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Microwave Assisted, Silica Gel Mediated, Solvent Free, Michael-Addition of Aryl Methyl Ketones with Chalcones for the Synthesis of 1,3,5-triarylpentane-1,5-diones

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Supplementary Information

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General

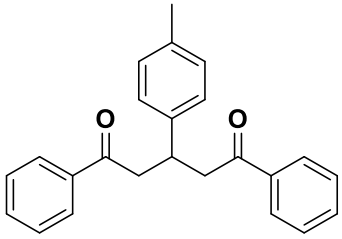
A CEM Discover microwave synthesizer (Model No: 908010) operating at 180/264 V and 50/60 Hz with microwave power maximum level of 300 W and microwave frequency of 2455 MHz was employed for the microwave-assisted experiments. Nuclear Magnetic Resonance (^{1}H, ^{13}C - NMR) spectra were recorded on 300 MHz spectrometer (Bruker) in $CDCl_3$ using TMS as an internal standard. Chemical shifts are reported in parts per million (δ), coupling constants (*J* values) are reported in Hertz (Hz) and spin multiplicities are indicated by the following symbols: s (singlet), d (doublet), dd (doublet of doublet), ddd (doublet of doublet of doublet), dt (doublet of triplet), t (triplet), p (pentet), m (multiplet). ^{13}C NMR spectra were routinely run with broadband decoupling. Pre coated silica gel on aluminium plates (Merck) were used for TLC analysis with a mixture of petroleum ether (60 – 80 °C) and ethyl acetate as the eluent. Electrospray ionization (ESI) mass spectra were obtained on an LCQ Fleet mass spectrometer, Thermo Fisher Instruments Limited, US and an Agilent mass spectrometer. HRMS was recorded on Brucker-Daltonics, Micro-TOF-Q II mass spectrometer. Elemental analyses were performed on a Perkin Elmer 2400 Series II Elemental CHNS analyser.

General procedure for the synthesis of 1,3,5-triarylpentan-1,5-diones (3)

A mixture of chalcone 1 (1.0 equiv), aryl methyl ketone 2 (1.0 equiv.) and Silica gel 60 (spherical, 63-200 μm) (3 g) was taken in a 10 mL quartz vial and placed in the microwave oven. The vial was sealed with a pressure cap and subjected to microwave irradiation. The irradiation was programmed between 100–120 °C, 120 W, 5 bar, for 10 min. The reaction was monitored by TLC using petroleum ether/ethyl acetate mixture (7:3) as the eluent. After the reaction mixture was cooled to room temperature, ethanol was added and reaction mixture was separated from the silica gel. The obtained crude was filtered, dried in vacuum and recrystallized from ethanol to afford 3. Then, the silica gel was carefully washed well with methanol : dichloromethane (1:1) and dried in hot air oven at 130 °C under reduced pressure for 1 h. The recovered silica gel could be recycled.

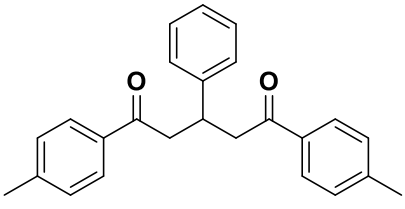
Characterization of compounds 3

3-(4-Methylphenyl)-1,5-diphenyl-1,5-pentanedione (3a)[1]



Isolated as colorless solid; mp: 120 – 121 ºC; IR (KBr): 3069, 3037, 2898, 1678 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.94 (dt, *J* = 3.5, 1.5 Hz, 3H), 7.53 (dd, *J* = 10.4, 4.2 Hz, 2H), 7.42 (dd, *J* = 11.5, 4.3 Hz, 3H), 7.28 – 7.20 (m, 3H), 7.12 (dd, *J* = 27.9, 8.1 Hz, 3H), 4.03 (p, *J* = 6.8 Hz, 1H), 3.48 (dd, *J* = 16.5, 6.9 Hz, 2H), 3.33 (dd, *J* = 16.6, 6.2 Hz, 2H), 2.28 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 198.6, 140.7, 136.9, 136.1, 132.9, 129.3, 128.5, 128.1, 127.2, 44.9, 36.8, 20.9. ESI-MS *m/z* calcd for $C_{24}H_{22}O_2$: 342.43 $[M]^+$; Found: 343.59 $[M+H]^+$. Anal. Calcd for $C_{24}H_{22}O_2$: C, 84.18; H, 6.48%. Found: C, 84.14; H, 6.52%.

1,5-Bis(4-methylphenyl)-3-phenyl-1,5-pentanedione (3b)[1]



Isolated as colorless solid; mp: 108 – 110 ºC; IR (KBr): 3059, 3028, 2891, 1679 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.88 (d, *J* = 8.1 Hz, 4H), 7.45 – 7.39 (m,6H), 4.00 (p, *J* = 6.8 Hz, 1H), 3.45 (dd, *J* = 16.9, 6.8 Hz, 2H), 3.27 (dd, *J* = 16.8, 7.2 Hz, 2H), 2.41 (s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ_C: 198.0, 144.0, 142.4, 134.3, 132.2, 129.3, 128.9, 128.7, 128.2, 44.6, 36.6, 21.6. ESI-MS *m/z* calcd for $C_{25}H_{24}O_2$: 356.46 $[M]^+$; Found: 357.21 $[M+H]^+$. Anal. Calcd for: C, 84.24; H, 6.79%. Found: C, 84.27; H, 6.75%.

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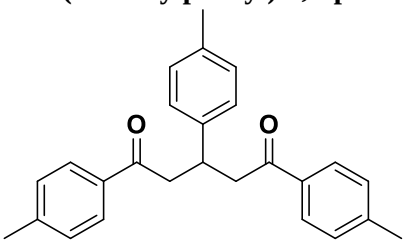
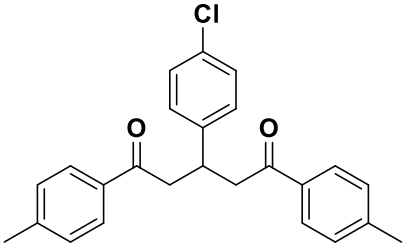
1,3,5-Tri(4-methylphenyl)-1,5-pentanedione (3c)[2]



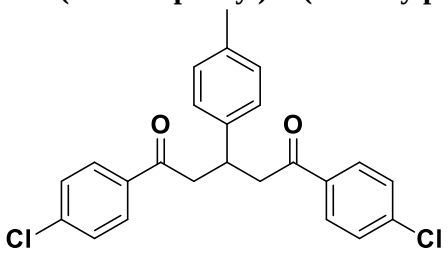
Isolated as colorless crystal; mp: 87 – 89 °C; IR (KBr): 3058, 3027, 2891, 1679 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ_H: 7.86 (d, *J* = 8.1 Hz, 4H), 7.24 (d, *J* = 8.1 Hz, 4H), 7.17 (d, *J* = 8.1 Hz, 2H), 7.08 (d, *J* = 8.1 Hz, 2H), 4.05 (p, *J* = 6.9 Hz, 1H), 3.45 (dd, *J* = 16.5, 6.9 Hz, 2H), 3.32 (dd, *J* = 16.5, 6.9 Hz, 2H), 2.39 (s, 6H), 2.28 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ_C: 198.3, 143.8, 140.9, 136.1, 134.4, 129.3, 129.2, 128.3, 127.3, 45.0, 36.9, 21.6, 21.0. ESI-MS *m/z* calcd for $C_{26}H_{26}O_2$: 370.49 $[M]^+$; Found: 371.77 $[M+H]^+$. Anal. Calcd for $C_{26}H_{26}O_2$: C, 84.29; H, 7.07%. Found: C, 84.32; H, 7.11%.

3-(4-Chlorophenyl)-1,5-di(4-methylphenyl)-1,5-pentanedione (3d)[1]



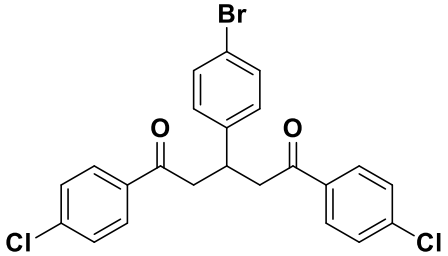
Isolated as colorless solid; mp: 113 – 114 °C; IR (KBr): 3065, 3031, 2888, 1677 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.70 (d, *J* = 8.6 Hz, 4H), 7.23 (d, *J* = 8.6 Hz, 4H), 6.96 (d, *J* = 8.0 Hz, 2H), 6.90 (d, *J* = 8.2 Hz, 2H), 3.80 (p, *J* = 6.9 Hz, 1H), 3.27 (dd, *J* = 16.6, 7.0 Hz, 2H), 3.10 (dd, *J* = 16.6, 7.0 Hz, 2H), 2.41 (s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 198.8, 143.9, 141.9, 136.1, 132.7, 129.6, 128.9, 128.7, 128.2, 44.5, 39.0, 21.1. HRMS (ESI) *m/z* calcd for $C_{25}H_{23}ClO_2$: 390.9019 $[M]^+$; Found: 390.9021 $[M]^+$. Anal. Calcd for $C_{25}H_{23}ClO_2$: C, 76.81; H, 5.93%. Found: C, 76.85; H, 5.99%.

1,5-Bis(4-chlorophenyl)-3-(4-methylphenyl)-1,5-pentanedione (3e)[2]



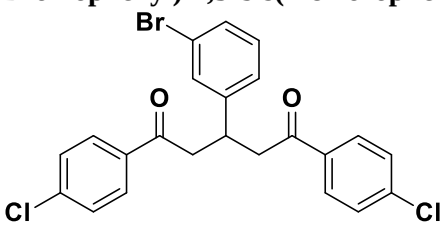
Isolated as colorless crystal; mp: 72 – 73 °C; IR (KBr): 3077, 3037, 2886, 1679 cm^{-1}; ^1H NMR (300 MHZ, $CDCl_3$) δ_H: 7.88 (d, *J* = 8.6 Hz, 4H), 7.40 (d, *J* = 8.6 Hz, 4H), 7.14 (d, *J* = 8.0 Hz, 2H), 7.07 (d, *J* = 8.0 Hz, 2H), 3.97 (p, *J* = 6.9 Hz, 1H), 3.44 (dd, *J* = 16.6, 7.0 Hz, 2H), 3.27 (dd, *J* = 16.6, 7.0 Hz, 2H), 2.28 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ_C: 197.4, 140.3, 139.5, 136.4, 135.3, 129.5, 129.3, 128.9, 127.2, 44.9, 36.9, 20.9. HRMS (ESI) *m/z* calcd for $C_{24}H_{20}Cl_2O_2$: 411.3204 $[M]^+$; Found: 411.3207 $[M]^+$. Anal. Calcd for $C_{24}H_{20}Cl_2O_2$: C, 70.08; H, 4.90%. Found: C, 70.12; H, 4.92%.

3-(4-Bromophenyl)-1,5-bis(4-chlorophenyl)-1,5-pentanedione (3f)[2]



Isolated as colorless solid; mp: 95 – 96 °C; IR (KBr): 3060, 3027, 2894, 1679 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.95 (d, *J* = 8.5 Hz, 2H), 7.87 (d, *J* = 8.5 Hz, 2H), 7.67 (d, *J* = 8.5 Hz, 2H), 7.50 (d, *J* = 8.5 Hz, 2H), 7.30 (dd, *J* = 14.7, 8.6 Hz, 4H), 4.08 (p, *J* = 7.2 Hz 1H), 3.52 (dd, *J* = 16.9, 6.8 Hz, 2H), 3.34 (dd, *J* = 17.4, 7.7 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 197.0, 141.9, 139.8, 135.4, 131.9, 129.6, 129.5, 129.0, 128.7, 44.7, 36.5. HRMS (ESI) *m/z* calcd for: 476.1899 $[M]^+$; Found: 476.1895 $[M]^+$. Anal. Calcd. for $C_{23}H_{17}BrCl_2O_2$; C, 58.01; H, 3.60%. Found: C, 58. 03; H, 3.58%.

3-(3-Bromophenyl)-1,5-bis(4-chlorophenyl)-1,5-pentanedione (3g)[2]



Isolated as colorless solid; mp: 92 – 93 °C; IR (KBr): 3052, 3031, 2882, 1675 cm^{-1}; ^1HNMR (300 MHz, $CDCl_3$) δ_H: 7.88 (d, *J* = 8.4 Hz, 4H), 7.42 – 7.12 (m, 8H), 4.06 (p, *J* = 6.9 Hz, 1H), 3.45 (dd, *J* = 17.0, 6.8 Hz, 2H), 3.28 (dd, *J* = 16.9, 6.9 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ_C: 196.8, 145.9, 139.6, 134.9, 130.4, 130.2, 129.9, 129.5, 128.9, 126.3, 122.7, 44.4, 36.6. HRMS (ESI) *m/z* calcd for: 476.1899 $[M]^+$; Found: 477.1894 $[M]^+$. Anal. Calcd. for $C_{23}H_{17}BrCl_2O_2$: C, 58.01; H, 3.60%. Found: C, 58.04; H, 3.64%.

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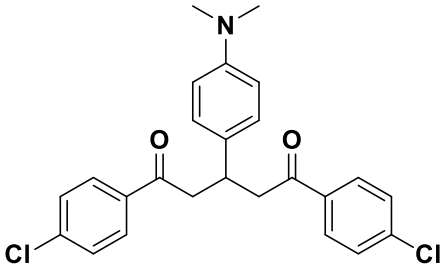
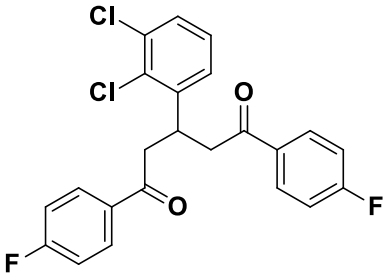
1,5-Bis(4-chlorophenyl)-3-[4-(dimethylamino)phenyl]-1,5-pentanedione (3h)[2]



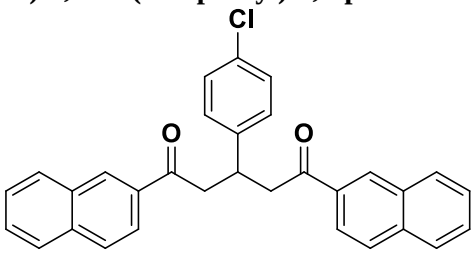
Isolated as pale yellow solid; mp: 82 – 84 °C; IR (KBr): 3061, 3029, 2894, 1679 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ_H: 7.89 (d, *J* = 8.4 Hz, 4H), 7.41 (d, *J* = 8.3 Hz, 4H), 7.11 (d, *J* = 8.6 Hz, 2H), 6.65 (d, *J* = 8.6 Hz, 2H), 3.91 (p, *J* = 7.0 Hz, 1H), 3.42 (dd, *J* = 16.4, 7.1 Hz, 2H), 3.25 (dd, *J* = 16.4, 6.9 Hz, 2H), 2.90 (s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ_C: 197.9, 149.6, 139.5, 135.4, 131.1, 129.7, 129.0, 128.1, 112.9, 45.4, 40.7, 36.6. HRMS (ESI) *m/z* calcd for $C_{25}H_{23}Cl_2NO_2$: 440.3616 $[M]^+$; Found: 440.3617 $[M]^+$. Anal. Calcd for $C_{25}H_{23}Cl_2NO_2$: C, 68.19; H, 5.26; N, 3.18%. Found: C, 68.14; H, 5.30; N, 3.22%.

3-(2,3-Dichlorophenyl)-1,5-bis(4-fluorophenyl)-1,5-pentanedione (3i)



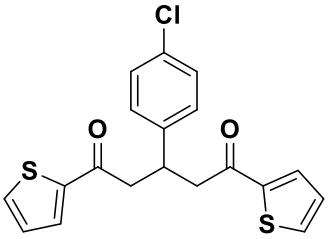
Isolated as colorless solid; mp: 70 – 72 °C; IR (KBr): 3063, 3031, 2896, 1677 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 8.02 – 7.97 (m, 4H), 7.31 (d, *J* = 7.8, 1H), 7.23 (d, *J* = 7.5 Hz, 1H), 7.16 – 7.09 (m, 5H), 4.56 (p, *J* = 6.9 Hz, 1H), 3.55 – 3.35 (m, 4H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 196.5, 167.6, 164.2, 147.0, 143.1, 133.1, 130.9, 130.7, 128.8, 127.3, 115.9, 115.6, 42.7, 34.8. HRMS (ESI) *m/z* calcd for $C_{23}H_{16}Cl_2F_2O_2$: 433.2747 $[M]^+$; Found: 433.2749 $[M]^+$. Anal. Calcd. for $C_{23}H_{16}Cl_2F_2O_2$: C, 63.76; H, 3.72%. Found: C, 63.81; H, 3.75%.

3-(4-c)-1,5-bis(2-naphthyl)-1,5-pentanedione (3j)[2]



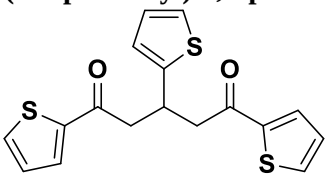
Isolated as colorless solid; mp: 120 – 122 °C; IR (KBr): 3057, 3028, 2889, 1679 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ_H: 8.50 (s, 2H), 8.01 - 7.83 (m, 8H), 7.63 - 7.51 (m, 4H), 7.30 – 7.23 (m, 4H), 4.19 (p, *J* = 6.9 Hz, 1H), 3.67 (dd, *J* = 16.7, 6.7 Hz, 2H), 3.47 (dd, *J* = 16.7, 7.2 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 198.2, 142.3, 135.6, 134.0, 132.5, 132.3, 129.9, 129.6, 128.9, 128.8, 128.5, 128.4, 127.7, 126.8, 123.8, 44.8, 36.8. HRMS (ESI) *m/z* calcd for: 462.9661 $[M]^+$; Found: 462.9665 $[M]^+$. Found: %.Anal. Calcd. for $C_{31}H_{23}ClO_2$: C, 80.42; H, 5.01%. Found: C, 80.38; H, 5.06%.

3-(4-Chlorophenyl)-1,5-bis(thiophene-2-yl)-1,5-pentanedione (3k)[2]



Isolated as colorless crystal; mp: 110 – 111 °C; IR (KBR) 3298, 3172, 2931, 2852, 1701 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ_H: 7.76 (dd, *J* = 3.8, 1.1 Hz, 2H), 7.64 (dd, *J* = 5.0, 1.1 Hz, 2H), 7.25-7.23 (m, 4H), 7.13 (dd, *J* = 4.9, 3.8 Hz, 2H), 4.09 (p, *J* = 6.0 Hz, 1H), 3.43 (dd, *J* = 16.2, 6.8 Hz, 2H), 3.26 (dd, *J* = 16.2, 7.4 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ_C: 190.9, 143.9, 141.6, 133.8, 132.3, 132.1, 128.8, 128.6, 128.1, 45.0, 37.0. HRMS (ESI) *m/z* calcd for $C_{19}H_{15}ClO_2S_2$: 374.9042 $[M]^+$; Found: 374.9047 $[M]^+$. Anal. Calcd for $C_{19}H_{15}ClO_2S_2$: C, 60.87; H, 4.03; S, 17.10%. Found: C, 60.89; H, 4.07; S, 17.12%.

1,3,5-Tris(thiophen-2-yl)-1,5-pentanedione (3l)



Isolated as colorless crystal; mp: 103 – 105 °C; IR (KBR) 3297, 3168, 2934, 2859, 1695 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ_H: 7.76 (d, *J* = 3.8 Hz, 2H), 7.62 (d, *J* = 4.9 Hz, 2H), 7.16 - 7.07 (m, 3H), 6.92 - 6.82 (m, 2H), 4.39 (p, *J* = 6.9 Hz, 1H), 3.47 (dd, *J* = 16.3, 6.8 Hz, 2H), 3.34 (dd, *J* = 16.2, 7.0 Hz, 2H).^{13}C NMR (75 MHz, $CDCl_3$) δ_C: 190.6, 143.8, 133.6, 132.0, 127.9, 126.5, 124.2, 123.2, 121.9, 45.7, 32.7. ESI-MS *m/z* calcd for $C_{17}H_{14}O_2S_3$: 346.47 $[M]^+$; Found: 347.22 $[M+H]^+$. Anal. Calcd. for $C_{17}H_{14}O_2S_3$; C, 58.93; H, 4.07; S, 27.76%. Found: C, 58.90; H, 4.12; S, 27.72%.

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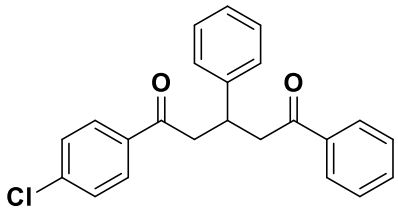
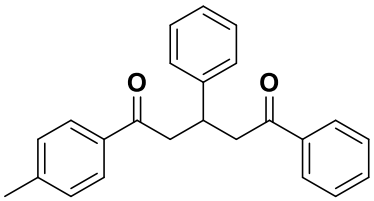
1-(4-Chlorophenyl)-3,5-diphenyl-1,5-pentanedione (3m)[2]



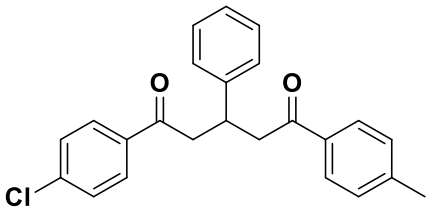
Isolated as colorless solid; mp: 97 – 99 ºC; IR (KBr): 3060, 3029, 2894, 1675, 1680 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ_H: 7.96 (d, *J* = 7.3 Hz, 2H), 7.90 (d, *J* = 8.6 Hz, 2H), 7.57 (t, *J* = 7.3 Hz, 1H), 7.49 – 7.41 (m, 4H), 7.33 – 7.22 (m, 5H), 4.10 (p, J = 6.0 Hz, 1H), 3.50 (dd, *J* = 16.8, 7.2 Hz, 2H), 3.34 – 3.27 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ_C: 198.4, 197.4, 143.5, 139.4, 136.7, 135.1, 133.1, 129.5, 128.8, 128.6, 128.5, 128.0, 127.4, 126.7, 44.8, 44.7, 37.1. HRMS (ESI) *m/z* calcd for $C_{23}H_{19}ClO_2$: 362.8488 $[M]^+$; Found: 362.8490 $[M]^+$. Anal. Calcd for $C_{23}H_{19}ClO_2$: C, 76.13; H, 5.28%. Found: C, 76.18; H, 5.25%.

1-(4-Methylphenyl)-3,5-diphenyl-1,5-pentanedione (3n)[3]



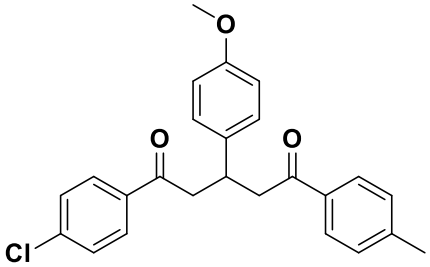
Isolated as colorless solid; mp: 99 – 101 ºC; IR (KBr): 3058, 3027, 2890, 1677, 1679 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 8.01 (dt, *J* = 3.5, 1.5 Hz, 3H), 7.59 (dt, *J* = 10.4, 1.4 Hz, 2H), 7.52 – 7.46 (m, 3H), 7.34 – 7.13 (m, 6H), 4.17 – 4.05 (m, 1H), 3.58 – 3.50 (m, 2H), 3.42 – 3.37 (m, 2H), 2.34 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 198.6, 198.5, 143.8, 140.7, 136.9, 136.1, 132.9, 129.3, 129.2, 128.5, 128.2, 128.1, 127.4, 127.2, 44.8, 37.2, 21.6. ESI-MS *m/z* calcd for $C_{24}H_{22}O_2$: 342.43 $[M]^+$; Found: 343.22 $[M+H]^+$. Anal. Calcd for $C_{24}H_{22}O_2$: C, 84.18; H, 6.48%. Found: C, 84.22; H, 6.43%.

1-(4-Chlorophenyl)-5-(4-methylphenyl)-3-phenyl-1,5-pentanedione (3o)



Isolated as colorless solid; mp: 101 – 102 ºC; IR (KBr): 3052, 3031, 2890, 1676, 1681 cm^{-1}; ^1H NMR (500 MHz, Chloroform) δ 7.65 (d, *J* = 7.5 Hz, 2H), 7.60 (d, *J* = 7.5 Hz, 2H), 7.35 – 7.25 (m, 6H), 7.23 – 7.15 (m, 3H), 3.63 (p, *J* = 6.8 Hz, 1H), 3.44 (dd, *J* = 12.4, 8.0 Hz, 1H), 3.37 (dd, *J* = 12.5, 3.3 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 198.51, 143.7, 143.6, 138.6, 135.8, 129.3, 128.9, 128.8, 128.2, 127.9, 127.5, 127.1, 44.2, 38.7, 20.8. HRMS (ESI) *m/z* calcd for: 376.8753 $[M]^+$; Found: 376.8756 $[M]^+$. Ana. Calcd. for $C_{24}H_{21}ClO_2$; C, 76.49; H, 5.62%. Found: C, 76.54; H, 5.68%.

1-(4-Chlorophenyl)-3-(4-methoxyphenyl)-5-(4-methylphenyl)-1,5-pentanedione (3p)



Isolated as colorless crystal; mp: 111 – 113 ºC; IR (KBr): 3066, 3029, 2898, 1678, 1681 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.89 (d, *J* = 6.7 Hz, 2H), 7.84 (d, *J* = 8.3 Hz, 2H), 7.40 (d, *J* = 6.7 Hz, 2H), 7.24 (d, *J* = 8.0 Hz, 2H), 7.17 (d, *J* = 8.7 Hz, 2H), 6.80 (d, *J* = 8.7 Hz, 2H), 4.02 – 3.91 (m, 1H), 3.75 (s, 3H), 3.43 (ddd, *J* = 16.6, 12.0, 6.8 Hz, 2H), 3.25 (ddd, *J* = 16.2, 14.0, 7.1 Hz, 2H), 2.40 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 198.3, 197.6, 158.4, 143.9, 139.5, 135.7, 135.4, 134.6, 129.6, 129.3, 128.9, 128.4, 128.3, 114.1, 55.2, 45.1, 45.0, 36.6, 21.7. HRMS (ESI) *m/z* calcd for: 406.9013 $[M]^+$; Found: 406.9017 $[M]^+$. Anal. Calcd. for $C_{25}H_{23}ClO_3$; C, 73.79; H, 5.70%. Found: C, 73.82; H, 5.68%.

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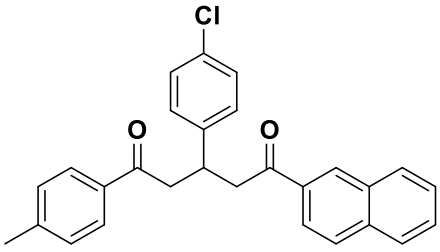
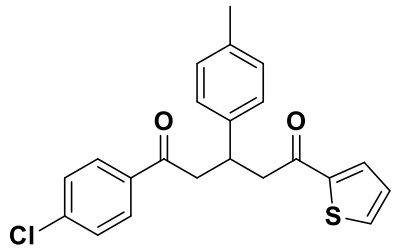
3-(4-Chlorophenyl)-1-(4-methylphenyl)-5-(2-naphthyl)-1,5-pentanedione (3q)



Isolated as colorless solid; mp: 107 – 109 ºC; IR (KBr): 3061, 3031, 2882, 1679, 1683 cm^{-1}; IR (KBr) 3288, 3068, 2947, 2852, 1718, 1696, 1654, 1091, 702 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ_H: 8.48 (s, 1H), 7.97 (t, *J* = 8.2 Hz, 2H), 7.89 – 7.84 (m, 4H), 7.64 – 7.51 (m, 3H), 7.25 (m, 5H), 4.16 (p, *J* = 6.9 Hz, 1H), 3.72 – 3.51 (m, 2H), 3.46 – 3.29 (m, 2H), 2.41 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ_C: 198.1, 197.8, 144.0, 142.4, 135.5, 134.2, 134.0, 132.4, 132.1, 129.8, 129.5, 129.3, 128.9, 128.7, 128.4, 128.4, 128.2, 127.7, 126.7, 123.7, 44.7, 44.6, 36.7, 21.6. HRMS (ESI) *m/z* calcd for: 426.9340 $[M]^+$; Found: 426.9343 $[M]^+$. Anal. Calcd. for $C_{28}H_{23}ClO_2$; C, 78.77; H, 5.43%. Found: C, 78.01; H, 5.48%.

1-(4-Chlorophenyl)-3-(4-methylphenyl)-5-(thiophene-2-yl)-1,5-pentanedione (3r)[2]



Isolated as colorless crystal; mp: 90 – 91 ºC; IR (KBR) 3288, 3171, 2939, 2849, 1705, 1698 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ_H: 7.90 (d, *J* = 6.8 Hz, 2H), 7.76 (dd, *J* = 3.8, 1.1 Hz, 1H), 7.63 (dd, *J* = 4.9, 1.1 Hz, 1H), 7.42 (d, *J* = 8.7 Hz, 2H), 7.18 – 7.08 (m, 5H), 4.04 (p, *J* = 6.0 Hz, 1H), 3.54 (dd, *J* = 15.0, 6.0 Hz, 1H), 3.43 (dd, *J* = 15.0, 6.0 Hz, 1H), 3.33-3.21 (m,2H) 2.30 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ_C: 197.7, 191.8, 144.6, 140.6, 139.8, 136.7, 135.5, 134.1, 132.5, 129.9, 129.7, 129.2, 128.5, 127.6, 46.0, 45.0, 37.5, 21.3. HRMS (ESI) *m/z* calcd for: 382.9031 $[M]^+$; Found: 382.9036 $[M]^+$. Anal. Calcd. for $C_{22}H_{19}ClO_2S$; C, 69.01; H, 5.00; S, 8.37%. Found: C, 69.06; H, 5.04; S, 8.40%.

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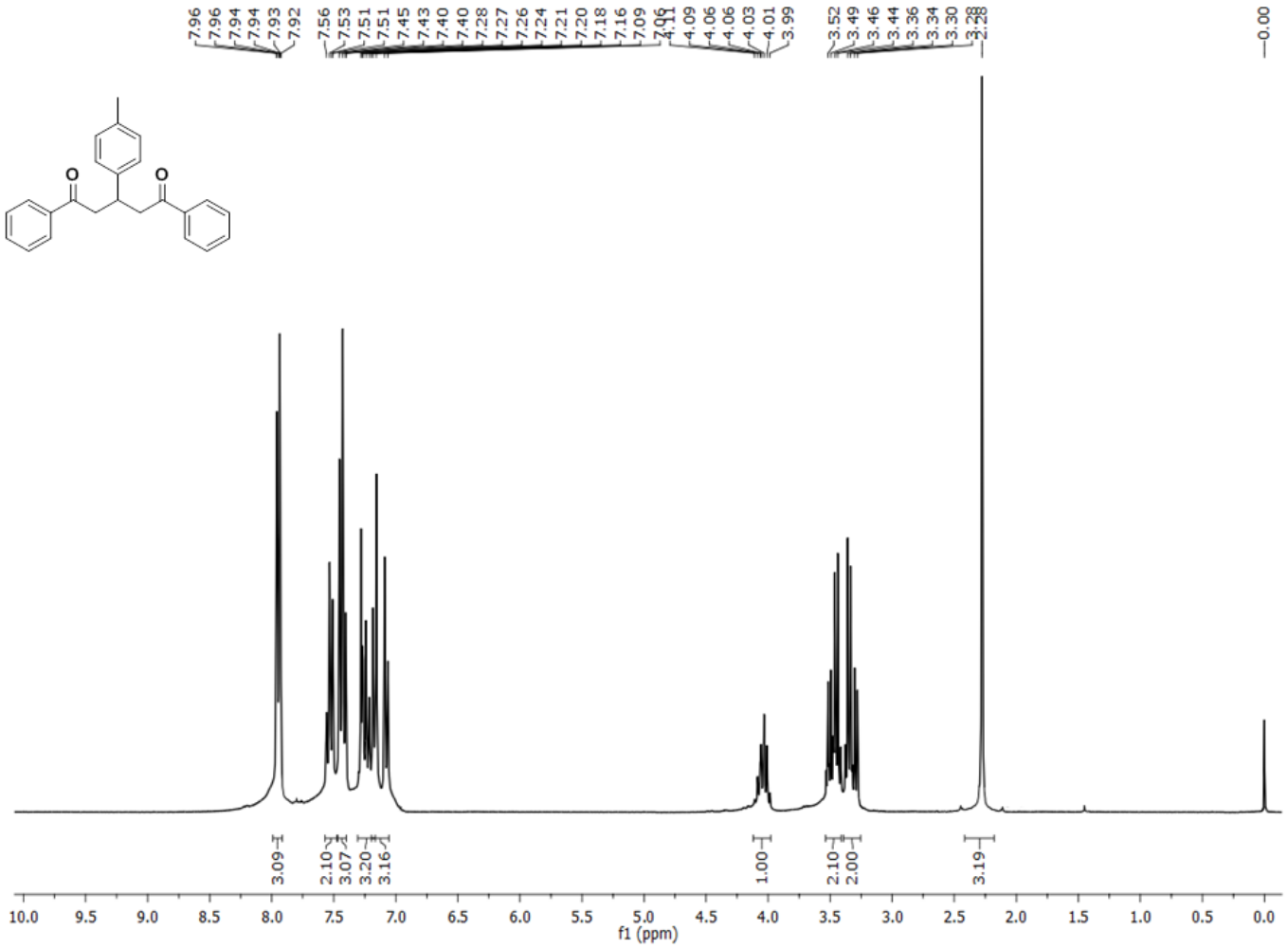

Fig. S1. ^{1}H NMR spectrum of compound 3a.

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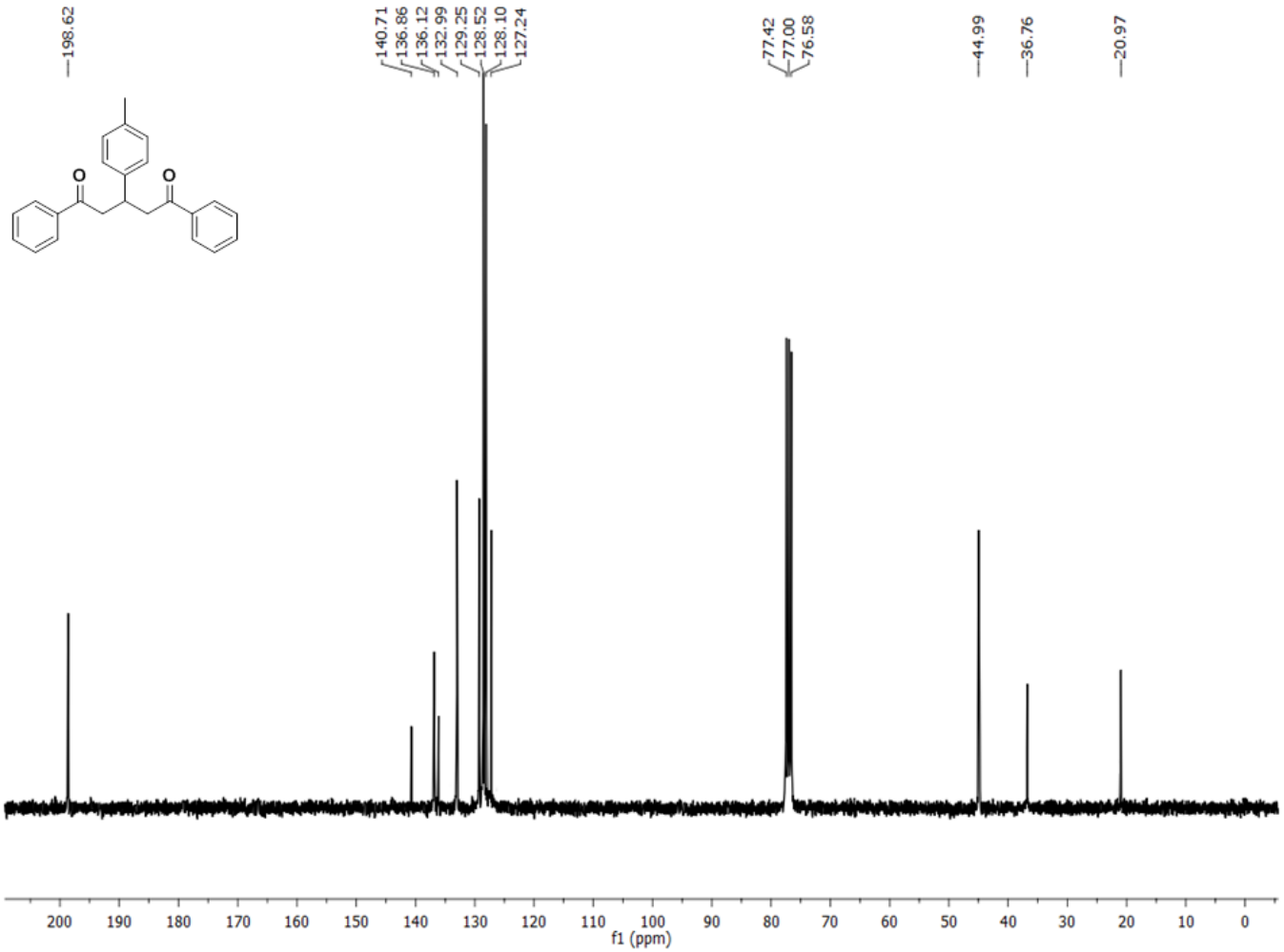

Fig. S2. ^{13}C NMR spectrum of compound 3a.

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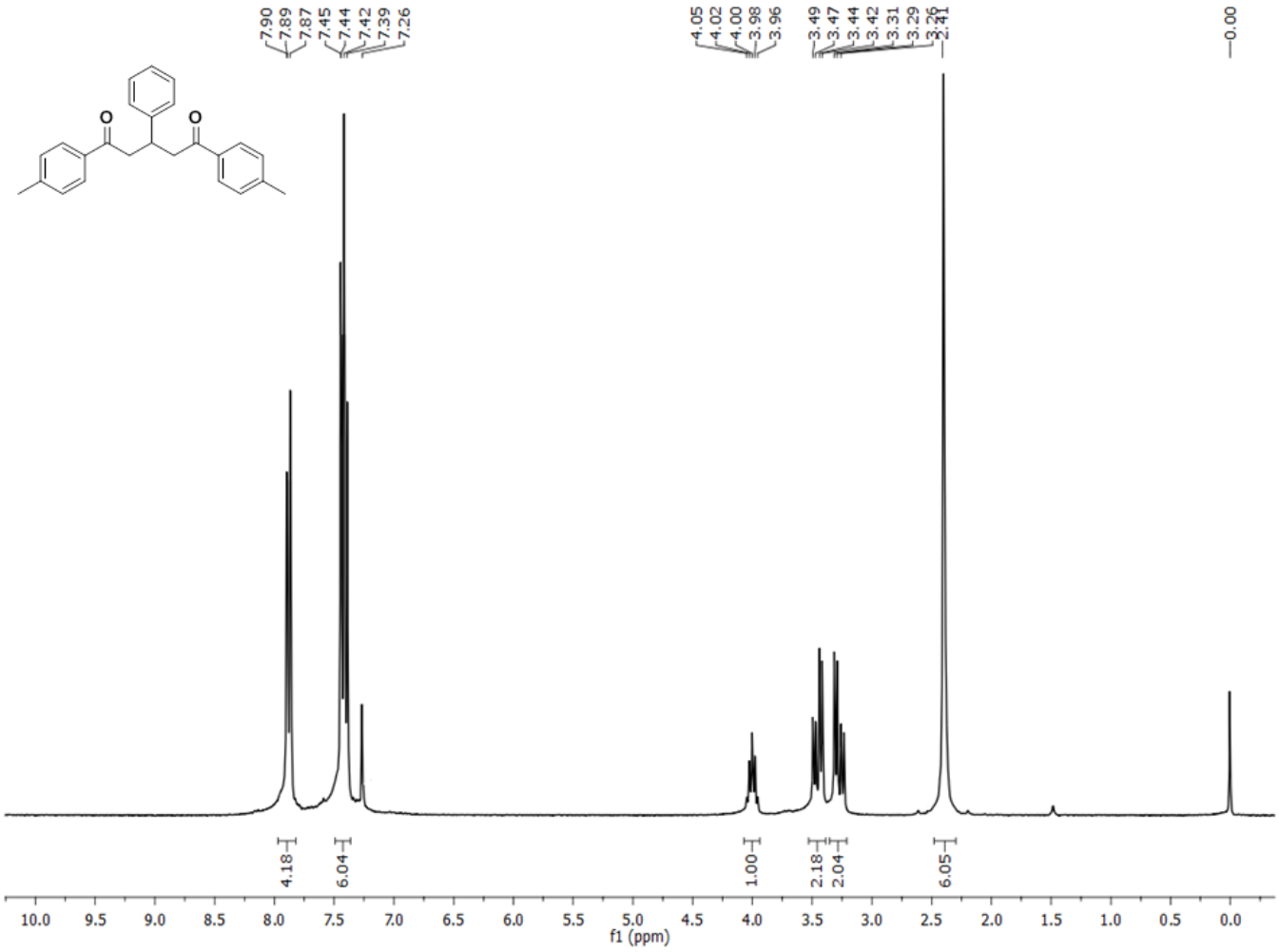

Fig. S3. ^{1}H NMR spectrum of compound 3b.

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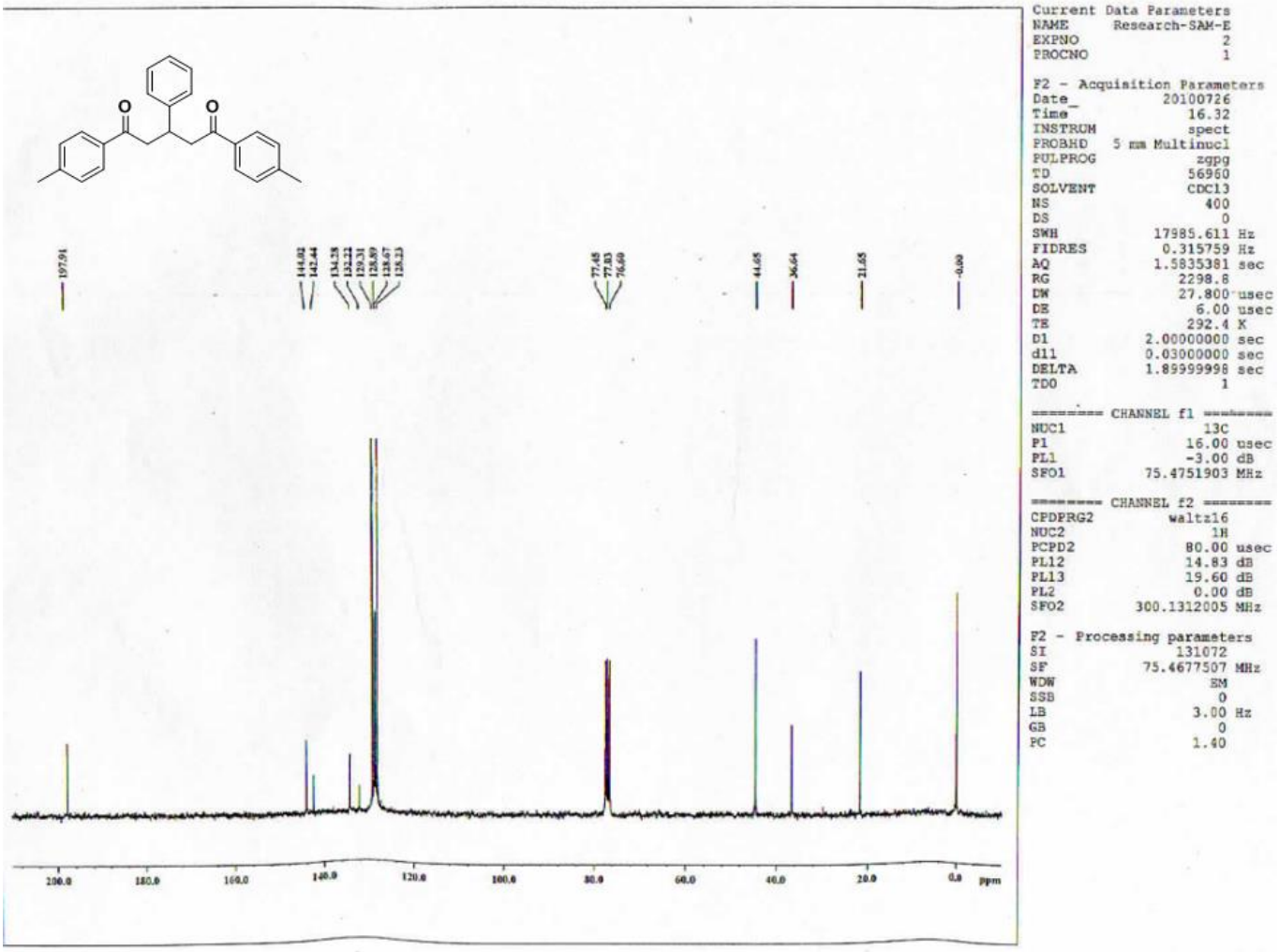

Fig. S4. ^{13}C NMR spectrum of compound 3b.

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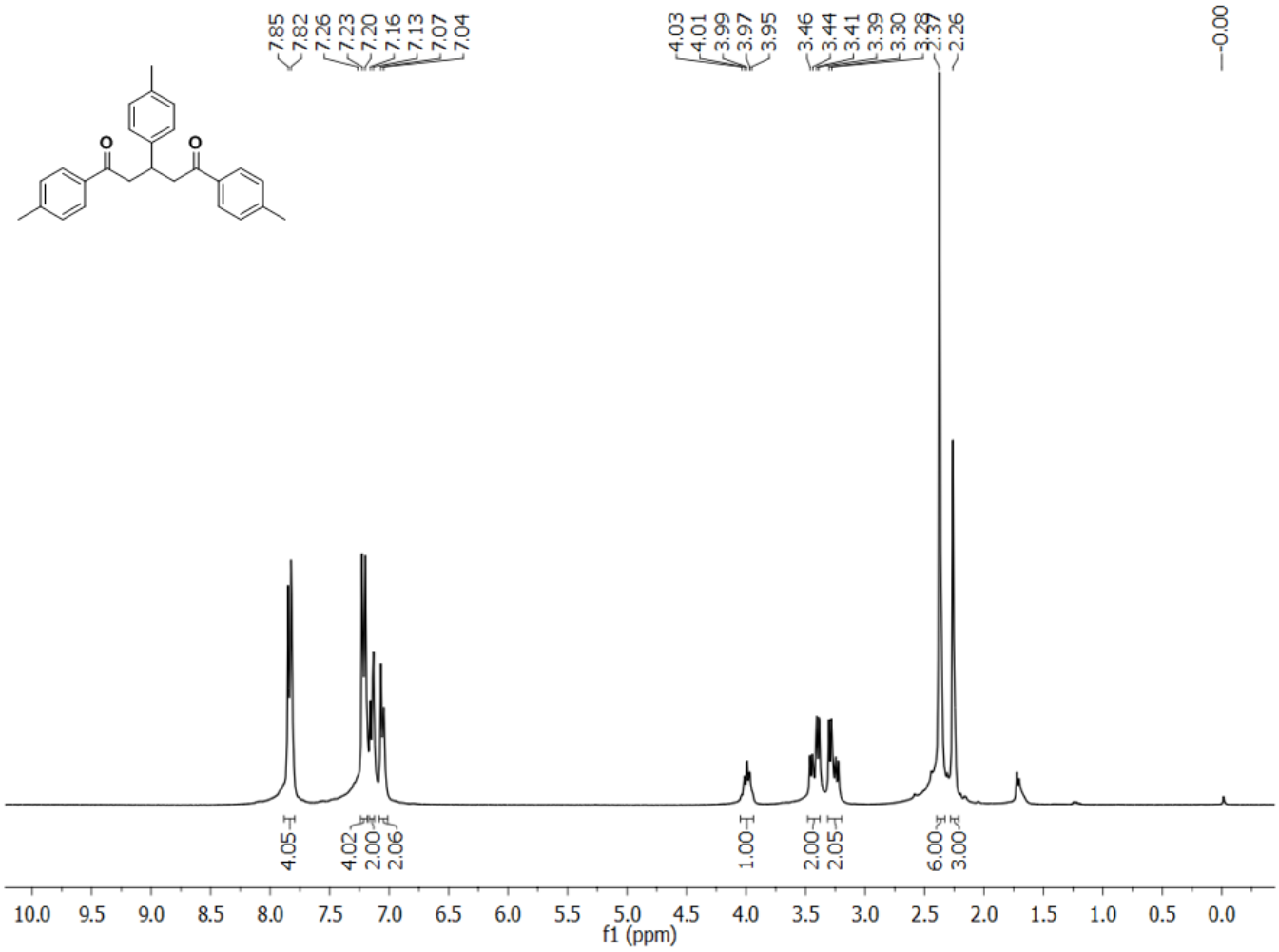

Fig. S5. ^{1}H NMR spectrum of compound 3c.

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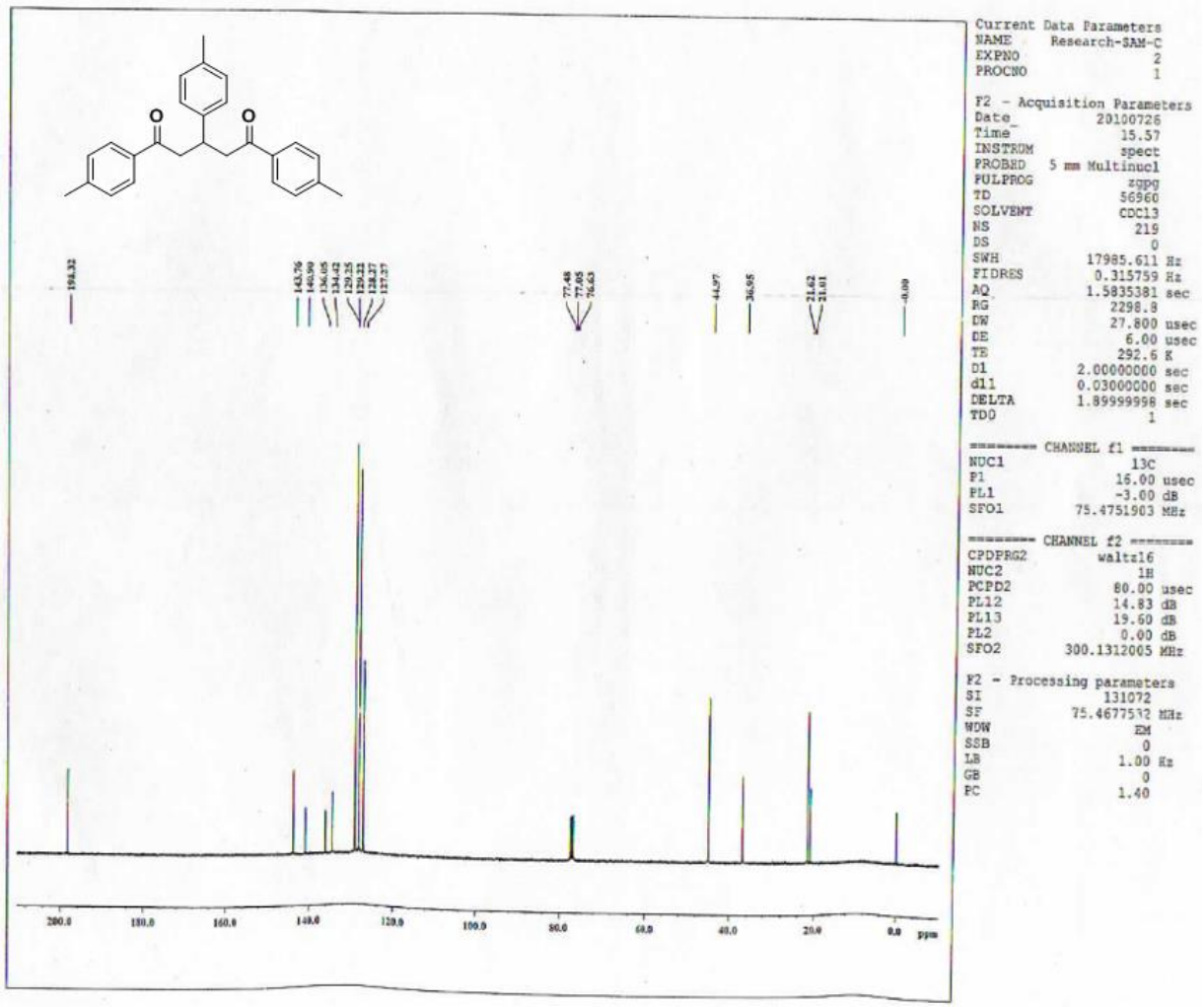

Fig. S6. ^{13}C NMR spectrum of compound 3c.

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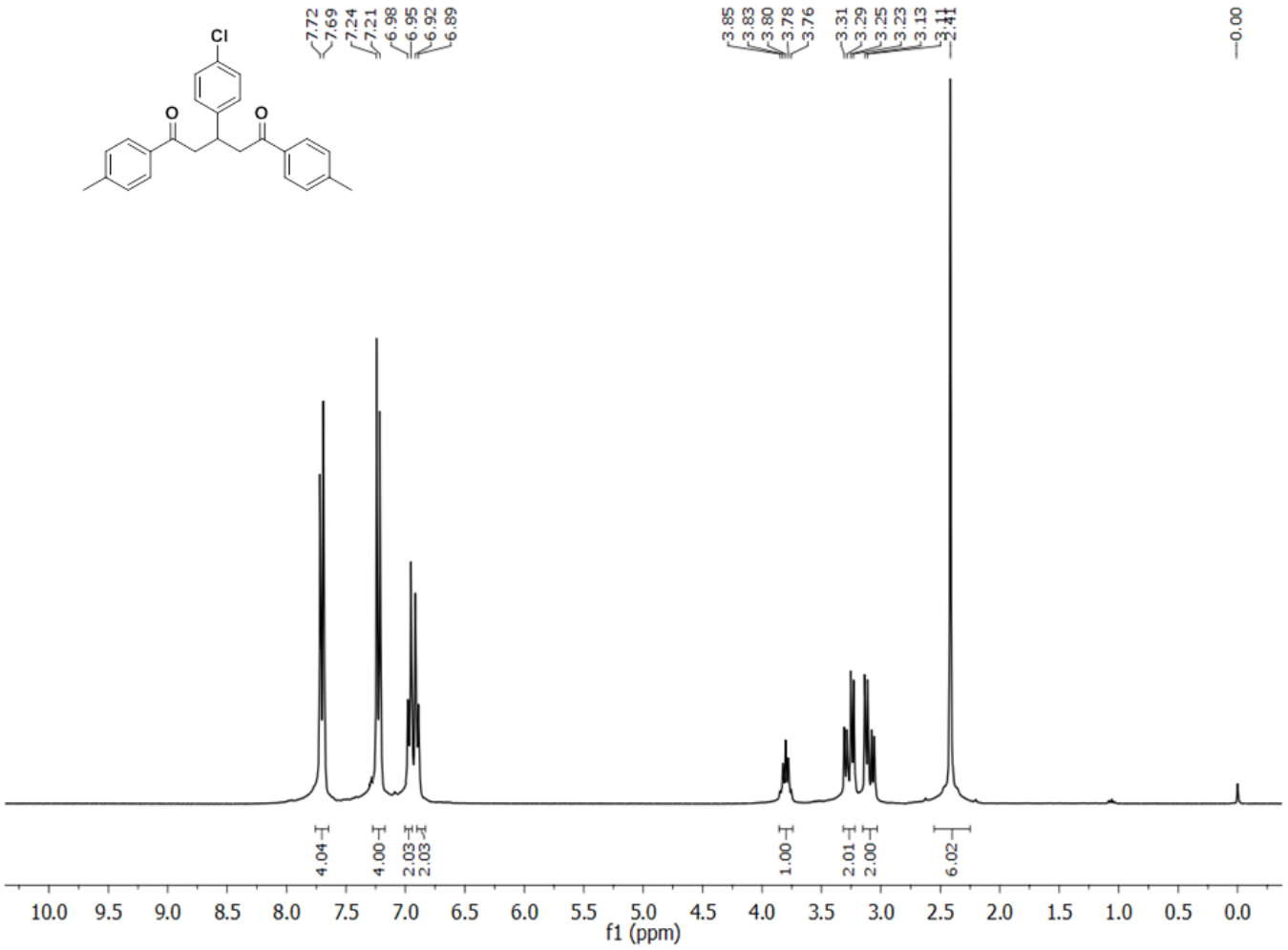

Fig. S7. ^{1}H NMR spectrum of compound 3d.

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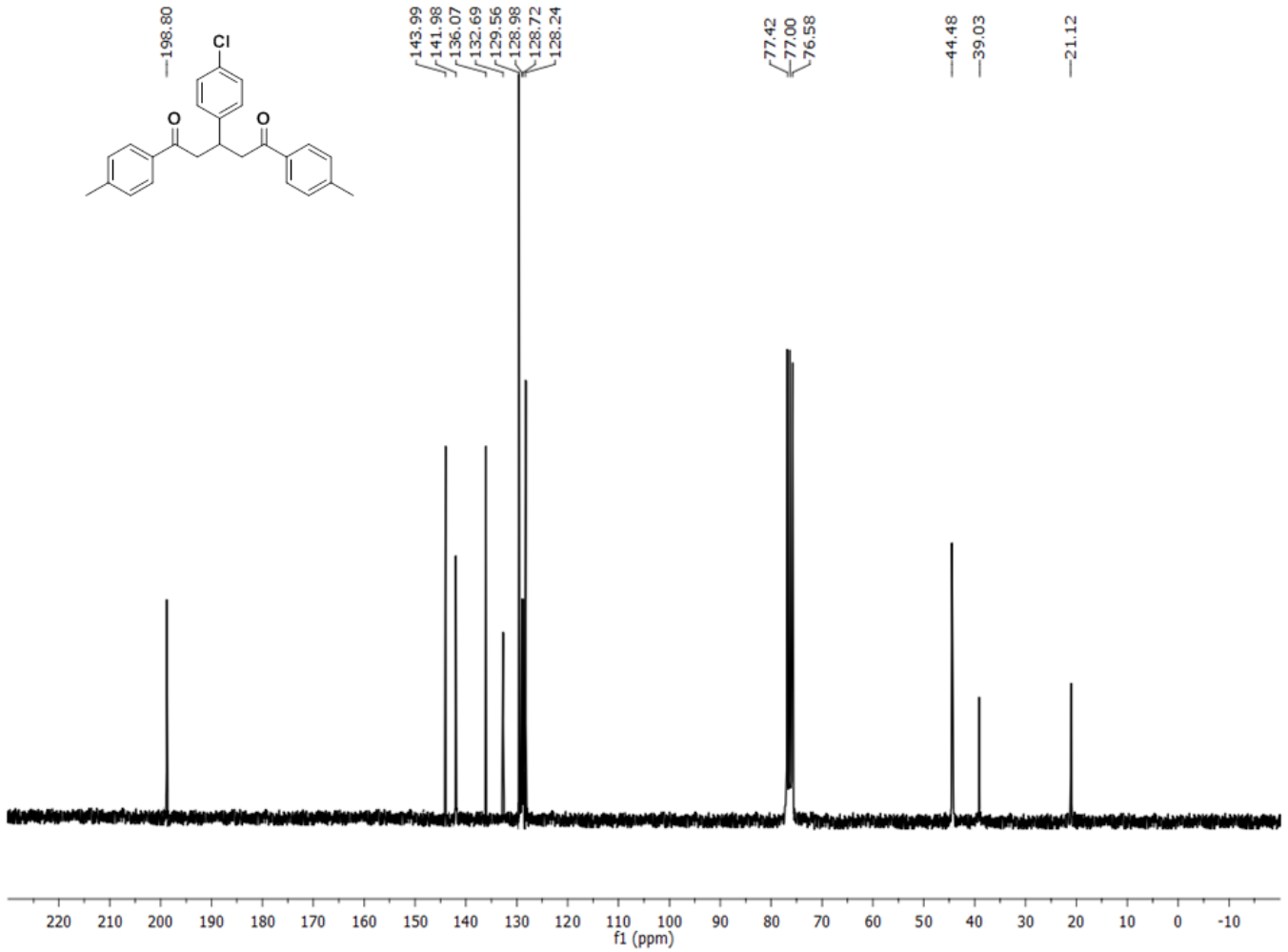

Fig. S8. ^{13}C NMR spectrum of compound 3d.

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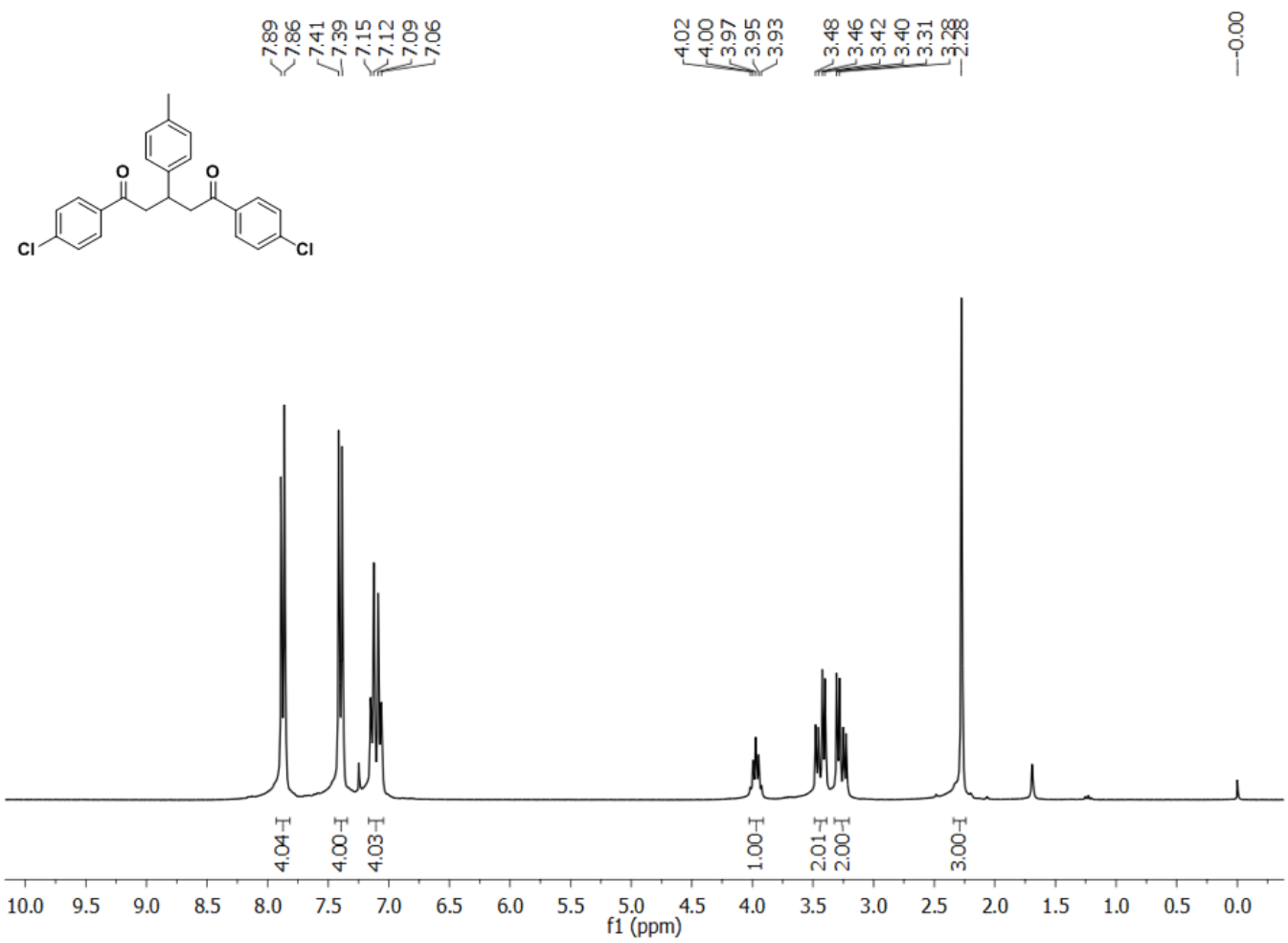

Fig. S9. ^{1}H NMR spectrum of compound 3e.

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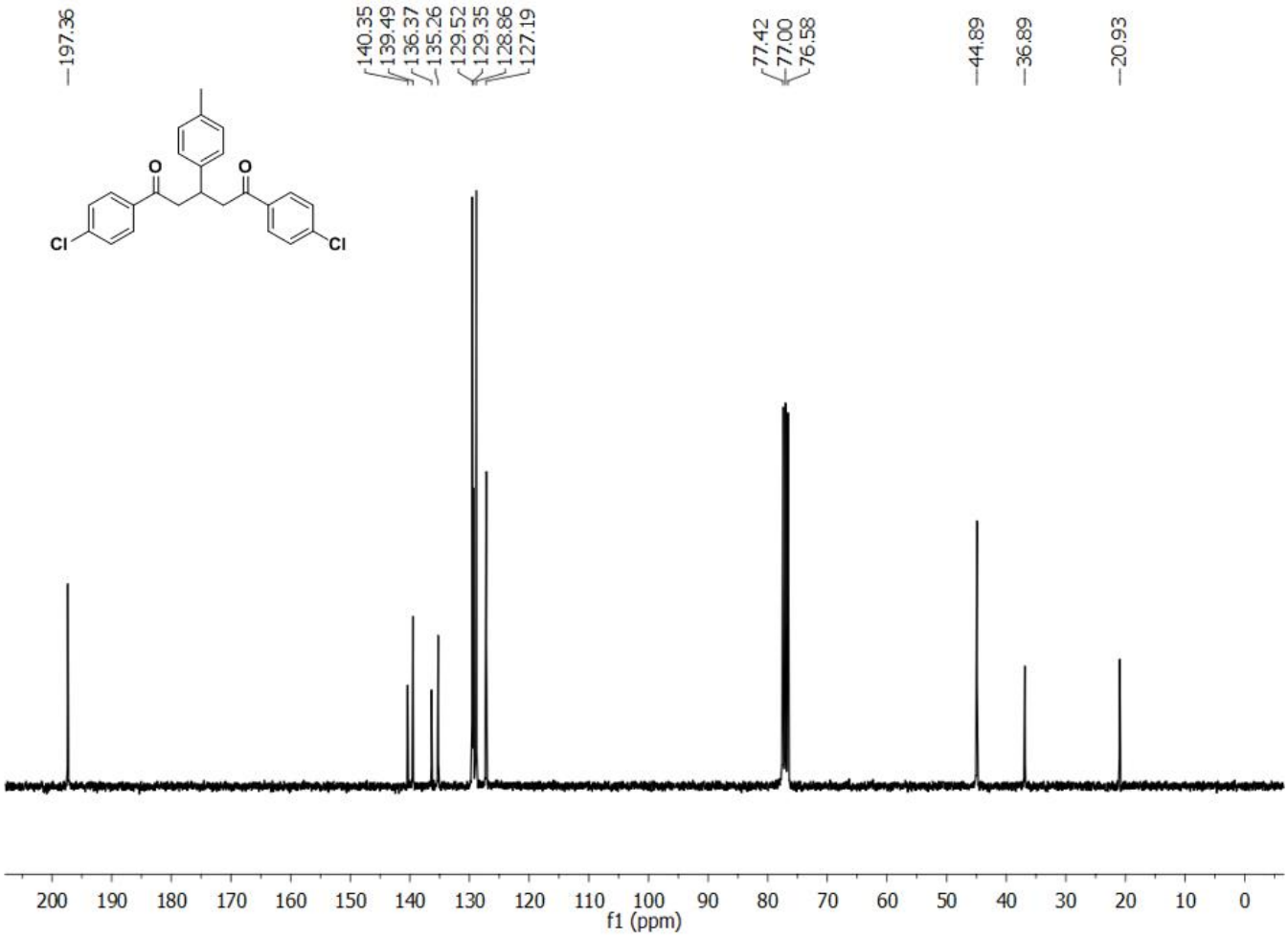

Fig. S10. ^{13}C NMR spectrum of compound 3e.

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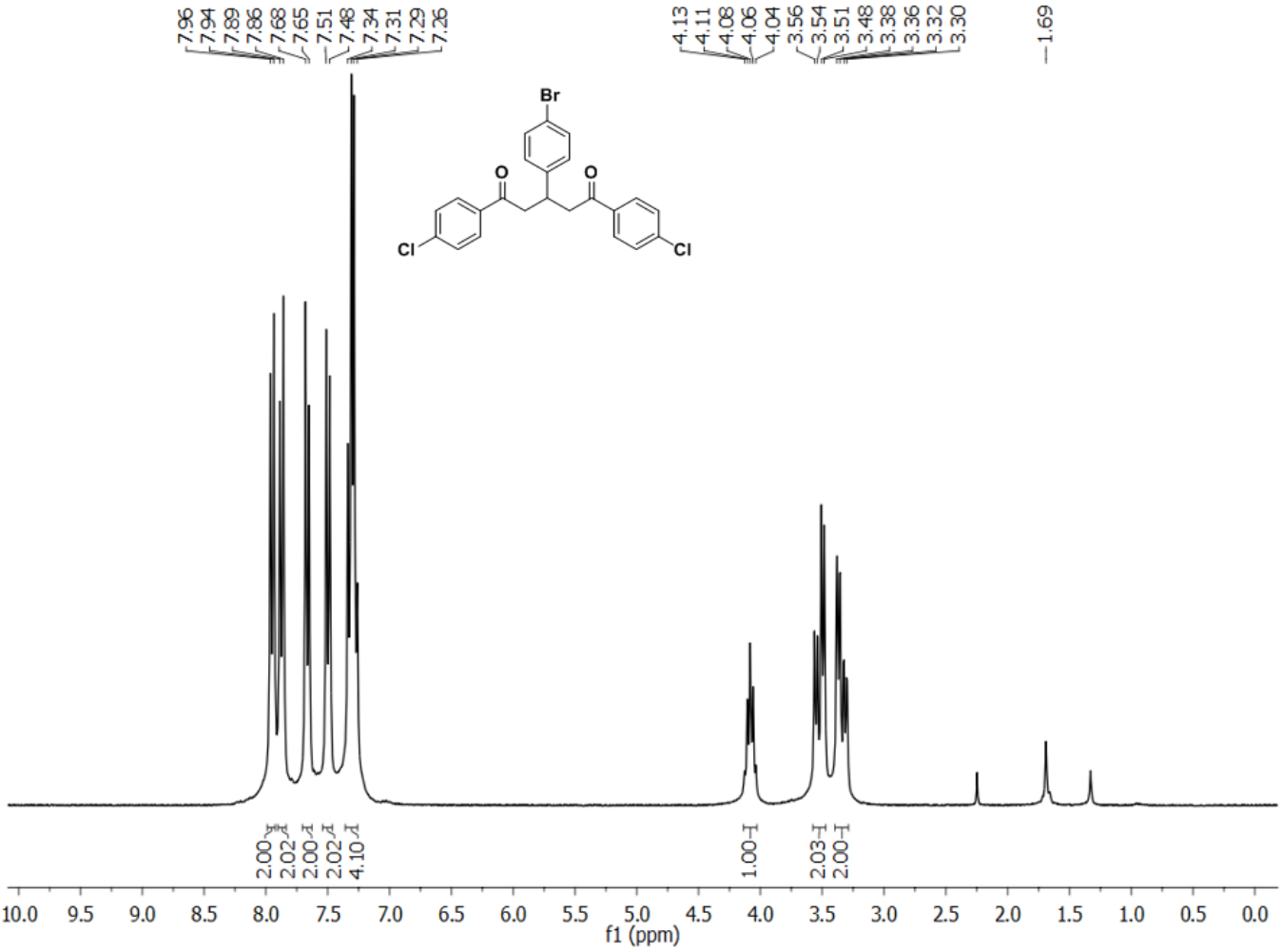

Fig. S11. ^{1}H NMR spectrum of compound 3f.

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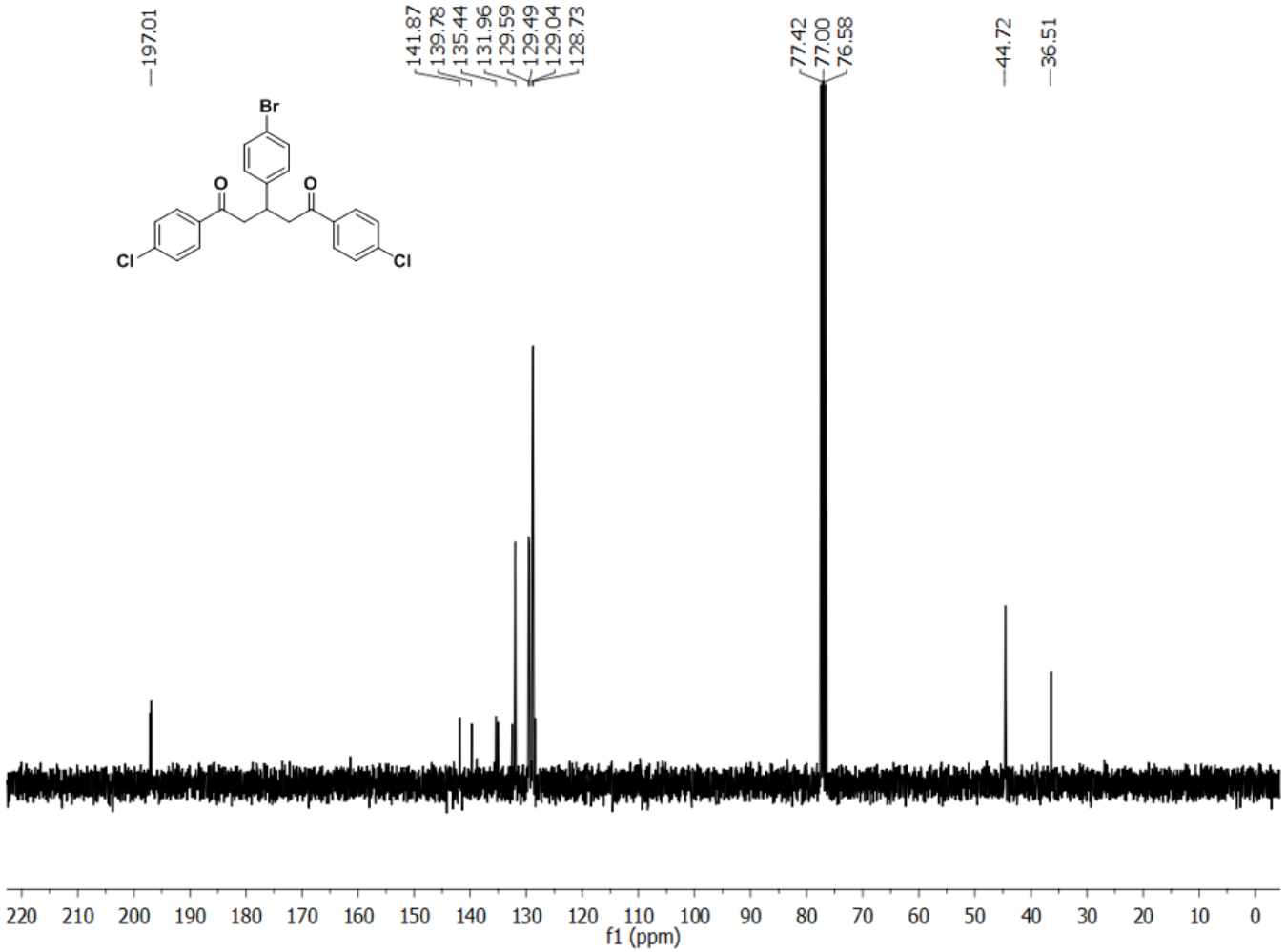

Fig. S12. ^{13}C NMR spectrum of compound 3f.

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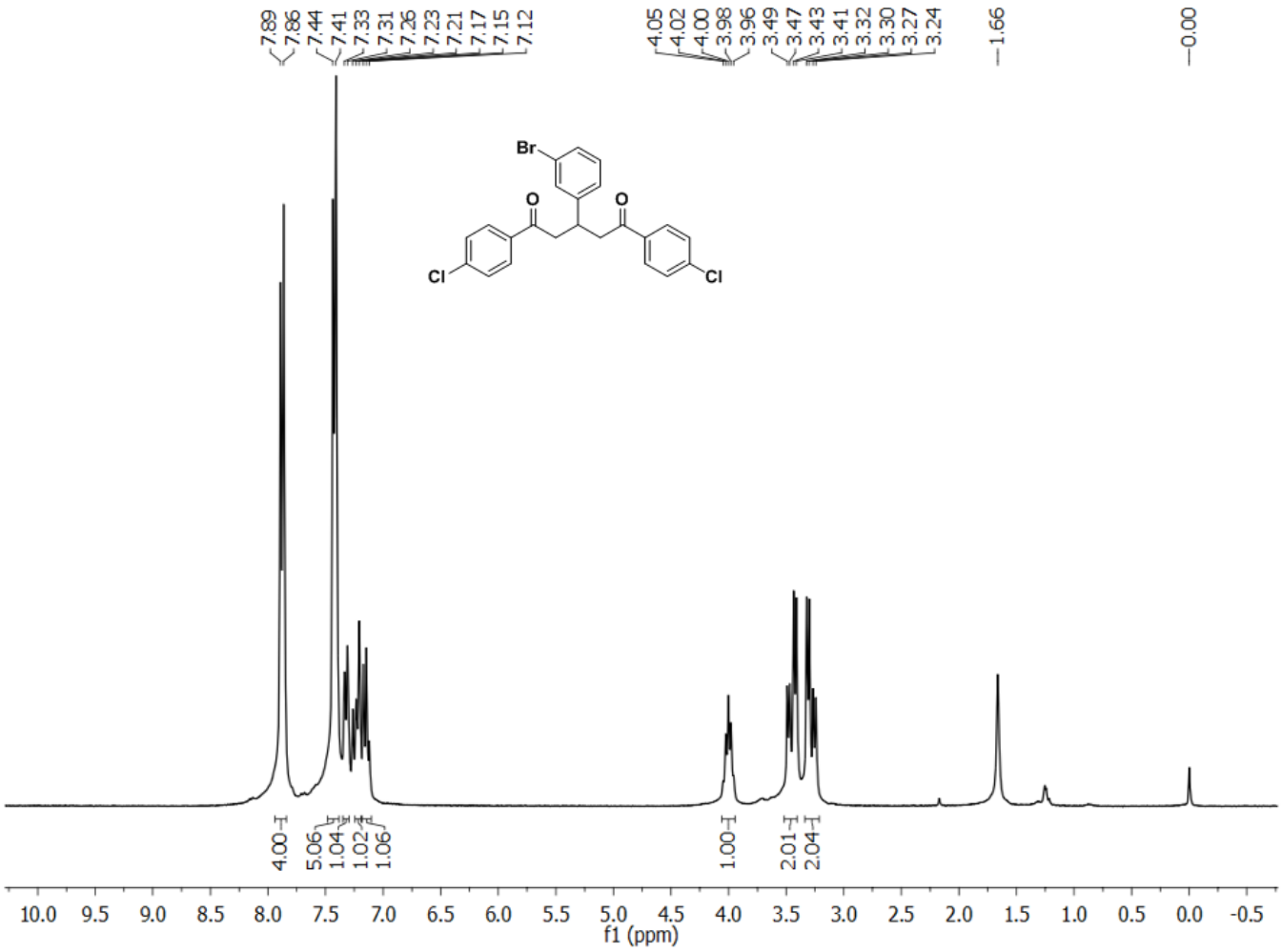

Fig. S13. ^{1}H NMR spectrum of compound 3g.

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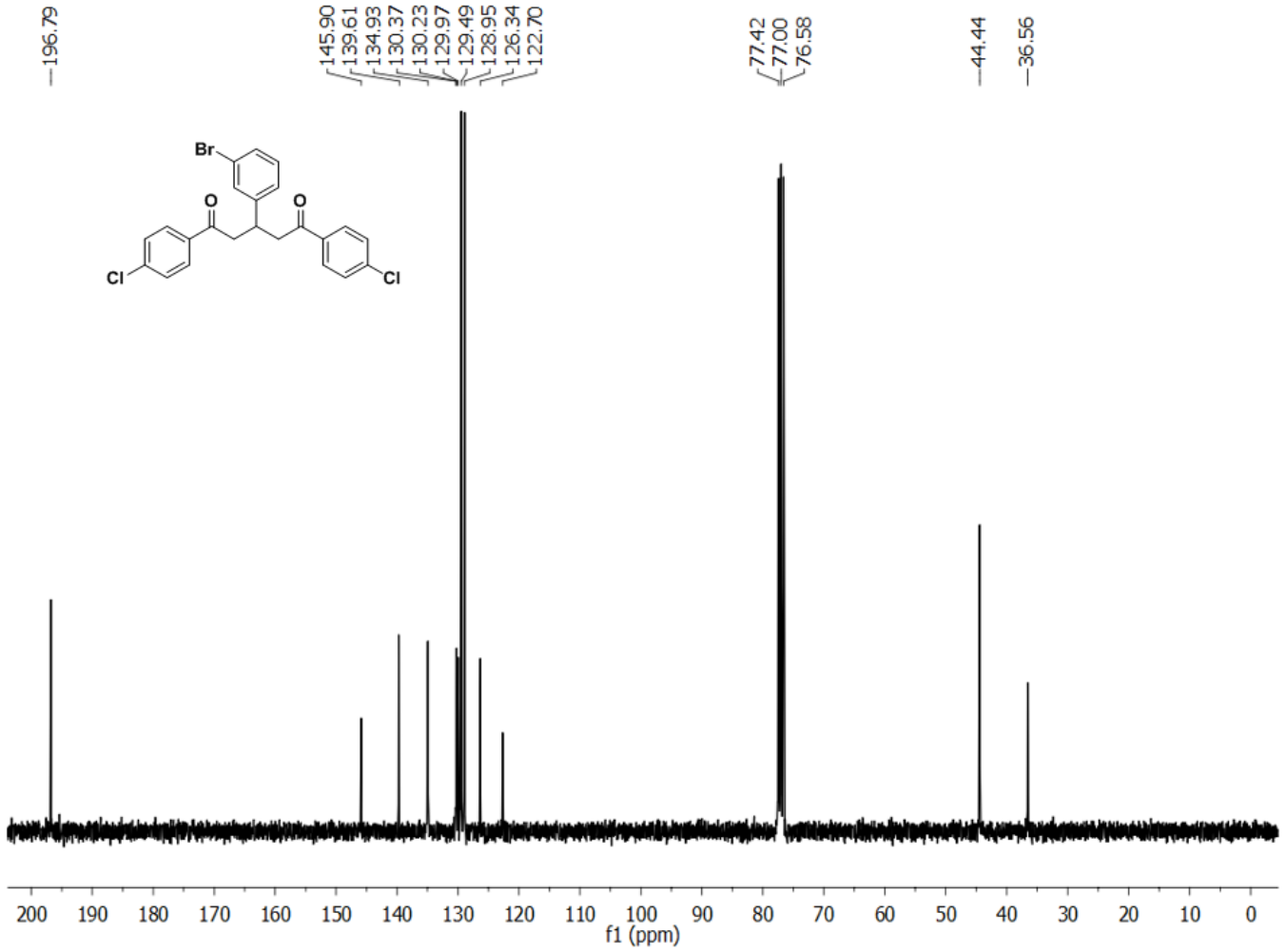

Fig. S14. ^{13}C NMR spectrum of compound 3g.

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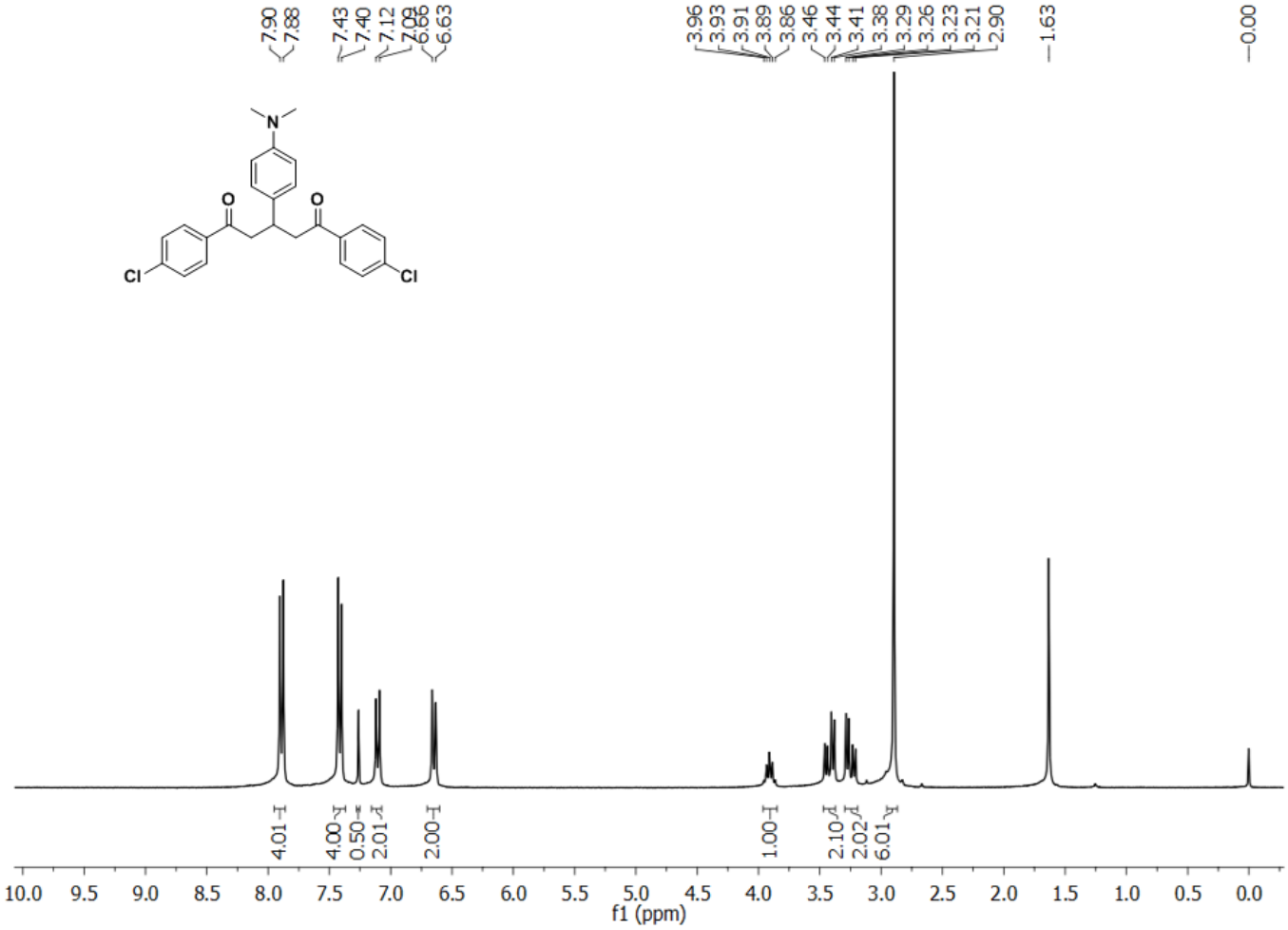

Fig. S15. ^{1}H NMR spectrum of compound 3h.

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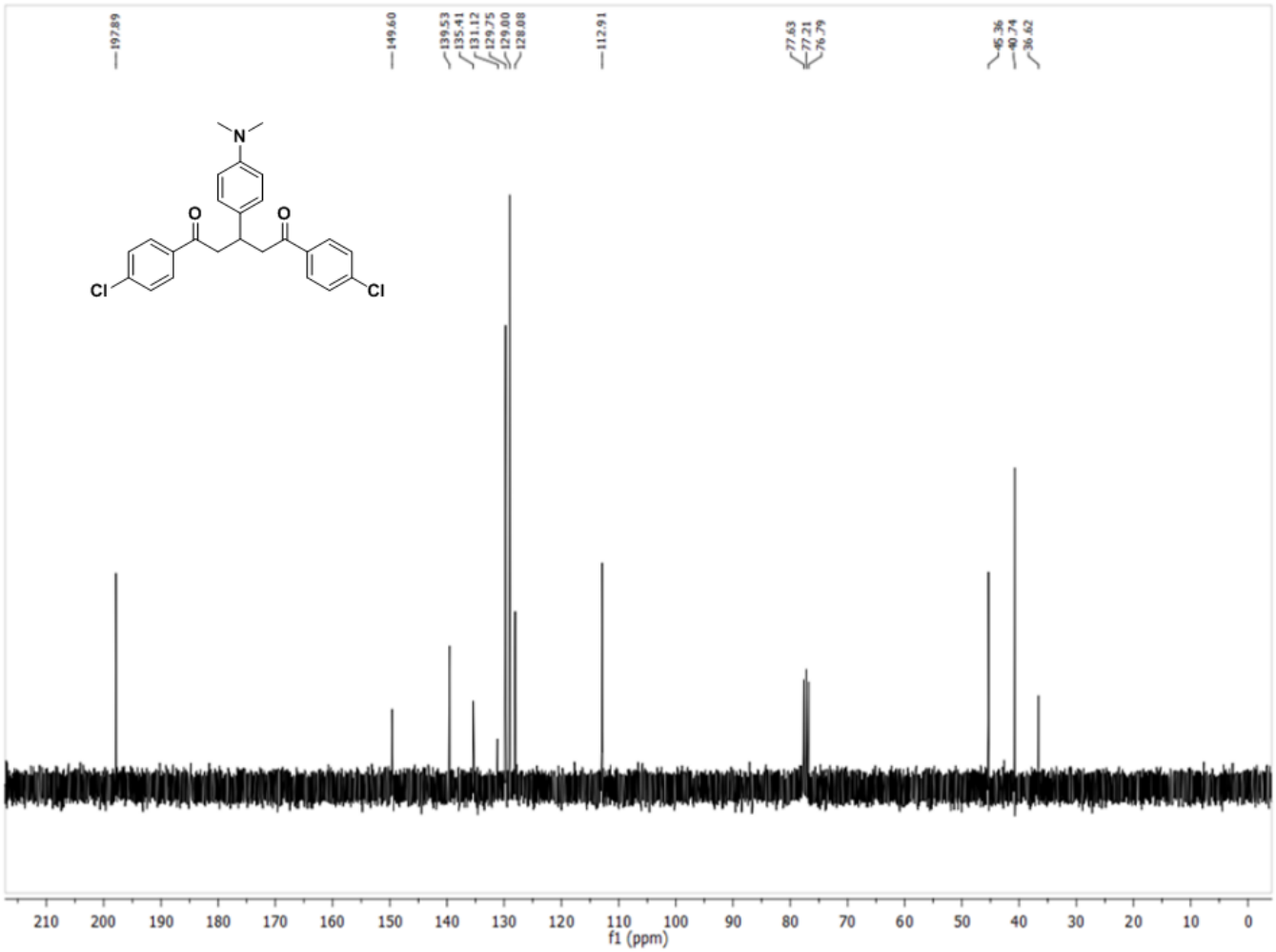

Fig. S16. ^{13}C NMR spectrum of compound 3h.

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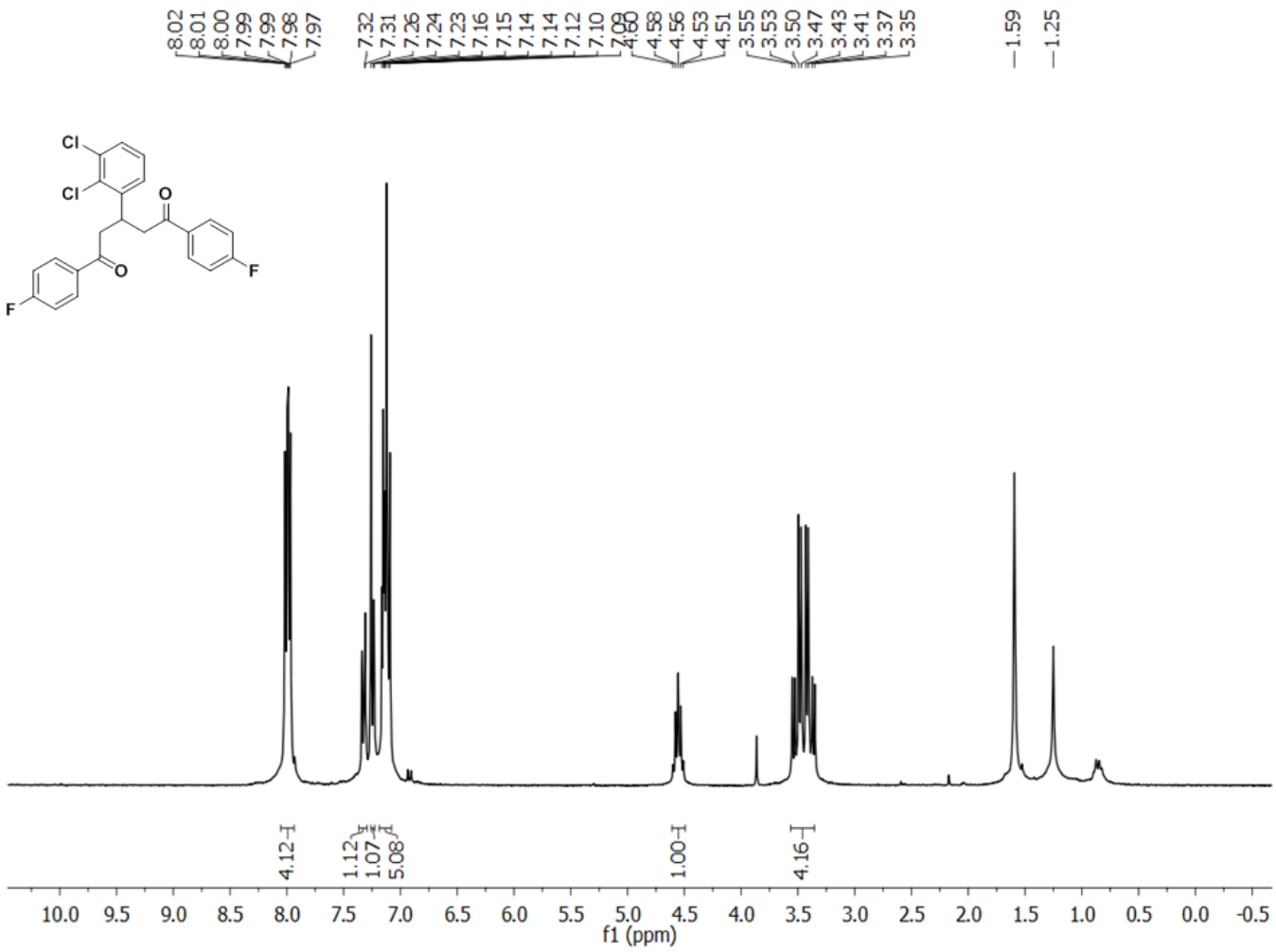

Fig. S17. ^{1}H NMR spectrum of compound 3i.

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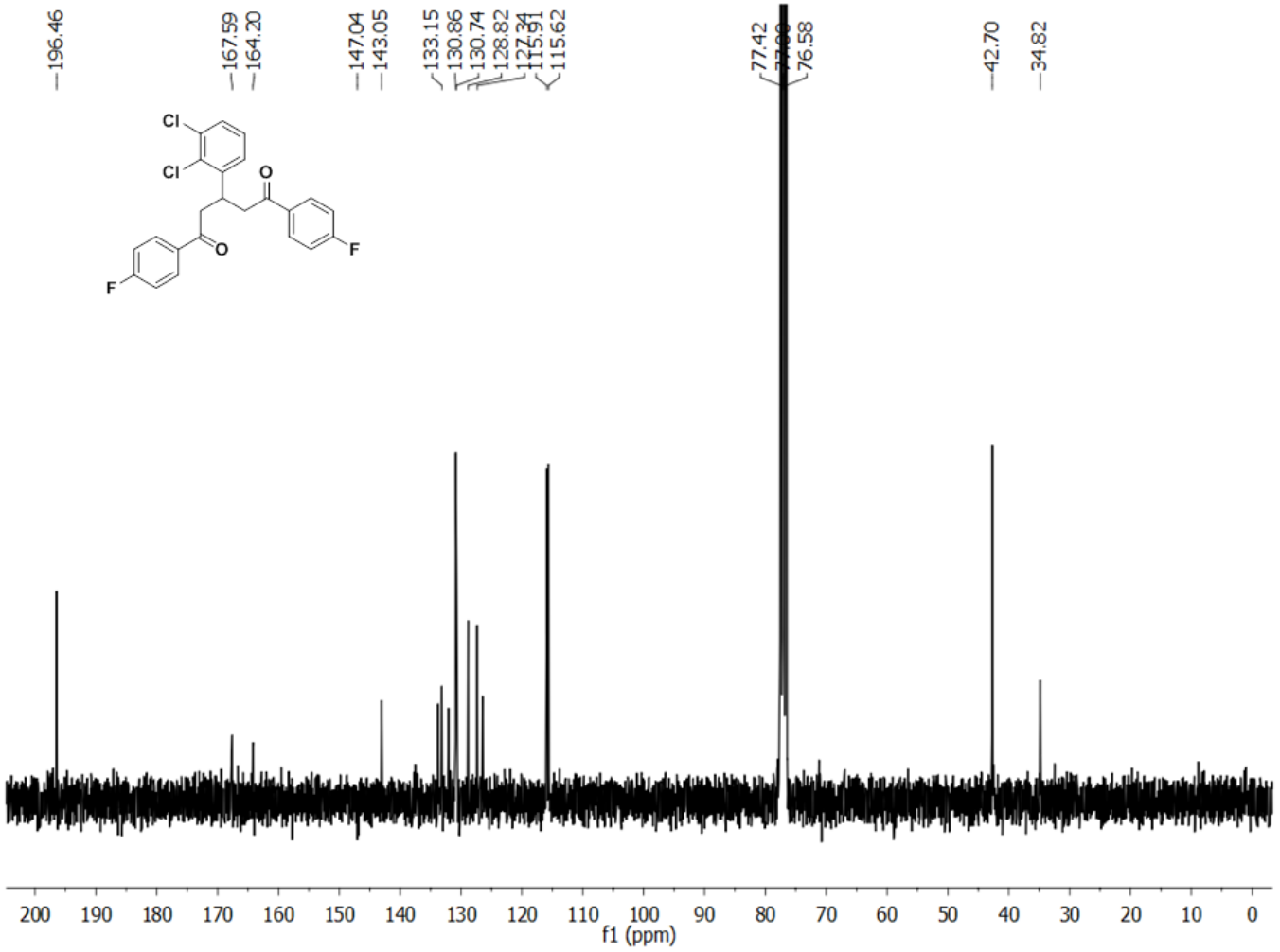

Fig. S18. ^{13}C NMR spectrum of compound 3i.

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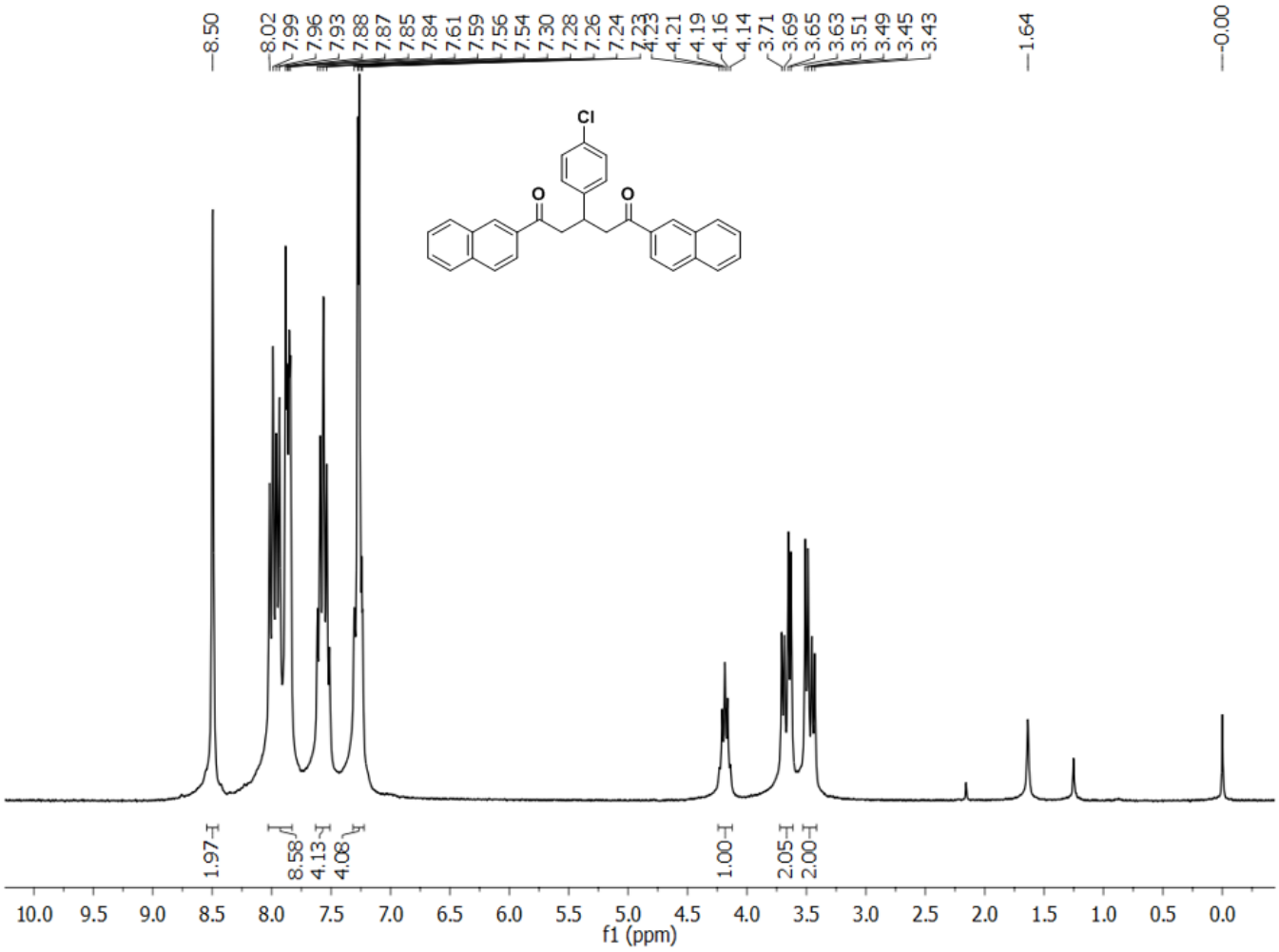

Fig. S19. ^{1}H NMR spectrum of compound 3j.

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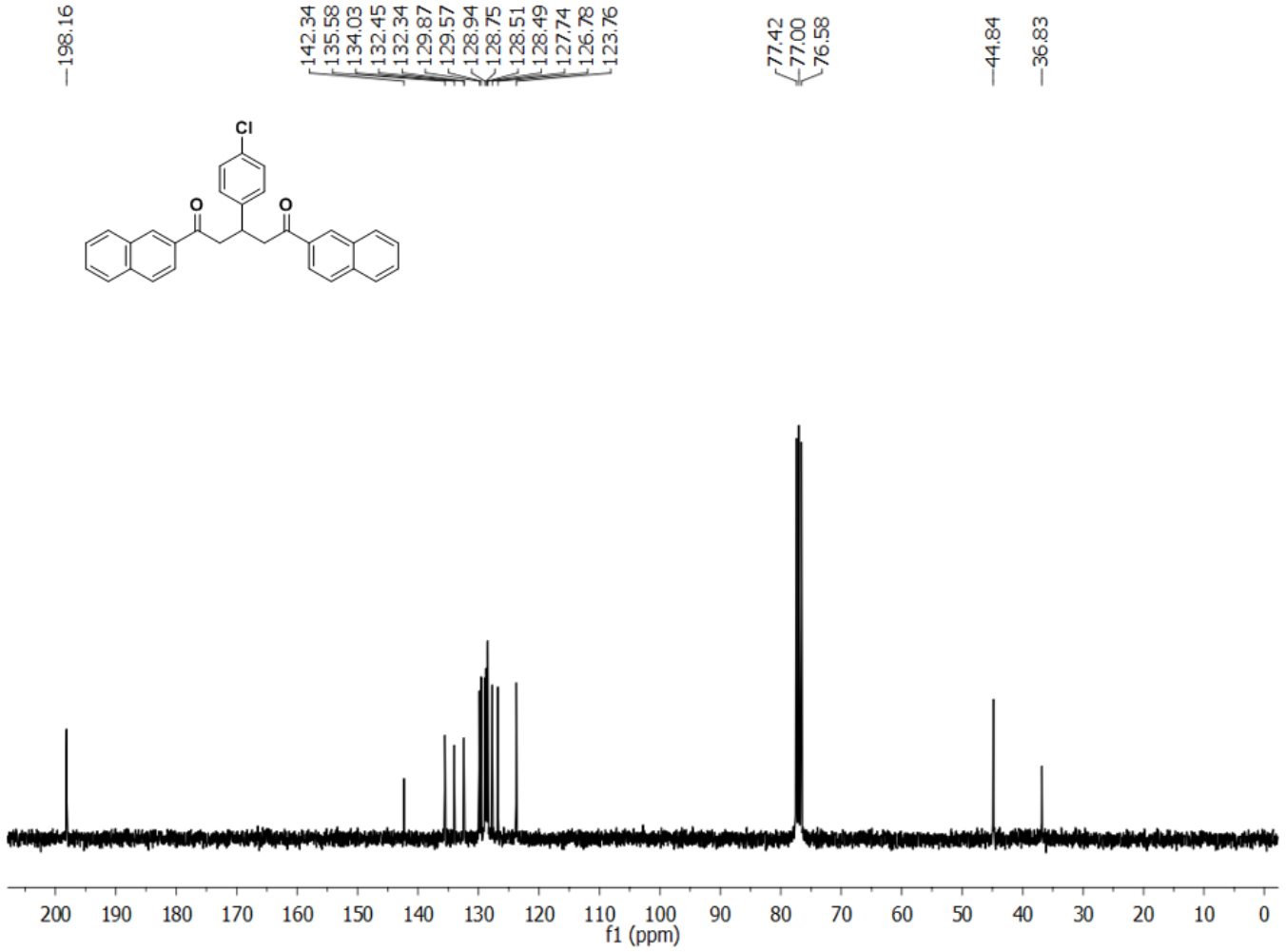

Fig. S20. ^{13}C NMR spectrum of compound 3j.

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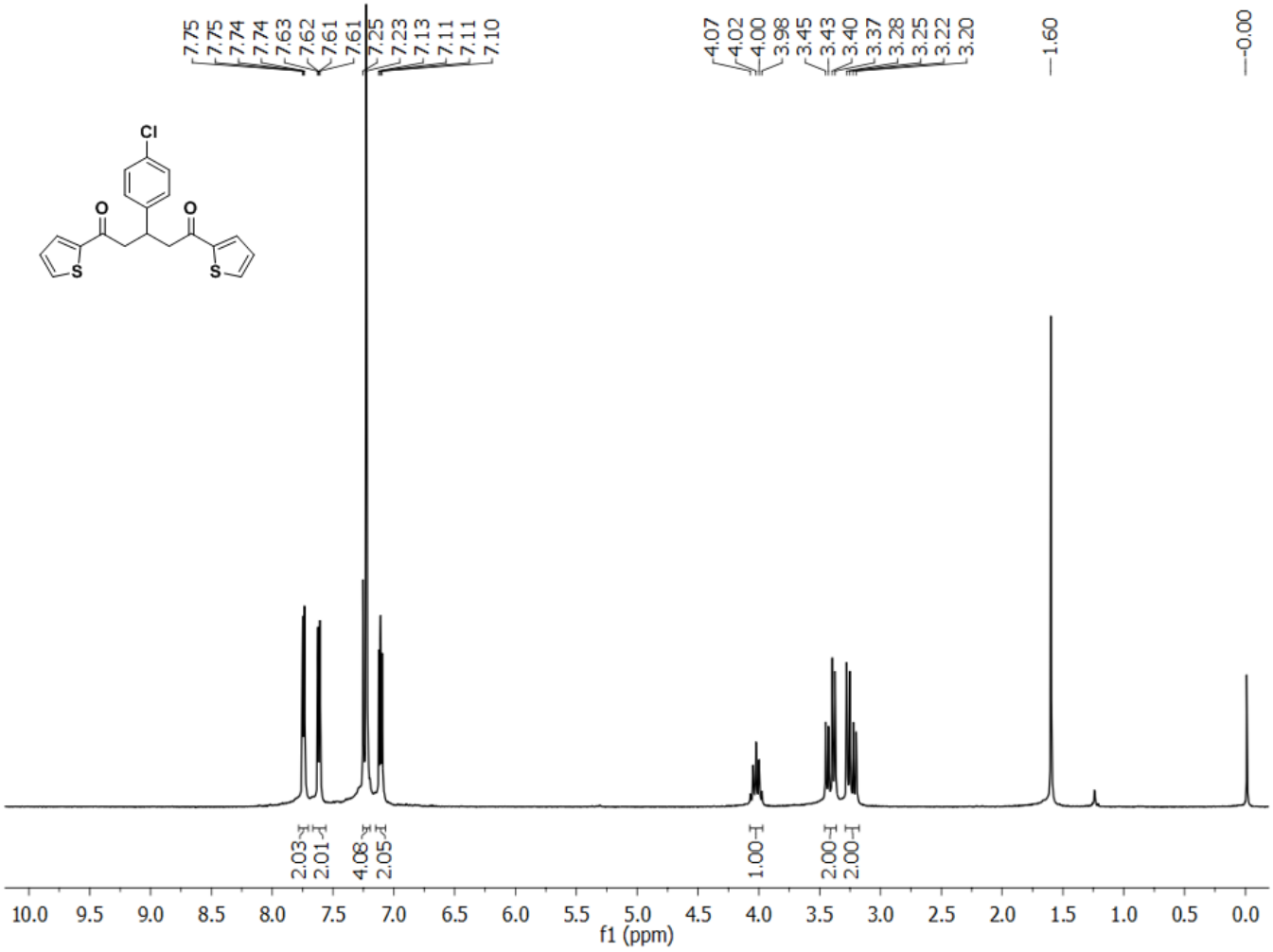

Fig. S21. ^{1}H NMR spectrum of compound 3k.

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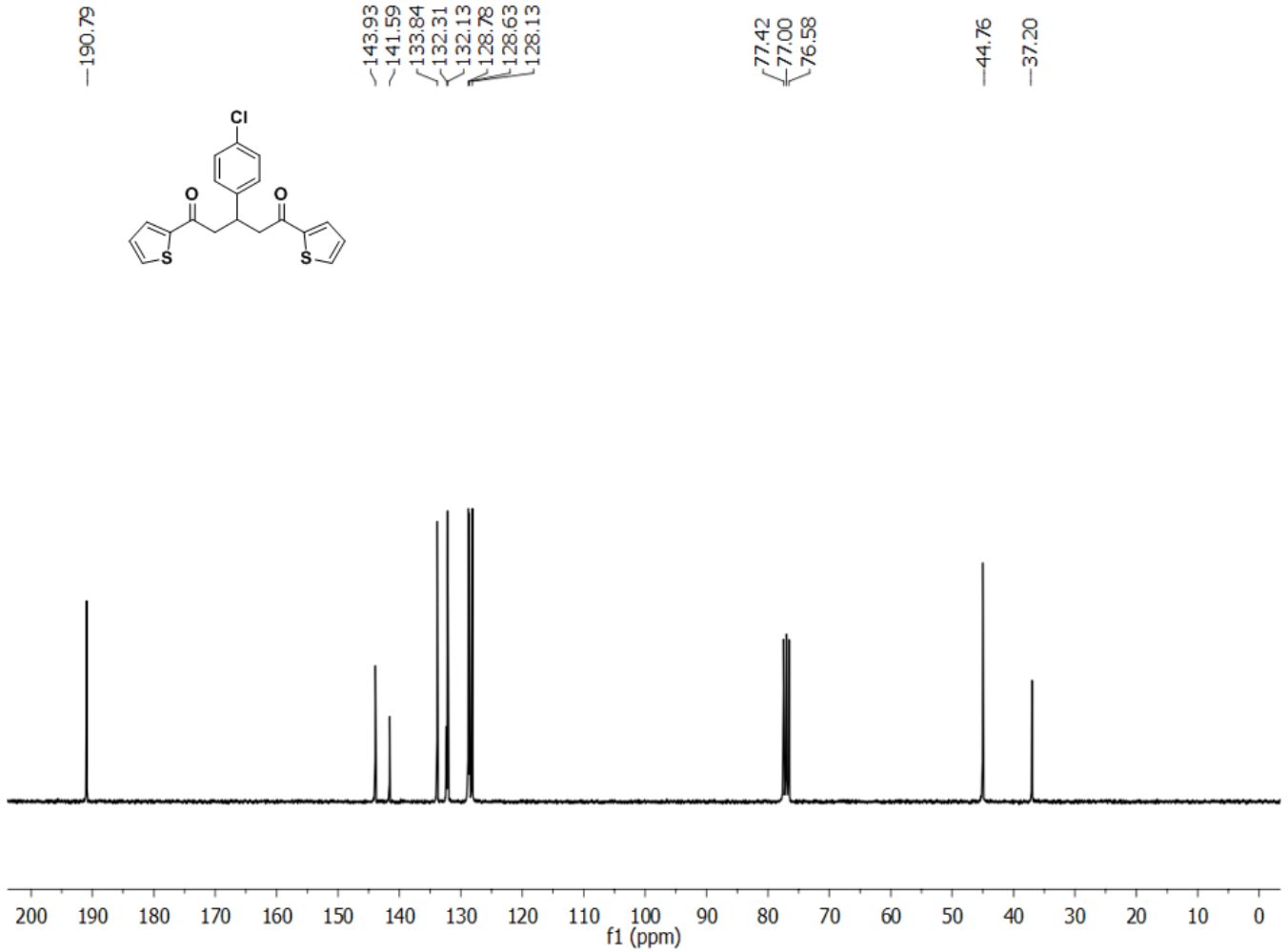

Fig. S22. ^{13}C NMR spectrum of compound 3k.

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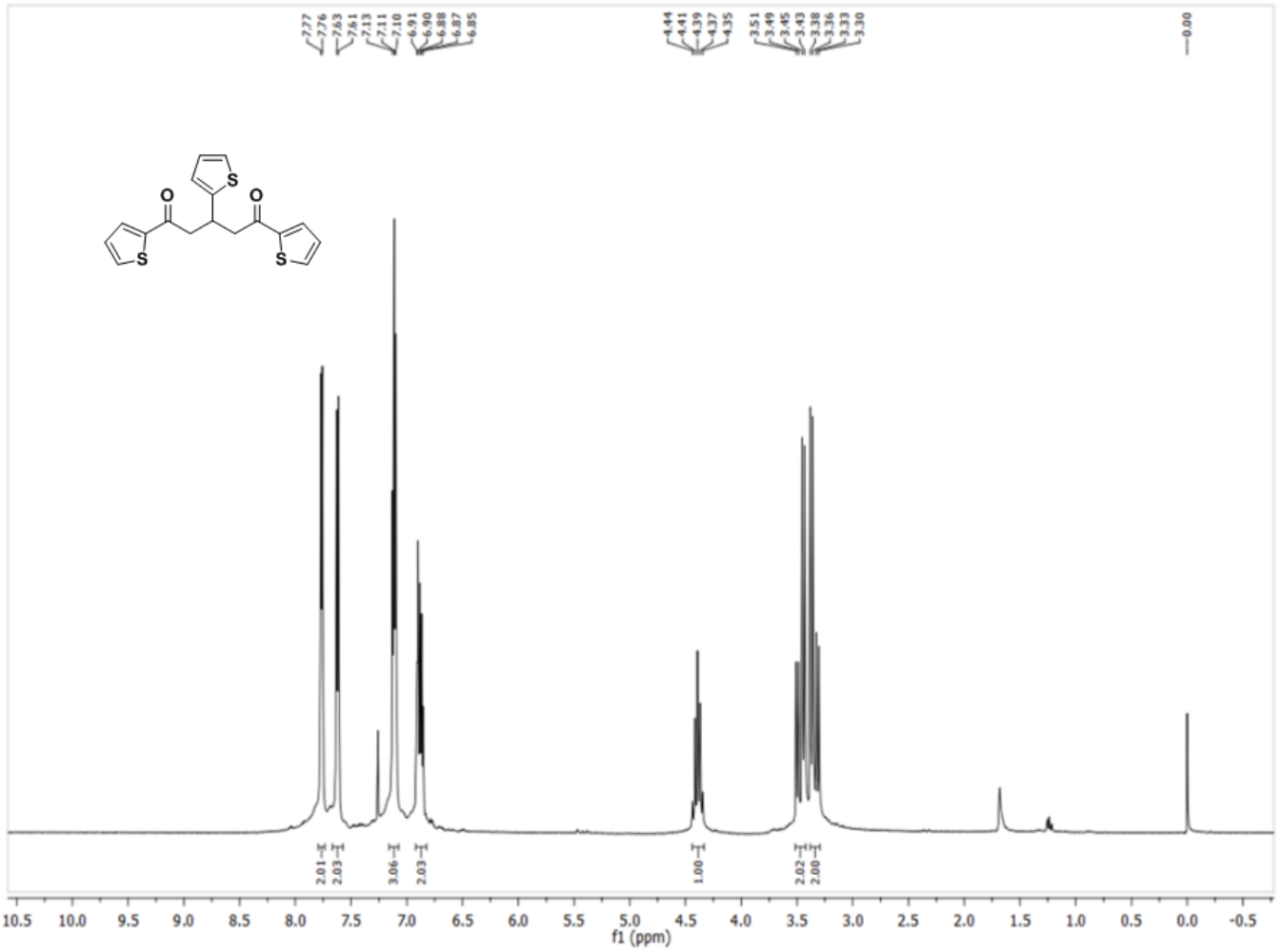

Fig. S23. ^{1}H NMR spectrum of compound 3l.

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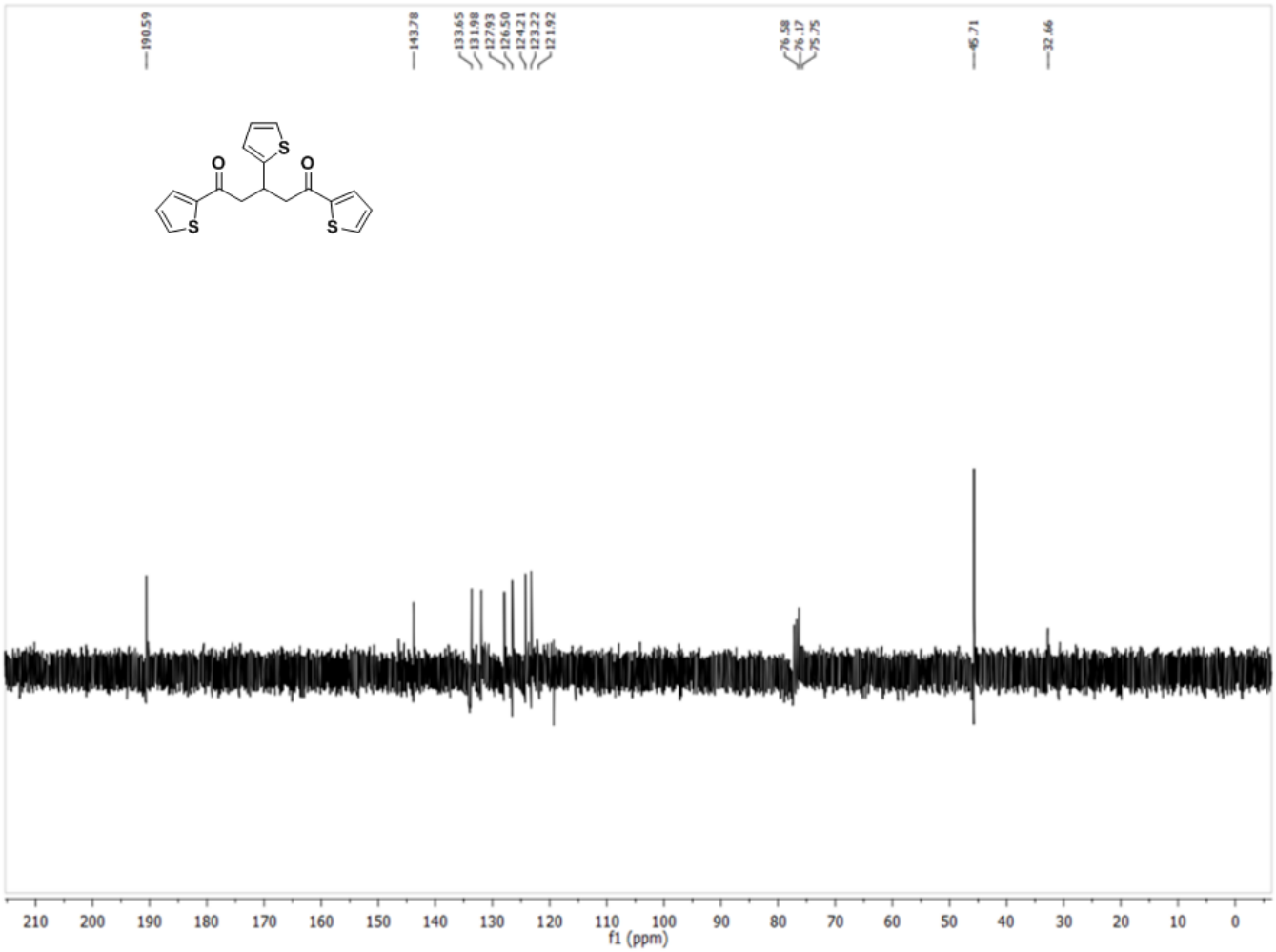

Fig. S24. ^{13}C NMR spectrum of compound 3l.

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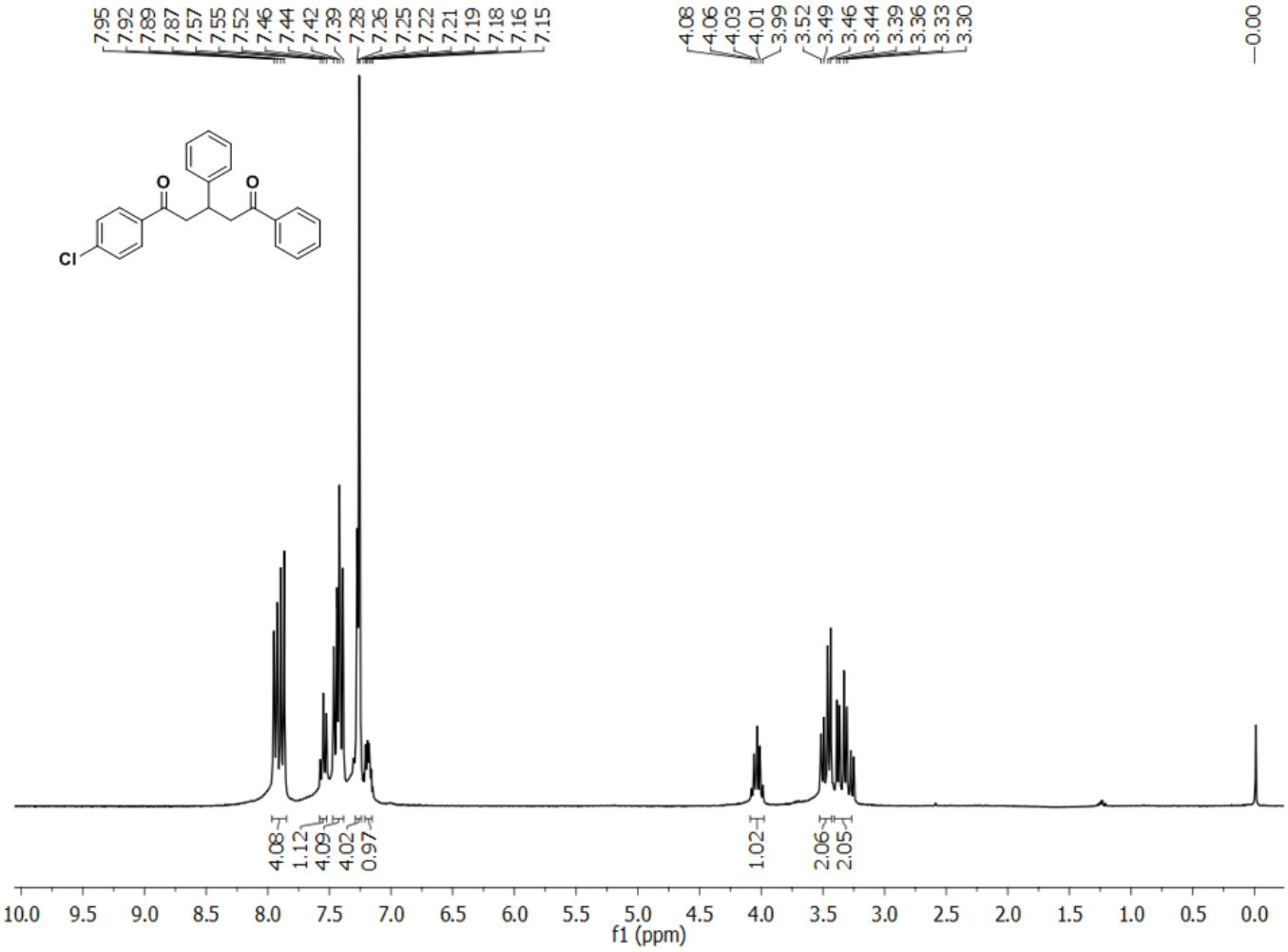

Fig. S25. ^{1}H NMR spectrum of compound 3m.

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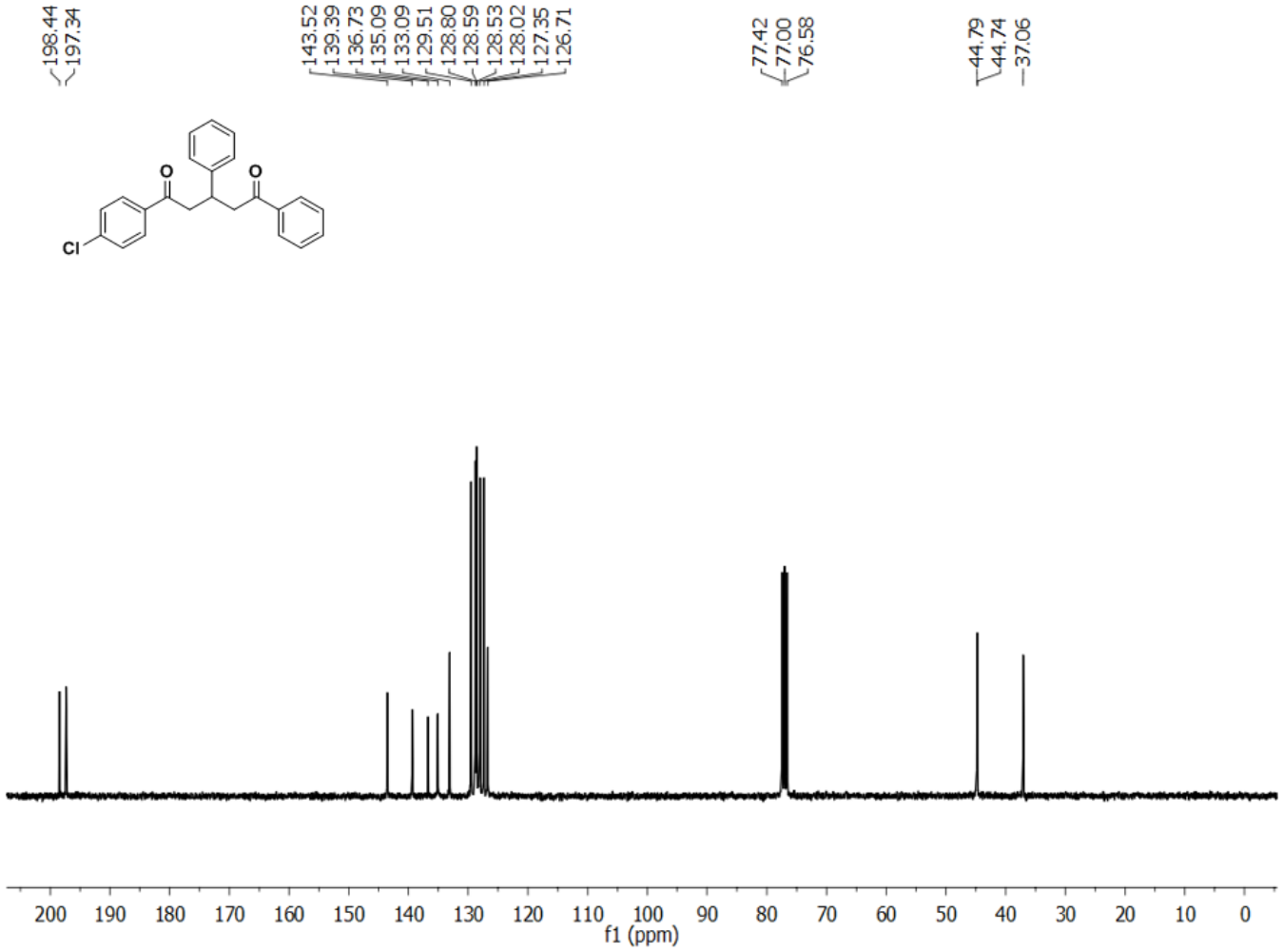

Fig. S26. ^{13}C NMR spectrum of compound 3m.

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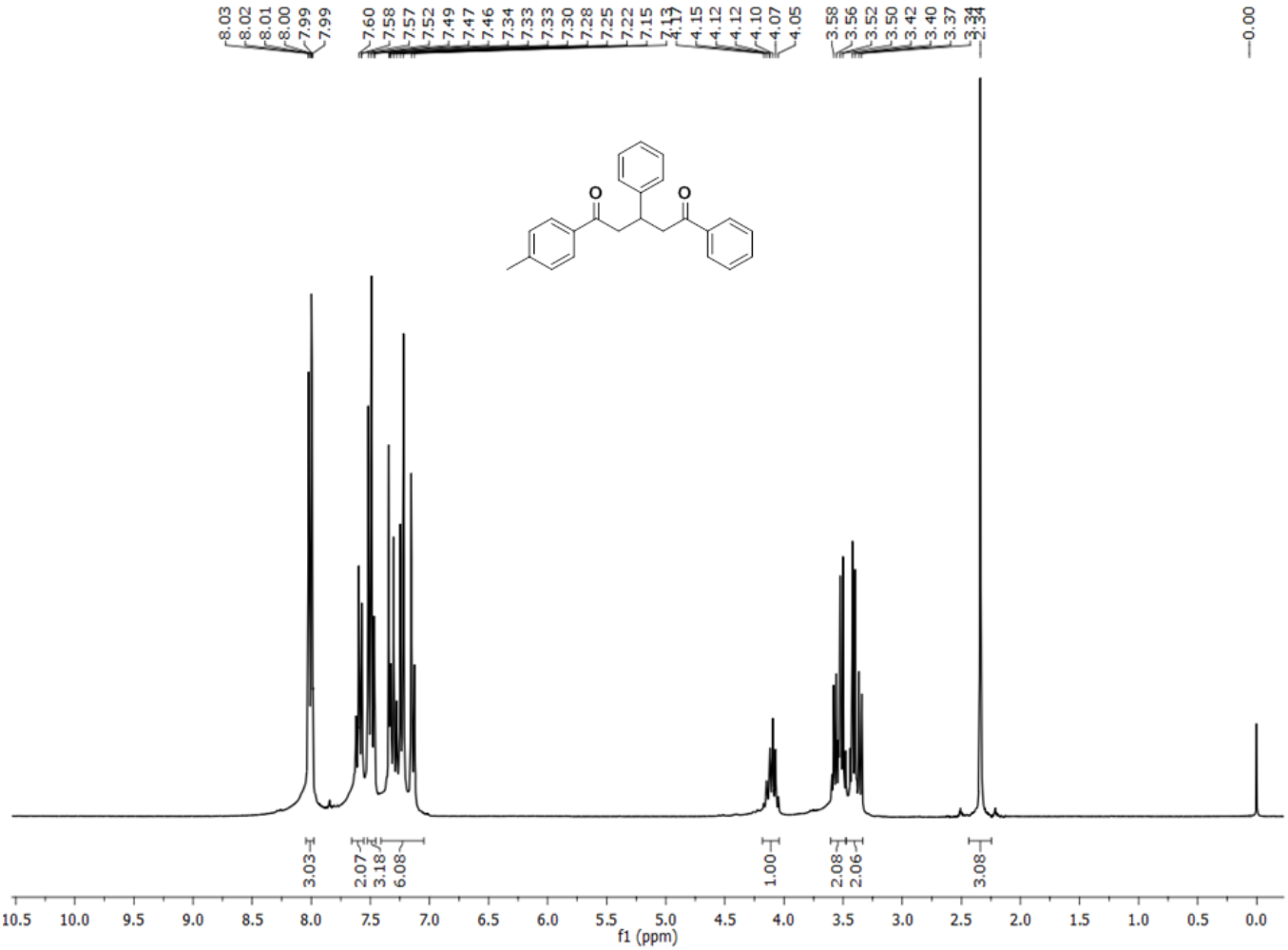

Fig. S27. ^{1}H NMR spectrum of compound 3n.

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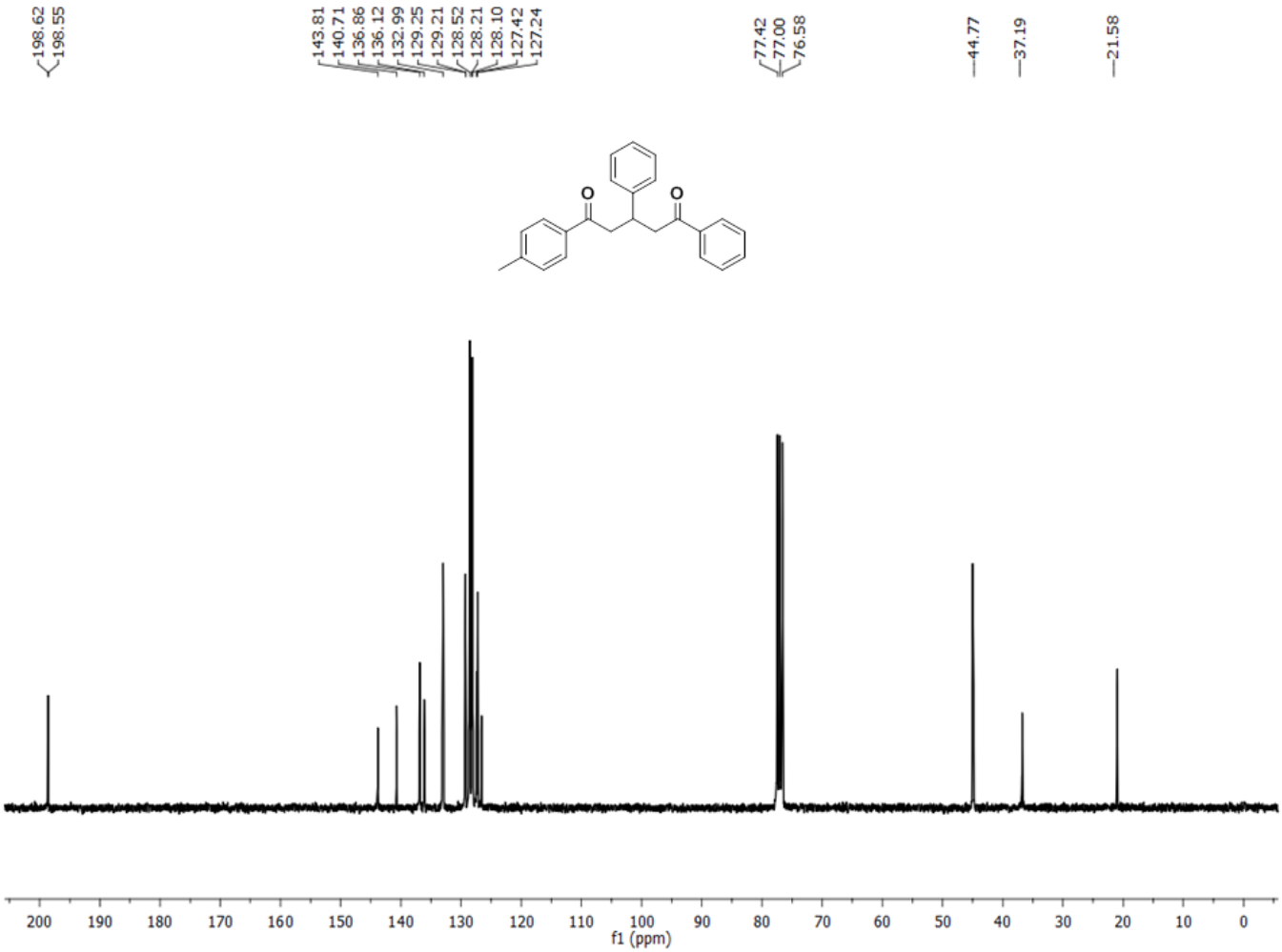

Fig. S28. ^{13}C NMR spectrum of compound 3n.

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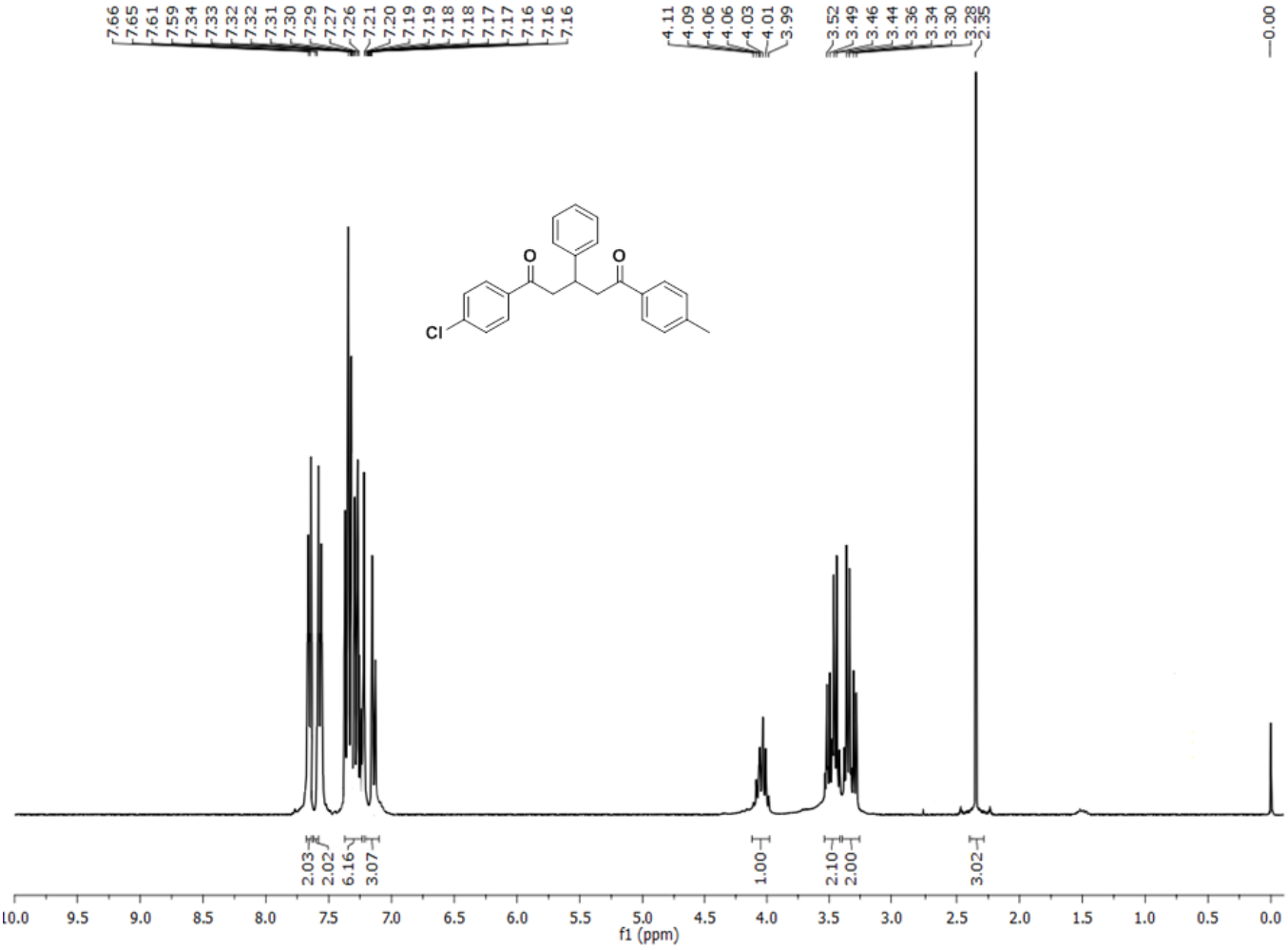

Fig. S29. ^{1}H NMR spectrum of compound 3o.

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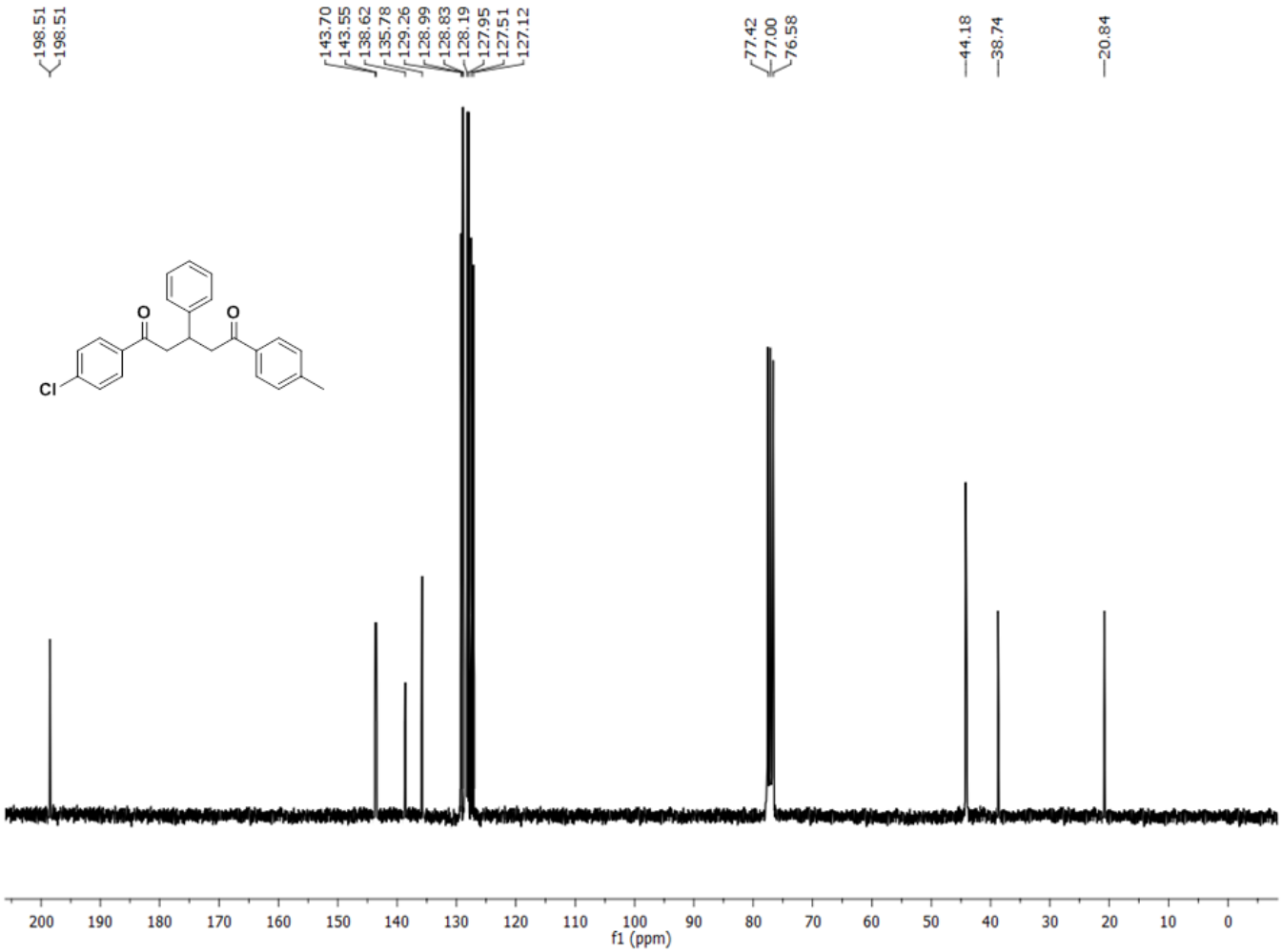

Fig. S30. ^{13}C NMR spectrum of compound 3o.

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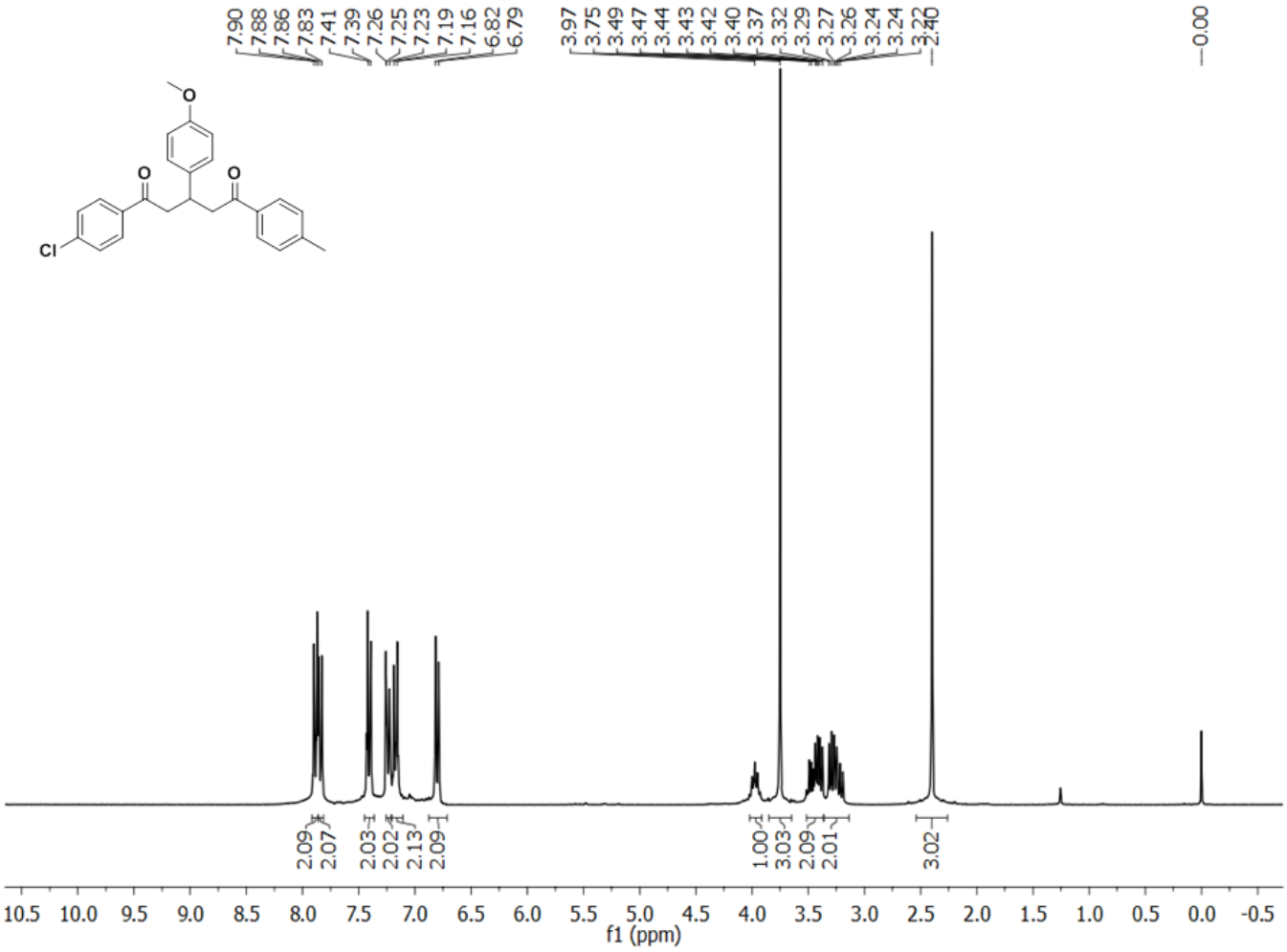

Fig. S31. ^{1}H NMR spectrum of compound 3p.

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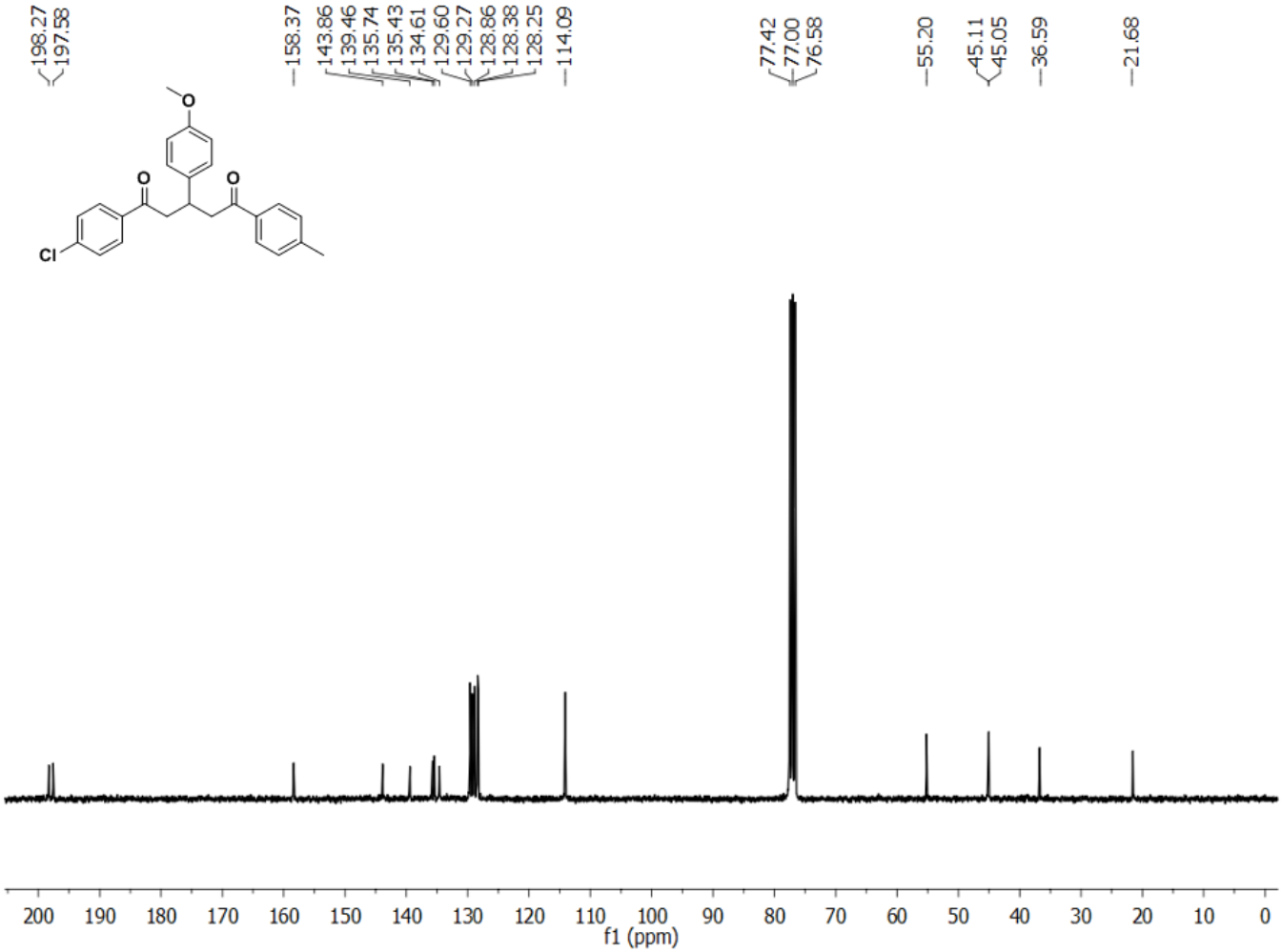

Fig. S32. ^{13}C NMR spectrum of compound 3p.

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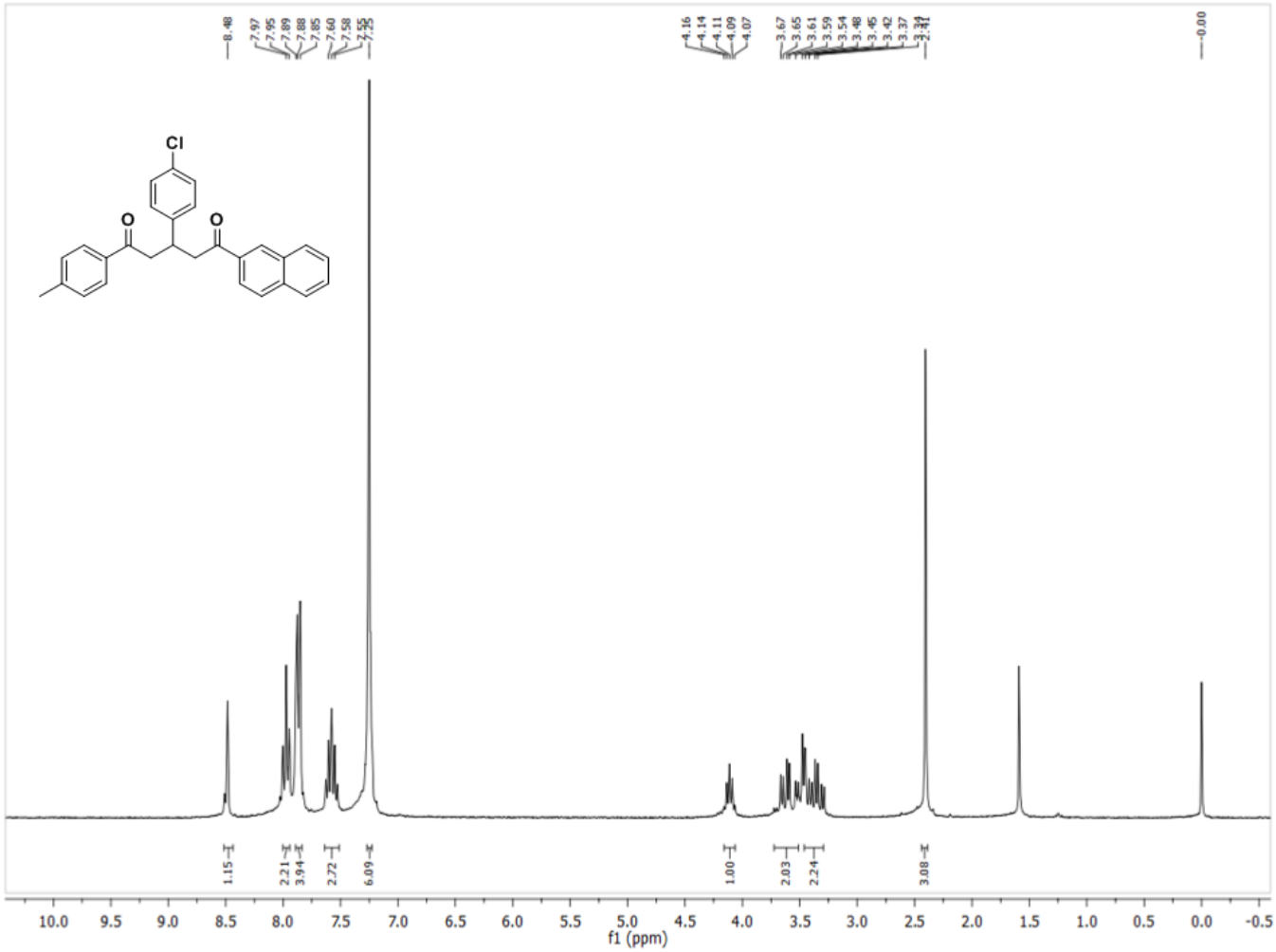

Fig. S33. ^{1}H NMR spectrum of compound 3q.

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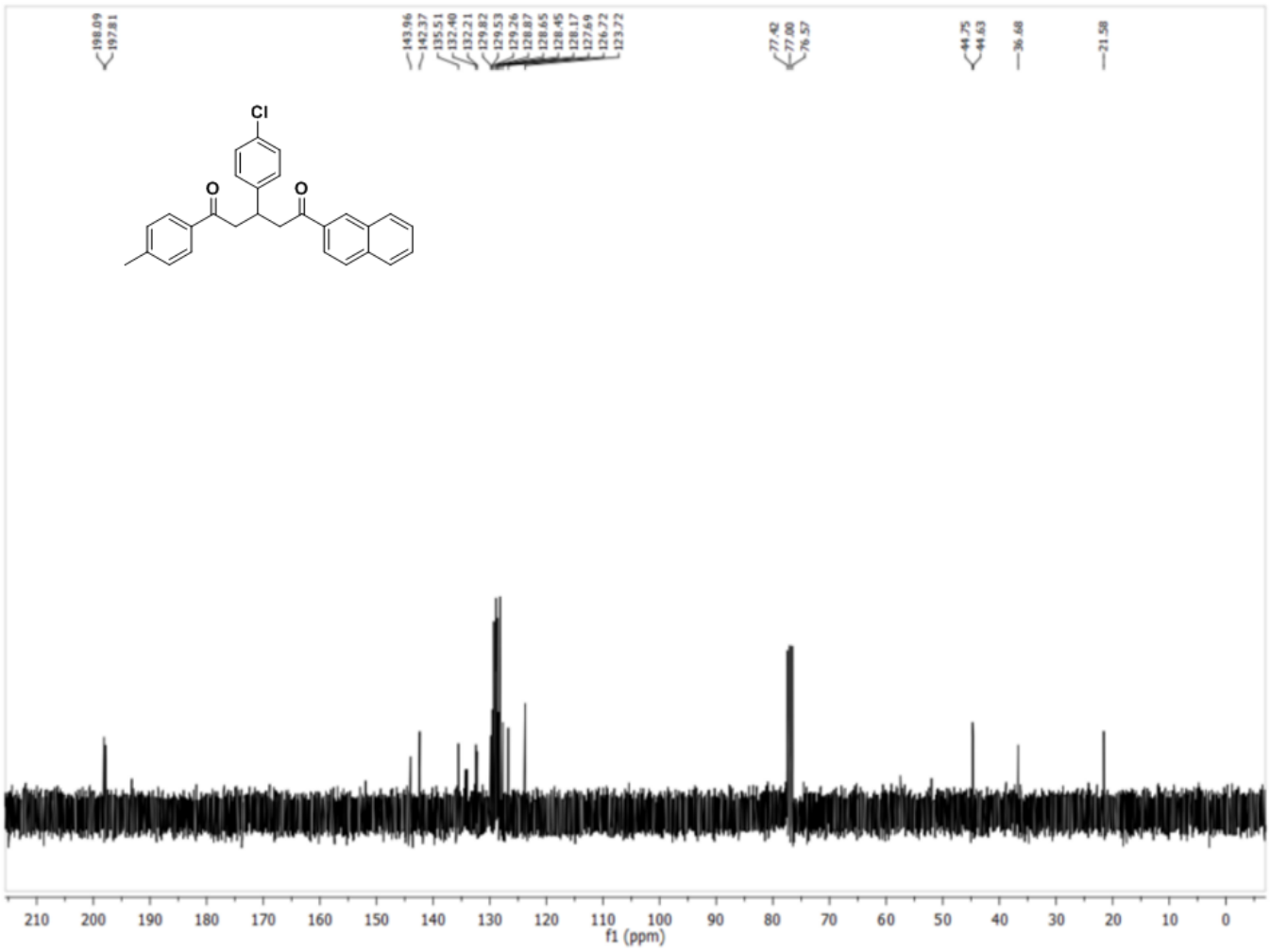

Fig. S34. ^{13}C NMR spectrum of compound 3q.

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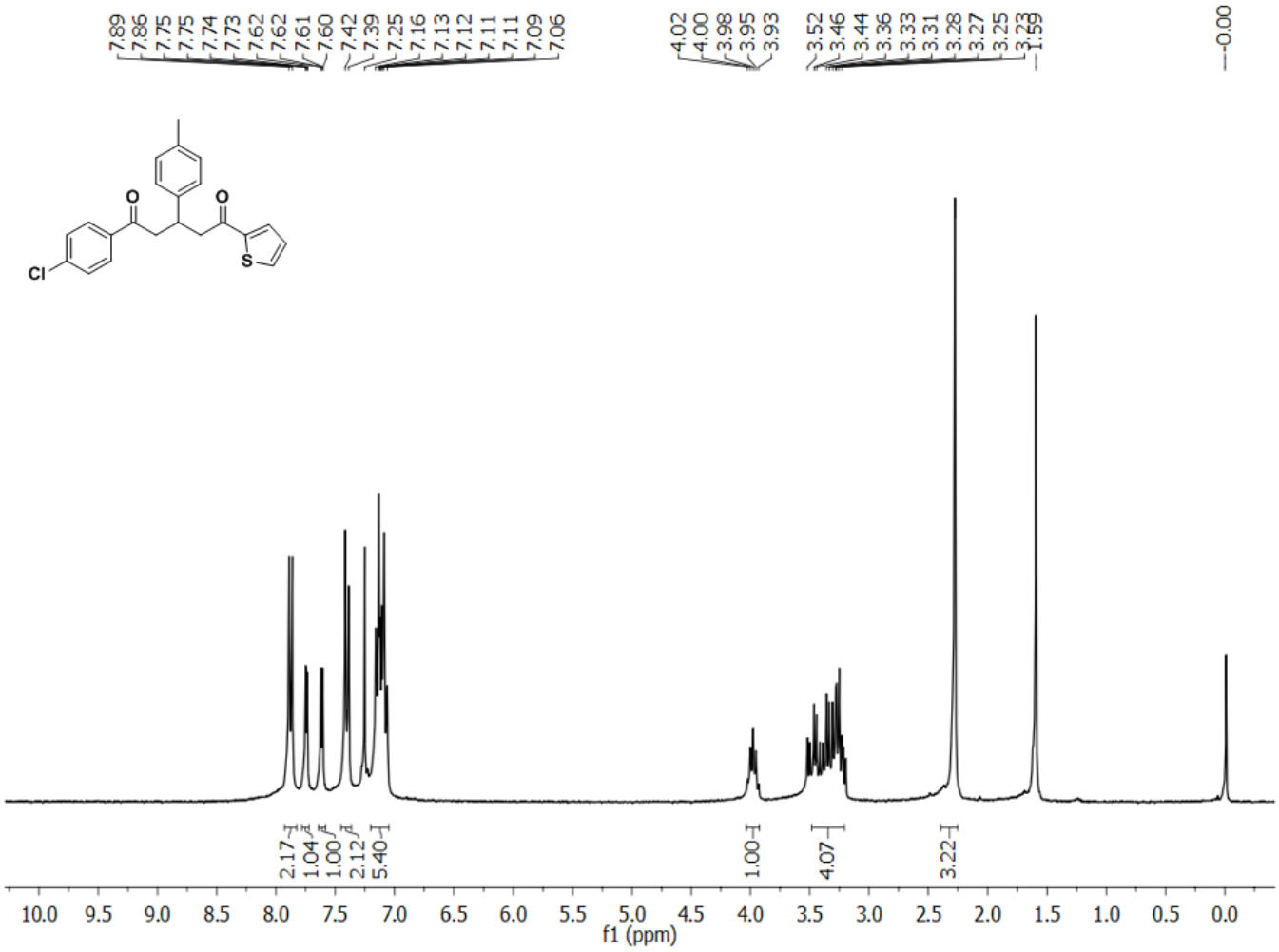

Fig. S35. ^{1}H NMR spectrum of compound 3r.

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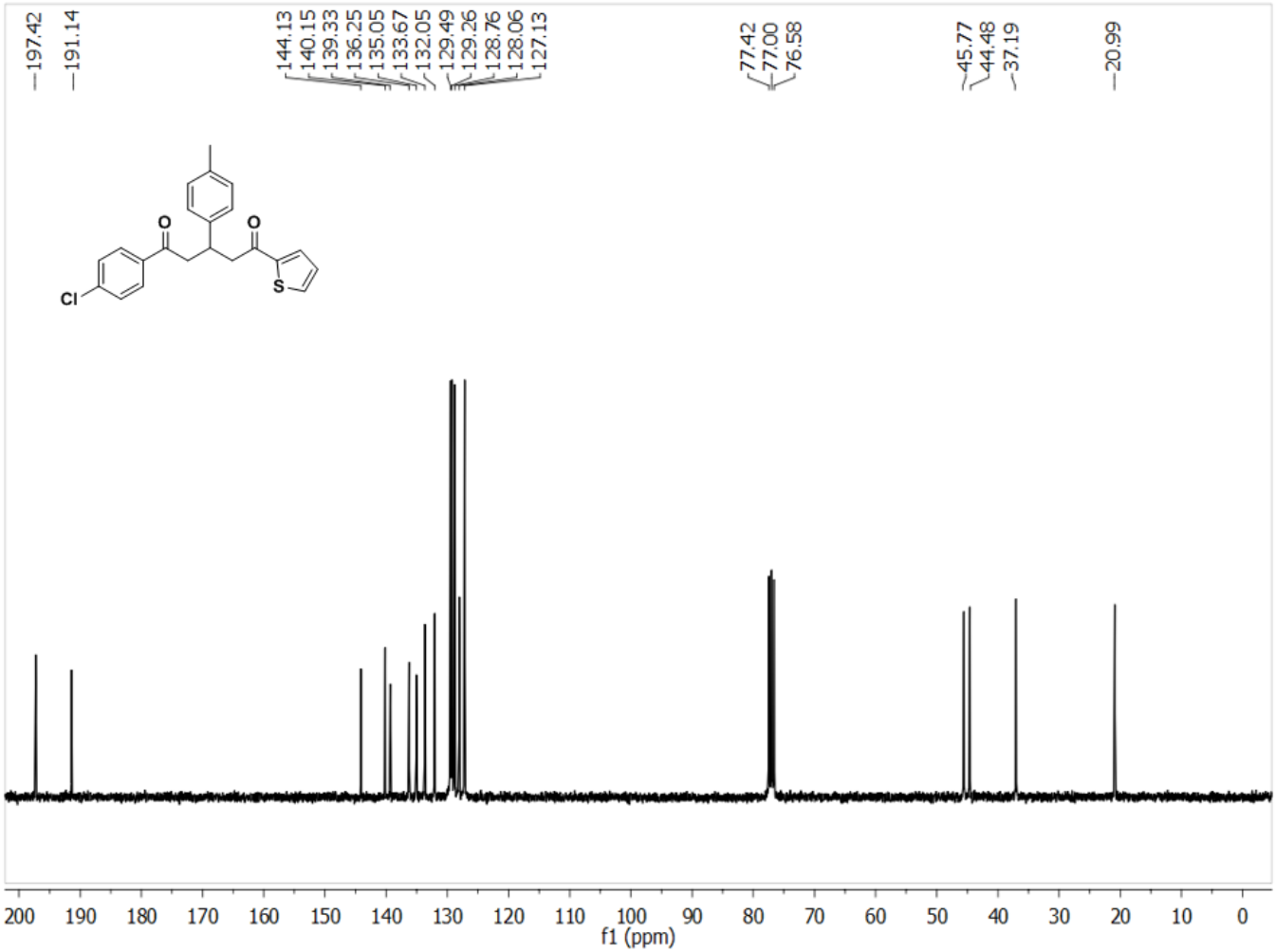

Fig. S36. ^{13}C NMR spectrum of compound 3r.

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