Regioselective Cu(I)-Catalyzed Intramolecular Hydroacylation and Dimerization of Propargylarylaldehydes

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Contents

Experimental Section

General and methods. Propargyl aryl aldehydes were prepared according to the previously reported procedures.[1-3] The pure products were achieved using silica gel column chromatography. Melting points were measured with a Buchi B-545 melting point apparatus. ^{1}H NMR (300 or 400 MHz) and ^{13}C NMR (76 or 100 MHz) spectra were recorded by a Bruker spectrometer (δ in ppm) in deuterated chloroform ($CDCl_3$) with TMS as the internal standard.

General procedure for the synthesis of α,β-enones. A mixture of *S*-propargyl quinoline-3-carbaldehydes (1.0 mmol), $CuSO_4 \cdot 5H_2O$ (10 mol%), and ascorbic acid (15 mol%) was dissolved in *t*-BuOH/H_2O (4:1). The resulting solution was stirred vigorously under reflux conditions for 24 h. The crud products were purified using column chromatography with *n*-hexane/ethyl acetate (8:2) as eluent.

3-Methyl-4H-thiopyrano[2,3-b]quinolin-4-one (**4a**): White solid, M.P. 145-148 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ_{ppm}= 2.64 (s, 3H), 7.51 (t, *J* = 7.20 Hz, 1H), 7.79 (t, *J* = 7.05 Hz, 1H), 7.91 (d, *J* = 8.40 Hz, 1H), 7.93 (s, 1H), 8.09 (d, *J* = 8.70 Hz, 1H), 8.61 (s, 1H). ^{13}C NMR (76 MHz, $CDCl_3$): δ_{ppm}= 26.6, 125.8, 126.0, 126.9, 128.6, 128.7, 130.9, 131.7, 133.7, 144.7, 148.4, 163.6, 192.2. [M+Na^+] for $C_{13}H_9NOS$: 250.0303.

3,9-Dimethyl-4H-thiopyrano[2,3-b]quinolin-4-one (**4b**): White solid, M.P.159-161 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ_{ppm}= 2.75 (s, 3H), 2.91 (s, 3H), 7.50 (t, *J* = 7.65 Hz, 1H), 7.70 (d, *J* = 6.90 Hz, 1H), 7.87 (d, *J* = 8.40 Hz, 1H), 8.01 (s, 1H), 8.68 (s, 1H). ^{13}C NMR (76 MHz, $CDCl_3$): δ_{ppm}= 18.4, 26.4, 125.5, 126.6, 126.9, 130.6, 131.3, 133.7, 136.4, 144.3, 147.8, 162.8, 191.8, 144.7, 148.4, 163.6, 192.2. [M+Na^+] for $C_{14}H_{11}NOS$: 264.0459.

3,7-Dimethyl-4H-thiopyrano[2,3-b]quinolin-4-one (**4c**): White solid, M.P. 168-170 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ_{ppm}= 2.57 (s, 3H), 2.69 (s, 3H), 7.63 (t, *J* = 6.60 Hz, 1H), 7.65 (s, 1H), 7.71 (s, 1H), 8.02 (d, *J* = 6.60 Hz, 1H), 8.57 (s, 1H). ^{13}C NMR (76 MHz, $CDCl_3$): δ_{ppm}= 21.6, 26.6, 125.9, 127.0, 127.1, 128.1, 131.7, 132.9, 133.5, 135.8, 144.4, 147.2, 162.7, 192.2. [M+Na^+] for $C_{14}H_{11}NOS$: 264.0459.

7-Methoxy-3-methyl-4H-thiopyrano[2,3-b]quinolin-4-one (**4d**): White solid, M.P. 213-215 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ_{ppm}= 3.21 (s, 3H), 3.82 (s, 3H), 6.85 (d, *J* = 2.70 Hz, 1H), 7.28 (dd, *J* = 2.70 Hz, *J* = 12.30 Hz, 1H), 7.60 (d, *J* = 9.30 Hz, 1H), 8.12 (s, 1H). ^{13}C NMR (76 MHz, $CDCl_3$): δ_{ppm}= 26.6, 55.6, 70.2, 104.8, 124.8, 124.8, 126.8, 129.9, 131.9, 144.6, 145.2, 157.2, 161.2, 192.4. [M+Na^+] for $C_{14}H_{11}NO_2S$: 280.0408.

7-Ethoxy-3-methyl-4H-thiopyrano[2,3-b]quinolin-4-one (**4e**): Yellow solid, M.P. 206-208 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ_{ppm}= 1.46 (t, *J* = 7.05 Hz, 3H), 2.62 (s, 3H), 4.10 (q, *J* = 6.90 Hz, *J* = 13.80 Hz, 3H), 7.06 (s, 1H), 7.40 (dd, *J* = 1.20 Hz, *J* = 2.40 Hz, 1H), 7.82 (s, 1H), 7.97 (d, *J* = 6.30 Hz, 1H), 8.42 (s, 1H). ^{13}C NMR (76 MHz, $CDCl_3$): δ_{ppm}= 14.7, 26.6, 29.7, 63.9, 105.4, 125.1, 126.8, 126.9, 129.7, 131.9, 156.6, 192.3. [M+Na^+] for $C_{15}H_{13}NO_2S$: 294.0565.

8-Chloro-7-methoxy-3-methyl-4H-benzo[g]thiochromen-4-one (**4f**): White solid, M.P. 182-185 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ_{ppm}= 2.62 (s, 3H), 4.00 (s, 3H), 7.16 (s,1H), 7.85 (s, 1H), 8.13 (s, 1H), 8.50 (s, 1H). ^{13}C NMR (76 MHz, $CDCl_3$): δ_{ppm}= 45.16, 73.0, 120.1, 120.3, 120.8, 125.7, 126.0,126.4, 126.6, 128.8, 129.7, 132.0, 133.5, 168.22. [M+Na^+]. for $C_{14}H_{10}ClNO_2S$: 314.0018.

9-Methyl-8H-benzo[h]thiopyrano[2,3-b]quinolin-8-one (**4g**): White solid, M.P. 120-123 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ_{ppm}= 2.64 (s, 3H), 7.70 (m, 4H), 7.83 (m, 2H), 7.91 (s, 3H), 8.52 (s, 1H), 9.32 (m, 1H). ^{13}C NMR (76 MHz, $CDCl_3$): δ_{ppm}= 23.0, 103.5, 115.3, 124.7, 126.5,126.7, 129.2, 129.8, 130.8, 131.4, 134.8, 135.0, 145.5, 146.1, 146.6, 157.2. [M+Na^+] for $C_{17}H_{11}NOS$: 300.049.

7-Fluoro-3-methyl-4H-thiopyrano[2,3-b]quinolin-4-one (**4h**): White solid, M.P. 187-190 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ_{ppm}= 2.66 (s, 3H), 7.55 (m, 2H), 7.90 (s, 1H), 8.09 (m, 1H), 8.57 (s, 1H). ^{13}C NMR (76 MHz, $CDCl_3$): δ_{ppm}= 25.9, 109.8, 120.4, 125.3, 129.9, 130.8, 131.0, 131.6, 144.3, 191.4. [M+Na^+] for $C_{13}H_8FNOS$: 268.0208.

7-Chloro-3-methyl-4H-thiopyrano[2,3-b]quinolin-4-one (**4i**): Yellow solid, M.P. 153-155 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ_{ppm}= 2.66 (s, 3H), 7.67 (dd, *J* = 1.80 Hz, *J* = 1.80 Hz, 3H), 7.91 (d, *J* = 2.70 Hz, 1H), 8.04 (d, *J* = 6.90 Hz, 1H), 8.55 (s, 1H). ^{13}C NMR (76 MHz, $CDCl_3$): δ_{ppm}= 26.8, 126.6, 126.9, 127.8, 128.4, 129.6, 130.4, 131.6, 133.1, 134.5, 134. 183.5. [M+Na^+] for $C_{13}H_8ClNOS$: 283.9913.

7-Bromo-3-methyl-4H-thiopyrano[2,3-b]quinolin-4-one (**4j**): Brown solid, M.P. 163-165 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ_{ppm}= 2.66 (s, 3H), 7.79 (dd, *J* = 2.40 Hz, *J* = 2.10 Hz, 1H), 7.92 (s, 1H), 7.97 (d, *J* = 6.30 Hz, 1H), 8.09 (d, *J* = 2.10 Hz, 1H), 8.54 (s, 1H). ^{13}C NMR (76 MHz, $CDCl_3$): δ_{ppm}= 29.9, 112.8, 125.6, 128.6, 129.4, 131.6, 132.4, 133.2, 182.1. [M+Na^+] for $C_{13}H_8BrNOS$: 327.9408.

General procedure for the synthesis of conjugated enynes. The O- or N-propargyl aryl aldehydes (2.0 mmol) was stirred with $CuSO_4 \cdot 5H_2O$ (10 mol%) in the presence of a catalytic amount of ascorbic acid (15 mol%) as an additive for 24 h under reflux *t*-BuOH/H_2O (4:1). The crud products were purified using column chromatography with *n*-hexane/ethyl acetate (8:2) as eluent.

(E/Z)-2,2'-(Hex-2-en-4-yne-1,6-diylbis(oxy))dibenzaldehyde (100/0) (**12a**): White solid, M.P. 101-103 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ_{ppm}= 4.68 (d, *J* = 3.60 Hz,2H), 4.93 (s, 2H), 5.89 (dd, *J* = 2.00 Hz, *J* = 16.00 Hz, 1H), 6.33 (m, 1H), 6.90 (d, *J* = 8.40 Hz, 1H), 7.05 (m, 3H), 7.52 (m, 2H), 7.82 (m, 2H), 10.46 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ_{ppm}= 57.1, 57.2, 67.7, 67.8, 84.5, 85.3, 111.6, 112.6, 113.3, 121.3,121.6, 125.2, 128.6, 128.8, 135.8, 138.2. [M+Na^+] for $C_{20}H_{16}O_4$: 343.0946.

(E/Z)-6,6'-(Hex-2-en-4-yne-1,6-diylbis(oxy))bis(3-methoxybenzaldehyde) (100/0) (**12b**): White solid, M.P. 153-155 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ_{ppm}= 3.74 (s, 3H), 3.75 (s, 3H), 4.59 (dd, *J* = 1.20 Hz, *J* = 4.80 Hz, 2H), 4.84 (d, *J* = 1.20 Hz, 2H), 5.82 (d, *J* = 16.00 Hz, 1H, *E* isomer), 6.26 (m, 1H), 6.83 (d, *J* = 9.20 Hz, 1H), 7.02 (m, 1H), 7.08 (m, 2H), 7.28 (m, 2H), 10.39 (s, 1H),

10.40 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ_{ppm}= 54.8, 57.2, 67.6, 83.9, 84.3, 109.3, 109.5, 110.4, 113.7, 114.8, 122.3, 122.4, 124.4, 125.2, 137.4, 153.0, 153.4, 153.6, 154.2, 188.2, 188.4. [M+Na^+] for $C_{22}H_{20}O_6$: 403.1158.

(E/Z)-2,2'-(Hex-2-en-4-yne-1,6-diylbis(oxy))bis(4-methoxybenzaldehyde) (70:30) (**12c**): White solid, M.P.160-162 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ_{ppm}= 3.79 (s, 3H), 3.80 (s, 3H), 4.61 (dd, *J* = 1.60 Hz, *J* = 4.80 Hz, *E* isomer), 4.75 (dd, *J* = 1.60 Hz, *J* = 6.40 Hz, *Z* isomer), 4.86 (d, *J* = 1.20 Hz, *E* isomer), 4.92 (d, *J* = 1.60 Hz, *Z* isomer), 5.73 (d, *J* =11.20 Hz, *Z* isomer), 5.86 (d, *J* =16.00 Hz, *E* isomer), 6.16 (m, *Z* isomer), 6.27 (m, *E* isomer), 6.31 (m, 1H), 6.51 (m, 3H), 7.76 (m, 2H), 10.24 (s, 1H), 10.25 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ_{ppm}= 55.6, 57.0, 7.28, 83.5, 90.2, 98.9, 99.8, 106.3, 106.5, 111.8, 119.2, 119.6, 130.6, 130.9, 138.7, 161.3, 162.3, 165.9, 166.0, 188.0, 188.2. [M+Na^+] for $C_{22}H_{20}O_6$: 403.1158.

(E/Z)-2,2'-(Hex-2-en-4-yne-1,6-diylbis(oxy)) bis(4-methylbenzaldehyde) (60:40) (**12d**): White solid, M.P. 112-115 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ_{ppm}= 2.25 (s, 3H), 2.27 (s, 3H), 4.61 (dd, *J* = 1.60 Hz, *J* = 4.80 Hz, *E* isomer), 4.71 (dd, *J* = 1.20 Hz, *J* = 6.00 Hz, *Z* isomer), 4.86 (d, *J* = 1.60 Hz, *E* isomer), 4.92 (d, *J* = 2.00 Hz, *Z* isomer), 5.70 (d, *J* =11.20 Hz, *Z* isomer), 5.84 (d, *J* = 16.00 Hz, *E* isomer), 6.15 (m, *Z* isomer), 6.28 (m, *E* isomer), 6.67 (d, *J* = 8.80 Hz, *Z* isomer), 6.76 (d, *J* = 8.40 Hz, *E* isomer), 6.95 (dd, *J* = 2.40 Hz, *J* = 8.40 Hz, 1H), 7.27 (m, 2H), 7.57 (m, 2H), 10.39 (s, 1H), 10.40 (s,1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ_{ppm}= 20.3, 29.7, 57.4, 68.0, 84.8, 85.3, 111.5, 112.8, 113.5, 128.6, 128.8, 130.8, 131.2, 136.4, 136.5, 138.3, 158.1, 158.5, 189.6, 189.8. [M+Na^+] for $C_{22}H_{20}O_4$: 371.1259.

(E/Z)-2,2'-(Hex-2-en-4-yne-1,6-diylbis(oxy))bis(4-chlorobenzaldehyde) (70:30) (**12e**): Yellow solid, M.P. 122-124 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ_{ppm}= 4.64 (dd, *J* = 2.00 Hz, *J* = 5.20 Hz, *E* isomer), 4.72 (dd, *J* = 1.60 Hz, *J* = 6.00 Hz, *Z* isomer), 4.89 (d, *J* = 1.60 Hz, *E* isomer), 4.95 (d, *J* = 2.00 Hz, *Z* isomer), 5.74 (d, *J* = 10.80 Hz, *Z* isomer), 5.83 (d, *J* = 16.00 Hz, *E* isomer), 6.16 (m, *Z* isomer), 6.27 (m, *E* isomer), 6.73 (d, *J* = 8.80 Hz, *Z* isomer), 6.83 (d, *J* = 8.80 Hz, 1H),

7.01 (dd, *J* = 1.60 Hz, J = 8.80 Hz, 1H), 7.42(m, 2H), 7.73 (m, 2H), 10.35 (s, 1H), 10.36 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ_{ppm}= 56.4, 67.1, 83.4, 84.5, 110.7, 113.3, 114.0, 125.0, 125.5, 126.1, 126.4, 127.2, 127.4, 134.2, 134.4, 136.8, 157.2, 157.7, 187.0, 187.2. [M+Na^+] for $C_{20}H_{14}Cl_2O_4$: 411.0167.

(E/Z)-2,2'-(Hex-2-en-4-yne-1,6-diylbis(oxy))bis(1-naphthaldehyde) (10:90) (**12f**): White solid, M.P. 155-157 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ_{ppm}= 4.79 (d, *J* = 8.40 Hz, 2H, *Z* isomer), 5.08 (s, 2H, *Z* isomer), 5.72 (d, *J* = 14.40 Hz, *Z* isomer), 5.86 (d, *J* = 21.60 Hz, *E* isomer), 6.18 (m, *Z* isomer), 6.88 (d, *J* = 12.00 Hz, *Z* isomer), 7.34 (m, 3H), 7.61 (m, 5H), 7.96 (m, 1H), 9.18 (t, *J* = 10.40 Hz, 2H), 10.75 (s, 1H), 10.82 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ_{ppm}= 58.0, 67.2, 83.8, 90.4, 111.8, 113.4, 114.2, 117.1, 118.3, 125.0, 125.1, 125.3, 128.3, 129.9, 137.2, 137.5, 138.5, 161.8, 162.7, 191.9, 192.0. [M+Na^+] for $C_{28}H_{20}O_4$: 443.1259.

(E/Z)-1,1'-(Hex-2-en-4-yne-1,6-diyl) bis(1H-indole-2-carbaldehyde) (0/100) (**12g**): White solid, M.P.112-115 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ_{ppm}= 5.29 (dd, *J* = 1.60 Hz, *J* = 8.80 Hz, 2H), 5.56 (d, *J* = 14.00 Hz, 1H, *Z* isomer), 5.66 (d, *J* = 2.80 Hz, 2H), 5.94 (m, 1H), 7.12 (m, 3H), 7.23 (m, 2H), 7.31 (s, 1H), 7.45 (m, 1H), 7.58 (d, *J* = 11.20 Hz, 2H), 7.66 (d, *J* = 9.20 Hz, 1H), 7.76 (d, *J* = 10.80 Hz, 1H), 9.82 (s, 1H), 9.91 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ_{ppm}= 34.8, 44.0, 76.6, 77.0, 77.5, 90.2, 110.8, 110.9, 111.0, 118.1, 121.1, 121.6, 123.3, 123.7, 127.1, 127.6, 134.6, 134.9, 138.7, 140.3. [M+Na^+]. for $C_{24}H_{18}N_2O_2$: 389.1266.

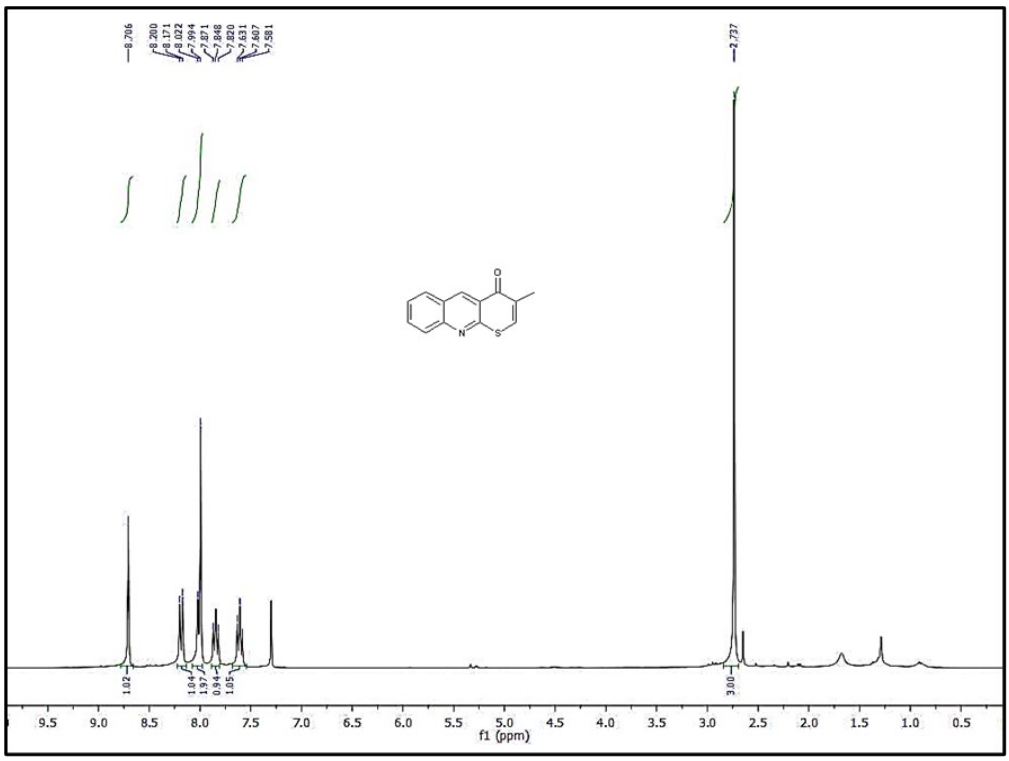
X-ray Crystallography

The colorless needle crystals of **12g** were obtained from methanol solvent. The crystals are crystallized in monoclinic space group *C2/c*. The X-ray data for the reported structure was collected at 100(12) K with Rigaku XtaLAB Synergy S Dualflex diffractometer with HyPix detector and a PhotonJet Cu-Kα radiation source (λ = 1. 54184 Å). The obtained data set was processed with *CrysAlisPro* software. [4] The structures were solved by direct methods and refined with the full-matrix least-squares method on F^2 with the use of SHELX2014 program packages.[5]

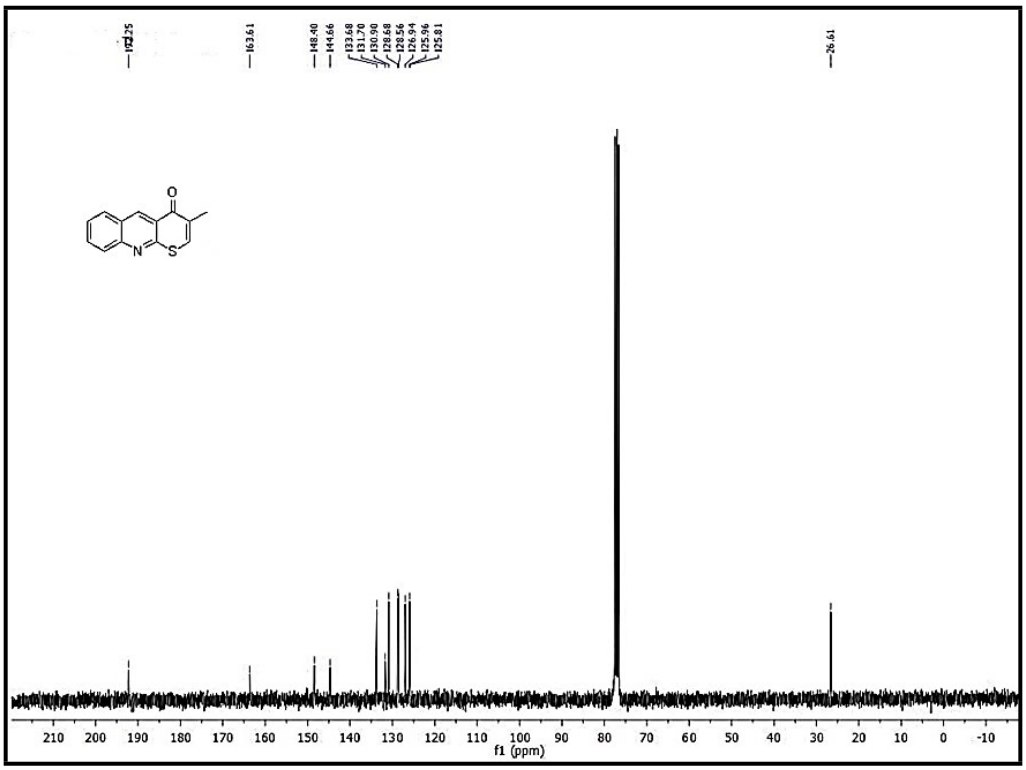
The data collection and refinement processes are summarized in Table 6. The structural data have been deposited at the Cambridge Crystallographic Data Centre: (CCDC No 2376902).

Table 6. Crystal data and structure refinement parameters for **12g**.

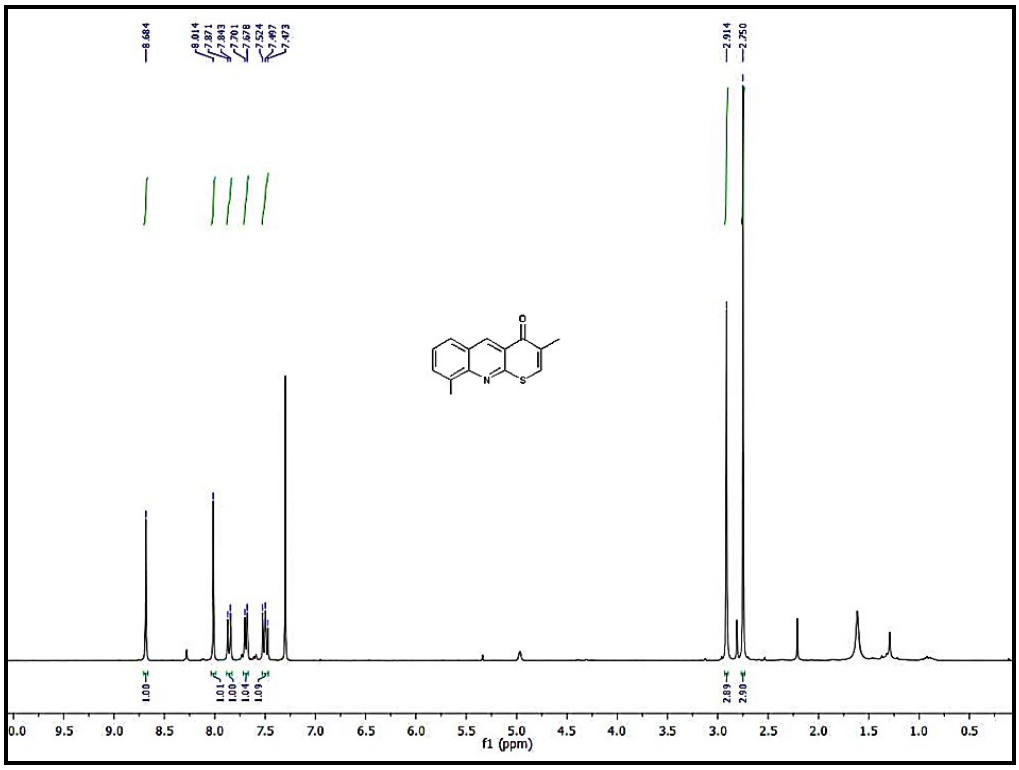
Compound	**12g**
Empirical formula	$C_{28}H_{26}N_2O_4$
Formula weight [g.mol^{-1}]	454.51
Crystal size [mm]	0.291 × 0.024 × 0.019
Crystal system	Monoclinic
Space group	*C*2/*c*
a [Å]	32.7304(13)
b [Å]	4.4327(2)
c [Å]	32.3534(13)
β [º]	96.961(4)
Volume [Å^3]	4659.4(3)
Z	8
Density (calc.) [g.cm^{-3}]	1.296
Absorption coefficient [mm^{-1}]	0.703
F(000)	1920
Θ range [º]	2.720 to 78.078
Reflections collected / unique	14873 / 4604 [R_{int} = 0.0774]
Index ranges *hkl*	-39<=h<=40, -5<=k<=4, -40<=l<=38
restraints/parameters	0 / 307
Goodness of fit on F^2	1.037
Final R indices [$I > 2\sigma(I)$]	R1 = 0.0938, wR2 = 0.2525
R indices (all data)	R1 = 0.1320, wR2 = 0.2860
Max electron density/e·Å^{-3}	0.629
Min electron density/e·Å^{-3}	-0.411



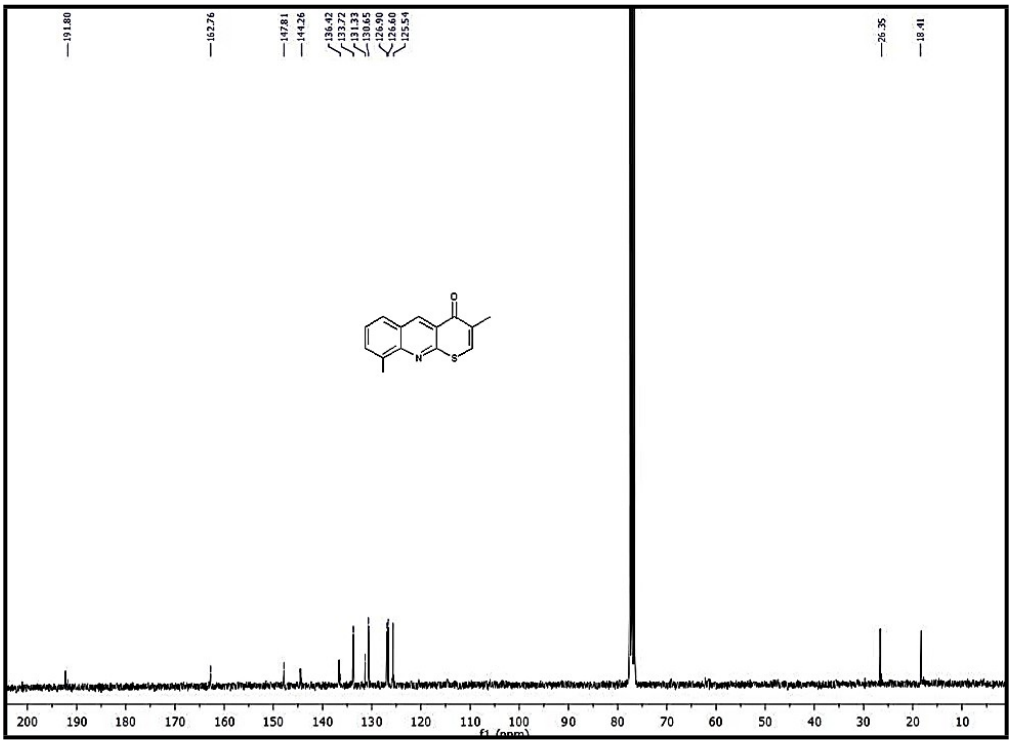
^{1}H NMR Spectrum of **4a**



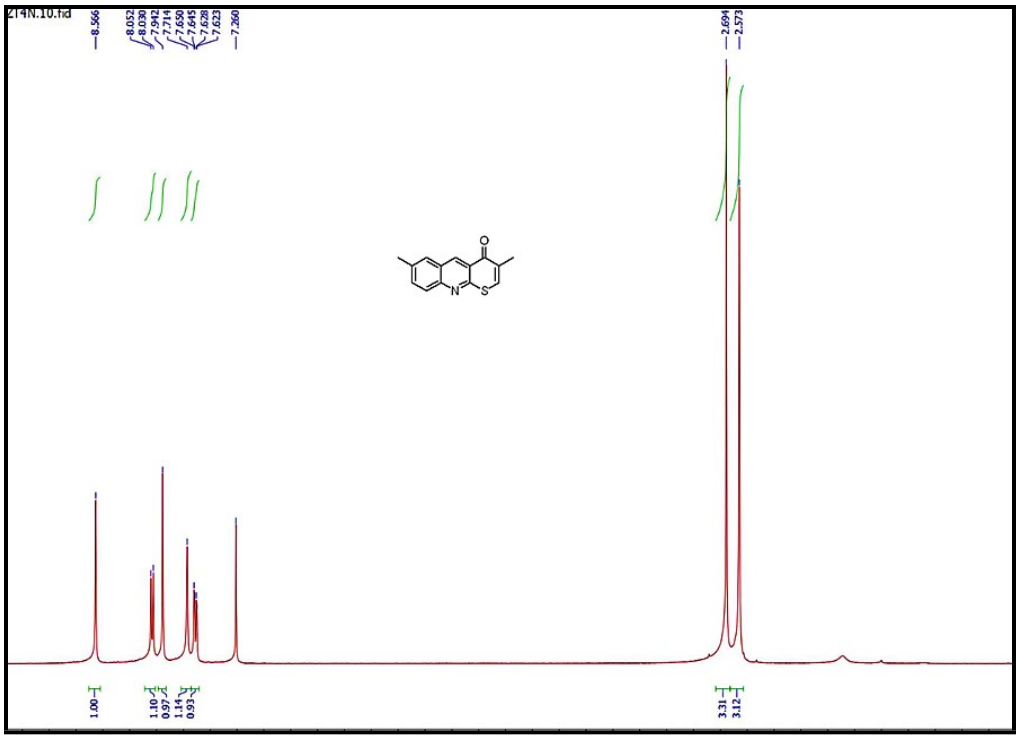
^{13}C NMR Spectrum of **4a**



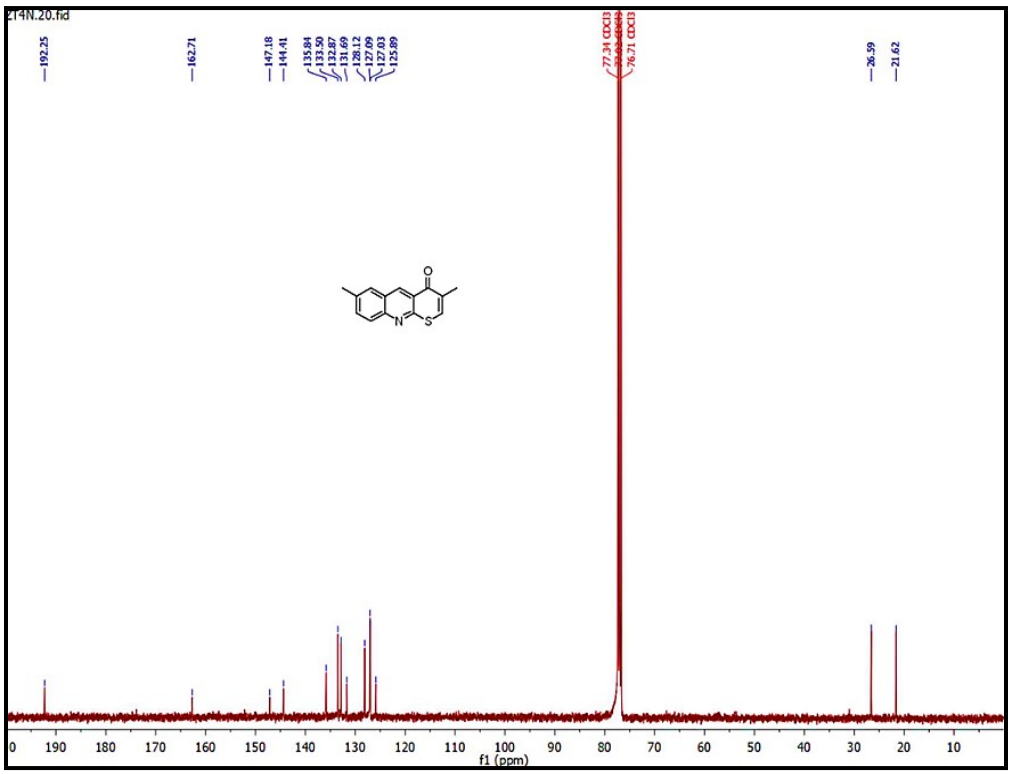
^{1}H NMR Spectrum of **4b**



^{13}C NMR Spectrum of **4b**

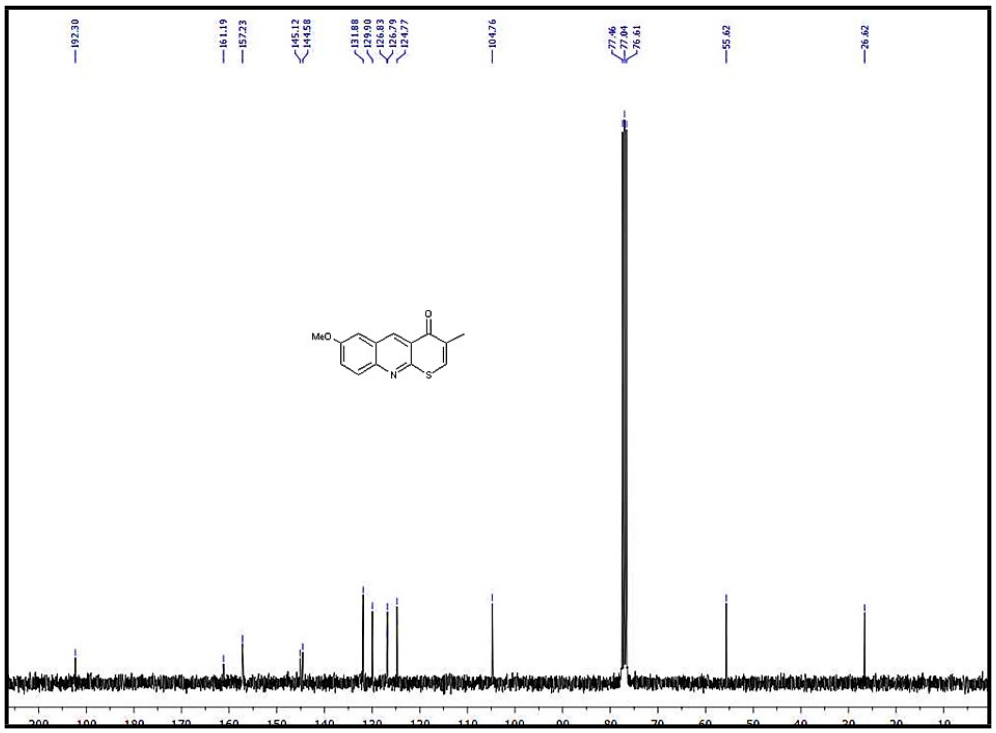


^{1}H NMR Spectrum of **4c**

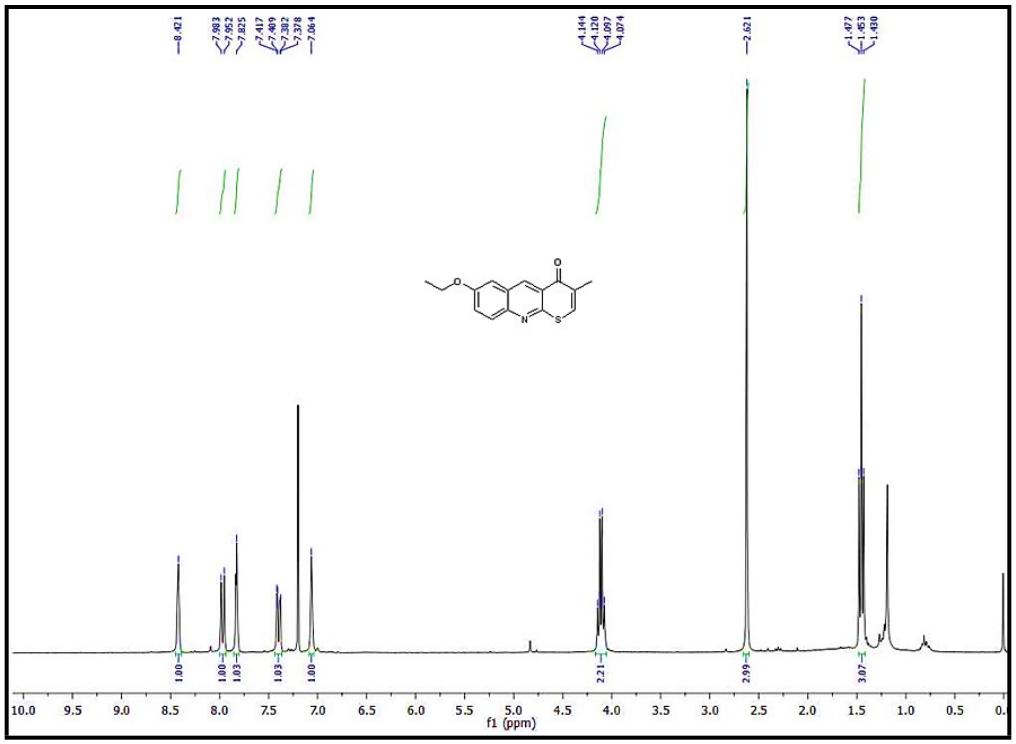


^{13}C NMR Spectrum of **4c**

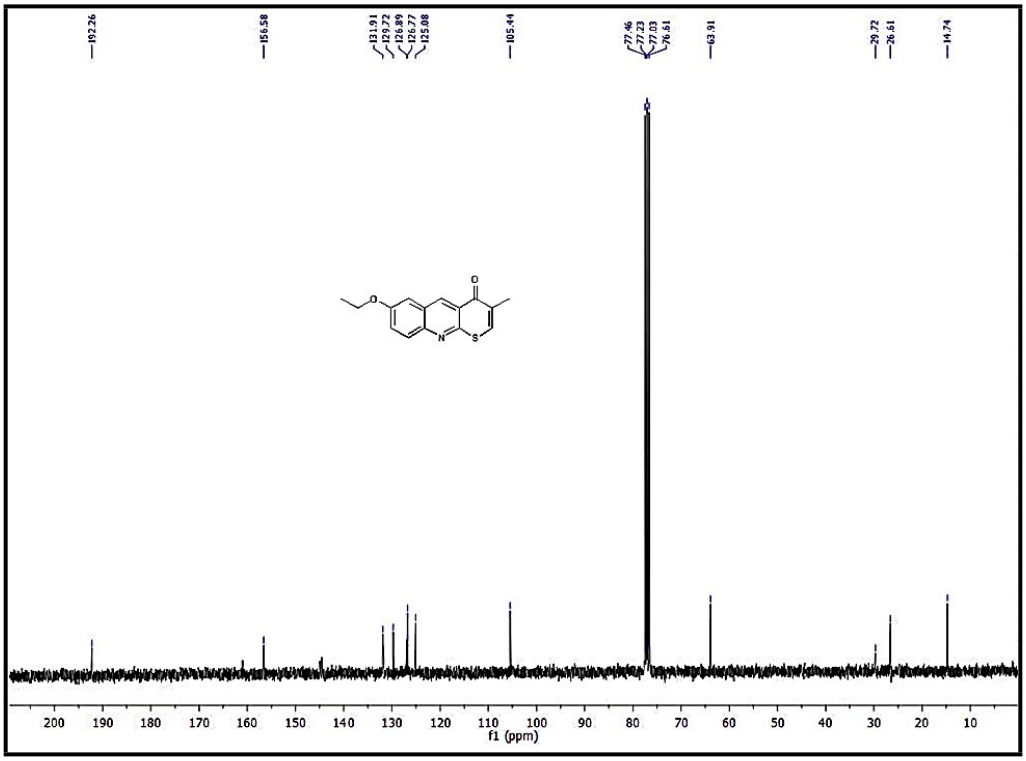
^{1}H NMR Spectrum of **4d**



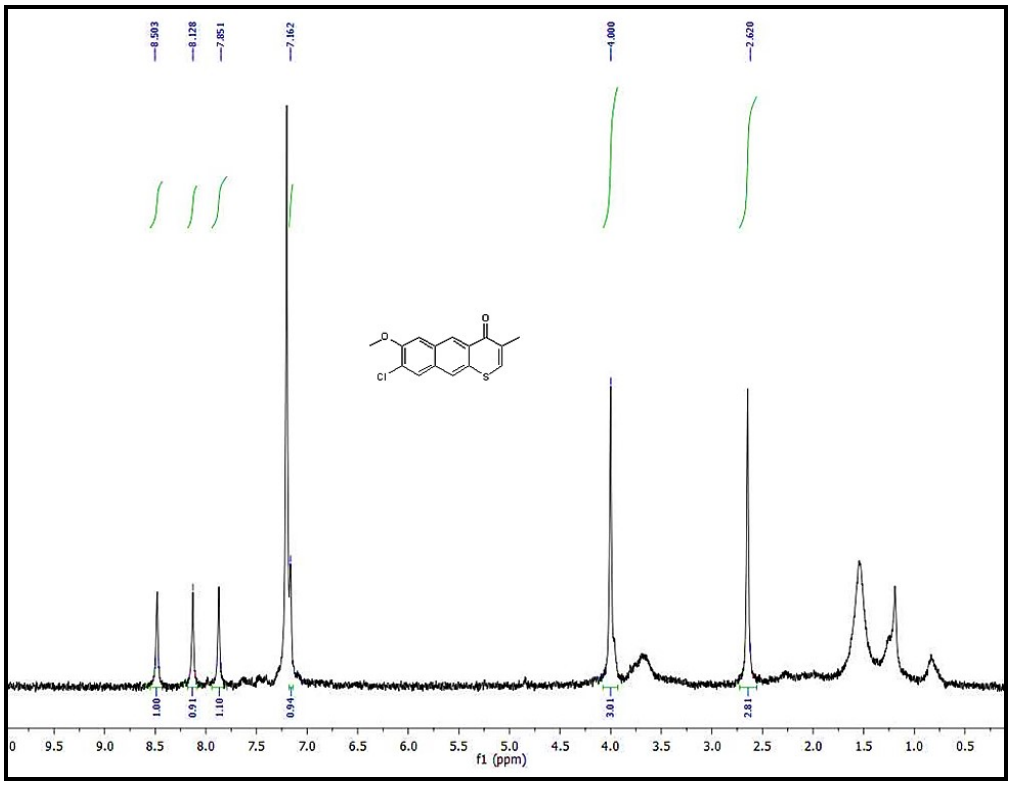
^{13}C NMR Spectrum of **4d**



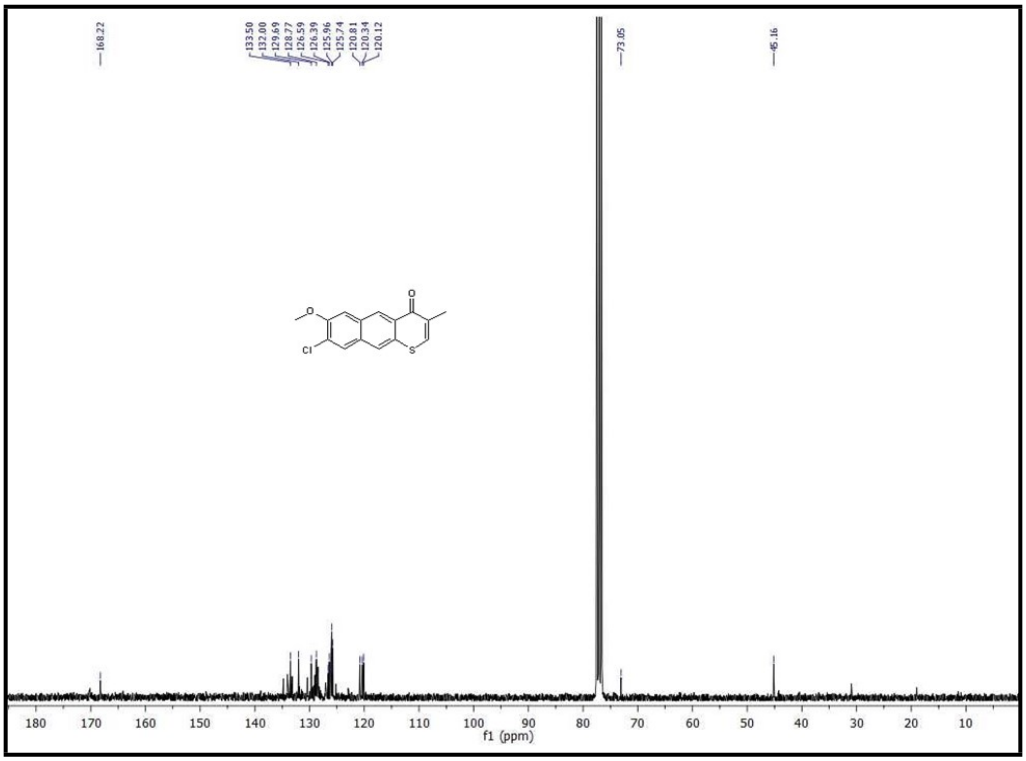
^{1}H NMR Spectrum of **4e**



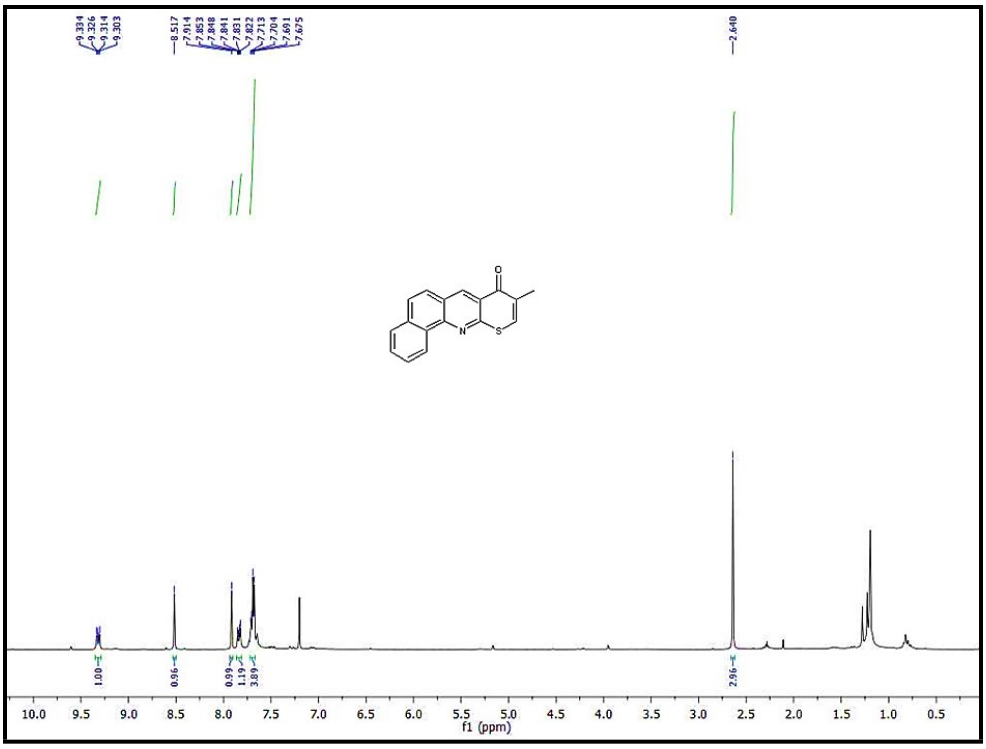
^{13}C NMR Spectrum of **4e**



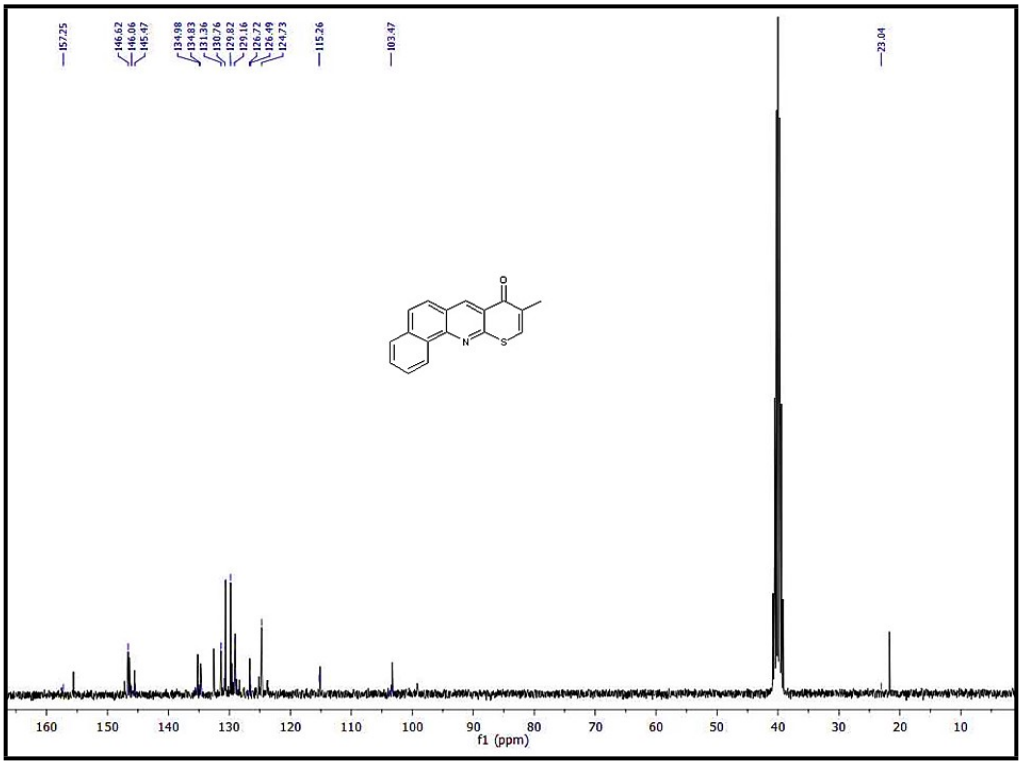
^{1}H NMR Spectrum of **4f**



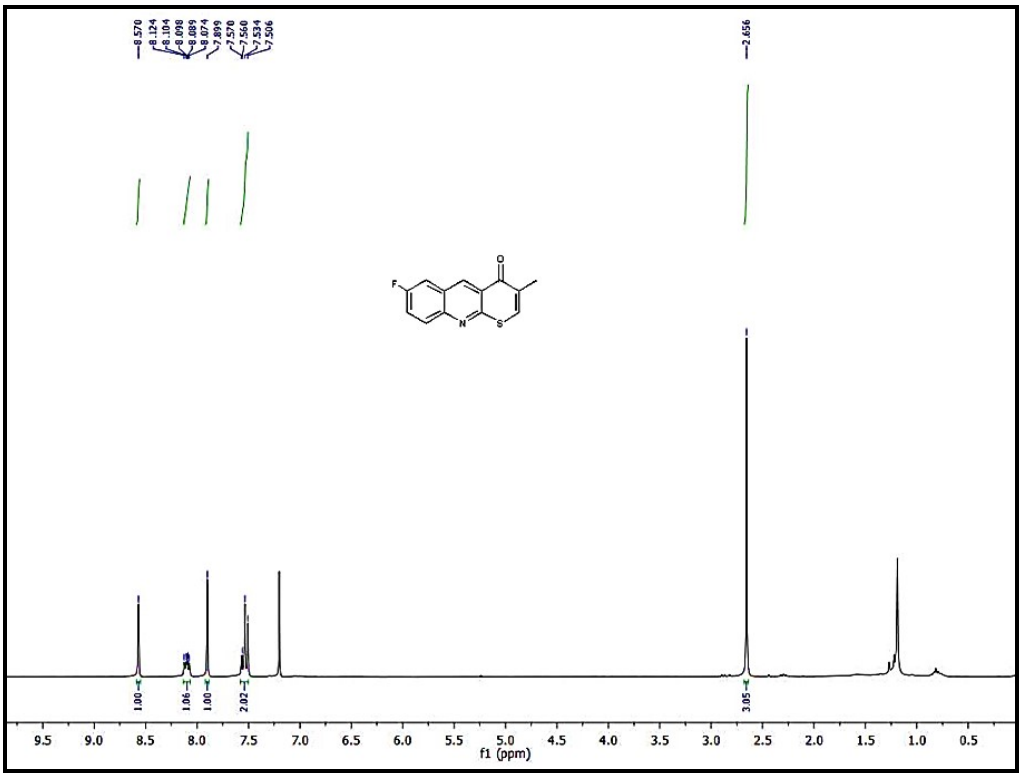
^{13}C NMR Spectrum of **4f**



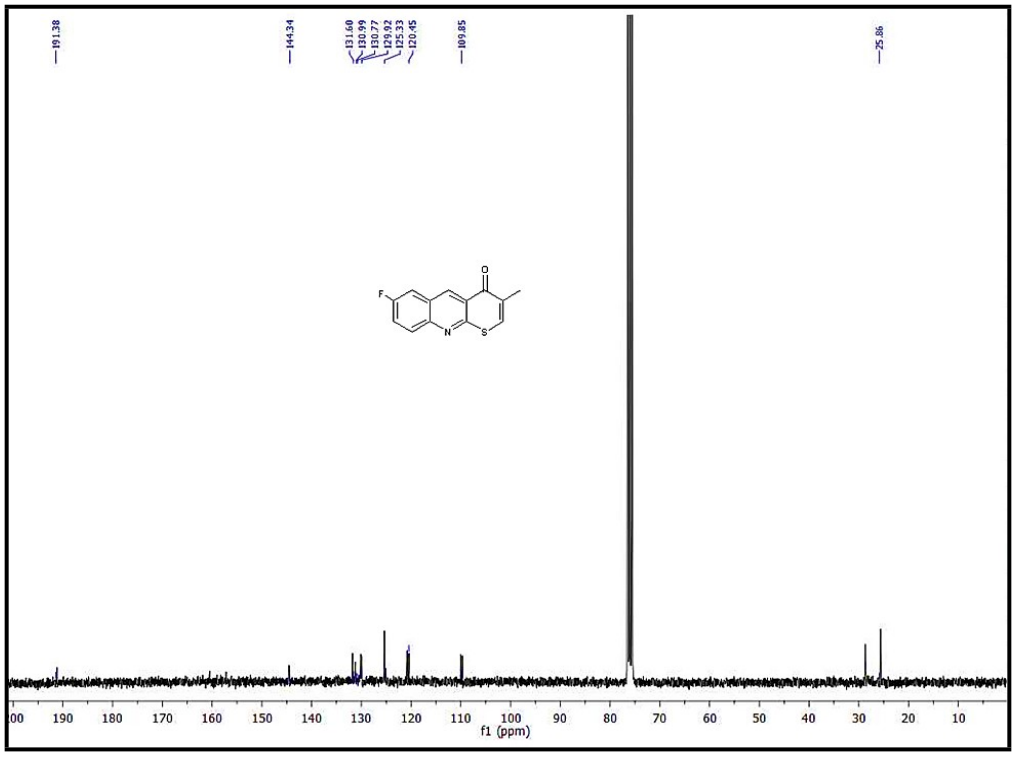
^{1}H NMR Spectrum of **4g**



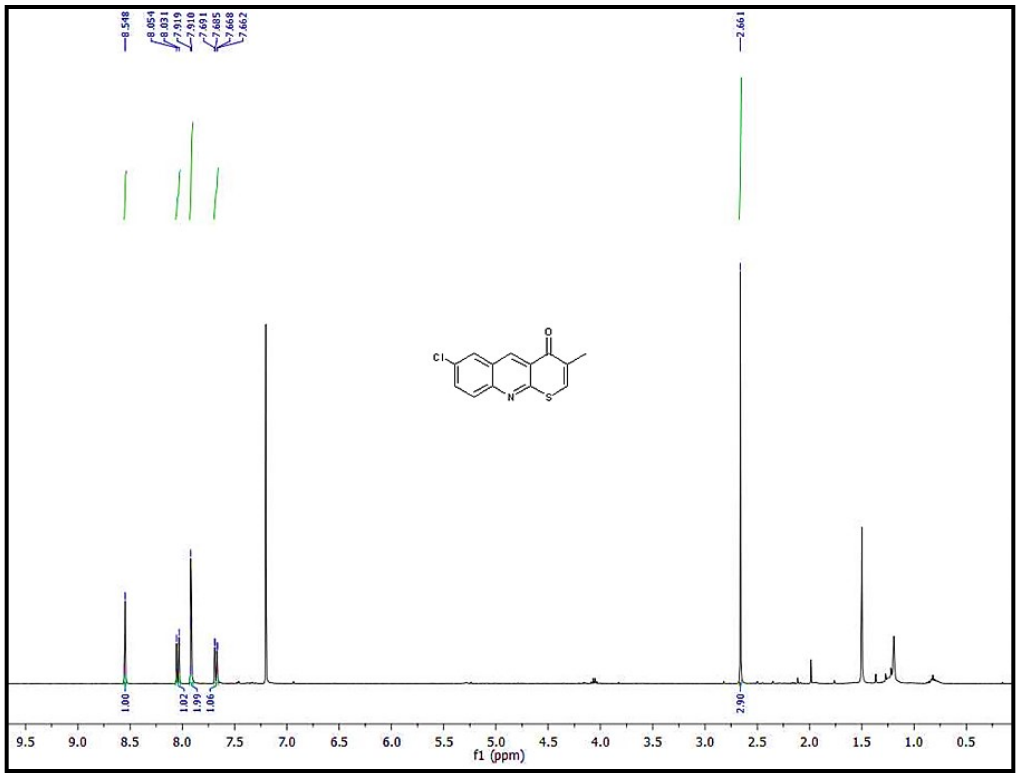
^{13}C NMR Spectrum of **4g**



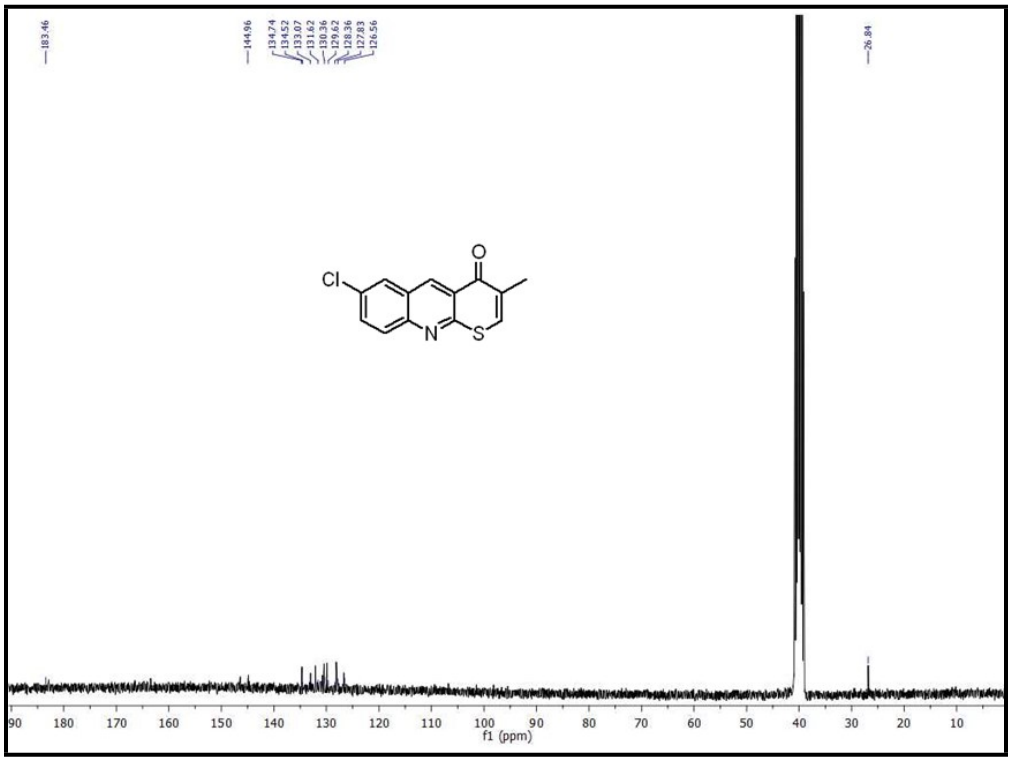
^{1}H NMR Spectrum of **4h**



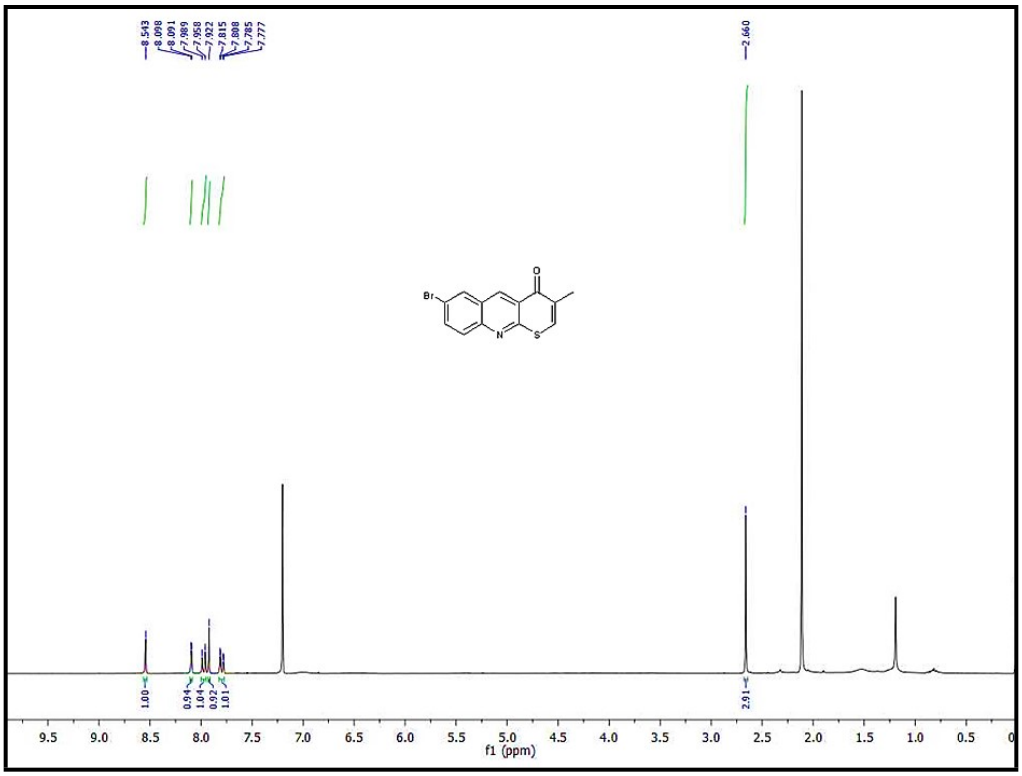
^{13}C NMR Spectrum of **4h**



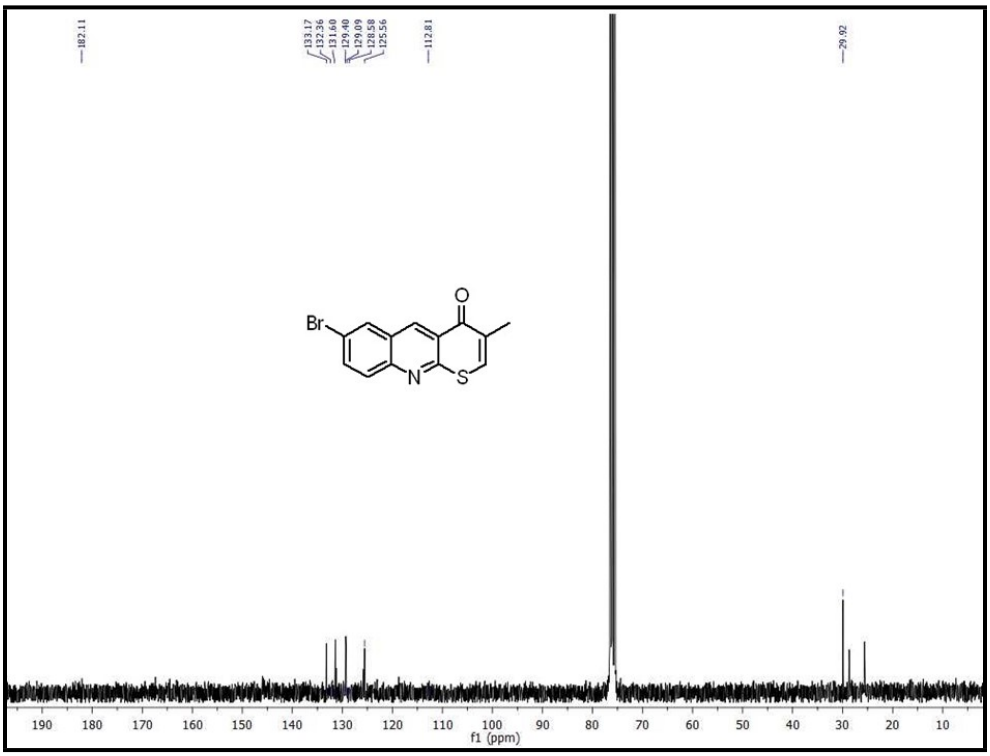
^{1}H NMR Spectrum of **4i**



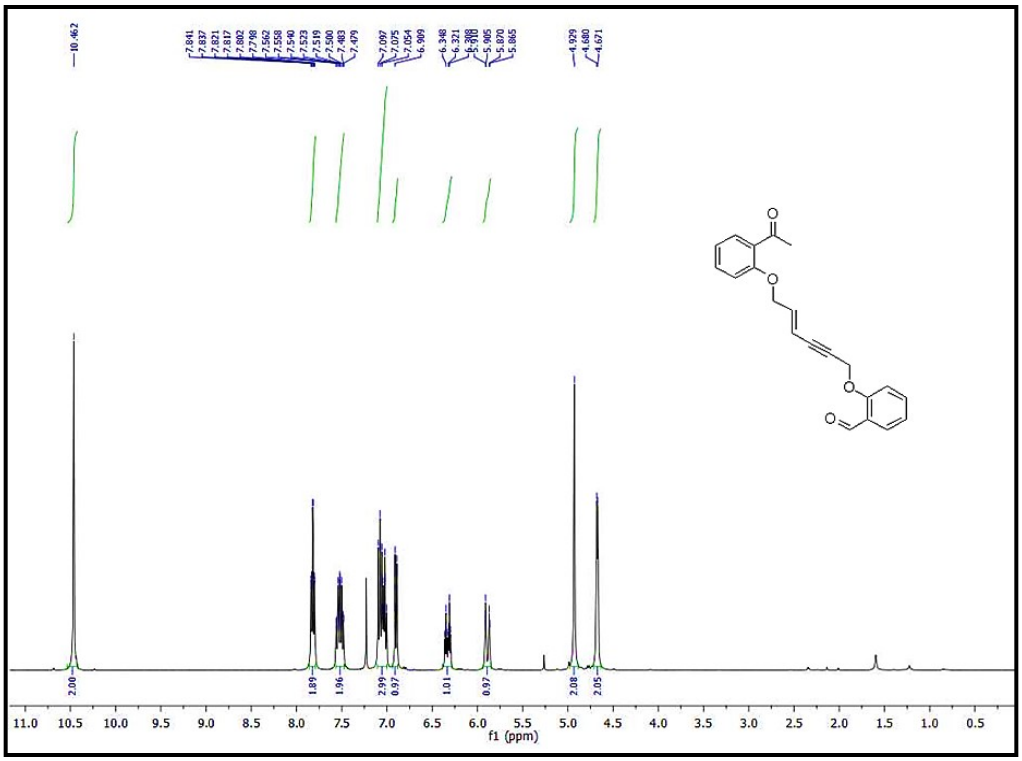
^{13}C NMR Spectrum of **4i**



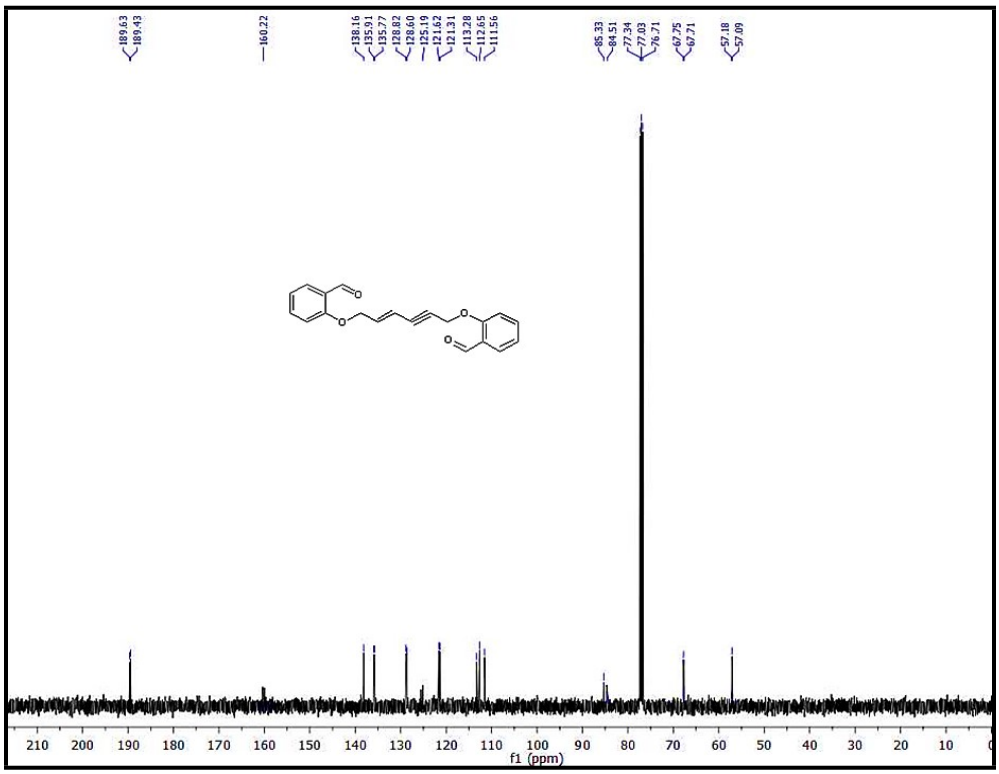
^{1}H NMR Spectrum of **4j**



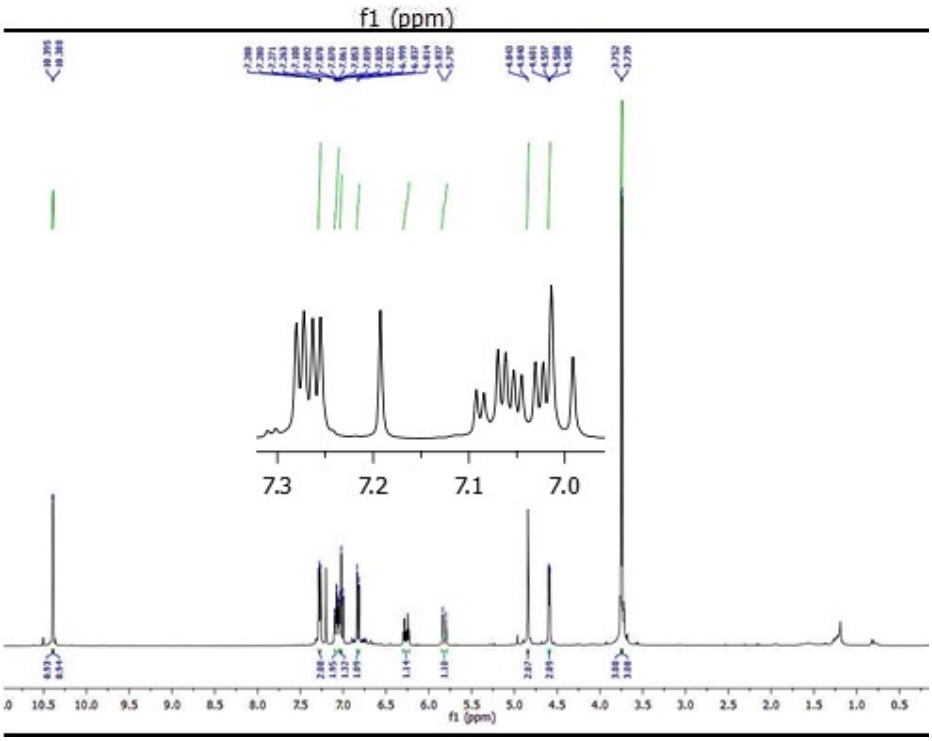
^{13}C NMR Spectrum of **4j**



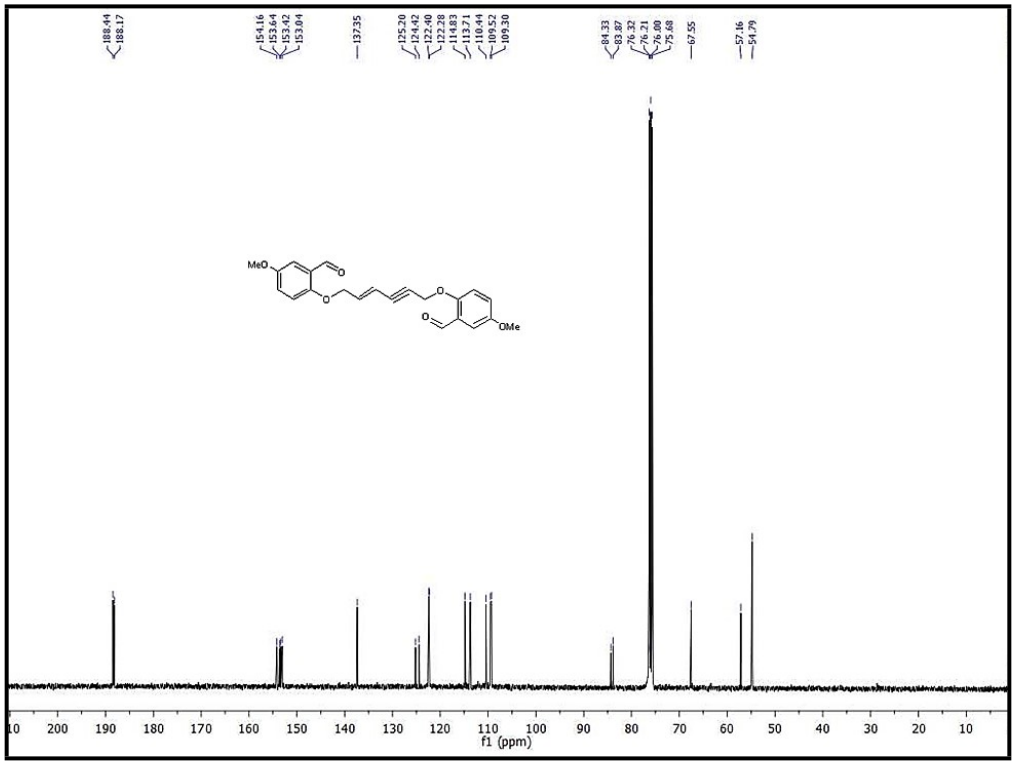
^{1}H NMR Spectrum of **12a**



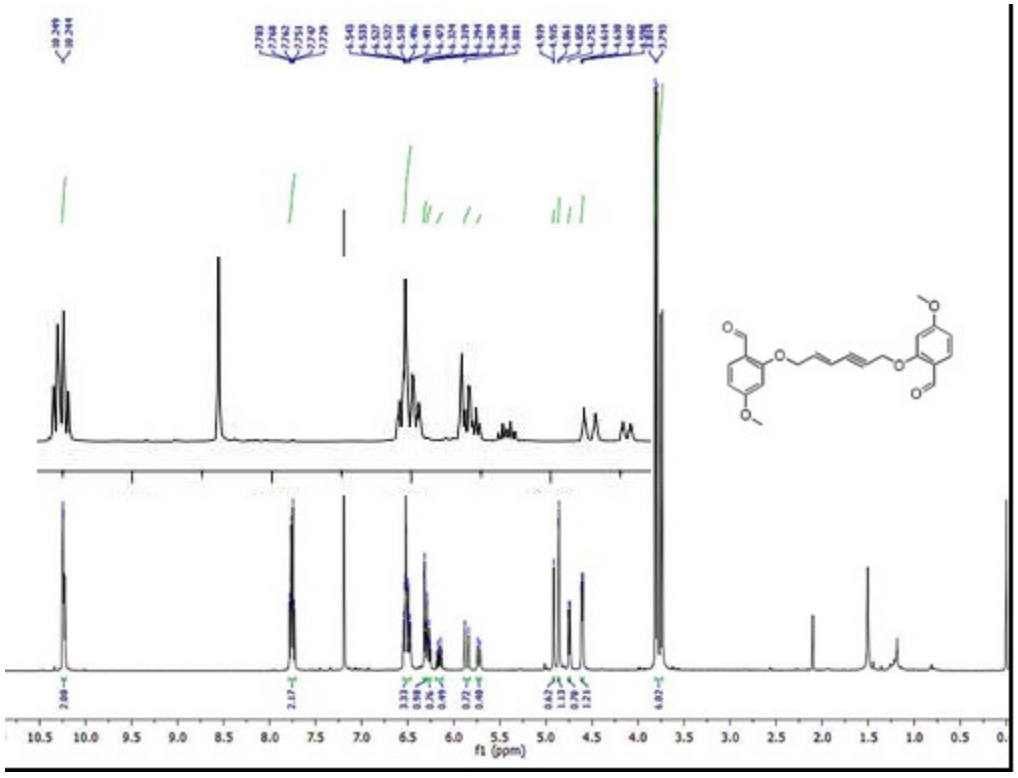
^{13}C NMR Spectrum of **12a**



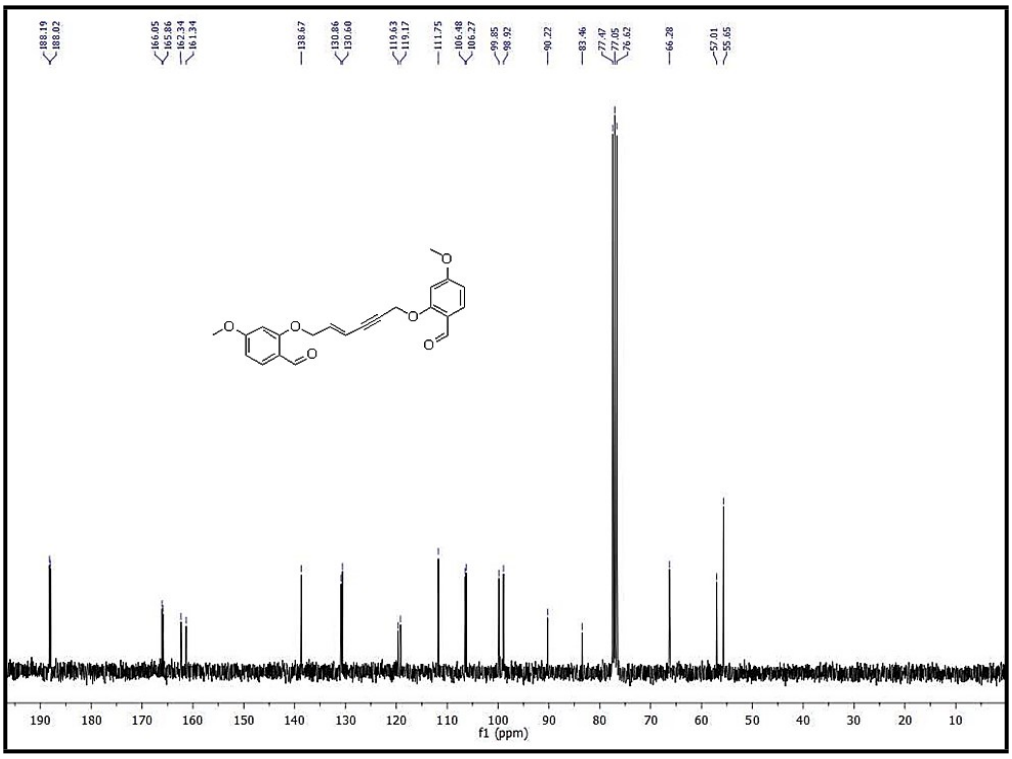
^{1}H NMR Spectrum of **12b**



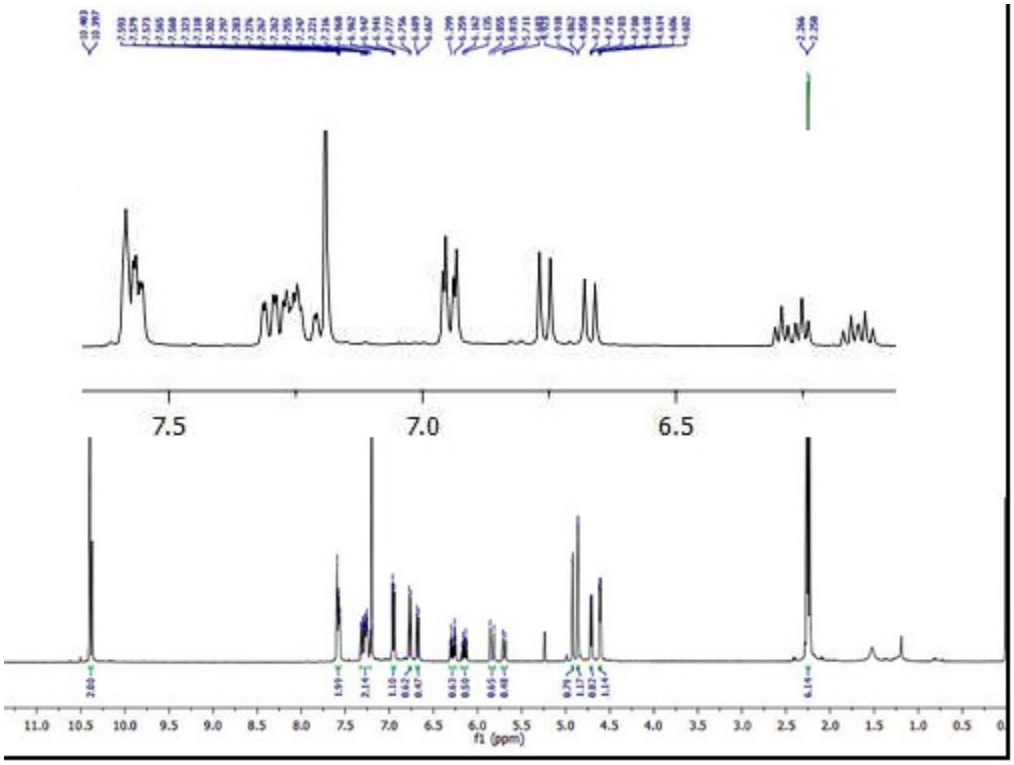
^{13}C NMR Spectrum of **12b**



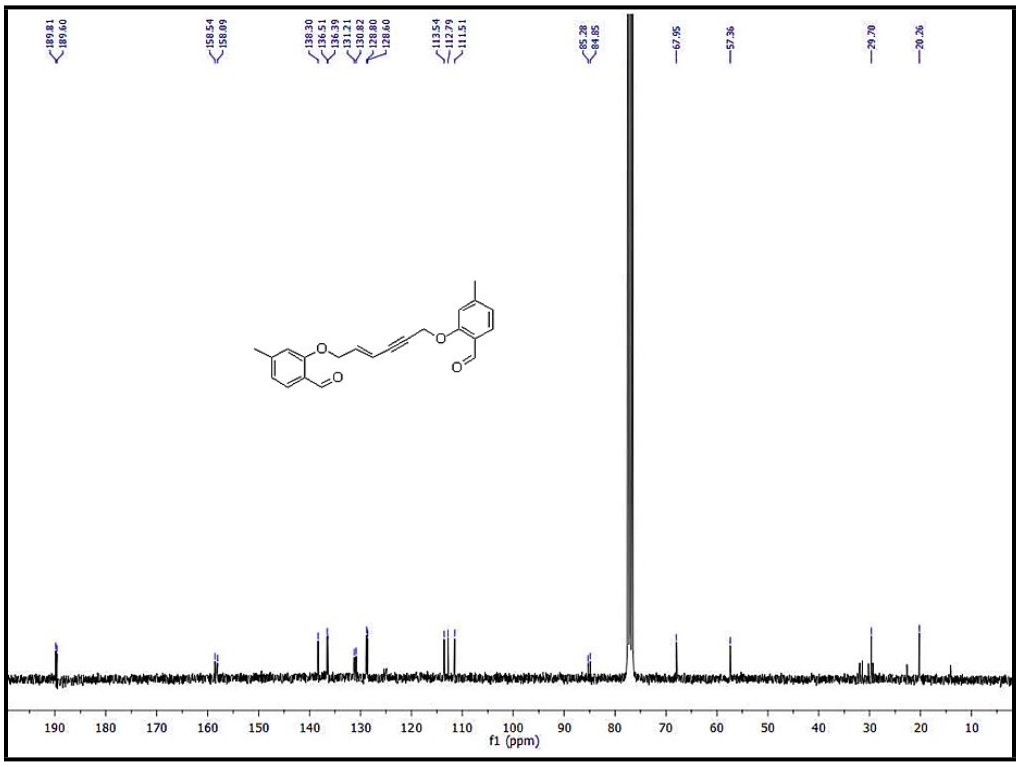
^{1}H NMR Spectrum of **12c**



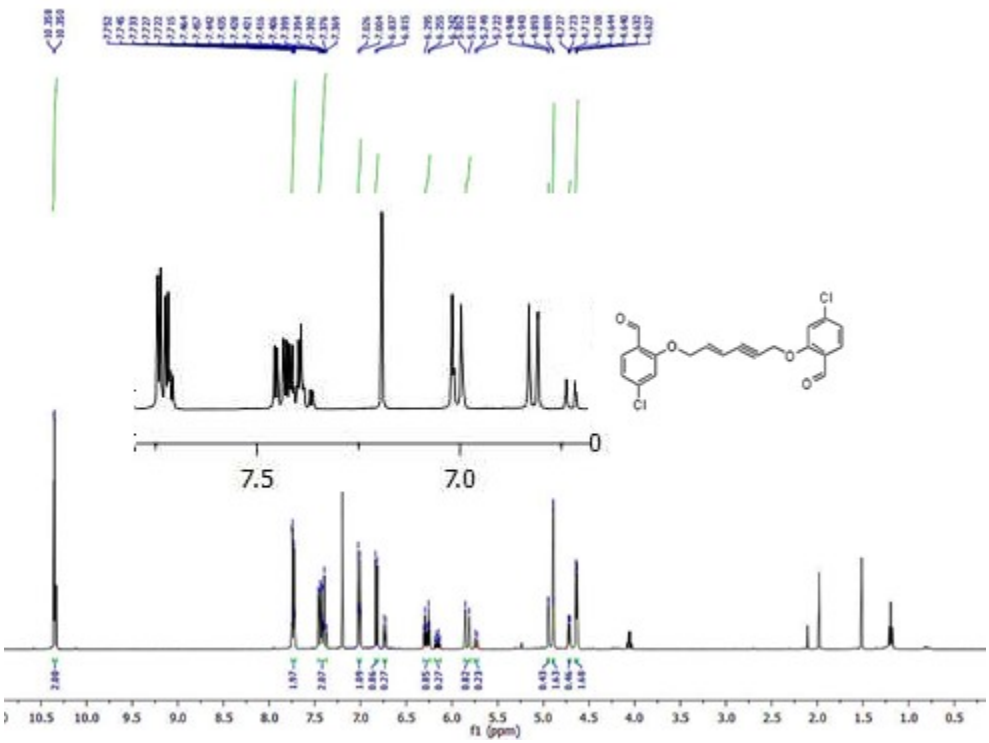
^{13}C NMR Spectrum of **12c**



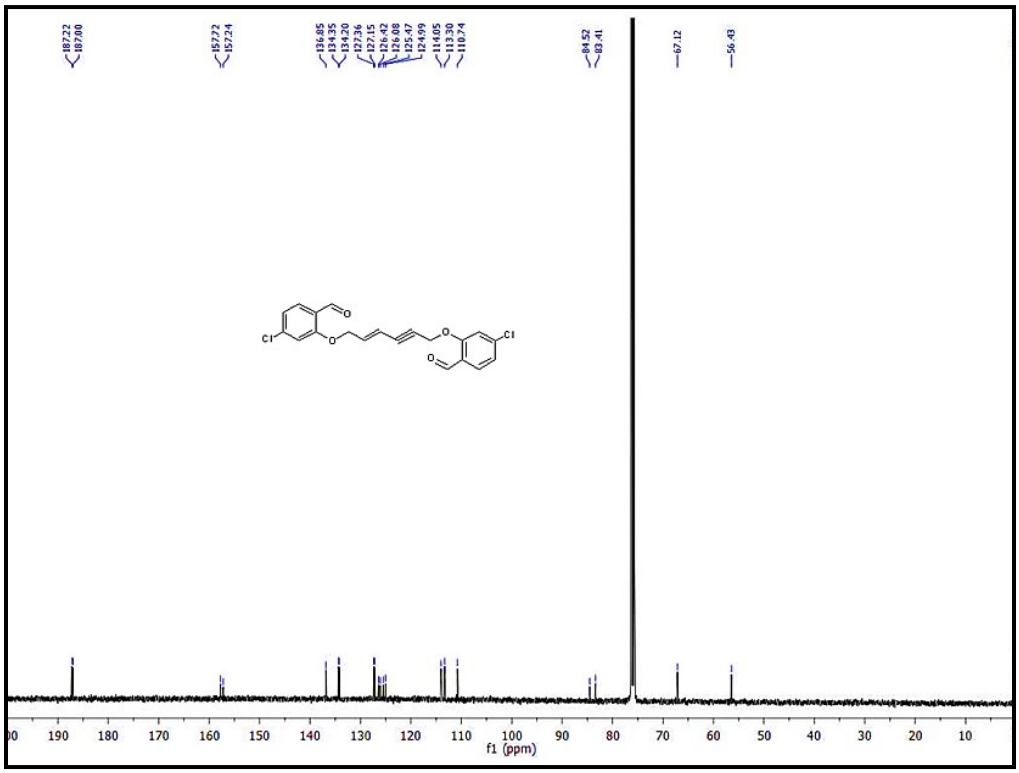
^{1}H NMR Spectrum of **12d**



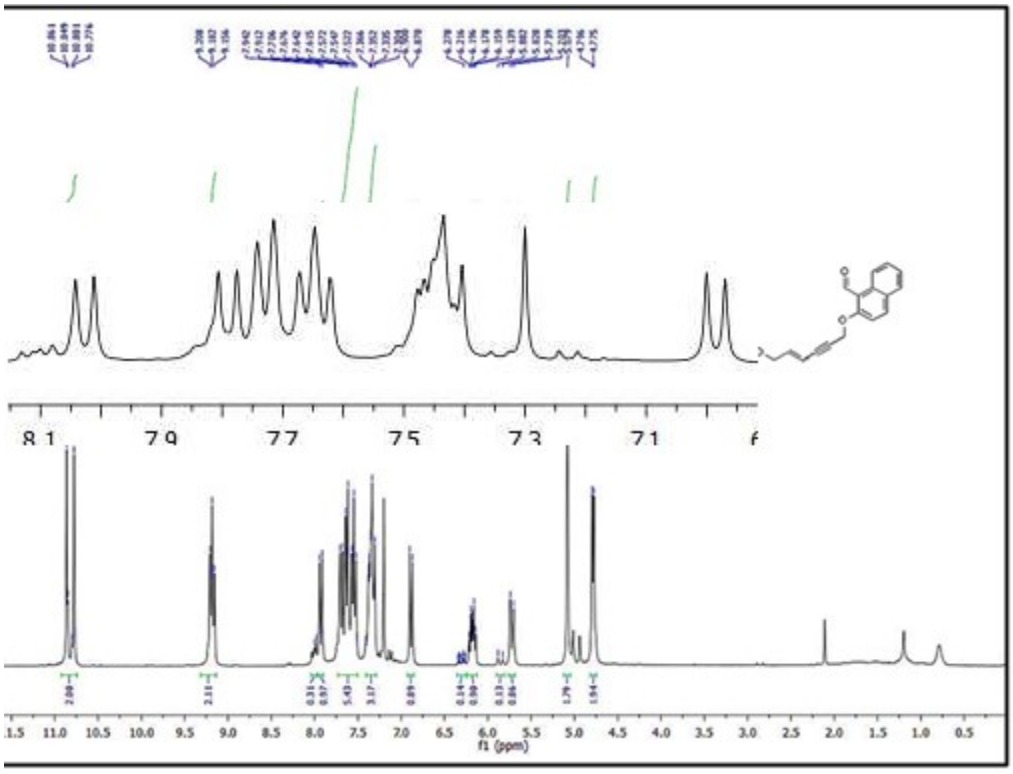
^{13}C NMR Spectrum of **12d**



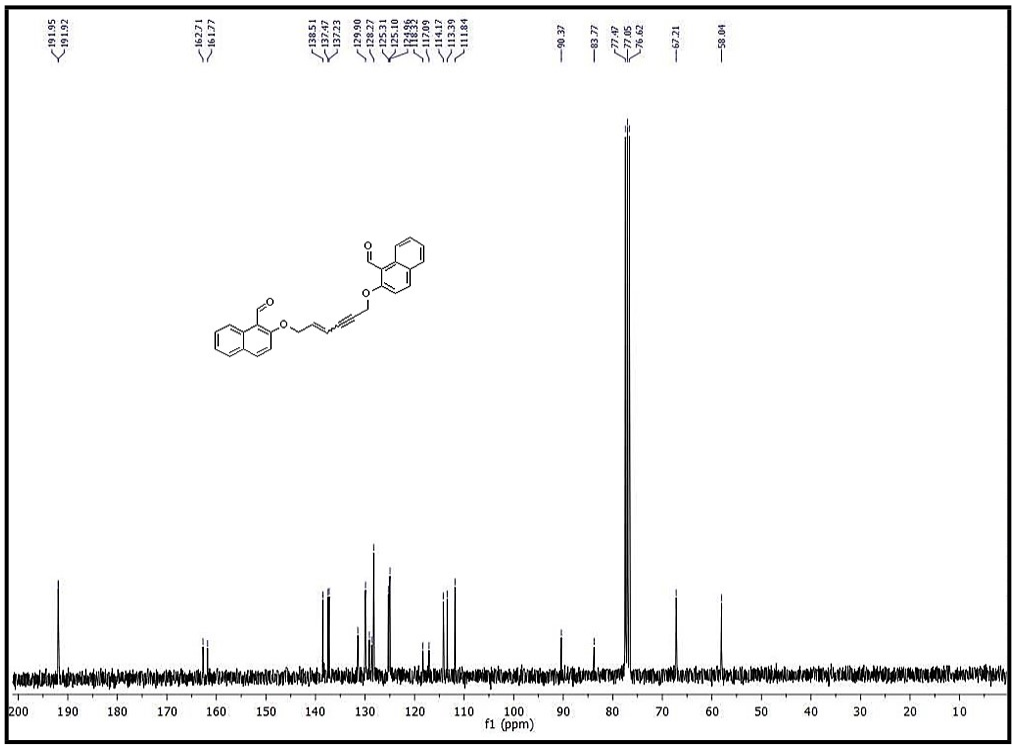
^{1}H NMR Spectrum of **12e**



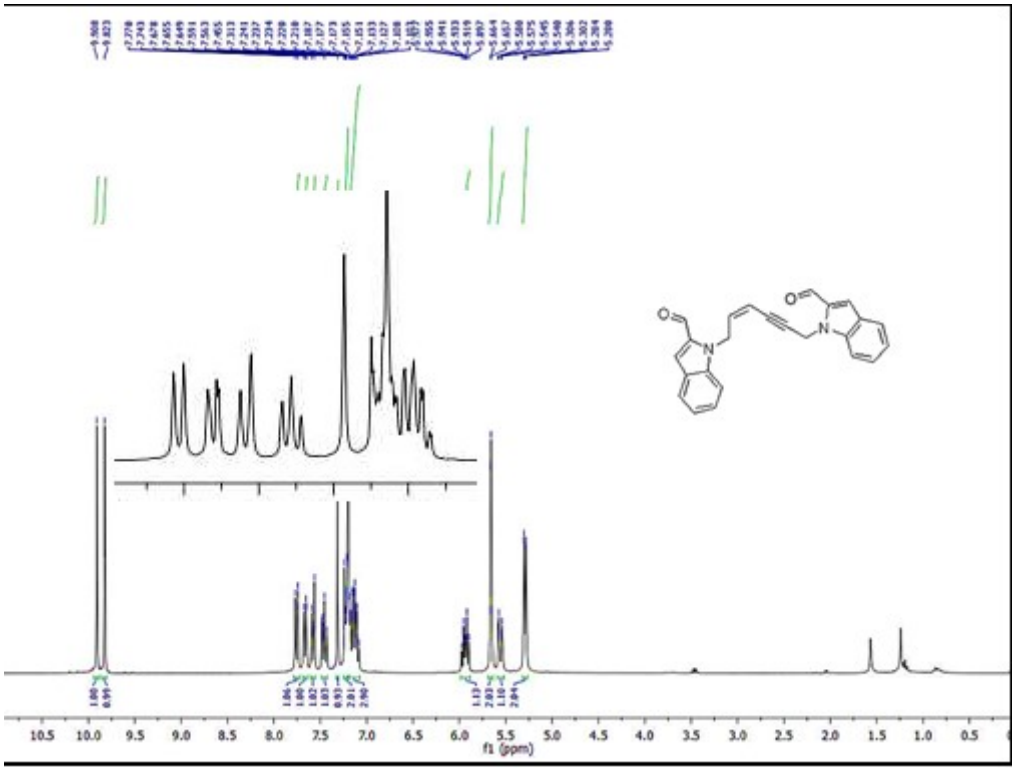
^{13}C NMR Spectrum of **12e**



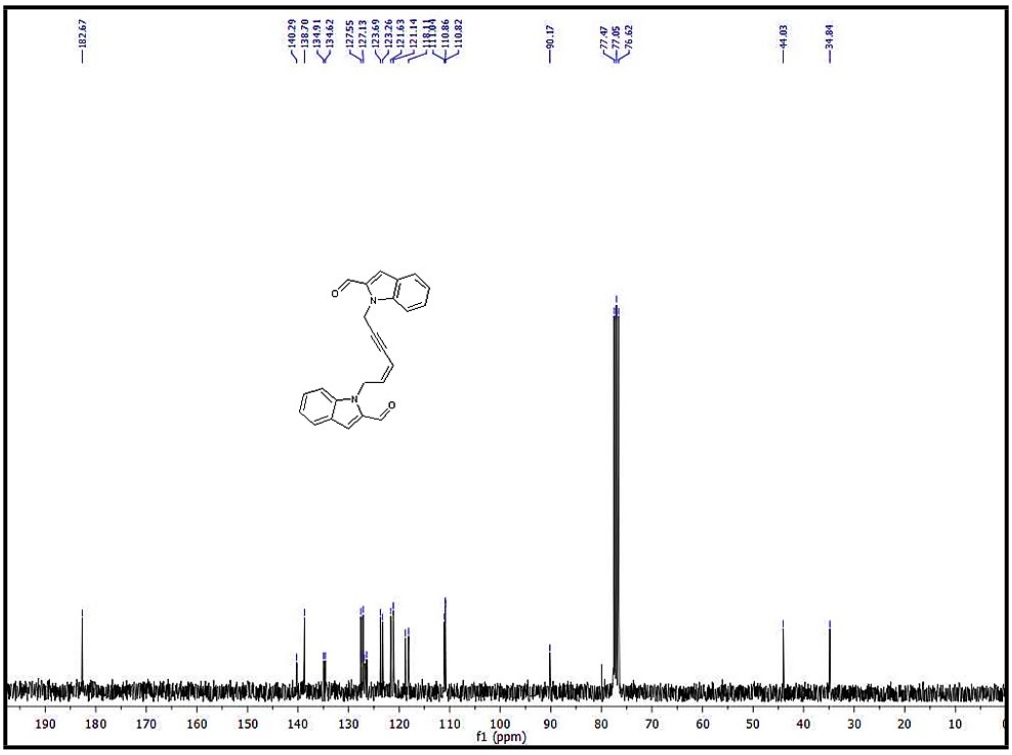
^{1}H NMR Spectrum of **12f**



^{13}C NMR Spectrum of **12f**



^{1}H NMR Spectrum of **12g**



^{13}C NMR Spectrum of **12g**

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