Ru(II)-catalysed regiospecific C-H/O-H oxidative annulation to access isochromeno[8,1-*ab*]phenazines: far red fluorescence and live cancer cell imaging

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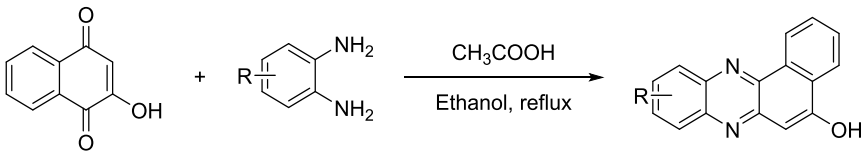
Electronic Supplementary Information

Contents

1. General information

Reagents and solvents were purchased from commercial sources. $[RuCl_2(p\text{-cymene})]_2$, $Cu(OAc)_2.H_2O$, $AgSbF_6$ were used as purchased without further purification, reagents otherwise noted. Column chromatography was performed on silica gel (100–200 mesh). Analytical TLC was performed on precoated aluminium sheets of silica gel 60F254 of 0.2 mm thickness. Melting points were determined in capillary tubes and are uncorrected. ^1H NMR (400 MHz) and ^{13}C (100 MHz) spectra were recorded in $CDCl_3$ solution with TMS as internal standard. High resolution mass spectra (HRMS-ESI) were recorded using high resolution mass spectrometer. UV-visible absorption spectra were measured using UV spectrophotometer. The steady state fluorescence measurements were measured using fluorescence spectrophotometer. Cell imaging was done using laser confocal microscope instrument.

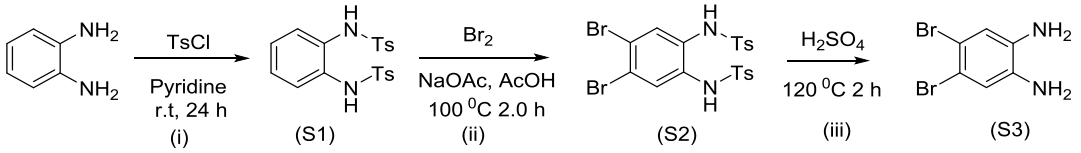
2. General procedure for synthesis of benzo[a]phenazin-5-ol 1a



Scheme S1. General procedure for synthesis of benzo[a]phenazin-5-ol 1a

A mixture of ortho-phenylenediamine 2.16 g (1.0 equiv), 2-hydroxy 1,4-napthaquinone 3.48 g (1.0 equiv) and (20 mol %) acetic acid in ethanol 50 ml was added heated at reflux for 2 hours. The reaction was cooled, the resulting solid was collected for filtration, washed with cold ethanol. The **benzo[a]phenazin-5-ol** was obtained as yellow solid with the yield of 95%.

3. General procedure for synthesis of 4,5-dibromobenzene-1,2-diamine[1-2]



Scheme S2. General procedure for synthesis of 4,5-dibromobenzene-1,2-diamine

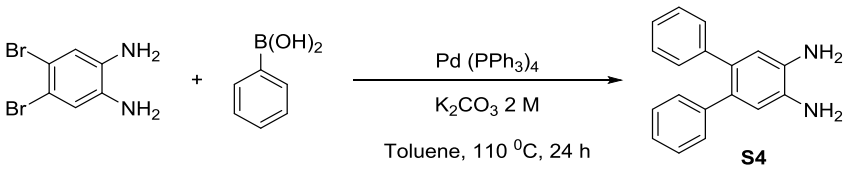
(i) Synthesis of *N,N′*-(1,2-Phenylene)bis-4-methylbenzenesulfonamide (S1): o-Phenylenediamine (1.0 equiv). Was added slowly to a solution of p-toluenesulfonyl chloride (2 equiv) in pyridine 50 mL which was cooled to 0 ^0C in a ice bath. The resulting mixture was stirred at room temperature for 24 h. After slow addition of 15 % aqueous HCl, a precipitate was formed. The solids was filtered by suction filtration. The solid was recrystallized from ethanol. After recrystallization,

compound N,N′-(1,2-Phenylene)bis(4-methylbenzenesulfonamide) was obtained as a pale solid 92 % of yield.

(ii) Synthesis of *N,N′*-(4,5-dibromo-1,2-phenylene)bis-4-methylbenzenesulfonamide (S2): A solution of N, N'-(1,2-Phenylene)bis (4-methylbenzenesulfonamide) 1equiv in glacial acetic was stirred and sodium acetate (2 equiv) added. Bromine (2 equiv) was added dropwise and the resulting dark solution then heated at 80°C for 3 hr. Water (50ml) was added and the resulting precipitate isolated by filtration and purified by recrystallization from acetic acid to give the required product N,N'-(4,5-dibromo-1,2-phenylene)bis(4-methylbenzenesulfonamide) Yield of 94%, white needles.

(iii) Synthesis of 4,5-dibromobenzene-1,2-diamine (S3): *N,N'-(*4,5-dibromo-1,2-phenylene)bis(4-methylbenzenesulfonamide) was dissolved and heated in concentrated sulphuric acid at 110 °C for about 15 min. After cooling to room temperature, the reaction mixture was poured into ice–water and neutralized with 50% NaOH solution until the color of the solution is off-white and lots of precipitate was formed. After filtration, the filtrate was recrystallized from methanol. The precipitate gave an off-white powder yield of 95%.

4. Reaction procedure for synthesis of [1,1':2',1''-terphenyl]-4',5'-diamine (S4): [3]



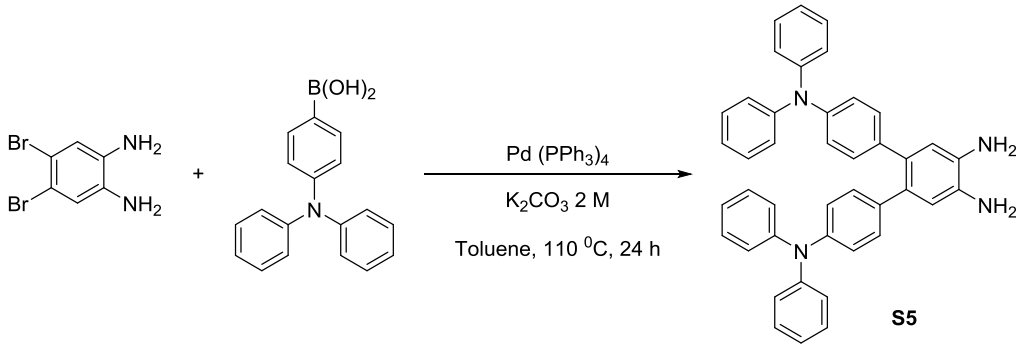
. **Scheme S3**. Reaction procedure for synthesis of [1,1':2',1"-terphenyl]-4',5'-diamine

4,5-dibromobenzene-1,2-diamine (0.500 mg 1.88mmol), phenylboronic acid (570 mg 2.0 equiv) and $Pd(PPh_3)_4$ (64.1 mg, 0.0555 mmol) were dissolved toluene (50 mL). Next, 2.0 M K_2CO_3 aqueous solution (15 mL) was added to the toluene solution under nitrogen atmosphere. And, the mixture was stirred at 85 ºC for 24 h. After finishing reaction, water was added to the toluene solution. Then, compounds were extracted with Ethyl acetate, and the organic layer was washed with brine and dried by anhydrous Na_2SO_4.Next, the organic solution was evaporated, and then the crude was purified by silica gel column chromatography (hexane/ethyl acetate) to afford compound **S4 (**507.6 mg, 1.95 mmol, 97%) as white solid.

5. Reaction procedure for synthesis *N*[4],*N*[4],*N*[4''],*N*[4'']-tetraphenyl-[1,1':2',1''-terphenyl]-4,4',4'',5'-tetraamine (S5):

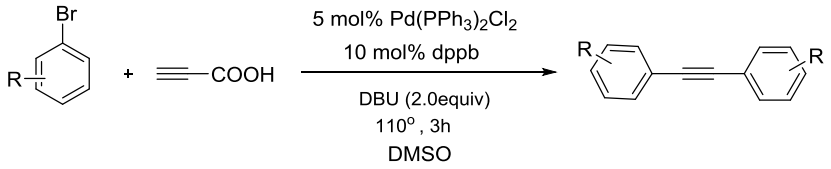
4, 5-Dibromobenzene-1,2-diamine (0.500 g 1.88 mmol), 4-(Diphenylamino)phenylboronic acid (1.08 g 2.00 equiv) and $Pd(PPh_3)_4$ (64.1 mg, 0.0555 mmol) were dissolved toluene (50 mL). Next, 2.0 M K_2CO_3 aqueous solution (15 mL) was added to the toluene solution under nitrogen atmosphere. And,

the mixture was stirred at 85 ºC for 24 h. After finishing reaction, water was added to the toluene solution. Then, compounds were extracted with Ethyl acetate, and the organic layer was washed with brine and dried by anhydrous Na_2SO_4. Next, the organic solution was evaporated, and then the crude was purified by silica gel column chromatography (hexane/ethyl acetate) to afford compound **S5 (**507.6 mg, 1.95 mmol, 97%) as white solid.



Scheme S4. Reaction procedure for synthesis $N^4,N^4,N^{4''},N^{4''}$-tetraphenyl-[1,1':2',1''-terphenyl]-4,4',4'',5'-tetraamine

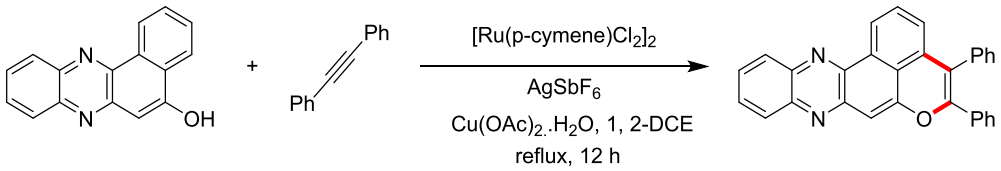
6. General procedure for synthesis of alkynes (2):[4]



Scheme S5. General procedure for synthesis of alkynes

Following the literature procedure, Pd $(PPh_3)_2Cl_2$ (105 mg, 0.15 mmol), 1,4- bis (diphenyl phosphino) butane (128 mg, 0.30 mmol), aryl halides (6.00 mmol), and propiolic acid (212 mg, 3.0 mmol) were combined with DBU (913 mg, 6.0 mmol) in a round bottom flask. DMSO (15.0 mL) was added and the reaction was maintained in the oxygen atmosphere. The resulting mixture was placed in an oil bath at 80 ^oC for 3 h. The reaction was poured in saturated ammonium chloride solution and extracted with ethyl acetate 3× 25 mL. The combined ethyl acetate layer was washed with brine solution, dried over anhydrous sodium sulphate, filtered, and the solvent were removed under vacuum. The resulting crude product was purified using flash column chromatography in silica gel 100-200 mesh using 5% EtOAc in pet ether.

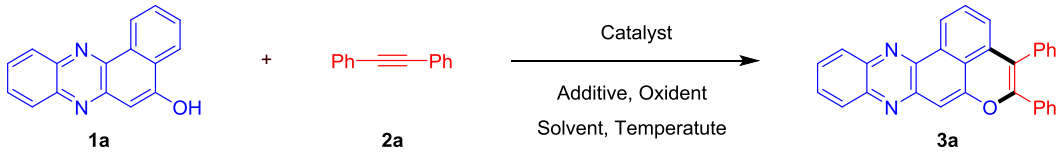
7. Experimental procedure for synthesis of isochromeno[8,1-ab]phenazine 3a



Scheme S6. Experimental procedure for synthesis of isochromeno[8,1-ab]phenazine

To an oven-dried 25 mL round bottom flask fitted with a reflux condenser were added benzo[*a*]phenazin-5-ol **1a** (73.88 mg, 0.3 mmol, 1.0 equiv), Diphenylacetylene **2a** (53.43 mg, 0.3 mmol, 1.0 equiv), [RuCl2(p-cymene)]$_2$ (9.18 mg, 0.015 mmol, 0.05 equiv), $AgSbF_6$ (20.62 mg, 0.06 mmol, 0.2 equiv) $Cu(OAc)_2.H_2O$ (59.89 mg, 0.3 mmol, 1.0 equiv) and in 1,2-dichloroethane 3.0 ml (1,2-DCE). The reaction mixture was refluxed for 12 h. After cooling to ambient temperature, the reaction mixture was diluted with CH_2Cl_2, filtered through celite and the filtrate was concentrated under reduced pressure. The crude residue was purified through a silica gel column using hexane and ethyl acetate (85:15) as eluent. Desired product orange solid **3a** 91.25 mg was obtained in 72 % of yield.

Table S1 Optimization of reaction condition of benzo[*a*]phenazin-5-ol with di-phenyl acetylene[a]



Entry	Catalyst	Additive	Oxidant (equiv.)	Solvent	Yield (%)[b]
1	$[RuCl_2(p\text{-cymene})]_2$	$AgSbF_6$	$Cu(OAc)_2.H_2O$	Toluene	14[c], 30
2	$[RuCl_2(p\text{-cymene})]_2$	$AgSbF_6$	$Cu(OAc)_2.H_2O$	Choro benzene	40
3	$[RuCl_2(p\text{-cymene})]_2$	$AgSbF_6$	$Cu(OAc)_2.H_2O$	1,4-dioxane	10
4	$[RuCl_2(p\text{-cymene})]_2$	$AgSbF_6$	$Cu(OAc)_2.H_2O$	*t*-AmOH	20
5	$[RuCl_2(p\text{-cymene})]_2$	$AgSbF_6$	$Cu(OAc)_2.H_2O$	CH_3OH	18
6	$[RuCl_2(p\text{-cymene})]_2$	$AgSbF_6$	$Cu(OAc)_2.H_2O$	DMF	30
7	$[RuCl_2(p\text{-cymene})]_2$	$AgSbF_6$	$Cu(OAc)_2.H_2O$	1,2- DCE	72
8	$[RuCl_2(p\text{-cymene})]_2$	$AgSbF_6$	$Cu(OAc)_2$	1,2- DCE	68
9	$[RuCl_2(p\text{-cymene})]_2$	$AgSbF_6$	$Ag(OAc)_2$	1,2- DCE	45
10	$[RuCl_2(p\text{-cymene})]_2$	$AgSbF_6$	Ag_2CO_3	1,2- DCE	25
11	$[RuCl_2(p\text{-cymene})]_2$	$AgSbF_6$	$K_2S_2O_8$	1,2- DCE	18

12	$[RuCl_2(p\text{-cymene})]_2$	$AgSbF_6$	$PhI(OAc)_2$	1,2- DCE	trace
13	$[RuCl_2(p\text{-cymene})]_2$	$AgSbF_6$	$Cu(OAc)_2.H_2O$ (0.5)	1,2- DCE	28
14	$[RuCl_2(p\text{-cymene})]_2$	$AgSbF_6$	-	1,2- DCE	-
15	$[RuCl_2(p\text{-cymene})]_2$	KPF_6	$Cu(OAc)_2.H_2O$	1,2- DCE	65
16	$[RuCl_2(p\text{-cymene})]_2$	$AgBF_4$	$Cu(OAc)_2.H_2O$	1,2- DCE	50
17	$[RuCl_2(p\text{-cymene})]_2$	-	$Cu(OAc)_2.H_2O$	1,2- DCE	10
18	$RuCl_2.\ 5H_2O$	$AgSbF_6$	$Cu(OAc)_2.H_2O$	1,2- DCE	trace
19	$[RhCp^*Cl_2]_2$	$AgSbF_6$	$Cu(OAc)_2.H_2O$	1,2- DCE	60
20	$Pd(OAc)_2$	-	$Cu(OAc)_2$	DMF	Trace

[a] Reaction conditions: **1a** (0.3 mmol), **2a** (0.3 mmol), catalyst (5.0 mol%), additive (20 mol%) and oxidant (1 equiv.) in the indicated solvent (2.0 mL) at reflux or 110 °C for 12 h under air. [b] Isolated yield. [c] Under nitrogen

8. ORTEP diagram of compound 3a

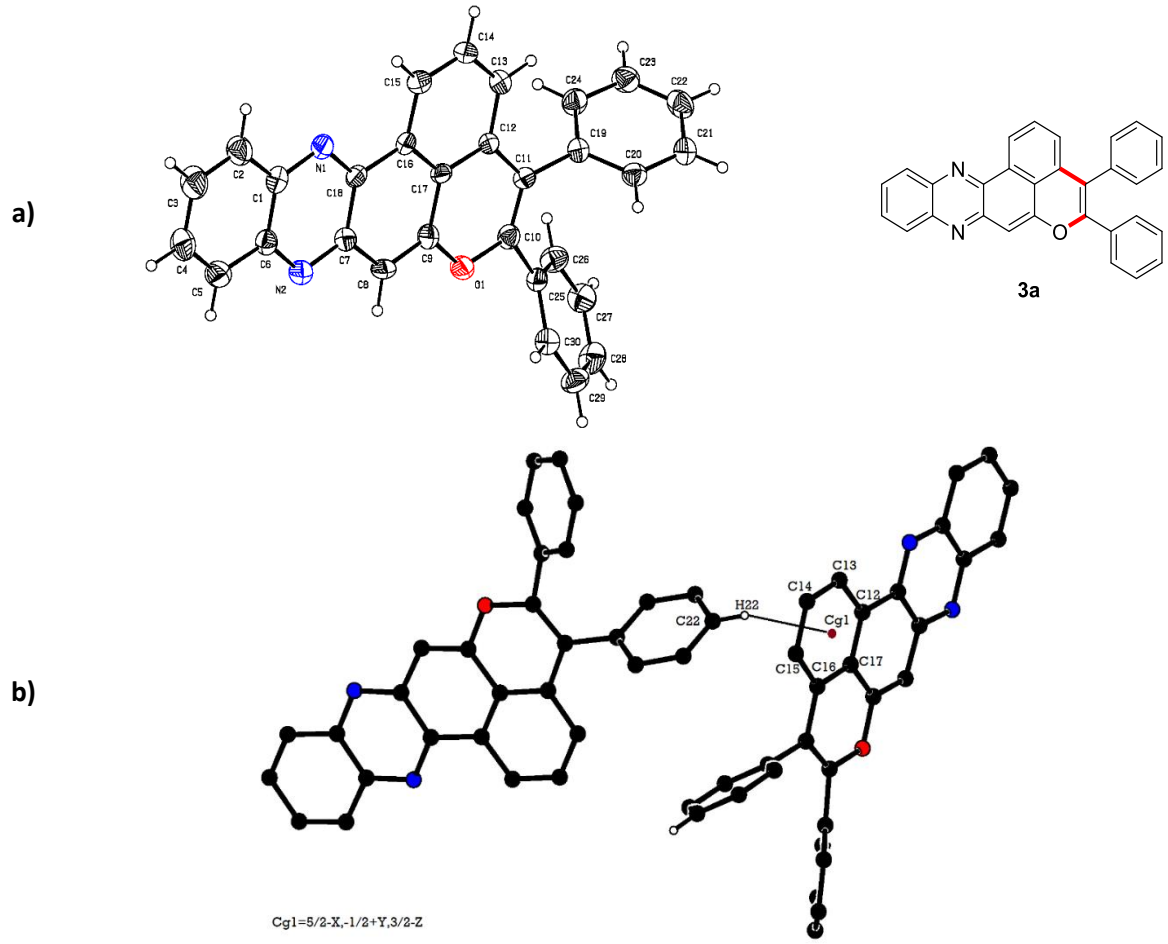
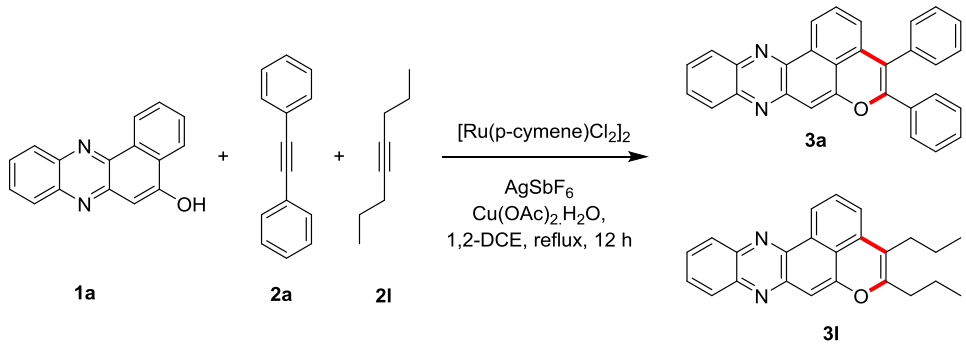


Figure S1. (a) ORTEP diagram of compound **3a** (CCDC Number: 1503529) with 30% probability ellipsoids. (b) Intermolecular π-π stacking interaction.

9. Competitive Experiments



Scheme S7. Competitive Experiments

To an oven-dried 25 mL round bottom flask fitted with a reflux condenser were added benzo[*a*]phenazin-5-ol **1a** (73.88 mg, 0.3 mmol, 1.0 equiv), Diphenylacetylene **2a** (53.43 mg, 0.3 mmol, 1.0 equiv), 2l (36.06 mg, 0.3 mmol, 1.0 equiv), $[RuCl2(p\text{-cymene})]_2$ (9.18 mg, 0.015 mmol, 0.05 equiv), $AgSbF_6$ (20.62 mg, 0.06 mmol, 0.2 equiv) $Cu(OAc)_2.H_2O$ (59.89 mg, 0.3 mmol, 1.0 equiv) and in 1,2-dichloroethane 3.0 ml (1,2-DCE). The reaction mixture was refluxed for 12 h. After cooling to ambient temperature, the reaction mixture was diluted with CH_2Cl_2, filtered through celite and the filtrate was concentrated under reduced pressure. The crude residue was purified through a silica gel column using hexane and ethyl acetate (85:15) as eluent. Desired product orange solid **3a** and **3l**. ^{1}H NMR analysis confirmed that the annulated product originating from the electron-deficient alkyne 2a was obtained in higher preposition 1.7:1.0 when compared with 2l.

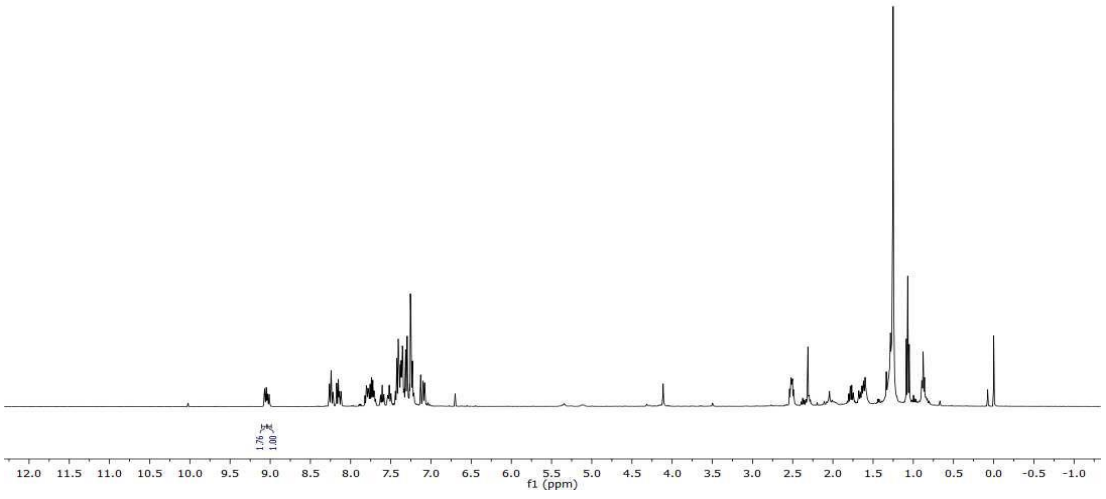
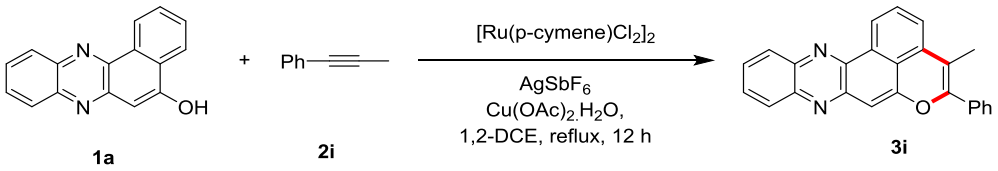


Figure S2. ^{1}HNMR of Competitive Experiments

10. Regioselectivity of the reaction



Scheme S8. Regioselectivity of the reaction

To an oven-dried 25 mL round bottom flask fitted with a reflux condenser were added benzo[a]phenazin-5-ol **1a** (73.88 mg, 0.3 mmol, 1.0 equiv),1-phenyl-1-propyne **2i** (34.84 mg, 0.3 mmol, 1.0 equiv), $[RuCl_2(p\text{-cymene})]_2$ (5.0 mol%), $Cu(OAc)_2 \cdot H_2O$ (1 equiv) and $AgSbF_6$ (20 mol%) in 1,2-DCE. The reaction mixture was refluxed for 12 h. After cooling to ambient temperature, the reaction mixture was diluted with CH_2Cl_2, filtered through celite and the filtrate was concentrated under reduced pressure. The crude residue was purified through a silica gel column using hexane and ethyl acetate (85:15) as eluent. Desired product orange solid **3i** 73.52 mg was obtained in 68 % of yield.. The regioselectivity of the annulated product was confirmed by NOE experiment.

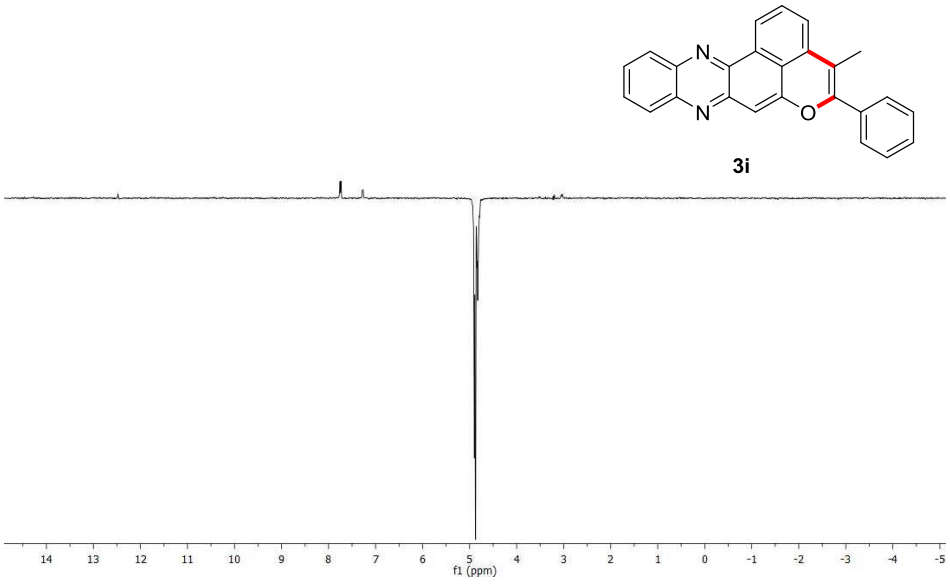


Figure S3. 1D NOE spectrum of compound 3i

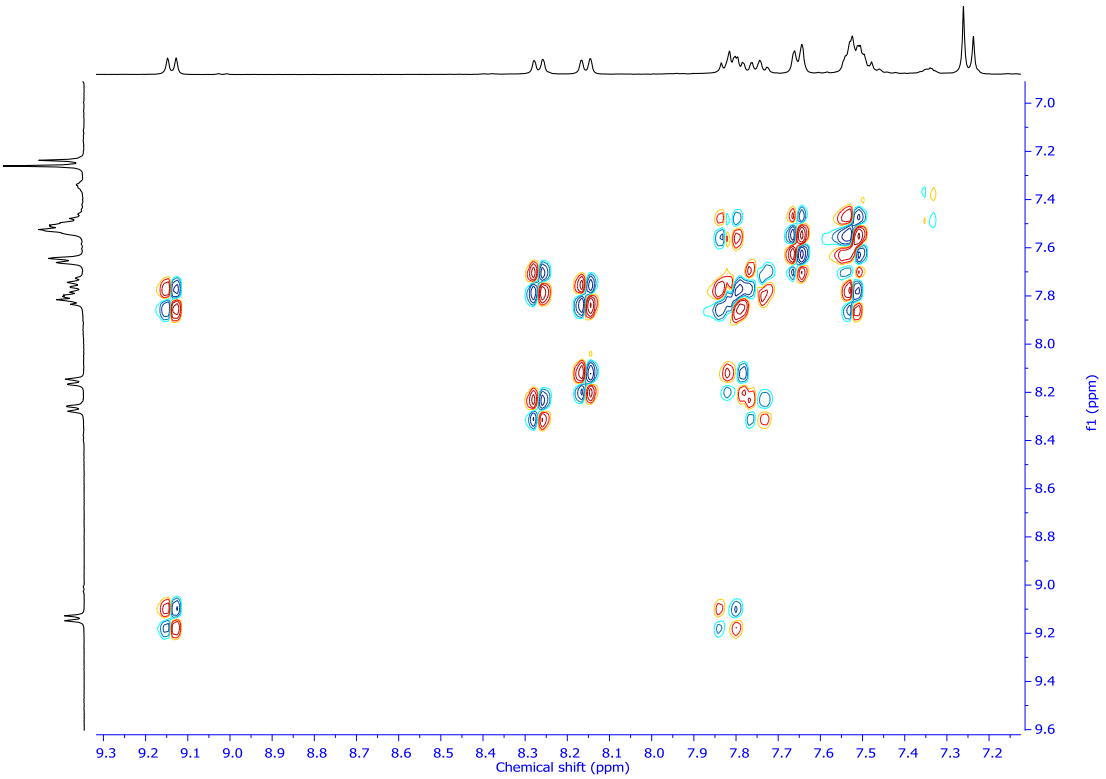


Figure S4. H-H COSY spectrum of compound 3i

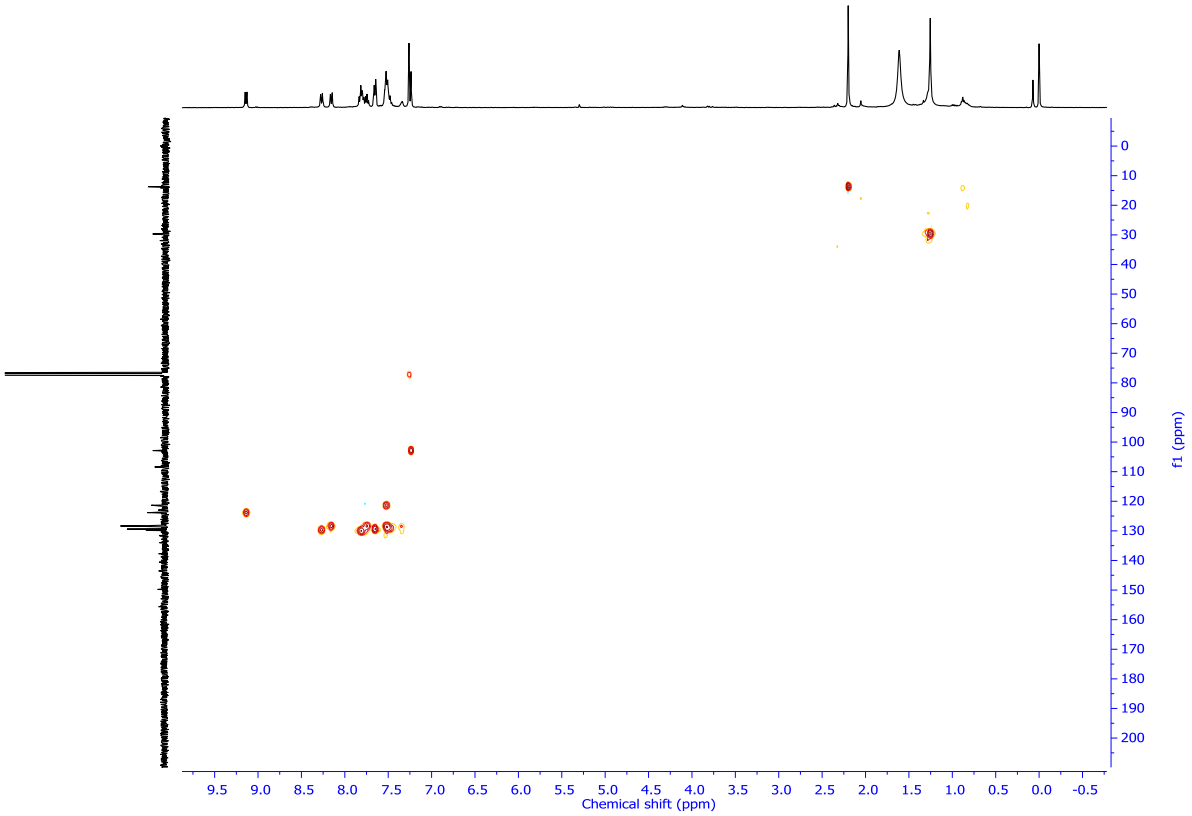


Figure S5. HSQC spectrum of compound 3i

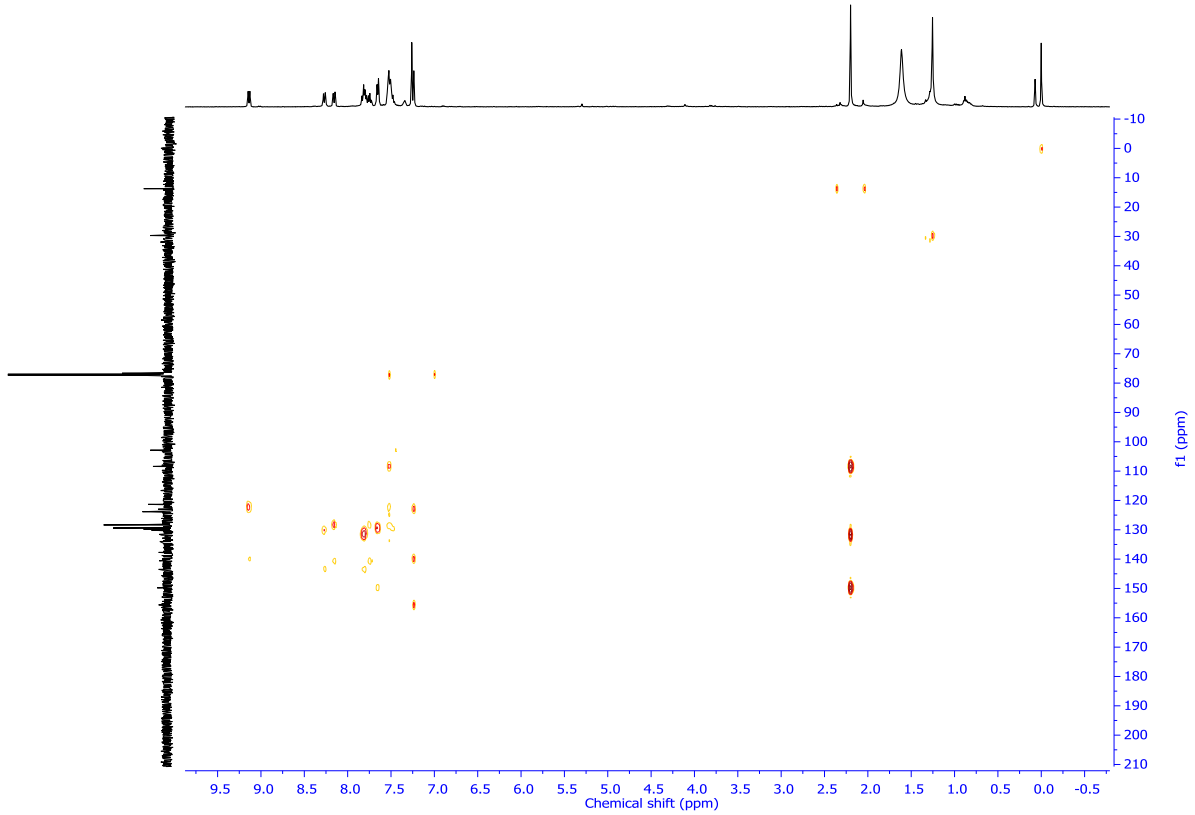
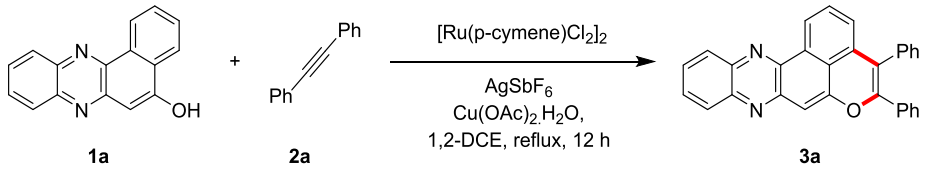


Figure S6. HMBC spectrum of compound 3i

11. Gram scale synthesize of 4,5-diphenylisochromeno[8,1-ab]phenazine



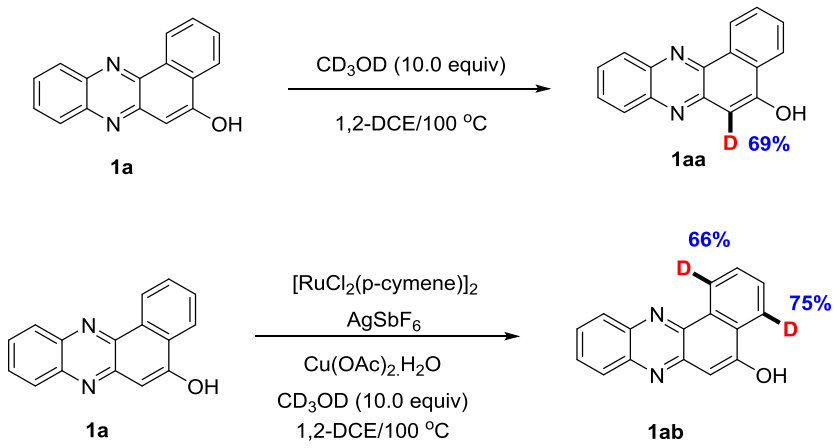
Scheme S9. Gram scale synthesize of 4,5-diphenylisochromeno[8,1-ab]phenazine

To an oven-dried 100 mL round bottom flask fitted with a reflux condenser were added benzo[a]phenazin-5-ol **1a** (1.03 g 4.2 mmol, 1.0 equiv), Diphenylacetylene **2a** (0.722 g, 4.2 mmol, 1.0 equiv), $[RuCl_2(p\text{-cymene})]_2$ (128 mg, 5.0 mol%), $Cu(OAc)_2{\cdot}H_2O$ (0.8350 g, 1.0 equiv) and $AgSbF_6$ (0.287 g, 0.2 equiv) in 1,2-DCE 50 ml. The reaction mixture was refluxed for 12 h. After cooling to ambient temperature, the reaction mixture was diluted with CH_2Cl_2, filtered through celite and the filtrate was concentrated under reduced pressure. The crude residue was purified through a silica gel column using hexane and ethyl acetate as eluent (85:15) to afford **3a** in 66% yield.

12. The preliminary mechanism study

To an oven-dried 25 mL round bottom flask fitted with a reflux condenser were added benzo[*a*]phenazin-5-ol **1** (0.3 mmol) and CD_3OD (10 equiv.) in 1,2-dichloroethane (1,2-DCE). This mixture was refluxed for 12 h. After cooling to ambient temperature, the solvent was evaporated under reduced pressure and the residue was purified by flash column chromatography on a silica gel to give the mixture **1a**/**1aa**. The mixture was analyzed using ^1H NMR spectrometer. The deuterium incorporation was estimated to be 69% as shown in the following scheme.

To an oven-dried 25 mL round bottom flask fitted with a reflux condenser were added benzo[*a*]phenazin-5-ol **1** (0.3 mmol), acetylene **2** (0.3 mmol), $[RuCl_2(p\text{-cymene})]_2$ (5.0 mol%), $Cu(OAc)_2 \cdot H_2O$ (1 equiv) $AgSbF_6$ (20 mol%) and CD_3OD (10 equiv.) in 1,2-dichloroethane (1,2-DCE). This mixture was refluxed for 12 h. After cooling to ambient temperature, the solvent was evaporated under reduced pressure and the residue was purified by flash column chromatography on a silica gel to give the mixture **1a**/**1ab**. The mixture was analyzed using ^1H NMR spectrometer. The deuterium incorporation was estimated to be 66% and 75% as shown in the following scheme.



Scheme S10. The preliminary mechanism study

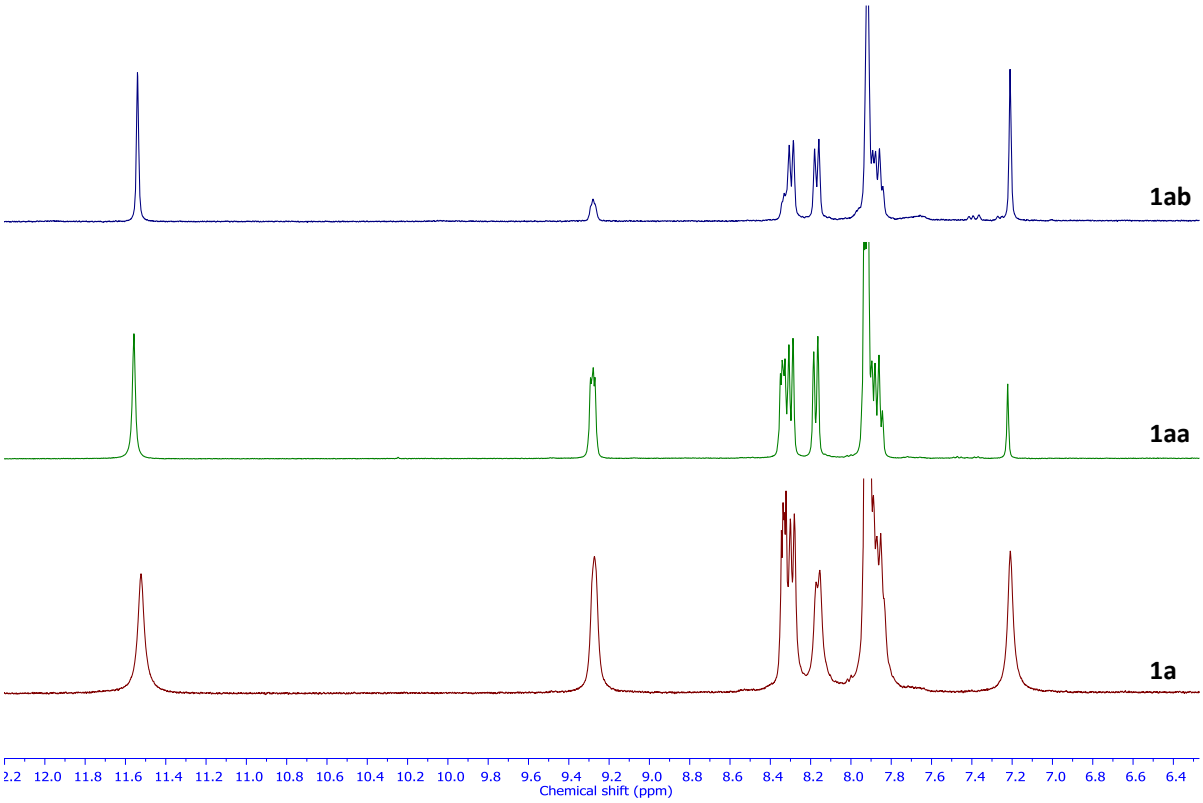
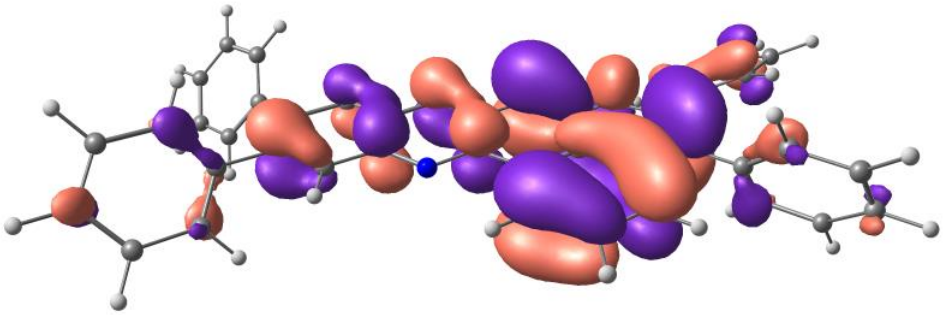


Figure S7. ^{1}H NMR spectrum of preliminary mechanism study

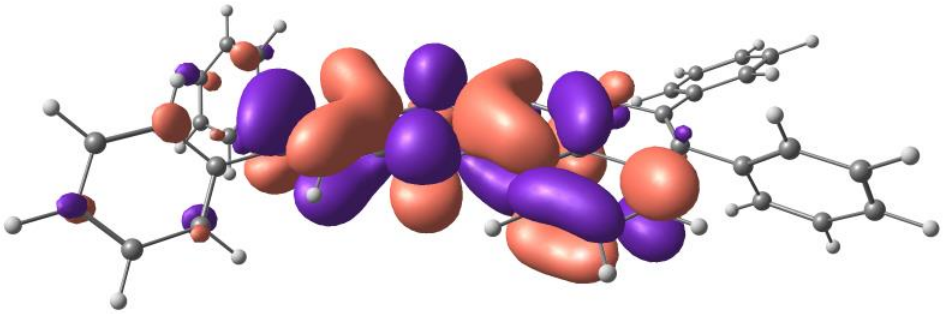
13. Molecular orbital amplitude plots of HOMO and LUMO levels of 3p

Density functional theory (DFT) calculation of the compound **3p** was carried out using Gaussian 09 program package. The ground state geometries of **3p** was optimized using DFT-B3LYP-6-311G basis set. In order to get the information about absorption properties TDDFT calculations ware carried out using the optimized geometries.

HOMO



LUMO:



E: 2.9468415951338 eV

HOMO: -5.1692517844812

LUMO: -2.2224101893474

Figure S8. HOMO and LUMO

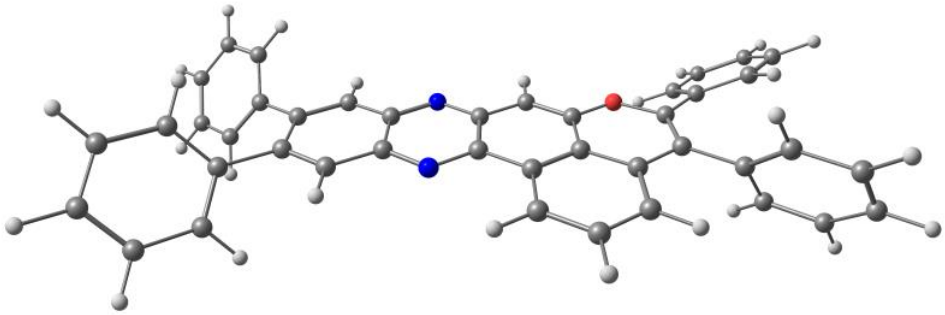
Table S2. Atom coordinates

1	6	-2.216690000	0.775436000	-0.026287000
2	6	-1.051180000	1.487909000	-0.027945000
3	6	0.196769000	0.791860000	-0.022550000
4	6	0.213915000	-0.656494000	-0.011451000
5	6	-1.045961000	-1.388371000	-0.006593000
6	6	-2.264480000	-0.660266000	-0.021291000
7	6	2.532209000	-0.629410000	-0.003026000
8	6	2.517575000	0.804183000	-0.020415000
9	6	3.756046000	1.497031000	-0.011826000
10	1	3.717931000	2.579707000	0.024382000
11	6	4.970805000	0.830145000	0.002963000
12	6	4.983424000	-0.621998000	-0.013575000
13	6	3.777760000	-1.305909000	-0.004994000
14	1	-1.050488000	2.569284000	-0.035181000
15	1	3.756212000	-2.389253000	-0.040543000
16	6	6.243031000	-1.419857000	-0.083780000
17	6	6.439241000	-2.505244000	0.792338000
18	6	7.227855000	-1.157484000	-1.056339000
19	6	7.585666000	-3.301323000	0.703280000
20	1	5.693655000	-2.713401000	1.553521000
21	6	8.370523000	-1.956310000	-1.147838000
22	1	7.071465000	0.574133000	1.748635000
23	6	8.555614000	-3.029896000	-0.267836000
24	1	7.721188000	-4.129000000	1.392610000
25	1	9.114929000	-1.742391000	-1.908412000
26	1	9.445766000	-3.646929000	-0.338902000
27	6	6.217808000	1.647426000	0.079941000
28	6	7.199690000	1.401451000	1.059587000
29	6	6.402672000	2.734335000	-0.796552000
30	6	8.329094000	2.218165000	1.157732000

31	1	7.091575000	-0.331273000	-1.745153000
32	6	7.536323000	3.547778000	-0.701306000
33	1	5.658395000	2.930586000	-1.562094000
34	6	8.503581000	3.292868000	0.276948000
35	1	9.071371000	2.017253000	1.923907000
36	1	7.663566000	4.376586000	-1.390799000
37	1	9.383515000	3.923794000	0.353121000
38	7	1.347097000	1.500310000	-0.026886000
39	7	1.367554000	-1.339103000	-0.001543000
40	6	-3.521342000	-1.329628000	-0.012938000
41	6	-4.753283000	-0.522537000	-0.026013000
42	6	-4.666743000	0.839550000	-0.049028000
43	6	-3.522765000	-2.731000000	0.033769000
44	6	-2.317026000	-3.449971000	0.046794000
45	6	-1.090180000	-2.796007000	0.022334000
46	1	-4.463742000	-3.265212000	0.062654000
47	1	-2.347999000	-4.534277000	0.079787000
48	1	-0.154368000	-3.340517000	0.032653000
49	8	-3.415784000	1.483578000	-0.029158000
50	6	-6.071789000	-1.225290000	0.049505000
51	6	-6.540149000	-1.997834000	-1.031076000
52	6	-6.854884000	-1.152299000	1.215597000
53	6	-7.765493000	-2.668023000	-0.950492000
54	1	-5.943501000	-2.065336000	-1.935754000
55	6	-8.080502000	-1.821587000	1.295225000
56	1	-6.499896000	-0.565065000	2.056119000
57	6	-8.540019000	-2.580507000	0.212724000
58	1	-8.114978000	-3.254739000	-1.794310000
59	1	-8.673465000	-1.751891000	2.201680000
60	1	-9.490745000	-3.100298000	0.274993000
61	6	-5.739878000	1.854730000	-0.079403000

62	6	-5.536854000	3.087452000	0.575812000
63	6	-6.948286000	1.651280000	-0.775134000
64	6	-6.524731000	4.073771000	0.557840000
65	1	-4.601716000	3.262831000	1.092949000
66	6	-7.931255000	2.643509000	-0.793513000
67	1	-7.114534000	0.726082000	-1.311241000
68	6	-7.727633000	3.855701000	-0.124046000
69	1	-6.353863000	5.013052000	1.074073000
70	1	-8.853313000	2.471208000	-1.339368000
71	1	-8.494116000	4.623974000	-0.139932000

Optimized geometry:



Possible packing arrangement:

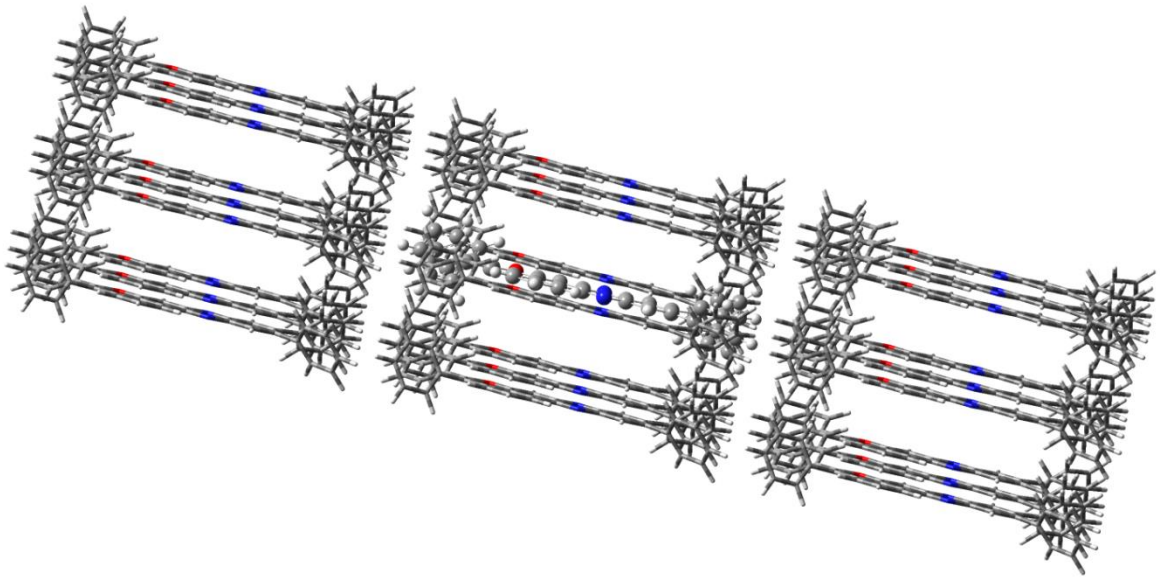


Figure S9. Optimized geometry and Possible packing arrangement:

14. Photophysical properties of compounds (3a-v):

Table S3. Photophysical properties of compounds (3a-v):

entry	$\lambda_{max,ab}$, (nm)		$\lambda_{max,em}$, (nm)		Stokes shift[e](cm^{-1})		Quantum Yield (Φ_F)[f]
	Solution[a]	Solid[b]	Solution[a,c]	Solid[b,d]	Solution[a]	Solid[b]	
3a	494	508	553	590	2160	2736	0.54
3b	498	511	559	585	2191	2475	0.43
3c	500	514	572	604	2517	2899	0.48
3d	490	504	544	619	2026	3686	0.35
3e	489	496	540	565	1931	2462	0.37
3f	488	501	539	565	1939	2261	0.39
3h	486	498	538	566	1989	2412	0.32
3i	495	497	554	566	2151	2453	0.45
3j	497	550	563	599	2359	1487	0.41
3k	501	527	563	579	2198	1704	0.47
3l	499	503	563	581	2278	2669	0.52
3m	503	510	567	588	2244	2601	0.65
3n	491	503	544	573	1984	2429	0.37
3o	518	529	580	616	2064	2670	0.12
3p	507	521	559	607	1835	2719	0.86
3q	503	509	551	582	1732	2464	0.23
3r	516	525	568	599	1774	2353	0.37
3s	501	512	594	630	3125	3658	0.17
3t	431	435	545	580	4853	5747	0.52
3u	526	532	609	640	2591	3172	0.28
3v	511	540	632	637	3747	2820	0.22

[a] In DCM solvent at 5 μM. [b] Thin film [c] Excitation wavelengh: 450-500 nm. [d] Excitation wavelengh: 450-540 nm. [e] Stokes shift= $\lambda_{max,ab}$ - $\lambda_{max,em}$ (cm^{-1}). [f]Determined by Fluorescein (Φ_F = 0.79) as a standard in 0.1M ethanol solution.

15. Absorption and fluorescent spectra of synthesised compounds:

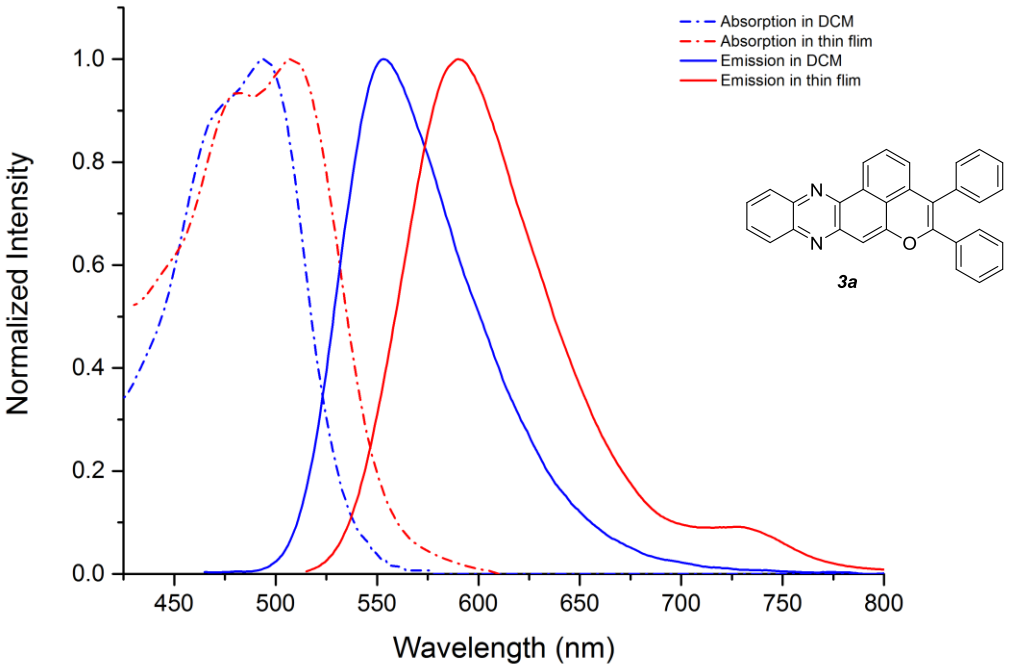


Figure S10. Absorption and fluorescent spectra of compound 3a

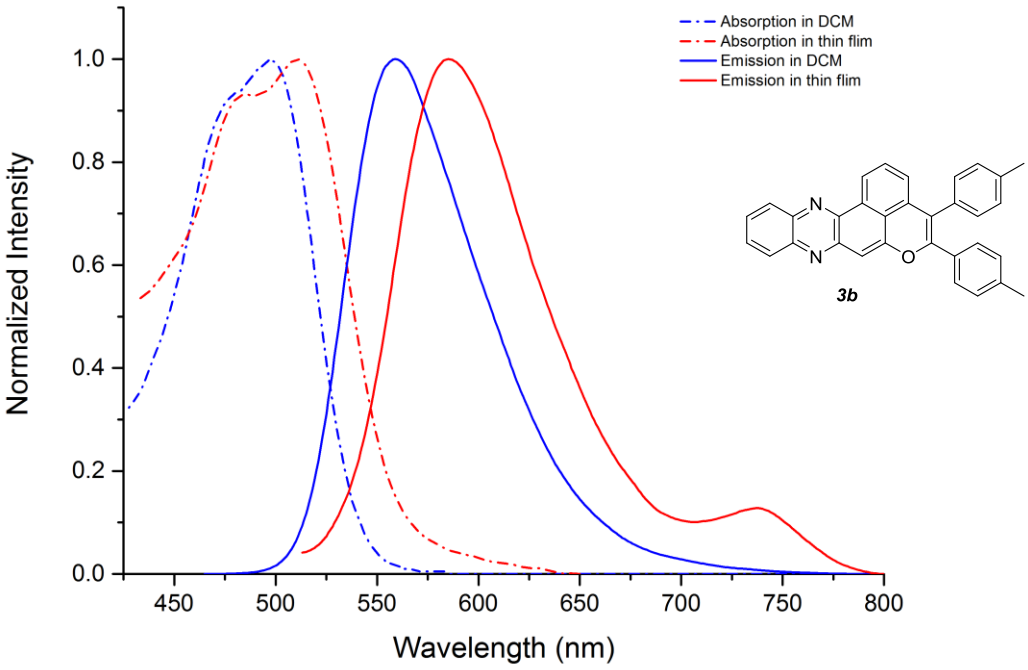


Figure S11. Absorption and fluorescent spectra of compound 3b

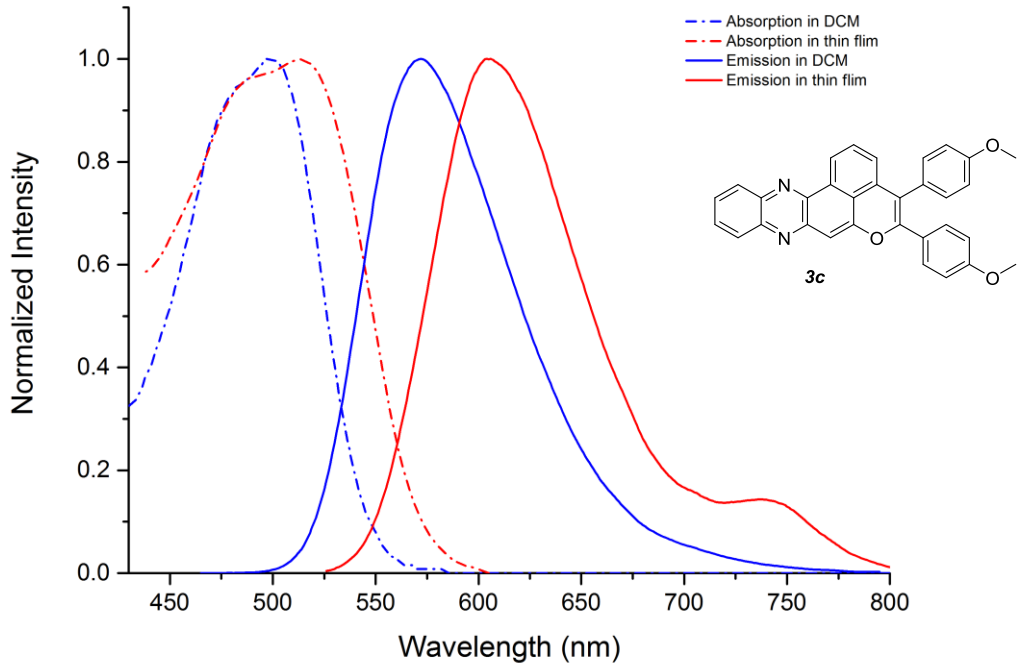


Figure S12. Absorption and fluorescent spectra of compound 3c

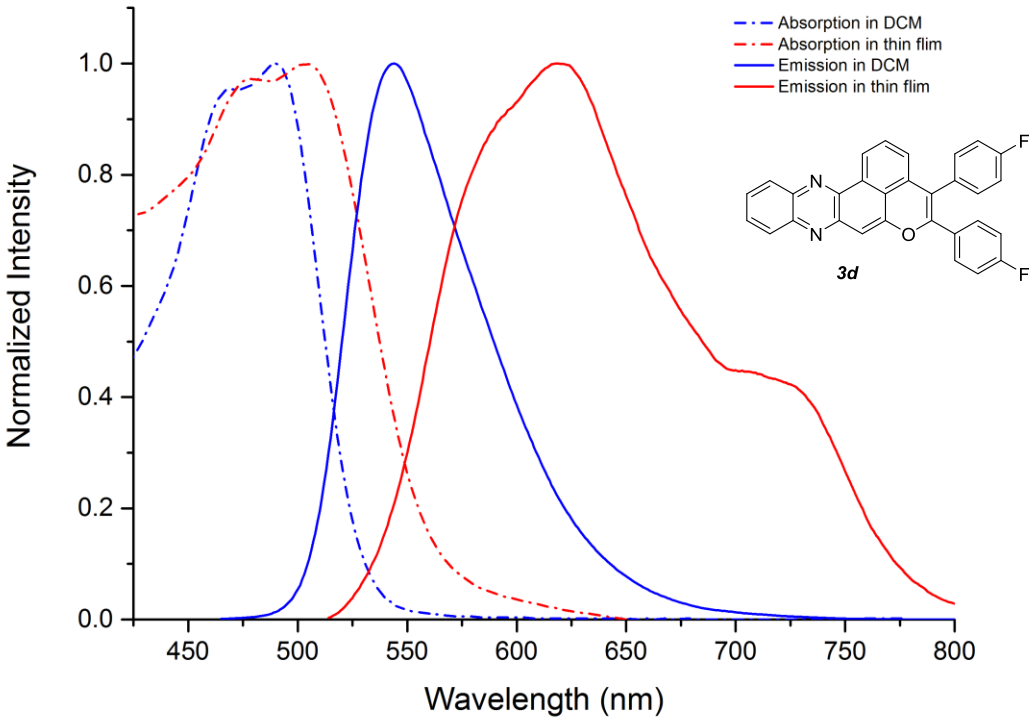


Figure S13. Absorption and fluorescent spectra of compound 3d

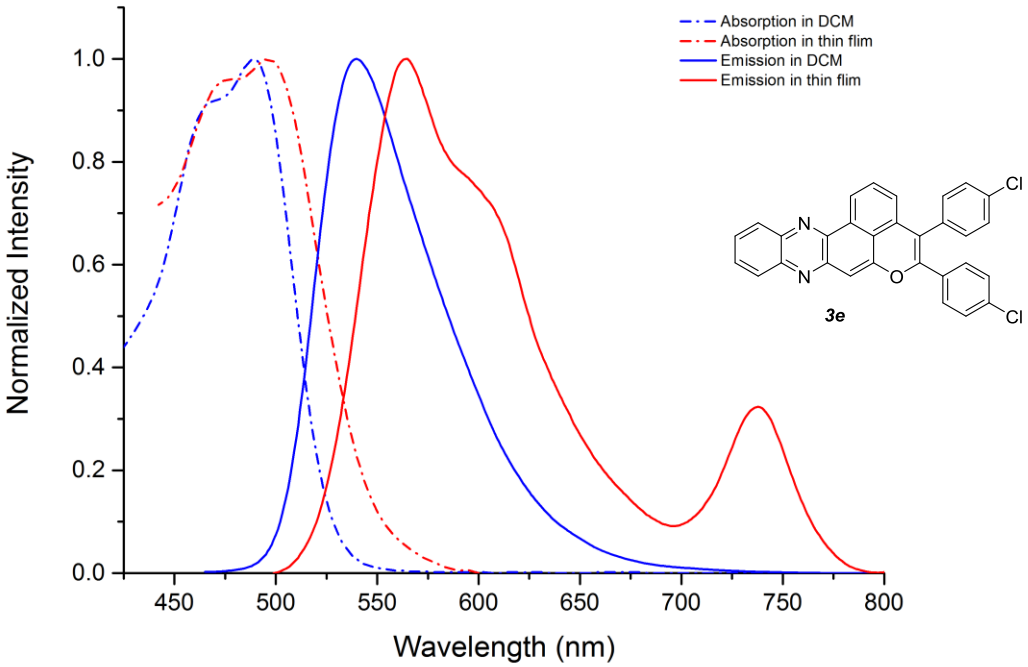


Figure S14. Absorption and fluorescent spectra of compound 3e

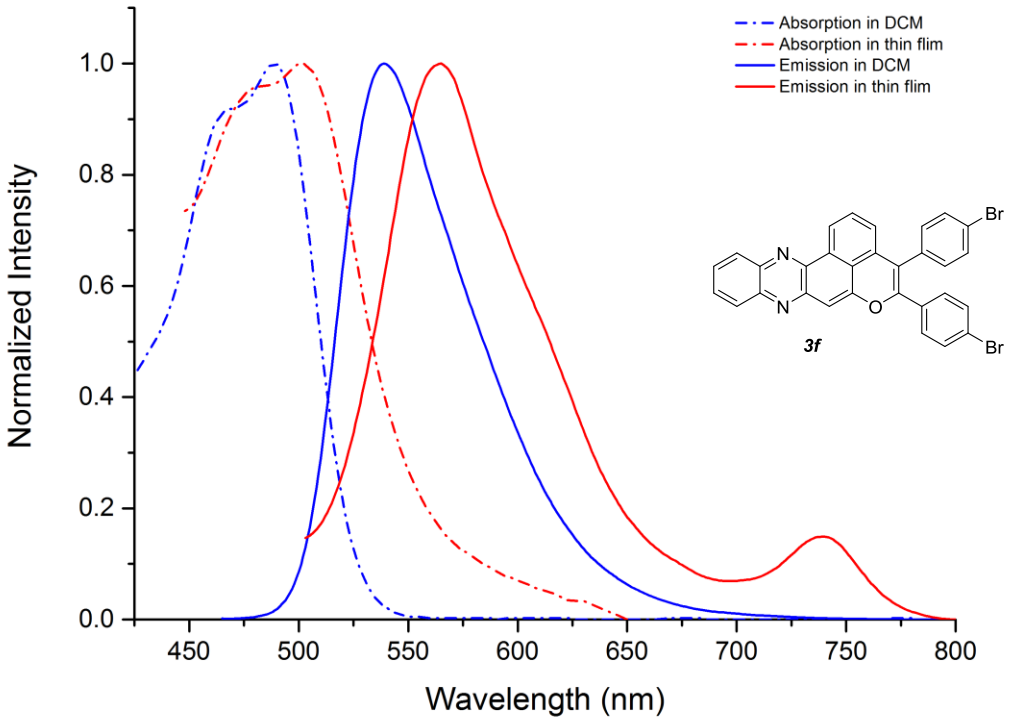


Figure S15. Absorption and fluorescent spectra of compound 3f

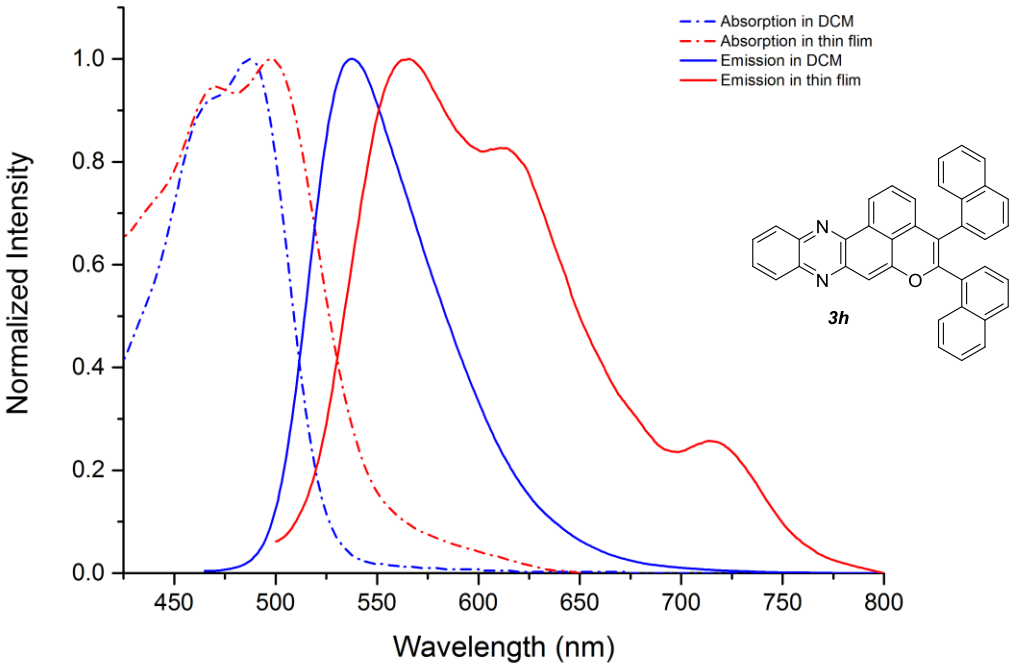


Figure S16. Absorption and fluorescent spectra of compound 3h

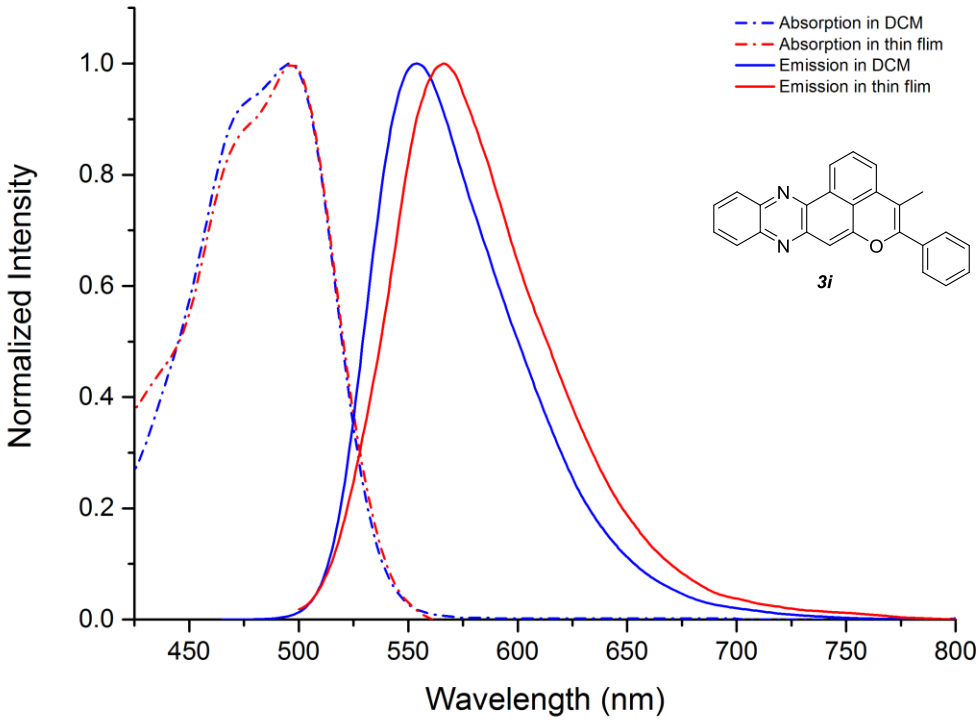


Figure S17. Absorption and fluorescent spectra of compound 3i

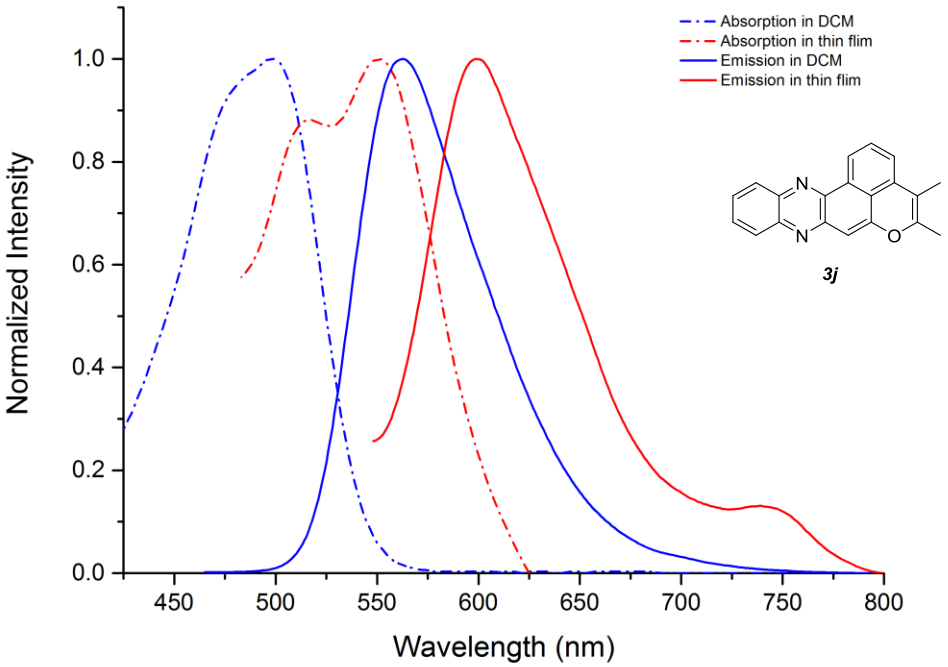


Figure S18. Absorption and fluorescent spectra of compound 3j

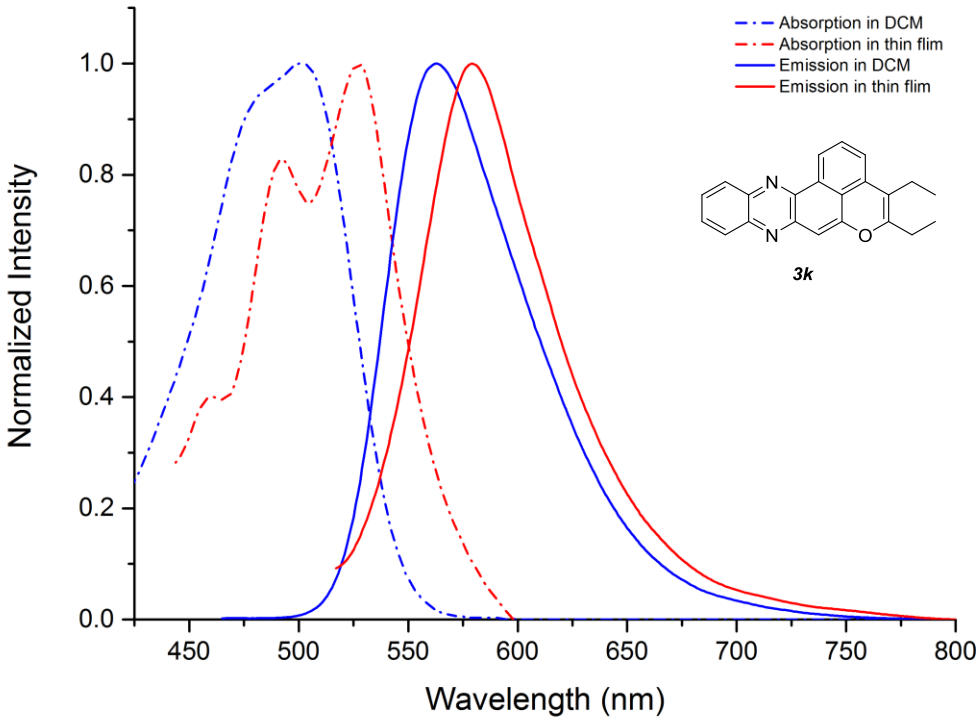


Figure S19. Absorption and fluorescent spectra of compound 3k

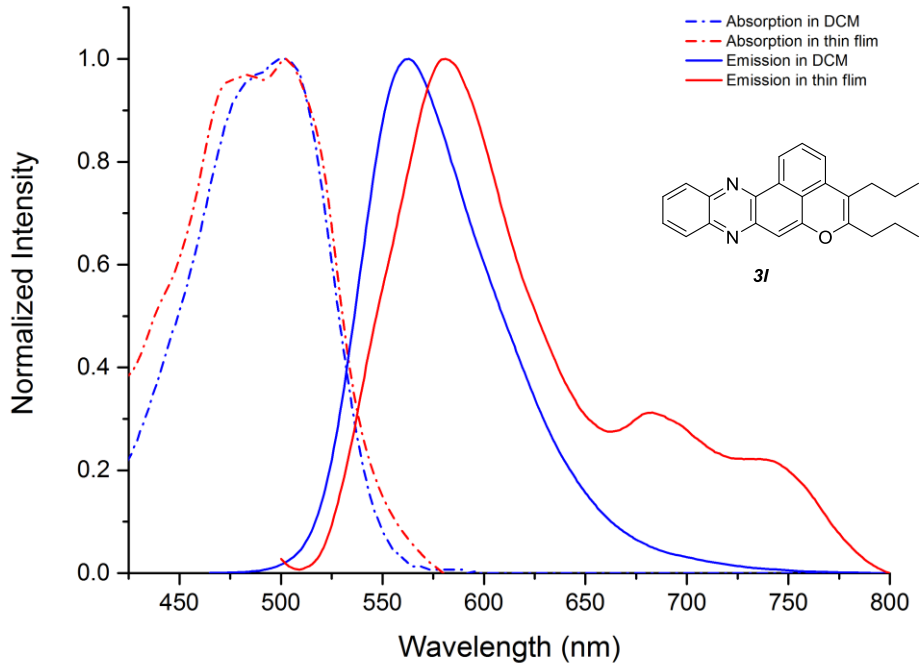


Figure S20. Absorption and fluorescent spectra of compound 3l

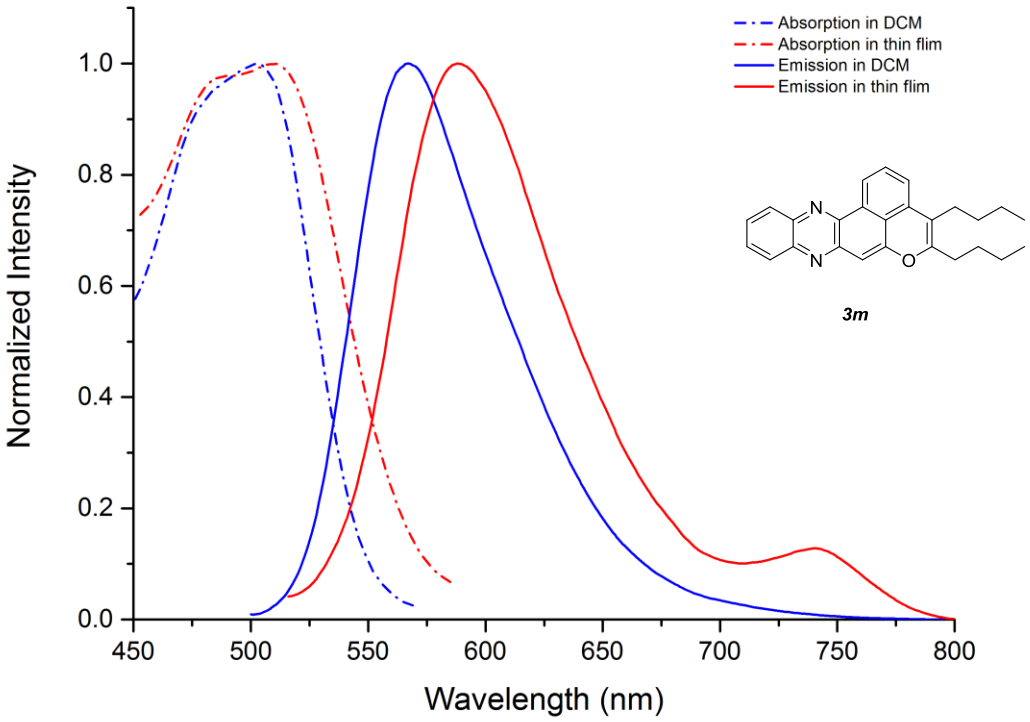


Figure S21. Absorption and fluorescent spectra of compound 3m

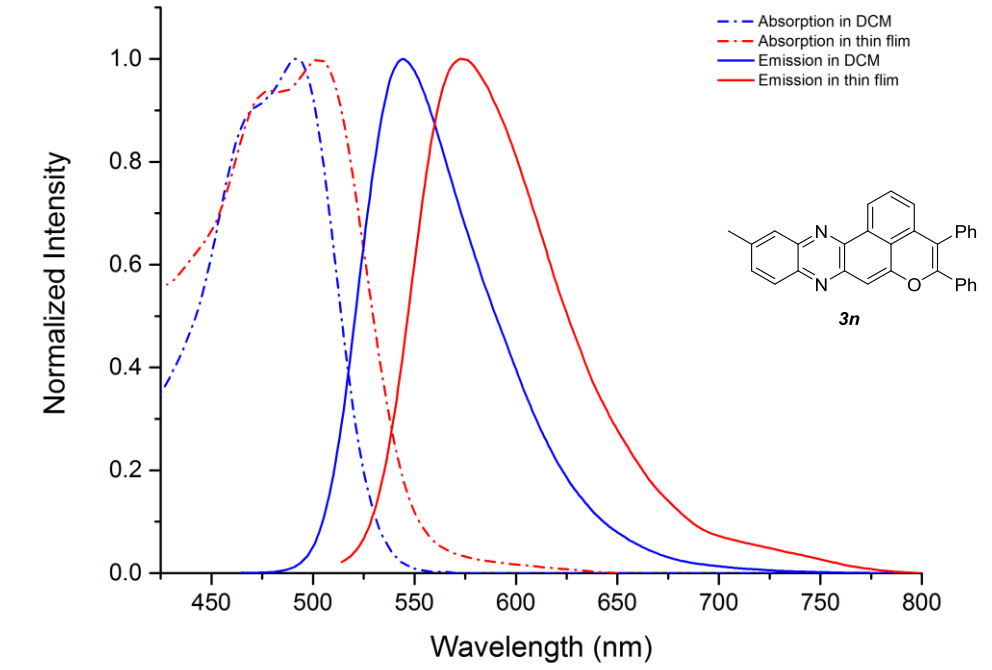


Figure S22. Absorption and fluorescent spectra of compound 3n

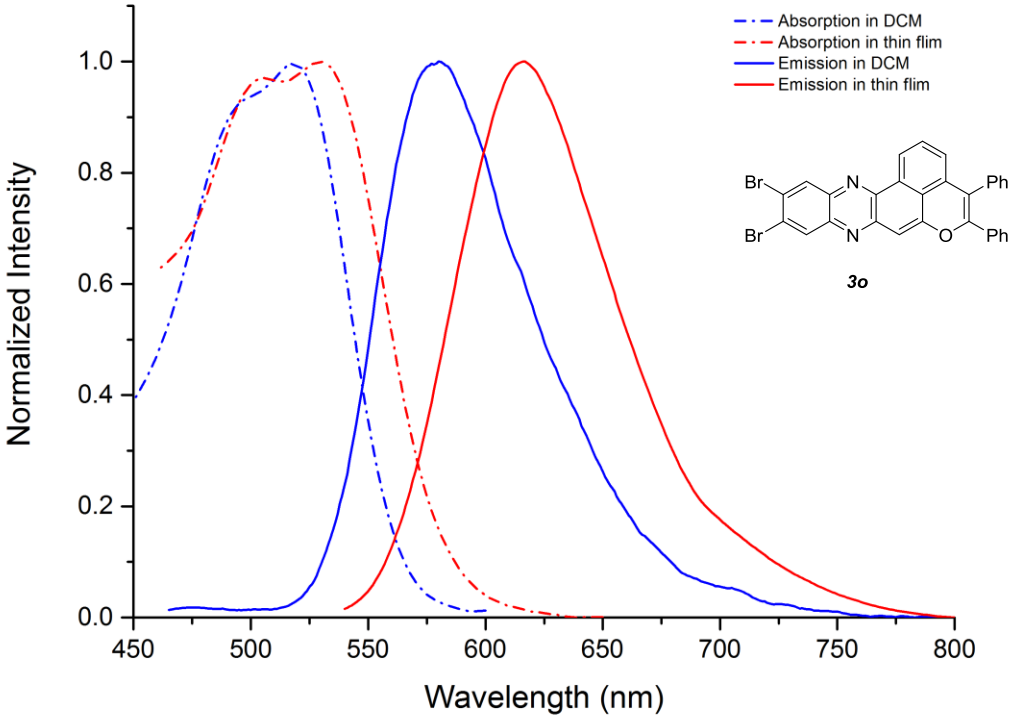


Figure S23. Absorption and fluorescent spectra of compound 3o

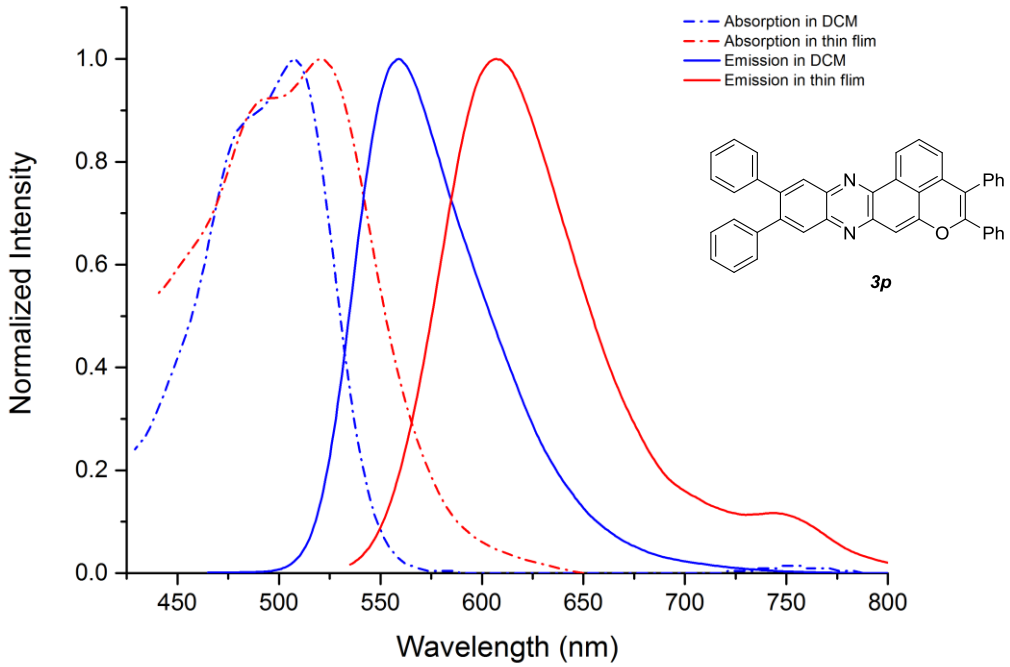


Figure S24. Absorption and fluorescent spectra of compound 3p

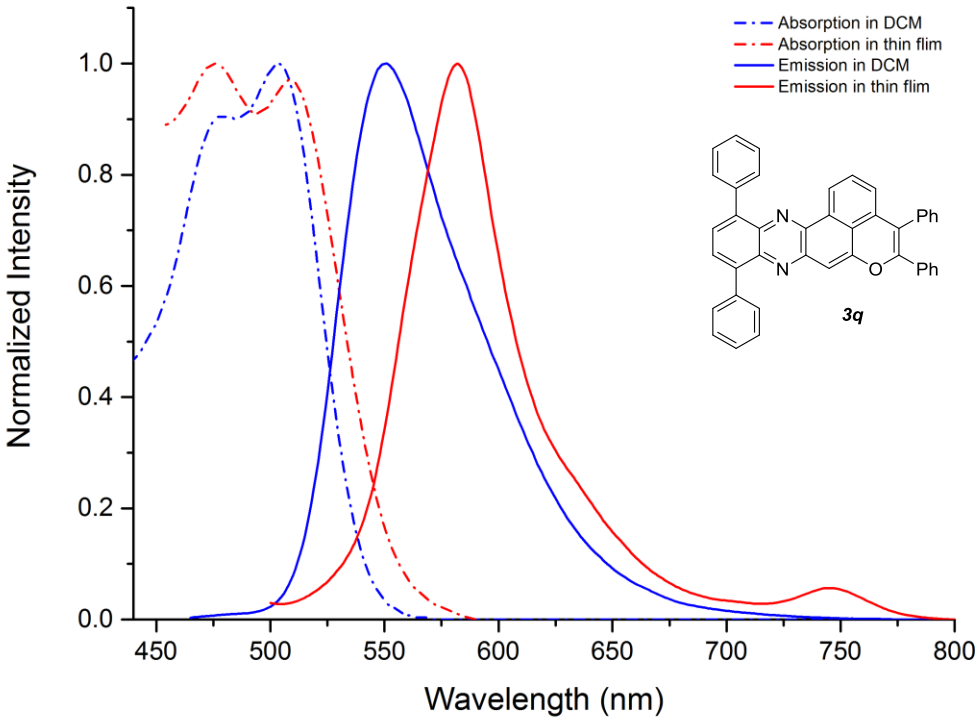


Figure S25. Absorption and fluorescent spectra of compound 3q

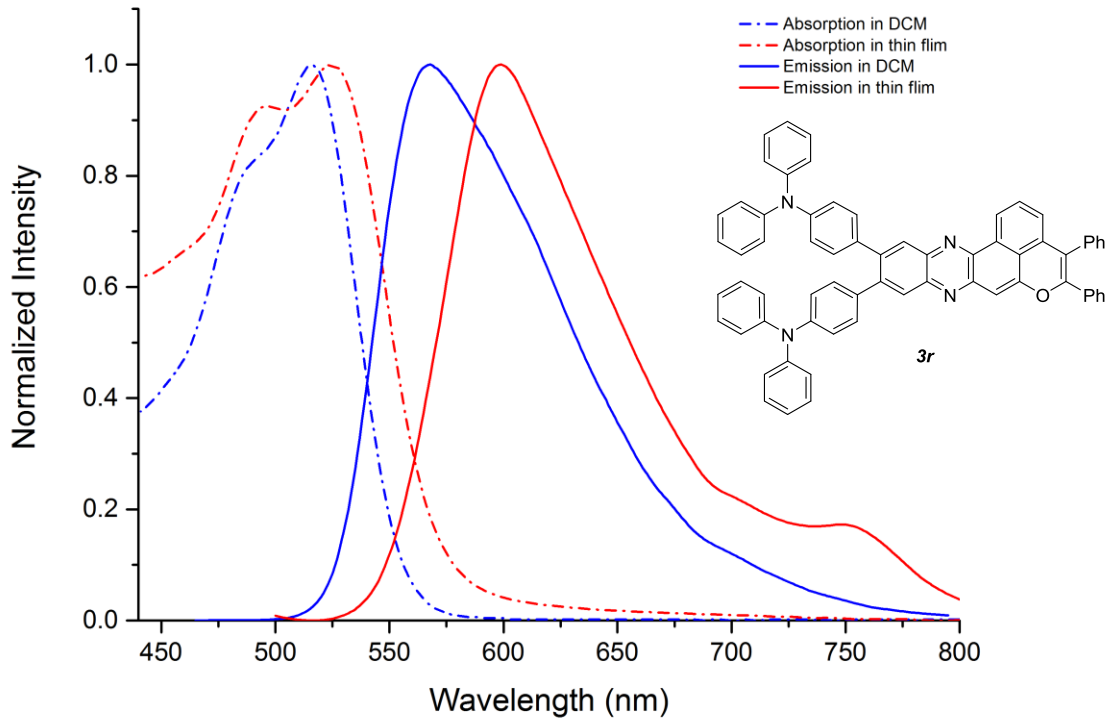


Figure S26. Absorption and fluorescent spectra of compound 3r

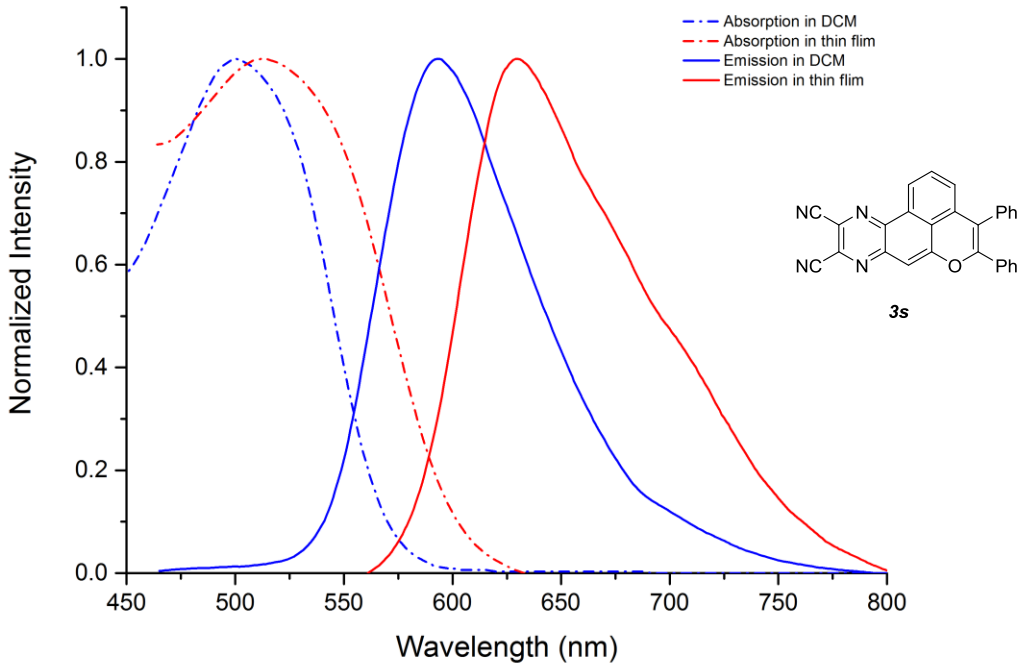


Figure S27. Absorption and fluorescent spectra of compound 3s

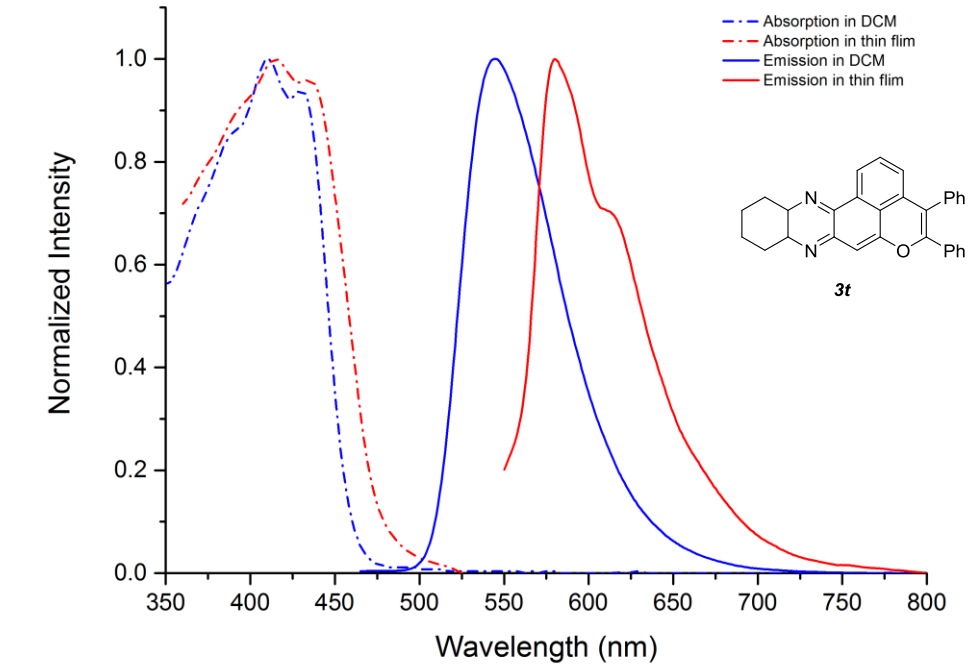


Figure S28. Absorption and fluorescent spectra of compound 3t

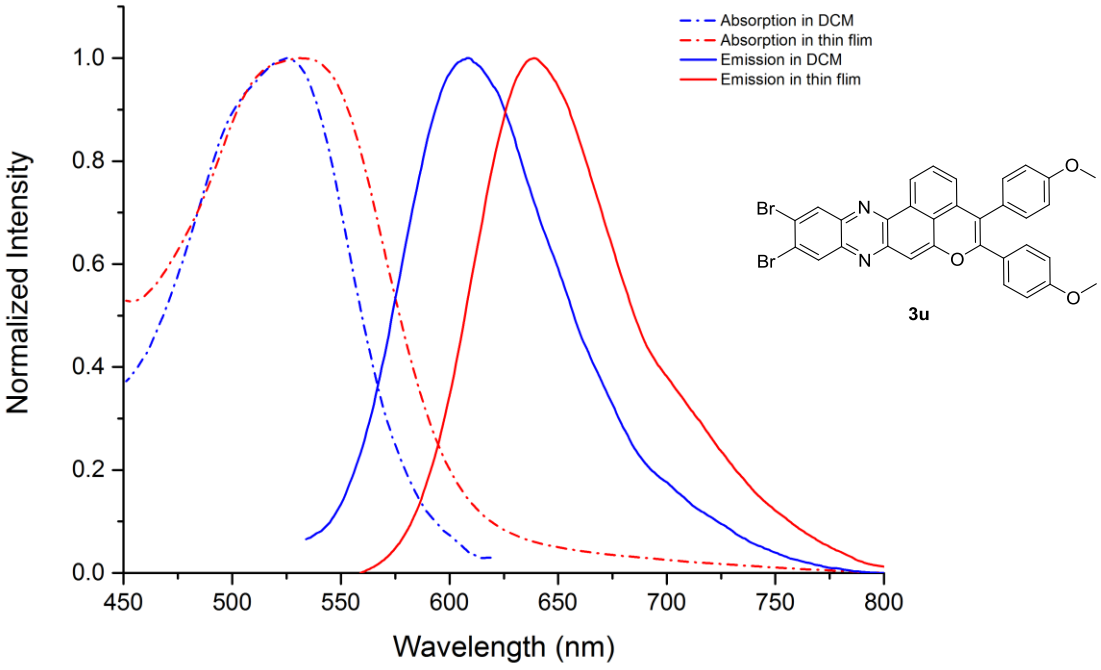


Figure S29. Absorption and fluorescent spectra of compound 3u

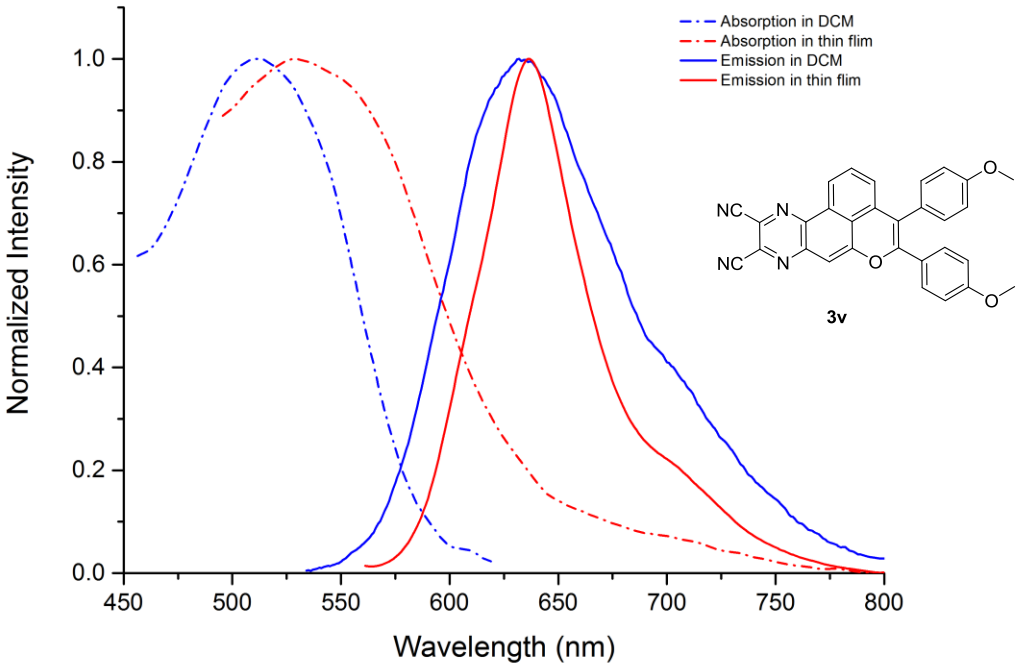


Figure S30. Absorption and fluorescent spectra of compound 3v

16. Solvatochromism

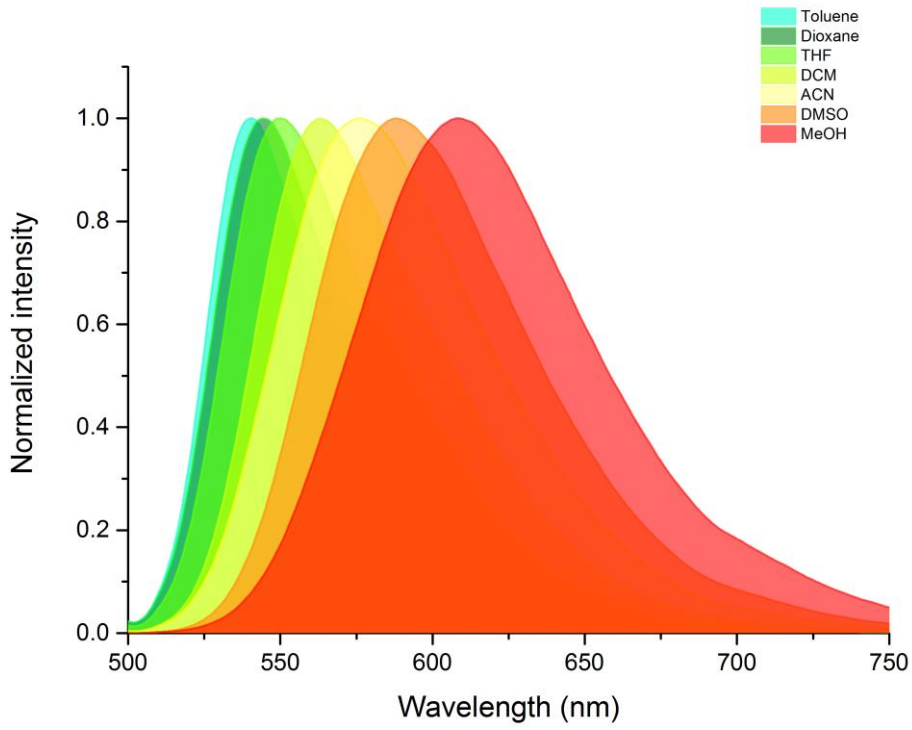


Figure S31. Solvatochromism spectra of compound 3p

17. Live cell imaging experiment

Fluorescence compounds of **3p** and **3t** were examined for intracellular imaging in A549, HepG2 and U937 cells. A549 and HepG2 cells were grown in DMEM, and U937 cells were grown in RMPI media with 10% fetal bovine serum, 1% penicillin/streptomycin at 37 °C under a 5% CO_2 atmosphere for 24 h. The cells were incubated at 37 °C with 10 μM of the fluorescence compounds of **3p** and **3t** for 30 min separately. After thorough washing with PBS, the cells were stained with nucleus staining dye DAPI (2 μM) for another 20 min. After that, the staining solution was replaced with fresh PBS to remove the remaining dye. Finally, the blue, green and red fluorescence images of A549, HepG2 and U937 cells were captured using a confocal laser scanning microscope (ZEISS, LSM710). Excitation wavelength for Green: 488 nm; Emission collection: 490-554 nm; Excitation wavelength for Red: 561 nm; Emission collection: 570-680 nm; Excitation wavelength for Blue: 405 nm; Emission collection: 410- 460 nm.

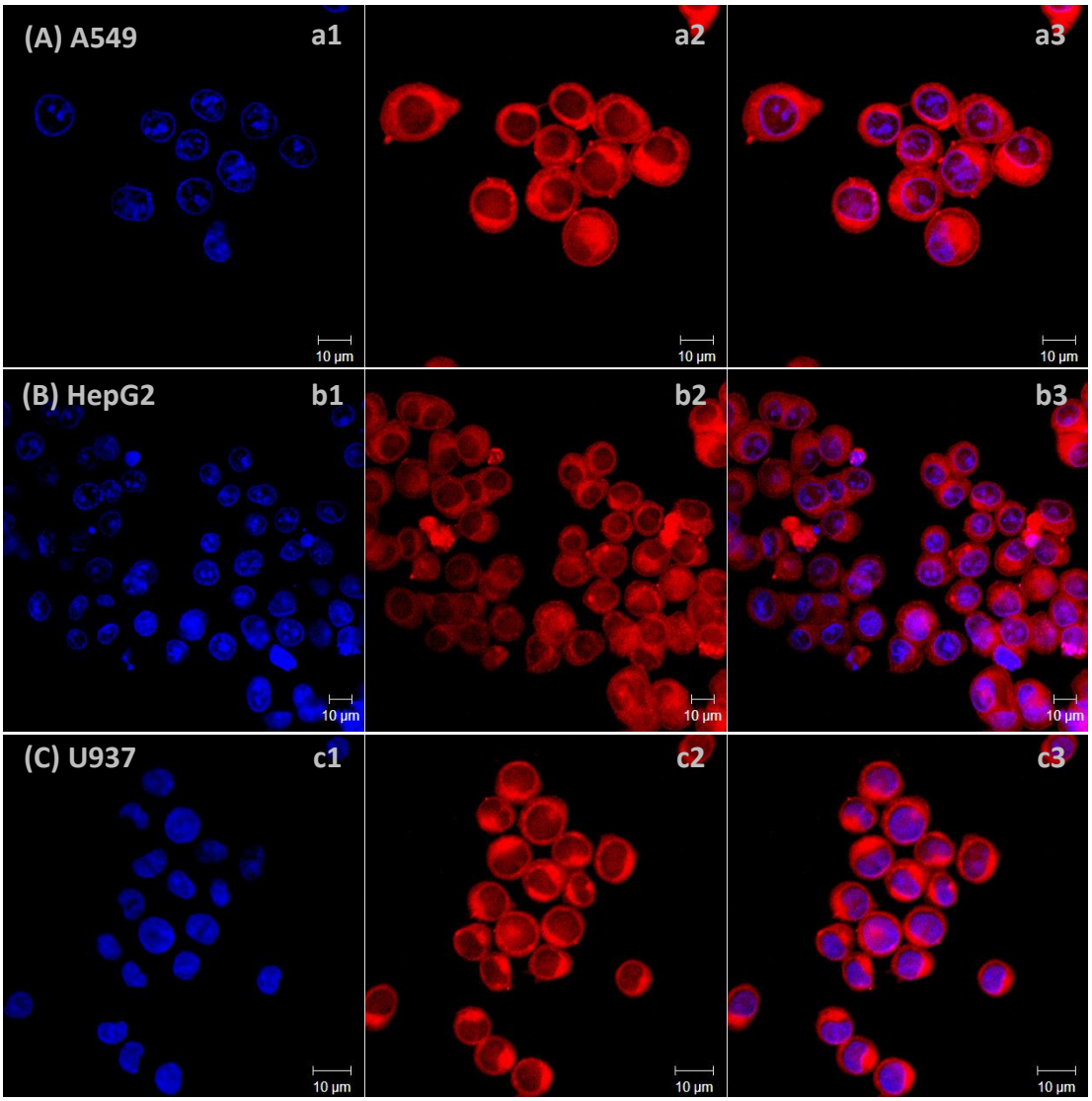


Figure S32. Live cell imaging of compound **3p** in A549, HepG2 and U937 cells. (A) Row 1 (a1–a3): A549 cells treated with 10 µM **3p** for 30 min. (B) Row 2 (b1–b3): HepG2 cells treated with 10 µM **3p** for 30 min. (C) Row 3 (c1–c3): U937 cells treated with 10 µM **3p** for 30 min. For A, B and C, column 1 (a1–c1): DAPI stained blue fluorescence images; column 2 (a2–c2): red fluorescence images; column 3 (a3–c3): merging of the blue and red fluorescence images. Scale bar = 10 µm. Excitation and emission wavelength: 405 nm and 410-460 nm for blue fluorescence images; 561 nm and 570-680 nm for red fluorescence images.

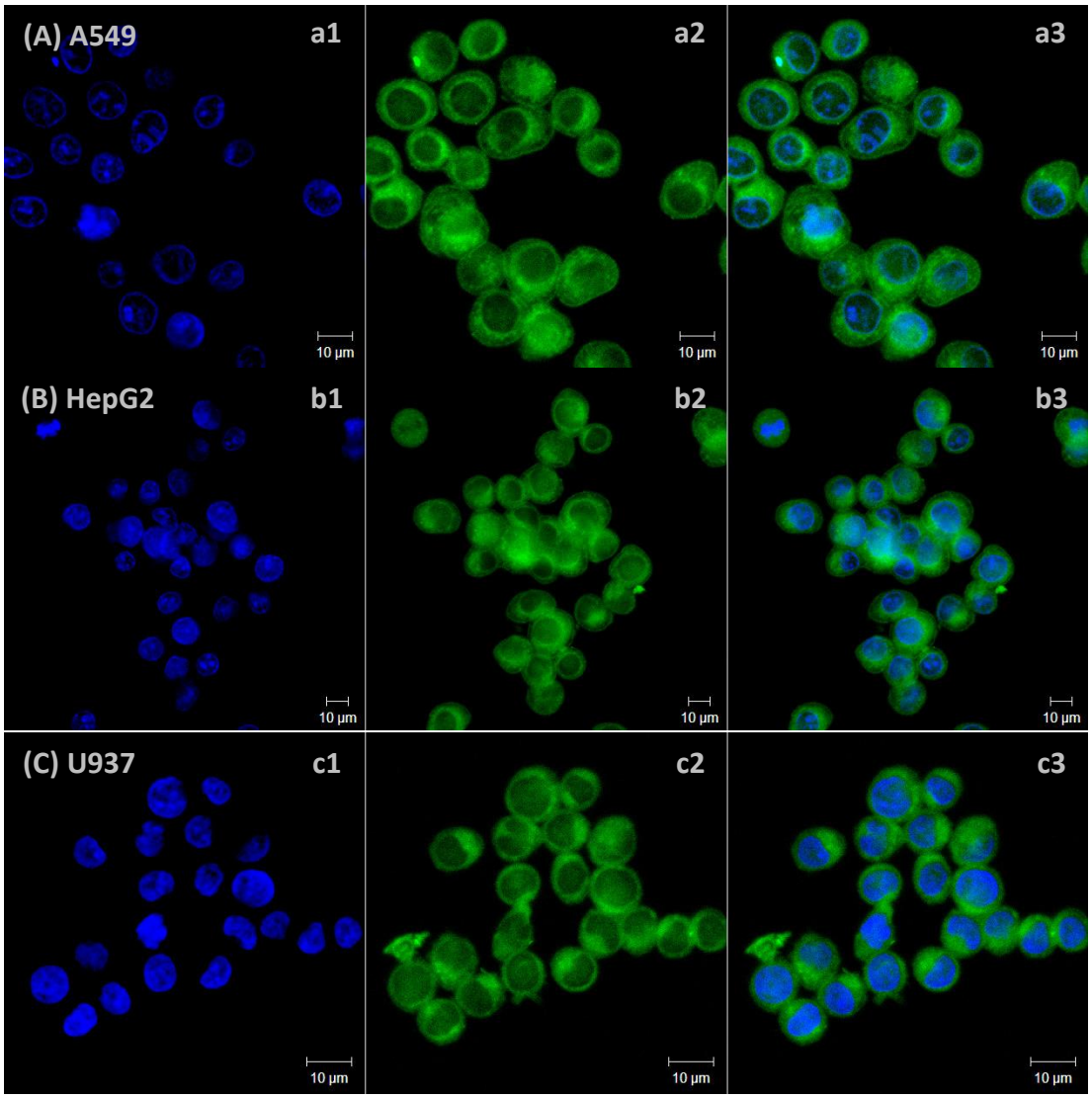


Figure S33. Live cell imaging of compound **3t** in A549, HepG2 and U937 cells. (A) Row 1 (a1–a3): A549 cells treated with 10 µM **3t** for 30 min. (B) Row 2 (b1–b3): HepG2 cells treated with 10 µM **3t** for 30 min. (C) Row 3 (c1–c3): U937 cells treated with 10 µM **3t** for 30 min. For A, B and C, column 1 (a1–c1): DAPI stained blue fluorescence images; column 2 (a2–c2): green fluorescence images; column 3 (a3–c3): merging of the blue and green fluorescence images. Scale bar = 10 µm. Excitation and emission wavelength: 405 nm and 410-460 nm for blue fluorescence images; 488 nm and 490-554 nm for green fluorescence images.

18. Cell viability assay

Cytotoxic properties of the synthesized compounds **3p** and **3t** were studied against A549, HepG2 and U937 cells. A549 and HepG2 cells were maintained in complete tissue culture medium DMEM and U937 cells were maintained in RPMI with 10% fetal bovine serum and 2 mM L-glutamine, along with antibiotics (about 100 International Unit per mL of penicillin, 100 μg per mL of streptomycin) with the pH adjusted to 7.2. Medium of 50 μL containing 5000 cells per well and different concentrations of synthesized compounds **3p** and **3t** were seeded in 96 well plates. The cells were cultivated at 37 °C with 5% CO_2 and 95% air in 100% relative humidity. AQueous One Solution reagent of 20 μL was added to each well of CellTiter 96® according to the manufacturer's guidelines and incubated at 37 °C for 1–4 h under a humidified, 5% CO_2 atmosphere. The cytotoxicity against cells was determined by measuring the absorbance of the converted dye at 490 nm in an ELISA reader. Cytotoxicity of each sample was expressed as an IC_{50} value.

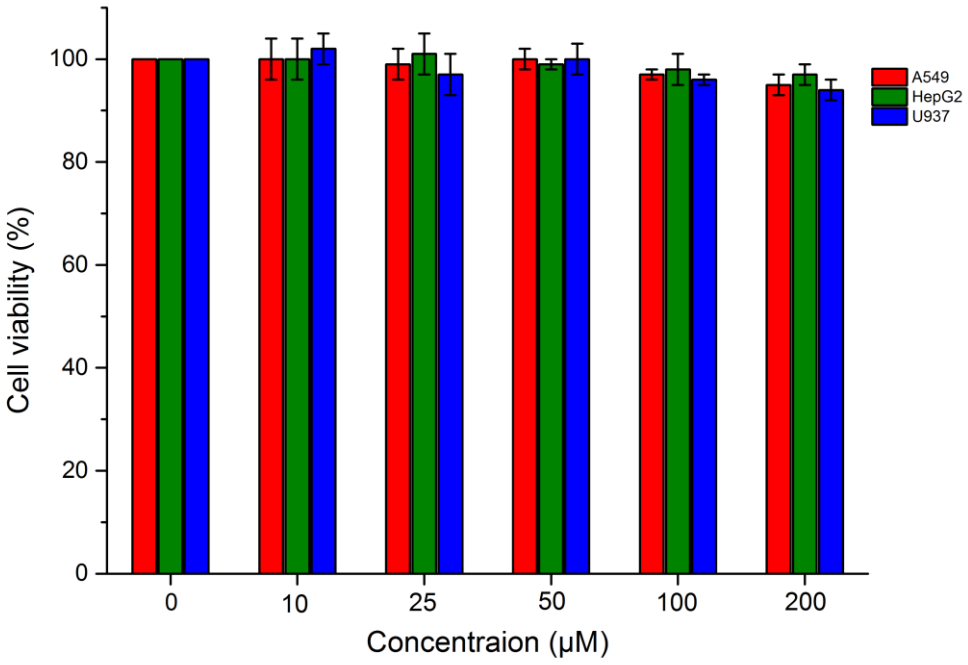


Figure S34. Cell viability assay in A549, HepG2 and U937 cells using MTS reagent. Cells have been incubated with Compound 3p (0-200 μM) for 48 h.

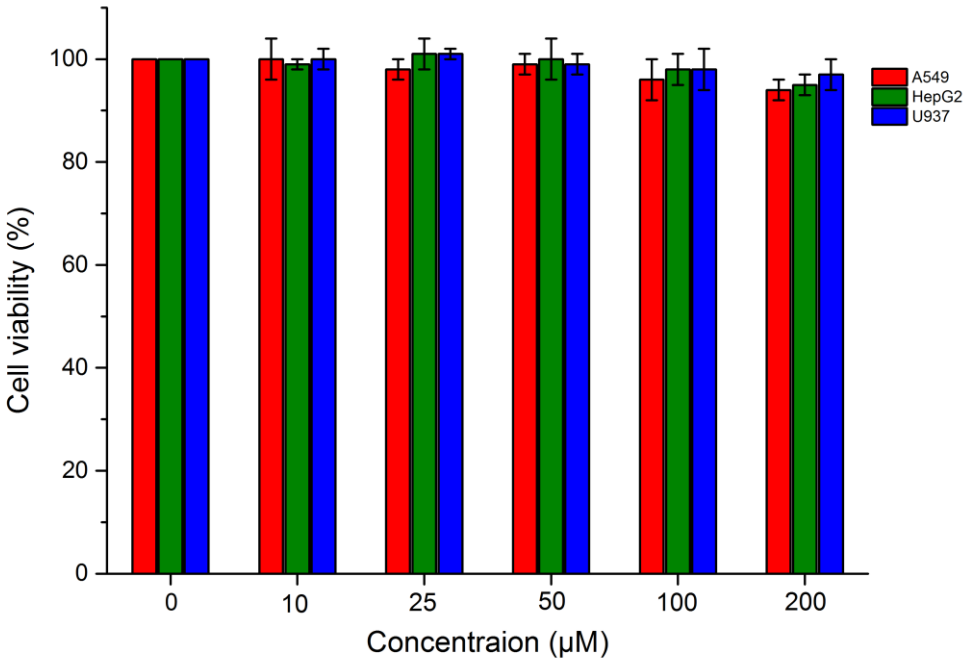
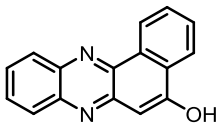


Figure S35. Cell viability assay in A549, HepG2 and U937 cells using MTS reagent. Cells have been incubated with Compound **3t** (0-200 μM) for 48 h.

19. Spectral Data for synthesized compounds

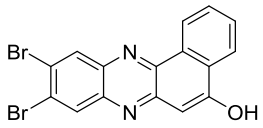
benzo[a]phenazin-5-ol (1a)



1a

Yield: 95 %, Yellow Solid; **^{1}H NMR (400 MHz, DMSO-d6)** δ 11.52 (s, 1H), 9.27 (s, 1H), 8.41 – 8.23 (m, 2H), 8.16 (d, *J* = 7.1 Hz, 1H), 7.99 – 7.80 (m, 4H), 7.21 (s, 1H) ppm; **^{13}C NMR (100 MHz, DMSO-d6)** δ 131. 5, 130.7, 130.7, 130.6, 130.2, 129.7, 129.3, 129.3, 126.8, 125.3, 124.0, 123.4, 121.2, 103.9 ppm.

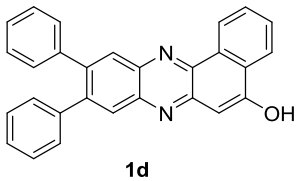
9,10-dibromobenzo[a]phenazin-5-ol (1c)



1c

Yield: 87 %, Yellow Solid; **^{1}H NMR (400 MHz, DMSO-d6)** δ 11.73 (s, 1H), 9.12 (dd, *J* = 6.5, 2.6 Hz, 1H), 8.58 (s, 1H), 8.48 (s, 1H), 8.31 – 8.27 (m, 1H), 7.95 – 7.87 (m, 2H), 7.09 (s, 1H) ppm; **^{13}C NMR (100 MHz, DMSO-d6**) δ 149.8, 142.1, 139.0, 133.4, 131.1, 131.1, 131.0, 129.5, 129.46, 125.9, 125.4, 123.6, 123.5, 103.5 ppm.

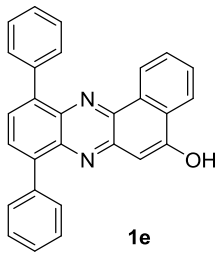
9,10-diphenylbenzo[a]phenazin-5-ol (1d)



1d

Yield: 82 %, Yellow Solid; **^{1}H NMR (400 MHz, DMSO-d6)** δ 11.58 (s, 1H), 9.26 (s, 1H), 8.33 (dd, *J* = 6.3, 2.9 Hz, 1H), 8.22 (s, 1H), 8.11 (s, 1H), 7.96 – 7.86 (m, 2H), 7.36 – 7.25 (m, 10H), 7.22 (s, 1H) ppm; **^{13}C NMR (100 MHz, DMSO-d6)** δ 165.8, 143.2, 140.5, 131.4, 130.6, 130.54, 130.1, 130.1, 129.2, 128.6, 127.7, 127.6, 125.3, 123.4, 123.42, 123.4, 104.0 ppm.

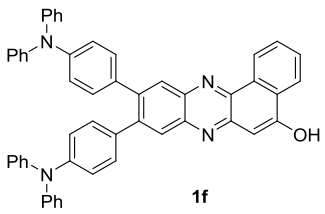
8,11-diphenylbenzo[a]phenazin-5-ol (1e)



1e

Yield: 79 %, Yellow Solid; **^{1}H NMR (400 MHz, DMSO-d6)** δ 11.49 (s, 1H), 8.95 (dd, *J* = 6.6, 2.5 Hz, 1H), 8.31 (dd, *J* = 6.6, 2.5 Hz, 1H), 7.98 – 7.91 (m, 4H), 7.89 – 7.82 (m, 4H), 7.63 (t, *J* = 7.6 Hz, 2H), 7.56 (dd, *J* = 14.3, 6.7 Hz, 3H), 7.50 (dd, *J* = 13.3, 5.9 Hz, 1H), 7.09 (s, 1H) ppm; **^{13}C NMR (100 MHz, DMSO-d6)** δ 157.58, 144.78, 140.97, 139.87, 139.08, 138.92, 138.87, 138.63, 137.76, 131.70, 131.39, 131.20, 130.45, 129.32, 129.24, 128.59, 128.43, 128.38, 128.04, 127.88, 125.30, 123.38, 104.11 ppm.

9,10-bis(4-(diphenylamino)phenyl)benzo[a]phenazin-5-ol (1f)

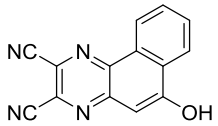


1f

Yield: 90 %, Red color solid; **^{1}H NMR (400 MHz, DMSO-d6)** δ 11.51 (s, 1H), 9.22 (d, *J* = 4.5 Hz, 1H), 8.33 – 8.25 (m, 1H), 8.17 (s, 1H), 8.06 (s, 1H), 7.90 – 7.84 (m, 2H), 7.28 (t, *J* = 7.6 Hz, 8H), 7.19 (s, 1H), 7.14 (d, *J* = 8.4 Hz, 4H), 7.08 – 7.00 (m, 12H), 6.90 (dd, *J* =

8.5, 1.3 Hz, 4H) ppm; **^{13}C NMR (100 MHz, DMSO-d6)** δ 157.3, 147.4, 147.4, 146.87, 146.7, 143.0, 142.3, 141.3, 139.9, 138.1, 134.5, 134.4, 131.4, 131.2, 130.4, 130.0, 129.6, 129.1, 128.4, 125.2, 124.5, 124.5, 123.74, 123.6, 122.8, 122.7, 104.0 ppm.

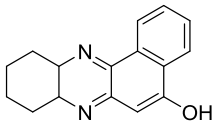
6-hydroxybenzo[f]quinoxaline-2,3-dicarbonitrile (1g)



1g

Yield: 82 %, Yellow Solid; **^{1}H NMR (400 MHz, DMSO-d6)** δ 12.45 (s, 1H), 8.93 – 8.89 (m, 1H), 8.34 (dd, *J* = 7.9, 1.1 Hz, 1H), 8.01 – 7.95 (m, 2H), 7.11 (s, 1H) ppm; **^{13}C NMR (100 MHz, DMSO-d6)** δ 161.6, 145.0, 137.6, 132.1, 131.5, 130.6, 129.6, 129.1, 125.8, 125.5, 123.6, 115.6, 115.1, 103.6 ppm.

7a,8,9,10,11,11a-hexahydrobenzo[a]phenazin-5-ol (1h)



1h

Yield: 86 %, Yellow Solid; **^{1}H NMR (400 MHz, DMSO-d6)** δ 8.10 – 8.05 (m, 1H), 8.03 (s, 1H), 7.93 – 7.87 (m, 1H), 7.63 – 7.54 (m, 2H), 5.54 (s, 1H), 3.28 (td, *J* = 11.6, 3.8 Hz, 1H), 2.93 (td, *J* = 11.2, 3.8 Hz, 1H), 2.33 (d, *J* = 5.6 Hz, 1H), 1.93 (d, *J* = 12.1 Hz, 1H), 1.77 (dd, *J* = 29.7, 7.5 Hz, 2H), 1.51 – 1.26 (m, 4H) ppm; **^{13}C NMR (100 MHz, DMSO-d6)** δ 181.8, 153.0, 146.0, 133.2, 132.3, 131.5, 131.1, 125.1, 123.9, 100.06, 62.8, 52.5, 32.3, 30.7, 25.4, 24.0 ppm.

20. Copies of ^{1}H and ^{13}C NMR spectrum of synthesized compounds (1a-h)

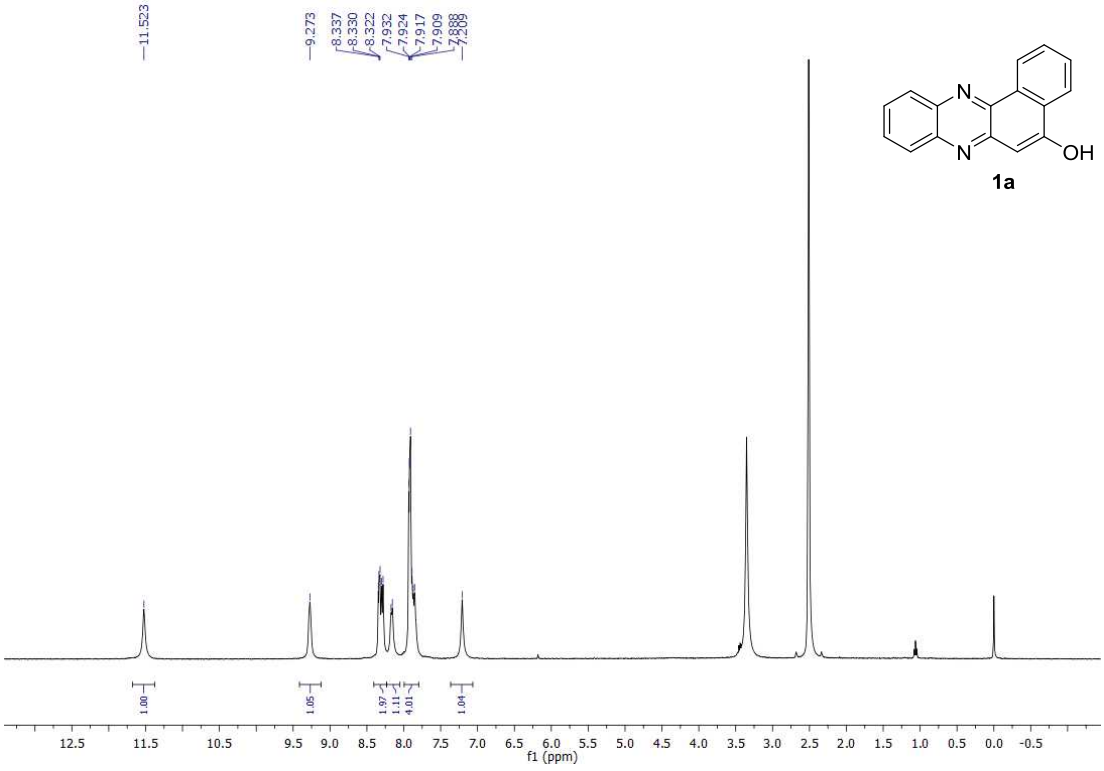


Figure S36. *^{1}H NMR spectrum of synthesized compound 1a in DMSO-d_6*

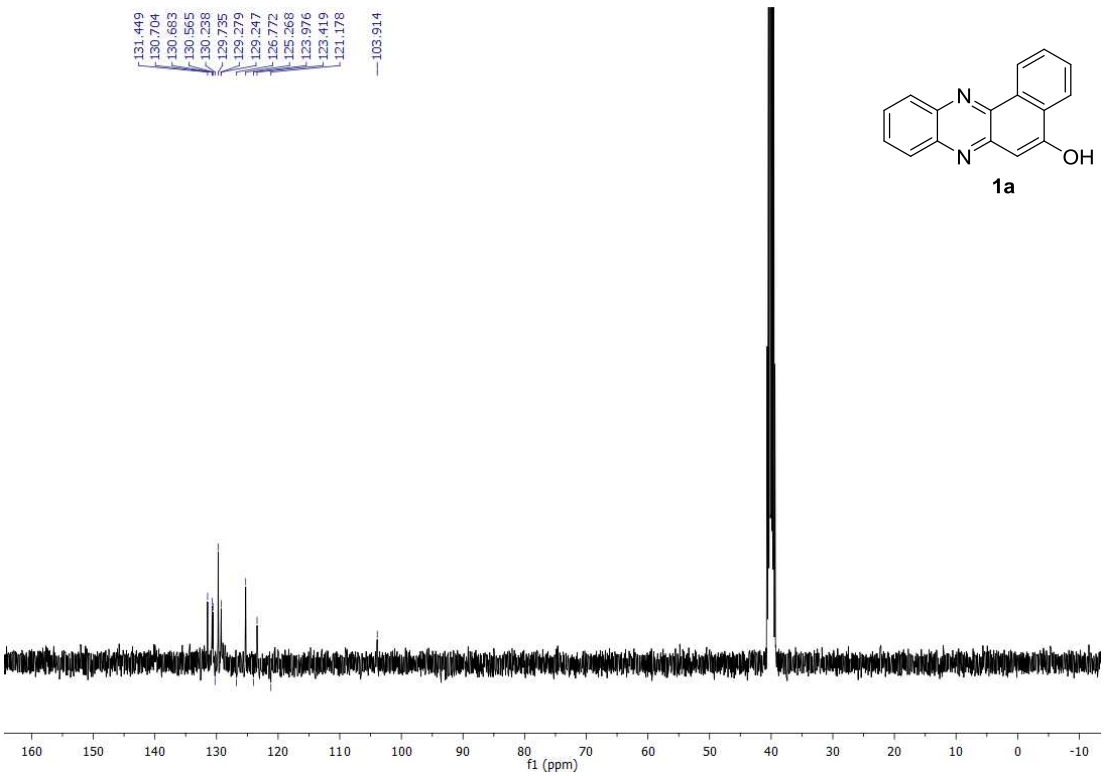


Figure S37. *^{13}C NMR spectrum of synthesized compound 1a in DMSO-d_6*

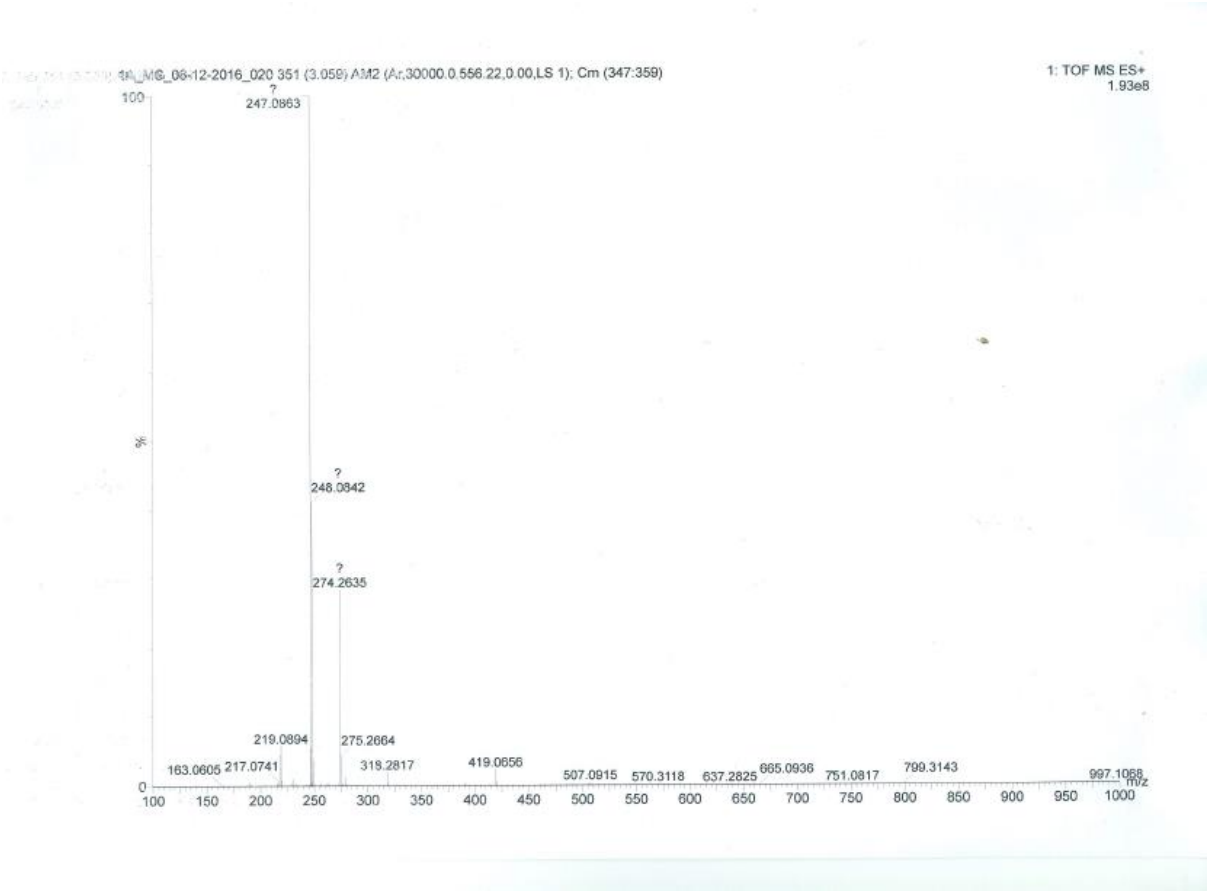


Figure S38. *HRMS spectrum of synthesized compound 1a*

Figure S39. *HRMS spectrum of synthesized compound 1b*

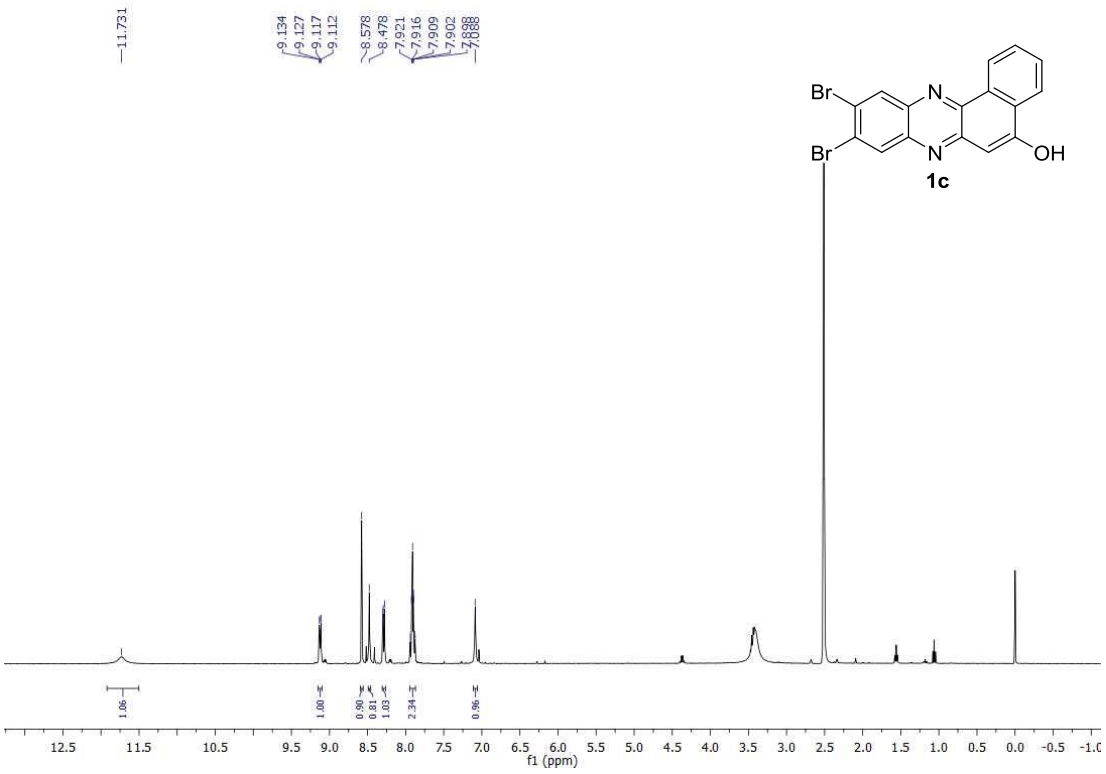


Figure S40. *^{1}H NMR spectrum of synthesized compound 1c in DMSO-d_6*

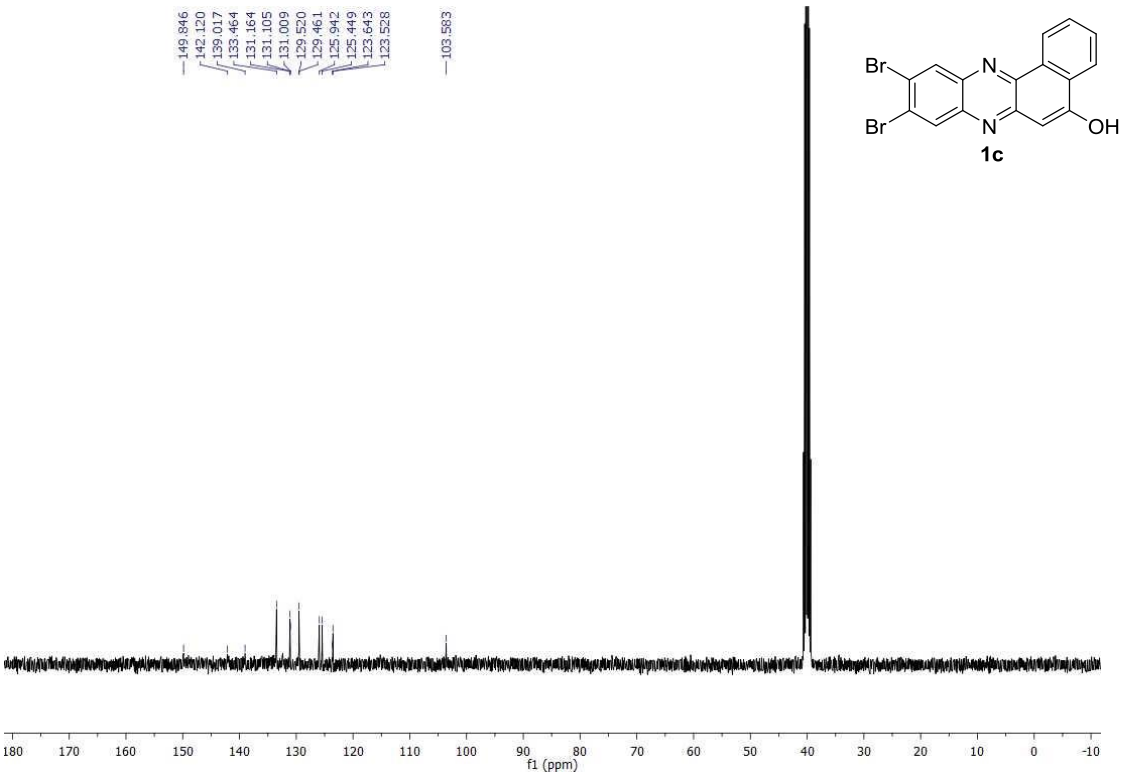


Figure S41. *^{13}C NMR spectrum of synthesized compound 1c in DMSO-d_6*

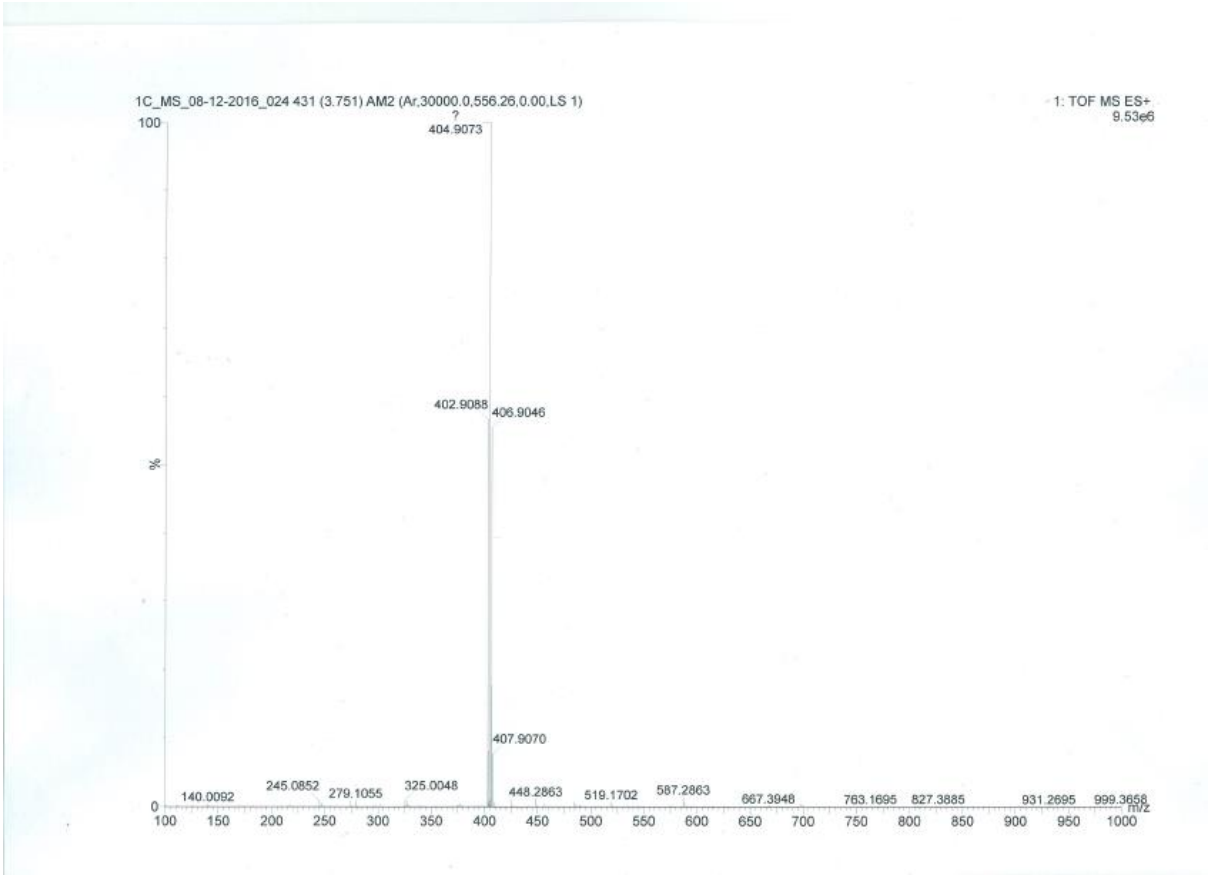


Figure S42. *HRMS spectrum of synthesized compound 1c*

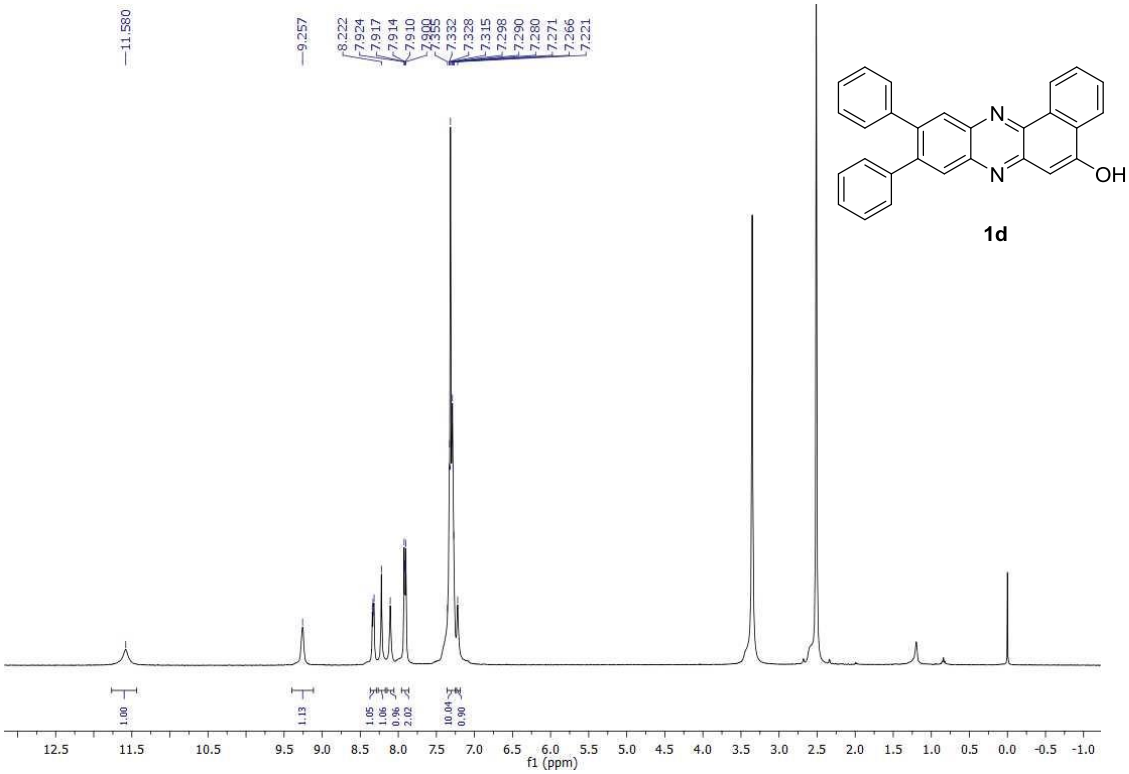


Figure S43. *^{1}H NMR spectrum of synthesized compound 1d in DMSO-d_6*

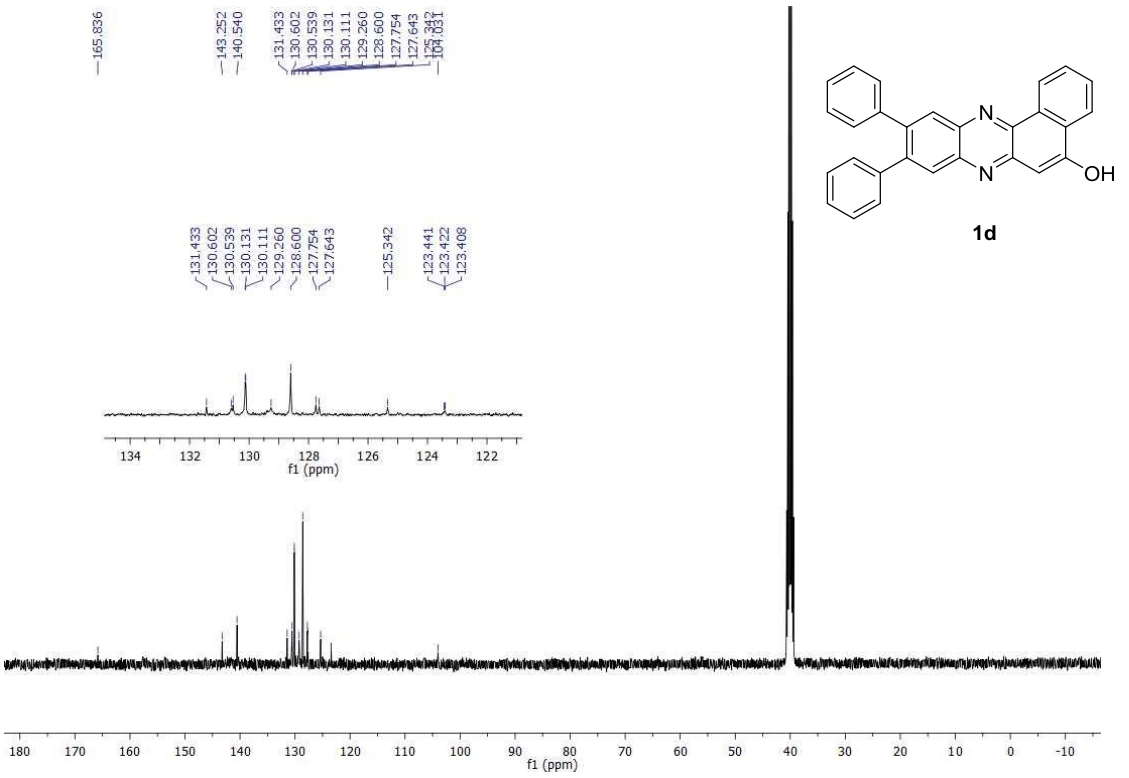


Figure S44. *^{13}C NMR spectrum of synthesized compound 1d in DMSO-d_6*

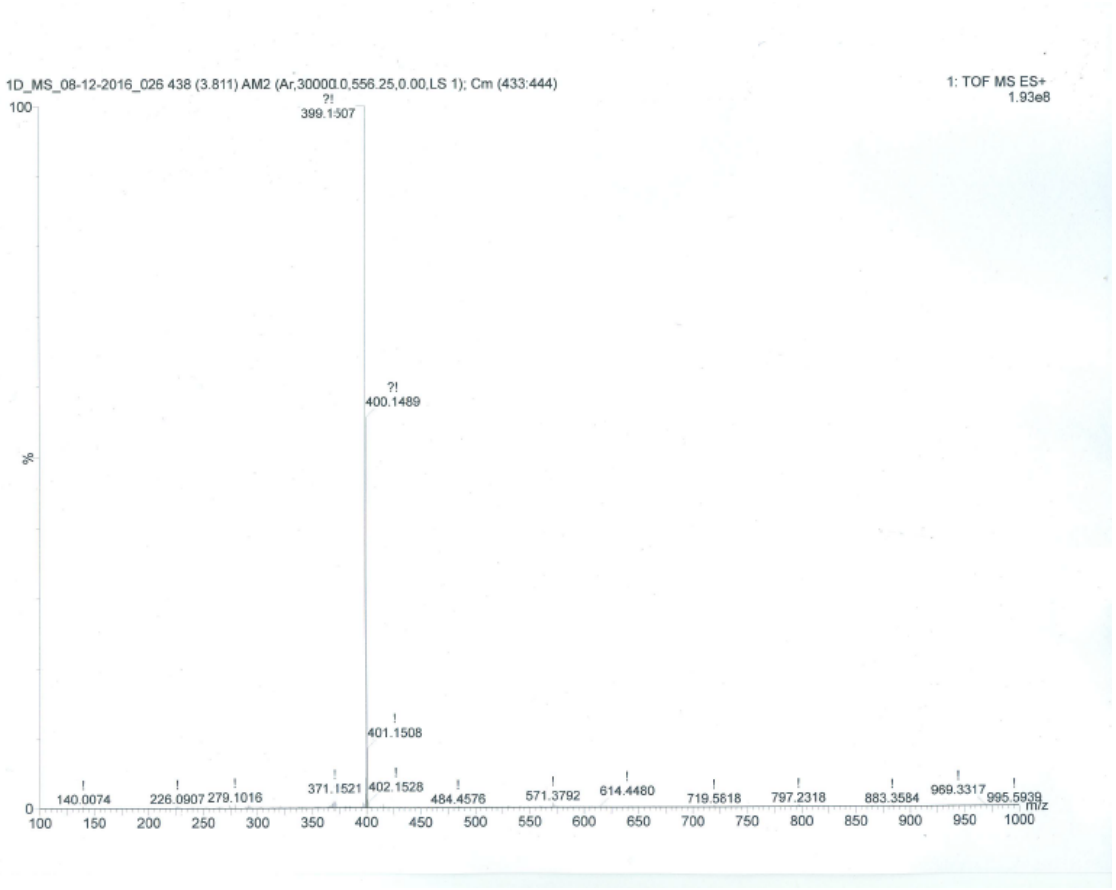


Figure S45. *HRMS spectrum of synthesized compound 1d*

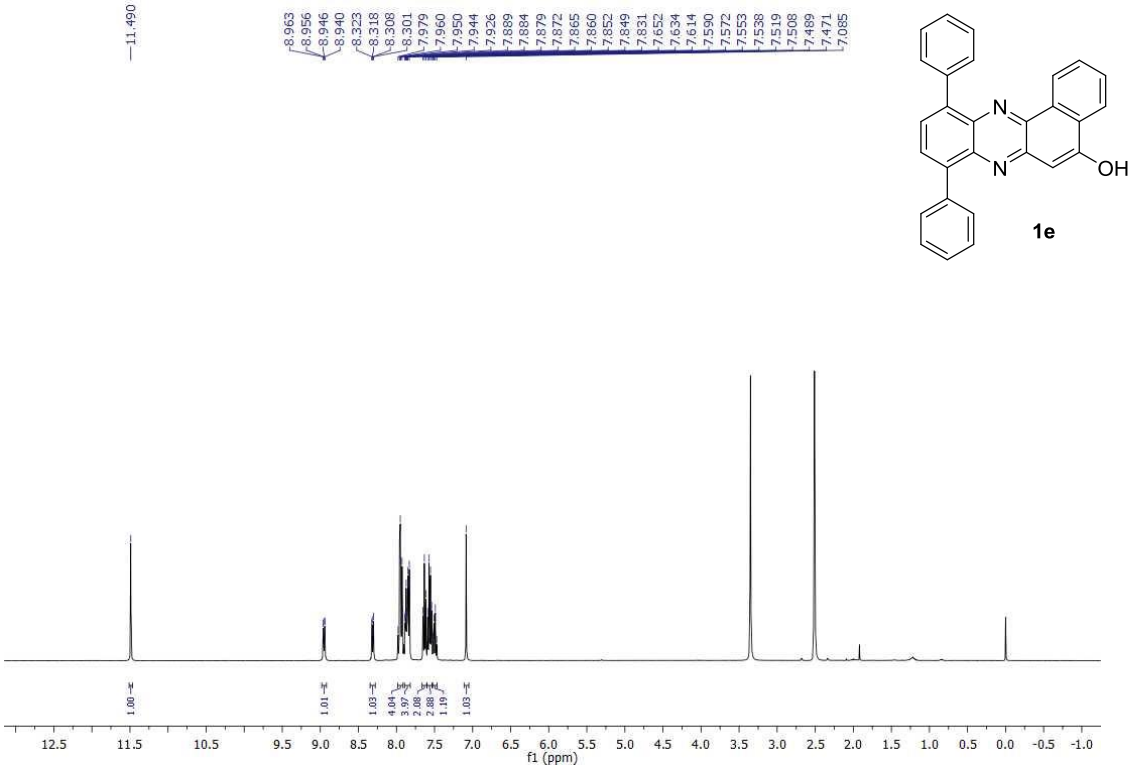


Figure S46. *^{1}H NMR spectrum of synthesized compound 1e in DMSO-d_6*

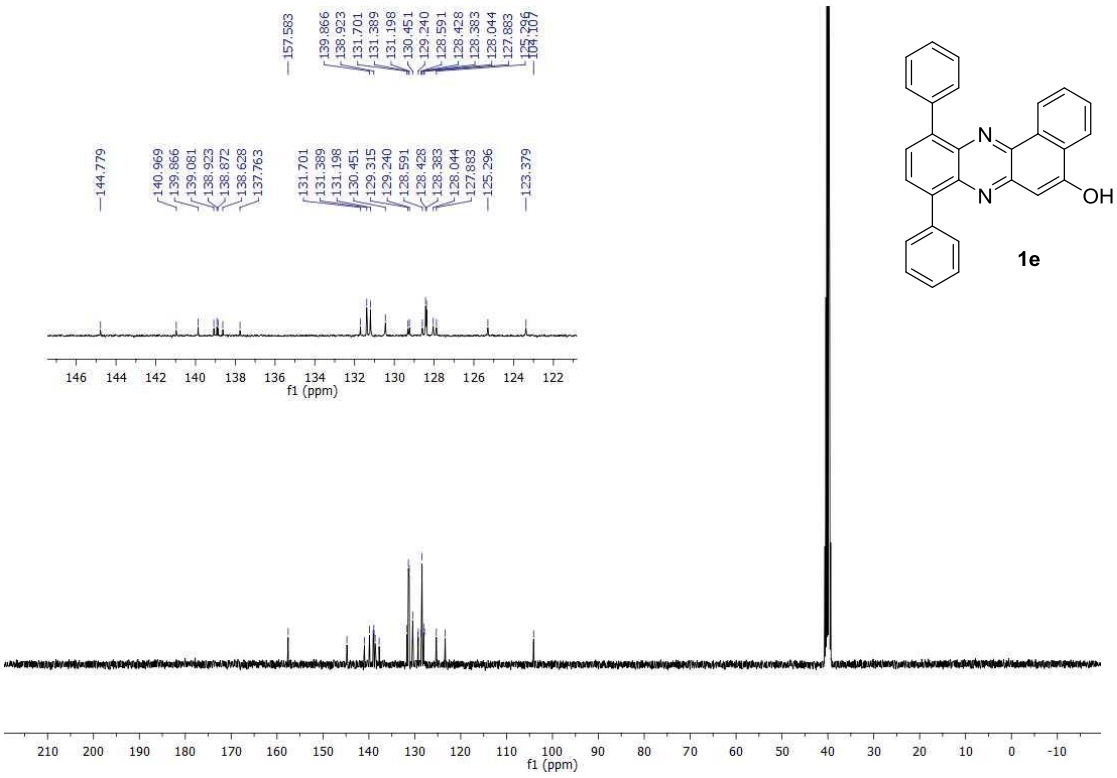


Figure S47. *^{13}C NMR spectrum of synthesized compound 1e in DMSO-d_6*

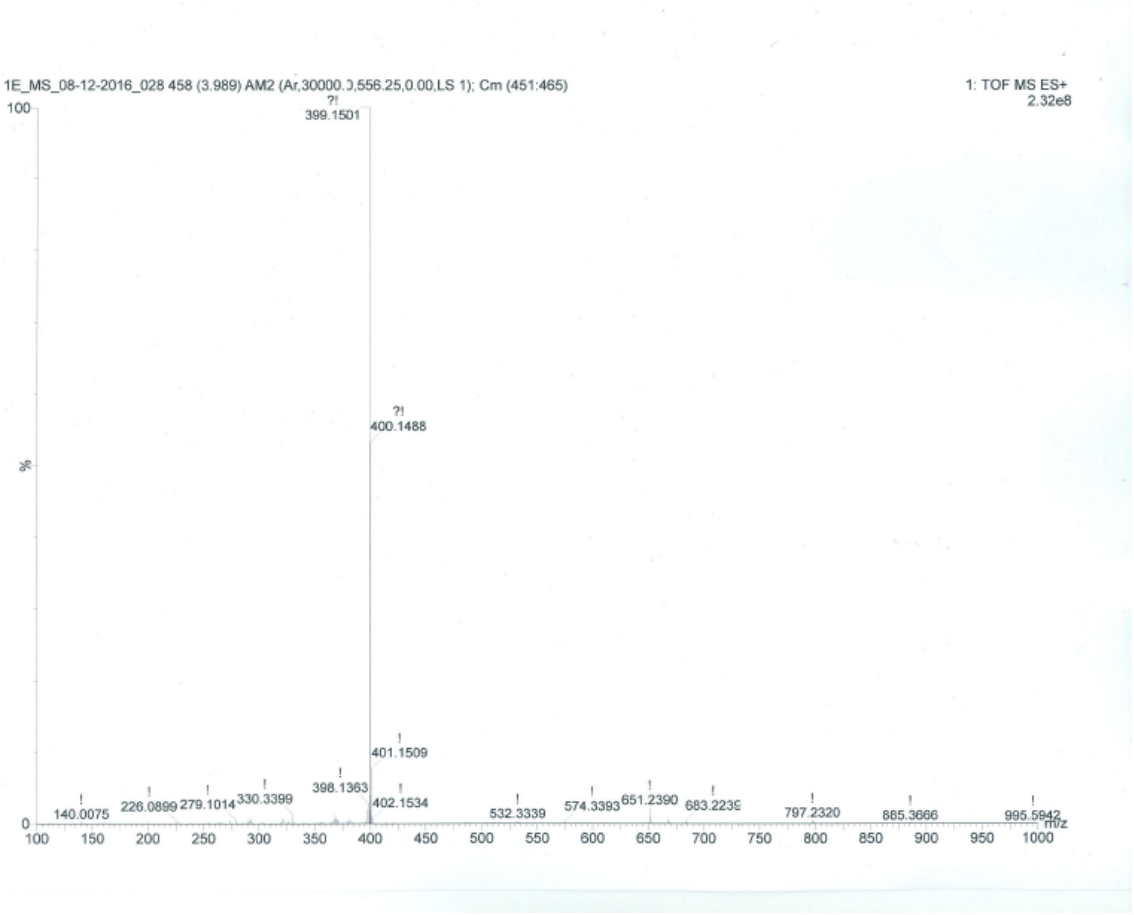


Figure S48. *HRMS spectrum of synthesized compound 1e*

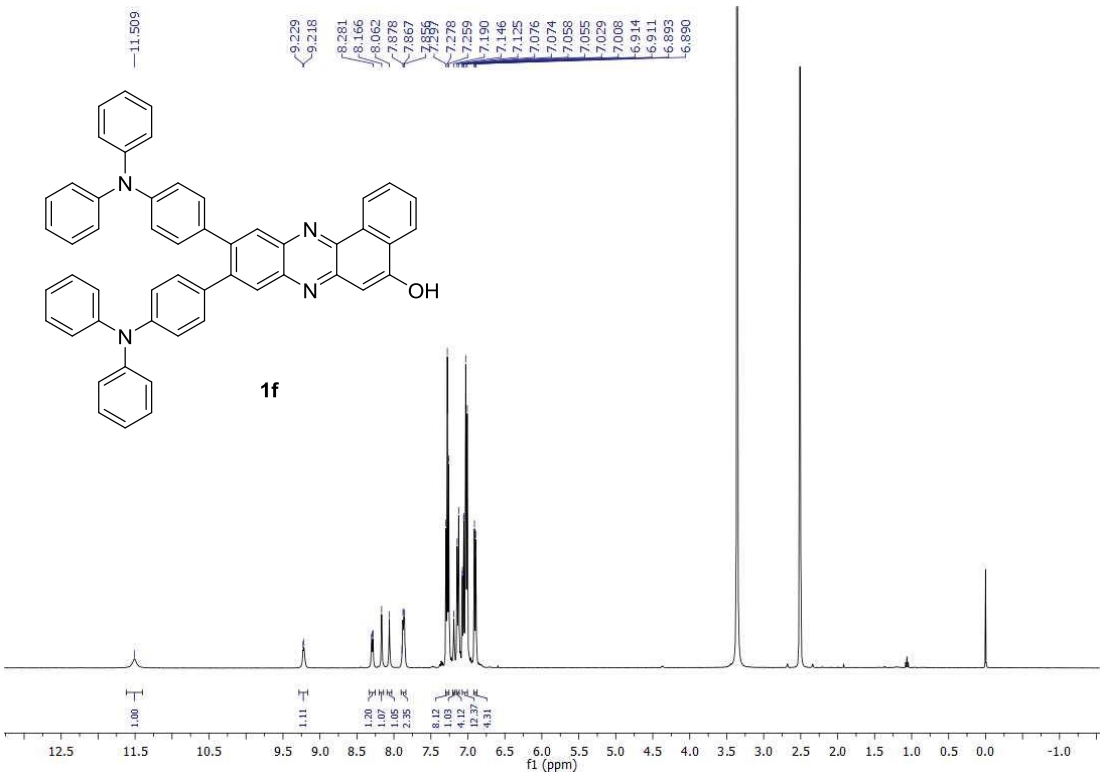


Figure S49. *[1]H NMR spectrum of synthesized compound 1f in DMSO-d_6*

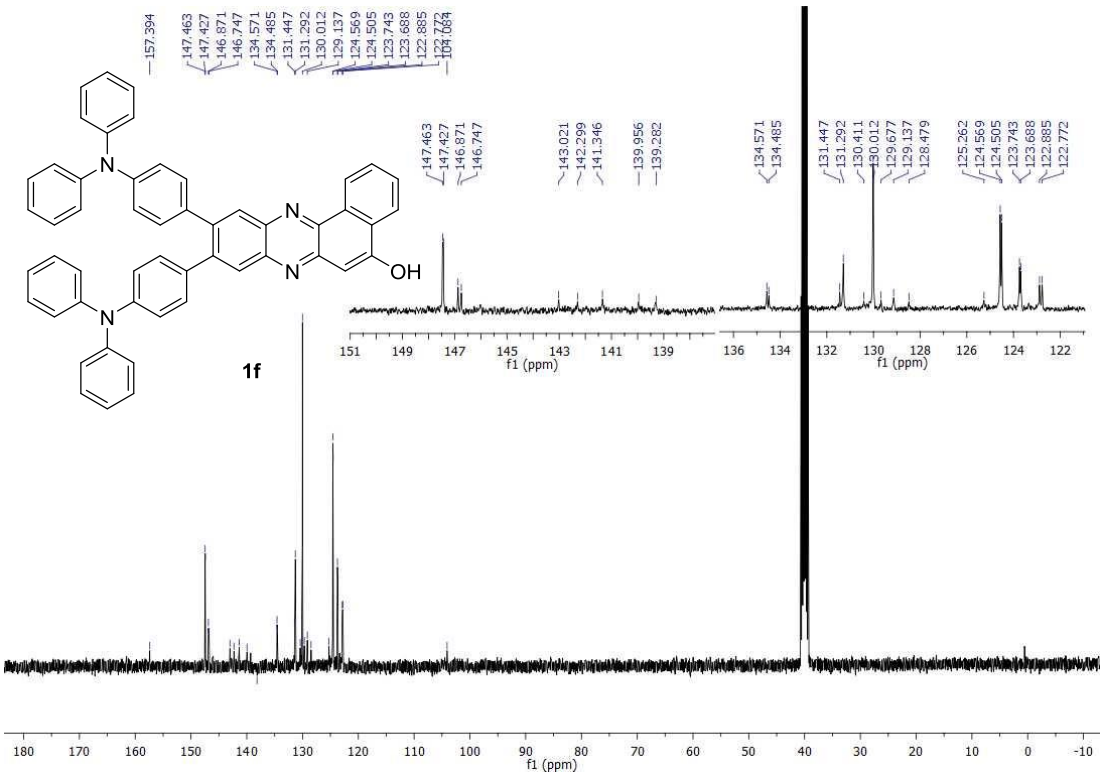


Figure S50. *[13]C NMR spectrum of synthesized compound 1f in DMSO-d_6*

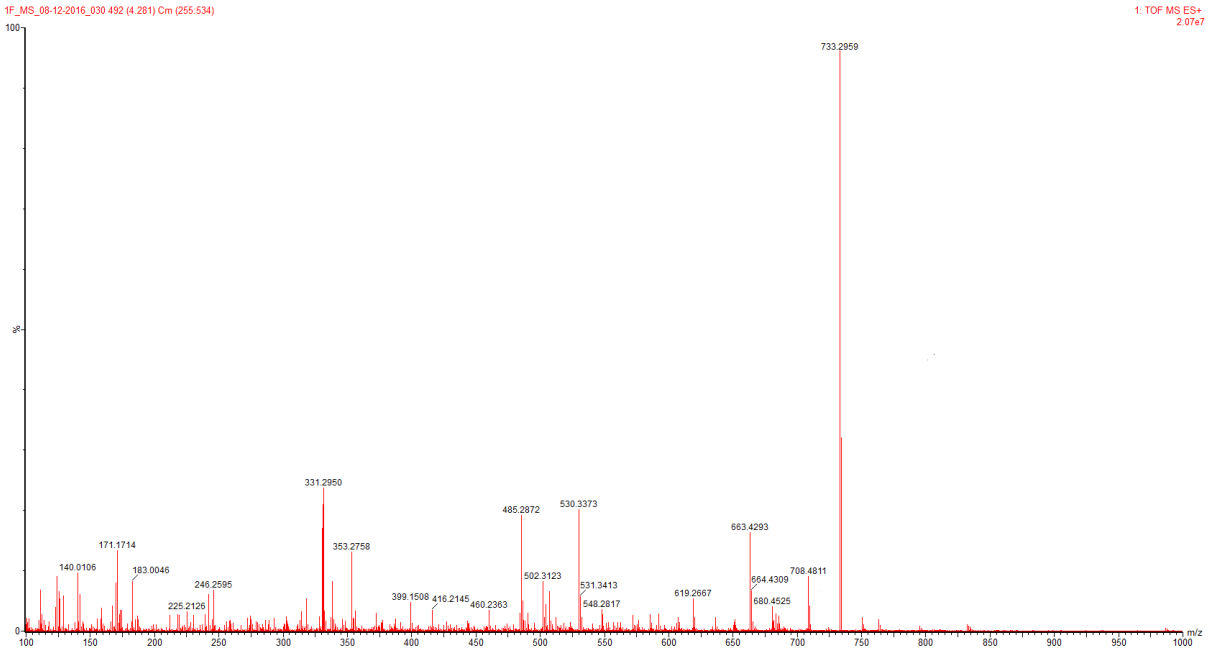


Figure S51. *HRMS spectrum of synthesized compound 1f*

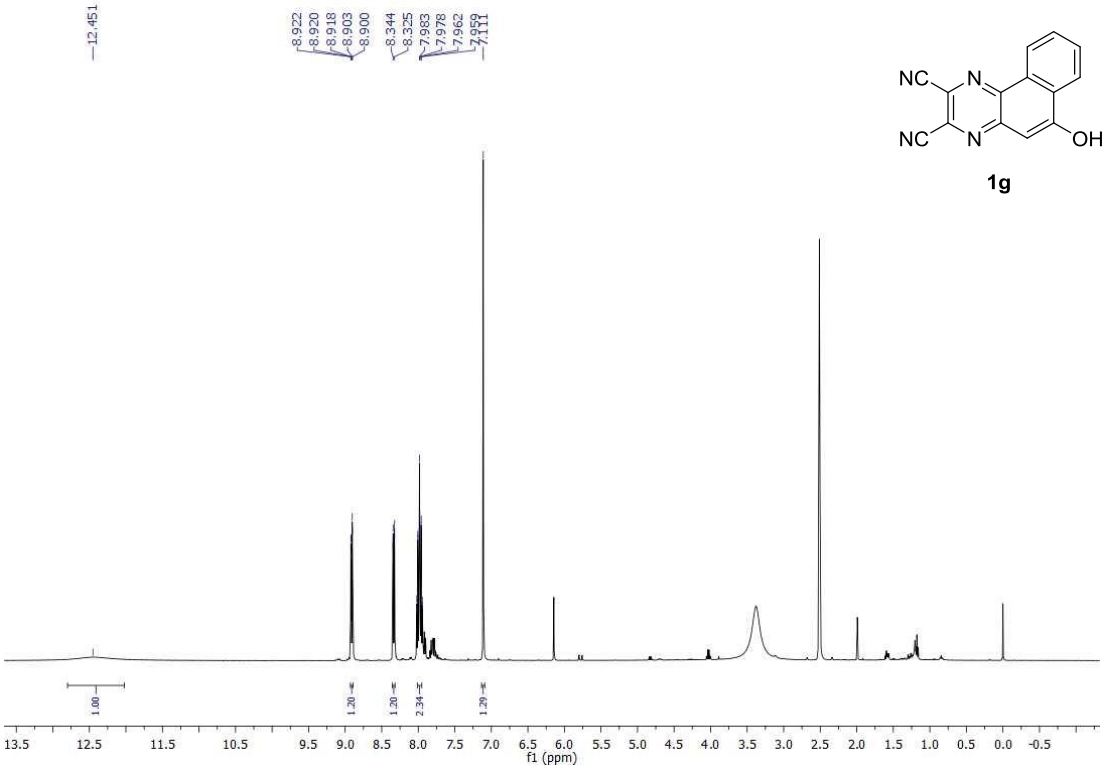


Figure S52. *[1]H NMR spectrum of synthesized compound 1g in DMSO-d_6*

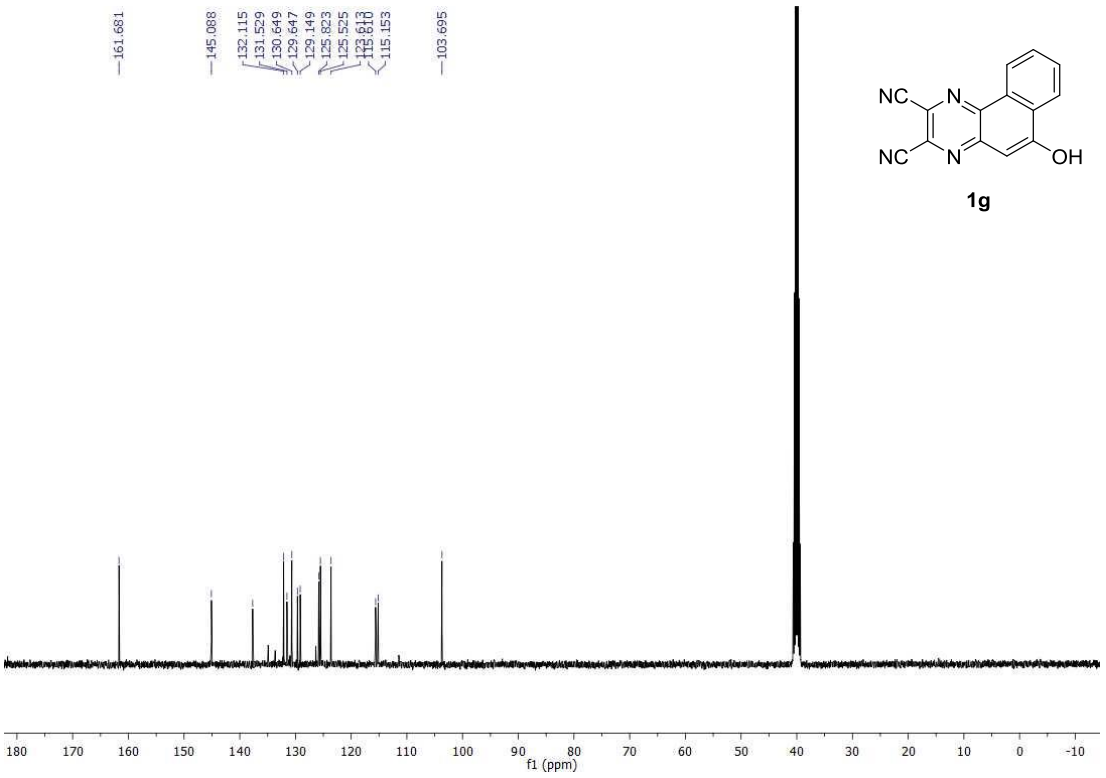


Figure S53. *[13]C NMR spectrum of synthesized compound 1g in DMSO-d_6*

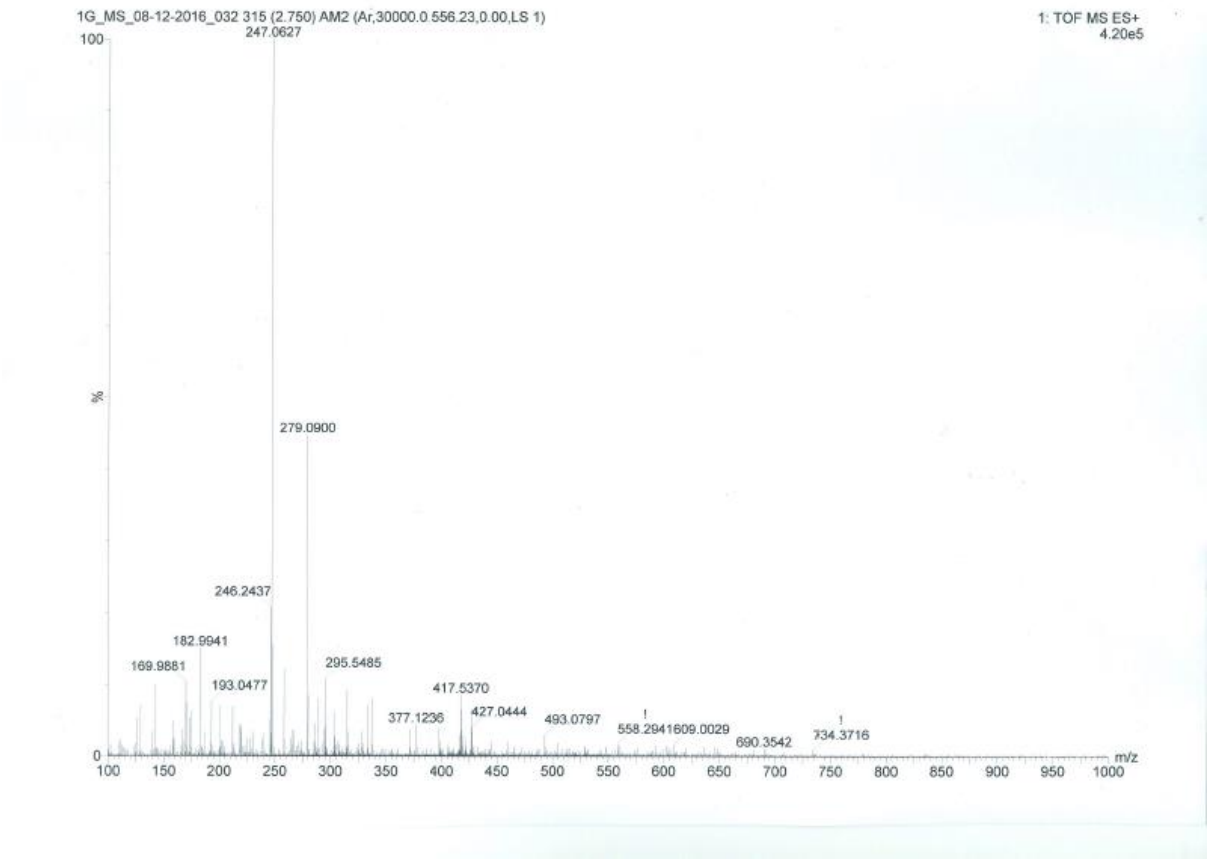


Figure S54. *HRMS spectrum of synthesized compound 1g*

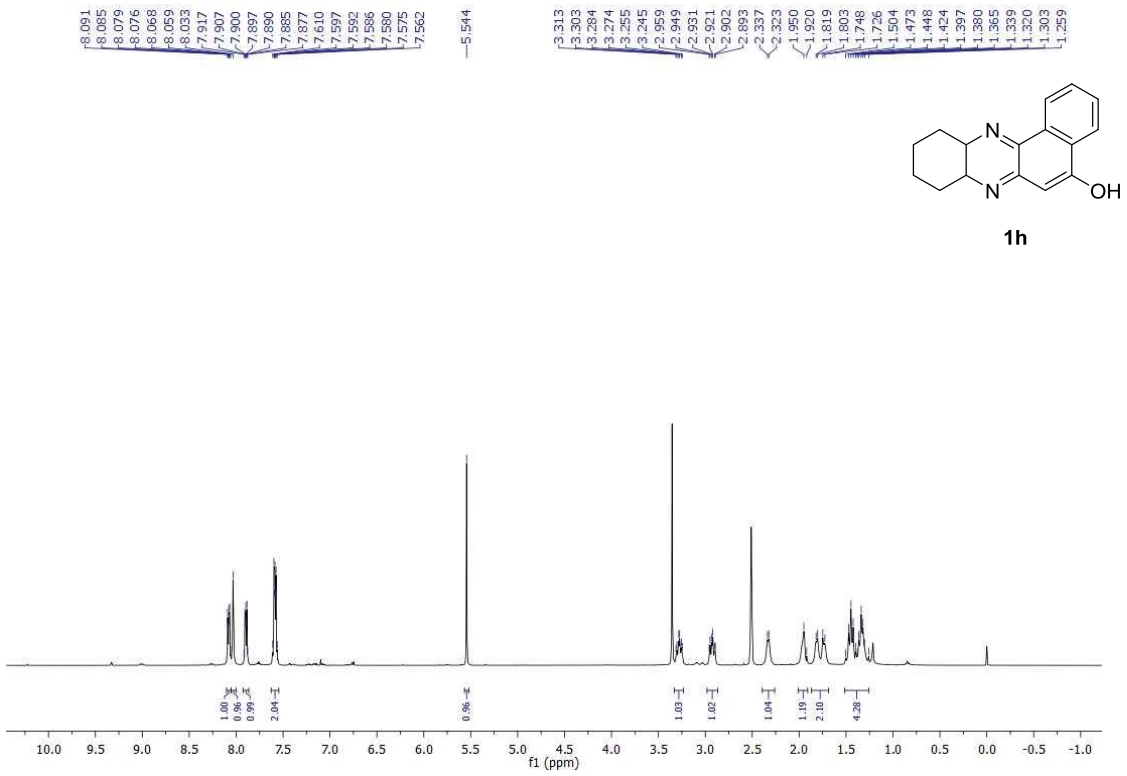


Figure S55. *^{1}H NMR spectrum of synthesized compound 1h in DMSO-d_6*

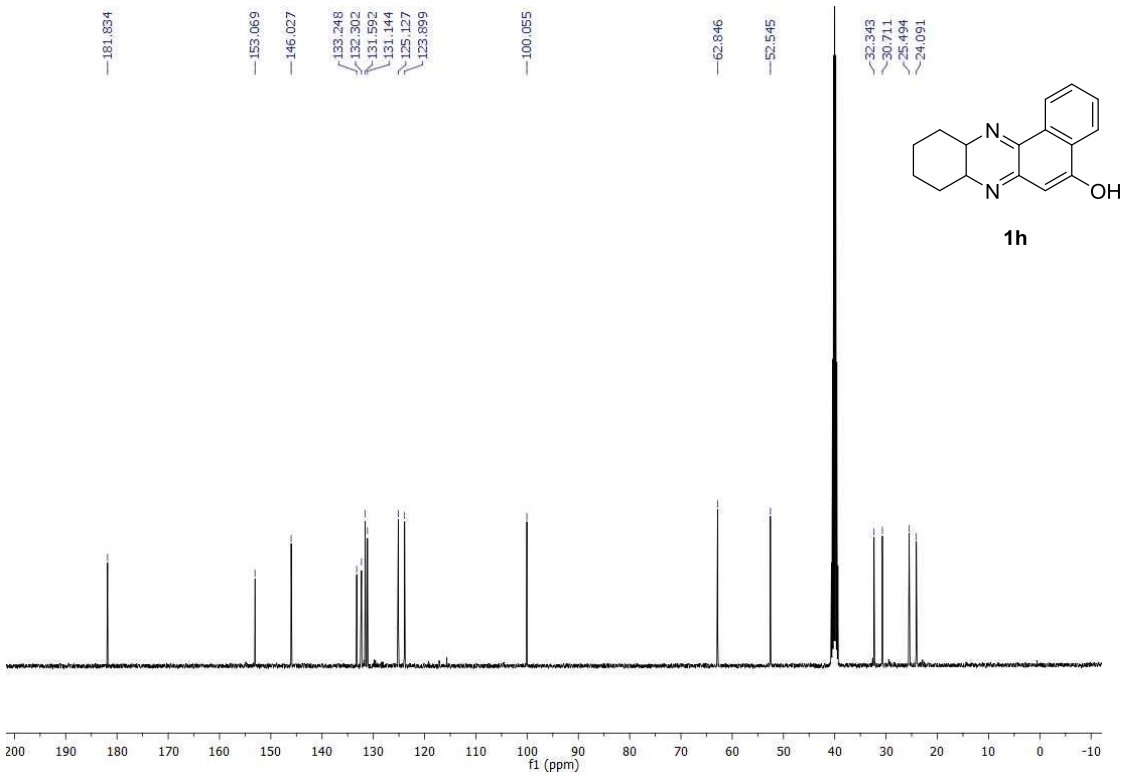


Figure S56. *^{13}C NMR spectrum of synthesized compound 1h in DMSO-d_6*

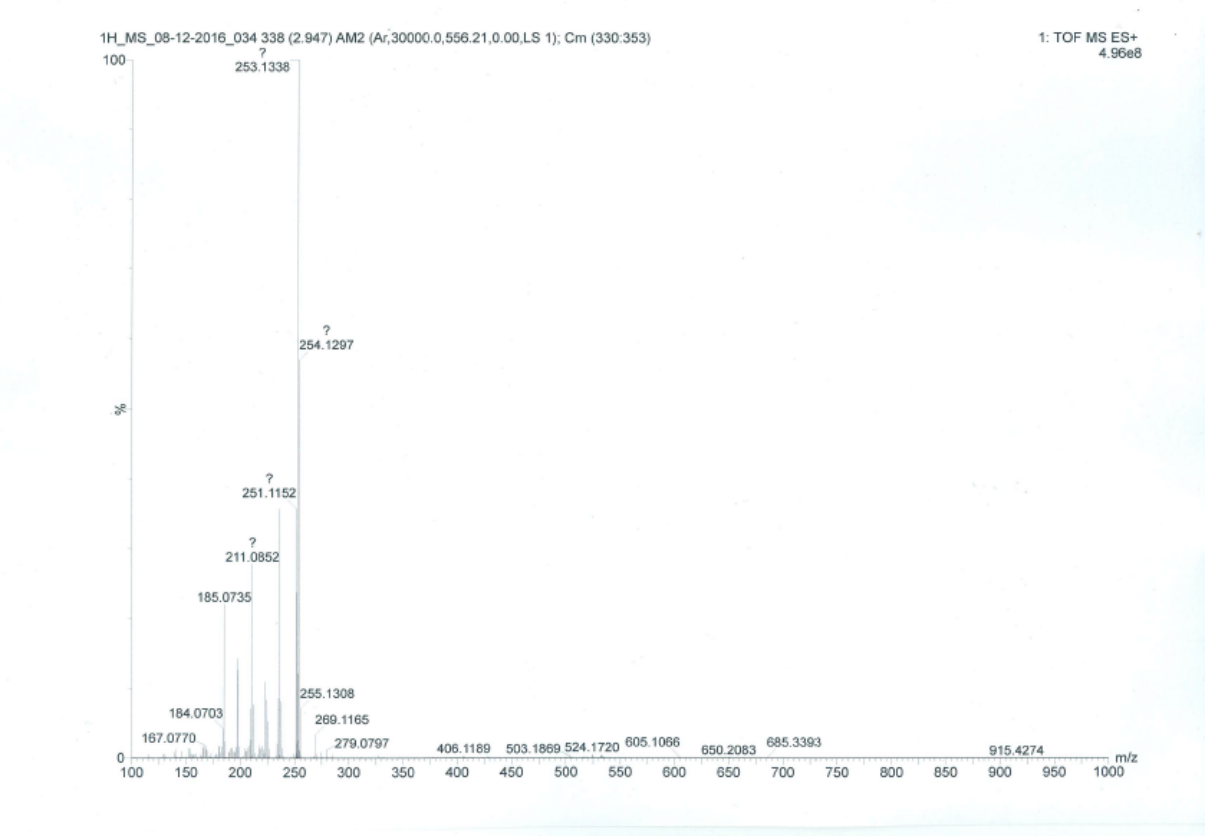


Figure S57. *HRMS spectrum of synthesized compound 1h*

21. Copies of ^{1}H, ^{13}C NMR and HRMS spectrum of synthesized compound (3a-s)

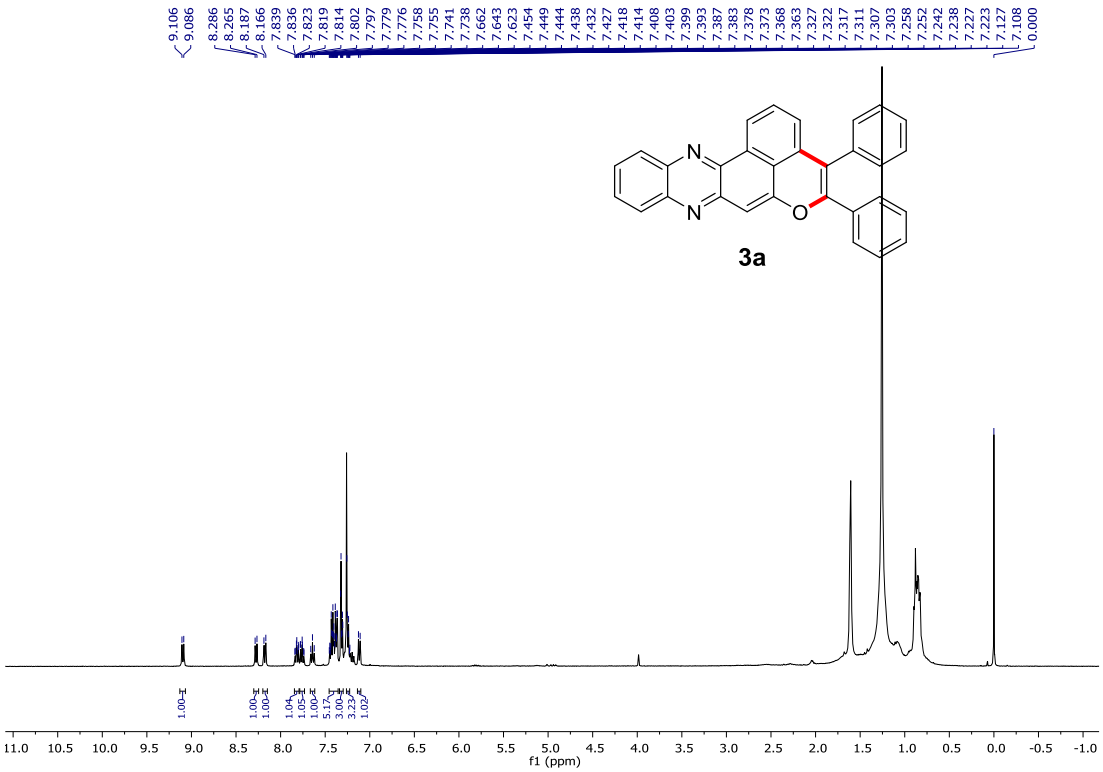


Figure S58. *^{1}H NMR spectrum of synthesized compound 3a in $CDCl_3$*

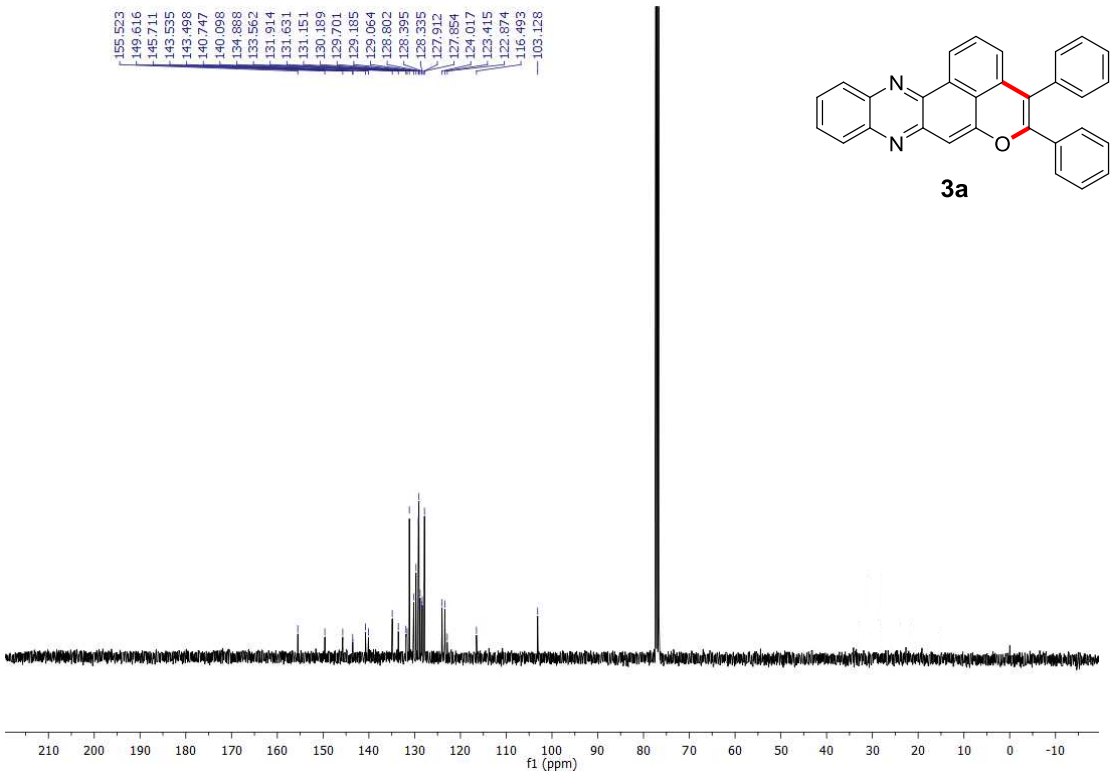


Figure S59. *^{13}C NMR spectrum of synthesized compound 3a in $CDCl_3$*

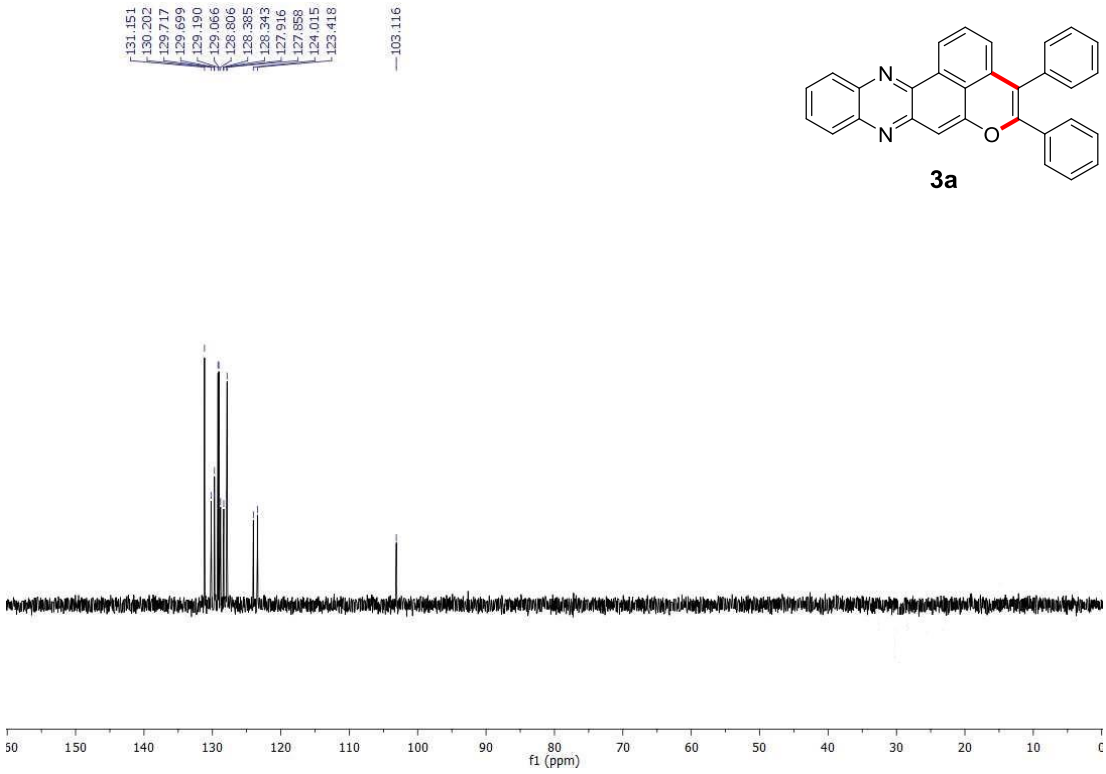


Figure S60. *DEPT-135 NMR spectrum of synthesized compound 3a in* $CDCl_3$

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Analysis Info

Analysis Name	D:\Data\2015\DrNagarajan\DEC\pps-F11.d	Acquisition Date	12/15/2015 11:37:45 AM
Method	TL-P.m	Operator	Ramu Sridhar
Sample Name	PPS-F11-ACN	Instrument	maXis 10138
Comment			

Acquisition Parameter

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Scan End	600 m/z	Set Collision Cell RF	350.0 Vpp	Set Divert Valve	Waste

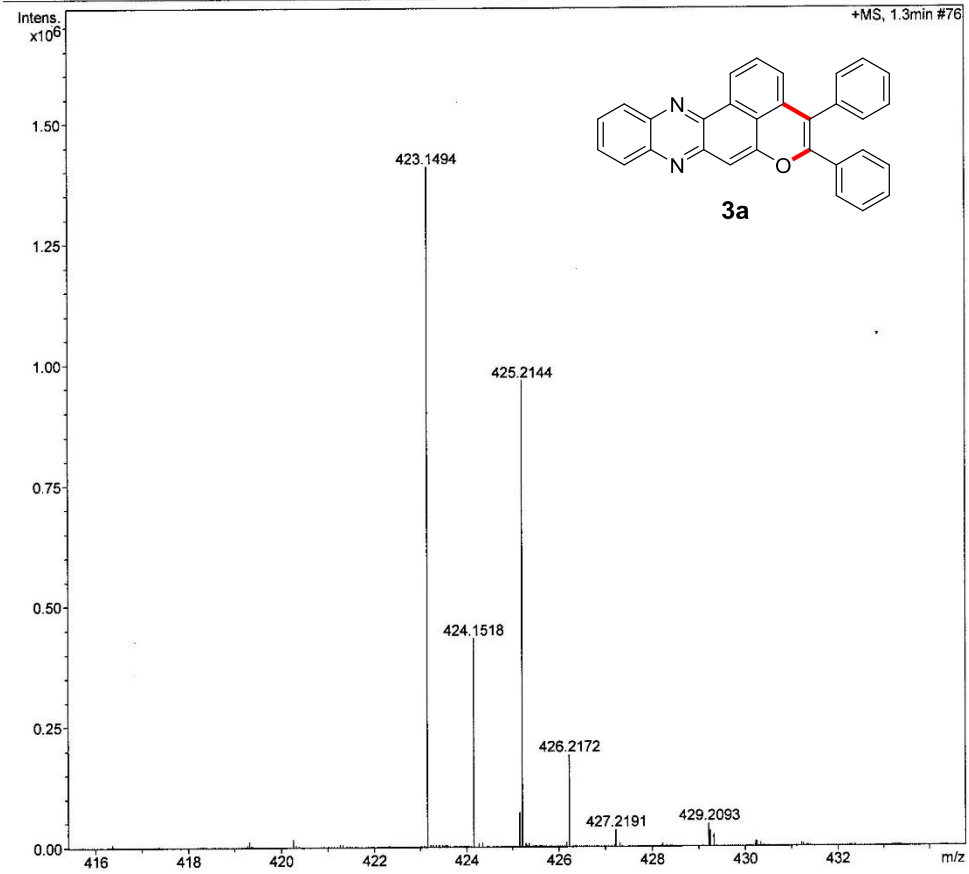

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Figure S61. *HRMS spectrum of synthesized compound 3a*

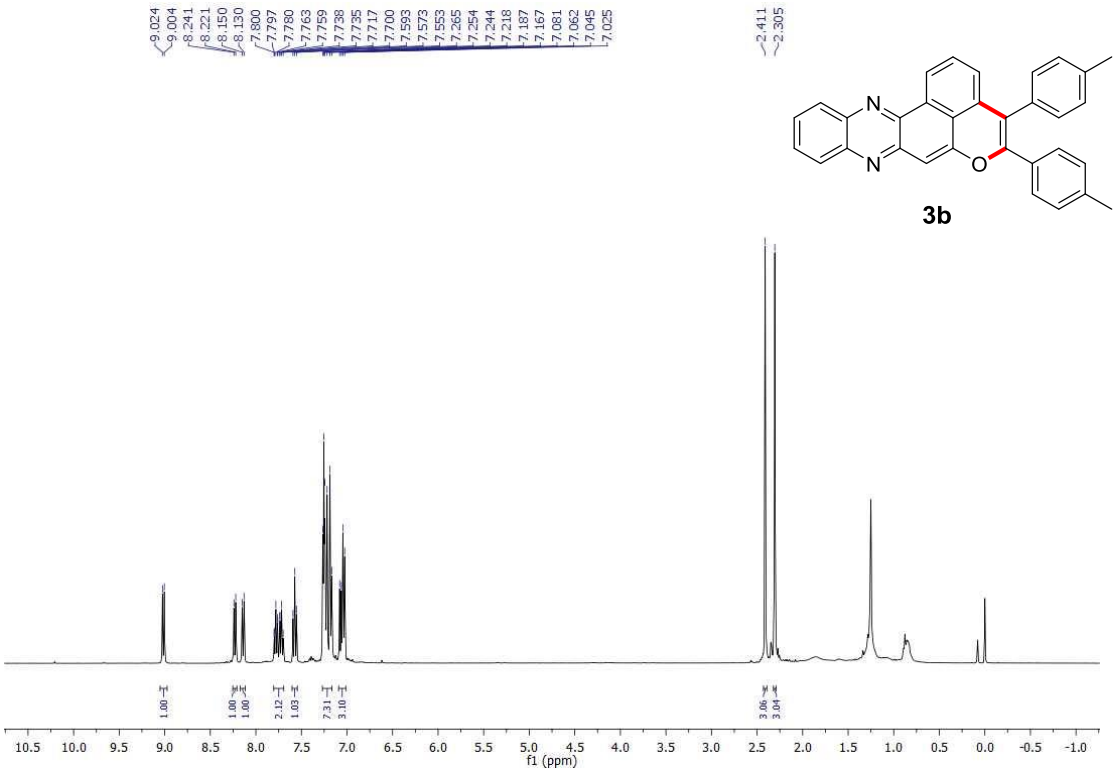

Figure S62. *^{1}H NMR spectrum of synthesized compound 3b in $CDCl_3$*

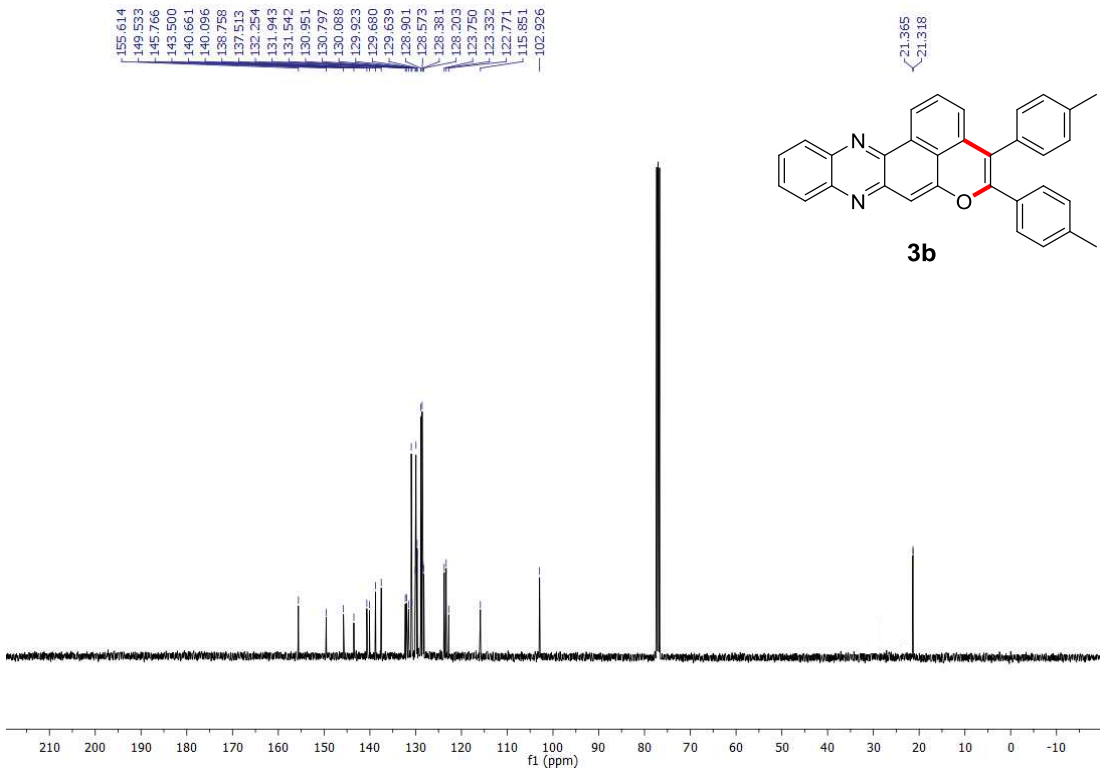

Figure S63. *^{13}C NMR spectrum of synthesized compound 3b in $CDCl_3$*

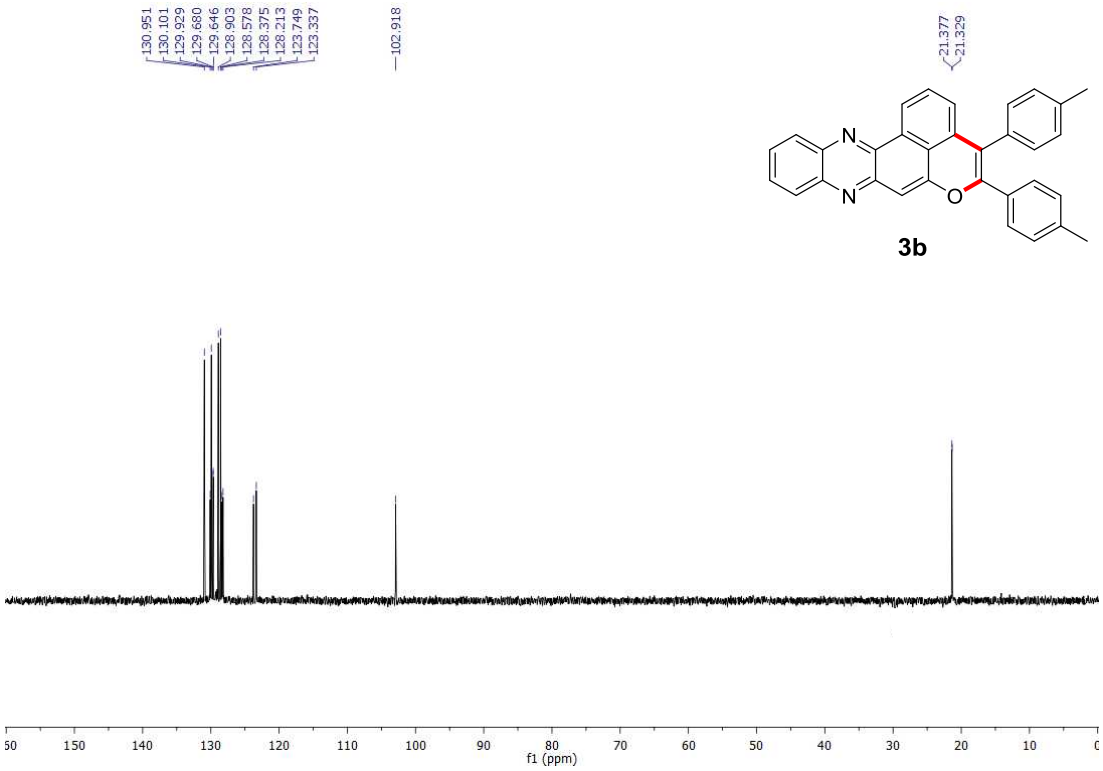

Figure S64. *DEPT-135 NMR spectrum of synthesized compound 3b in* $CDCl_3$

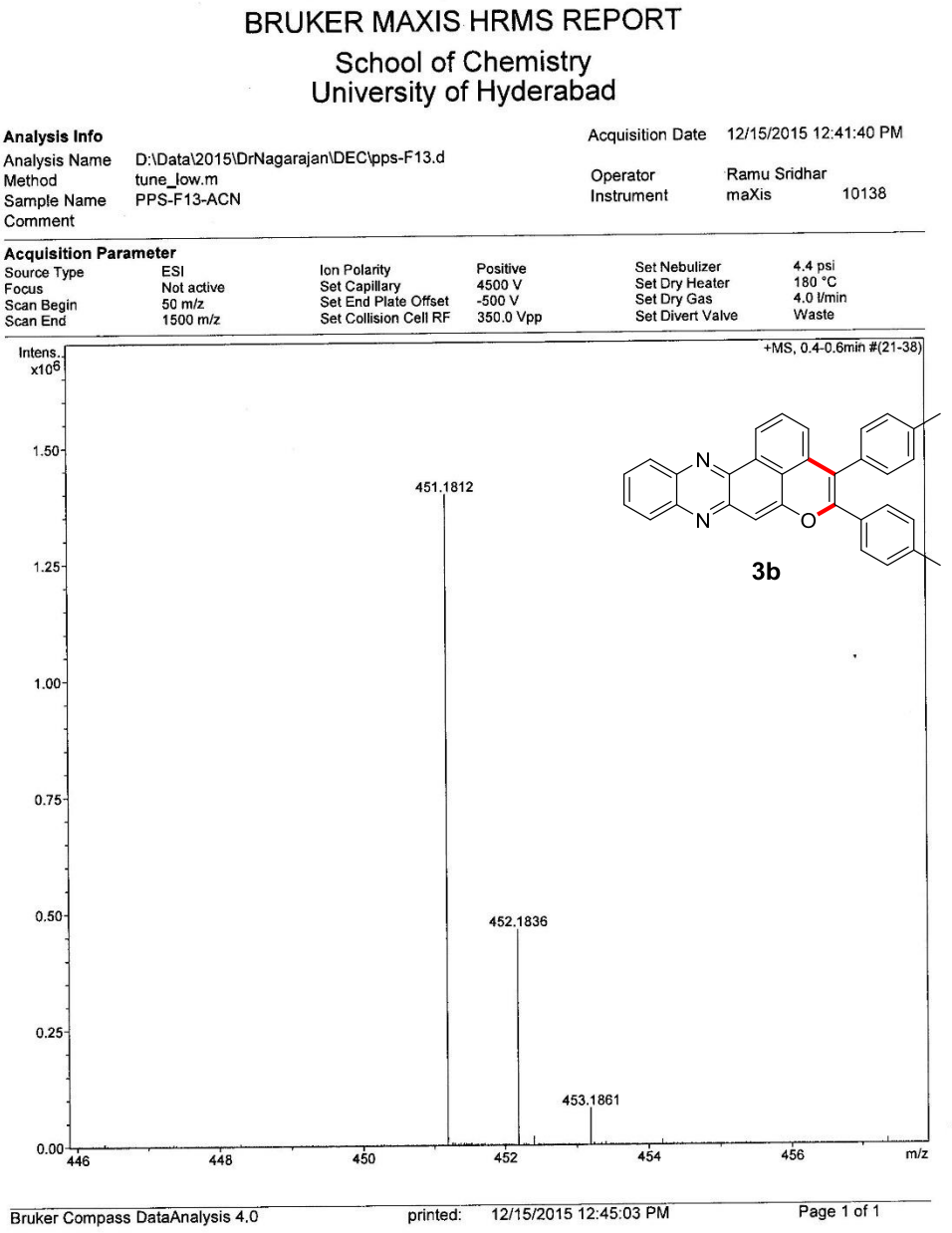

Figure S65. *HRMS spectrum of synthesized compound 3b*

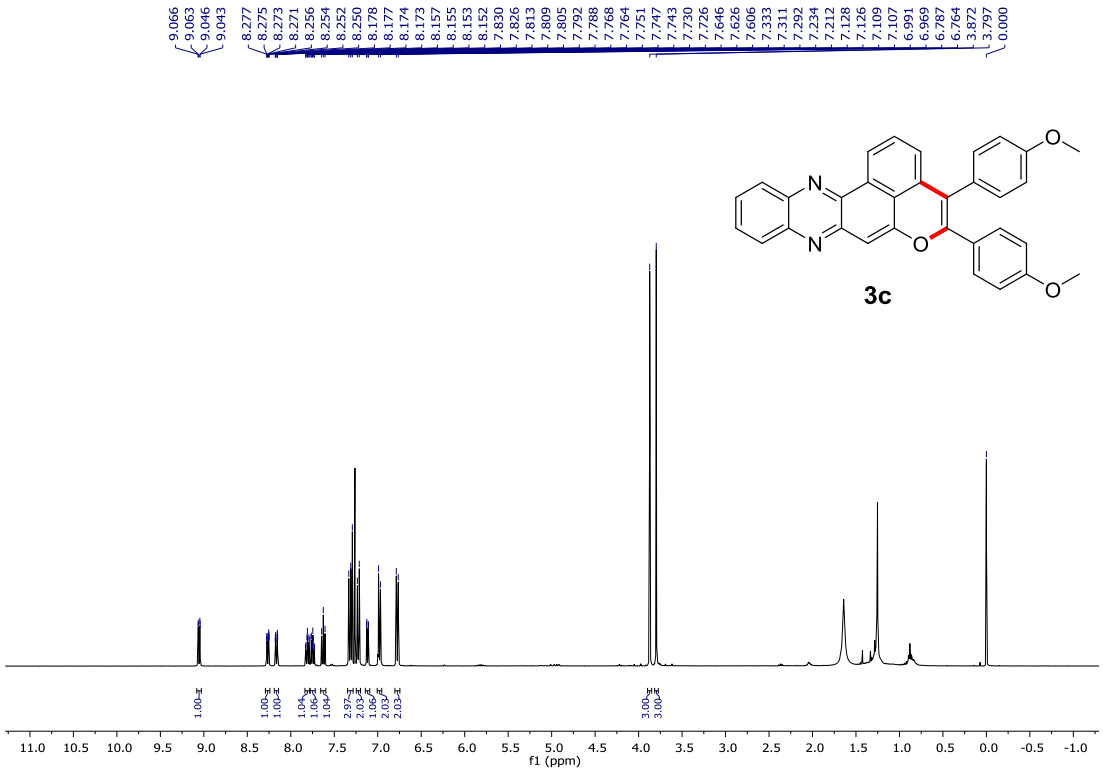

Figure S66. *^{1}H NMR spectrum of synthesized compound 3c in $CDCl_3$*

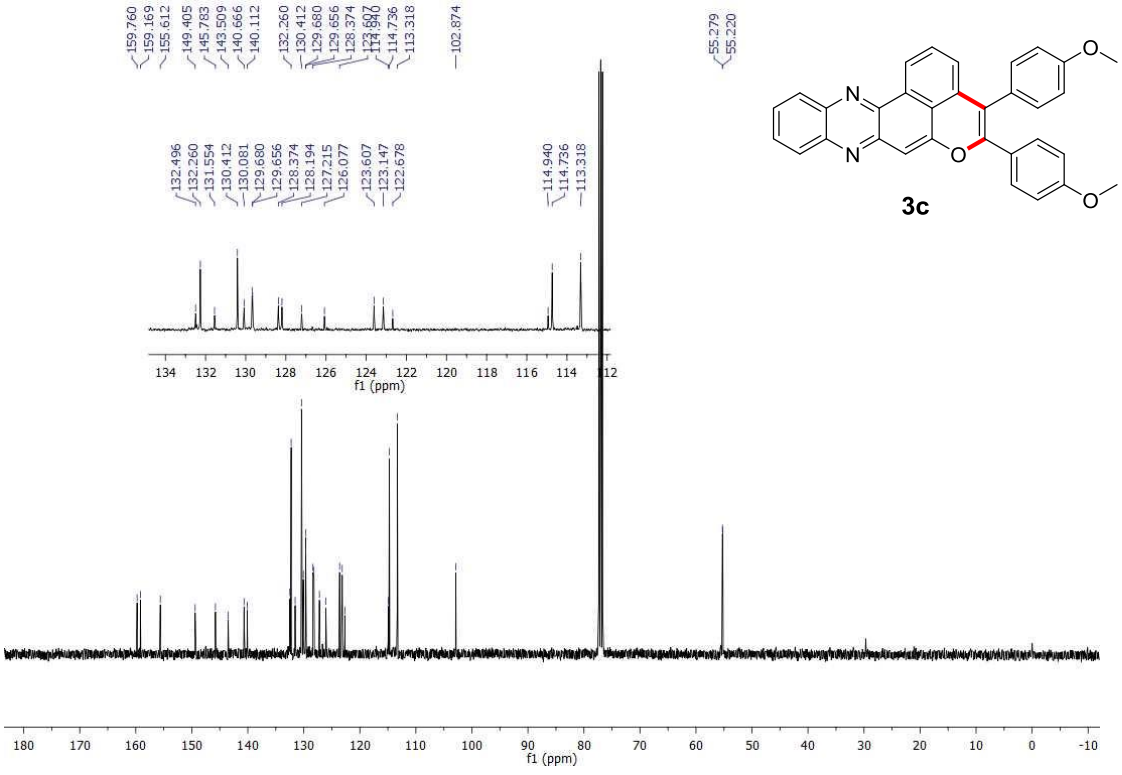

Figure S67. *^{13}C NMR spectrum of synthesized compound 3c in $CDCl_3$*

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Analysis Info

Analysis Name	D:\Data\2015\DrNagarajan\DEC\pps-F12.d	Acquisition Date	12/15/2015 12:50:35 PM
Method	tune_low.m	Operator	Ramu Sridhar
Sample Name	PPS-F12-ACN	Instrument	maXis 10138
Comment			

Acquisition Parameter

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Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1500 m/z	Set Collision Cell RF	350.0 Vpp	Set Divert Valve	Waste

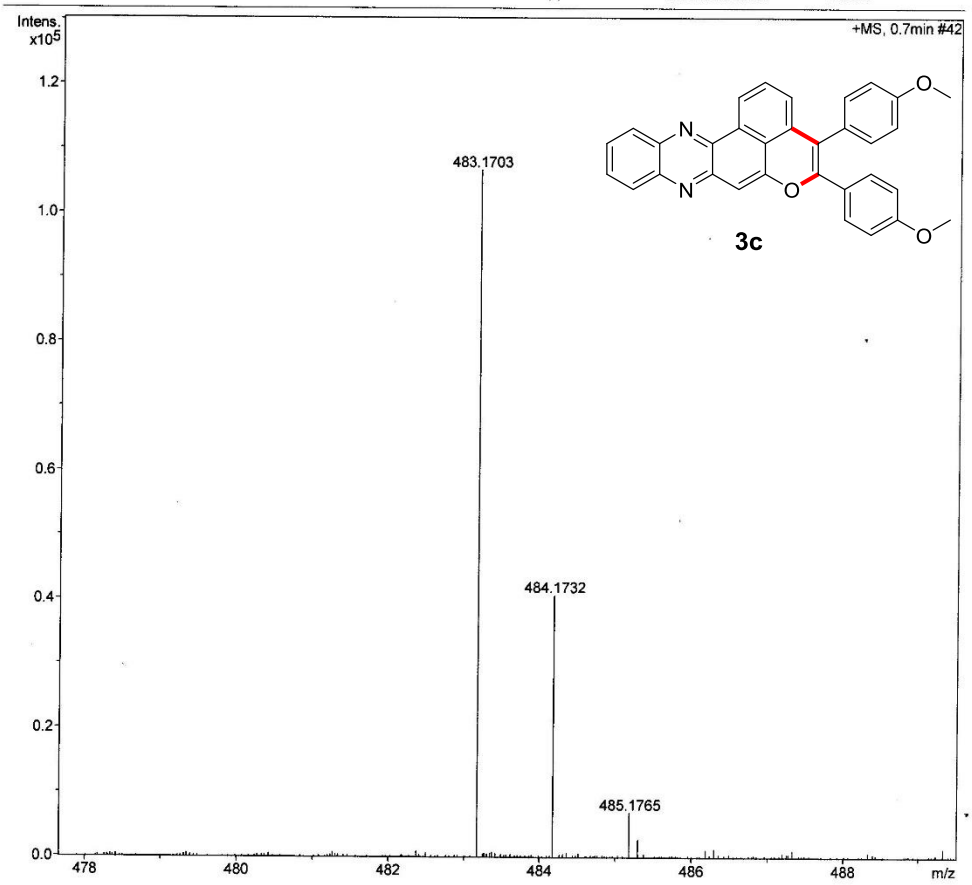

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Figure S68. *HRMS spectrum of synthesized compound 3c*

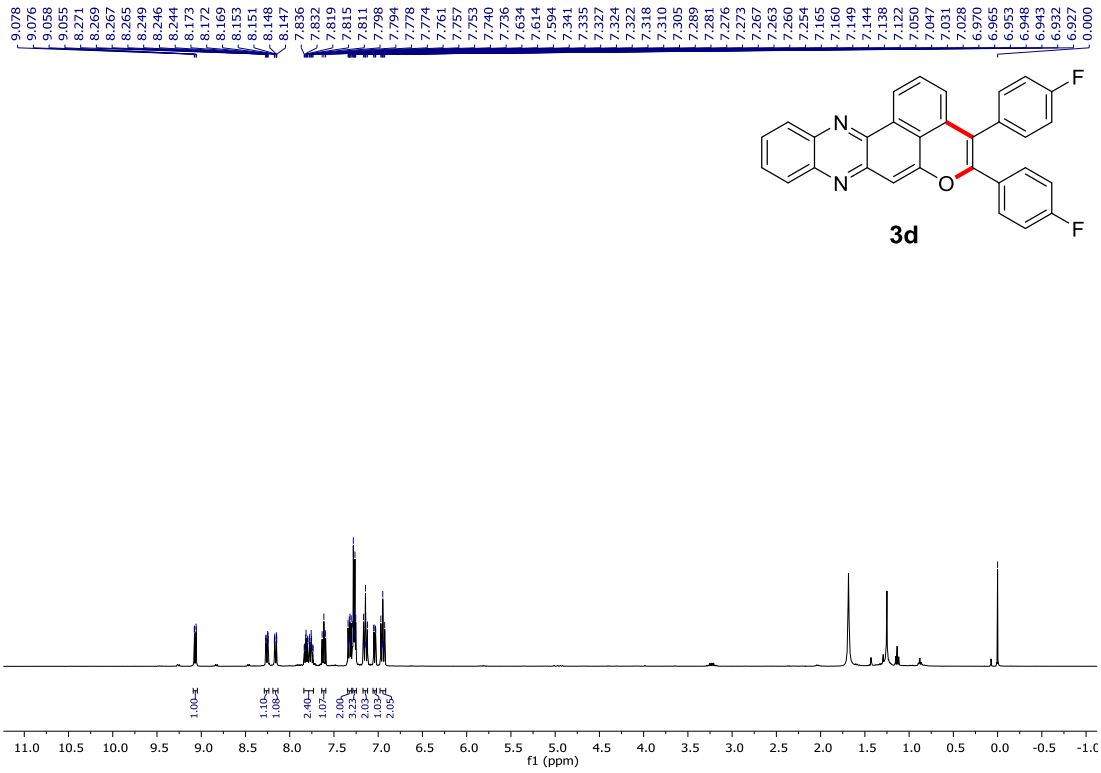

Figure S69. *^{1}H NMR spectrum of synthesized compound 3d in $CDCl_3$*

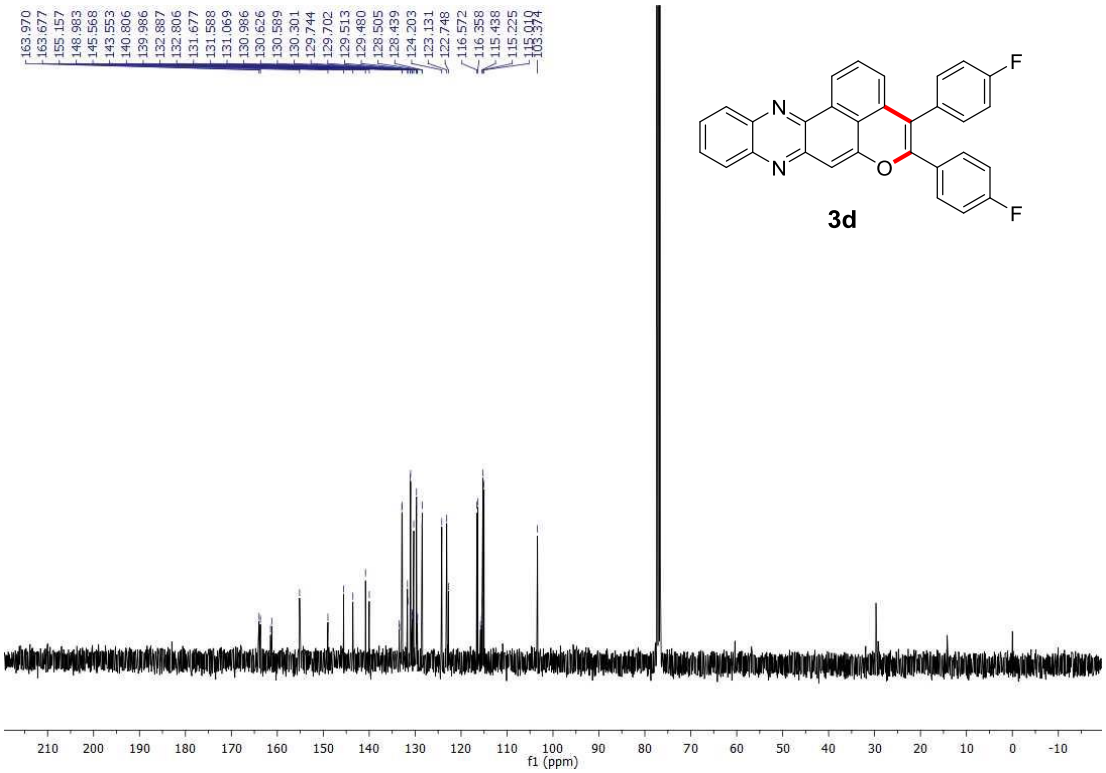

Figure S70. *^{13}C NMR spectrum of synthesized compound 3d in $CDCl_3$*

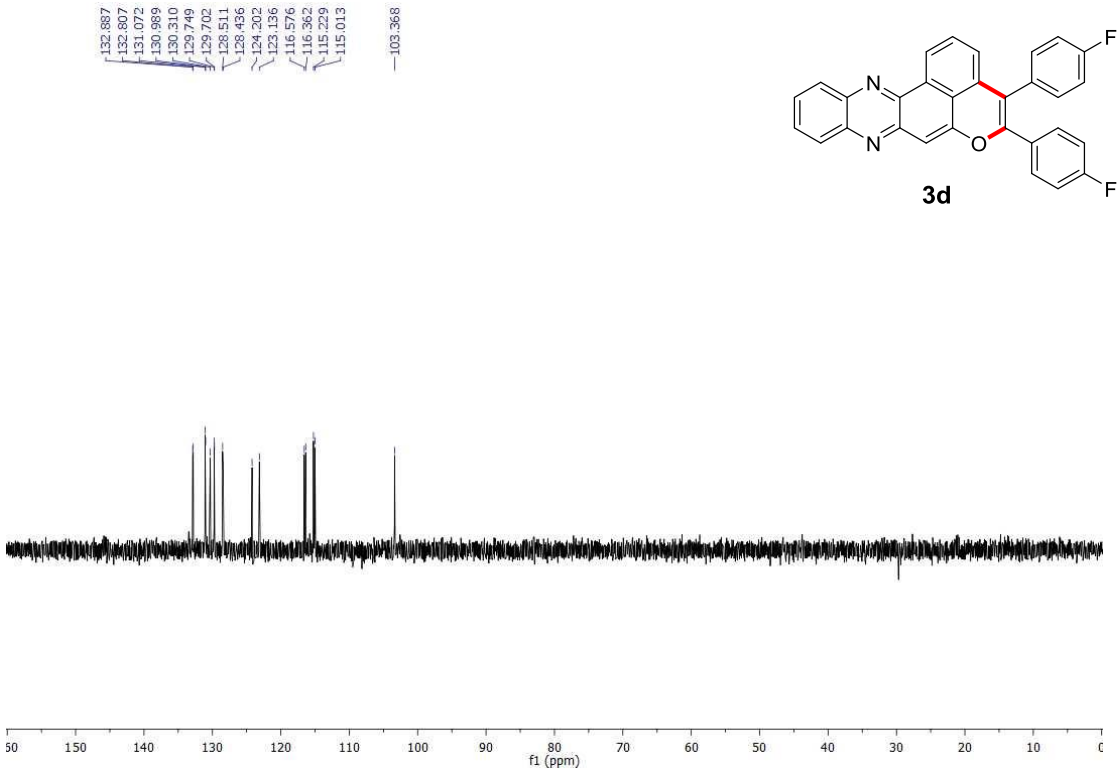

Figure S71. *DEPT-135 NMR spectrum of synthesized compound 3d in $CDCl_3$*

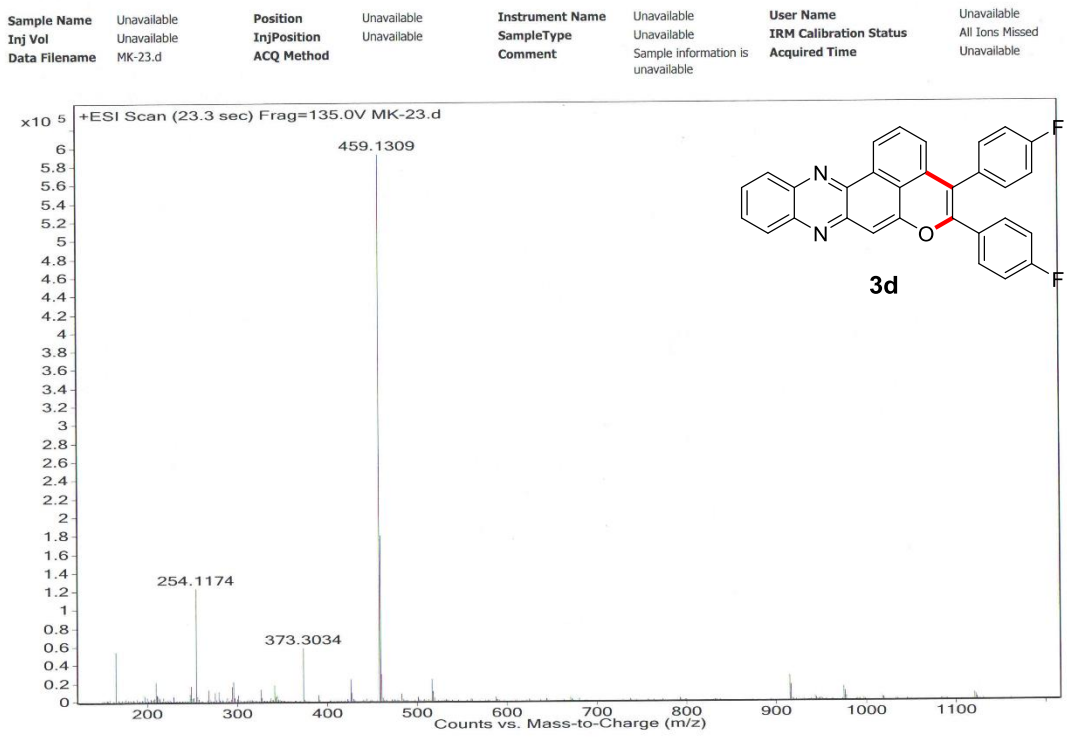

Figure S72. *HRMS spectrum of synthesized compound 3d*

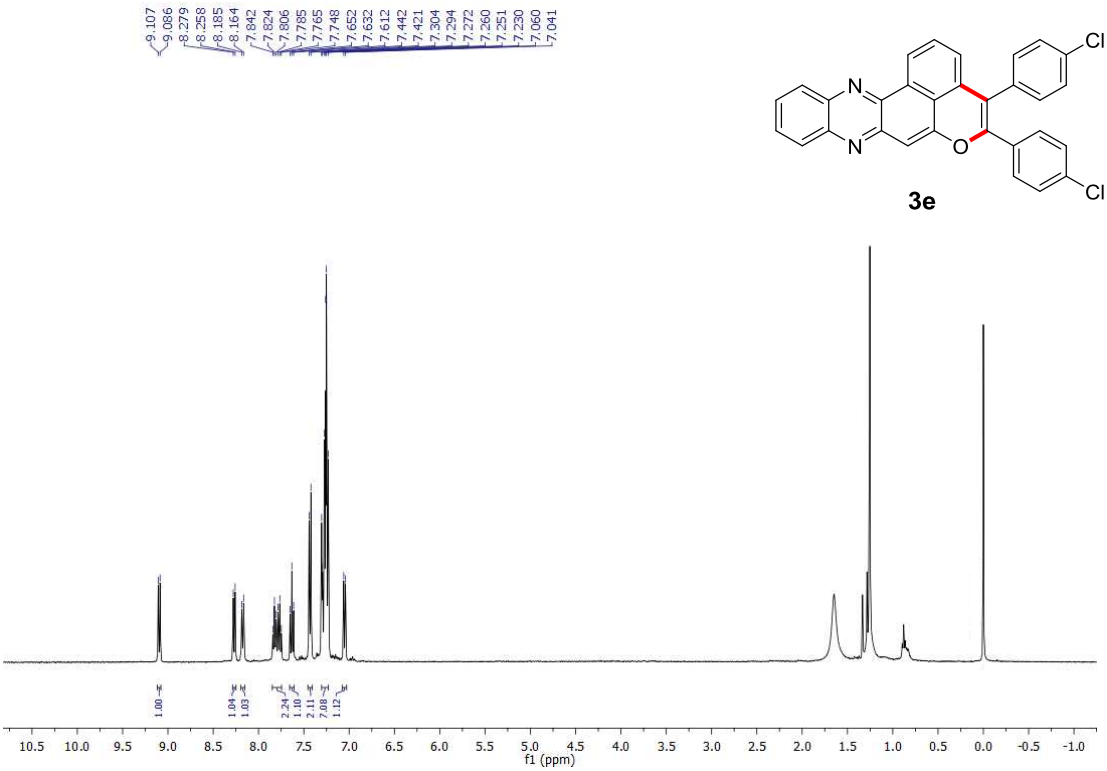

Figure S73. *^{1}H NMR spectrum of synthesized compound 3e in $CDCl_3$*

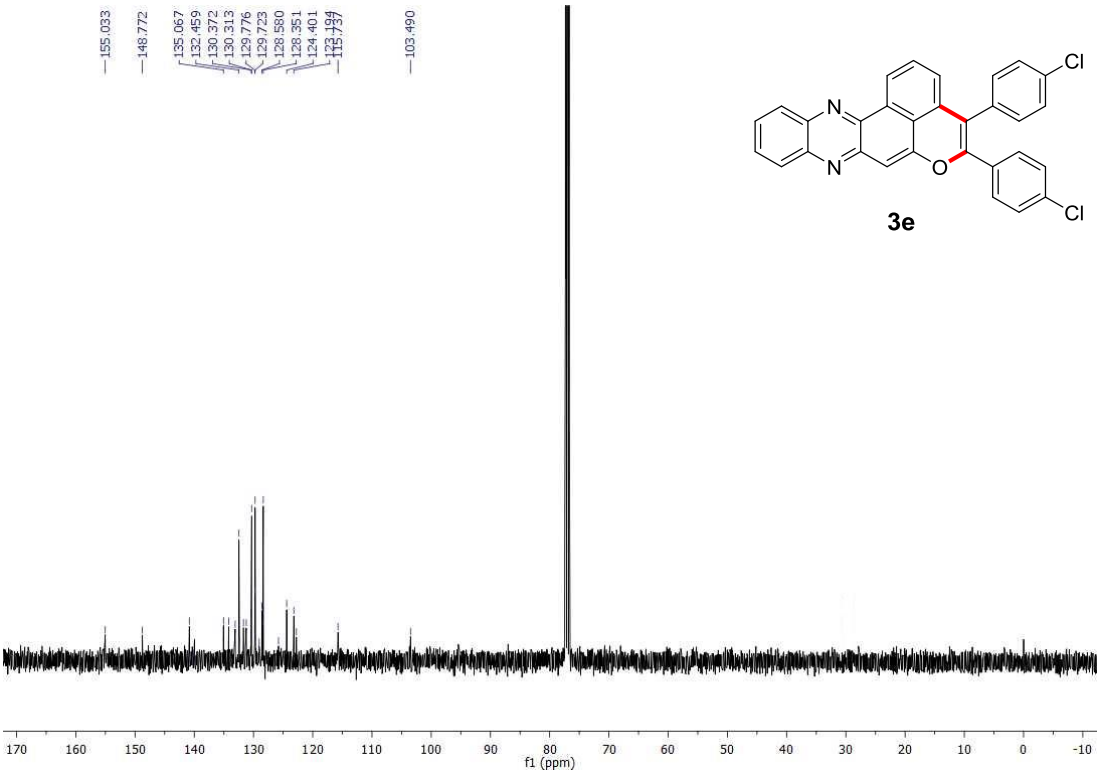

Figure S74. *^{13}C NMR spectrum of synthesized compound 3e in $CDCl_3$*

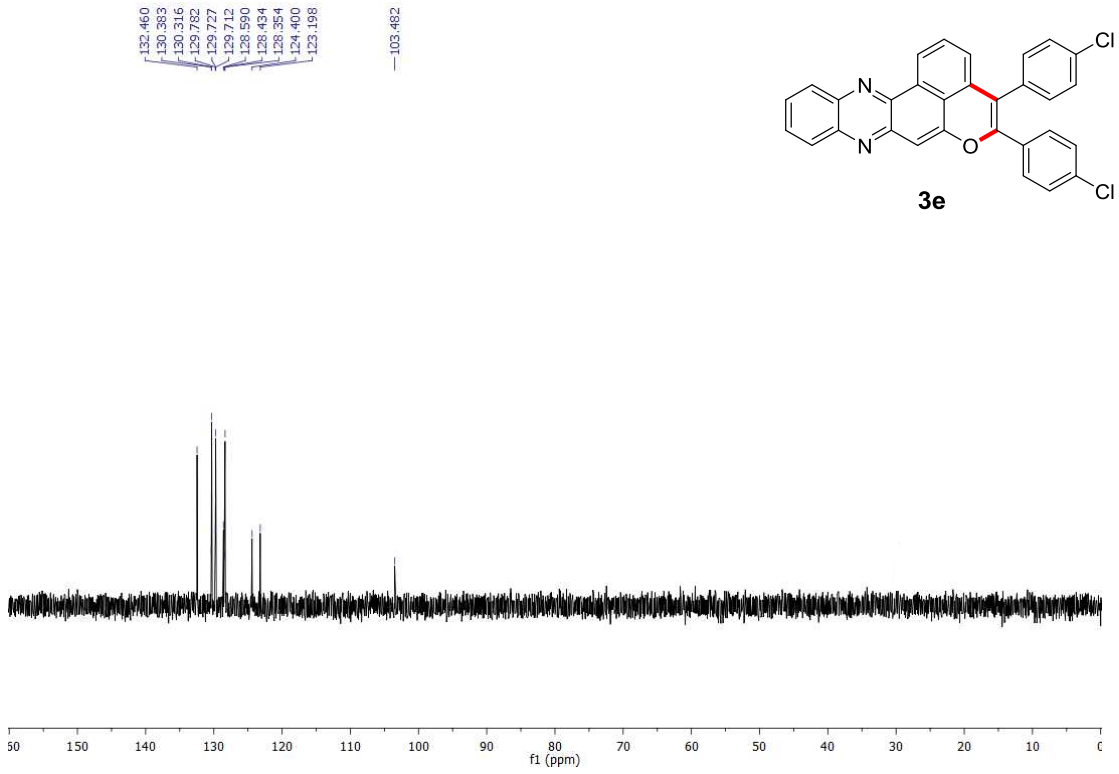

Figure S75. *DEPT-135 NMR spectrum of synthesized compound 3e in $CDCl_3$*

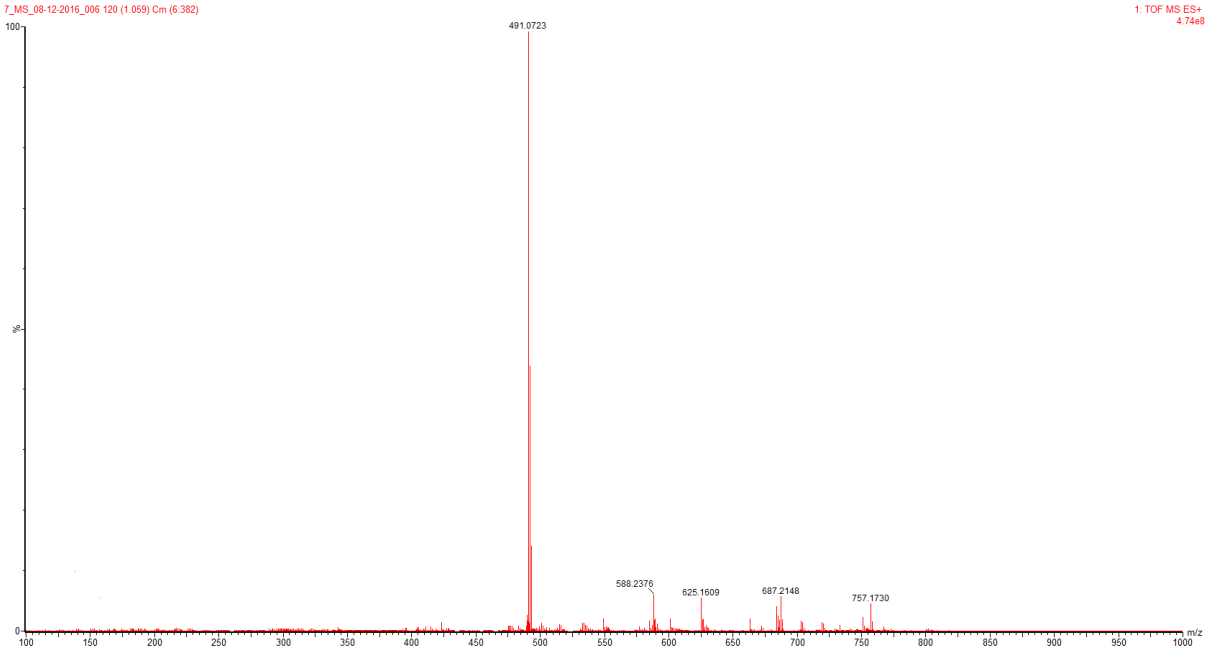

Figure S76. *HRMS spectrum of synthesized compound 3e*

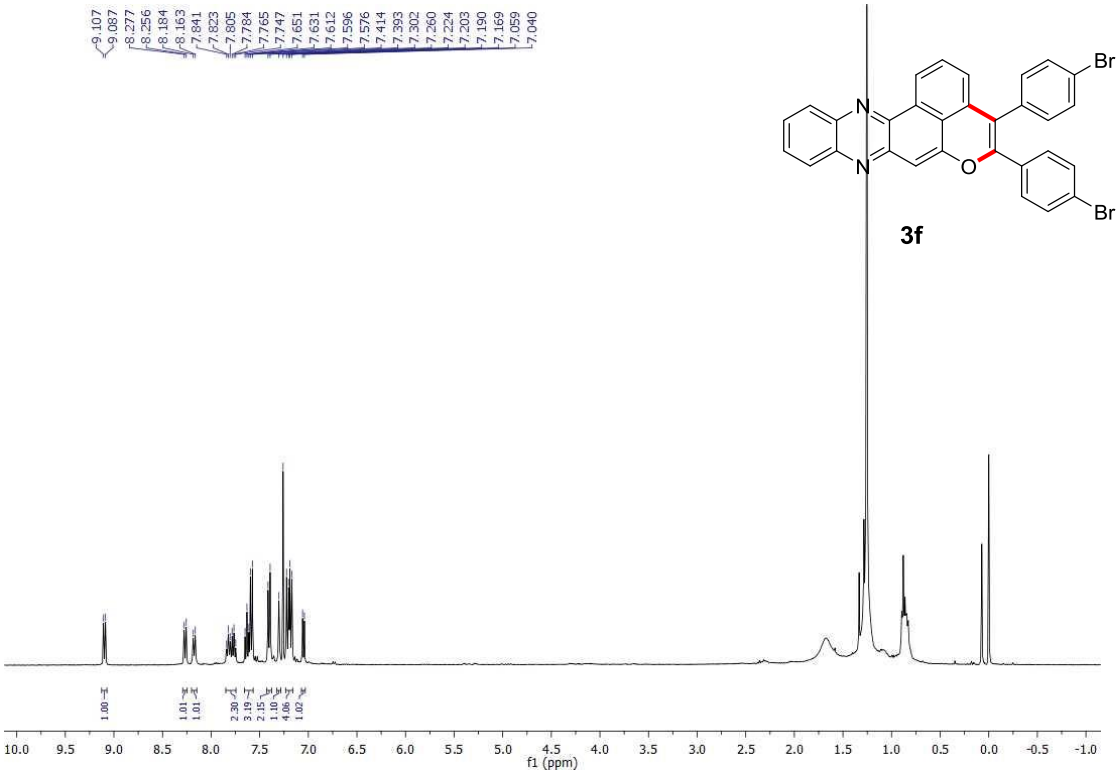

Figure S77. *^{1}H NMR spectrum of synthesized compound 3f in $CDCl_3$*

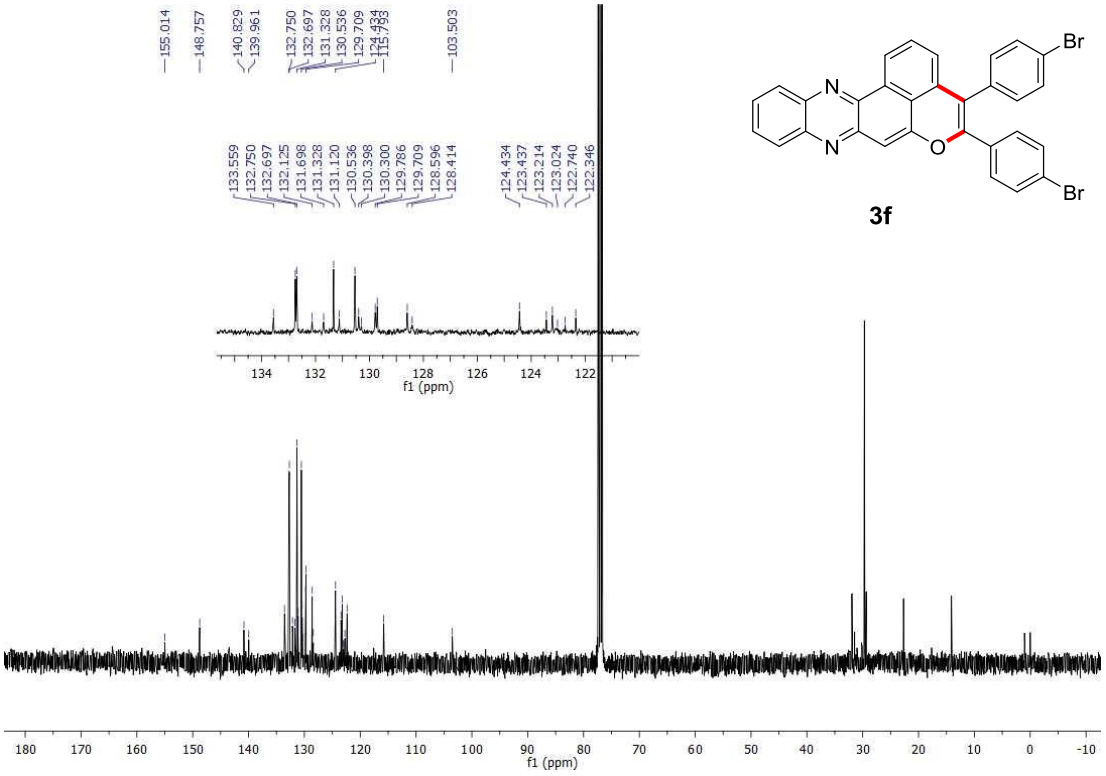

Figure S78. *^{13}C NMR spectrum of synthesized compound 3f in $CDCl_3$*

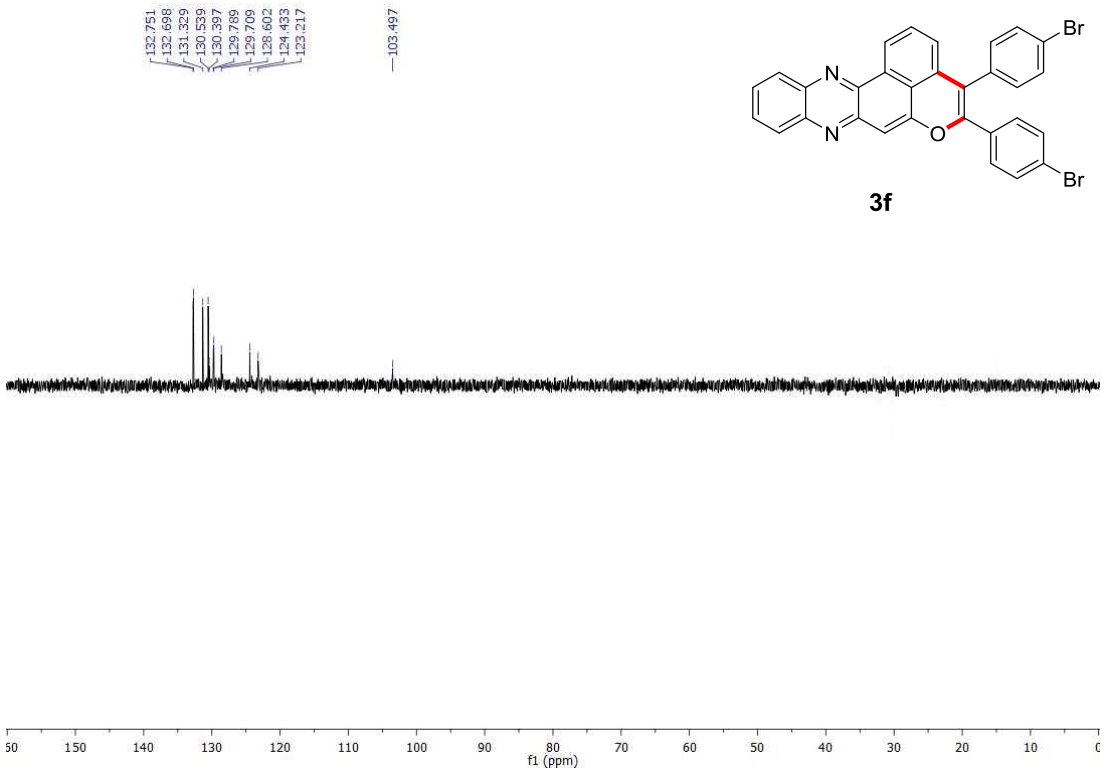

Figure S79. *DEPT-135 NMR spectrum of synthesized compound 3f in $CDCl_3$*

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Analysis Info

Analysis Name	D:\Data\2015\DrNagarajan\DEC\pps-F14.d	Acquisition Date	12/15/2015 1:02:26 PM
Method	tune_low.m	Operator	Ramu Sridhar
Sample Name	PPS-F14-ACN	Instrument	maXis 10138
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	4.4 psi
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1500 m/z	Set Collision Cell RF	350.0 Vpp	Set Divert Valve	Waste

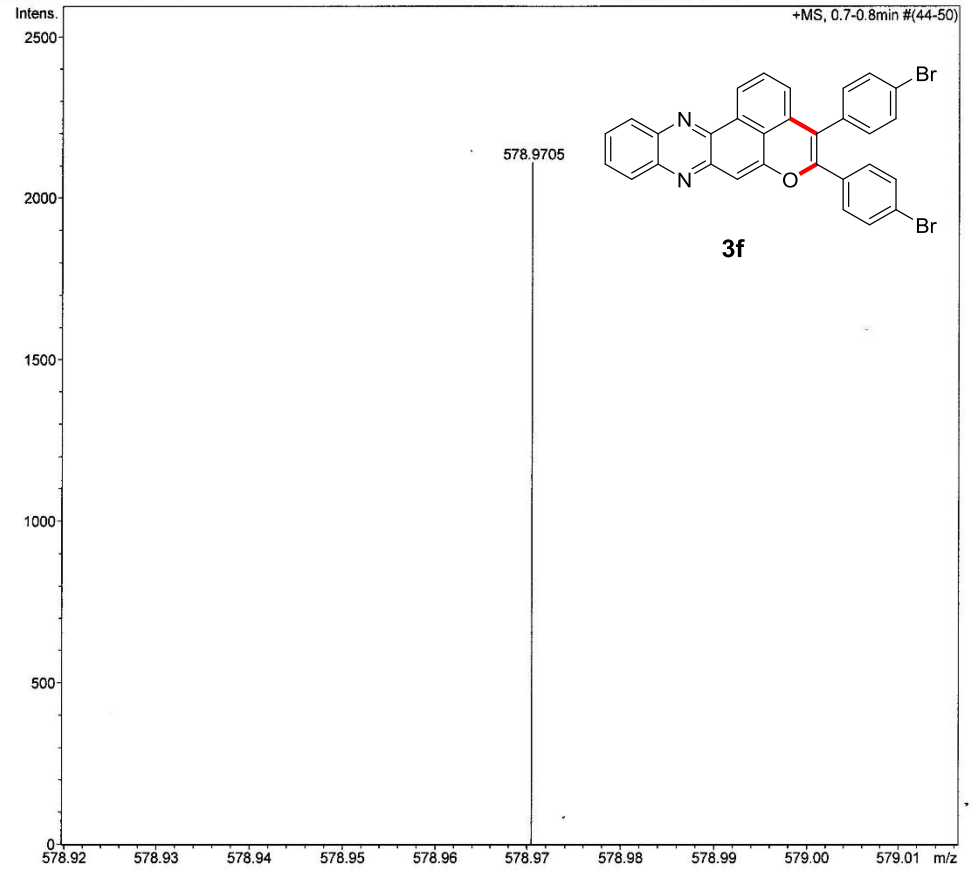

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Figure S80. *HRMS spectrum of synthesized compound 3f*

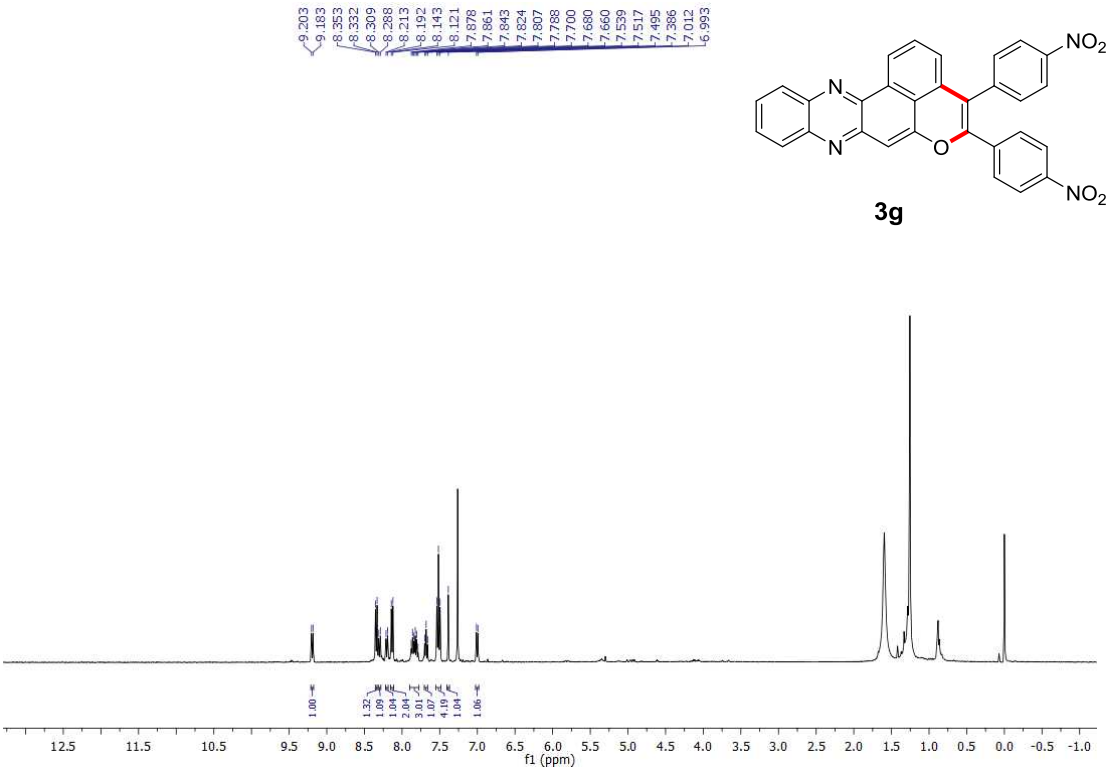

Figure S81. *^{1}H NMR spectrum of synthesized compound 3g in $CDCl_3$*

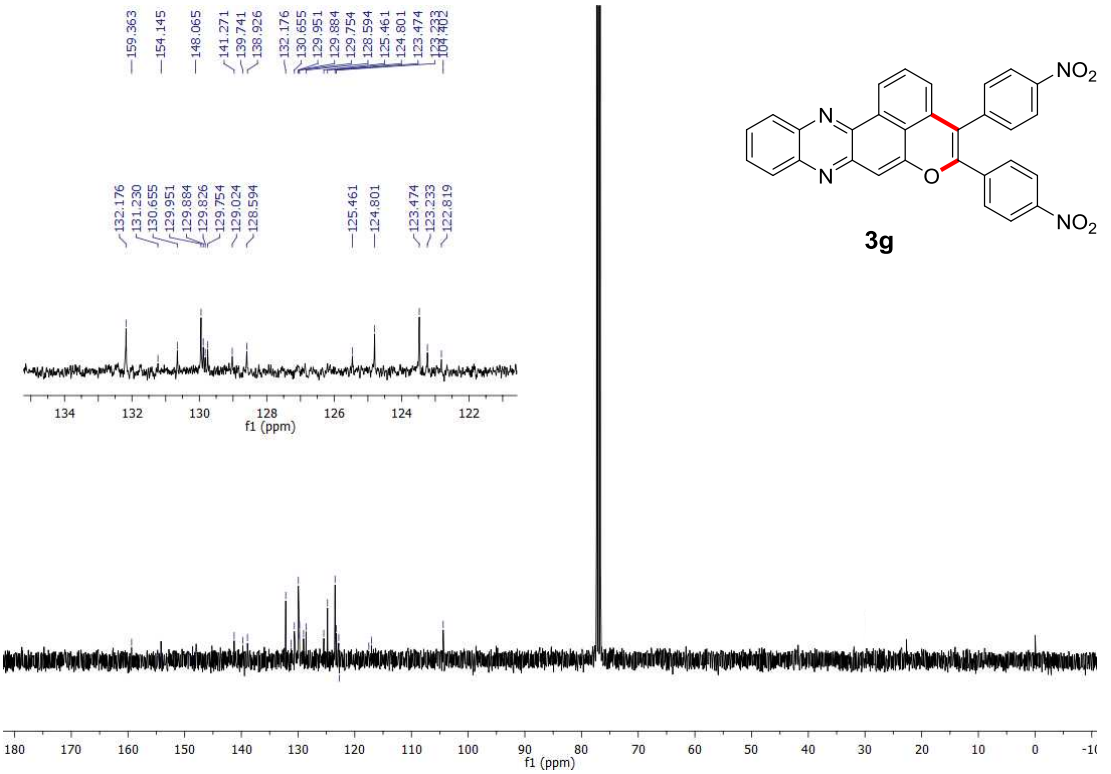

Figure S82. *^{13}C NMR spectrum of synthesized compound 3g in $CDCl_3$*

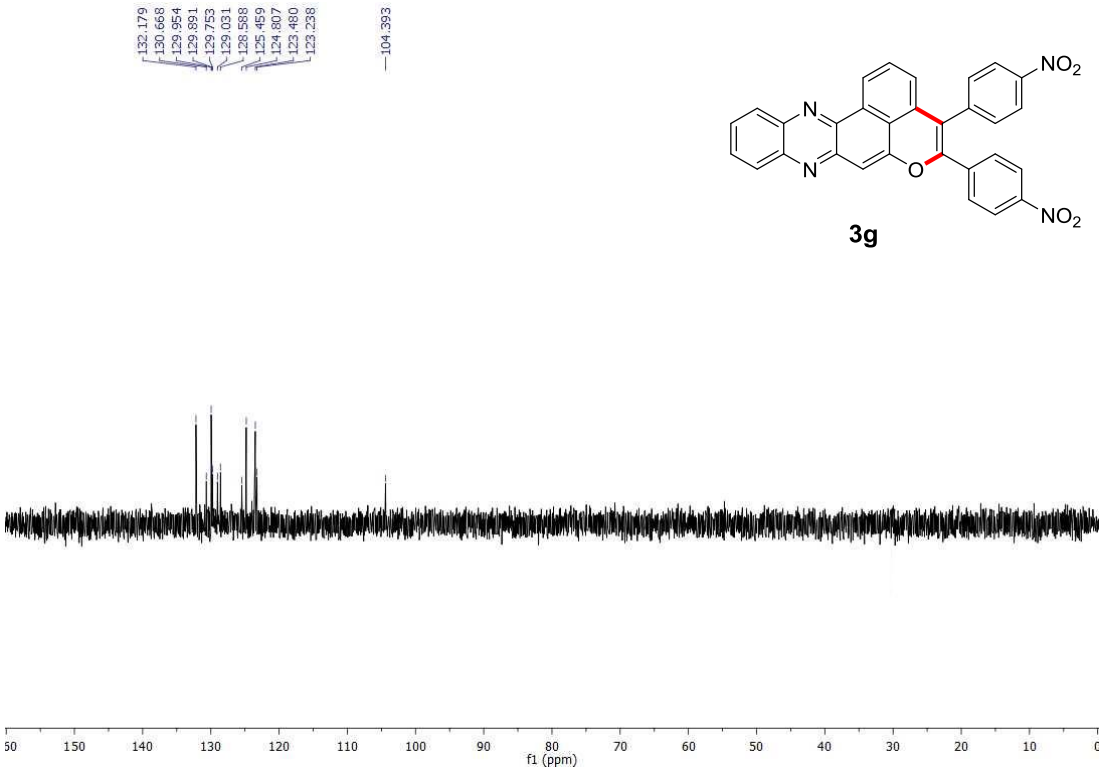

Figure S83. *DEPT-135 NMR spectrum of synthesized compound 3g in $CDCl_3$*

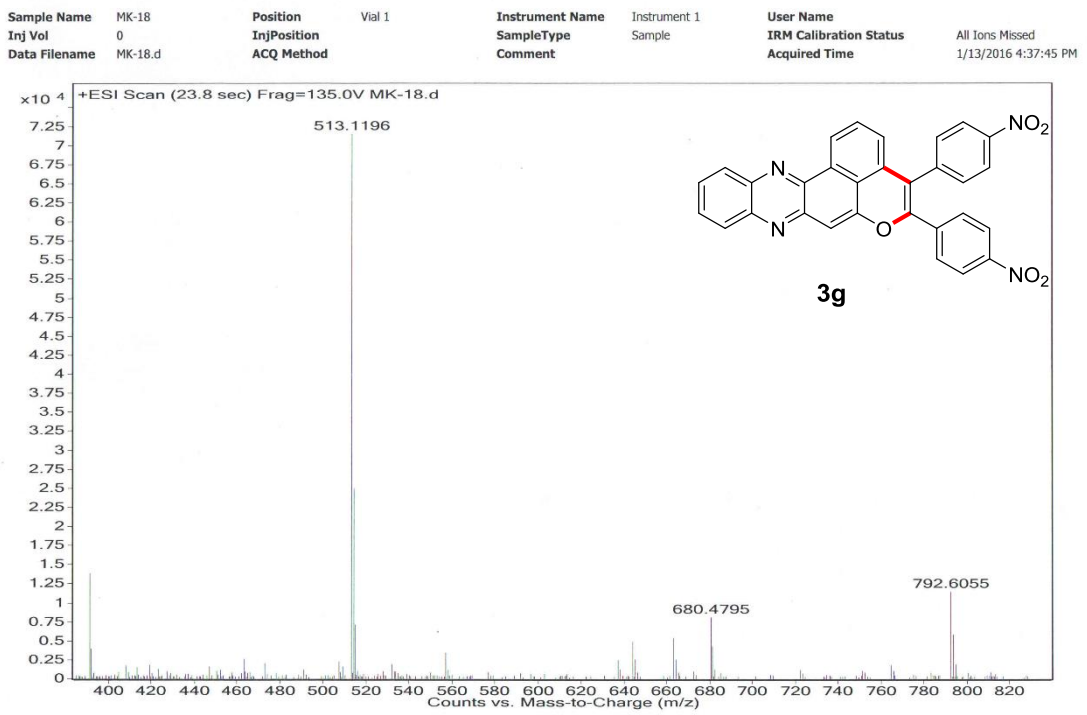

Figure S84. *HRMS spectrum of synthesized compound 3g*

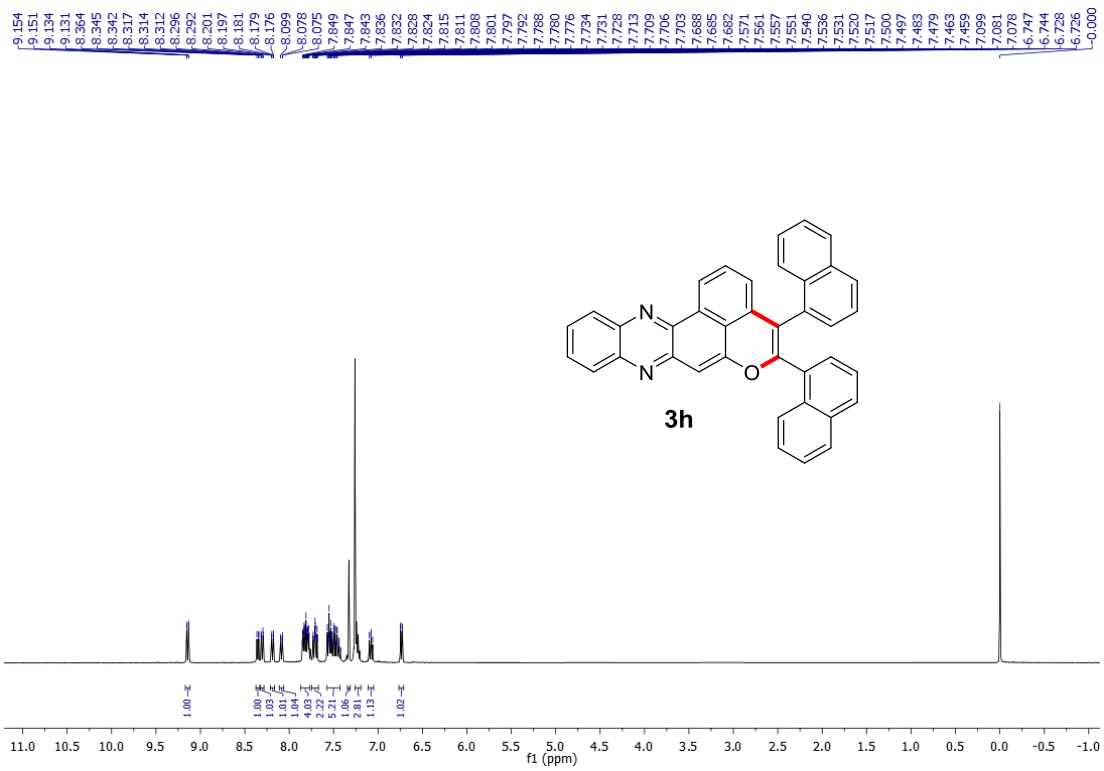

Figure S85. *^{1}H NMR spectrum of synthesized compound 3h in $CDCl_3$*

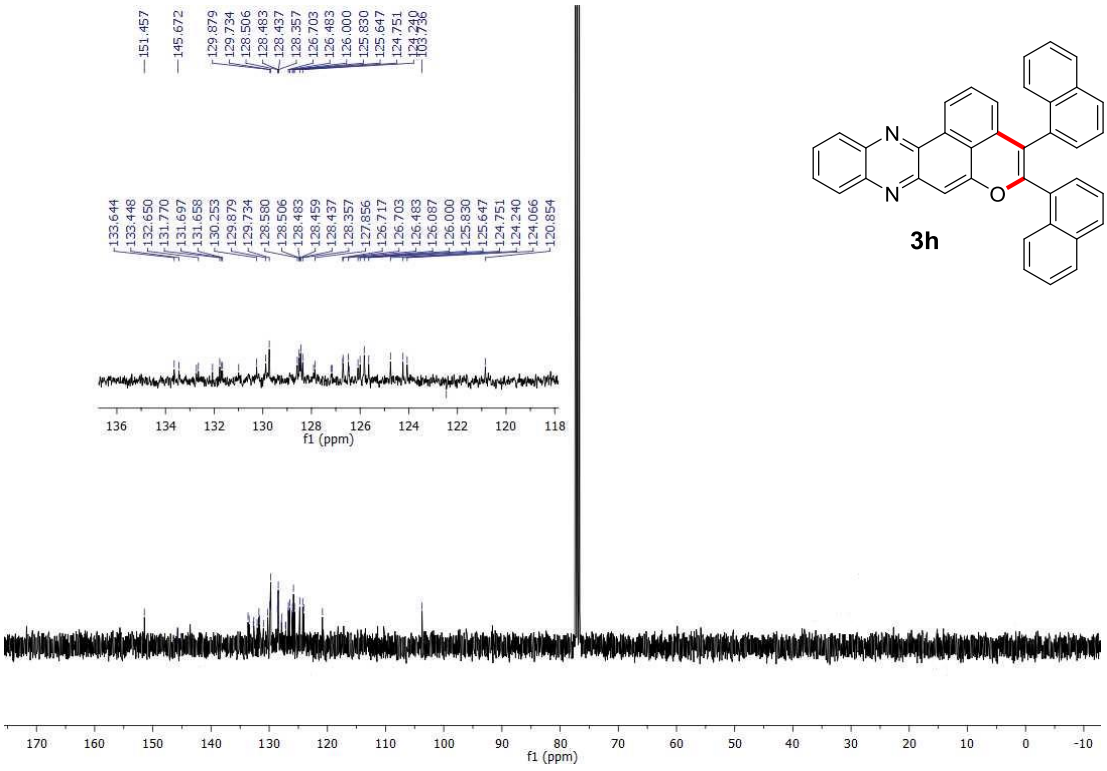

Figure S86. *^{13}C NMR spectrum of synthesized compound 3h in $CDCl_3$*

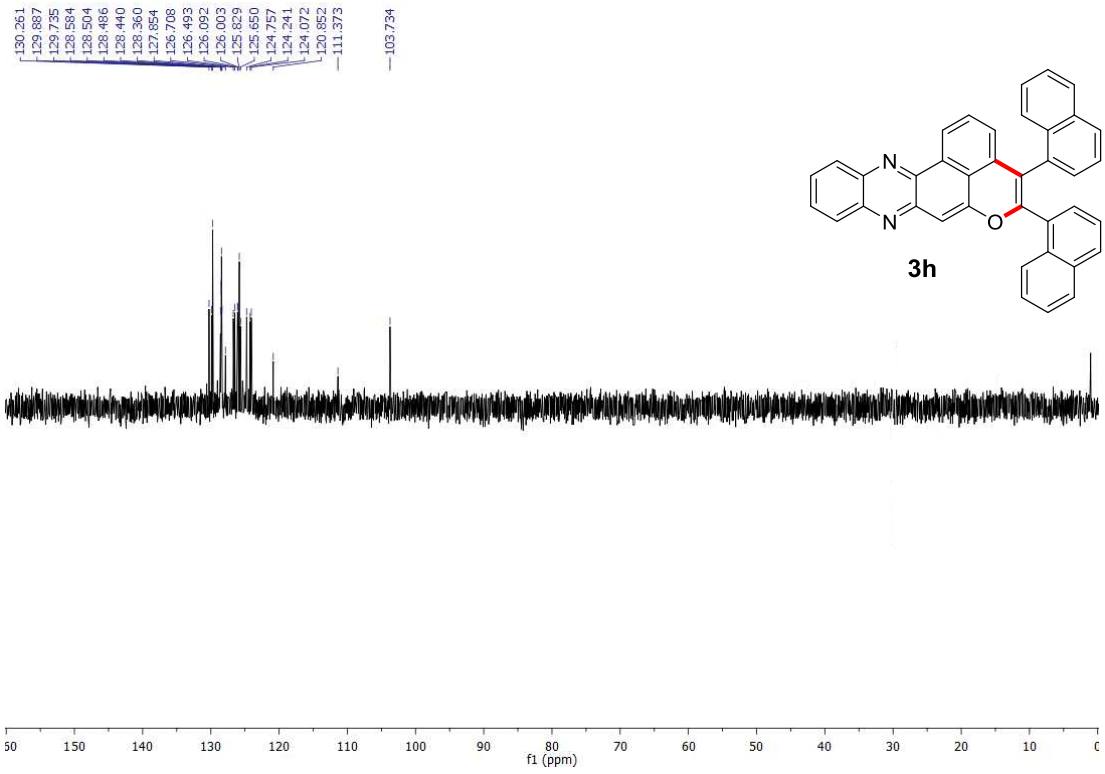

Figure S87. *DEPT-135 NMR spectrum of synthesized compound 3h in $CDCl_3$*

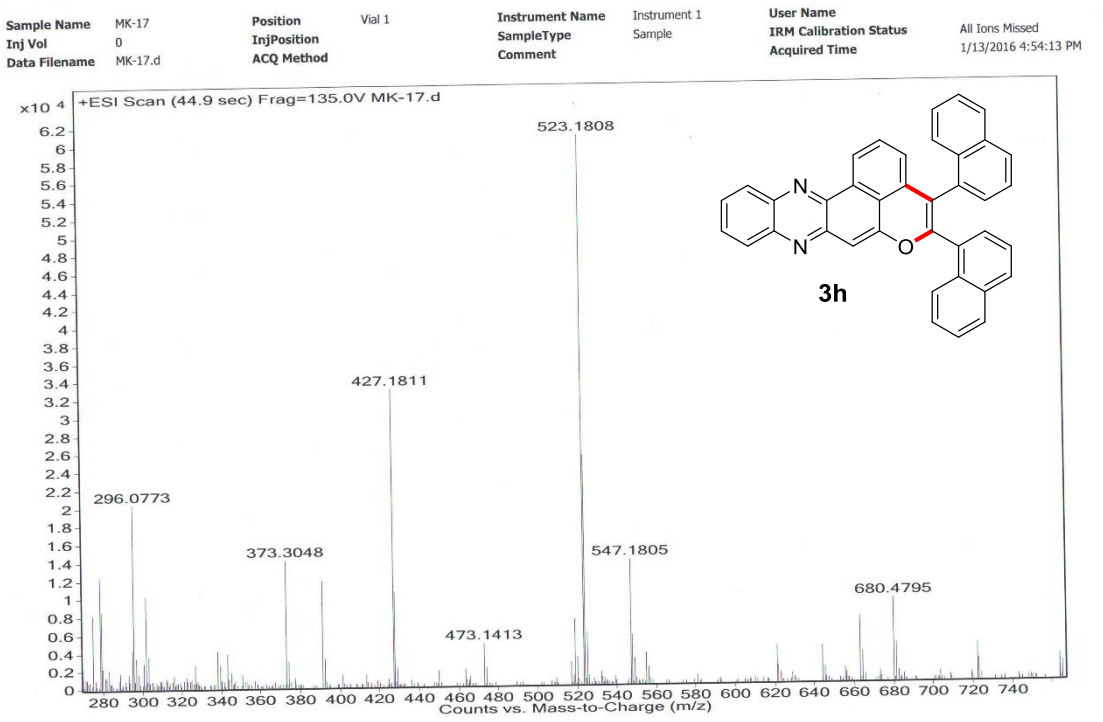

Figure S88. *HRMS spectrum of synthesized compound 3h*

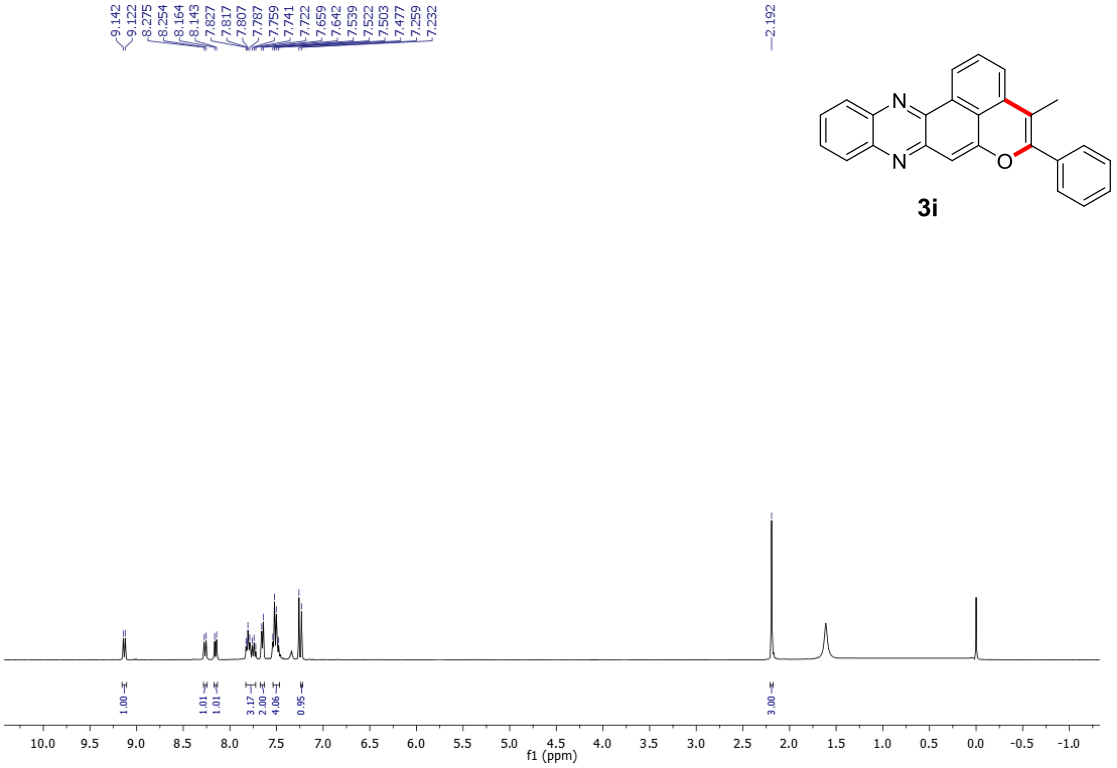

Figure S89. *^{1}H NMR spectrum of synthesized compound 3i in $CDCl_3$*

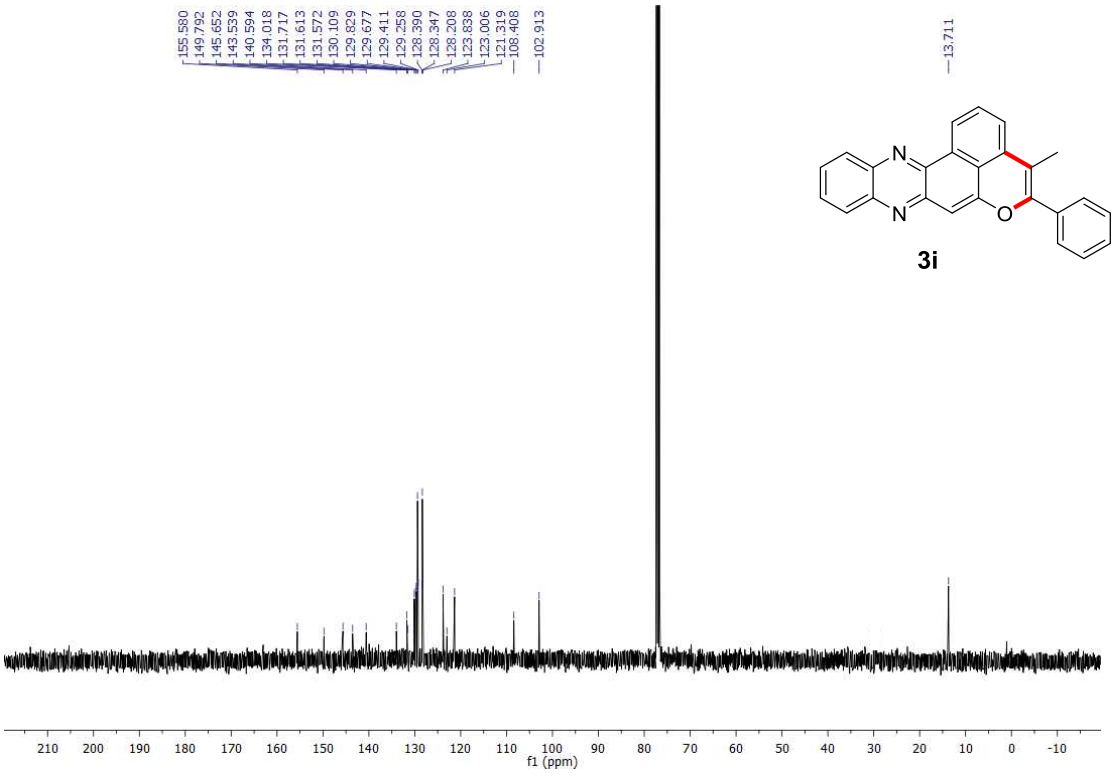

Figure S90. *^{13}C NMR spectrum of synthesized compound 3i in $CDCl_3$*

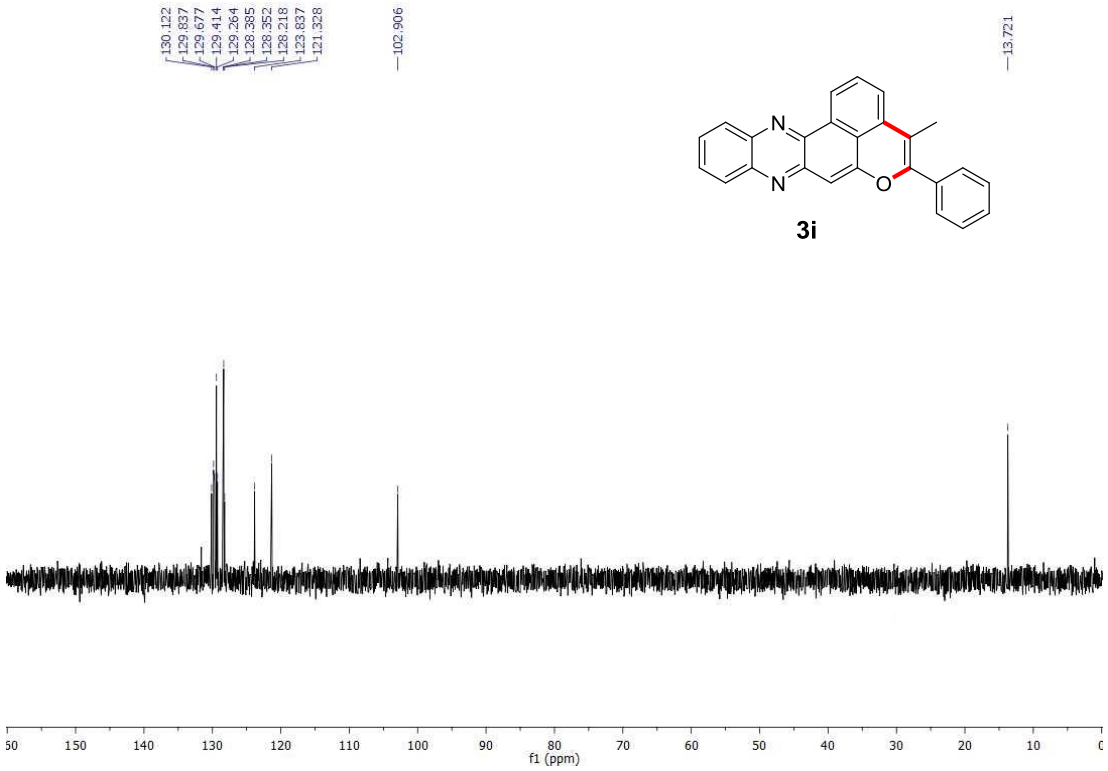

Figure S91. *DEPT-135 NMR spectrum of synthesized compound 3i in* $CDCl_3$

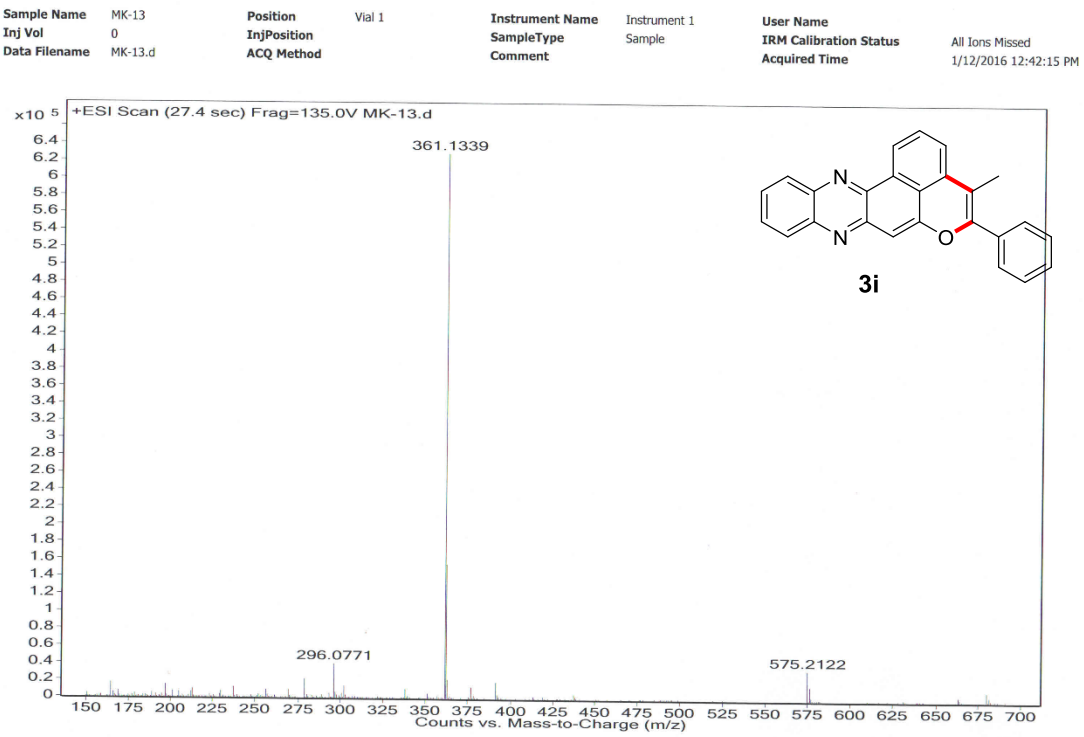

Figure S92. *HRMS spectrum of synthesized compound 3i*

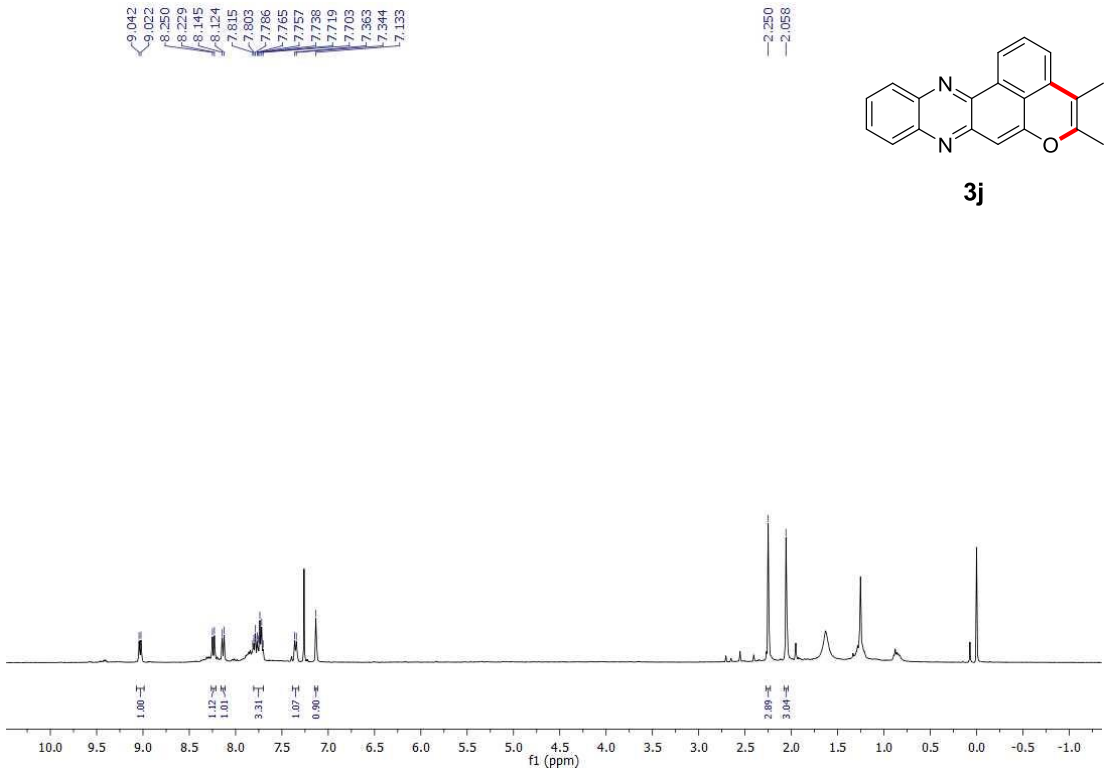

Figure S93. *^{1}H NMR spectrum of synthesized compound 3j in $CDCl_3$*

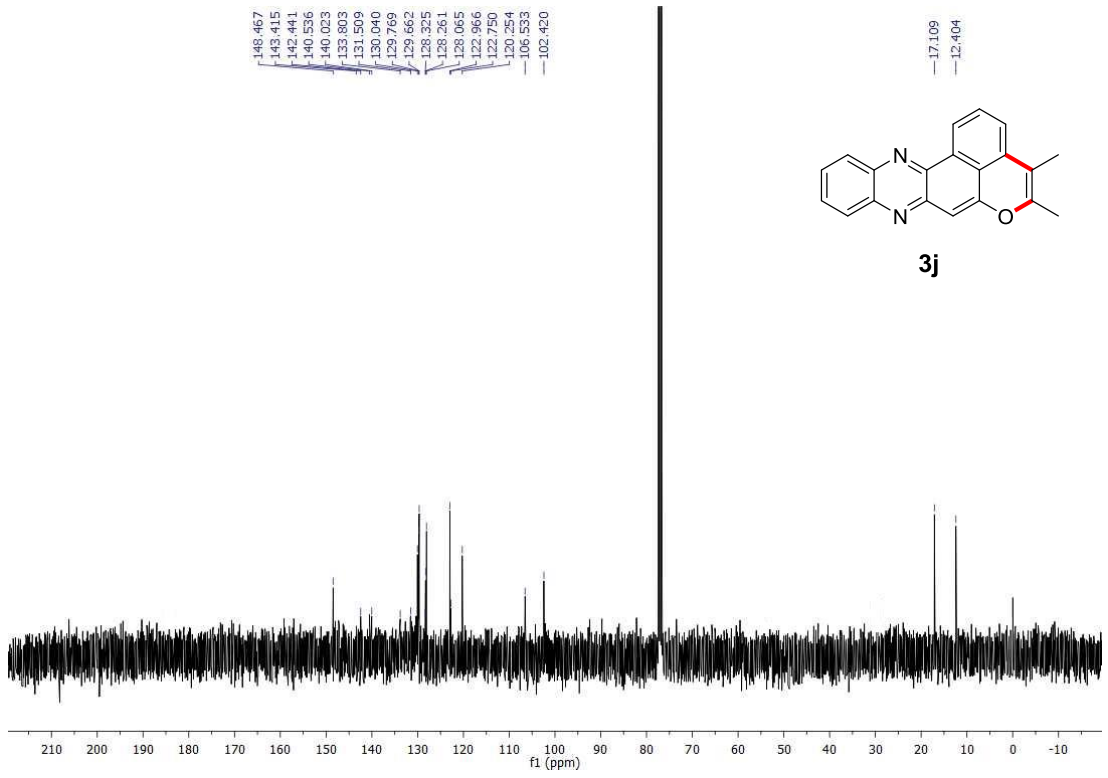

Figure S94. *^{13}C NMR spectrum of synthesized compound 3j in $CDCl_3$*

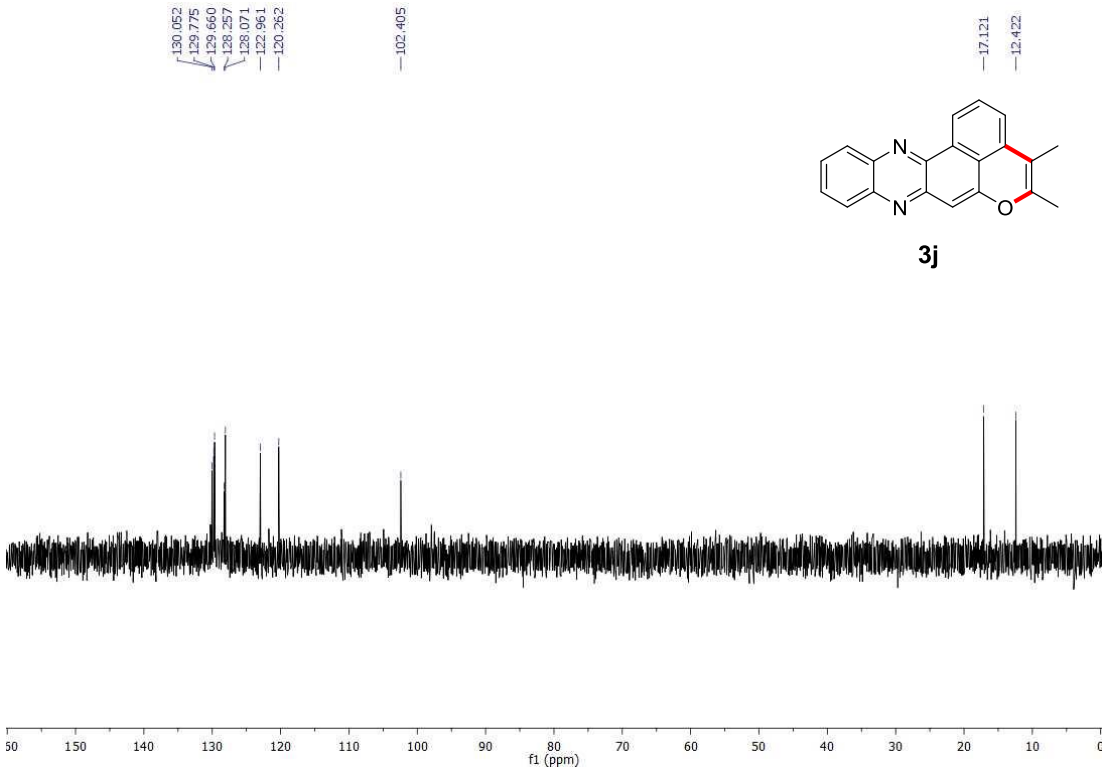

Figure S95. *DEPT-135 NMR spectrum of synthesized compound 3j in* $CDCl_3$

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Analysis Info

Analysis Name	D:\Data\2015\DrNagarajan\DEC\pps-F15.d	Acquisition Date	12/15/2015 11:24:24 AM
Method	TL-P.m	Operator	Ramu Sridhar
Sample Name	PPS-F15-ACN	Instrument	maXis 10138
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	4.4 psi
Focus	Not active	Set Capillary	3800 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	600 m/z	Set Collision Cell RF	350.0 Vpp	Set Divert Valve	Waste

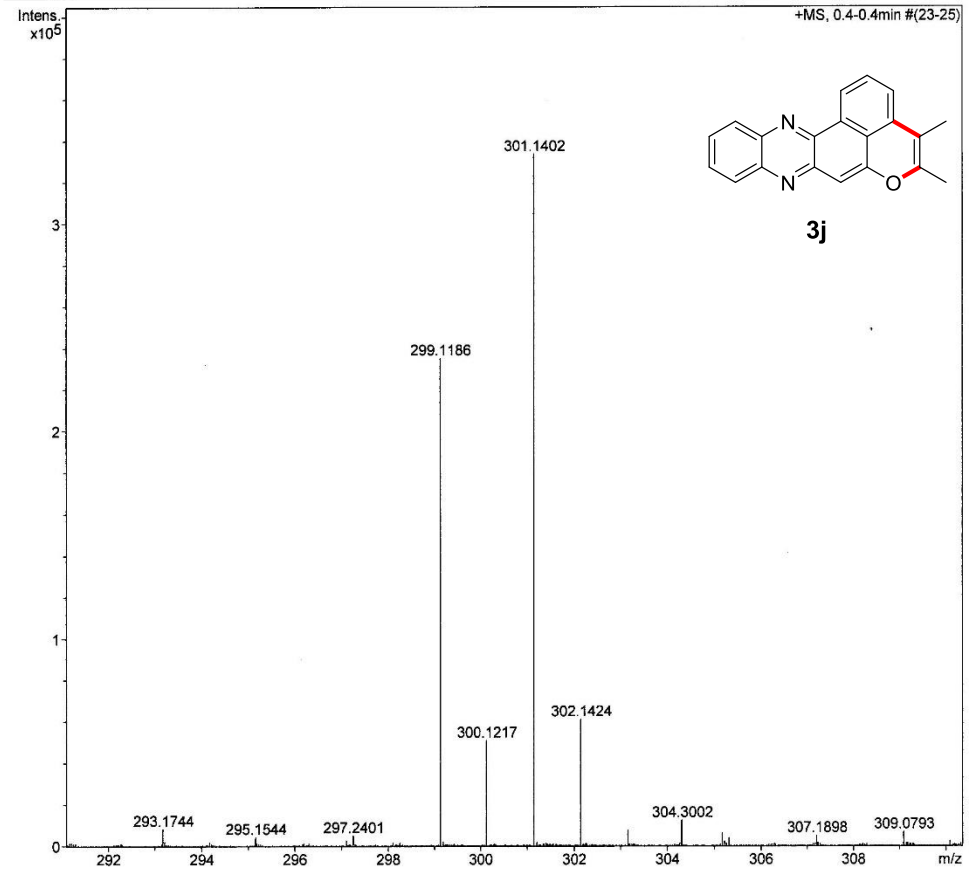

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Figure S96. *HRMS spectrum of synthesized compound 3j*

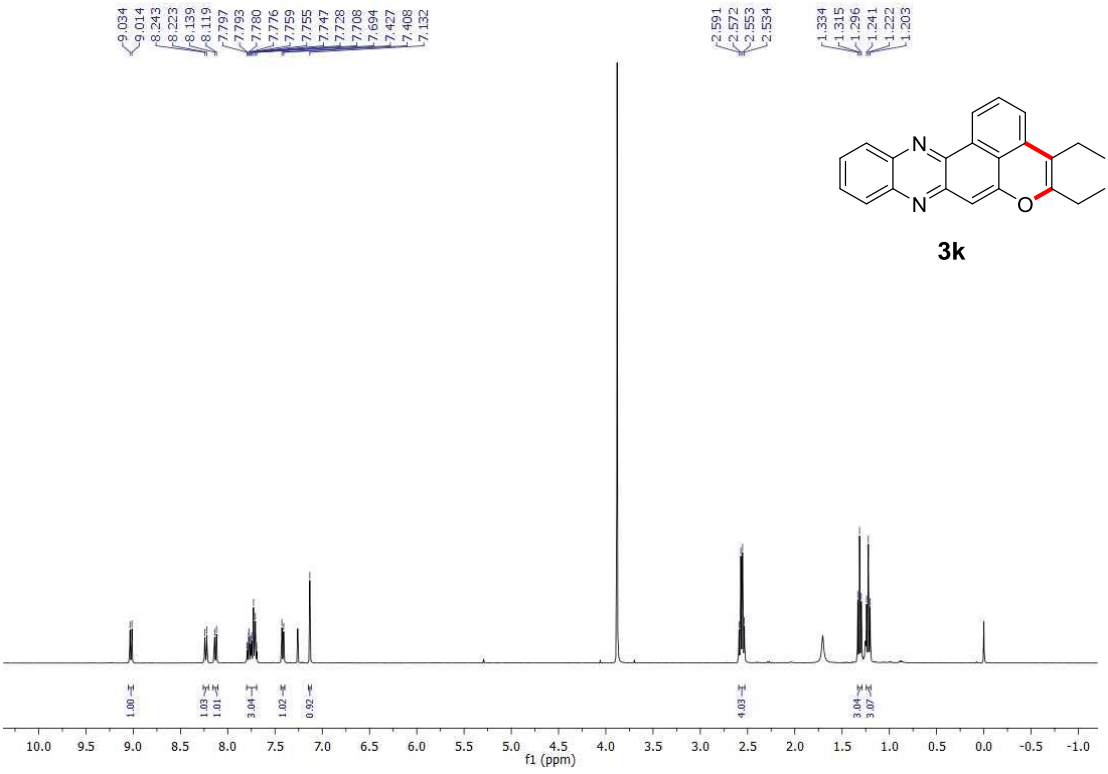

Figure S97. *^{1}H NMR spectrum of synthesized compound 3k in $CDCl_3$*

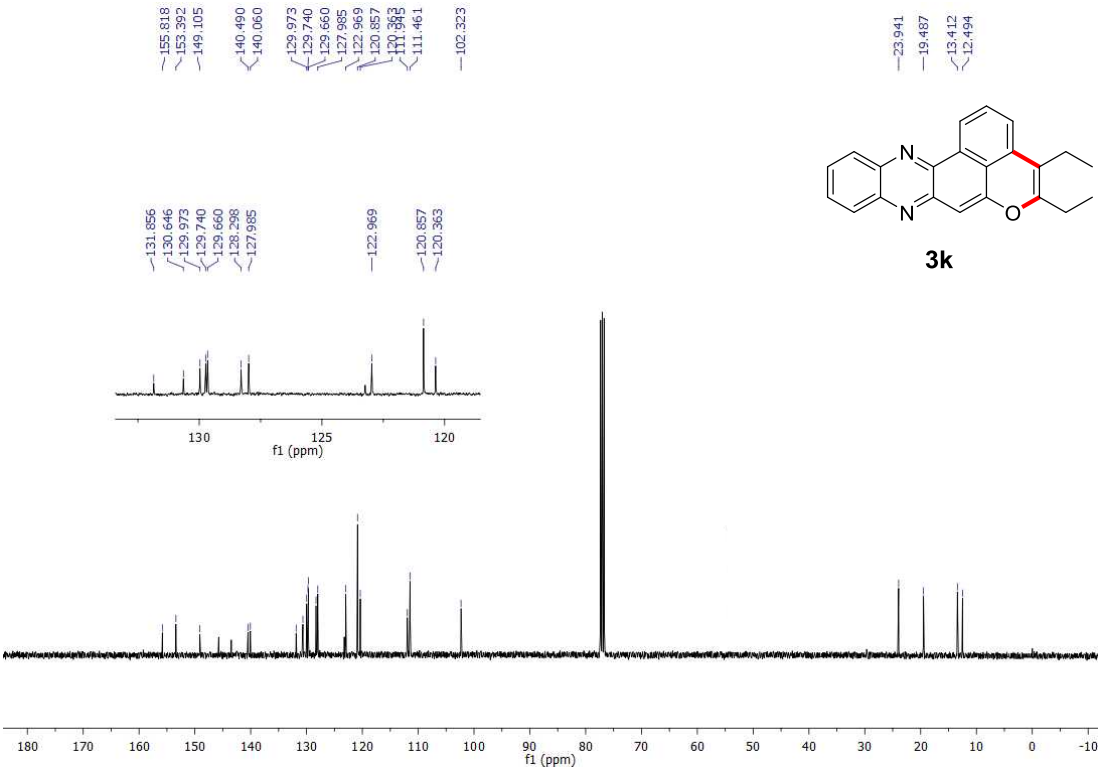

Figure S98. *^{13}C NMR spectrum of synthesized compound 3k in $CDCl_3$*

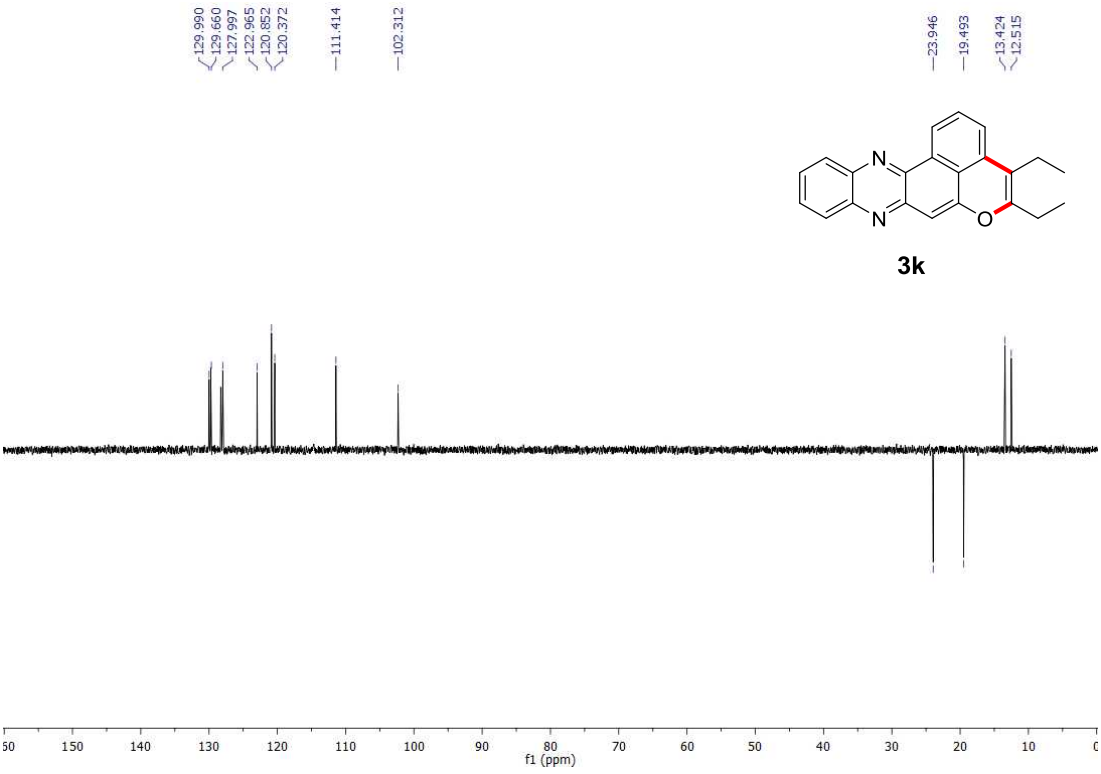

Figure S99. *DEPT-135 NMR spectrum of synthesized compound 3k in $CDCl_3$*

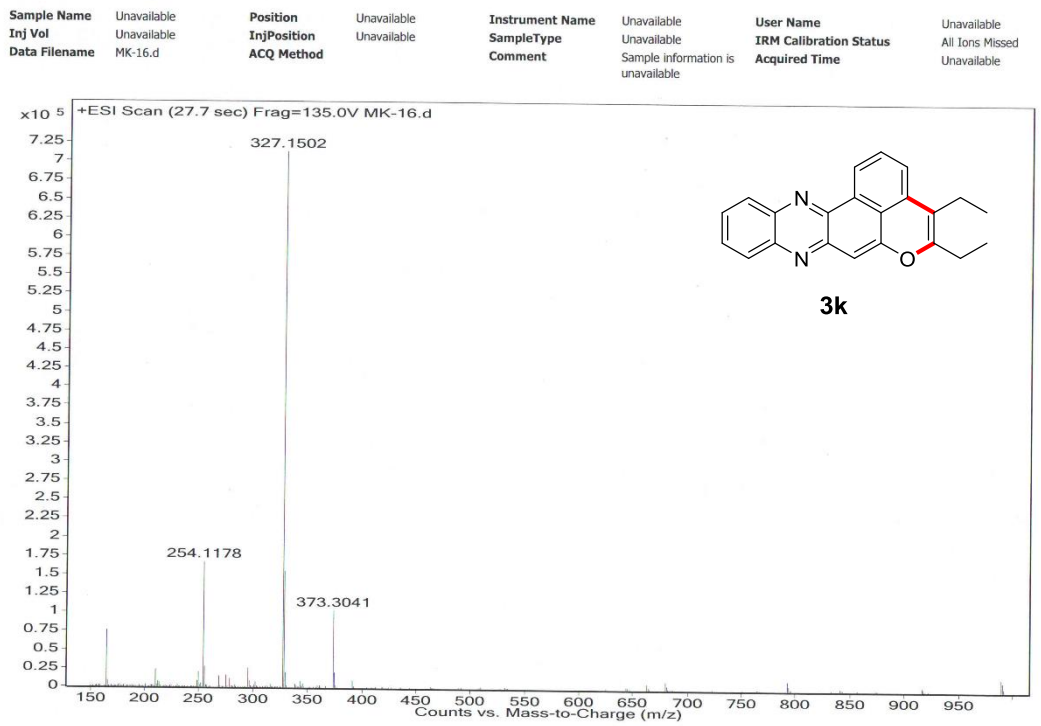

Figure S100. *HRMS spectrum of synthesized compound 3k*

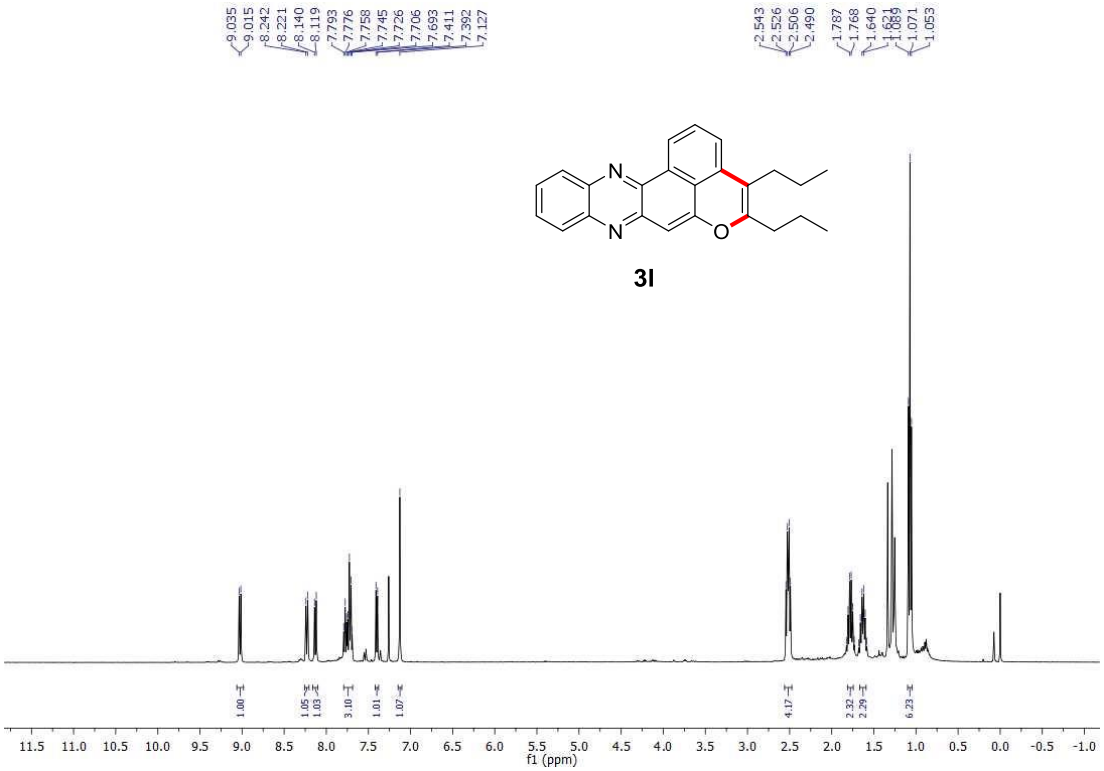

Figure S101. *^{1}H NMR spectrum of synthesized compound 3l in $CDCl_3$*

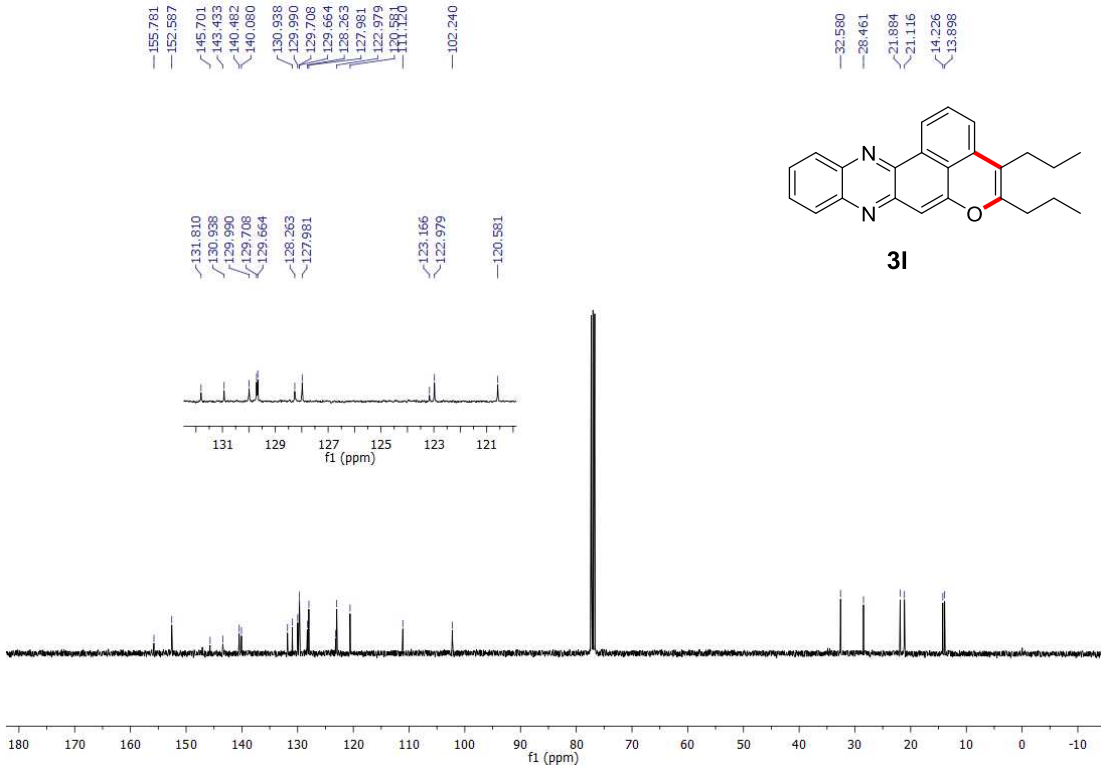

Figure S102. *^{13}C NMR spectrum of synthesized compound 3l in $CDCl_3$*

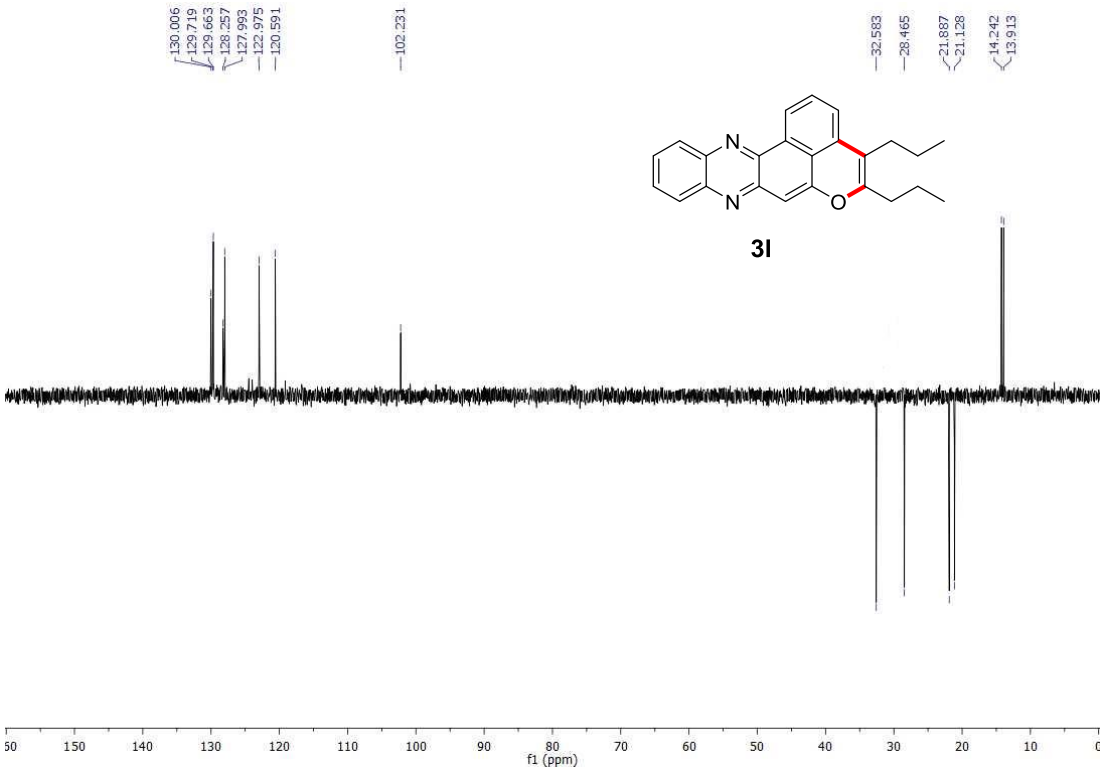

Figure S103. *DEPT-135 NMR spectrum of synthesized compound 3l in* $CDCl_3$

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Analysis Info

Analysis Name	D:\Data\2015\DrNagarajan\DEC\PPS-F16.d	Acquisition Date	12/18/2015 1:04:15 PM
Method	tune_low.m	Operator	Ramu Sridhar
Sample Name	PPS-F16-ACN	Instrument	maXis 10138
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	4.4 psi
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1500 m/z	Set Collision Cell RF	350.0 Vpp	Set Divert Valve	Waste

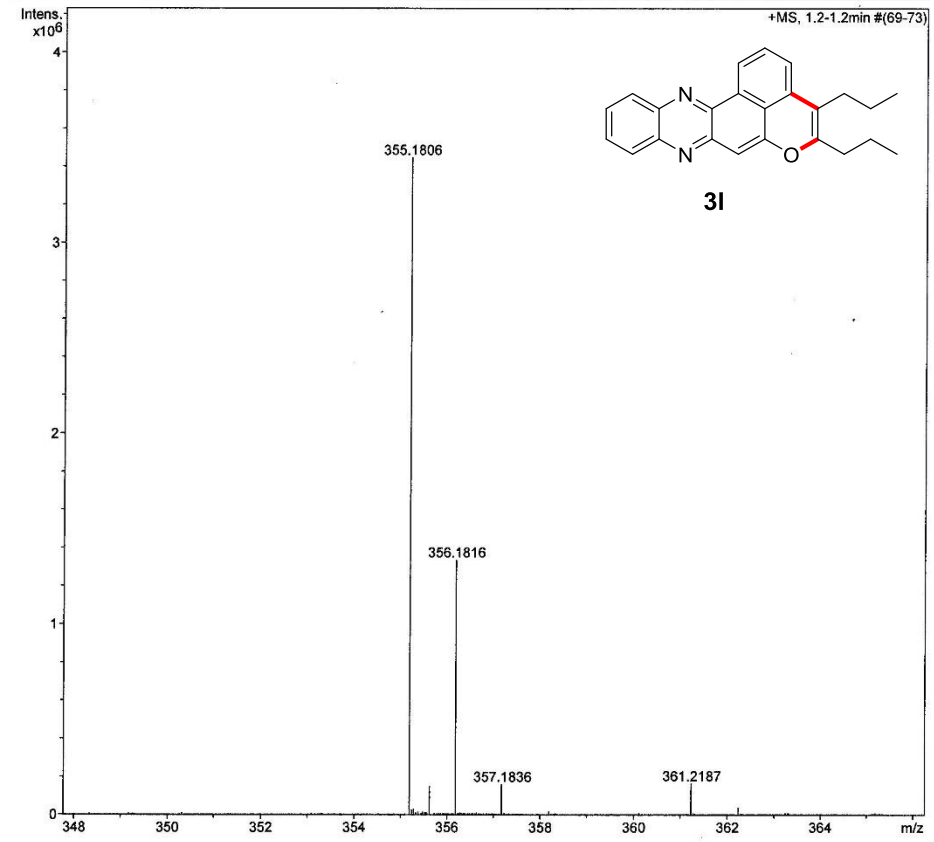

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Figure S104. *HRMS spectrum of synthesized compound 3l*

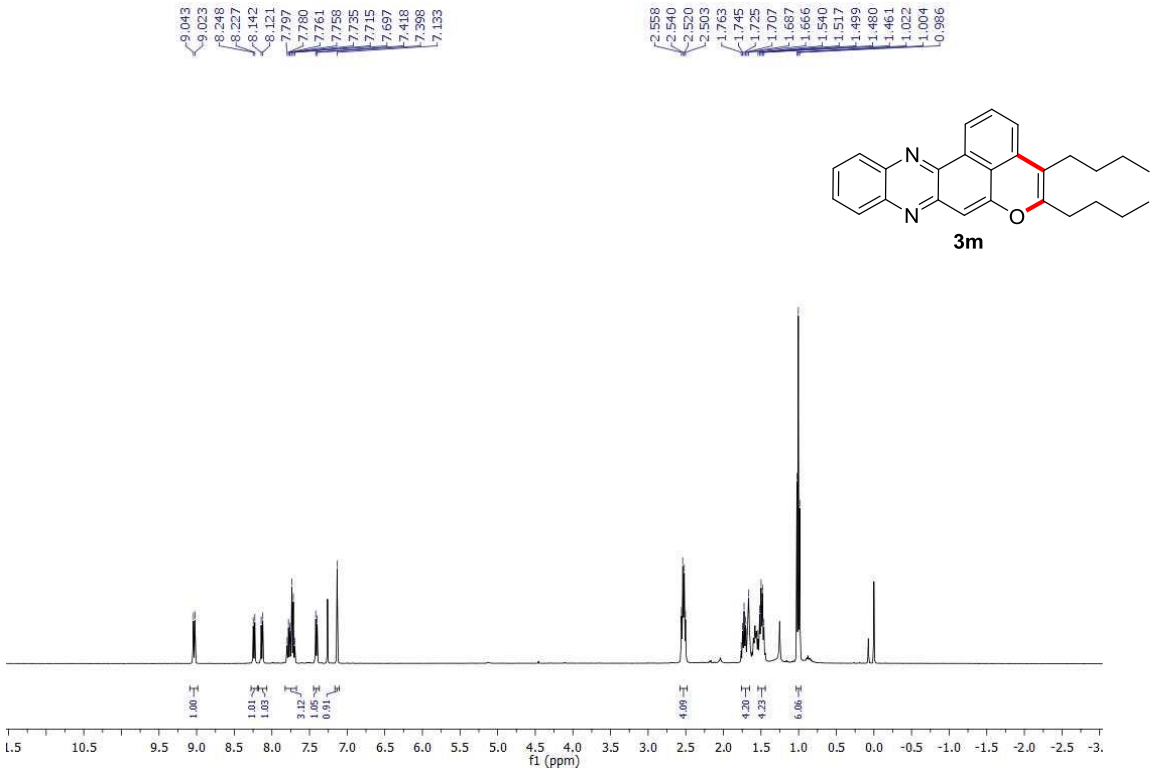

Figure S105. *^{1}H NMR spectrum of synthesized compound 3m in $CDCl_3$*

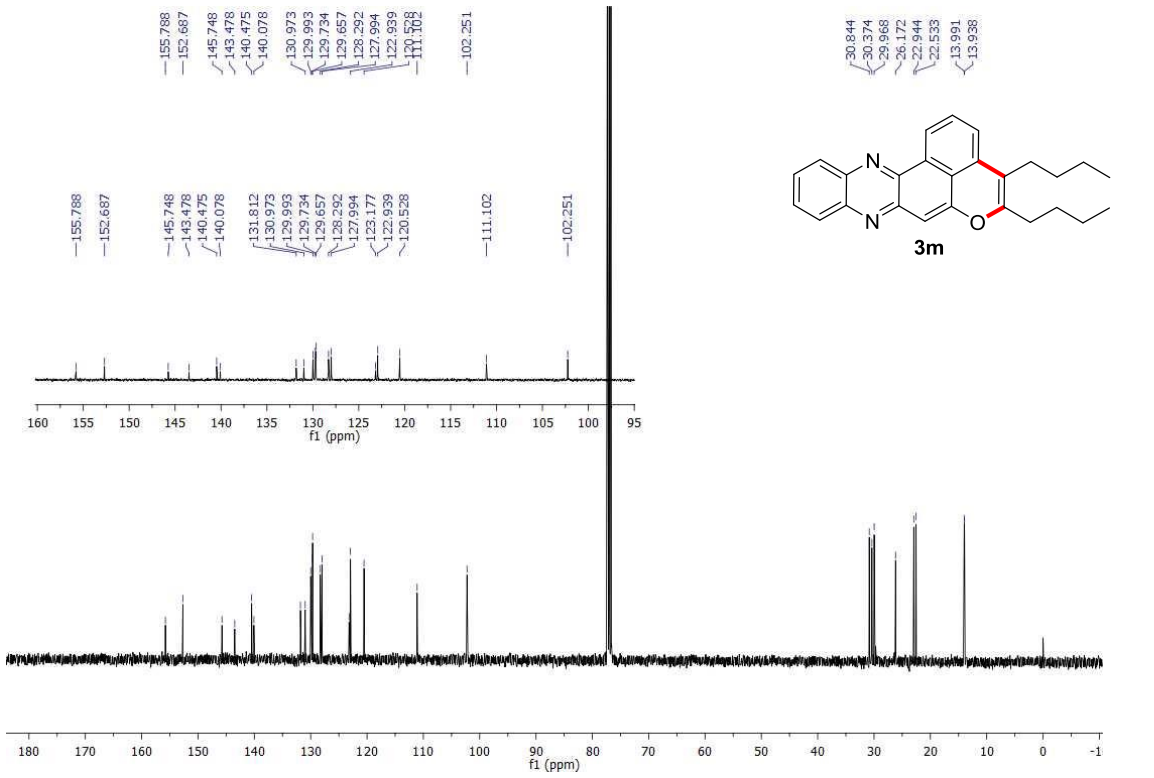

Figure S106. *^{13}C NMR spectrum of synthesized compound 3m in $CDCl_3$*

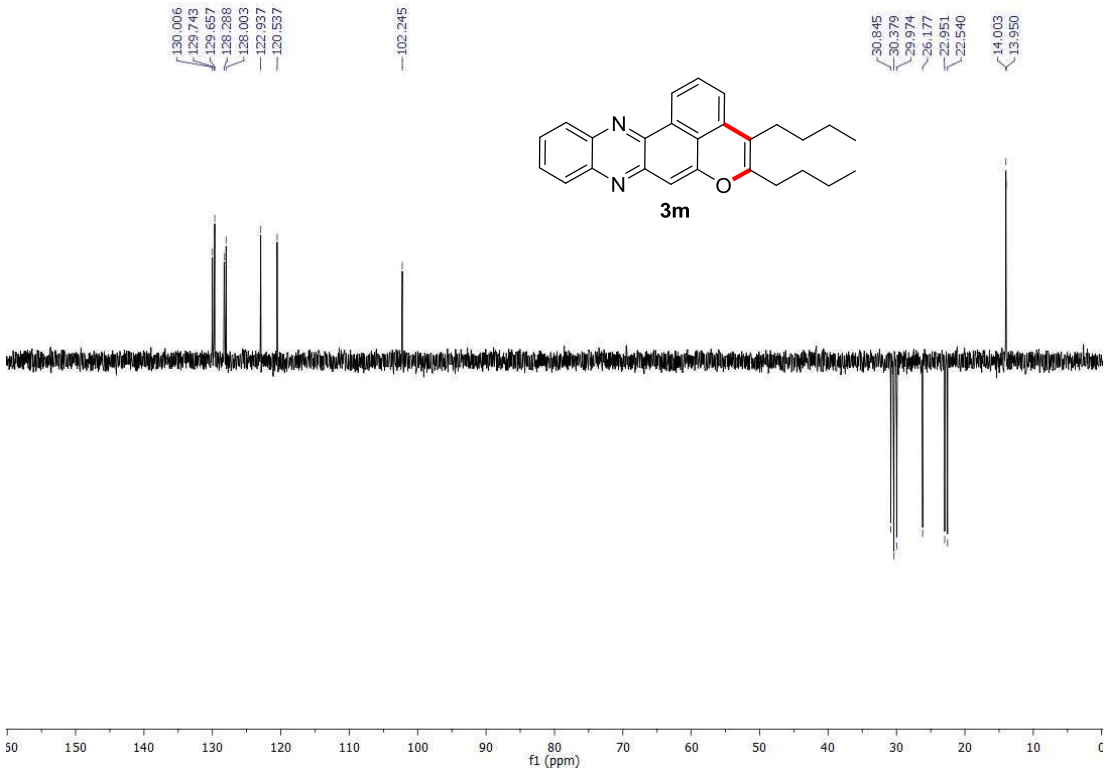

Figure S107. *DEPT-135 NMR spectrum of synthesized compound 3m in $CDCl_3$*

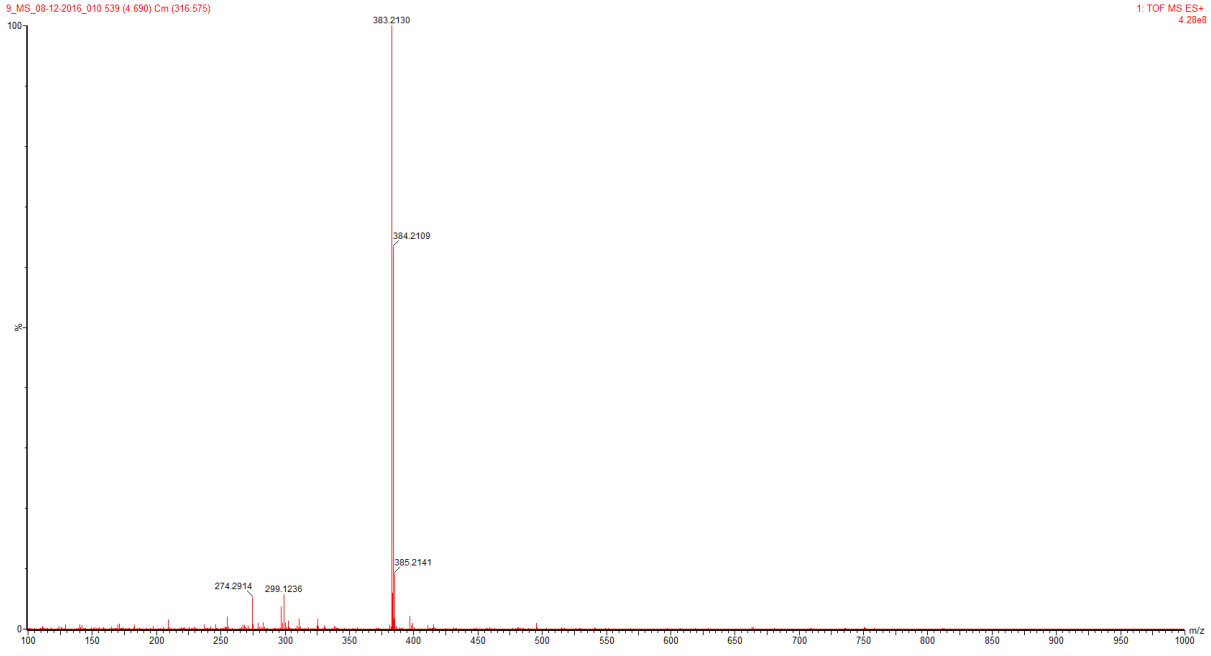

Figure S108. *HRMS spectrum of synthesized compound 3m*

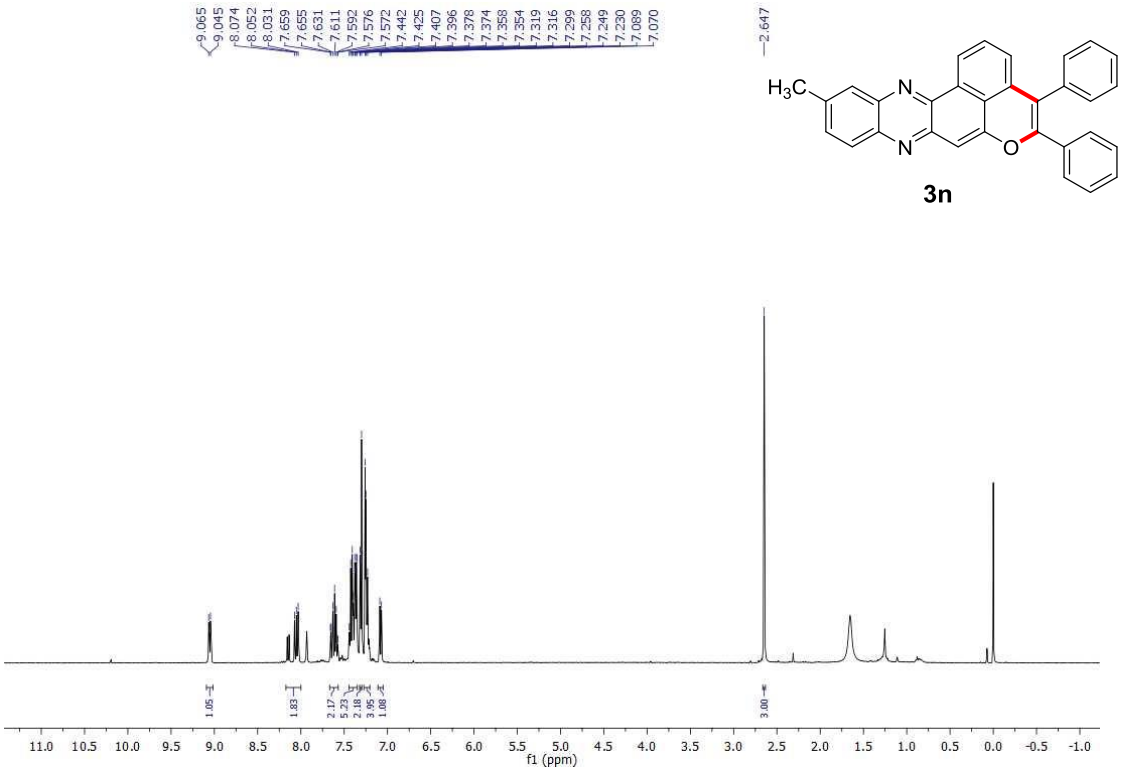

Figure S109. *^{1}H NMR spectrum of synthesized compound 3n in $CDCl_3$*

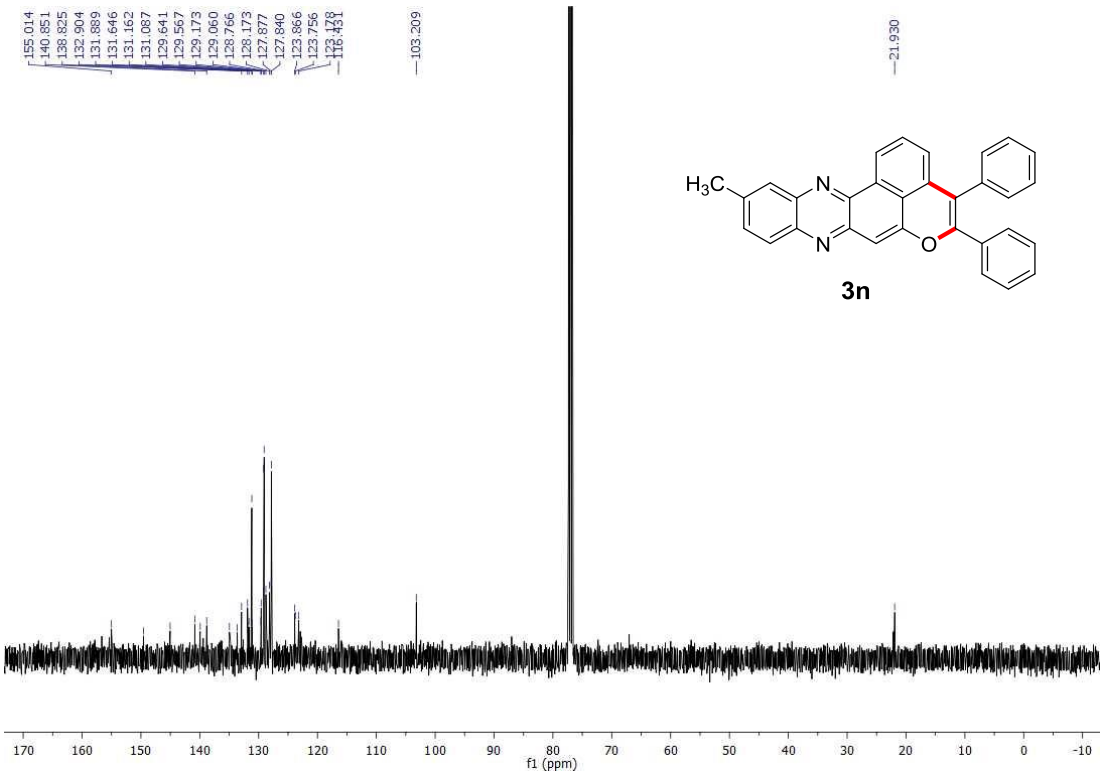

Figure S110. *^{13}C NMR spectrum of synthesized compound 3n in $CDCl_3$*

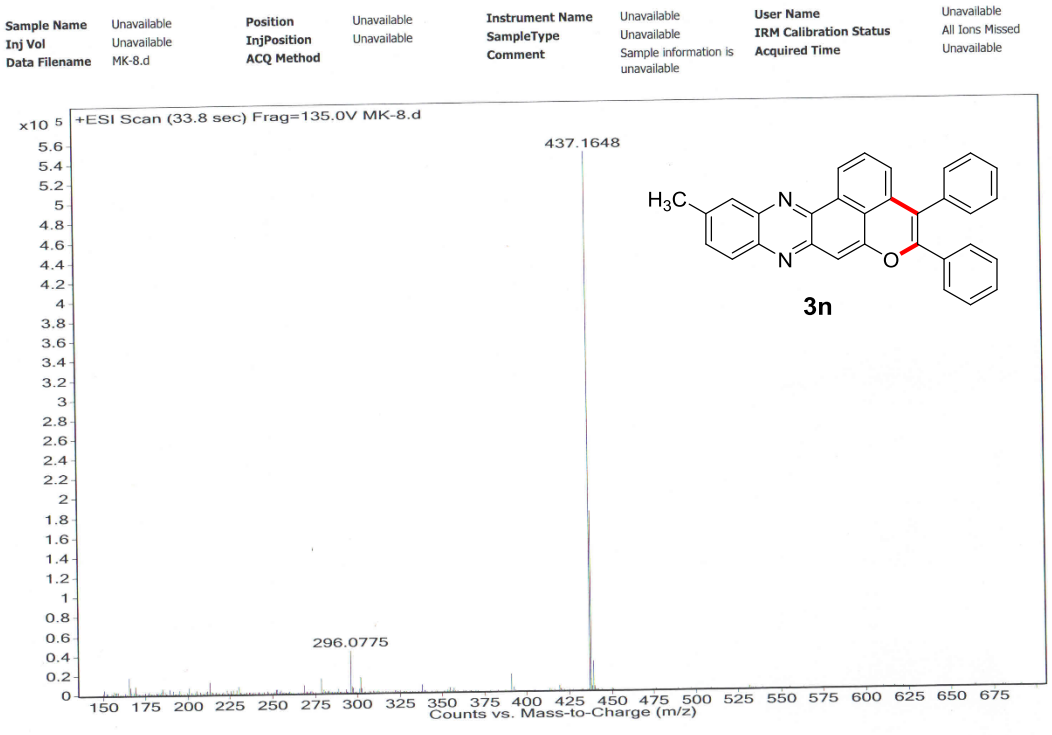

Figure S111. *HRMS spectrum of synthesized compound 3n*

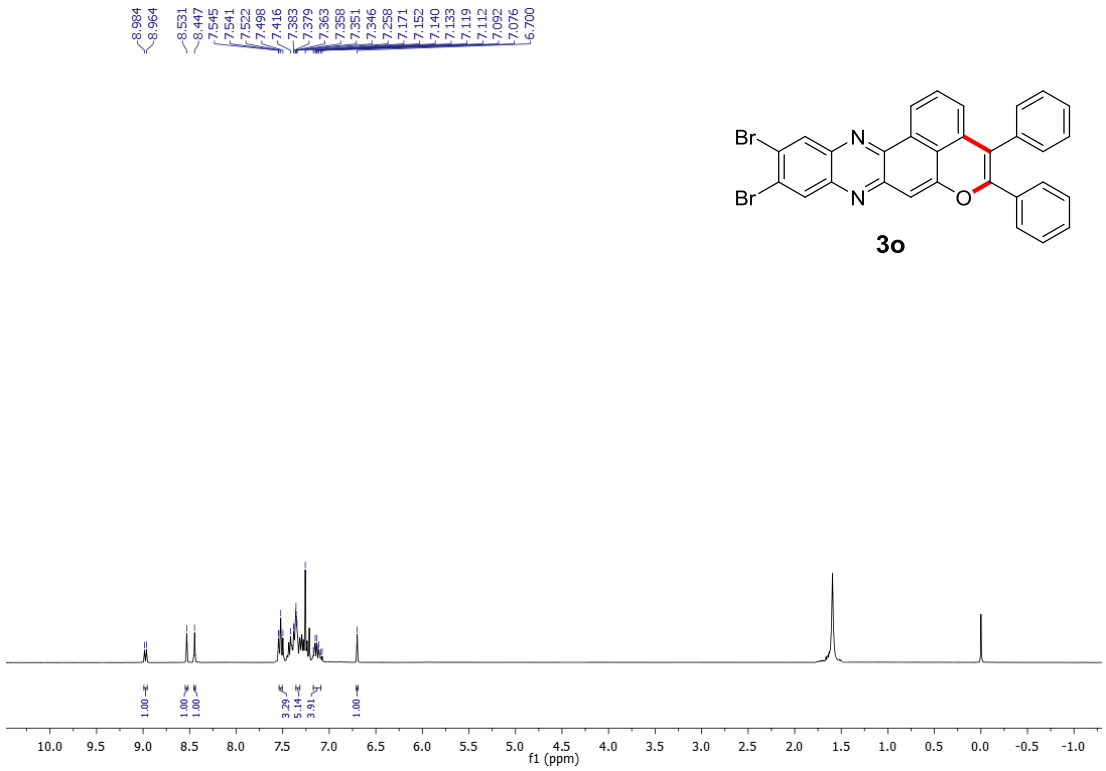

Figure S112. *^{1}H NMR spectrum of synthesized compound 3o in $CDCl_3$*

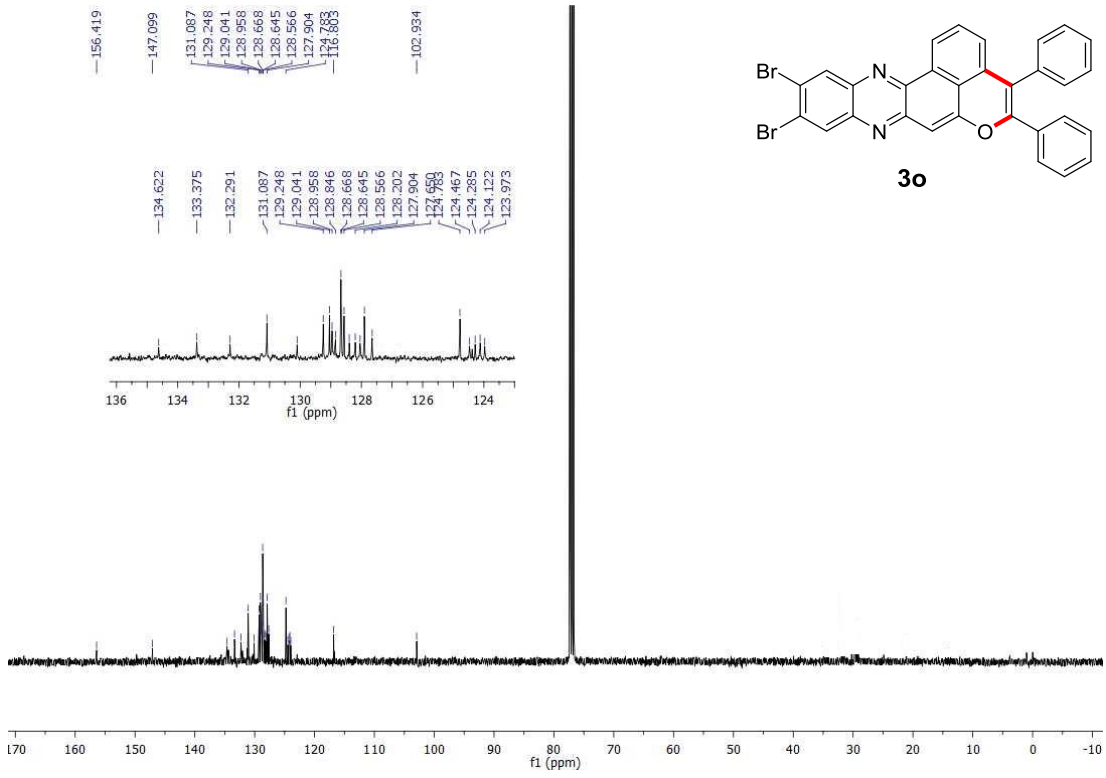

Figure S113. *^{13}C NMR spectrum of synthesized compound 3o in $CDCl_3$*

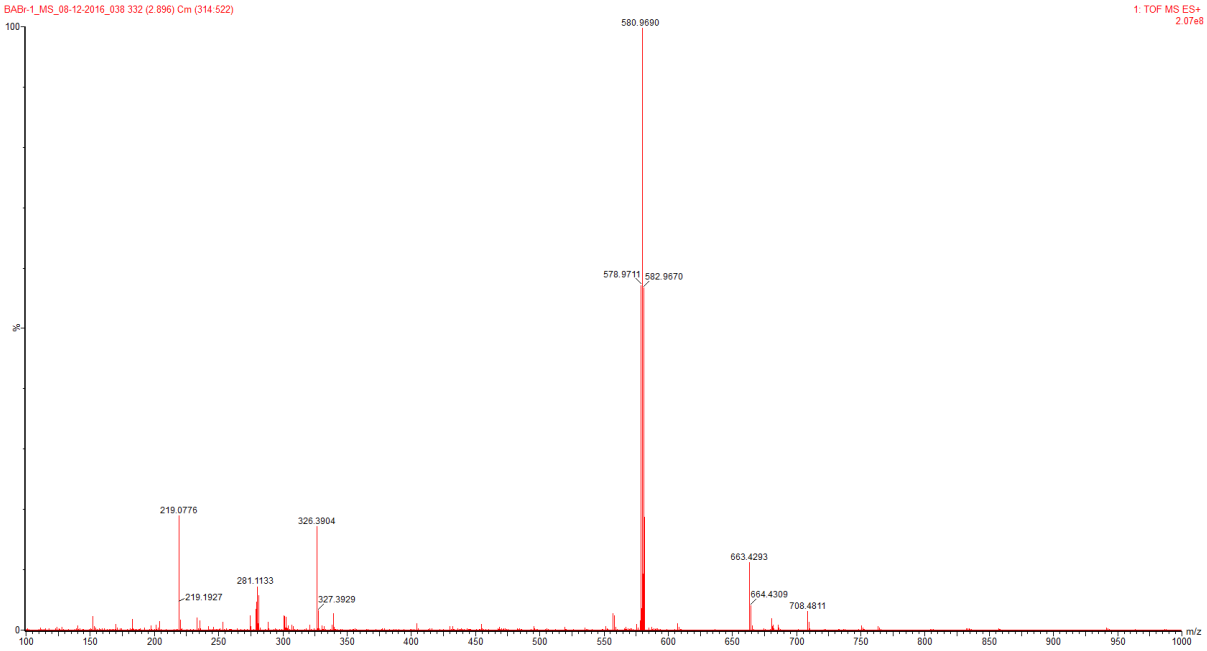

Figure S114. *HRMS spectrum of synthesized compound 3o*

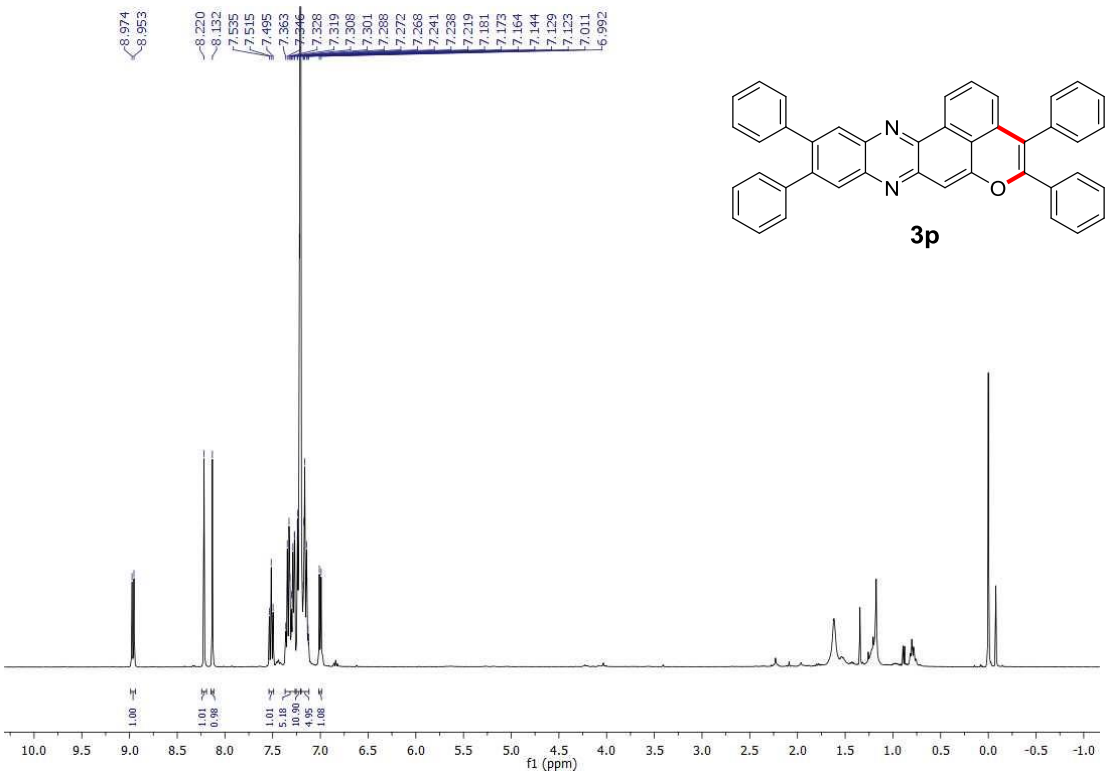

Figure S115. *[1]H NMR spectrum of synthesized compound 3p in $CDCl_3$*

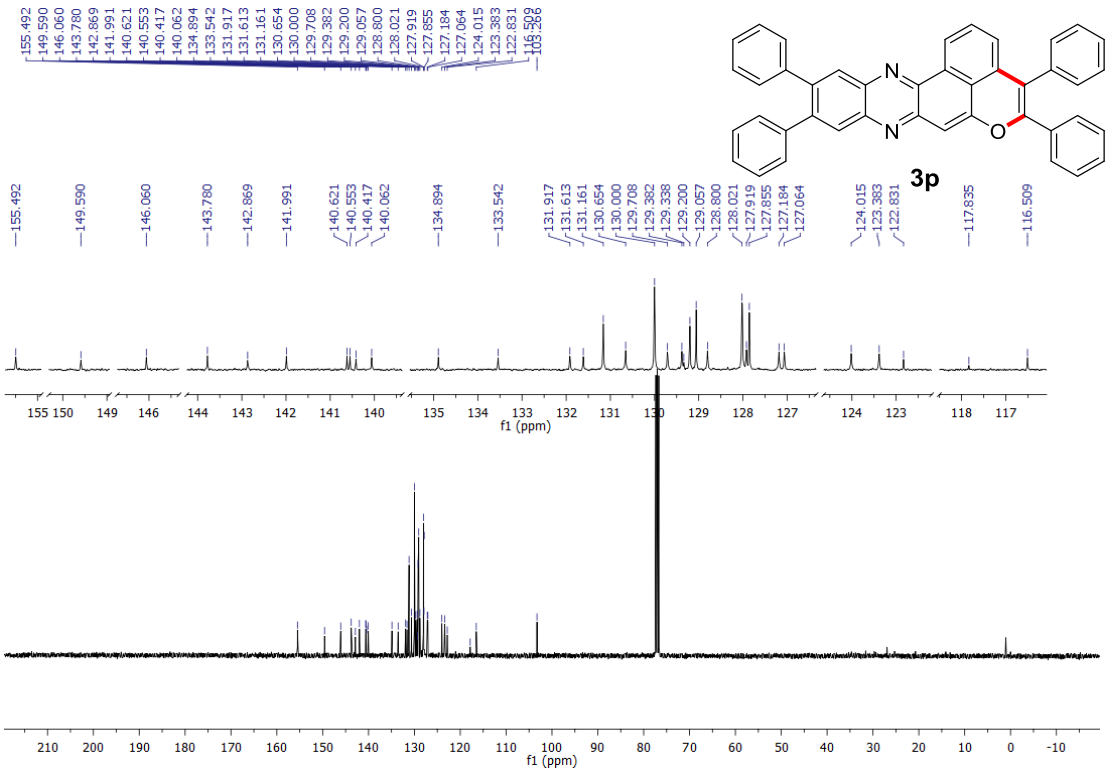

Figure S116. *[13]C NMR spectrum of synthesized compound 3p in $CDCl_3$*

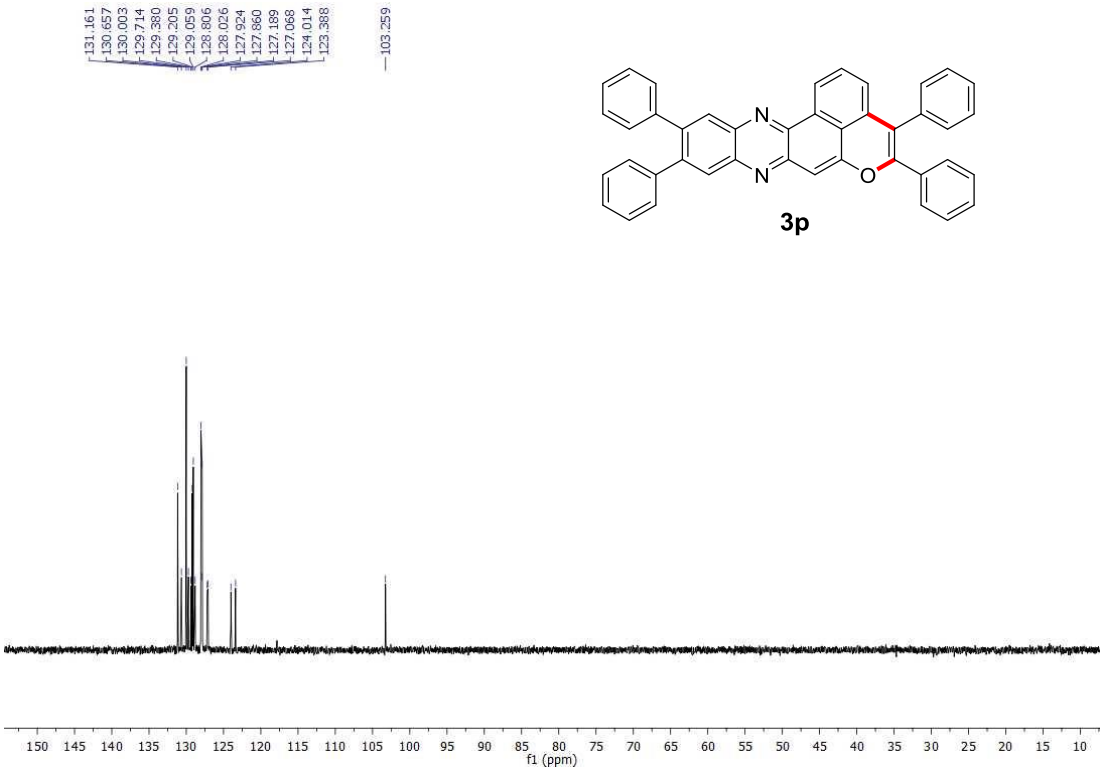

Figure S117. *DEPT-135 NMR spectrum of synthesized compound 3p in $CDCl_3$*

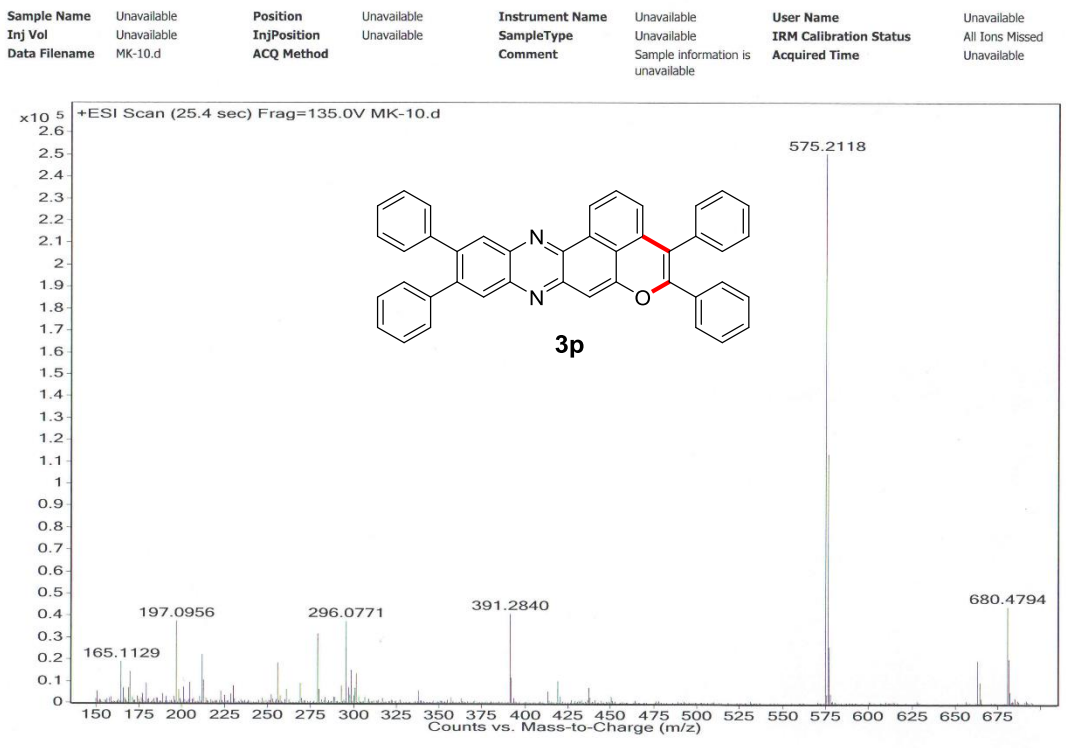

Figure S118. *HRMS spectrum of synthesized compound 3p*

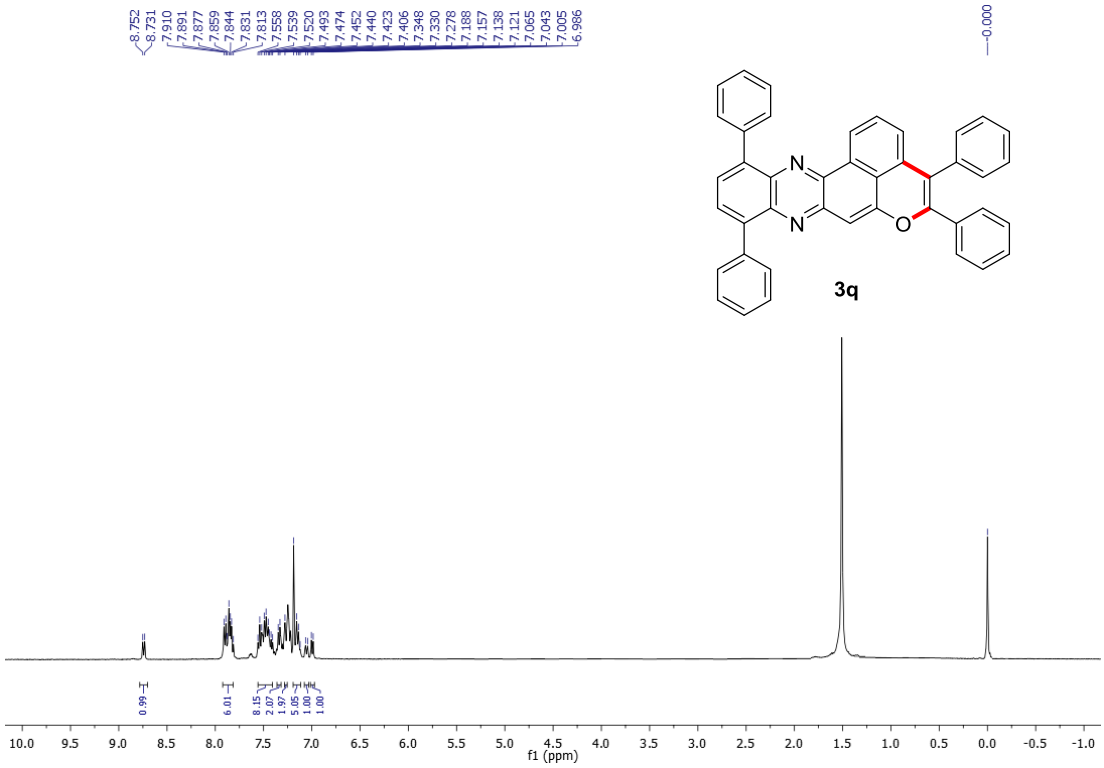

Figure S119. *^{1}H NMR spectrum of synthesized compound 3q in $CDCl_3$*

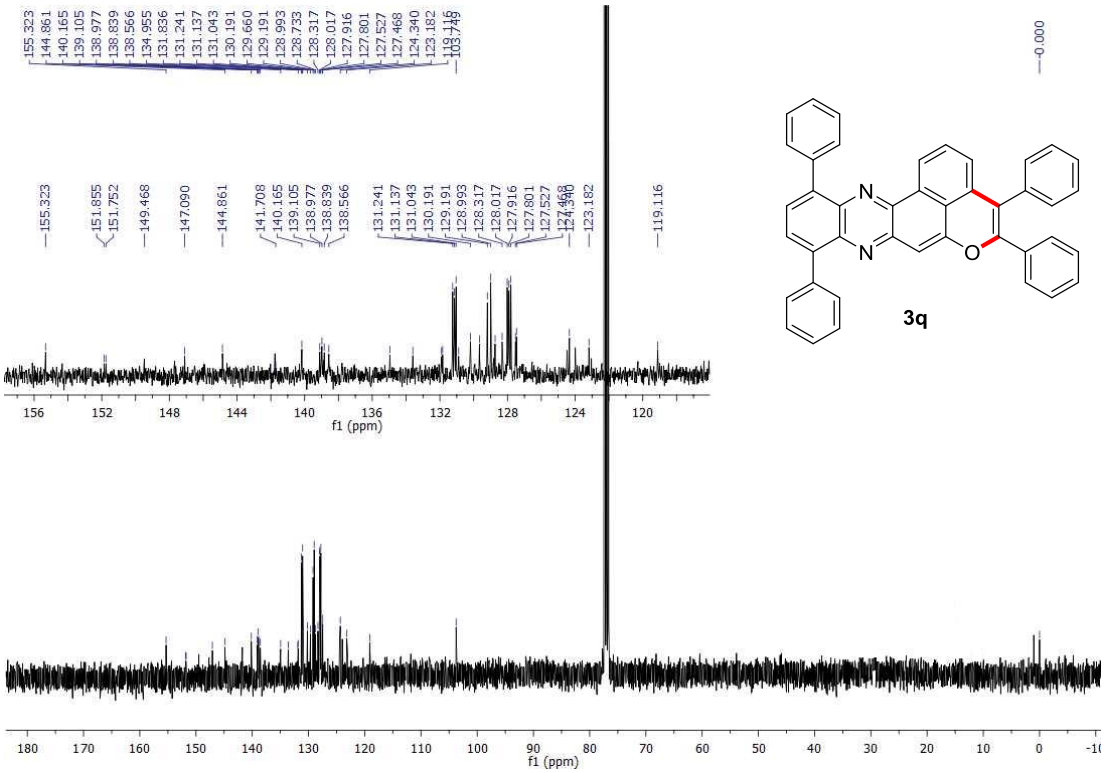

Figure S120. *^{13}C NMR spectrum of synthesized compound 3q in $CDCl_3$*

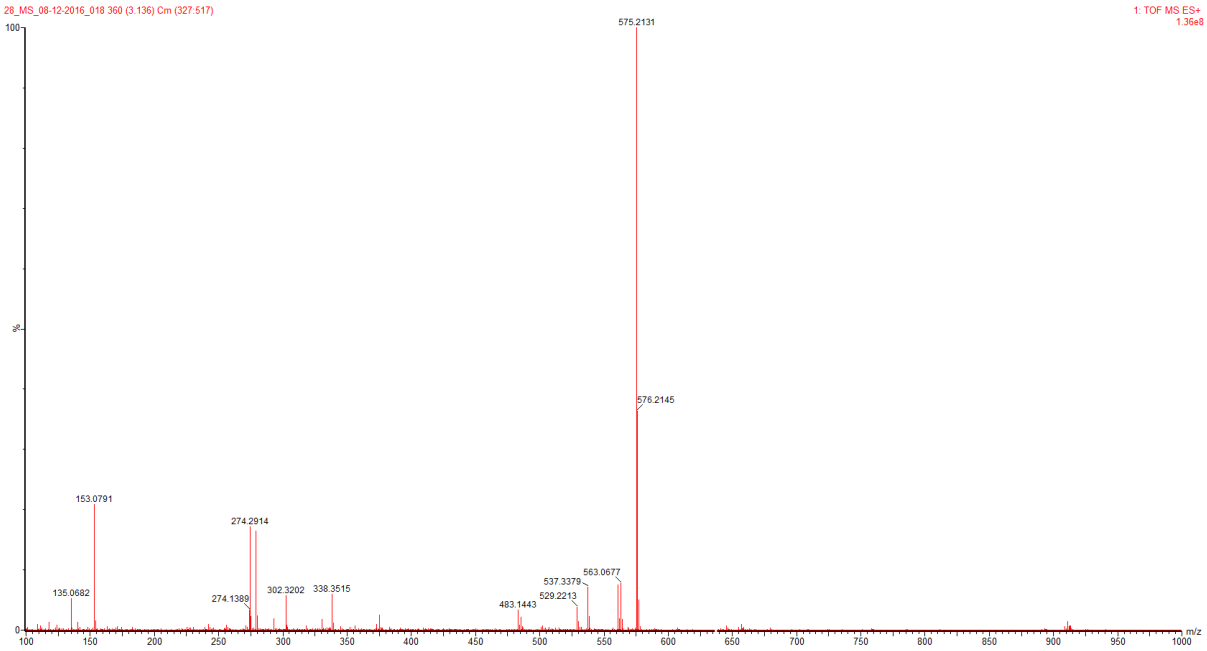

Figure S121. *HRMS spectrum of synthesized compound 3q*

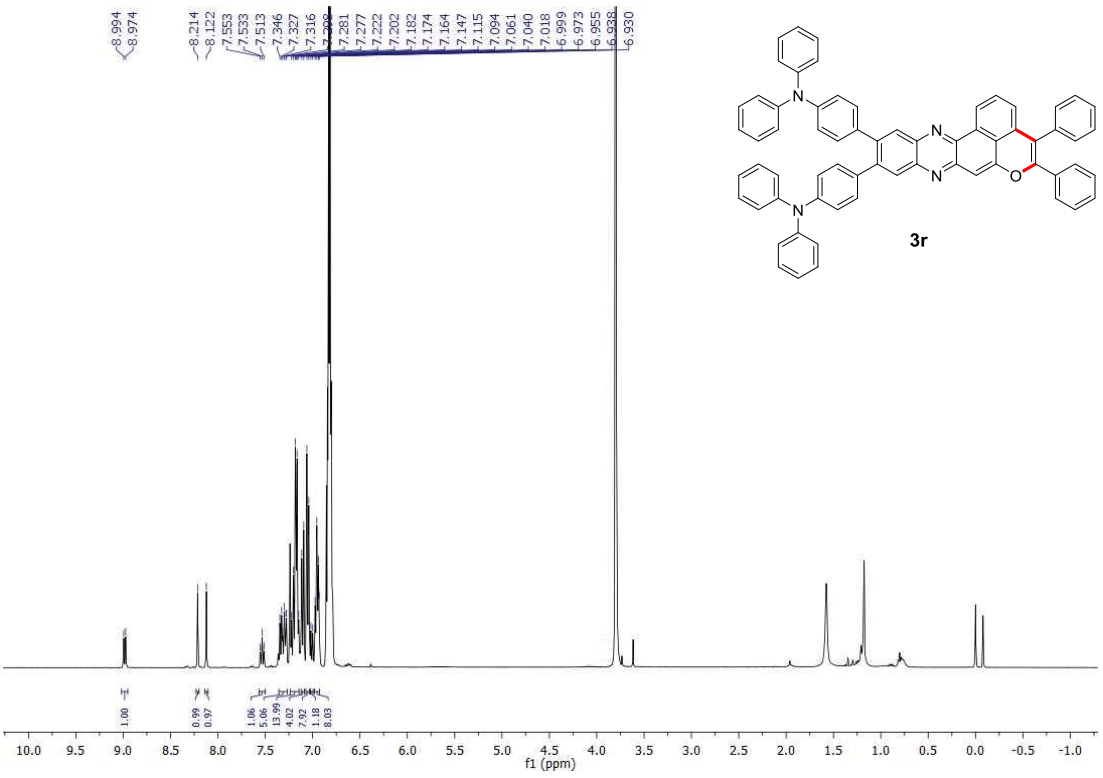

Figure S122. *^{1}H NMR spectrum of synthesized compound 3r in $CDCl_3$*

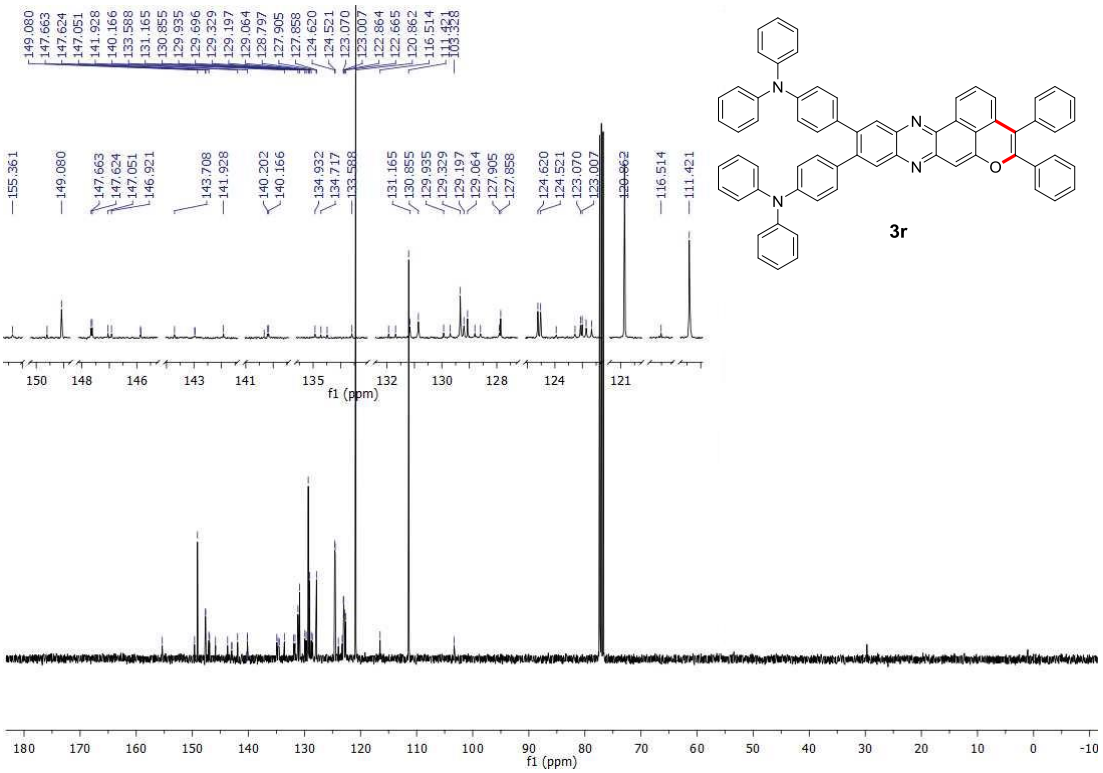

Figure S123. *^{13}C NMR spectrum of synthesized compound 3r in $CDCl_3$*

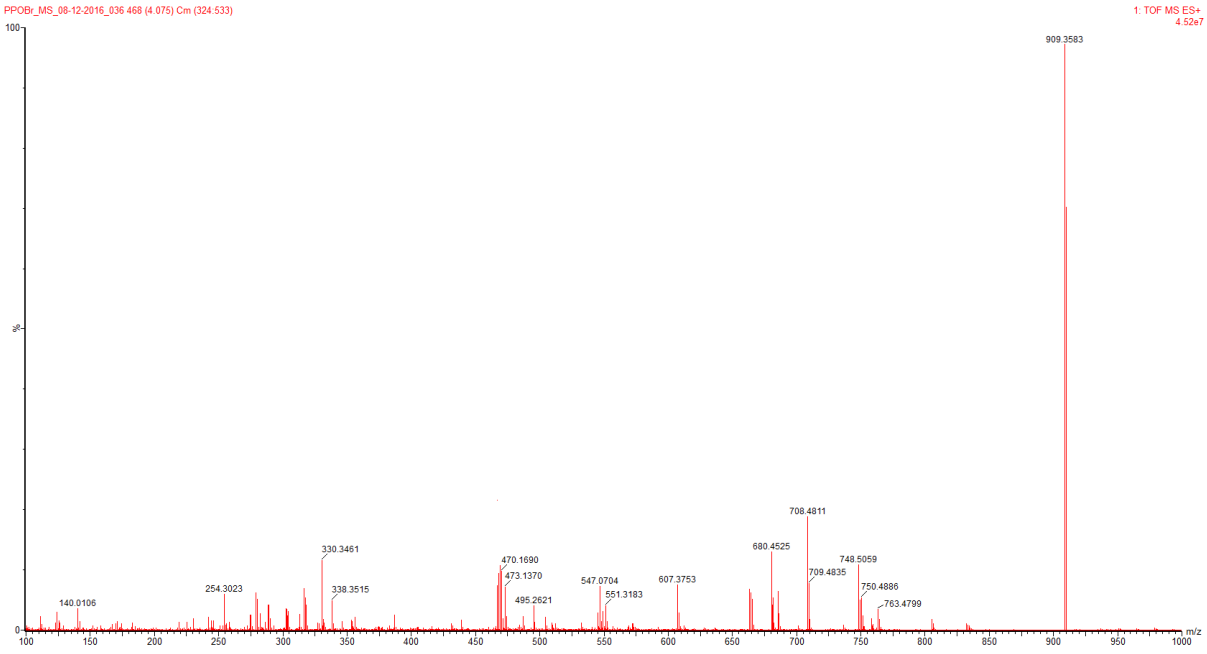

Figure S124. *HRMS spectrum of synthesized compound 3r*

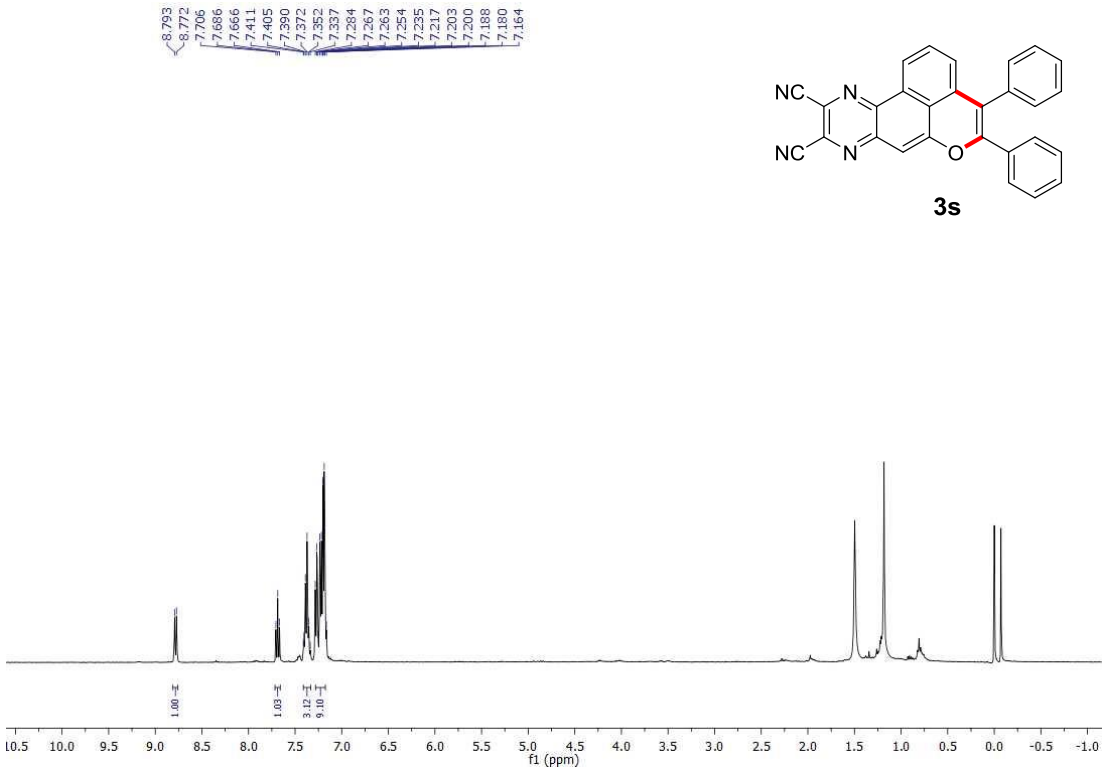

Figure S125. *^{1}H NMR spectrum of synthesized compound 3s in $CDCl_3$*

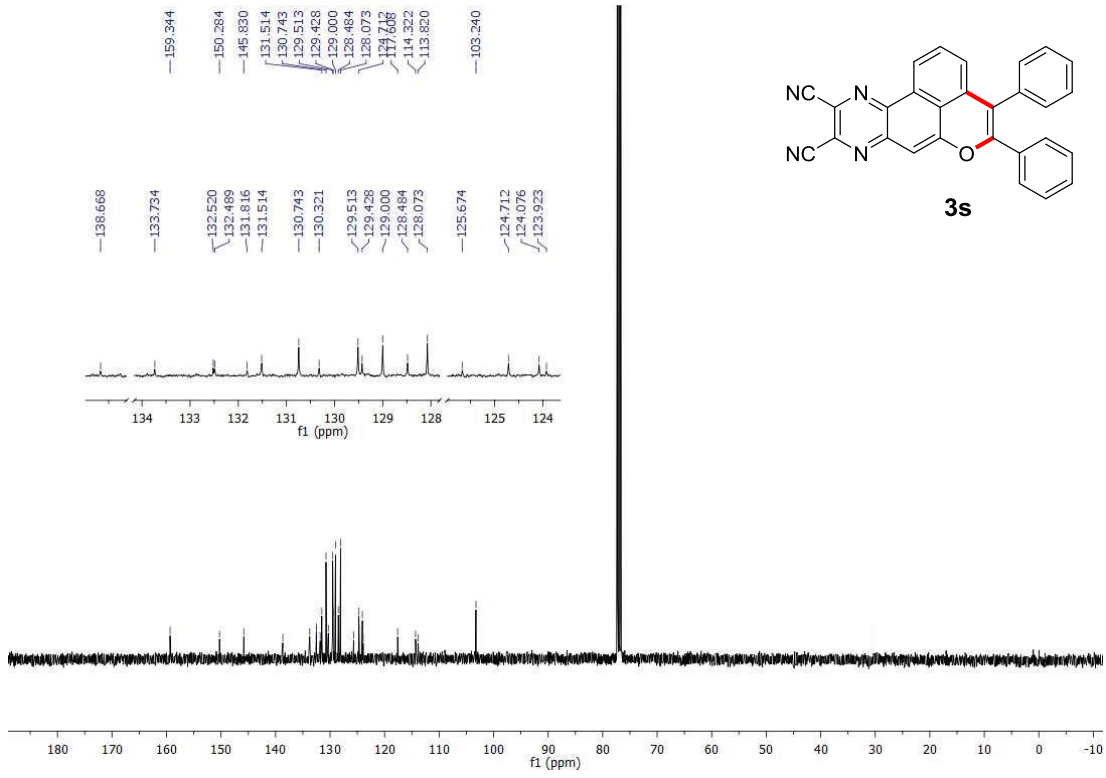

Figure S126. *^{13}C NMR spectrum of synthesized compound 3s in $CDCl_3$*

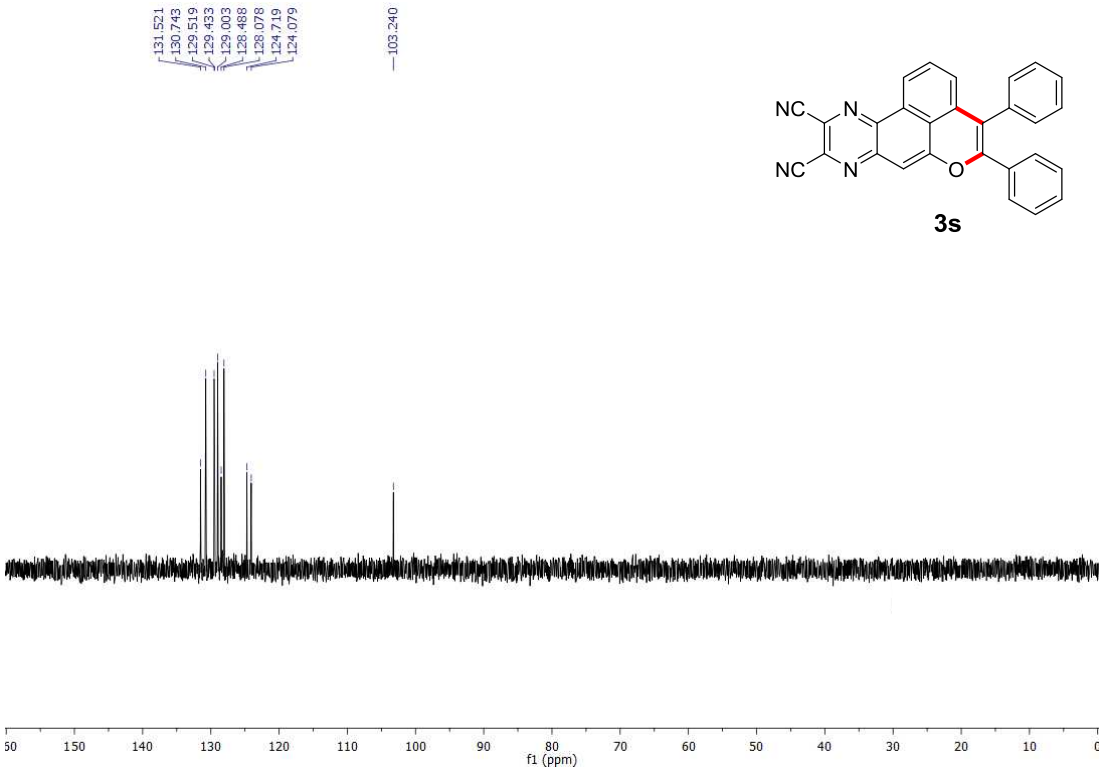

Figure S127. *DEPT-135 NMR spectrum of synthesized compound 3s in $CDCl_3$*

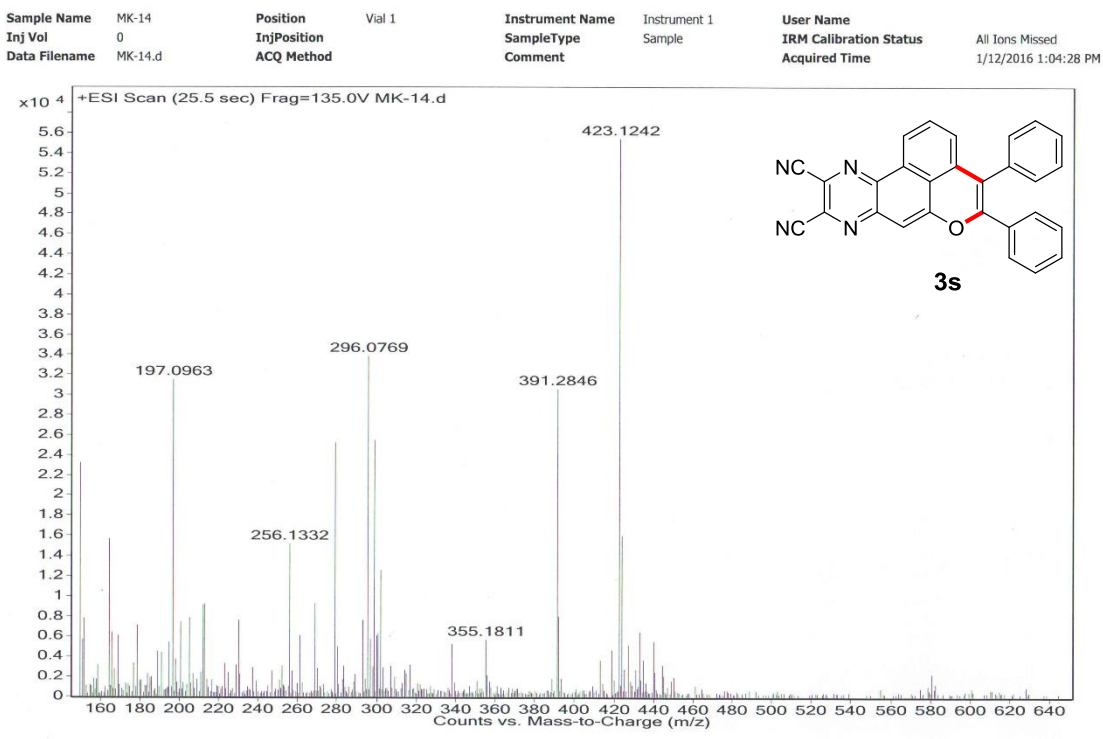

Figure S128. *HRMS spectrum of synthesized compound 3s*

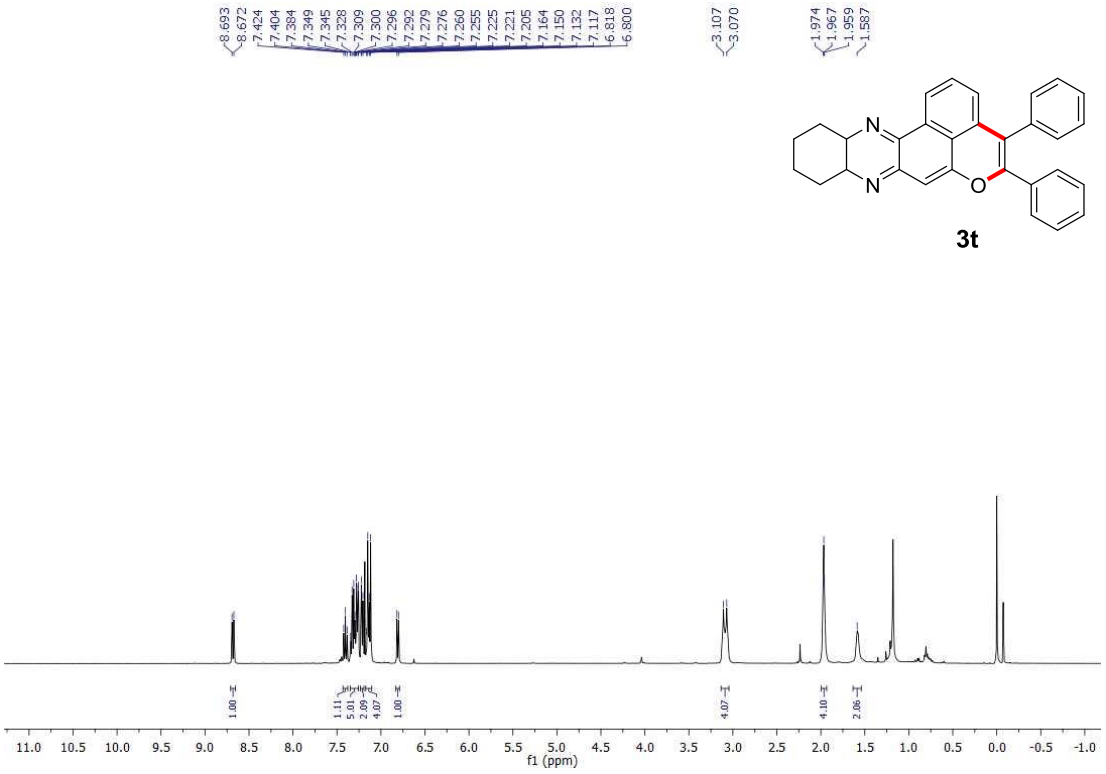

Figure 129. *^{1}H NMR spectrum of synthesized compound 3t in $CDCl_3$*

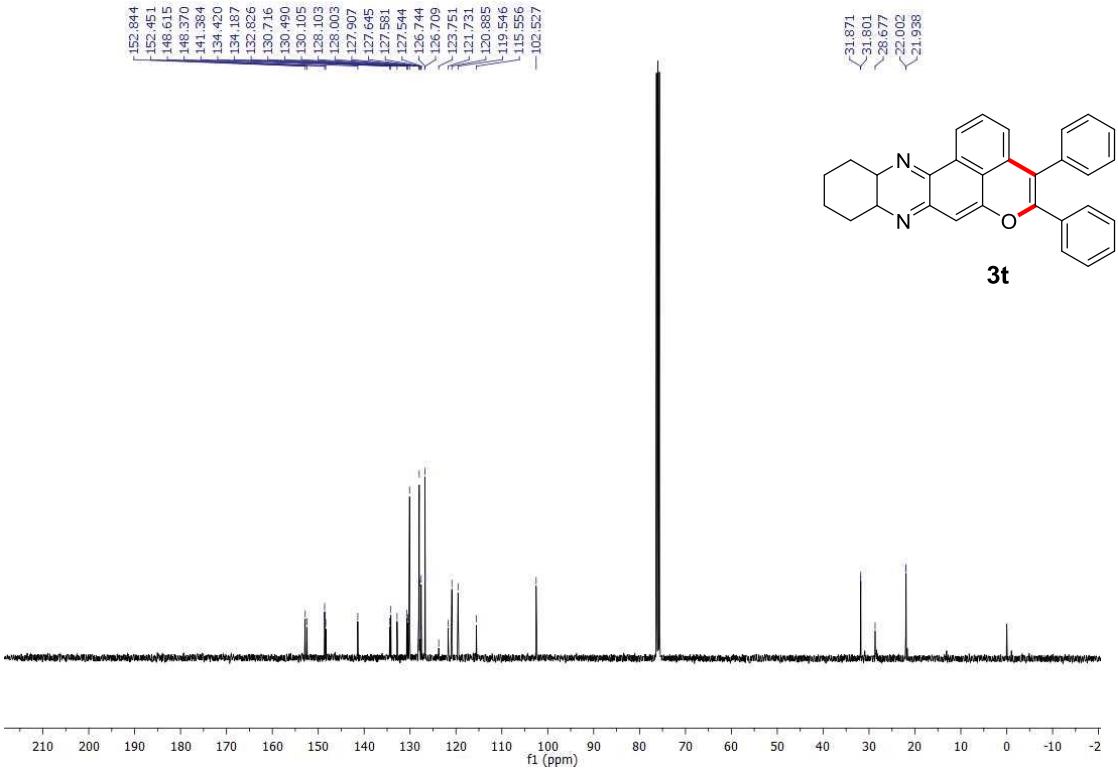

Figure S130. *^{13}C NMR spectrum of synthesized compound 3t in $CDCl_3$*

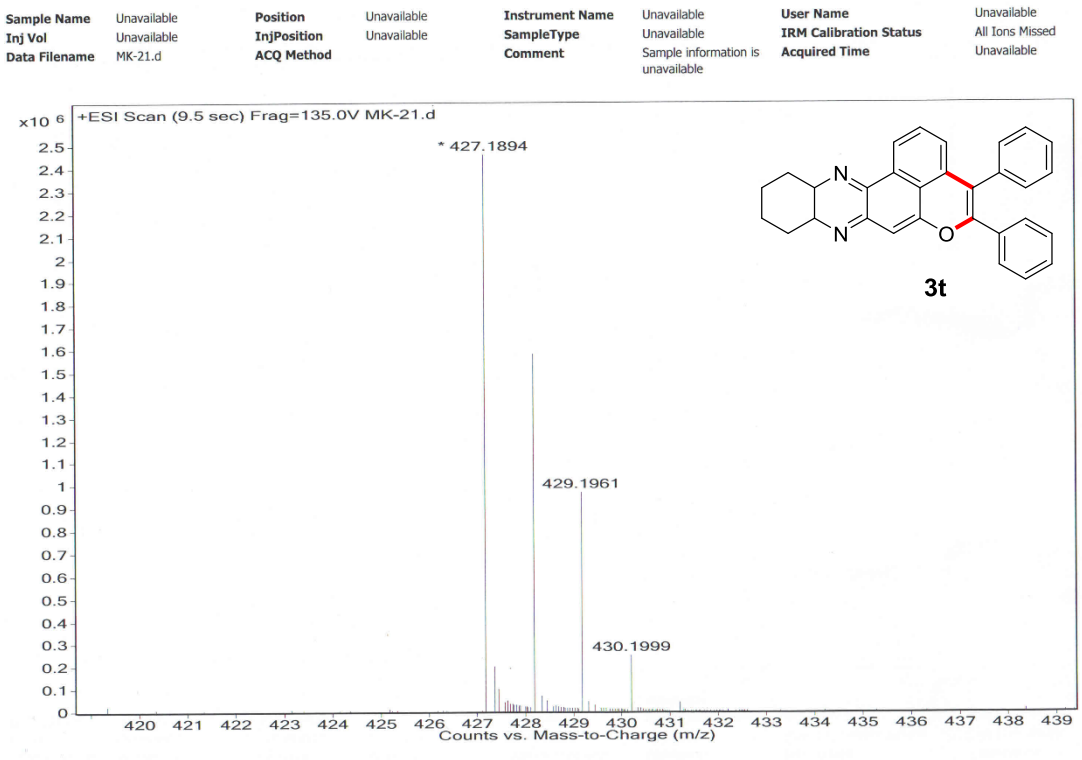

Figure S131. *HRMS spectrum of synthesized compound 3t*

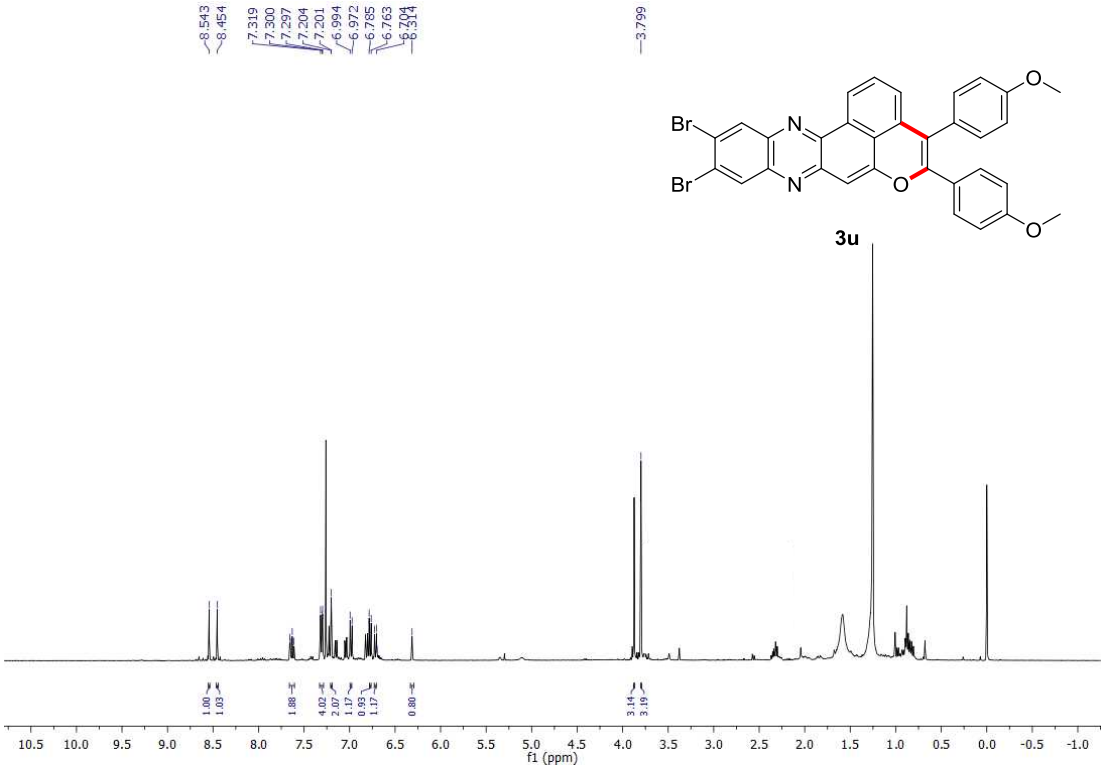

Figure S132. *^{1}H NMR spectrum of synthesized compound 3u in $CDCl_3$*

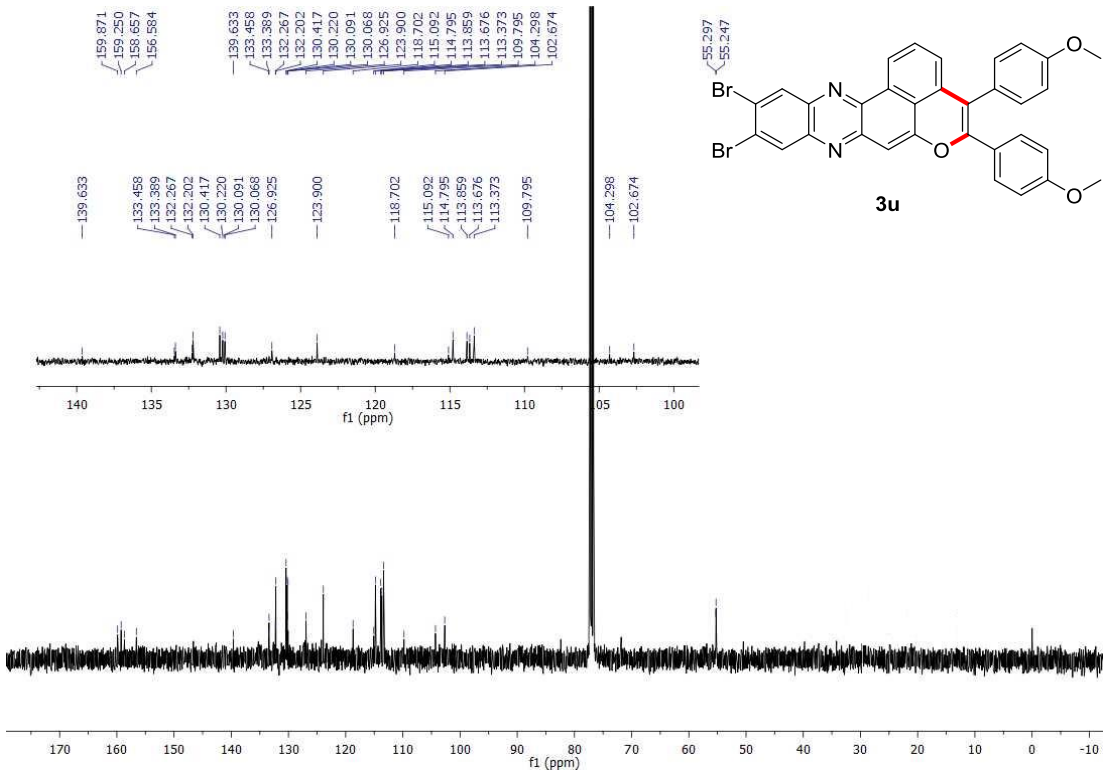

Figure S133. *^{13}C NMR spectrum of synthesized compound 3u in $CDCl_3$*

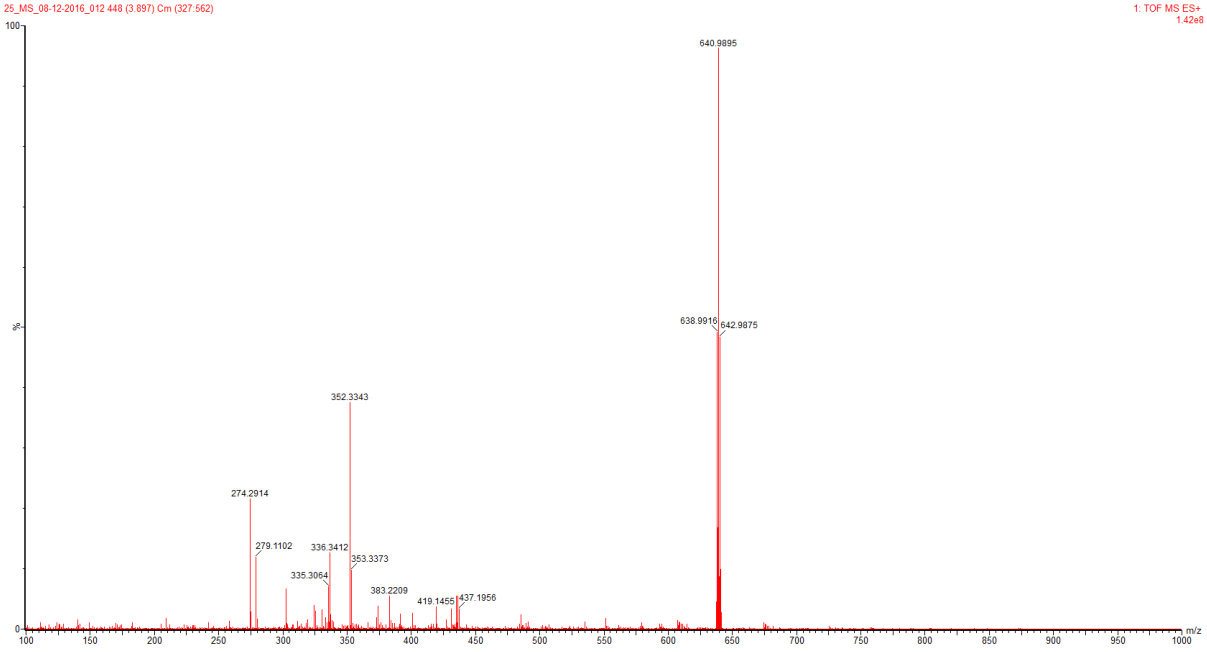

Figure S134. *HRMS spectrum of synthesized compound 3u*

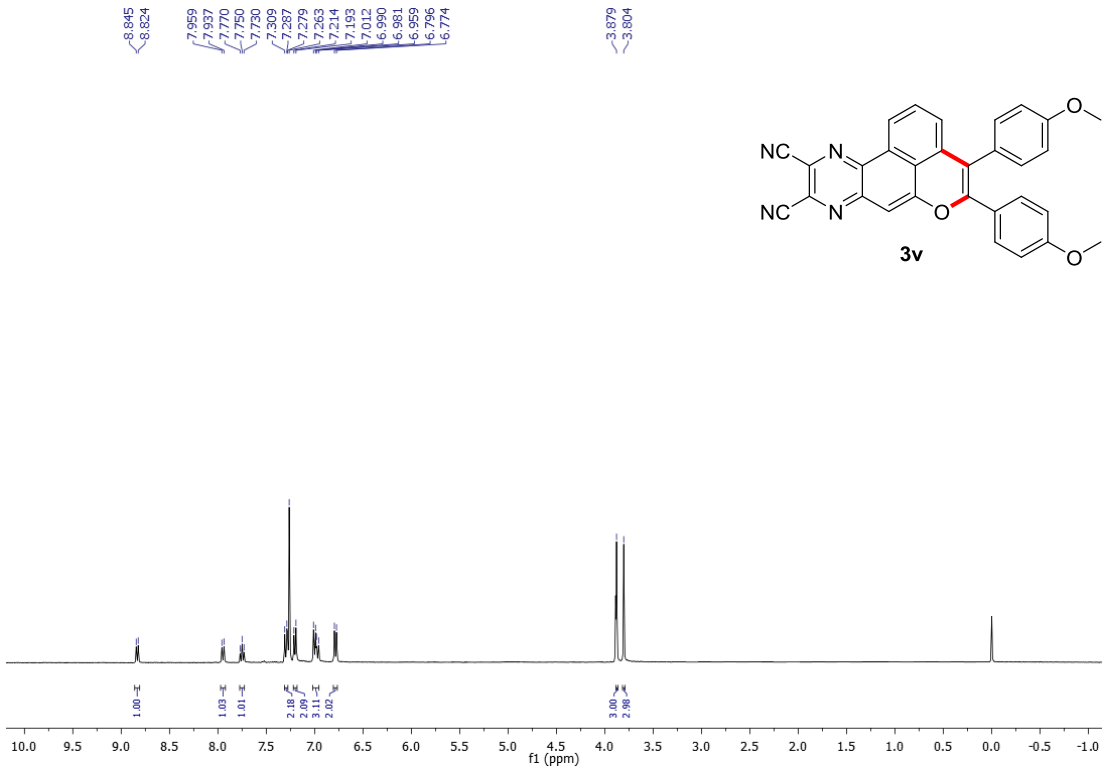

Figure S135. *^{1}H NMR spectrum of synthesized compound 3v in $CDCl_3$*

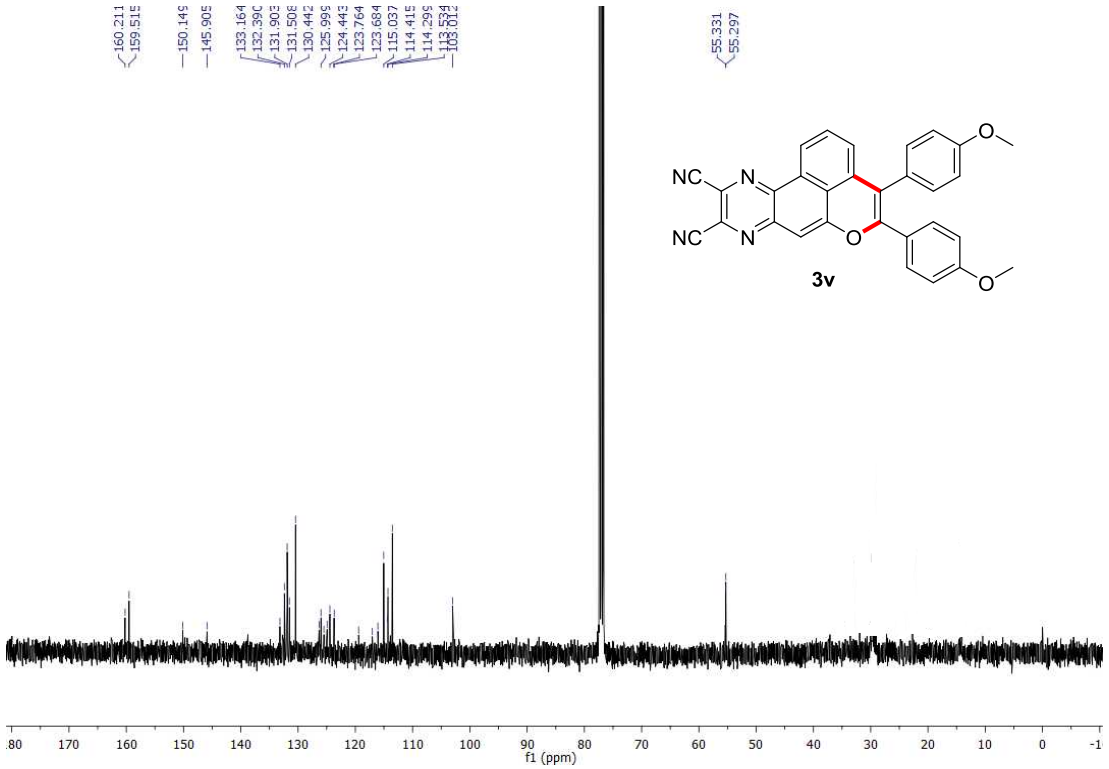

Figure S136. *^{13}C NMR spectrum of synthesized compound 3v in $CDCl_3$*

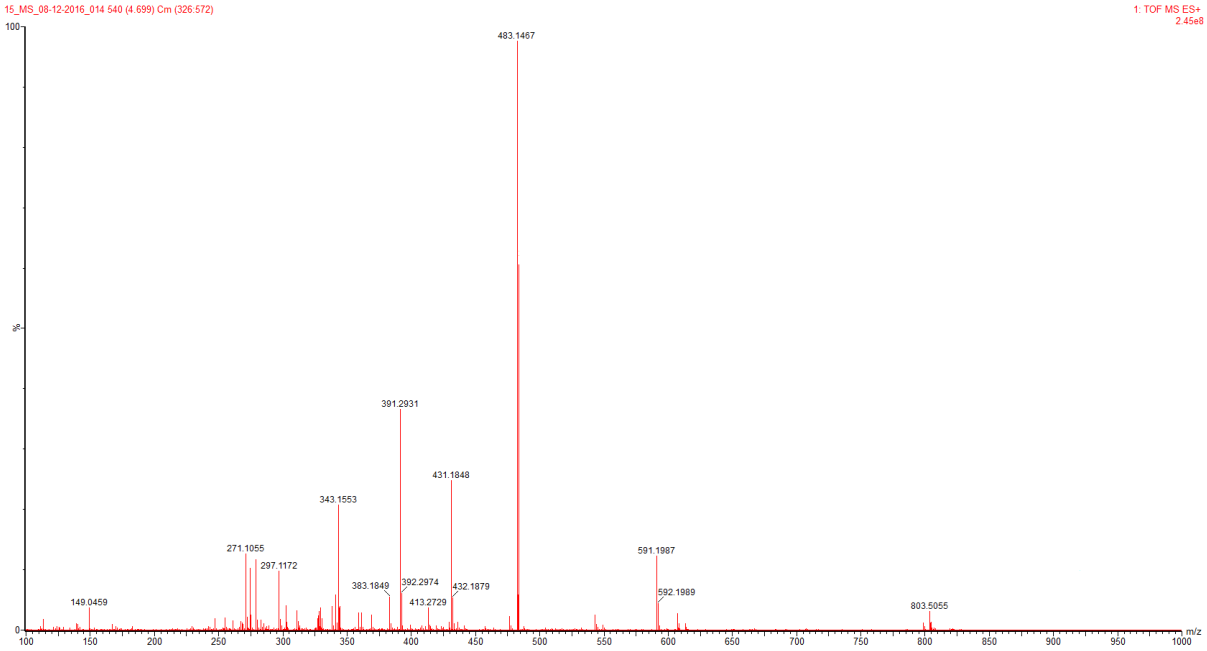

Figure S137. *HRMS spectrum of synthesized compound 3v*

22. References

1. R. C. Elderfield, V. B. Meyer, *J. Am. Chem. Soc*. 1954, *76*, 1887-1891.
2. G. W. H. Cheeseman, *J. Chem. Soc*. **1962**, 1170-1176.
3. B. Gao, Y. Liu, Y. Geng, Y. Cheng, L. Wang, X. Jing, F. Wang, *Tetrahedron Lett.* **2009**, *50*, 1649-1652.
4. K. Park, G. Bae, J. Moon, J. Choe, K. H. Song, S. Lee, *J. Org. Chem.* **2010**, *75*, 6244.