Supporting Information

Synthesis of γ-Benzopyranone by TfOH-promoted Regioselective Cyclization of *o*-Alkynoylphenols

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General Techniques

All commercially available reagents were used as received. Dry THF and DCM (Kanto Chemical Co.) were obtained by passing commercially available pre-dried, oxygen-free formulations.

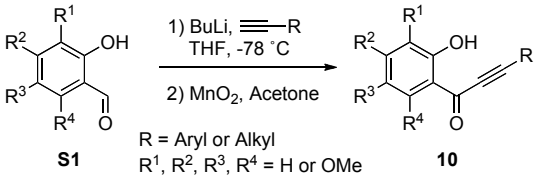
All reactions in solution-phase were monitored by thin-layer chromatography carried out on 0.2 mm E. Merck silica gel plates (60F-254) with UV light, and visualized with anisaldehyde, 10% ethanolic phosphomolybdic acid. Silica gel 60N (Kanto Chemical Co. 100~210 μm) was used for column chromatography.

^{1}H NMR spectra (400 MHz) and ^{13}C NMR spectra (100 MHz) were recorded on JEOL JNM-Al400 spectrometers in the indicated solvent. Chemical shifts (δ) are reported in units parts per million (ppm) relative to the signal for internal tetramethylsilane (0 ppm for ^{1}H) for solutions in $CDCl_3$. NMR spectral data are reported as follows: chloroform (7.26 ppm for ^{1}H) or chloroform-*d* (77.0 ppm for ^{13}C), when internal standard is not indicated. Multiplicities are reported by the following abbreviations: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), dd (double doublet), dt (double triplet), ddd (double double doublet), ddt (double double triplet), dddd (double double double doublet), brs (broad singlet), brd (broad doublet), *J* (coupling constants in Hertz).

Mass spectra and high-resolution mass spectra were measured on JEOL JMS-DX303 and MS-AX500 instruments. IR spectra were recorded on a Shimazu FTIR-8400. Only the strongest and/or structurally important absorption are reported as the IR data afforded in cm^{-1}. All Melting points were determined with Yazawa Micro Melting Point BY-2 and are not corrected.

Experimental procedure

General procedure for the synthesis of *o*-alkynoylphenol derivatives



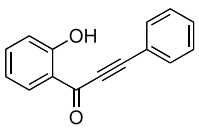
To a solution of phenylacetylene derivative (2.20 equiv.) in dry THF (4 ml/mmol) was added BuLi (1.60 M in hexane solution, 2.20 equiv.) dropwisely at –78 ˚C under argon. After being stirred at the same temperature for 1 h, to the reaction mixture was added a solution of aldehyde **S1** in dry THF (1 ml/mmol) dropwisely at –78 ˚C under argon and stirred continuously at the same temperature for 1 h. After being stirred at 0 ˚C for 30 min, the reaction mixture was quenched with saturated aqueous NH_4Cl at 0 ˚C. The organic layer was separated and the aqueous layer was extracted with ethyl acetate. The combined organic layers were washed with saturated aqueous $NaHCO_3$, brine, dried over $MgSO_4$ and filtered. The filtrate was concentrated in vacuo to afford the propargylalcohol. The crude alcohol was used for next reaction without further purification.

To a solution of crude alcohol in acetone (1 ml/mmol) was added MnO_2 (5.00 equiv.) at room temperature under argon. After being stirred at the same temperature, the reaction mixture was filtered thorough a pad of Celite® and the filtrate was concentrated in vacuo. The resulting residue was purified by column chromatography on silica gel to afford *o*-alkynoylphenol derivatives **10**.

2-(3-Phenylpropynoyl)phenol (10a)

Yield: 71%, a yellowish solid (Melting point 66–67 ˚C)

^{1}H NMR (400 MHz, $CDCl_3$) δ 11.8 (s, 1H), 8.14 (dd, 1H, *J* = 8.4, 1.2 Hz), 7.62 (d, 2H, *J* = 7.2 Hz), 7.50 (m, 4H), 6.98 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 182.2, 162.8, 137.1, 133.1, 133.0, 131.2, 128.7, 120.7, 119.6, 119.4, 118.1, 96.0, 85.7; FT-IR (Neat) 3582, 3062, 2206, 1623, 1594, 1203 cm^{-1}; HRFABMS calcd for $C_{15}H_{11}O_2$ (M^++H) 223.0756, found 223.0765.

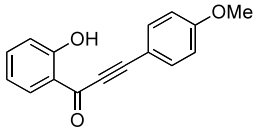


10a

2-[3-(4-Methoxyphenyl)propynoyl]phenol (10b)

Yield: 90%, a yellowish solid (Melting point 79–80˚C)

^{1}H NMR (400 MHz, $CDCl_3$) δ 11.8 (s, 1H), 8.12 (dd, 1H, *J* = 8.0, 1.6 Hz), 7.64 (d, 2H, *J* = 8.8 Hz), 7.51 (ddd, 1H, *J* = 8.0, 1.6 Hz), 6.97 (m, 4H), 3.87 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 182.1, 162.6, 161.9, 136.8, 135.2, 132.8, 120.8, 119.2, 118.0, 114.5, 111.4, 97.3, 85.8, 55.5; FT-IR (Neat) 3582, 2921, 2186, 1624, 1593, 1254 cm^{-1}; HREIMS calcd for $C_{16}H_{12}O_3$ 252.0786, found 252.0777.

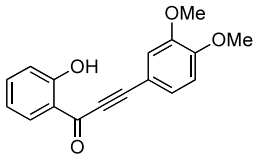


10b

2-[3-(3, 4-Dimethoxyphenyl)propynoyl]phenol (10c)

Yield: 77%, a yellowish solid (Melting point 119–120 ˚C)

^{1}H NMR (400 MHz, $CDCl_3$) δ 11.8 (s, 1H), 8.11 (dd, 1H, *J* = 8.4, 1.6 Hz), 7.52 (ddd, 1H, *J* = 8.4, 7.8, 1.6 Hz), 7.35 (dd, 1H, *J* = 8.4, 2.0 Hz), 7.15 (d, 1H, *J* = 2.0 Hz), 6.99 (m, 2H), 6.90 (d, 1H, *J* = 8.4 Hz), 3.95 (s, 3H), 3.93 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 182.1, 162.7, 152.0, 148.9, 136.9, 132.9, 127.7, 120.7, 119.3, 118.1, 115.3, 111.4, 111.1, 97.4, 85.4, 56.0, 55.9; FT-IR (Neat) 3584, 2954, 2188, 1623, 1592, 1249 cm^{-1}; HREIMS calced for $C_{17}H_{14}O_4$ 282.0892, found 282.0894.

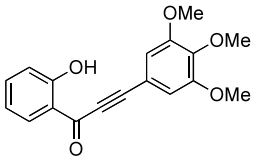


10c

2-[3-(3, 4, 5-Trimethoxyphenyl)propynoyl]phenol (10d)

Yield: 65%, a yellowish solid (Melting point 137–139 ˚C)

^{1}H NMR (400 MHz, $CDCl_3$) δ 11.8 (s, 1H), 8.11 (dd, 1H, *J* = 8.4, 1.6 Hz), 7.54 (ddd, 1H, *J* = 8.4, 7.6, 1.6 Hz), 7.01 (m, 2H), 6.93 (s, 2H), 3.92 (s, 3H), 3.91 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 182.1, 162.8, 153.2, 141.4, 137.1, 132.9, 120.7, 119.3, 118.1, 114.2, 110.5, 96.6, 85.2, 61.1, 56.3; FT-IR (Neat) 2997, 2193, 1628, 1575, 1246 cm^{-1}; HREIMS calced for $C_{18}H_{16}O_5$ 312.0998, found 312.0999.

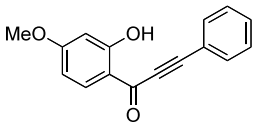


10d

5-Methoxy-2-(3-phenylpropynoyl)phenol (10e)

Yield: 90%, a yellowish solid (Melting point 105–106 ˚C)

^{1}H NMR (400 MHz, $CDCl_3$) δ 12.2 (s, 1H), 8.00 (d, 1H, *J* = 8.8 Hz), 7.67 (m, 2H), 7.45 (m, 3H), 6.53 (dd, 1H, *J* = 8.8, 2.4 Hz), 6.44 (d, 1H, *J* = 2.4 Hz), 3.87 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 180.3, 167.0, 165.7, 134.7, 133.0, 130.9, 128.7, 120.0, 115.3, 108.5, 100.6, 95.1, 85.6, 55.7; FT-IR (Neat) 3586, 2941, 2204, 1632, 1588, 1274 cm^{-1}; HREIMS calced for $C_{16}H_{12}O_3$ 252.0786, found 252.0773.

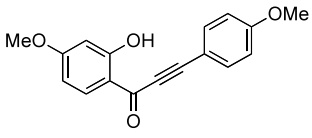


10e

5-Methoxy-2-[3-(4-methoxyphenyl)propynoyl]phenol (10f)

Yield: 54%, a yellowish solid (Melting point 122–123 ˚C)

^{1}H NMR (400 MHz, $CDCl_3$) δ 12.3 (s, 1H), 7.98 (d, 1H, *J* = 8.8 Hz), 7.62 (d, 2H, *J* = 8.8 Hz), 6.93 (d, 2H, *J* = 8.8 Hz), 6.52 (dd, 1H, *J* = 8.8, 2.4 Hz), 6.43 (d, 1H, *J* = 2.4 Hz), 3.78 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 180.2, 166.6, 165.5, 161.7, 134.9, 134.5, 115.2, 114.4, 111.6, 108.3, 100.5, 96.4, 85.5, 55.7, 55.5; FT-IR (Neat) 3582, 3395, 2916, 2197, 1628, 1605, 1264 cm^{-1}; HREIMS calced for $C_{17}H_{14}O_4$ 282.0892, found 282.0888.

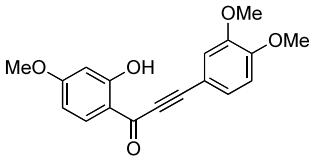


10f

5-Methoxy-2-[3-(3, 4-dimethoxyphenyl)propynoyl]phenol (10g)

Yield: 88%, a yellowish solid (Melting point 154–156 ˚C)

^{1}H NMR (400 MHz, $CDCl_3$) δ 12.3(s, 1H), 7.98 (d, 1H, *J* = 8.8 Hz), 7.32 (dd, 1H, *J* = 8.4, 2.0 Hz), 7.14 (d, 1H, *J* = 2.0 Hz), 6.89 (d, 1H, *J* = 8.4 Hz), 6.53 (dd, 1H, *J* = 8.8, 2.4 Hz), 6.43 (d, 1H, *J* = 2.4 Hz), 3.93 (s, 3H), 3.92 (s, 3H), 3.87 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 180.3, 166.8, 165.6, 151.8, 148.9, 134.5, 127.4, 115.3, 115.2, 111.8, 111.1, 108.4, 100.5, 96.4, 85.2, 56.0, 55.7; FT-IR (Neat) 3586, 2936, 2191, 1628, 1584, 1253 cm^{-1}; HREIMS calced for $C_{18}H_{16}O_5$ 312.0998, found 312.0989.

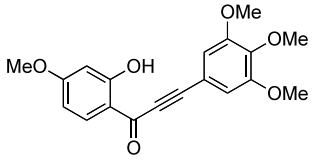


10g

5-Methoxy-2-[3-(3, 4, 5-trimethoxyphenyl)propynoyl]phenol (10h)

Yield: 92%, a yellowish solid (Melting point 113–115 ˚C)

^{1}H NMR (400 MHz, $CDCl_3$) δ 12.2 (s, 1H), 7.98 (d, 1H, *J* = 8.8 Hz), 6.90 (s, 2H), 6.54 (dd, 1H, *J* = 8.8, 2.4 Hz), 6.44 (d, 1H, *J* = 2.4 Hz), 3.91 (s, 3H), 3.90 (s, 6H), 3.87 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 180.1, 166.9, 165.7, 153.3, 141.2, 134.5, 115.2, 114.5, 110.4, 108.5, 100.5, 95.6, 85.0, 61.0, 56.3, 55.7; FT-IR (Neat) 3587, 2940, 2195, 1628, 1577, 1255 cm^{-1}; HREIMS calced for $C_{19}H_{18}O_6$ 342.1103, found 342.1110.

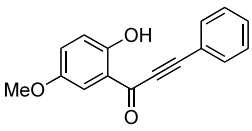


10h

4-Methoxy-2-[3-(4-methoxyphenyl)propynoyl]phenol (10i)

Yield: 86%, a yellowish solid (Melting point 88–89 ˚C)

^{1}H NMR (400 MHz, $CDCl_3$) δ 11.4 (s, 1H), 7.68 (m, 2H), 7.59 (d, 1H, *J* = 3.2 Hz), 7.52 (m, 1H), 7.45 (m, 2H), 7.17 (dd, 1H, *J* = 8.8, 3.2 Hz), 6.95 (d, 1H, *J* = 8.8 Hz), 3.86 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 181.7, 157.4, 152.0, 133.1, 131.2, 128.8, 125.4, 120.3, 119.7, 119.1, 114.7, 96.0, 85.7, 55.9; FT-IR (Neat) 3587, 3010, 2199, 1635, 1587, 1258 cm^{-1}; HREIMS calced for $C_{16}H_{12}O_3$ 252.2647, found 252.0778.

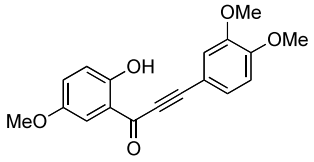


10i

4-Methoxy-2-[3-(3, 4-dimethoxyphenyl)propynoyl]phenol (10j)

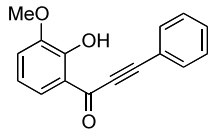
Yield: 64%, a yellowish solid (Melting point 129–131 ˚C)

^{1}H NMR (400 MHz, $CDCl_3$) δ 11.5(s, 1H), 7.59 (d, 1H, *J* = 3.2 Hz), 7.33 (dd, 1H, *J* = 8.4, 1.6 Hz), 7.16 (m, 2H), 6.95 (d, 1H, *J* = 9.6 Hz), 6.90 (d, 1H, *J* = 8.4 Hz), 3.94 (s, 3H), 3.92 (s, 3H), 3.87 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 181.7, 157.3, 152.1, 152.0, 148.9, 127.6, 125.0, 120.3, 119.1, 115.3,115.0, 111.5, 111.2, 97.4, 85.5, 56.1, 56.0, 55.9; FT-IR (Neat) 3584, 2937, 2191, 1581, 1251 cm^{-1}; HREIMS calced for $C_{18}H_{16}O_5$ 312.0998, found 312.0989.



10j

6-Methoxy-2-[3-(4-methoxyphenyl)propynoyl]phenol (10k)

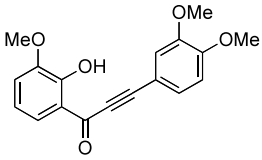


10k

Yield: 69%, a yellowish solid (Melting point 118-120°C)

^{1}H NMR (400 MHz, $CDCl_3$) δ 12.0 (s, 1H), 7.73 (dd, 1H, *J* = 8.4, 1.2 Hz), 7.70 (m, 2H), 7.48 (m, 3H), 7.11 (dd, 1H, *J* = 8.4, 1.2 Hz), 6.94 (d, 1H, *J* = 8.4 Hz), 3.93 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 182.4, 153.2, 148.6, 133.1, 131.1, 128.7, 124.0, 120.8, 119.6, 118.7, 117.8, 96.1, 85.9, 56.2; FT-IR (Neat) 3586, 3374, 2204, 1613, 1592, 1343, 1221 cm^{-1}; HREIMS calced for $C_{16}H_{12}O_3$ 252.0786, found 252.0780.

6-Methoxy-2-[3-(3, 4-dimethoxyphenyl)propynoyl]phenol (10l)

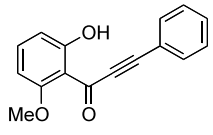


10l

Yield: 71%, a yellowish solid (Melting point 134–135 °C)

^{1}H NMR (400 MHz, $CDCl_3$) δ 12.1 (s, 1H), 7.72 (d, 1H, *J* = 7.6 Hz), 7.35 (dd, 1H, *J* = 8.4, 1.6 Hz), 7.16 (d, 1H, *J* = 1.6 Hz), 7.10 (d, 1H, *J* = 7.6 Hz), 6.94 (t, 1H, *J* = 7.6 Hz), 6.90 (d, 1H, *J* = 8.4 Hz), 3.94 (s, 3H), 3.93 (s, 3H), 3.92 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 182.4, 153.2, 152.1, 148.9, 148.7, 127.7, 124.0, 120.8, 118.6, 117.6, 115.3, 111.5, 111.1, 97.5, 85.8, 56.3, 56.1, 56.0; FT-IR (Neat) 3413, 2188, 1578, 1514, 1254 cm^{-1}; HREIMS calced for $C_{18}H_{16}O_5$, 312.0998, found 312.0999.

3-Methoxy-2-[3-(4-methoxyphenyl)propynoyl]phenol (10m)

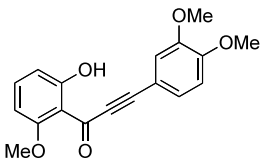


10m

Yield: 21%, a yellowish solid (Melting point 80–81 °C)

^{1}H NMR (400 MHz, $CDCl_3$) δ 12.8 (s, 1H), 7.64 (m, 2H), 7.42 (m, 4H), 6.57 (d, 1H, *J* = 8.4 Hz), 6.41 (d, 1H, *J* = 8.4 Hz), 3.97 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 180.0, 165.1, 161.3, 137.5, 133.1, 130.7, 128.6, 120.8, 112.0, 110.4, 101.4, 95.5, 90.0, 55.8; FT-IR (Neat) 3582, 2203, 1620, 1582, 1242 cm^{-1}; HREIMS calced for $C_{16}H_{12}O_3$ 252.0786, found 252.0786.

3-Methoxy-2-[3-(3, 4-dimethoxyphenyl)propynoyl]phenol (10n)



10n

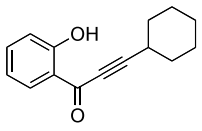
Yield: 32%, a yellowish solid (Melting point 123–125 °C)

^{1}H NMR (400 MHz, $CDCl_3$) δ 12.9 (s, 1H), 7.38 (t, 1H, *J* = 8.4 Hz), 7.27(dd, 1H, *J* = 8.4, 1.6 Hz), 7.13 (d, 1H, *J* = 1.6 Hz), 6.88 (d, 1H, *J* = 8.4 Hz), 6.57 (d, 1H, *J* = 8.4 Hz), 6.41 (d, 1H, *J* = 8.4 Hz), 3.98 (s, 3H), 3.93 (s, 3H), 3.91 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 179.3, 165.0, 161.1, 151.6, 148.8, 137.1, 127.4, 115.6, 112.7, 112.0, 111.1, 110.5, 101.4, 96.8, 89.8, 56.0, 55.8; FT-IR (Neat) 3586, 3389, 2936, 2188, 1618, 1582, 1245 cm^{-1}; HRMS [EI] calced for $C_{18}H_{16}O_5$ 312.0998, found 312.0999.

2-(3-Cyclohexylpropynoyl)phenol (10o)

Yield: 93%, colorless oil

^{1}H NMR (400 MHz, $CDCl_3$) δ 11.8 (s, 1H), 8.01 (dd, 1H, *J* = 8.0, 1.6 Hz), 7.49 (ddd, 1H, *J* = 7.8, 1.6 Hz), 6.97 (d, 1H, *J* = 7.8 Hz), 6.94 (t, 1H, *J* = 8.0 Hz), 2.73 (m, 1H), 1.41-1.95 (m, 10H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 182.8, 162.7, 136.8, 133.2, 119.2, 118.0, 11.8, 108.2, 103.5, 31.6, 29.5, 25.6, 24.7; FT-IR (Neat) 2933, 2855, 2210, 1624, 1598, 1243 cm^{-1}; HREIMS calced for $C_{15}H_{16}O_2$ 228.1150, found 228.1154.

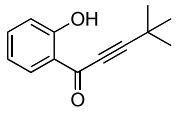


10o

2-(4,4-Dimethylpent-2-ynoyl)phenol (10p)

Yield: 92%, colorless oil

^{1}H NMR (400 MHz, $CDCl_3$) δ 11.8 (s, 1H), 7.97 (dd, 1H, *J* = 8.2, 1.6 Hz), 7.49 (ddd, 1H, *J* = 8.2, 7.8, 1.6 Hz), 6.96 (d, 1H, *J* = 7.8, 0.8 Hz), 6.94 (ddd, 1H, *J* = 8.2, 0.8 Hz), 1.39 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 182.7, 162.7, 136.8, 133.1, 120.7, 119.2, 117.9, 106.9, 30.7, 30.0, 28.1; FT-IR (Neat) 3347, 2216, 1625, 1600, 1256 cm^{-1}; HREIMS calced for $C_{13}H_{14}O_2$ 202.0994, found 202.0996.

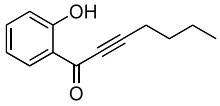


10p

2-(Hept-2-ynoyl)phenol (10q)

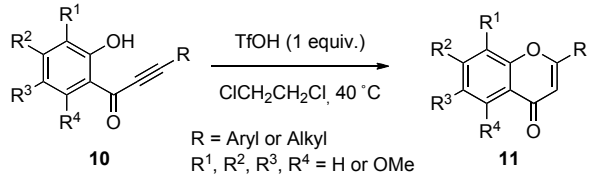
Yield: 67%, colorless oil

^{1}H NMR (400 MHz, $CDCl_3$) δ 11.7 (s, 1H), 8.01 (dd, 1H, *J* = 7.6, 2.0 Hz), 7.49 (ddd, 1H, *J* = 8.4, 7.6, 2.0 Hz), 6.96 (d, 1H, *J* = 8.4 Hz), 6.94 (t, 1H, *J* = 7.6 Hz), 2.53 (t, 3H, *J* = 7.4 Hz), 1.67 (m, 2H), 1.52 (m, 2H), 0.97 (t, 3H, *J* = 7.4 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 182.6, 162.7, 136.8, 133.1, 120.7, 119.2, 118.0, 99.9, 78.5, 29.7, 22.1, 19.0, 13.5; FT-IR (Neat) 3413, 2232, 2212, 1626, 1598, 1245 cm^{-1}; HREIMS calced for $C_{13}H_{14}O_2$ 202.0994, found 202.0996.



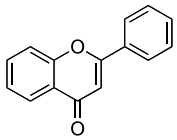
10q

General Procedure for the Synthesis of Flavonoid Derivatives by TfOH-promoted cyclization



To a solution of *o*-alkynoylphenol **10** in 1,2-dichloroethane (4 ml/mmol) was added TfOH (1.00 equiv.) at 0 ˚C under argon. After being stirred at 40 ˚C, the reaction mixture was quenched with saturated aqueous $NaHCO_3$ at 0 ˚C. The organic layer was separated and the aqueous layer was extracted once with ethyl acetate. The combined organic layers were washed with brine, dried over $MgSO_4$ and filtered. The filtrate was concentrated in vacuo, and the resulting residue was purified by column chromatography on silica gel to afford flavonoid derivative **11**.

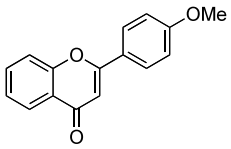
2-Phenyl-4H-chromen-4-one (11a)


11a

Yield: 80%, white solid (Melting point 95–97 ˚C)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (dd, 1H, *J* = 8.0, 1.6 Hz), 7.94 (m, 2H), 7.71 (ddd, 1H, *J* = 8.0, 1.6 Hz), 7.55 (m, 4H), 7.43 (ddd, 1H, *J* = 8.0, 1.6 Hz), 6.84 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.9, 162.9, 155.8, 133.4, 131.3, 128.7, 125.9, 125.2, 124.8, 123.6, 117.8, 107.1; FT-IR (Neat) 3589, 3440, 1670, 1604, 1254 cm^{-1}; HREIMS calced for $C_{15}H_{10}O_2$ 222.0681, found 222.0679.

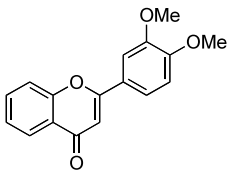
2-(4-Methoxyphenyl)-4H-chromen-4-one (11b)


11b

Yield: 90%, white solid (Melting point 134–136 ˚C)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.21 (dd, 1H, *J* = 8.0, 1.6 Hz), 7.87 (d, 2H, *J* = 8.8 Hz), 7.67 (ddd, 1H, *J* = 8.0, 1.6 Hz), 7.53 (d, 1H, *J* = 8.0 Hz), 7.40 (t, 1H, *J* = 8.0 Hz), 7.01 (d, 2H, *J* = 8.8 Hz), 6.74 (s, 1H, e), 3.89 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.2, 163.3, 162.3, 156.0, 133.5, 127.9, 125.9, 125.0, 124.0, 123.9, 117.8, 114.4, 106.1, 55.5; FT-IR (Neat) 3582, 3420, 2925, 1648,1608, 1268 cm^{-1}; HREIMS calced for $C_{16}H_{12}O_3$ 252.0786, found 252.0782.

2-(3,4-Dimethoxyphenyl)-4H-chromen-4-one (11c)


11c

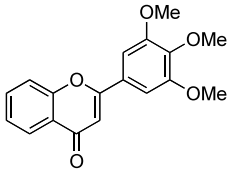
Yield: 90%, white solid (Melting point 156–157 ˚C)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.22 (dd, 1H, *J* = 8.4, 1.2 Hz), 7.68 (ddd, 1H, *J* = 8.4, 1.2 Hz), 7.55 (d, 2H, *J* = 8.4 Hz), 7.41 (m, 2 H), 6.98 (d, 1H, *J* = 8.4 Hz), 6.75 (s, 1H), 3.98 (s, 3H), 3.96 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.2, 163.3, 156.1, 152.1, 149.3, 133.5, 125.6, 125.1, 124.2, 123.9, 120.0, 117.8, 111.2, 108.9, 106.4, 56.1, 56.0; FT-IR (Neat) 3582, 342, 1641, 1602, 1269 cm^{-1}; HREIMS calced for $C_{17}H_{14}O_4$ 282.0892, found 282.0895.

2-(3,4,5-Trimethoxyphenyl)-4H-chromen-4-one (11d)

Yield: 96%, white solid (Melting point 176–178 °C)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.24 (dd, 1H, *J* = 8.0, 1.2 Hz), 7.71 (dd, 1H, *J* = 8.0, 1.2 Hz), 7.59 (dd, 1H, *J* = 8.0, 1.6 Hz), 7.43 (ddd, 1H, *J* = 8.0, 1.6 Hz), 7.15 (s, 2H), 6.78 (s, 1H), 3.97 (s, 6H), 3.94 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.3, 163.3, 156.2, 153.6, 141.3, 133.7, 127.0, 125.7, 125.3, 123.9, 118.0, 107.4, 103.8, 61.0, 56.4; FT-IR (Neat) 2939, 1653, 1604, 1244 cm^{-1}; HREIMS calced for $C_{18}H_{16}O_5$ 312.0998, found 312.0993.

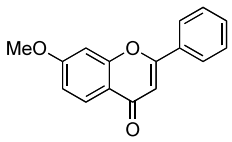


11d

7-Methoxy-2-phenyl-4H-chromen-4-one (11e)

Yield: 60%, white solid (Melting point 105–106 °C)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 (d, 1H, *J* = 8.8 Hz), 7.90 (m, 2H), 7.51 (m, 3H), 6.98 (m, 2H), 6.76 (s, 1H), 3.93 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.8, 164.2, 163.0, 158.0, 131.8, 131.4, 129.0, 127.0, 126.1, 117.8, 114.4, 107.5, 100.4, 55.8; FT-IR (Neat) 3447, 3065, 1642, 1607,1238 cm^{-1}; HREIMS calced for $C_{16}H_{12}O_3$ 252.0786, found 252.0858.

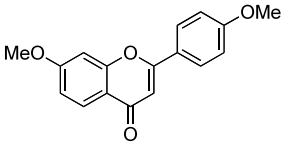


11e

7-Methoxy-2-(4-methoxyphenyl)-4H-chromen-4-one (11f)

Yield: 61%, white solid (Melting point 149–150 °C)

^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.12 (d, 1H, *J* = 8.8 Hz), 7.85 (d, 2H, *J* = 9.2 Hz), 7.01 (d, 2H, *J* = 9.2 Hz), 6.98 (m, 2H), 6.68 (s, 1H), 3.93 (s, 3H), 3.89 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.8, 164.0, 163.0, 162.2, 157.9, 127.8, 127.0, 124.1, 117.8, 114.4, 114.1, 106.1, 100.4, 55.7, 55.5; FT-IR (Neat) 3361, 2925, 1626, 1607, 1258, 1180, 1165 cm^{-1}; HREIMS calced for $C_{17}H_{14}O_4$ 282.0892, found 282.0883.

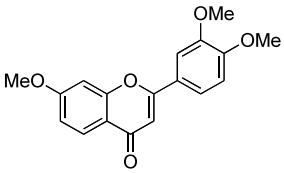


11f

7-Methoxy-2-(3,4-dimethoxyphenyl)-4H-chromen-4-one (11g)

Yield: 82%, white solid (Melting point 173–174 °C)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (d, 1H, *J* = 8.8 Hz), 7.54 (dd, 1H, *J* = 8.4, 2.0 Hz), 7.36 (d, 1H, *J* = 2.0 Hz), 6.97 (m, 3H), 6.69 (s, 1H), 3.98 (s, 3H), 3.96 (s, 3H), 3.94 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.7, 163.9, 162.9, 157.7, 151.8, 149.1, 126.9, 124.3, 119.7, 117.7, 114.1, 111.1, 108.7, 106.3, 100.4, 56.1, 56.0, 55.9; FT-IR (Neat) 3422, 2931, 1628, 1602, 1269 cm^{-1}; HREIMS calced for $C_{18}H_{16}O_5$ 312.0998, found 312.0998.

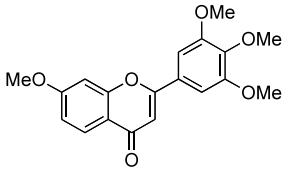


11g

7-Methoxy-2-(3,4,5-triimethoxyphenyl)-4H-chromen-4-one (11h)

Yield: 73%, white solid (Melting point 192–196 ˚C)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (d, 1H, *J* = 8.8 Hz), 7.12 (s, 2H), 6.99 (m, 2H), 6.71 (s, 1H), 3.97 (s, 6H), 3.95 (s, 3H), 3.94 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.7, 164.2, 162.9, 157.9, 153.5, 141.1, 127.1, 127.0, 117.8, 114.4, 107.3, 103.7, 100.5, 61.0, 56.3, 55.8; FT-IR (Neat) 3385, 2934, 1626, 1601, 1254, 1126 cm^{-1}; HREIMS calced for $C_{19}H_{18}O_6$ 324.1103, found 324.1103.

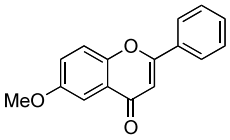


11h

6-Methoxy-2-phenyl-4H-chromen-4-one (11i)

Yield: 91%, white solid (Melting point 165-167 ˚C)

^{1}H NMR (400 MHz, $CDCl_3$) d 7.91 (m, 2H), 7.55 (d, 1H, *J* = 3.2 Hz), 7.52 (m, 4H), 7.28 (dd, 1H, *J* = 9.2, 3.2 Hz), 6.81 (s, 1H), 3.90 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.2, 163.1, 156.9, 151.3, 131.8, 131.4, 129.0, 126.2, 124.5, 123.7, 119.4, 106.8, 104.8, 55.9; FT-IR (Neat) 3001, 1639, 1606, 1489, 1361 cm^{-1}; HREIMS calced for $C_{16}H_{12}O_3$ 252.0786, found 252.0767.

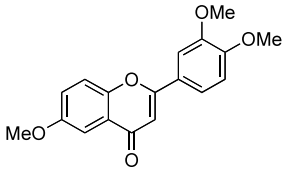


11i

6-Methoxy-2-(3,4-dimethoxyphenyl)-4H-chromen-4-one (11j)

Yield: 92%, white solid (Melting point 205–207 ˚C)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 (d, 1H, *J* = 3.2 Hz, a), 7.56 (dd, 1H, *J* = 8.4, 2.0 Hz), 7.51 (d, 1H, *J* = 9.2 Hz), 7.39 (d, 1H, *J* = 2.0 Hz), 7.29 (dd, 1H, *J* = 9.2, 3.2 Hz), 6.99 (d, 1H, *J* = 8.4 Hz), 6.76 (s, 1H), 3.99 (s, 3H), 3.97 (s, 3H), 3.92 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.0, 163.0, 156.8, 151.8, 150.8, 149.1, 124.4, 124.2, 123.4, 119.8, 119.3, 111.1, 108.7, 105.7, 104.8, 56.0, 55.9; FT-IR (Neat) 2922, 1643, 1616, 1263 cm^{-1}; HREIMS calced for $C_{18}H_{16}O_5$ 312.0998, found 312.0971.

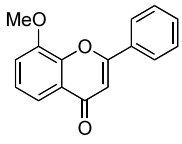


11j

8-Methoxy-2-phenyl-4H-chromen-4-one (11k)

Yield: 91%, white solid (Melting point 202–203 ˚C)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (m, 2H), 7.77 (dd, 1H, *J* = 8.0, 1.2 Hz), 7.52 (m, 3H), 7.34 (t, 1H, *J* = 8.0 Hz), 7.18 (dd, 1H, *J* = 8.0, 1.2 Hz), 6.83 (s, 1H), 4.02 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.4, 162.9, 149.1, 146.6, 131.8, 131.5, 129.0, 126.3, 124.9, 124.6, 116.4, 114.4, 107.3, 56.3; FT-IR (Neat) 3586, 3395, 3065, 1638, 1582, 1573, 1273 cm^{-1}; HREIMS calced for $C_{16}H_{12}O_3$ 252.0786, found 252.0780.

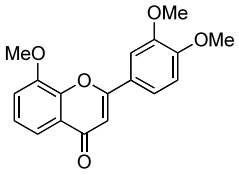


11k

8-Methoxy-2-(3,4-dimethoxyphenyl)-4H-chromen-4-one (11l)

Yield: 46%, white solid (Melting point 148–149 °C)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (dd, 1H, *J* = 8.4, 1.6 Hz), 7.62 (dd, 1H, *J* = 8.8, 1.9 Hz), 7.45 (d, 1H, *J* = 1.9 Hz), 7.31 (t, 1H, *J* = 8.4 Hz), 7.18 (dd, 1H, *J* = 8.4, 1.6 Hz), 6.99 (d, 1H, *J* = 8.8 Hz), 6.76 (s, 1H), 4.02 (s, 3H), 3.99 (s, 3H), 3.96 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.3, 162.9, 152.0, 149.2, 149.0, 146.5, 124.9, 124.7, 124.3, 120.0, 116.0, 114.3, 111.1, 108.9, 106.2, 56.4, 56.0, 56.0; FT-IR (Neat) 3397, 1637, 1516, 1269 cm^{-1}; HREIMS calced for $C_{18}H_{16}O_5$ 312.0998, found 312.0994.

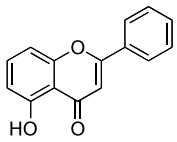


11l

5-Hydroxy-2-phenyl-4H-chromen-4-one (11m)

Yield: 71%, yellowish solid (Melting point 160–162°C)

^{1}H NMR (400 MHz, $CDCl_3$) δ 12.6 (s, 1H), 7.92 (dd, 2H, *J* = 6.4, 1.6 Hz), 7.54 (m, 4H), 7.00 (dd, 1H, *J* = 8.2, 0.6 Hz), 6.81 (dd, 1H, *J* = 8.2, 0.6 Hz), 6.74 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 183.6, 164.6, 160.8, 156.5, 135.4, 132.0, 131.2, 129.1, 126.4, 111.5, 110.9, 107.0, 106.1; FT-IR (Neat) 3586, 3384, 2920, 1656, 1613, 1257cm^{-1}; HREIMS calced for $C_{15}H_{10}O_3$ 238.0630, found 238.0617.

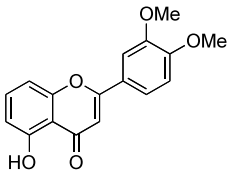


11m

5-Hydroxy-2-(3,4-dimethoxyphenyl)-4H-chromen-4-one (11n)

Yield: 90% yellowish solid (Melting point 169–171 °C)

^{1}H NMR (400 MHz, $CDCl_3$) δ 12.6 (s, 1H), 7.53 (m, 2H), 7.36 (s, 1H), 6.98 (d, 2H, *J* = 8.4 Hz), 6.80 (d, 1H, *J* = 8.4 Hz), 6.65 (s, 1H), 3.98 (s, 3H), 3.97 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 183.3, 164.4, 160.7, 156.3, 152.4, 149.3, 135.1, 123.6, 120.2, 111.3, 111.2, 110.7, 108.9, 106.9, 104.8, 56.1; FT-IR (Neat) 3589, 3397, 2931, 1653, 1594, 1253 cm^{-1}; HREIMS calced for $C_{17}H_{14}O_5$ 298.0841, found 298.0840.

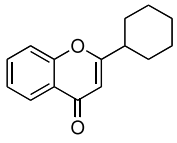


11n

2-Cyclohexyl-chromen-4H-one (11o)

Yield: 73%, colorless oil

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.18 (d, 1H, *J* = 8.0 Hz), 7.64 (t, 1H, *J* = 8.0 Hz), 7.43 (d, 1H, *J* = 8.0 Hz), 7.37 (t, 1H, *J* = 8.0 Hz), 6.17 (s, 1H), 2.53 (tt, 1H, *J* = 11.4, 3.4 Hz), 1.26-2.06 (m, 10H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.6, 173.4, 156.5, 133.3, 125.6, 124.8, 123.8, 117.8, 107.9, 42.8, 30.4, 25.8, 25.7; FT-IR (Neat) 3477, 2930, 2855, 1655, 1466, 1221 cm^{-1}; HREIMS calced for $C_{15}H_{16}O_2$ 228.1150, found 228.1148.

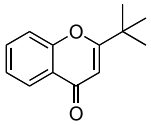


11o

2-*tert*-Butyl-chromen-4H-one (11p)

Yield: 67%, colorless oil

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.18 (dd, 1H, *J* = 8.0, 1.6 Hz), 7.65 (dt, 1H, *J* = 8.0, 1.6 Hz), 7.42 (d, 1H, *J* = 8.0 Hz), 7.37 (d, 1H, *J* = 8.0 Hz), 6.29 (s, 1H), 1.39 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.9, 176.0, 156.4, 133.4, 125.5, 124.8, 123.4, 117.8, 106.7, 36.4, 27.8; FT-IR (Neat) 3389, 1654, 1466, 1360, 1134 cm^{-1}; HREIMS calced for $C_{13}H_{14}O_2$ 202.0994, found 202.0987.

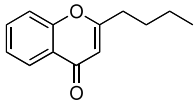


11p

2-Butyl-chromen-4H-one (11q)

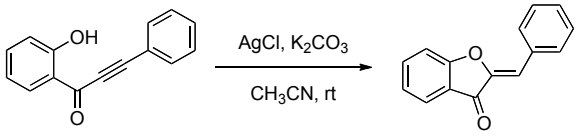
Yield: 75%, colorless oil

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.18 (dd, 1H, *J* = 8.2, 1.4 Hz), 7.62 (dt, 1H, *J* = 8.2, 1.4 Hz), 7.42 (d, 1H, *J* = 8.2 Hz), 7.37 (t, 1H, *J* = 8.2 Hz), 6.18 (s, 1H), 2.62 (t, 2H, *J* = 7.2 Hz), 1.73 (m, 2H), 1.44 (m, 2H), 0.97 (t, 3H, *J* = 7.2 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.3, 169.8, 156.5, 133.3, 125.6, 124.8, 123.7, 117.8, 109.8, 34.0, 28.8, 22.1, 13.7; FT-IR (Neat) 3585, 1656, 1465, 1383, 1221 cm^{-1}; HREIMS calced for $C_{13}H_{14}O_2$ 202.0994, found 202.0987.



11q

2-Benzylidene-1-benzofuran-3-one (Aurene) (12a)

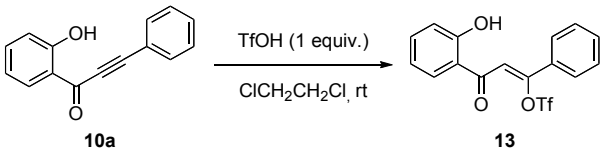


10a **12a**

To a suspention of *o*-alkynoylphenol (1 equiv.) and K_2CO_3 (1.00 equiv.) in acetonitrile (4 ml/mmol) was added AgCl (0.500 equiv.) at 0 ˚C under argon. After being stirred at room temperature, the reaction mixture was filtered thorough a pad of Celite® and the filtrate was concentrated in vacuo. The resulting residue was purified by column chromatography on silica gel (eluted with hexane/ethyl acetate = 9/1) to afford aurone **12a** (90% yield) as a colorless solid (Melting point 98–99 ˚C).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (d, 2H, *J* = 6.8 Hz), 7.81 (dd, 1H, *J* = 7.6, 0.8 Hz), 7.66 (dt, 1H, *J* = 7.6, 0.8 Hz), 7.43 (m, 3H), 7.33 (d, 1H, *J* = 7.6 Hz), 7.22 (t, 2H, *J* = 7.6 Hz), 6.90 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 184.7, 166.0, 146.8, 136.8, 132.2, 131.5, 129.8, 128.8, 124.6, 123.4, 121.6, 113.0, 112.9; FT-IR (Neat) 3480, 2810, 1709, 1654, 1597 cm^{-1}; HRFABMS calcd for $C_{15}H_{12}O_2$ (M^++H) 223.0756, found 223.0758.

2-(3-Phenyl-3-trifluoromethanesulfonylpropenoyl)phenol (13)



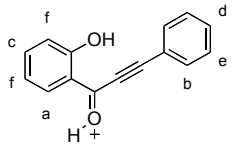
To a solution of *o*-alkynoylphenol **10a** (100 mg, 0.45 mmol) in 1,2-dichloroethane (1.8 mL, 4 ml/mmol) was added TfOH (1.00 equiv., 40 μL, 0.45 mmol) at 0 ˚C under argon. After being stirred at room temperature, the reaction mixture was quenched with saturated aqueous $NaHCO_3$ at 0 ˚C. The organic layer was separated and the aqueous layer was extracted once with ethyl acetate. The combined organic layers were washed with brine, dried over $MgSO_4$ and filtered. The filtrate was concentrated in vacuo, and then the resulting residue was purified by column chromatography on silica gel (eluted with hexane/ethyl acetate = 9/1) to afford **11a** (23 mg, 23%) and vinyl triflate **13** (69 mg, 41%) as a yellowish oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 12.1 (s, 1H), 7.75 (dd, 1H, *J* = 8.4, 1.2 Hz), 7.68 (m, 2H), 7.53 (m, 4H), 7.18 (s, 1H), 7.04 (dd, 1H, *J* = 8.4, 0.8 Hz), 6.94 (dt, 1H, *J* = 8.0, 0.8 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 191.8, 163.4, 154.5, 137.2, 132.1, 129.9, 129.2, 126.7, 119.9, 119.2, 118.9, 118.2 (q, *J* = 322 Hz) 113.8; FT-IR (Neat) 3063, 1651, 1624, 1226 cm^{-1}; HRFABMS calcd for $C_{16}H_{12}F_3O_5S$ (M^++H) 373.0358, found 373.0345.

Assignments of the signals in Figure 2

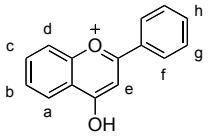
Spectrum B

8.21 (dd, 1H, *J* = 8.1, 1.2 Hz, a), 7.83 (dd, 2H, *J* = 8.0, 1.2 Hz, b)
7.76 (ddd, 1H, *J* = 7.6, 1.2 Hz, c), 7.65 (dd, 1H, *J* = 8.0, 1.2 Hz, d)
7.53 (t, 2H, *J* = 8.0 Hz, e), 7.16 (m, 2H, f)



Spectrum D

8.44 (dd, 1H, *J* = 8.2, 1.6 Hz, a), 8.29(d, 2H, *J* = 8.0 Hz, f)
8.21 (ddd, 1H, *J* = 8.8, 7.2, 1.6 Hz, c), 8.18 (s, 1H, e)
8.07 (dd, 1H, *J* = 8.8, 0.8 Hz, d), 7.86 (ddd, 1H, *J* = 8.2, 7.2, 0.8 Hz, b)
7.80 (m, 1H, h), 7.70 (dd, 2H, *J* = 8.0, 7.6 Hz, g)



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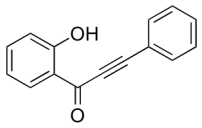
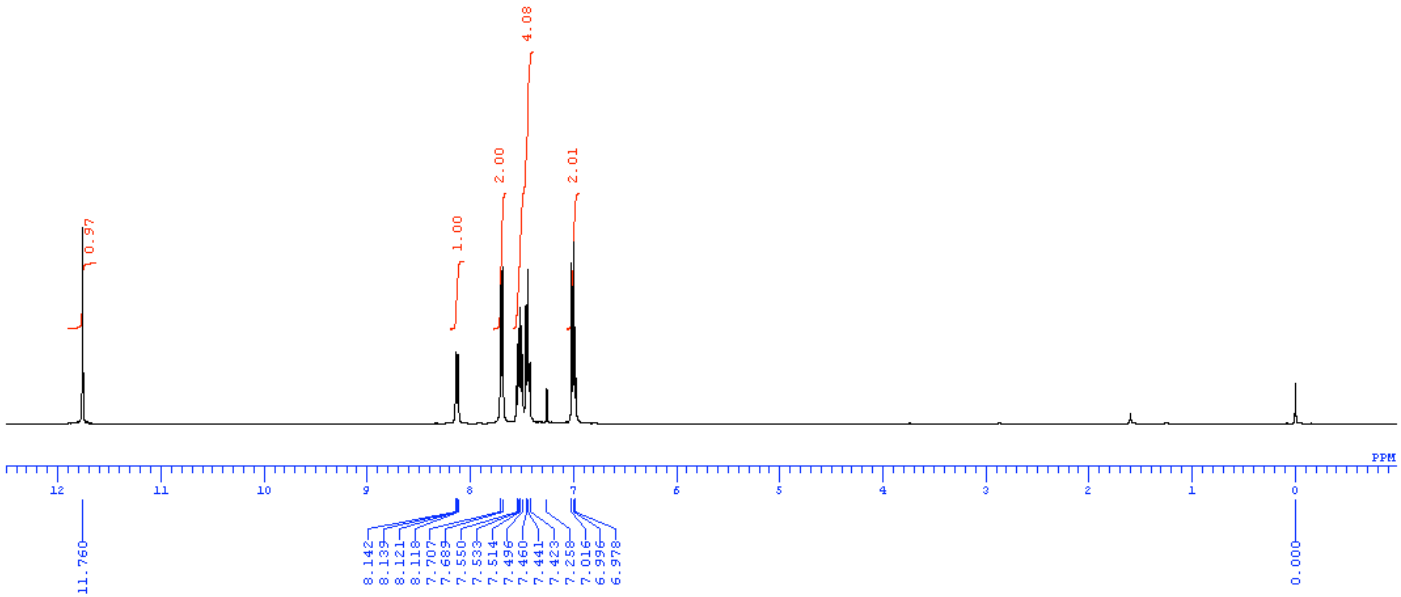


10a

^{1}H NMR ($CDCl_3$)



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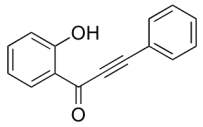
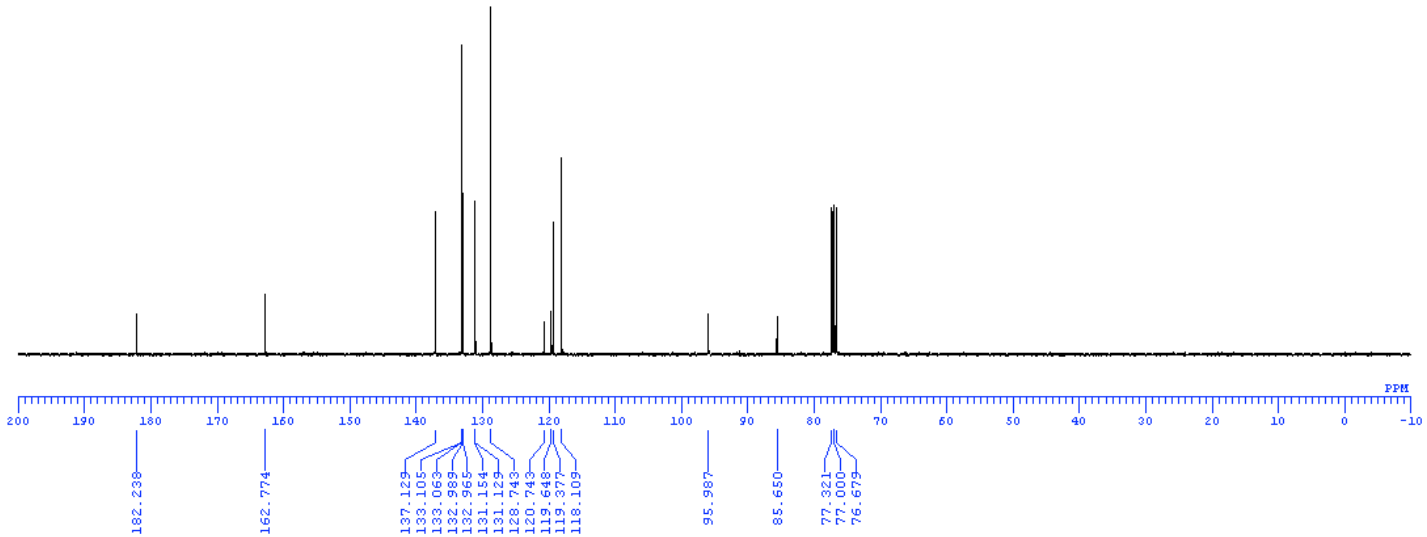
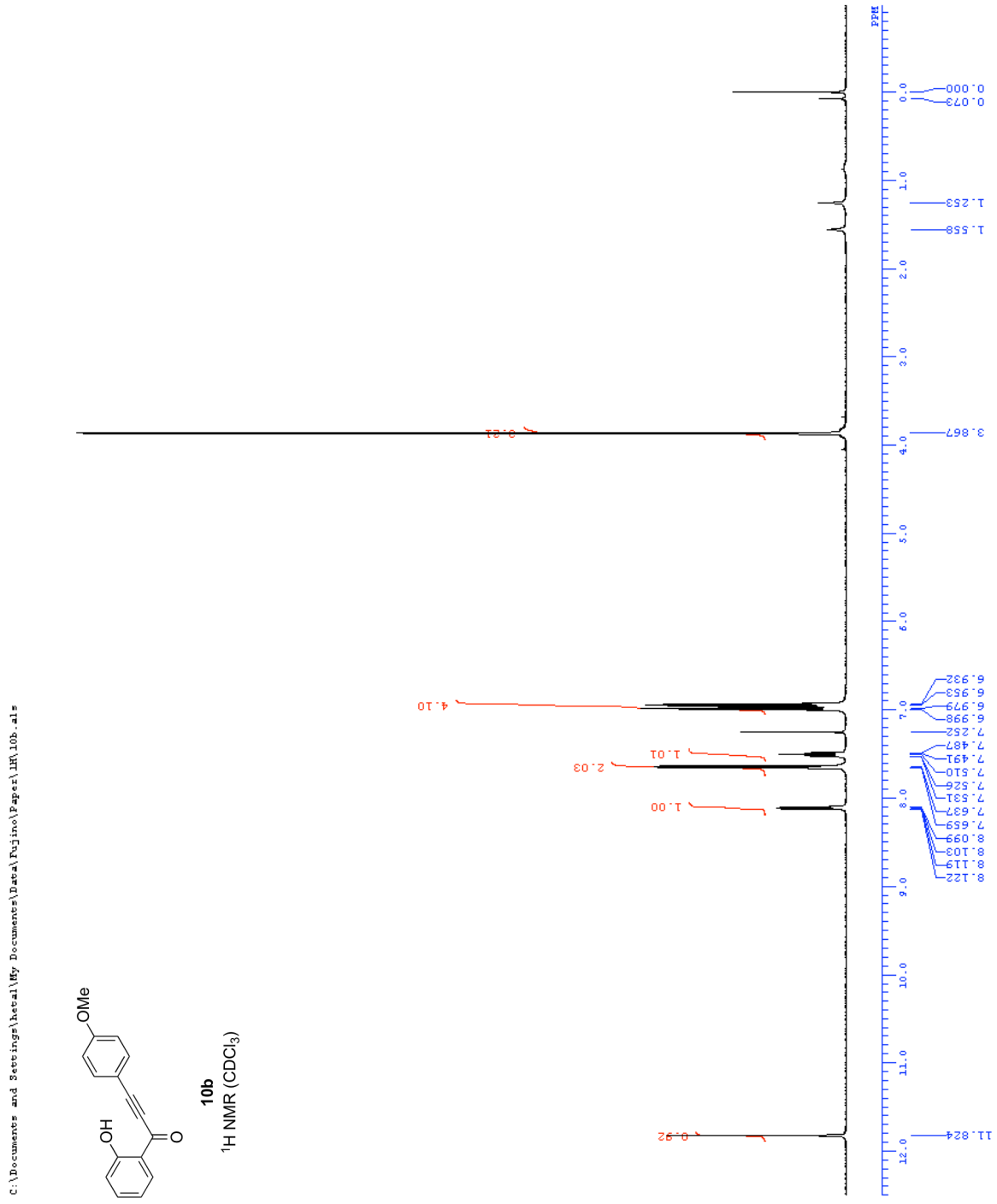


10a

^{13}C NMR ($CDCl_3$)

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OMe
OH
O
10b
^{1}H NMR ($CDCl_3$)
PPM
0.000
0.073
1.253
1.558
3.867
6.932
6.953
6.979
6.998
7.252
7.487
7.491
7.510
7.526
7.531
7.637
7.659
8.099
8.103
8.119
8.122
11.824
0.92
1.00
2.03
1.01
4.10
9.21

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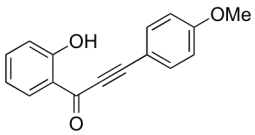
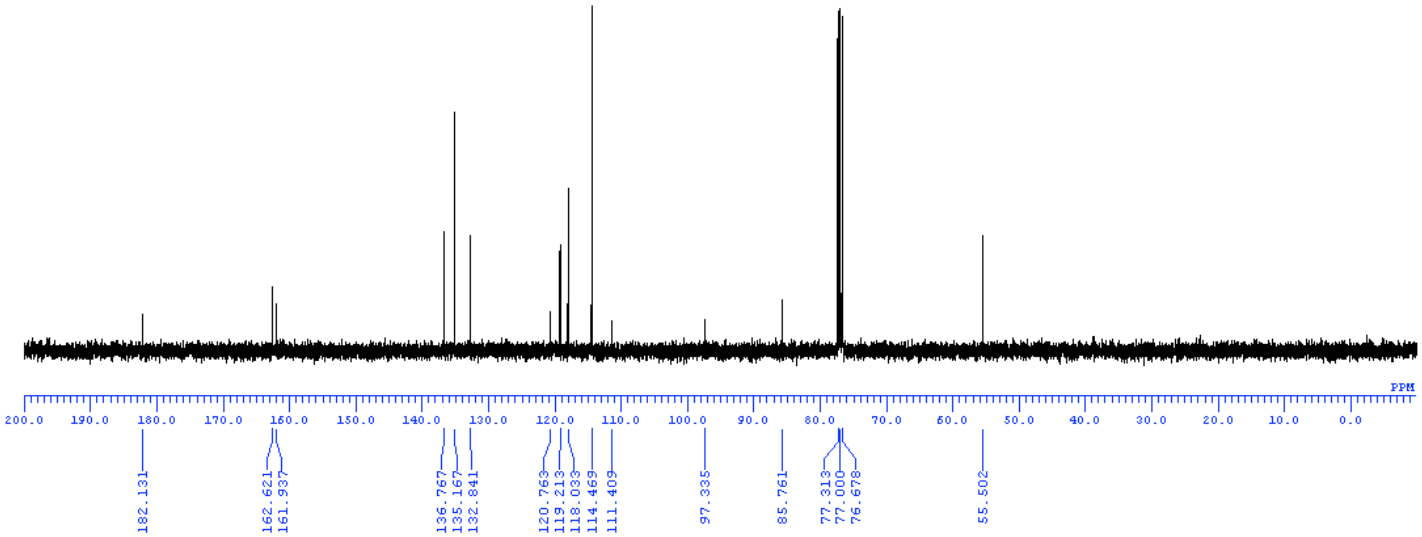
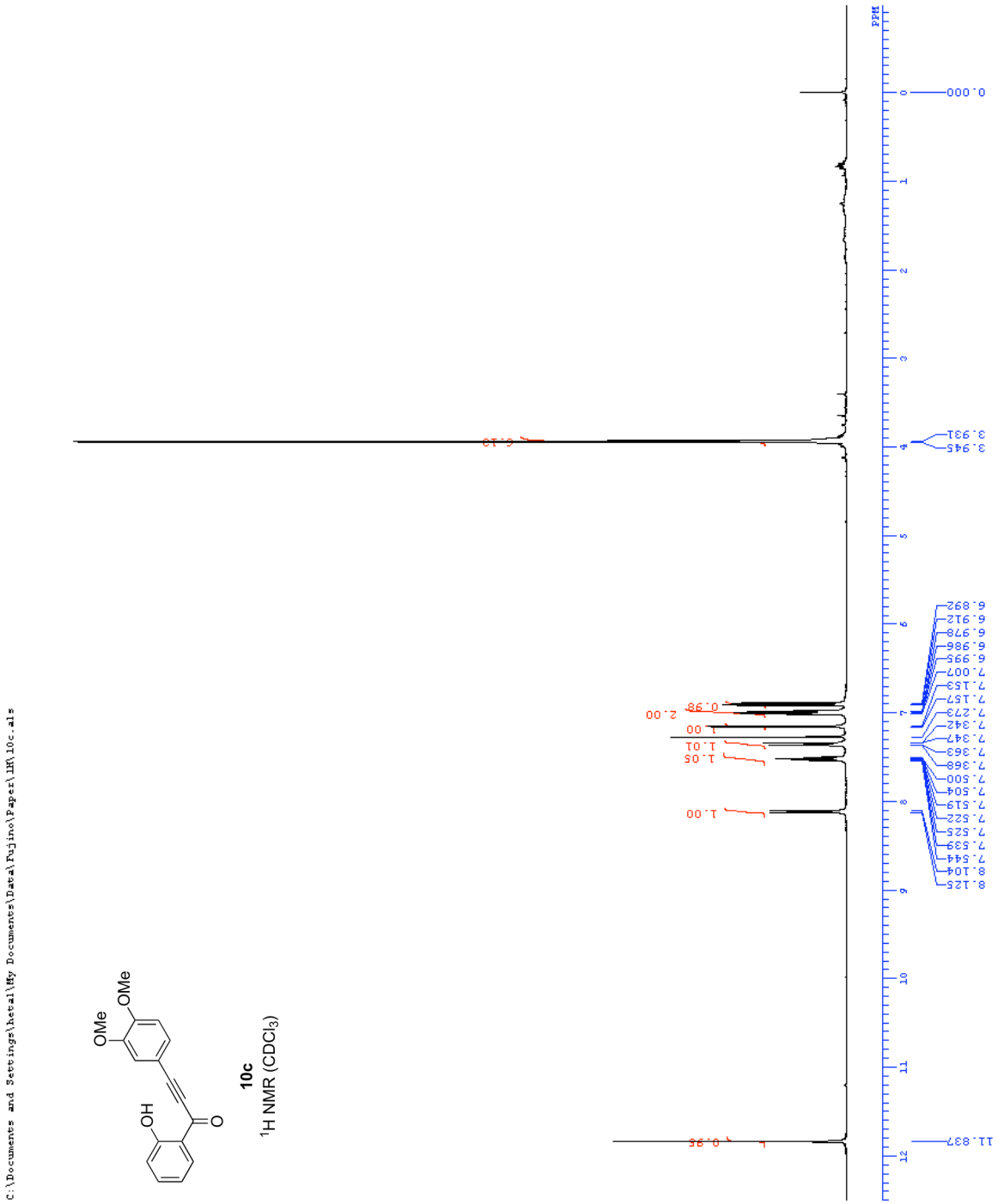


10b

^{13}C NMR ($CDCl_3$)




C:\Documents and Settings\hetal\My Documents\Data\Pujino\Paper\1H\10c.als
OMe
OMe
OH
O
10c
^{1}H NMR ($CDCl_3$)
PPM

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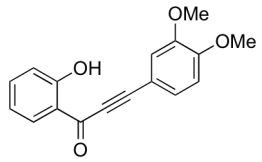
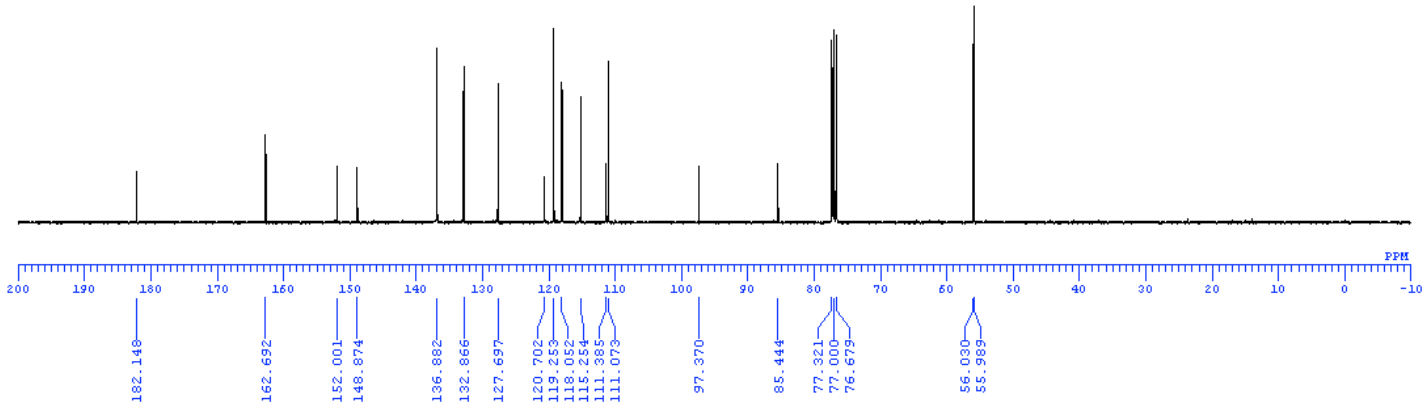


10c

^{13}C NMR ($CDCl_3$)



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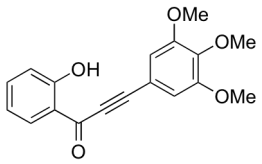

10d

^{1}H NMR ($CDCl_3$)

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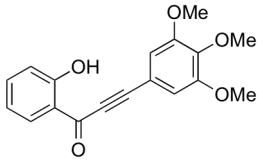
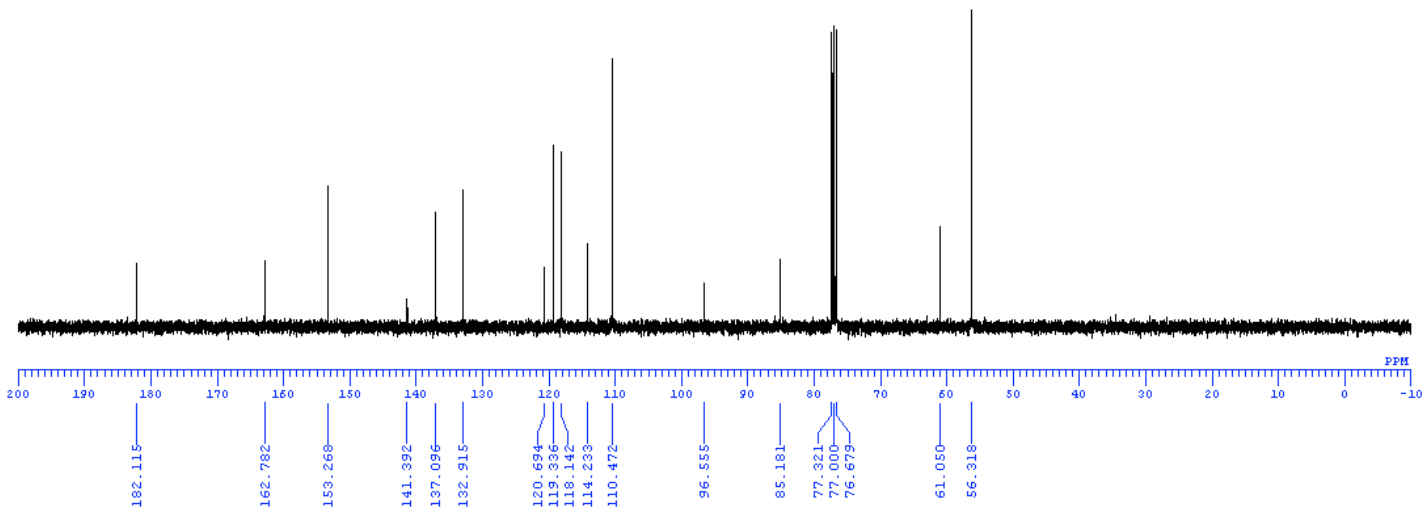
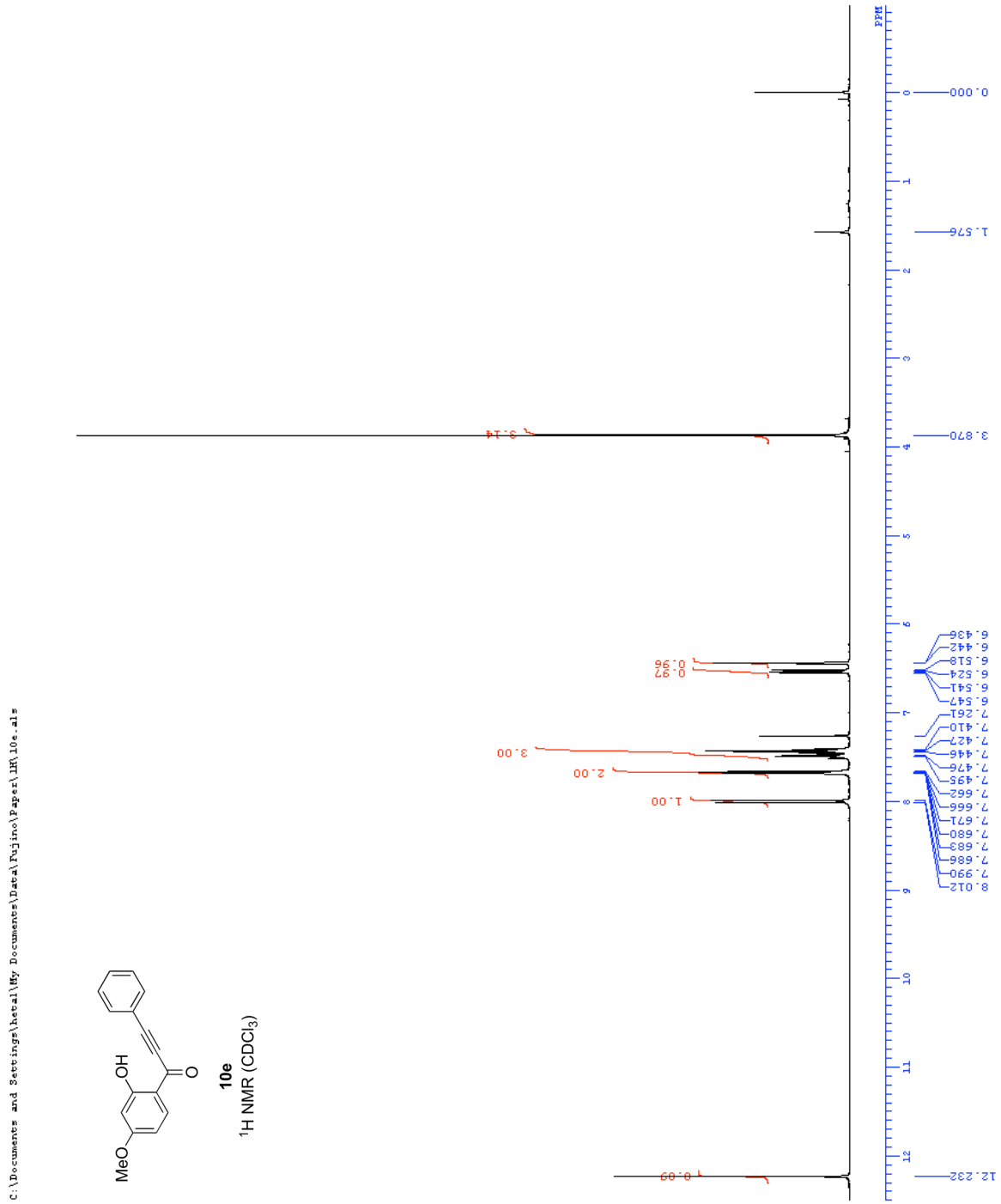


10d

^{13}C NMR ($CDCl_3$)

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OH
O
MeO
10e
1H NMR (CDCl3)
PPM
0.000
1.576
3.870
12.232
3.14
0.97
0.96
3.00
2.00
1.00
1.09
8.012
7.990
7.686
7.683
7.680
7.671
7.666
7.662
7.495
7.476
7.446
7.427
7.410
7.261
6.547
6.541
6.524
6.518
6.442
6.436

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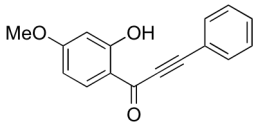
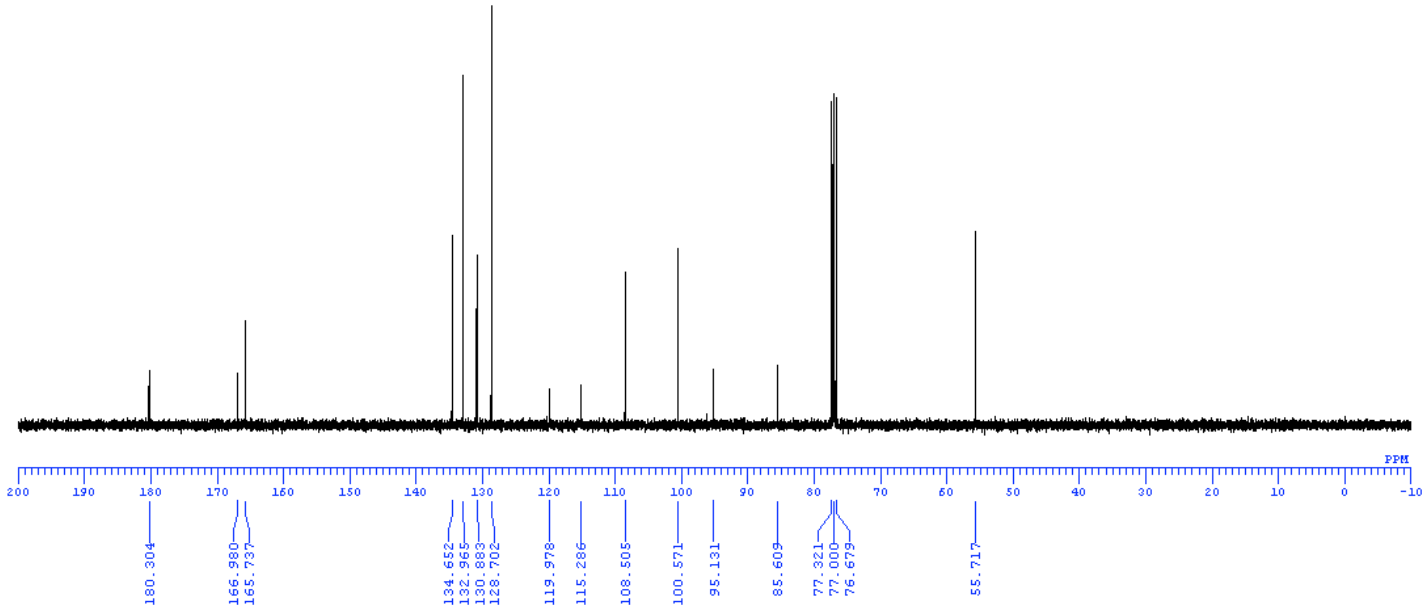


10e

^{13}C NMR ($CDCl_3$)



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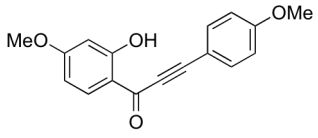

10f

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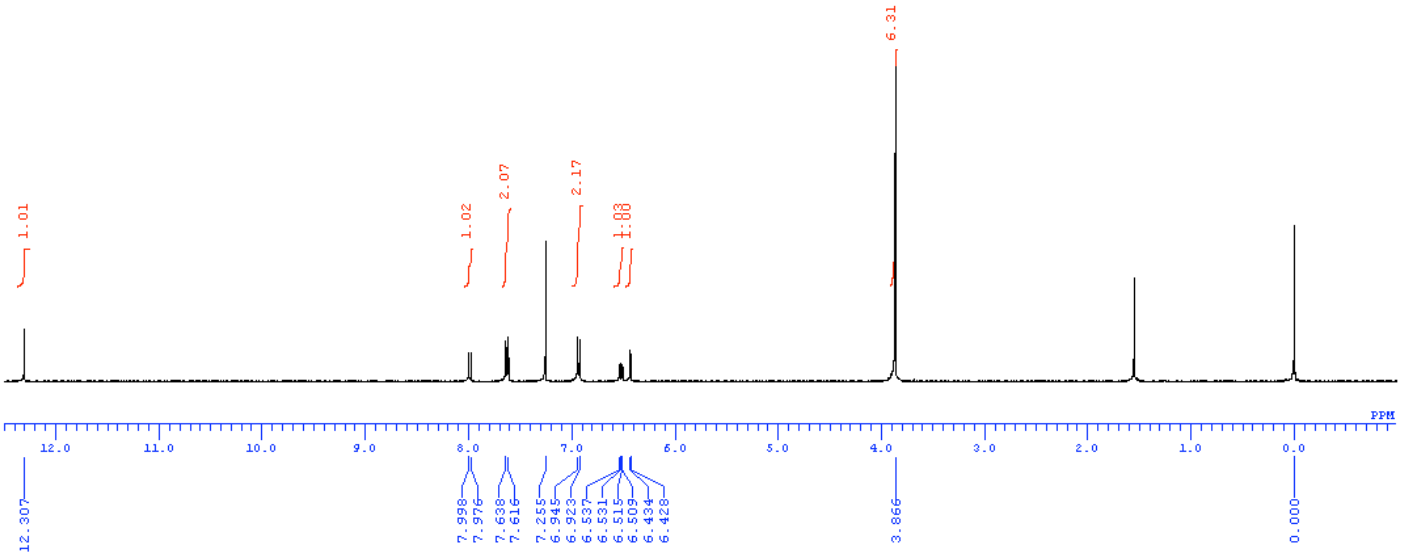

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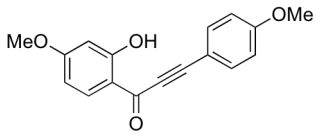
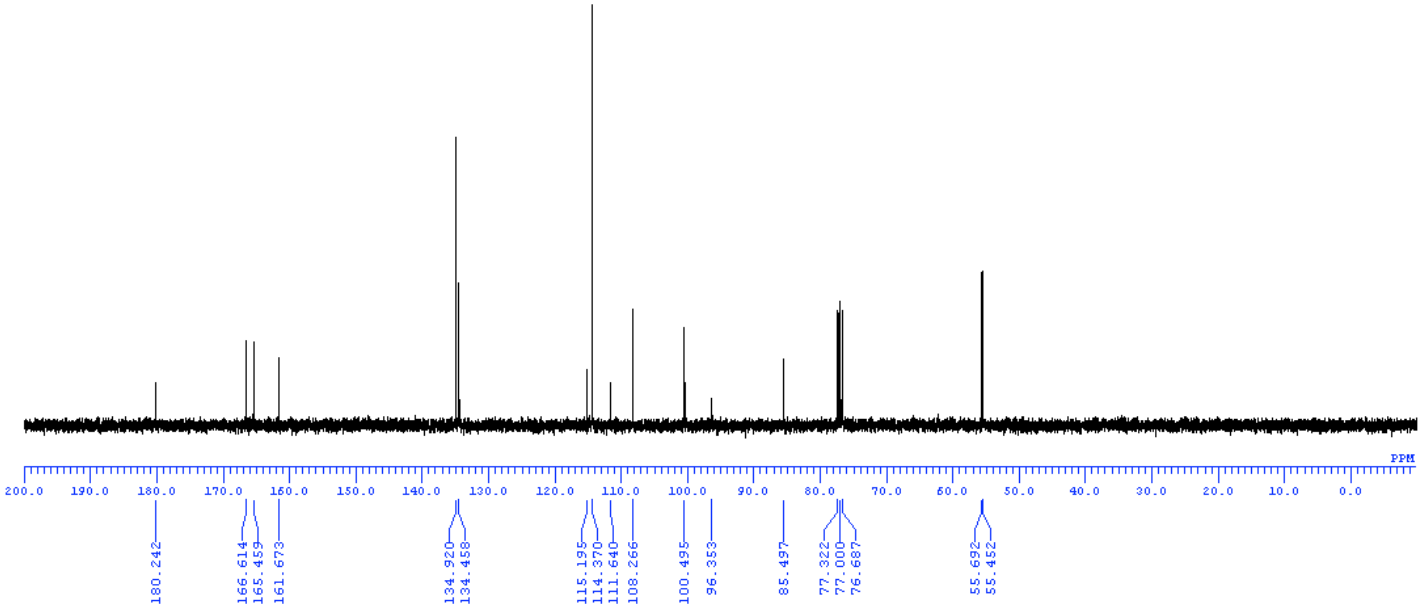


10f

^{13}C NMR ($CDCl_3$)



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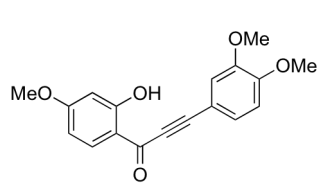

10g

^{1}H NMR ($CDCl_3$)

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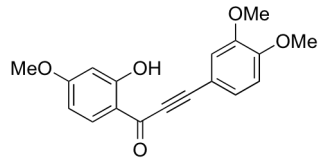

10g

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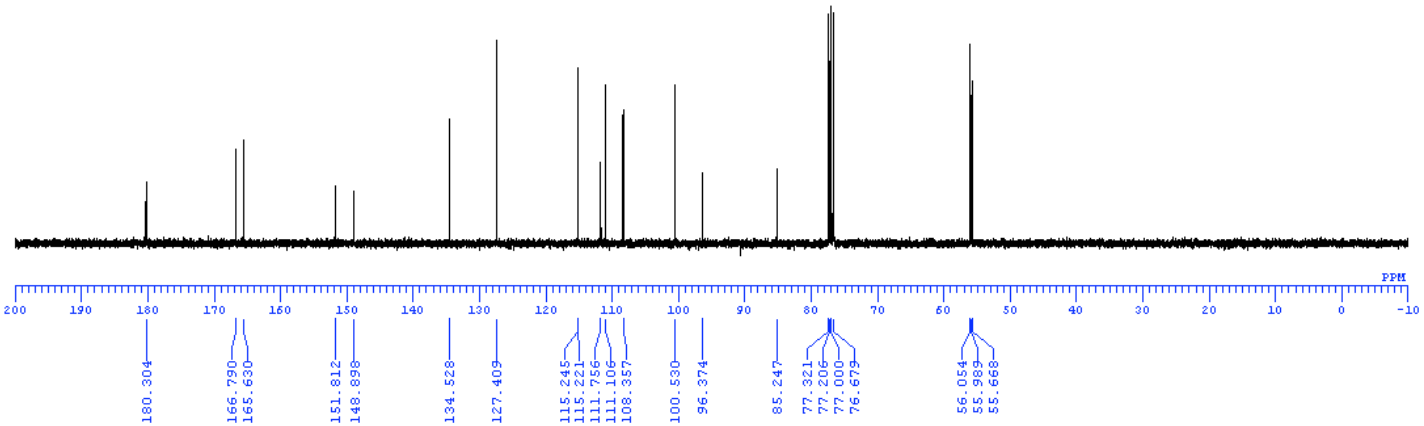

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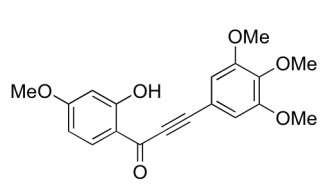

10h

^{1}H NMR ($CDCl_3$)

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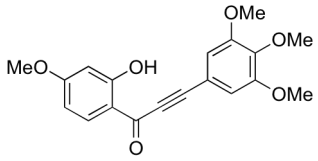
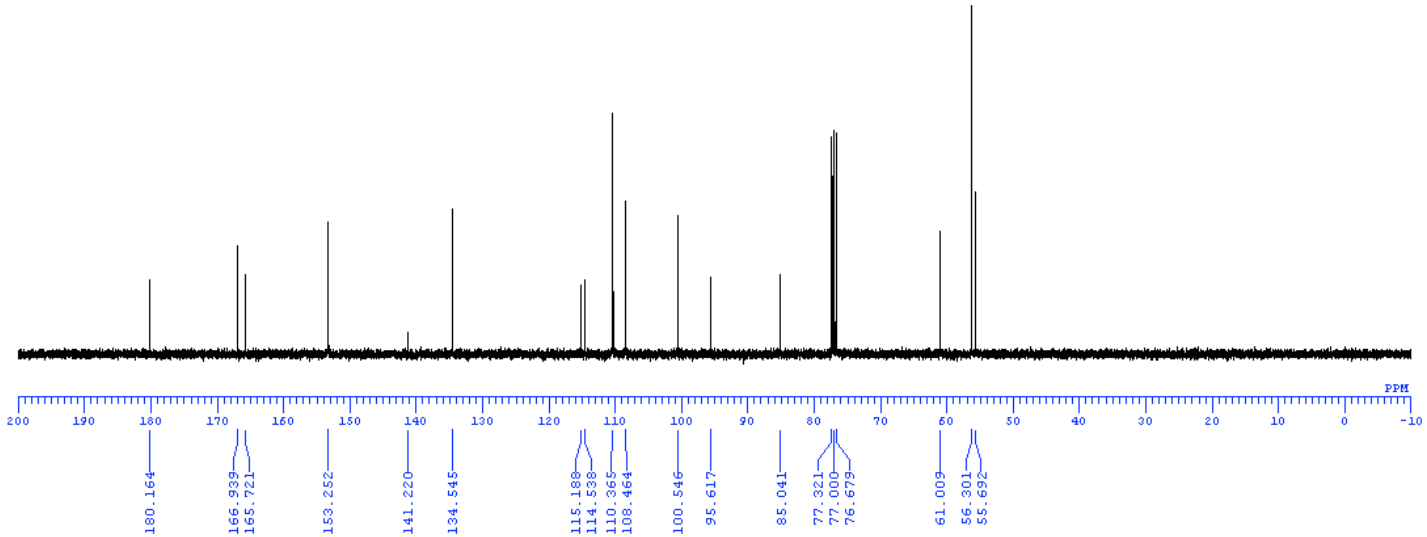


10h

^{13}C NMR ($CDCl_3$)



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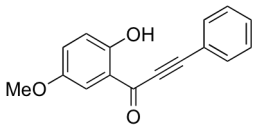
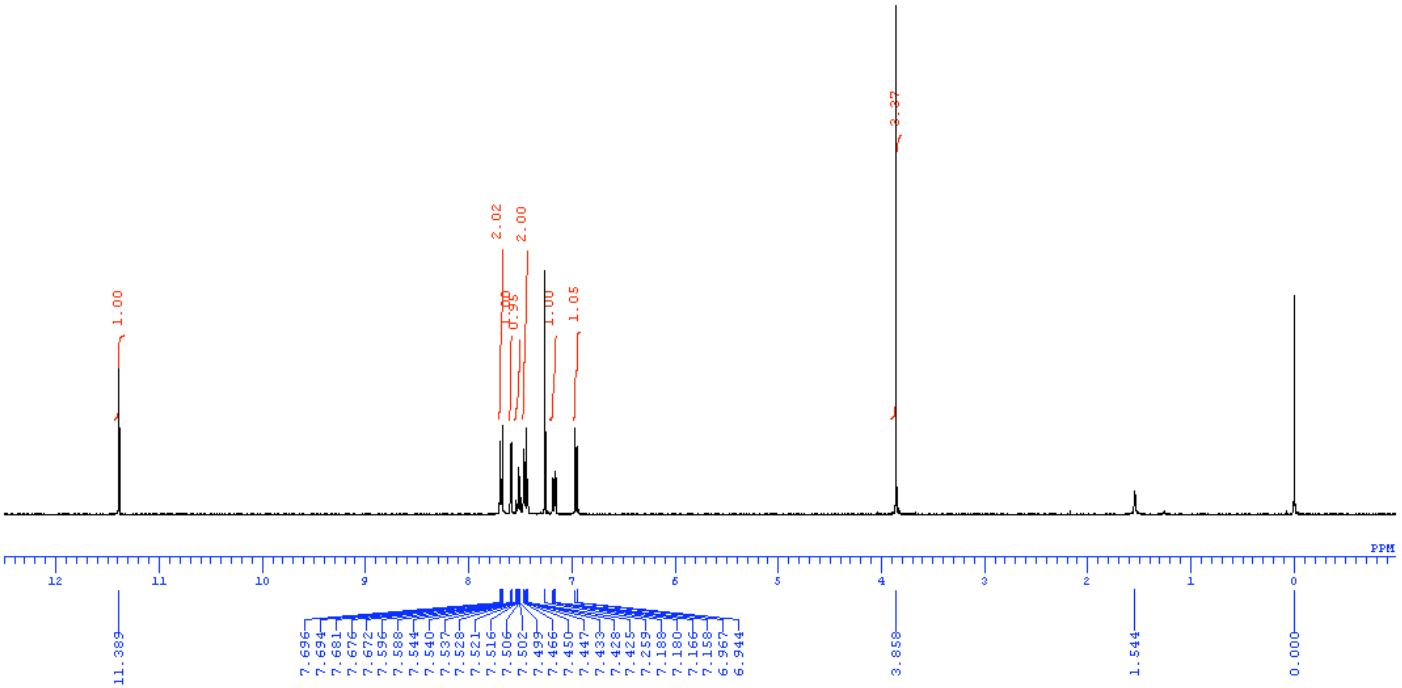


10i

^{1}H NMR ($CDCl_3$)



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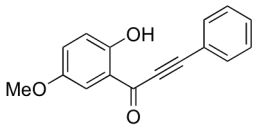
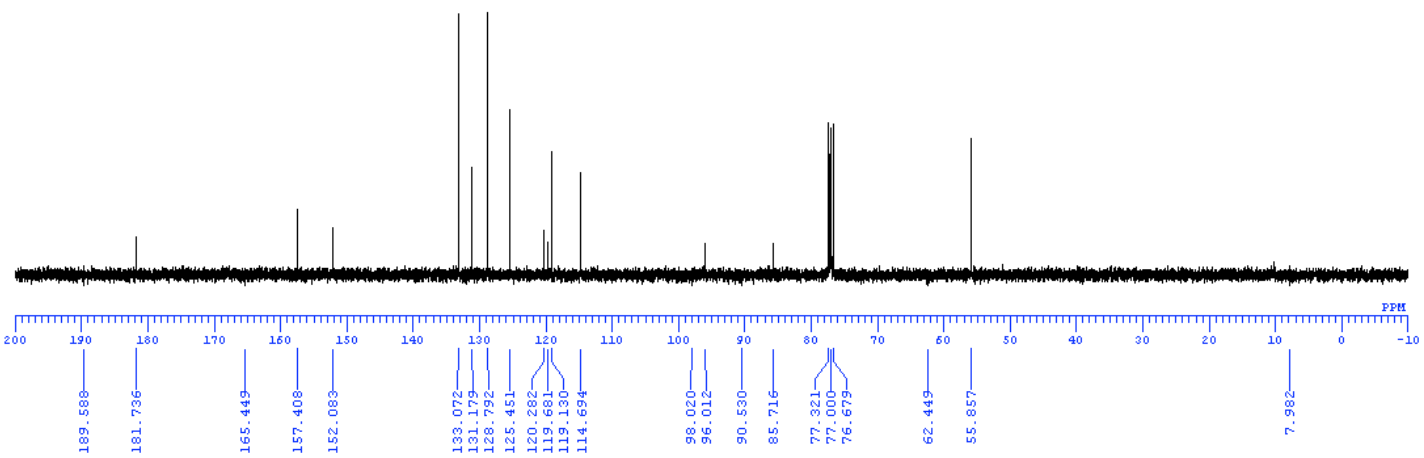


10i

^{13}C NMR ($CDCl_3$)



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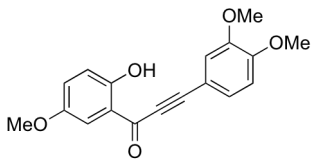

10j

^{1}H NMR ($CDCl_3$)

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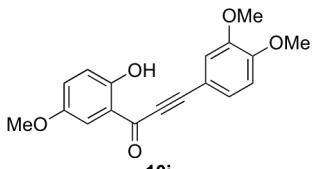
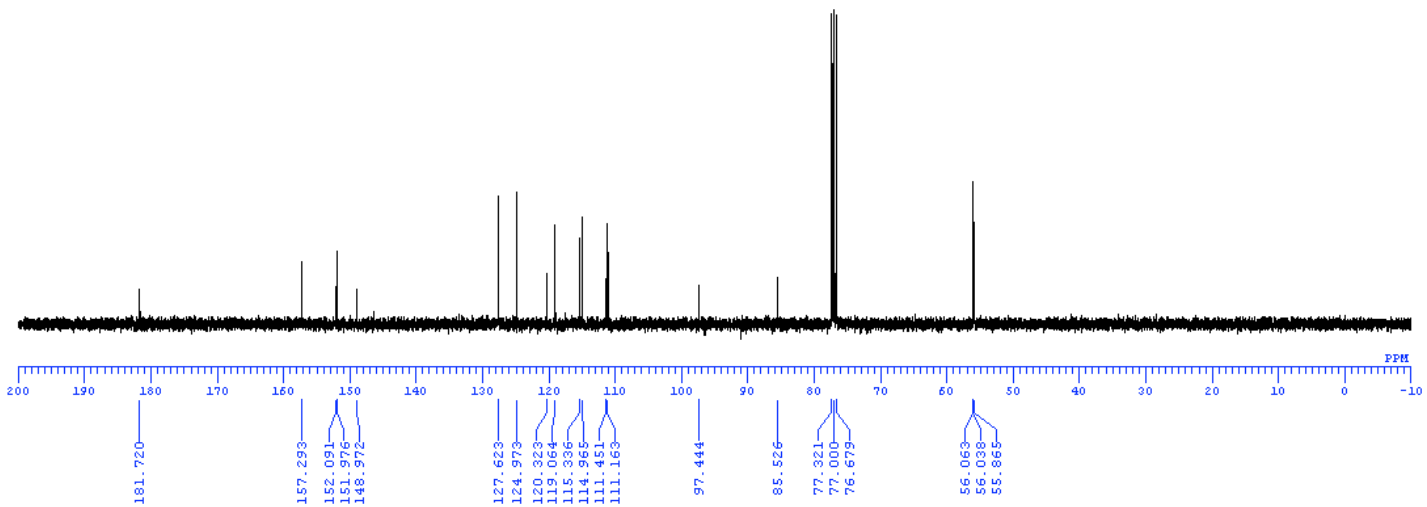
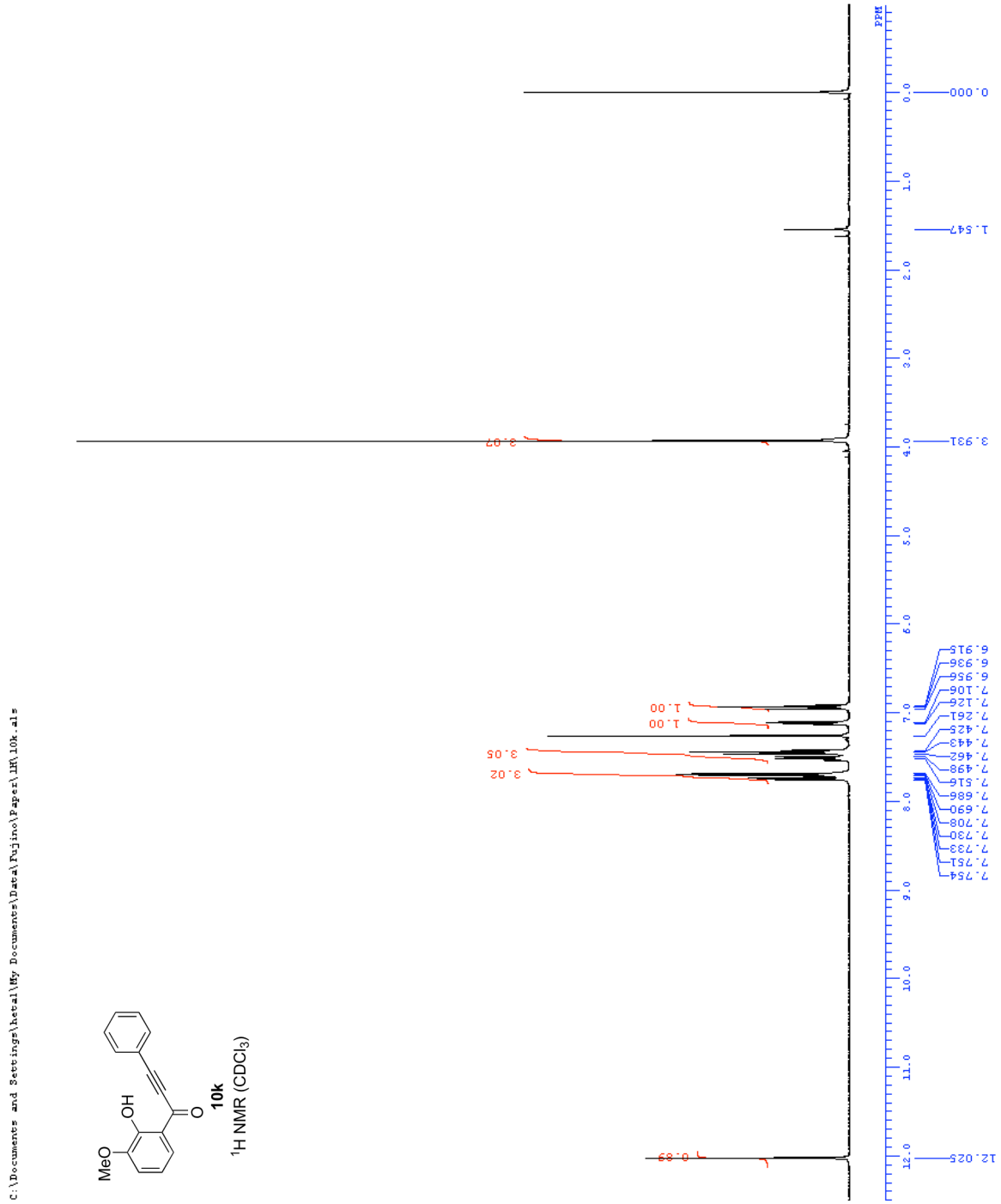


10j

^{13}C NMR ($CDCl_3$)




C:\Documents and Settings\hetal\My Documents\Data\Fujino\Paper\1H\10k.als
MeO
OH
O
10k
^{1}H NMR ($CDCl_3$)
PPM
0.000
1.547
3.931
6.915
6.936
6.956
7.106
7.126
7.261
7.425
7.443
7.462
7.498
7.516
7.686
7.690
7.708
7.730
7.733
7.751
7.754
12.025
3.97
1.00
1.00
3.05
3.02
0.89

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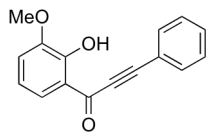
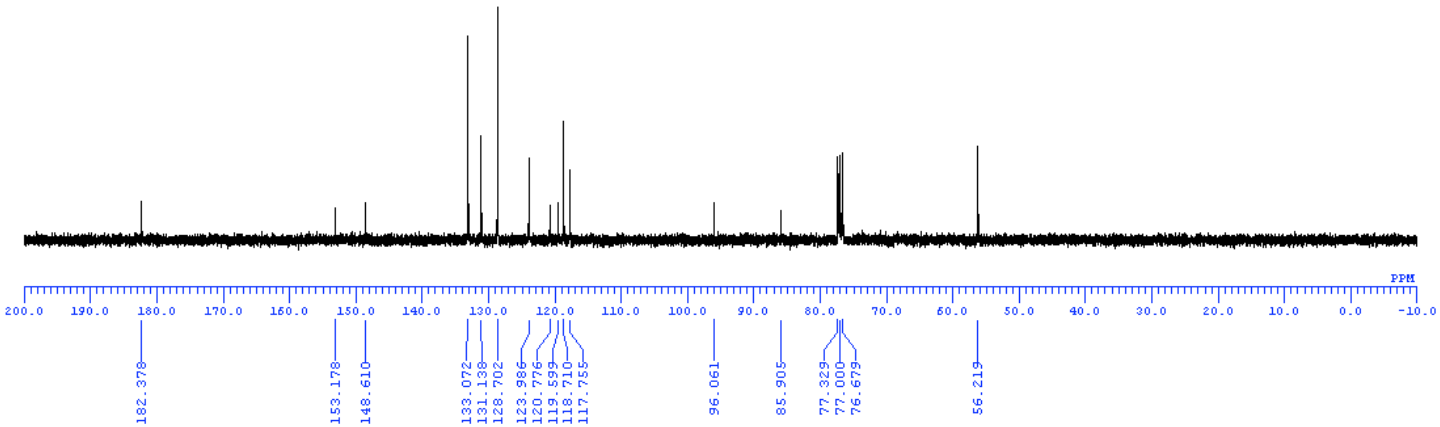


10k

^{13}C NMR ($CDCl_3$)



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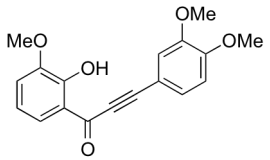

10l

^{1}H NMR ($CDCl_3$)

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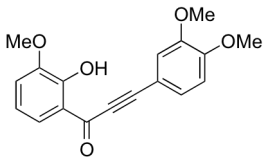
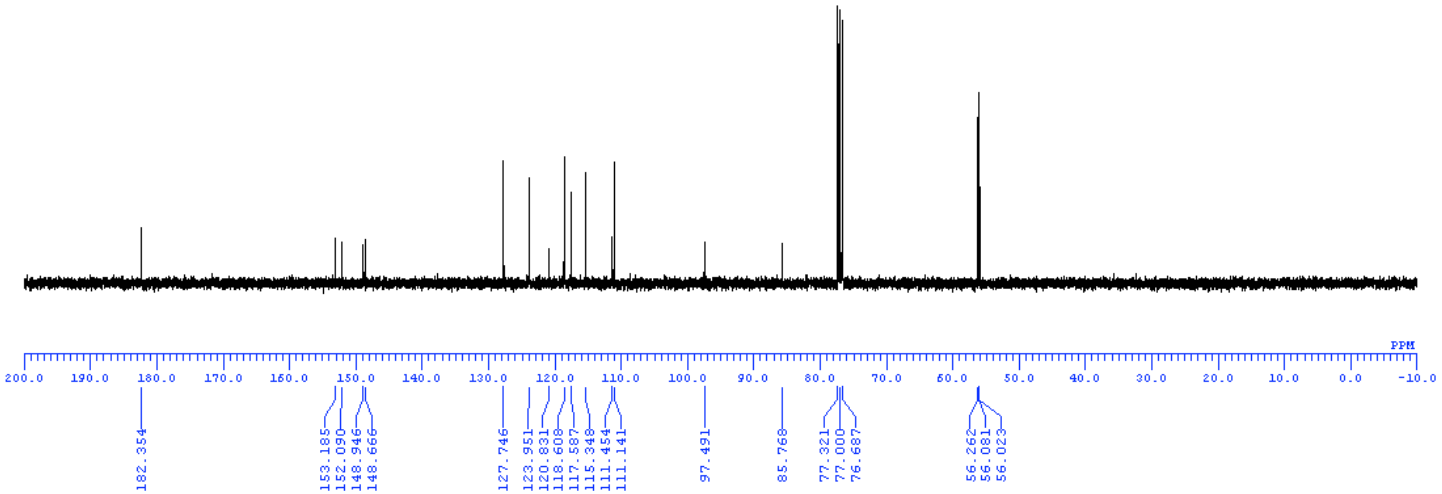
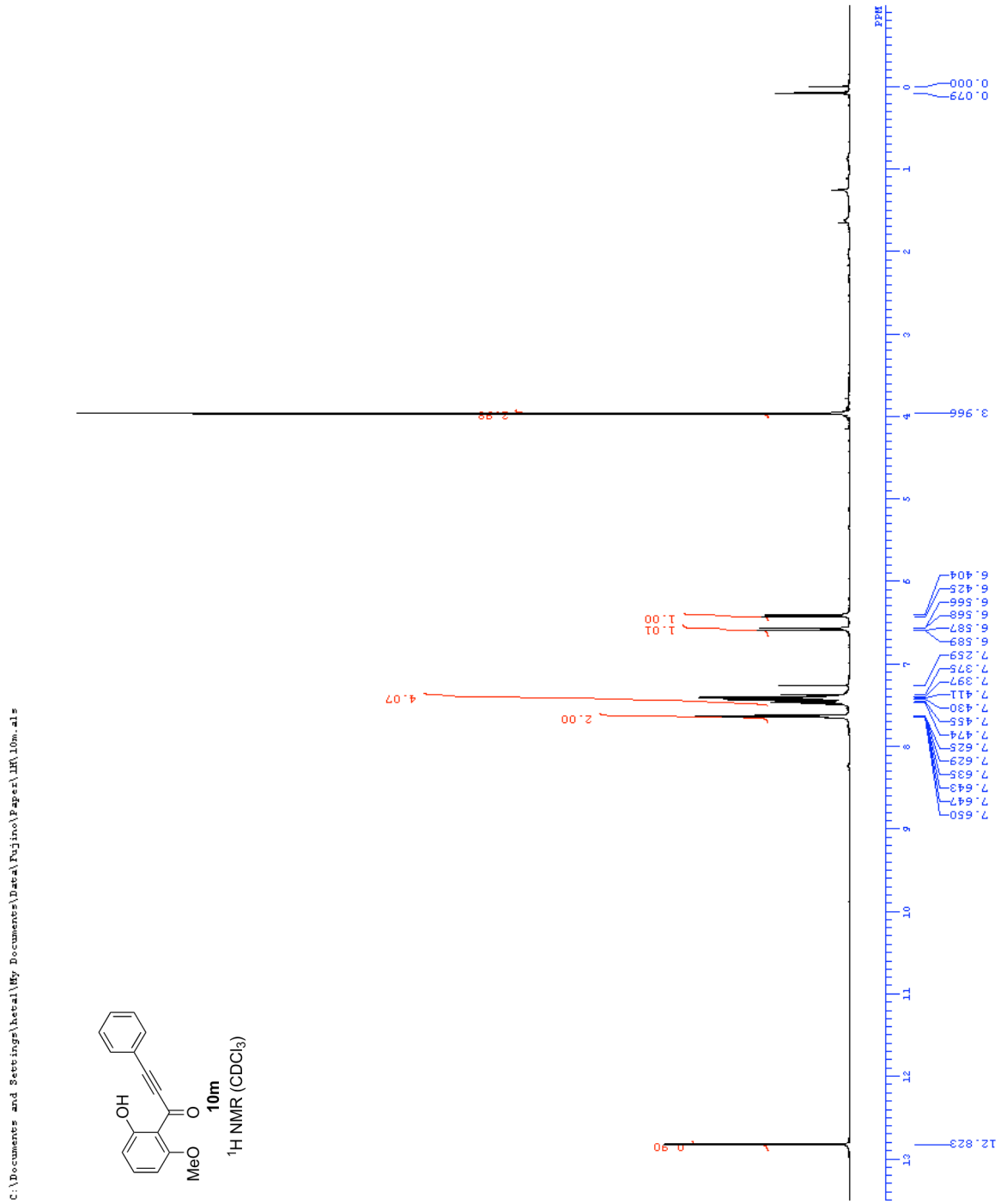
auto



10l

^{13}C NMR ($CDCl_3$)

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OH
O
MeO
10m
1H NMR (CDCl3)
PPM
0.000
0.079
3.966
6.404
6.425
6.566
6.568
6.587
6.589
7.259
7.375
7.397
7.411
7.430
7.455
7.474
7.625
7.629
7.635
7.643
7.647
7.650
12.823
2.99
1.01
1.00
4.07
2.00
0.90

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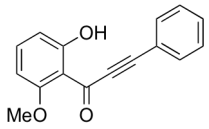
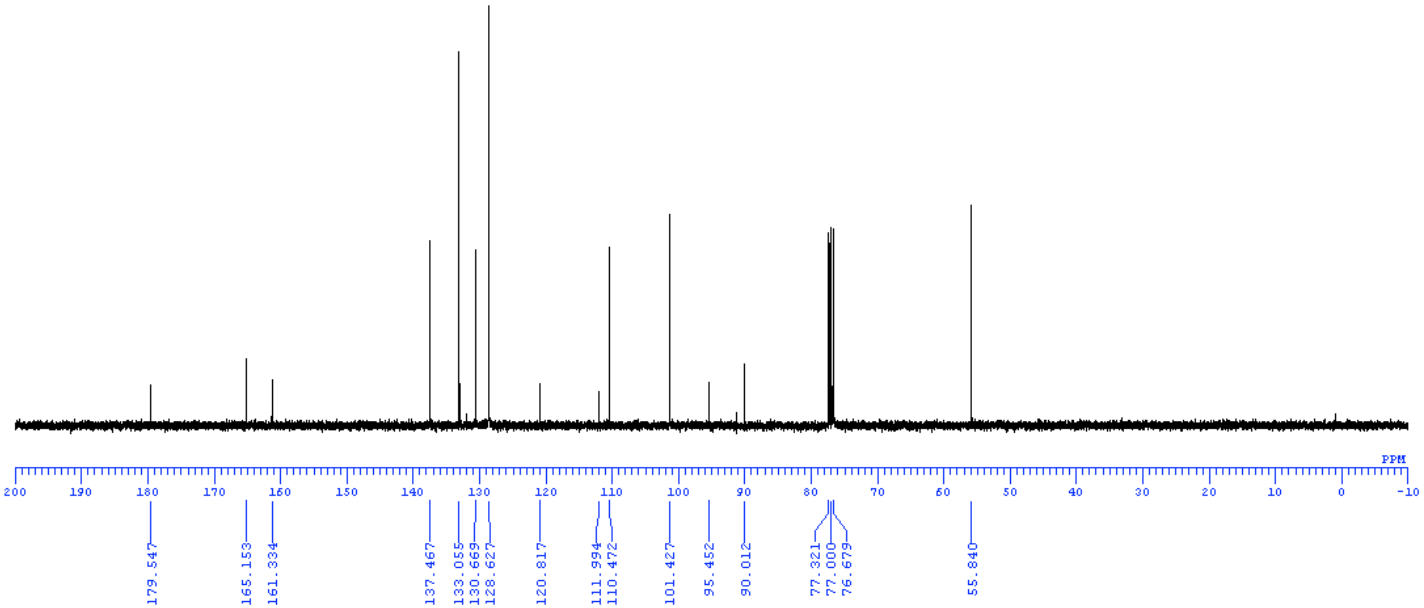


10m

^{13}C NMR ($CDCl_3$)



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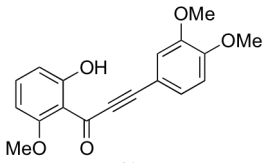
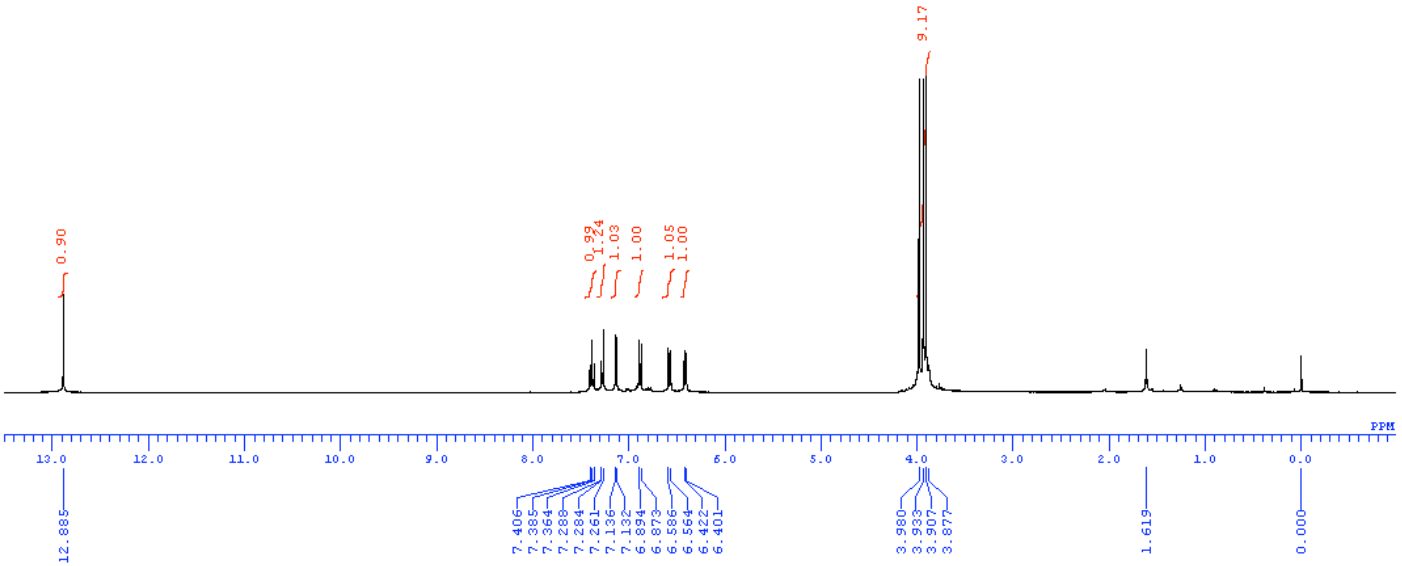


10n

^{1}H NMR ($CDCl_3$)



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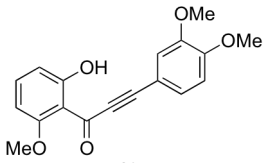
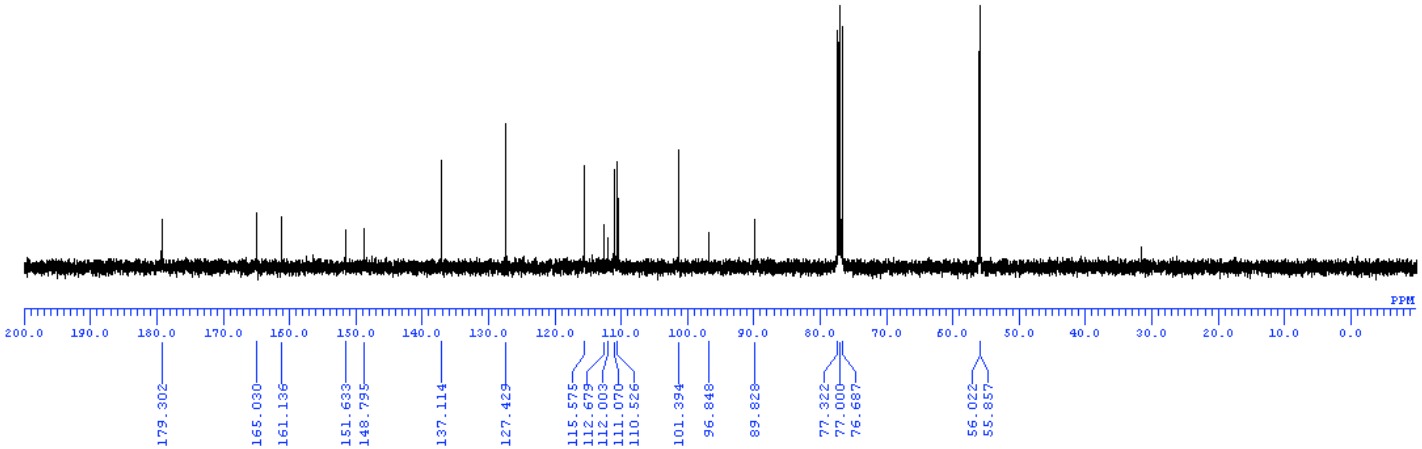
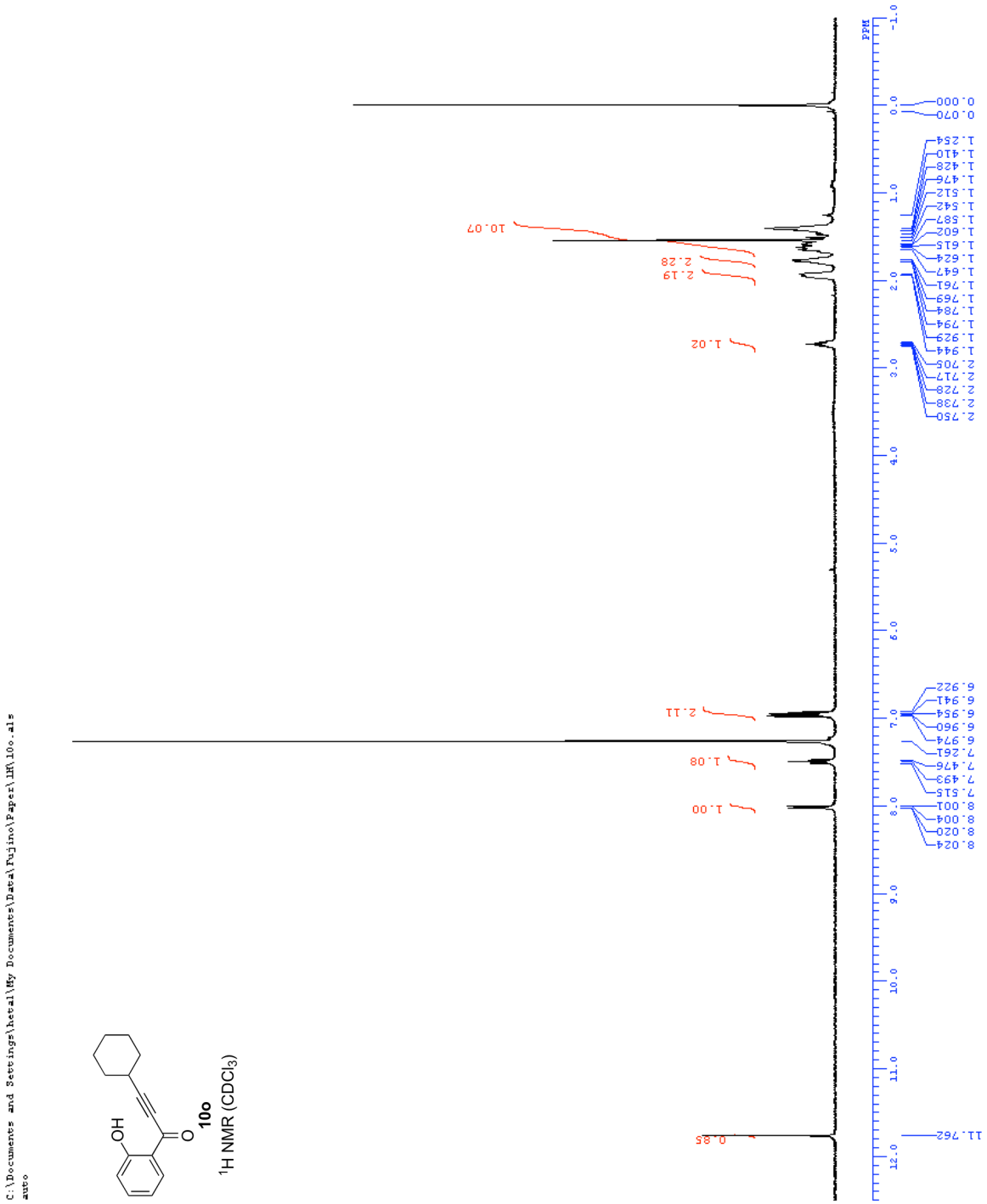
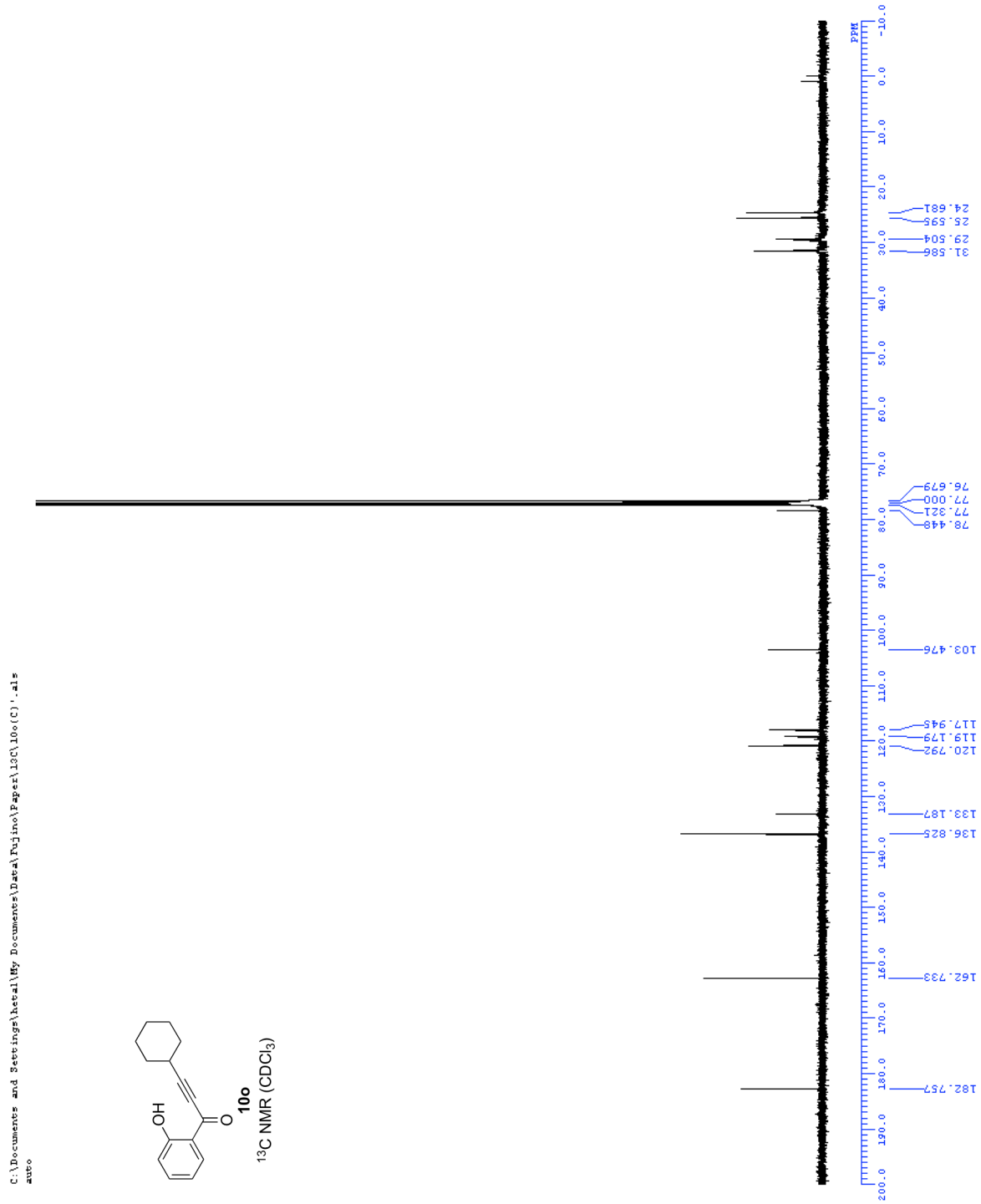


10n

^{13}C NMR ($CDCl_3$)




C:\Documents and Settings\hetal\My Documents\Data\Fujino\Paper\1H\10o.als
auto
OH
O
10o
^{1}H NMR ($CDCl_3$)
0.85
1.00
1.08
2.11
1.02
2.19
2.28
10.07
PPM
11.762
8.024
8.020
8.004
8.001
7.515
7.493
7.476
7.261
6.974
6.960
6.954
6.941
6.922
2.750
2.738
2.728
2.717
2.705
1.944
1.929
1.794
1.784
1.769
1.761
1.647
1.624
1.615
1.602
1.587
1.542
1.512
1.476
1.428
1.410
1.254
0.070
0.000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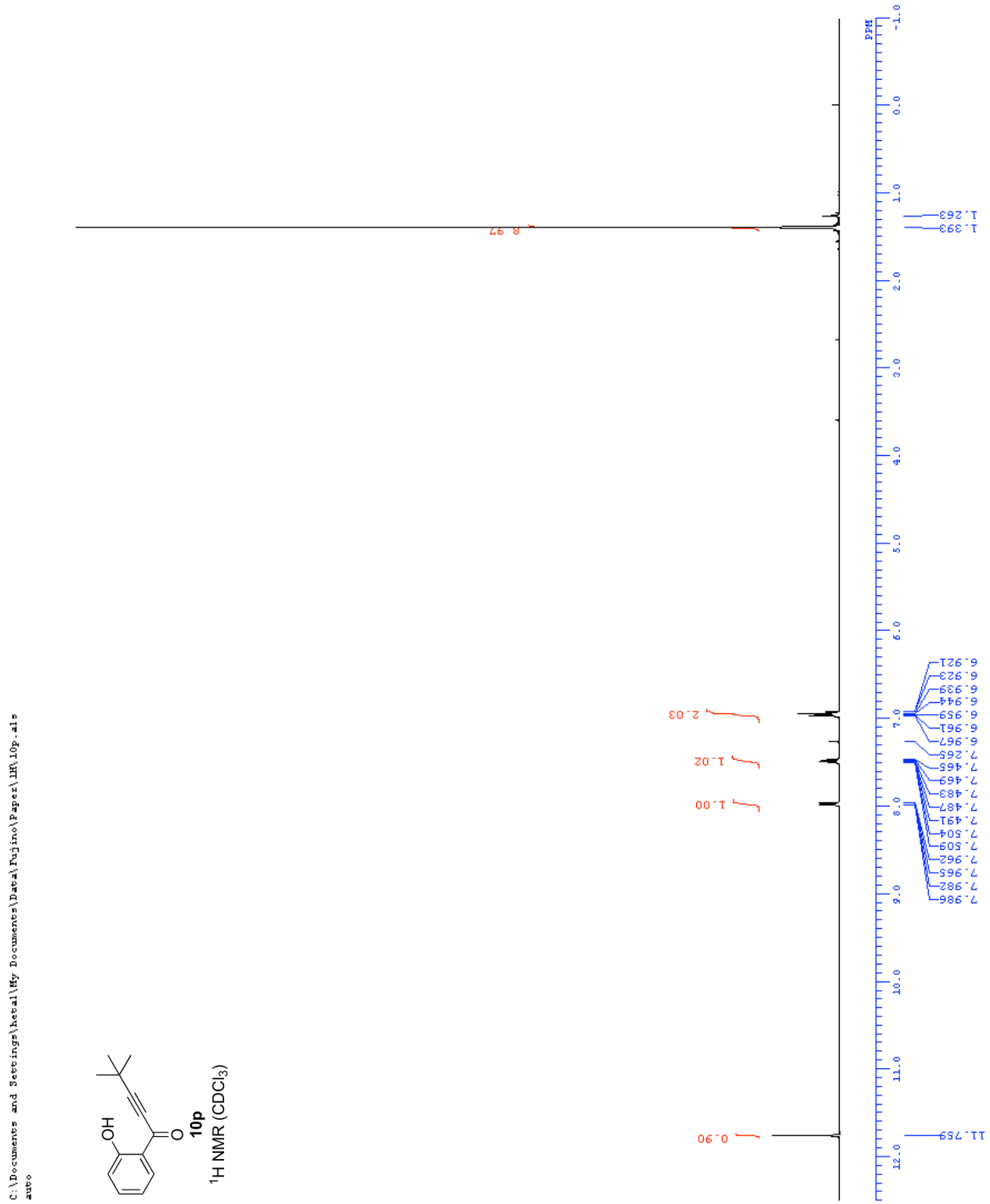
auto
OH
O
10o
13C NMR (CDCl3)
PPM
-10.0
0.0
10.0
20.0
30.0
40.0
50.0
60.0
70.0
80.0
90.0
100.0
110.0
120.0
130.0
140.0
150.0
160.0
170.0
180.0
190.0
200.0
24.681
25.595
29.504
31.586
76.679
77.000
77.321
78.448
103.476
117.945
119.179
120.792
133.187
136.825
162.733
182.757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auto
OH
O
10p
^{1}H NMR ($CDCl_3$)
8.97
2.03
1.02
1.00
0.90
11.759
7.986
7.982
7.965
7.962
7.509
7.504
7.491
7.487
7.483
7.469
7.465
7.265
6.967
6.961
6.959
6.944
6.939
6.923
6.921
1.393
1.263
PPM

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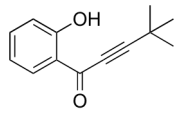
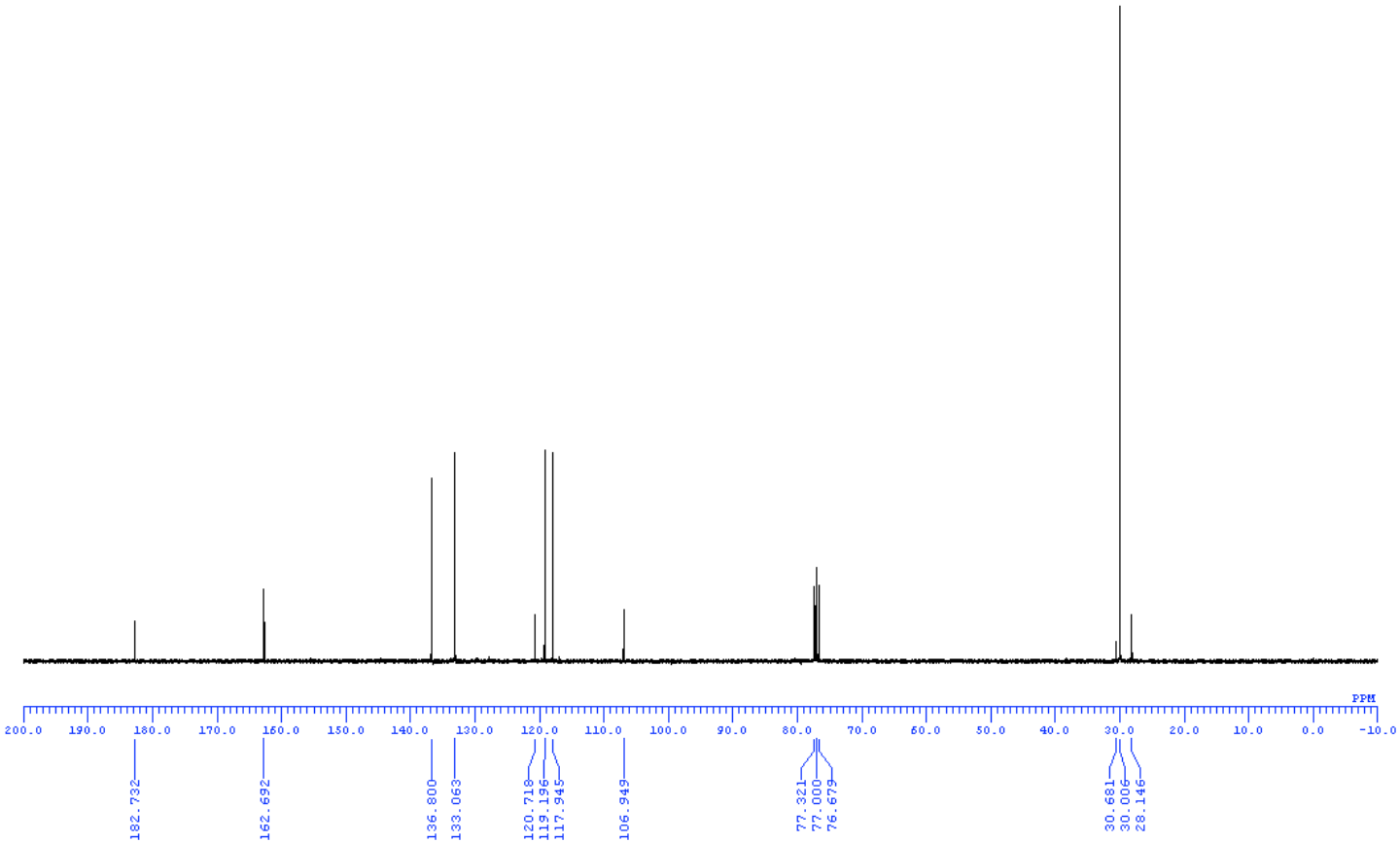
auto



10p

^{13}C NMR ($CDCl_3$)



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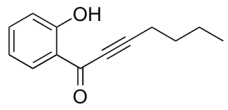
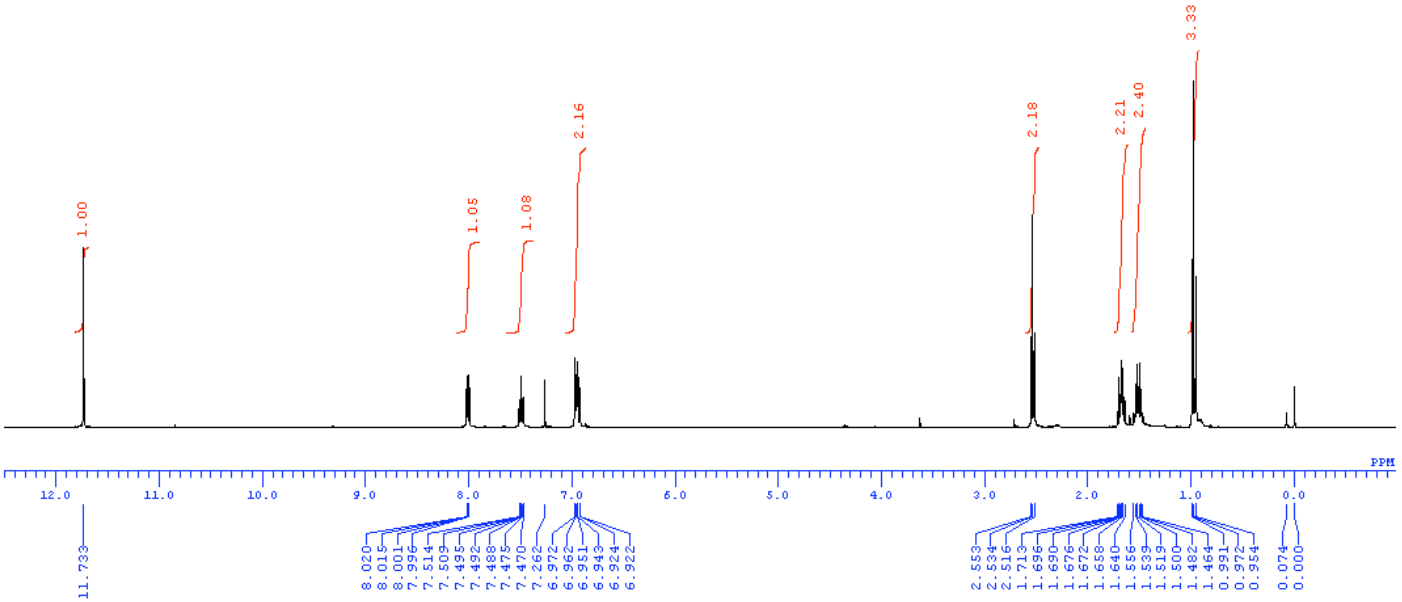
auto



10q

^{1}H NMR ($CDCl_3$)



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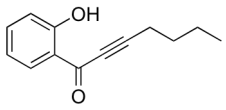
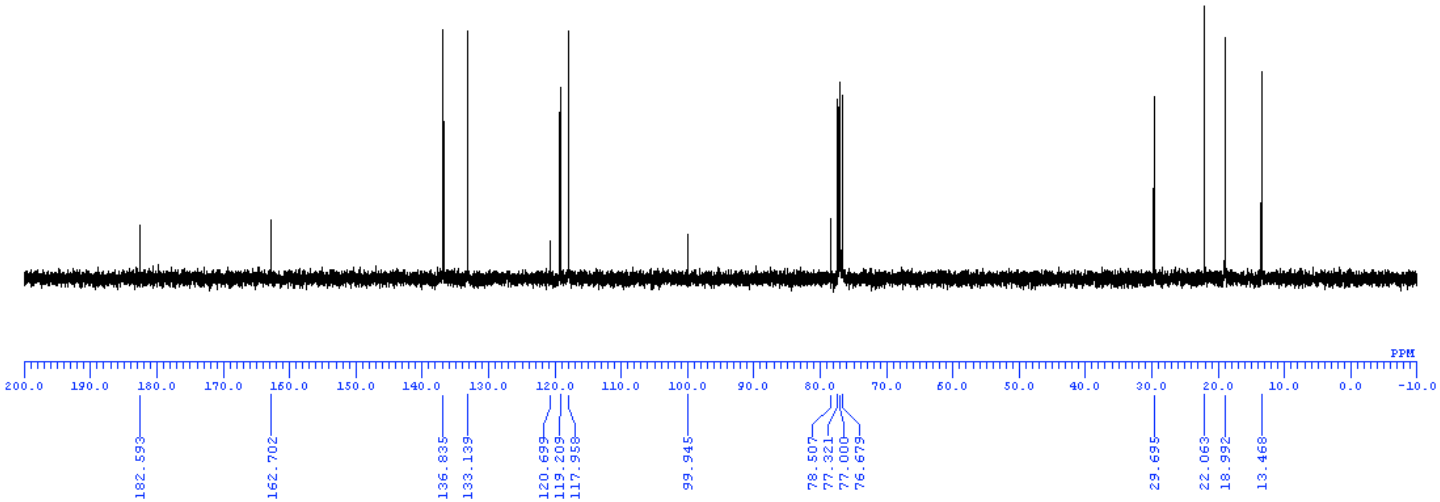


10q

^{13}C NMR ($CDCl_3$)



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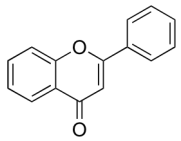
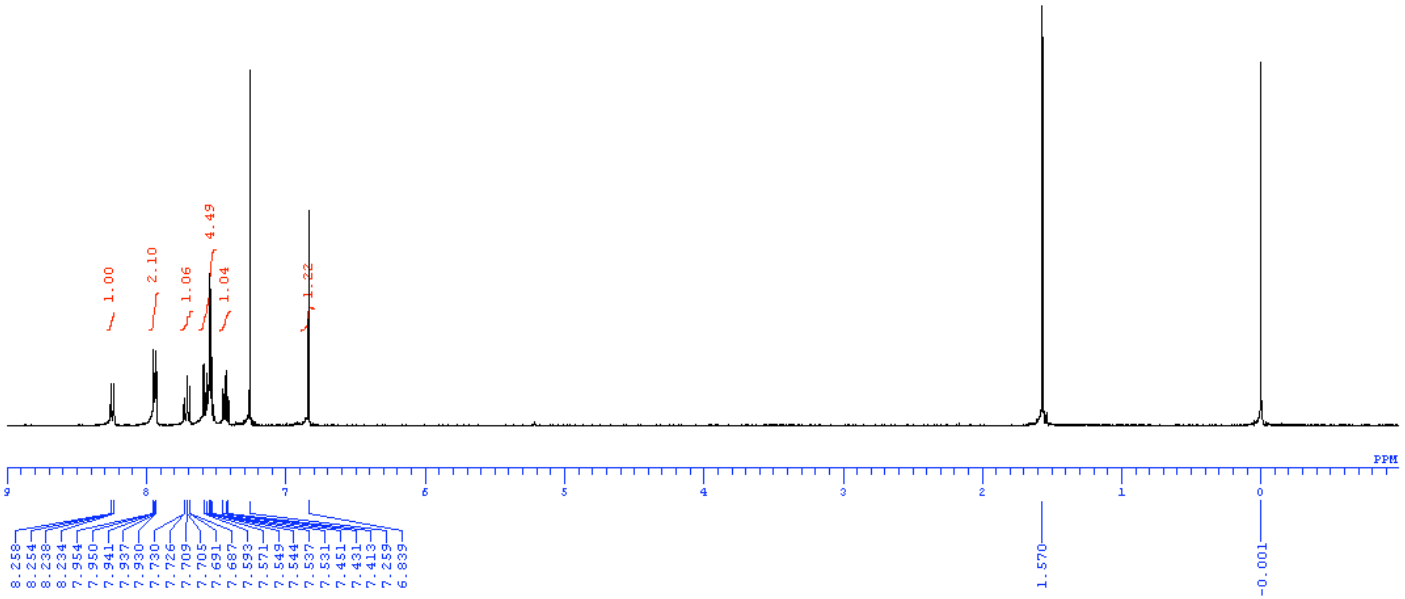


11a

^{1}H NMR ($CDCl_3$)



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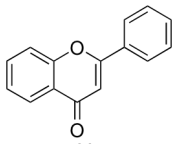
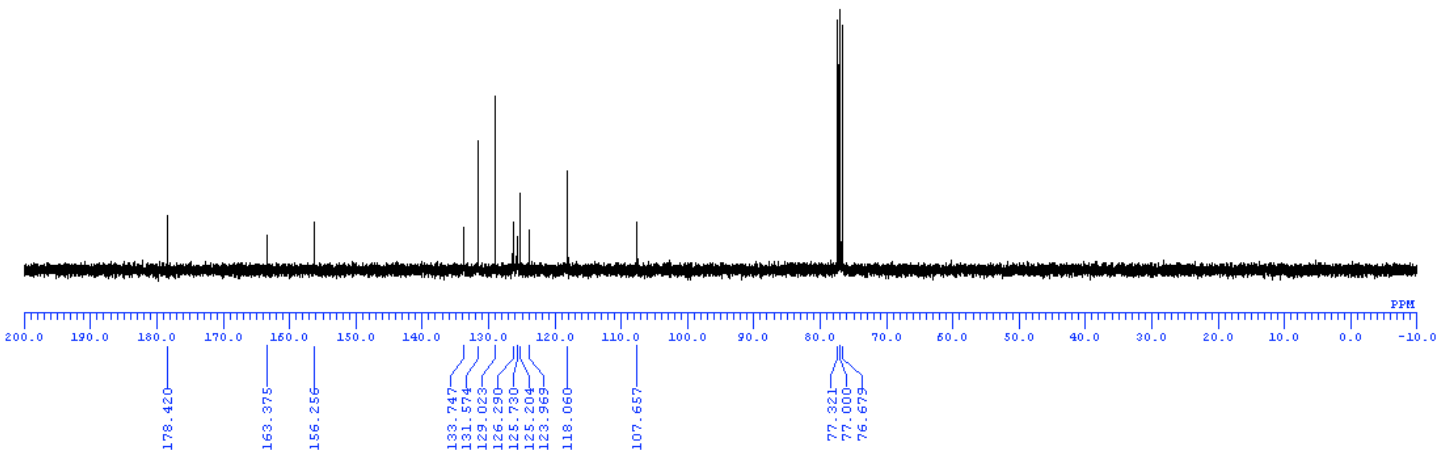


11a

^{13}C NMR ($CDCl_3$)



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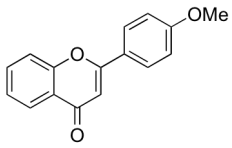
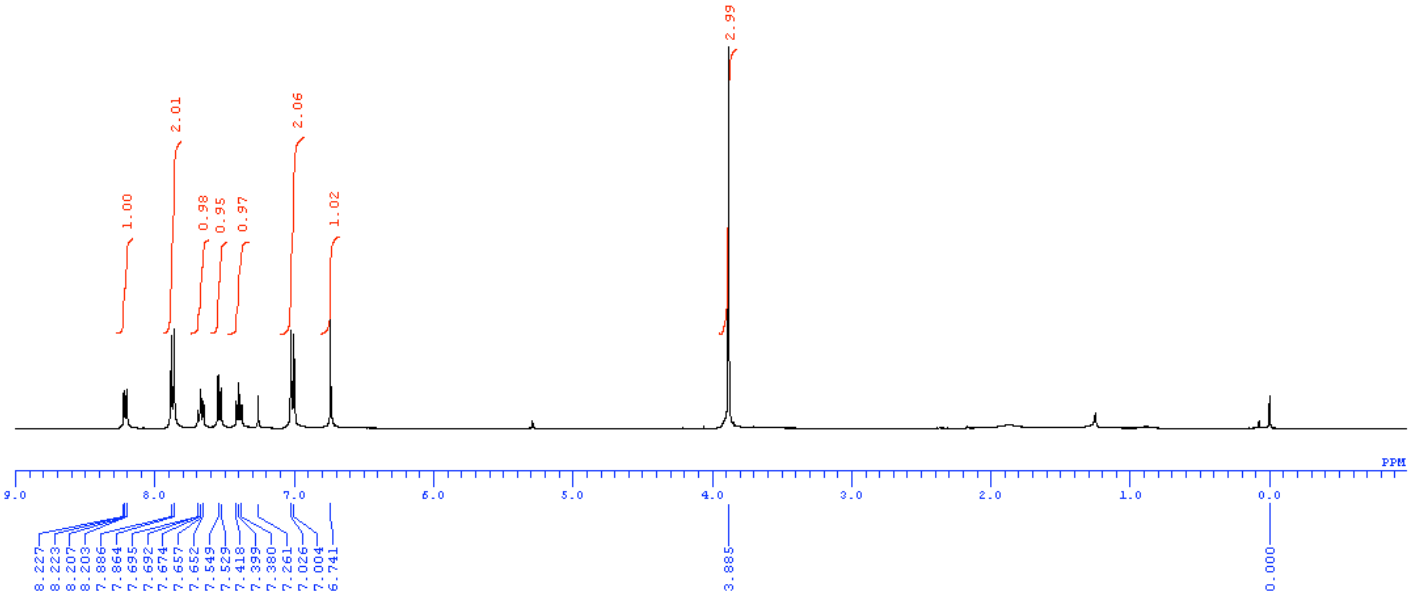


11b

^{1}H NMR ($CDCl_3$)



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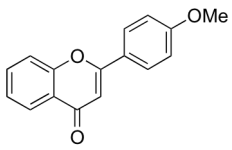
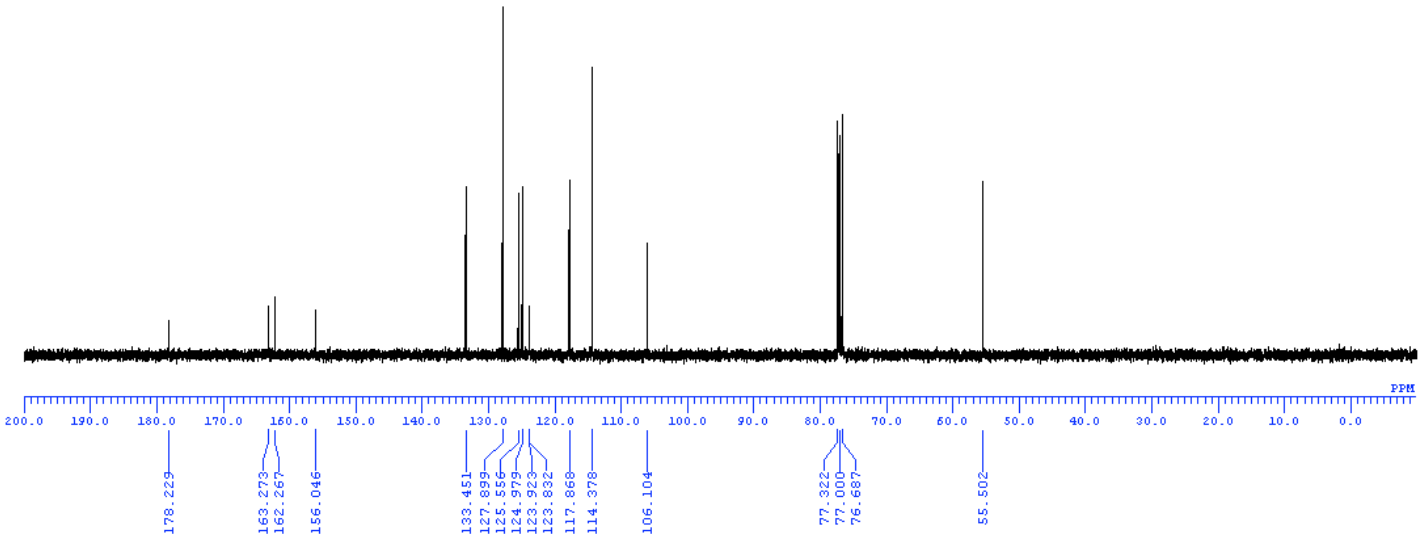


11b

^{13}C NMR ($CDCl_3$)



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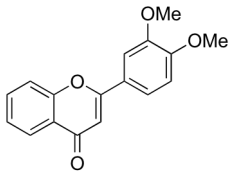
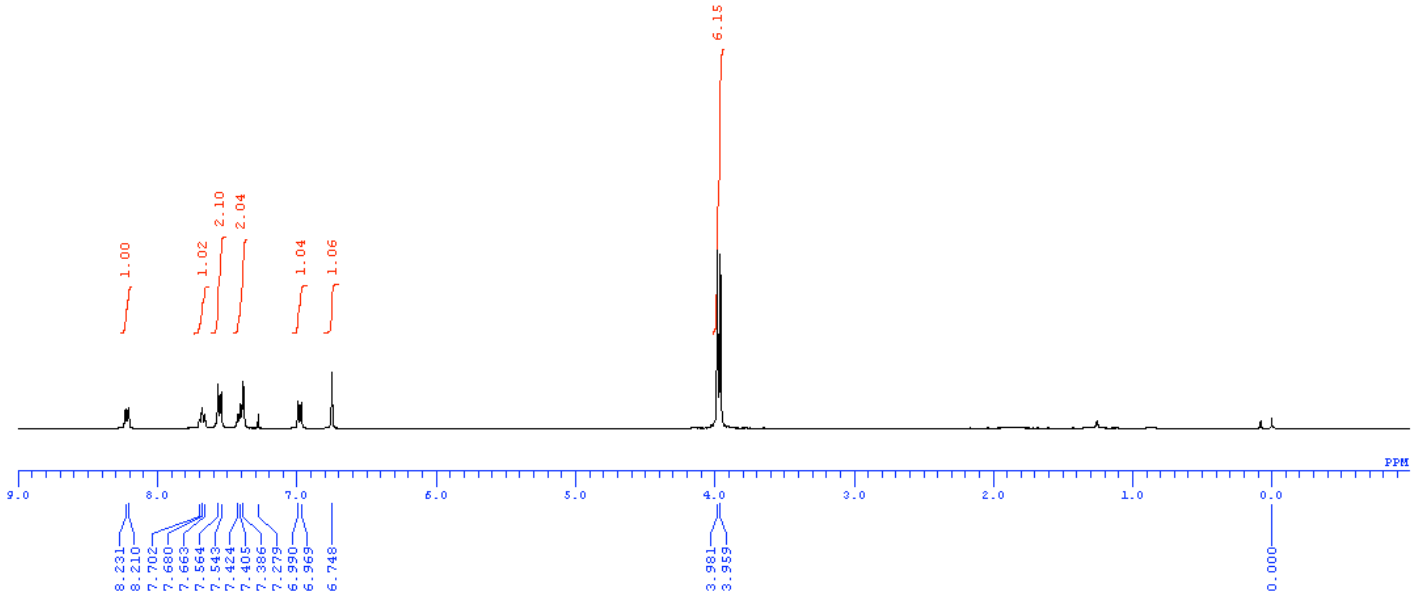


11c

^{1}H NMR ($CDCl_3$)



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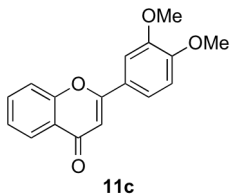
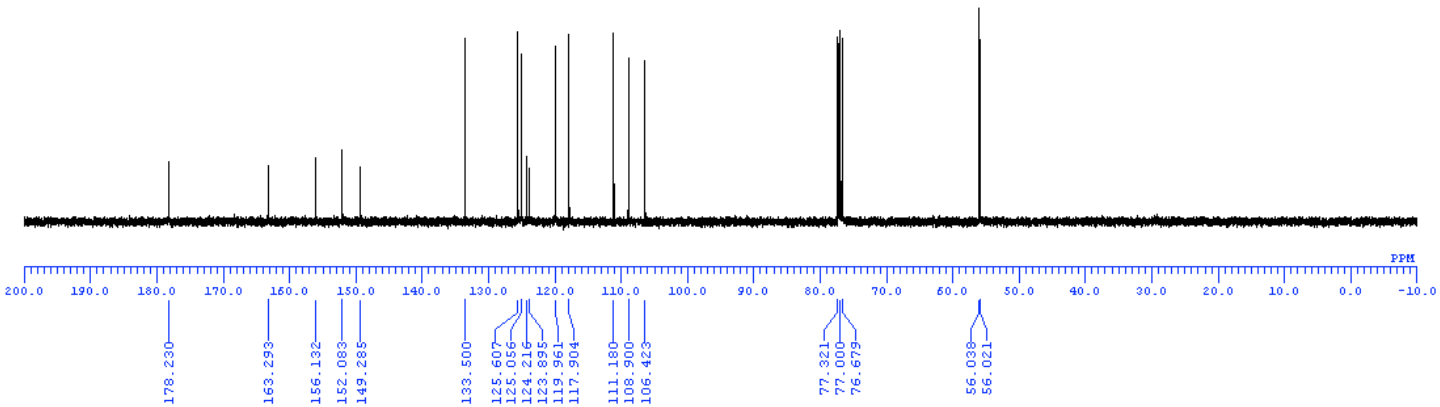


11c

^{13}C NMR ($CDCl_3$)



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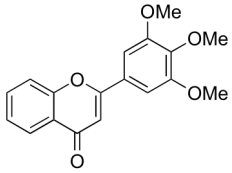
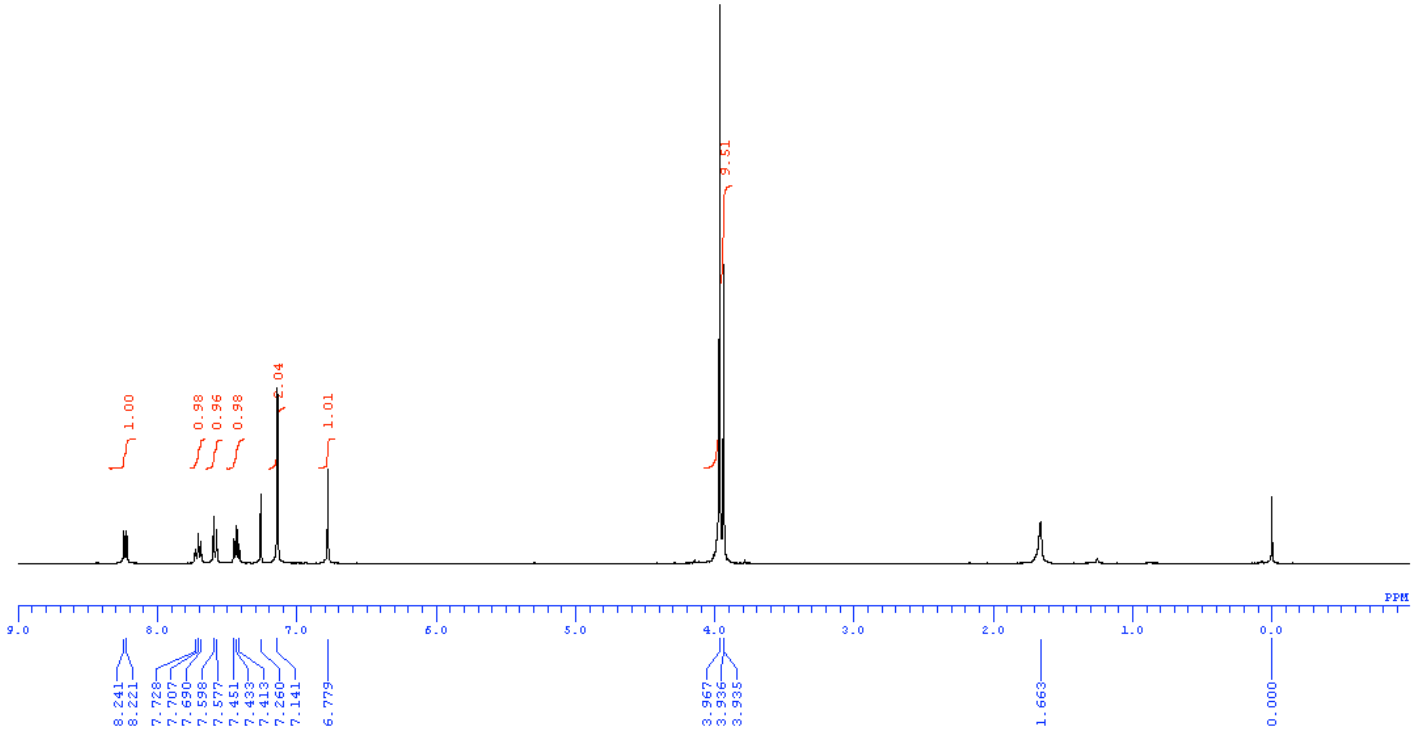


11d

^{1}H NMR ($CDCl_3$)



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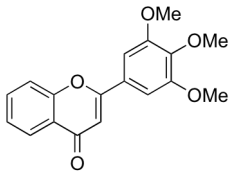
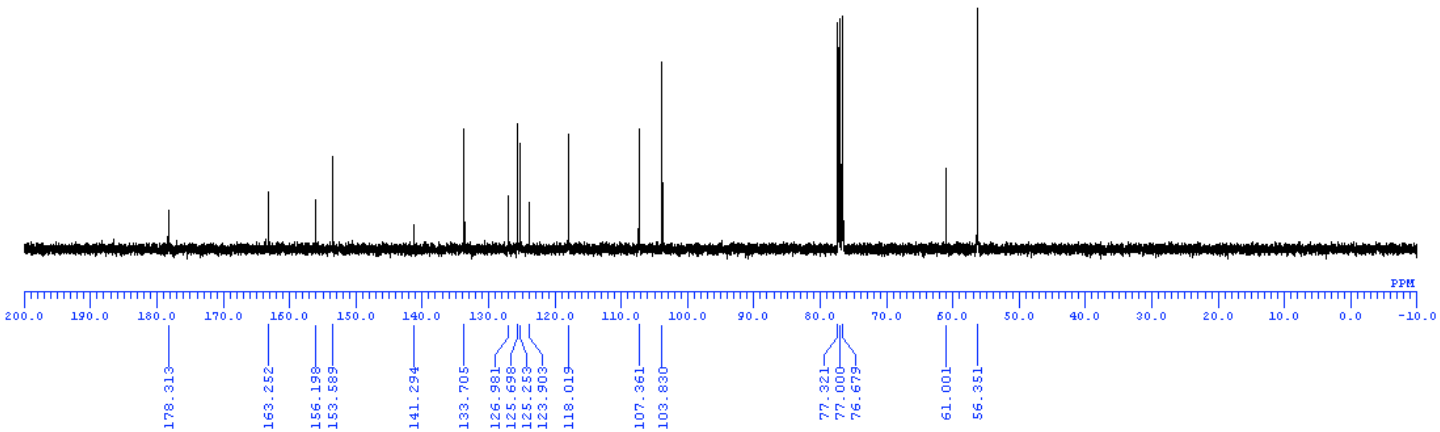


11d

^{13}C NMR ($CDCl_3$)



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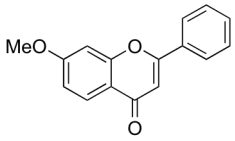
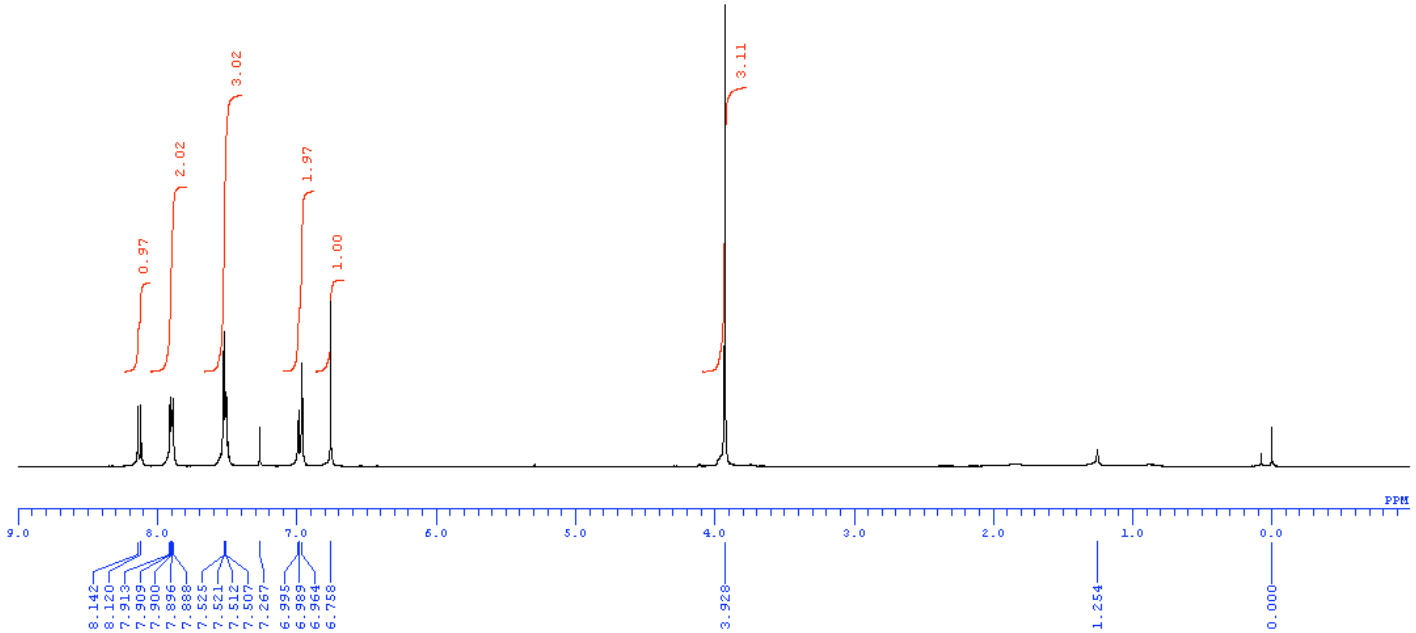


11e

^{1}H NMR ($CDCl_3$)



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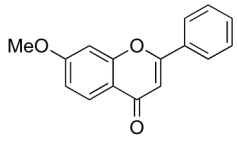
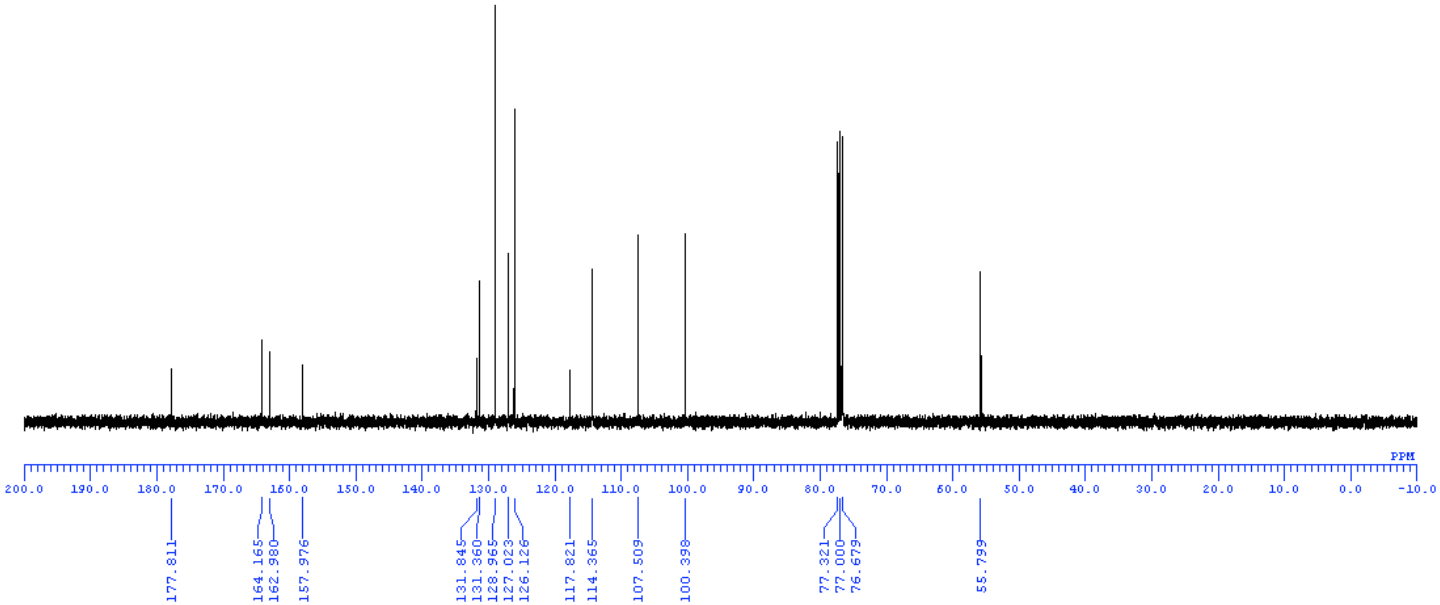


11e

^{13}C NMR ($CDCl_3$)



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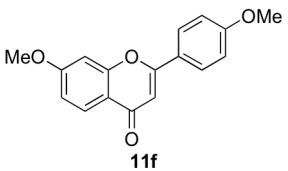
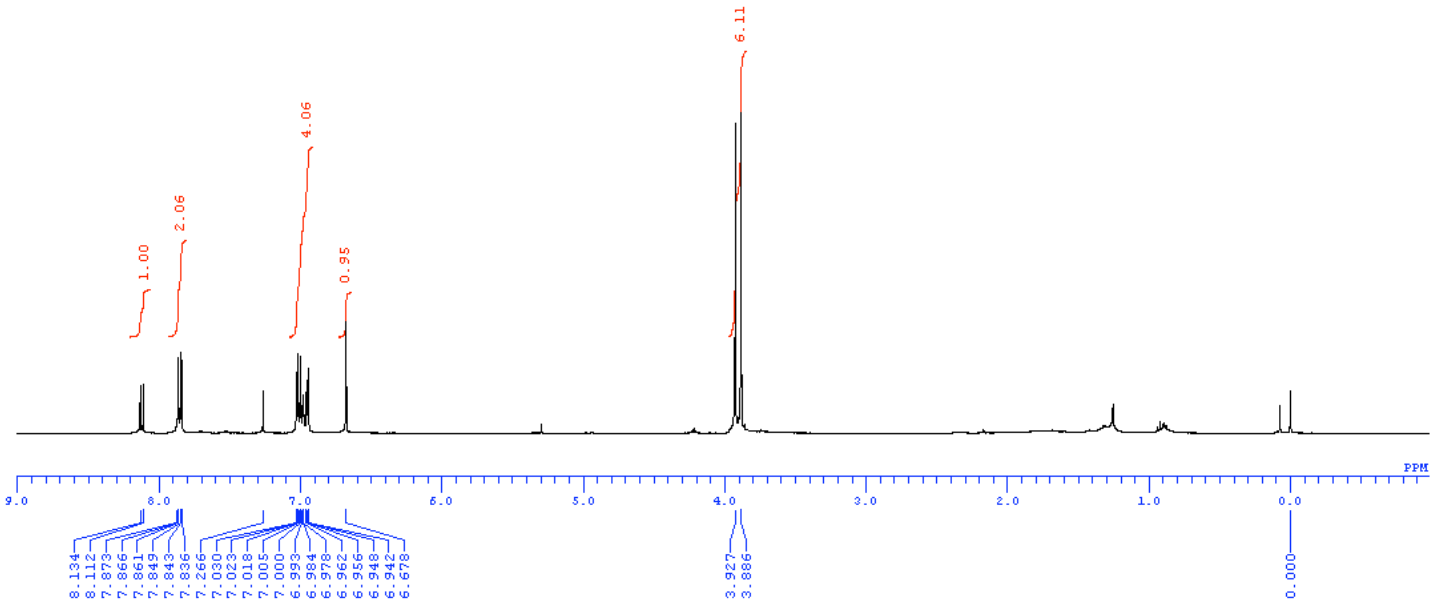


11f

^{1}H NMR ($CDCl_3$)



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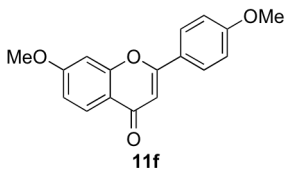
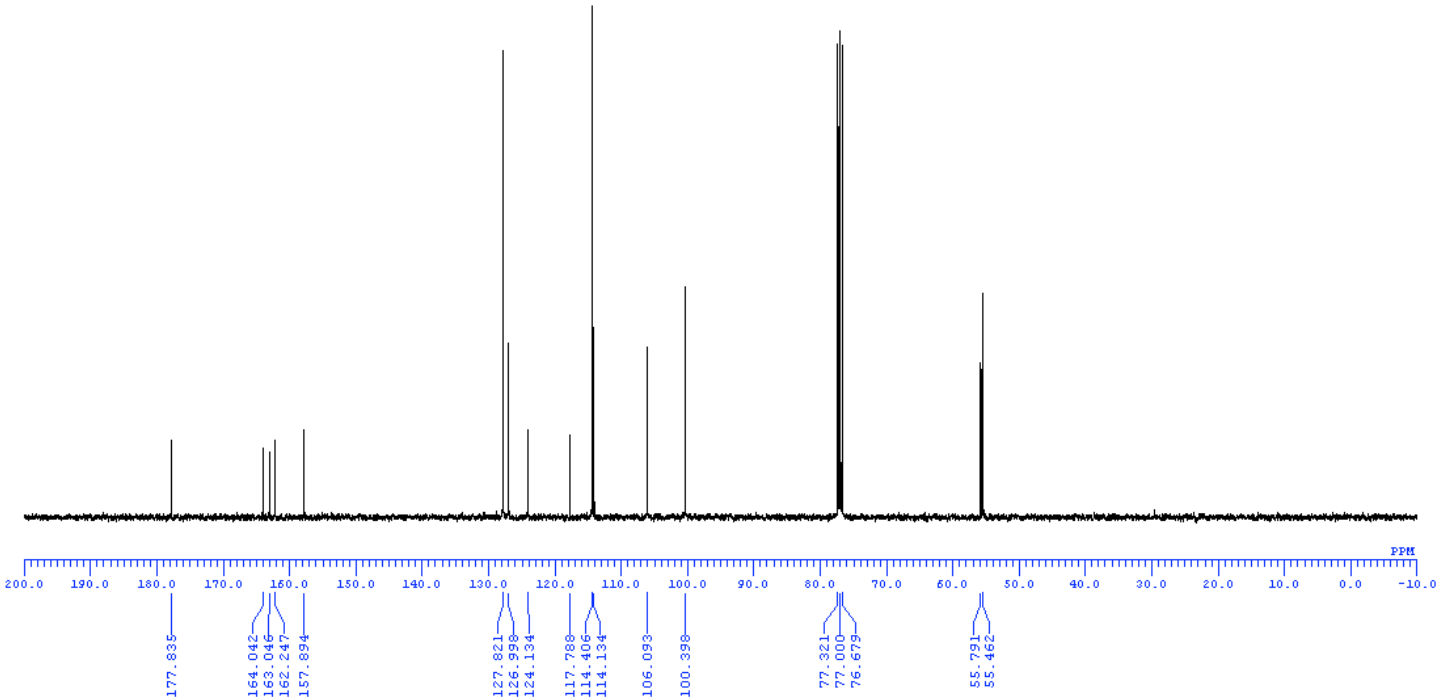


11f

^{13}C NMR ($CDCl_3$)



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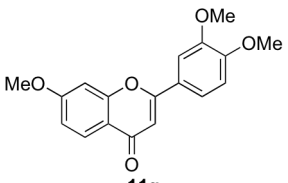
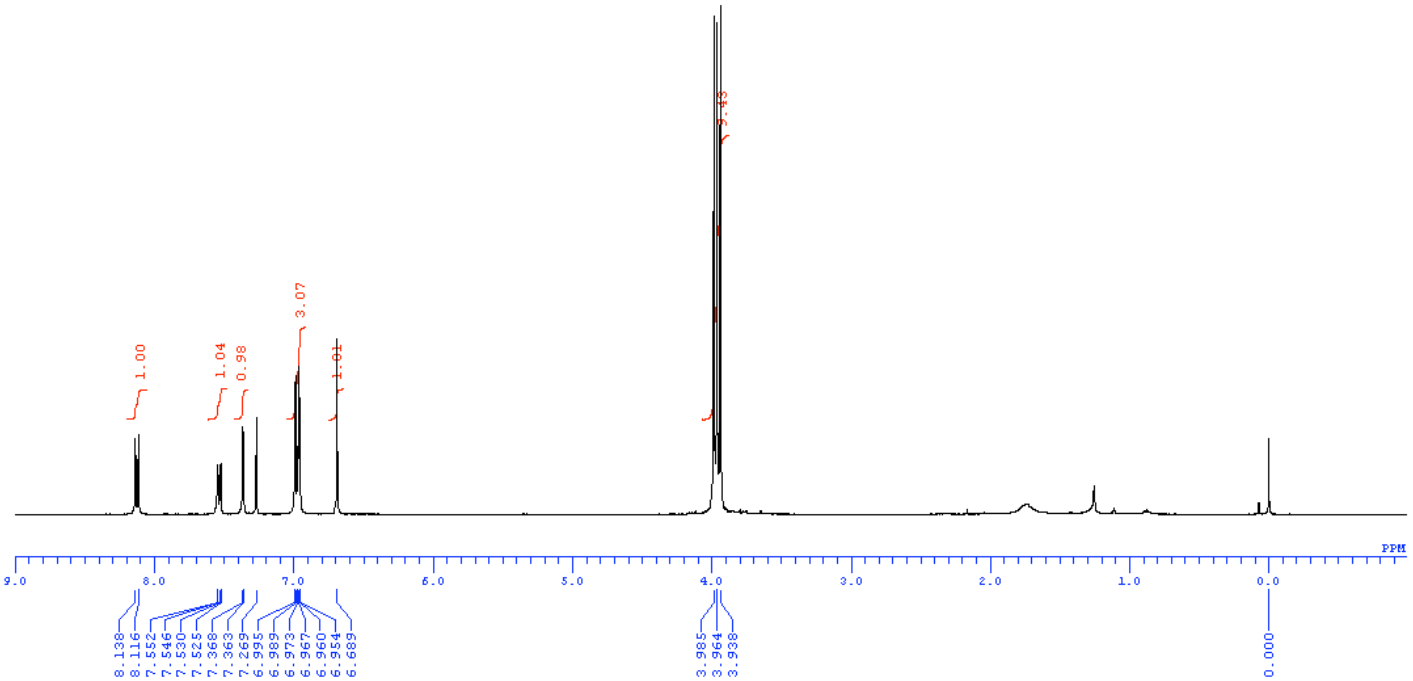


11g

^{1}H NMR ($CDCl_3$)



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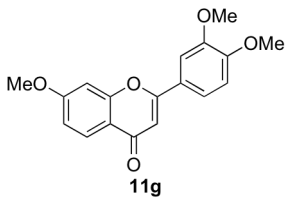
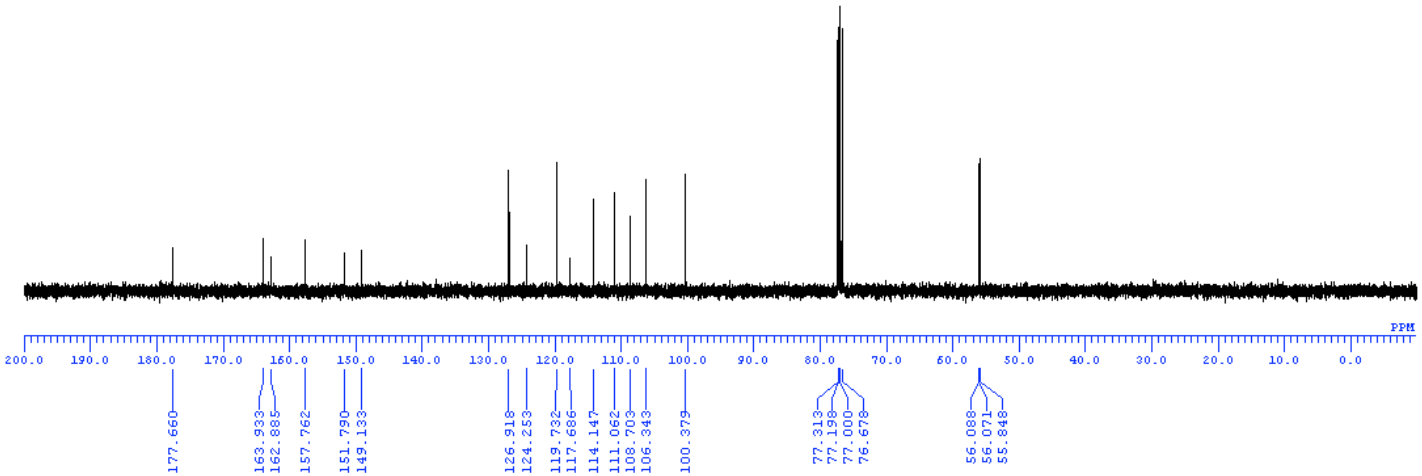
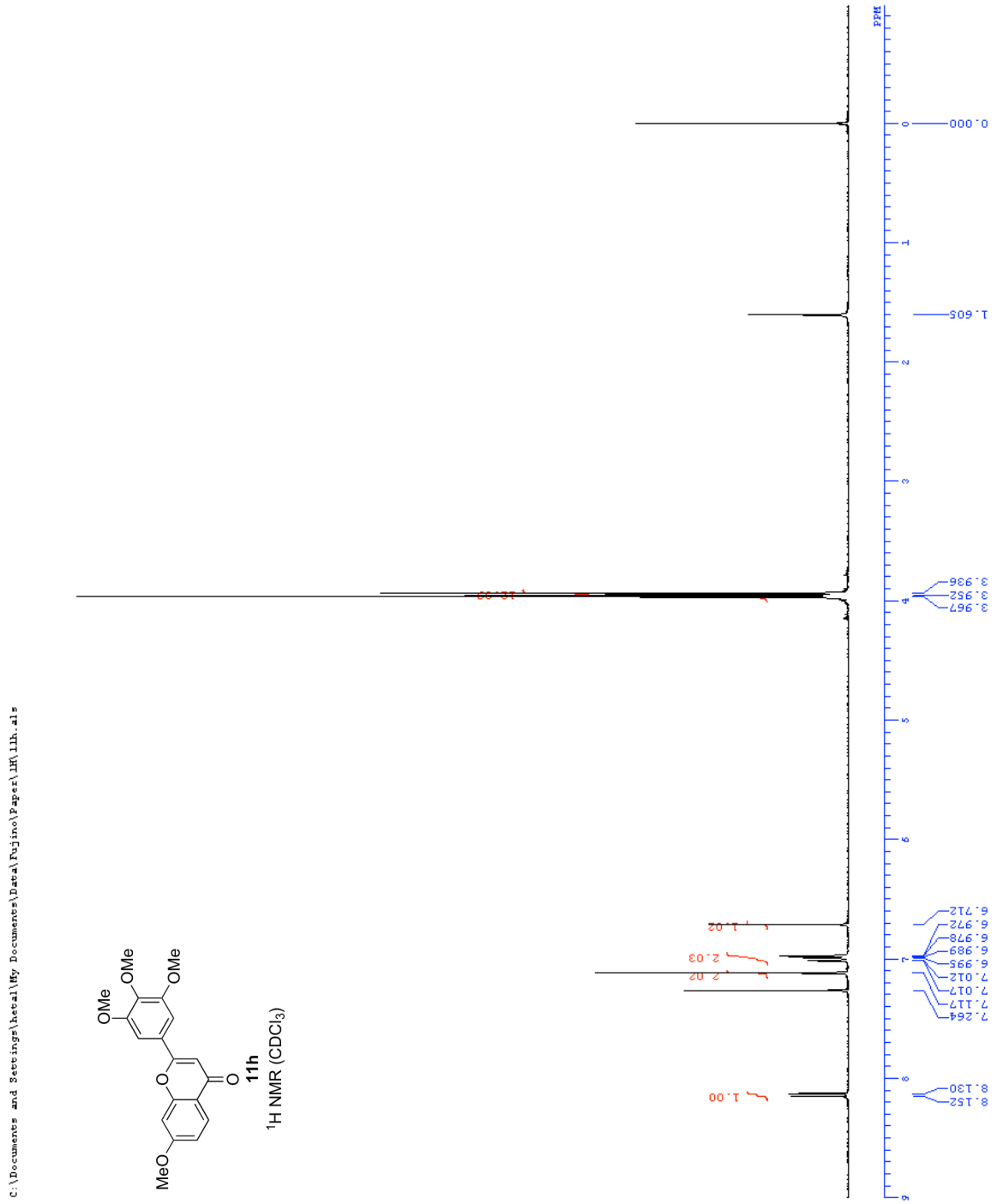


11g

^{13}C NMR ($CDCl_3$)




C:\Documents and Settings\hetal\My Documents\Data\Pujino\Paper\1H\11h.als
OMe
OMe
OMe
MeO
O
O
11h
1H NMR (CDCl3)
PPM
0.000
1.605
3.936
3.952
3.967
6.712
6.972
6.978
6.989
6.995
7.012
7.017
7.117
7.264
8.130
8.152
1.00
2.03
2.02
1.02

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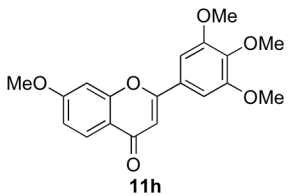
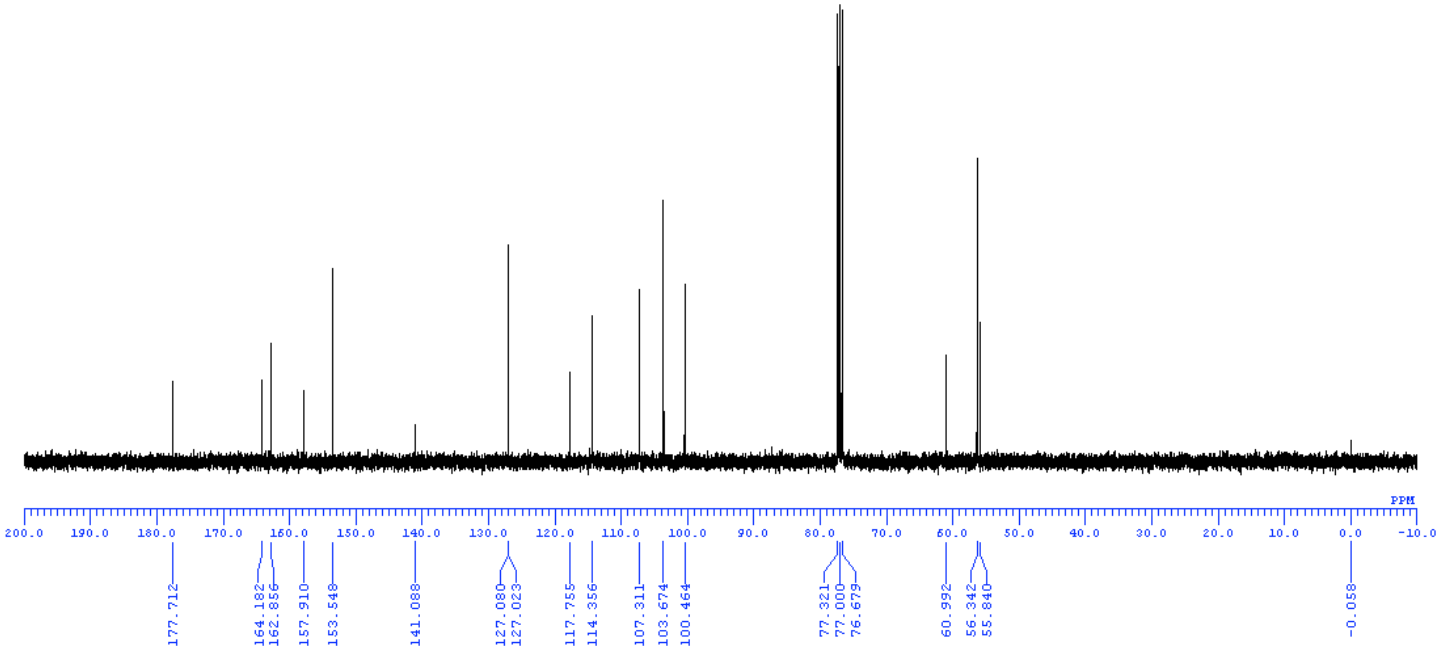


11h

^{13}C NMR ($CDCl_3$)



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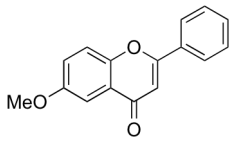
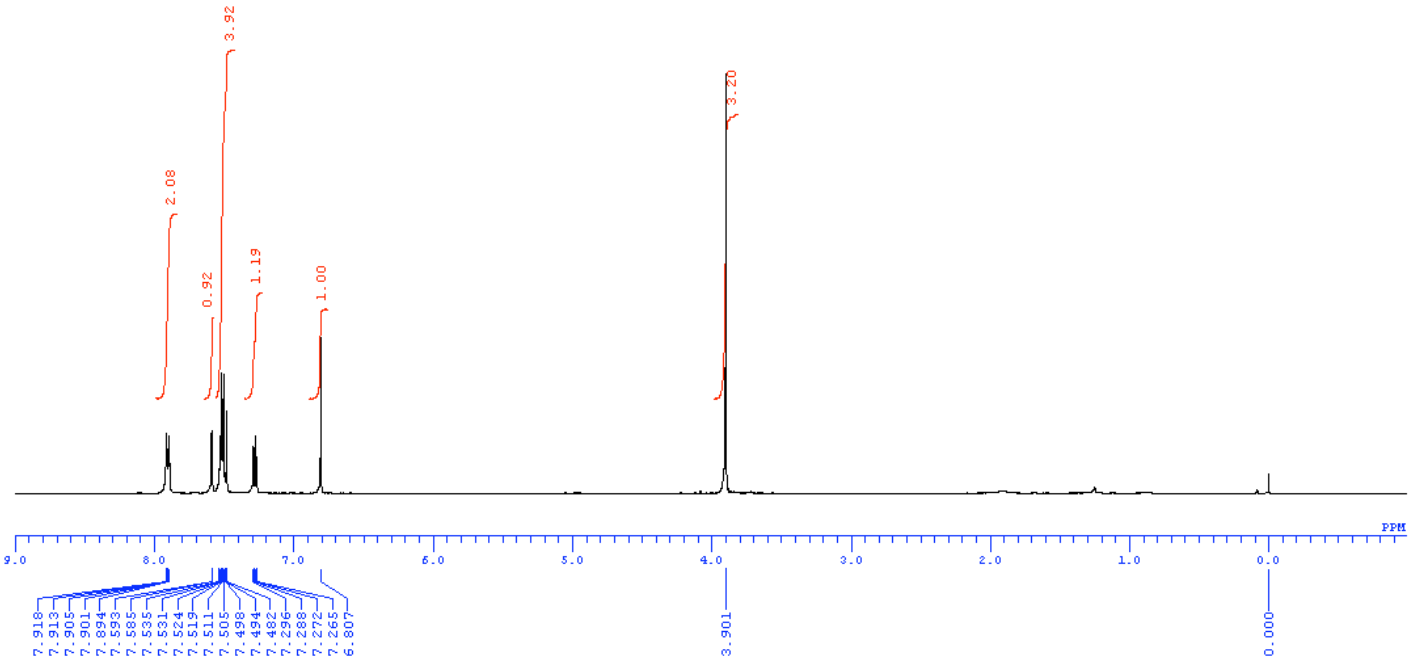


11i

^{1}H NMR ($CDCl_3$)



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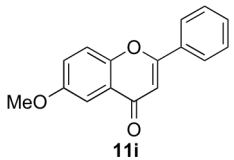
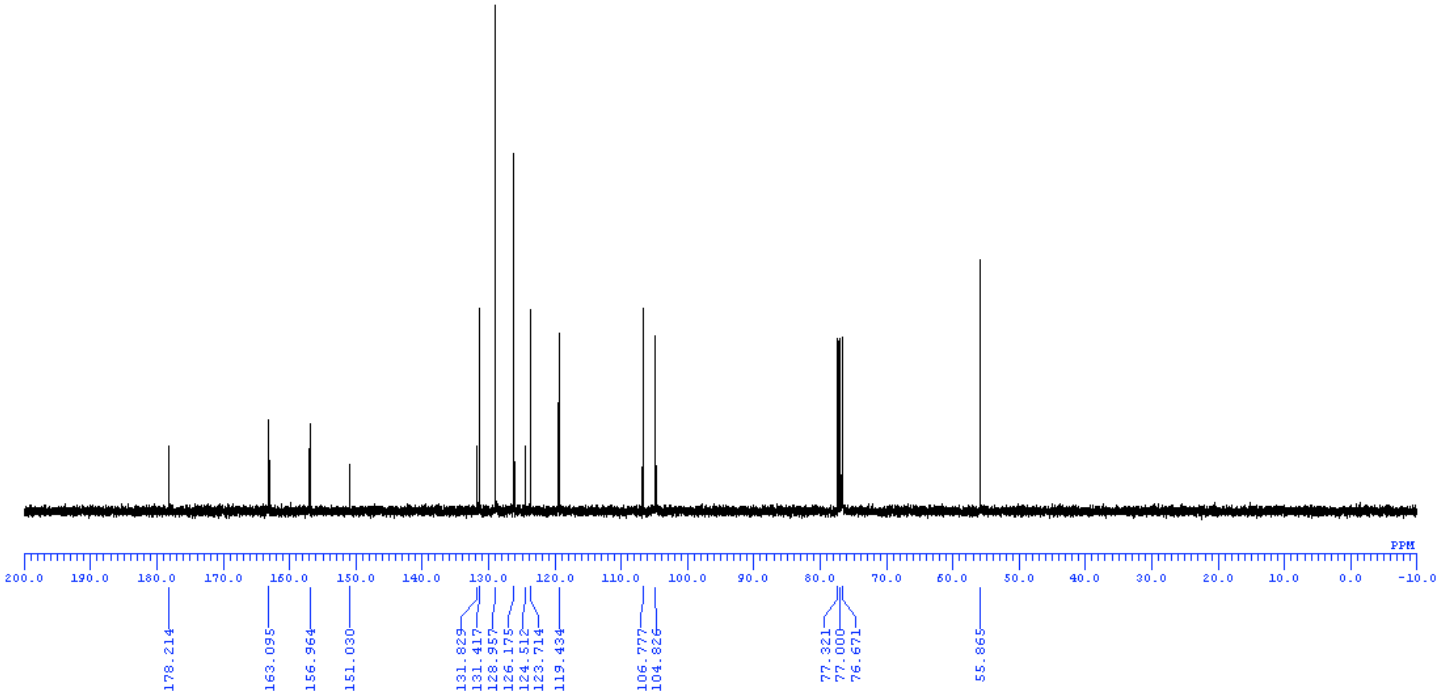
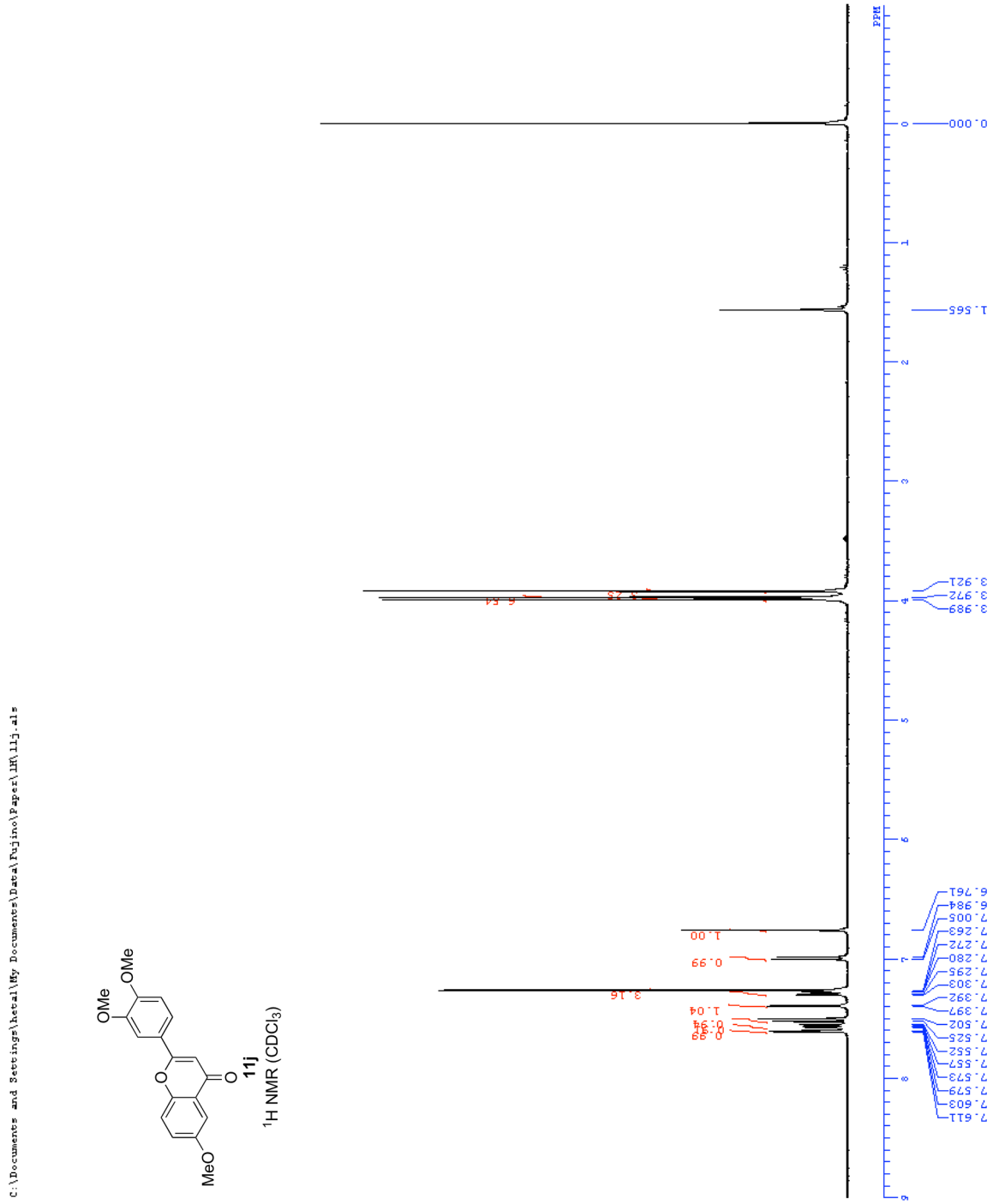


11i

^{13}C NMR ($CDCl_3$)




C:\Documents and Settings\hetal\My Documents\Data\Pujino\Paper\1H\11j.als
OMe
OMe
O
O
MeO
11j
1H NMR (CDCl3)
PPM
0.000
1.565
3.921
3.972
3.989
6.761
6.984
7.005
7.263
7.272
7.280
7.295
7.303
7.392
7.397
7.502
7.525
7.552
7.557
7.573
7.579
7.603
7.611
1.00
0.99
3.16
1.04
0.89
0.94

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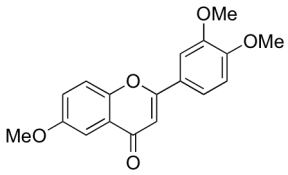
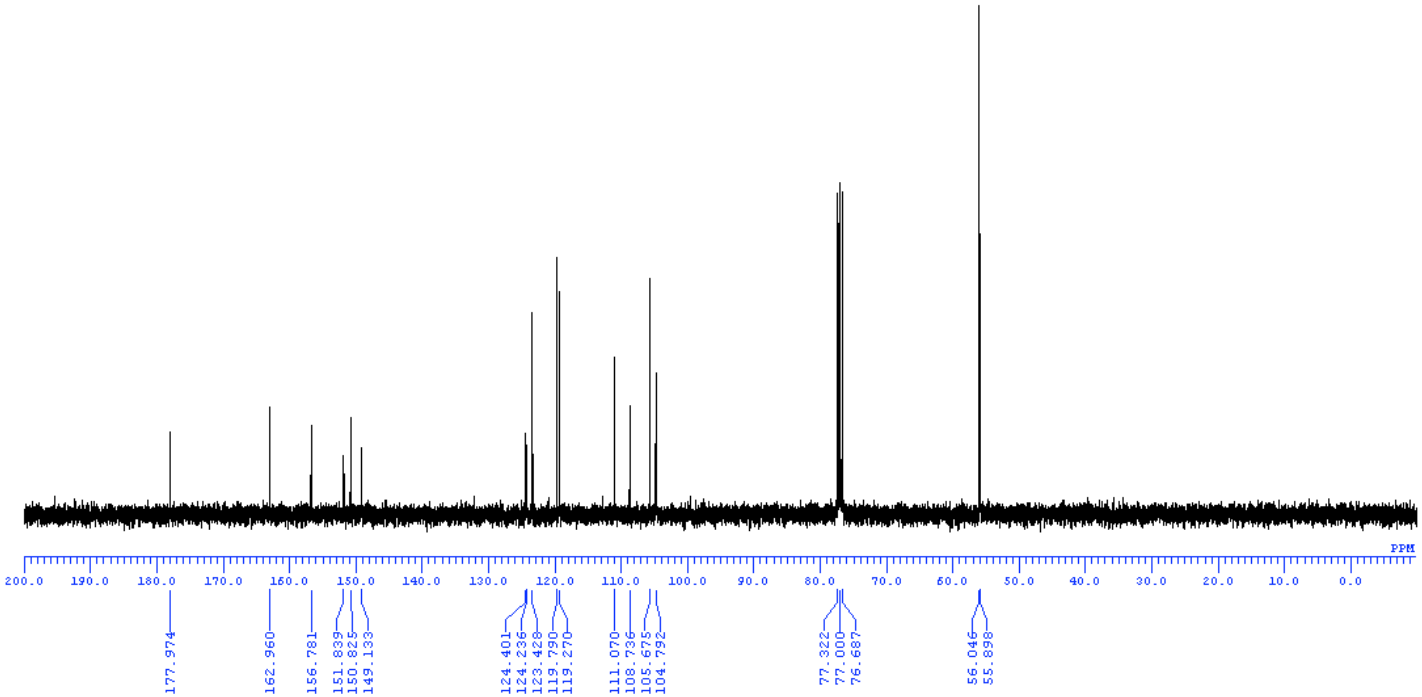


11j

^{13}C NMR ($CDCl_3$)



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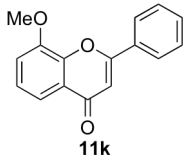
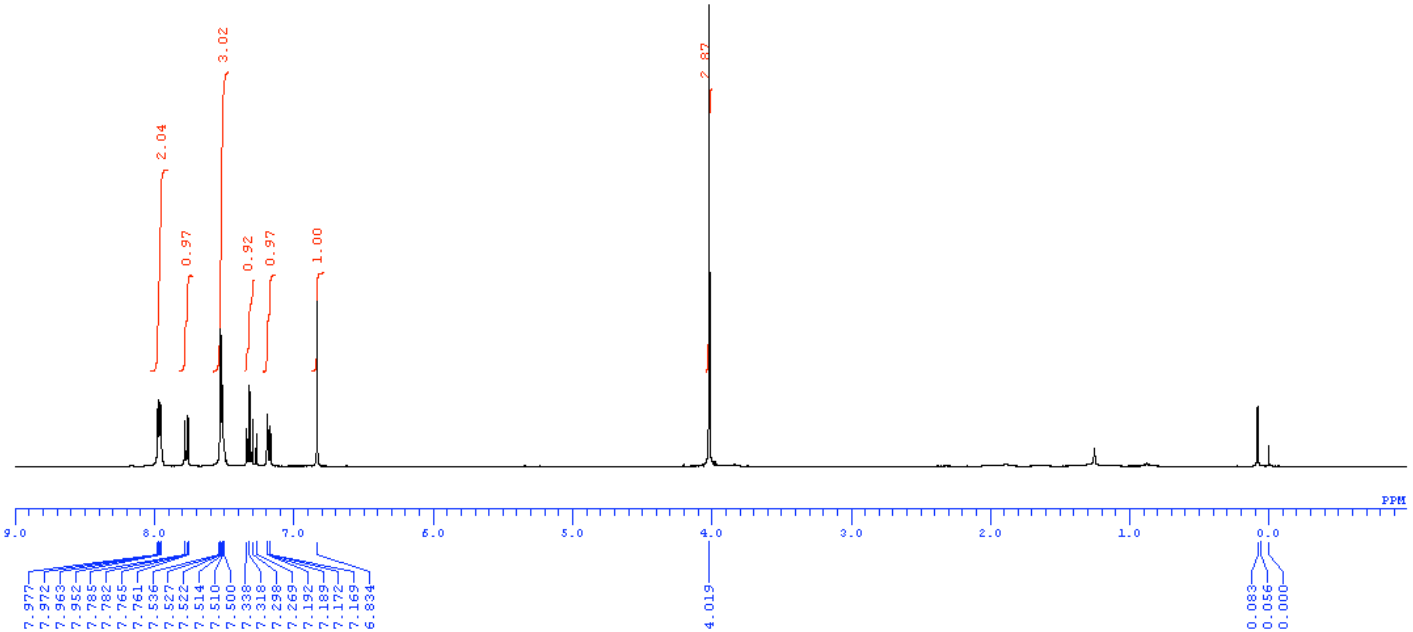


11k

^{1}H NMR ($CDCl_3$)



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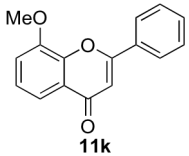
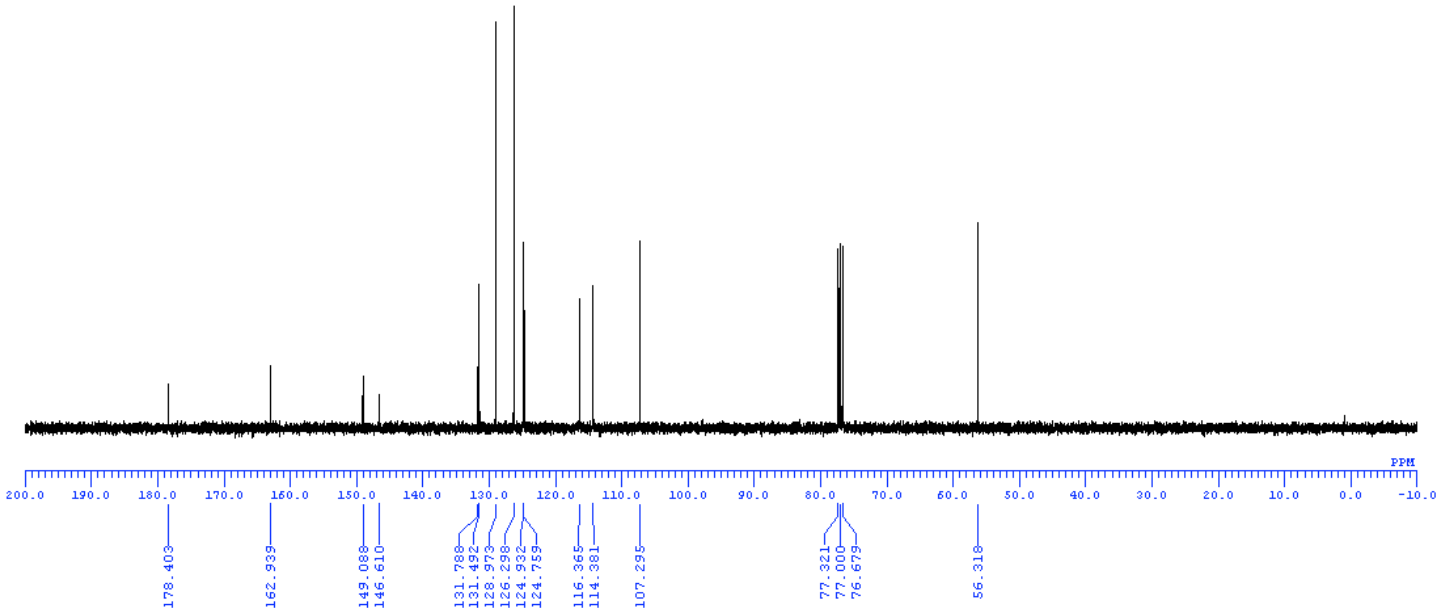
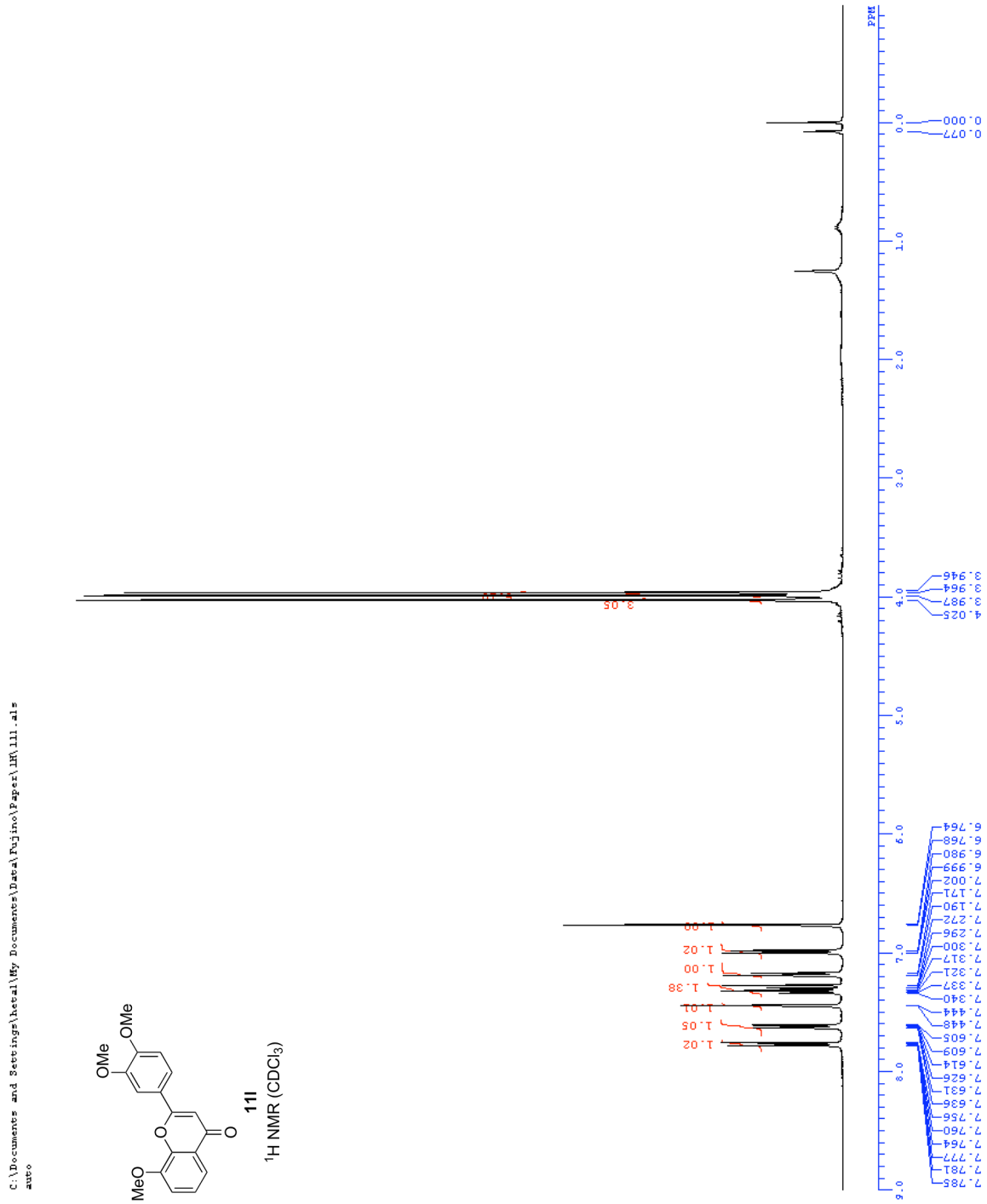


11k

^{13}C NMR ($CDCl_3$)




C:\Documents and Settings\hetal\My Documents\Data\Fujino\Paper\1H\111.als
auto
OMe
OMe
MeO
O
O
11l
1H NMR (CDCl3)
3.05
1.02
1.05
1.01
1.38
1.00
1.02
1.00
4.025
3.987
3.964
3.946
0.077
0.000
PPM

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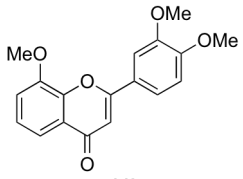
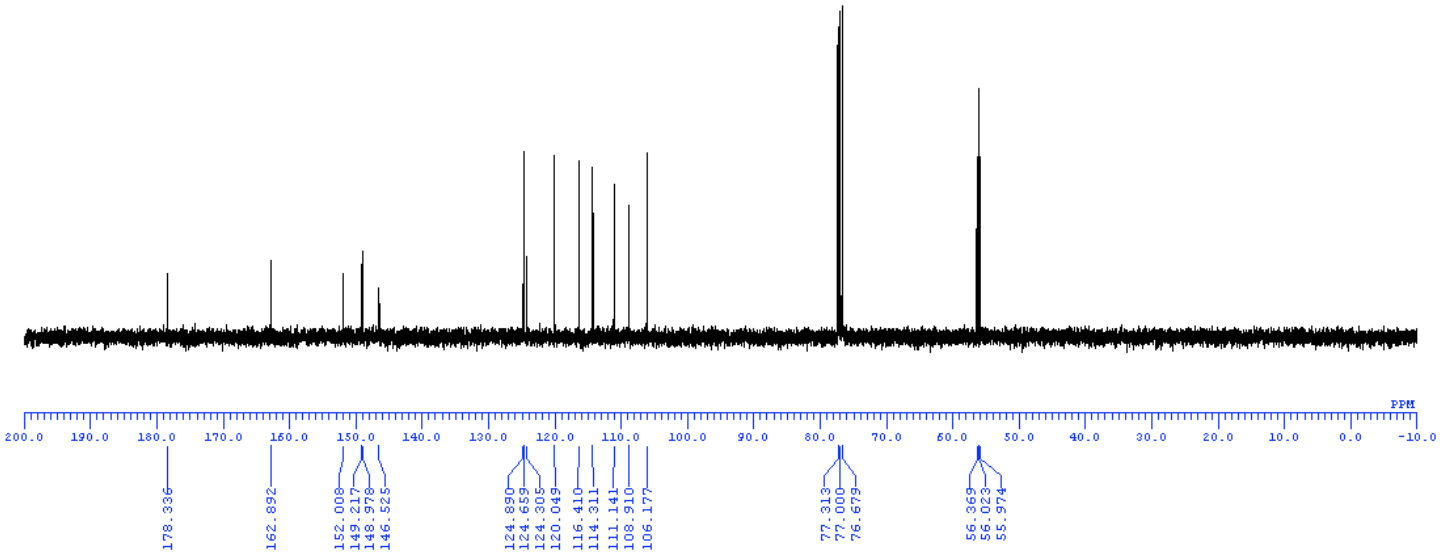


11l

^{13}C NMR ($CDCl_3$)



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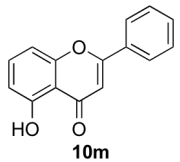
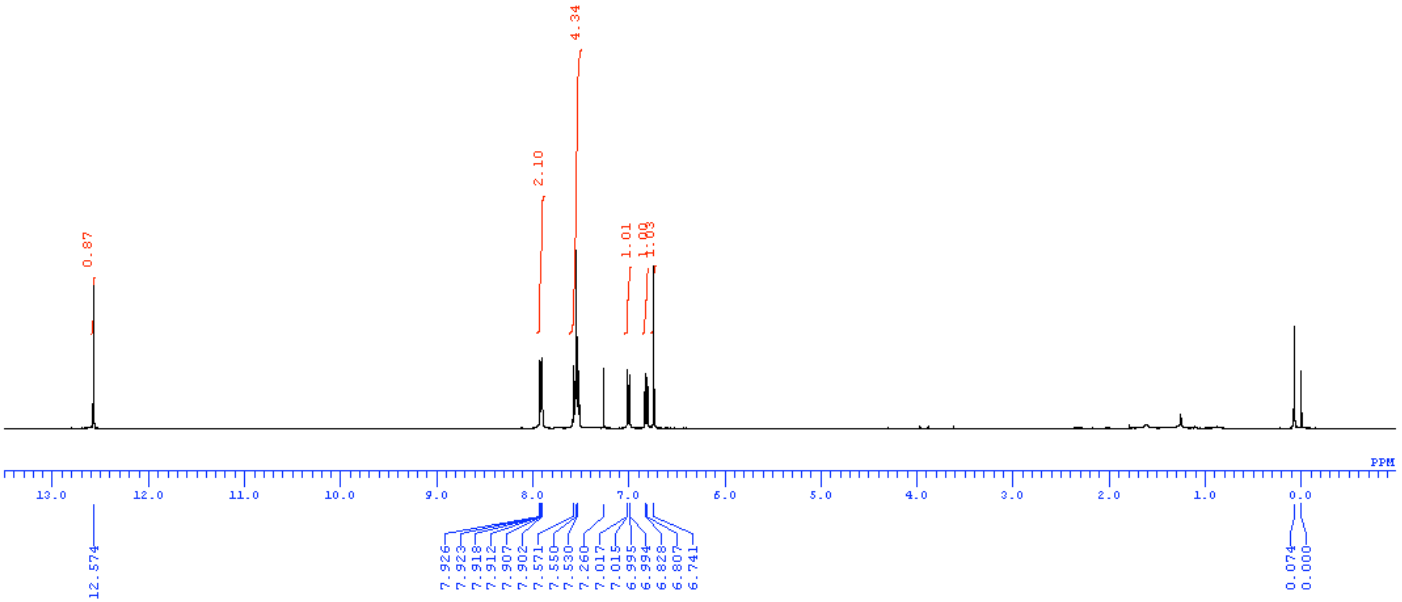


10m

^{1}H NMR ($CDCl_3$)



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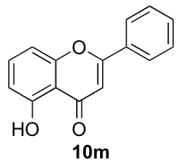
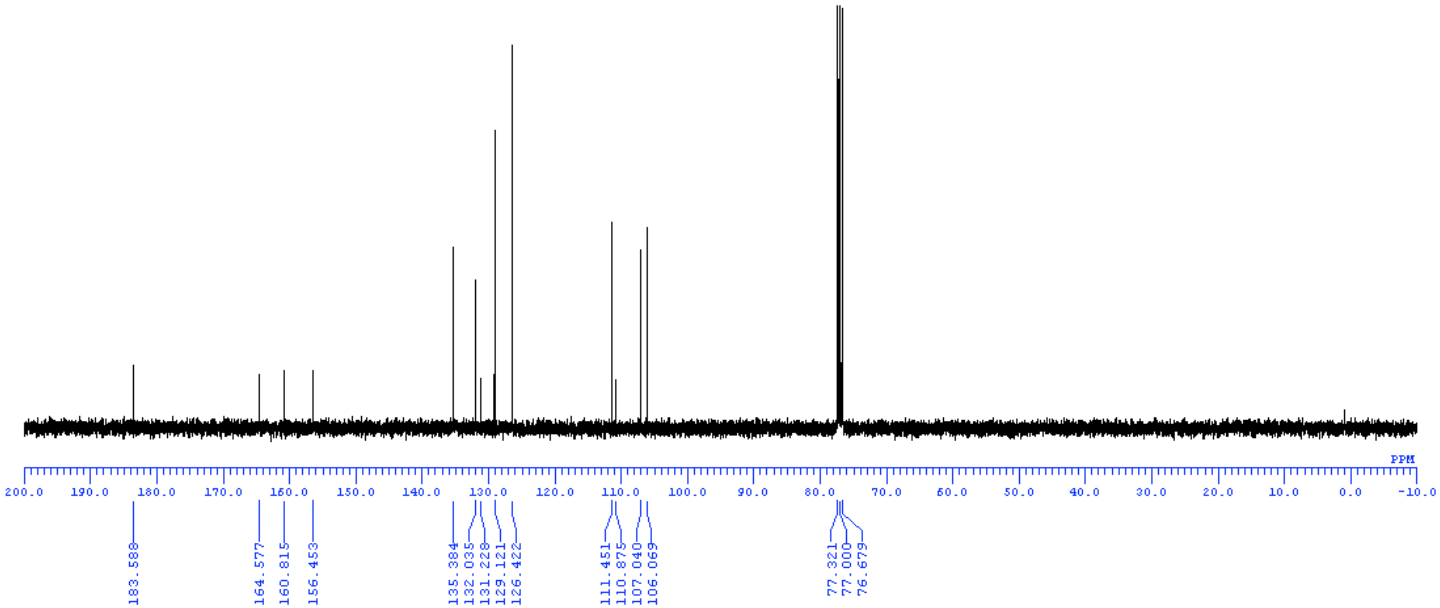


10m

^{13}C NMR ($CDCl_3$)



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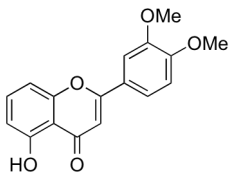
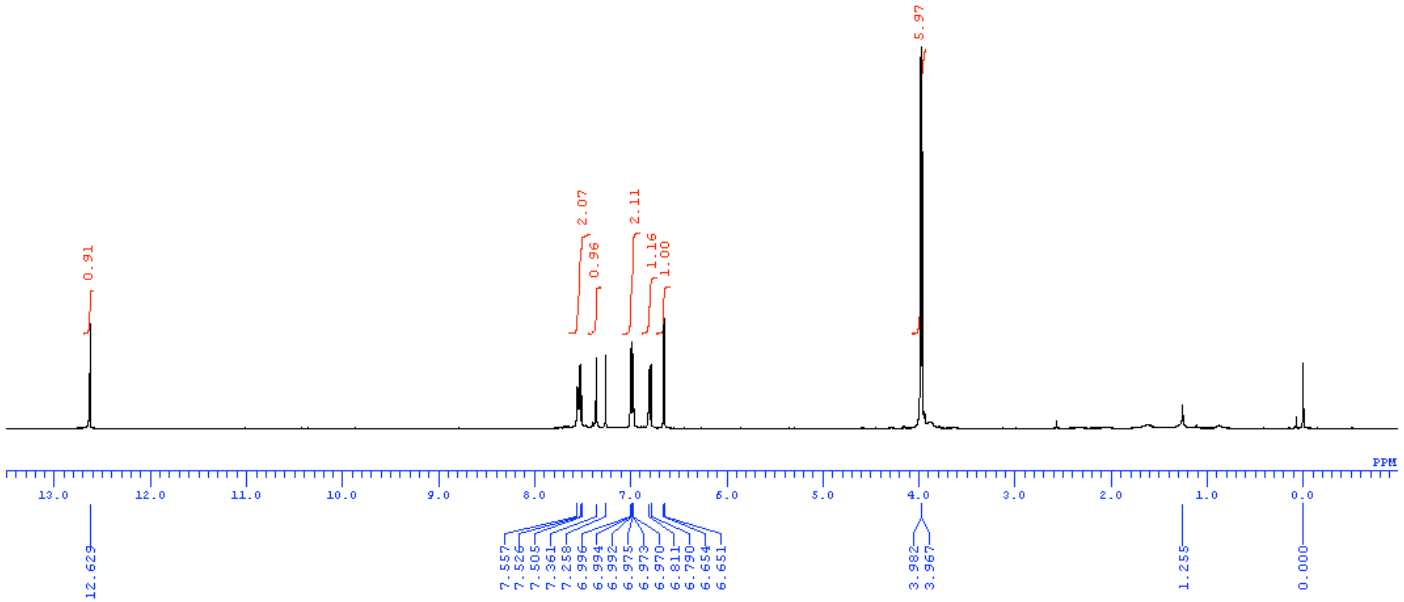


11n

^{1}H NMR ($CDCl_3$)



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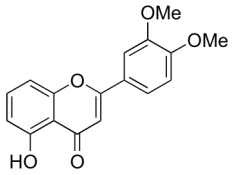
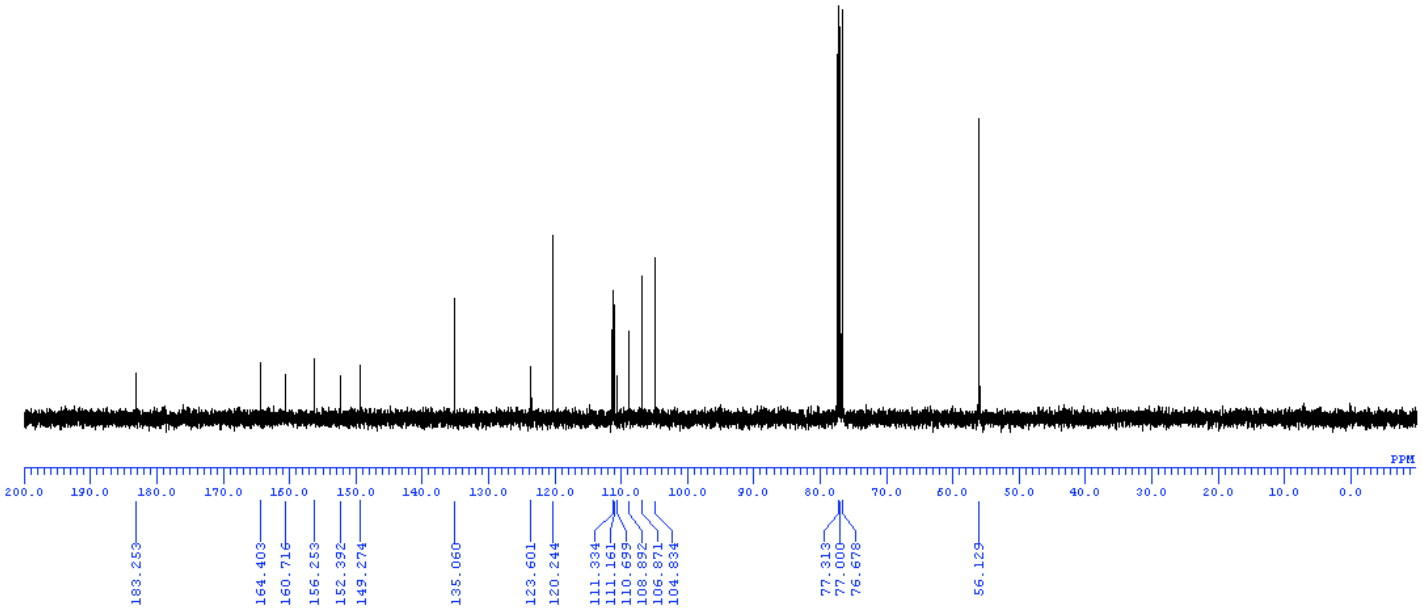


11n

^{13}C NMR ($CDCl_3$)



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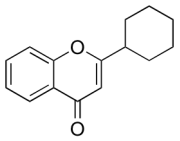
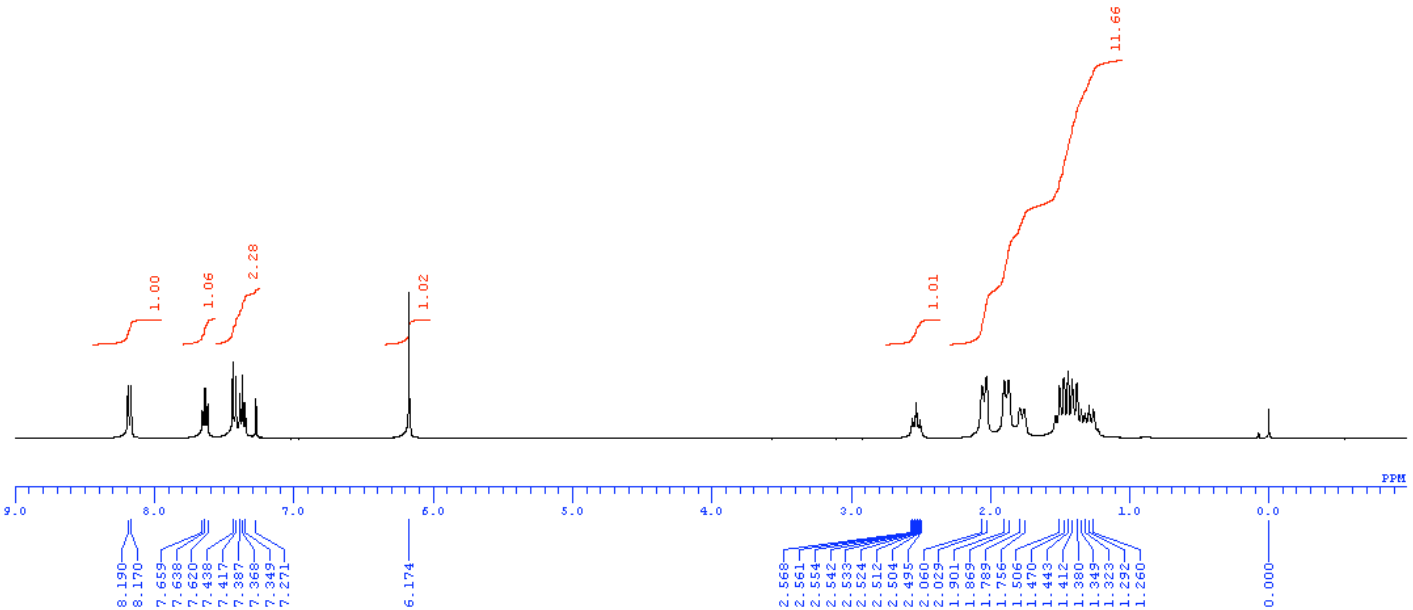


11o

^{1}H NMR ($CDCl_3$)



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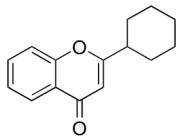
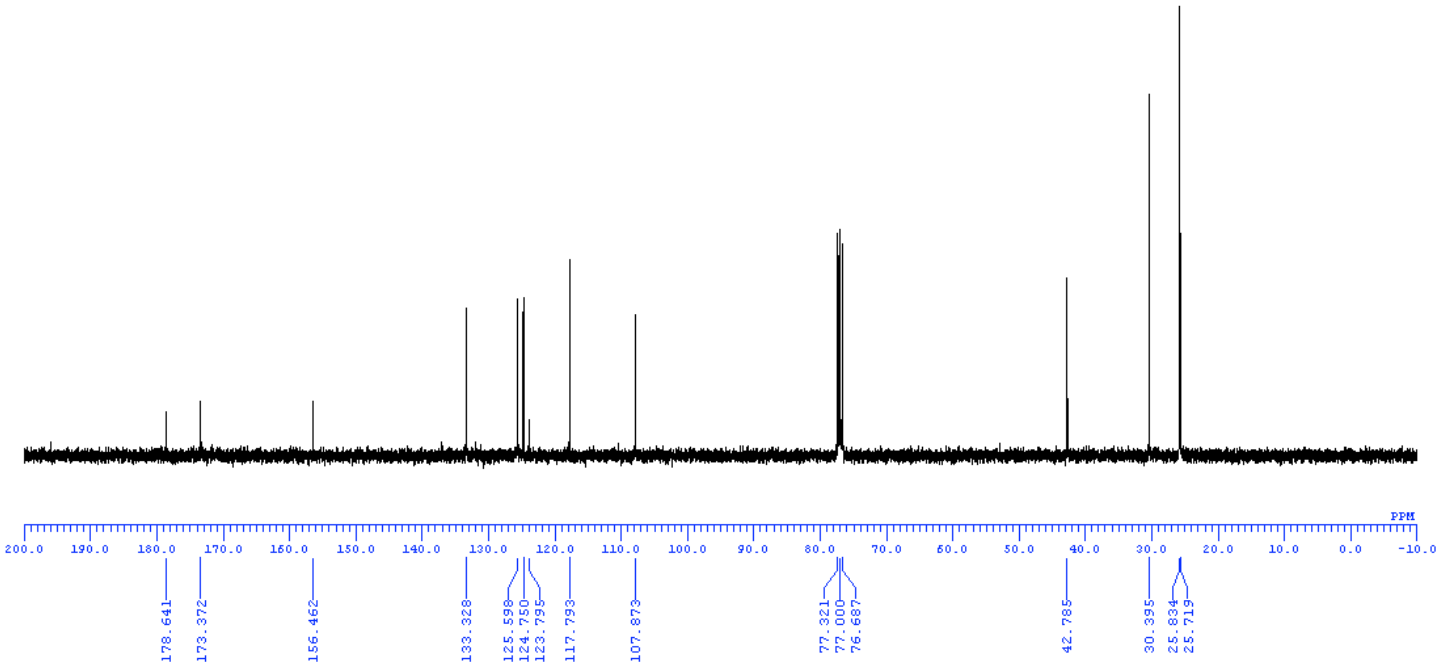
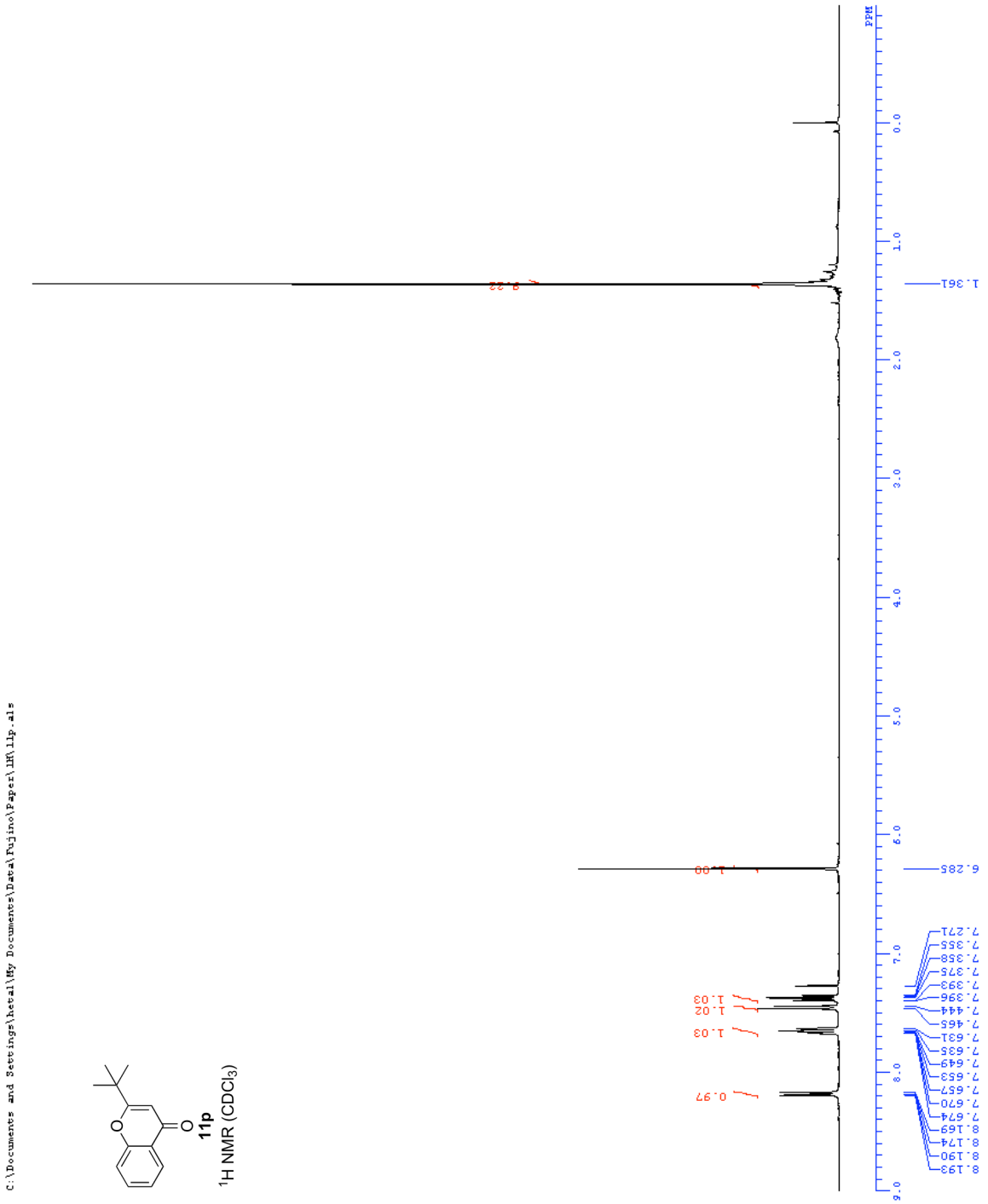


11o

^{13}C NMR ($CDCl_3$)

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11p
1H NMR (CDCl3)
PPM
0.0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
1.361
6.285
7.271
7.355
7.358
7.375
7.393
7.396
7.444
7.465
7.631
7.635
7.649
7.653
7.657
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7.674
8.169
8.174
8.190
8.193
0.97
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1.02
1.03
1.00
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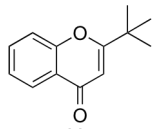
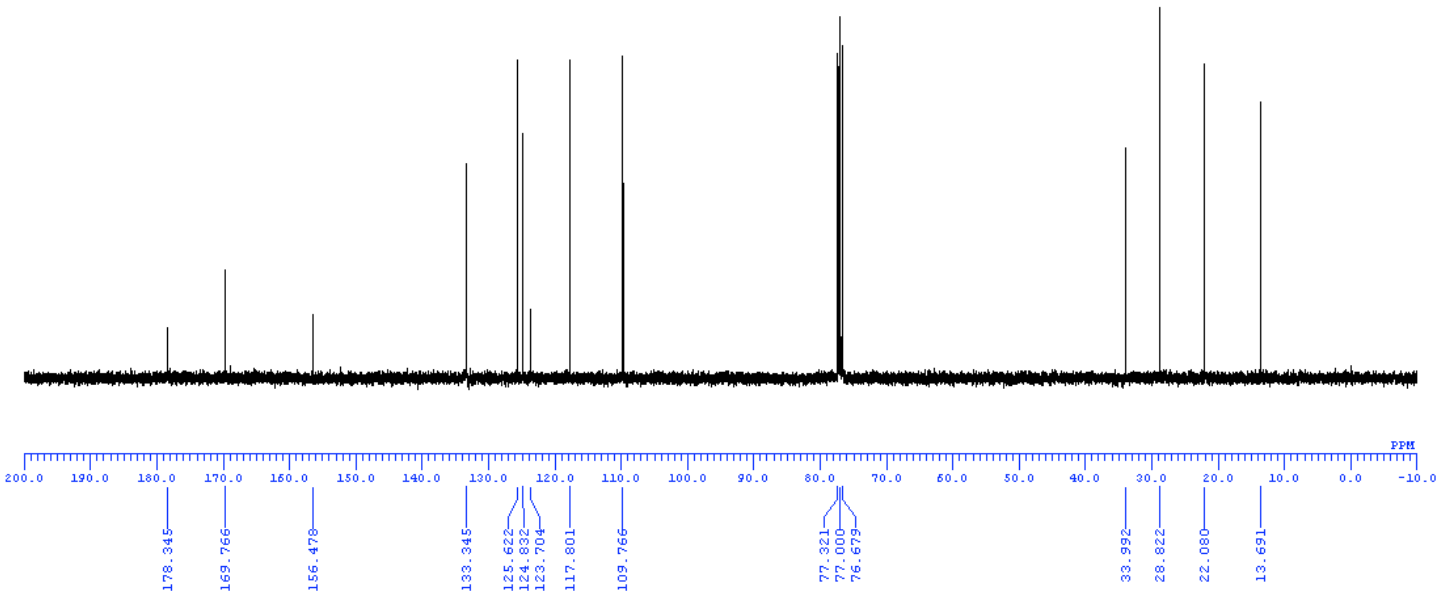


11p

^{13}C NMR ($CDCl_3$)



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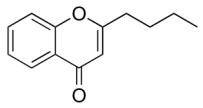
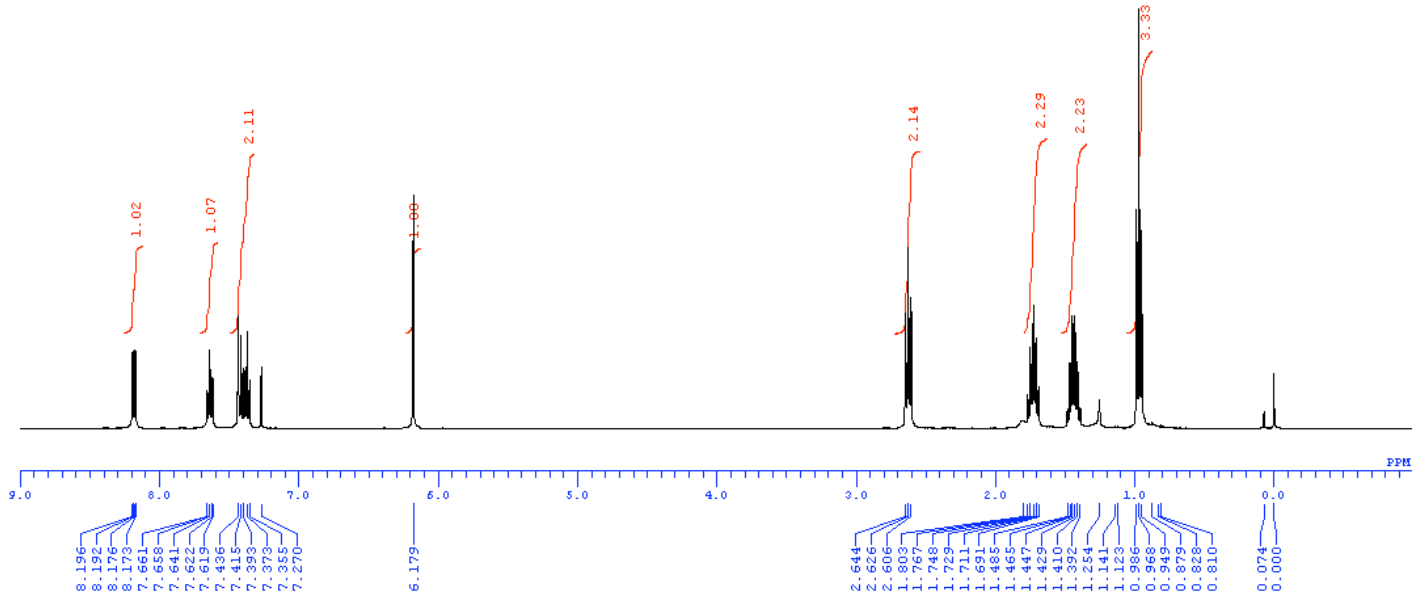
YS-787-crude



11q

^{1}H NMR ($CDCl_3$)



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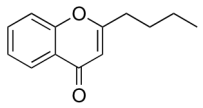
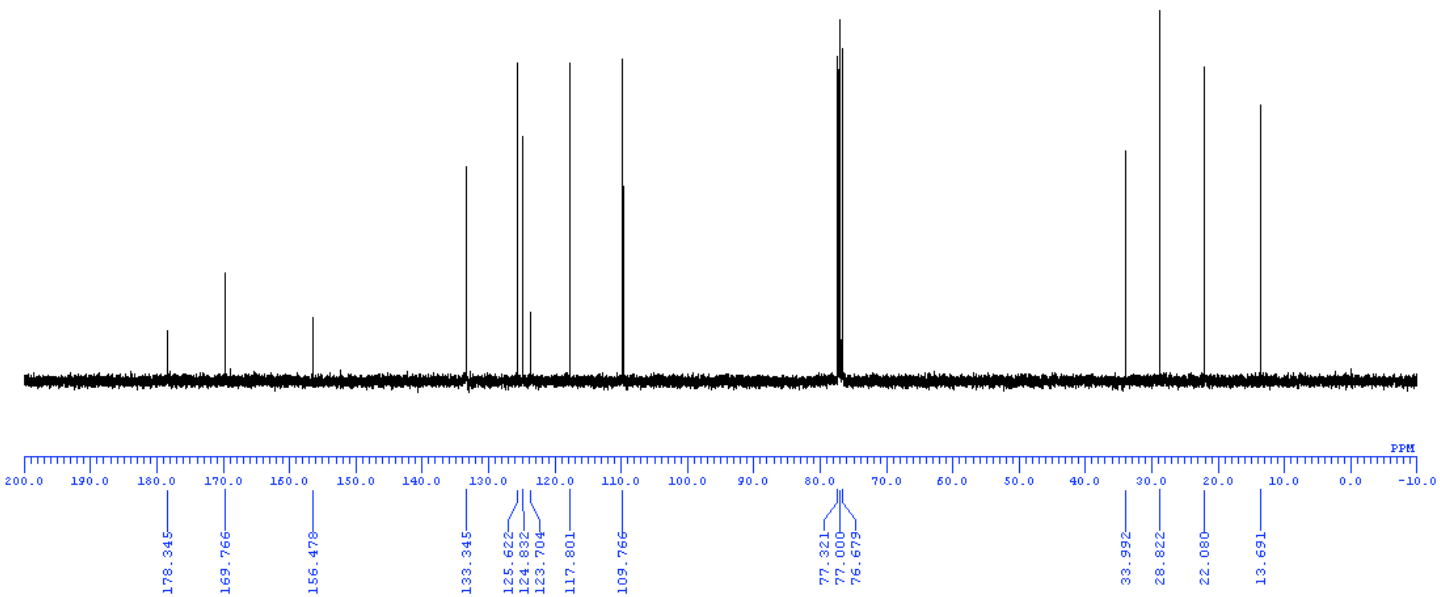


11q

^{13}C NMR ($CDCl_3$)



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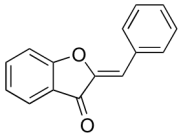
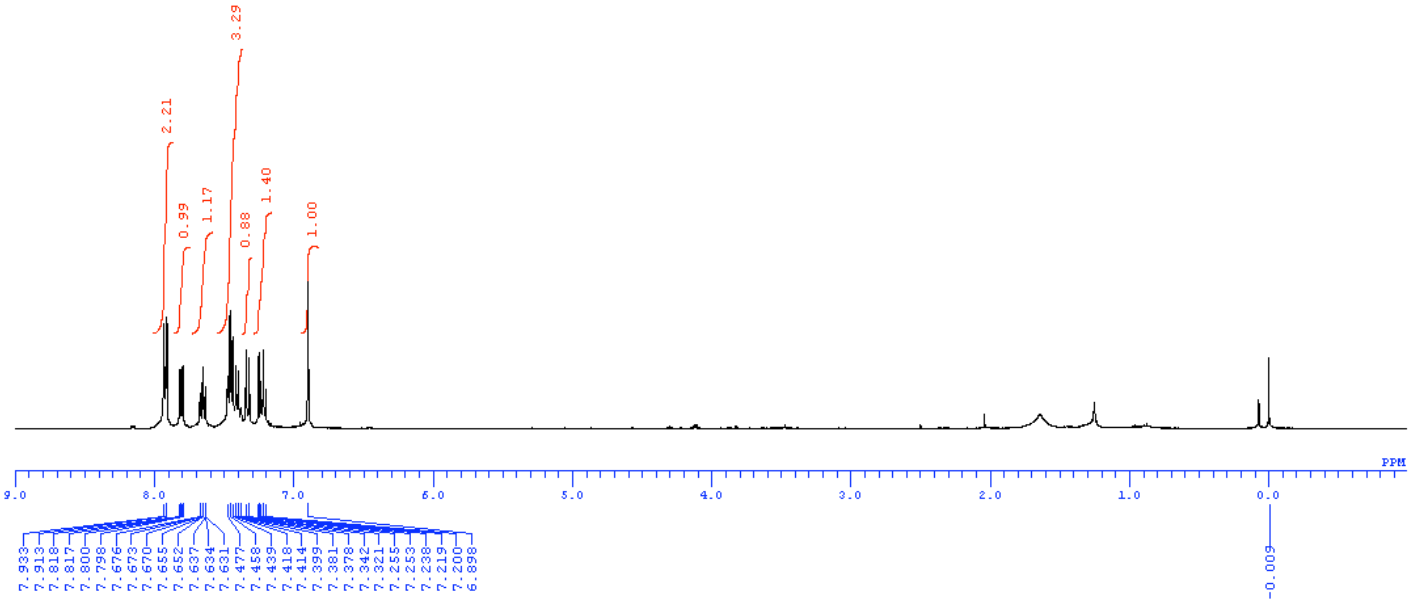


12a

^{1}H NMR ($CDCl_3$)



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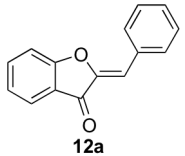
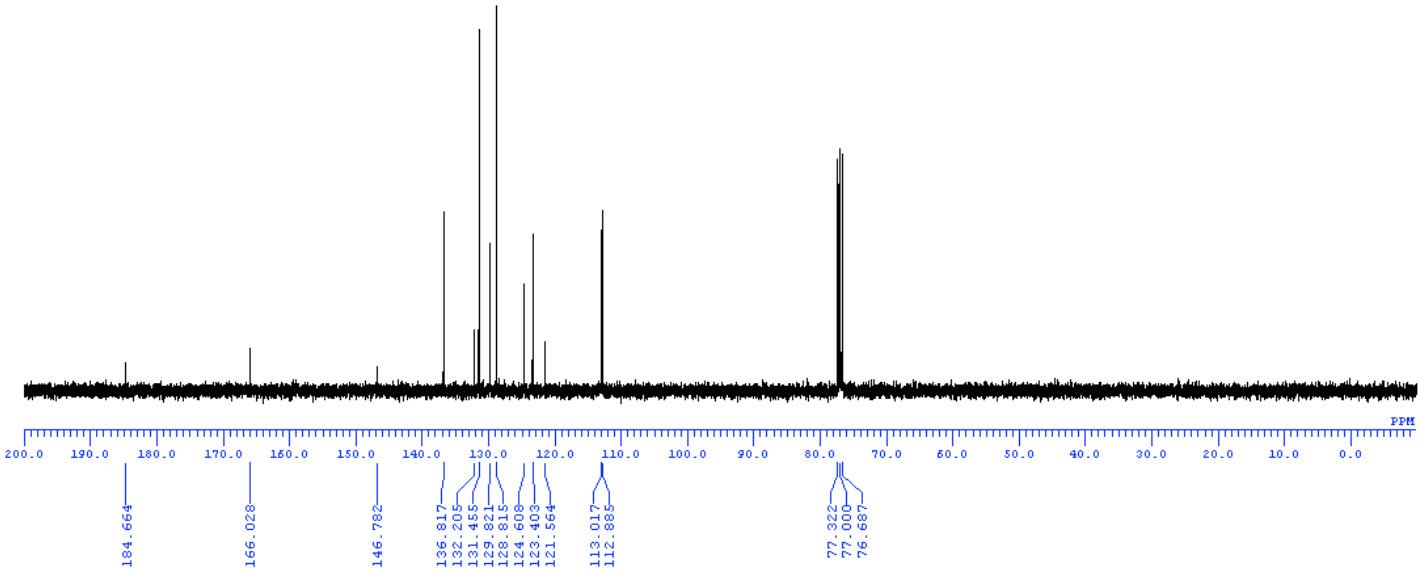


12a

^{13}C NMR ($CDCl_3$)



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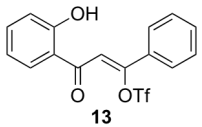
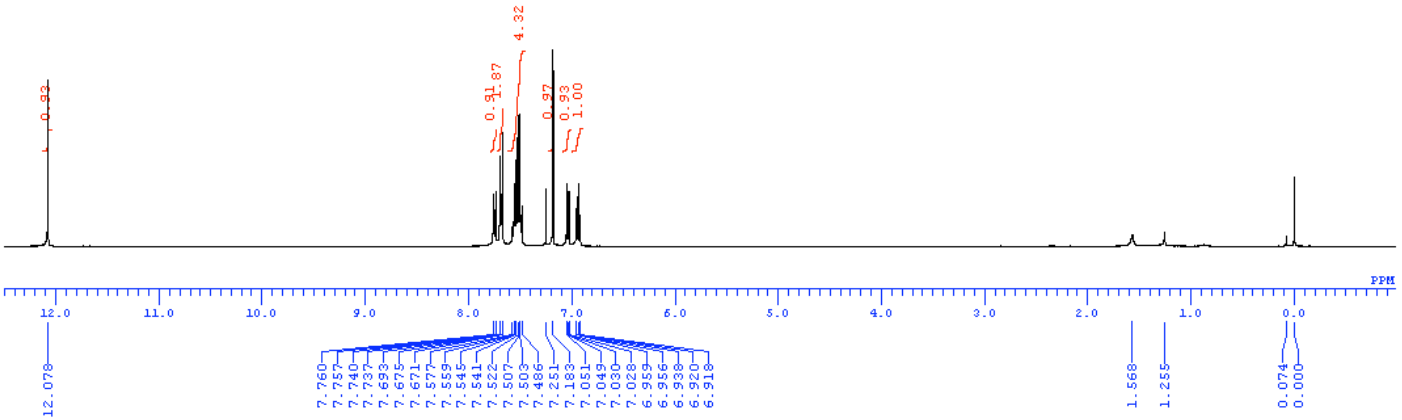


13

^{1}H NMR ($CDCl_3$)



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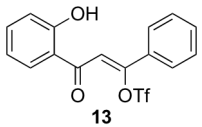

13

^{13}C NMR ($CDCl_3$)