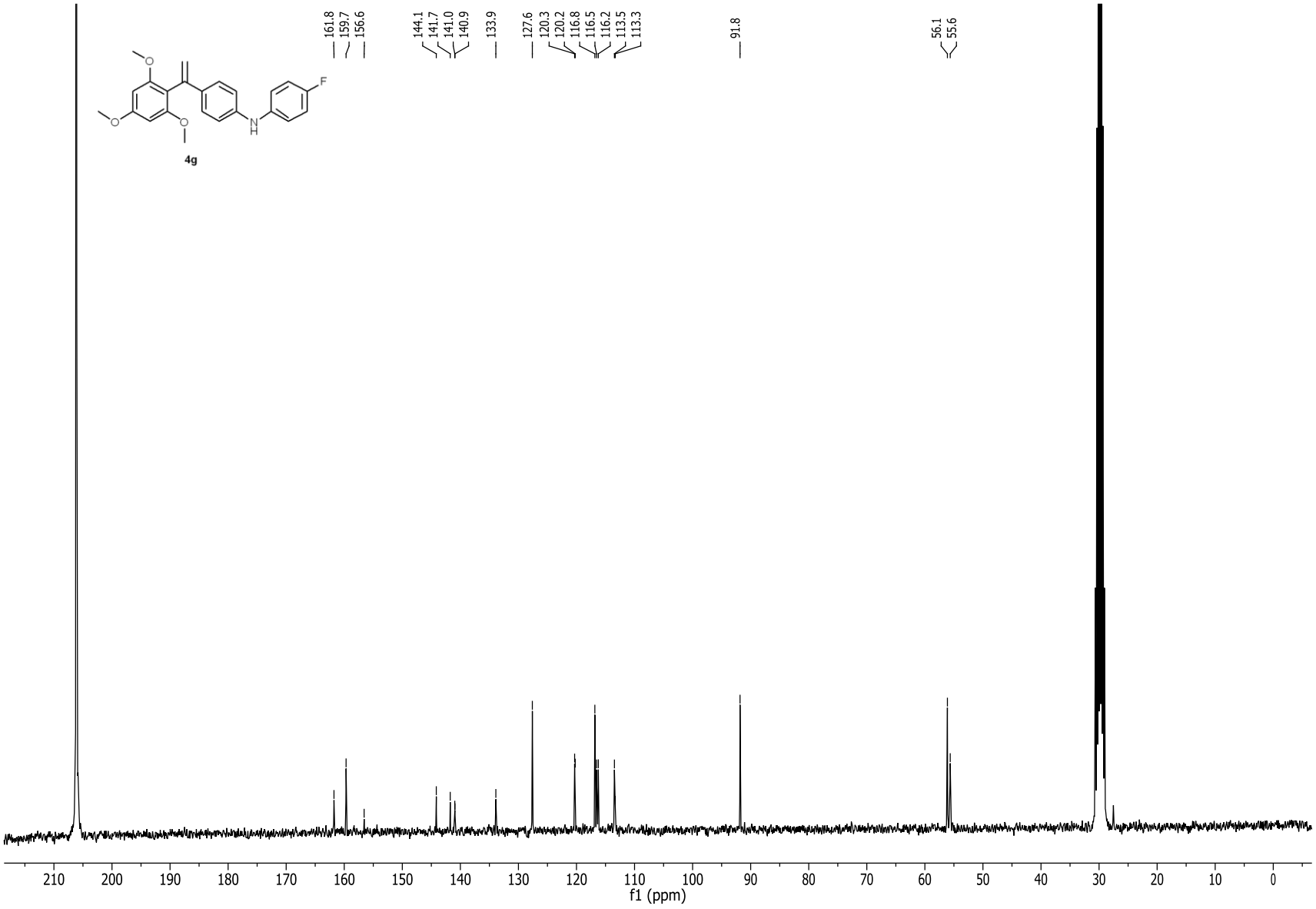

7.24
7.24
7.22
7.22
7.21
7.04
7.03
7.02
7.02
7.00
6.99
6.98
6.97
6.96
6.86
6.85
6.84
6.83
6.82
6.81
6.74
6.73
6.72
6.72
6.71
6.70
6.69
6.54
6.24
5.48
5.48
5.30
5.30
3.78
3.75
3.74
HN
4e
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


161.8
159.1
155.7
142.9
141.3
137.9
132.8
131.3
128.0
122.0
121.9
119.3
115.3
115.2
113.6
92.0
56.3
55.7
55.6
HN
4e
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

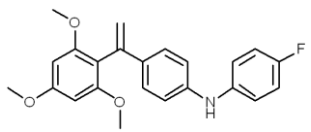

7.16
7.13
7.06
7.04
6.83
6.81
6.73
6.70
6.63
6.29
5.73
5.73
4.88
4.87
3.84
3.67
2.23
2.18
NH
4f
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


4f
161.73
159.69
145.12
142.28
141.84
136.72
133.22
131.56
127.46
127.03
123.56
121.17
116.87
113.09
91.83
56.12
55.63
21.16
17.73
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

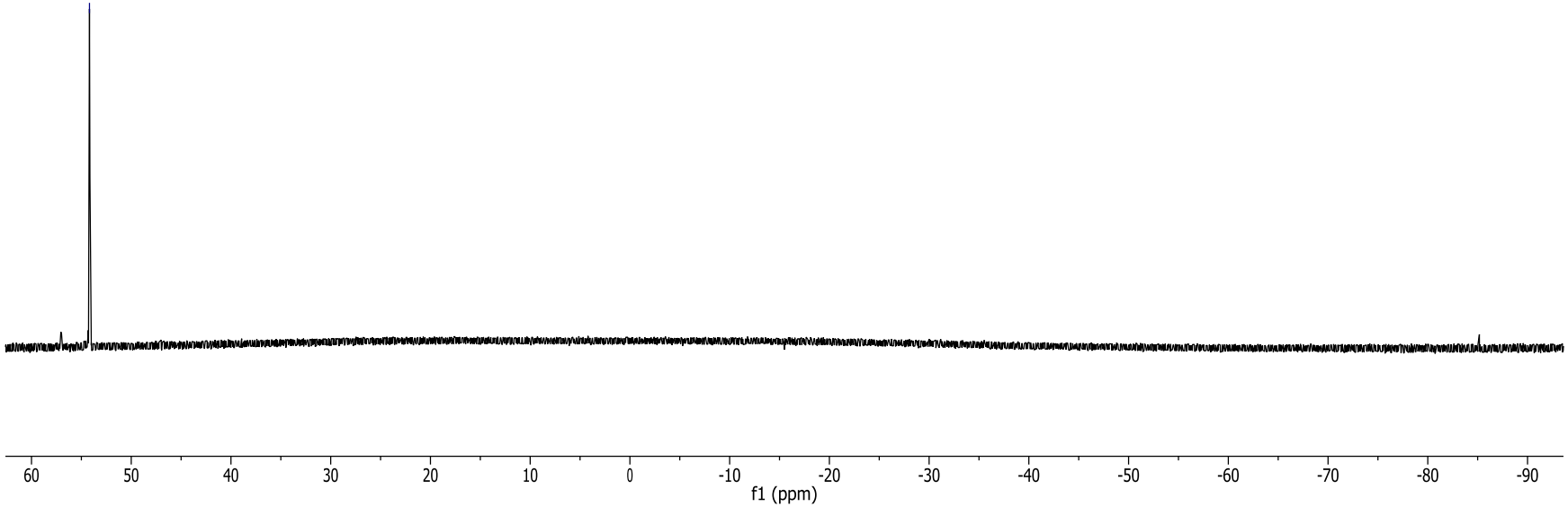

7.32
7.19
7.18
7.18
7.16
7.15
7.14
7.13
7.12
7.11
7.10
7.09
7.06
7.06
7.05
7.04
7.03
7.02
7.01
7.00
6.99
6.98
6.97
6.96
6.95
6.94
6.93
6.92
6.91
6.29
5.74
5.74
4.90
4.90
3.84
3.67
O
O
O
N
H
F
4g
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

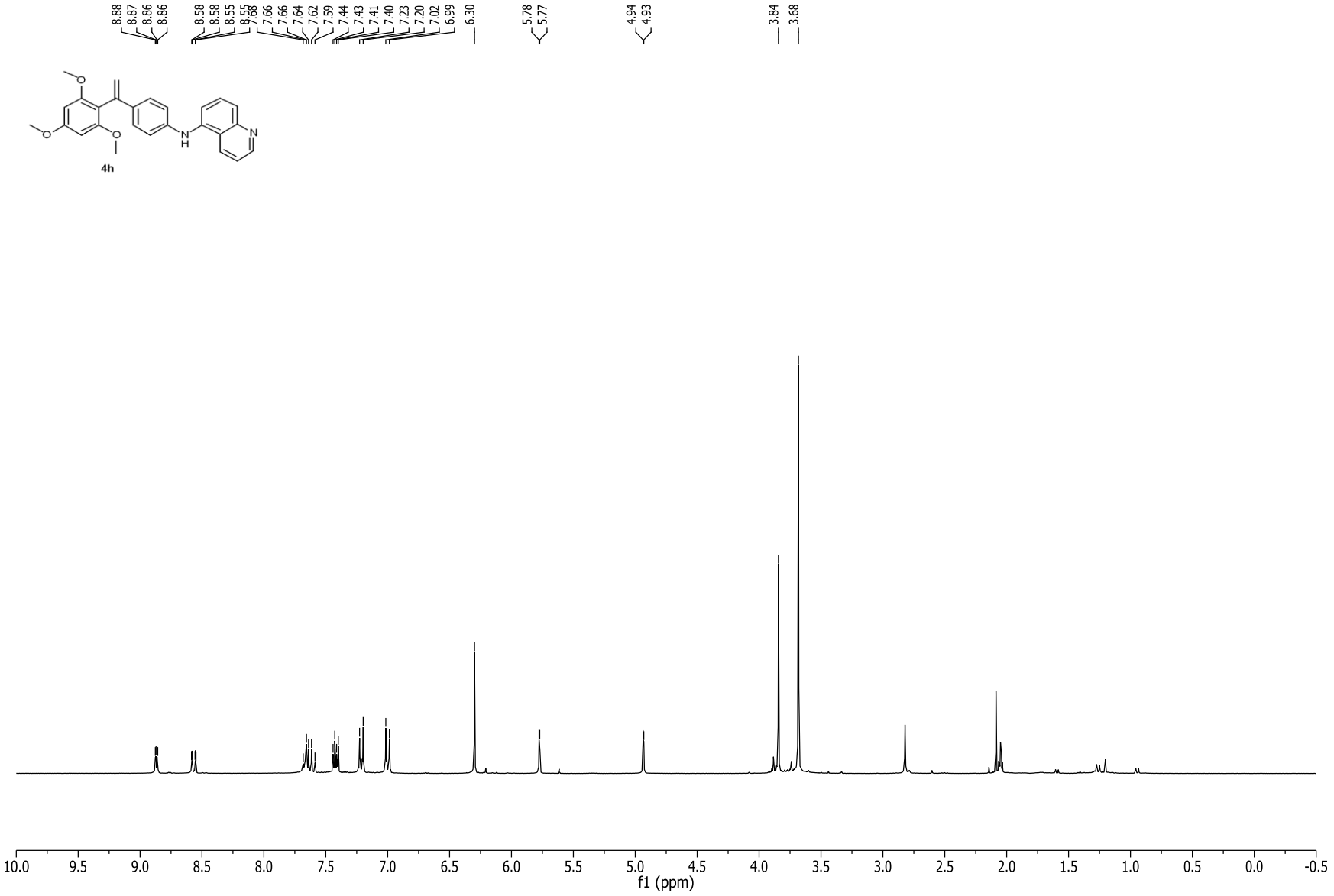

161.8
159.7
156.6
144.1
141.7
141.0
140.9
133.9
127.6
120.3
120.2
116.8
116.5
116.2
113.5
113.3
91.8
56.1
55.6
O
O
O
N
H
F
4g
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

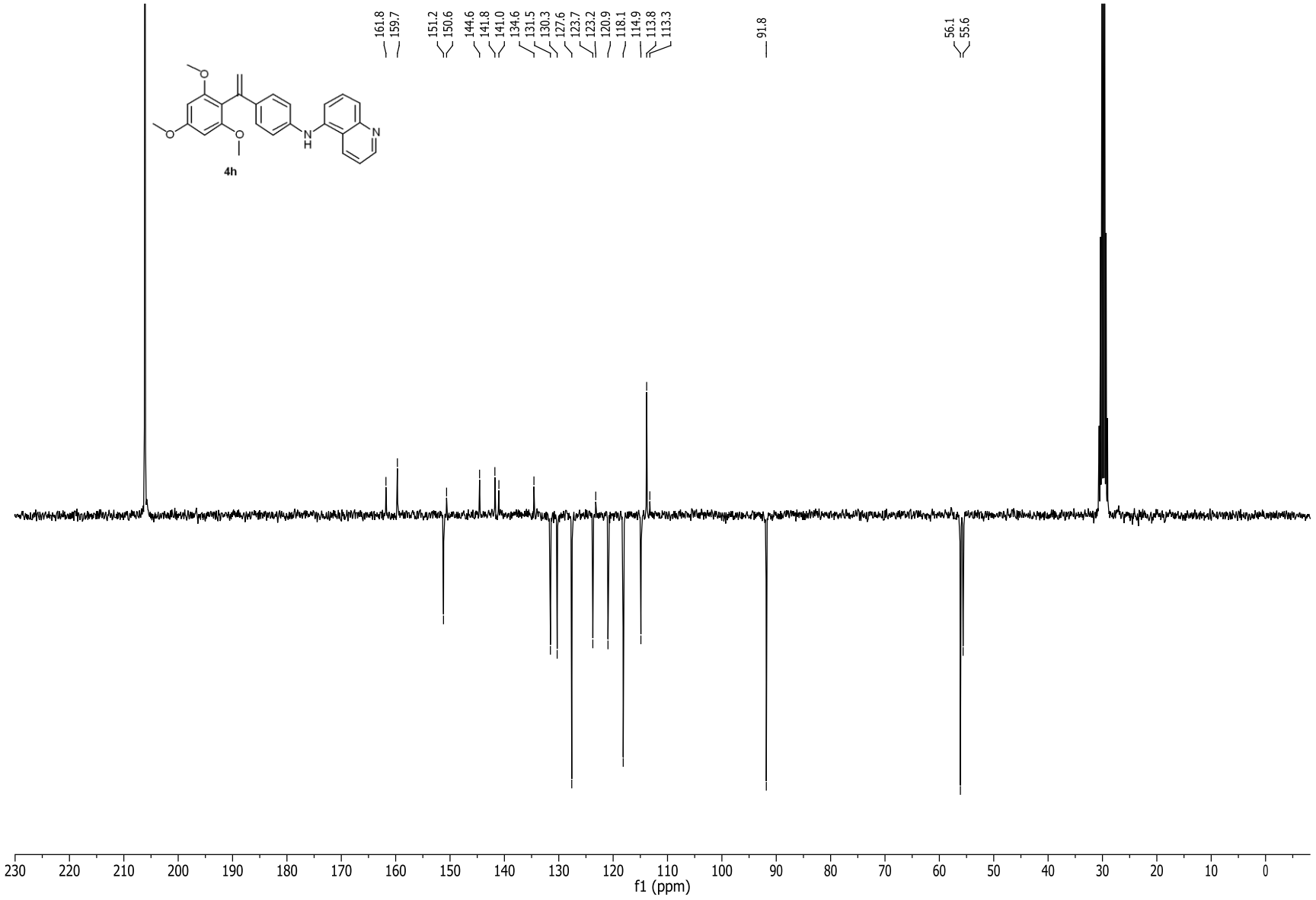
54.19

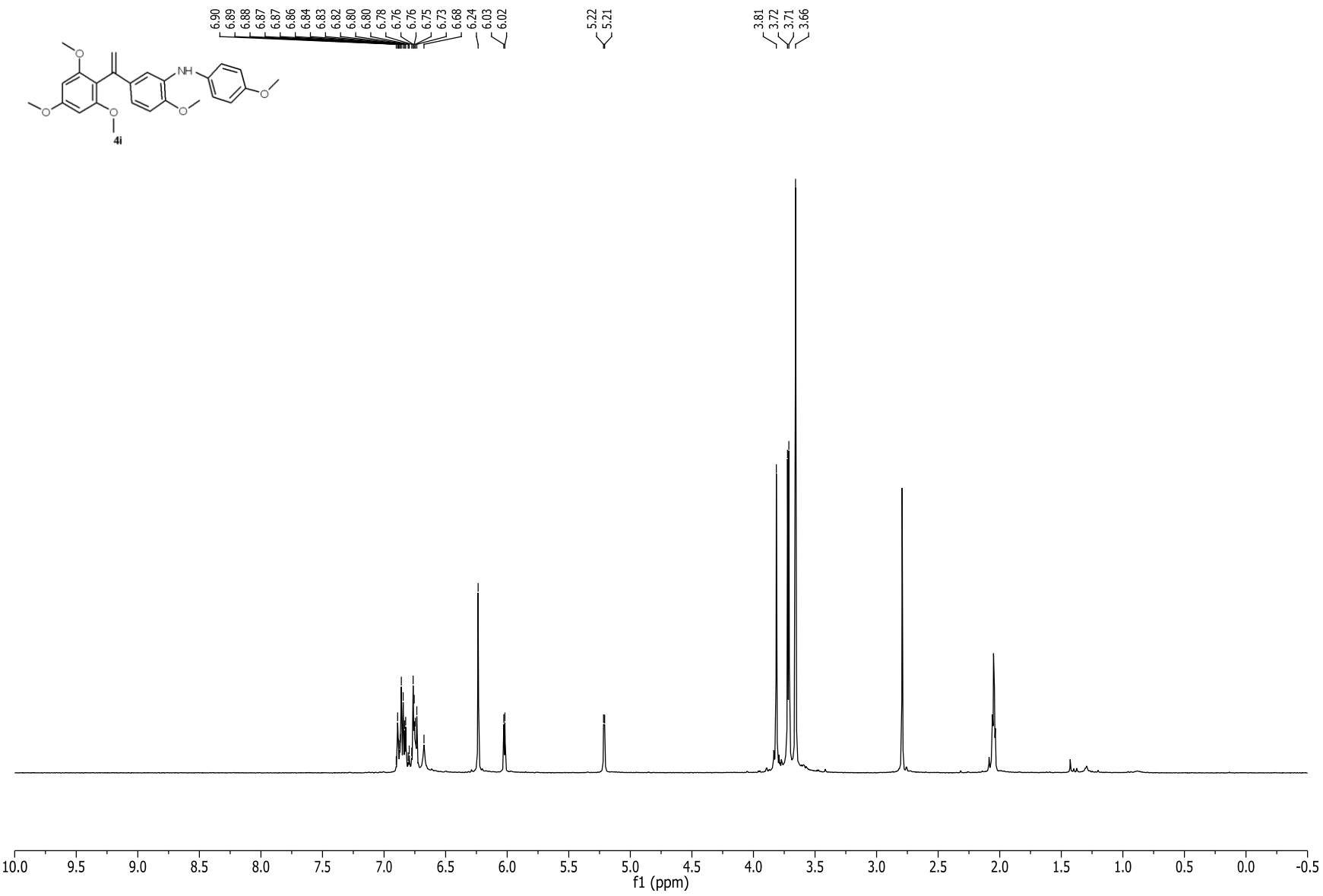


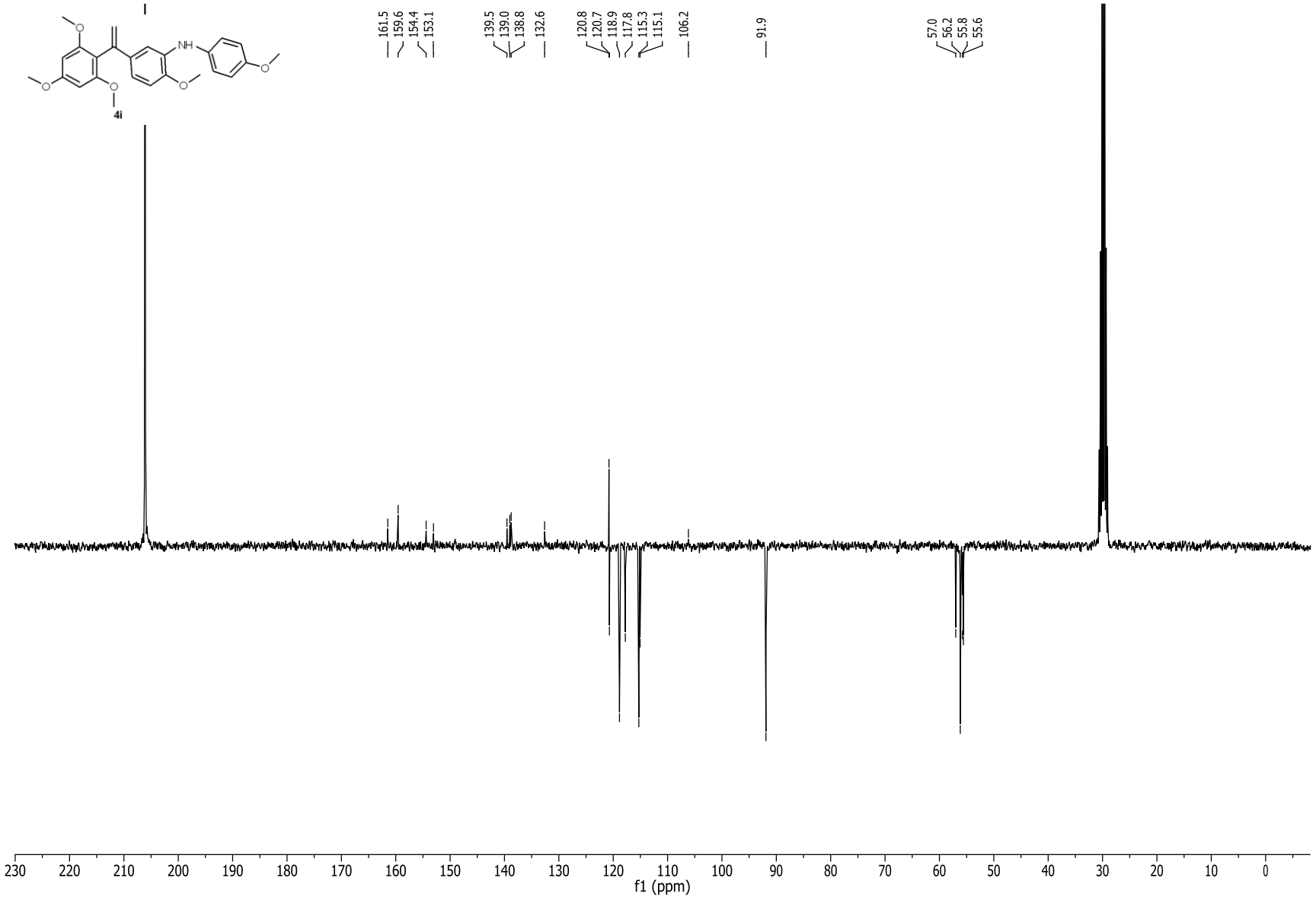
4g

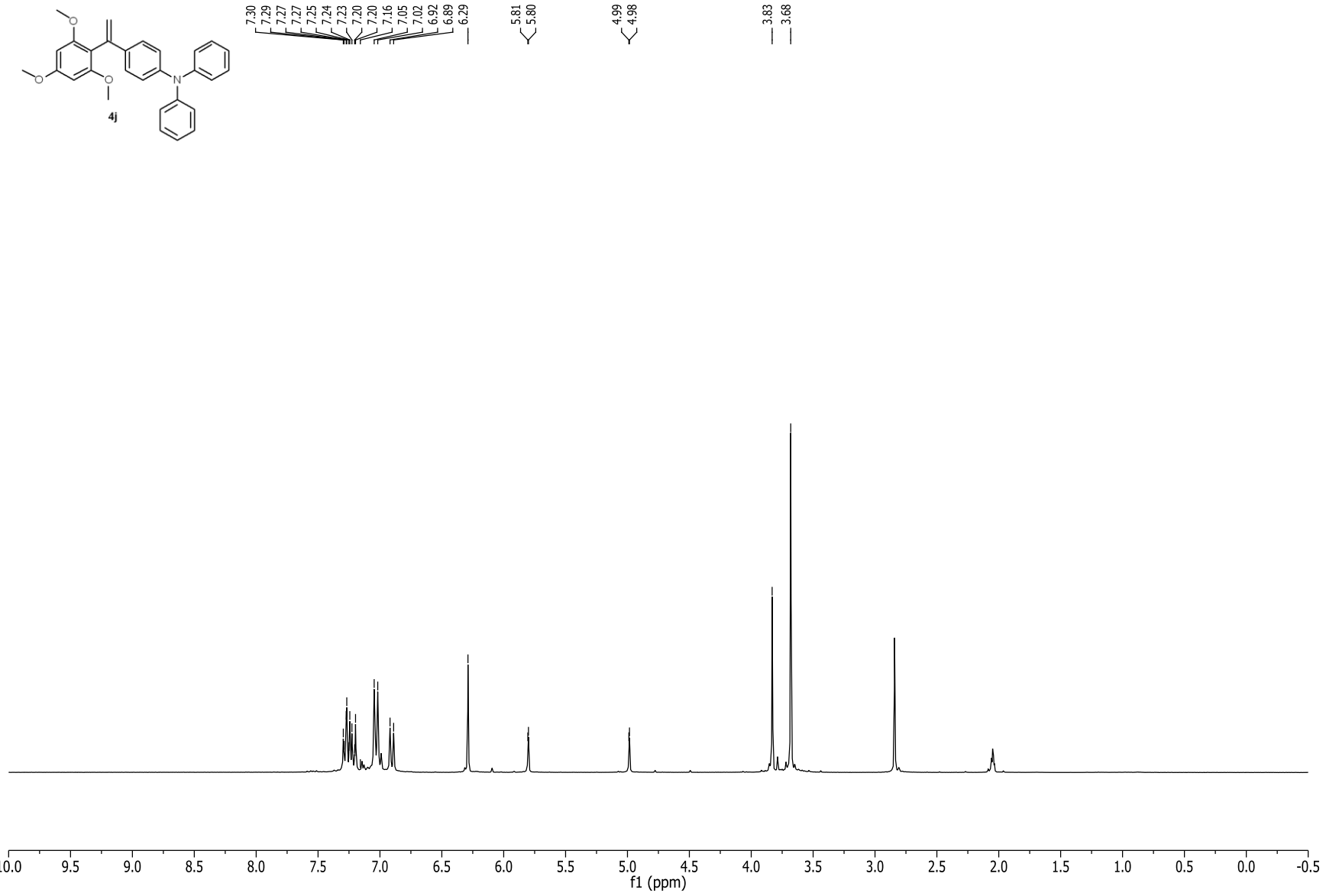


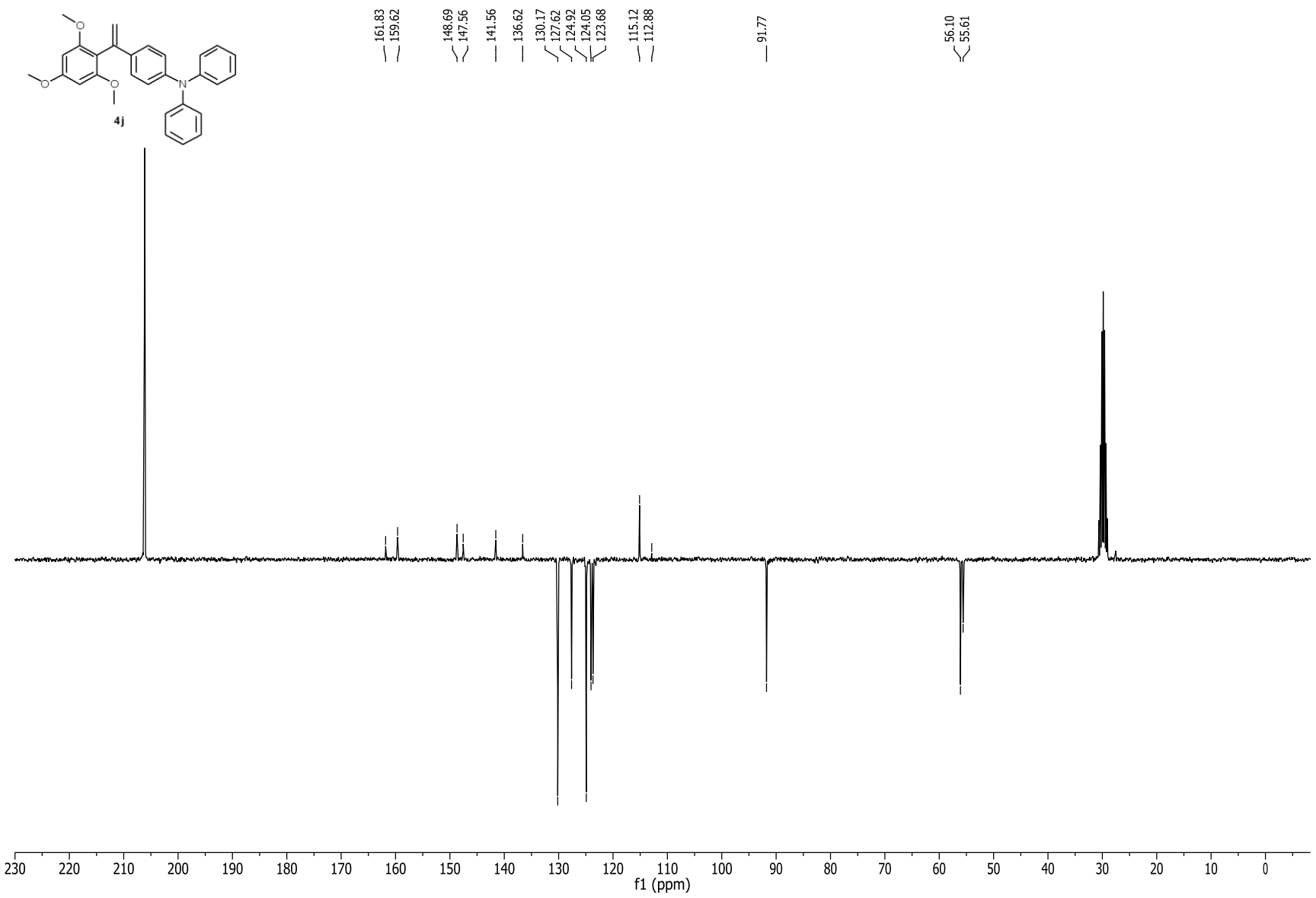

8.88
8.87
8.86
8.86
8.58
8.58
8.55
8.55
7.68
7.66
7.66
7.64
7.62
7.59
7.44
7.43
7.41
7.40
7.23
7.20
7.02
6.99
6.30
5.78
5.77
4.94
4.93
3.84
3.68
O
O
O
N
H
N
4h
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

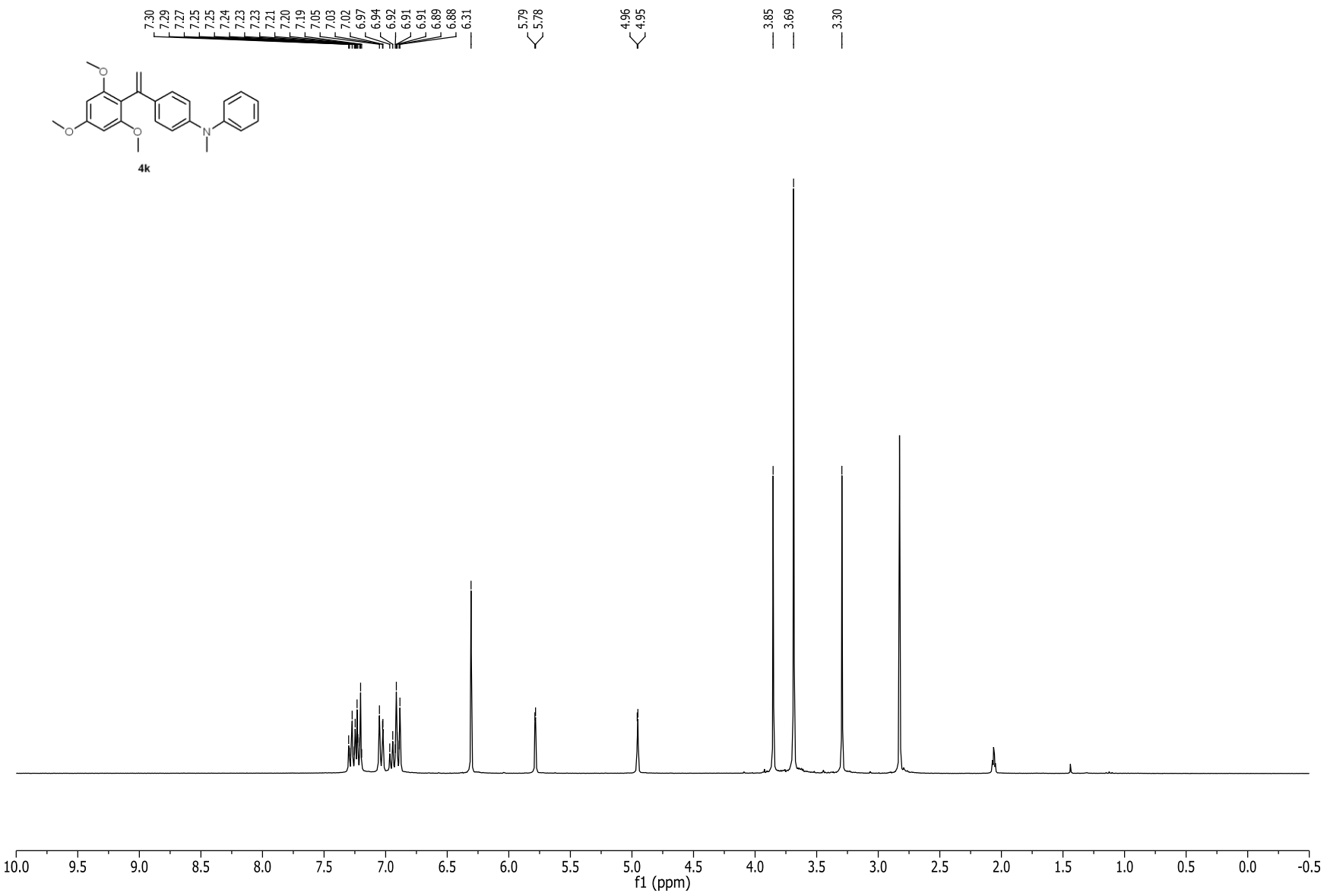

161.8
159.7
151.2
150.6
144.6
141.8
141.0
134.6
131.5
130.3
127.6
123.7
123.2
120.9
118.1
114.9
113.8
113.3
91.8
56.1
55.6
4h
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

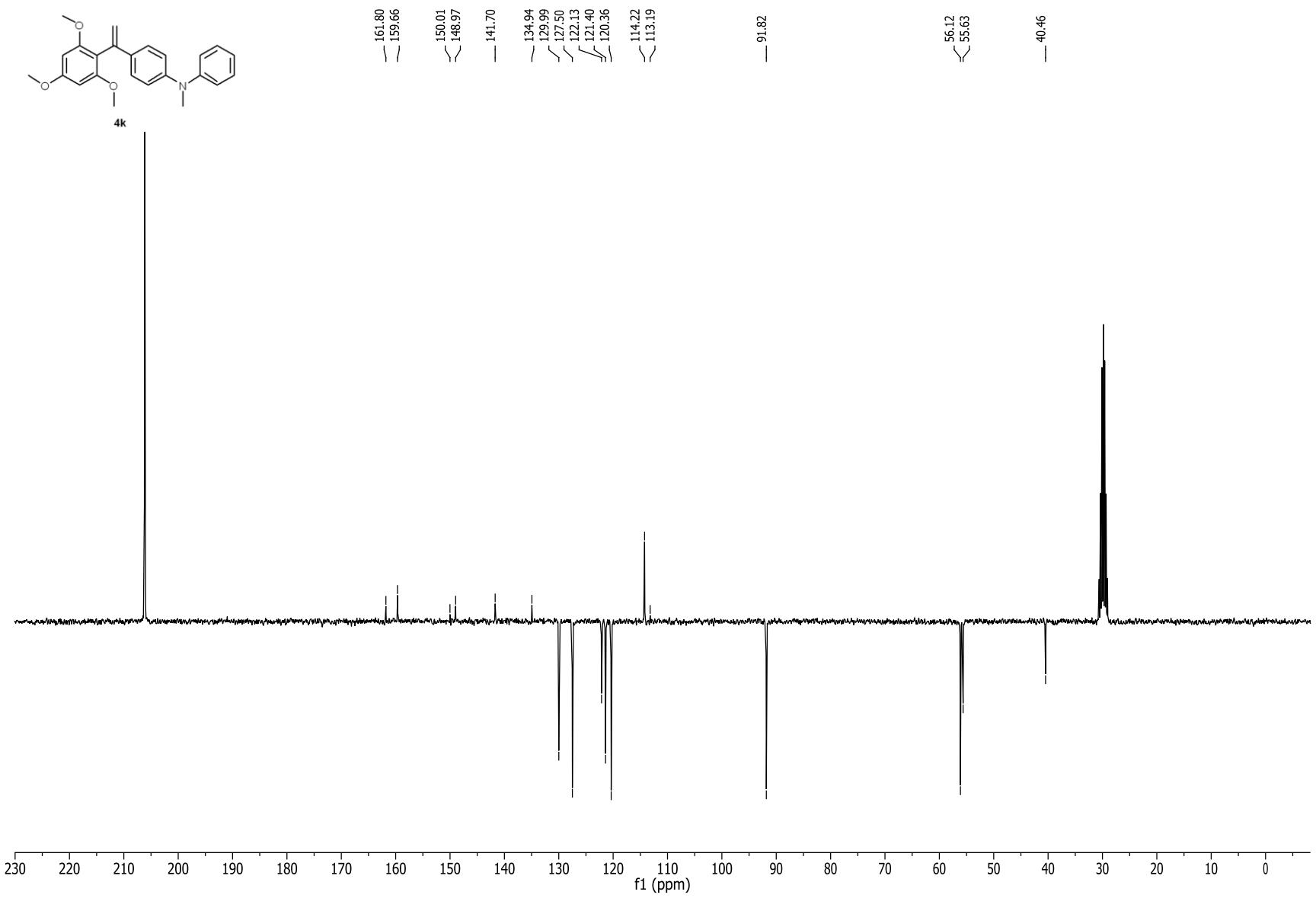

6.90
6.89
6.88
6.87
6.87
6.86
6.84
6.83
6.82
6.80
6.80
6.78
6.76
6.76
6.75
6.73
6.68
6.24
6.03
6.02
5.22
5.21
3.81
3.72
3.71
3.66
NH
4i
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

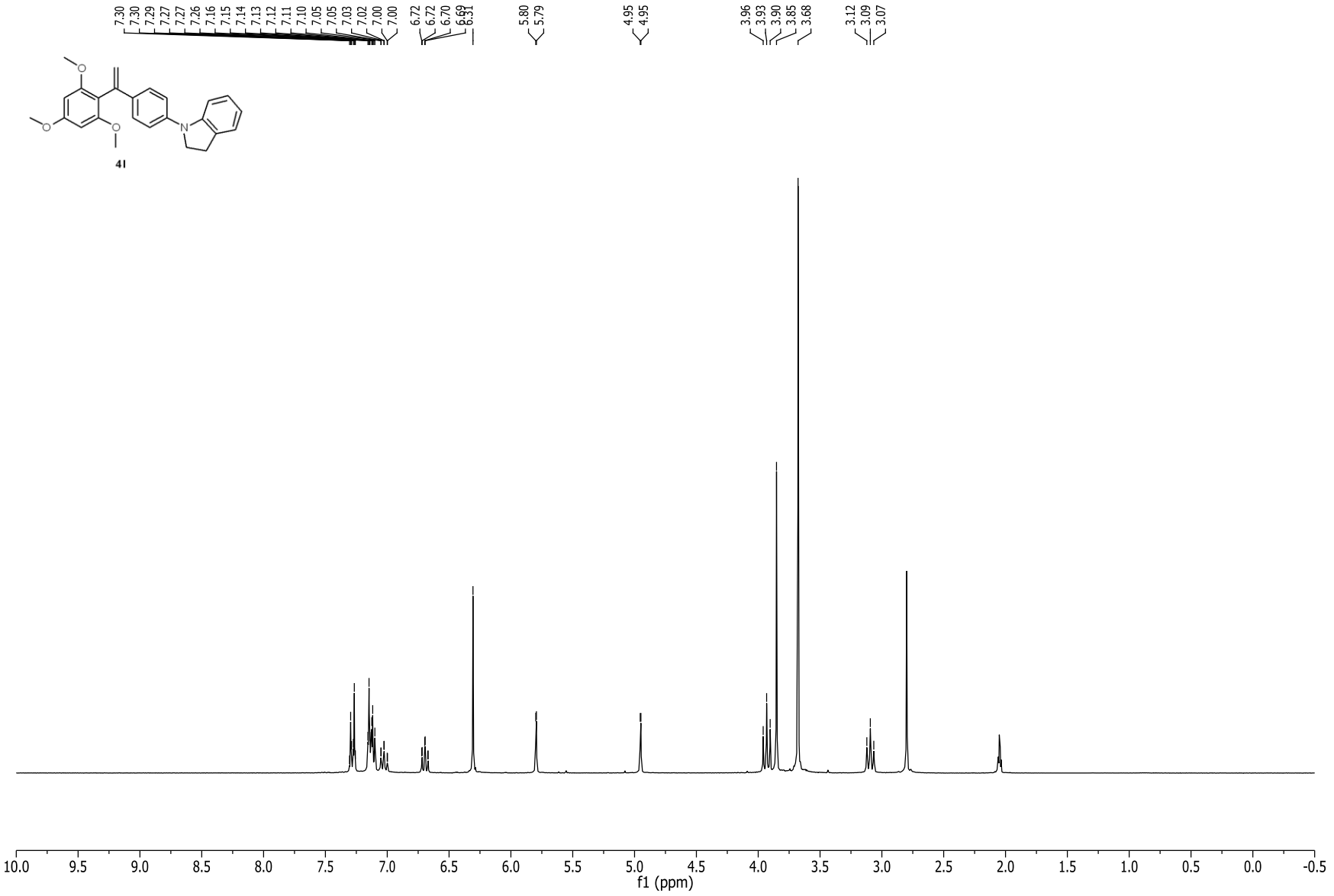

4i
161.5
159.6
154.4
153.1
139.5
139.0
138.8
132.6
120.8
120.7
118.9
117.8
115.3
115.1
106.2
91.9
57.0
56.2
55.8
55.6
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

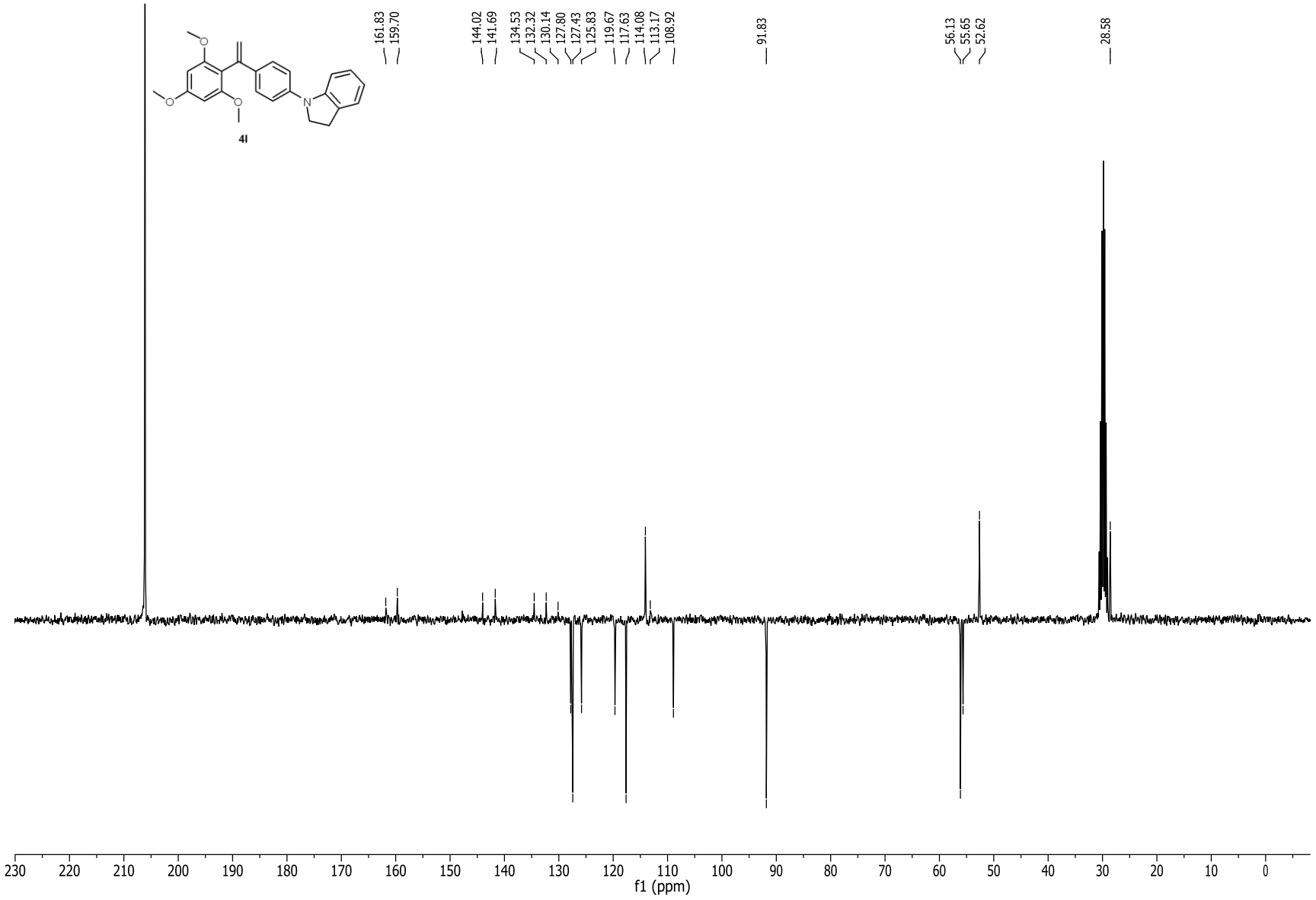

4j
7.30
7.29
7.27
7.27
7.25
7.24
7.23
7.20
7.20
7.16
7.05
7.02
6.92
6.89
6.29
5.81
5.80
4.99
4.98
3.83
3.68
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

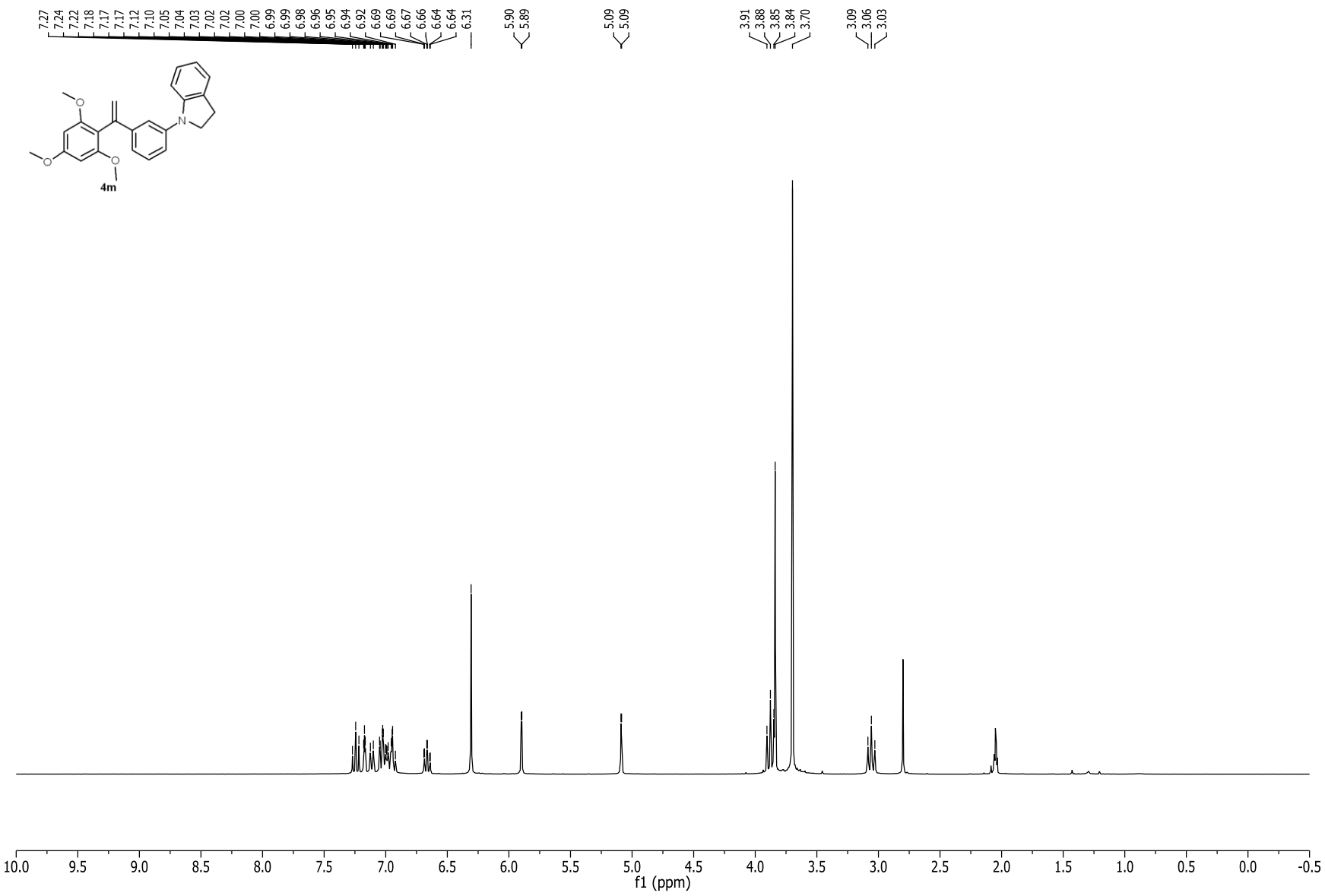

4j
161.83
159.62
148.69
147.56
141.56
136.62
130.17
127.62
124.92
124.05
123.68
115.12
112.88
91.77
56.10
55.61
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

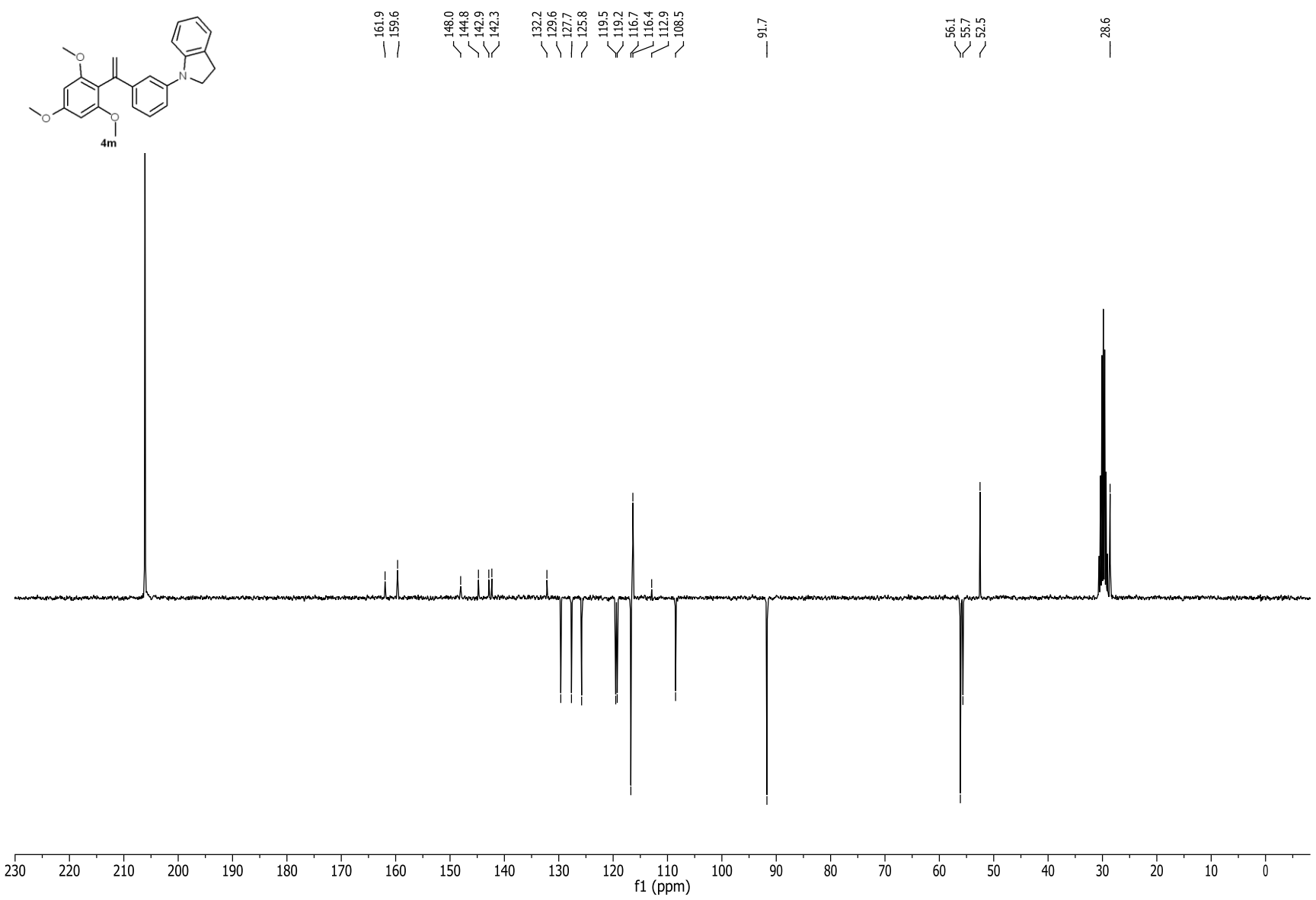

7.30
7.29
7.27
7.25
7.25
7.24
7.23
7.23
7.21
7.20
7.19
7.05
7.03
7.02
6.97
6.94
6.92
6.91
6.91
6.89
6.88
6.31
5.79
5.78
4.96
4.95
3.85
3.69
3.30
4k
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

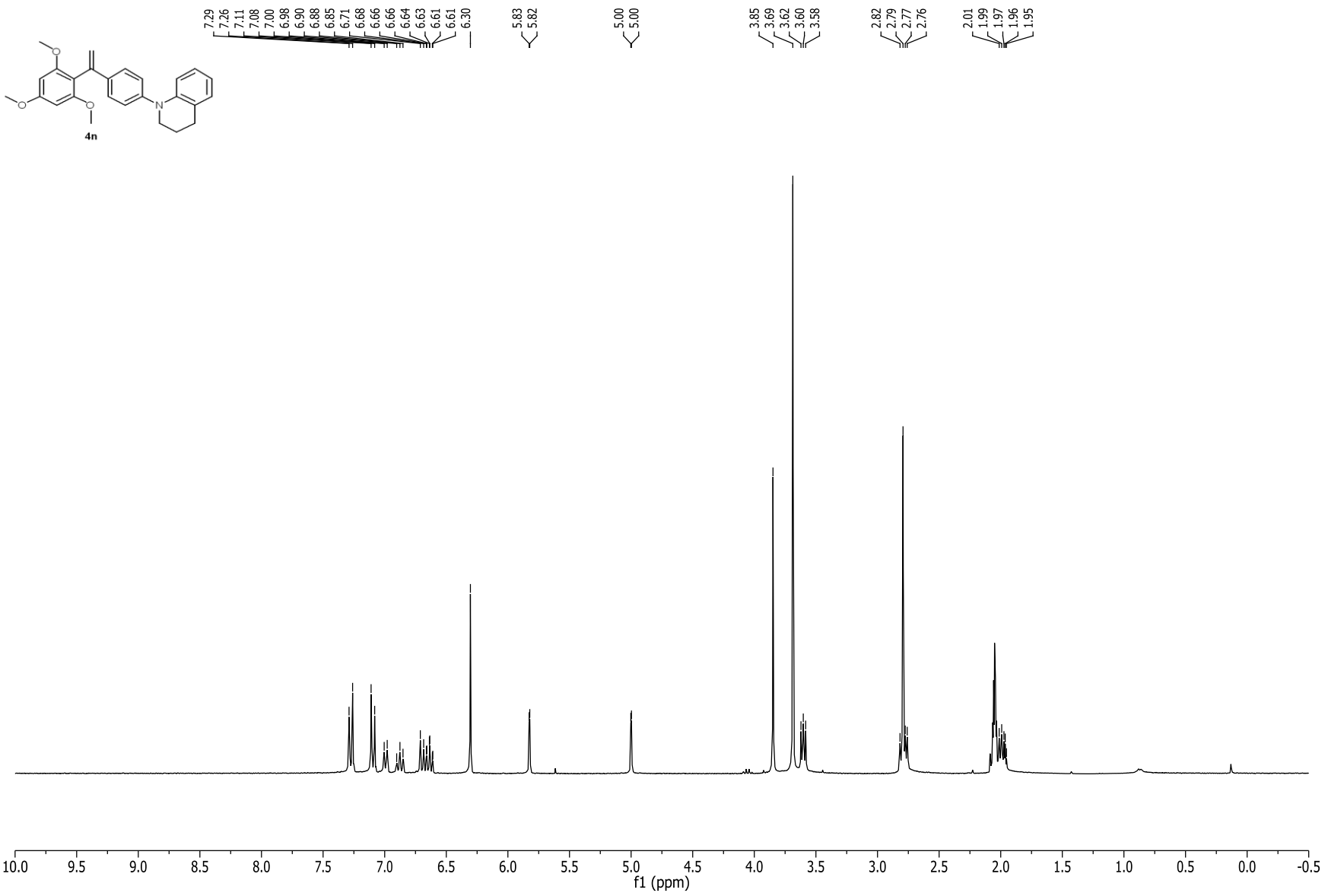

4k
161.80
159.66
150.01
148.97
141.70
134.94
129.99
127.50
122.13
121.40
120.36
114.22
113.19
91.82
56.12
55.63
40.46
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

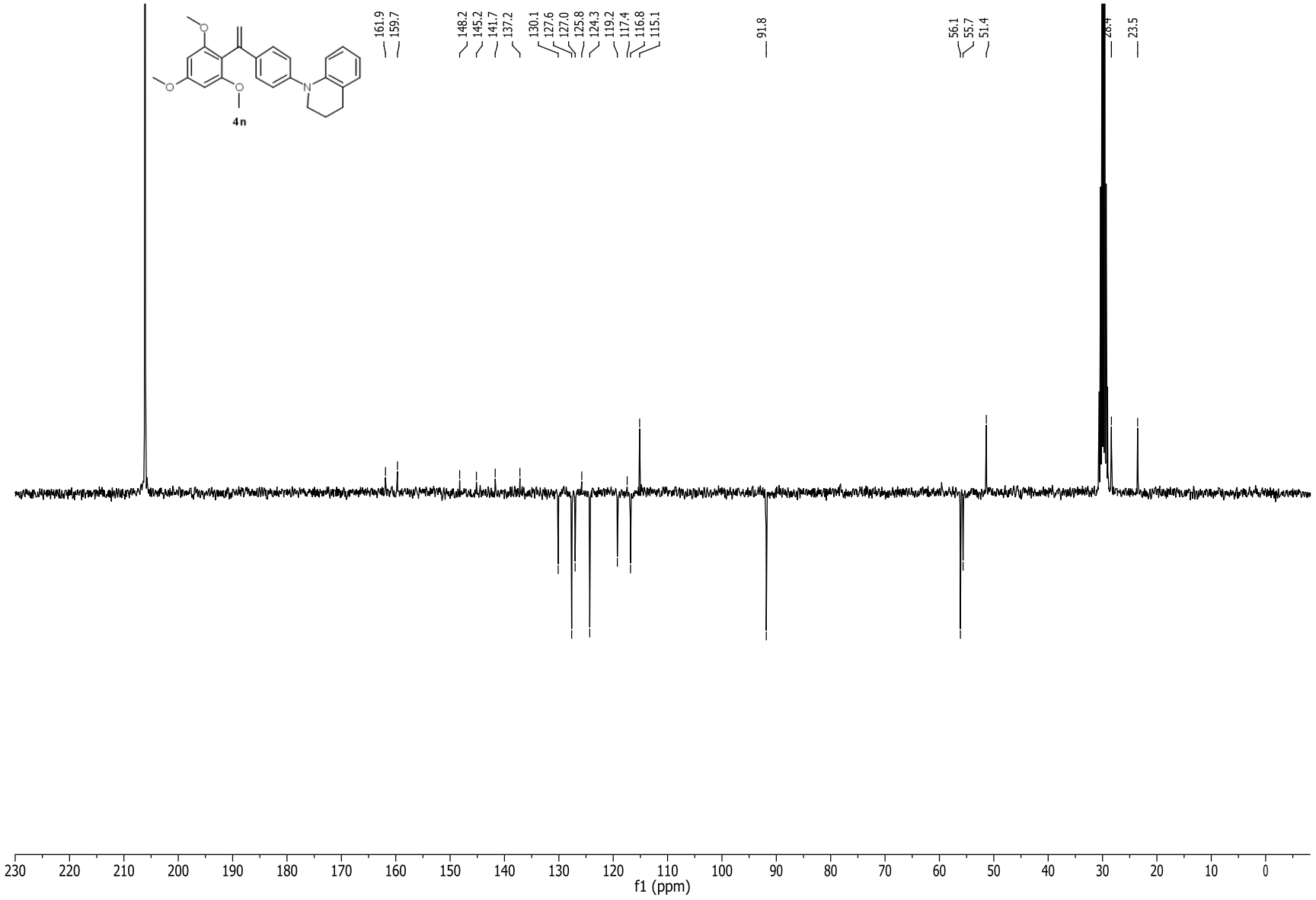

7.30
7.30
7.29
7.27
7.27
7.26
7.16
7.15
7.14
7.13
7.12
7.11
7.10
7.05
7.05
7.03
7.02
7.00
7.00
6.72
6.72
6.70
6.69
6.69
5.80
5.79
4.95
4.95
3.96
3.93
3.90
3.85
3.68
3.12
3.09
3.07
O
O
O
N
4l
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

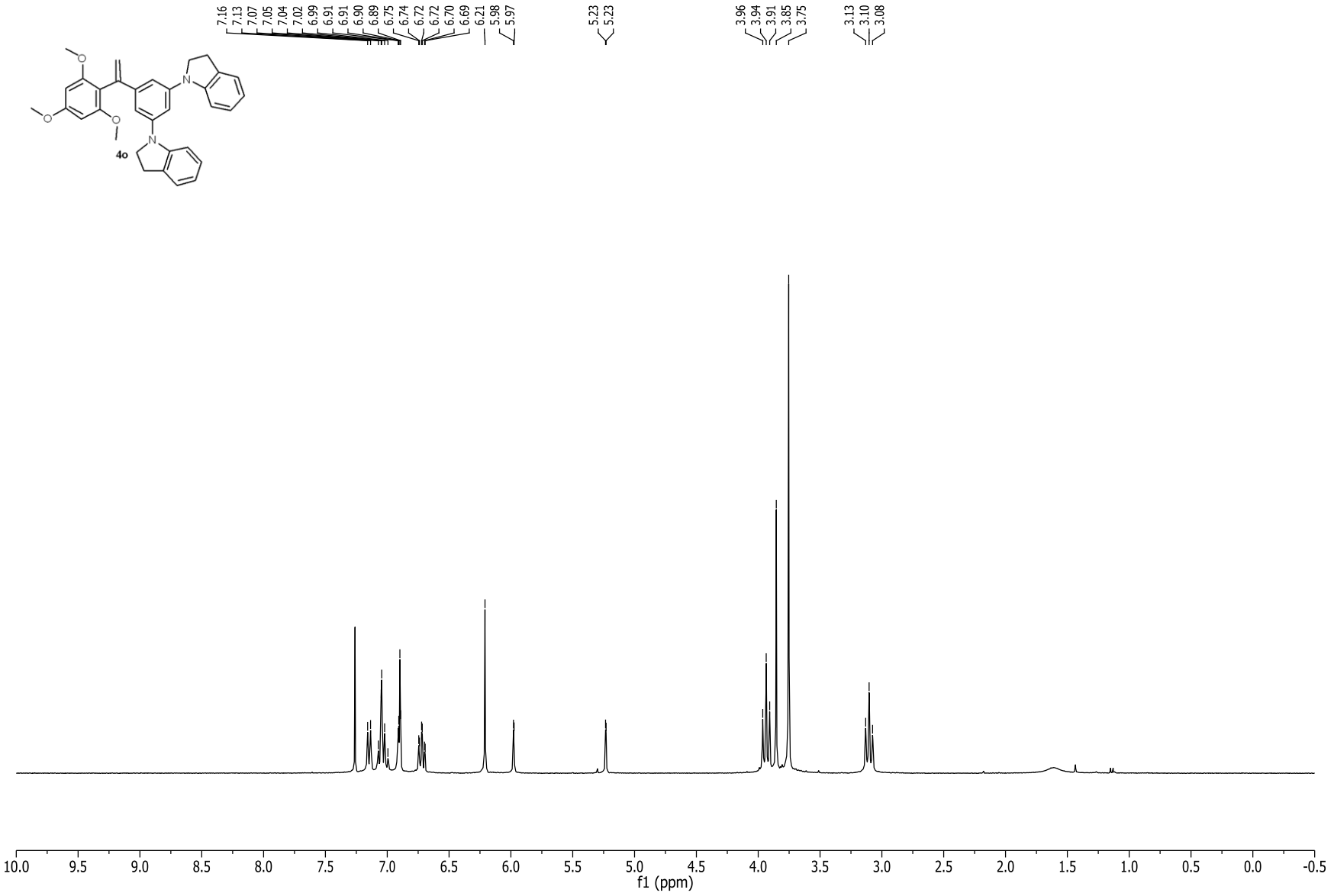

161.83
159.70
144.02
141.69
134.53
132.32
130.14
127.80
127.43
125.83
119.67
117.63
114.08
113.17
108.92
91.83
56.13
55.65
52.62
28.58
4l
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

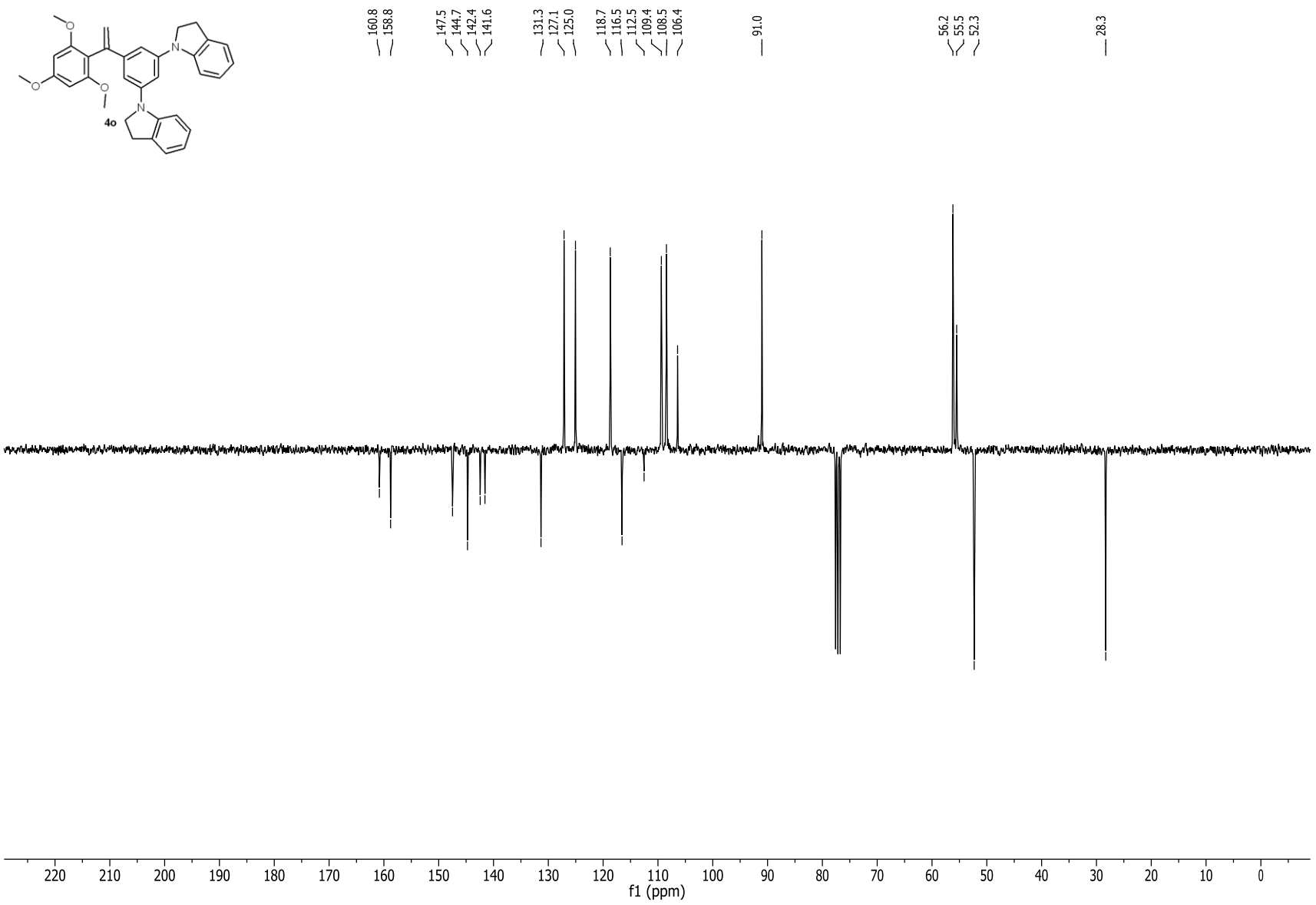

7.27
7.24
7.22
7.18
7.17
7.17
7.12
7.10
7.05
7.04
7.03
7.02
7.02
7.00
7.00
6.99
6.99
6.98
6.96
6.95
6.94
6.92
6.69
6.69
6.67
6.66
6.64
6.64
6.31
5.90
5.89
5.09
5.09
3.91
3.88
3.85
3.84
3.70
3.09
3.06
3.03
4m
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

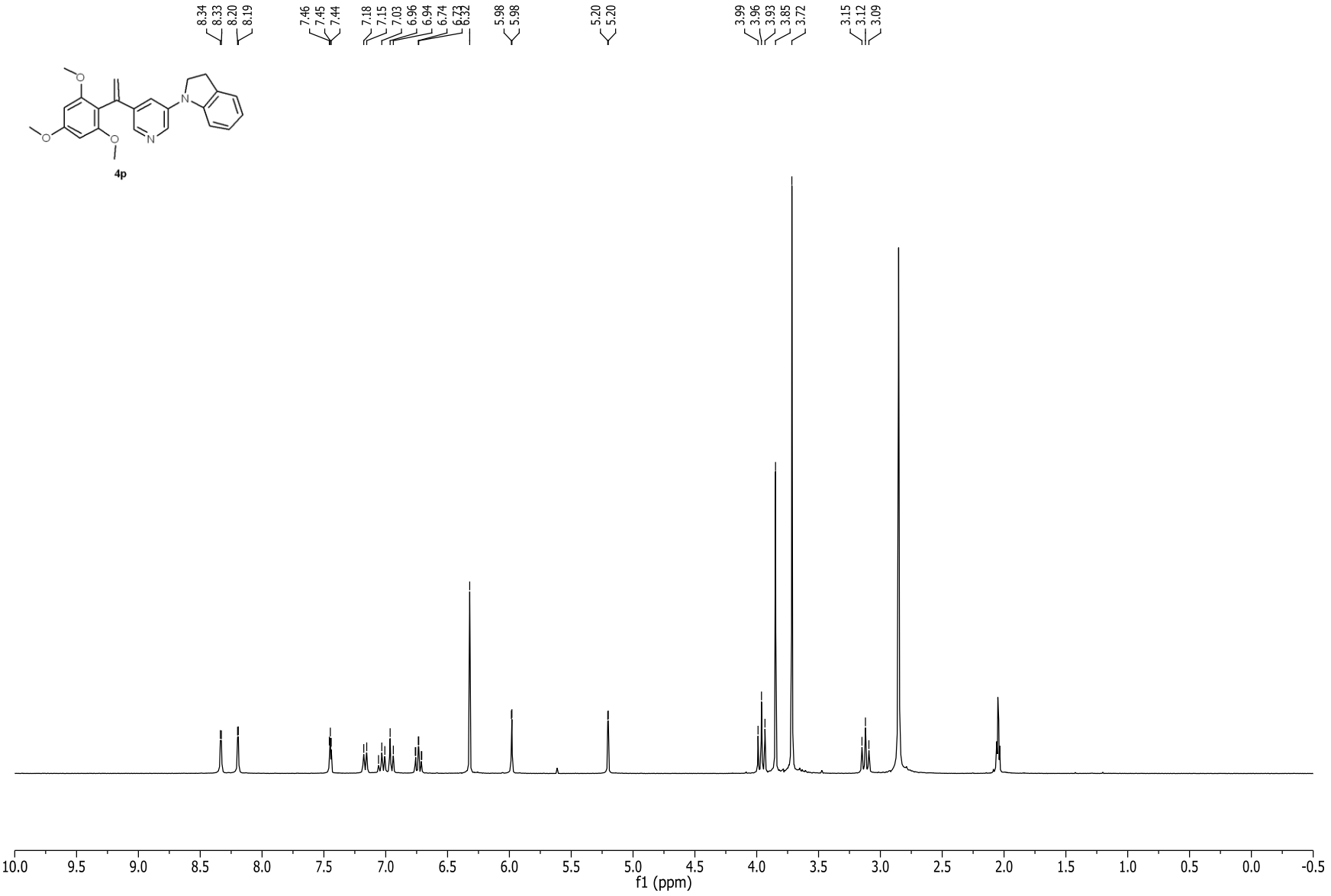

4m
161.9
159.6
148.0
144.8
142.9
142.3
132.2
129.6
127.7
125.8
119.5
119.2
116.7
116.4
112.9
108.5
91.7
56.1
55.7
52.5
28.6
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

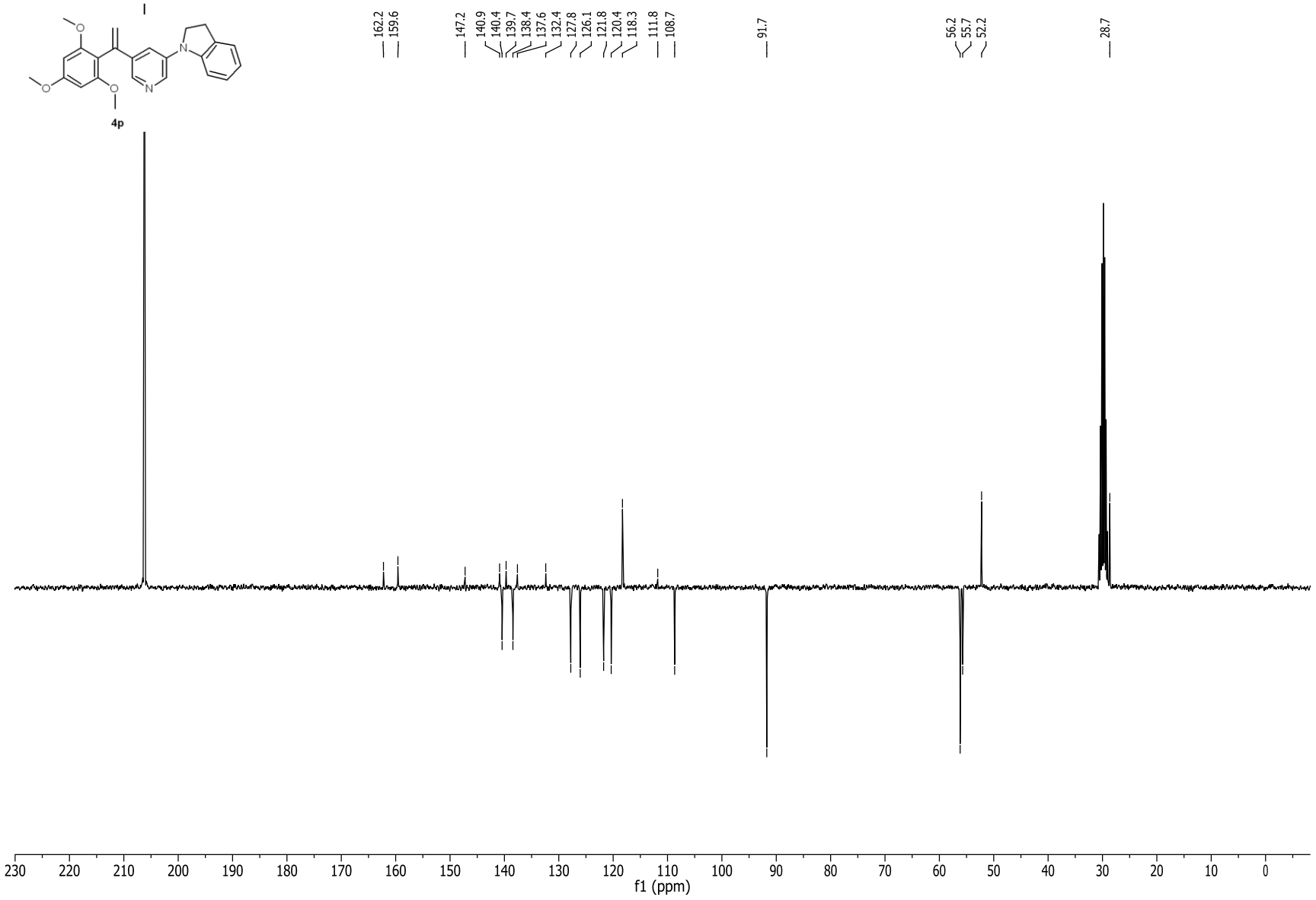

7.29
7.26
7.11
7.08
7.00
6.98
6.90
6.88
6.85
6.71
6.68
6.66
6.66
6.64
6.63
6.61
6.61
6.30
5.83
5.82
5.00
5.00
3.85
3.69
3.62
3.60
3.58
2.82
2.79
2.77
2.76
2.01
1.99
1.97
1.96
1.95
4n
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

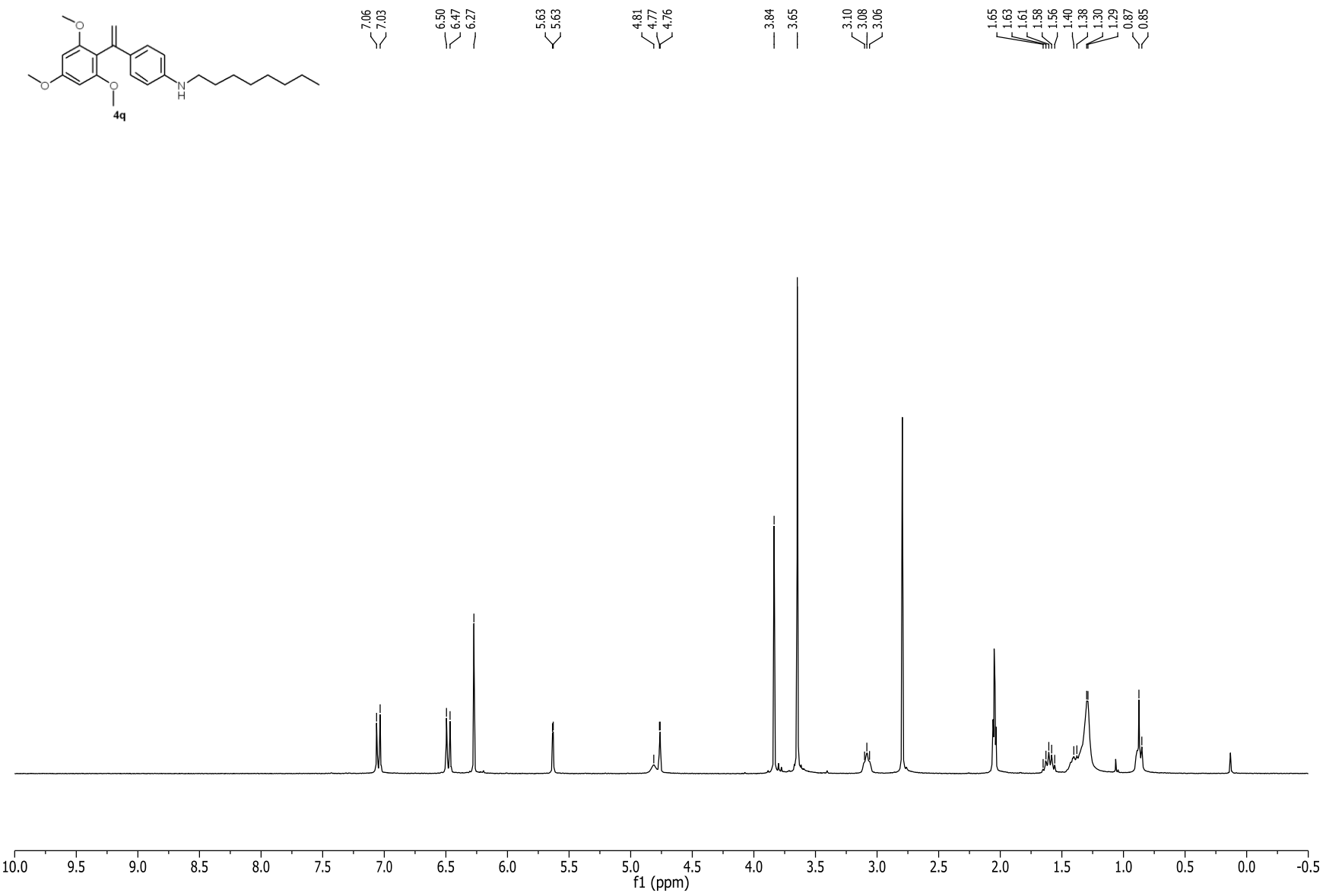

4n
161.9
159.7
148.2
145.2
141.7
137.2
130.1
127.6
127.0
125.8
124.3
119.2
117.4
116.8
115.1
91.8
56.1
55.7
51.4
26.4
23.5
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

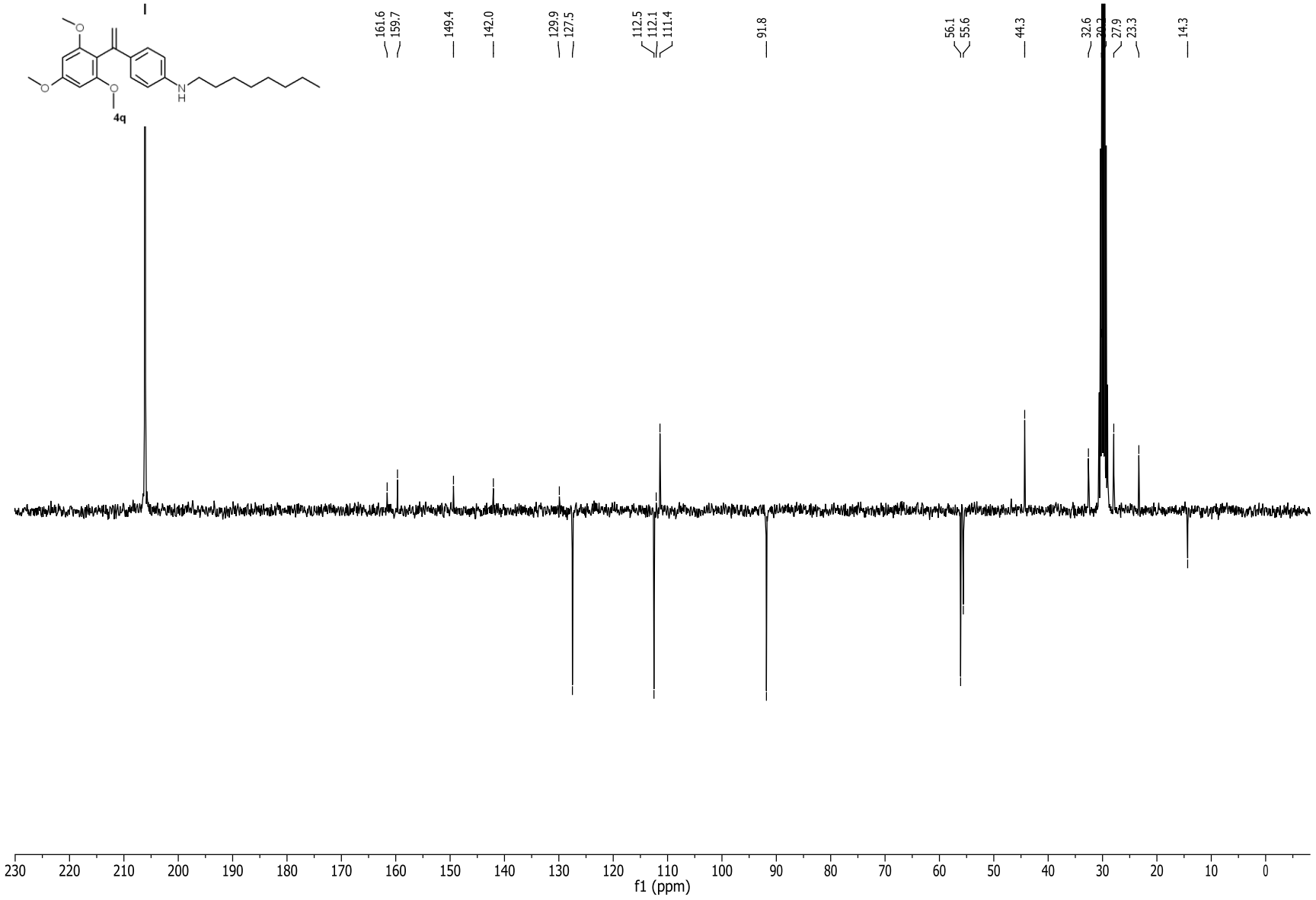

7.16
7.13
7.07
7.05
7.04
7.02
6.99
6.91
6.91
6.90
6.89
6.75
6.74
6.72
6.72
6.70
6.69
6.21
5.98
5.97
5.23
5.23
3.96
3.94
3.91
3.85
3.75
3.13
3.10
3.08
4o
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

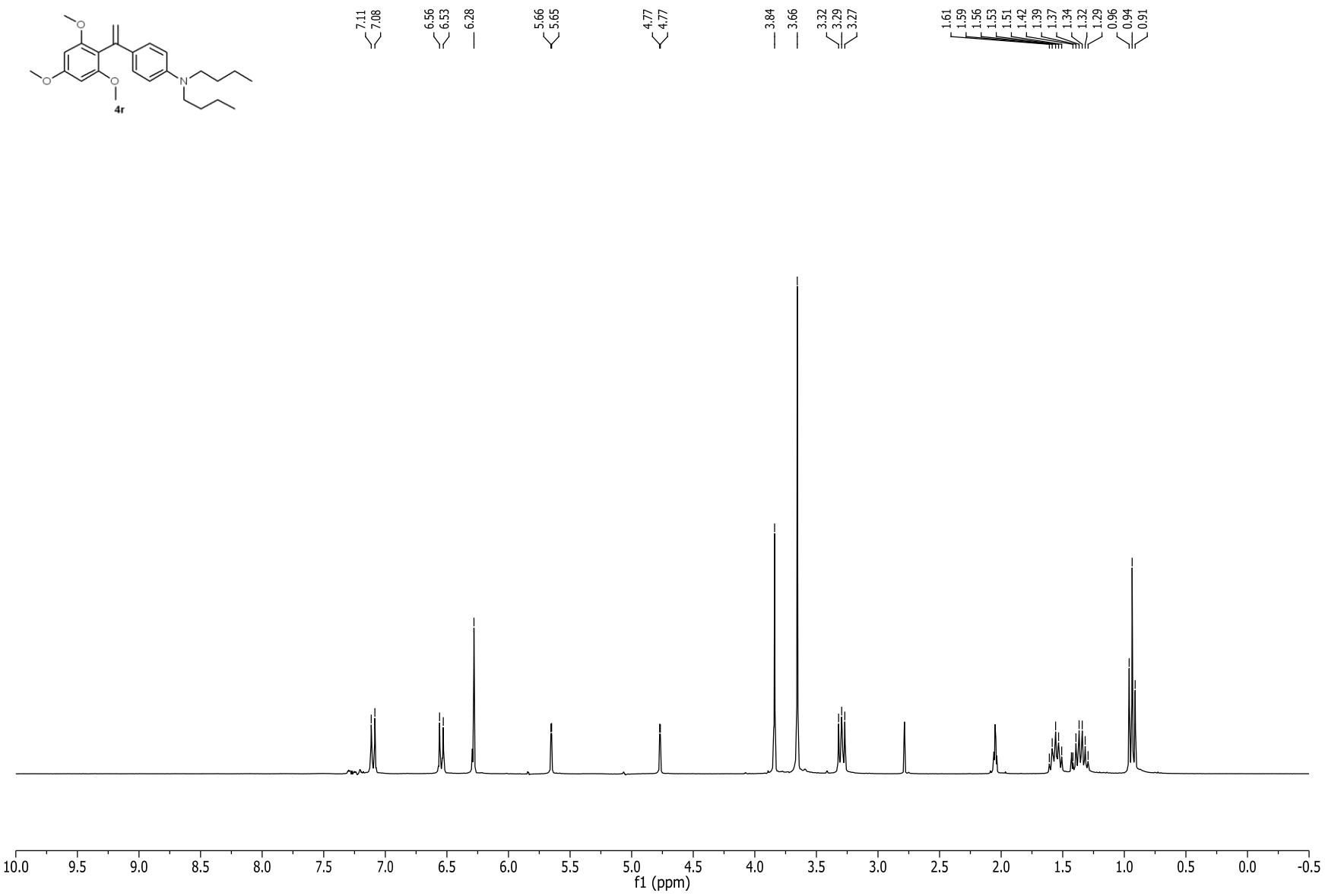

4o
160.8
158.8
147.5
144.7
142.4
141.6
131.3
127.1
125.0
118.7
116.5
112.5
109.4
108.5
106.4
91.0
56.2
55.5
52.3
28.3
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

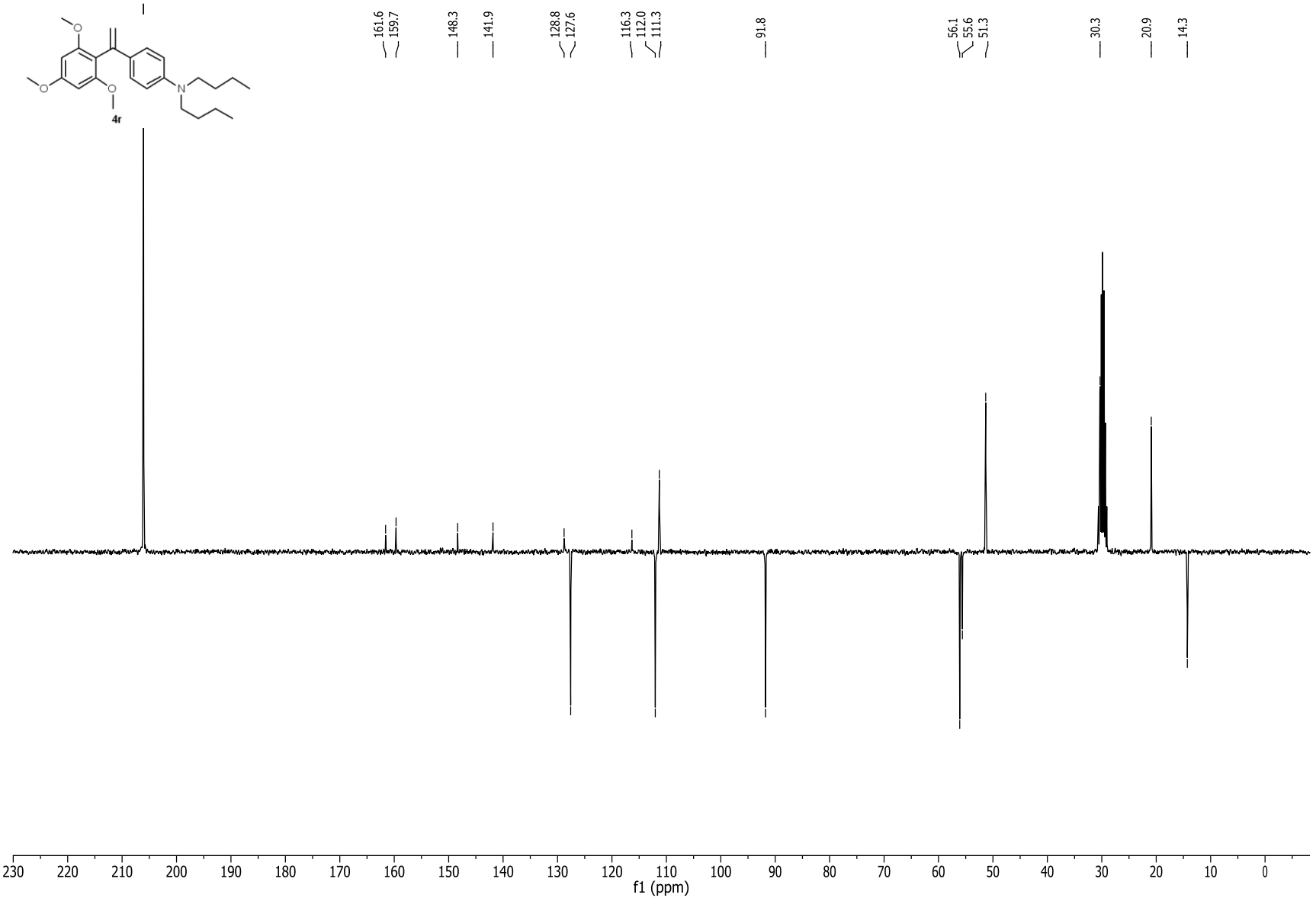

8.34
8.33
8.20
8.19
7.46
7.45
7.44
7.18
7.15
7.03
6.96
6.94
6.74
6.73
6.32
5.98
5.98
5.20
5.20
3.99
3.96
3.93
3.85
3.72
3.15
3.12
3.09
4p
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

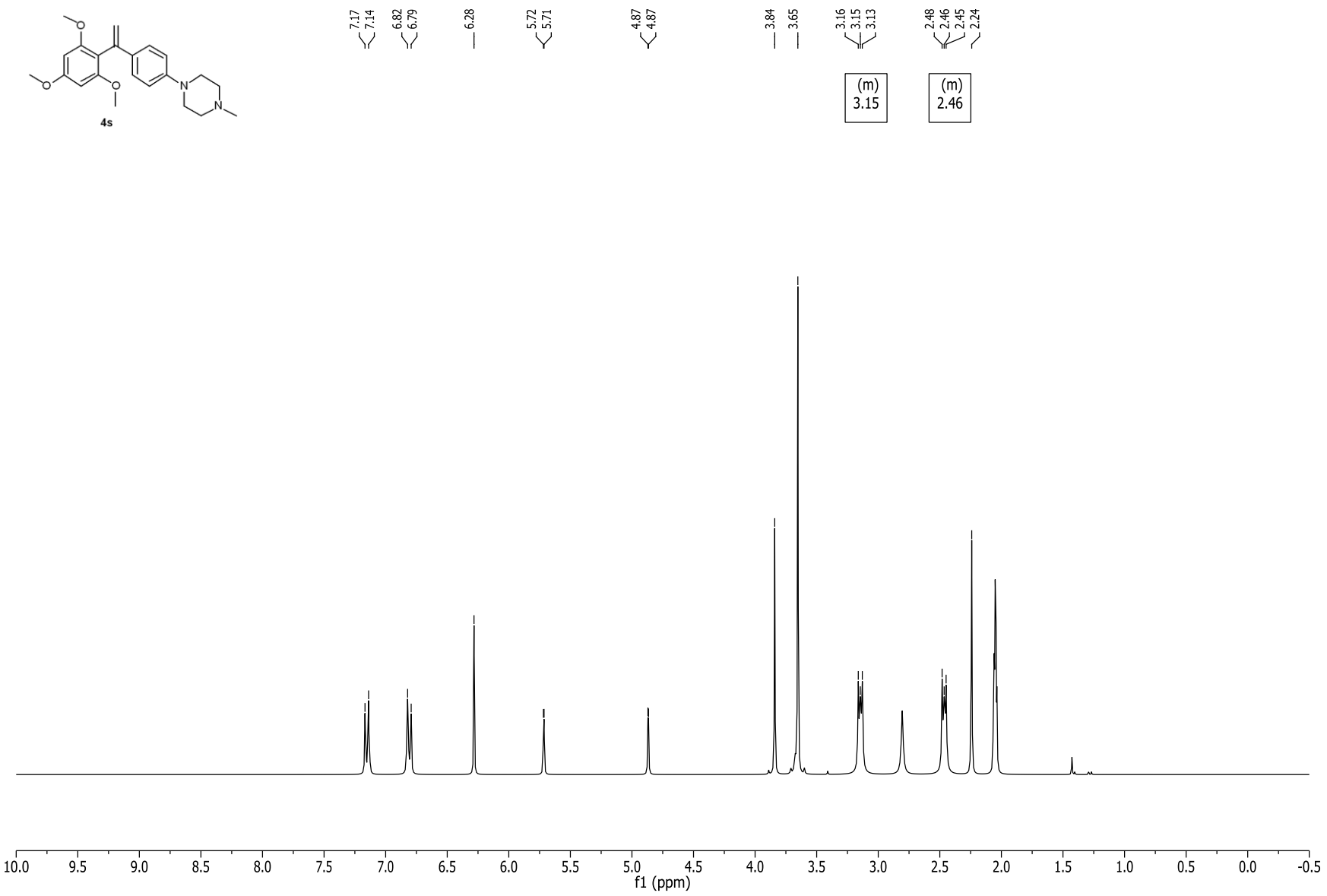

4p
162.2
159.6
147.2
140.9
140.4
139.7
138.4
137.6
132.4
127.8
126.1
121.8
120.4
118.3
111.8
108.7
91.7
56.2
55.7
52.2
28.7
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

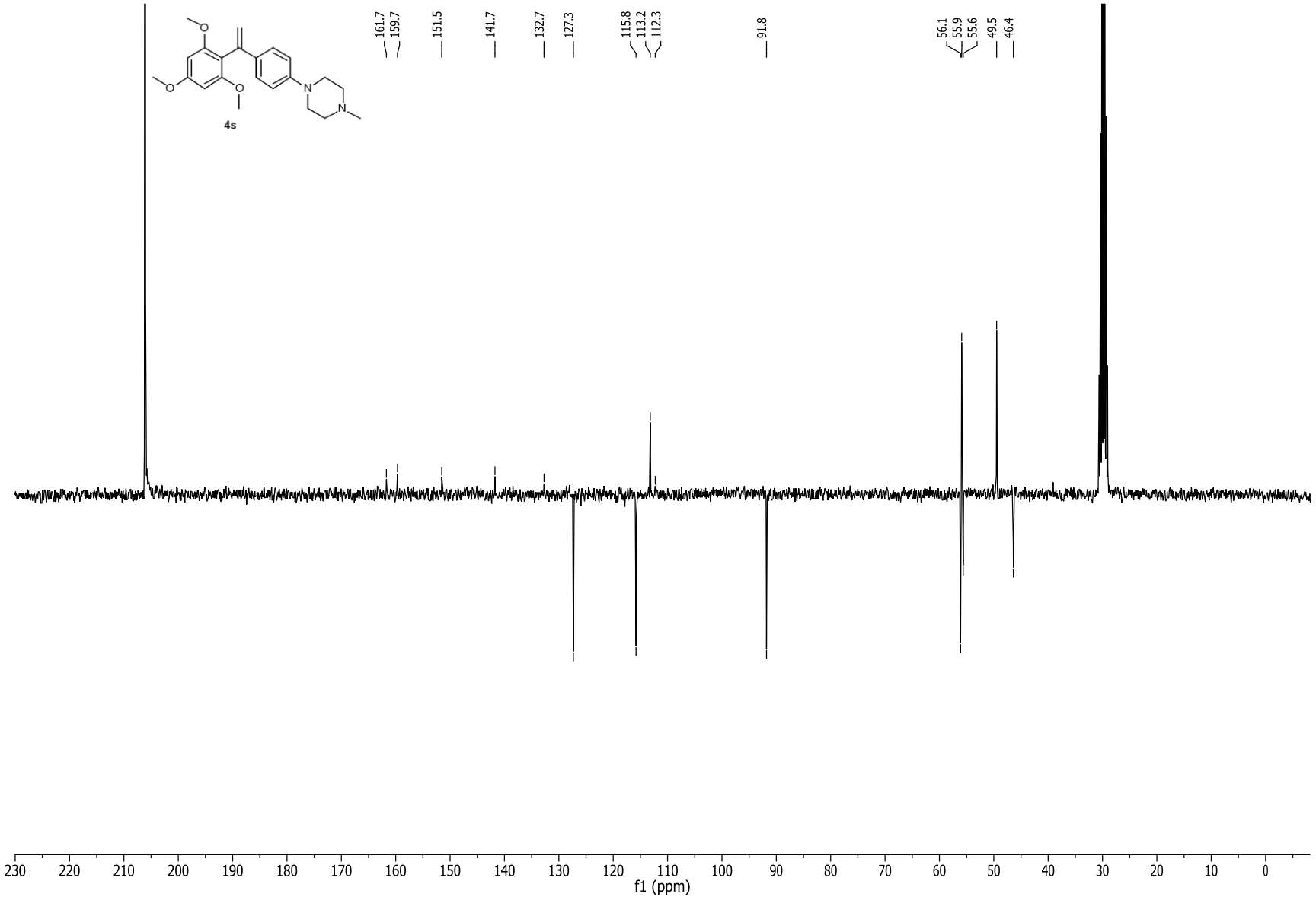

4q
7.06
7.03
6.50
6.47
6.27
5.63
5.63
4.81
4.77
4.76
3.84
3.65
3.10
3.08
3.06
1.65
1.63
1.61
1.58
1.56
1.40
1.38
1.30
1.29
0.87
0.85
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

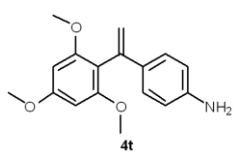

4q
161.6
159.7
149.4
142.0
129.9
127.5
112.5
112.1
111.4
91.8
56.1
55.6
44.3
32.6
30.3
27.9
23.3
14.3
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


4r
7.11
7.08
6.56
6.53
6.28
5.66
5.65
4.77
4.77
3.84
3.66
3.32
3.29
3.27
1.61
1.59
1.56
1.53
1.51
1.42
1.39
1.37
1.34
1.32
1.29
0.96
0.94
0.91
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


4r
161.6
159.7
148.3
141.9
128.8
127.6
116.3
112.0
111.3
91.8
56.1
55.6
51.3
30.3
20.9
14.3
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

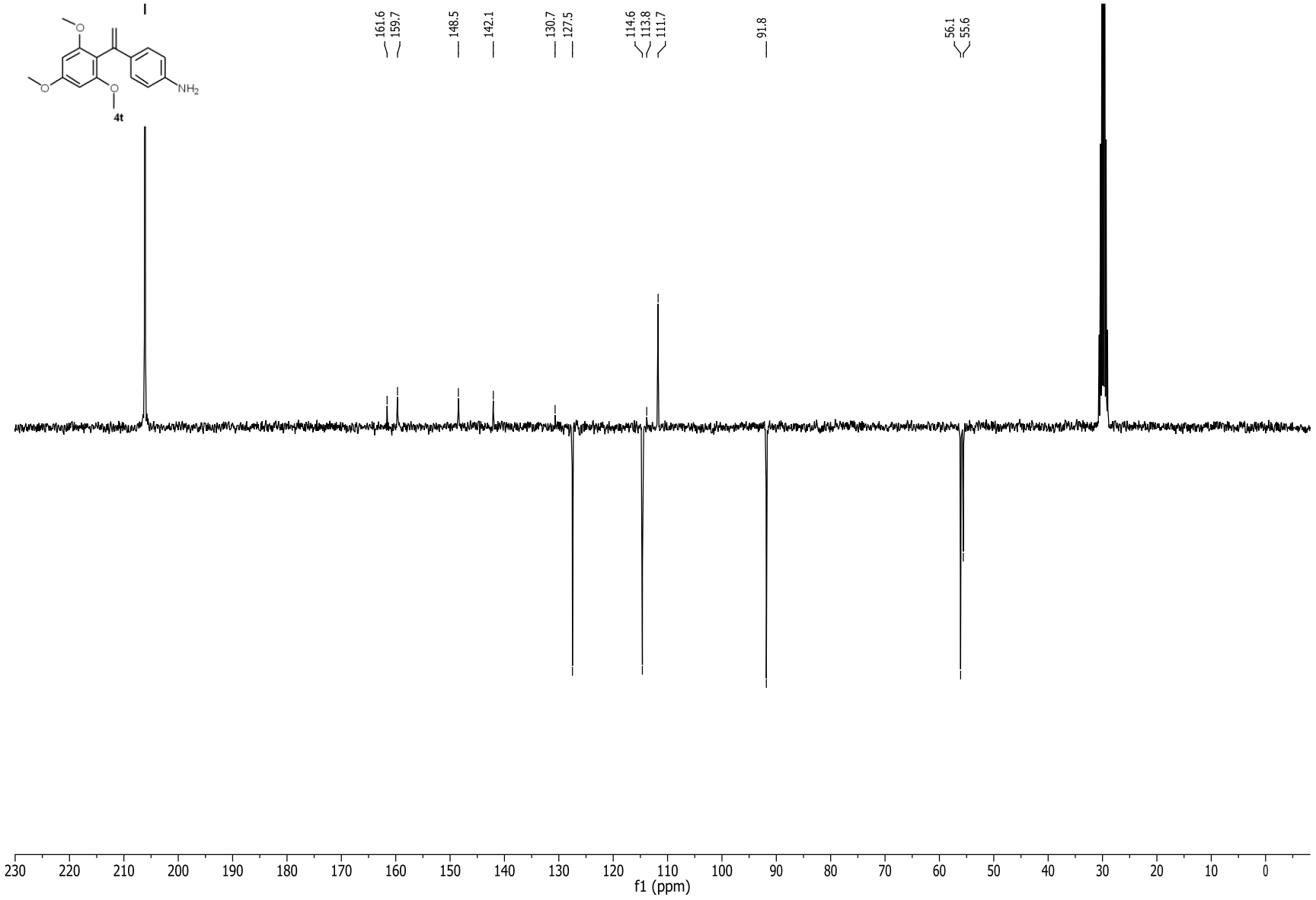

4s
7.17
7.14
6.82
6.79
6.28
5.72
5.71
4.87
4.87
3.84
3.65
3.16
3.15
3.13
2.48
2.46
2.45
2.24
(m)
3.15
(m)
2.46
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

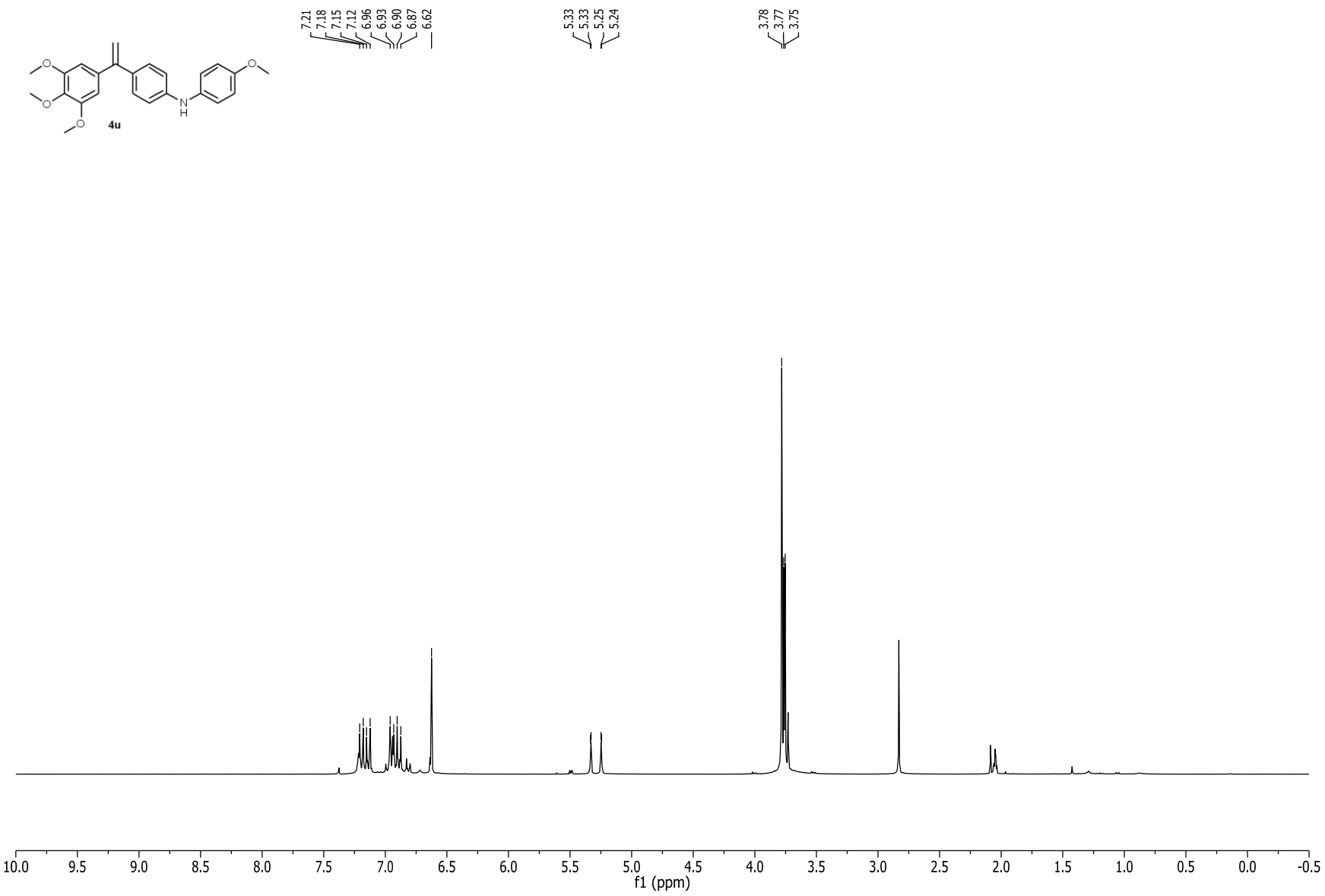

4s
161.7
159.7
151.5
141.7
132.7
127.3
115.8
113.2
112.3
91.8
56.1
55.9
55.6
49.5
46.4
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

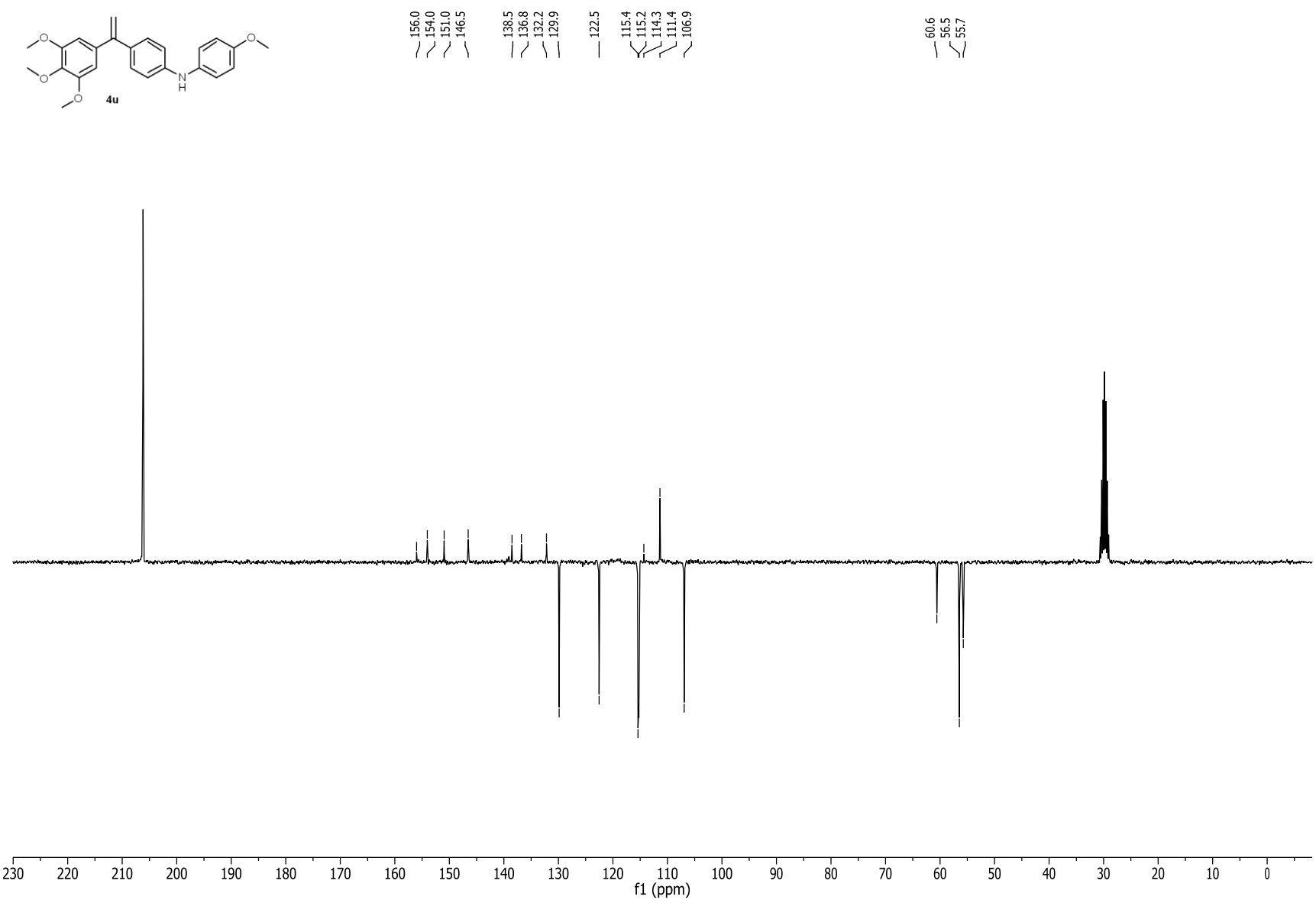

O
O
O
NH2

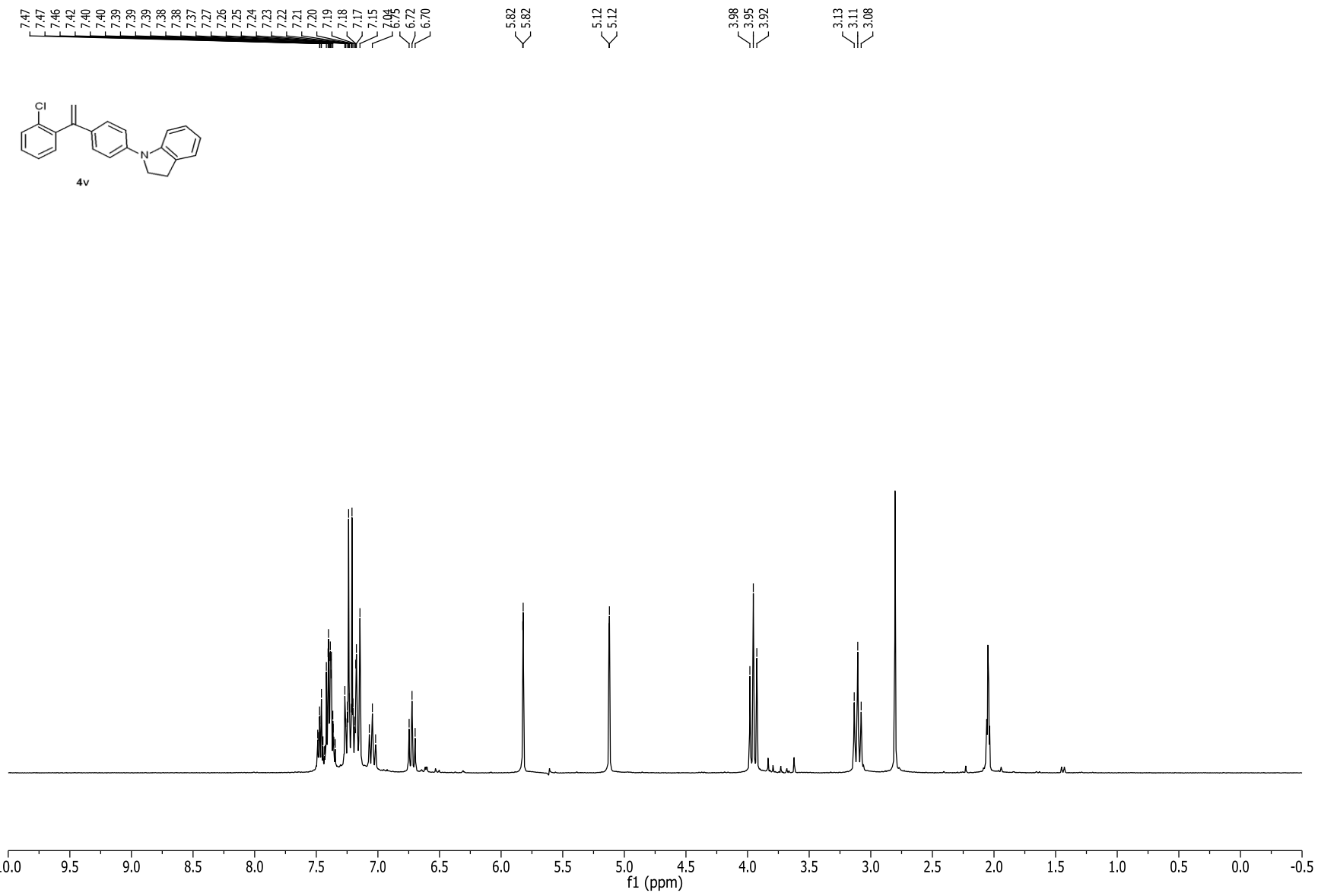
4t

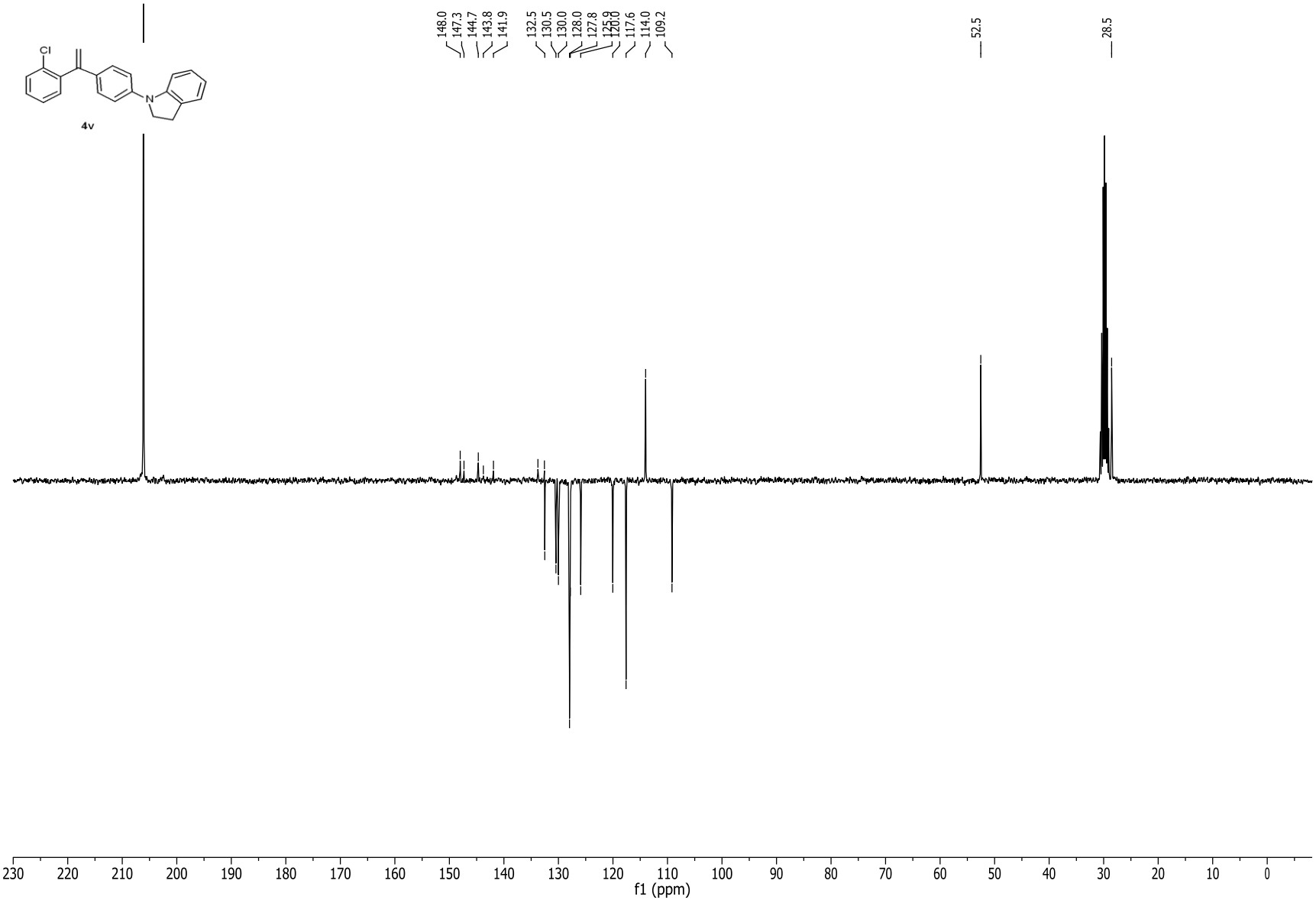
7.02
7.00
6.54
6.51
6.27
5.64
5.63
4.78
4.78
4.55
3.83
3.65
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

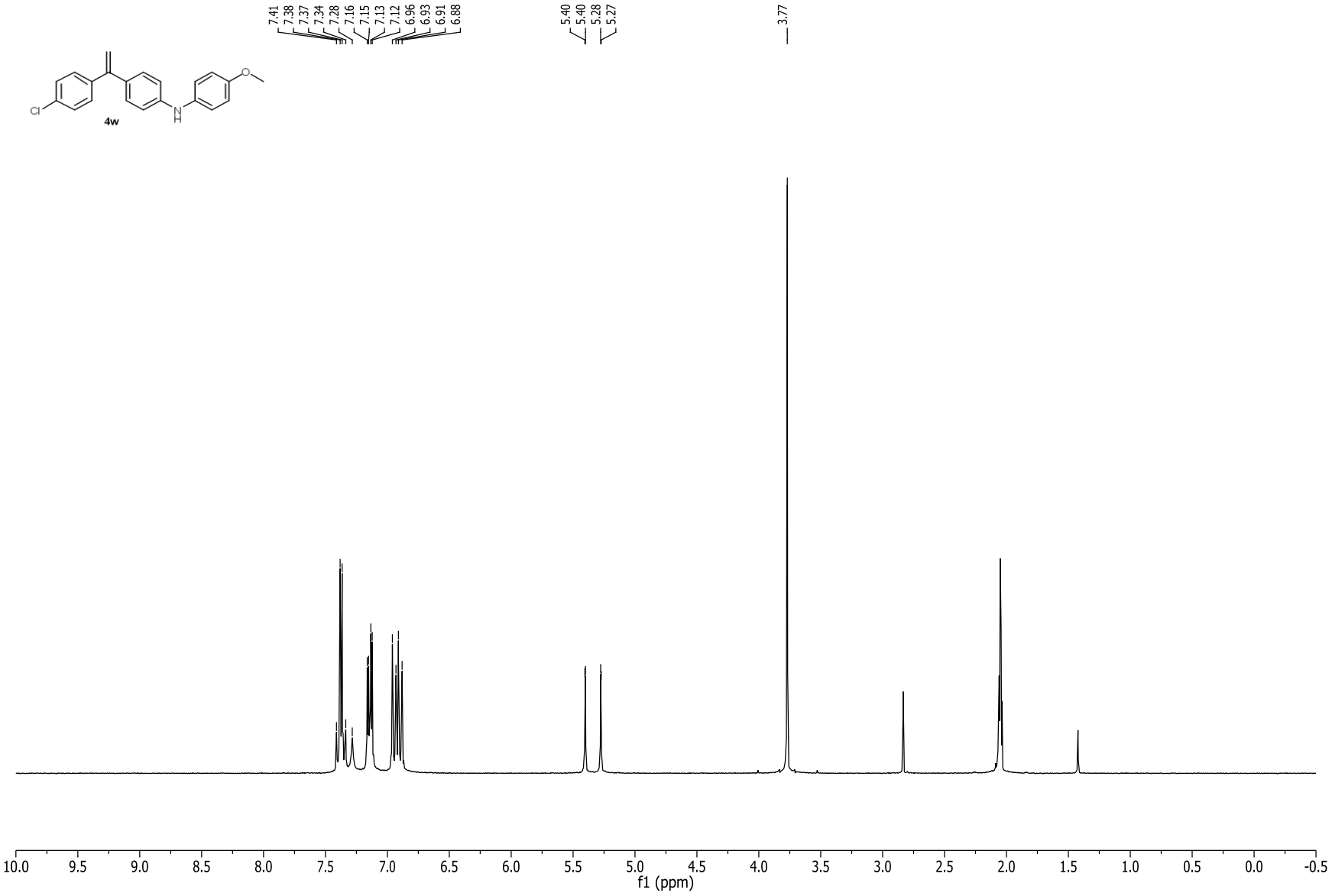

4t
161.6
159.7
148.5
142.1
130.7
127.5
114.6
113.8
111.7
91.8
56.1
55.6
NH2
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

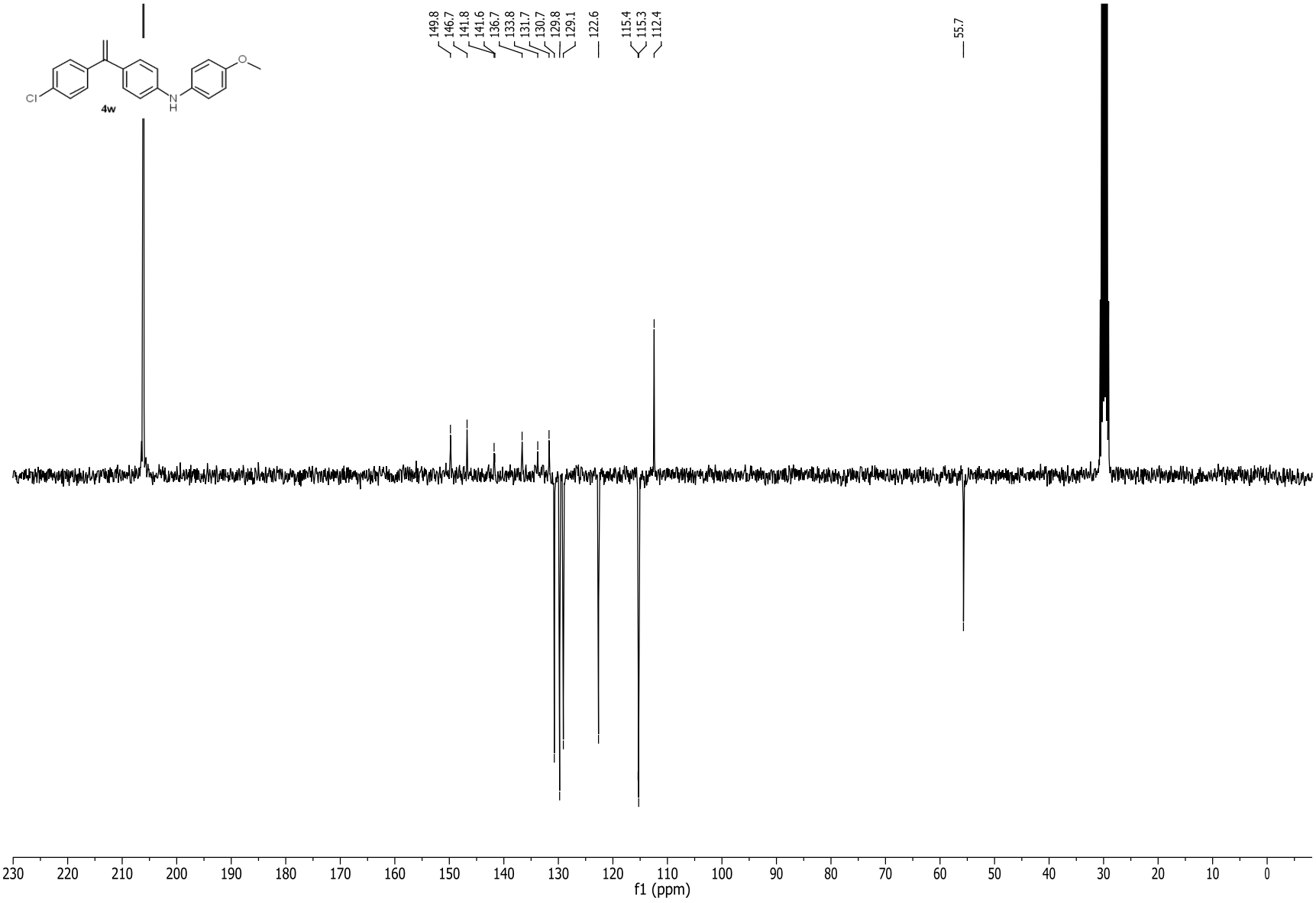

7.21
7.18
7.15
7.12
6.96
6.93
6.90
6.87
6.62
5.33
5.33
5.25
5.24
3.78
3.77
3.75
4u
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

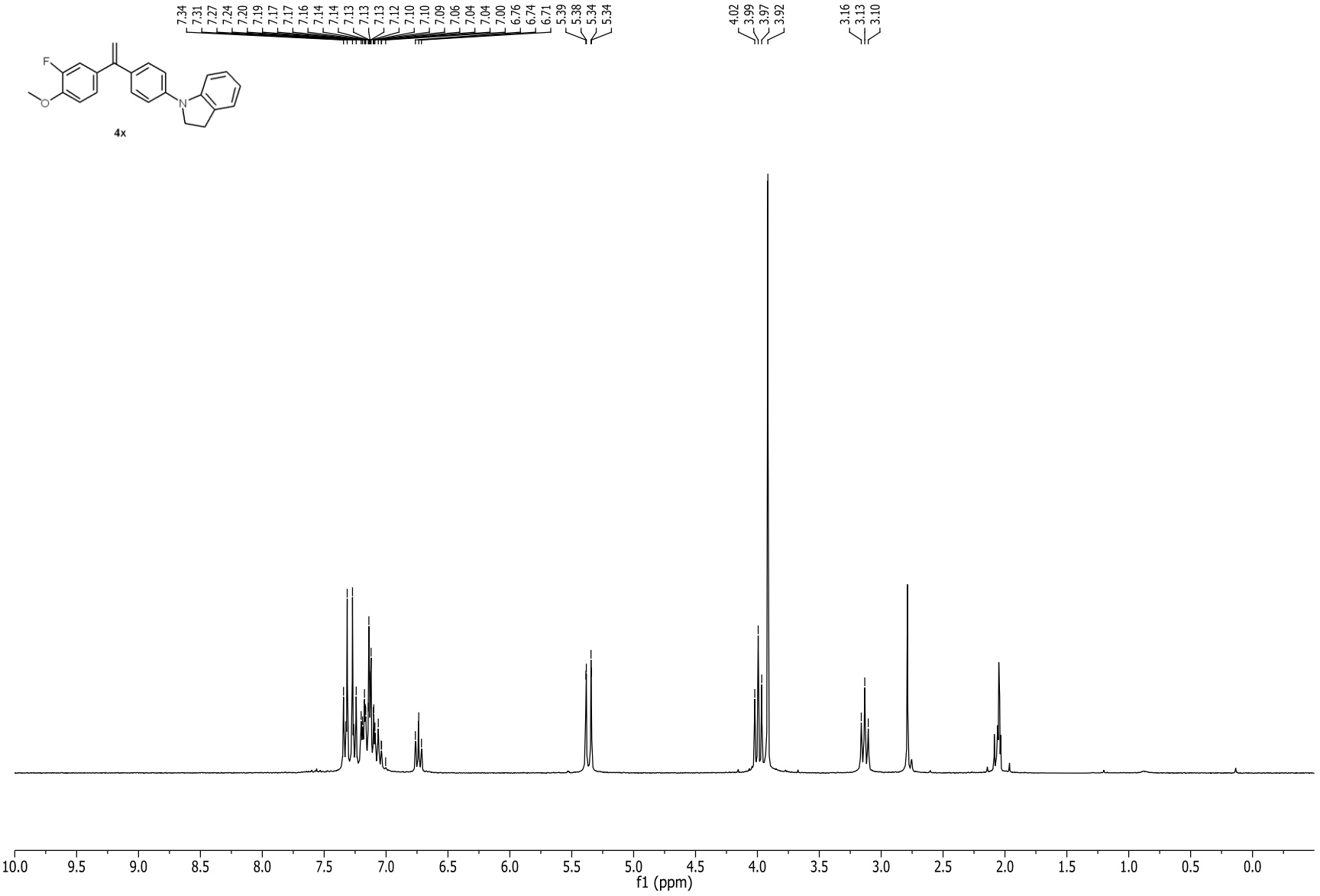

4u
156.0
154.0
151.0
146.5
138.5
136.8
132.2
129.9
122.5
115.4
115.2
114.3
111.4
106.9
60.6
56.5
55.7
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

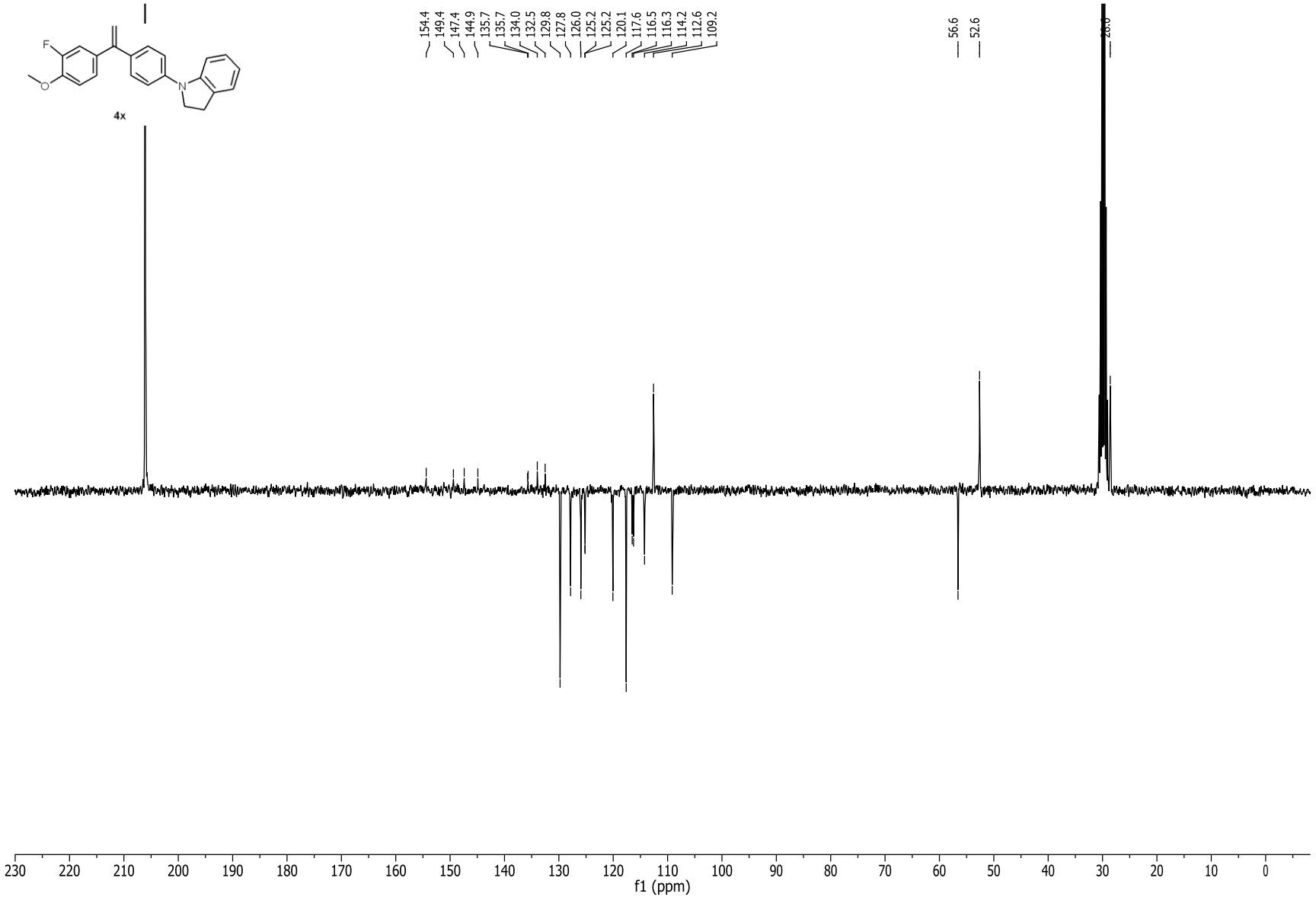

7.47
7.47
7.46
7.42
7.40
7.40
7.39
7.39
7.39
7.38
7.38
7.37
7.27
7.26
7.25
7.24
7.23
7.22
7.21
7.20
7.19
7.18
7.17
7.15
7.04
6.75
6.72
6.70
5.82
5.82
5.12
5.12
3.98
3.95
3.92
3.13
3.11
3.08
Cl
N
4v
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


Cl
N
4v
148.0
147.3
144.7
143.8
141.9
132.5
130.5
130.0
128.0
127.8
125.9
125.0
117.6
114.0
109.2
52.5
28.5
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

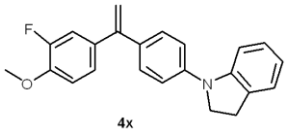

7.41
7.38
7.37
7.34
7.28
7.16
7.15
7.13
7.12
6.96
6.93
6.91
6.88
5.40
5.40
5.28
5.27
3.77
Cl
N
H
O
4w
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


149.8
146.7
141.8
141.6
136.7
133.8
131.7
130.7
129.8
129.1
122.6
115.4
115.3
112.4
55.7
Cl
N
H
O
4w
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

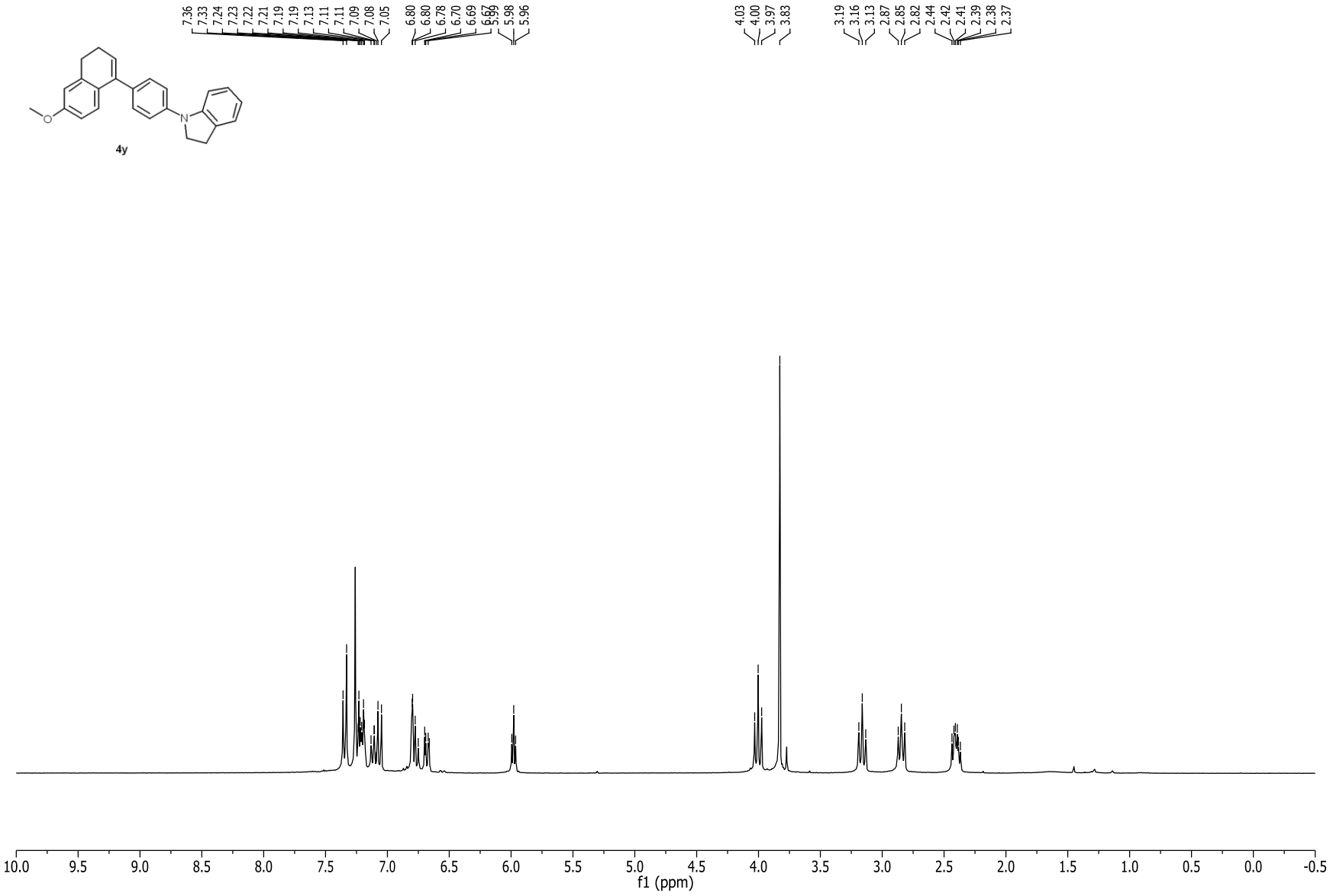

7.34
7.31
7.27
7.24
7.20
7.19
7.17
7.17
7.16
7.14
7.14
7.13
7.13
7.13
7.12
7.10
7.10
7.09
7.06
7.04
7.04
7.00
6.76
6.74
6.71
5.39
5.38
5.34
5.34
4.02
3.99
3.97
3.92
3.16
3.13
3.10
F
O
N
4x
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

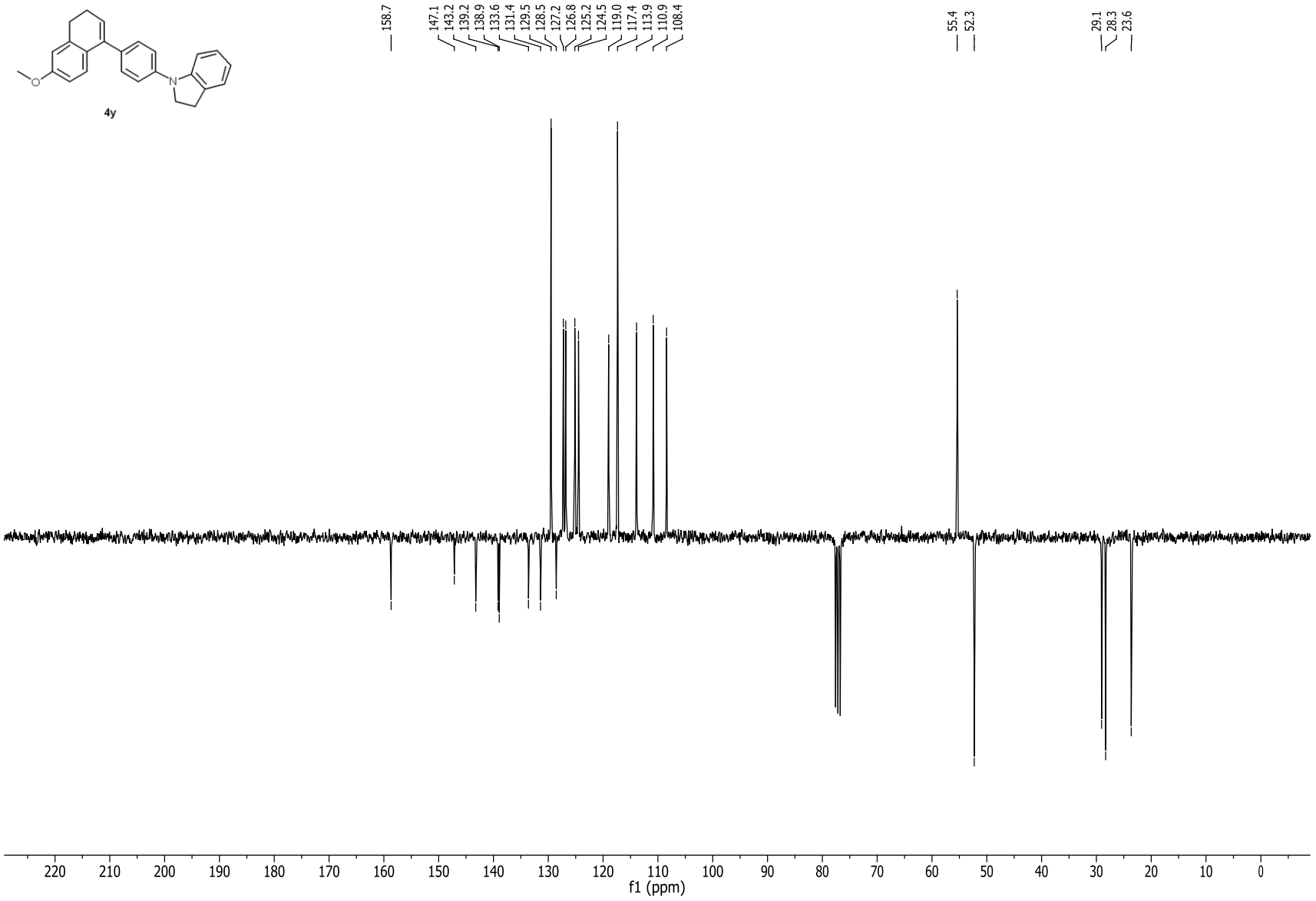

F
O
N
4x
154.4
149.4
147.4
144.9
135.7
135.7
134.0
132.5
129.8
127.8
126.0
125.2
125.2
120.1
117.6
116.5
116.3
114.2
112.6
109.2
56.6
52.6
26.0
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

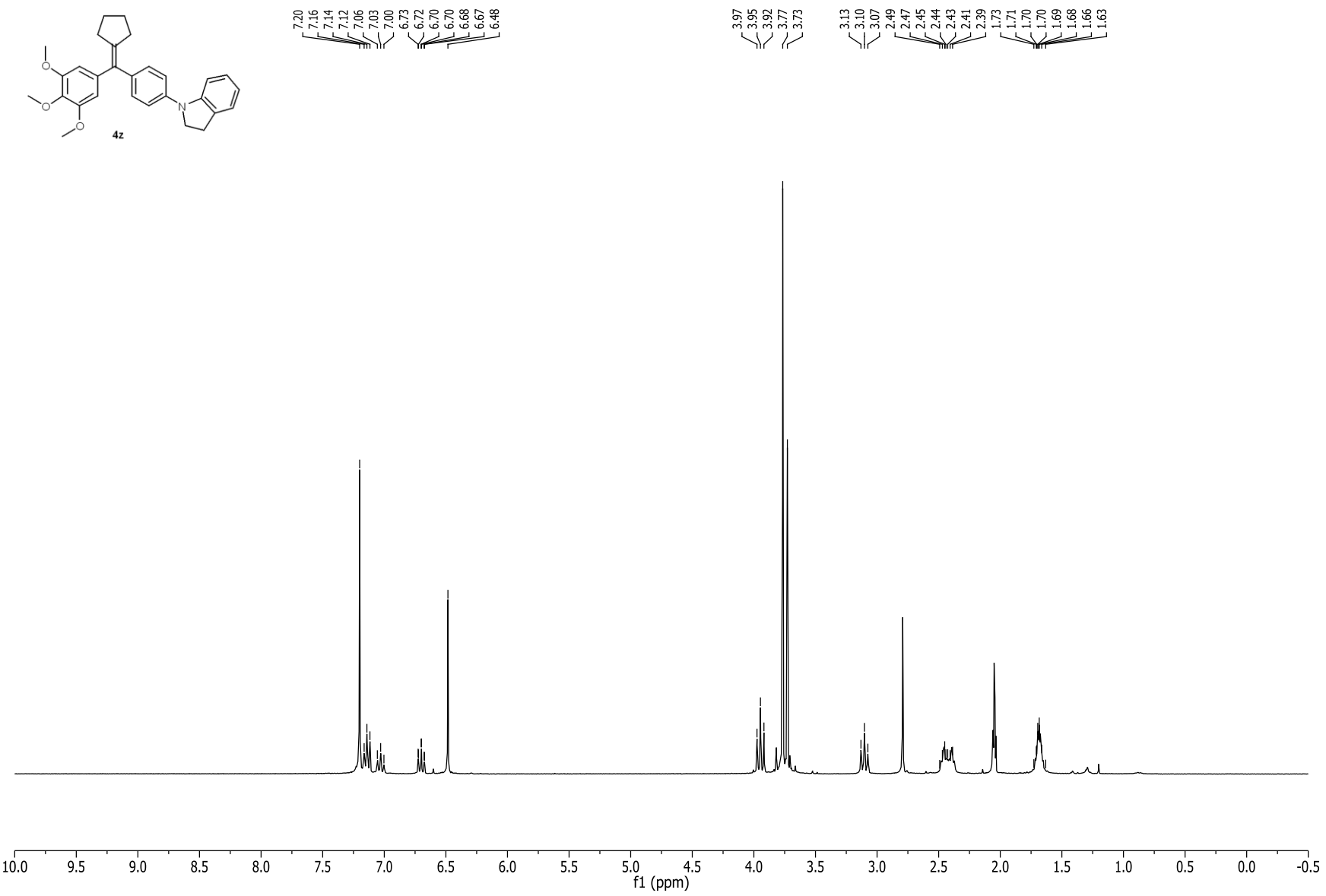
2012.11.05
mr402
F19CPDsw Aceton v biocis 11

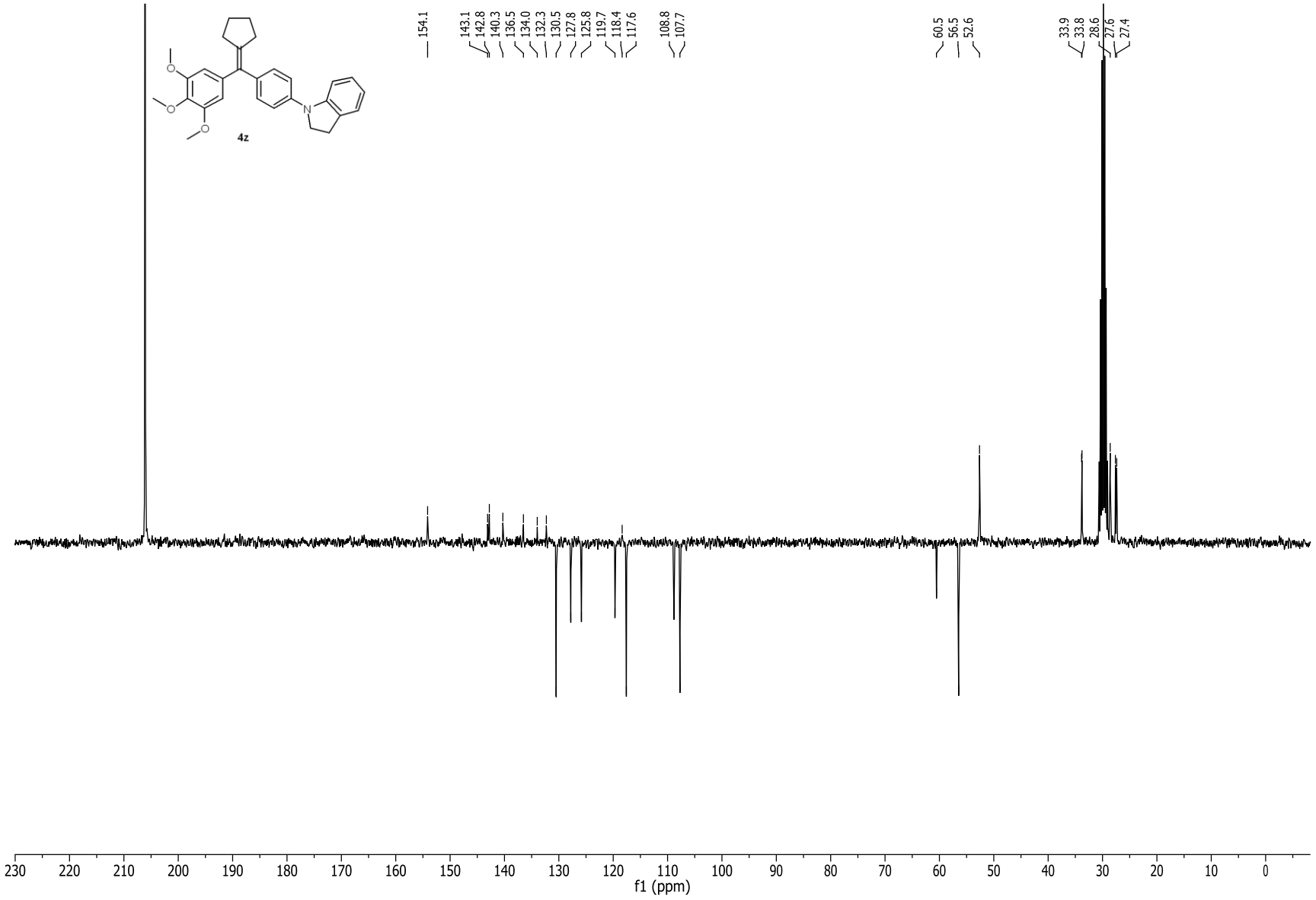


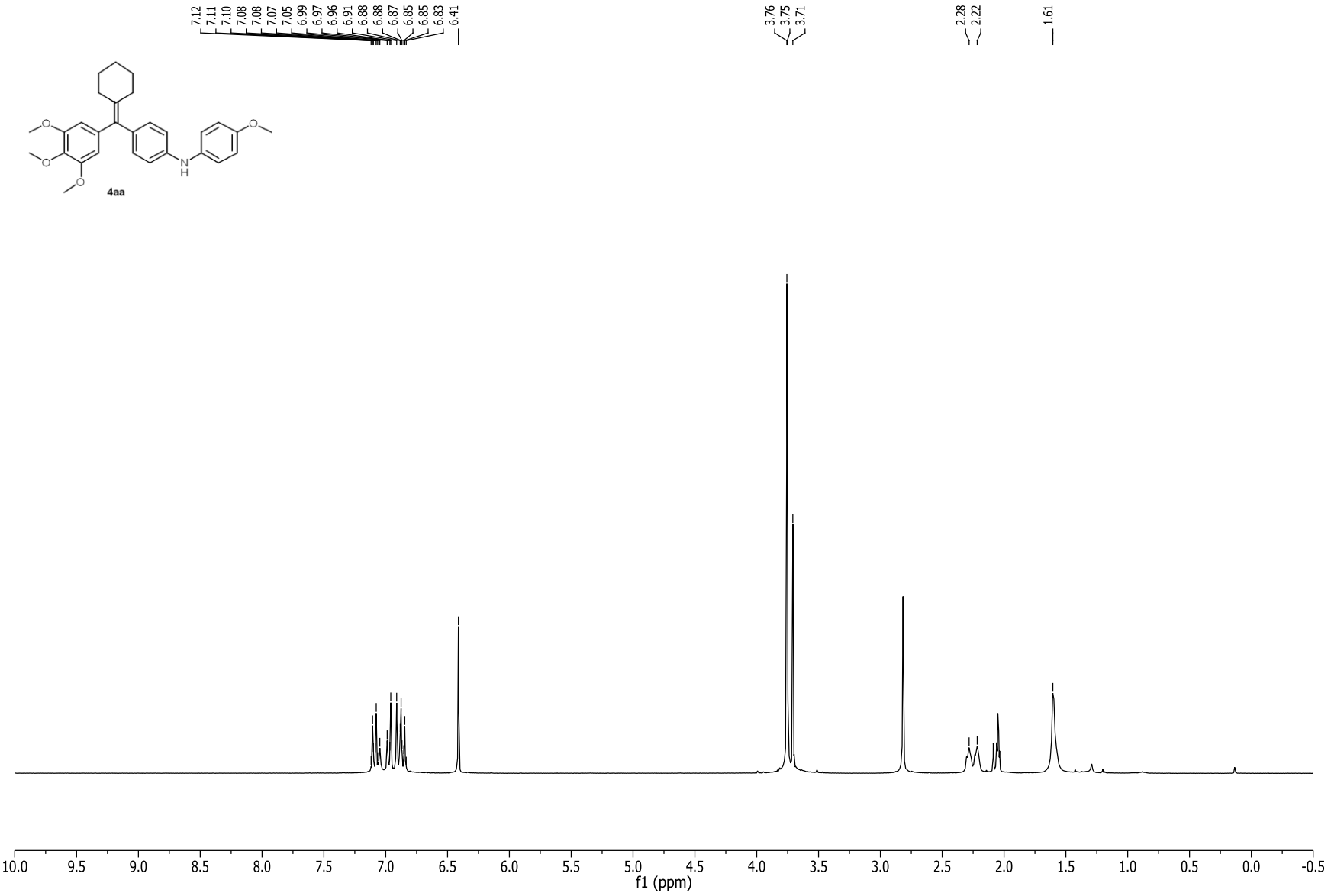
4x

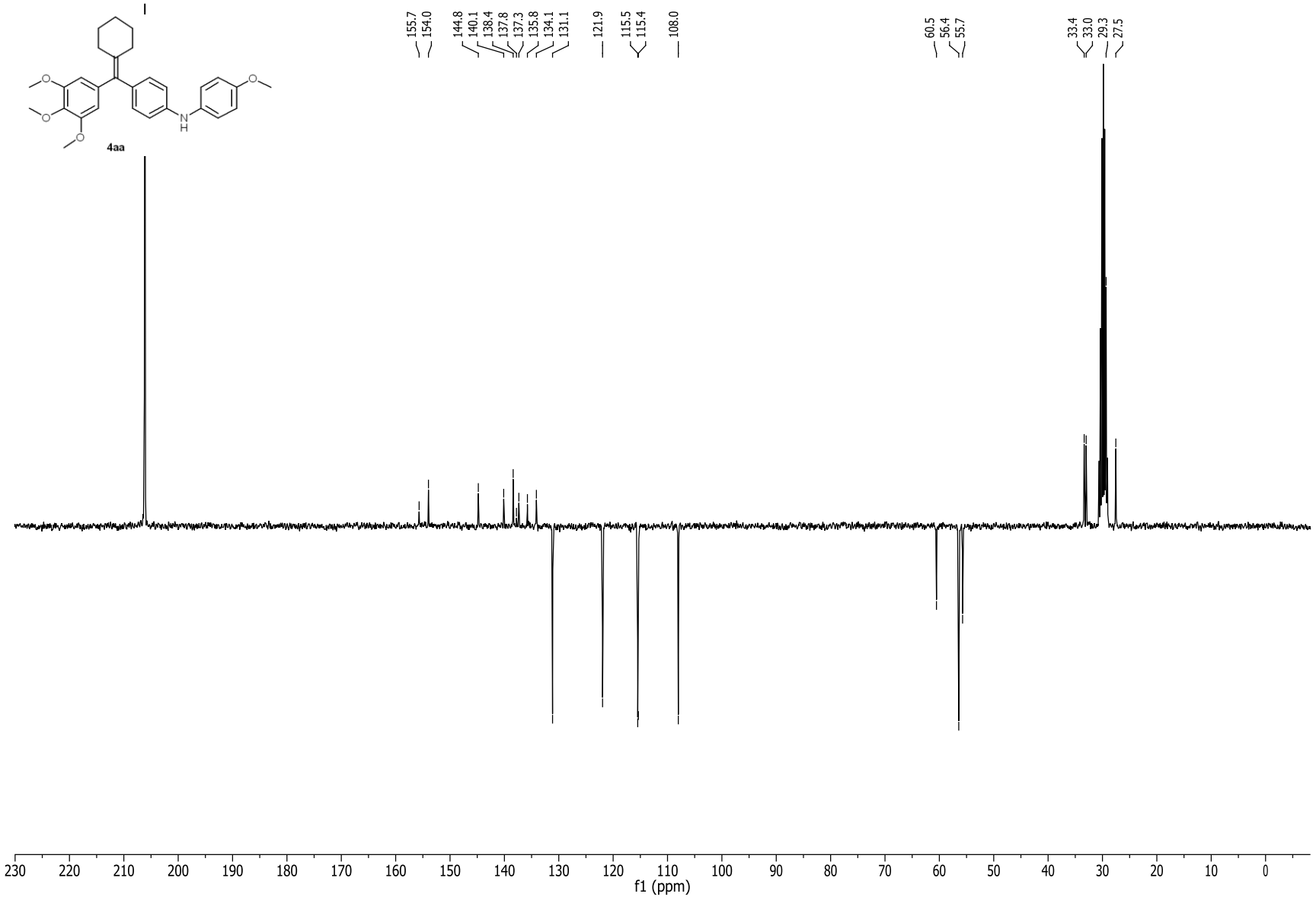

7.36
7.33
7.24
7.23
7.22
7.21
7.19
7.19
7.13
7.11
7.11
7.09
7.08
7.05
6.80
6.80
6.78
6.70
6.69
5.98
5.96
4.03
4.00
3.97
3.83
3.19
3.16
3.13
2.87
2.85
2.82
2.44
2.42
2.41
2.39
2.38
2.37
O
N
4y
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

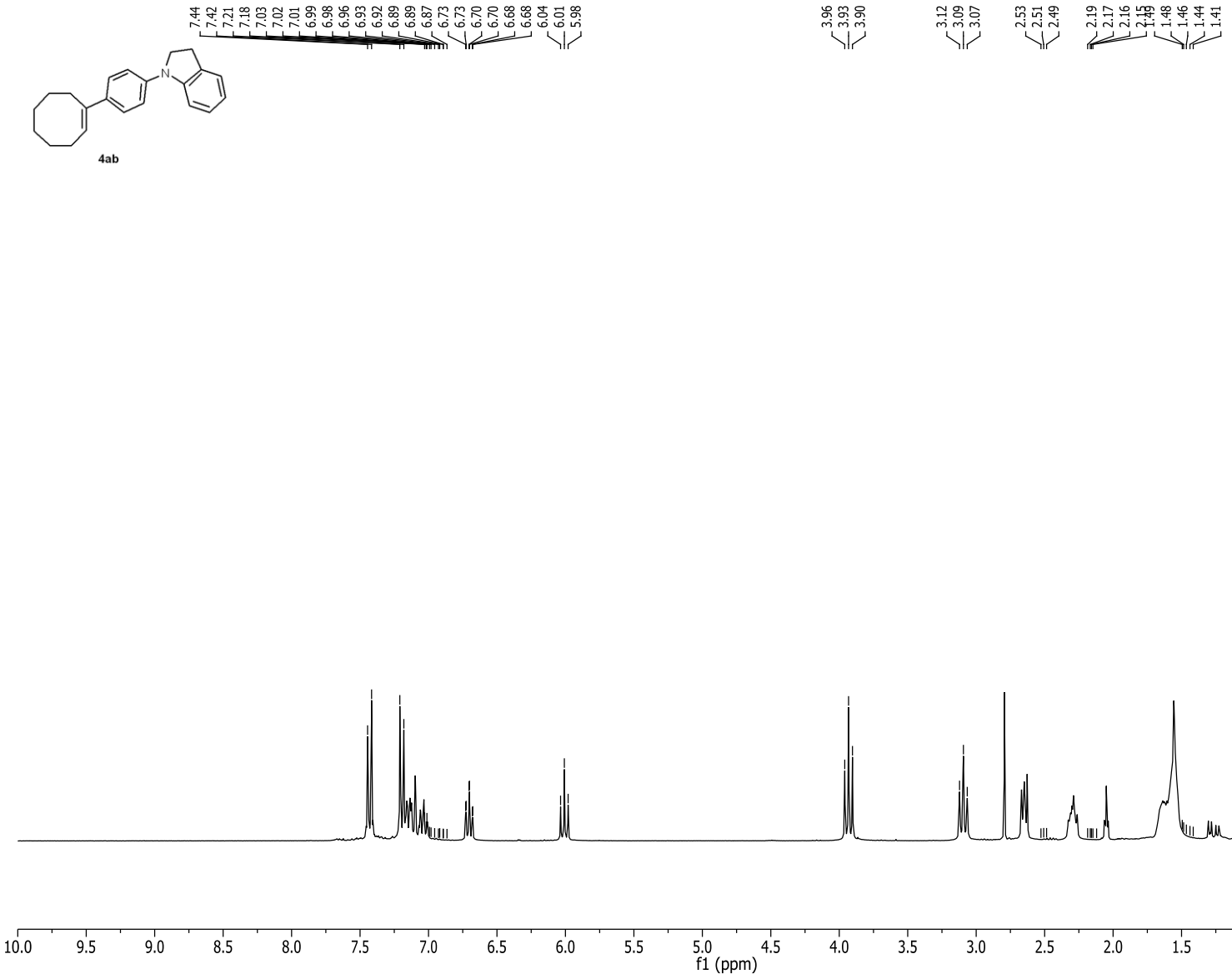

4y
158.7
147.1
143.2
139.2
138.9
133.6
131.4
129.5
128.5
127.2
126.8
125.2
124.5
119.0
117.4
113.9
110.9
108.4
55.4
52.3
29.1
28.3
23.6
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

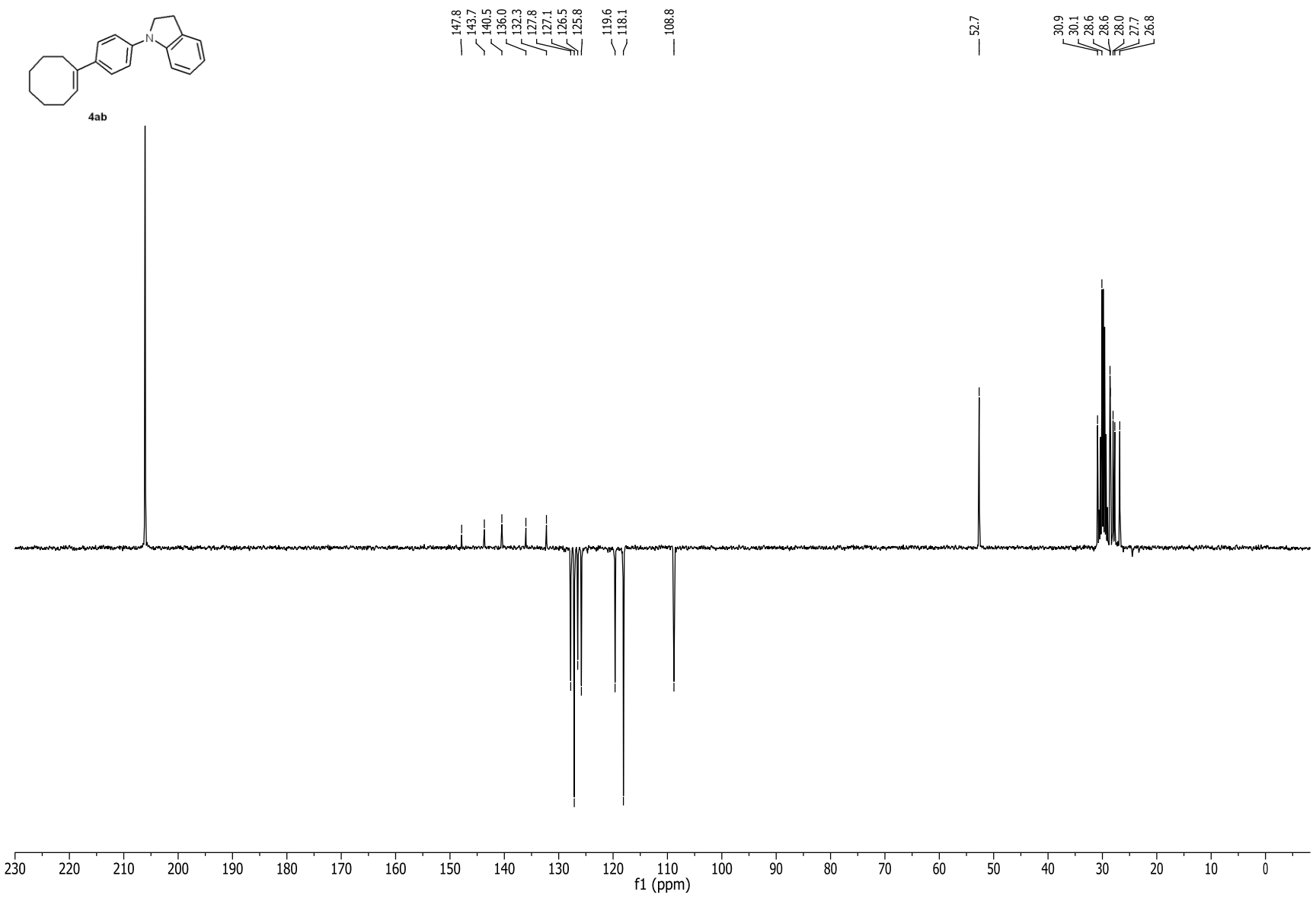

7.20
7.16
7.14
7.12
7.06
7.03
7.00
6.73
6.72
6.70
6.70
6.68
6.67
6.48
3.97
3.95
3.92
3.77
3.73
3.13
3.10
3.07
2.49
2.47
2.45
2.44
2.43
2.41
2.39
1.73
1.71
1.70
1.70
1.69
1.68
1.66
1.63
4z
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

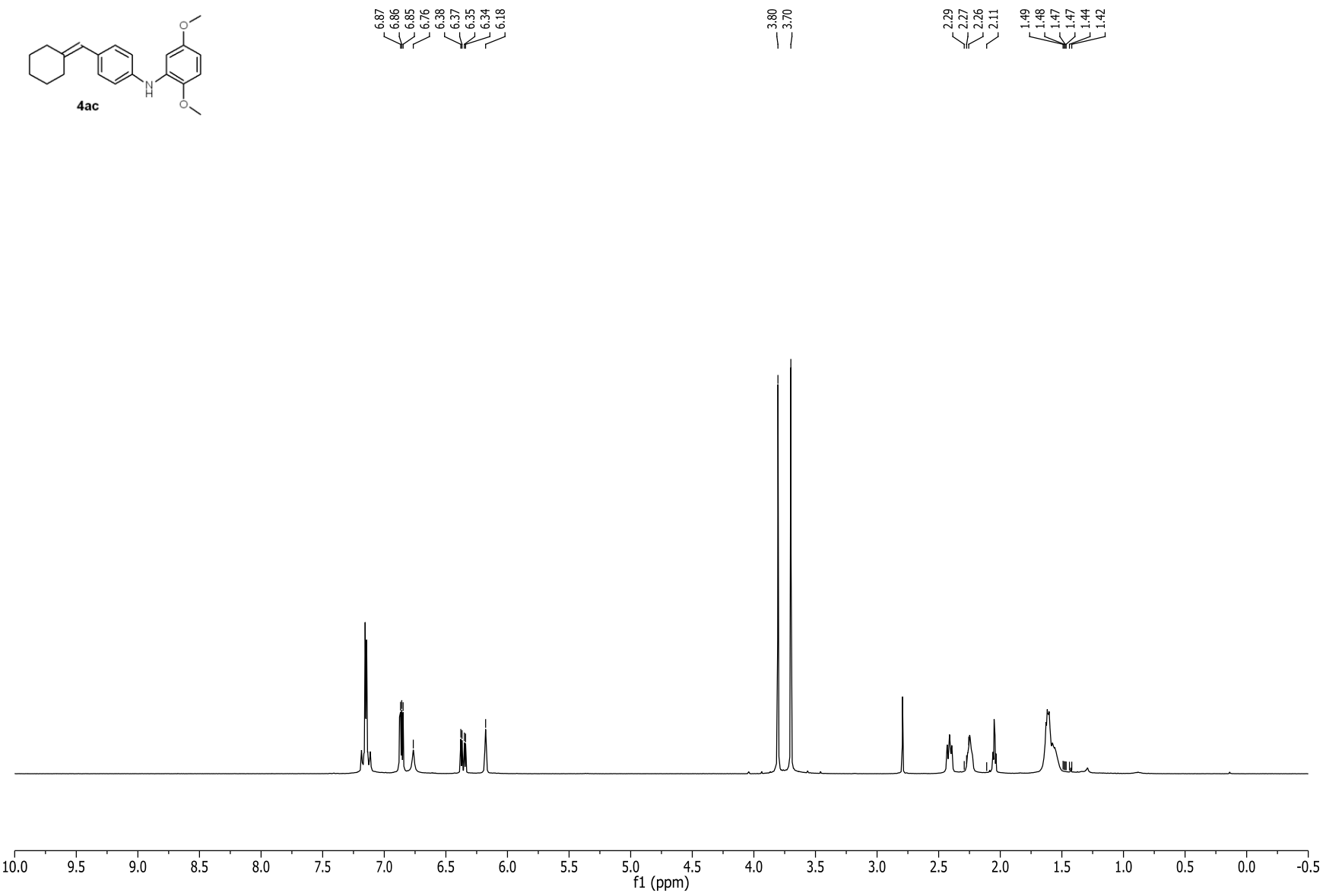

4z
154.1
143.1
142.8
140.3
136.5
134.0
132.3
130.5
127.8
125.8
119.7
118.4
117.6
108.8
107.7
60.5
56.5
52.6
33.9
33.8
28.6
27.6
27.4
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

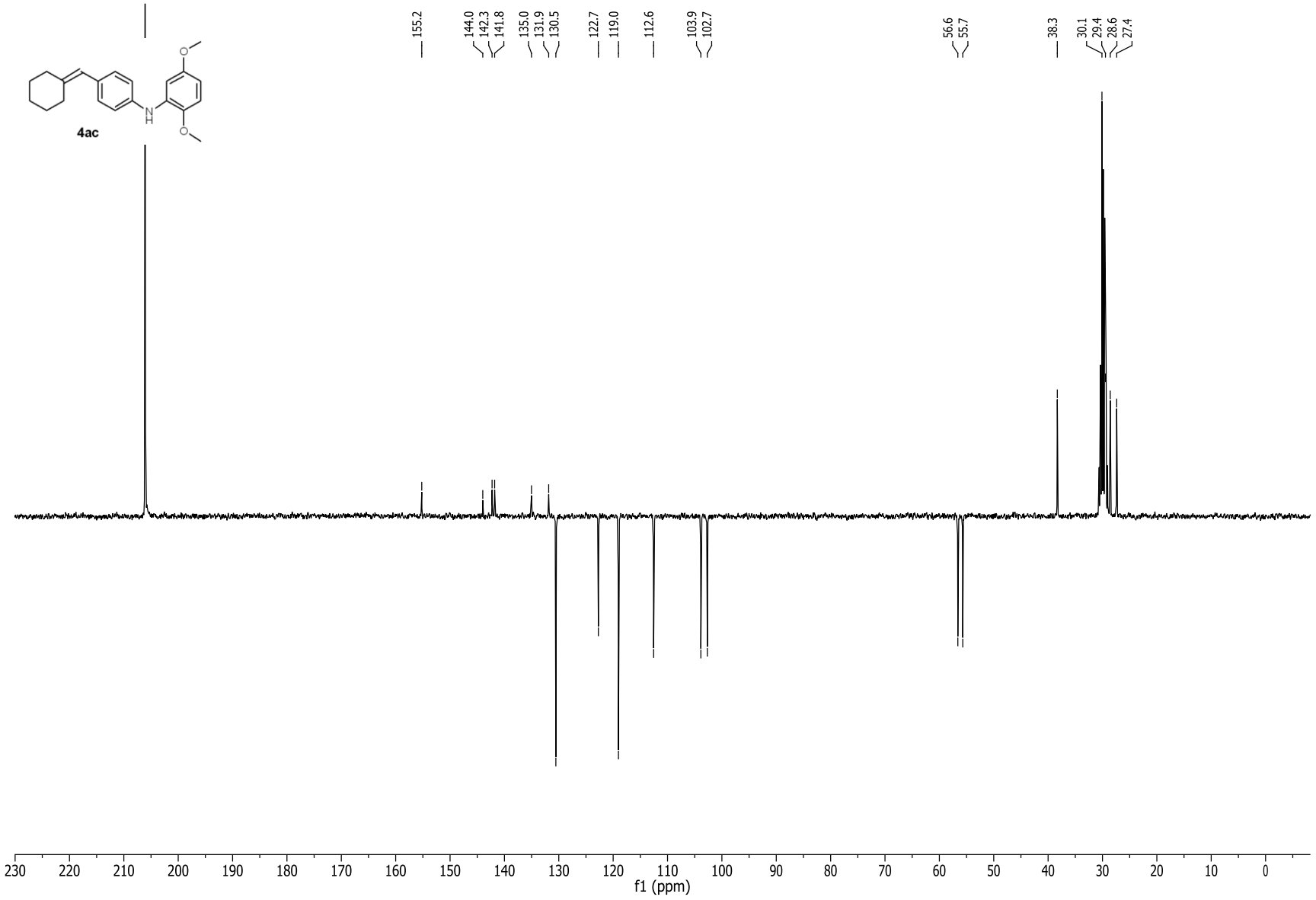

7.12
7.11
7.10
7.08
7.08
7.07
7.05
6.99
6.97
6.96
6.91
6.88
6.88
6.87
6.85
6.85
6.83
6.41
3.76
3.75
3.71
2.28
2.22
1.61
4aa
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


4aa
155.7
154.0
144.8
140.1
138.4
137.8
137.3
135.8
134.1
131.1
121.9
115.5
115.4
108.0
60.5
56.4
55.7
33.4
33.0
29.3
27.5
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.44
7.42
7.21
7.18
7.03
7.02
7.01
6.99
6.98
6.96
6.93
6.92
6.89
6.89
6.87
6.73
6.73
6.70
6.70
6.68
6.68
6.04
6.01
5.98
3.96
3.93
3.90
3.12
3.09
3.07
2.53
2.51
2.49
2.19
2.17
2.16
2.15
1.49
1.48
1.46
1.44
1.41
N
4ab
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


4ab
147.8
143.7
140.5
136.0
132.3
127.8
127.1
126.5
125.8
119.6
118.1
108.8
52.7
30.9
30.1
28.6
28.6
28.0
27.7
26.8
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


4ac
6.87
6.86
6.85
6.76
6.38
6.37
6.35
6.34
6.18
3.80
3.70
2.29
2.27
2.26
2.11
1.49
1.48
1.47
1.47
1.44
1.42
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


4ac
155.2
144.0
142.3
141.8
135.0
131.9
130.5
122.7
119.0
112.6
103.9
102.7
56.6
55.7
38.3
30.1
29.4
28.6
27.4
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)