Supporting Information

A general two-step one-pot synthesis of ynones from α-keto acids and 1-iodoalkynes

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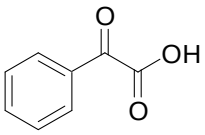
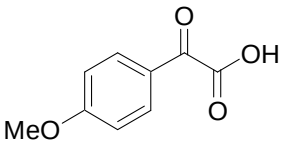
The structures of the starting material 2a-2m



2a



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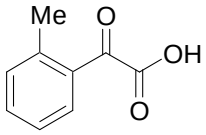

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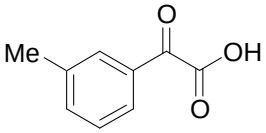

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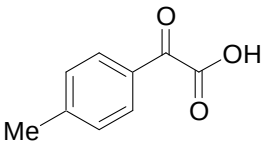

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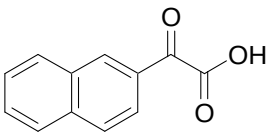

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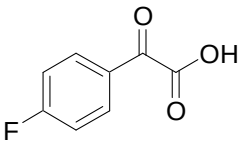

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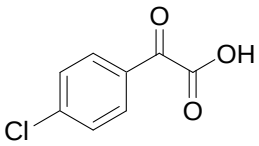

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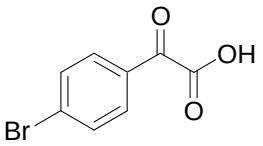
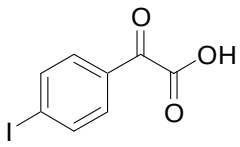


2i



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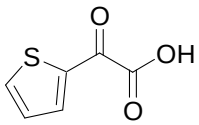
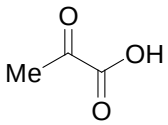
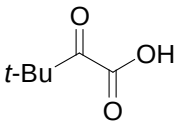


2k

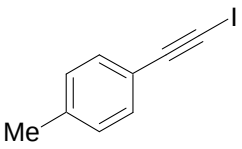


2l

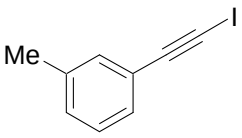


2m

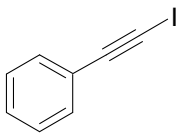
The structures of the starting material 6a-6h



6a



6b



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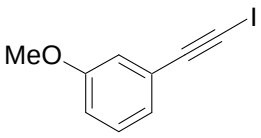

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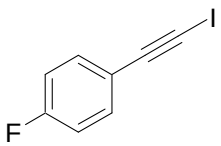

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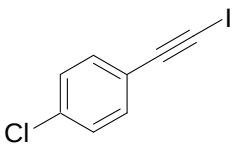

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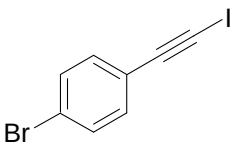
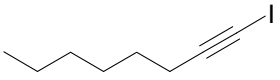


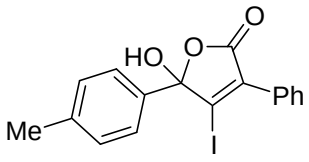
6g



6h

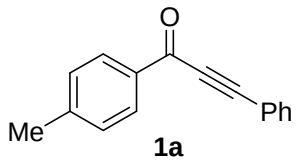
General method

All melting points were determined on a Yanaco melting point apparatus and are uncorrected. IR spectra were recorded as KBr pellets on a Nicolet FT-IR 5DX spectrometer. All ^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz) spectra were recorded in $CDCl_3$. TMS was used as an internal reference and *J* values are given in Hz. HR-MS were obtained on a Bruker micrOTOF-Q II spectrometer. PE is petroleum ether (60–90 ^{o}C). All α-keto acids[1] (**2a-2m**) and 1-iodoalkynes[2] (**6a-6h**) are known compounds. They were purchased directly or were prepared according to the reported procedures.



7a

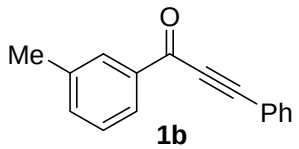
Synthesis of 3-phenyl-4-iodo-5-(4-methylphenyl)-5-hydroxy furan-2(5*H*)-one (7a). A mixture of 1-iodo-2-(4-methylphenyl)-ethyne (**6a**, 0.6 mmol, 145 mg), 2-oxo-2-phenylacetic acid (**2a**, 0.5 mmol, 75 mg) and $BF_3 \cdot Et_2O$ (0.1 mmol, 14 mg) in toluene (2 mL) was stirred at 40 °C for 1 h (monitored by TLC). After the reaction was cooled down to room temperature, it was quenched by addition of brine (15 mL) and the resultant mixture was extracted with EtOAc (3 × 15 mL). The combined organic layers were washed with brine (2 × 15 mL) and dried over $MgSO_4$. The solvent was removed by vacuum and the residue was purified by flash chromatography (silica gel, 25% EtOAc in PE) to give 186 mg (95%) of the desired product **7a** as a white solid, mp 152–154 ^{o}C. IR (KBr) *v* 3264, 1744 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70–7.63 (m, 2H), 7.46–7.38 (m, 5H), 7.25–7.20 (m, 2H), 4.64 (s, 1H), 2.37 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.5, 139.9, 136.6, 132.8, 129.8, 129.3 (2C), 129.0, 128.9 (2C), 128.4 (2C), 126.3 (2C), 125.7, 106.0, 21.3. HRMS *m/z* (ESI) calcd for $C_{17}H_{13}IO_3$, $(M+Na)^+$ 414.9802; found, 414.9803.



1a

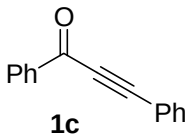
1-(4-Methylphenyl)-3-phenylprop-2-yn-1-one (1a). A mixture of 1-iodo-2-(4-methylphenyl)-ethyne (**6a**, 0.6 mmol, 145 mg), 2-oxo-2-phenylacetic acid (**2a**, 0.5 mmol, 75 mg) and $BF_3 \cdot Et_2O$ (0.1 mmol, 14 mg) in toluene (2 mL) was stirred at 40 °C for 1 h (monitored by TLC). After it was cooled down to room temperature, powder K_2CO_3 (1 mmol, 138 mg) and DMSO (1 mL) were added. The resultant mixture was stirred at 60 °C for another 20 min and was then cooled down to room temperature. The mixture was poured into water (15 mL) and was extracted with EtOAc (3 x 15 mL). The combined organic layers were washed with brine (2 x 15 mL) and dried over $MgSO_4$. The solvent was removed by vacuum and the residue was purified by a flash chromatography (silica gel, 5% EtOAc in PE) to give 102 mg (93%) of **1a** as a yellow solid, mp 64–66 ^oC (lit.[3] 68–70 ^oC). ^1H NMR (400 MHz, $CDCl_3$) δ 8.11 (d, J = 8.1 Hz, 2H), 7.68–7.66 (m, 2H), 7.50–7.44 (m, 1H), 7.44–7.38 (m, 2H), 7.31–7.29 (m, 2H), 2.43 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.6, 145.2, 134.5, 132.9 (2C), 130.6, 129.6 (2C), 129.3 (2C), 128.6 (2C), 120.1, 92.5, 86.9, 21.8.

The ynones **1b-1z** were synthesized by the similar procedure.



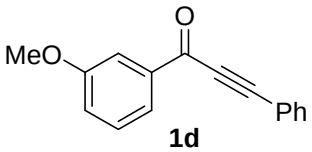
1b

1-(3-Methylphenyl)-3-phenylprop-2-yn-1-one (1b). 99 mg (90%), yellow oil.[4] ^1H NMR (400 MHz, $CDCl_3$) δ 8.06–8.00 (m, 2H), 7.69 (d, J = 7.3 Hz, 2H), 7.50–7.47 (m, 1H), 7.45–7.39 (m, 4H), 2.45 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.2, 138.5, 136.8, 135.0, 133.0 (2C), 130.7, 129.7, 128.6 (2C), 128.5, 127.1, 120.1, 92.8, 87.0, 21.3.



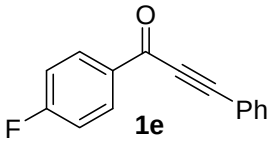
1c

A typical procedure for synthesis of 1,3-diphenylprop-2-yn-1-one (1c). 96 mg (93%), yellow solid, mp 45–47 °C (lit.[3] 46–48 °C). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.25–8.19 (m, 2H), 7.71–7.65 (m, 2H), 7.65–7.58 (m, 1H), 7.55–7.45 (m, 3H), 7.44–7.37 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.9, 136.8, 134.1, 133.0 (2C), 130.7, 129.5 (2C), 128.6 (2C), 128.5 (2C), 120.0, 93.1, 86.8.



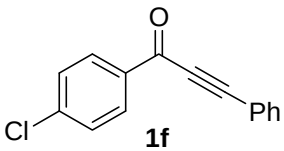
1d

1-(3-Methoxyphenyl)-3-phenylprop-2-yn-1-one (1d). 95 mg (80%), yellow solid, mp 56–58 °C (lit.[3] 59–61 °C). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88–7.84 (m, 1H), 7.70–7.67 (m, 3H), 7.51–7.47 (m, 1H), 7.45–7.40 (m, 3H), 7.20–7.16 (m, 1H), 3.88 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.7, 159.7, 138.2, 133.0 (2C), 130.8, 129.6, 128.6 (2C), 122.8, 120.9, 120.0, 112.7, 92.9, 86.9, 55.4.



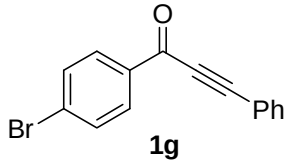
1e

1-(4-Fluorophenyl)-3-phenylprop-2-yn-1-one (1e). 98 mg (87%), yellow solid, mp 56–58 °C (lit.[4] 47–49 °C). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.29–8.21 (m, 2H), 7.72–7.65 (m, 2H), 7.51–7.47 (m, 1H), 7.45–7.41 (m, 2H), 7.22–7.16 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.3, 166.4 (d, $J_{C\text{-}F}$ = 251.7 Hz), 133.3 (d, $J_{C\text{-}F}$ = 0.95 Hz), 133.0 (2C), 132.2 (d, $J_{C\text{-}F}$ = 10.5 Hz, 2C), 130.9, 128.7 (2C), 119.9, 115.8 (d, $J_{C\text{-}F}$ = 21.9 Hz, 2C), 93.3, 86.5.



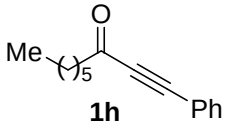
1f

1-(4-Chlorophenyl)-3-phenylprop-2-yn-1-one (1f). 111 mg (92%), yellow solid, mp 97–99 °C (lit.[3] 103–105 °C). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17–8.12 (m, 2H), 7.70–7.64 (m, 2H), 7.52–7.45 (m, 3H), 7.45–7.39 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.5, 140.6, 135.2, 133.0 (2C), 130.9, 130.8 (2C), 128.9 (2C), 128.7 (2C), 119.8, 93.6, 86.5.



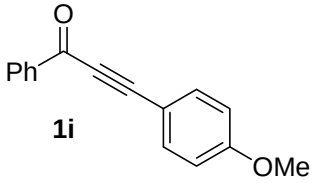
1g

1-(4-Bromophenyl)-3-phenylprop-2-yn-1-one (1g). 120 mg (84%), yellow solid, mp 106–108 °C (lit.[4] 110–111 °C). ^{1}H NMR (400 MHz, $CDCl_3$), δ 8.07 (d, J = 8.5, 2H), 7.69–7.64 (m, 4H), 7.53–7.47 (m, 1H), 7.44–7.41 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.8, 135.6, 133.1 (2C), 131.9 (2C), 131.0, 130.9 (2C), 129.5, 128.7 (2C), 119.8, 93.6, 86.5.



1h

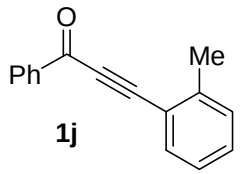
1-Phenylnon-1-yn-3-one (1h). 46 mg (43%), coroless oil.[5] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58–7.56 (m, 2H), 7.49–7.42 (m, 1H), 7.41–7.35 (m, 2H), 2.66 (t, J = 7.4 Hz, 2H), 1.78–1.70 (m, 2H), 1.39–1.27 (m, 6H), 0.95–0.85 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 188.3, 133.0 (2C), 130.6, 128.6 (2C), 120.0, 90.5, 87.8, 45.5, 31.5, 28.6, 24.1, 22.4, 14.0.



1i

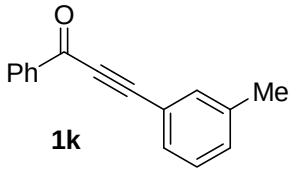
1-Phenyl-3-(4-methoxyphenyl)prop-2-yn-1-one (1i). 100 mg (85%), yellow solid, mp 79–80 °C (lit.[3] 80–81 °C). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.22 (d, J = 7.6 Hz, 2H), 7.67–7.58 (m, 3H), 7.52–7.49 (m, 2H), 6.92 (d, J = 8.7, 2H), 3.84 (s, 3H);

^{13}C NMR (100 MHz, $CDCl_3$) δ 177.9, 161.7, 136.9, 135.1 (2C), 133.8, 129.4 (2C), 128.5 (2C), 114.3 (2C), 111.8, 94.3, 86.8, 55.3.



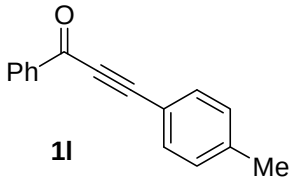
1j

1-Phenyl-3-(2-Methylphenyl)prop-2-yn-1-one (1j). 96 mg (87%), yellow oil.[3] ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.25–8.23 (m, 2H), 7.66–7.61 (m, 2H), 7.54–7.50 (m, 2H), 7.39–7.35 (m, 1H), 7.29–7.21 (m, 2H), 2.59 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.0, 142.1, 137.0, 134.0, 133.6, 130.8, 129.8, 129.5 (2C), 128.6 (2C), 125.9, 119.9, 92.1, 90.7, 20.8.



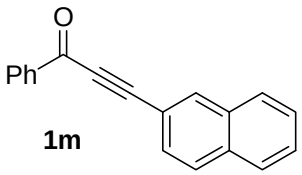
1k

1-Phenyl-3-(3-Methylphenyl)prop-2-yn-1-one (1k). 99 mg (90%), yellow oil.[6] ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.24–8.21 (m, 2H), 7.64–7.60 (m, 1H), 7.53–7.48 (m, 4H), 7.32–7.29 (m, 2H), 2.38 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.0, 138.5, 136.9, 134.0, 133.5, 131.7, 130.2, 129.5 (2C), 128.6 (3C), 119.8, 93.5, 86.6, 21.1.



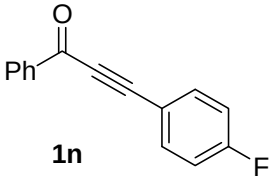
1l

1-Phenyl-3-(4-methylphenyl)prop-2-yn-1-one (1l). 105 mg (95%), yellow solid, mp 68–70 ^{o}C (lit.[3] 69–71 ^{o}C). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.22 (m, 2H), 7.64–7.60 (m, 1H), 7.60–7.57 (m, 2H), 7.52–7.49 (m, 2H), 7.23–7.21 (m, 2H), 2.39 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.0, 141.5, 136.9, 134.0, 133.1 (2C), 129.5 (2C), 129.4 (2C), 128.5 (2C), 116.9, 93.8, 86.7, 21.7.



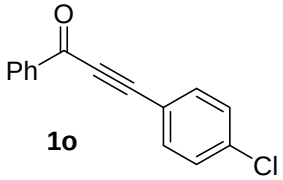
1m

1-Phenyl-3-(naphthalen-2-yl)prop-2-yn-1-one (1m). 96 mg (75%), white solid, mp 86–88 °C (lit.[7] 81–83 °C). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.29–8.20 (m, 3H), 7.88–7.81 (m, 3H), 7.68–7.60 (m, 2H), 7.59–7.49 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.9, 136.8, 134.3, 134.1, 133.8, 132.6, 129.5 (2C), 128.6 (2C), 128.4, 128.3, 128.1, 127.9, 127.8, 127.0, 117.2, 93.6, 87.1.



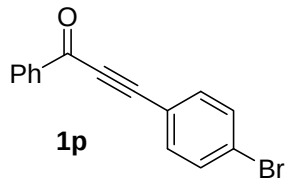
1n

1-Phenyl-3-(4-fluorophenyl)-prop-2-yn-1-one (1n). 100 mg (89%), white solid, mp 70–72 °C (lit.[8] 73–75 °C). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.22–8.20 (m, 2H), 7.71–7.62 (m, 3H), 7.54–7.50 (m, 2H), 7.15–7.09 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.8, 163.9 (d, $J_{C\text{-}F}$ = 254.7 Hz), 136.7, 135.3 (d, $J_{C\text{-}F}$ = 9.0 Hz, 2C), 134.2, 129.5 (2C), 128.6 (2C), 116.3 (d, $J_{C\text{-}F}$ = 22.4 Hz, 2C), 116.2 (d, $J_{C\text{-}F}$ = 3.5 Hz), 91.9, 86.8.



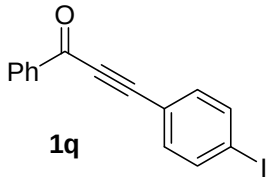
1o

1-Phenyl-3-(4-chlorophenyl)prop-2-yn-1-one (1o). 95 mg (80%), pale yellow solid, mp 104–106 °C (lit.[6] 106–107 °C). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.21–8.19 (m, 2H), 7.66–7.60 (m, 3H), 7.54–7.50 (m, 2H), 7.42–7.39 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.8, 137.2, 137.0, 134.2 (3C), 129.5 (2C), 129.1 (2C), 128.6 (2C), 118.6, 91.6, 87.5.



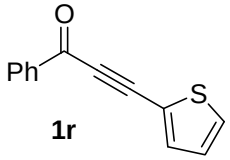
1p

1-Phenyl-3-(4-bromophenyl)prop-2-yn-1-one (1p). 135 mg (95%), white solid, mp 110–112 °C (lit.[3] 115–116 °C). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.23–8.18 (m, 2H), 7.67–7.61 (m, 1H), 7.60–7.49 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.8, 136.7, 134.3 (2C), 134.2, 132.1 (2C), 129.6 (2C), 128.7 (2C), 125.6, 119.0, 91.6, 87.7.



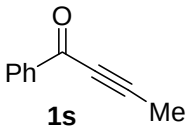
1q

1-Phenyl-3-(4-iodophenyl)prop-2-yn-1-one (1q). 128 mg (77%), yellow solid, mp 56–58 °C (lit.[9] 118–120 °C). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.20–8.18 (m, 2H), 7.77–7.75 (m, 2H), 7.65–7.61 (m, 1H), 7.53–7.49 (m, 2H), 7.39–7.37 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.7, 137.9 (2C), 136.6, 134.2, 134.1 (2C), 129.5 (2C), 128.6 (2C), 119.4, 97.7, 91.7, 87.8.



1r

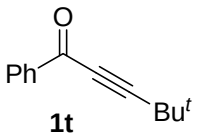
1-Phenyl-3-(thiophen-2-yl)prop-2-yn-1-one (1r). 88 mg (83%), yellow oil.[10] ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.22–8.16 (m, 2H), 7.65–7.62 (m, 1H), 7.60–7.59 (m, 1H), 7.54–7.51 (m, 3H), 7.12–7.10 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.5, 136.7, 136.6, 134.1, 131.7, 129.4 (2C), 128.6 (2C), 127.8, 119.8, 91.6, 87.0.



1s

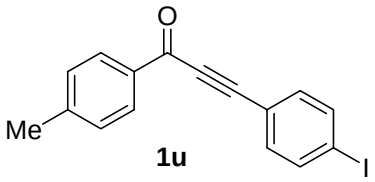
1-Phenylbut-2-yn-1-one (1s). 47 mg (65%), colorless oil.[11] ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15 (d, J = 7.8 Hz, 2H), 7.60 (t, J = 7.3 Hz, 1H), 7.48 (t, J = 7.7 Hz, 2H),

2.17 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.3, 136.8, 133.9, 129.6 (2C), 128.5 (2C), 92.5, 78.9, 4.3.



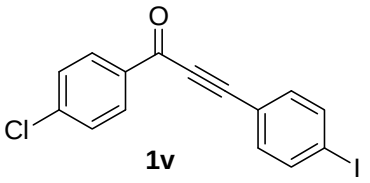
1t

1-Phenyl-4,4-dimethylpent-2-yn-1-one (1t). 68 mg (73%), coroless oil.[12] ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (d, J = 7.7 Hz, 2H), 7.58 (t, J = 7.4 Hz, 1H), 7.46 (t, J = 7.6 Hz, 2H), 1.38 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.1, 136.8, 133.7, 129.3 (2C), 128.3 (2C), 103.7, 77.9, 30.0 (3C), 27.8.



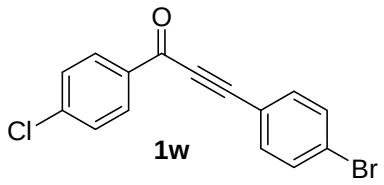
1u

1-(4-Methylphenyl)-3-(4-iodophenyl)-prop-2-yn-1-one (1u). 130 mg (75%), yellow solid, mp 120–122 ^{o}C. IR (KBr) v 2214, 1636 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10–8.04 (m, 2H), 7.78–7.72 (m, 2H), 7.38–7.33 (m, 2H), 7.28 (m, 2H), 2.43 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.4, 145.3, 137.9 (2C), 134.3, 134.1 (2C), 129.6 (2C), 129.3 (2C), 119.6, 97.5, 91.2, 87.9, 21.8. HRMS *m/z* (ESI) calcd for $C_{16}H_{12}IO^+$, $(M+H)^+$ 346.9927; found 346.9925.



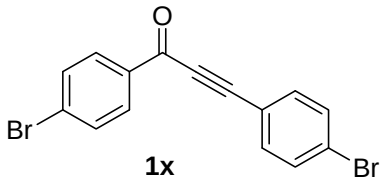
1v

1-(4-Chlorophenyl)-3-(4-iodophenyl)prop-2-yn-1-one (1v). 84 mg (46%), white solid, mp 183–185 ^{o}C. IR (KBr) v 2219, 1643 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.16–8.10 (m, 2H), 7.82–7.76 (m, 2H), 7.52–7.47 (m, 2H), 7.42–7.36 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.4, 140.9, 138.0 (2C), 135.1, 134.2 (2C), 130.8 (2C), 129.1 (2C), 119.3, 97.9, 92.3, 87.5. HRMS *m/z* (ESI) calcd for $C_{15}H_9ClIO^+$, $(M+H)^+$ 366.9381; found 366.9383.



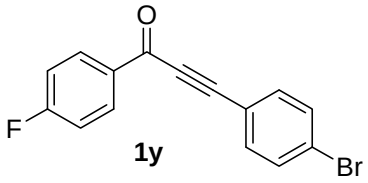
1w

1-(4-Chlorophenyl)-3-(4-bromophenyl)-prop-2-yn-1-one (1w). 116 mg (73%), white solid, mp 175–177 °C. IR (KBr) ν 2200, 1626 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08–8.02 (m, 2H), 7.69–7.64 (m, 2H), 7.64–7.58 (m, 2H), 7.45–7.39 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.6, 137.4, 135.5, 134.3 (2C), 132.0 (2C), 130.9 (2C), 129.7, 129.2 (2C), 118.3, 92.2, 87.2. HRMS *m/z* (ESI) calcd for $C_{15}H_9BrClO^+$, $(M+H)^+$ 318.9520; found, 318.9521.



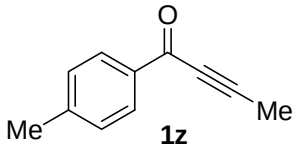
1x

1,3-Bis(4-bromophenyl)prop-2-yn-1-one (1x). 138 mg (76%), yellow solid, mp 187–189 °C (lit.[13] 187–188 °C). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08–8.01 (m, 2H), 7.70–7.63 (m, 2H), 7.60–7.50 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.6, 135.5, 134.3 (2C), 132.1 (2C), 132.0 (2C), 130.9 (2C), 129.7, 125.8, 118.7, 92.2, 87.3.



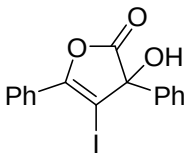
1y

1-(4-Fluorophenyl)-3-(4-bromophenyl)prop-2-yn-1-one (1y). 118 mg (78%), white solid, mp 138–140 °C. IR (KBr) ν 2199, 1624 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09–8.02 (m, 2H), 7.72–7.64 (m, 4H), 7.17–7.09 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.7, 164.1 (d, $J_{C\text{-}F}$ = 253 Hz), 135.6, 135.4 (d, $J_{C\text{-}F}$ = 9 Hz, 2C), 132.0 (2C), 130.9 (2C), 129.6, 116.3 (d, $J_{C\text{-}F}$ = 23 Hz, 2C), 116.0 (d, $J_{C\text{-}F}$ = 4 Hz), 92.5, 86.5. HRMS *m/z* (ESI) calcd for $C_{15}H_9BrFO^+$, $(M+H)^+$ 302.9815; found, 302.9817.



1z

1-(4-Methylphenyl)but-2-yn-1-one (1z). 52 mg (66%), yellow oil. IR (KBr) ν 2243, 1643 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.03 (d, J = 8.2 Hz, 2H), 7.26 (d, J = 8.0 Hz, 2H), 2.42 (s, 3H), 2.14 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.9, 144.9, 134.5, 129.7 (2C), 129.2 (2C), 91.9, 79.0, 21.8, 4.3. HRMS *m/z* (ESI) calcd for $C_{11}H_{11}O^+$, $(M+H)^+$ 159.0804; found 159.0802.



8c

Preparation and isolation of 3-hydroxy-3,5-diphenyl-4-iodo-furan-2(3*H*)-one (8c). A mixture of 1-iodo-2-phenylethyne (**6c**, 0.6 mmol, 137 mg), 2-oxo-2-phenylacetic acid (**2a**, 0.5 mmol, 75 mg) and $BF_3{\cdot}Et_2O$ (0.1 mmol, 14 mg) in toluene (2 mL) was stirred at 25 °C for 1 h (monitored by TLC). After the reaction was quenched by addition of brine (15 mL), the resultant mixture was extracted with EtOAc (3 × 15 mL). The combined organic layers were washed with brine (2 × 15 mL) and dried over $MgSO_4$. The solvent was removed by vacuum and the residue was purified by flash chromatography (silica gel, 10% EtOAc in PE) to give 40 mg (21%) of the desired product **8c** as a white solid, mp 107–109 °C (lit.[14] 109–110 °C); ^1H NMR (400 MHz, $CDCl_3$) δ 8.08 (dd, J = 7.8, 1.7 Hz, 2H), 7.53–7.45 (m, 5H), 7.45–7.38 (m, 3H), 3.23 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.5, 151.8, 137.0, 131.2, 129.3, 128.9 (2C), 128.5 (2C), 127.8 (2C), 127.5, 125.4 (2C), 81.5, 73.5.

References

[1] Xu, W.; Chen, Q.-Y. *J. Org. Chem.* **2002**, *67*, 9421–9427.

[2] Kuldeep, W.; Yang, C.; West, P. R.; Deming, K. C.; Chemburkar, S. R.; Reddy, R. E. *Synth. Commun.* **2008**, *38*, 4434–4444.

[3] Liu, J.; Peng, X.; Sun, W.; Zhao, Y.; Xia, C. *Org. Lett.* **2008**, *10*, 3933–3936.

[4] Tan, H.; Li, H.; Ji, W.; Wang, L. *Angew. Chem. Int. Ed.* **2015**, *54*, 8374–8377.

[5] Liu, X.; Yu, L.; Luo, M.; Zhu, J.; Wei, W. *Chem. Eur. J.* **2015**, *21*, 8745-8749.

[6] Gandeepan, P.; Parthasarathy, K.; Su, T.-H.; Cheng, C.-H. *Adv. Synth. Catal.* **2012**, *354*, 457–468.

[7] Chen, J.-Y.; Lin, T.-.C.; Chen, S.-C.; Chen, A.-J.; Mou, C.-Y.; Tsai, F.-Y. *Tetrahedron* **2009**, *65*, 10134–10141.

[8] Geden, J. V.; Pancholi, A. K.; Shipman, M. *J. Org. Chem.* **2013**, *78*, 4158−4164.

[9] Dermenci, A.; Whittaker, R. E.; Gao, Y.; Cruz, F. A.; Yu, Z.-X.; Dong, G. *Chem. Sci.* **2015**, *6*, 3201−3210.

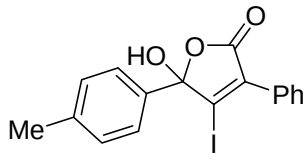
[10] Negishi, E.-I.; Qian, M.; Zeng, F.; Anastasia, L.; Babinski, D. *Org. Lett.* **2003**, *5*, 1597–1600.

[11] Suffert, J.; Toussaint, D. *J. Org. Chem.* **1995**, *60*, 3550–3553.

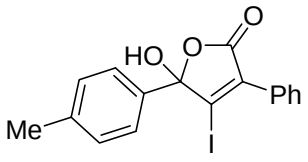
[12] Ahmed, M. S. M.; Mori, A. *Org. Lett.* **2003**, *5*, 3057–3060.

[13] Kim, W.; Park, K.; Park, A.; Choe, J.; Lee, S. *Org. Lett.* **2013**, *15*, 1654–1657.

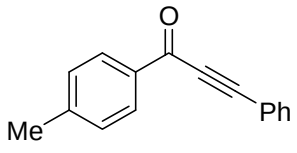
[14] Just, Z. W.; Larock, R. C. *J. Org. Chem.* **2008**, *73*, 2662–2667.



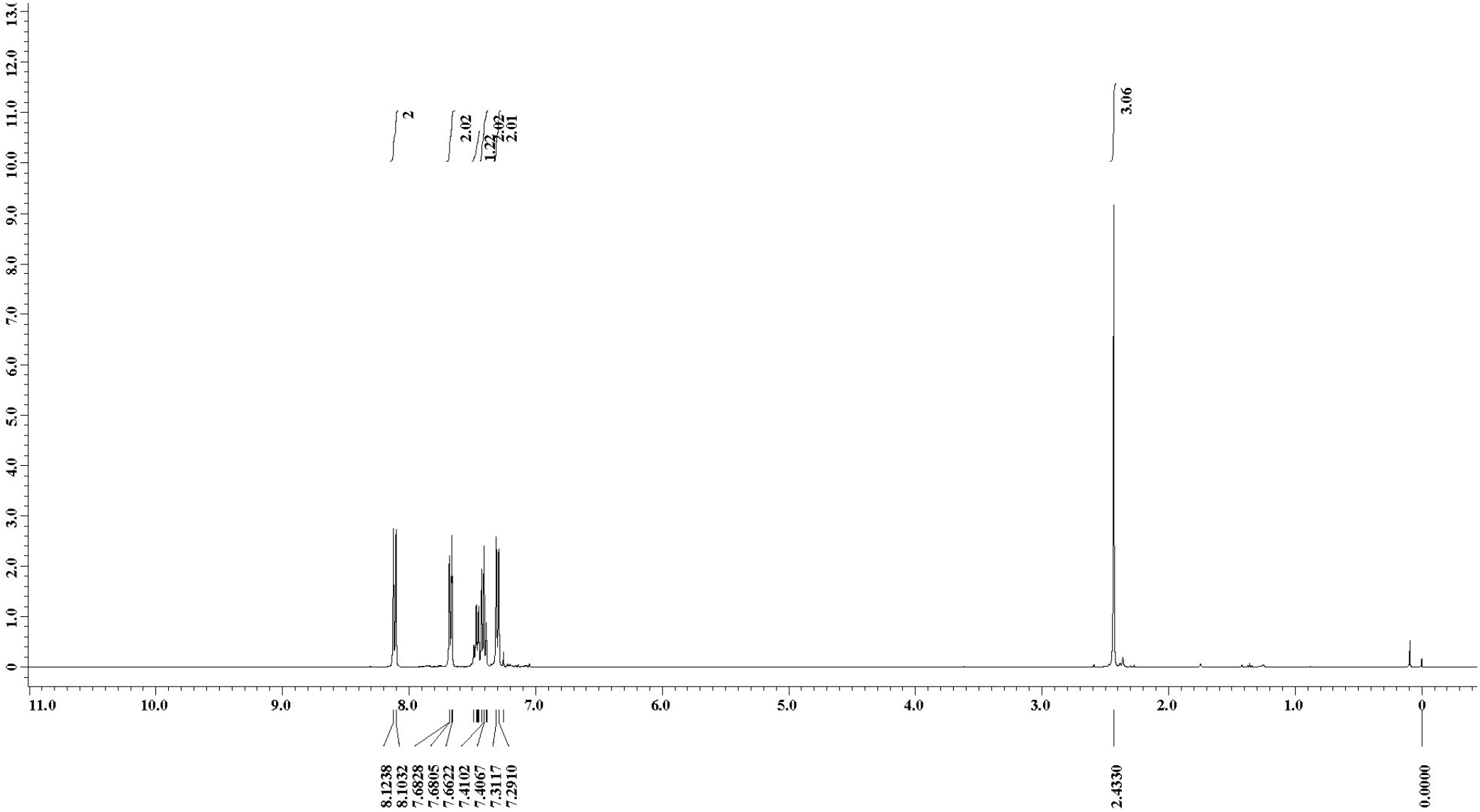
7a, ^{1}H NMR (400 MHz, $CDCl_3$)

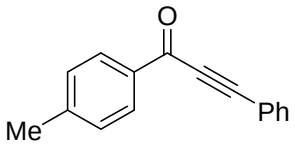


7a, ^{13}C NMR (100 MHz, $CDCl_3$)

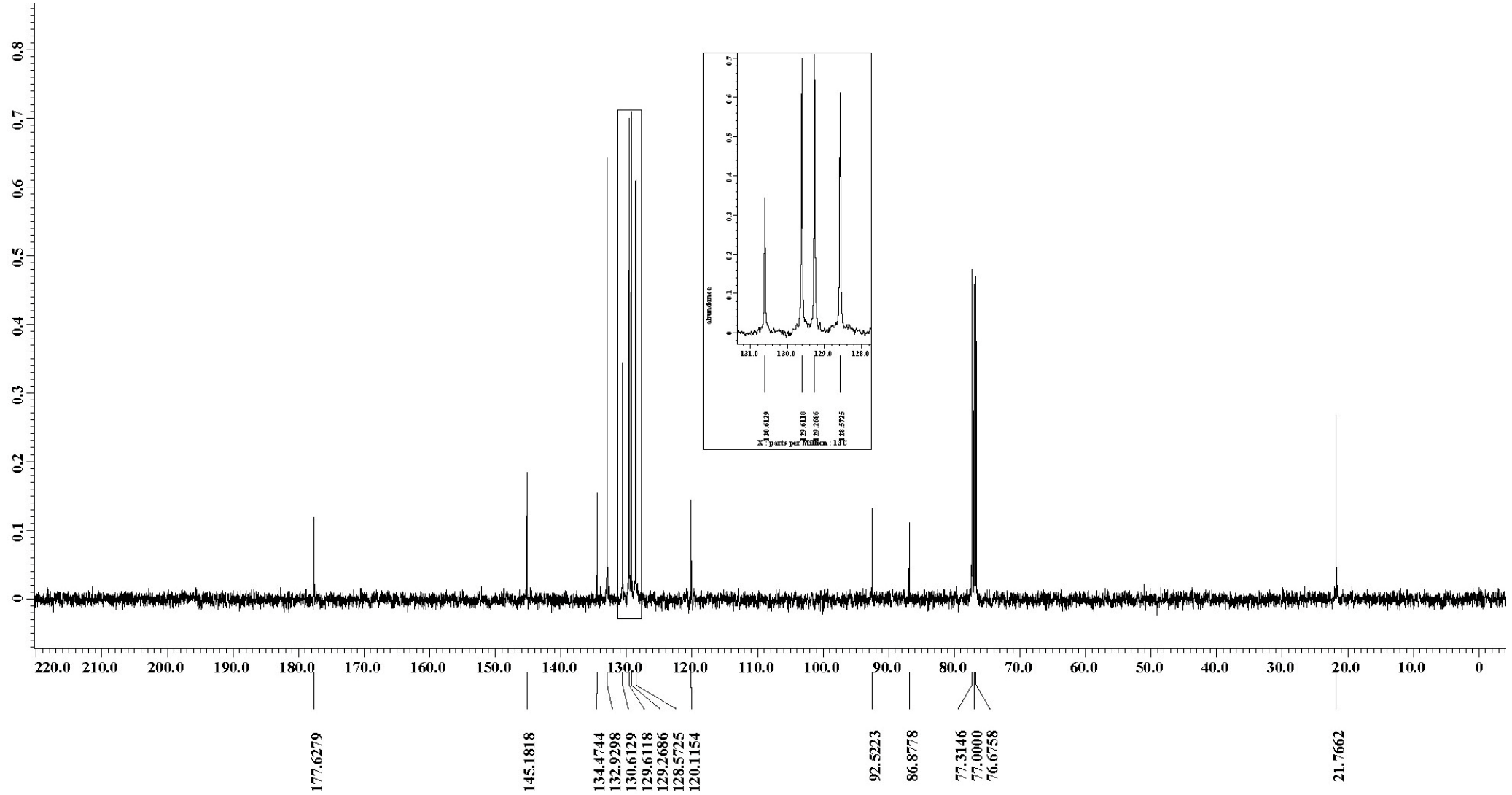


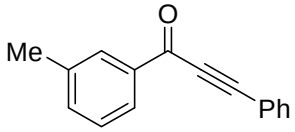
1a, ^{1}H NMR (400 MHz, $CDCl_3$)



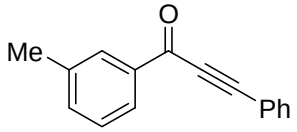


1a, ^{13}C NMR (100 MHz, $CDCl_3$)

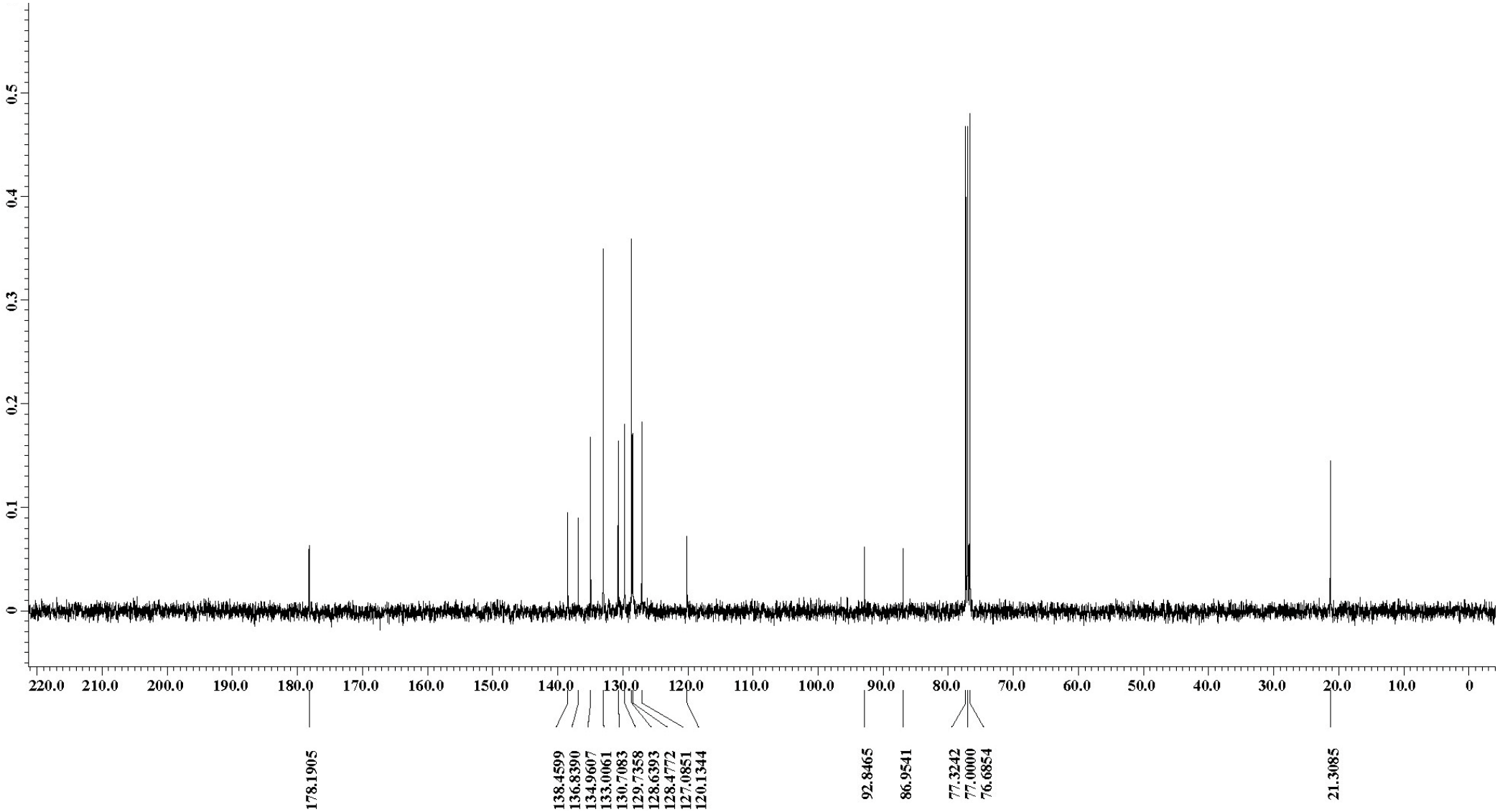


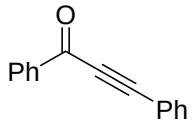


1b, ^{1}H NMR (400 MHz, $CDCl_3$)

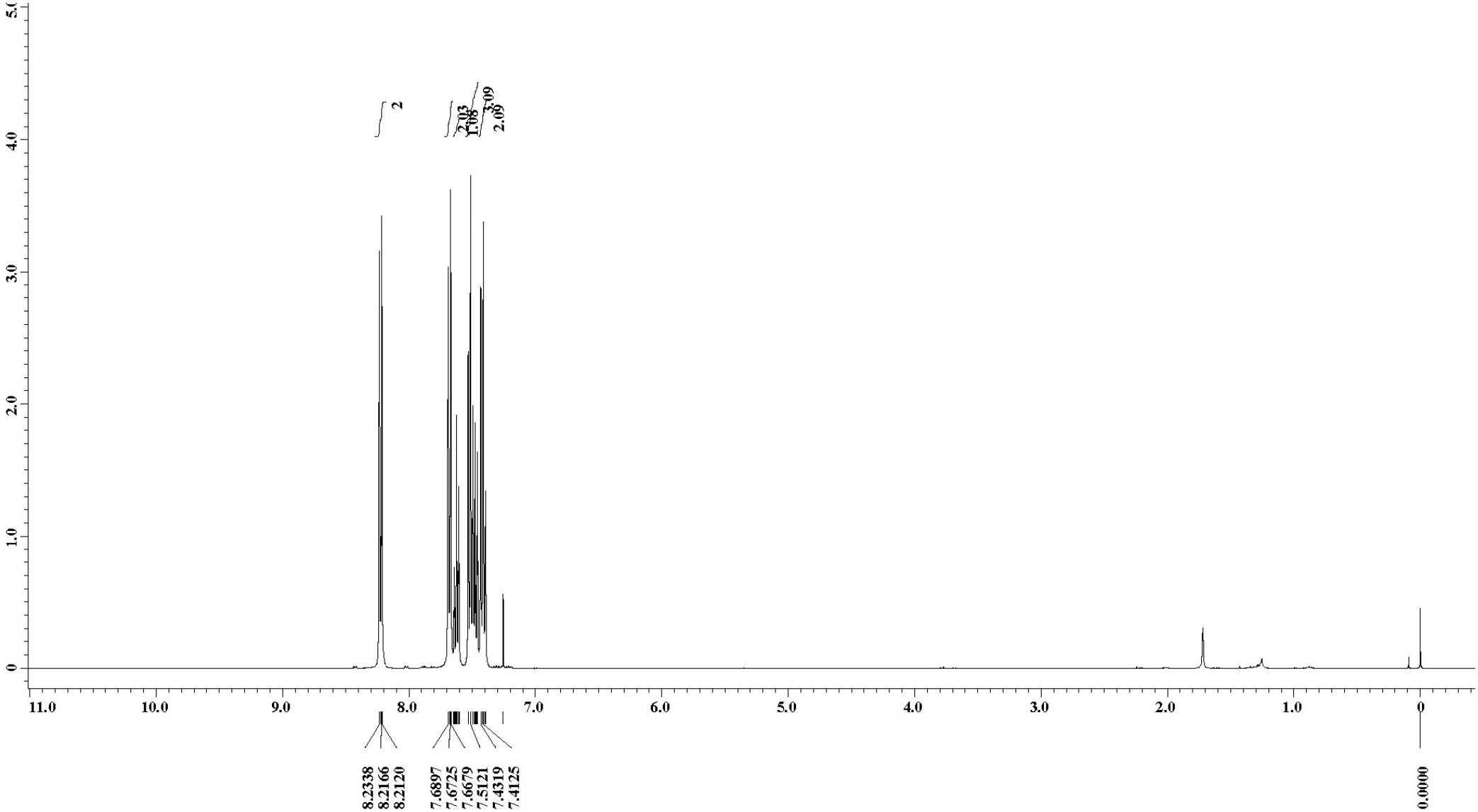


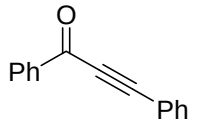
1b, ^{13}C NMR (100 MHz, $CDCl_3$)



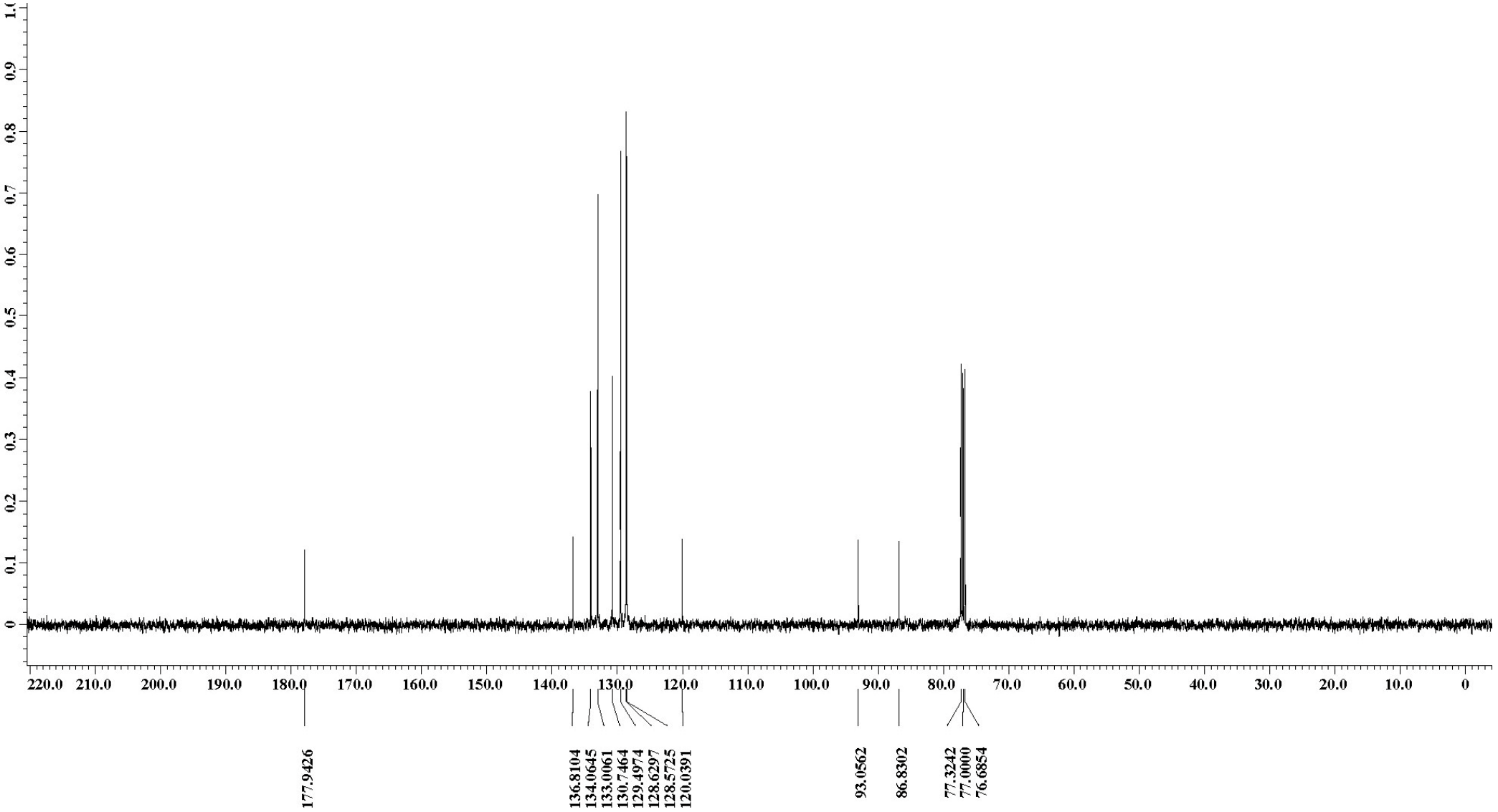


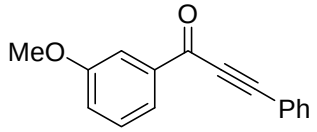
1c, ^{1}H NMR (400 MHz, $CDCl_3$)



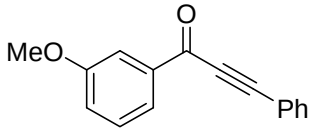


1c, ^{13}C NMR (100 MHz, $CDCl_3$)

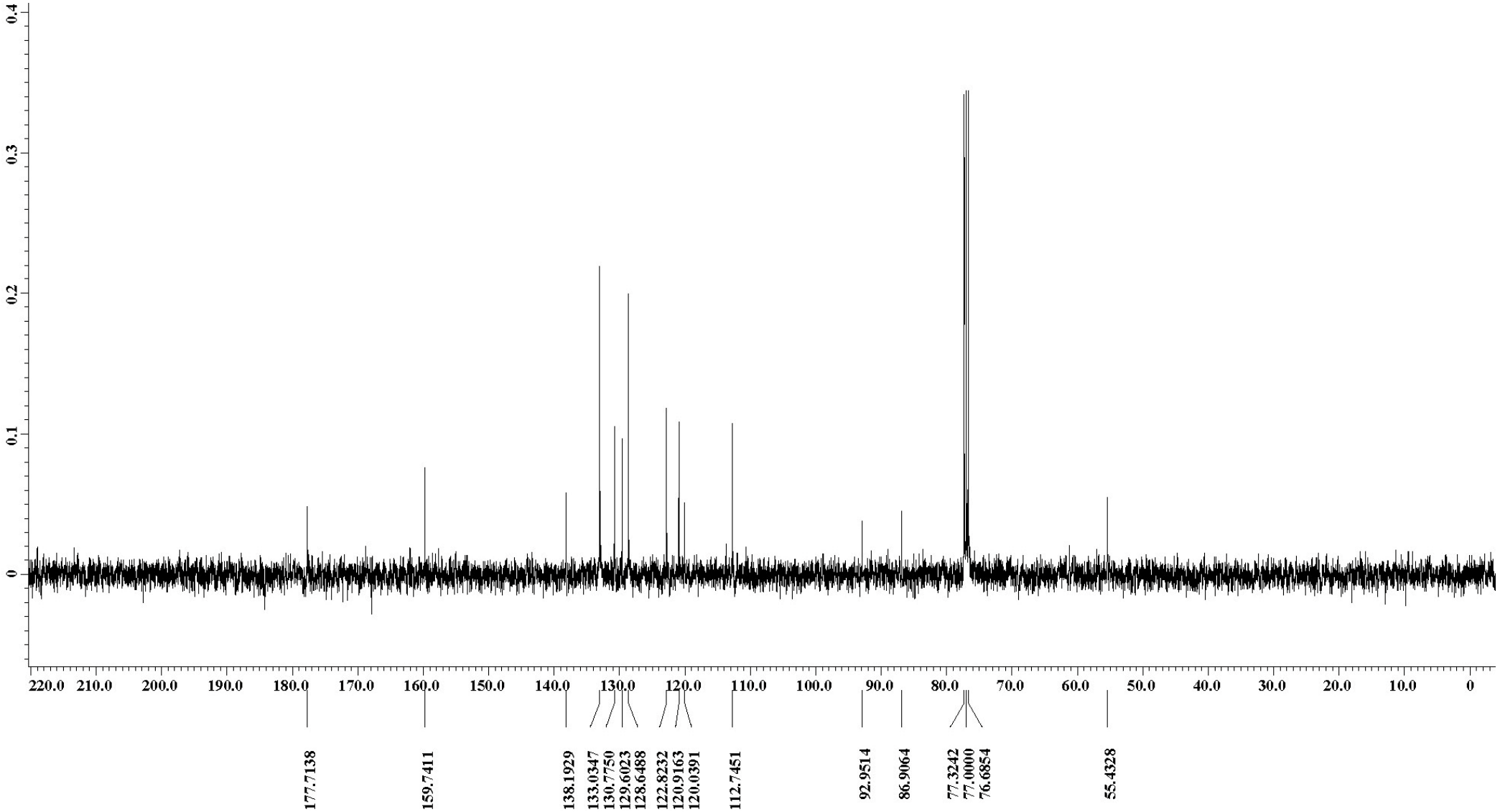


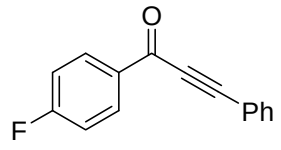


1d, ^{1}H NMR (400 MHz, $CDCl_3$)

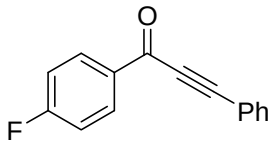


1d, ^{13}C NMR (100 MHz, $CDCl_3$)

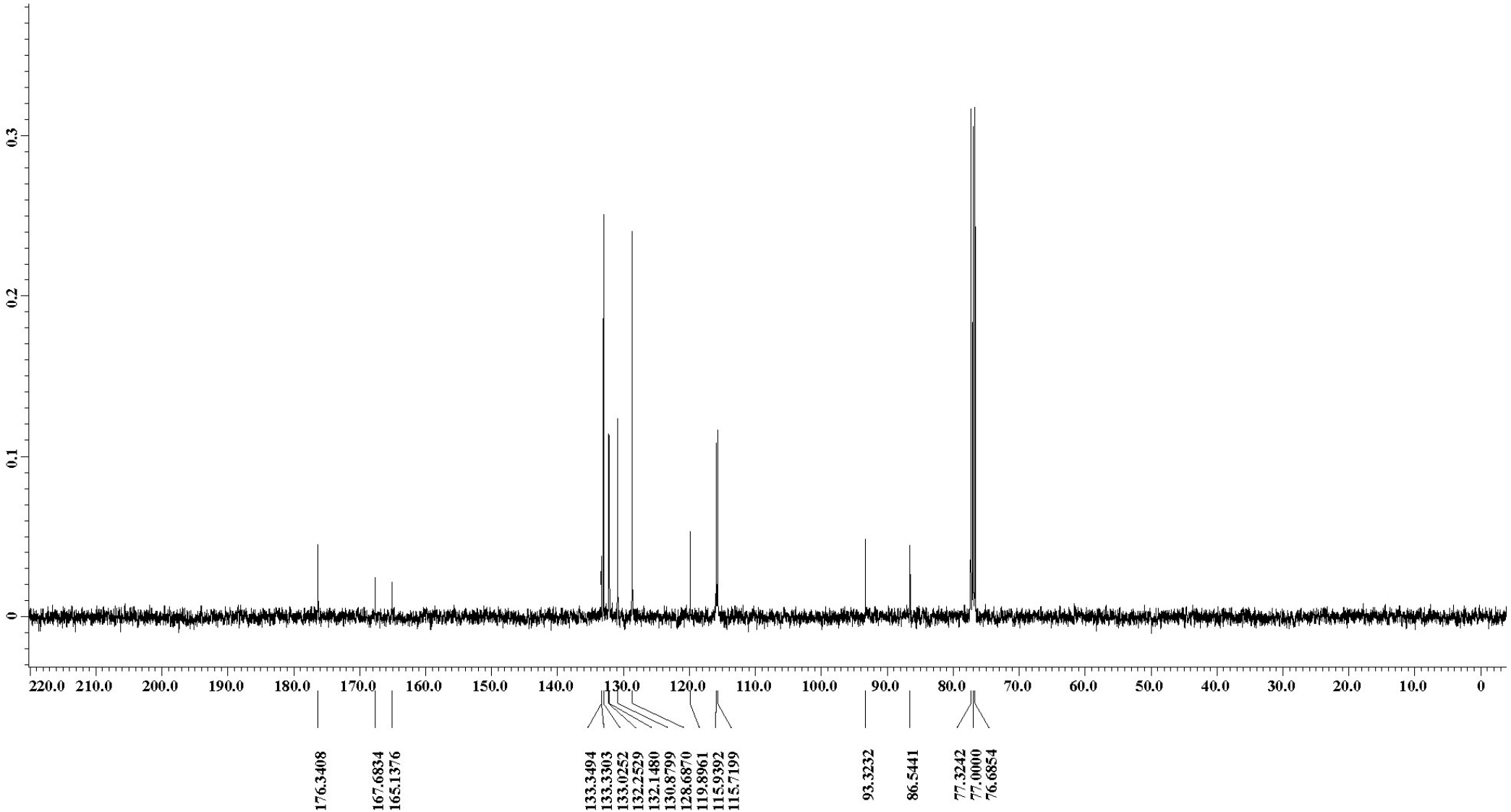


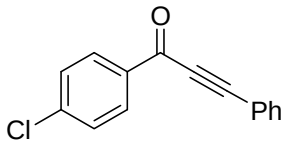


1e, ^{1}H NMR (400 MHz, $CDCl_3$)

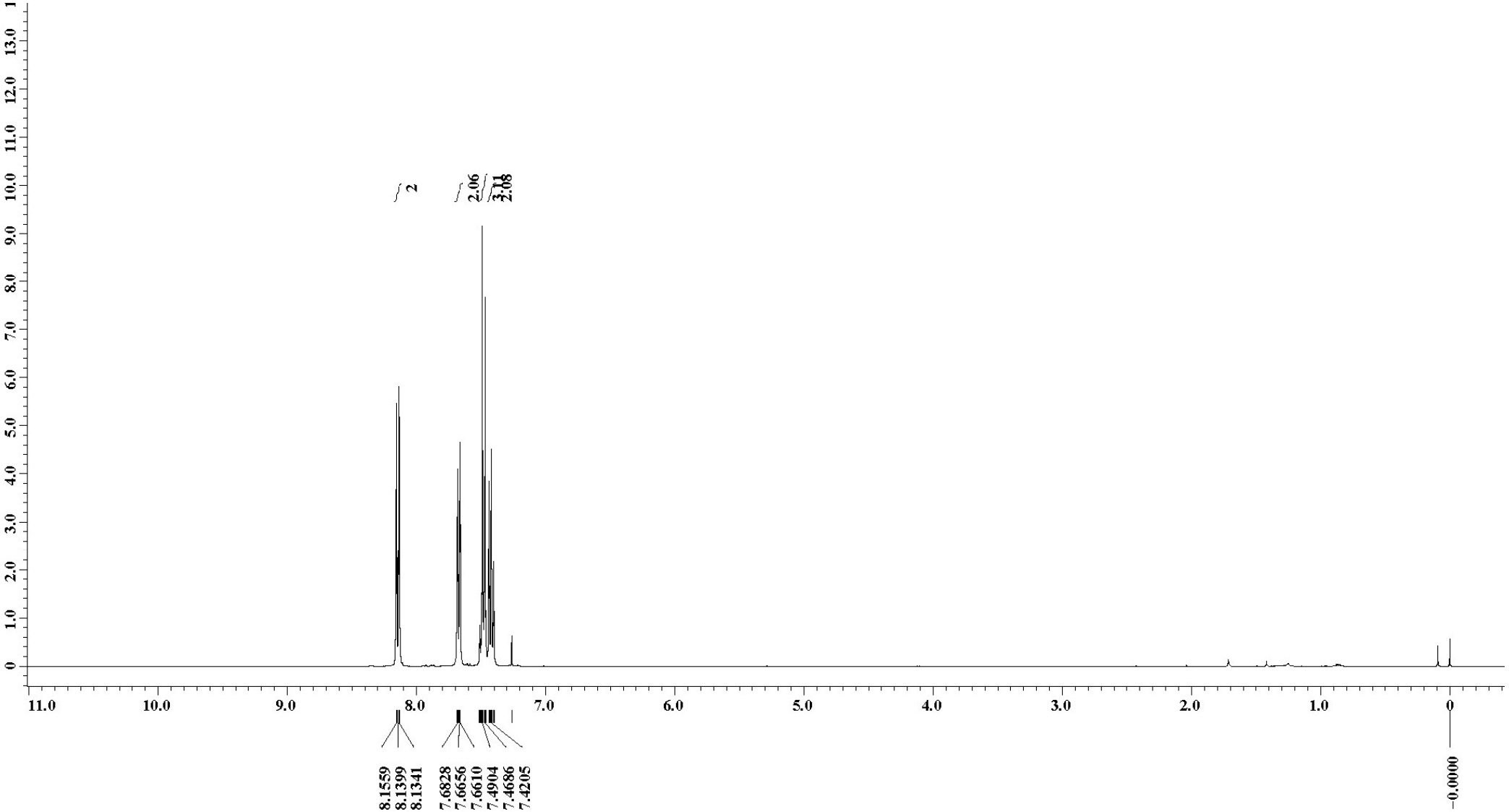


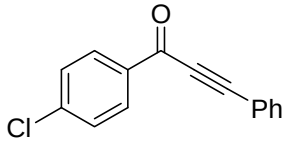
1e, ^{13}C NMR (100 MHz, $CDCl_3$)



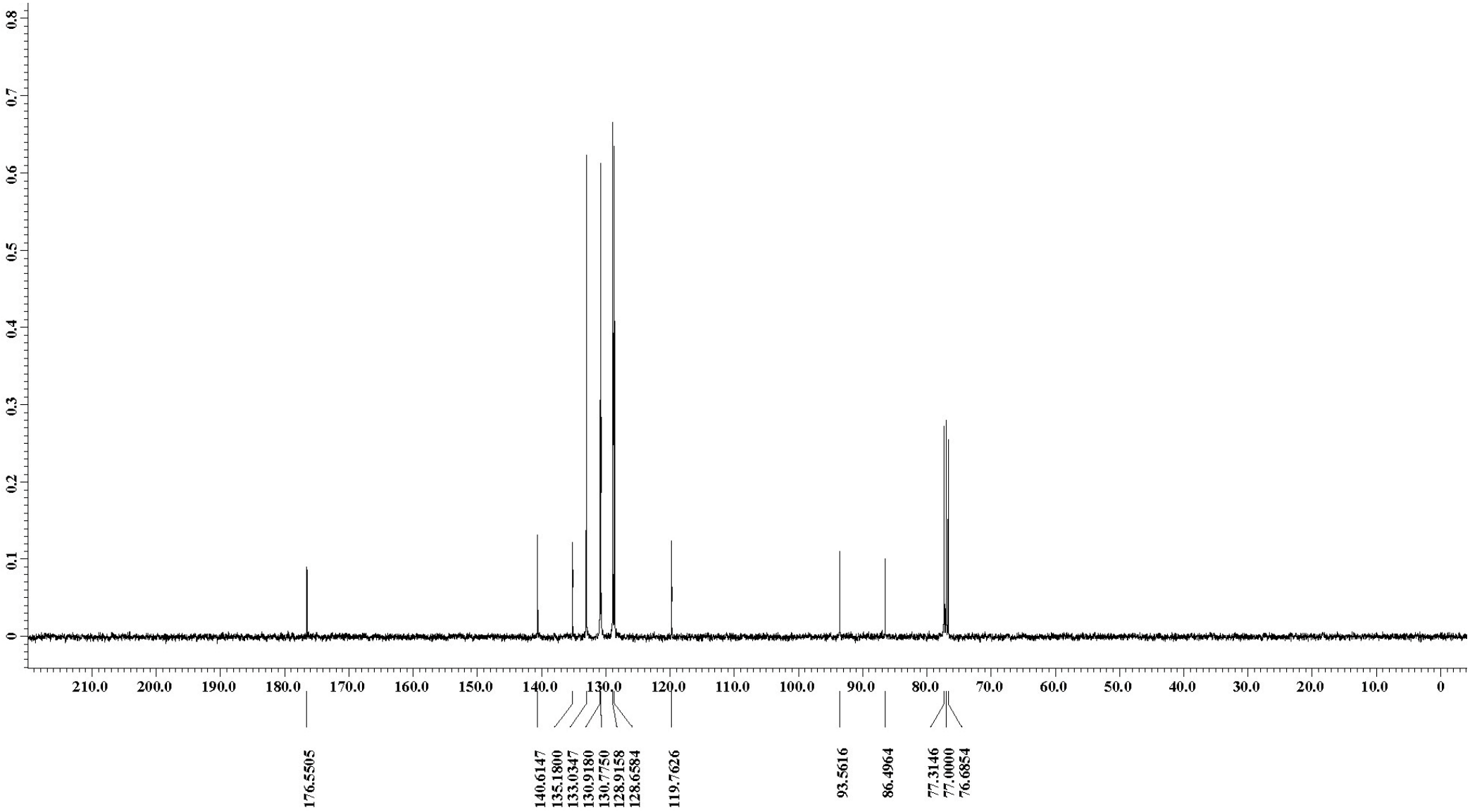


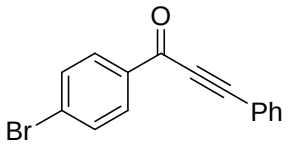
1f, ^{1}H NMR (400 MHz, $CDCl_3$)



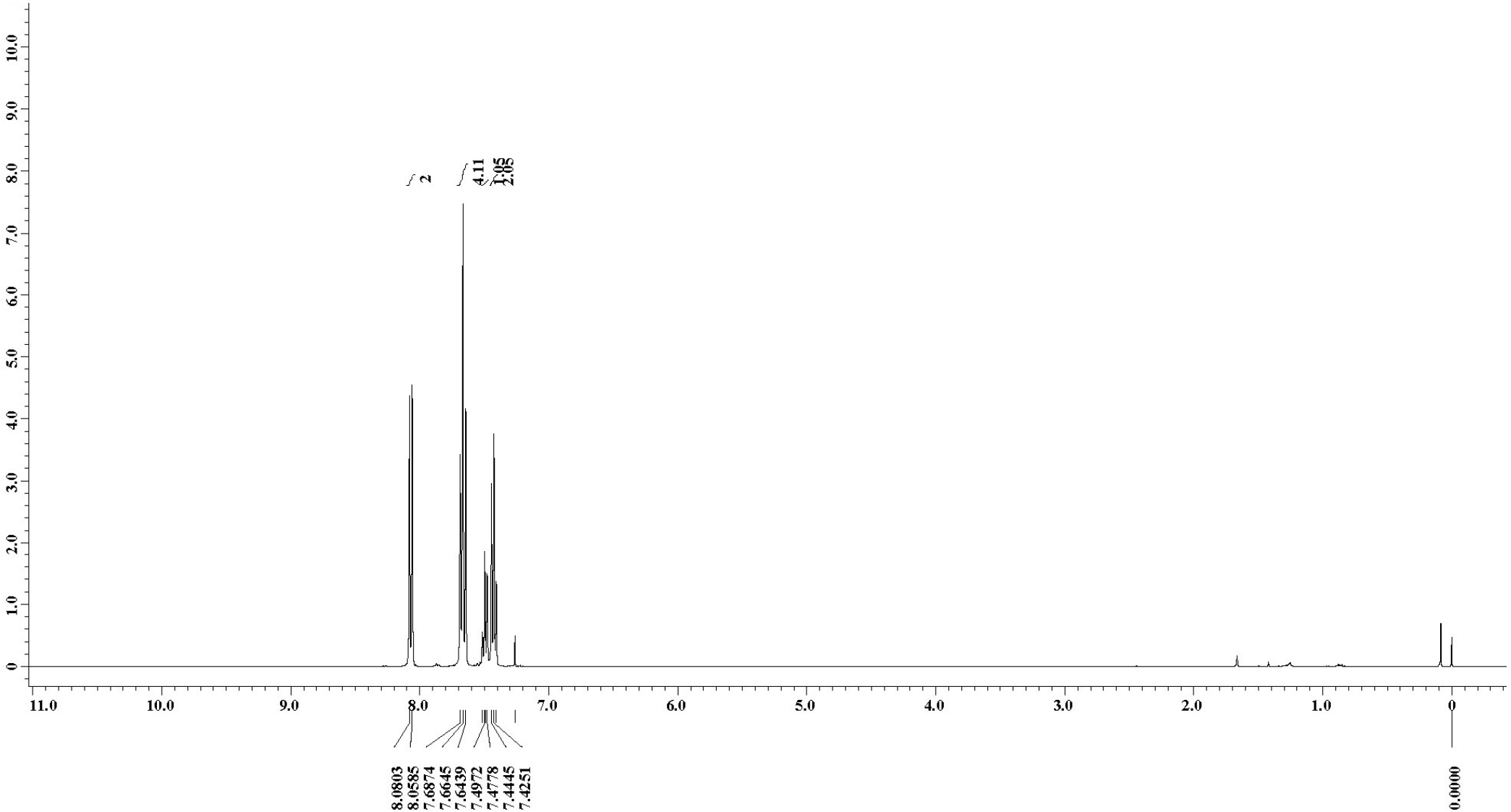


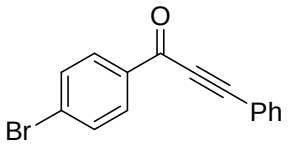
1f, ^{13}C NMR (100 MHz, $CDCl_3$)



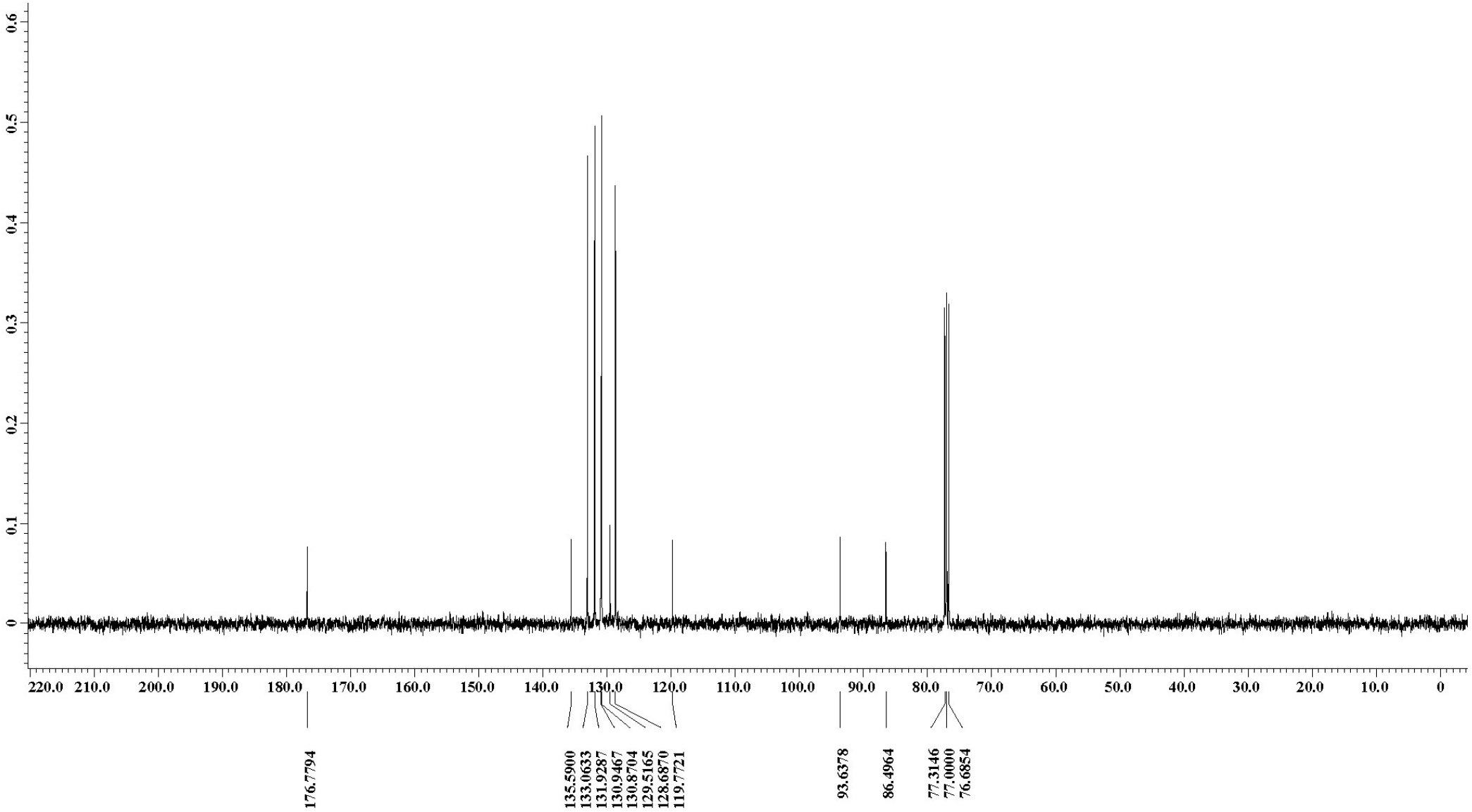


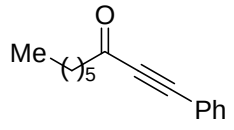
1g, ^{1}H NMR (400 MHz, $CDCl_3$)



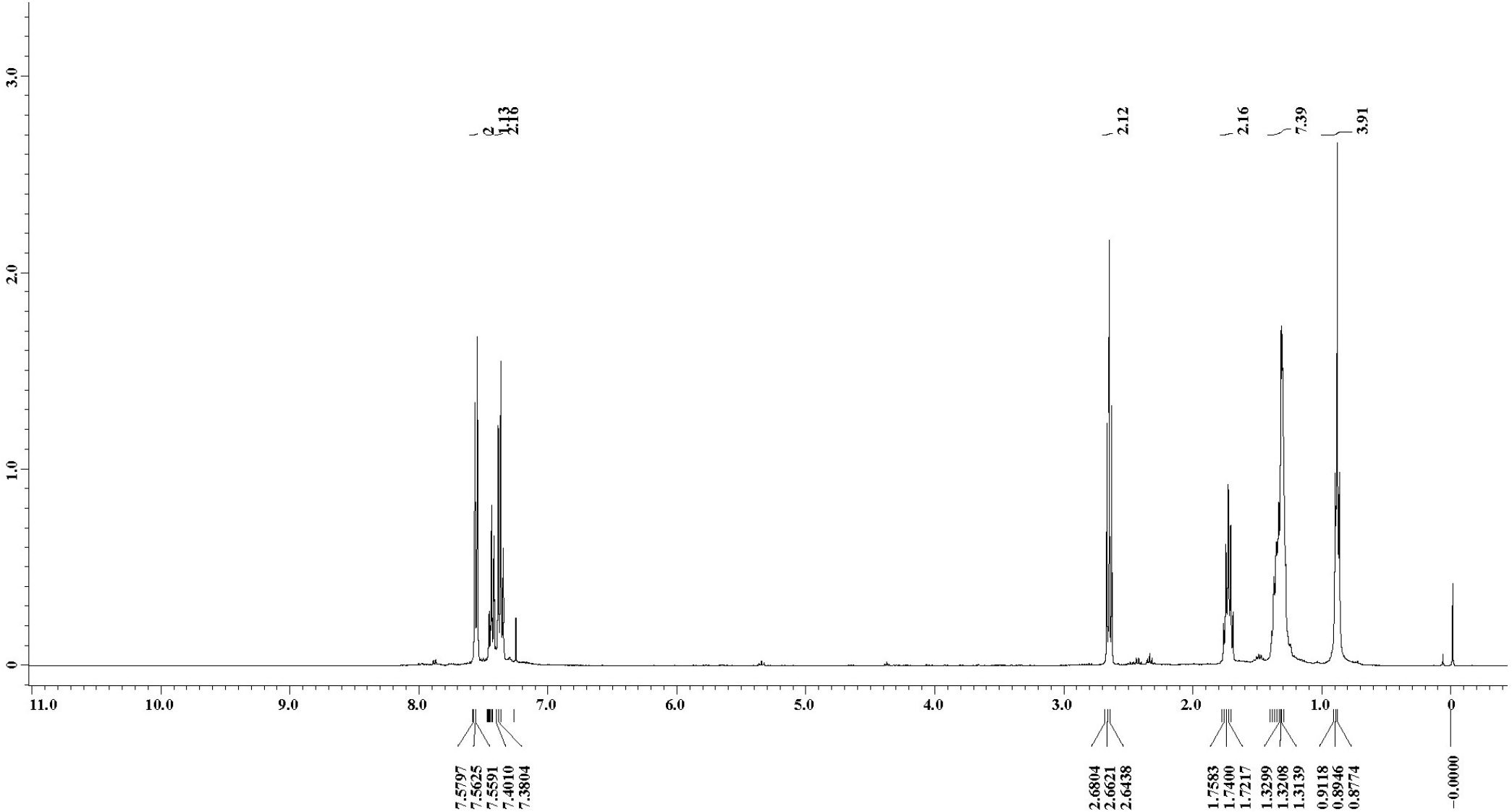


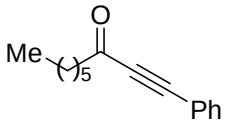
1g, ^{13}C NMR (100 MHz, $CDCl_3$)



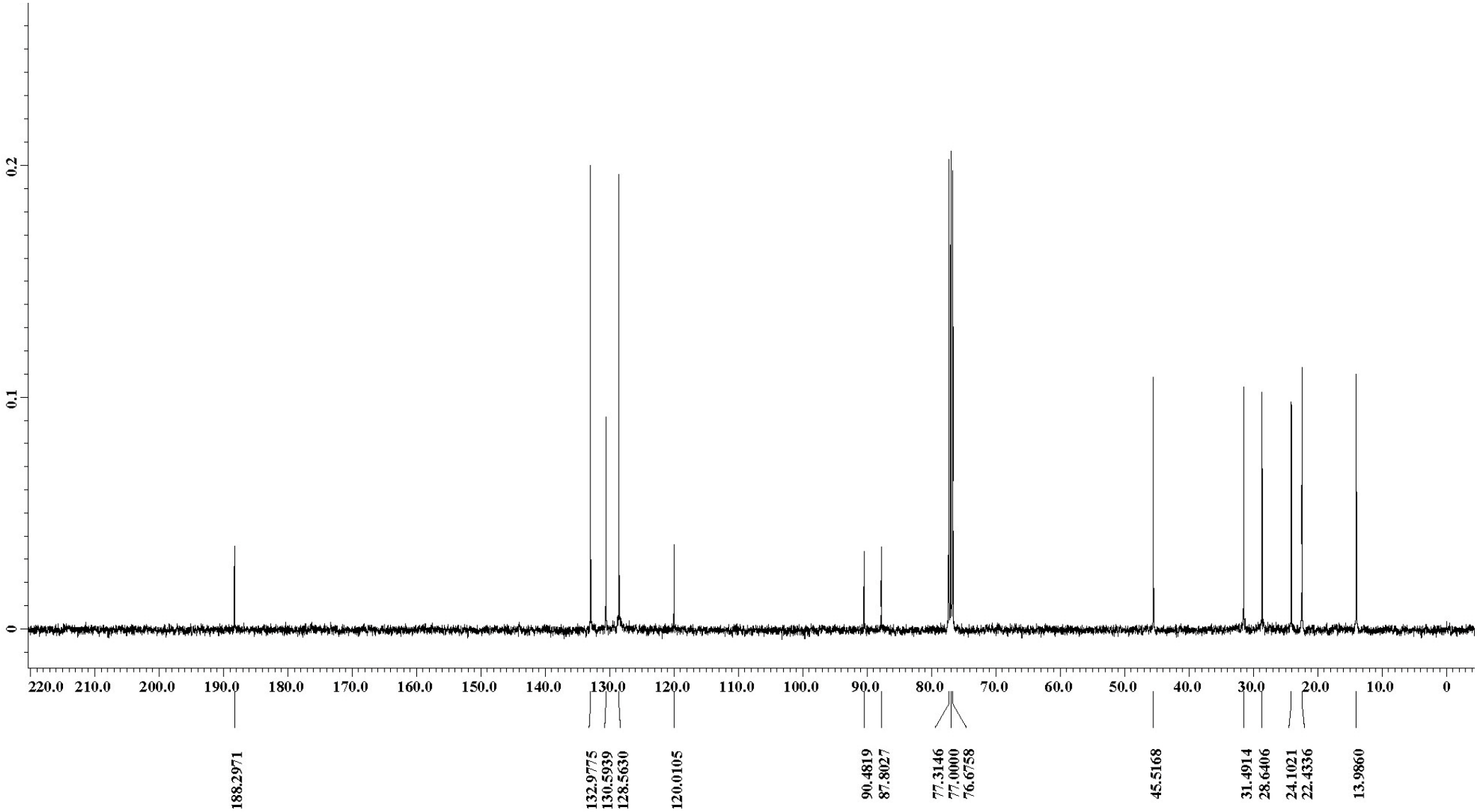


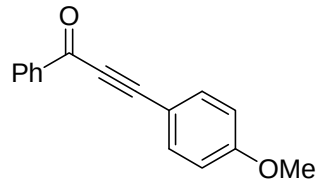
1h, ^{1}H NMR (400 MHz, $CDCl_3$)



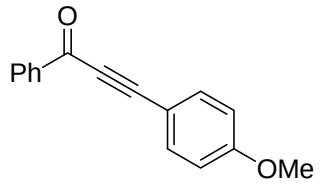


1h, ^{13}C NMR (100 MHz, $CDCl_3$)

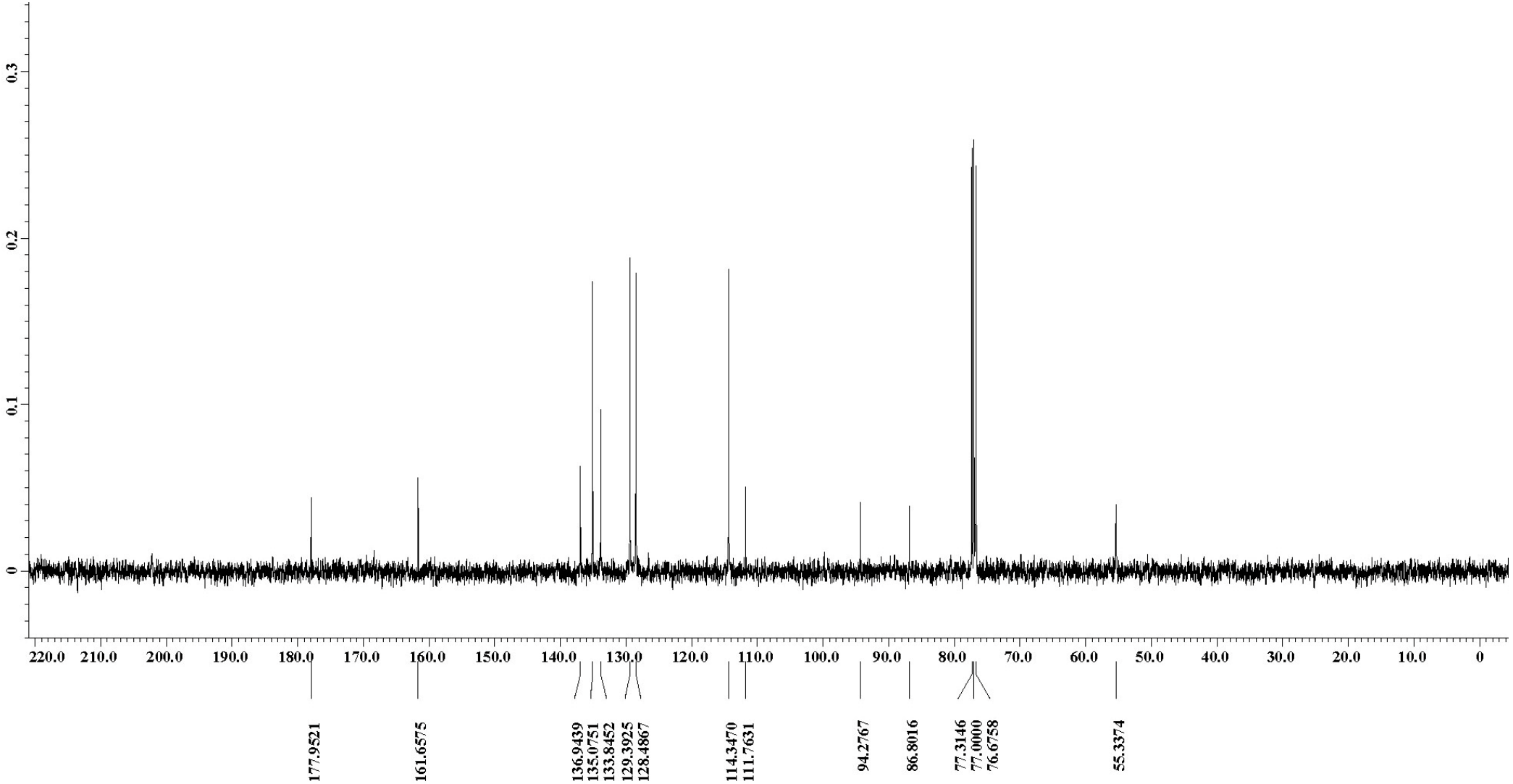


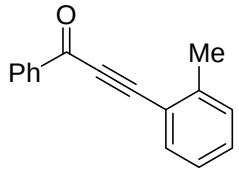


1i, ^{1}H NMR (400 MHz, $CDCl_3$)

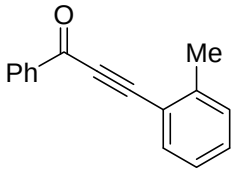


1i, ^{13}C NMR (100 MHz, $CDCl_3$)

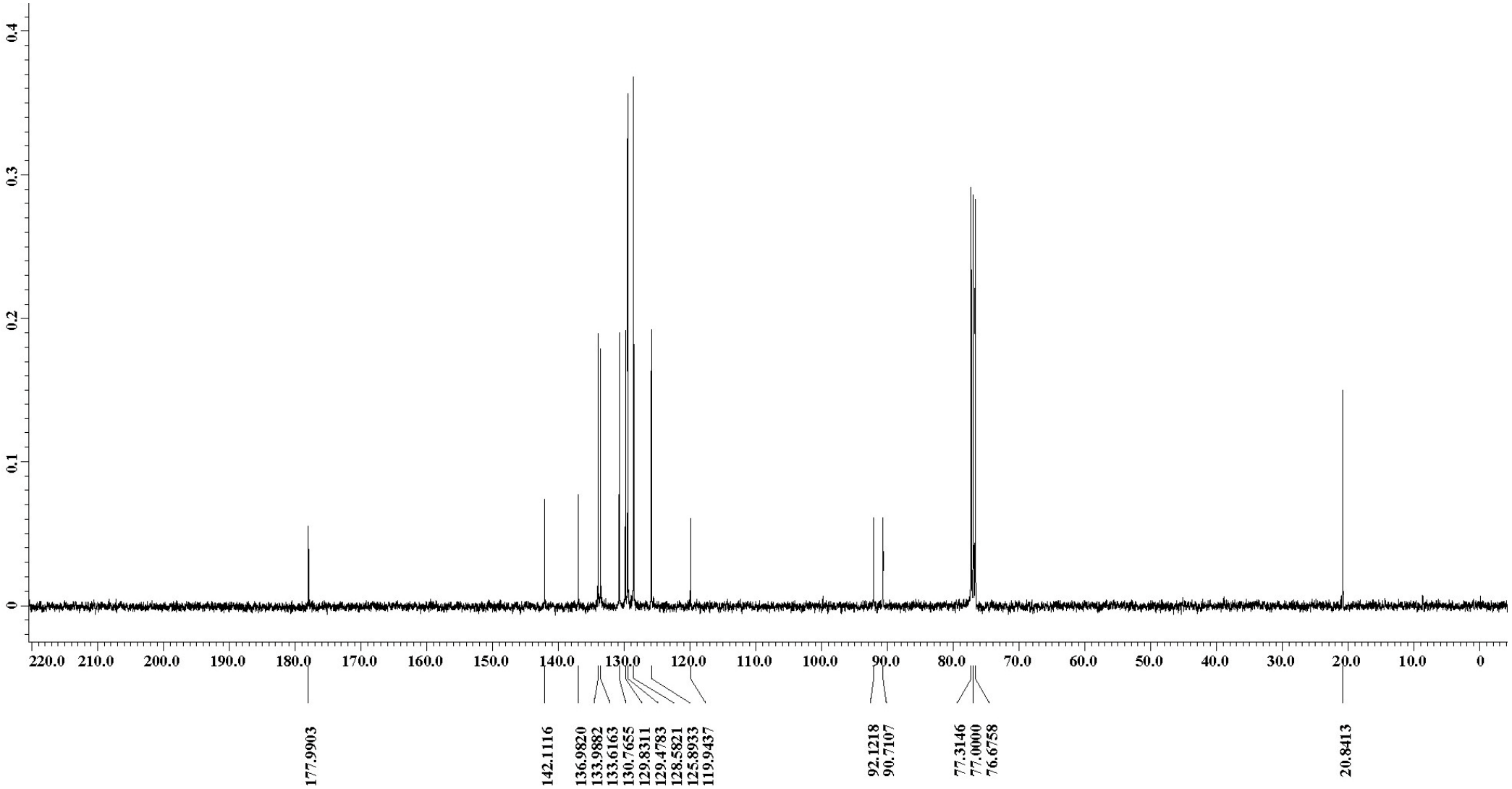


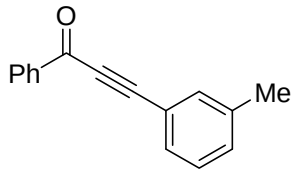


1j, ^{1}H NMR (400 MHz, $CDCl_3$)

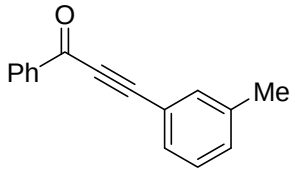


1j, ^{13}C NMR (100 MHz, $CDCl_3$)

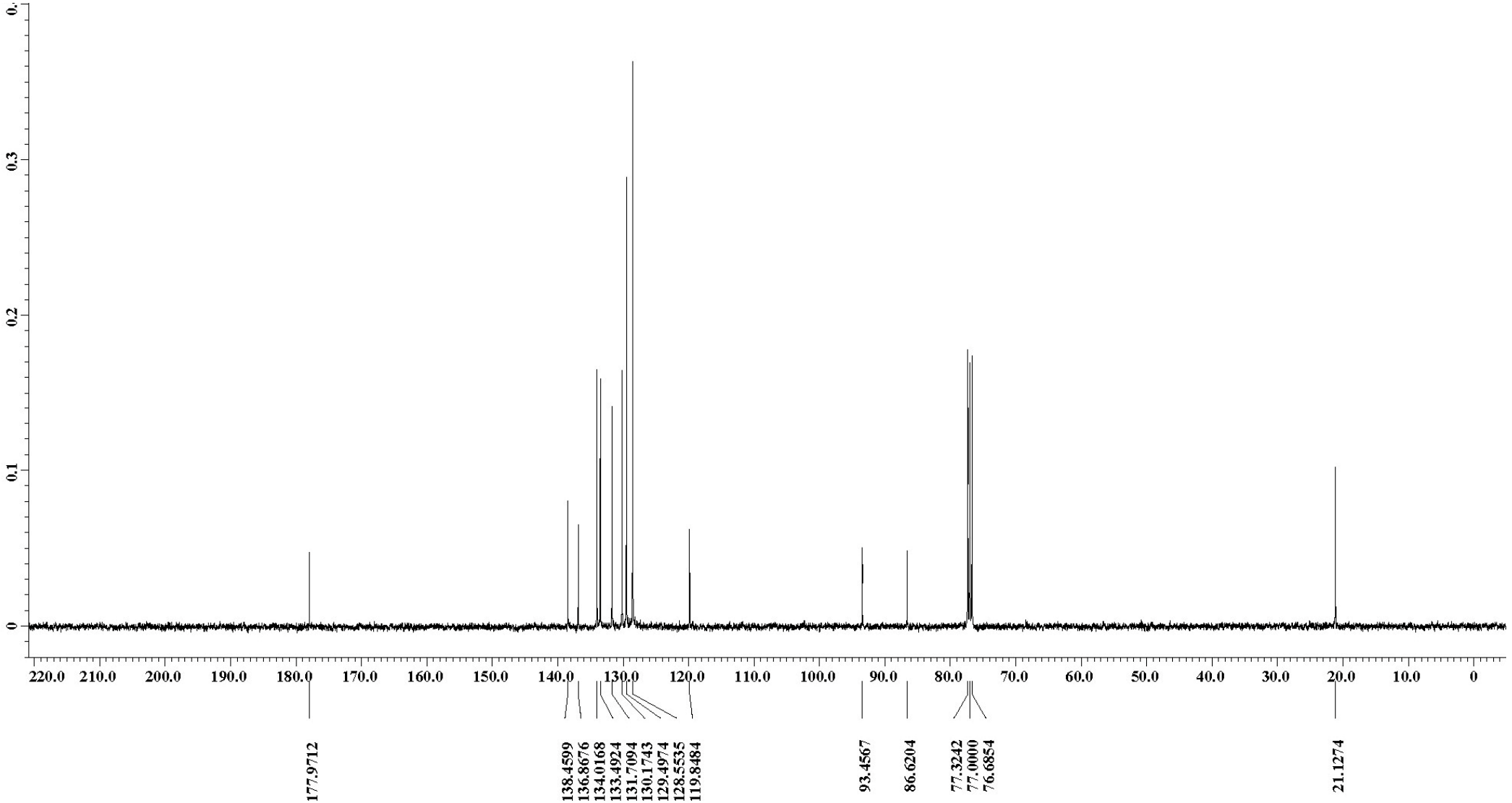


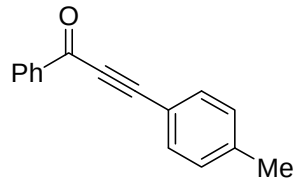


1k, ^{1}H NMR (400 MHz, $CDCl_3$)

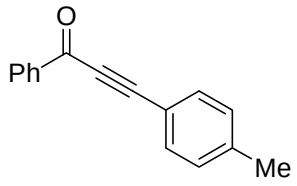


1k, ^{13}C NMR (100 MHz, $CDCl_3$)

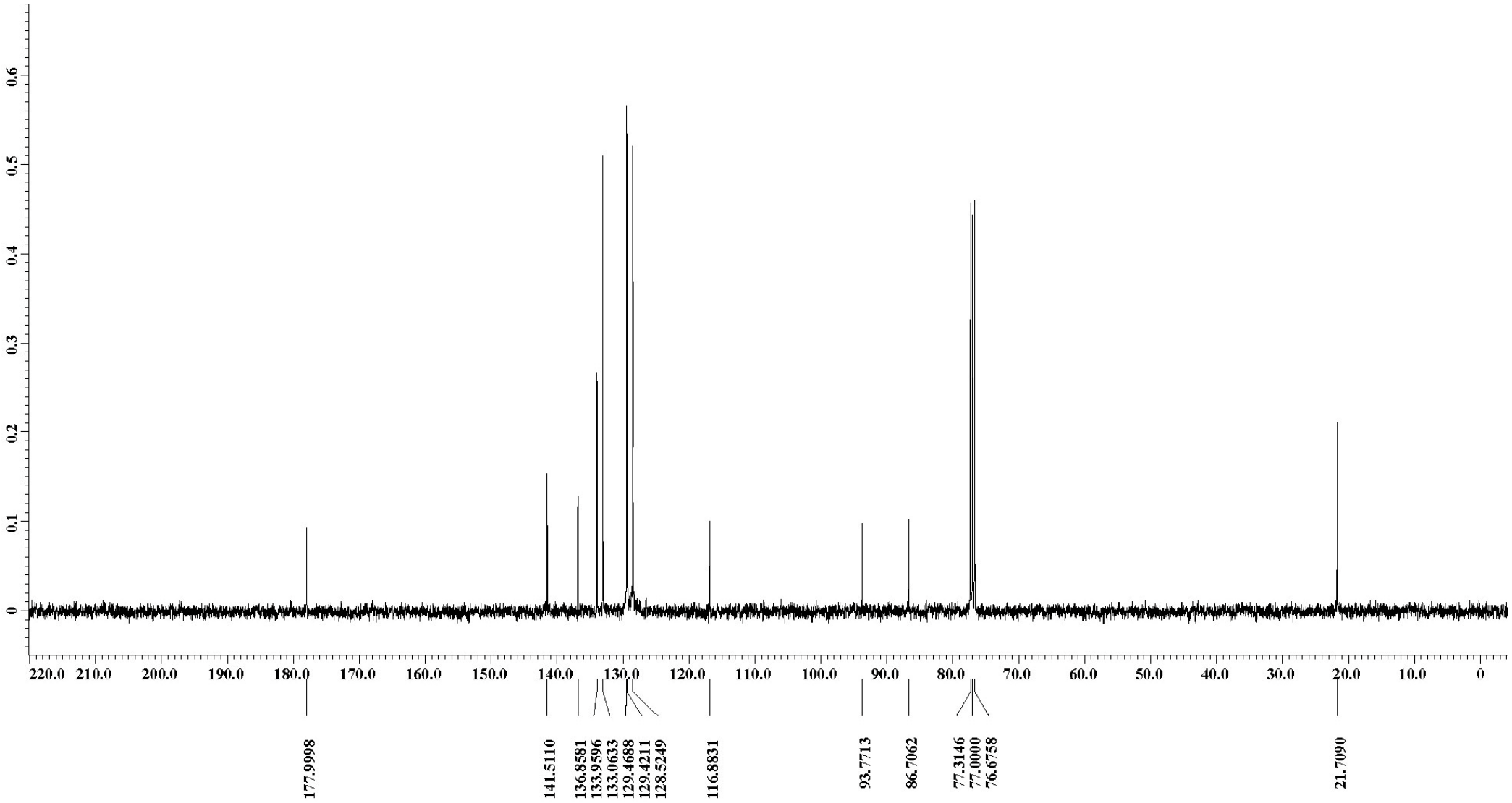


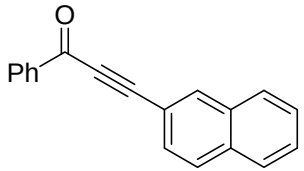


1l, ^{1}H NMR (400 MHz, $CDCl_3$)

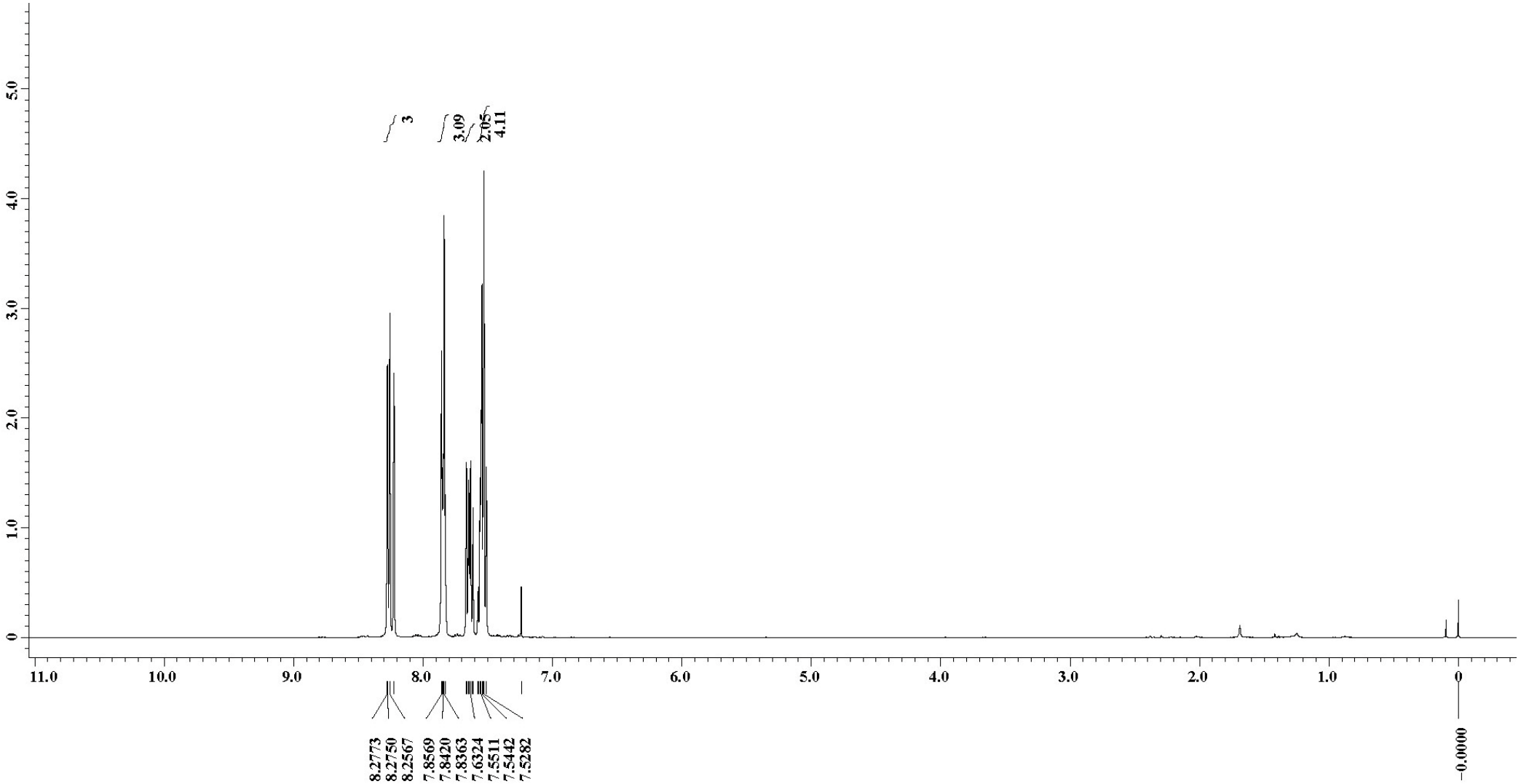


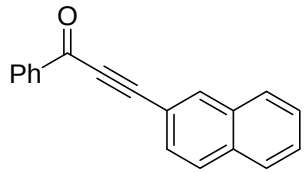
1l, ^{13}C NMR (100 MHz, $CDCl_3$)



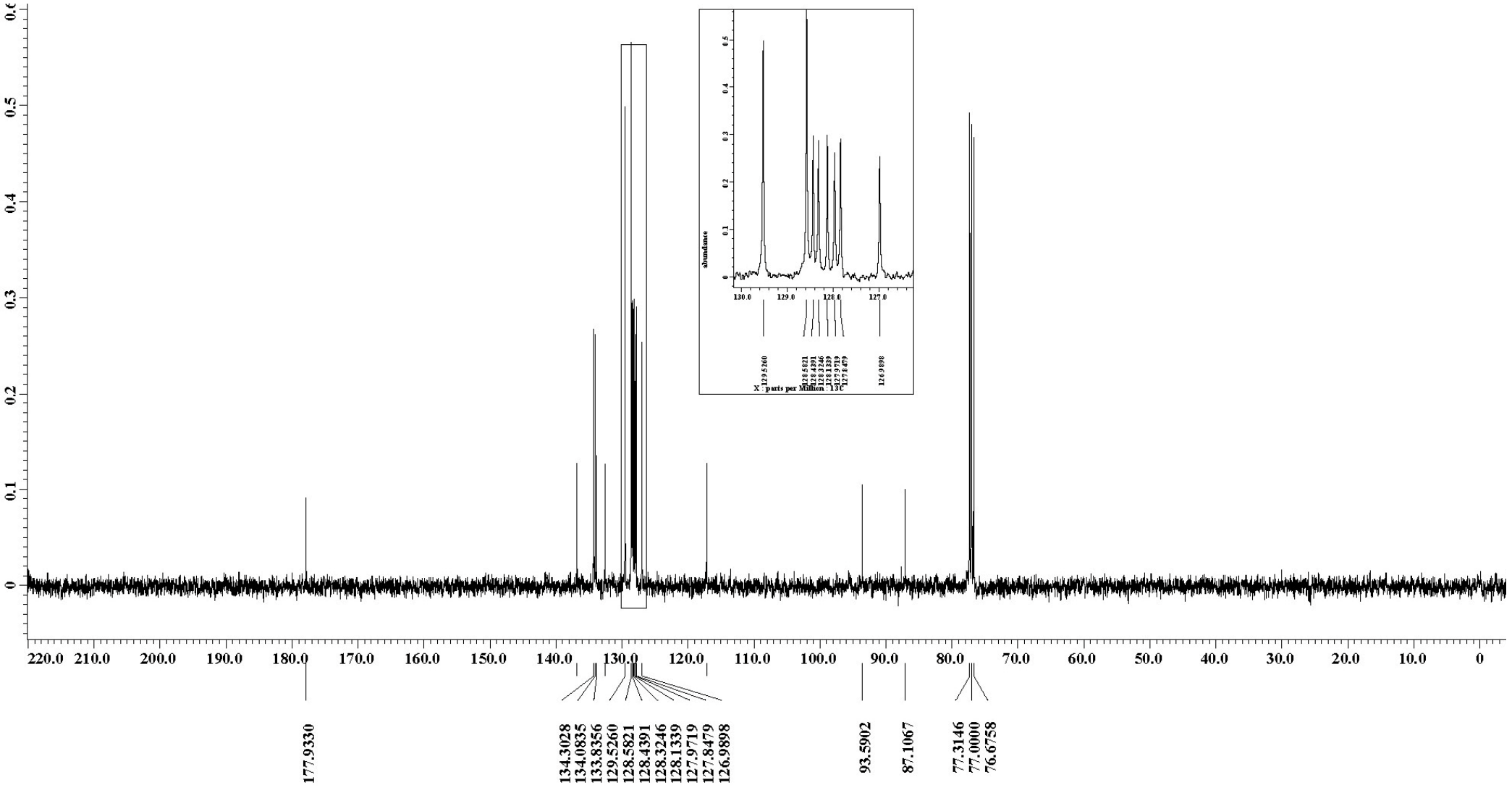


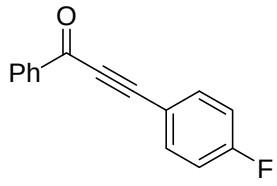
1m, ^{1}H NMR (400 MHz, $CDCl_3$)



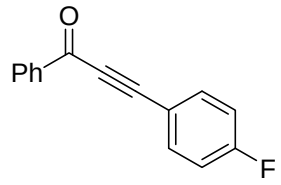


1m, ^{13}C NMR (100 MHz, $CDCl_3$)

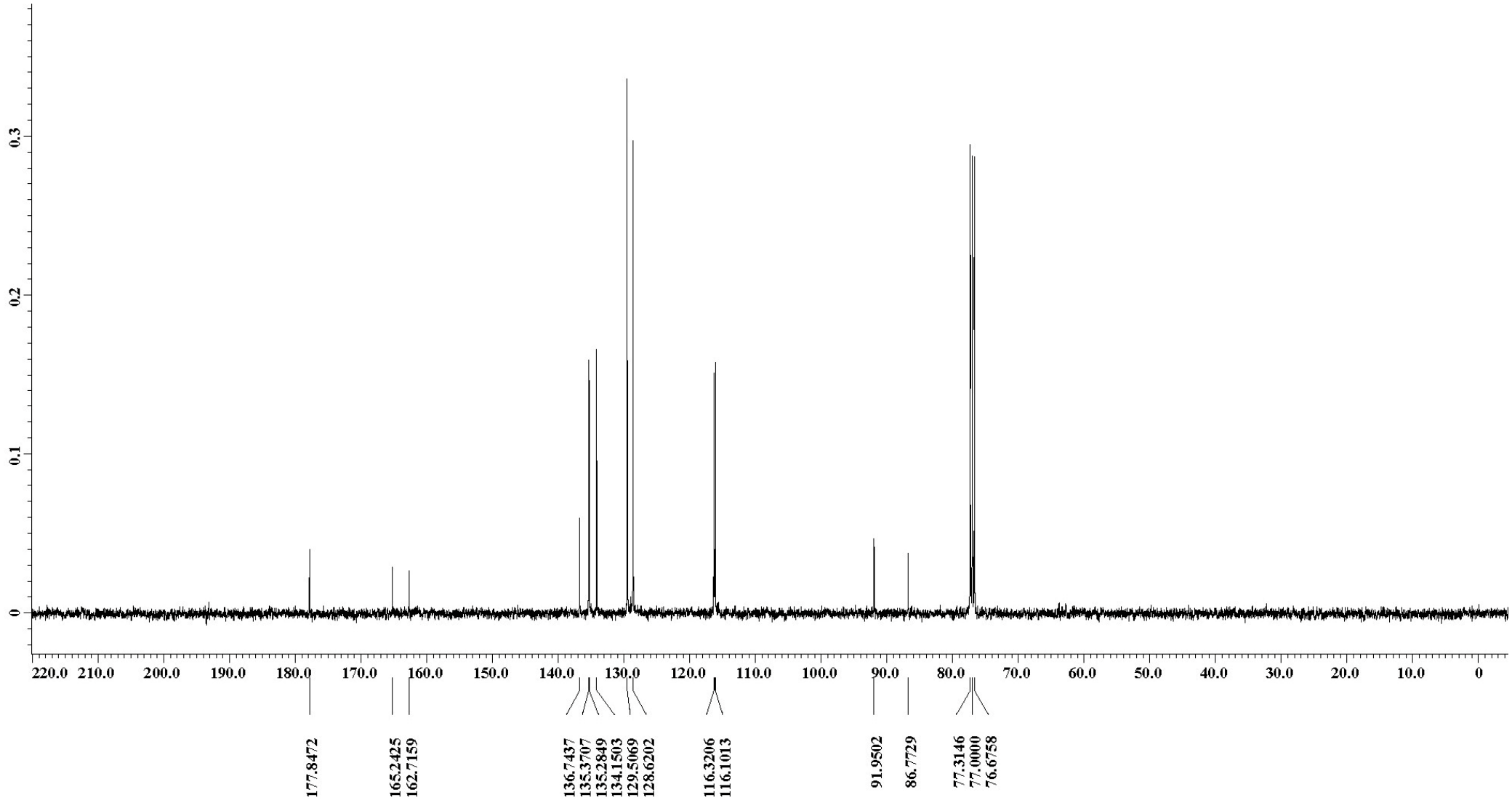


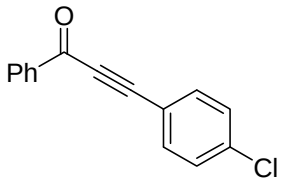


1n, ^{1}H NMR (400 MHz, $CDCl_3$)

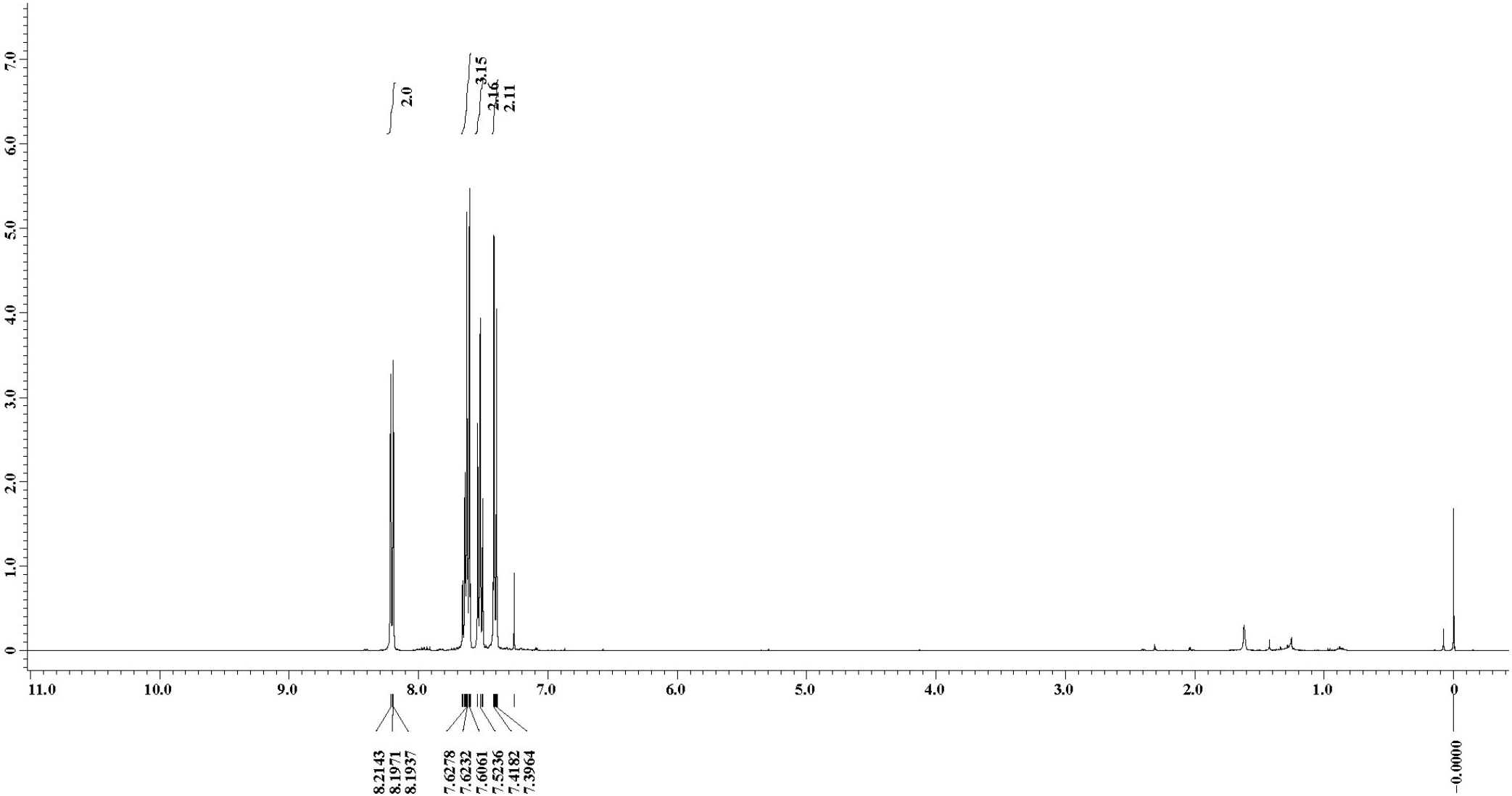


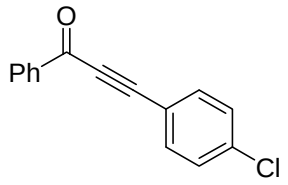
1n, ^{13}C NMR (100 MHz, $CDCl_3$)



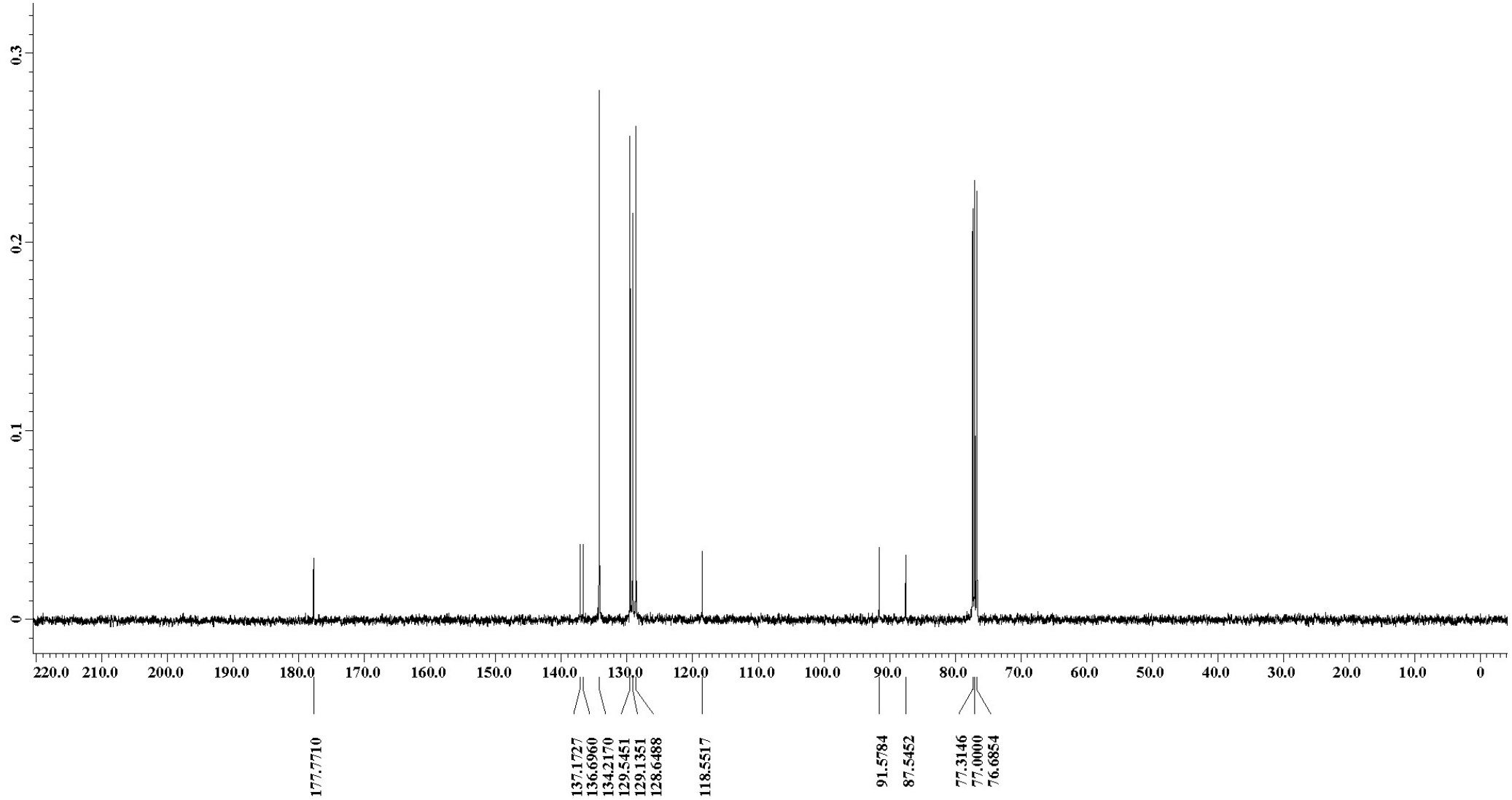


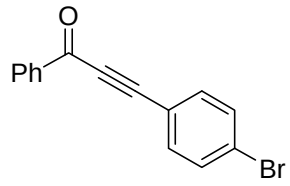
1o, ^{1}H NMR (400 MHz, $CDCl_3$)



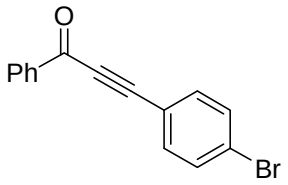


1o, ^{13}C NMR (100 MHz, $CDCl_3$)

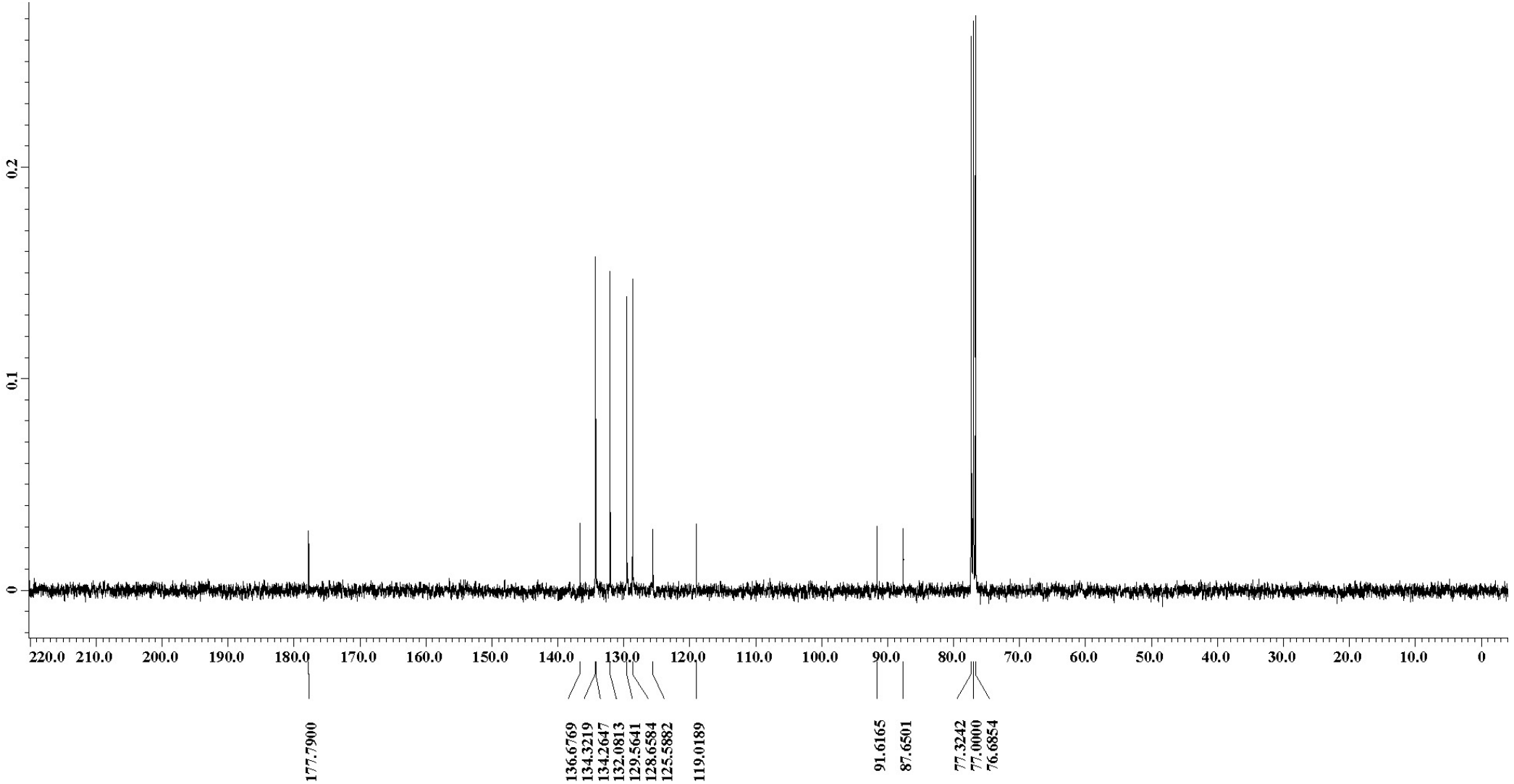


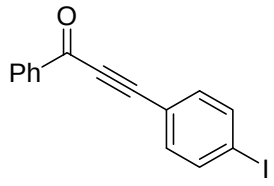


1p, ^{1}H NMR (400 MHz, $CDCl_3$)

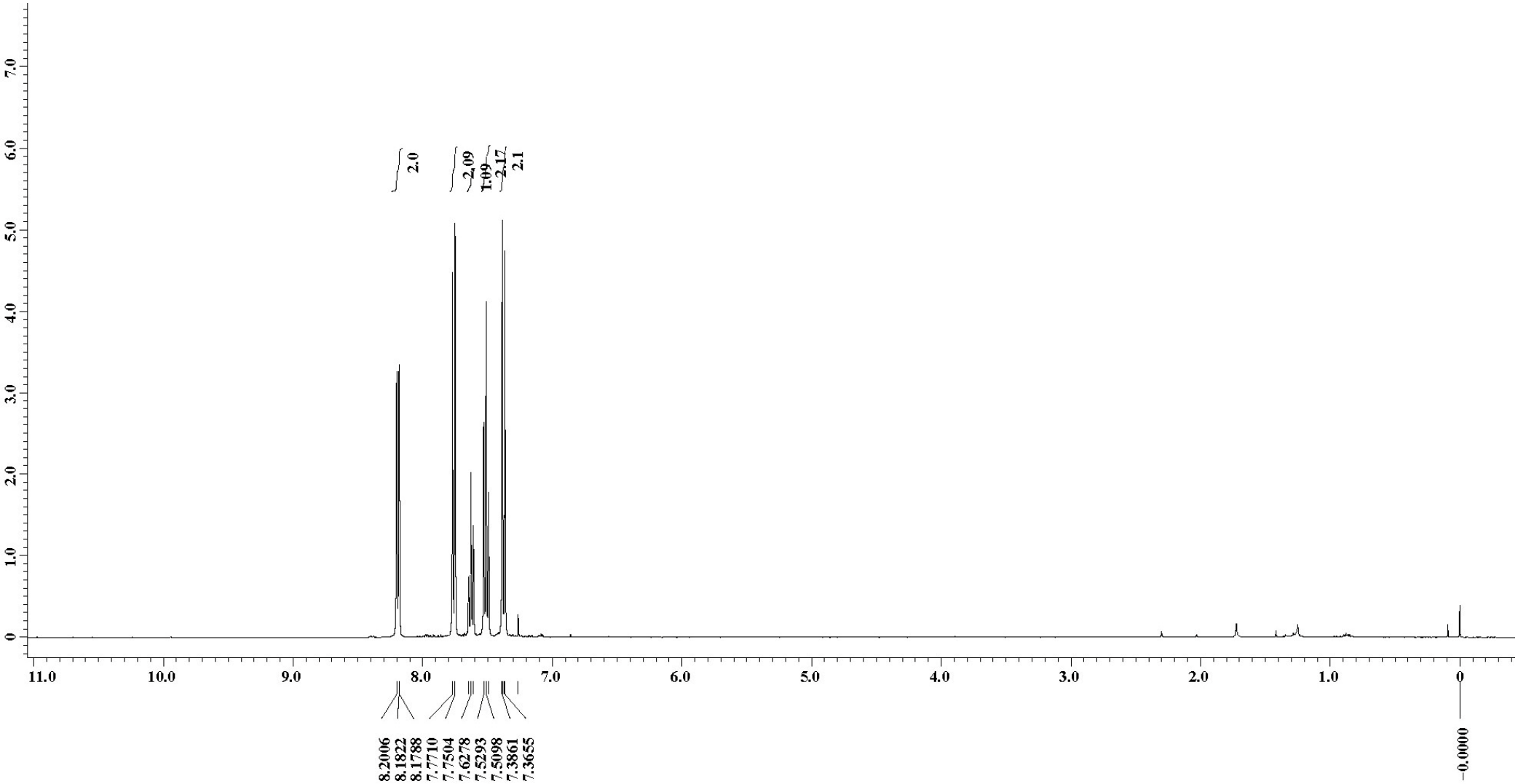


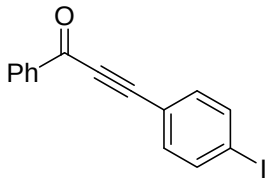
1p, ^{13}C NMR (100 MHz, $CDCl_3$)



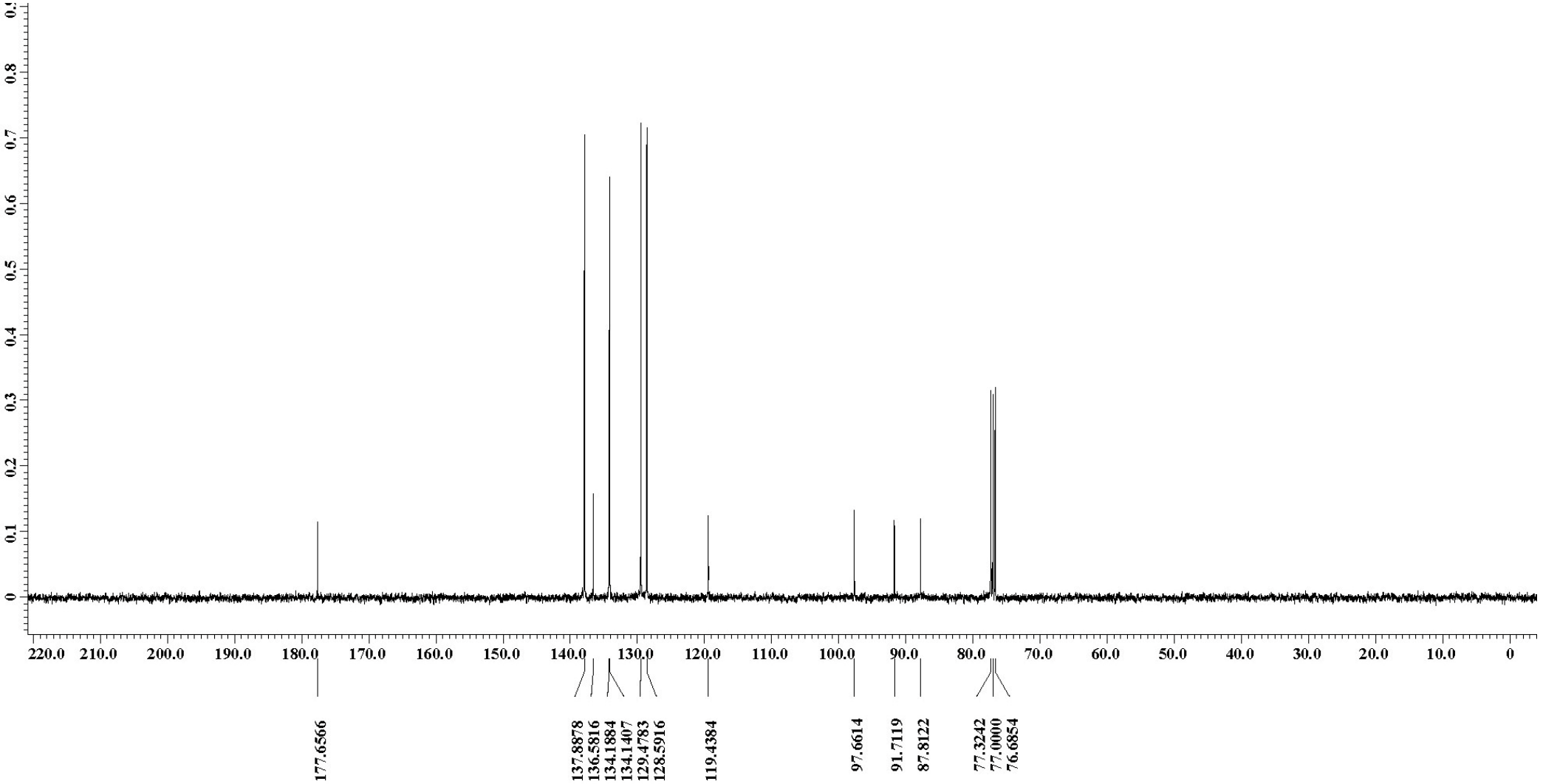


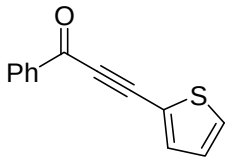
1q, ^{1}H NMR (400 MHz, $CDCl_3$)



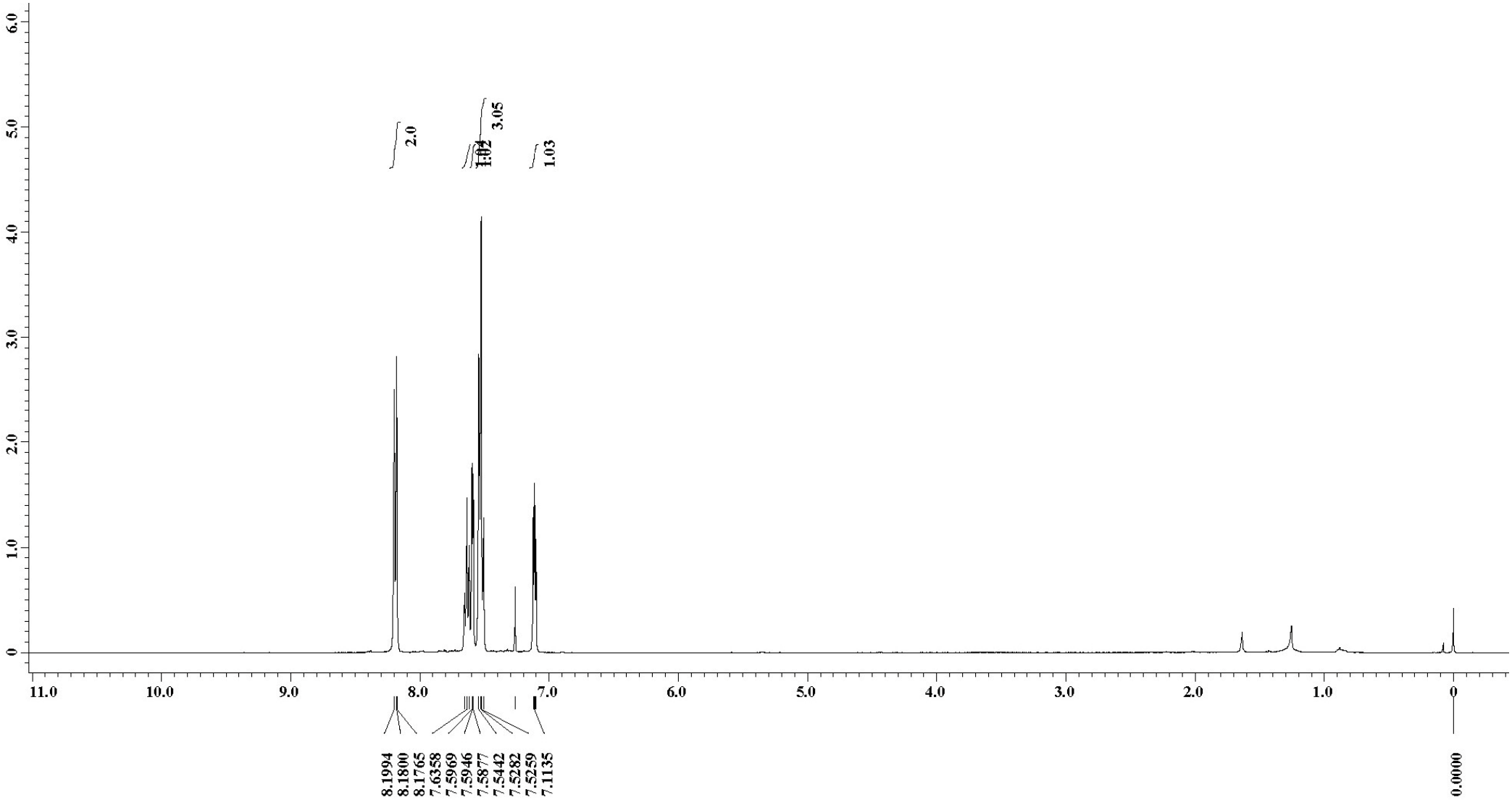


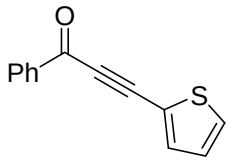
1q, ^{13}C NMR (100 MHz, $CDCl_3$)



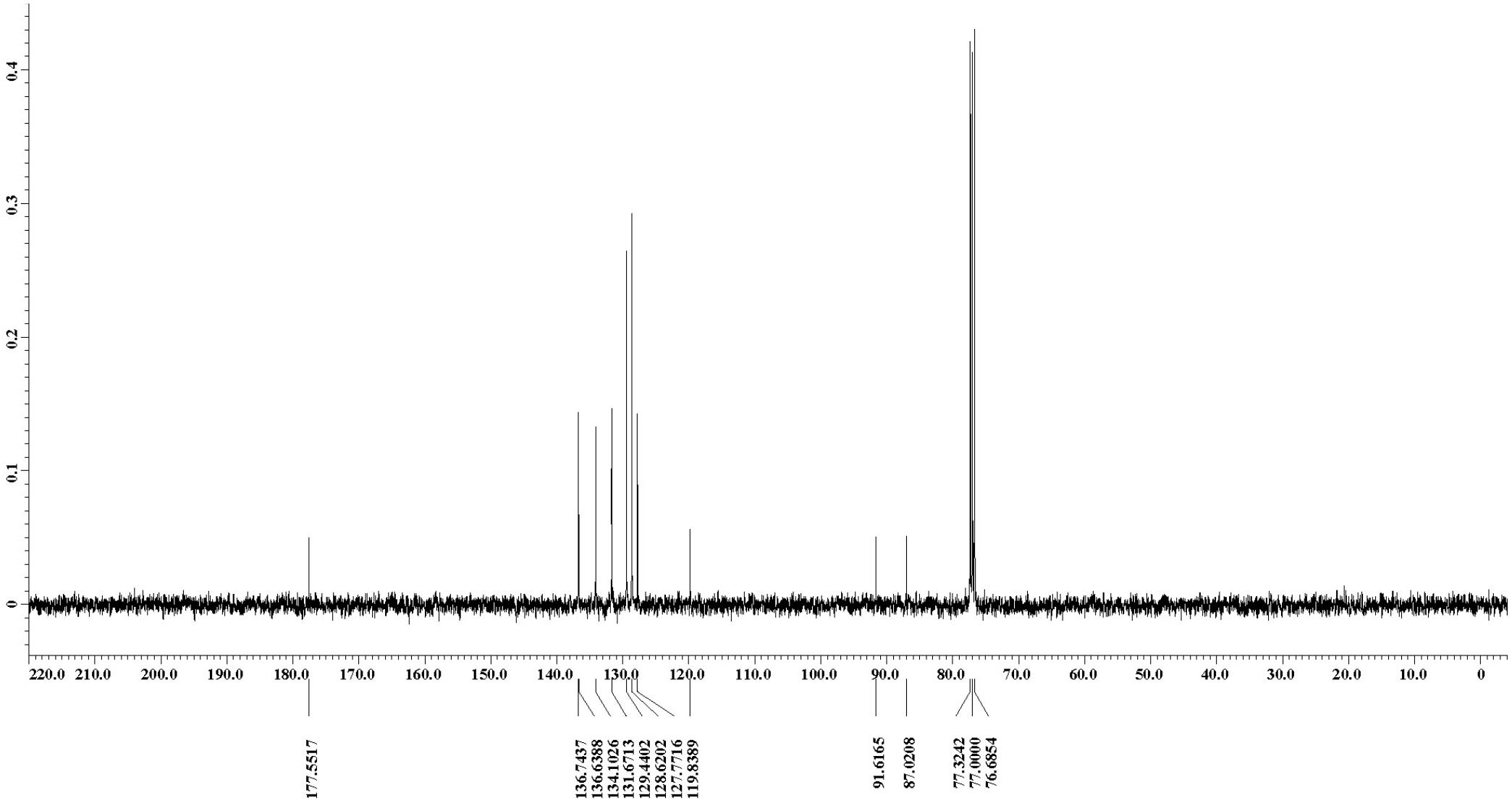


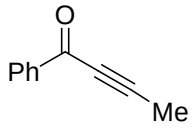
1r, ^{1}H NMR (400 MHz, $CDCl_3$)



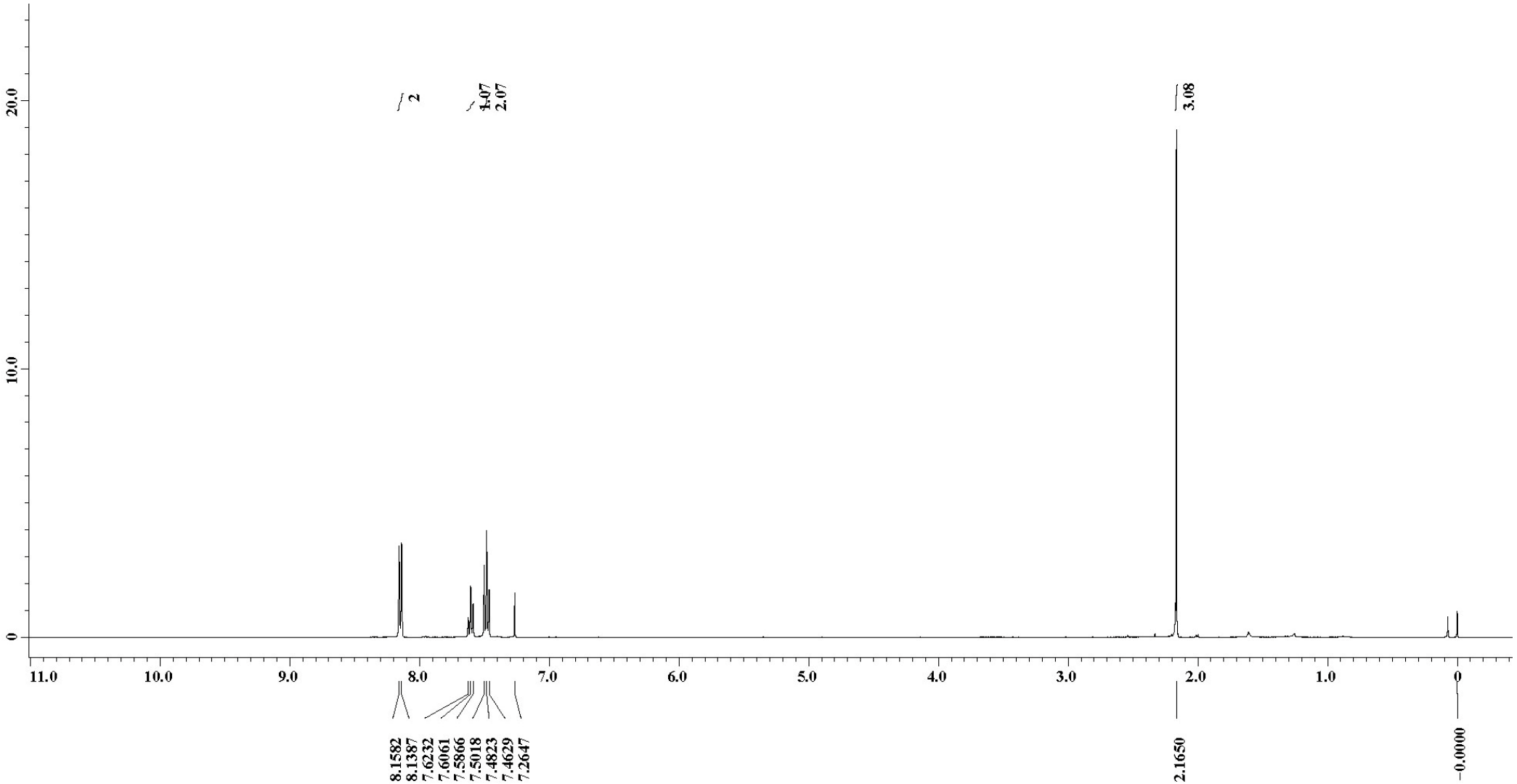


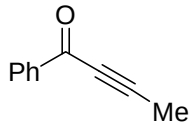
1r, ^{13}C NMR (100 MHz, $CDCl_3$)



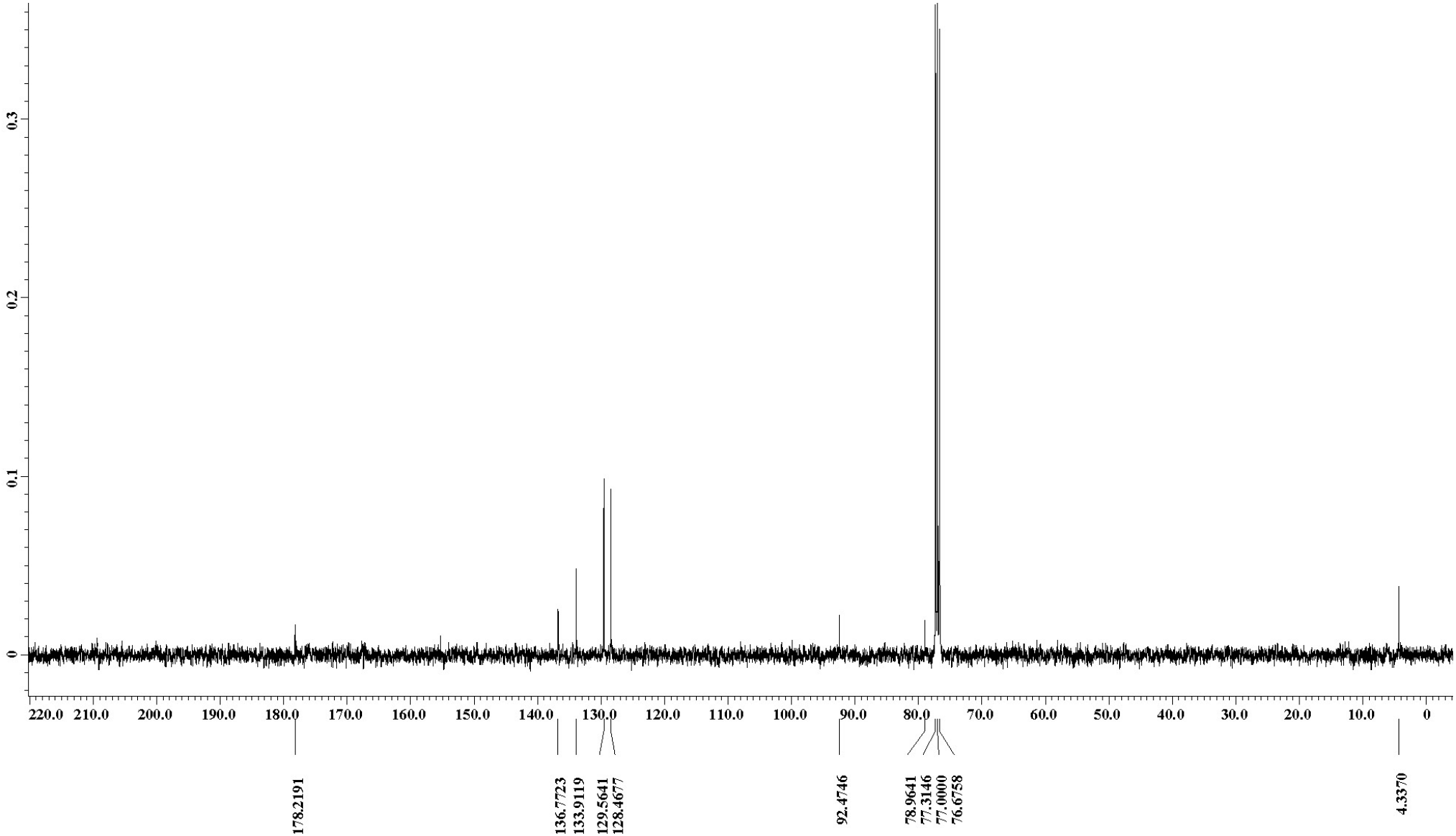


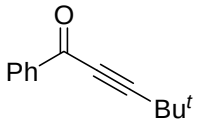
1s, ^{1}H NMR (400 MHz, $CDCl_3$)



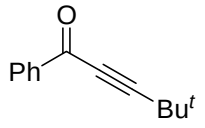


1s, ^{13}C NMR (100 MHz, $CDCl_3$)

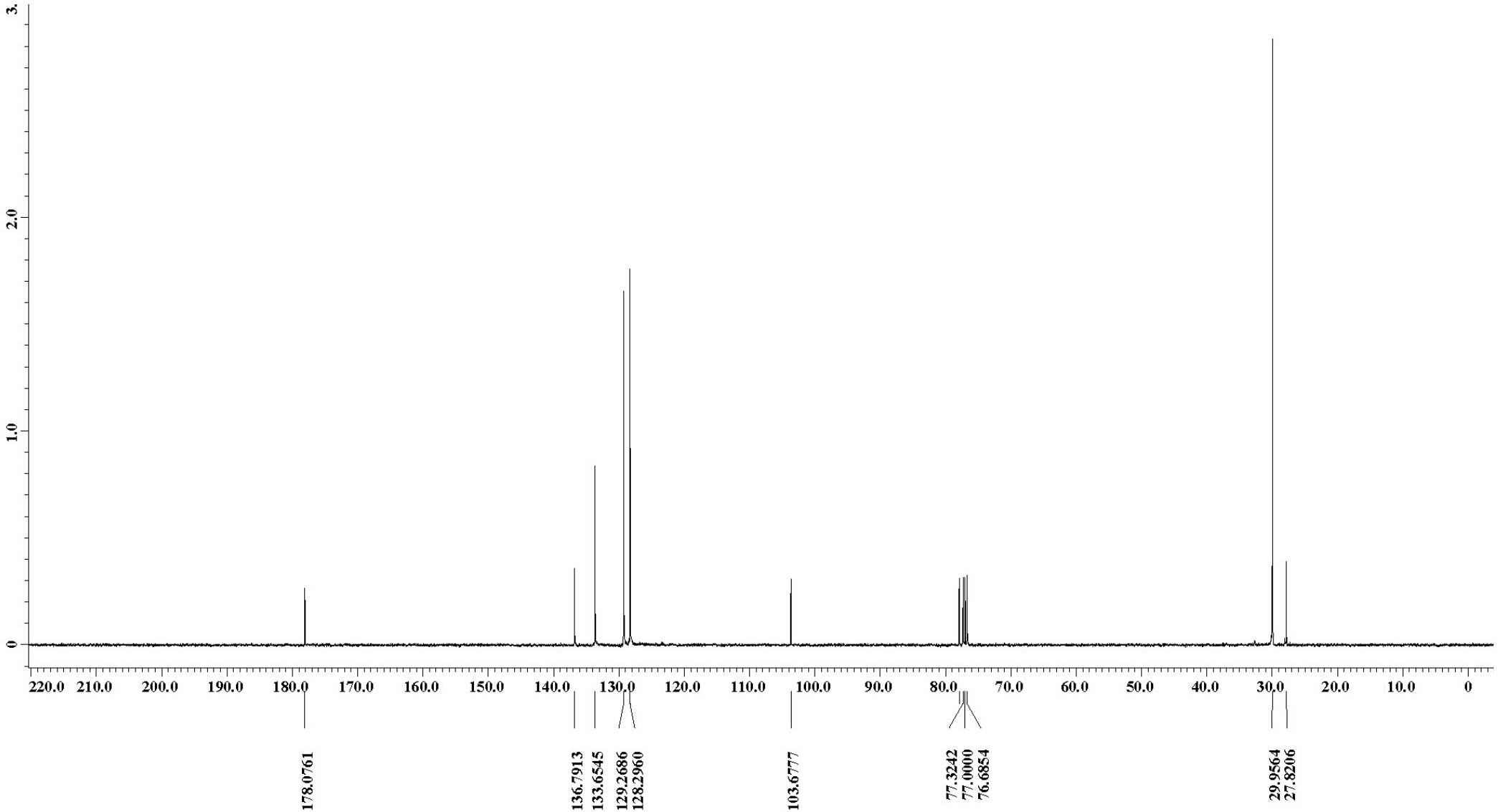


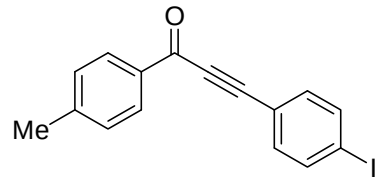


1t, ^{1}H NMR (400 MHz, $CDCl_3$)

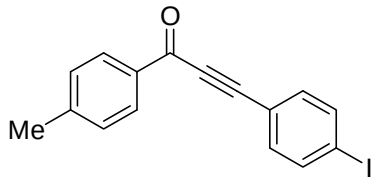


1t, ^{13}C NMR (100 MHz, $CDCl_3$)

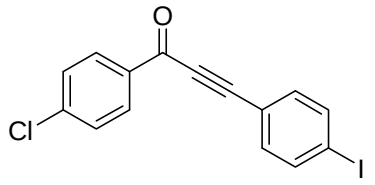




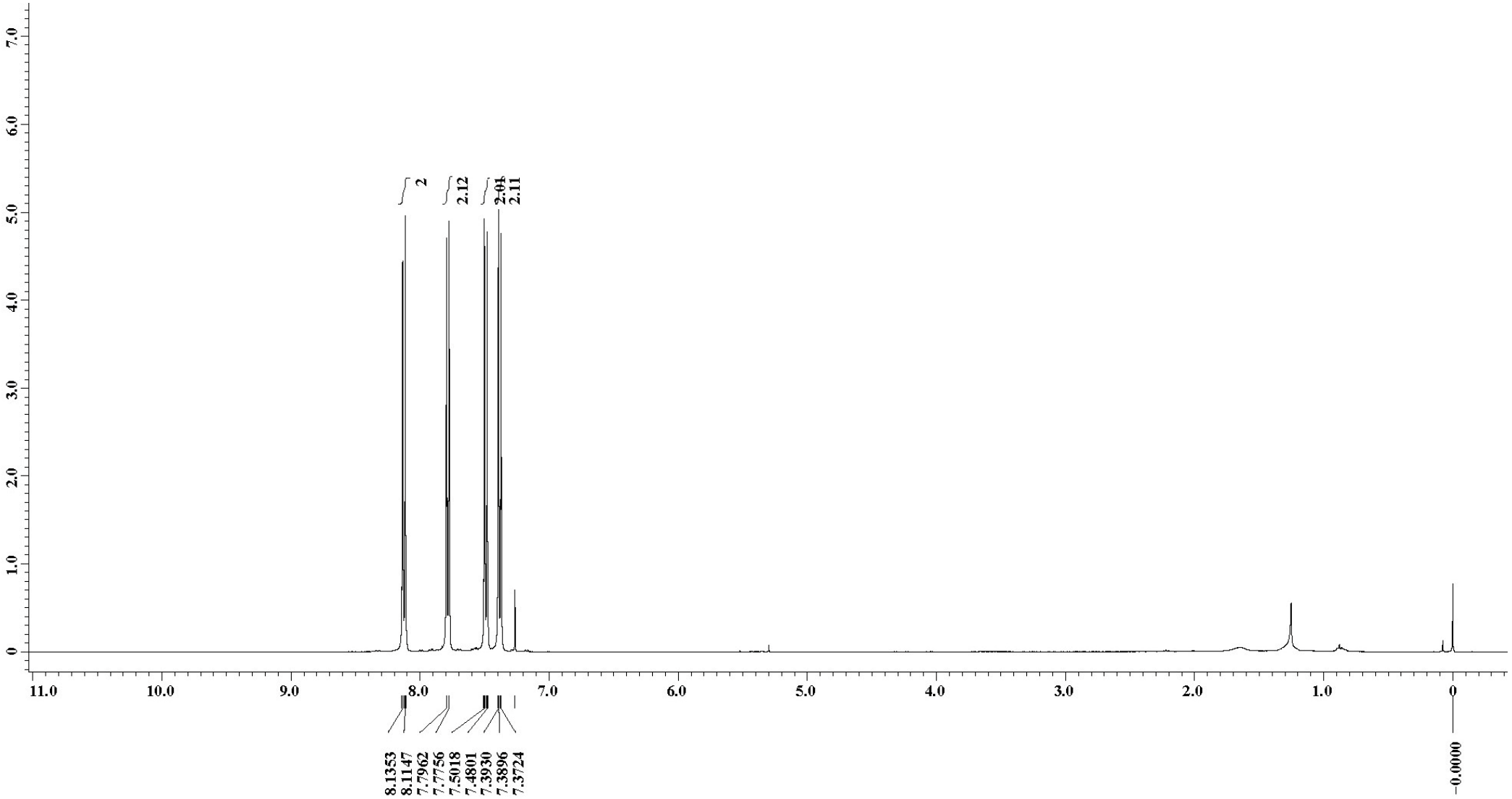
1u, ^{1}H NMR (400 MHz, $CDCl_3$)

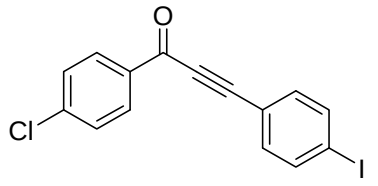


1u, ^{13}C NMR (100 MHz, $CDCl_3$)

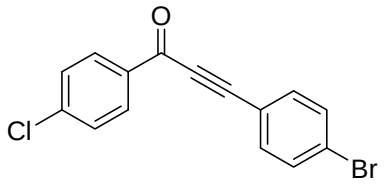


1v, ^{1}H NMR (400 MHz, $CDCl_3$)

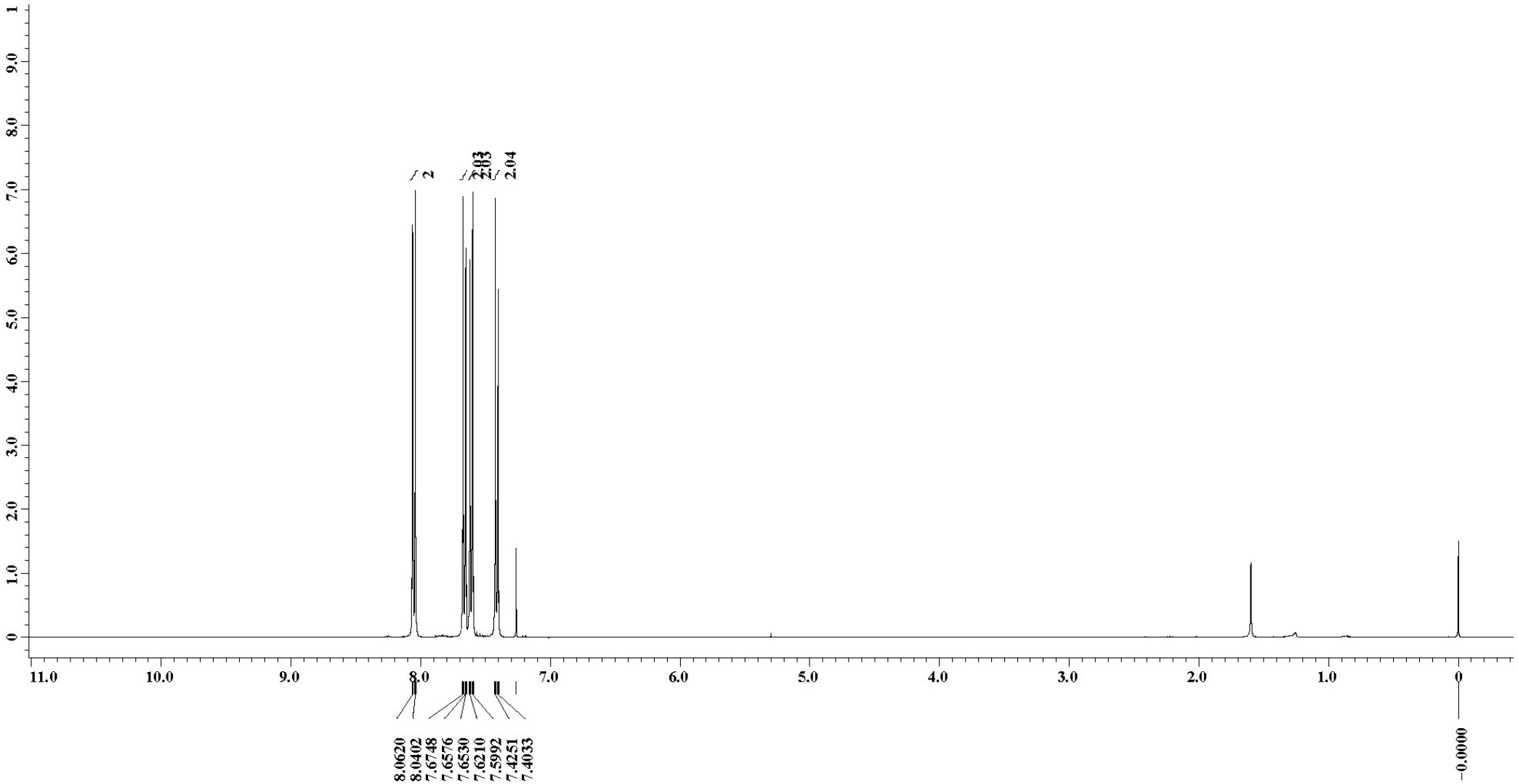


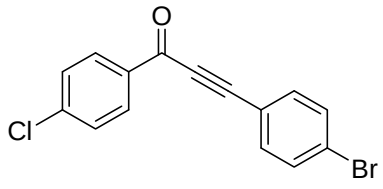


1v, ^{13}C NMR (100 MHz, $CDCl_3$)

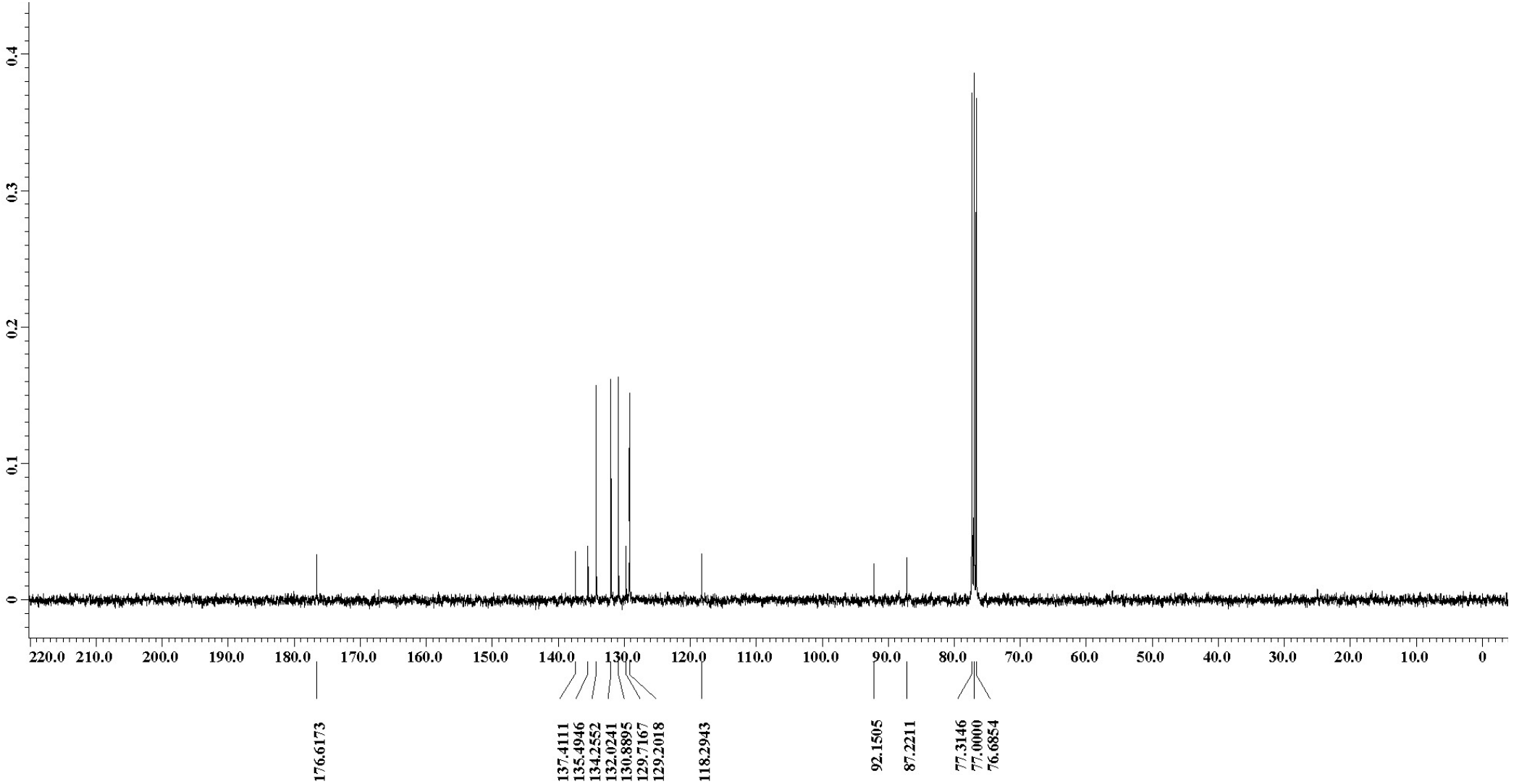


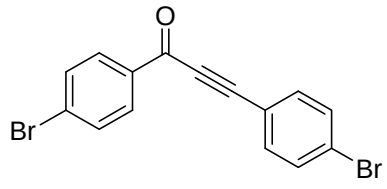
1w, ^{1}H NMR (400 MHz, $CDCl_3$)



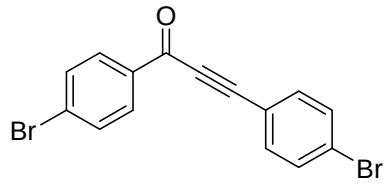


1w, ^{13}C NMR (100 MHz, $CDCl_3$)

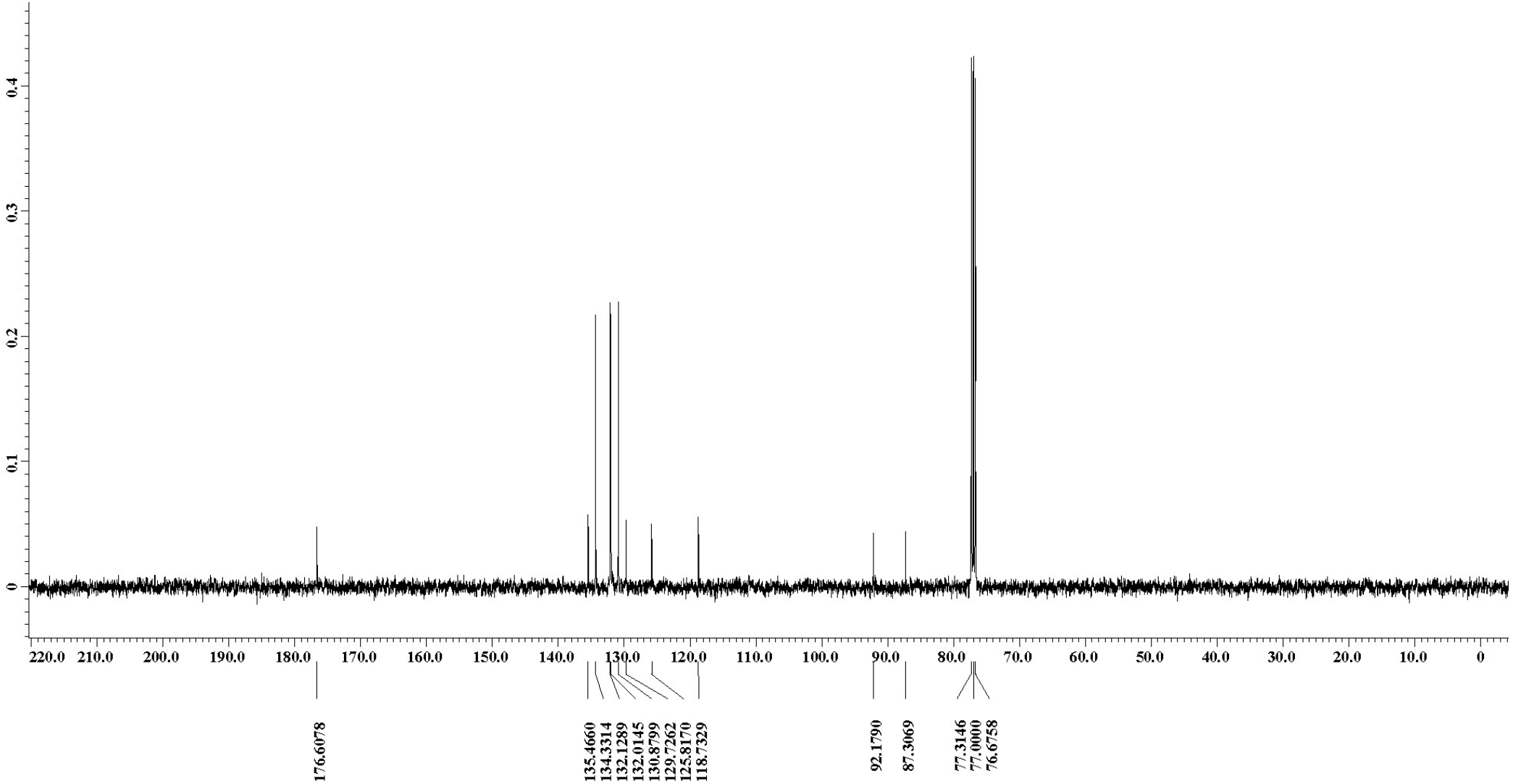


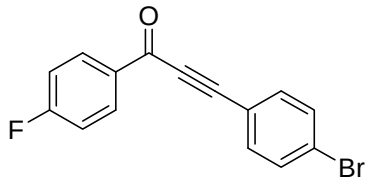


1x, ^{1}H NMR (400 MHz, $CDCl_3$)

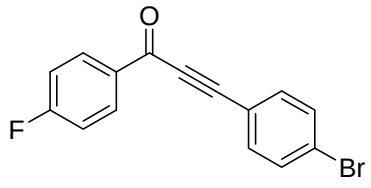


1x, ^{13}C NMR (100 MHz, $CDCl_3$)

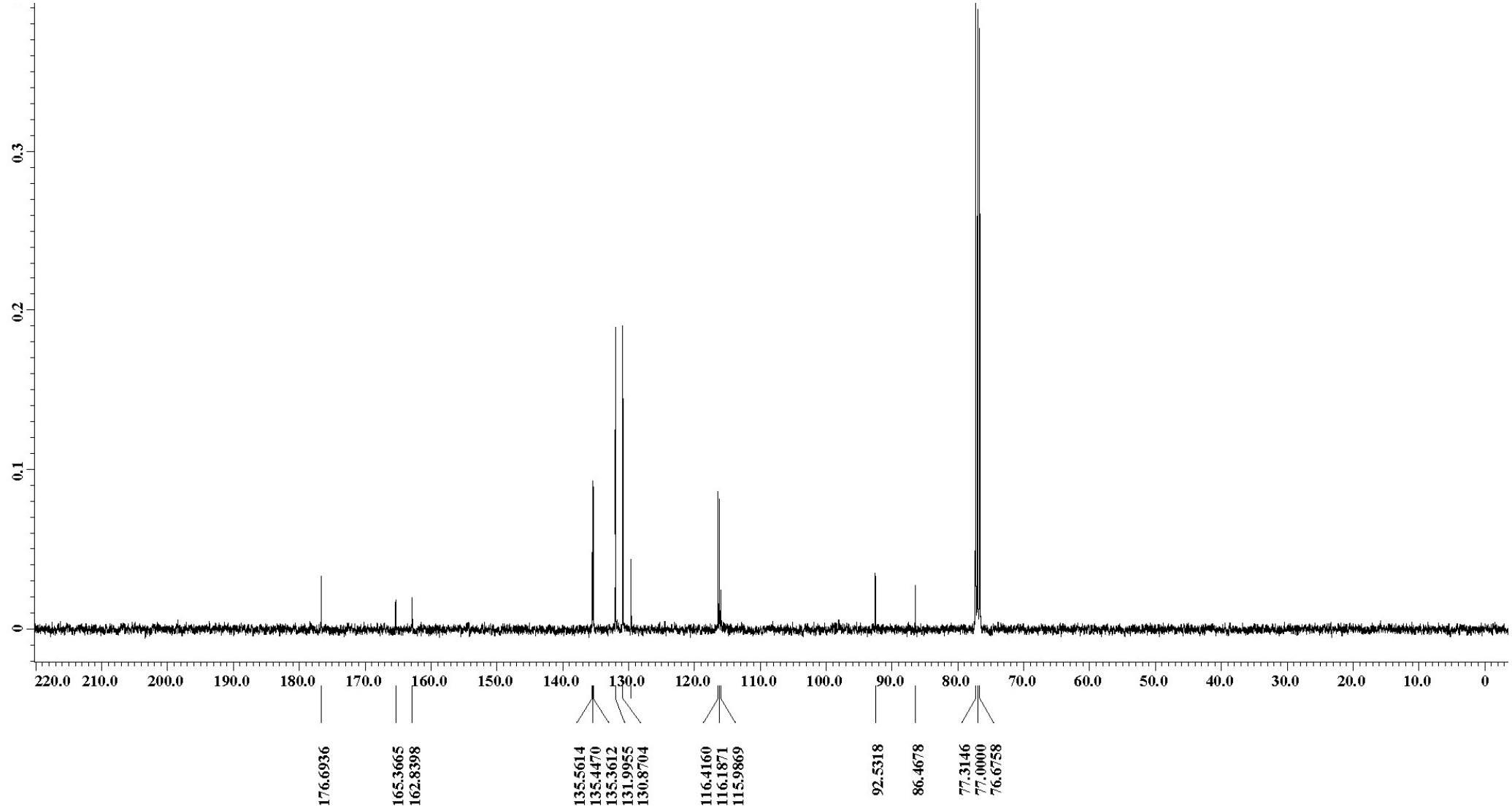


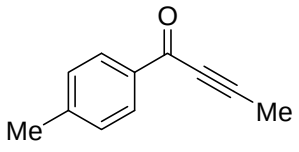


1y, ^{1}H NMR (400 MHz, $CDCl_3$)

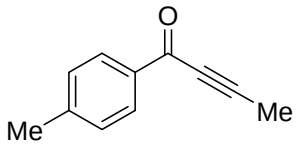


1y, ^{13}C NMR (100 MHz, $CDCl_3$)

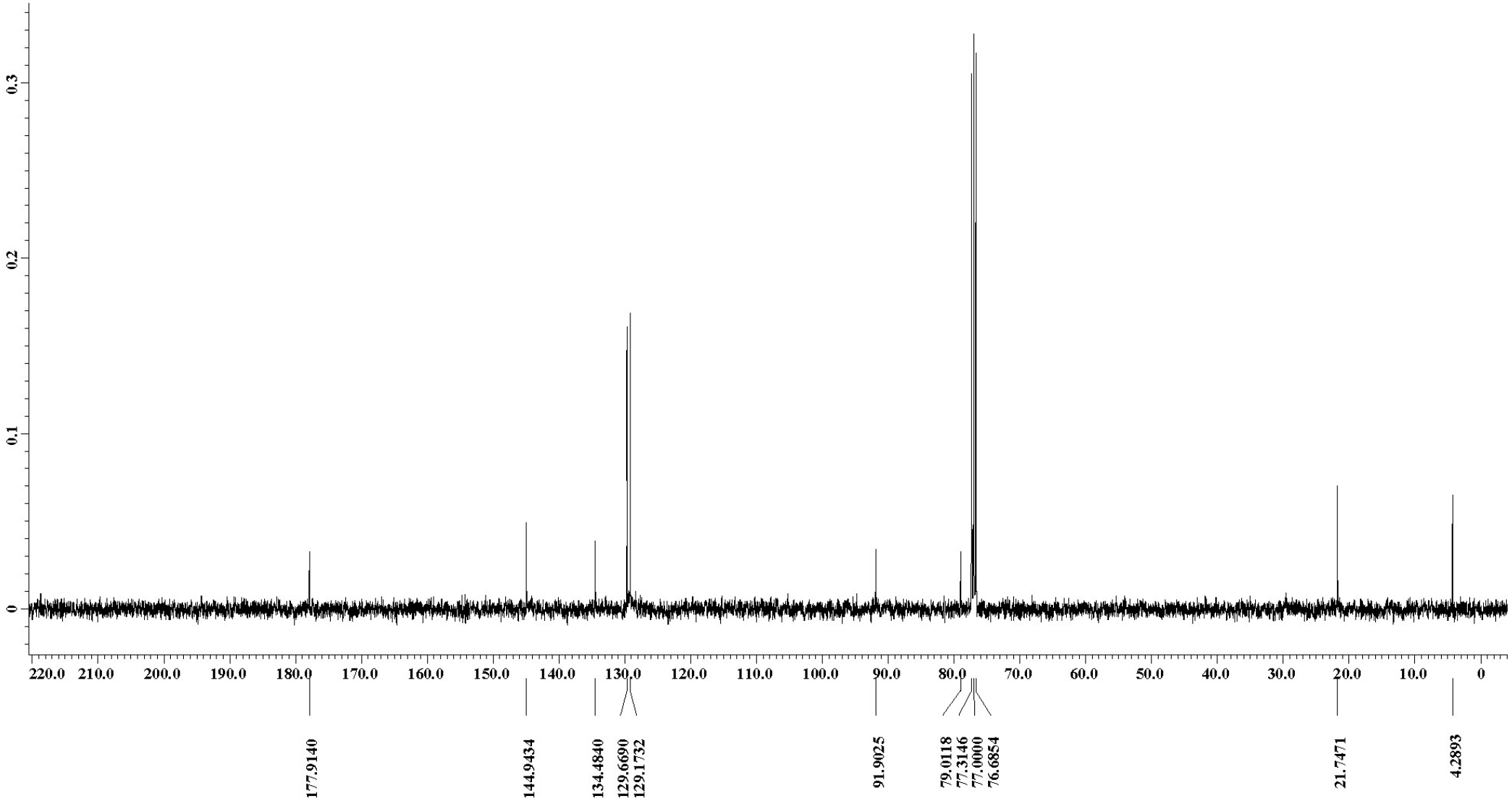


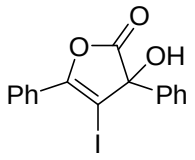


1z, ^{1}H NMR (400 MHz, $CDCl_3$)

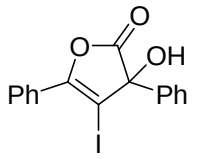


1z, ^{13}C NMR (100 MHz, $CDCl_3$)





8c, ^{1}H NMR (400 MHz, $CDCl_3$)



8c, ^{13}C NMR (100 MHz, $CDCl_3$)