Sustainable Carbon Nanoarchetectonics: Biomass Derived Carbon Nano-Onion/Magnetite Composite as a Reusable Catalyst for Solvent-free Synthesis of Benzoxazinones and Benzthioxazinones

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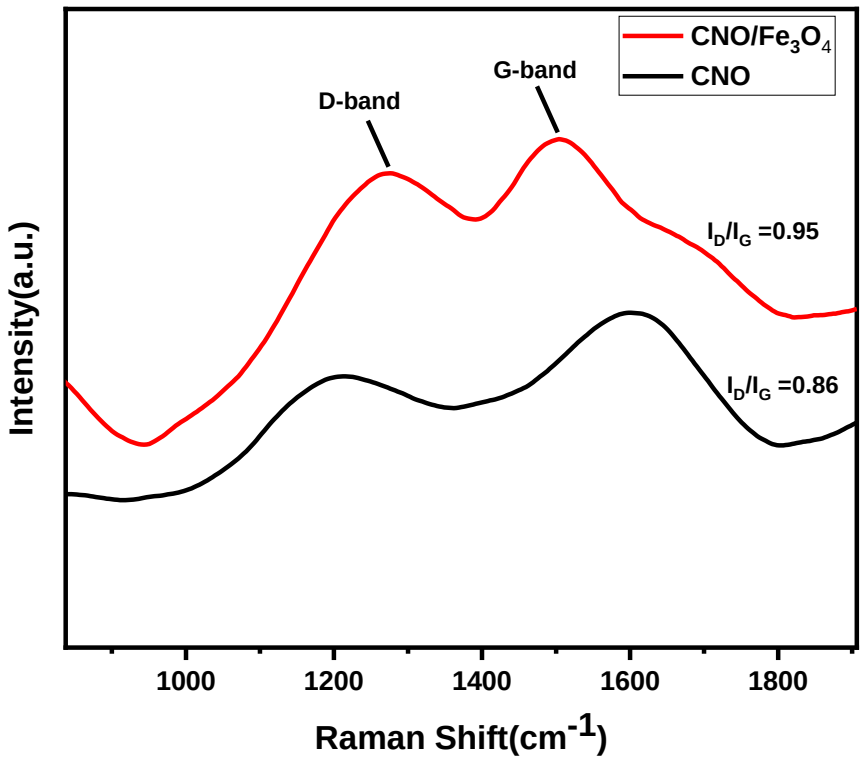


Fig.S1: Raman Spectra of CNO and CNO/Fe_3O_4

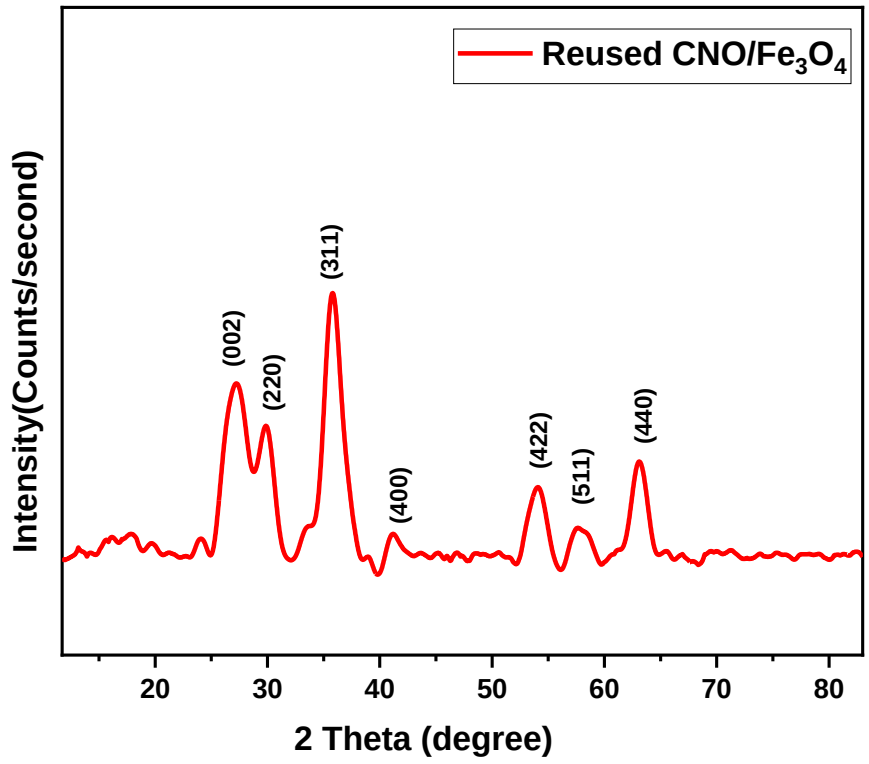


Fig.S2: PXRD Spectrum of recycled CNO/Fe$_3$O$_4$

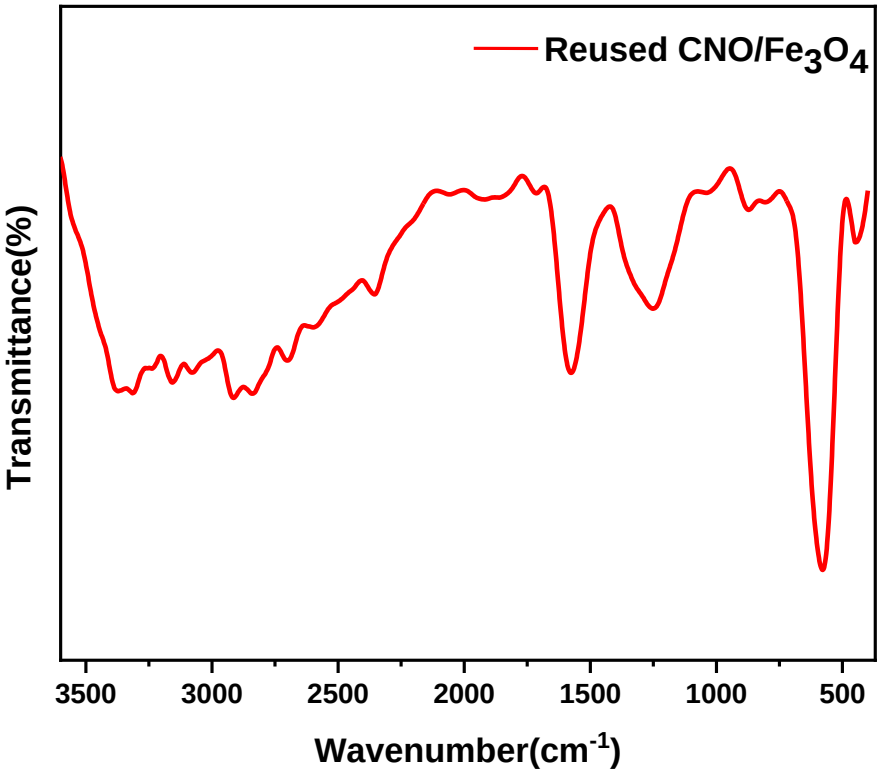
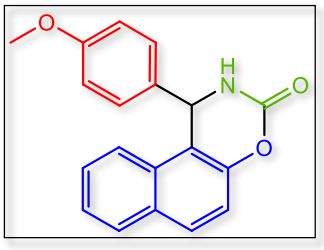


Fig.S3: FTIR Spectrum of recycled CNO/Fe$_3$O$_4$

Spectral data of synthesized products

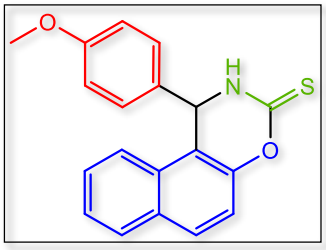
Compound Characterization.

1-(4-methoxyphenyl)-1,2-dihydro-3H-naphtho[1,2-e][1,3]oxazin-3-one(4a)



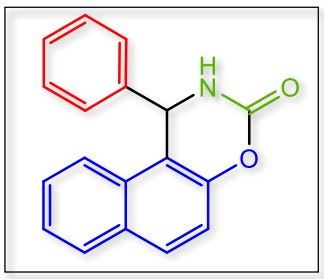
^{1}H NMR (400 MHz, DMSO-d_6) δ 8.78 (d, J = 3.2 Hz, 1H), 8.00 – 7.93 (m, 2H), 7.80 (dt, J = 7.8, 0.8 Hz, 1H), 7.50 – 7.42 (m, 2H), 7.37 (d, J = 8.8 Hz, 1H), 7.23 (d, J = 8.6 Hz, 2H), 6.88 (d, J = 8.8 Hz, 2H), 6.14 (d, J = 3.2 Hz, 1H), 3.69 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 159.34, 149.80, 147.80, 135.51, 130.89, 130.55, 129.37, 129.06, 128.67, 127.75, 125.48, 123.61, 117.32, 114.78, 114.71, 55.57, 53.69. HRMS (ESI) m/z: [M+H]+ calcd for C19H15NO3 306.1125; found 306.1125

1-(4-methoxyphenyl)-1,2-dihydro-3H-naphtho[1,2-e][1,3]oxazine-3-thione(4b)



^{1}H NMR (400 MHz, DMSO-d_6) δ 8.78 (s, 1H), 8.00 – 7.93 (m, 2H), 7.80 (dd, J = 8.2, 1.4 Hz, 1H), 7.49 (s, 2H), 7.37 (d, J = 8.8 Hz, 1H), 7.22 (d, J = 8.8 Hz, 2H), 6.88 (d, J = 8.8 Hz, 2H), 6.14 (d, J = 3.2 Hz, 1H), 3.69 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 159.32, 149.81, 147.78, 135.50, 130.88, 130.56, 129.35, 129.07, 128.67, 127.76, 125.49, 123.61, 117.32, 114.77, 114.70, 55.56, 53.67.

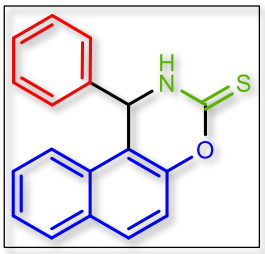
1-phenyl-1,2-dihydro-3H-naphtho[1,2-e][1,3]oxazin-3-one(4c)



^{1}H NMR (400 MHz, DMSO-d_6) δ 8.87 (d, J = 3.1 Hz, 1H), 8.00 (d, J = 9.0 Hz, 1H), 7.95 (dd, J = 7.7, 1.7 Hz, 1H), 7.82 (dd, J = 8.3, 1.4 Hz, 1H), 7.51 – 7.43 (m, 2H), 7.39 (d, J = 8.9 Hz, 1H), 7.33 (d, J = 4.3 Hz, 4H), 7.29 – 7.25 (m, 1H), 6.20 (d, J = 3.1 Hz, 1H). ^{13}C NMR (100

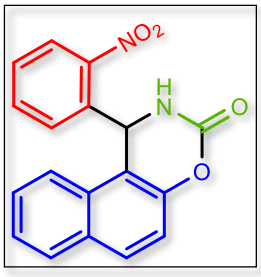
MHz, DMSO-d_6) δ 149.79, 147.90, 143.35, 130.89, 130.70, 129.42, 129.36, 129.10, 128.48, 127.82, 127.44, 125.55, 123.57, 117.34, 114.53, 54.25.

1-phenyl-1,2-dihydro-3H-naphtho[1,2-e][1,3]oxazine-3-thione(4d)



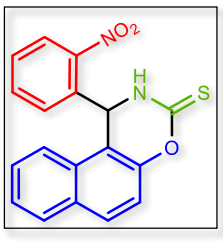
^{1}H NMR (400 MHz, DMSO-d_6) δ 8.89 (d, *J* = 2.8 Hz, 1H), 8.02 (d, *J* = 9.0 Hz, 1H), 7.98 – 7.95 (m, 1H), 7.57 (d, *J* = 1.3 Hz, 1H), 7.51 (td, *J* = 7.2, 1.5 Hz, 2H), 7.46 (d, *J* = 1.5 Hz, 1H), 7.39 (d, *J* = 8.9 Hz, 1H), 7.35 – 7.25 (m, 3H), 7.21 (d, *J* = 2.0 Hz, 1H), 6.52 (d, *J* = 2.9 Hz, 1H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 149.14, 148.30, 139.97, 132.17, 131.12, 130.89, 130.54, 130.30, 129.33, 129.28, 128.82, 128.11, 125.61, 122.75, 117.33, 112.96, 52.21.

1-(2-nitrophenyl)-1,2-dihydro-3H-naphtho[1,2-e][1,3]oxazin-3-one(4e)



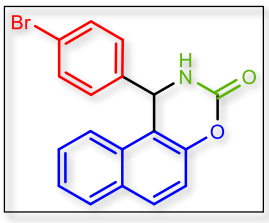
^{1}H NMR (400 MHz, DMSO-d_6) δ 8.77 (d, *J* = 2.8 Hz, 1H), 8.07 – 7.99 (m, 2H), 7.94 – 7.90 (m, 1H), 7.59 – 7.49 (m, 3H), 7.44 – 7.38 (m, 2H), 7.36 (d, *J* = 8.9 Hz, 1H), 7.11 (dd, *J* = 7.7, 1.6 Hz, 1H), 6.91 (d, *J* = 2.8 Hz, 1H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 148.81, 136.57, 135.34, 131.53, 130.34, 129.33, 128.36, 125.88, 125.76, 122.87, 117.35, 112.08, 49.72.

1-(2-nitrophenyl)-1,2-dihydro-3H-naphtho[1,2-e][1,3]oxazine-3-thione(4f)



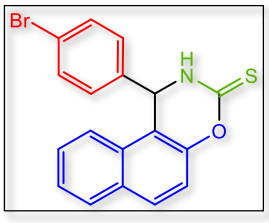
^{1}H NMR (400 MHz, DMSO-d_6) δ 8.83 (d, *J* = 3.6 Hz, 1H), 8.05 (d, *J* = 3.6 Hz, 2H), 7.97 (s, 1H), 7.74 (dd, *J* = 10.2, 4.6 Hz, 1H), 7.62 (d, *J* = 4.8 Hz, 1H), 7.47 – 7.44 (m, 2H), 7.40 (t, *J* = 3.8 Hz, 2H), 7.17 (t, *J* = 3.6 Hz, 1H), 6.97 (d, *J* = 3.6 Hz, 1H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 148.82, 148.01, 136.58, 135.33, 131.54, 130.96, 130.34, 130.22, 129.33, 128.36, 125.89, 125.77, 122.87, 117.35, 112.10, 49.73.

1-(4-bromophenyl)-1,2-dihydro-3H-naphtho[1,2-e][1,3]oxazin-3-one(4g)



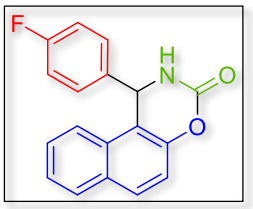
^{1}H NMR (400 MHz, DMSO-d_6) δ 8.94 (d, *J* = 3.2 Hz, 1H), 8.05 (d, *J* = 9.2 Hz, 1H), 8.00 (dd, *J* = 7.6, 1.8 Hz, 1H), 7.83 (d, *J* = 8.2 Hz, 1H), 7.61 – 7.56 (m, 2H), 7.54 – 7.48 (m, 2H), 7.43 (d, *J* = 8.8 Hz, 1H), 7.33 – 7.29 (m, 2H), 6.28 (d, *J* = 3.2 Hz, 1H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 149.61, 147.94, 142.63, 132.36, 130.90, 129.71, 129.26, 129.15, 127.94, 125.64, 123.50, 121.67, 117.36, 113.98, 53.55.

1-(4-bromophenyl)-1,2-dihydro-3H-naphtho[1,2-e][1,3]oxazine-3-thione(4h)



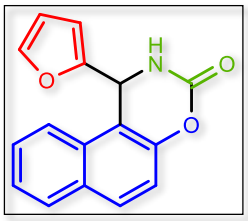
^{1}H NMR (400 MHz, DMSO-d_6) δ 8.90 (d, *J* = 3.2 Hz, 1H), 8.00 (d, *J* = 9.2 Hz, 1H), 7.95 (dd, *J* = 7.6, 1.8Hz, 1H), 7.80 – 7.77 (m, 1H), 7.55 – 7.52 (m, 2H), 7.49 – 7.43 (m, 2H), 7.38 (d, *J* = 8.8 Hz, 1H), 7.29 – 7.25 (m, 2H), 6.23 (d, *J* = 3.2 Hz, 1H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 149.61, 147.95, 142.64, 132.36, 132.18, 131.77, 130.90, 129.71, 129.26, 129.15, 127.93, 125.63, 123.50, 121.67, 117.35, 113.98, 53.55.

1-(4-fluorophenyl)-1,2-dihydro-3H-naphtho[1,2-e][1,3]oxazin-3-one(4i)



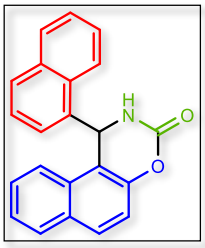
^{1}H NMR (400 MHz,DMSO-d_6) δ 8.88 (d, *J* = 3.2 Hz, 1H), 8.00 (d, *J* = 9.2 Hz, 1H), 7.96 (dd, *J* = 7.6, 1.8 Hz, 1H), 7.82 – 7.78 (m, 1H), 7.48 (ddd, *J* = 9.2, 7.6, 1.4 Hz, 2H), 7.40 – 7.33 (m, 3H), 7.17 (t, *J* = 8.8 Hz, 2H), 6.24 (d, *J* = 3.2 Hz, 1H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 160.84, 149.68, 147.88, 139.62, 130.90, 130.82, 129.61, 129.53, 129.28, 129.13, 127.89, 125.60, 123.53, 117.35, 116.33, 116.12, 114.32, 53.42.

1-(furan-2-yl)-1,2-dihydro-3H-naphtho[1,2-e][1,3]oxazin-3-one(4j)



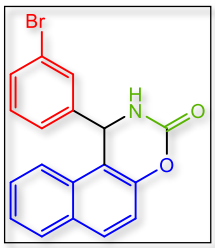
^{1}H NMR (400 MHz, DMSO-d$_6$) δ 8.86 (d, *J* = 3.2 Hz, 1H), 8.01 – 7.94 (m, 3H), 7.54 (dt, *J* = 6.4, 1.6 Hz, 2H), 7.50 – 7.45 (m, 1H), 7.33 (d, *J* = 8.8 Hz, 1H), 6.46 (d, *J* = 3.2 Hz, 1H), 6.38 – 6.33 (m, 2H). ^{13}C NMR (100 MHz, DMSO-d$_6$) δ 154.21, 150.33, 148.03, 143.59, 130.78, 130.74, 129.34, 129.05, 127.86, 125.59, 123.28, 117.27, 112.47, 110.99, 107.70, 48.94.

1-(naphthalen-1-yl)-1,2-dihydro-3H-naphtho[1,2-e][1,3]oxazin-3-one(4k)



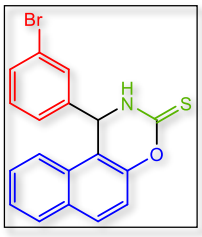
^{1}H NMR (400 MHz, DMSO-d$_6$) δ 8.93 (s, 1H), 8.71 (d, *J* = 8.6 Hz, 1H), 8.03 (dd, *J* = 13.2, 8.6 Hz, 2H), 7.96 (d, *J* = 8.2 Hz, 1H), 7.88 (d, *J* = 8.2 Hz, 1H), 7.73 (d, *J* = 7.8 Hz, 1H), 7.65 (d, *J* = 7.6 Hz, 1H), 7.48 – 7.31 (m, 5H), 7.13 (s, 1H), 7.02 (d, *J* = 7.2 Hz, 1H). ^{13}C NMR (100 MHz, DMSO-d$_6$) δ 149.66, 148.61, 138.86, 134.12, 130.94, 130.78, 130.46, 129.55, 129.27, 129.16, 127.80, 127.40, 126.71, 126.32, 125.77, 125.53, 123.94, 123.27, 117.32, 114.64, 50.11.

1-(3-bromophenyl)-1,2-dihydro-3H-naphtho[1,2-e][1,3]oxazin-3-one(4l)



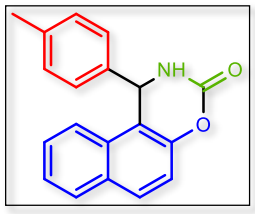
^{1}H NMR (400 MHz, DMSO-d$_6$) δ 8.92 (d, *J* = 3.2 Hz, 1H), 8.01 (d, *J* = 8.8 Hz, 1H), 7.95 (dd, *J* = 7.8, 1.4 Hz, 1H), 7.82 (dd, *J* = 8.4, 1.4 Hz, 1H), 7.61 (t, *J* = 1.8 Hz, 1H), 7.53 – 7.45 (m, 3H), 7.39 (d, *J* = 8.8 Hz, 1H), 7.27 (t, *J* = 7.8 Hz, 1H), 7.23 – 7.19 (m, 1H), 6.26 (d, *J* = 3.2 Hz, 1H). ^{13}C NMR (100 MHz, DMSO-d$_6$) δ 149.62, 148.04, 145.80, 131.77, 131.43, 131.01, 130.89, 130.40, 129.27, 129.16, 128.01, 126.33, 125.69, 123.50, 122.49, 117.36, 113.76, 53.53.

1-(3-bromophenyl)-1,2-dihydro-3H-naphtho[1,2-e][1,3]oxazine-3-thione(4m)



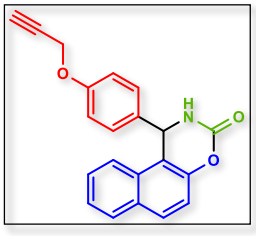
^{1}H NMR (400 MHz, DMSO-d_6) δ 8.92 (d, *J* = 3.2 Hz, 1H), 8.01 (d, *J* = 8.8 Hz, 1H), 7.95 (d, *J* = 7.6 Hz, 1H), 7.82 (d, *J* = 8.2 Hz, 1H), 7.60 (t, *J* = 1.8 Hz, 1H), 7.47 (dd, *J* = 7.8, 5.6 Hz, 3H), 7.39 (d, *J* = 8.8 Hz, 1H), 7.27 (s, 1H), 7.22 (s, 1H), 6.26 (d, *J* = 3.2 Hz, 1H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 149.16, 147.58, 145.34, 135.60, 133.09, 131.76, 131.32, 130.98, 130.90, 130.55, 130.43, 129.93, 128.80, 128.71, 128.29, 127.56, 125.88, 125.23, 123.03, 122.03, 121.74, 116.90, 113.29, 53.07.

1-(p-tolyl)-1,2-dihydro-3H-naphtho[1,2-e][1,3]oxazin-3-one(4n)



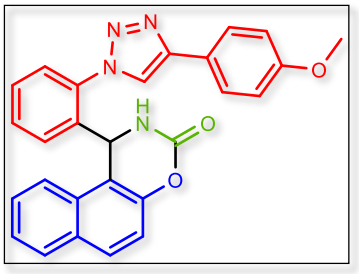
^{1}H NMR (400 MHz, DMSO-d_6) δ 8.81 (d, *J* = 3.2 Hz, 1H), 7.97 (d, *J* = 8.8 Hz, 1H), 7.93 (dd, *J* = 8.2, 1.4 Hz, 1H), 7.79 (dd, *J* = 8.2, 1.4 Hz, 1H), 7.45 (ddd, *J* = 9.2, 7.8, 1.4 Hz, 2H), 7.37 (d, *J* = 8.8 Hz, 1H), 7.20 – 7.18 (m, 2H), 7.12 (d, *J* = 7.8 Hz, 2H), 6.14 (d, *J* = 3.2 Hz, 1H), 2.22 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 149.30, 147.36, 139.97, 137.28, 130.38, 130.10, 129.40, 128.88, 128.65, 128.57, 128.30, 127.26, 126.84, 125.72, 124.99, 123.09, 116.81, 114.15, 53.53, 20.58.

1-(4-(prop-2-yn-1-yloxy)phenyl)-1,2-dihydro-3H-naphtho[1,2-e][1,3]oxazin-3-one(4o)



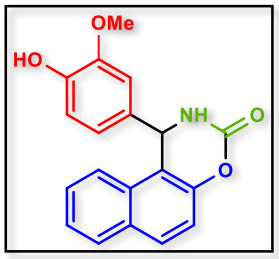
^{1}H NMR (400 MHz, DMSO-d_6) δ 8.80 (d, *J* = 3.2 Hz, 1H), 7.97 (dd, *J* = 15.2, 8.4 Hz, 2H), 7.81 (d, *J* = 8.2 Hz, 1H), 7.47 (t, *J* = 9.2 Hz, 2H), 7.38 (d, *J* = 8.8 Hz, 1H), 7.24 (d, *J* = 8.2 Hz, 3H), 6.94 (d, *J* = 8.2 Hz, 2H), 6.15 (d, *J* = 3.2 Hz, 1H), 4.74 (d, *J* = 2.2 Hz, 2H), 3.52 (s, 1H).^{13}C NMR (100 MHz, DMSO-d_6) δ 157.24, 149.80, 147.80, 136.25, 132.18, 132.00, 130.88, 130.60, 129.34, 129.08, 128.64, 127.80, 125.52, 123.60, 117.33, 115.61, 114.67, 79.59, 78.75, 55.83, 53.63

1-(2-(4-(4-methoxyphenyl)-1H-1,2,3-triazol-1-yl)phenyl)-1,2-dihydro-3H-naphtho[1,2-e][1,3]oxazin-3-one(4p)



^{1}H NMR (400 MHz, DMSO-d_6) δ 8.62 (s, 1H), 8.50 (s, 1H), 7.95 – 7.93 (m, 1H), 7.88 (d, *J* = 8.6 Hz, 2H), 7.82 (s, 2H), 7.64 (s, 2H), 7.53 – 7.51 (m, 1H), 7.31 (s, 1H), 7.20 (s, 2H), 7.07 – 7.04 (m, 4H), 3.81 (s, 3H).^{13}C NMR (100 MHz, DMSO-d_6) δ 159.66, 159.57, 152.87, 146.60, 134.82, 130.11, 129.67, 129.04, 127.92, 127.34, 123.49, 123.30, 122.59, 122.47, 119.27, 114.83, 114.54, 55.66, 48.91.

1-(4-hydroxy-3-methoxyphenyl)-1,2-dihydro-3H-naphtho[1,2-e][1,3]oxazin-3-one(4q)



^{1}H NMR (400 MHz, DMSO) δ 9.07 (s, 1H), 8.73 (d, *J* = 3.0 Hz, 1H), 7.99 – 7.93 (m, 2H), 7.82 (d, *J* = 8.2 Hz, 1H), 7.47 (ddd, *J* = 9.6, 7.8, 1.4 Hz, 2H), 7.37 (d, *J* = 8.8 Hz, 1H), 7.02 (d, *J* = 2.2 Hz, 1H), 6.67 (d, *J* = 8.2 Hz, 1H), 6.52 (dd, *J* = 8.2, 2.2 Hz, 1H), 6.08 (d, *J* = 3.2 Hz, 1H), 3.73 (s, 3H). ^{13}C NMR (100 MHz, DMSO) δ 167.44, 149.87, 148.08, 147.81, 146.80, 134.31, 130.85, 130.49, 129.50, 129.03, 127.72, 125.48, 123.73, 119.58, 117.28, 116.11, 114.73, 112.08, 56.12, 54.09.

^{1}H NMR & ^{13}C NMR spectra

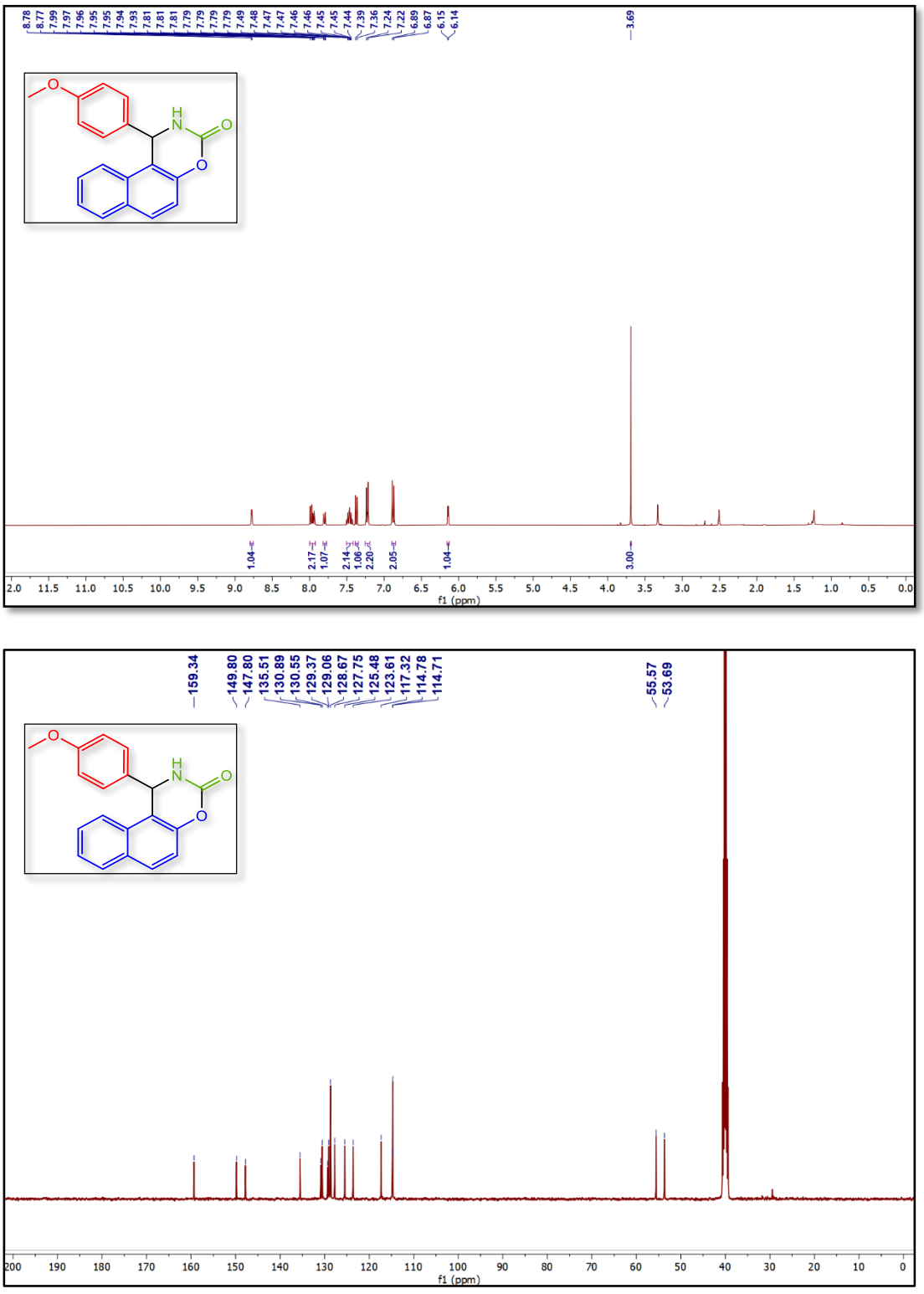


Fig. S4: ^{1}H NMR & ^{13}C NMR Spectra of 4a

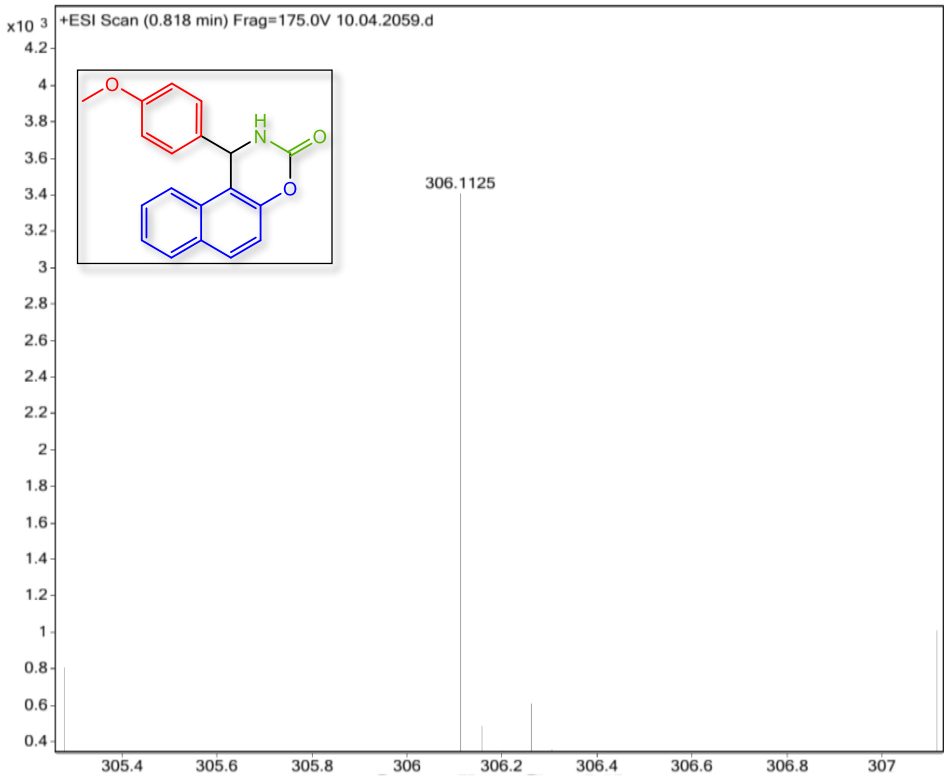


Fig. S5: HRMS Spectrum of 4a

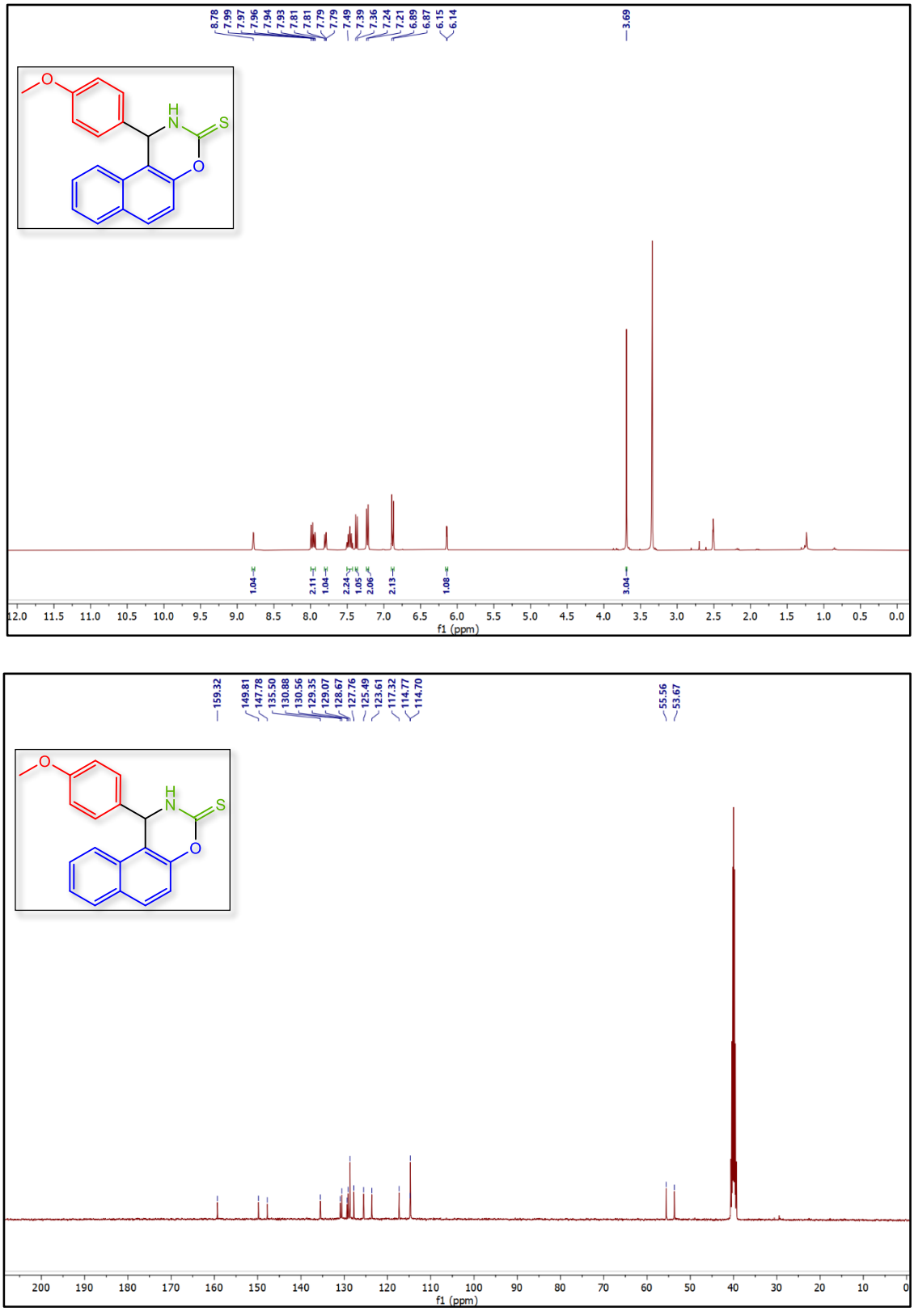
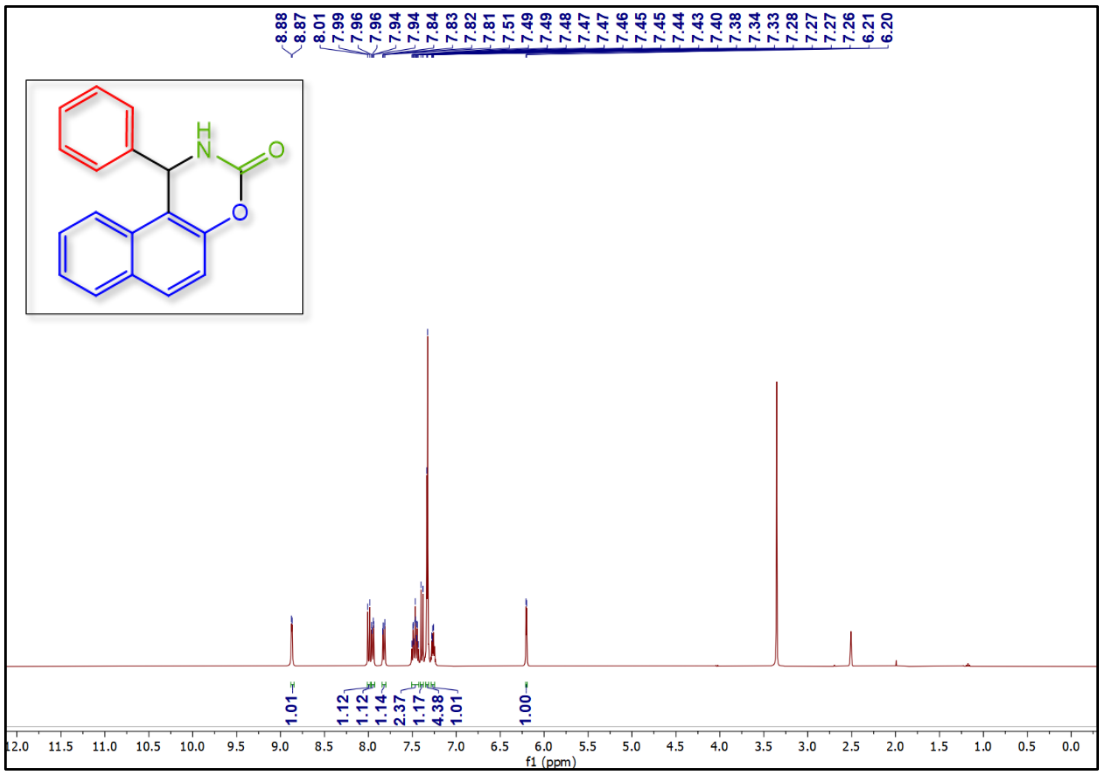


Fig. S6: ^{1}H NMR & ^{13}C NMR Spectra of 4b



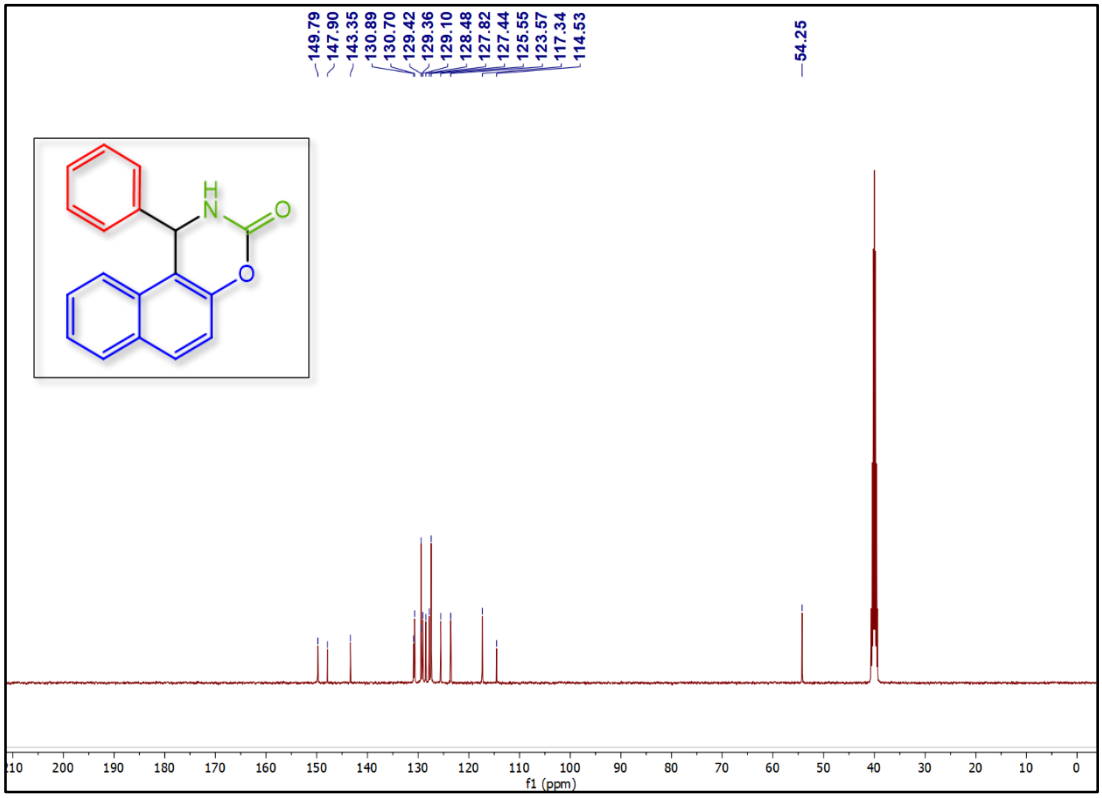


Fig. S7: ^{1}H NMR & ^{13}C NMR Spectra of 4c

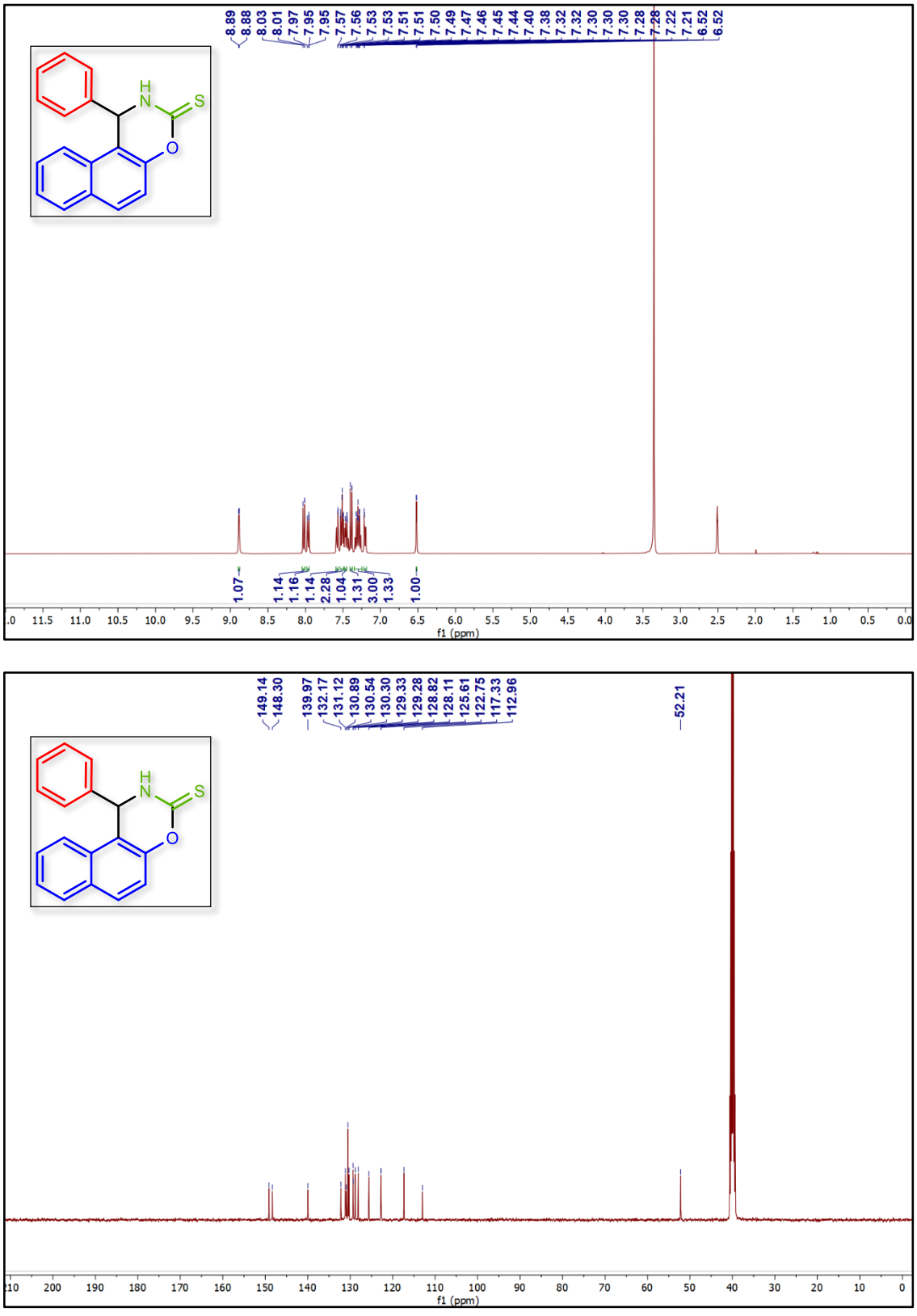


Fig. S8: ^{1}H NMR & ^{13}C NMR Spectra of 4d

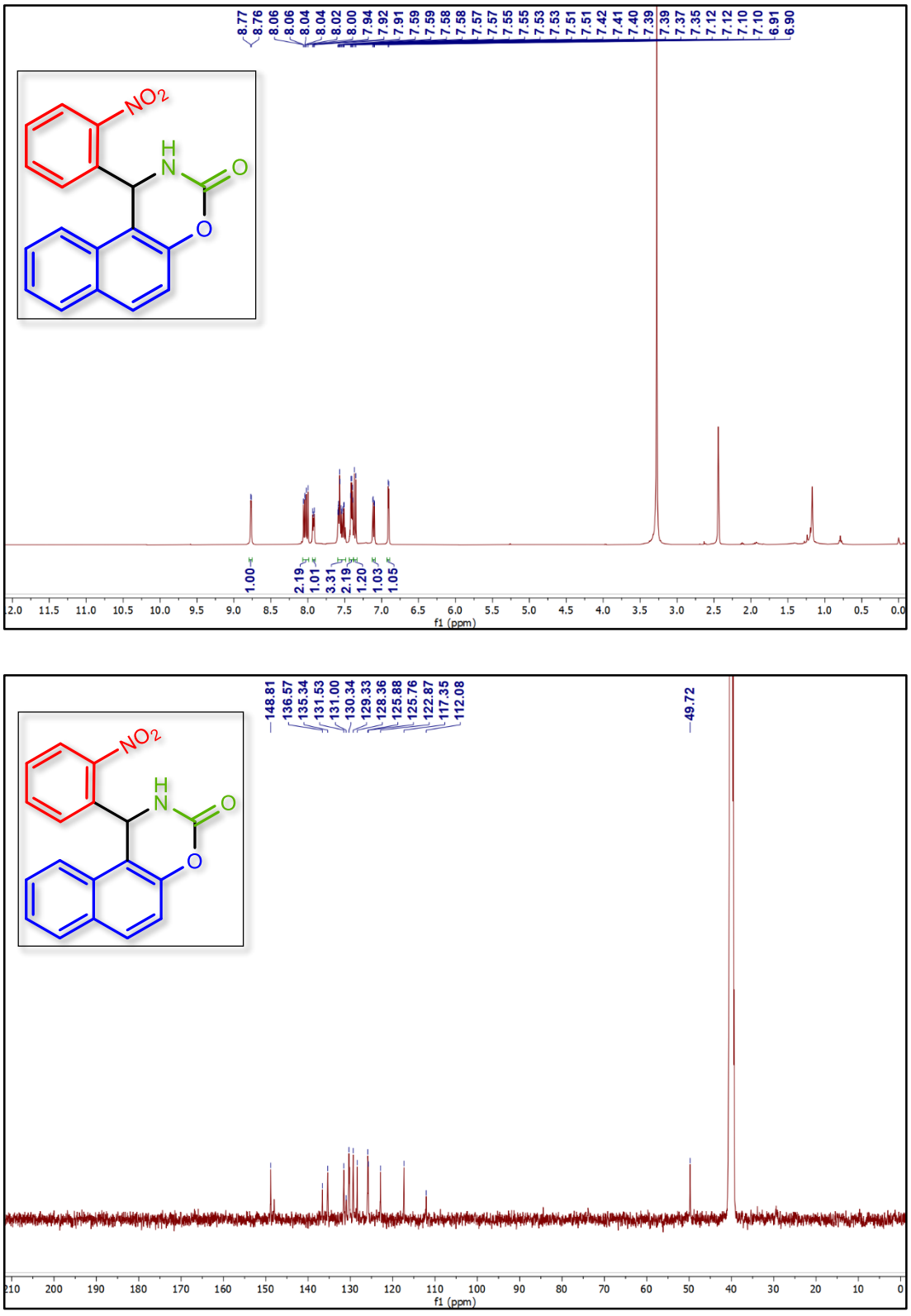


Fig. S9: ^{1}H NMR & ^{13}C NMR Spectra of 4e

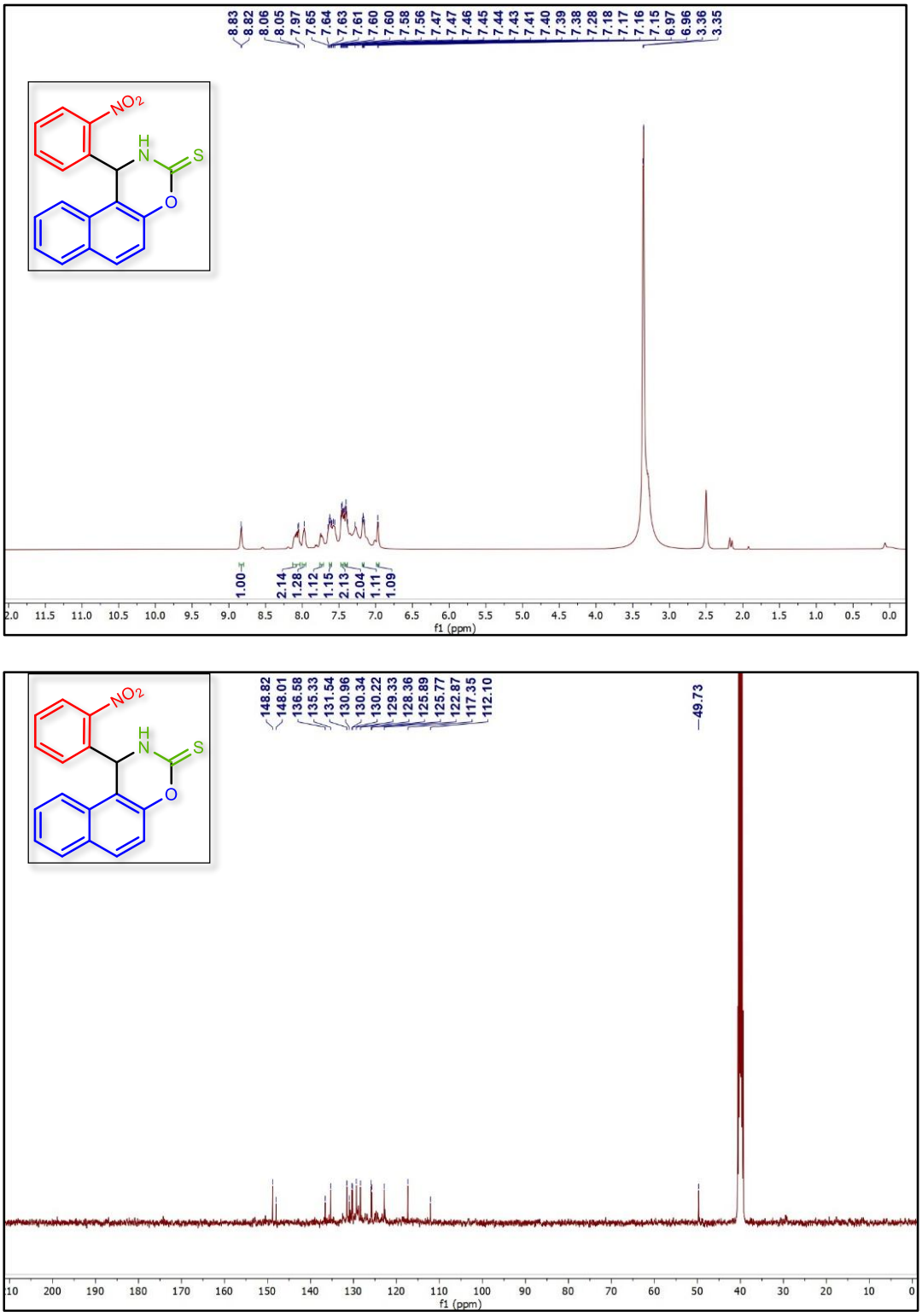


Fig. S10: ^{1}H NMR & ^{13}C NMR Spectra of 4f

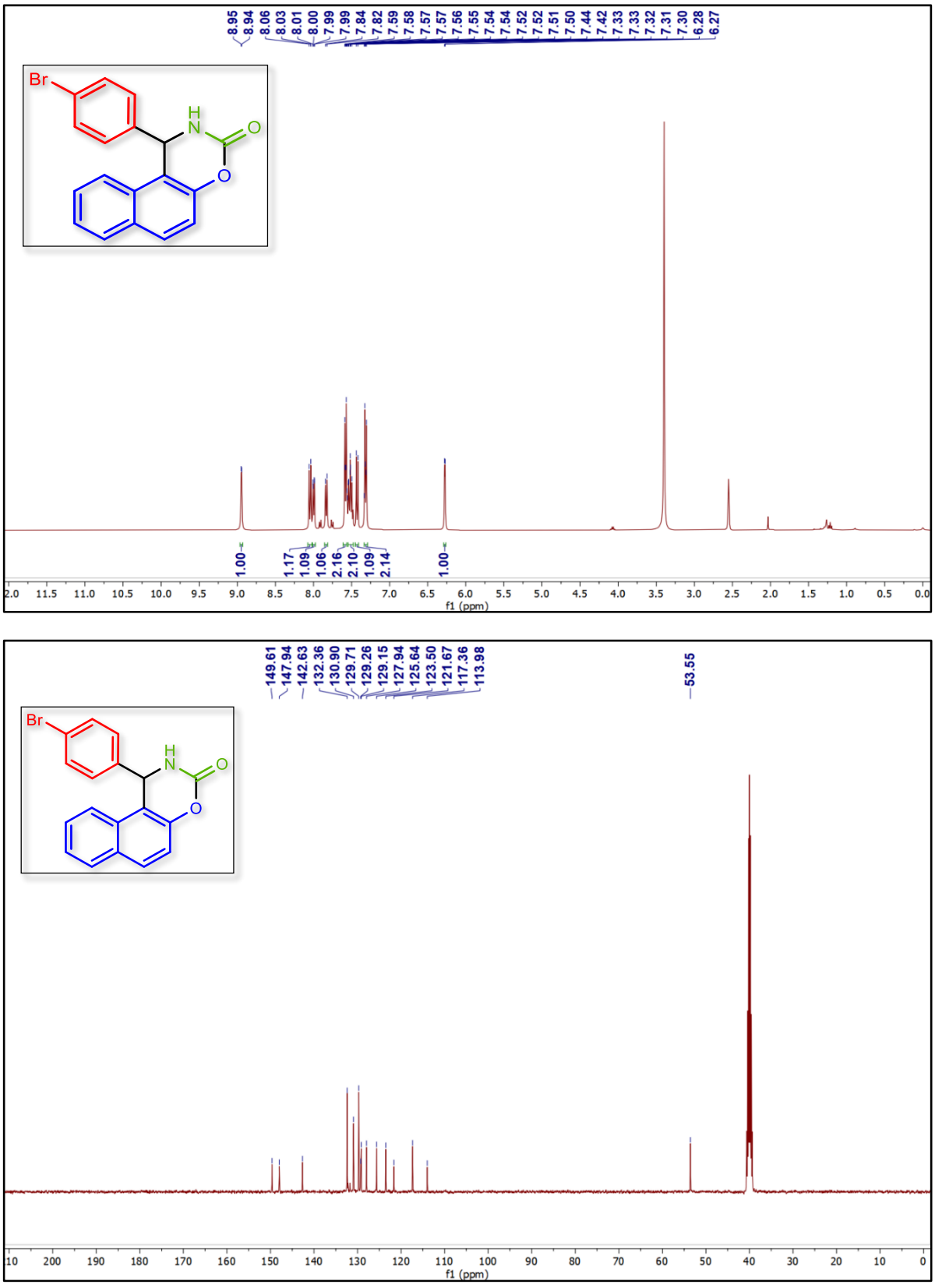


Fig. S11: ^{1}H NMR & ^{13}C NMR Spectra of 4g

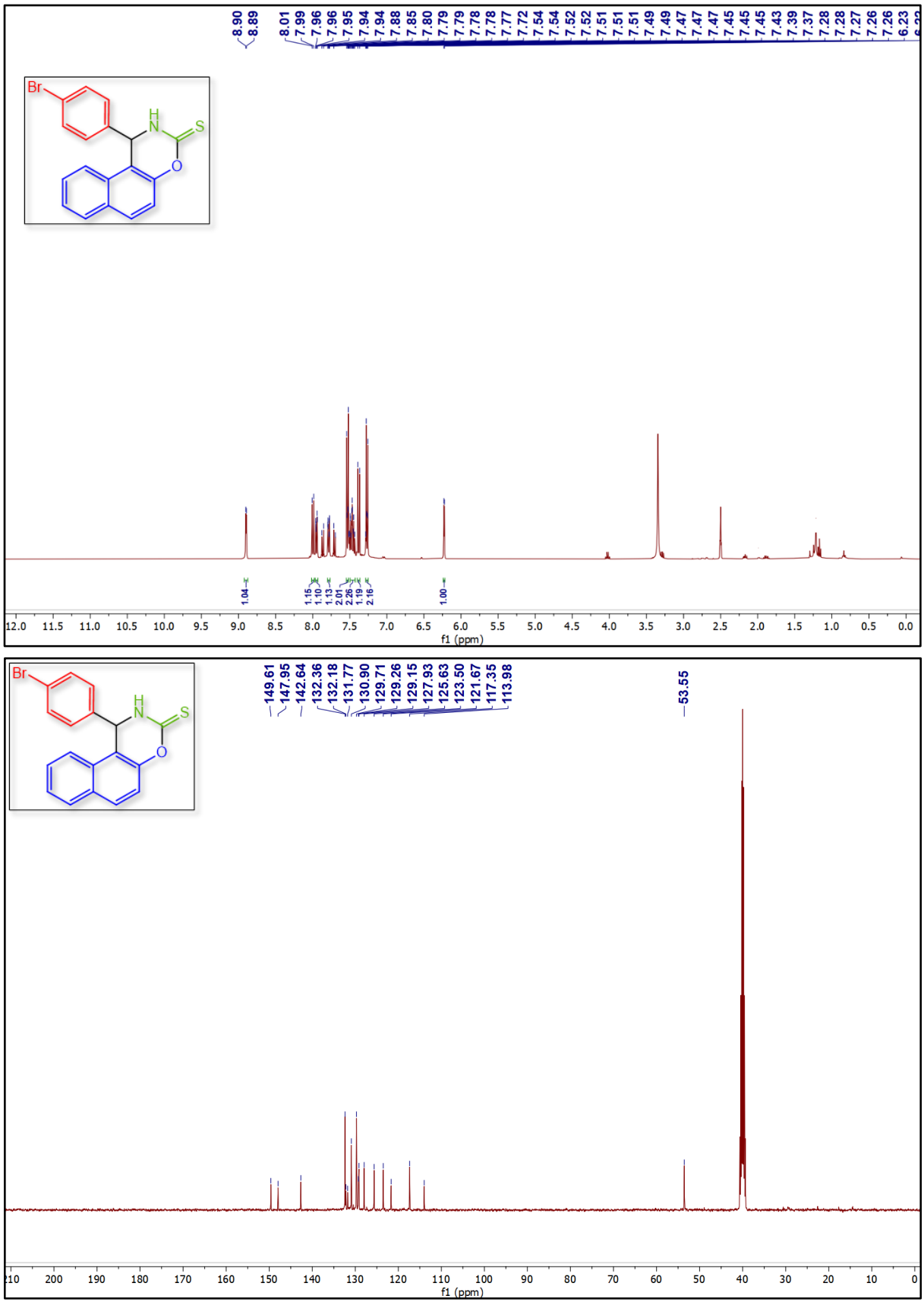
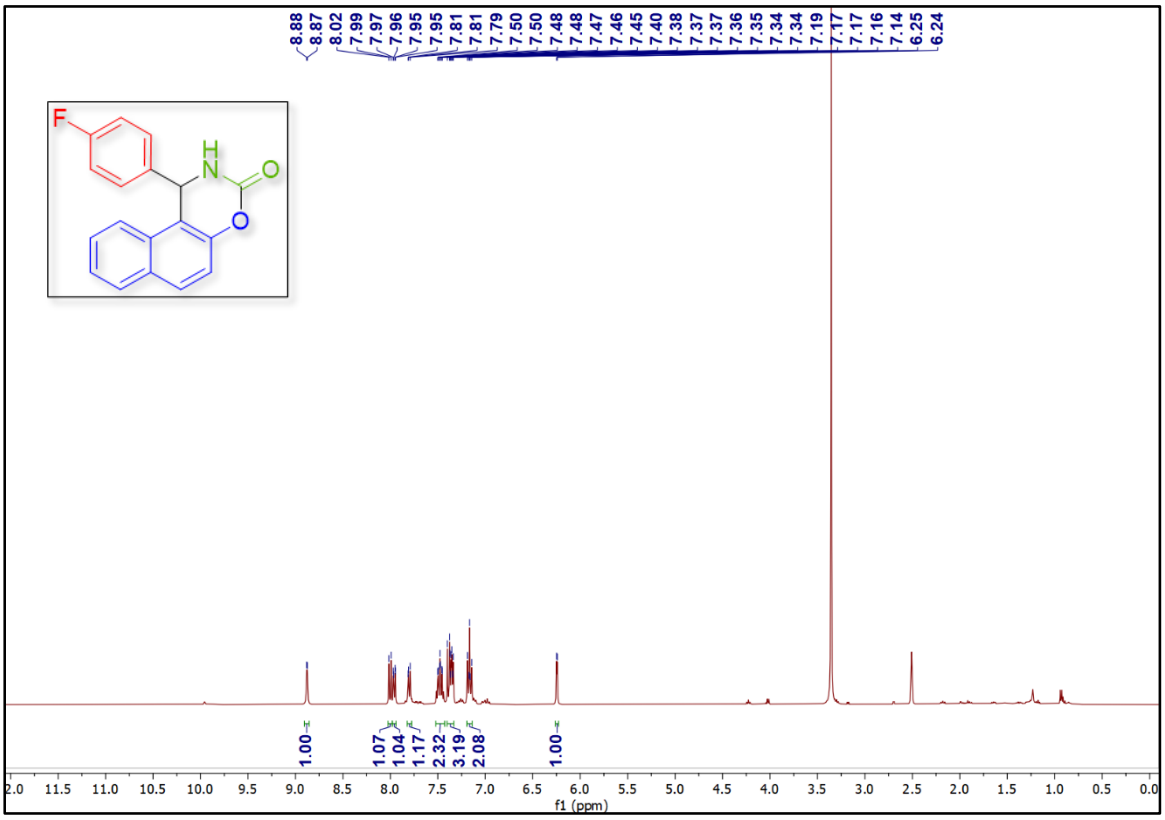


Fig. S12: ^{1}H NMR & ^{13}C NMR Spectra of 4h



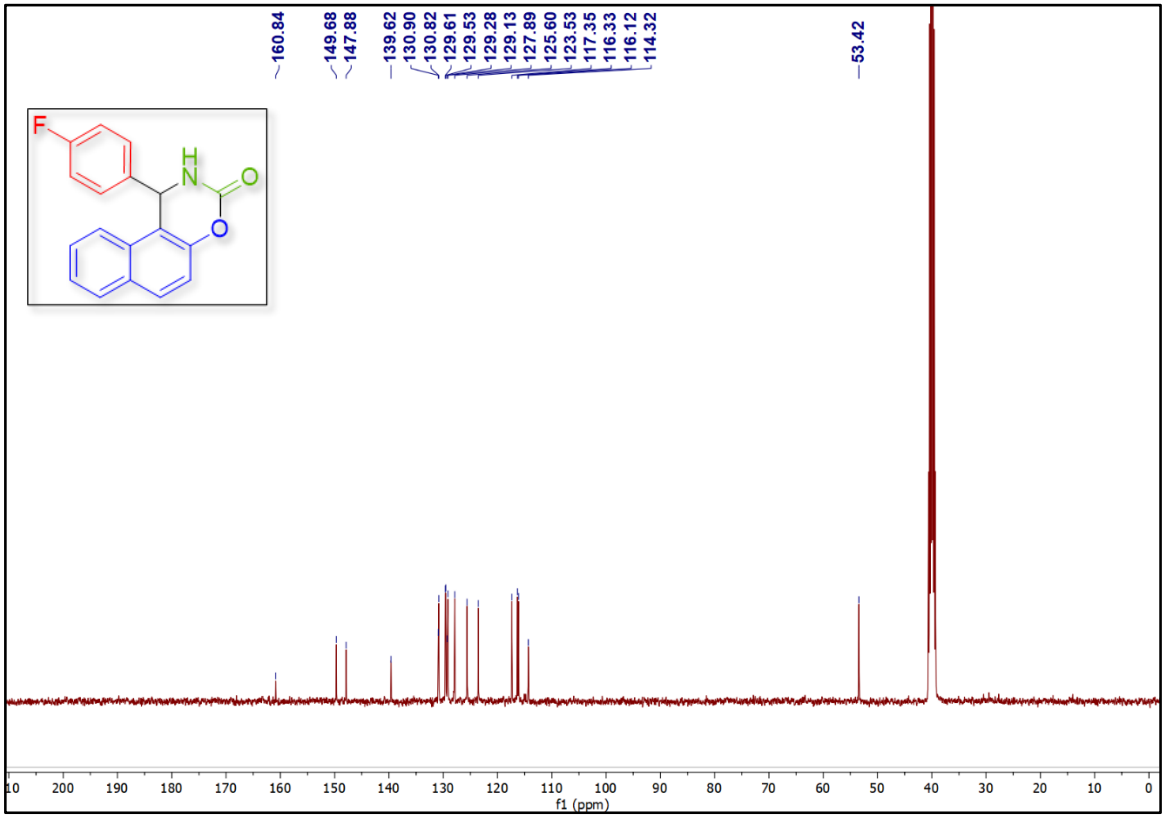


Fig. S13: ^{1}H NMR & ^{13}C NMR Spectra of 4i

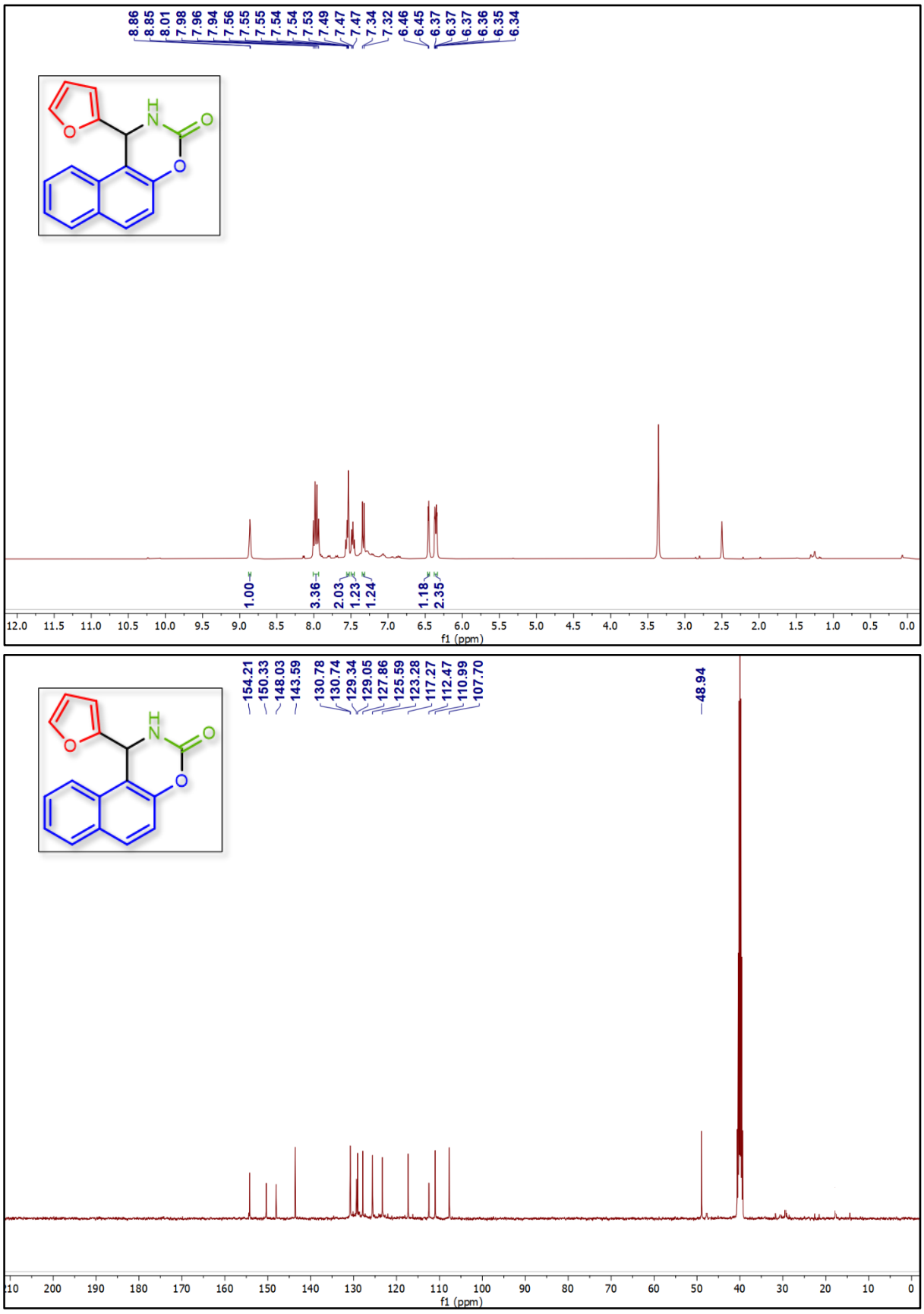
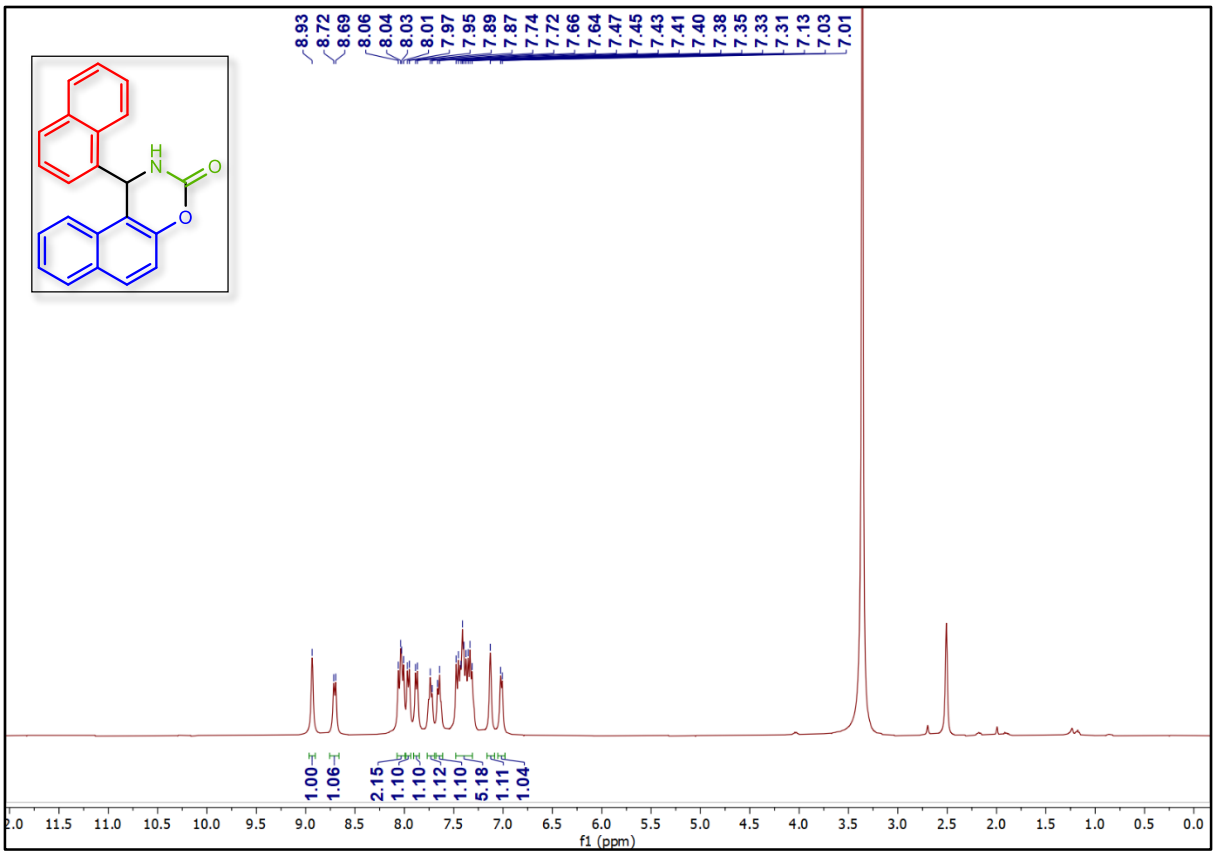


Fig. S14: ^{1}H NMR & ^{13}C NMR Spectra of 4j



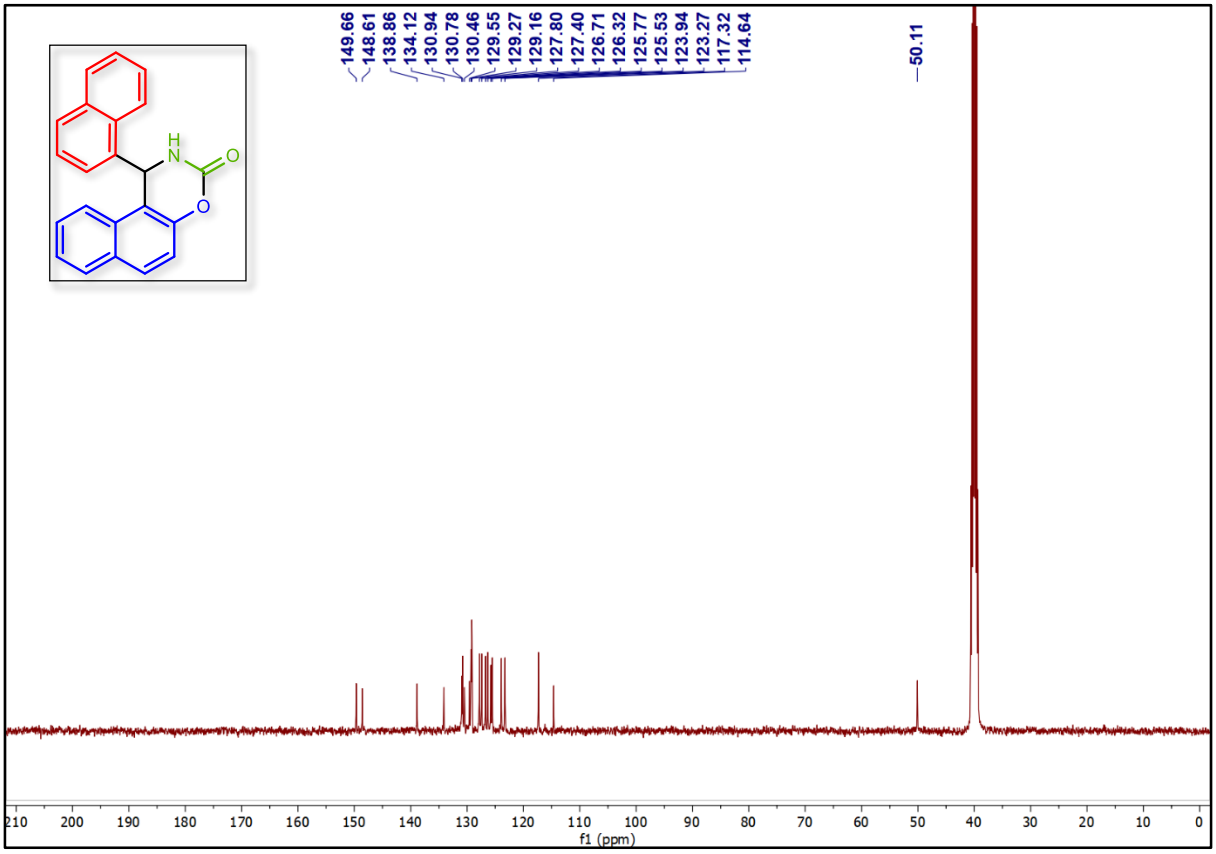


Fig. S15: ^{1}H NMR & ^{13}C NMR Spectra of 4k

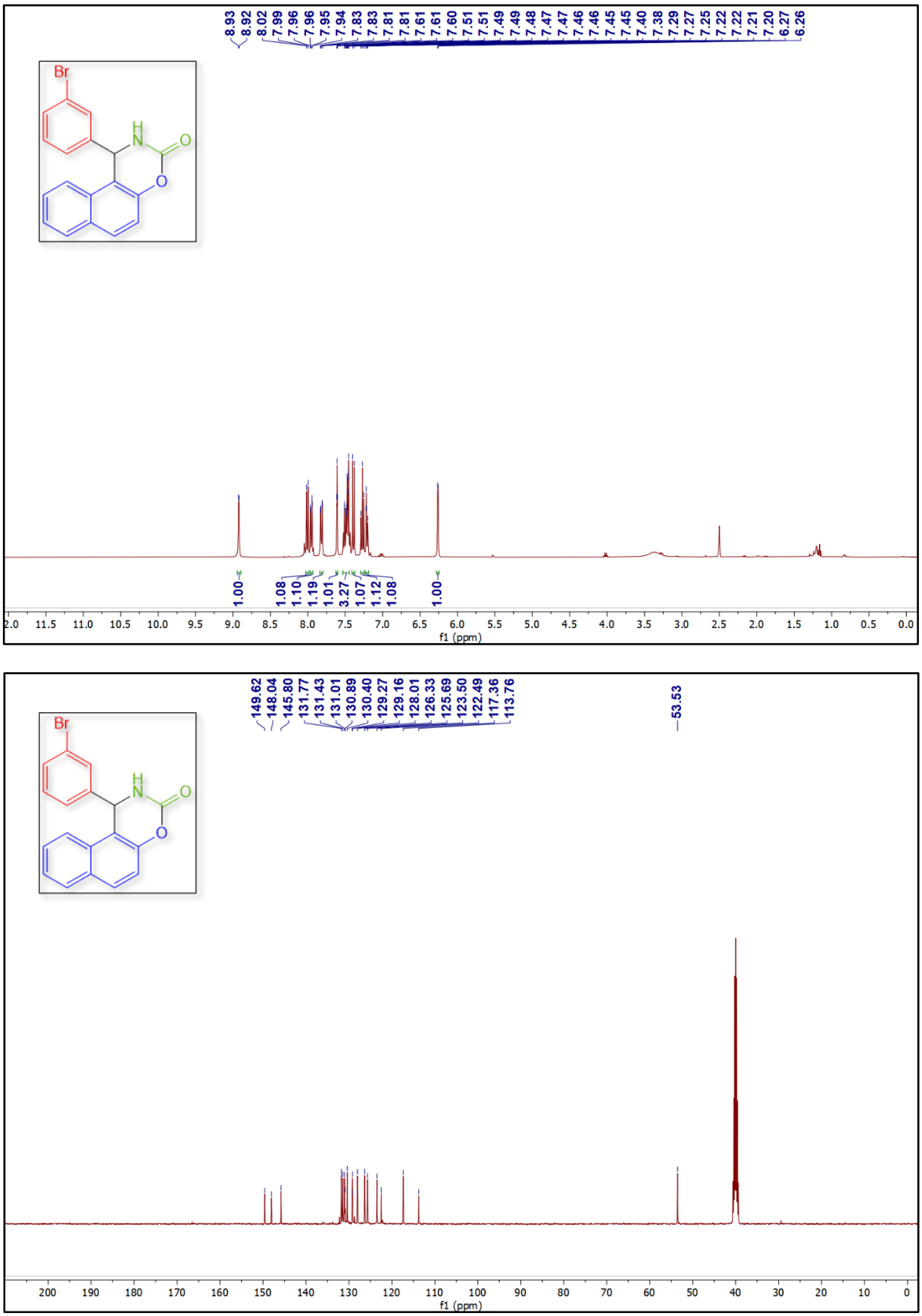
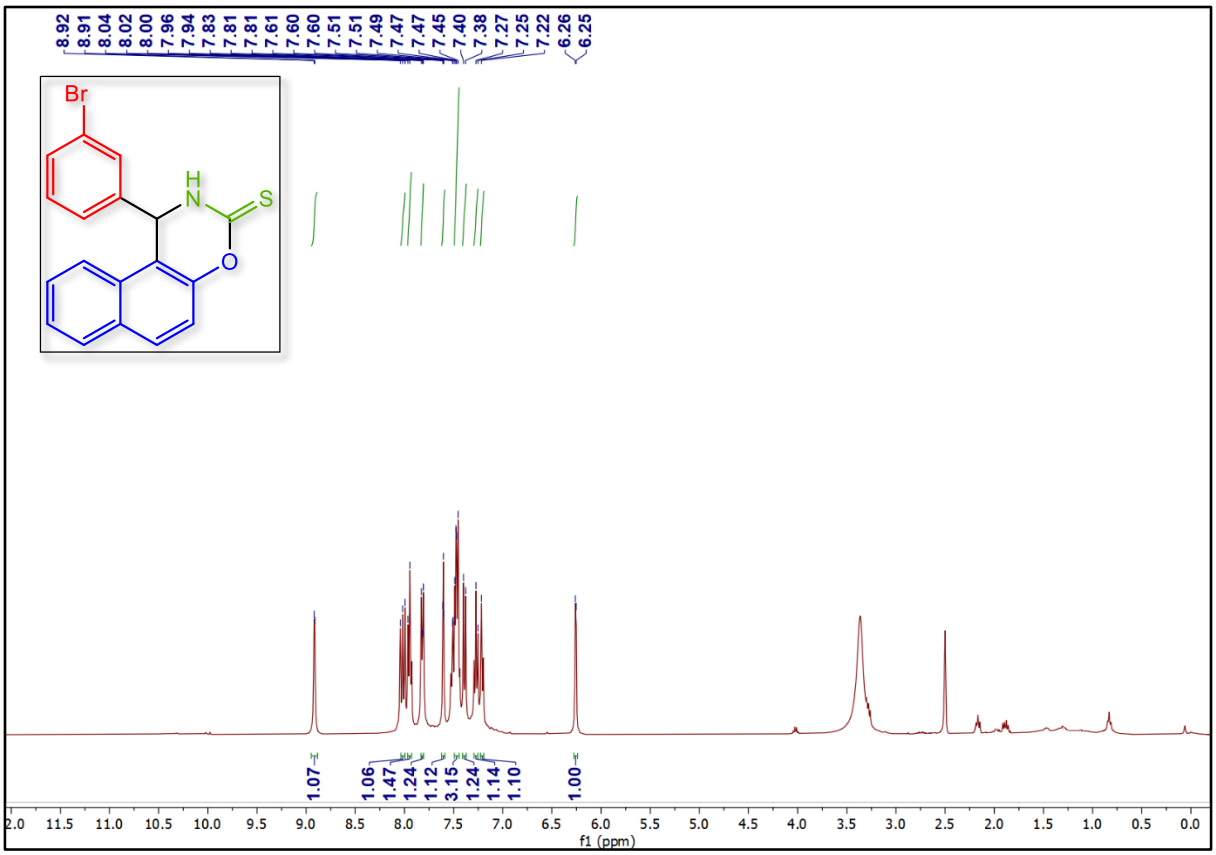


Fig. S16: ^{1}H NMR & ^{13}C NMR Spectra of 4l



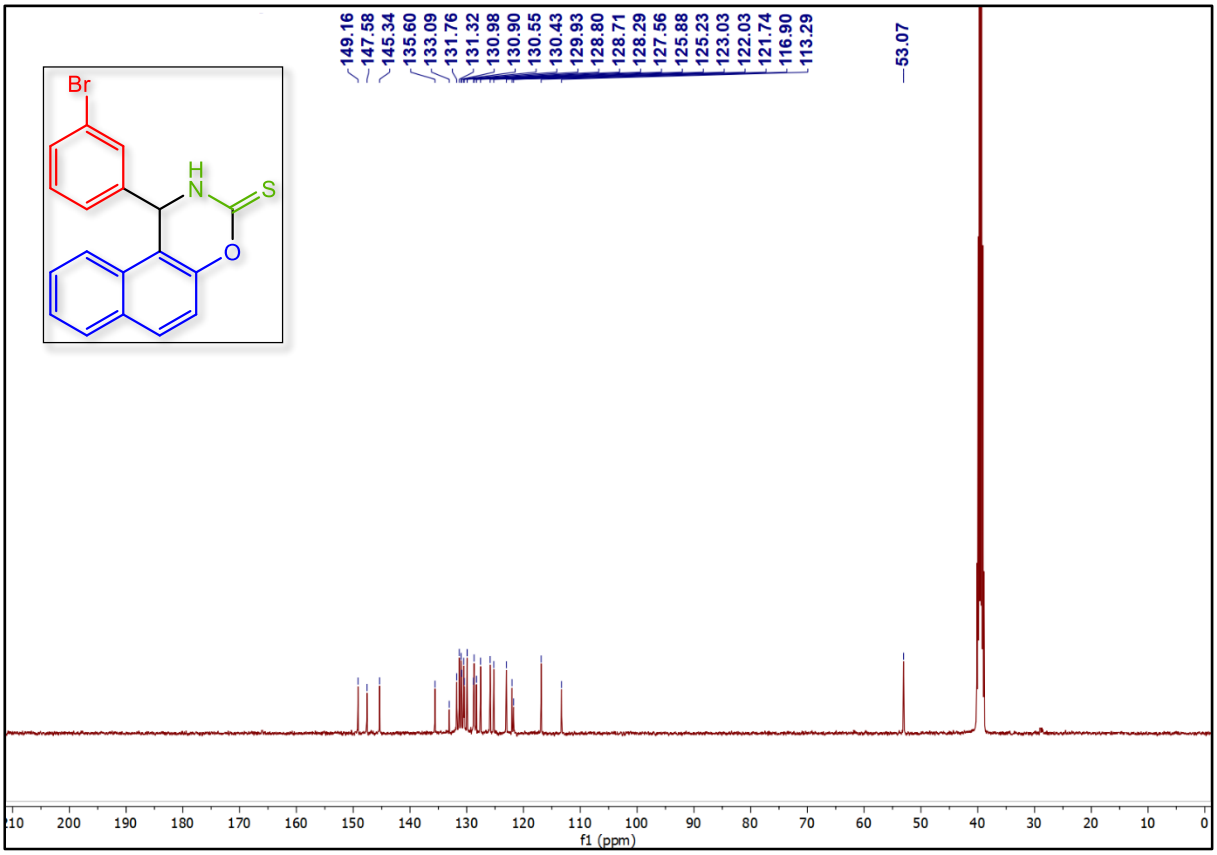
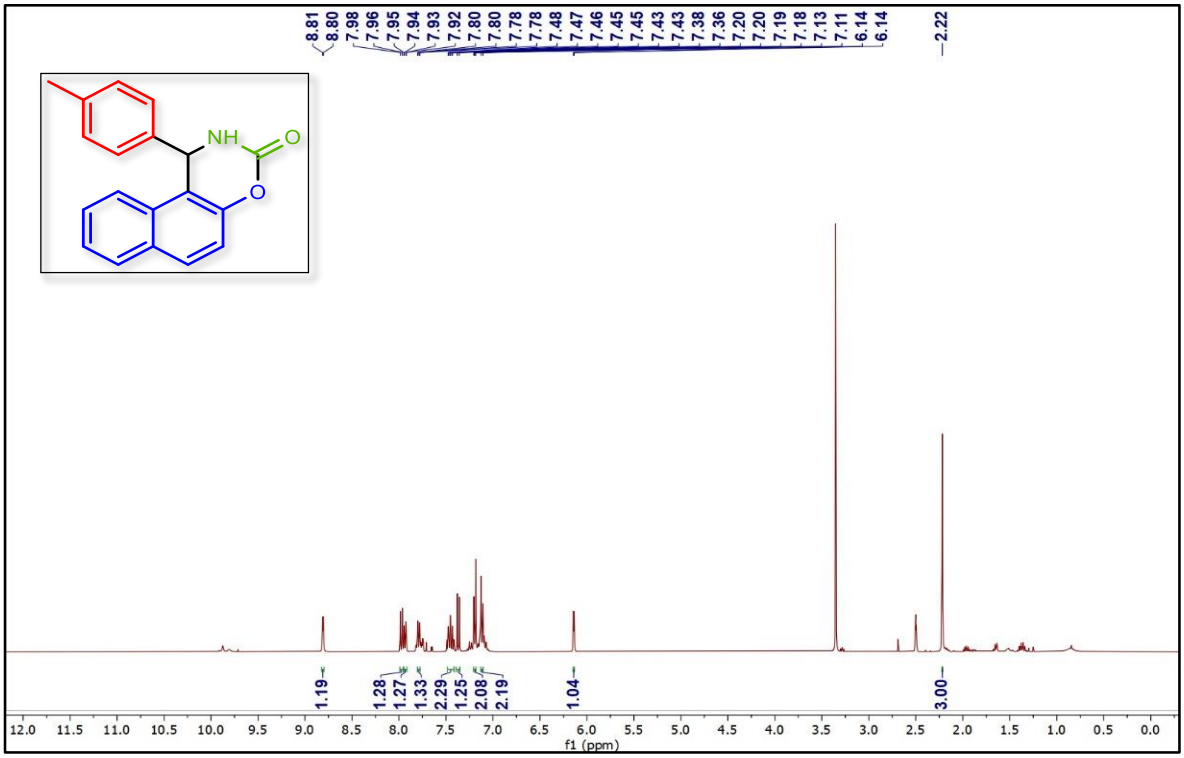


Fig. S17: ^{1}H NMR & ^{13}C NMR Spectra of 4m



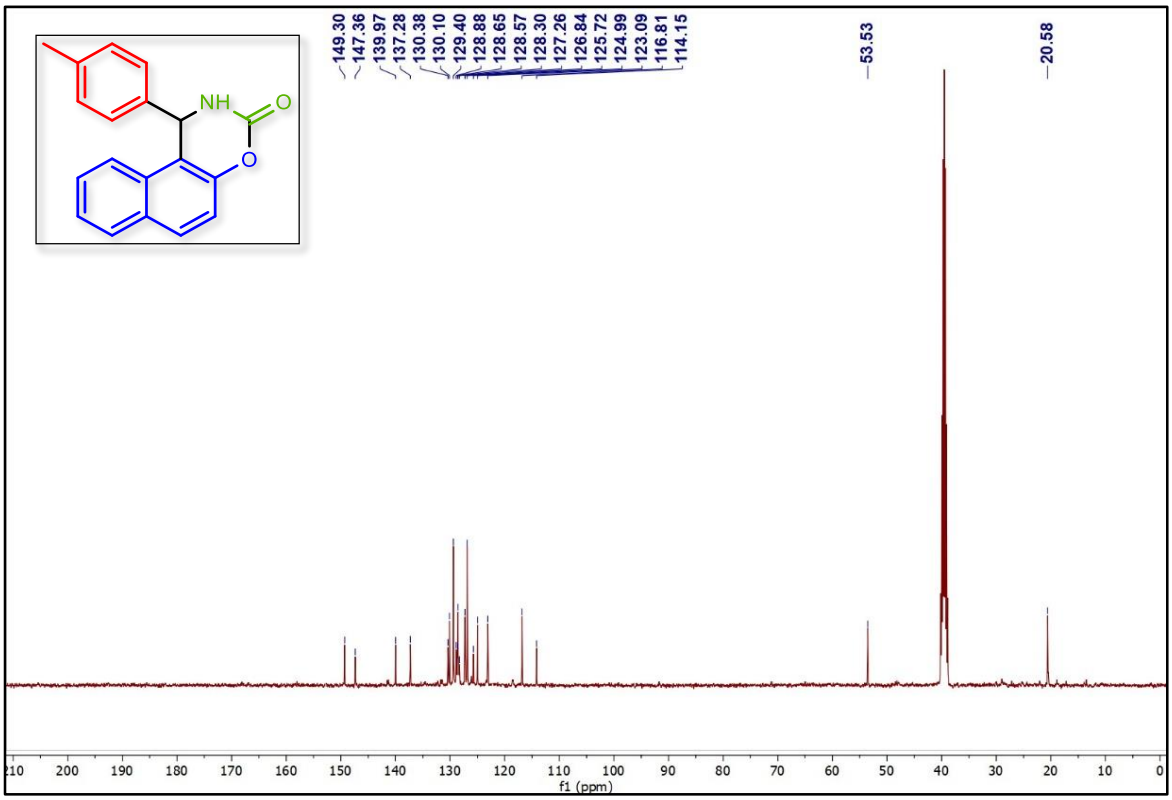


Fig. S18: ^{1}H NMR & ^{13}C NMR Spectra of 4n

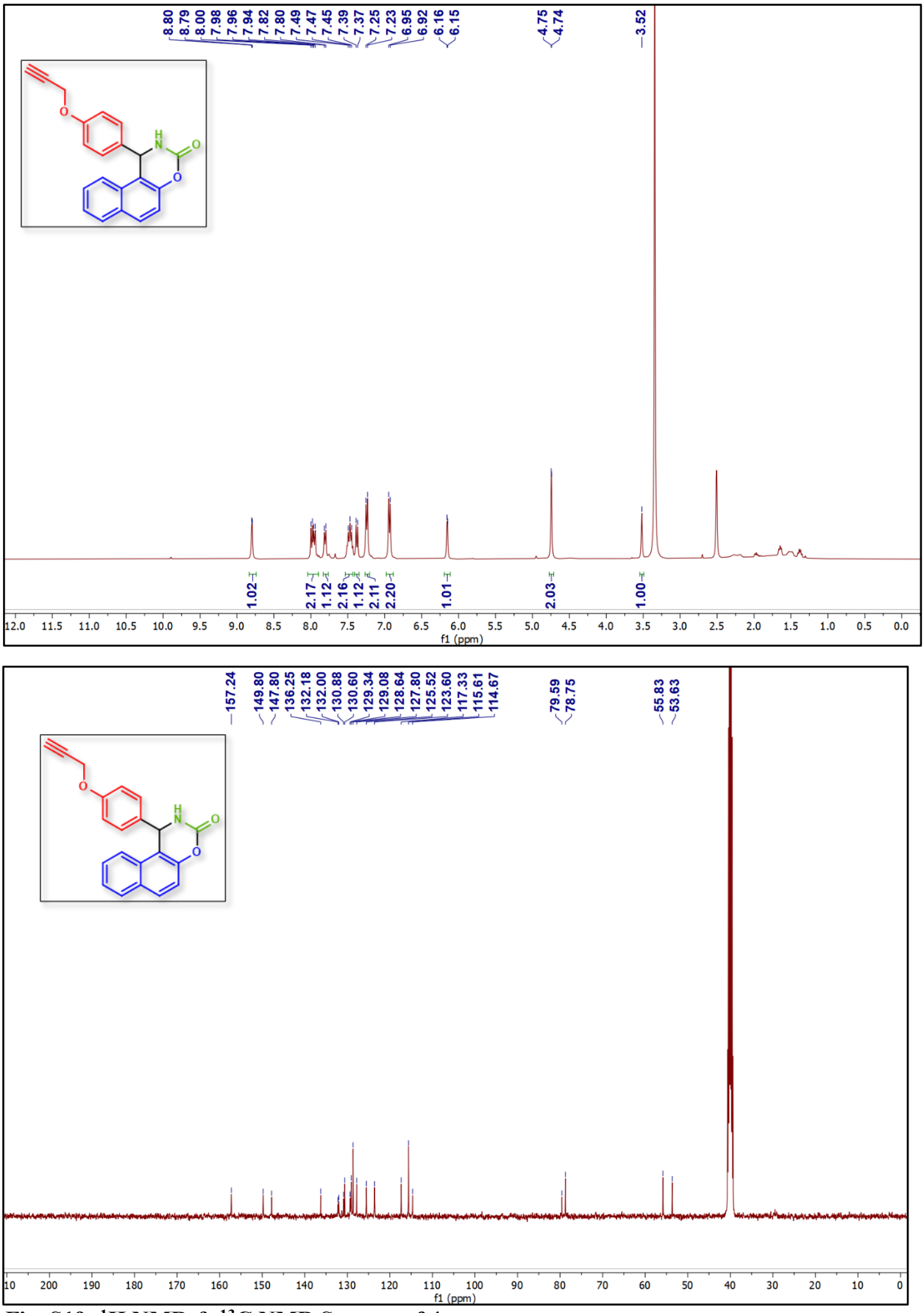


Fig. S19: ^{1}H NMR & ^{13}C NMR Spectra of 4o

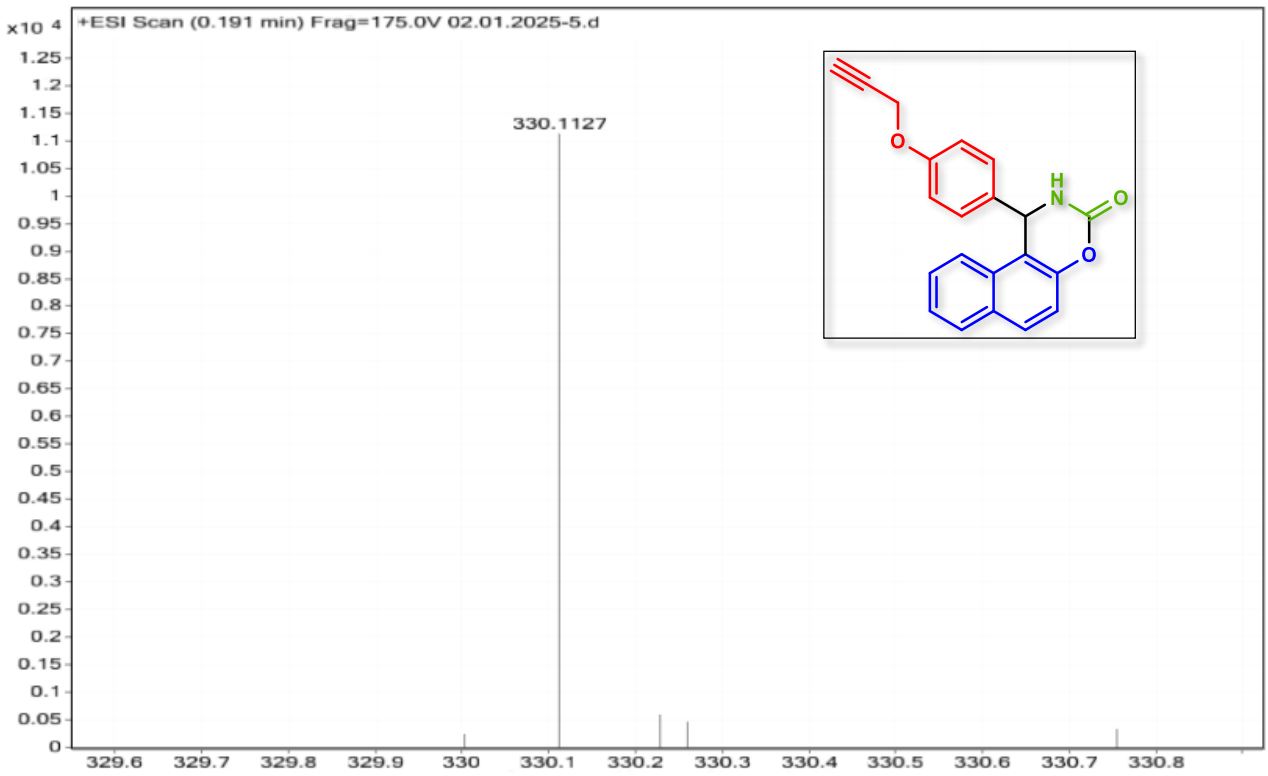
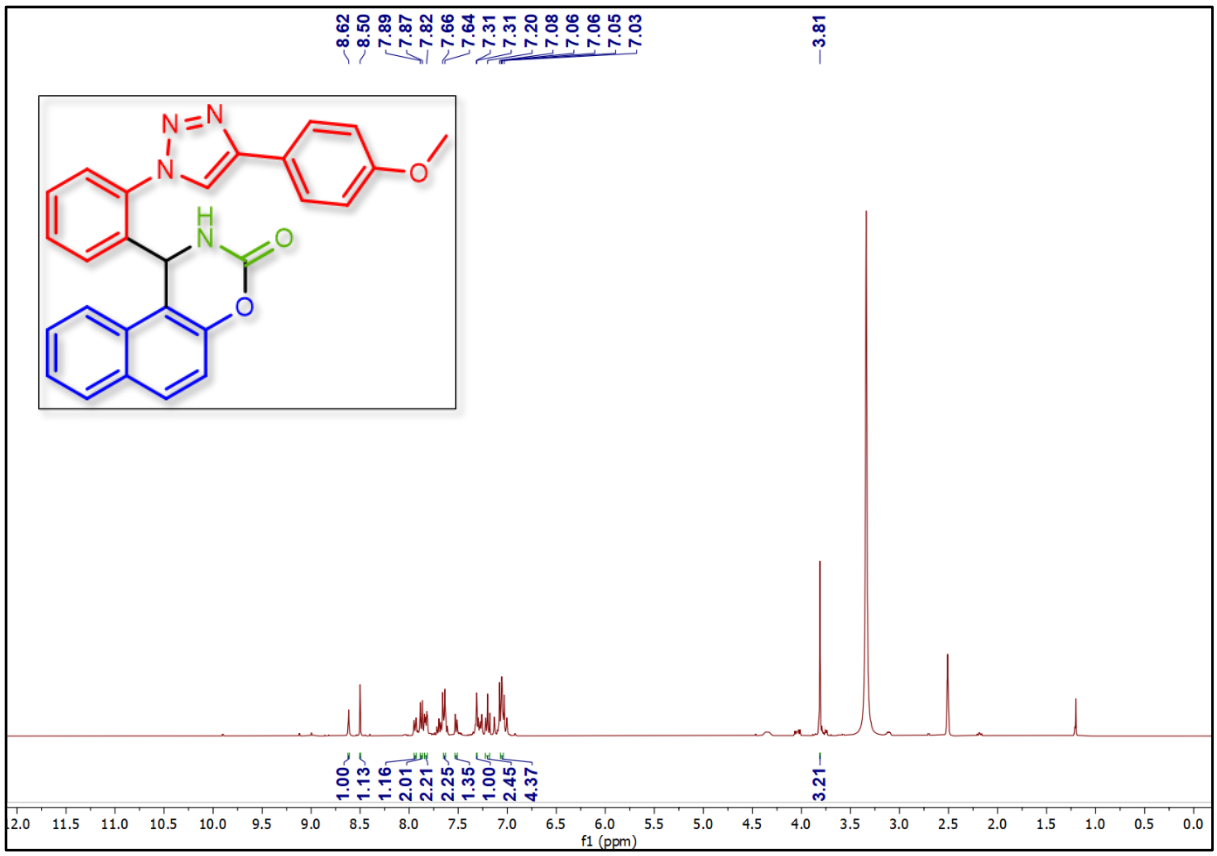


Fig. S20: HRMS Spectrum of 4o



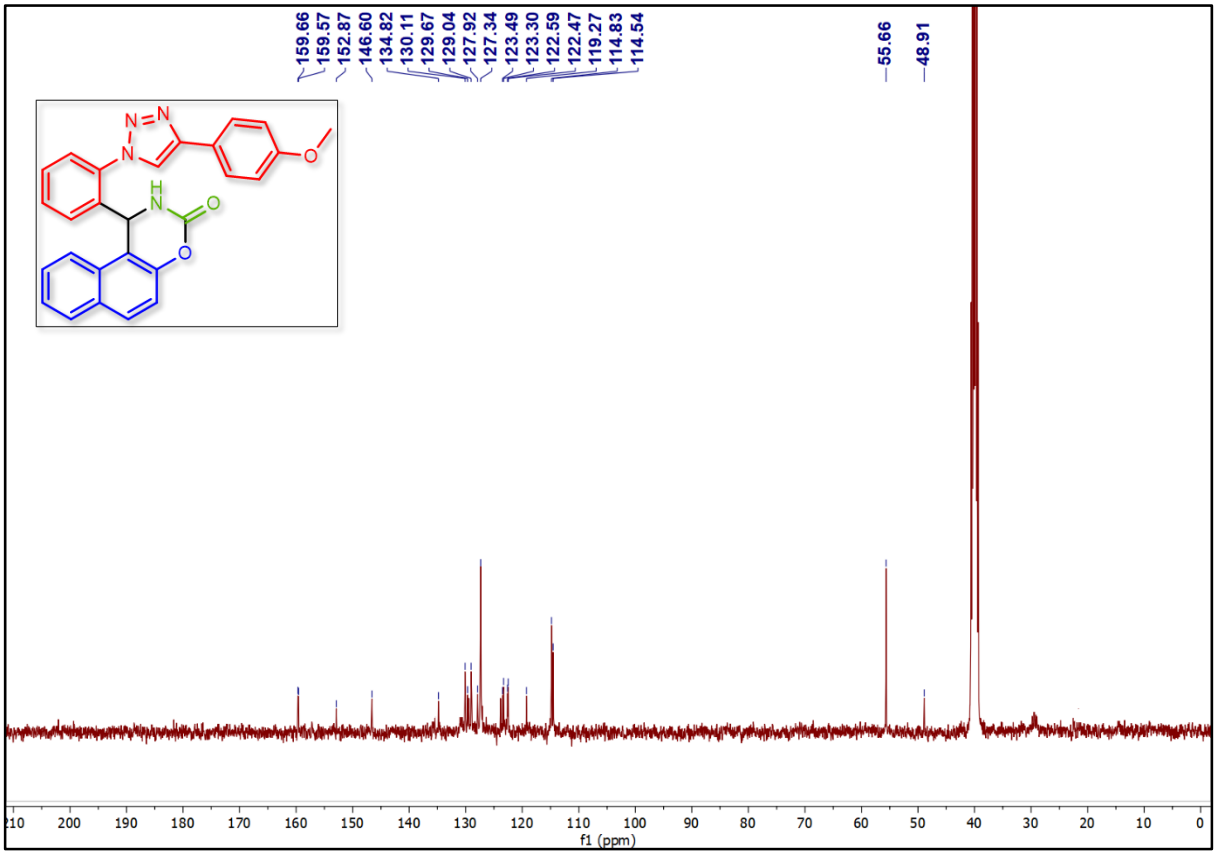


Fig. S21: ^{1}H NMR & ^{13}C NMR Spectra of 4p

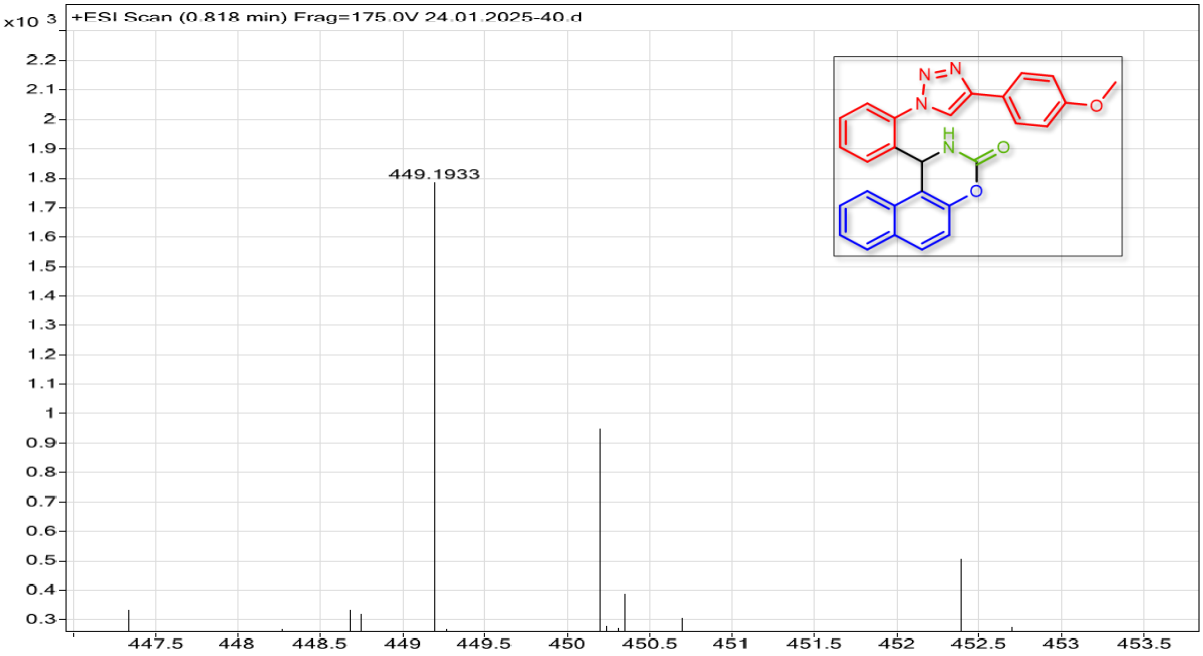
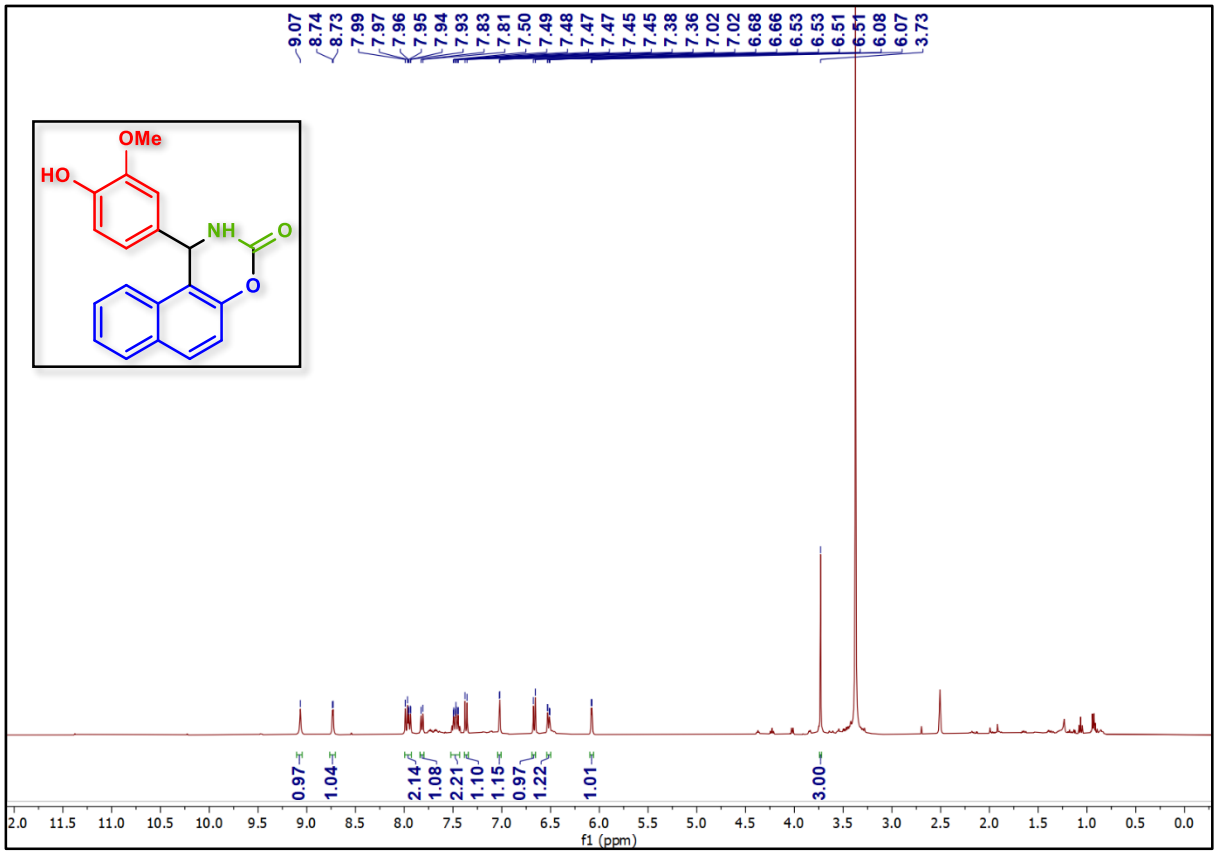


Fig. S22: HRMS Spectrum of 4p



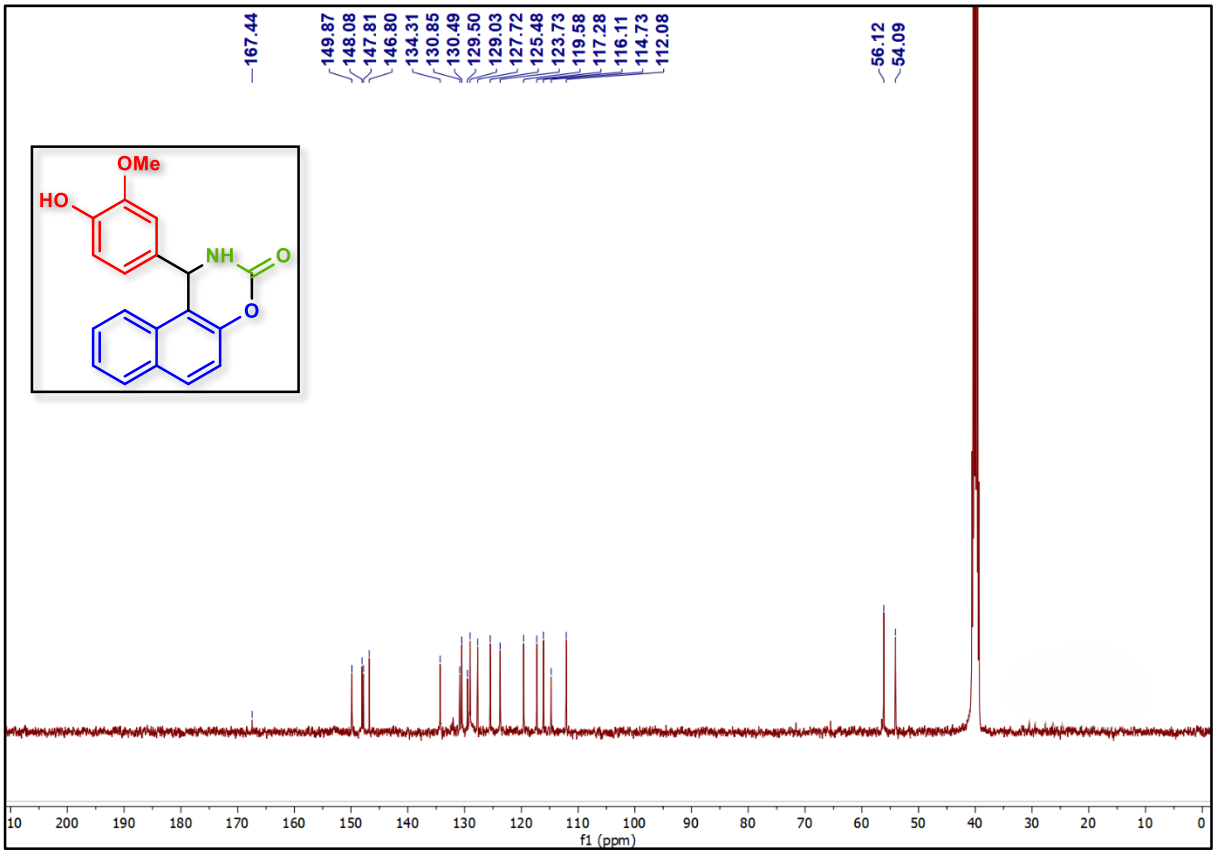
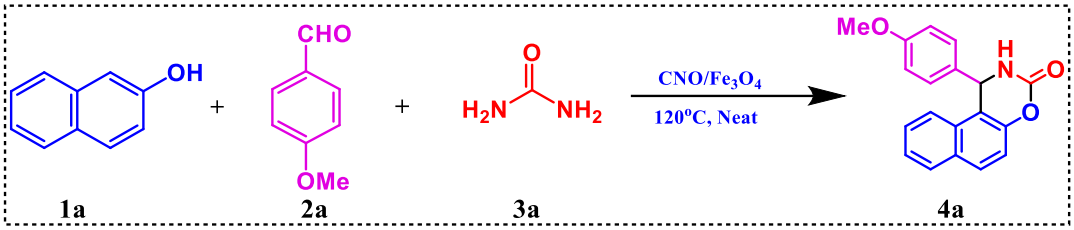


Fig. S23: ^{1}H NMR & ^{13}C NMR Spectra of 4q

Table S1: Calculation of product yield for the model reaction



Reactant	Amount (mmol)	Molecular Weight	Mass Used
2-Naphthol	1.00	144.17	144 mg
Anisaldehyde	1.00	136.15	136 mg
Urea	1.20	60.06	72 mg

After completion of the reaction, the resulting product was isolated and purified.

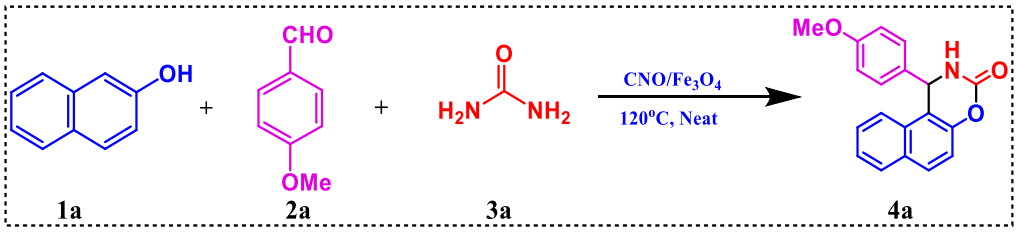
Product	Formula	MW	Expected Mass (mg)	Actual Mass (mg)	Yield (%)
1-(4-methoxyphenyl)-1,2-dihydro-3H-naphtho[1,2-e][1,3]oxazin-3-one	$C_{19}H_{15}NO_3$	305.33	305.33	290.00	94.98%

$$percent\ yield = \frac{Mass\ of\ isolated\ product}{theoretical\ mass\ based\ on\ limiting\ reagent} X100$$

$$= (290.00 / 305.33) \times 100$$

$$= 94.98\%$$

Table S2: Control Experiment: Comparative Catalytic Performance of CNO, Fe_3O_4 and CNO/ Fe_3O_4 for model reaction



Catalyst	Reaction Time	Product Yield (%)
CNO	1 hour	44%
Fe_3O_4	1 hour	29%
CNO/Fe_3O_4 Composite	**45 minutes**	**95%**

Table S3. Surface area and porosity from BET and BJH Analysis

Sample	BET Surface Area (m^2g^{-1})	Average Pore Diameter (nm)	Total Pore Volume (cm^3g^{-1})
CNO	204.60	5.09	0.261
Fe_3O_4 (by co-precipitation)	175.93	6.44	0.283
CNO/Fe_3O_4	113.84	18.09	0.515

Fig. S24: Reaction Setup Showing Controlled Flame Position and Thermal Profile

Mechanistic Validation

Experimental confirmation of ammonia (NH_3) formation was achieved through a combination of qualitative tests. The initial evidence was observed via the color change of litmus paper from red to blue, indicating the basic nature of the released gas. This was further confirmed using copper (II) sulphate impregnated paper, which turned deep blue upon exposure to NH_3 due to the formation of a tetraamminecopper (II) complex, serving as a sensitive indicator. Additionally, the introduction of a glass rod dipped in hydrochloric acid (HCl) into the reaction zone resulted in the formation of dense white fumes of ammonium chloride (NH_4Cl), providing clear visual confirmation of ammonia release. All these tests were successfully performed and validated experimentally and supporting images are shown below.

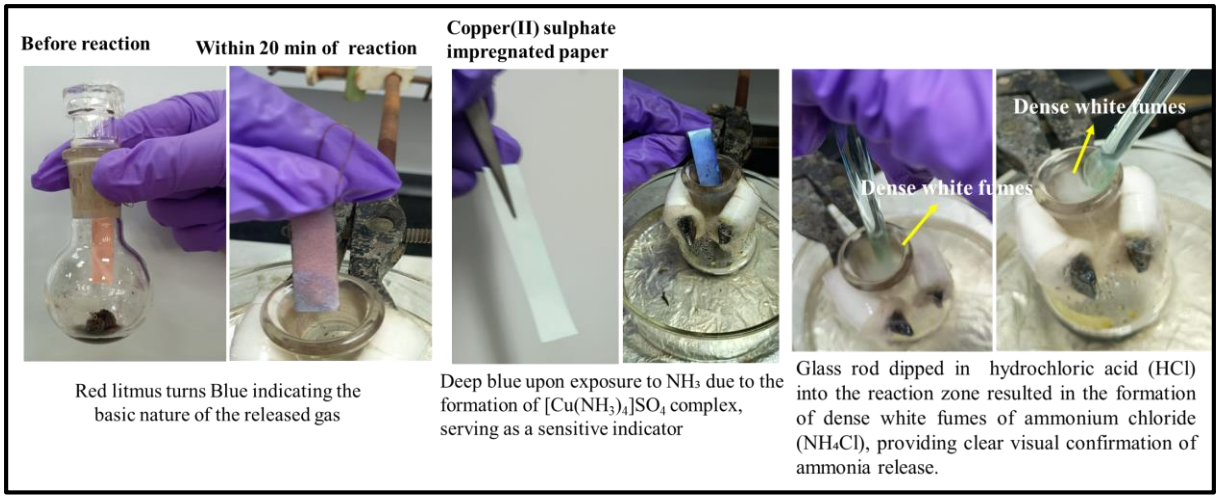


Fig S25: Experimental validation of ammonia (NH_3) formation through a combination of qualitative tests

NMR and FTIR analysis

After 15 minutes of reaction, ethyl acetate was added to extract the crude reaction mixture, followed by rotary evaporation to remove ethylacetate. The crude reaction mixture was then analyzed by ^{1}H NMR in DMSO-d_6, revealing a distinct imine proton signal at 8.81 ppm, consistent with the formation of an N-acylimine intermediate-I. Supporting this, the ^{13}C NMR Spectra showed a peak at 164.70 ppm, indicative of a conjugated C=N.

To further validate presence of C=N, FTIR analysis was performed on crude reaction mixture FTIR Spectra exhibited an absorption band at 1644.32 cm^{-1}, which aligns well with the C=N stretching vibration typically observed in imine-containing compounds. Together, these spectral features strongly suggest the successful formation of the N-acylimine intermediate-I during the progress of the reaction

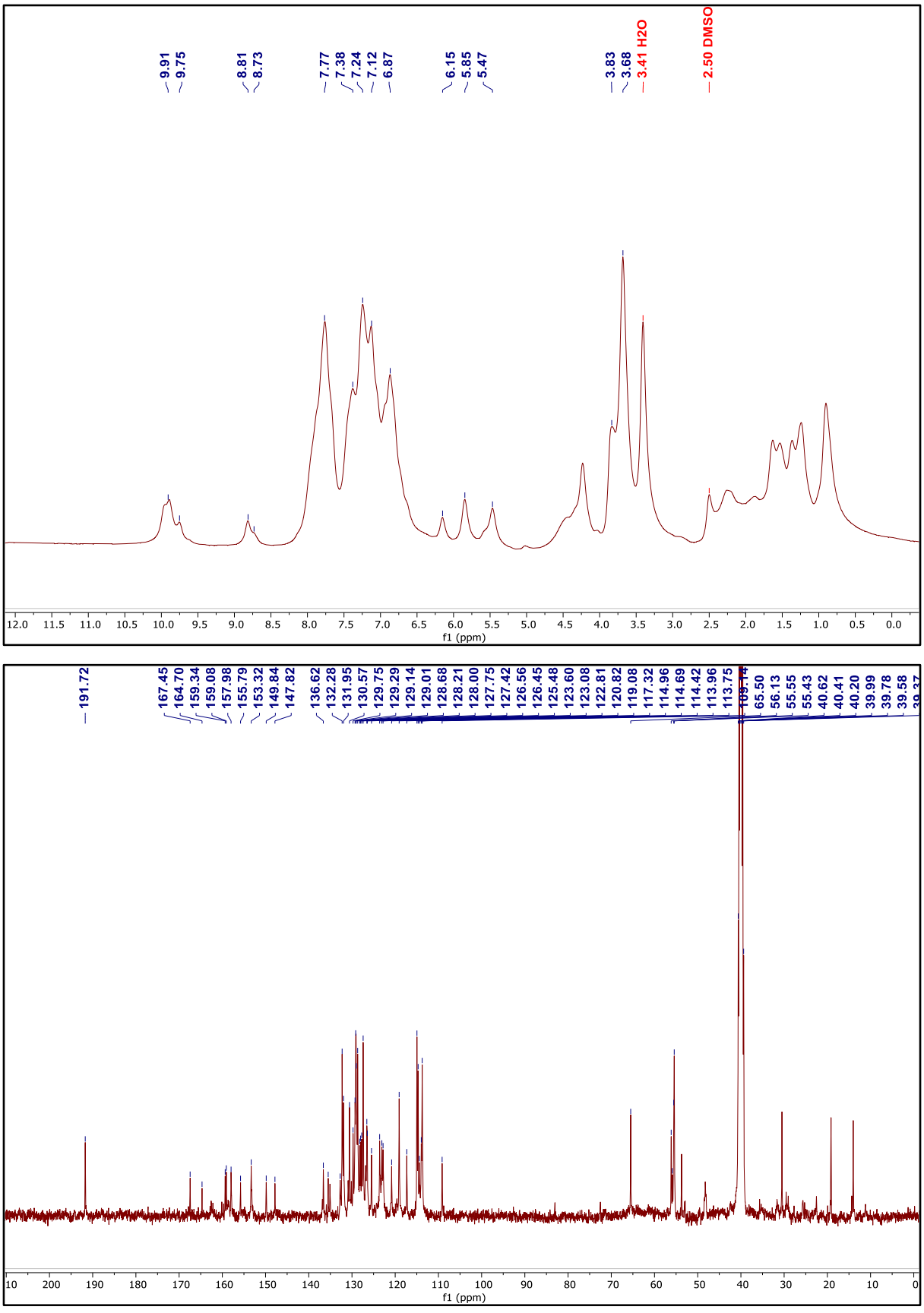


Fig. S26: ^{1}H NMR & ^{13}C NMR spectra of crude reaction mixture

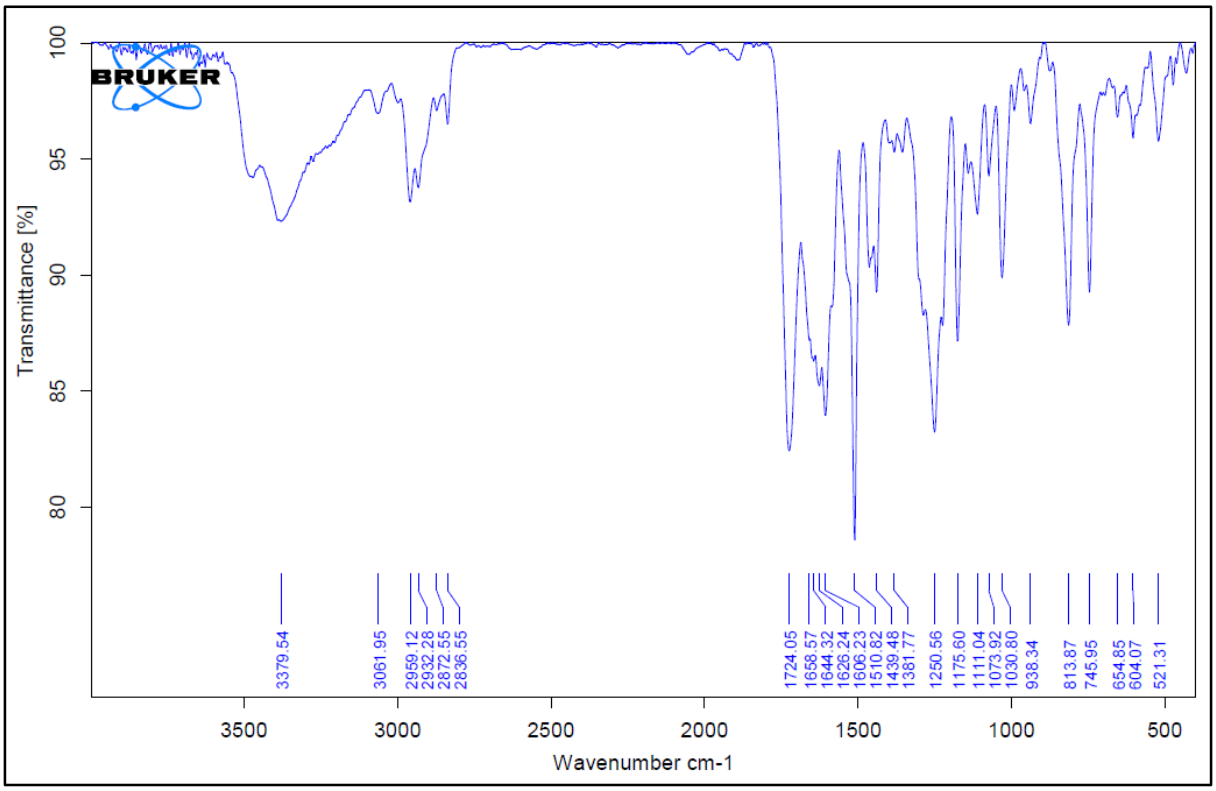


Fig. S27: FTIR Spectrum of crude reaction mixture