Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

For

$FeCl_3$ promoted ring size dictating diversity-oriented synthesis (DOS) of *N*-heterocycles using *in situ* generated cyclic imines and enamines

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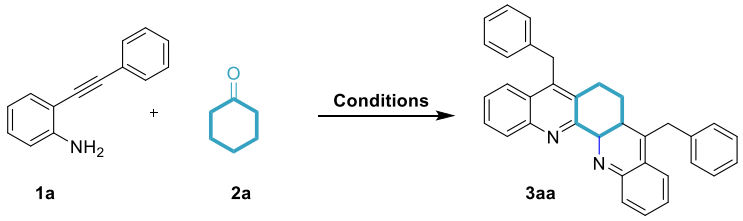
Table of Contents

1) General Information

Starting materials were prepared according to the literature reported protocols.[1] ^{1}H, ^{13}C and DEPT NMR spectra were recorded on a 400 MHz Varian Unity Plus or Varian Mercury plus spectrometer or JEOL 400 MHz. The chemical shift (δ) values are reported in parts per million (ppm), and the coupling constants (*J*) are given in Hz. The spectra were recorded using $CDCl_3$ as a solvent. ^{1}H NMR chemical shifts are referenced to tetramethylsilane (TMS) (0 ppm). ^{13}C NMR was referenced to $CDCl_3$ (77.0 ppm). The abbreviations used are as follows: s, singlet; d, doublet; t, triplet; q, quartet; dd, doublet of doublet; ddd, doublet of doublet of doublet; dt, doublet of triplets; td, triplet of doublet; m, multiplet. Mass spectra and high-resolution mass spectra (HRMS) was measured using the ESI (FT-MS solariX) at National Sun Yat-Sen University, Kaohsiung, Taiwan or LTQ Orbitrap XL (Thermo Fischer Scientific) at National Chung Hsing University. Melting points were determined on an EZ-Melt (Automated melting point apparatus). All products reported showed ^{1}H NMR spectra in agreement with the assigned structures. Reaction progress and product mixtures were routinely monitored by TLC using Merck TLC aluminum sheets (silica gel 60 F254). Column chromatography was carried out with 230–400 mesh silica gel 60 (Merck)/neutral alumina and a mixture of hexane/ethyl acetate or hexane as an eluent.

2) Studies on reaction parameters

Table -S1 Optimization of reaction conditions for dibenzo[*b,j*][1,10]phenanthrolines [a,b]



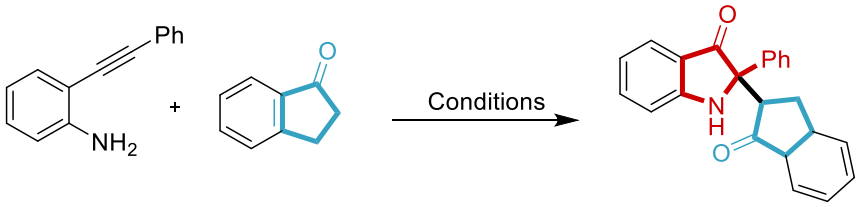
1a 2a 3aa

Entry	Lewis acids (equiv.)	Oxidant/Additive (equiv.)	Solvent	Time, h	Yield (%)[b]
1	$FeCl_3$ (0.2)	--	DMSO	10	<10
2[c]	$FeCl_3$ (0.2)	O_2	DMSO	14	24
3[d]	$FeCl_3$ (0.2)	TBHP(2.0)	DMSO	8	trace
4[e]	$FeCl_3$ (0.2)	DTBP(2.0)	DMSO	11	13
5[f]	$FeCl_3$ (0.2)	NCS(1.2)	DMSO	8	22
6	$FeCl_3$ (0.5)	--	DMSO	14	45
7	$FeCl_3$ (1.0)	--	DMSO	10	58
8	**$FeCl_3$ (1.5)**	**--**	**DMSO**	**8**	**83**
9	$FeCl_3$ (2.0)	--	DMSO	8	62
10	$FeCl_3.6\ H_2O$ (1.5)	--	DMSO	8	47
11	$FeCl_2$ (1.5)	--	DMSO	8	15
12	$FeBr_3$ (1.5)		DMSO	14	0
13	$AlCl_3$ (1.5)	--	DMSO	14	40
14	I_2 (1)	--	DMSO	14	0
15	$FeCl_3$ (1.5)	CH_3COOH (2.0)	DMSO	8	45
16	$FeCl_3$ (1.5)	C_6H_5COOH (2.0)	DMSO	9	66
17	$FeCl_3$ (1.5)	DDQ	DMSO	14	0
18[g]	--	--	DMSO	14	0
19	$FeCl_3$ (1.5)	--	Ethanol	10	40
20	$FeCl_3$ (1.5)	--	DMF	14	traces
21	$FeCl_3$ (1.5)	--	Toluene	14	0
22	$FeCl_3$ (1.5)	--	Dioxane	14	0
23[h]	$FeCl_3$ (1.5)	--	H_2O	14	0
24[I]	$FeCl_3$ (1.5)	--	DMSO	14	50
25	Zn, In, Cu, chlorides		DMSO	14	0

[a] All reactions were carried out using **1a** (2.1 mmol), **2a** (1.0 mmol), $FeCl_3$ (1.5 mmol) and solvent (0.5 mL) at 110 °C and the mixtures were stirred at indicated time unless otherwise noted. [b] Isolated yield. [c] reaction performed under O_2 atmosphere. [d] both 70% Aq. TBHP and 5.5 M TBHP in decane were used. [e] Di-*tert*-butyl-peroxide (DTBP). [f] *N*-Chlorosuccinimide. [g] reaction performed without $FeCl_3$. [h] 10 equiv. DMSO was used. [I] Reaction performed at 90 °C.

Initially, the reaction between 2-ethynyl aniline (**1a**) and cyclohexanone (**2a**) in the presence of 20 mol% $FeCl_3$ in DMSO at 110 °C afforded the desired product **3aa** less than 10% (Table-1, entry 1). Even though we introduce different oxidants or additives in reaction conditions, it couldn't help to improve the yield of the reaction (Table-1, entry 2-5). When we increased the $FeCl_3$ quantity from a catalytic amount to stoichiometric amount, (Table-1, entry 6-8) considerable improvement in the reaction yield was observed (Table-1, entry 8). However, further increasing the amount of $FeCl_3$ to 2 equivalent suppressed product formation (Table-1, entry 9). We screened the reaction with different iron salts such as $FeCl_3.6H_2O$, $FeCl_2$, $FeBr_3$ (Table-1, entry 10-12) and these all produce inferior yields. It confirmed the anhydrous $FeCl_3$ is the best choice for this reaction (Table-1, entry 8). Replacing $FeCl_3$ with $AlCl_3$ in the reaction medium resulted in 40% of **3aa**. Stoichiometric amount of I_2 failed to promote the reaction (Table-1, entry 14). The addition of CH_3COOH and C_6H_5COOH in the reaction provided the moderate yield of 44 and 65% (Table-1, entry 15-16). Subsequent investigation of the reaction under the stoichiometric amount of DDQ ended up with sluggish reaction mixture (Table-1, entry 17). In the absence of $FeCl_3$, **3aa** was not formed, which confirmed the role of $FeCl_3$ to promote the reaction (Table-1, entry 18). Further, we examined the reaction with different solvents, we found only ethanol to afford the 40% of the required product (Table-1, entry 19-23). While the reaction was carried out in the slightly lower temperature (90 °C), product formation was suppressed (Table-1, entry 24). Other common metal chlorides such as Cu, Zn, In were inferior to activate the reaction (Table-1, entry 25).

Table -S2 Optimization of reaction conditions for 2(-inden-2-yl)-3-oxo-indolines derivatives[a]



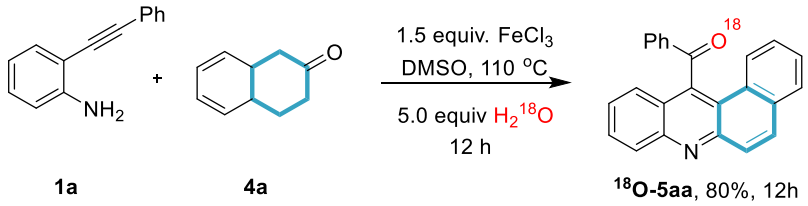
1a **6a** **7aa**

Entry	Lewis acids (equiv.)	Solvent	Time, h	Yield (%)[b]
1	$FeCl_3$ (1.5)	DMSO	8	80
2	$FeCl_3$ (0.7)	DMSO	8	48
3	$FeCl_3$ (0.4)	DMSO	8	25
4	$FeCl_3$ (0.1)	DMSO	8	<10
5	$FeCl_3$ (2.0)	DMSO	8	75
6[c]	$FeCl_3$ (1.5)	DMSO	8	22
7[d]	$FeCl_3$ (0.1)	DMSO	8	ND
8[e]	**$FeCl_3$ (1.5)**	**DMSO**	**5**	**88**
9	$FeCl_3.6H_2O$ (1.5)	DMSO	10	20
10	$FeBr_3$ (1.5)	DMSO	10	ND
11	$Fe(acac)_3$ (1.5)	DMSO	10	ND
12	$Fe(OTf)_3$ (1.5)	DMSO	10	20
13	FePC (1.5)	DMSO	10	ND
14	$AlCl_3$ (1.5)	DMSO	10	ND
15	$InCl_3.H_2O$ (1.5)	DMSO	10	45
16	$AgNO_3$ (1.5)	DMSO	10	ND
17	$Cu(OTf)_2$ (1.5)	DMSO	10	<10
18	$BF_3.OEt_2$ (2)	DMSO	7	65
19	$ZnCl_2$ (1.5)	DMSO	10	ND
20	$FeCl_3$ (1.5)	DMF	10	ND
21	$FeCl_3$ (1.5)	Toluene	10	ND
22	$FeCl_3$ (1.5)	Dioxane	10	ND
23	$FeCl_3$ (1.5)	H_2O	10	ND
24	$FeCl_3$ (1.5)	DCE	10	ND
25	$FeCl_3$ (1.5)	*t*-Butanol	10	ND
26[f]	$FeCl_3$ (1.5)	DMSO	8	ND
27[g]	--	DMSO	8	ND

[a] All reactions were carried out using **1a** (1 mmol), **2a** (1.5 mmol), $FeCl_3$ (1.5 mmol) and solvent (1 mL) at 110 °C and reaction mixture were stirred at indicated time unless otherwise noted. [b] Isolated yield. [c] reaction performed under O_2 atmosphere. [d] both 70% Aq. TBHP and 5.5 M TBHP in decane was used. [e] Reaction performed under N_2 atmosphere. [f] Reaction carried out in 70 °C. [g] Without $FeCl_3$ reaction was performed.

3) Mechanistic studies

(1) ^{18}O isotope labeling experiment of compound **5aa**.



In an oven dried 15 ml seal tube 2-(phenylethynyl) aniline **1a** (193 mg, 1 mmol) was added in 1 ml of dry DMSO followed by the sequential addition of anhydrous $FeCl_3$ (243 mg, 1.5 mmol) β-tetralone **4a** (219 mg, 1.5 mmol) and $H_2{}^{18}O$ (90 µl, 5 equivalent). The reaction mixture was allowed to stir at 110 °C for 12 h. After the completion, the reaction mixture was cooled to room temperature and diluted with 25.0 mL of cooled water. The water layer was extracted with (3X25 mL) of ethyl acetate and the combined ethyl acetate layer was given brine wash (1X10 mL). The final ethyl acetate layer was dried over sodium sulfate and concentrated under reduced pressure to get the crude compound. The crude product submitted for GC-MS analysis. The obtained crude was purified using column chromatography by eluting from hexane to 8-15% ethyl acetate/hexane to afford pure of benzo[*a*]acridin-12-yl(phenyl)methanone as a brown color solid.

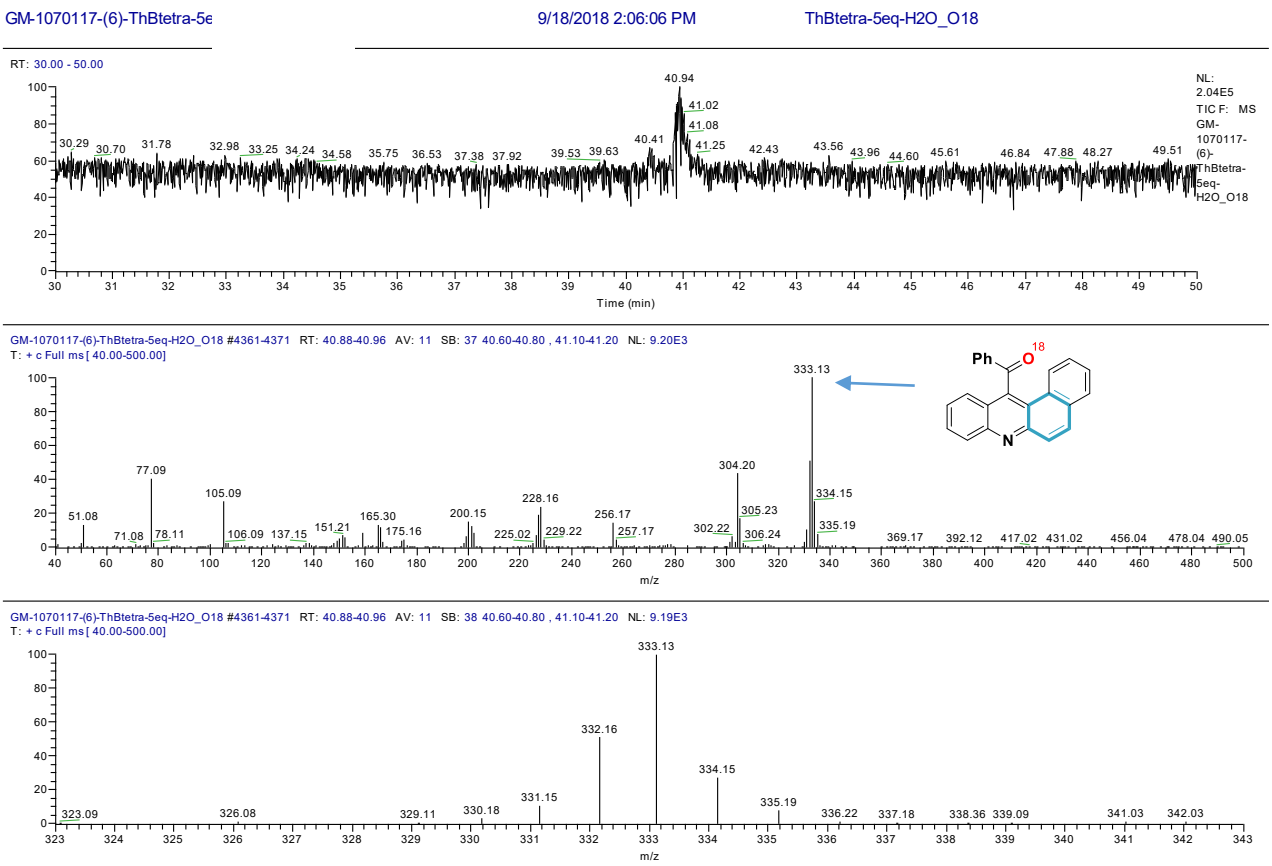
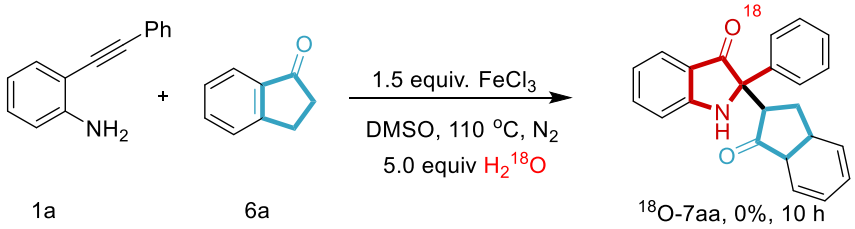


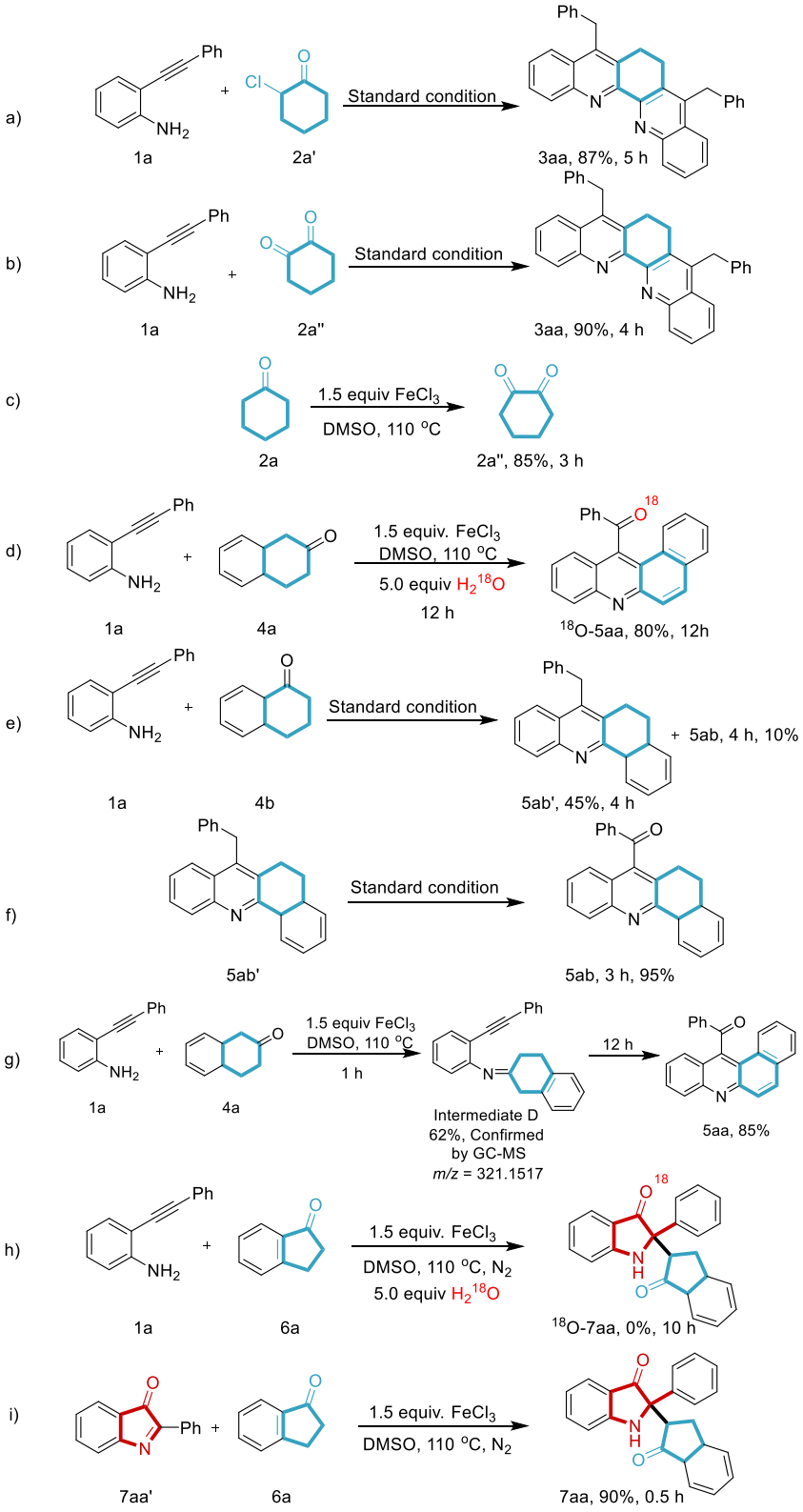
Figure S1. GC-MS spectra of the ^{18}O- and ^{16}O-labeling products

(2) ^{18}O isotope labeling experiment of compound **7aa**.



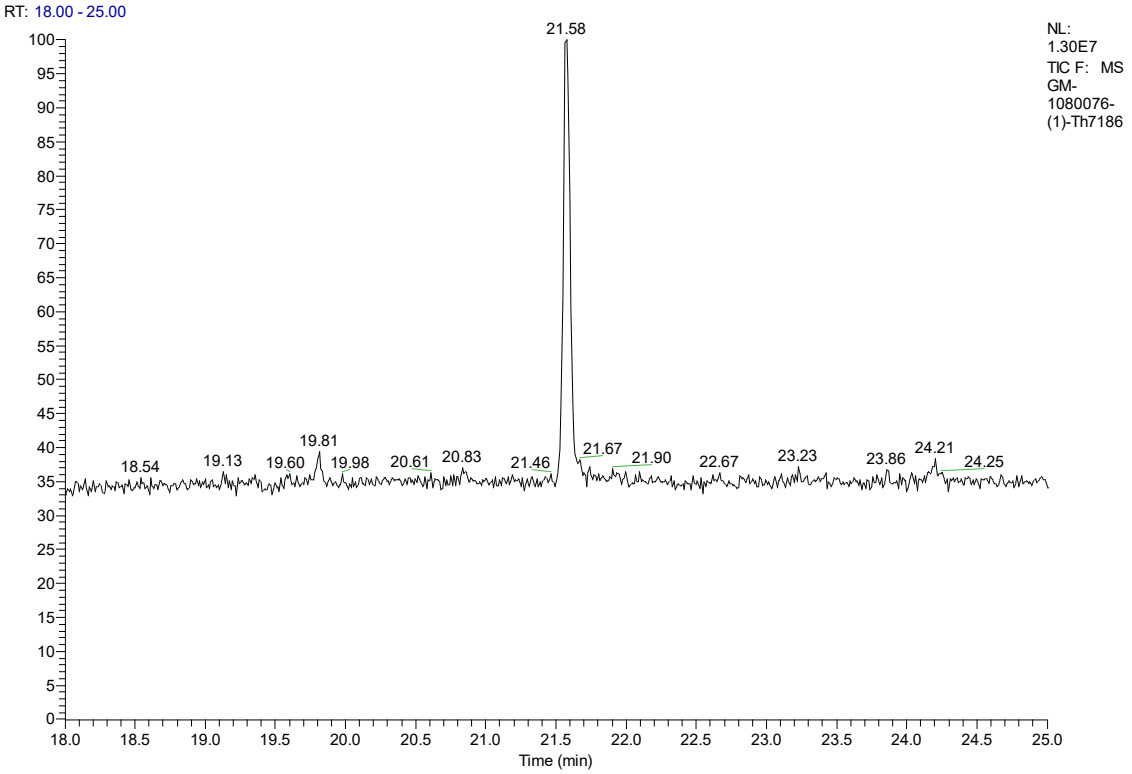
In an oven dried 15 ml seal tube 2-(phenylethynyl) aniline **1a** (193 mg, 1 mmol) was added in 1 ml of DMSO followed by the sequential addition of anhydrous $FeCl_3$ (243 mg, 1.5 mmol), 2,3-dihydro-1*H*-inden-1-one **6a** (198 mg, 1.5 mmol) and $H_2{}^{18}O$ (90 µl, 5 equivalent).. The reaction mixture was allowed to stir at 110 °C for 7 h at nitrogen atmosphere. We did not observed any product formation (**7aa**) in presence of 5 equivalent of $H_2{}^{18}O$.

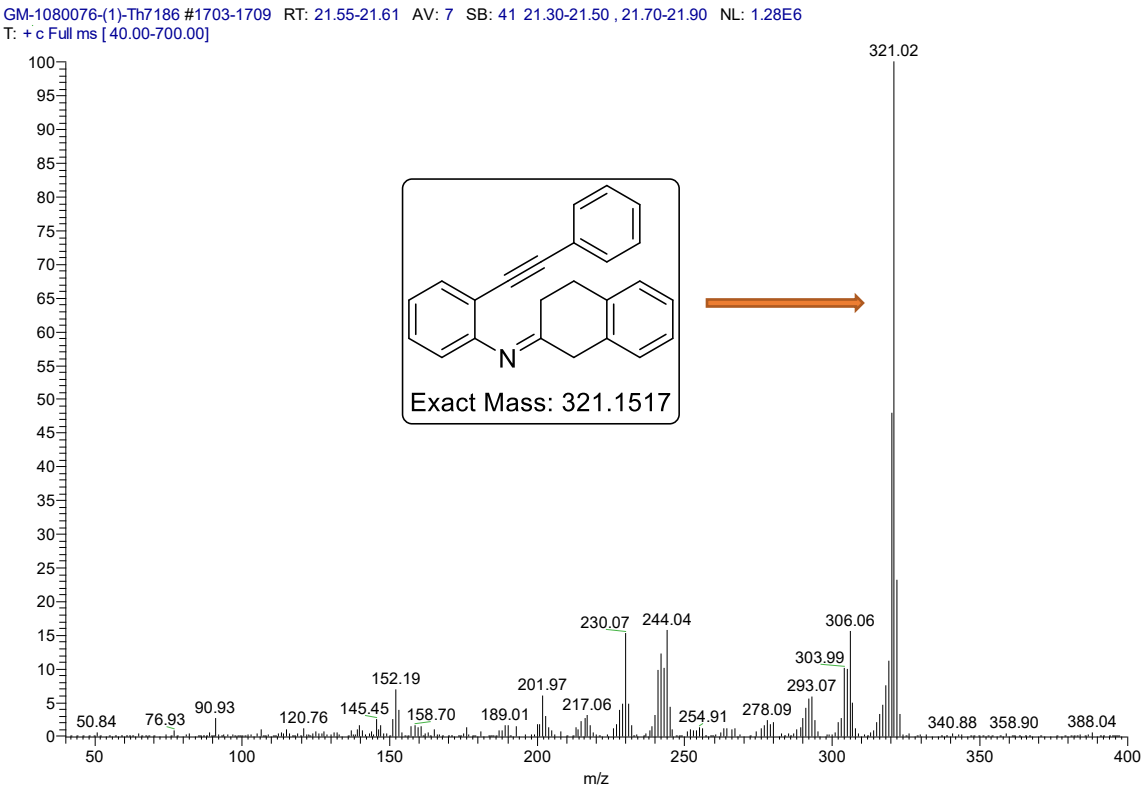
Scheme 1 Control Experiments



To understand the reaction mechanism of these all annulation reactions, we carried out various control studies. In case of dibenzo[*b,j*][1,10]phenanthrolines cyclization reactions, we envisioned 1,2-cyclohexadione and 2-chlorocyclohexan-1-one were possible intermediates. This hypothesis was confirmed by control studies (Scheme 1, a and b). The cyclohexanone treated under the standard reaction conditions in the absence of *o*-alkynylaniline, it converted into 1,2-cyclohexadione. These results support the *in situ* generation of 1,2-cyclohexadione in the reaction medium (Scheme 1, c). ^{18}O labelling studies with benzoylated acridines confirmed that water was the source of oxygen atom in the carbonyl group (Scheme 1, d). At the short reaction time (4 hr), we isolated the cyclized intermediate **5ab'** and the desired product **5ab** in the reaction of 2-(phenylethynyl)aniline **1a** and α-tetralone **4b** (Scheme 1, e). When this cyclized intermediate **5ab'** subjected to our standard reaction conditions, it readily underwent benzylic oxidation to afford the final product **5ab** in 95% yield (Scheme 1, f). These results ruled out the *in situ* generation of 1,2-dicarborbonyl group from oxidation of alkyne and consecutive Friedlander type cyclization.[3a] when we have monitored the reaction in short reaction time (1 hour) through GC-MS analysis, we found 62% of imine intermediate J was formed *in situ* in reaction medium. Further continue the reaction up to 12 hour, we isolated the product **5aa** with 85% yield. It confirm our proposed mechanism of imine formation as an initiation step of aerobic dehydrogenative aromatization reaction (Scheme 1, g). The ^{18}O labelling studies with dihydro-1*H*-indenone was failed, even the desired product **7aa** was not formed in the presence of water This result rationalized the source of oxygen should be DMSO (Scheme 1, h). Furthermore, our anticipated intermediate 2-phenyl-3*H*-indol-3-one **7a'** efficiently reacted with dihydro-1*H*-indenone and transformed into our desired product **7aa** (Scheme 1, i).

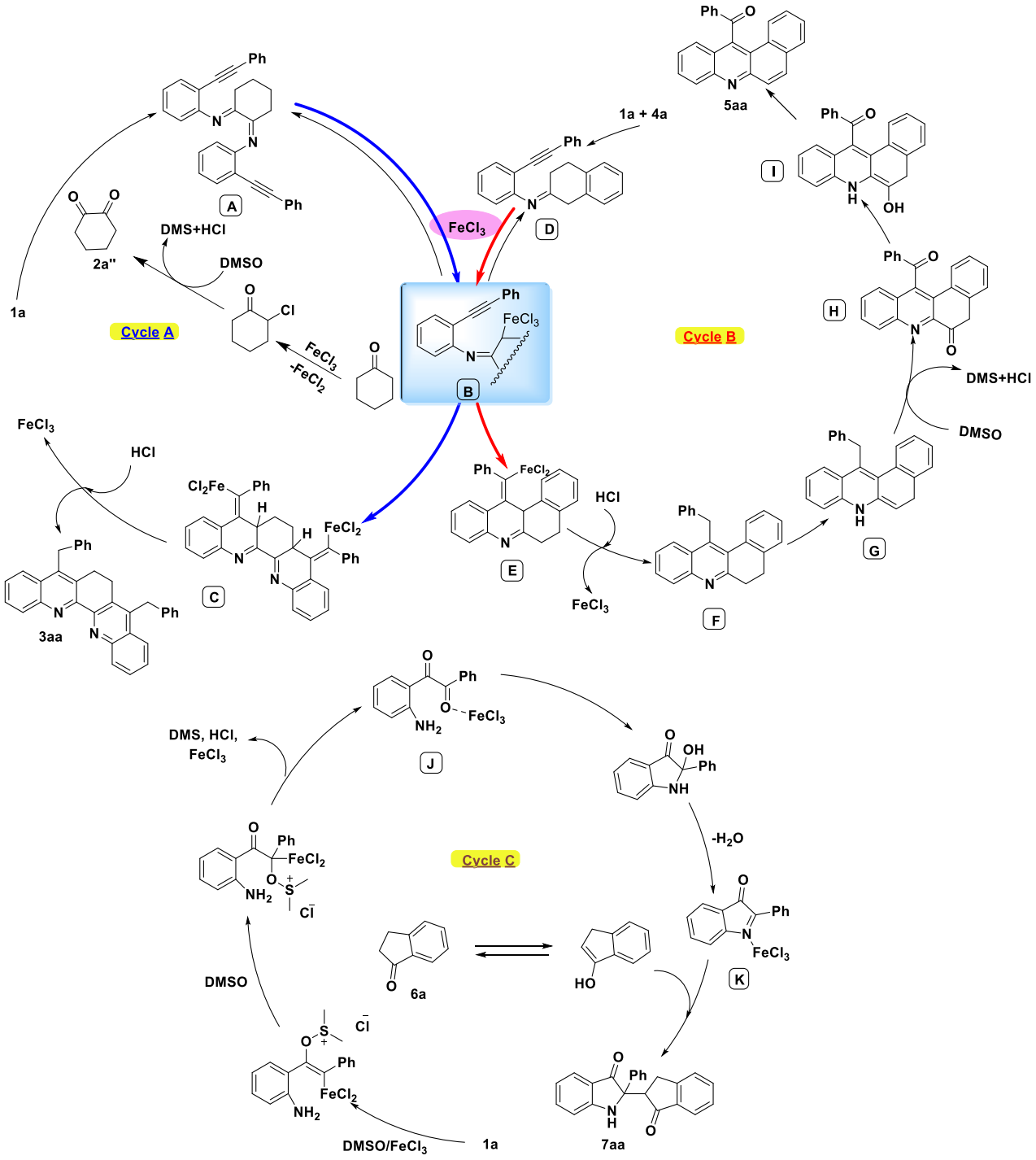
Figure S2. GC-MS spectra of the imine intermediate **D**



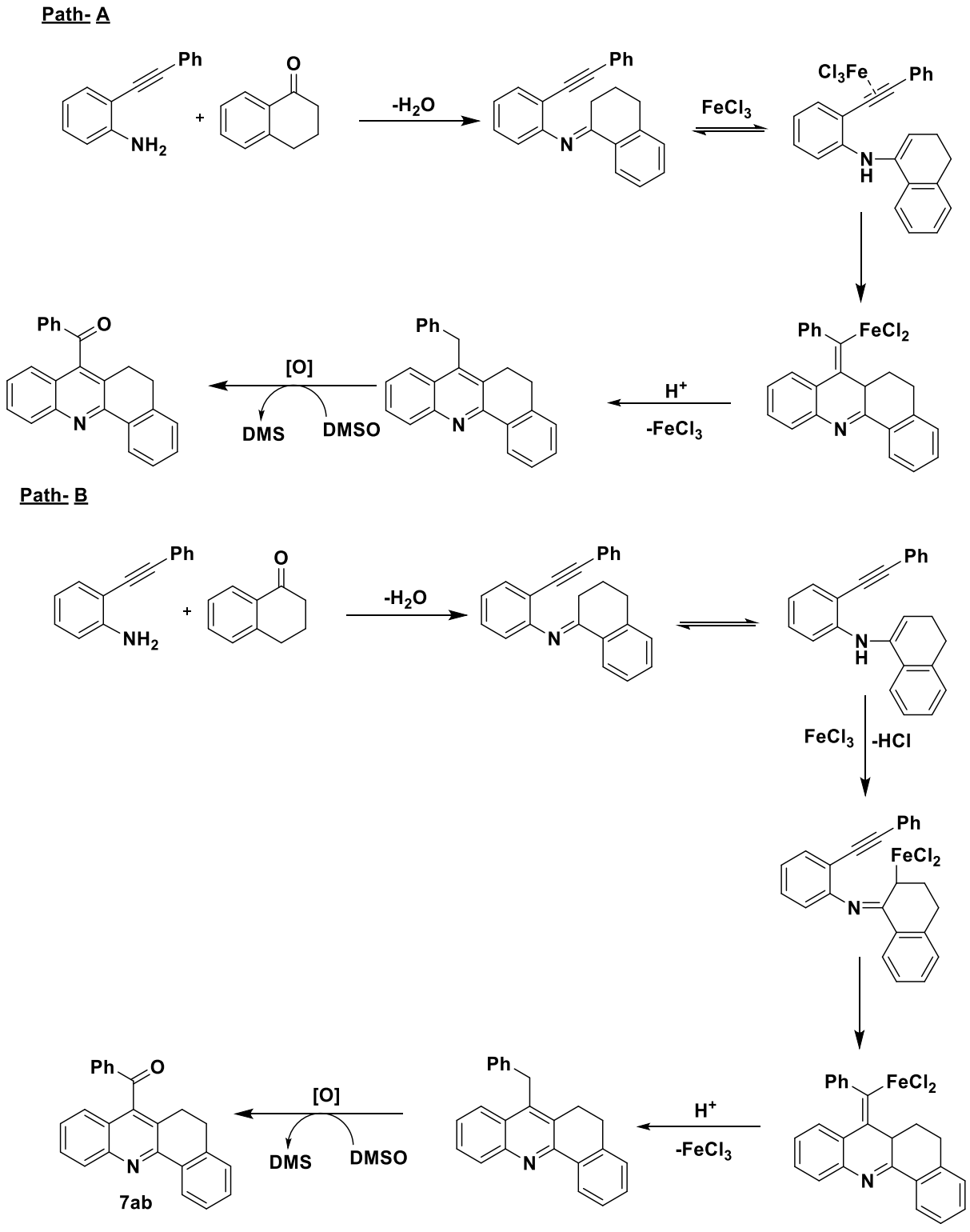


4) Reaction Mechanisms

i) Alternative mechanism for dibenzo[*b,j*][1,10]phenanthroline and benzoylated benzo[*a*] acridine derivatives formation.



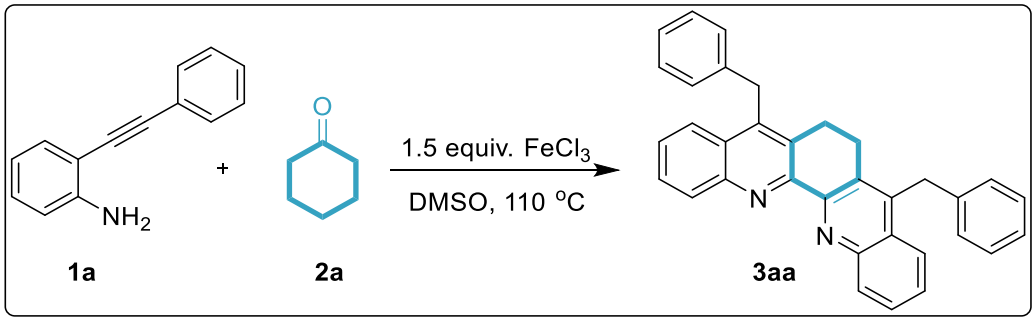
ii) Mechanism for formation of benzoylated benzo[*c*]acridine formation.



2) General Experimental Procedure and Spectral Characterization.

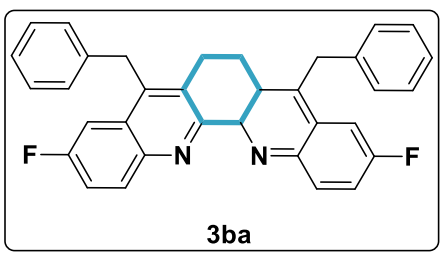
2.1 General procedure (A) for synthesis of dibenzo[*b,j*][1,10]phenanthroline.

Preparation of 5,8-dibenzyl-6,7-dihydrodibenzo[*b,j*][1,10]phenanthroline (**3aa**).



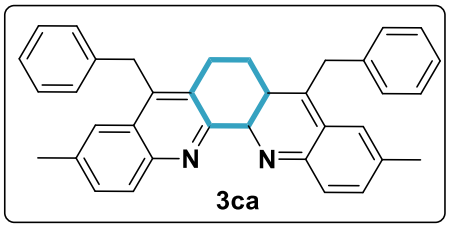
In an oven dried 15 ml seal tube 2-(phenylethynyl) aniline **1a** (405 mg, 2.1 mmol) was added in 1 ml of DMSO followed by the sequential addition of anhydrous $FeCl_3$ (243 mg, 1.5 mmol) and cyclohexanone **2a** (98 mg, 1 mmol). The reaction mixture was allowed to stir at 110 °C for 8 h. After the completion, the reaction mixture was cooled to room temperature and diluted with 25.0 mL of ice water. The water layer was extracted with (3X25 mL) of ethyl acetate and the combined ethyl acetate layer was given brine wash (1X10 mL). The final ethyl acetate layer was dried over sodium sulfate and concentrated under reduced pressure to get the crude compound. The obtained crude was purified using column chromatography by eluting from hexane to 30-55% ethyl acetate/hexane to afford pure of 5,8-dibenzyl-6,7-dihydrodibenzo[*b,j*][1,10]phenanthroline (**3aa**) as an brown color solid (374 mg, 81%); m.p. 230-232 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.49 (d, *J* = 8.5 Hz, 2H), 7.99 (d, *J* = 7.8 Hz, 2H), 7.70 (ddd, *J* = 8.3, 6.8, 1.2 Hz, 2H), 7.53 (ddd, *J* = 8.2, 6.8, 1.2 Hz, 2H), 7.26 – 7.14 (m, 6H), 7.07 (d, *J* = 7.0 Hz, 4H), 4.59 (s, 4H), 3.13 (s, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 152.29, 147.84, 141.98, 138.45, 131.74, 131.13, 128.66, 128.54, 127.90, 127.32, 126.35, 123.69, 33.32, 24.95. HRMS (ESI) calcd for $C_{34}H_{27}N_2$ $[M+H]^+$: 463.2167; found: 463.2168.

5,8-dibenzyl-3,10-difluoro-6,7-dihydrodibenzo[*b,j*][1,10]phenanthroline (**3ba**). Following the general procedure (A) for 14 h on a 1.0 mmol scale, giving the compound as an brown color solid (273 mg, 55%); m.p. 235-237 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ

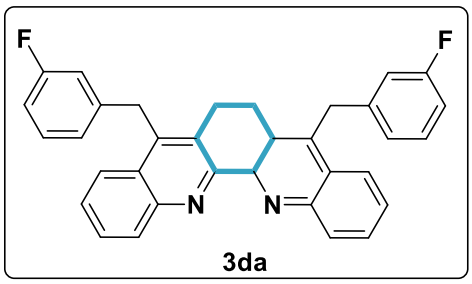


8.47 (dd, J = 9.3, 5.7 Hz, 2H), 7.58 (dd, J = 10.3, 2.7 Hz, 2H), 7.48 (ddd, J = 9.3, 8.0, 2.7 Hz, 2H), 7.26 – 7.16 (m, 6H), 7.06 (d, J = 6.9 Hz, 4H), 4.52 (s, 4H), 3.14 (s, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.32 (d, J_F = 248.7 Hz), 151.70, 145.07, 141.59 (d, J_F = 5.8 Hz), 137.92, 134.27 (d, J_F = 9.4 Hz), 131.79, 128.92, 128.84, 127.85, 126.60, 119.05 (d, J_F = 25.8 Hz), 107.44 (d, J = 22.9 Hz), 33.60, 24.98; ^{19}F NMR (376 MHz, $CDCl_3$) δ -111.03 (s, 2F); HRMS (ESI) calcd for $C_{34}H_{24}F_2N_2$ $[M+H]^+$: 498.1908; found: 462.2096.

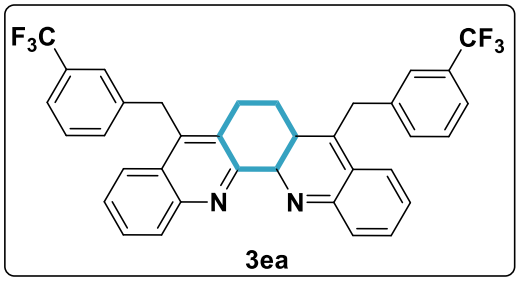
5,8-dibenzyl-3,10-dimethyl-6,7-dihydrodibenzo[*b,j*][1,10]phenanthroline (3ca). Following the general procedure (A) for 7 h on a 1.0 mmol scale, giving the compound as an off-white solid (441 mg, 90%); m.p. 290-291 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.37 (d, J = 8.6 Hz, 2H), 7.73 (s, 2H), 7.53 (dd, J = 8.7, 1.6 Hz, 2H), 7.25 – 7.14 (m, 6H), 7.07 (d, J = 7.1 Hz, 4H), 4.54 (s, 4H), 3.06 (s, 4H), 2.50 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 151.46, 146.34, 140.83, 138.46, 136.97, 131.31, 130.91, 130.63, 128.49, 127.76, 127.72, 126.11, 122.40, 33.04, 24.79, 21.96; HRMS (EI) calcd for : $C_{36}H_{30}N_2$ $[M^+]$: 490.2409; found: 490.2408.



5,8-bis(3-fluorobenzyl)-6,7-dihydrodibenzo[*b,j*][1,10]phenanthroline (3da). Following the general procedure (A) for 6 h on a 1.0 mmol scale, giving the compound as an brown color solid (418 mg, 85%); m.p. 210-211 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (dd, J = 8.5, 0.7 Hz, 2H), 7.72 (ddd, J = 8.4, 6.8, 1.3 Hz, 2H), 7.55 (ddd, J = 8.3, 6.8, 1.3 Hz, 2H), 7.20 (td, J = 8.0, 6.0 Hz, 2H), 6.91 – 6.84 (m, 4H), 6.78 (dd, J = 9.9, 1.7 Hz, 4H), 4.57 (s, 4H), 3.12 (s, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.06 (d, J_F = 246.5 Hz), 152.30, 147.95, 141.22, 141.03 (d, J_F = 7.2 Hz), 131.89, 131.09, 130.19 (d, J_F = 8.4 Hz), 128.75, 127.77, 127.58, 123.56 (d, J_F = 2.9 Hz), 123.50, 114.95 (d, J_F = 21.7 Hz), 113.44 (d, J_F = 21.1 Hz), 33.08, 24.97; ^{19}F NMR (376 MHz, $CDCl_3$) δ -112.56 (s, 2F); HRMS (EI) calcd for $C_{34}H_{24}F_2N_2$ $[M^+]$: 498.1907; found: 498.1898.



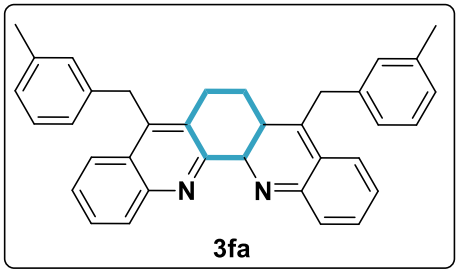
5,8-bis(3-(trifluoromethyl)benzyl)-6,7-dihydrodibenzo[*b,j*][1,10]phenanthroline (3ea).



3ea

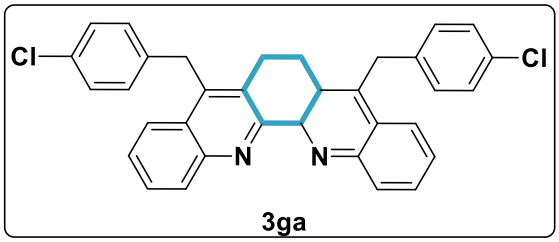
Following the general procedure (A) for 10 h on a 1.0 mmol scale, giving the compound as an off-white color solid (466 mg, 78 %); m.p. 241-242 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.51 (d, *J* = 7.8 Hz, 2H), 7.93 (d, *J* = 8.3 Hz, 2H), 7.73 (td, *J* = 7.2, 1.2 Hz, 2H), 7.55 (td, *J* = 7.2, 1.2 Hz, 2H), 7.44 (d, *J* = 8.6 Hz, 4H), 7.31 (dd, *J* = 9.9, 5.5 Hz, 2H), 7.14 (d, *J* = 7.8 Hz, 2H), 4.64 (s, 4H), 3.11 (s, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 152.33, 148.01, 140.83, 139.41, 132.00, 131.06, 130.91, 129.27, 128.84, 127.72, 127.67, 124.71 (q, J_F = 3.8 Hz), 123.43 (q, J_F = 3.7 Hz), 123.35, 33.09, 25.02; ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.05 (s, 6F); HRMS (EI) calcd for $C_{36}H_{24}F_6N_2$ [M^+]: 598.1843; found: 598.1849.

5,8-bis(3-methylbenzyl)-6,7-dihydrodibenzo[*b,j*][1,10]phenanthroline (3fa). Following the general procedure (A) for 8 h on a 1.0 mmol scale, giving the compound as an brown color solid (382 mg, 78%); m.p. 254-256 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.47(d, *J* = 7.8 Hz, 2H), 7.98 (d, *J* = 7.8 Hz, 2H), 7.69 (ddd, *J* = 8.0, 6.8, 1.2 Hz, 2H),, 7.51 (ddd, *J* = 8.3, 6.8, 1.3 Hz, 2H), 7.10 (t, *J* = 7.7 Hz, 2H), 6.97 (d, *J* = 7.4 Hz, 2H), 6.85 (d, *J* = 8.3 Hz, 4H), 4.54 (s, 4H), 3.11 (s, 4H), 2.21 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 152.34, 147.84, 142.06, 138.40, 138.31, 131.68, 131.17, 128.61, 128.51, 127.96, 127.26, 127.10, 124.94, 123.75, 33.25, 24.97, 21.33; HRMS (ESI) calcd for: $C_{36}H_{31}N_2$ [M+H]$^+$: 491.2481; found: 491.2481.



3fa

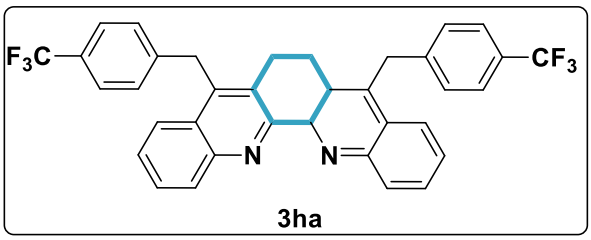
5,8-bis(4-chlorobenzyl)-6,7-dihydrodibenzo[*b,j*][1,10]phenanthroline (3ga). Following the general procedure (A) for 7 h on a 1.0 mmol scale, giving the compound as an off-white color solid (254 mg, 48%); m.p. 259-260 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50 (d, *J* = 7.9 Hz, 2H), 7.94 (d, *J*



3ga

= 8.0 Hz, 2H), 7.72 (ddd, *J* = 8.0, 6.8, 1.2 Hz, 2H), 7.54 (ddd, *J* = 8.2, 6.9, 1.2 Hz, 2H), 7.24 – 7.16 (m, 4H), 7.00 (d, *J* = 8.5 Hz, 4H), 4.55 (s, 4H), 3.09 (s, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 152.36, 147.99, 141.41, 136.93, 132.26, 131.96, 131.02, 129.25, 128.87, 128.72, 127.74, 127.56, 123.49, 32.71, 24.98; HRMS (EI) calcd for $C_{34}H_{24}Cl_2N_2$ [M^+]: 530.1316; found: 530.1312.

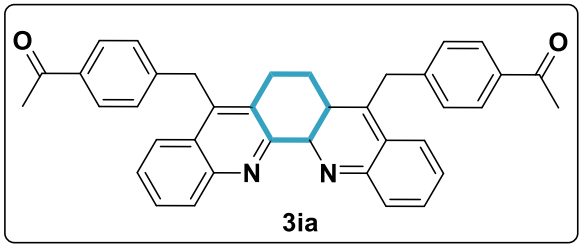
5,8-bis(4-(trifluoromethyl)benzyl)-6,7-dihydrodibenzo[*b*,*j*][1,10]phenanthroline (3ha).



3ha

Following the general procedure (A) for h on a 1.0 mmol scale, giving the compound as a white color solid (484 mg, 81%); m.p. 252-253 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.53 (dd, *J* = 8.5, 0.7 Hz, 2H), 7.94 (d, *J* = 7.9 Hz, 2H), 7.75 (ddd, *J* = 8.4, 6.8, 1.3 Hz, 2H), 7.57 (ddd, *J* = 8.3, 6.8, 1.3 Hz, 2H), 7.51 (d, *J* = 8.1 Hz, 4H), 7.20 (d, *J* = 8.1 Hz, 4H), 4.66 (s, 4H), 3.12 (s, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 152.35, 148.05, 142.58, 140.87, 132.07, 131.05, 128.85, 128.24, 127.73, 125.69 (q, J_F = 3.7 Hz), 123.38, 33.19, 25.03; ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.38 (s, 6F); HRMS (EI) calcd for $C_{36}H_{24}F_6N_2$ [M^+]: 598.1843; found: 598.1846.

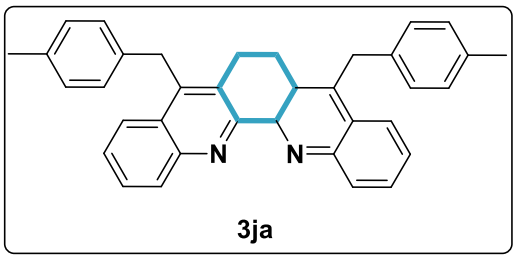
1,1'-(((6,7-dihydrodibenzo[*b*,*j*][1,10]phenanthroline-5,8-diyl)bis(methylene))bis(4,1-phenylene)) bis(ethan-1-one) (3ia).



3ia

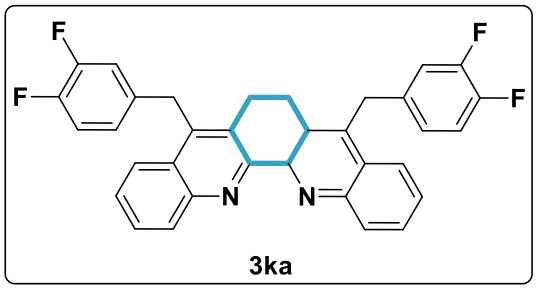
Following the general procedure (A) for 6 h on a 1.0 mmol scale, giving the compound as brown color solid (425 mg, 78%); m.p. 178-180 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.49 (dd, *J* = 8.5, 0.8 Hz, 2H), 7.93 (dd, *J* = 8.5, 0.7 Hz, 2H), 7.86 – 7.79 (m, 4H), 7.72 (ddd, *J* = 8.3, 6.8, 1.3 Hz, 2H), 7.54 (ddd, *J* = 8.3, 6.8, 1.3 Hz, 2H), 7.15 (d, *J* = 8.5 Hz, 4H), 4.63 (s, 4H), 3.09 (s, 4H), 2.52 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.53, 152.29, 147.97, 144.09, 141.07, 135.54, 131.91, 131.04, 128.83, 128.14, 127.73, 127.65, 123.44, 33.39, 26.51, 25.01. HRMS (ESI) calcd for $C_{38}H_{31}N_2O_2$ [M+H]$^+$: 547.2380; found: 547.2380.

5,8-bis(4-methylbenzyl)-6,7-dihydrodibenzo[*b,j*][1,10]phenanthroline (3ja) Following the general procedure (A) for 7 h on a 1.0 mmol scale, giving the compound as a brown color solid (313 mg, 64%); m.p. 253-254 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50 (dd, *J* = 19.0, 8.6 Hz, 2H), 7.98 (d, *J* = 8.0 Hz, 2H), 7.69 (ddd, *J* = 8.0, 6.8, 1.2 Hz, 2H), 7.51 (ddd, *J* = 8.2, 6.8, 1.2 Hz, 2H), 7.03 (d, *J* = 7.9 Hz, 4H), 6.96 (d, *J* = 8.1 Hz, 4H), 4.54 (s, 4H), 3.12 (s, 4H), 2.27 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 152.27, 147.78, 142.06, 135.80, 135.28, 131.62, 131.01, 129.23, 128.37, 127.83, 127.68, 127.14, 123.64, 32.81, 24.85, 20.79. HRMS (ESI) calcd for: $C_{36}H_{31}N_2$ $[M+H]^+$: 491.2481; found: 491.2481.



3ja

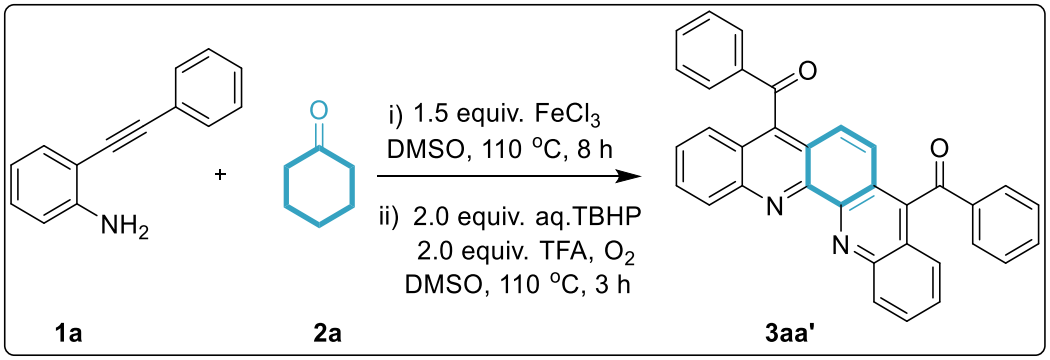
5,8-bis(3,4-difluorobenzyl)-6,7-dihydrodibenzo[*b,j*][1,10]phenanthroline (3ka). Following the general procedure (A) for7 h on a 1.0 mmol scale, giving the compound as brown color solid (480 mg, 90%); m.p. 264-266 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.55 – 8.47 (m, 2H), 7.92 (d, *J* = 8.2 Hz, 2H), 7.77 – 7.71 (m, 2H), 7.66 – 7.43 (m, 3H), 7.02 (dd, *J* = 18.0, 8.4 Hz, 2H), 6.89 – 6.76 (m, 3H), 4.54 (s, 4H), 3.11 (s, 4H); ^{13}C NMR (101 MHz, cdcl$_3$) δ 150.15 (d, J_F = 432.1 Hz), 140.99, 131.98, 131.60, 130.94, 129.42 (d, *J* = 59.2 Hz), 128.87, 128.20 (d, J_F = 30.2 Hz), 127.74, 127.60, 123.73, 118.28 (d, J_F = 17.9 Hz), 117.49 (d, J_F = 17.1 Hz), 116.84 (d, J_F = 17.5 Hz), 32.46, 24.95; ^{19}F NMR (376 MHz, $CDCl_3$) δ -140.58 (s, 1F), -136.93 (s, 1F), 134.19 (s, 1F), -125.96 (s, 1F); HRMS (EI) calcd for $C_{34}H_{22}F_4N_2$ $[M^+]$: 534.1719 ; found: 534.1725.



3ka

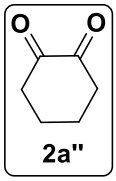
Procedure for synthesis of dibenzo[*b,j*][1,10]phenanthroline-5,8-diylbis(phenyl methanone) (3aa').

In an oven dried 10 ml seal tube 2-(phenylethynyl) aniline **1a** (40 mg, 2.1 mmol) was added in 250 µl of DMSO followed by the sequential addition of anhydrous $FeCl_3$ (24 mg, 1.5 mmol) and cyclohexanone **2a** (9 µl, 0.1 mmol). The reaction mixture was allowed to stir at 110 °C for 8 h. After the completion, the reaction mixture was cooled to room temperature and diluted with 10.0 mL of ice water. The water layer was extracted with (3X10 mL) of ethyl



acetate and the combined ethyl acetate layer was given brine wash (1X10 mL). The final ethyl acetate layer was dried over sodium sulfate and concentrated under reduced pressure to get the crude compound. The crude product directly carried to next step without further purification. In an oven dried 15 ml seal tube was loaded crude 5,8-dibenzyl-6,7-dihydrodibenzo[*b,j*] [1,10] phenanthroline **3aa** (consider the crude had 80% product, 36 mg 1 mmol) in 250 µl of DMSO followed by the addition of 70% TBHP in water (19 µl, 2 mmol) and trifluoroacetic acid (14 µl, 2 mmol). The resulting solution was sealed under O_2 and stirred at 110 °C for 3 h. After cooling to room temperature, the reaction mixture was diluted with 5 mL of water and neutralized with 10% aqueous $NaHCO_3$ solution. The aqueous layer was extracted with (3X5 mL) of ethyl acetate and the combined organic layer was given brine wash (2X5 mL). The final organic layer was evaporated in a rotary evaporator to remove the volatile components. The residue was purified by column chromatography on silica gel (40% ethyl acetate/ hexane to 60% ethyl acetate/hexane) to afford pure dibenzo[*b,j*][1,10]phenanthroline-5,8-diylbis(phenyl methanone) (**3aa'**) as a yellow color solid (21 mg, 45%); m.p. 255-256 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.53 (d, *J* = 8.8 Hz, 2H), 7.82 – 7.77 (m, 8H), 7.62 (dd, *J* = 12.9, 6.5 Hz, 3H), 7.55 – 7.48 (m, 3H), 7.49 – 7.39 (m, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.29, 151.67, 147.95, 143.63, 136.47, 134.82, 131.52, 130.11, 129.78, 129.71, 129.30, 128.39, 128.32, 125.25, 124.69. HRMS (ESI) calcd for $C_{34}H_{21}N_2O_2$ $[M+H]^+$: 489.1596; found: 489.1597.

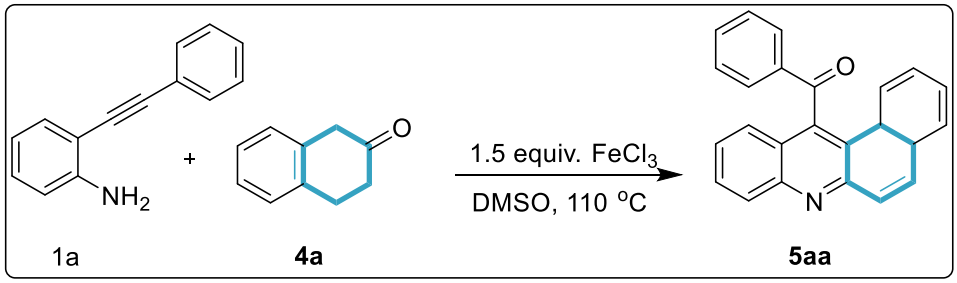
Cyclohexane-1,2-dione (2a'').



Semi solid, ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.24 – 6.11 (m, 1H), 6.01 (s, 1H), 2.55 (td, *J* = 6.8, 3.8 Hz, 2H), 2.48 – 2.34 (m, 2H), 2.15 – 1.87 (m, 2H).

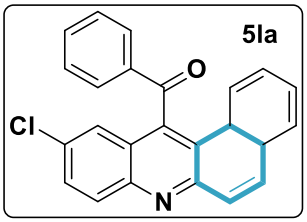
General procedure (B) for synthesis of Benzoylated Acridines.

Preparation benzo[*a*]acridin-12-yl(phenyl)methanone (**5aa**).



In an oven dried 15 ml seal tube 2-(phenylethynyl) aniline **1a** (193 mg, 1 mmol) was added in 1 ml of DMSO followed by the sequential addition of anhydrous $FeCl_3$ (243 mg, 1.5 mmol) and α-tetralone **4a** (219 mg, 1.5 mmol). The reaction mixture was allowed to stir at 110 °C for 12 h. After the completion, the reaction mixture was cooled to room temperature and diluted with 25.0 mL of cooled water. The water layer was extracted with (3X25 mL) of ethyl acetate and the combined ethyl acetate layer was given brine wash (1X10 mL). The final ethyl acetate layer was dried over sodium sulfate and concentrated under reduced pressure to get the crude compound. The obtained crude was purified using column chromatography by eluting from (8% ethyl acetate/ hexane to 15% ethyl acetate/hexane) afford pure benzo[*a*]acridin-12-yl(phenyl)methanone (**5aa**) as a brown color solid (300 mg, 91%); m.p. 213-214 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.36 (d, *J* = 8.6 Hz, 1H), 8.33 (d, *J* = 8.3 Hz, 1H), 8.08 (d, *J* = 9.2 Hz, 1H), 7.97 (d, *J* = 9.2 Hz, 1H), 7.92 – 7.74 (m, 5H), 7.63 – 7.47 (m, 3H), 7.43 – 7.34 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 200.15, 149.66, 147.26, 142.55, 136.72, 134.60, 133.24, 132.55, 130.20, 129.67, 129.41, 129.27, 129.20, 128.97, 128.71, 127.82, 127.67, 127.27, 126.94, 125.37, 123.62, 120.72; HRMS (EI) calcd for $C_{24}H_{15}NO$ [M^+]: 333.1153; found: 333.1152.

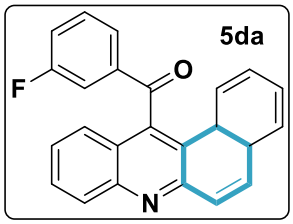
(10-chlorobenzo[*a*]acridin-12-yl)(phenyl)methanone (5la). Following the general procedure



(B) for 12 h on a 1.0 mmol scale, giving the compound as a yellow color solid (220 mg, 60%); m.p. 221-222 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.35 (d, *J* = 8.4 Hz, 1H), 8.27 (d, *J* = 9.6 Hz, 1H), 8.06 (d, *J* = 9.2 Hz, 1H), 7.99 (d, *J* = 9.2 Hz, 1H), 7.87 (dd, *J* = 7.8, 1.2 Hz, 1H), 7.80 (d, *J* = 7.2 Hz, 2H), 7.78 – 7.72 (m, 2H), 7.64 – 7.53 (m, 2H), 7.48 – 7.34 (m, 3H); ^{13}C

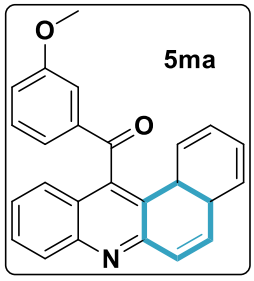
NMR (101 MHz, $CDCl_3$) δ 199.50, 149.76, 145.64, 141.51, 136.34, 134.85, 133.54, 132.89, 132.69, 131.38, 131.09, 129.69, 129.33, 129.29, 128.83, 128.41, 128.18, 127.87, 127.40, 123.98, 123.88, 121.28; HRMS (EI) calcd for $C_{24}H_{14}ClNO$ [M^+]: 367.0763; found: 367.0755.

Benzo[*a*]acridin-12-yl(3-fluorophenyl)methanone (5da). Following the general procedure (B)



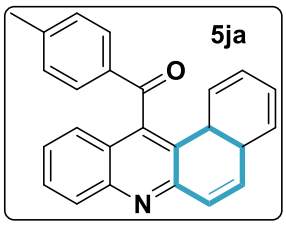
for 10 h on a 1.0 mmol scale, giving the compound as a yellow color solid (298 mg, 85%); m.p. 180-181 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.36 (d, J = 8.3 Hz, 1H), 8.32 (dd, J = 8.4, 0.7 Hz, 1H), 8.11 (d, J = 9.2 Hz, 1H), 8.01 (d, J = 9.2 Hz, 1H), 7.90 (dd, J = 7.8, 1.4 Hz, 1H), 7.85 (ddd, J = 8.6, 6.7, 1.3 Hz, 1H), 7.77 – 7.74 (m, 1H), 7.64 (td, J = 9.2, 2.0 Hz, 1H), 7.61 – 7.51 (m, 2H), 7.49 – 7.39 (m, 2H), 7.38 – 7.28 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.91, 163.06 (d, J_F = 249.6 Hz), 149.63, 147.28, 141.67, 138.71 (d, J_F = 6.2 Hz), 133.30, 132.61, 130.97 (d, J_F = 7.6 Hz), 129.56, 129.39, 128.99, 128.54, 127.98, 127.51, 127.34, 127.15, 125.72 (d, J_F = 3.0 Hz), 125.07, 123.42, 121.76 (d, J_F = 21.6 Hz), 120.69, 115.82 (d, J_F = 22.4 Hz); ^{19}F NMR (376 MHz, $CDCl_3$) δ -110.45 (s, 1F); HRMS (EI) calcd for $C_{24}H_{14}FNO$ [M^+]: 351.1059; found: 351.1063.

Benzo[*a*]acridin-12-yl(3-methoxyphenyl)methanone (5ma). Following the general procedure



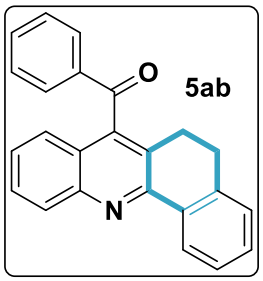
(B) for 24 h on 1.0 mmol scale, giving the compound as a brown color solid (326 mg, 90%); m.p. 164-165 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.33 (t, J = 9.2 Hz, 2H), 8.08 (d, J = 9.2 Hz, 1H), 7.98 (d, J = 9.2 Hz, 1H), 7.87 (dd, J = 7.8, 1.3 Hz, 1H), 7.82 (td, J = 6.4, 1.2 Hz, 1H), 7.77 (ddd, J = 8.7, 1.2, 0.7 Hz, 1H), 7.61 (s, 1H), 7.55 (td, J = 7.6, 1.2 Hz, 1H), 7.53 – 7.48 (m, 1H), 7.40 (ddd, J = 8.5, 7.2, 1.5 Hz, 1H), 7.24 – 7.16 (m, 2H), 7.12 (ddd, J = 7.7, 2.6, 1.7 Hz, 1H), 3.82 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 199.64, 159.89, 149.34, 146.90, 142.24, 137.72, 132.90, 132.22, 129.93, 129.85, 129.07, 128.96, 128.65, 128.38, 127.48, 127.35, 126.95, 126.59, 125.04, 123.35, 122.71, 120.88, 120.41, 112.65, 55.16; HRMS (EI) calcd for $C_{25}H_{17}NO_2$ [M^+]: 363.1259; found: 363.1254.

(10-methylbenzo[*a*]acridin-12-yl)(phenyl)methanone (5ja). Following the general procedure



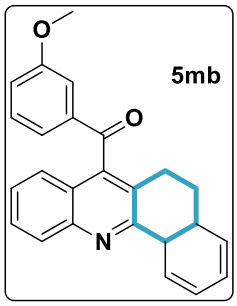
(B) for 12 h on 1.0 mmol scale, giving the compound as a yellow color solid (301 mg, 87%); m.p. 160-161 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.40 (d, *J* = 8.4 Hz, 1H), 8.32 (d, *J* = 8.6 Hz, 1H), 8.08 (d, *J* = 9.2 Hz, 1H), 7.97 (d, *J* = 9.2 Hz, 1H), 7.88 – 7.83 (m, 1H), 7.83 – 7.80 (m, 1H), 7.80 – 7.76 (m, 1H), 7.73 (d, *J* = 7.8 Hz, 2H), 7.51- 7.47 (m, 2H), 7.44 – 7.34 (m, 1H), 7.17 (d, *J* = 8.1 Hz, 2H), 2.35 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 199.69, 149.66, 147.25, 145.81, 142.79, 134.40, 133.15, 132.48, 130.10, 129.90, 129.78, 129.36, 129.19, 128.98, 128.75, 127.71, 127.67, 127.22, 126.81, 125.44, 123.65, 120.62, 21.80; HRMS (EI) calcd for $C_{25}H_{17}NO$ [M^+]: 347.1310; found: 347.1316.

(5,6-dihydrobenzo[*c*]acridin-7-yl)(phenyl)methanone (5ab). Following the general procedure



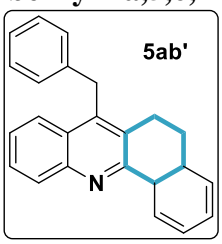
(B) for 14 h on 1.0 mmol scale giving the compound as a yellow color solid (234 mg, 70%); m.p. 125-126 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.62 (d, *J* = 7.7 Hz, 1H), 8.21 (d, *J* = 8.5 Hz, 1H), 7.87 (d, *J* = 8.1 Hz, 2H), 7.77 – 7.58 (m, 1H), 7.55 – 7.34 (m, 6H), 7.26 – 7.23 (m, 2H), 2.87 (s, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.95, 153.03, 147.20, 142.61, 139.05, 136.55, 134.54, 134.33, 130.06, 129.94, 129.68, 129.20, 129. 12, 127.88, 127.47, 126.84, 126.59, 126.26, 124.67, 124.53, 27.77, 26.25. HRMS (ESI) calcd for $C_{24}H_{18}NO$ [M+H]$^+$: 336.1384; found: 336.1382.

benzo[*c*]acridin-7-yl(3-methoxyphenyl)methanone (5mb). Following the general procedure (B)



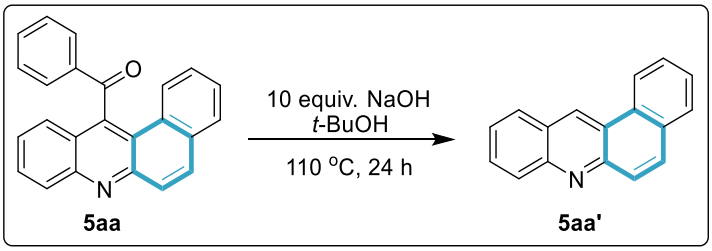
for 48 h on 1.0 mmol scale, giving the compound as a brown color solid (237 mg, 65%); m.p. 150-152 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.62 (dd, *J* = 7.7, 1.2 Hz, 1H), 8.20 (ddd, *J* = 8.5, 1.2, 0.6 Hz, 1H), 7.67 (ddd, *J* = 8.4, 6.8, 1.5 Hz, 1H), 7.54 (d, *J* = 2.3Hz, 1H), 7.51 – 7.38 (m, 4H), 7.38 – 7.26 (m, 2H), 7.24 (d, *J* = 7.3 Hz, 1H), 7.17 (ddd, *J* = 7.8, 2.7, 1.5 Hz, 1H), 3.85 (s, 3H), 2.92 (s, 4H); ^{13}C NMR (101 MHz,$CDCl_3$) δ 197.62, 160.06, 152.83, 147.03, 142.51, 138.88, 137.76, 134.17, 129.98, 129.89, 129.77, 129.03, 127.71, 127.30, 126.68, 126.42, 126.12, 124.51, 124.40, 122.91, 121.10, 112.59, 55.38, 27.65, 26.12; HRMS (ESI) calcd for $C_{25}H_{20}NO_2$ [M+H]$^+$: 366.1486; found: 366.1489.

7-benzyl-4a,5,6,12b-tetrahydrobenzo[*c*]acridine (5ab'). Following the general procedure (B)



for 4 h on 1.0 mmol scale, giving the compound yellow color solid; m.p. 109-110 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.59 (dd, *J* = 7.7, 1.2 Hz, 1H), 8.16 (dd, *J* = 8.4, 0.8 Hz, 1H), 7.93 (dd, *J* = 8.5, 0.8 Hz, 1H), 7.63 (ddd, *J* = 8.3, 6.8, 1.3 Hz, 1H), 7.47 – 7.39 (m, 2H), 7.35 (td, *J* = 7.4, 1.5 Hz, 1H), 7.26 – 7.24 (m, 1H), 7.24 – 7.20 (m, 2H), 7.19 – 7.14 (m, 1H), 7.13 – 7.05 (m, 2H), 4.53 (s, 2H), 3.08 (dd, *J* = 8.6, 5.4 Hz, 2H), 2.94 (dd, *J* = 8.3, 5.6 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 152.90, 147.18, 141.27, 138.92, 138.65, 134.93, 130.10, 129.42, 129.28, 128.53, 128.25, 127.86, 127.41, 127.35, 127.14, 126.24, 126.16, 126.10, 123.86, 33.20, 28.03, 25.38; HRMS (ESI) calcd for $C_{24}H_{20}N$ $[M+H]^+$: 322.1589; found: 322.1590.

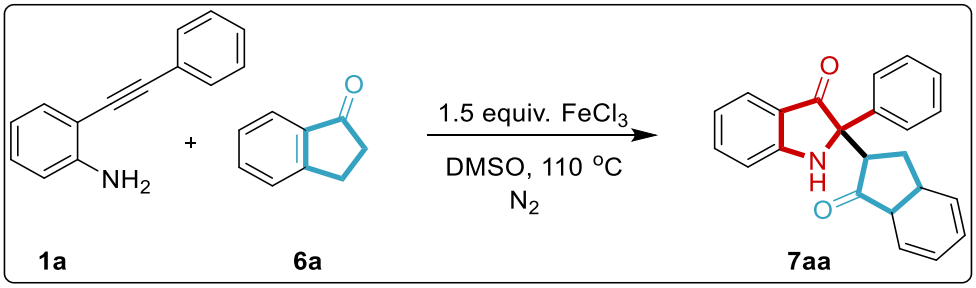
Procedure for synthesis of benzo[*a*]acridine (5aa').



In an oven dried 15 ml seal tube benzo[*a*]acridin-12-yl(phenyl)methanone **5aa** (333 mg, 1 mmol) was added in 1 ml of *t*-butanol followed by the addition of NaOH (400 mg, 10 mmol). The reaction mixture was allowed to stir at 110 °C for 24 h. After the completion, the reaction mixture was cooled to room temperature and diluted with 25.0 mL of cooled water. The water layer was extracted with (3X25 mL) of ethyl acetate and the combined ethyl acetate layer was given brine wash (1X10 mL). The final ethyl acetate layer was dried over sodium sulfate and concentrated under reduced pressure to get the crude compound. The obtained crude was purified using column chromatography by eluting from hexane to 8-15% ethyl acetate/hexane to afford benzo[*a*]acridine (**5aa'**) as a brown color solid (170 mg, 74%); m.p. 102-103 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.42 (s, 1H), 8.75 (d, *J* = 8.1 Hz, 1H), 8.26 (dd, *J* = 8.7, 0.7 Hz, 1H), 8.09 (dd, *J* = 8.3, 0.5 Hz, 1H), δ 8.00 (d, *J* = 9.3 Hz, 1H), 7.95 (d, *J* = 9.3 Hz, 1H), 7.89 (dd, *J* = 7.7, 1.3 Hz, 1H), 7.81 (ddd, *J* = 8.3, 6.7, 1.4 Hz, 1H), 7.76 – 7.57 (m, 3H; ^{13}C NMR (101 MHz, $CDCl_3$) δ 149.33, 148.15, 132.53, 131.22, 130.49, 130.08, 129.96, 129.01, 128.85, 128.40, 128.26, 127.70, 127.44, 126.49, 126.04, 124.14, 122.87; HRMS (ESI) calcd for $C_{17}H_{12}N$ $[M+H]^+$: 230.0966; found: 230.0964.

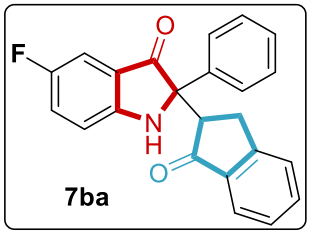
General procedure (C) for synthesis of 2-(indenyl-2-yl)-3-Oxoindoline.

Preparation of 2-(1-oxo-2,3-dihydro-1*H*-inden-2-yl)-2-phenylindolin-3-one (**7aa**).



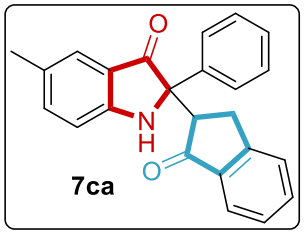
In an oven dried 15 ml seal tube 2-(phenylethynyl) aniline **1a** (193 mg, 1 mmol) was added in 1 ml of DMSO followed by the sequential addition of anhydrous $FeCl_3$ (243 mg, 1.5 mmol) and 2,3-dihydro-1*H*-inden-1-one **6a** (198 mg, 1.5 mmol). The reaction mixture was allowed to stir at 110 °C for 7 h at nitrogen atmosphere. After the completion, the reaction mixture was cooled to room temperature and diluted with 25.0 mL of cooled water. The water layer was extracted with (3X25 mL) of ethyl acetate and the combined ethyl acetate layer was given brine wash (1X10 mL). The final ethyl acetate layer was dried over sodium sulfate and concentrated under reduced pressure to get the crude compound. The obtained crude was purified using column chromatography by eluting from hexane to 10-16% ethyl acetate/hexane to afford pure of 2-(1-oxo-2,3-dihydro-1*H*-inden-2-yl)-2-phenylindolin-3-one (**7aa**) as a yellow color solid (298 mg, 88%); m.p. 215-216 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (t, *J* = 7.5 Hz, 2H), 7.61 – 7.52 (m, 3H), 7.46 (ddd, *J* = 8.3, 7.2, 1.3 Hz, 1H), 7.40 (d, *J* = 7.7 Hz, 1H), 7. 35 – 7.26 (m, 3H), 7.26 – 7.22 (m, 1H), 6.92 (d, *J* = 8.2 Hz, 1H), 6.87 (t, *J* = 7.6 Hz, 1H), 5.88 (s, 1H), 3.68 (dd, *J* = 8.3, 4.9 Hz, 1H), 3.35 (dd, *J* = 17.9, 8.4 Hz, 1H), 3.15 (dd, *J* = 18.0, 4.9 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.30, 200.25, 189.74, 167.70, 159.77, 153.45, 137.09, 136.91, 136.57, 135.06, 128.71, 127.89, 127.56, 126.39, 126.18, 125.28, 124.08, 120.71, 119.56, 112.14, 71.53, 53.88, 29.57; HRMS (EI) calcd for $C_{23}H_{17}NO_2$ [M^+]: 339.1259; found: 339.1263.

5-fluoro-2-(1-oxo-2,3-dihydro-1*H*-inden-2-yl)-2-phenylindolin-3-one (7ba). Following the general procedure (C) for 8 h on 1.0 mmol scale, giving the compound as a brown color solid (160 mg, 45%); m.p. 172-174 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (d, *J* = 7.6 Hz, 1H), 7.56 (td, *J* = 7.6, 1.2 Hz, 1H), 7.54 – 7.49 (m, 2H), 7.39 (d, *J* = 7.7 Hz, 1H), 7.36 – 7.26 (m, 4H),

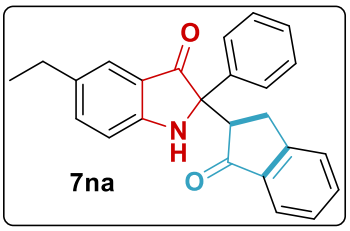


7.25 – 7.18 (m, 2H), 6.88 (dd, J = 8.8, 3.8 Hz, 1H), 5.68 (s, 1H), 3.71 (dd, J = 8.3, 4.9 Hz, 1H), 3.33 (dd, J = 17.9, 8.3 Hz, 1H), 3.08 (dd, J = 17.9, 4.8 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.06, 199.86, 158.07, 155.69, 154.83 (d, J_F = 294.8 Hz), 156.31, 153.36, 136.55 (d, J_F = 20.0 Hz), 135.14, 128.83, 128.06, 127.75, 127.65, 126.39, 126.03, 125.09, 124.96 (d, J_F = 25.5 Hz), 113.22 (d, J_F = 7.6 Hz), 110.15 (d, J_F = 22.7 Hz), 72.68, 54.13, 29.58; ^{19}F NMR (376 MHz, $CDCl_3$) δ -124.08 (s, 1F); HRMS (EI) calcd for $C_{23}H_{16}FNO_2$ [M^+]: 357.1165; found: 357.1157.

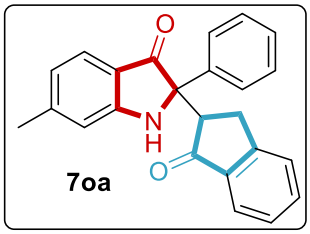
5-methyl-2-(1-oxo-2,3-dihydro-1*H*-inden-2-yl)-2-phenylindolin-3-one (7ca). Following the general procedure (C) for 5.5 h on 1.0 mmol scale, giving the compound as yellow color solid (289 mg, 82%); m.p. 212-213 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 (d, J = 7.7 Hz, 1H), 7.57 – 7.50 (m, 3H), 7.47 (s, 1H), 7.38 (d, J = 7.7 Hz, 1H), δ 7.37 – 7.26 (m, 4H), 7.23 (ddd, J = 7.3, 3.6, 1.3 Hz, 1H), 6.82 (d, J = 8.3 Hz, 1H), 5.66 (s, 1H), 3.70 (dd, J = 8.3, 4.9 Hz, 1H), 3.32 (dd, J = 17.9, 8.3 Hz, 1H), 3.09 (dd, J = 17.8, 4.8 Hz, 1H), 2.29 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.07, 200.21, 158.18, 153.36, 138.39, 137.20, 136.57, 134.91, 129.11, 128.67, 127.76, 127.47, 126.31, 126.04, 124.59, 124.01, 120.91, 112.01, 71.96, 53.90, 29.64, 20.51; HRMS (EI) calcd for $C_{24}H_{19}NO_2$ [M^+]: 353.1415; found: 353.1424.



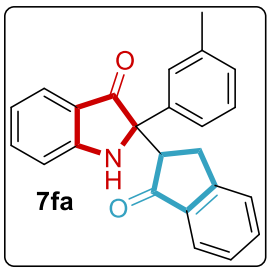
5-ethyl-2-(1-oxo-2,3-dihydro-1*H*-inden-2-yl)-2-phenylindolin-3-one (7na). Following the general procedure (C) for 9 h on a 1.0 mmol scale, giving the compound as an yellow color solid (275 mg, 75%); m.p. 213-214 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (d, J = 7.7 Hz, 1H), 7.61 – 7.46 (m, 4H), 7.39 (dd, J = 7.7, 0.8 Hz, 1H), 7.35 – 7.26 (m, 4H), 7.24 – 7.20 (m, 1H), 6.85 (d, J = 8.3 Hz, 1H), 5.71 (s, 1H), 3.68 (dd, J = 8.3, 4.9 Hz, 1H), 3.33 (dd, J = 17.9, 8.2 Hz, 1H), 3.12 (dd, J = 17.8, 5.0 Hz, 1H), 2.60 (q, J = 7.5 Hz, 2H), 1.34 – 0.85 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.11, 200.22, 158.28, 153.31, 137.45, 137.08, 136.50, 135.54, 134.85, 128.55, 127.67, 127.39, 126.23, 126.02, 123.93, 123.27, 120.78, 112.01, 71.85, 53.76, 29.52, 27.87, 15.38; HRMS (ESI) calcd for $C_{25}H_{21}NO_2$ [M^+]: 367.1572; found: 367.1577.



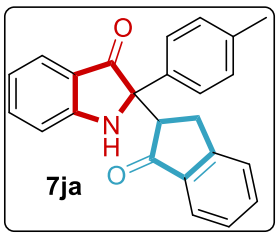
6-methyl-2-(1-oxo-2,3-dihydro-1*H*-inden-2-yl)-2-phenylindolin-3-one (7oa). Following the general procedure (C) for 9 h on a 1.0 mmol scale, giving the compound as a white solid (282 mg, 80%); m.p. 258-259 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (d, *J* = 7.6 Hz, 1H), 7.62 – 7.47 (m, 4H), 7.39 (d, *J* = 7.7 Hz, 1H), 7.36 – 7.27 (m, 3H), 7.24 – 7.20 (m, 1H), 6.78 – 6.65 (m, 2H), 5.81 (s, 1H), 3.64 (dd, *J* = 8.3, 4.9 Hz, 1H), 3.34 (dd, *J* = 17.9, 8.3 Hz, 1H), 3.14 (dd, *J* = 17.9, 4.9 Hz, 1H), 2.35 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.39, 199.52, 160.25, 153.48, 148.71, 137.18, 136.64, 135.01, 128.66, 127.81, 127.52, 126.39, 126.19, 124.99, 124.07, 121.36, 118.50, 112.24, 71.74, 53.80, 29.62, 22.47; HRMS (EI) calcd for $C_{24}H_{19}NO_2$ [M^+]: 353.1415; found: 353.1417.



2-(1-oxo-2,3-dihydro-1*H*-inden-2-yl)-2-(m-tolyl)indolin-3-one (7fa). Following the general procedure (C) for 10 h on a 1.0 mmol scale, giving the compound as a yellow color solid (317 mg, 90%); m.p. 193-194 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.71 – 7.63 (m, 2H), 7.54 (td, *J* = 7.6, 1.2 Hz, 1H), 7.44 (ddd, *J* = 8.3, 7.1, 1.3 Hz, 1H), 7.38 (d, *J* = 7.7 Hz, 1H), 7.32 (dd, *J* = 9.7, 5.1 Hz, 3H), 7.17 (t, *J* = 7.7 Hz, 1H), 7.05 (d, *J* = 7.5 Hz, 1H), 6.97 – 6.84 (m, 2H), 5.80 (s, 1H), 3.69 (dd, *J* = 8.3, 5.0 Hz, 1H), 3.32 (dd, *J* = 17.9, 8.4 Hz, 1H), 3.09 (dd, *J* = 17.9, 4.9 Hz, 1H), 2.30 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.47, 200.60, 160.12, 153.73, 138.75, 137.31, 137.26, 136.93, 135.29, 129.00, 128.92, 127.85, 127.05, 126.69, 125.57, 124.37, 123.44, 121.12, 119.81, 112.40, 71.95, 54.30, 30.02, 21.93; HRMS (ESI) calcd for $C_{24}H_{19}NO_2$ [M^+]: 353.1415; found: 353.1418.

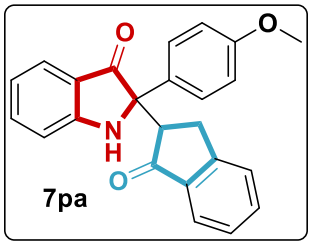


2-(1-oxo-2,3-dihydro-1*H*-inden-2-yl)-2-(p-tolyl)indolin-3-one (7ja). Following the general procedure (C) for 12 h on a 1.0 mmol scale, giving the compound as a brown color solid (307 mg, 87%); m.p. 183-184 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 (dd, *J* = 7.6, 3.3 Hz, 2H), 7.56 (td, *J* = 8.0, 1.2 Hz, 1H), 7.49 – 7.37 (m, 4H), 7.33 (t, *J* = 7.5 Hz, 1H), 7.09 (d, *J* = 8.0 Hz, 2H), 6.90 (d, *J* = 8.2 Hz, 1H), 6.86 (t, *J* = 7.4 Hz, 1H), 5.84 (s, 1H), δ 3.65 (dd, *J* = 8.3, 4.9 Hz, 1H), 3.34 (dd, *J* = 17.8, 8.4 Hz, 1H), 3.13 (dd, *J* = 17.6, 4.9 Hz, 1H), 2.27 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.23, 200.27, 159.65, 153.34, 137.55, 136.87, 136.49, 134.87,

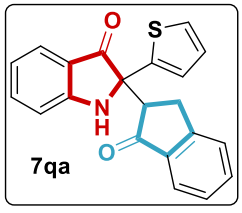


133.78, 129.30, 127.40, 126.26, 125.93, 125.14, 123.93, 119.33, 111.97, 53.69, 29.50, 20.78; HRMS (EI) calcd for $C_{24}H_{19}NO_2$ [M^+]: 353.1415; found: 353.1422.

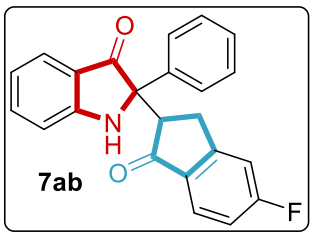
2-(4-methoxyphenyl)-2-(1-oxo-2,3-dihydro-1*H*-inden-2-yl)indolin-3-one (7pa). Following the general procedure (C) for 12 h on a 1.0 mmol scale, giving the compound a red color solid (295 mg, 80%); m.p. 189-190 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (t, *J* = 6.8 Hz, 2H)), 7.54 (t, *J* = 7.1 Hz, 1H), 7.47 – 7.36 (m, 4H), 7.31 (t, *J* = 7.4 Hz, 1H), 6.89 – 6.79 (m, 4H), 5.99 (s, 1H), 3.73 (s, 3H), 3.57 (dd, *J* = 8.2, 5.0 Hz, 1H), 3.32 (dd, *J* = 17.9, 8.3 Hz, 1H), 3.16 (dd, *J* = 17.9, 4.8 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.46, 200.47, 159.58, 159.02, 153.42, 136.91, 136.45, 134.90, 128.58, 127.38, 127.35, 126.26, 125.10, 123.88, 120.52, 119.26, 113.90, 70.92, 55.07, 53.67, 29.37. HRMS (EI) calcd for $C_{24}H_{19}NO_3$ [M^+]: 369.1364; found: 369.1372.



2-(1-oxo-2,3-dihydro-1*H*-inden-2-yl)-2-(thiophen-2-yl)indolin-3-one (7qa). Following the general procedure (C) for 10 h on a 1.0 mmol scale, giving the compound as a brown color solid (243 mg, 65%); m.p. 164-165°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 (t, *J* = 7.6 Hz, 2H), 7.58 (td, *J* = 7.6, 1.2, 1H), 7.50 (ddd, *J* = 8.4, 7.1, 1.4 Hz, 1H), 7.46 (d, *J* = 7.6, 1H), 7.34 (dd, *J* = 11.0, 3.9 Hz, 1H), 7.15 (ddd, *J* = 6.3, 4.4, 1.2 Hz, 2H), 6.97 (d, *J* = 8.4, 1H), 6.91 – 6.85 (m, 2H), 6.54 (s, 1H), 3.59 (dd, *J* = 17.6, 4.2 Hz, 1H), 3.43 – 3.24 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.17, 199.24, 159.51, 154.09, 137.54, 136.49, 135.35, 127.53, 126.91, 126.49, 125.43, 125.37, 125.02, 124.06, 119.94, 119.74, 112.71, 69.73, 53.73, 28.94. HRMS (EI) calcd for $C_{21}H_{15}NO_2S$ [M^+]: 345.0823; found: 345.0832.

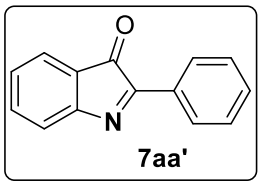


2-(5-fluoro-1-oxo-2,3-dihydro-1*H*-inden-2-yl)-2-phenylindolin-3-one (7ab). Following the general procedure (C) for 14 h on a 1.0 mmol scale, giving the compound as a brown color solid (178 mg, 50%); m.p. 172-174 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 – 7.63 (m, 2H), 7.53 (dd, *J* = 5.3, 3.3 Hz, 2H), 7.47 (ddd, *J* = 8.3, 7.2, 1.3 Hz, 1H), 7.34 – 7.27 (m, 2H), 7.26 – 7.17 (m, 1H), 7.10 – 6.98 (m, 2H), 6.93 (d, *J* = 8.2 Hz, 1H), 6.87 (t, *J* = 7.4 Hz, 1H), 5.92 (s,



1H), 3.65 (dd, J = 8.2, 4.9 Hz, 1H), 3.32 (dd, J = 18.2, 8.3 Hz, 1H), 3.16 (dd, J = 18.1, 4.7 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 202.53, 200.08, 167.31 (d, J_F = 257.2 Hz), 159.75, 156.37 (d, J = 10.3 Hz), 137.19, 136.60, 128.75, 127.99, 126.43 (d, J = 10.6 Hz), 126.18, 125.30, 120.67, 119.67, 115.99 (d, J = 23.8 Hz), 114.29, 113.04 (d, J = 22.3 Hz), 112.23, 71.35, 54.02, 29.45; ^{19}F NMR (376 MHz, $CDCl_3$) δ -101.68 (s, 1F); HRMS (EI) calcd for $C_{23}H_{16}FNO_2$ [M^+]: 357.1165; found: 357.1167.

2-phenyl-3H-indol-3-one (7aa') The 2-phenyl-3H-indol-3-one was prepared according to the reported procedures.[2] Red color solid; m.p. 158-159 ^oC; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.39 – 8.36 (m, 2H), 7.57 – 7.47 (m, 5H), 7.42 (d, J = 7.2 Hz, 1H), 7.25(d, J = 8.0 Hz, 1H).



7aa'

6) References

1. (a) E. Chong, S. A. Blum, *J. Am. Chem. Soc*. 2015, **137,** 10144-10147; (b) T. Saito, H. Nihei, T. Otani, T. Suyama, N. Furukawa, M. Saito, *Chem. Commun.* 2008, **0**, 172-174; (c) G. K. Dhandabani, M. R. Mutra and J.-J. Wang, *Adv. Synth. Catal*. 2018, **360**, 4754-4763.

2. (a) J.-S. Li, Y.-J. Liu, G.-W. Zhang and J.-A. Ma, *Org. Lett.*, 2017, **19**, 6364-6367; (b) J.-X. Liu, Q.-Q. Zhou, J.-G. Deng and Y.-C. Chen, *Org. Biomol. Chem.*, 2013, ***11***, 8175-8178; (c) P. Li, W. Yong, R. Sheng, W. Rao, X. Zhu and X. Zhang, *Adv. Synth. Catal.,* 2019, **361**, 201-207.

3. (a) G. C. Senadi, G. K. Dhandabani, W.-P. Hu and J.-J. Wang, *Green Chem.,* 2016, **18**, 6241-6245; (b) G. K. Dhandabani, M. R. Mutra, J.-J. Wang, *Adv. Synth. Catal.,* 2018, **360**, 4754-4763; (c) G. C. Senadi, T.-Y. Lu, G. K. Dhandabani J.-J. Wang, *Org. Lett.,* 2017, **19**, 1172-1175; (d) M. R. Mutra, G. K. Dhandabani, J.-J. Wang, *Adv. Synth. Catal.,* 2018, **360**, 3960-3968; (e) S. S. K. Boominathan, G. C. Senadi, J. K. Vandavasi, J. Y.-F. Chen and J.-J. Wang, *Chem. Eur. J.,* 2015, **21**, 3193-3197; (f) G. C. Senadi and J.-J. Wang, *Green Chem.,* 2018, **20**, 3420-3425.

7) X-ray Crystallography Data

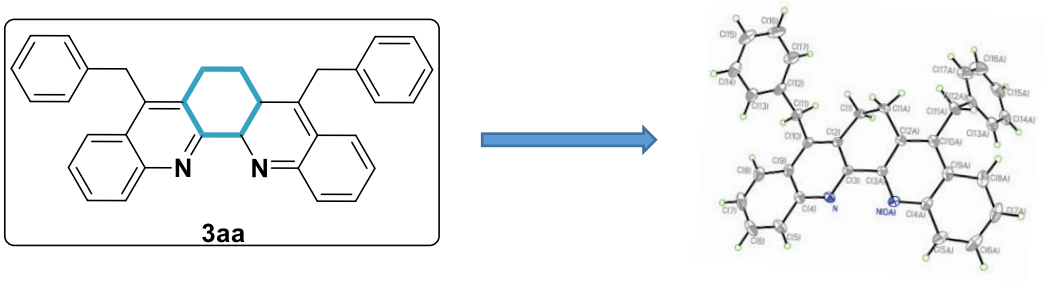


Table 1. Crystal data and structure refinement for th3175.

Identification code	th3175	
Empirical formula	C34 H26 N2	
Formula weight	462.57	
Temperature	150(2) K	
Wavelength	0.71073 Å	
Crystal system	monoclinic	
Space group	C 2/c	
Unit cell dimensions	a = 20.9893(8) Å	α= 90°.
	b = 11.0183(4) Å	β= 97.8192(16)°.
	c = 11.5218(4) Å	γ = 90°.
Volume	2639.83(17) $Å^3$	
Z	4	
Density (calculated)	1.164 Mg/m^3	
Absorption coefficient	0.068 mm^{-1}	
F(000)	976	
Crystal size	0.520 x 0.300 x 0.190 mm^3	
Theta range for data collection	3.570 to 26.389°.	
Index ranges	-26<=h<=26, -13<=k<=13, -14<=l<=14	
Reflections collected	23416	
Independent reflections	2696 [R(int) = 0.0438]	
Completeness to theta = 25.242°	99.8 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9281 and 0.8284	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2696 / 0 / 163	
Goodness-of-fit on F^2	1.024	
Final R indices [I>2sigma(I)]	R1 = 0.0531, wR2 = 0.1434	

R indices (all data)	R1 = 0.0688, wR2 = 0.1544
Extinction coefficient	n/a
Largest diff. peak and hole	0.335 and -0.403 e.Å^{-3}

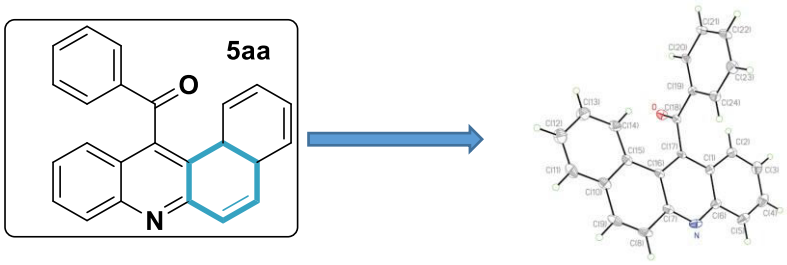


Table 1. Crystal data and structure refinement for th4036.

Identification code	th4036	
Empirical formula	C24 H19.90 N O0.45	
Formula weight	329.51	
Temperature	150(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 9.2367(4) Å	α= 104.8513(17)°.
	b = 10.2565(5) Å	β= 109.5128(15)°.
	c = 11.0747(6) Å	γ = 108.1988(15)°.
Volume	859.89(7) Å^3	
Z	2	
Density (calculated)	1.273 Mg/m^3	
Absorption coefficient	0.075 mm^{-1}	
F(000)	349	
Crystal size	0.520 x 0.420 x 0.210 mm^3	
Theta range for data collection	3.203 to 26.384°.	
Index ranges	-11<=h<=11, -12<=k<=12, -13<=l<=13	
Reflections collected	28959	
Independent reflections	3517 [R(int) = 0.0419]	
Completeness to theta = 25.242°	99.7 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9281 and 0.8922	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3517 / 0 / 238	

Goodness-of-fit on F^2	1.005
Final R indices [I>2sigma(I)]	R1 = 0.0425, wR2 = 0.1020
R indices (all data)	R1 = 0.0598, wR2 = 0.1139
Extinction coefficient	n/a
Largest diff. peak and hole	0.190 and -0.218 e.$Å^{-3}$

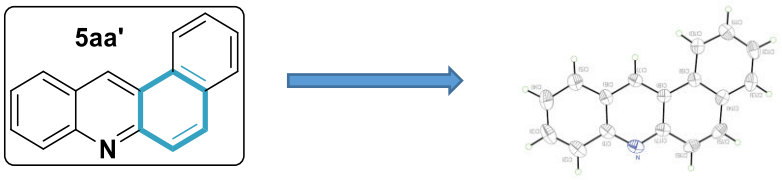


Table 1. Crystal data and structure refinement for th6194.

Identification code	th6194	
Empirical formula	C17 H11 N	
Formula weight	229.27	
Temperature	150(2) K	
Wavelength	0.71073 Å	
Crystal system	orthorhombic	
Space group	P 21 21 21	
Unit cell dimensions	a = 4.5356(4) Å	α= 90°.
	b = 17.1340(14) Å	β= 90°.
	c = 17.5416(14) Å	γ = 90°.
Volume	1363.2(2) $Å^3$	
Z	4	
Density (calculated)	1.117 Mg/m^3	
Absorption coefficient	0.065 mm^{-1}	
F(000)	480	
Crystal size	0.450 x 0.120 x 0.050 mm^3	
Theta range for data collection	3.324 to 26.413°.	
Index ranges	-5<=h<=5, -21<=k<=21, -21<=l<=21	
Reflections collected	18945	
Independent reflections	2789 [R(int) = 0.0583]	
Completeness to theta = 25.242°	99.3 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9281 and 0.8151	

Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2789 / 0 / 163
Goodness-of-fit on F^2	1.032
Final R indices [I>2sigma(I)]	R1 = 0.0509, wR2 = 0.1307
R indices (all data)	R1 = 0.0781, wR2 = 0.1496
Absolute structure parameter	1.1(10)
Extinction coefficient	n/a
Largest diff. peak and hole	0.164 and -0.197 e.Å^{-3}

Table 1. Crystal data and structure refinement for th5094.

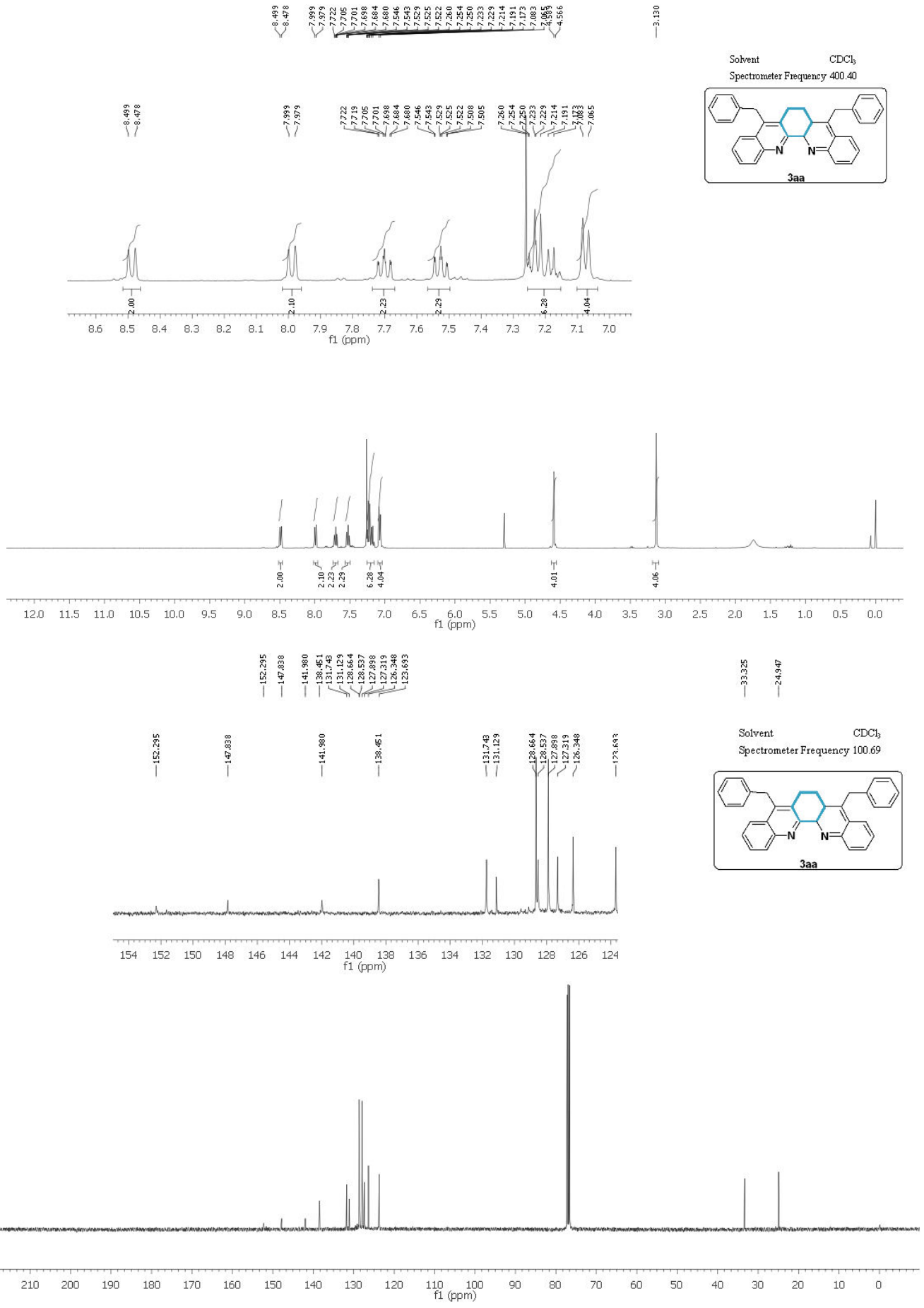
Identification code	th5094	
Empirical formula	C23 H16 F N O2	
Formula weight	357.37	
Temperature	150(2) K	
Wavelength	0.71073 Å	
Crystal system	trigonal	
Space group	R 3 c	
Unit cell dimensions	a = 29.6855(15) Å	α= 90°.
	b = 29.6855(15) Å	β= 90°.
	c = 11.124(2) Å	γ = 120°.
Volume	8489.4(18) Å^3	
Z	18	
Density (calculated)	1.258 Mg/m^3	
Absorption coefficient	0.087 mm^{-1}	
F(000)	3348	
Crystal size	0.350 x 0.100 x 0.070 mm^3	
Theta range for data collection	2.745 to 26.387°.	
Index ranges	-36<=h<=37, -36<=k<=37, -13<=l<=13	

Reflections collected	41148
Independent reflections	3846 [R(int) = 0.1156]
Completeness to theta = 25.242°	99.8 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9281 and 0.8247
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3846 / 1 / 249
Goodness-of-fit on F^2	1.060
Final R indices [I>2sigma(I)]	R1 = 0.0523, wR2 = 0.0995
R indices (all data)	R1 = 0.0912, wR2 = 0.1127
Absolute structure parameter	-0.1(5)
Extinction coefficient	0.00066(16)
Largest diff. peak and hole	0.232 and -0.239 e.$Å^{-3}$

Table 1. Crystal data and structure refinement for th5094.

Identification code	th5094
Empirical formula	C23 H16 F N O2
Formula weight	357.37
Temperature	150(2) K
Wavelength	0.71073 Å
Crystal system	trigonal
Space group	R 3 c
Unit cell dimensions	a = 29.6855(15) Å α= 90°.
	b = 29.6855(15) Å β= 90°.
	c = 11.124(2) Å γ= 120°.
Volume	8489.4(18) Å3
Z	18
Density (calculated)	1.258 Mg/m3
Absorption coefficient	0.087 mm-1
F(000)	3348
Crystal size	0.350 x 0.100 x 0.070 mm3
Theta range for data collection	2.745 to 26.387°.
Index ranges	-36<=h<=37, -36<=k<=37, -13<=l<=13
Reflections collected	41148
Independent reflections	3846 [R(int) = 0.1156]
Completeness to theta = 25.242°	99.8 %

Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9281 and 0.8247
Refinement method	Full-matrix least-squares on F2
Data / restraints / parameters	3846 / 1 / 249
Goodness-of-fit on F2	1.060
Final R indices [I>2sigma(I)]	R1 = 0.0523, wR2 = 0.0995
R indices (all data)	R1 = 0.0912, wR2 = 0.1127
Absolute structure parameter	-0.1(5)
Extinction coefficient	0.00066(16)
Largest diff. peak and hole	0.232 and -0.239 e.Å-3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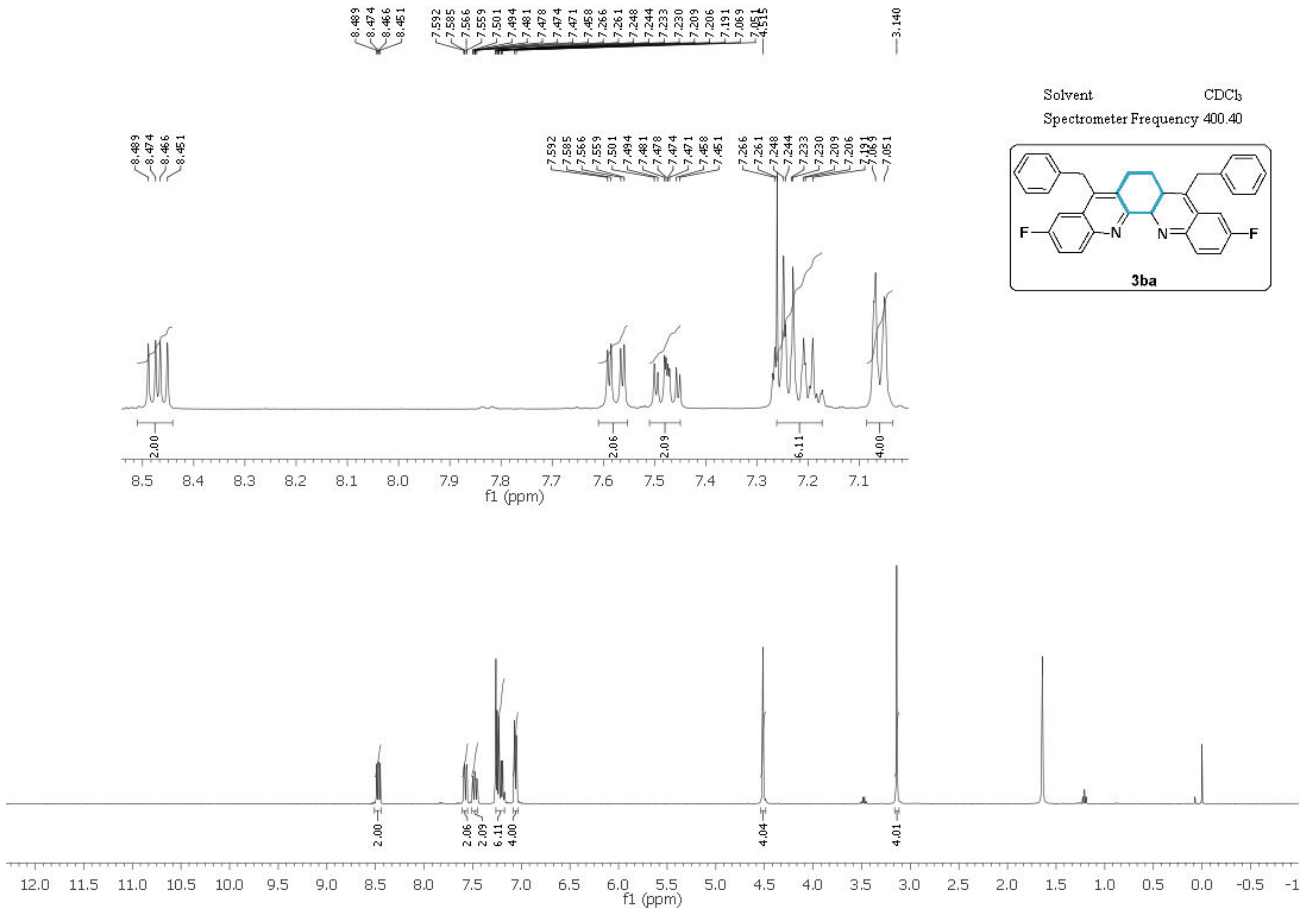
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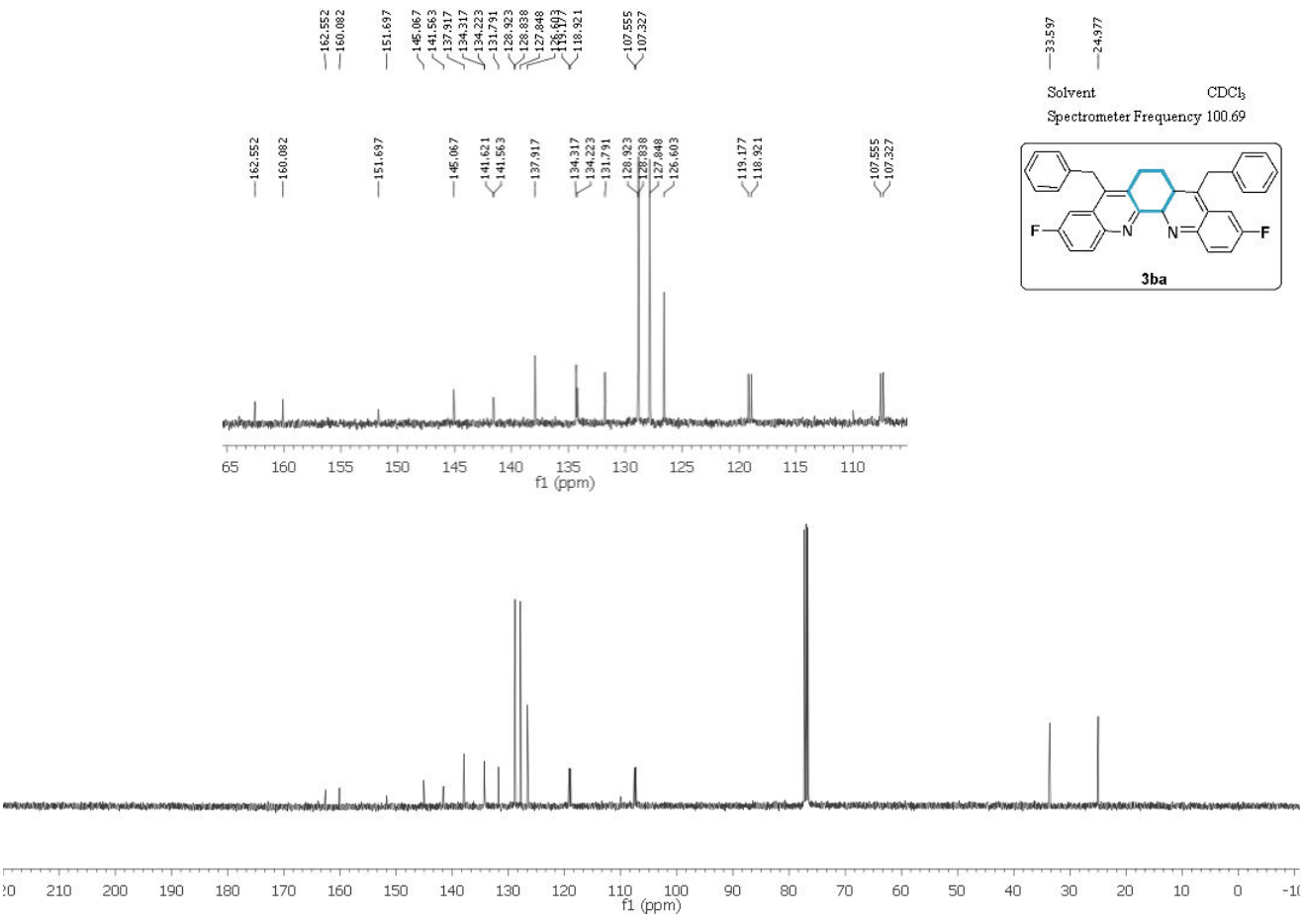
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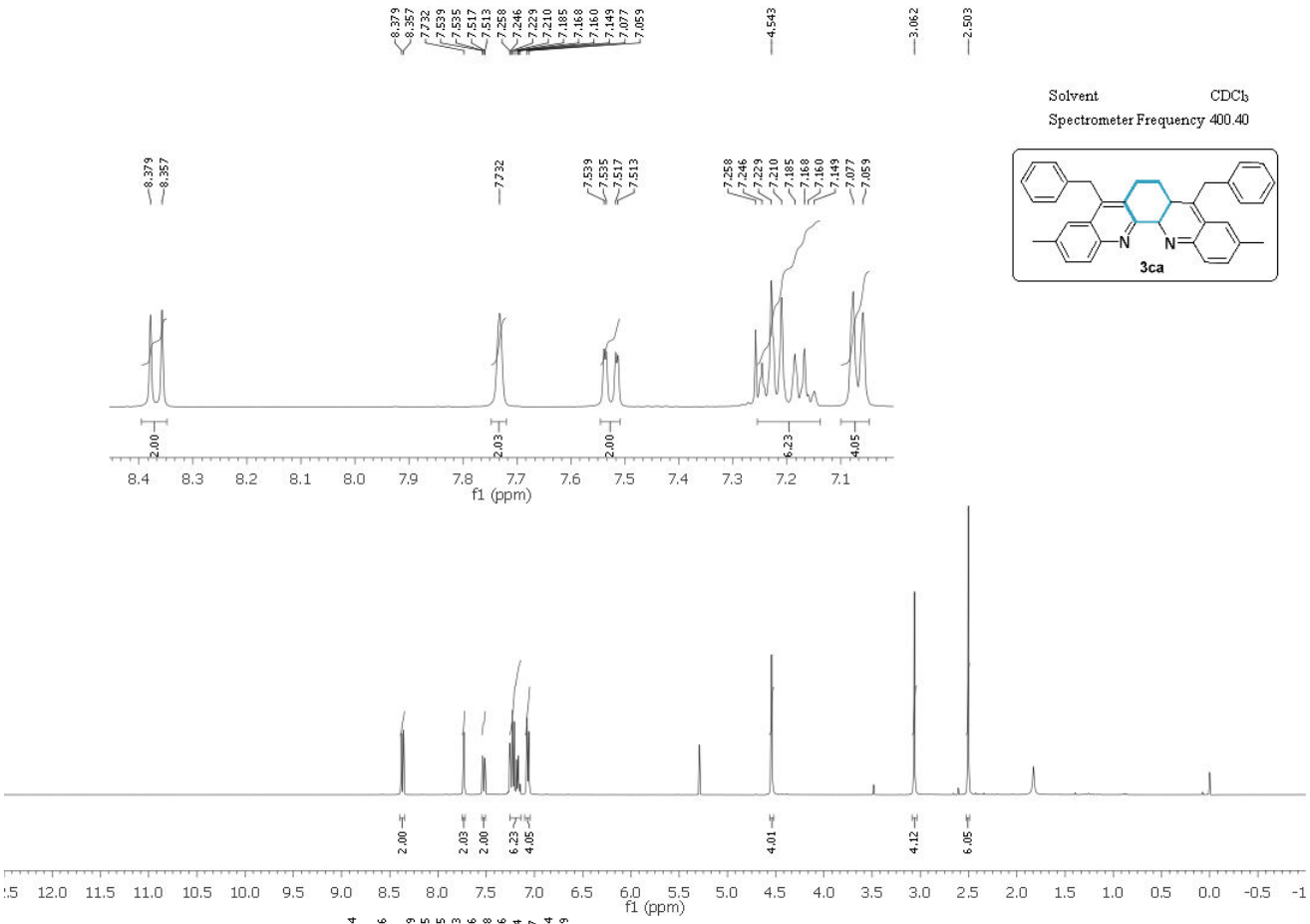
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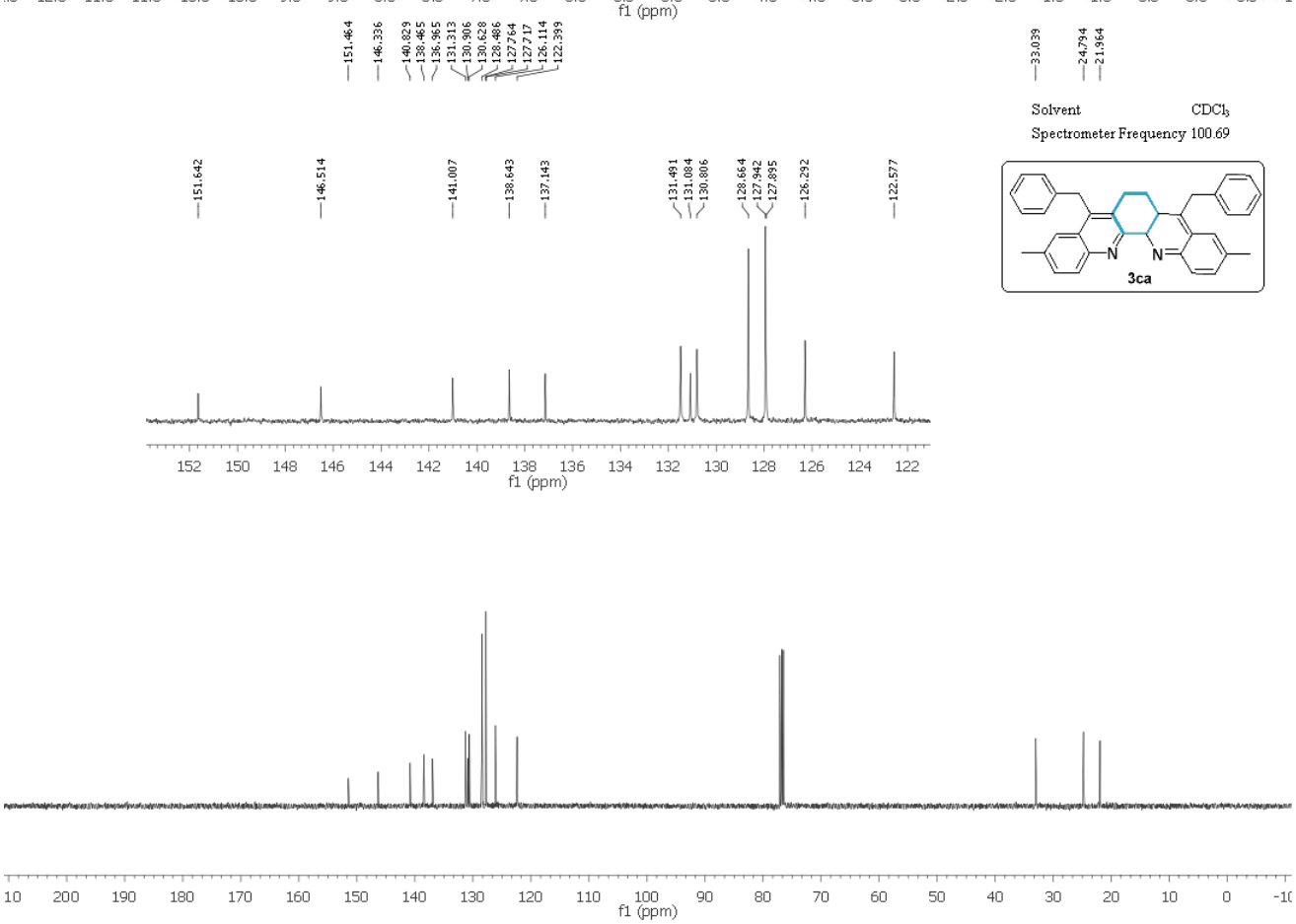
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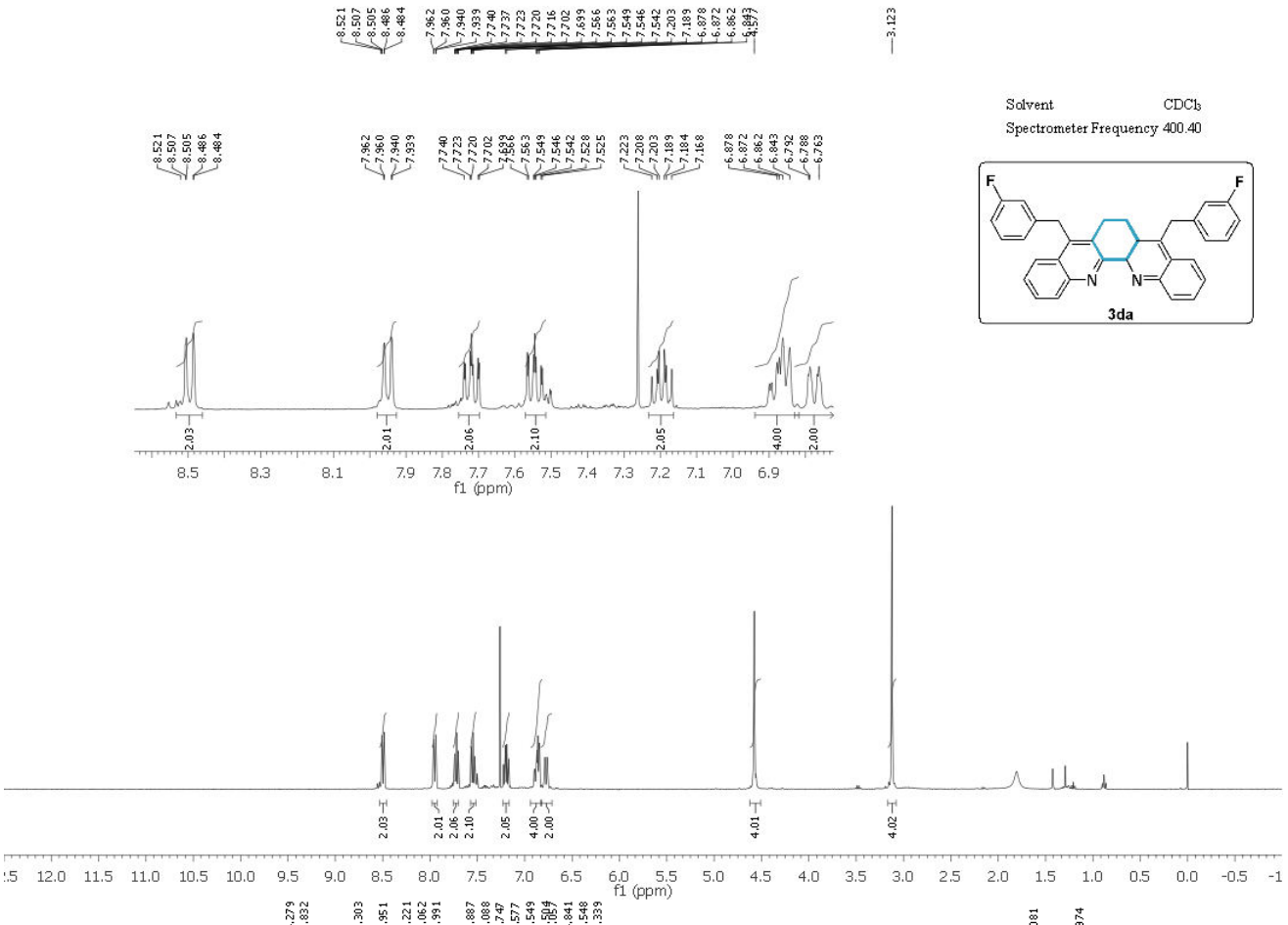
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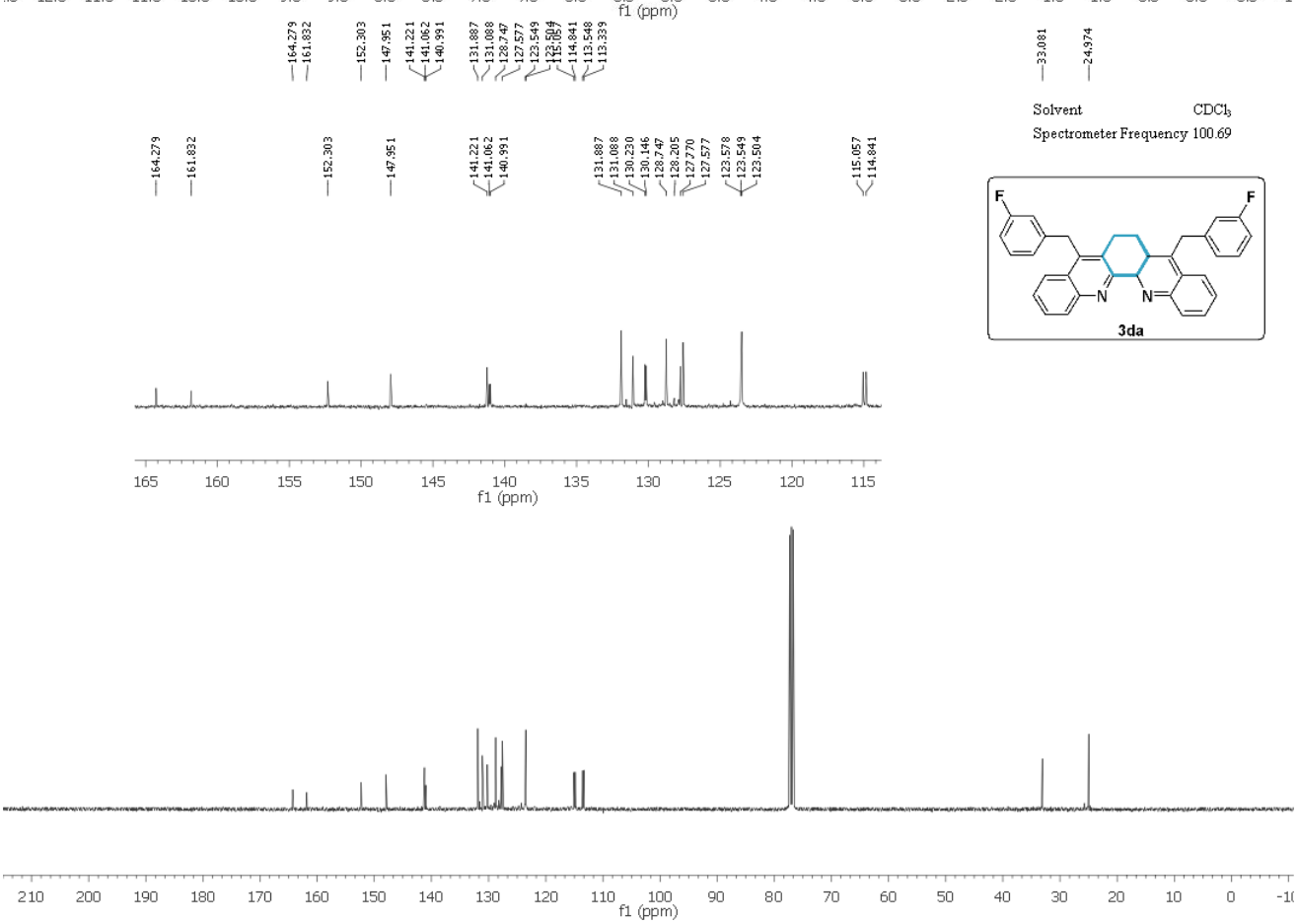
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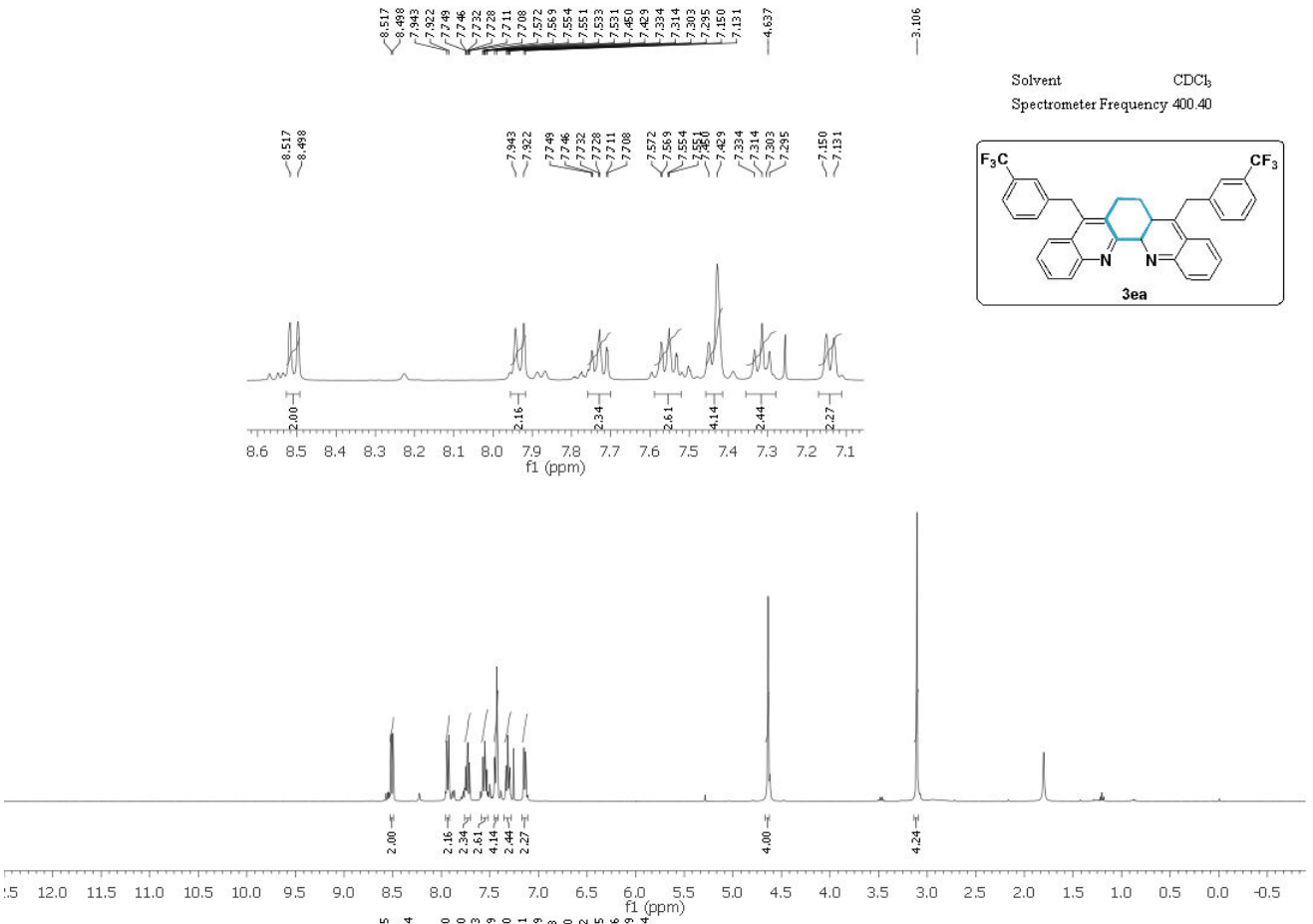
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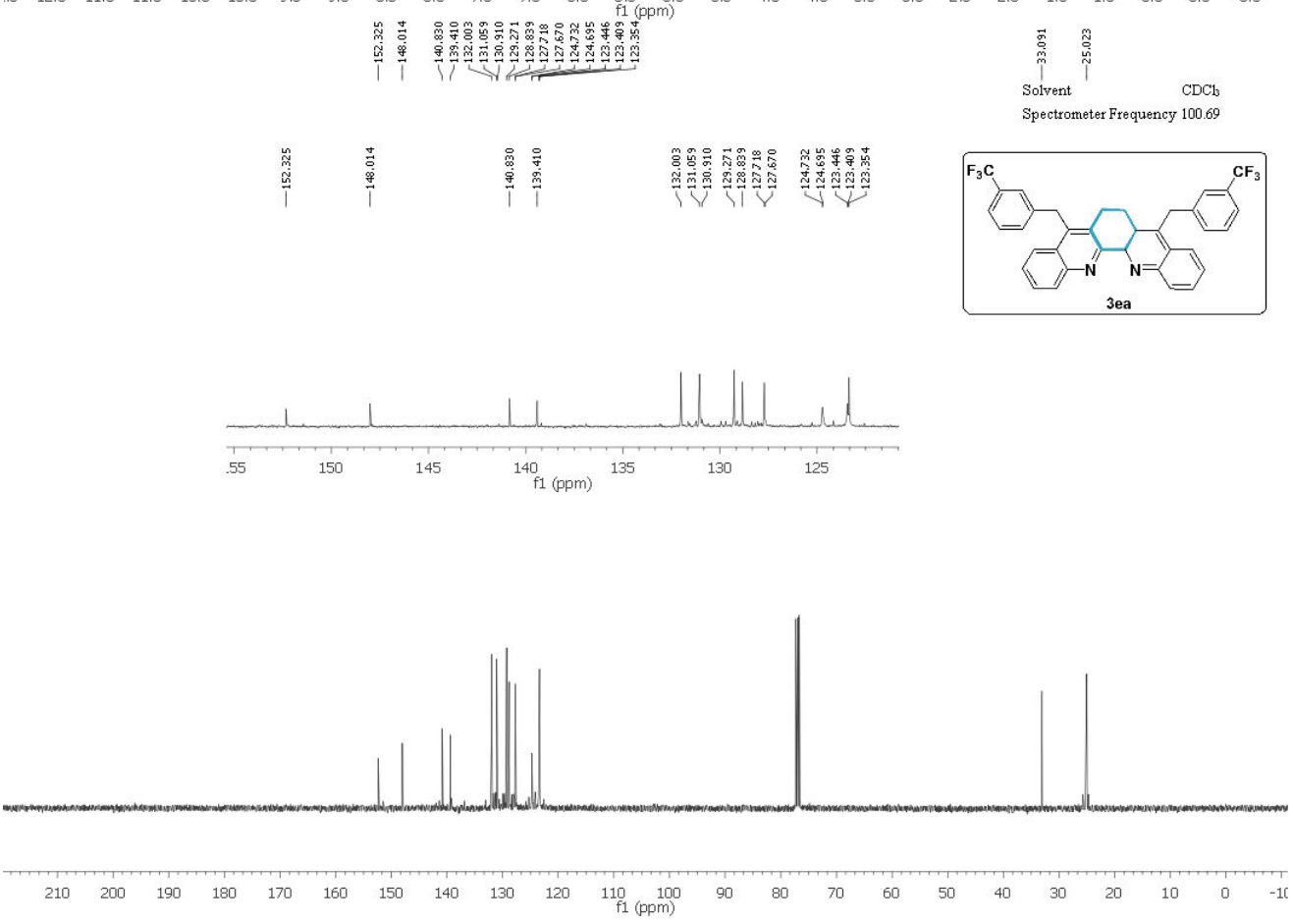
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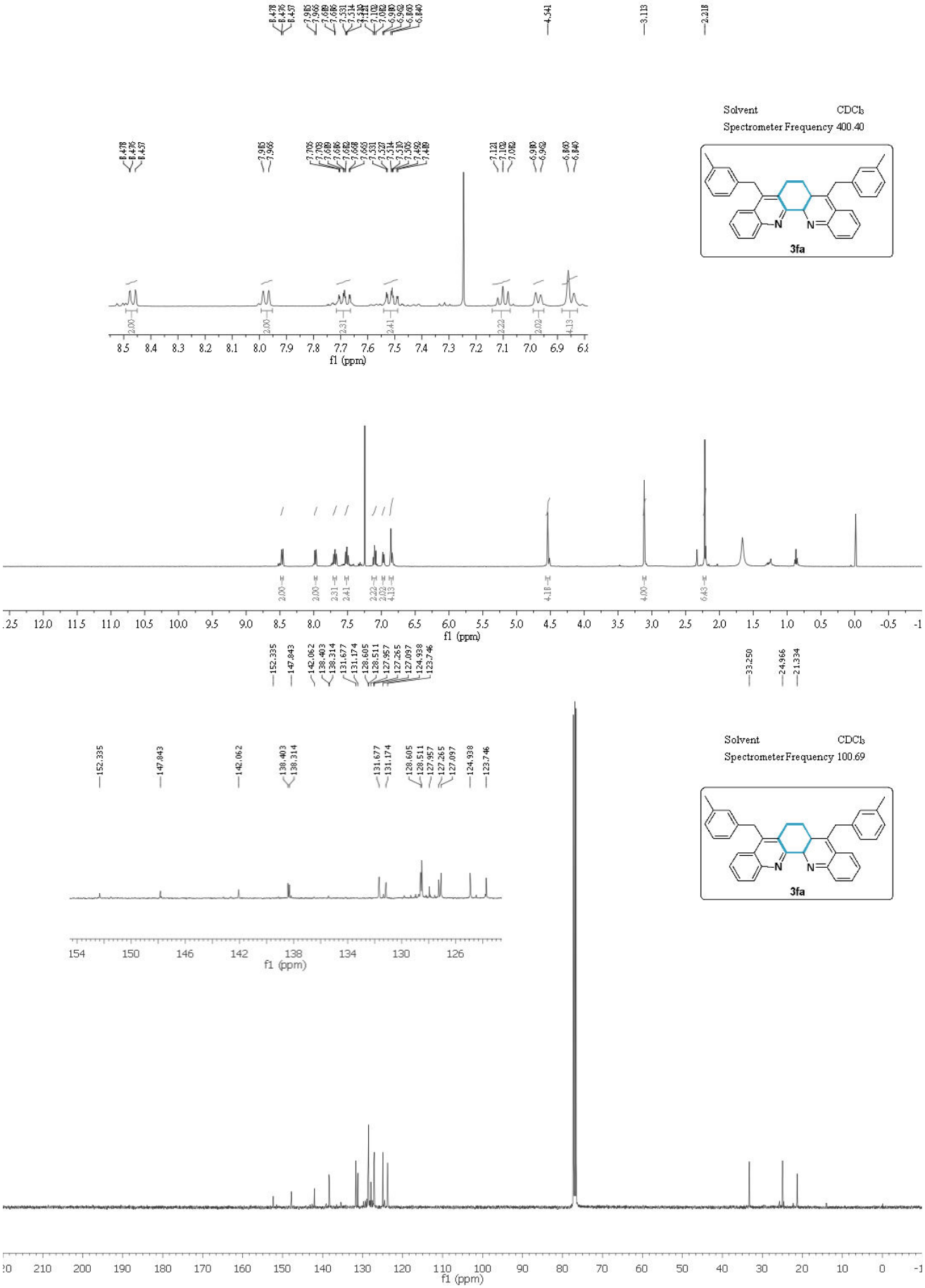
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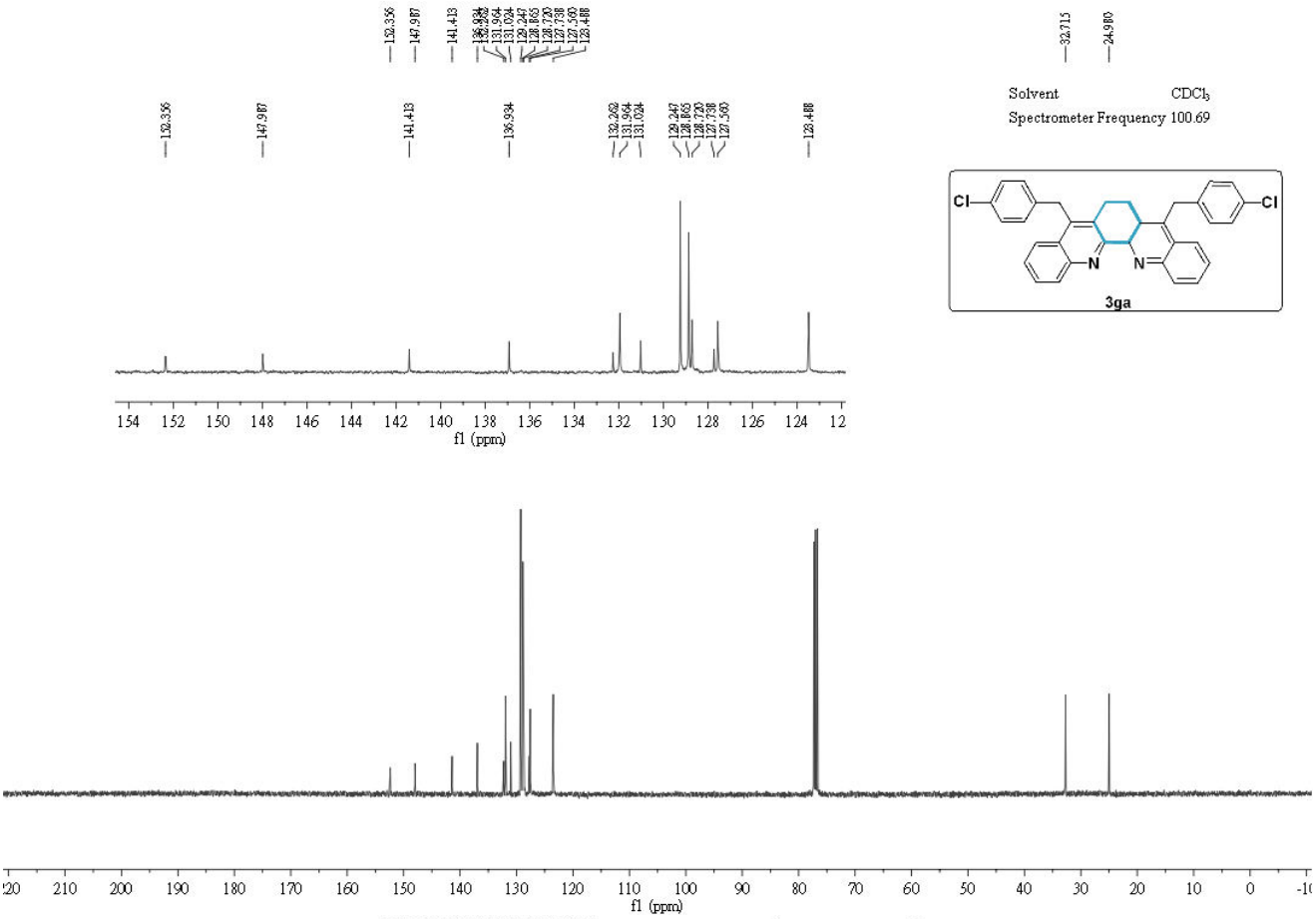
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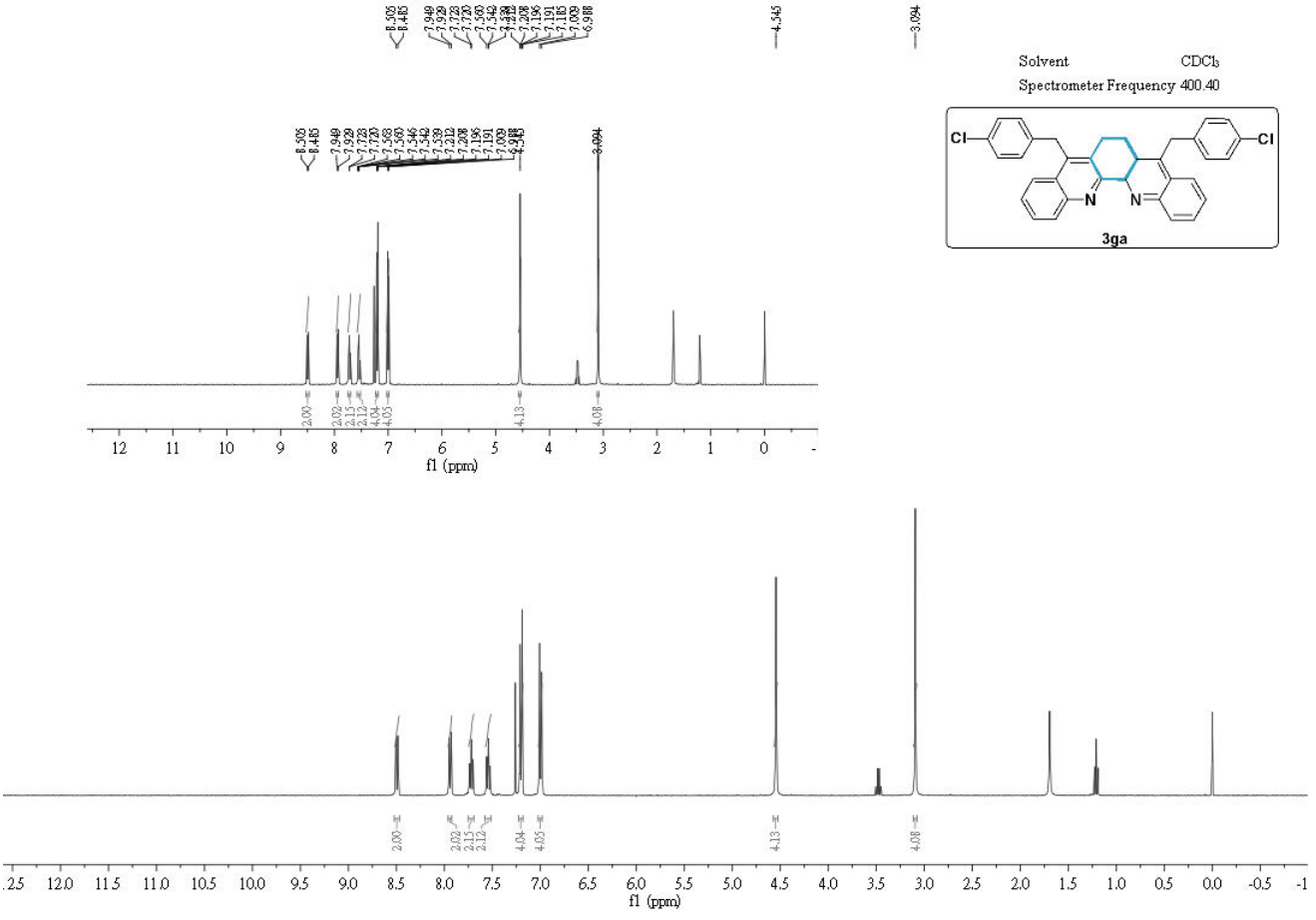
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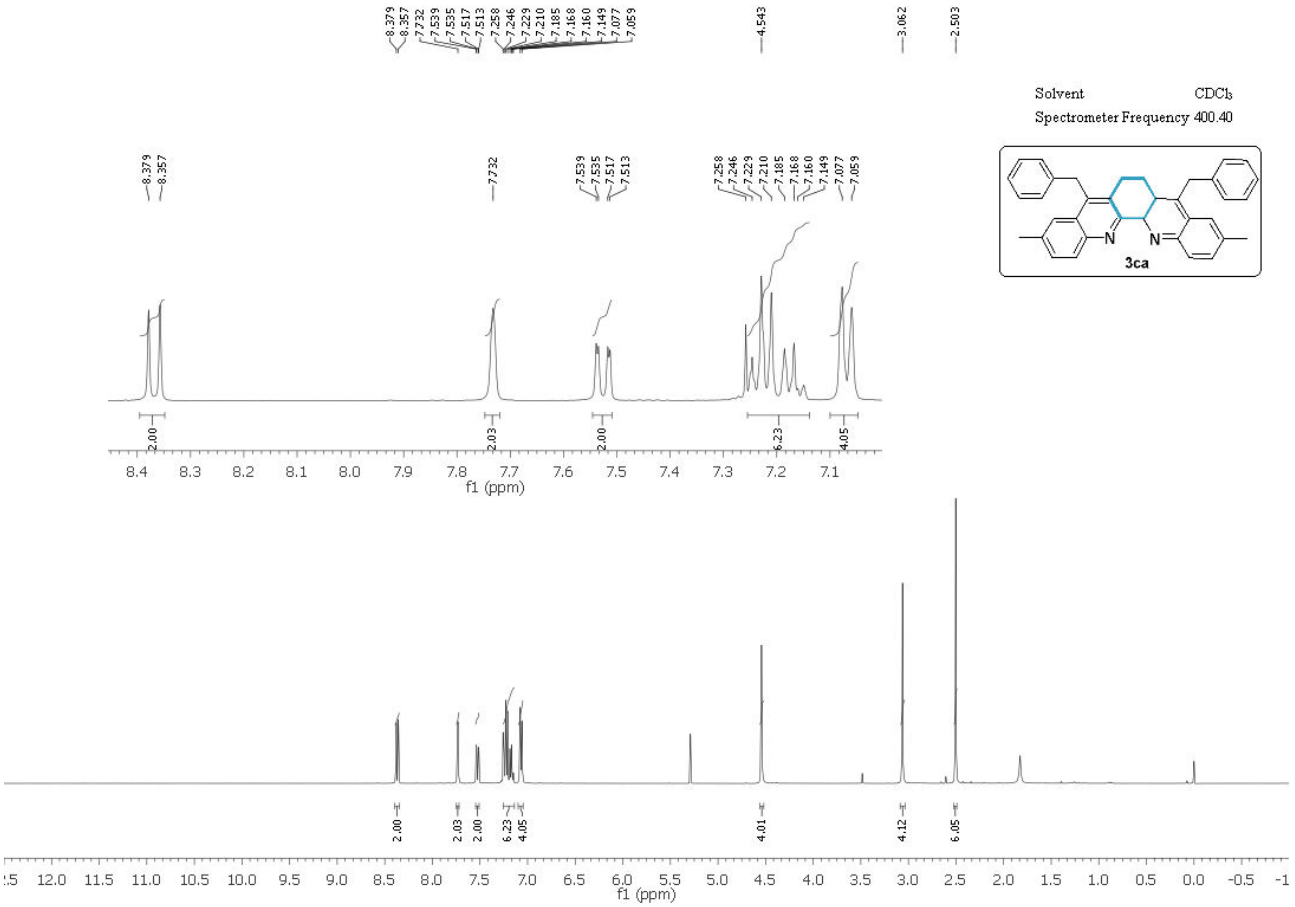
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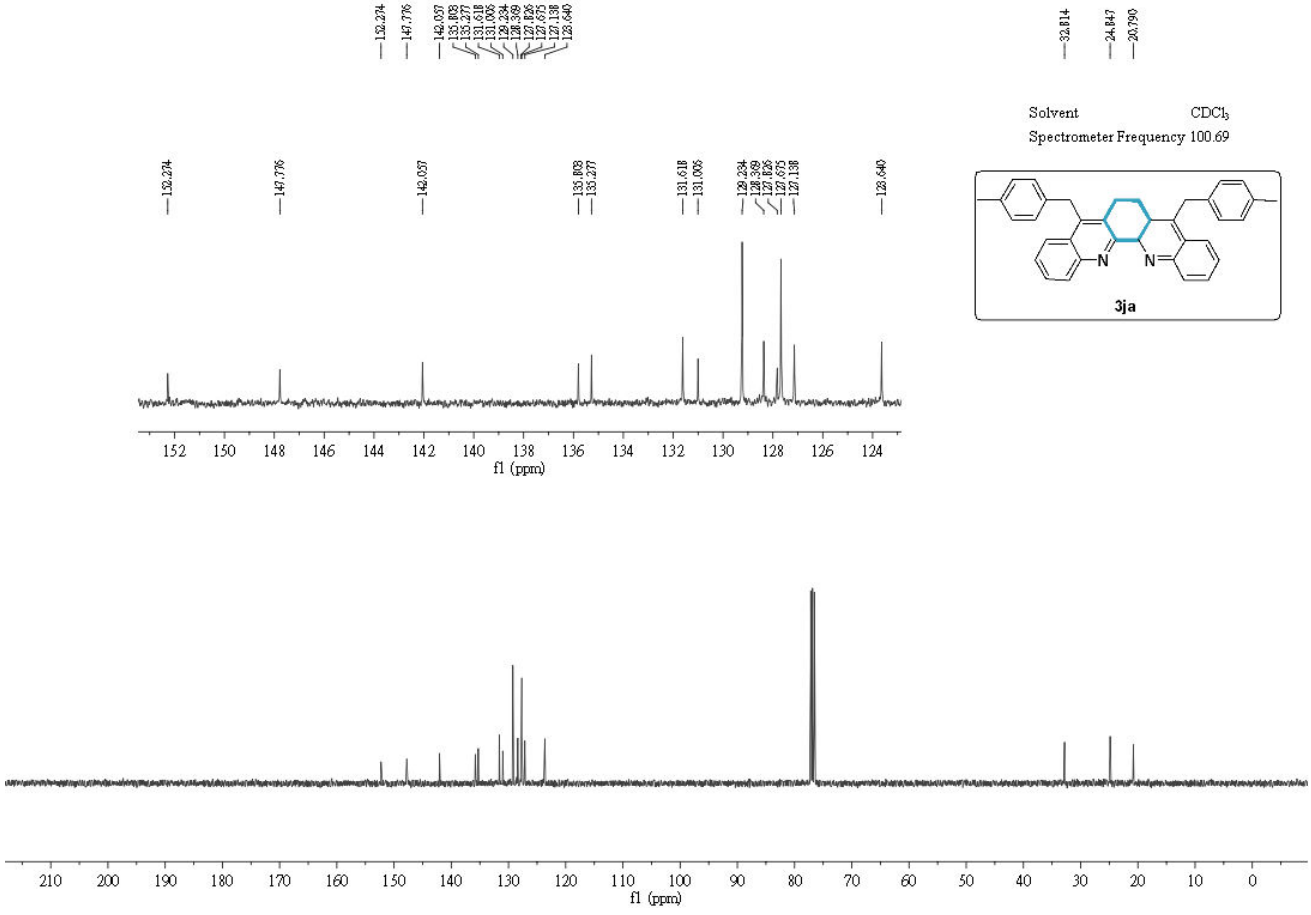
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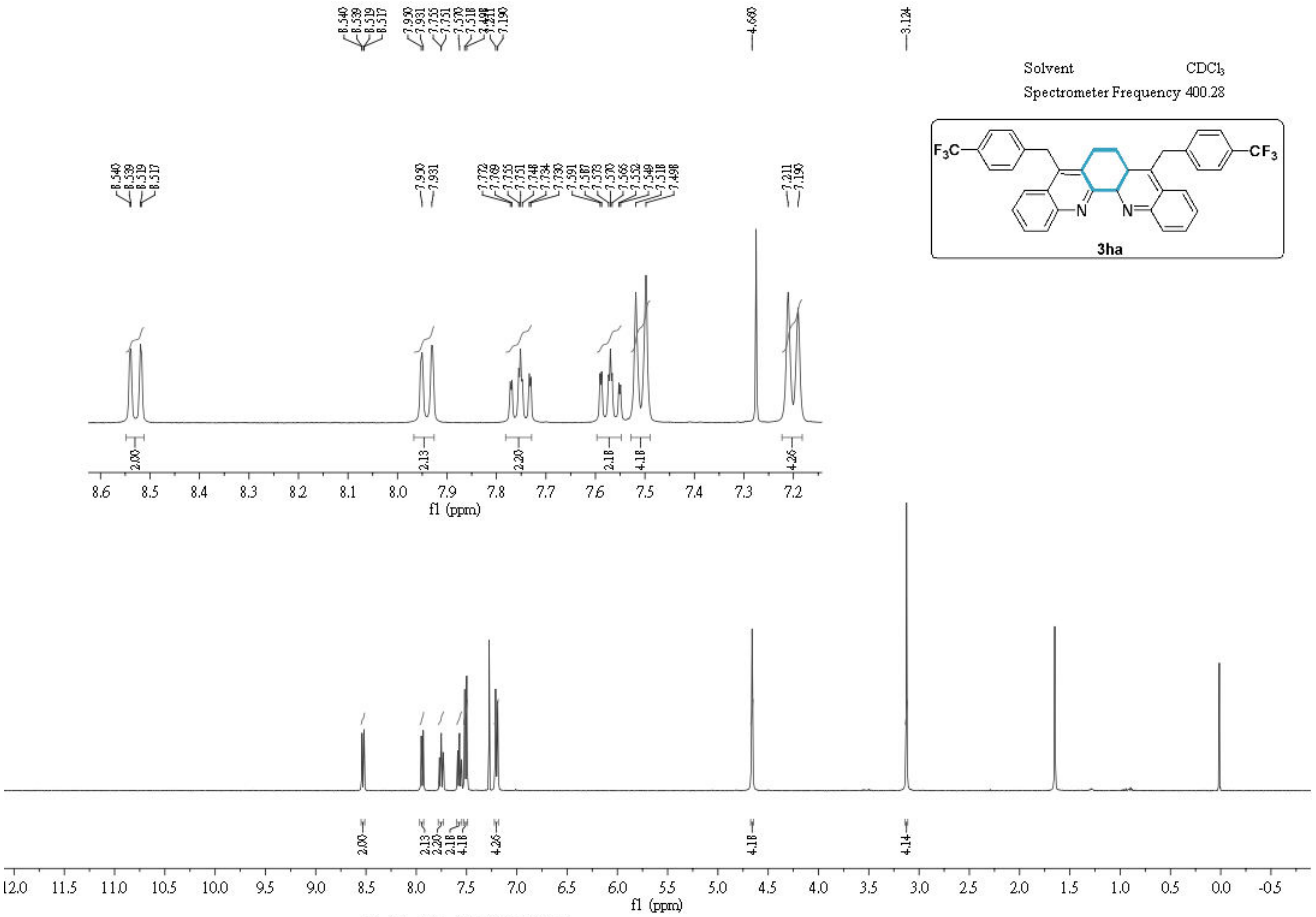
f1 (ppm)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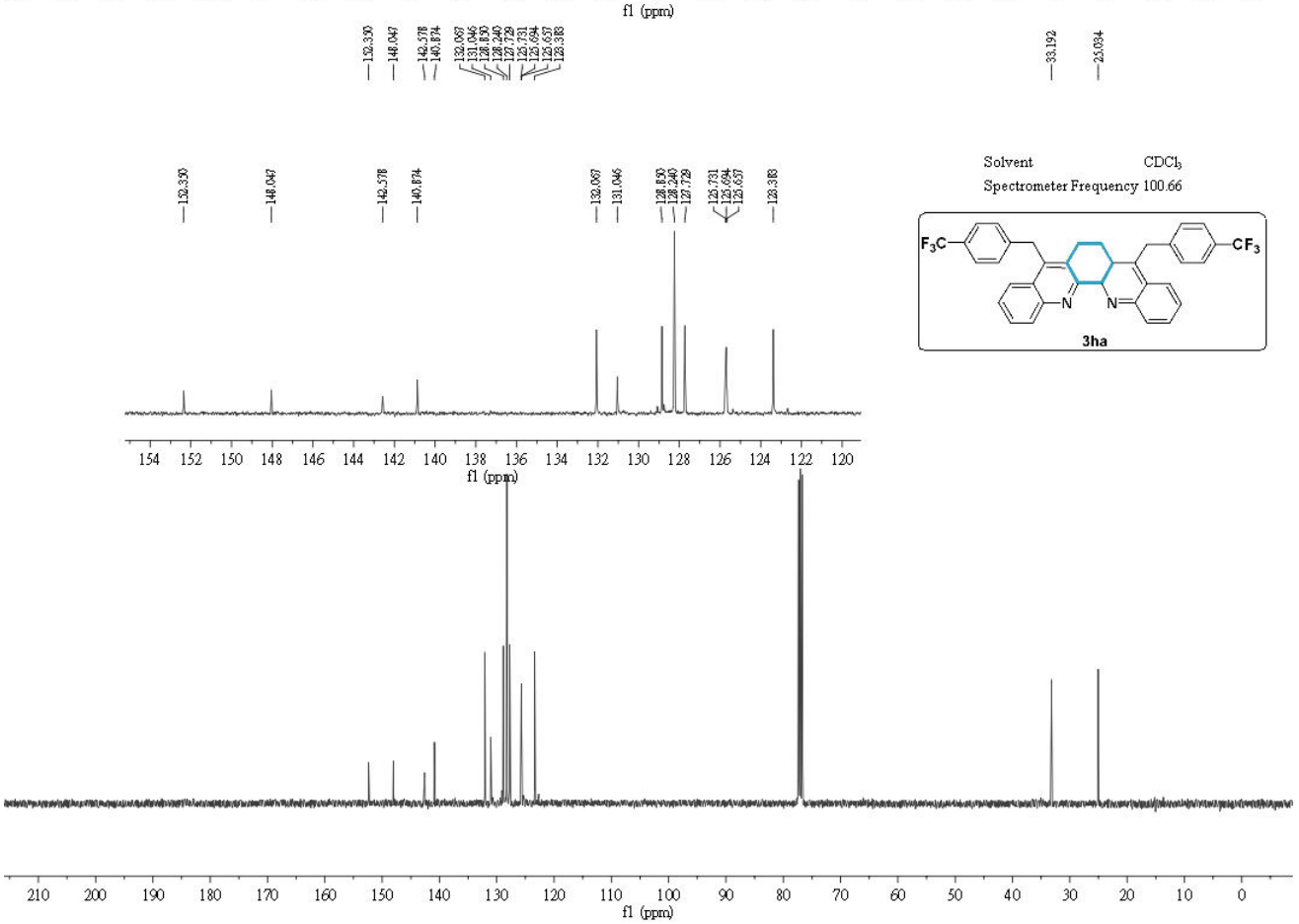
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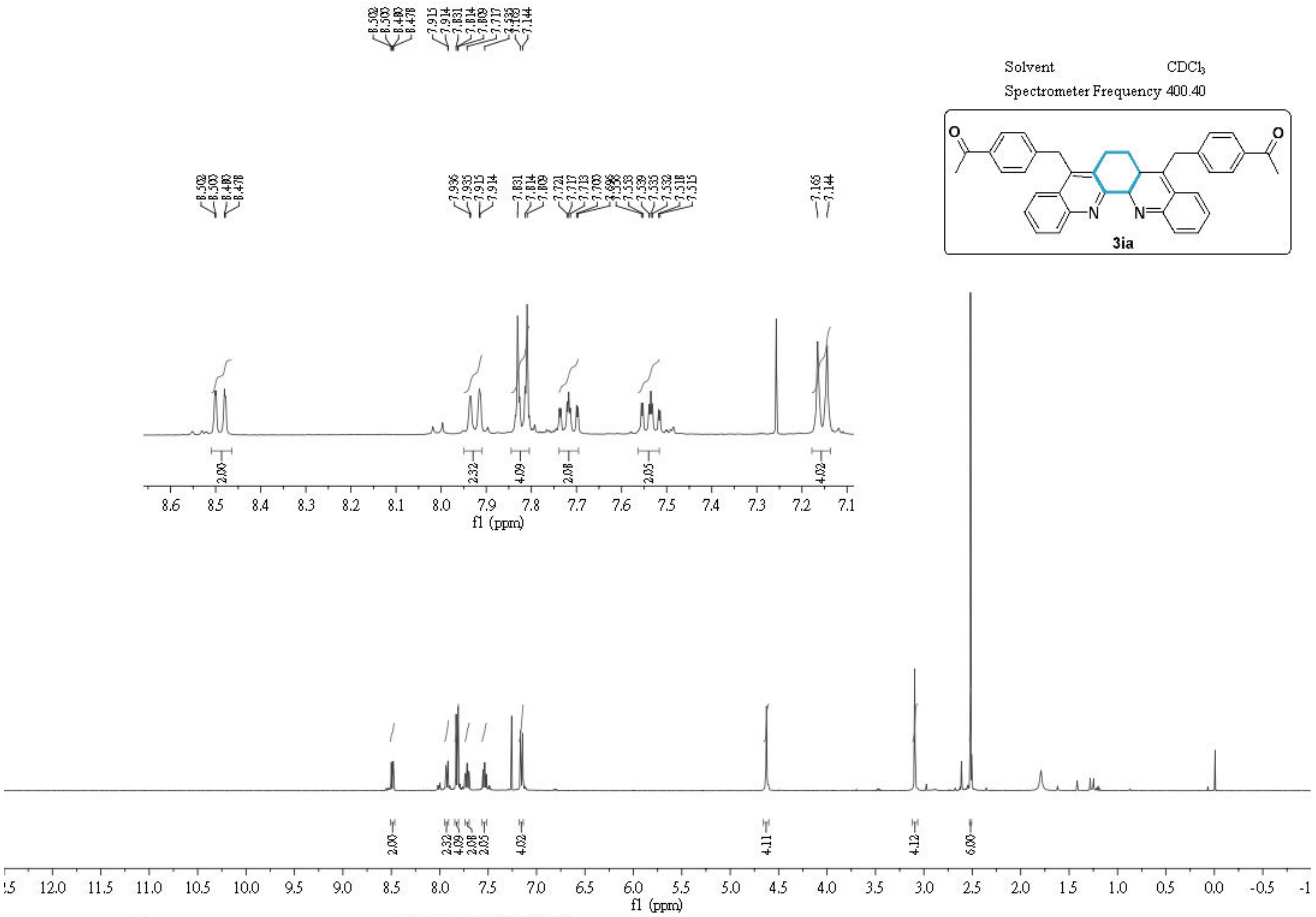
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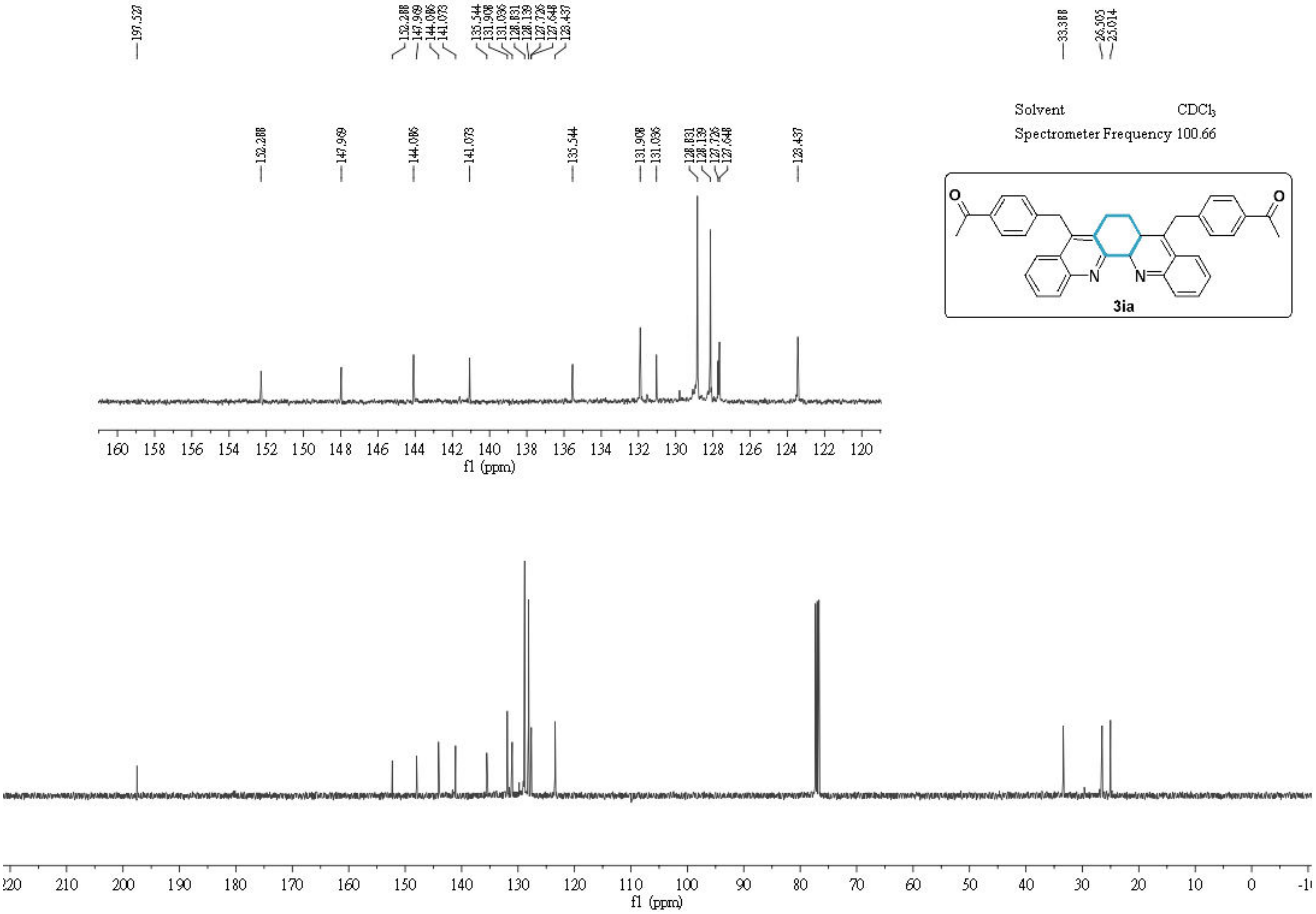
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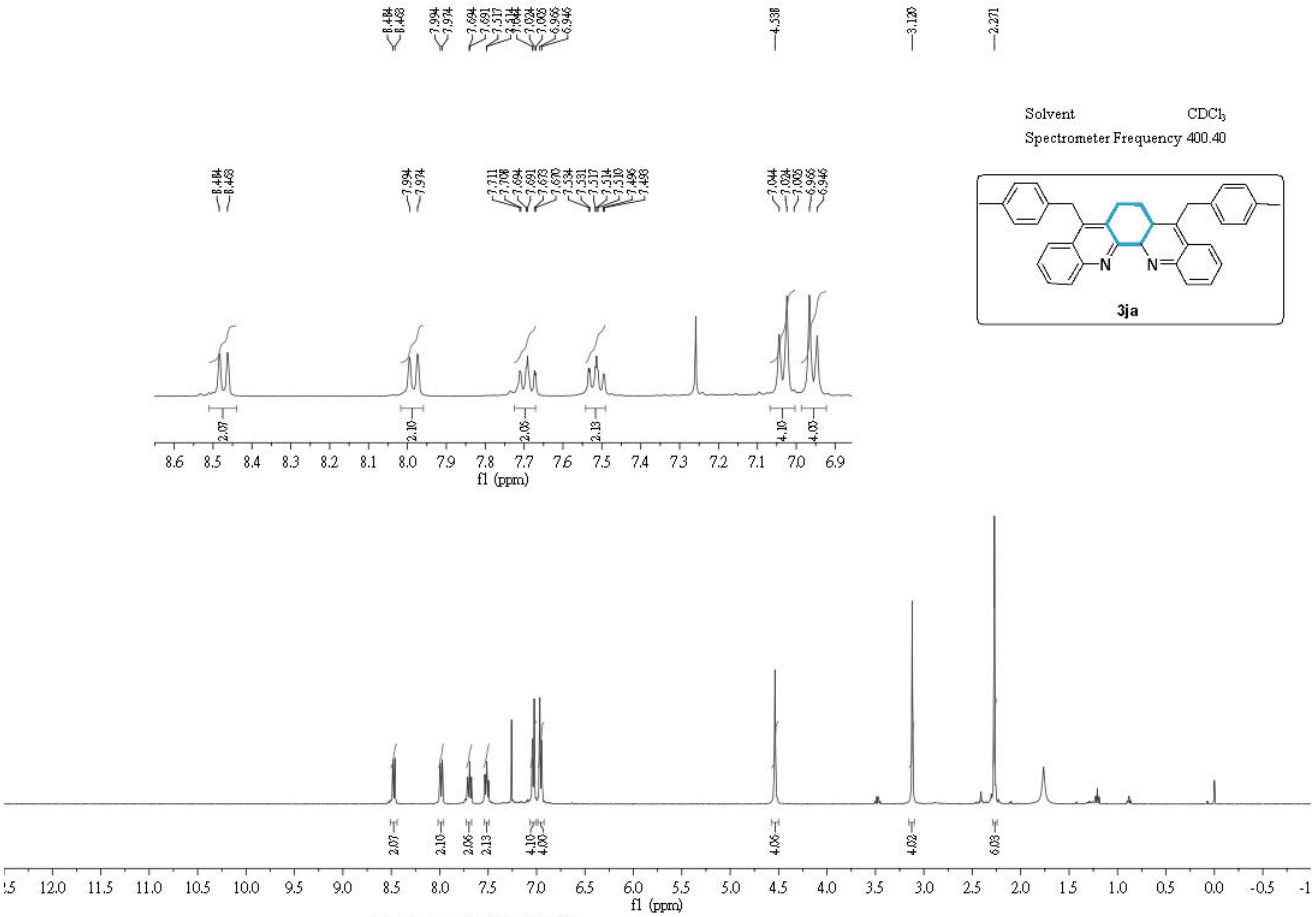
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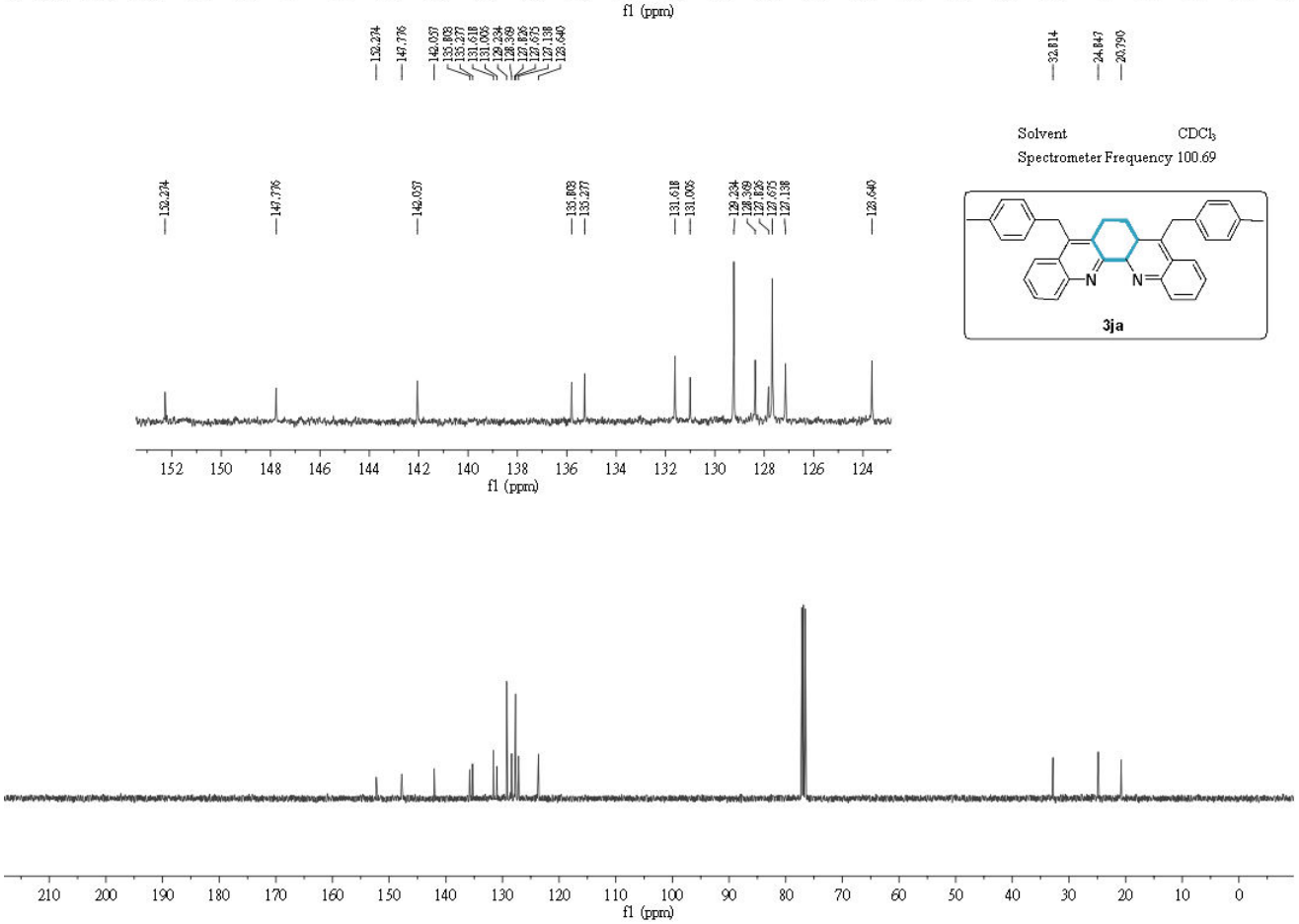
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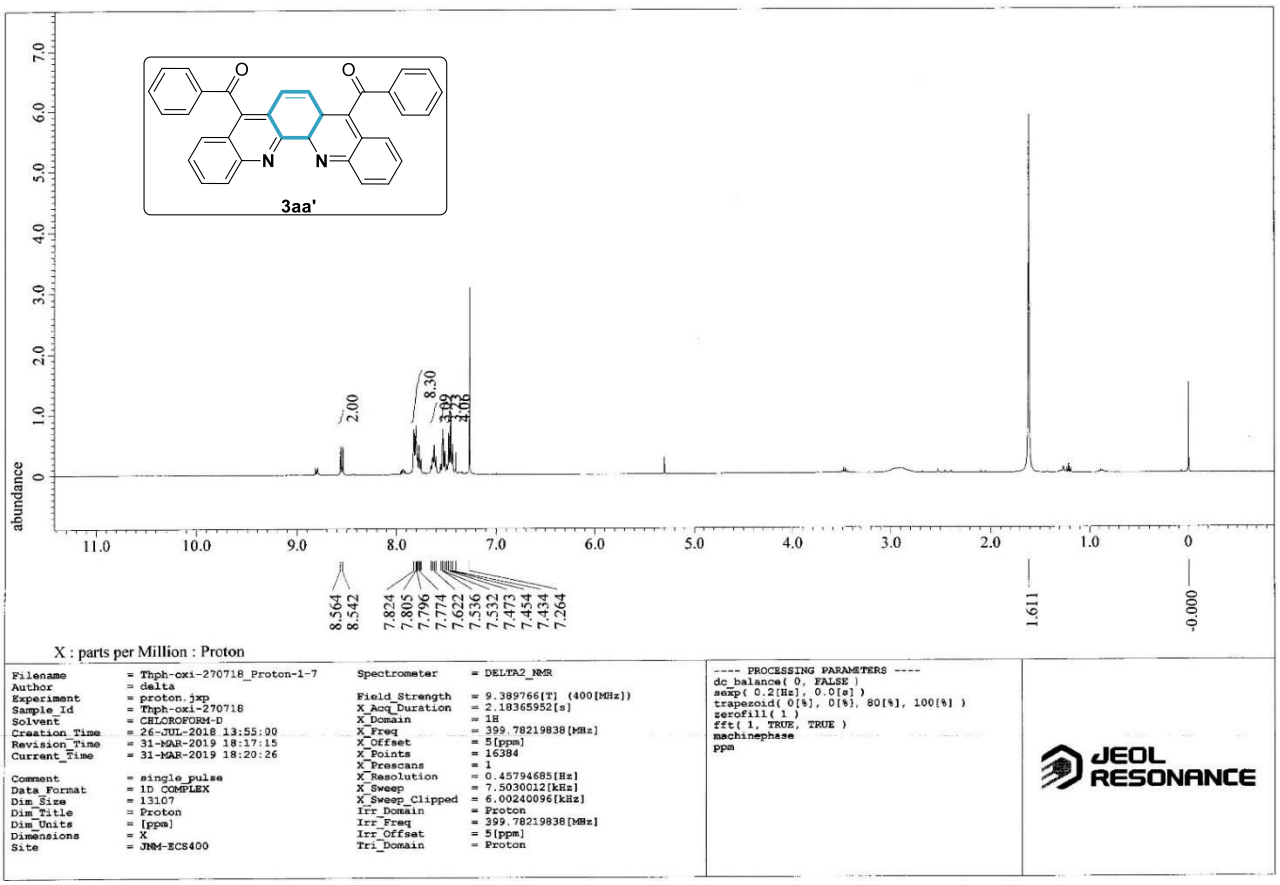
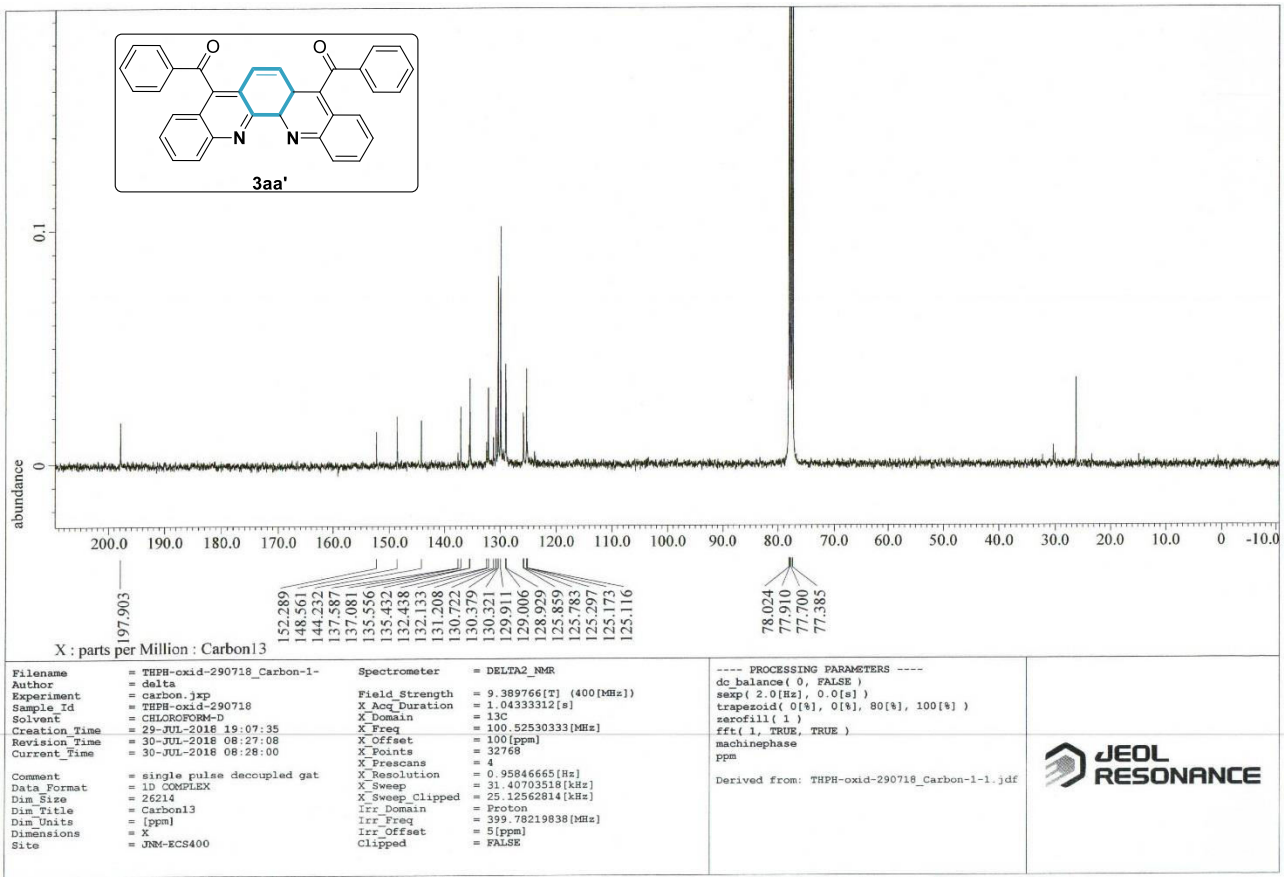
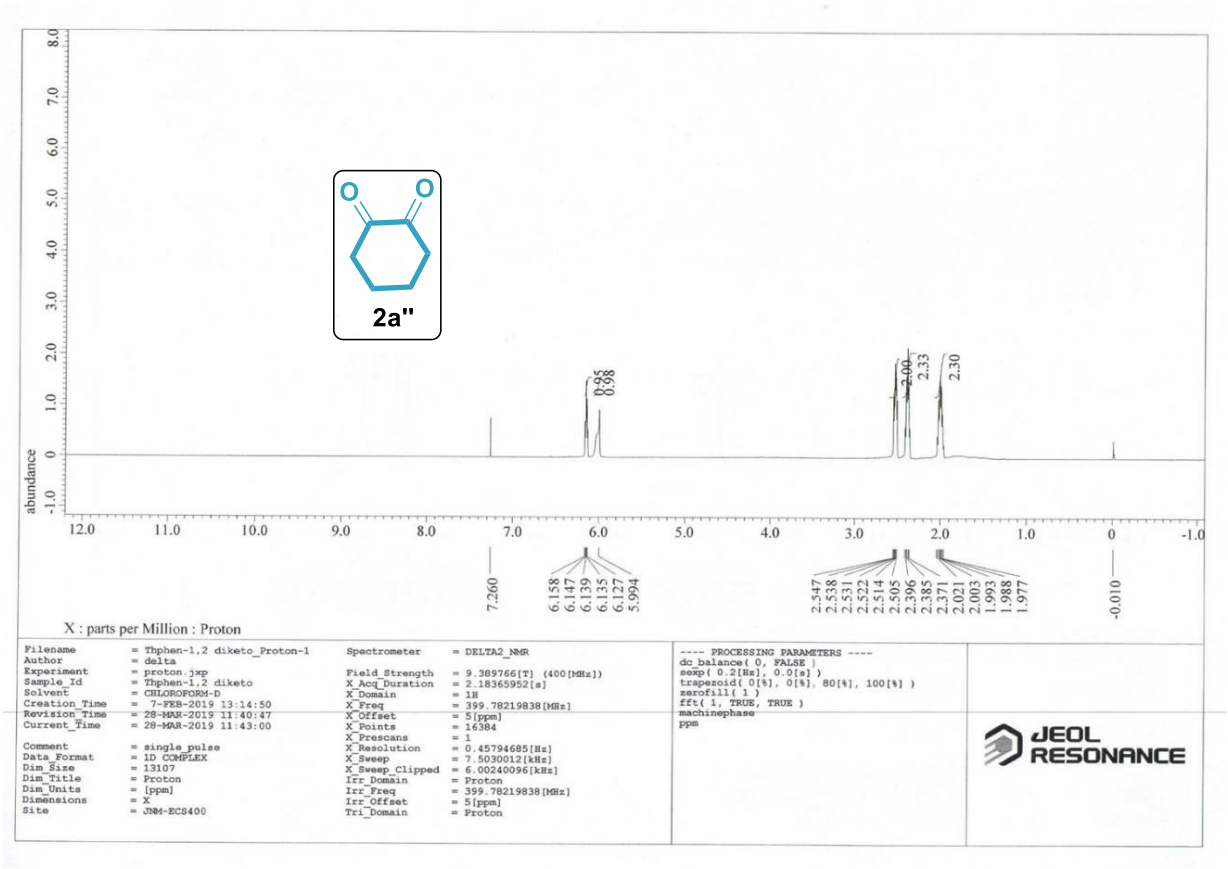
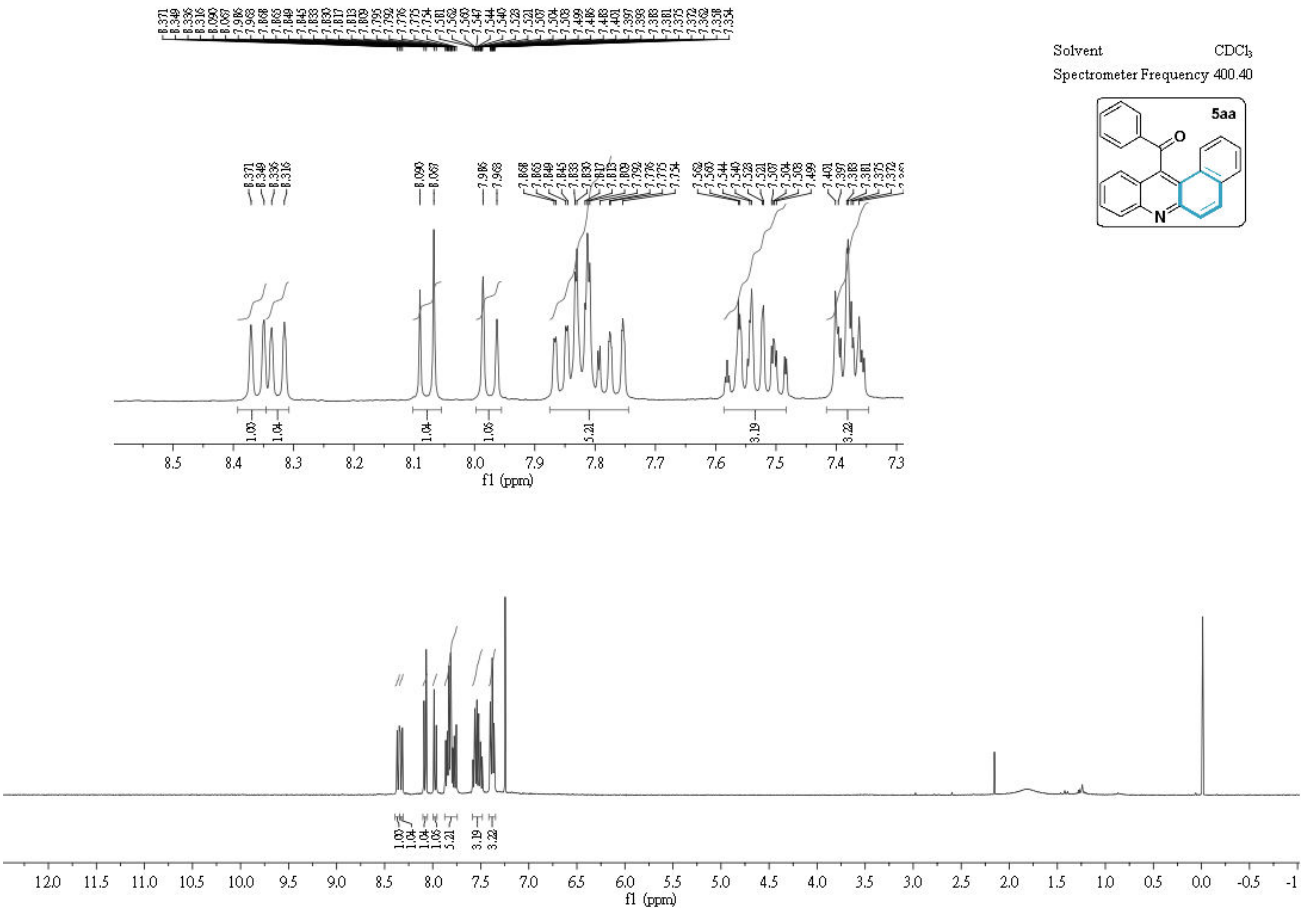
3ja
f1 (ppm)

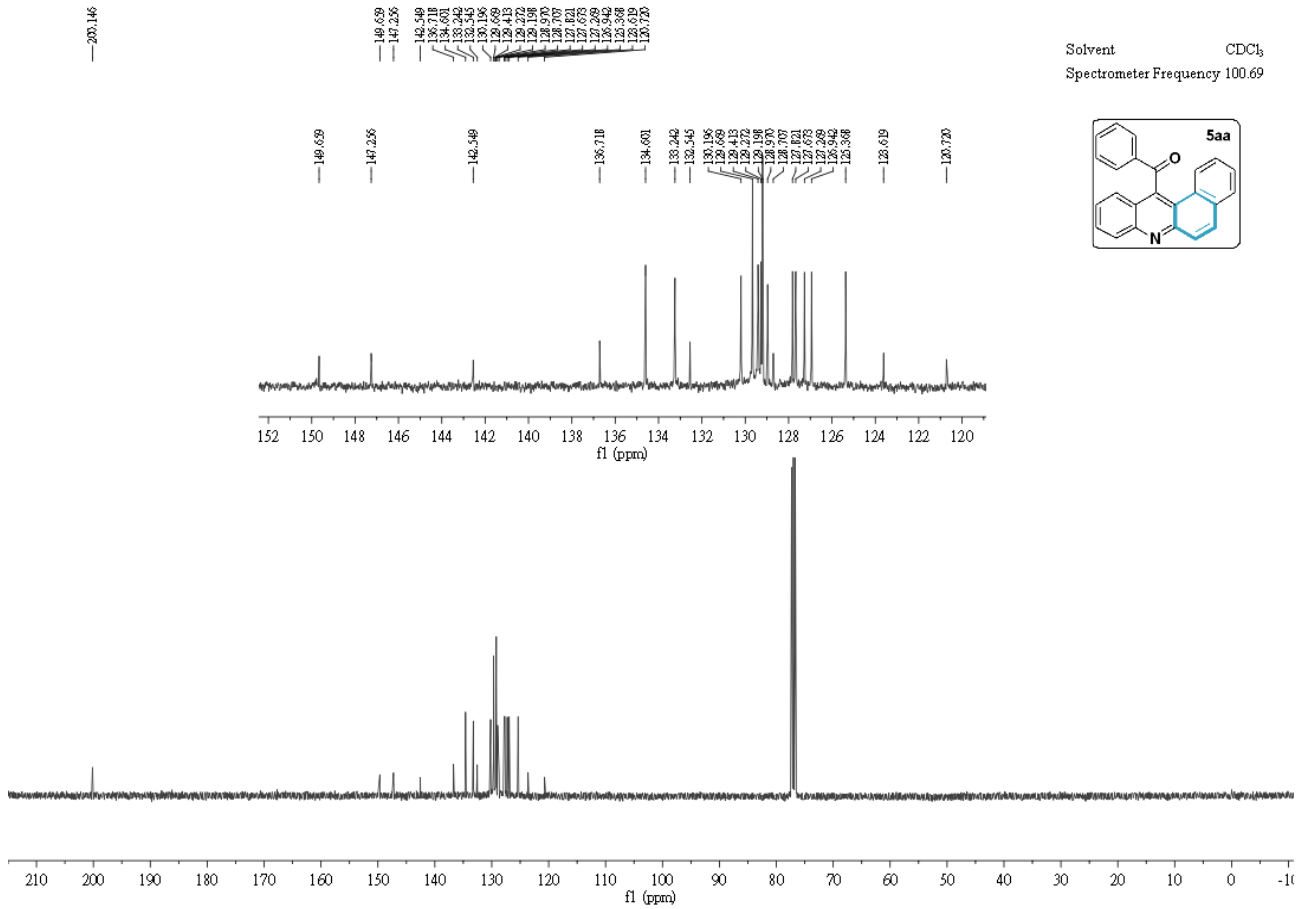

3aa'
abundance
2.00
8.30
1.03
1.03
1.06
8.564
8.542
7.824
7.805
7.796
7.774
7.622
7.536
7.532
7.473
7.454
7.434
7.264
1.611
-0.000
X : parts per Million : Proton
Filename = Thph-oxi-270718_Proton-1-7
Author = delta
Experiment = proton.jxp
Sample_Id = Thph-oxi-270718
Solvent = CHLOROFORM-D
Creation_Time = 26-JUL-2018 13:55:00
Revision_Time = 31-MAR-2019 18:17:15
Current_Time = 31-MAR-2019 18:20:26
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECS400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
JEOL RESONANCE


3aa'
abundance
197.903
152.289
148.561
144.232
137.587
137.081
135.556
135.432
132.438
132.133
131.208
130.722
130.379
130.321
129.911
129.006
128.929
125.859
125.783
125.297
125.173
125.116
78.024
77.910
77.700
77.385
X : parts per Million : Carbon13
Filename = THPH-oxid-290718_Carbon-1-
Author = delta
Experiment = carbon.jxp
Sample_Id = THPH-oxid-290718
Solvent = CHLOROFORM-D
Creation_Time = 29-JUL-2018 19:07:35
Revision_Time = 30-JUL-2018 08:27:08
Current_Time = 30-JUL-2018 08:28:00
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECS400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.04333312[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
Derived from: THPH-oxid-290718_Carbon-1-1.jdf
JEOL RESONANCE

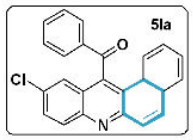

2a"
abundance
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
0.95
0.98
2.00
2.33
2.30
7.260
6.158
6.147
6.139
6.135
6.127
5.994
2.547
2.538
2.531
2.522
2.514
2.505
2.396
2.385
2.371
2.021
2.003
1.993
1.988
1.977
-0.010
X : parts per Million : Proton
Filename = Thphen-1,2 diketo_Proton-1
Author = delta
Experiment = proton.jxp
Sample_Id = Thphen-1,2 diketo
Solvent = CHLOROFORM-D
Creation_Time = 7-FEB-2019 13:14:50
Revision_Time = 28-MAR-2019 11:40:47
Current_Time = 28-MAR-2019 11:43:00
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECS400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
JEOL RESONANCE

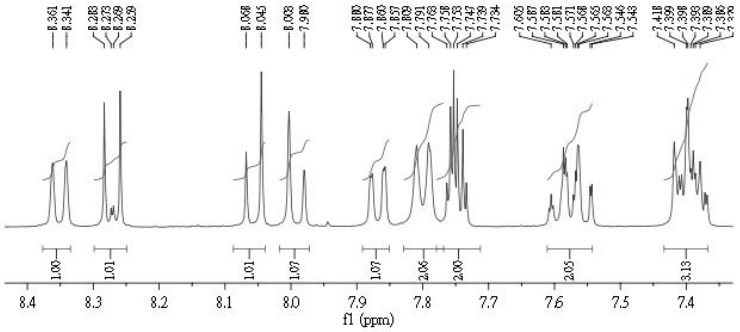

Solvent CDCl3
Spectrometer Frequency 400.40
5aa
f1 (ppm)

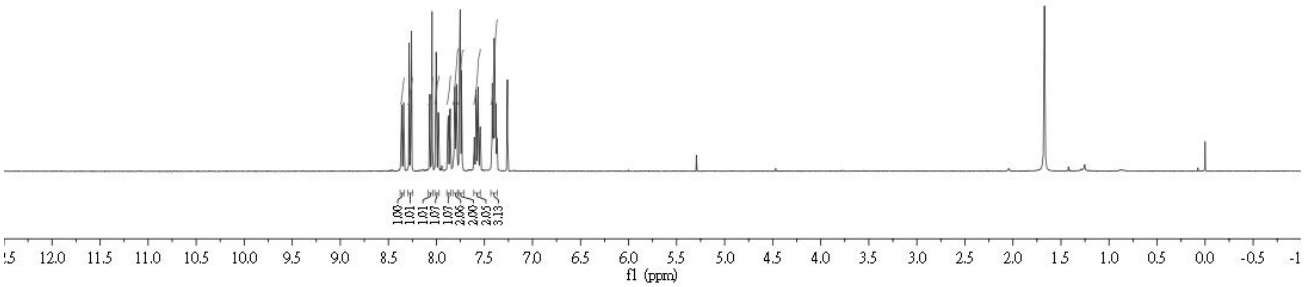
f1 (ppm)

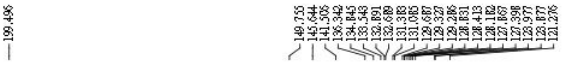

Solvent CDCl3
Spectrometer Frequency 100.69
5aa
f1 (ppm)

Solvent CDCl3
Spectrometer Frequency 400.40

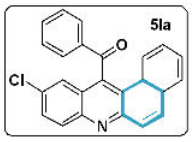


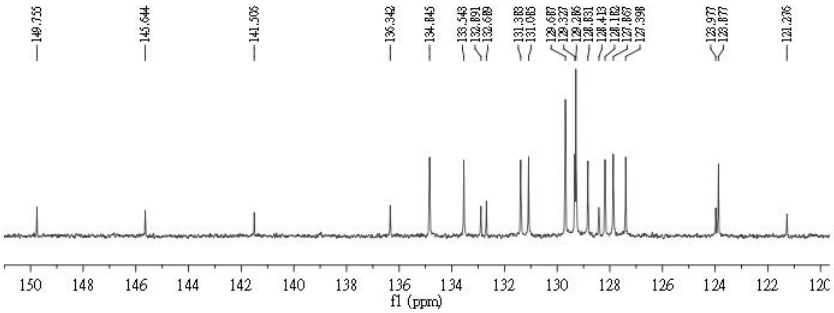


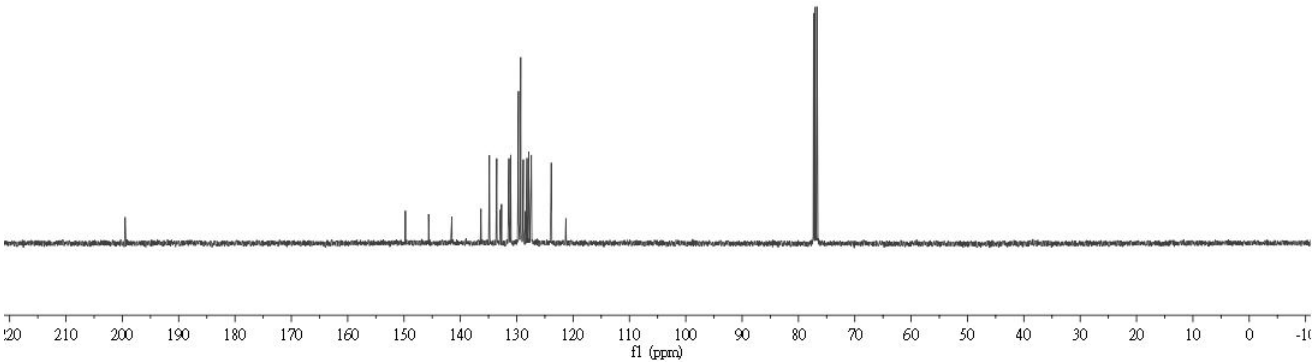


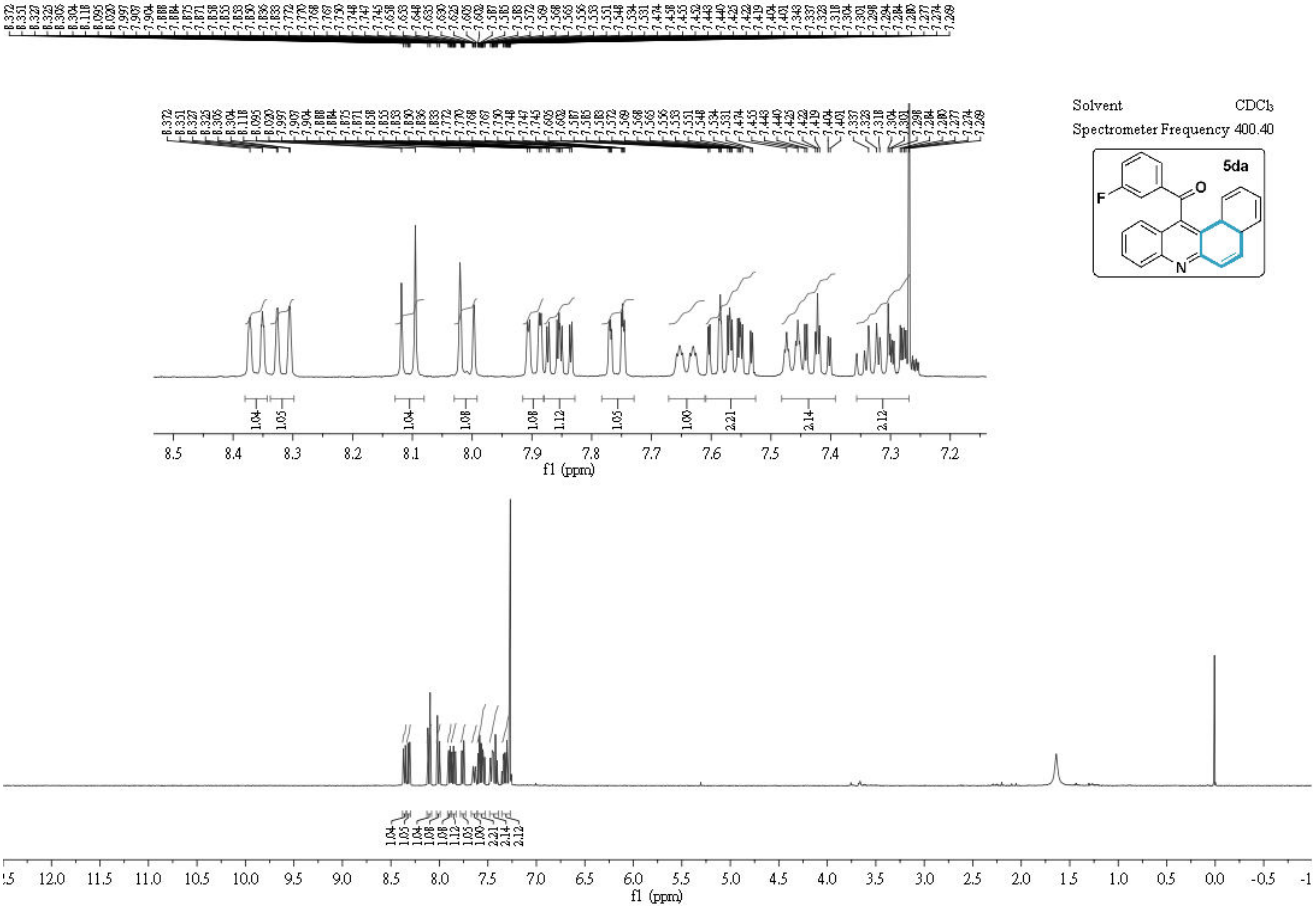


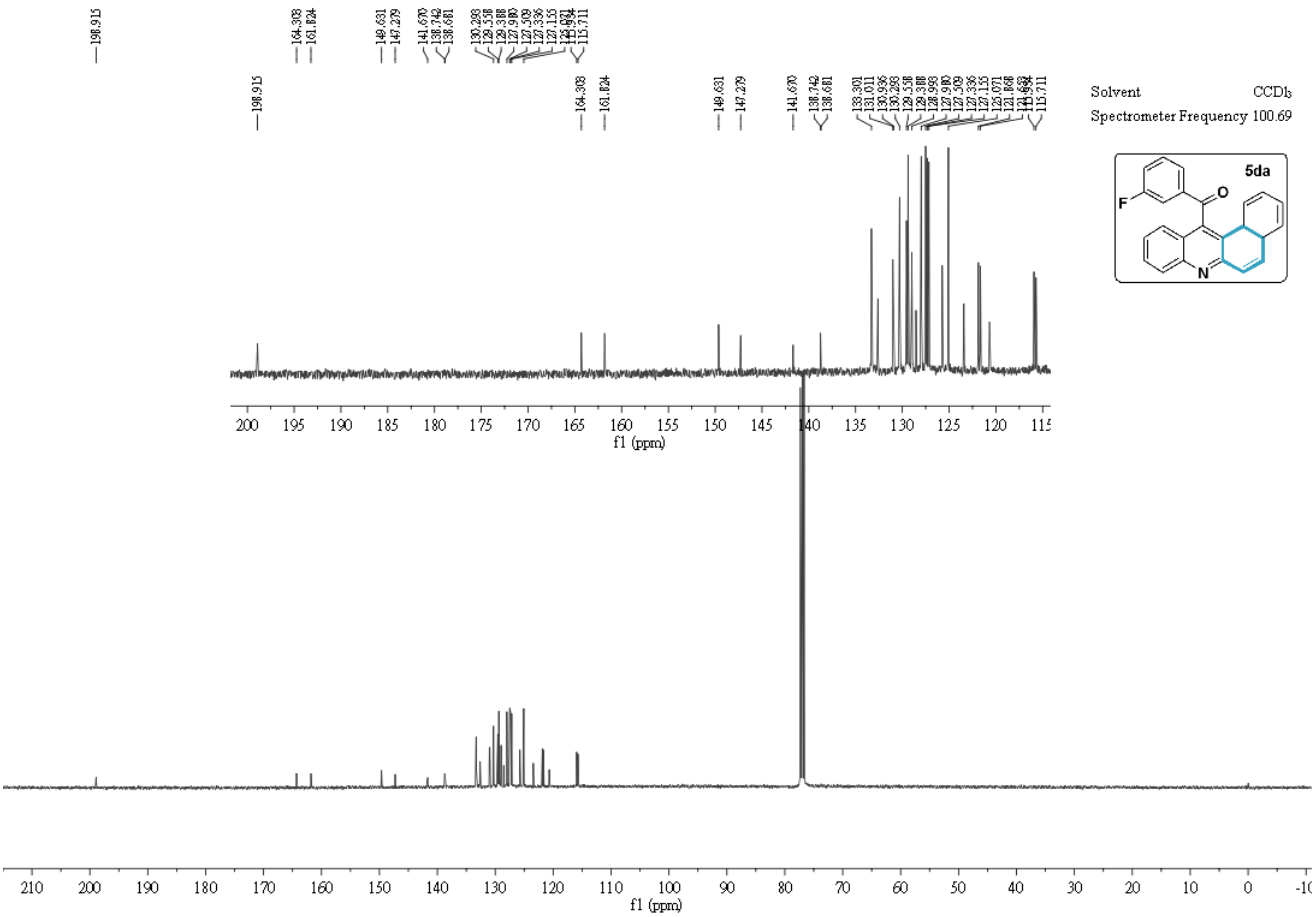
Solvent CDCl3
Spectrometer Frequency 100.69

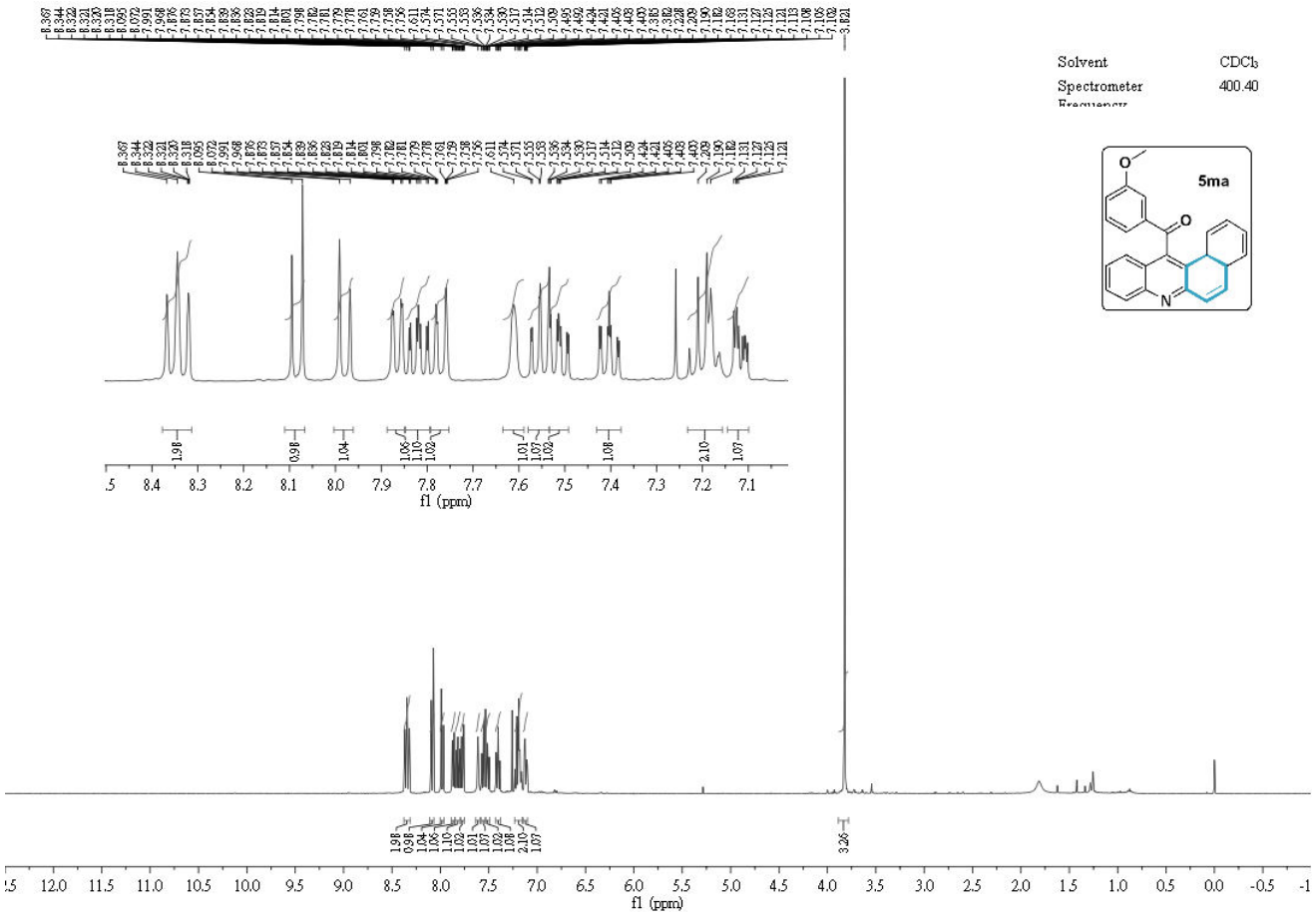


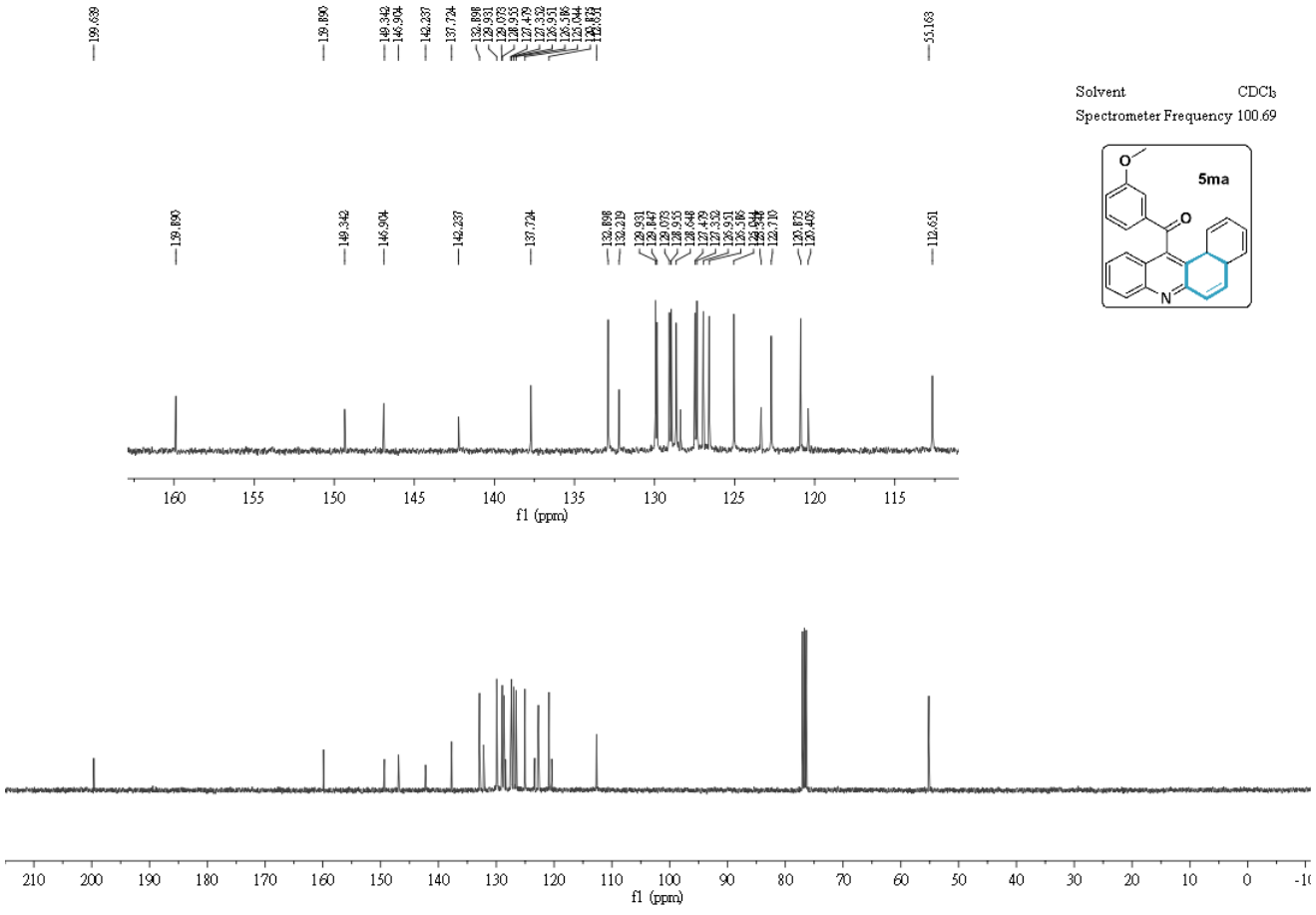


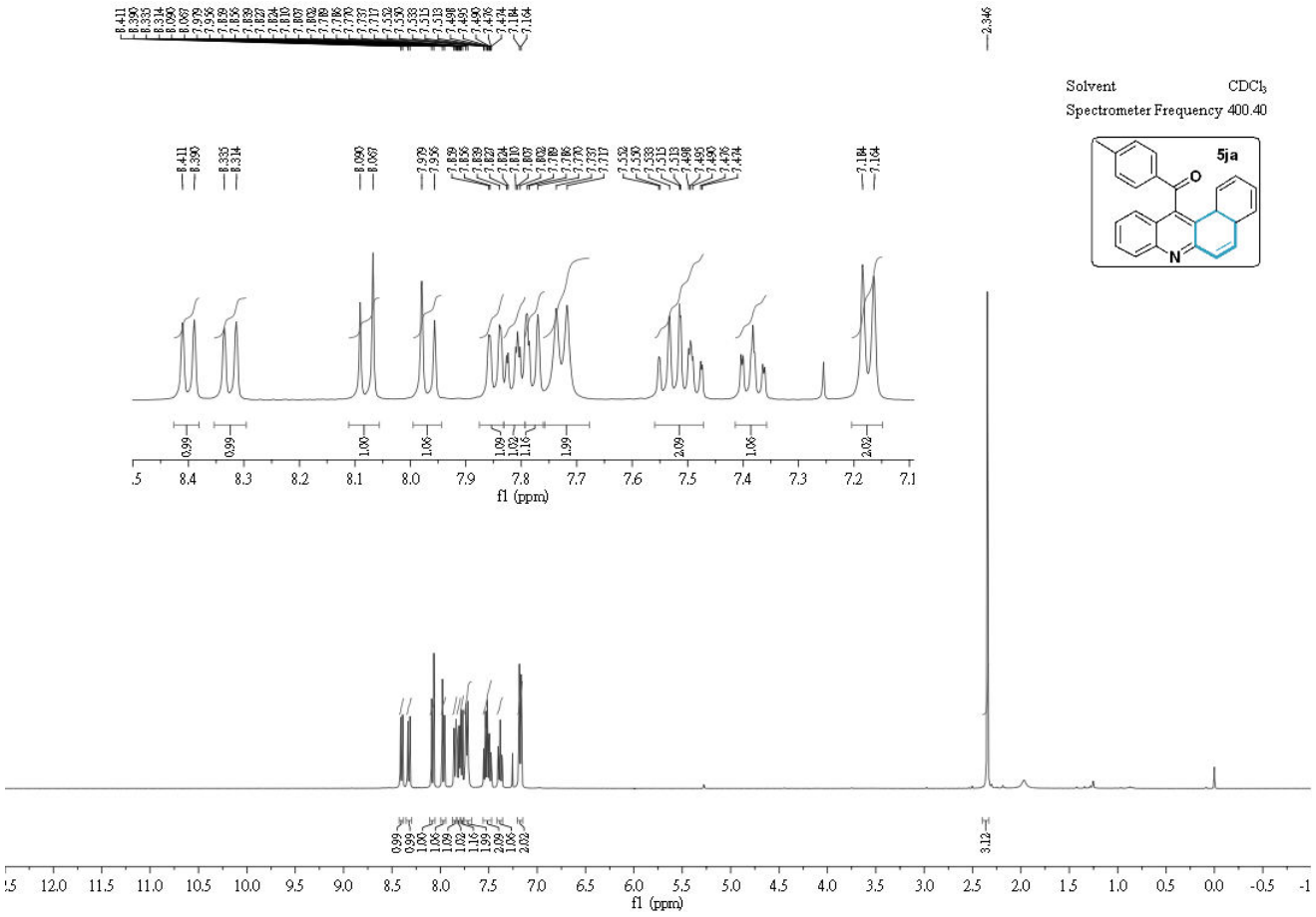


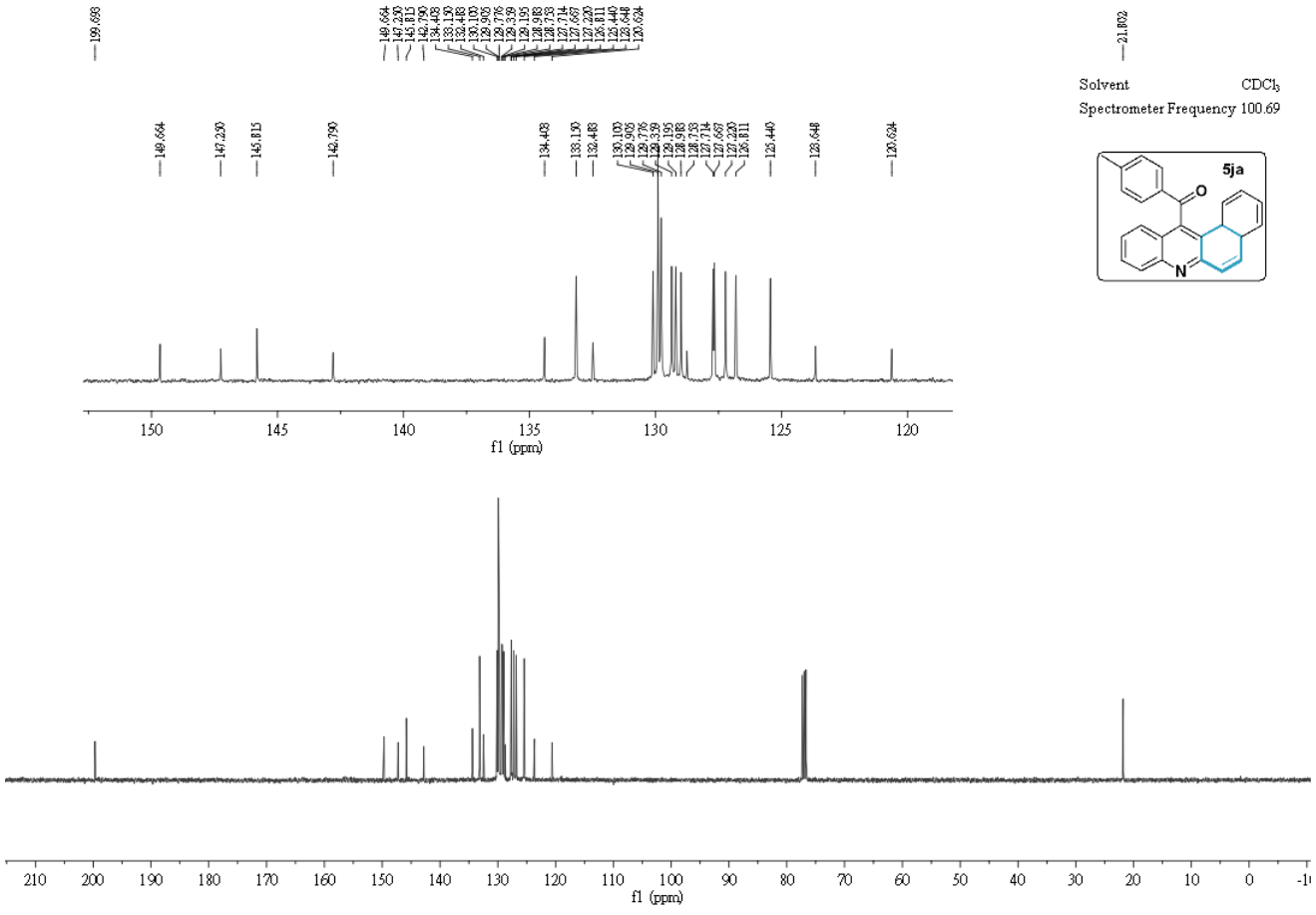

Solvent CDCl3
Spectrometer Frequency 400.40
5da
f1 (ppm)

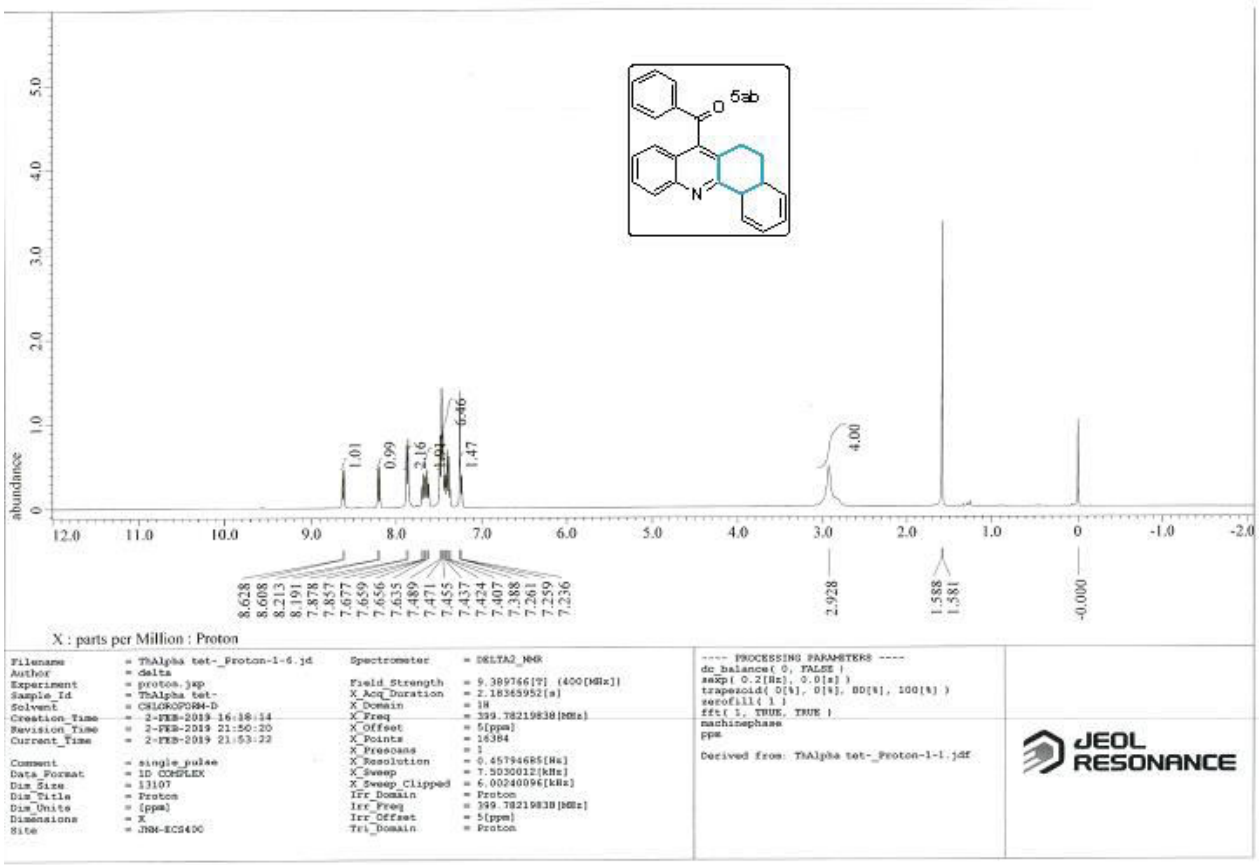

Solvent CCDl3
Spectrometer Frequency 100.69
5da
f1 (ppm)

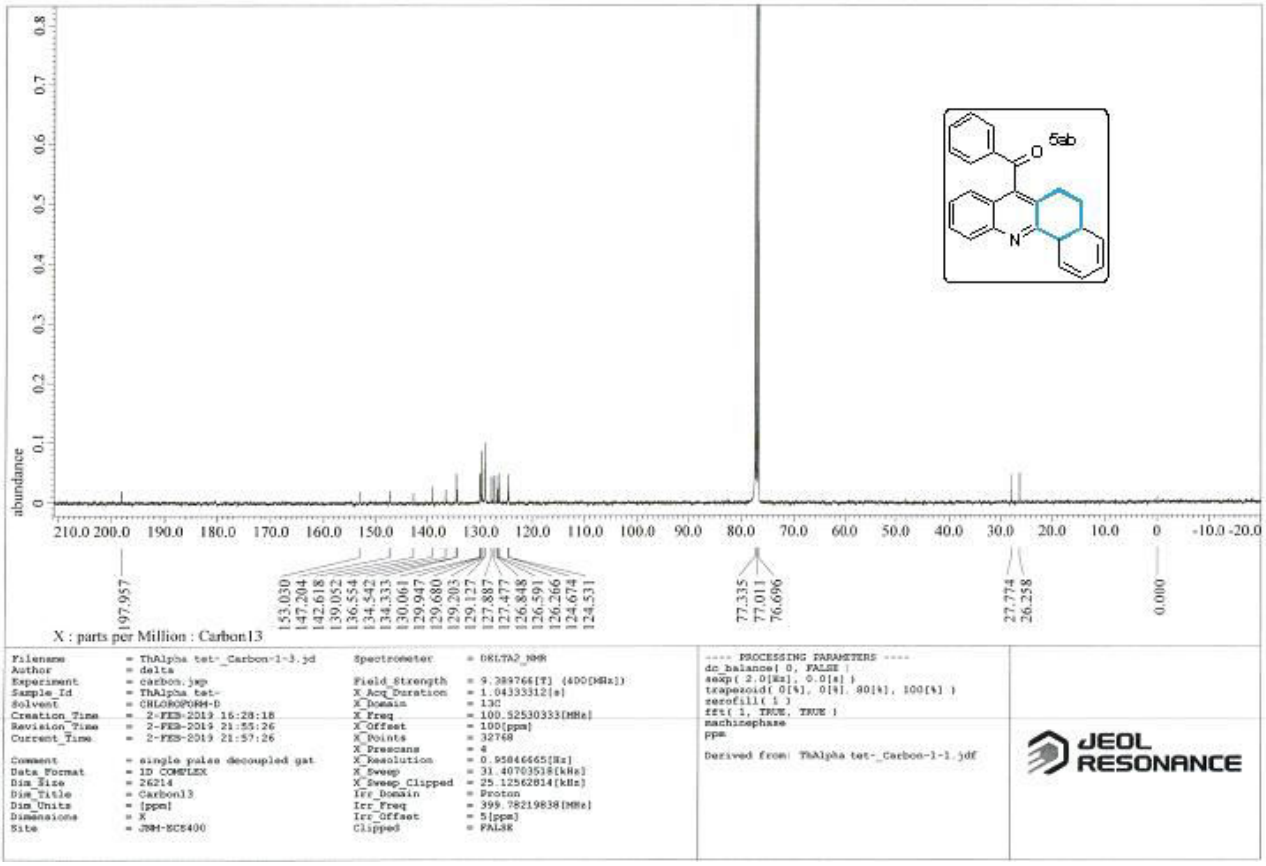

Solvent CDCl3
Spectrometer 400.40
5ma
f1 (ppm)

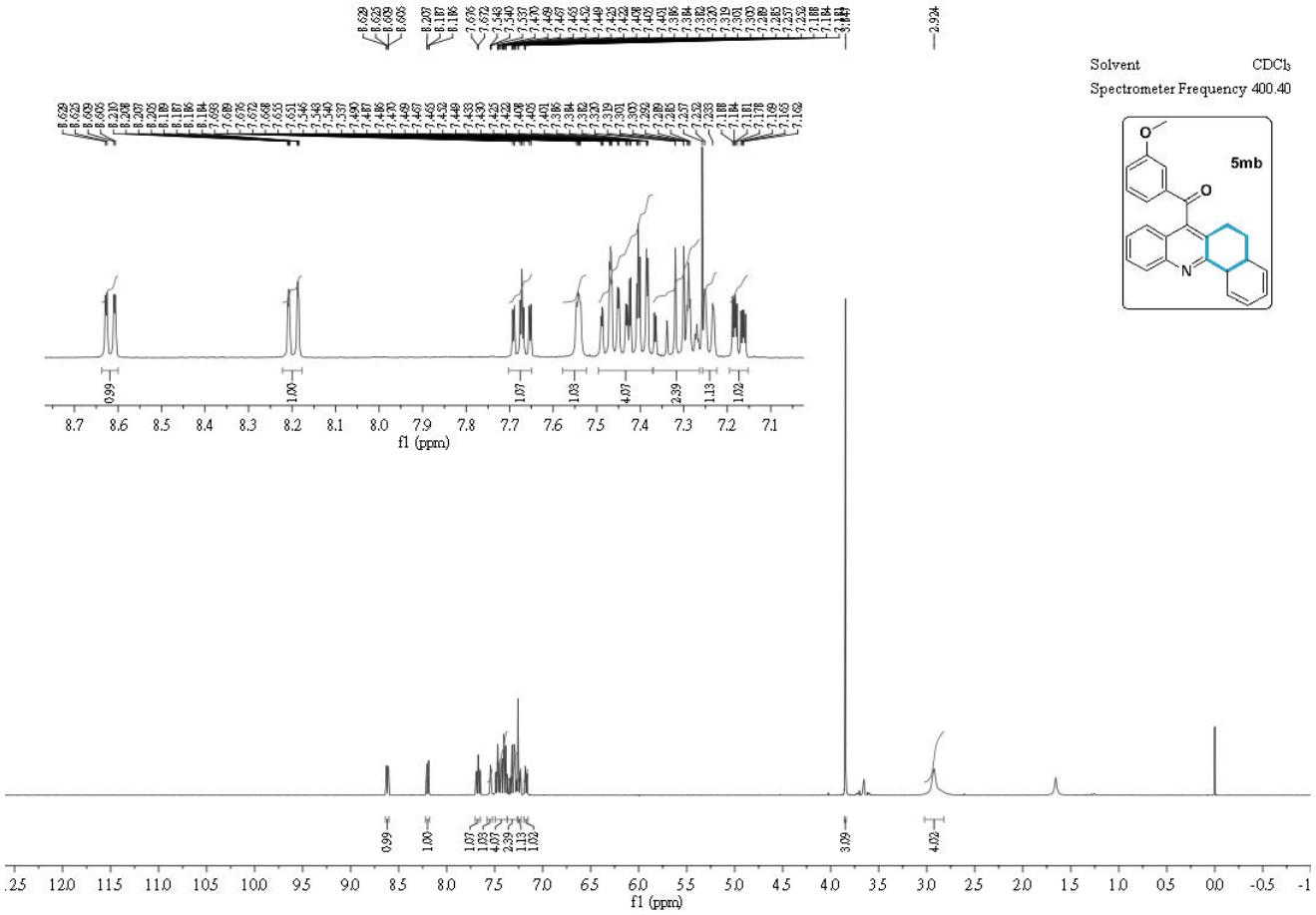

Solvent CDCl3
Spectrometer Frequency 100.69
5ma
f1 (ppm)

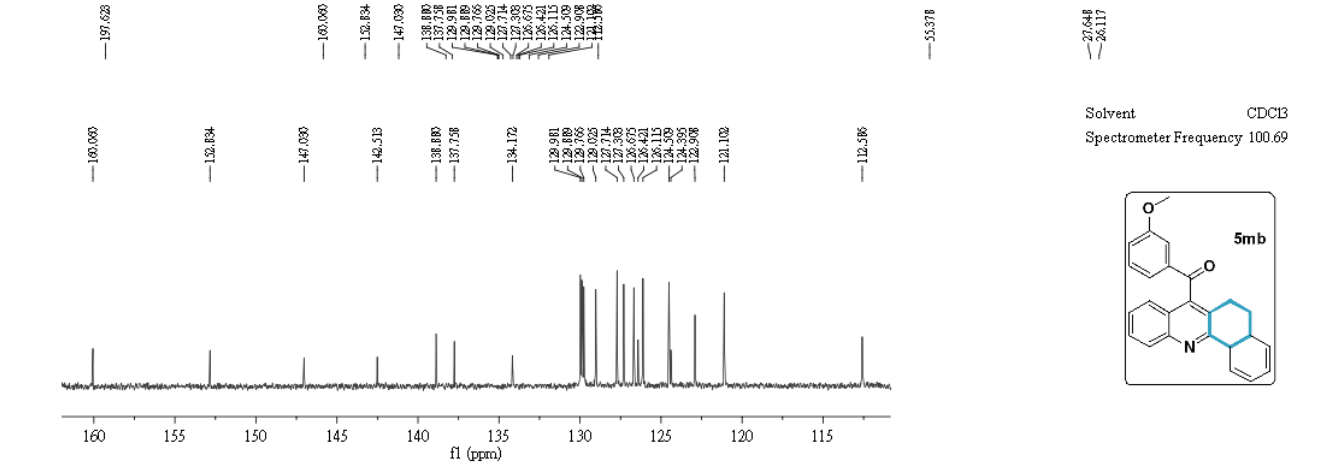

Solvent CDCl3
Spectrometer Frequency 400.40
5ja
f1 (ppm)

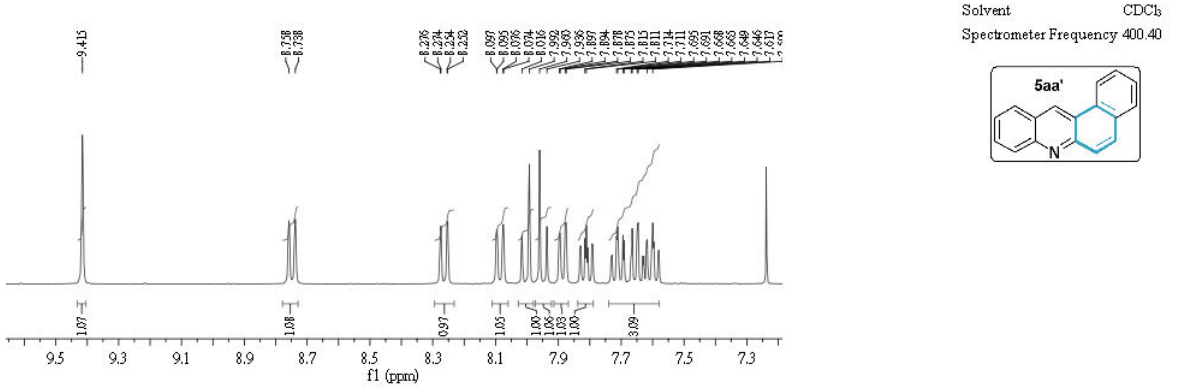

Solvent CDCl3
Spectrometer Frequency 100.69
5ja
f1 (ppm)

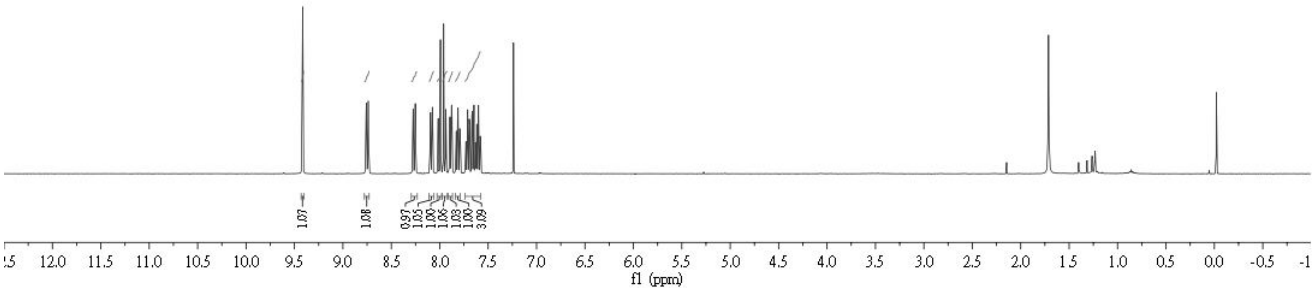

5ab
O
N
abundance
1.01
0.99
2.16
1.01
6.46
1.47
4.00
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0 -1.0 -2.0
8.628
8.608
8.213
8.191
7.878
7.857
7.677
7.659
7.656
7.635
7.489
7.471
7.455
7.437
7.424
7.407
7.388
7.261
7.259
7.236
2.928
1.588
1.381
-0.000
X : parts per Million : Proton
Filename = ThAlpha tet-_Proton-1-6.jd
Author = delta
Experiment = proton.jxp
Sample_Id = ThAlpha tet-
Solvent = CHLOROFORM-D
Creation_Time = 2-FEB-2019 16:18:14
Revision_Time = 2-FEB-2019 21:50:20
Current_Time = 2-FEB-2019 21:53:22
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECS400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.50300012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
Derived from: ThAlpha tet-_Proton-1-1.jdf
JEOL RESONANCE

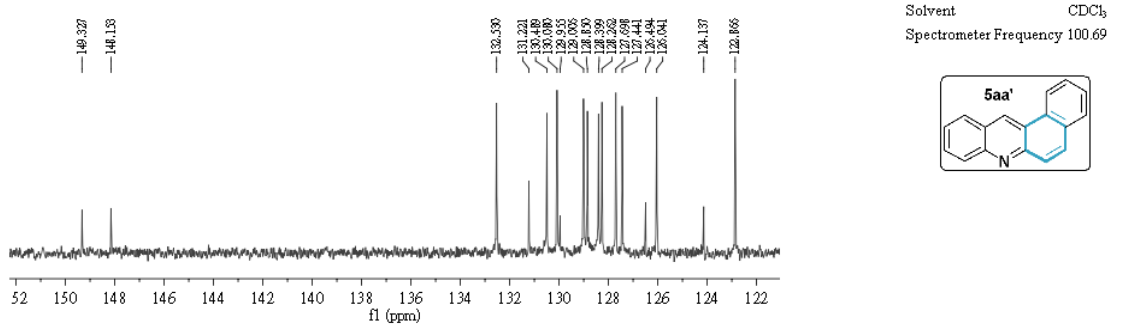

5ab
O
N
abundance
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
197.957
153.030
147.204
142.618
139.052
136.554
134.542
134.333
130.061
129.947
129.680
129.203
129.127
127.887
127.477
126.848
126.591
126.266
124.674
124.531
77.335
77.011
76.696
27.774
26.258
0.000
X : parts per Million : Carbon13
Filename = ThAlpha tet-_Carbon-1-3.jd
Author = delta
Experiment = carbon.jxp
Sample_Id = ThAlpha tet-
Solvent = CHLOROFORM-D
Creation_Time = 2-FEB-2019 16:28:18
Revision_Time = 2-FEB-2019 21:55:26
Current_Time = 2-FEB-2019 21:57:26
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECS400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.04333312[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
Derived from: ThAlpha tet-_Carbon-1-1.jdf
JEOL RESONANCE

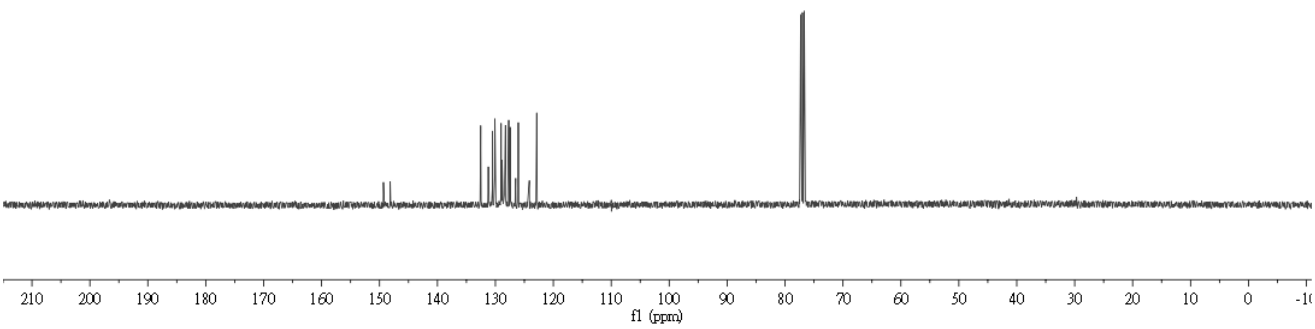

Solvent CDCl3
Spectrometer Frequency 400.40
5mb
f1 (ppm)

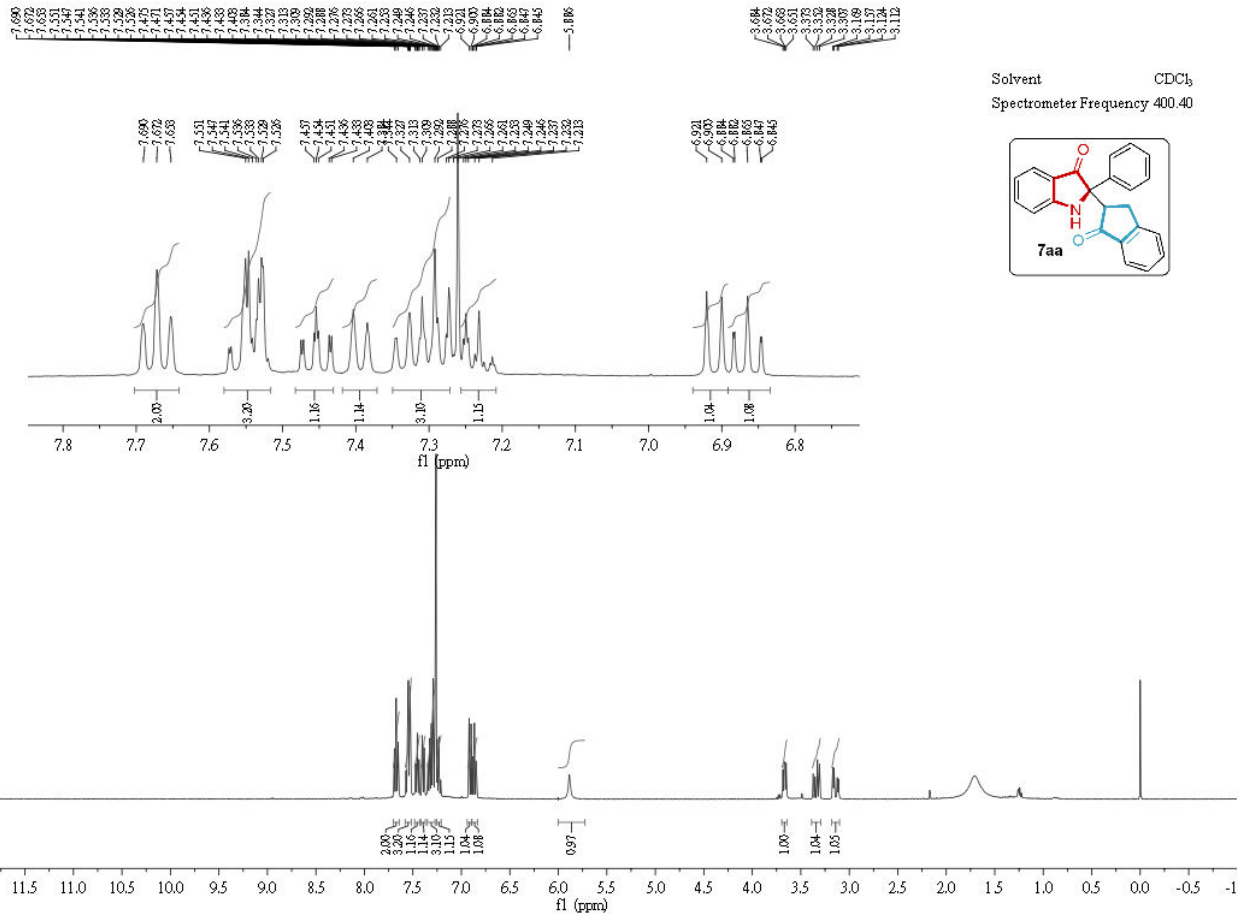

Solvent CDCl3
Spectrometer Frequency 100.69
5mb
f1 (ppm)

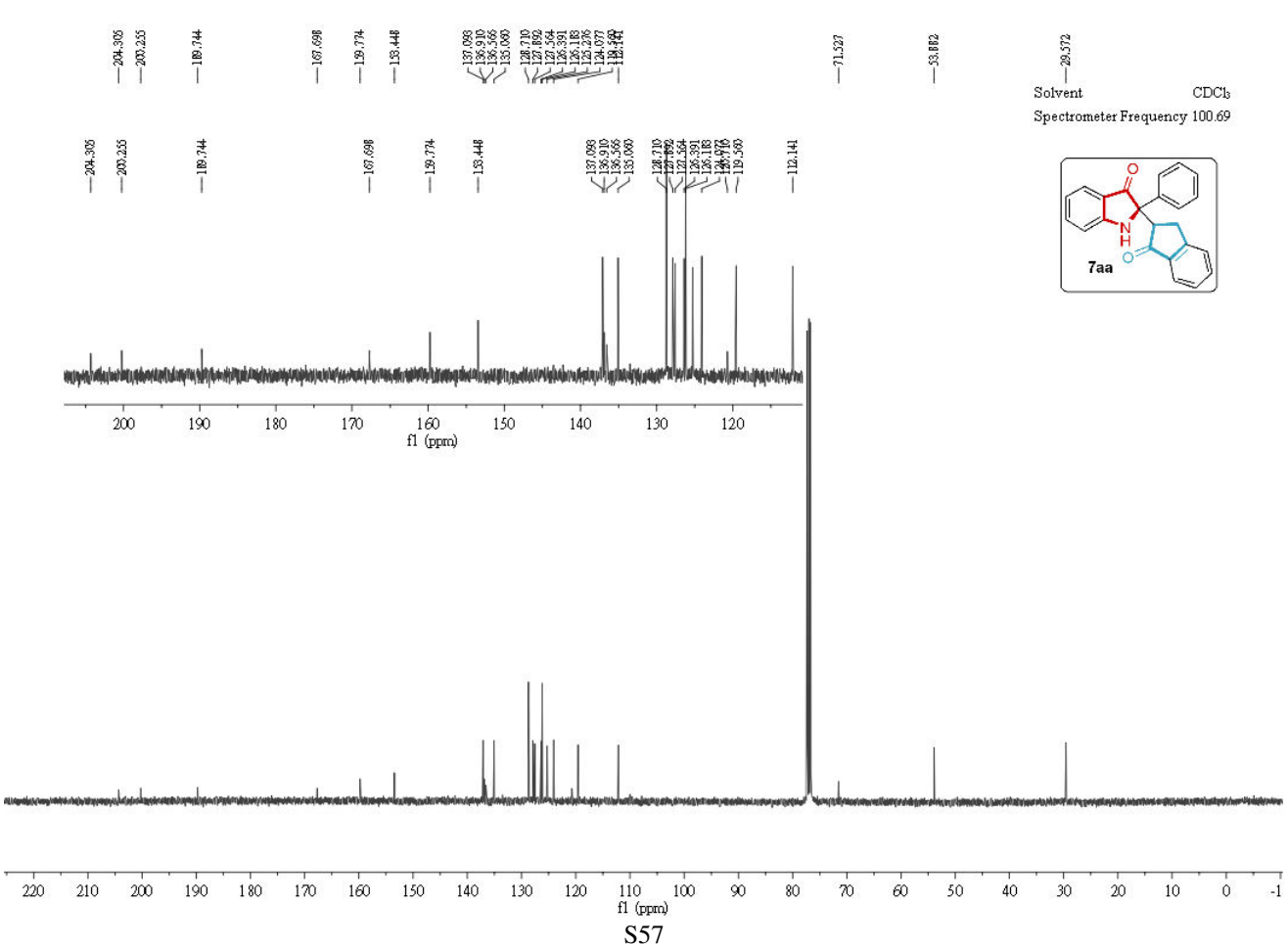

Solvent CDCl3
Spectrometer Frequency 400.40
5aa'
f1 (ppm)

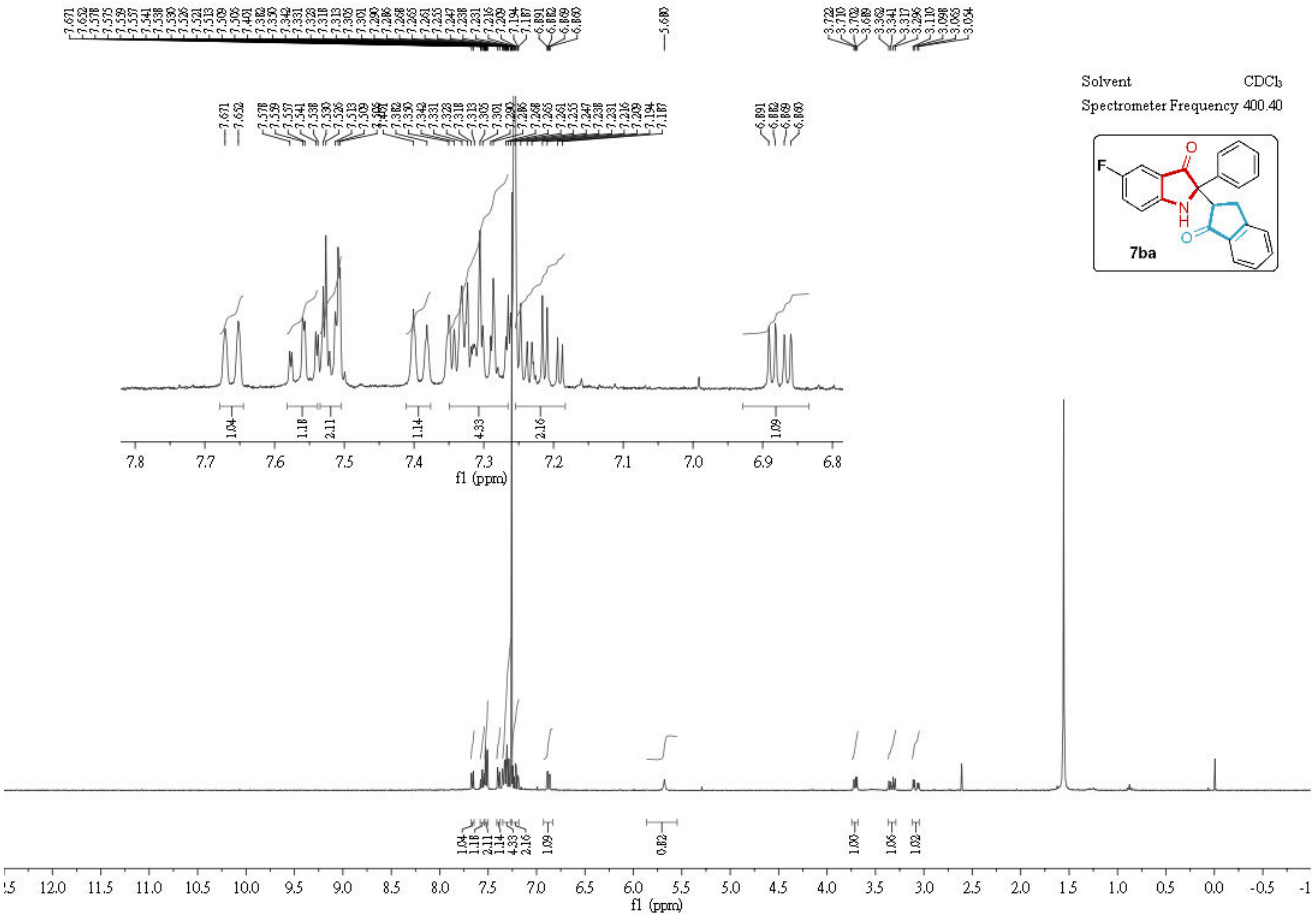

f1 (ppm)

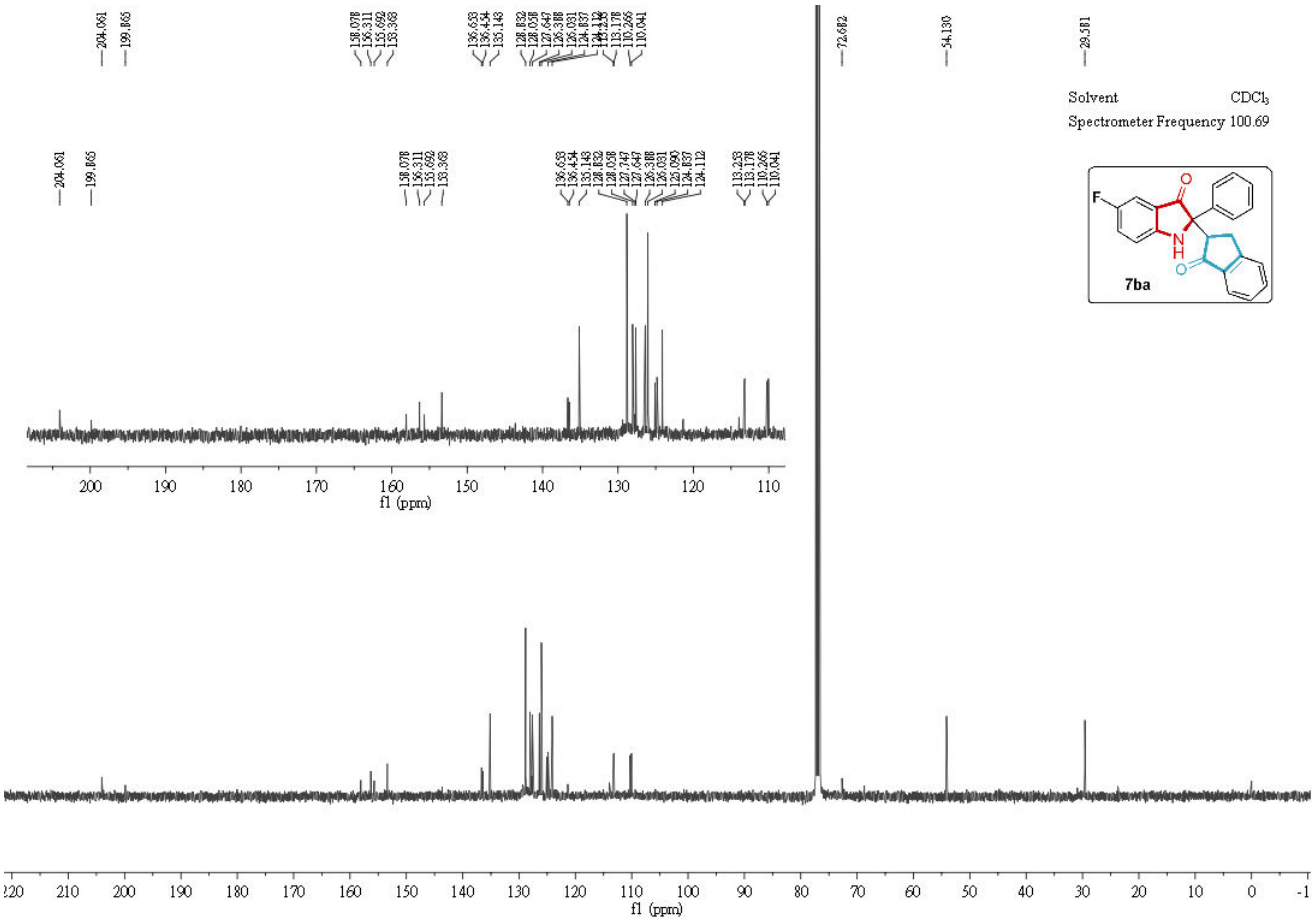

Solvent CDCl3
Spectrometer Frequency 100.69
5aa'
f1 (ppm)

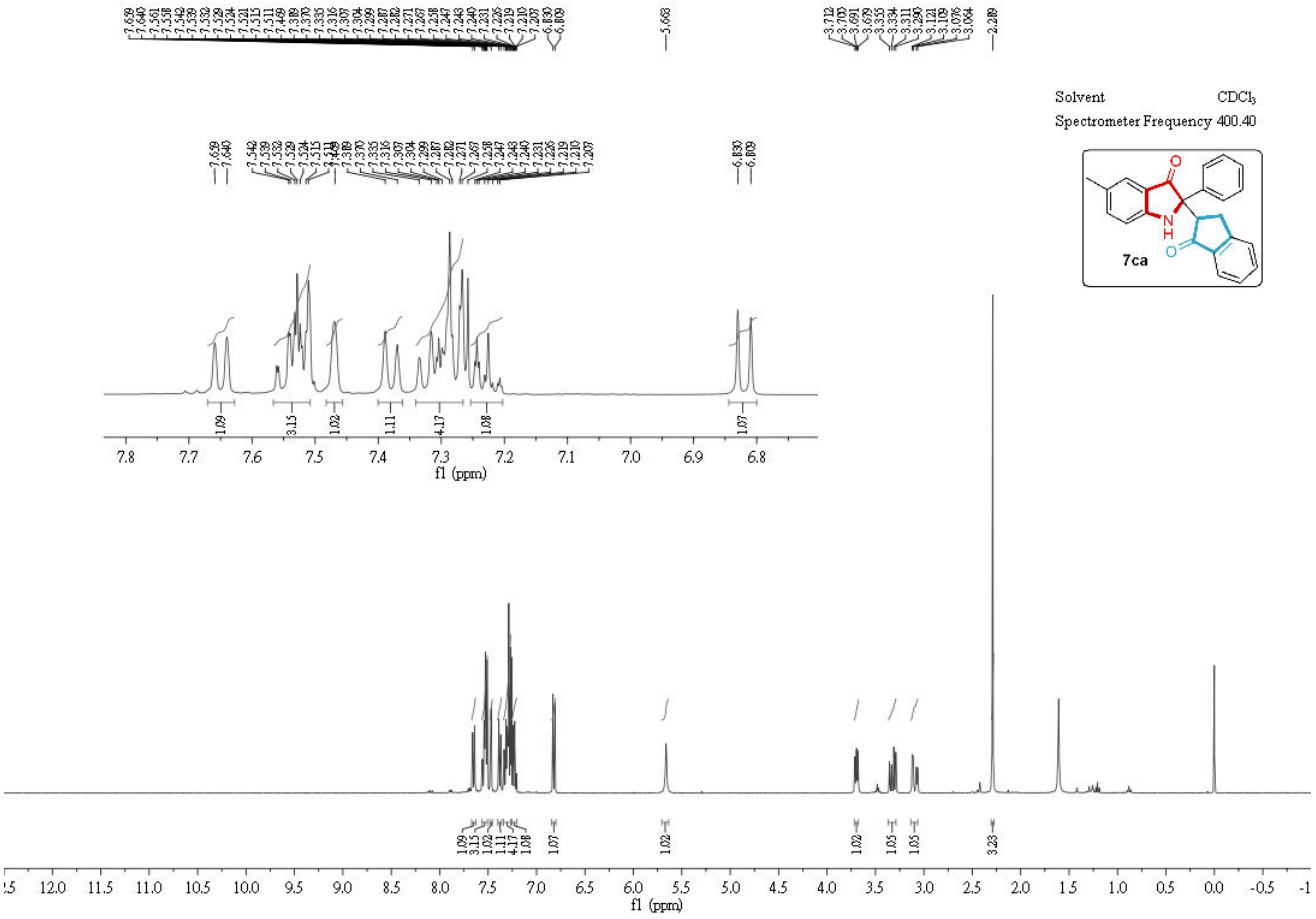

f1 (ppm)

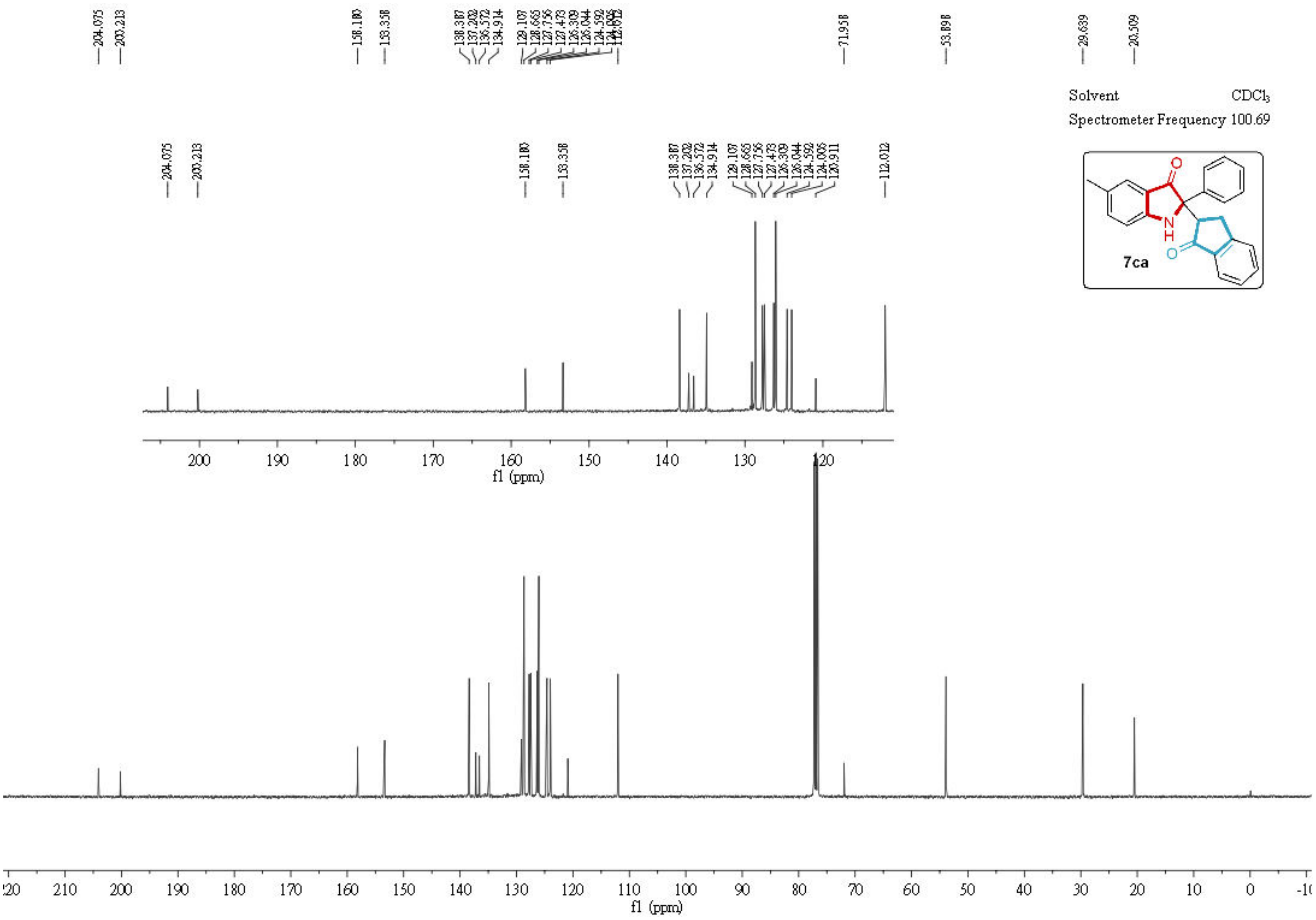

Solvent CDCl3
Spectrometer Frequency 400.40
7aa
f1 (ppm)

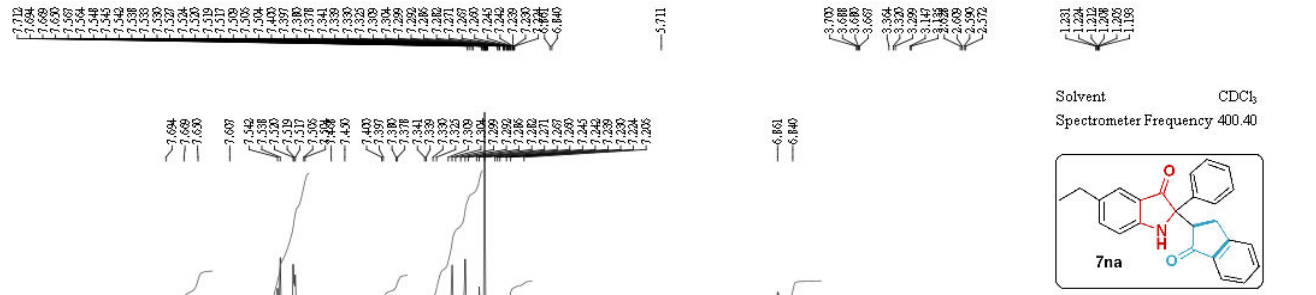

Solvent CDCl3
Spectrometer Frequency 100.69
7aa
f1 (ppm)

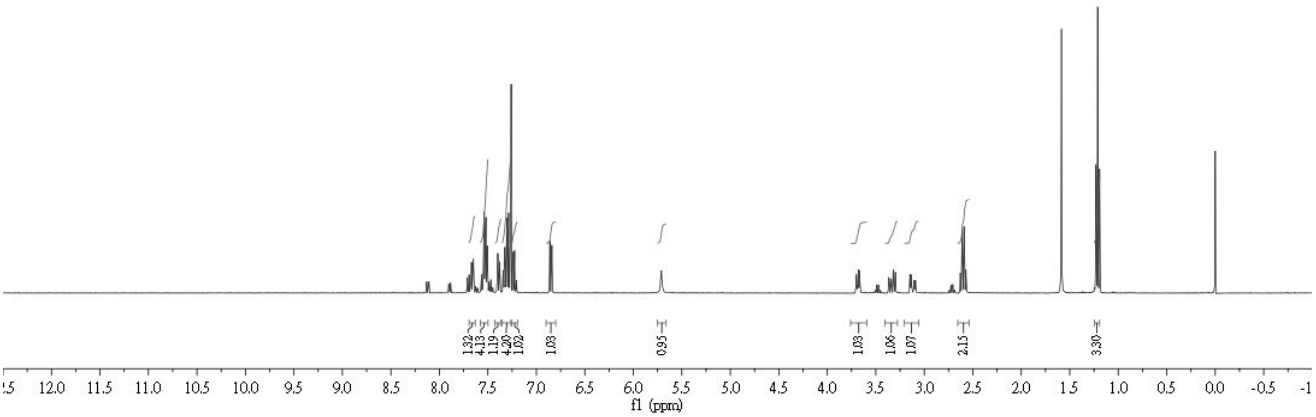

Solvent CDCl3
Spectrometer Frequency 400.40
7ba
f1 (ppm)

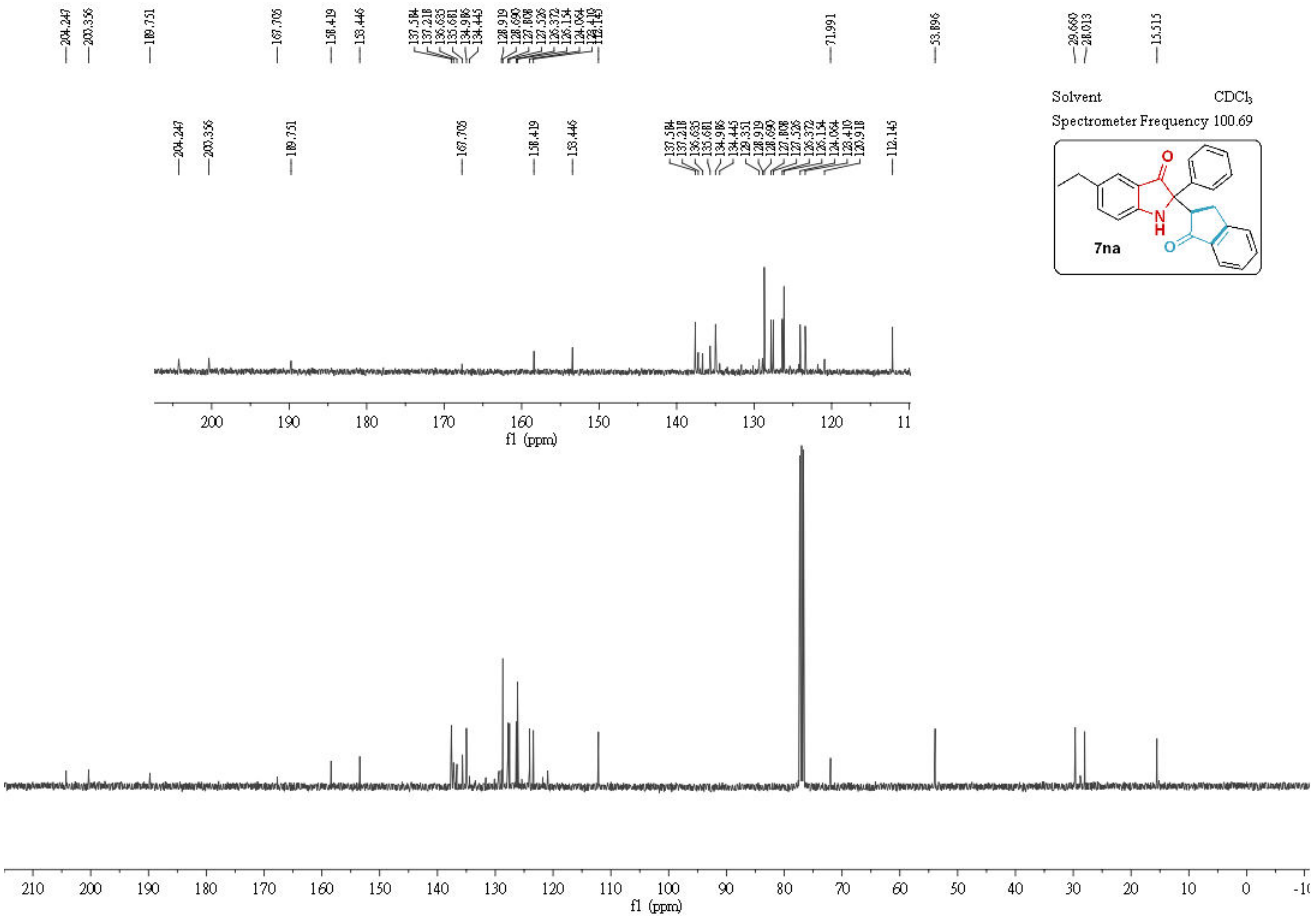

Solvent CDCl3
Spectrometer Frequency 100.69
7ba
f1 (ppm)

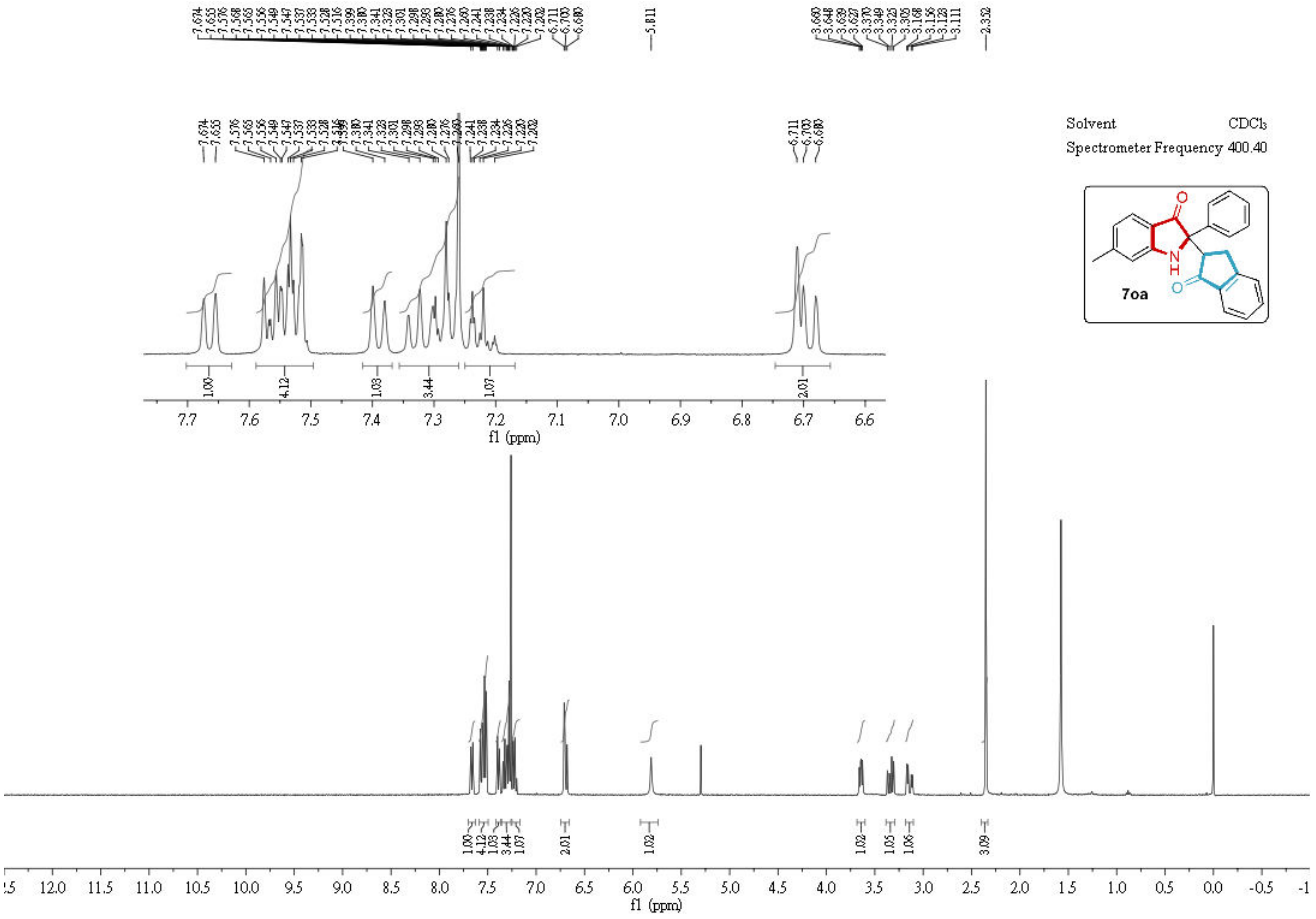

Solvent CDCl3
Spectrometer Frequency 400.40
7ca
f1 (ppm)

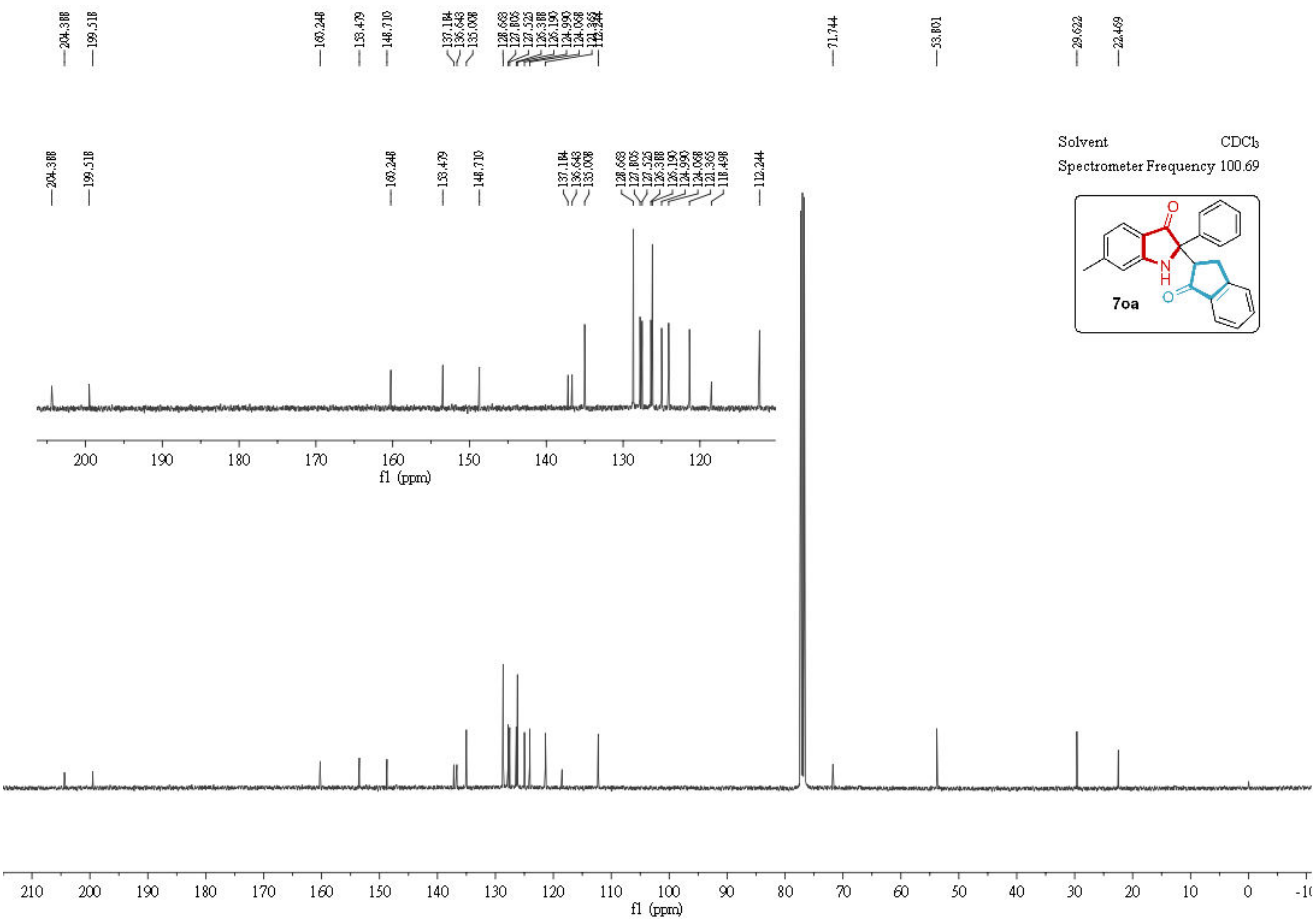

Solvent CDCl3
Spectrometer Frequency 100.69
7ca
f1 (ppm)

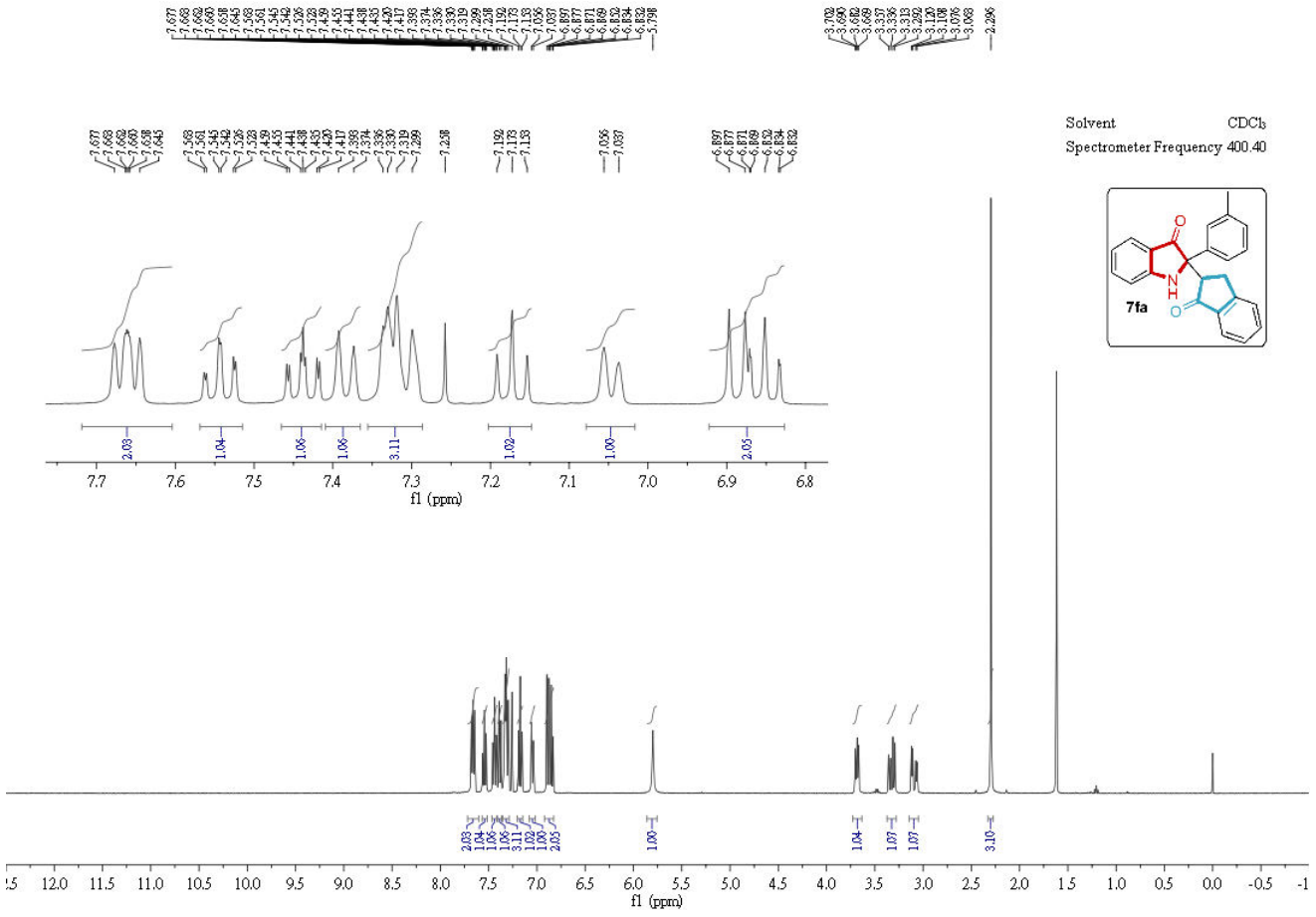

Solvent CDCl3
Spectrometer Frequency 400.40
7na
f1 (ppm)

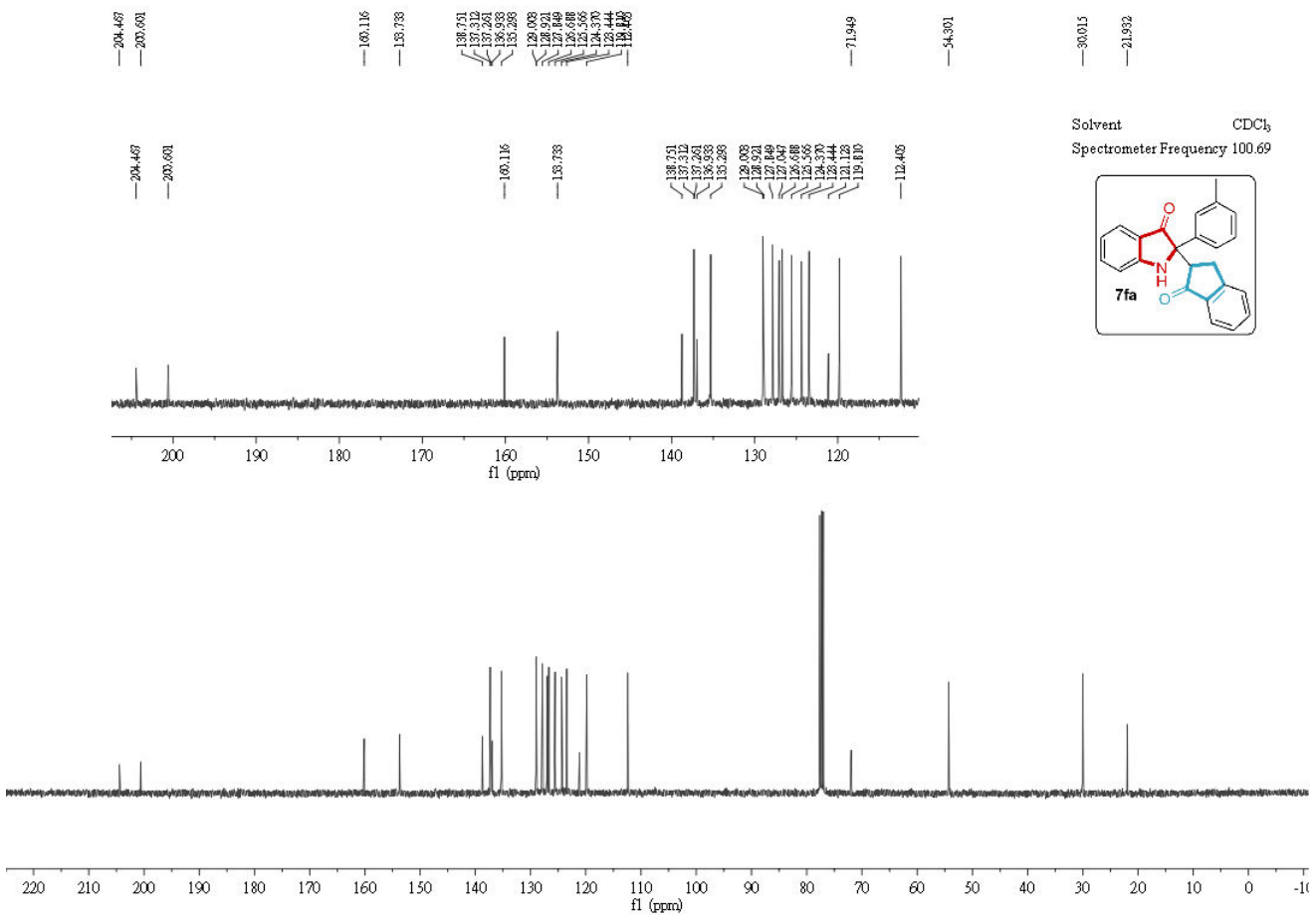

f1 (ppm)

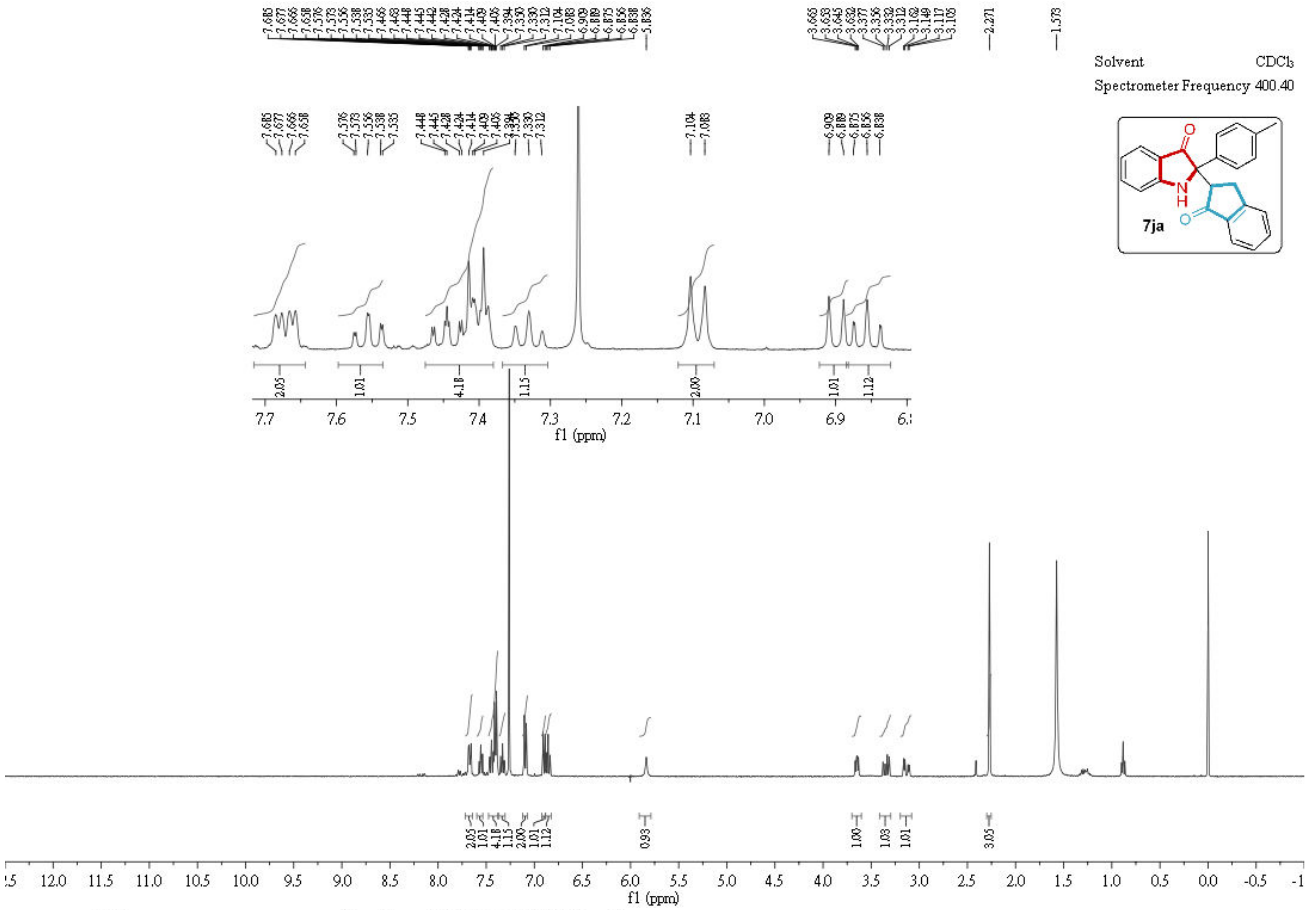

Solvent CDCl3
Spectrometer Frequency 100.69
7na
f1 (ppm)
f1 (ppm)

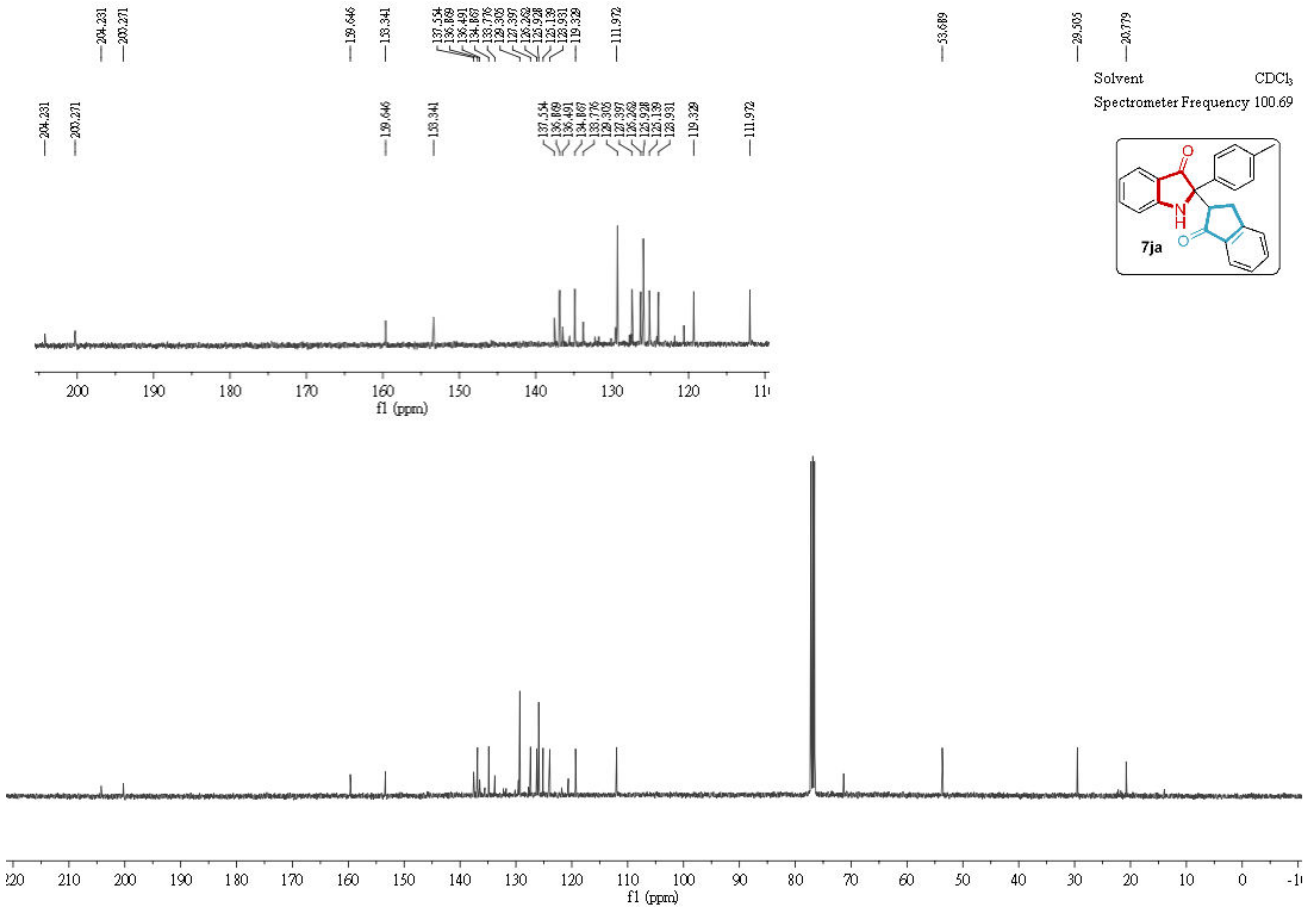

Solvent CDCl3
Spectrometer Frequency 400.40
7oa
f1 (ppm)

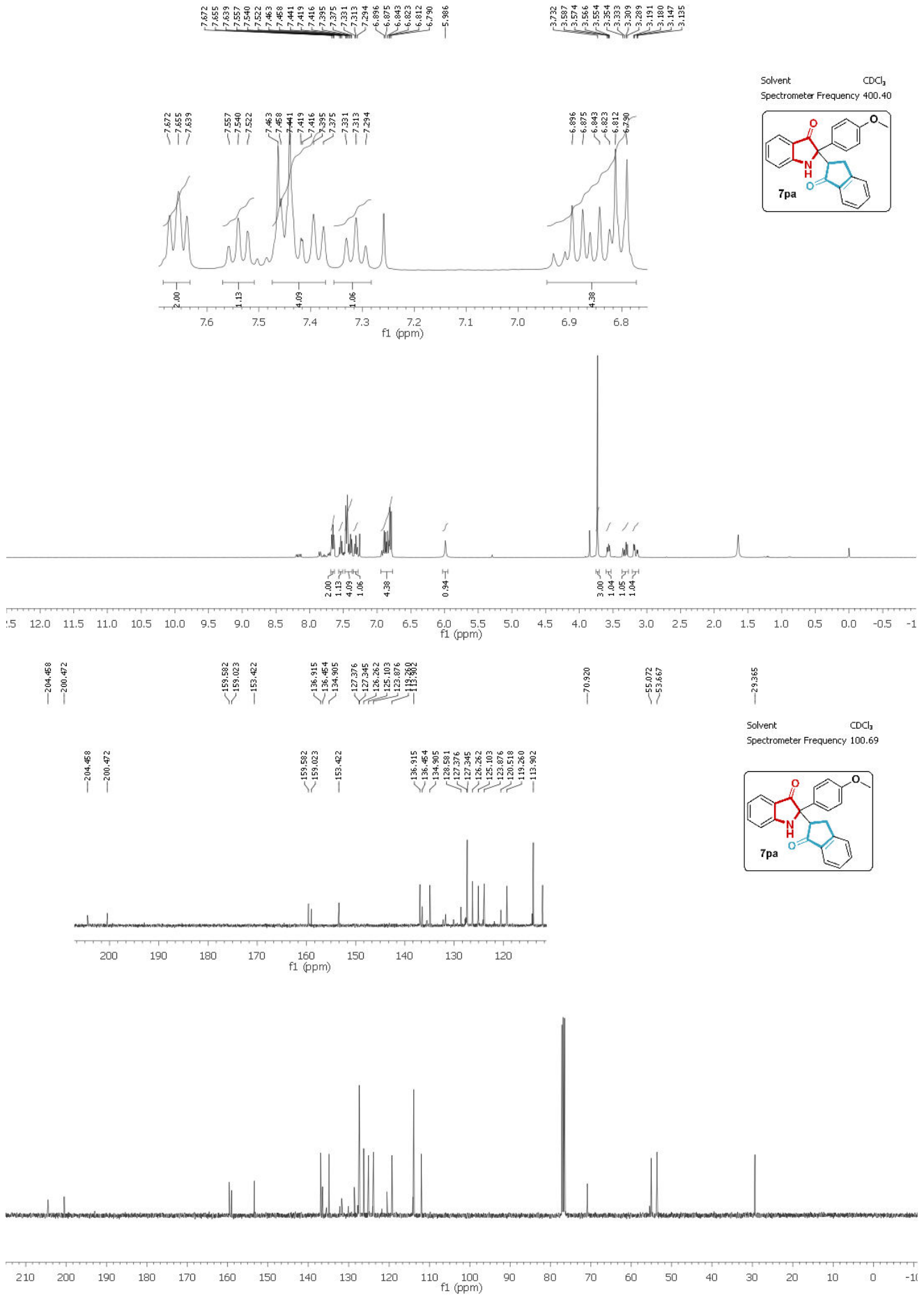

Solvent CDCl3
Spectrometer Frequency 100.69
7oa
f1 (ppm)

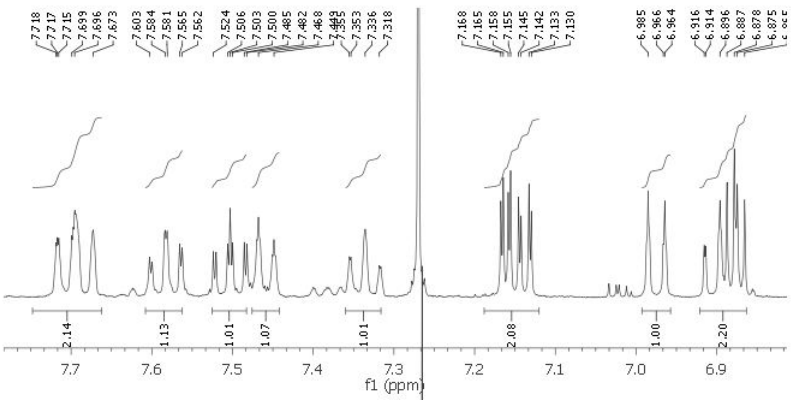

Solvent CDCl3
Spectrometer Frequency 400.40
7fa
f1 (ppm)

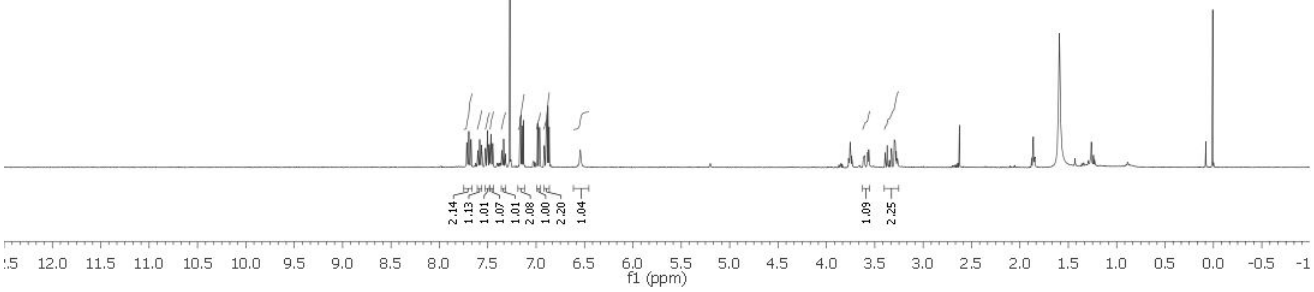

Solvent CDCl3
Spectrometer Frequency 100.69
7fa
f1 (ppm)

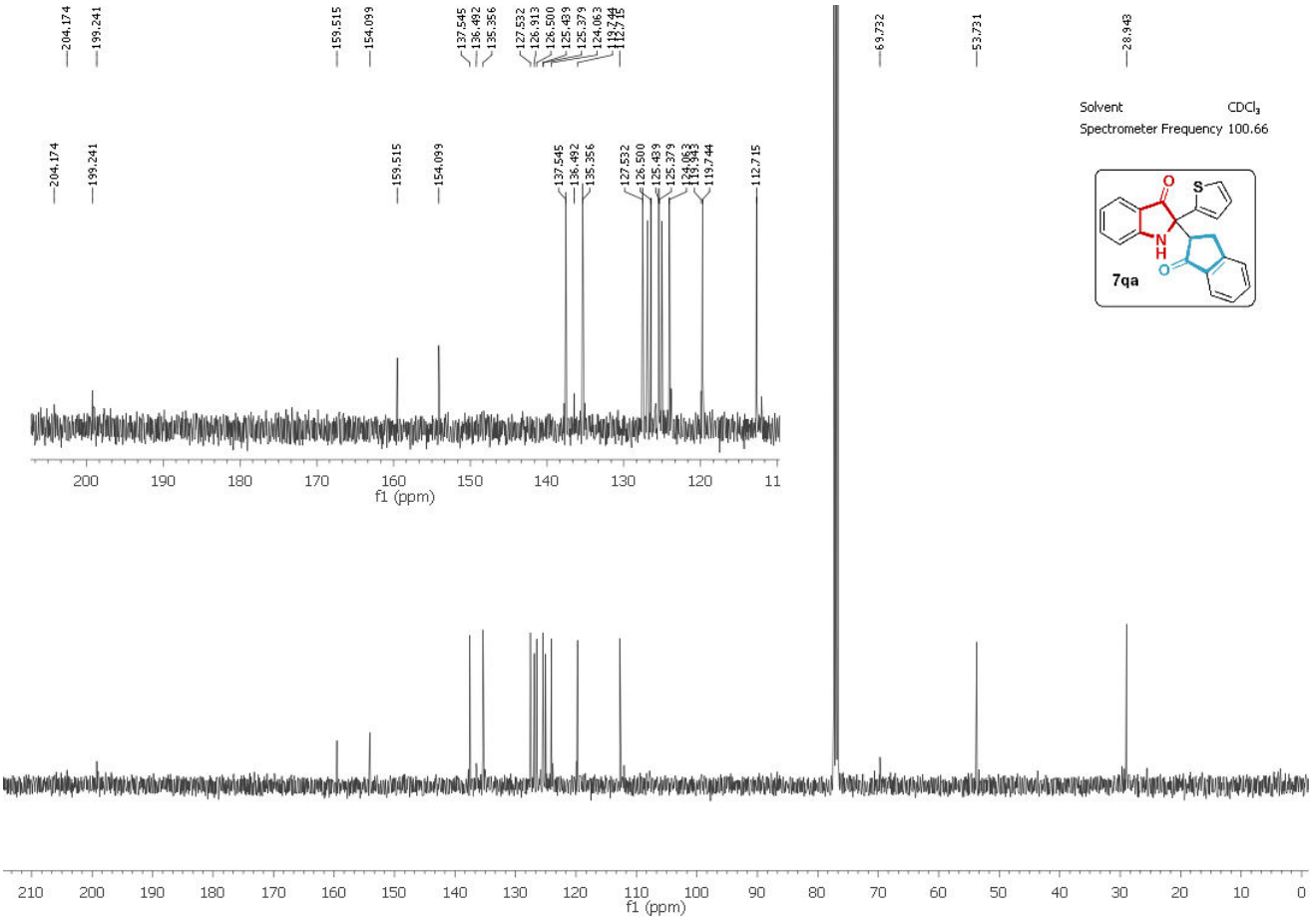

Solvent CDCl3
Spectrometer Frequency 400.40
7ja
f1 (ppm)

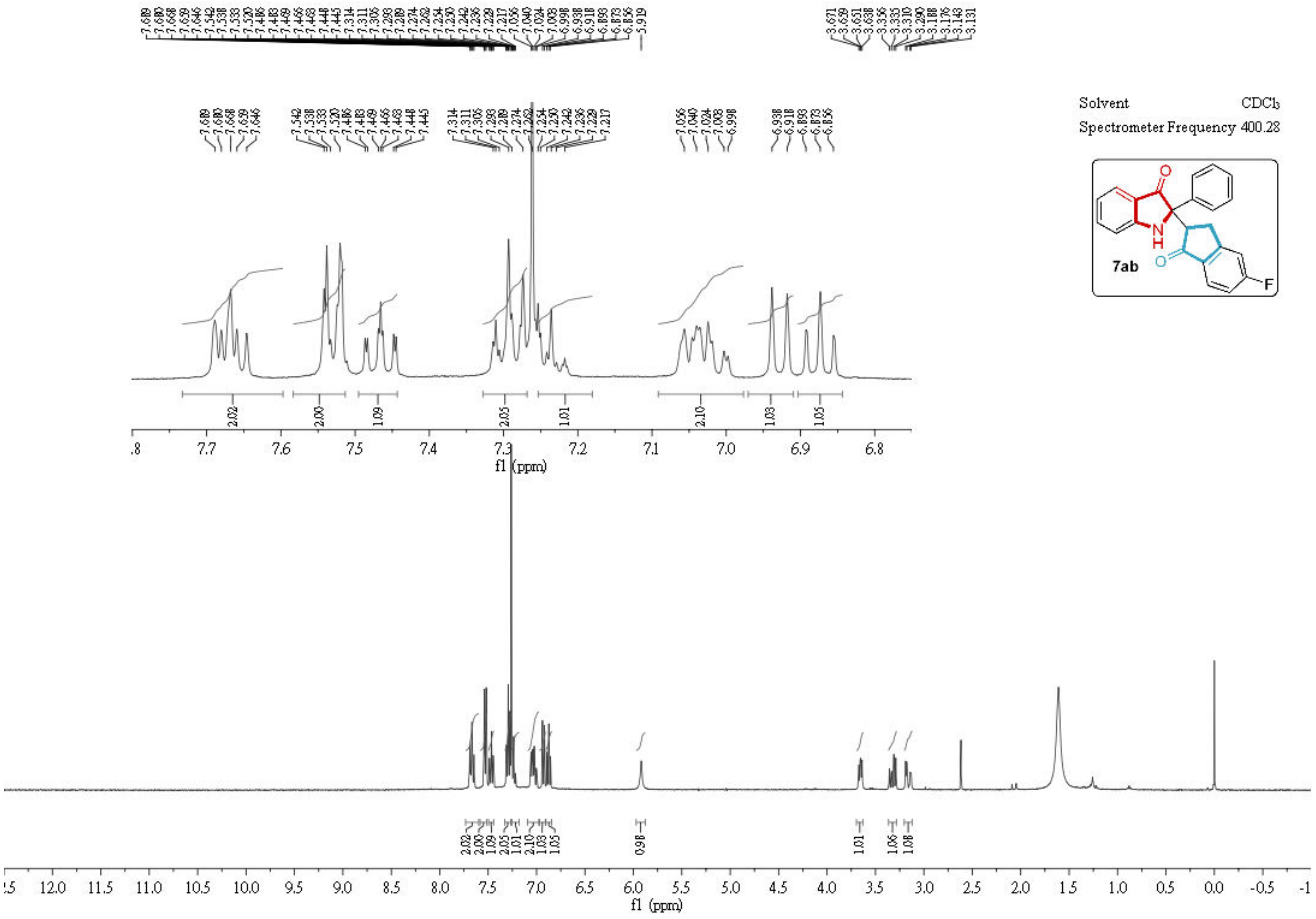

Solvent CDCl3
Spectrometer Frequency 100.69
7ja
f1 (ppm)

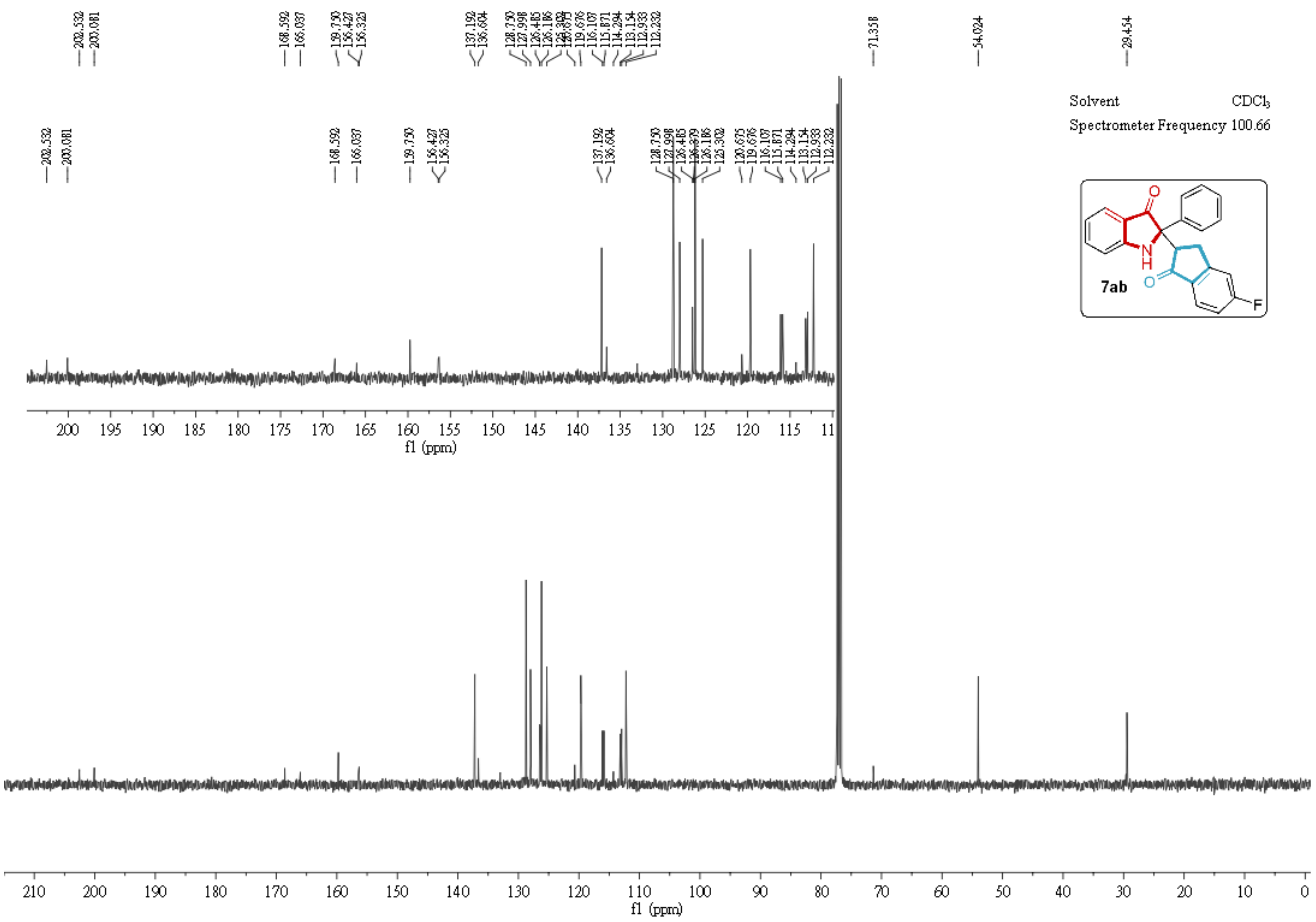

Solvent CDCl3
Spectrometer Frequency 400.40
7pa
f1 (ppm)
Solvent CDCl3
Spectrometer Frequency 100.69
7pa
f1 (ppm)

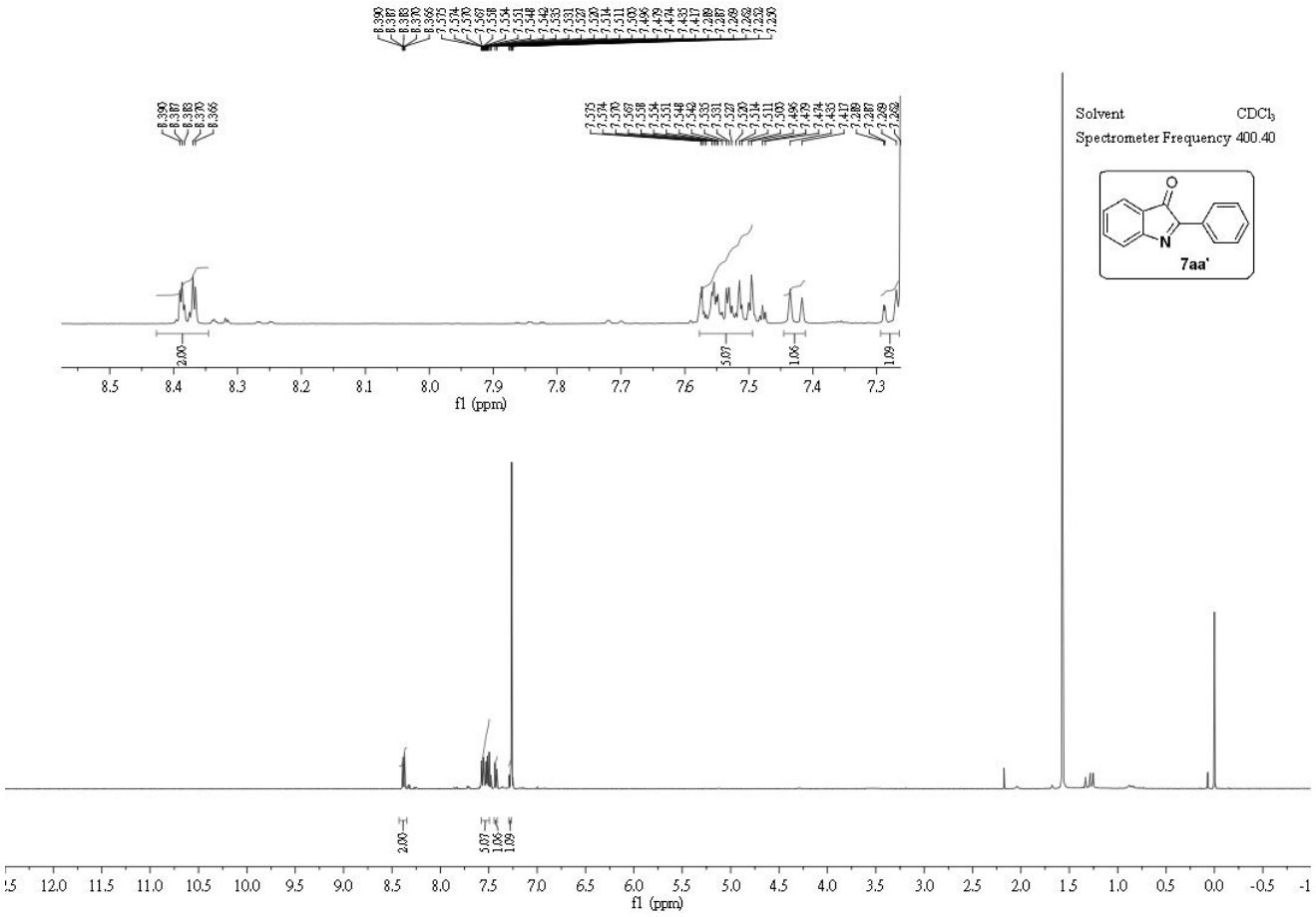

Solvent CDCl3
Spectrometer Frequency 400.40
7qa
f1 (ppm)

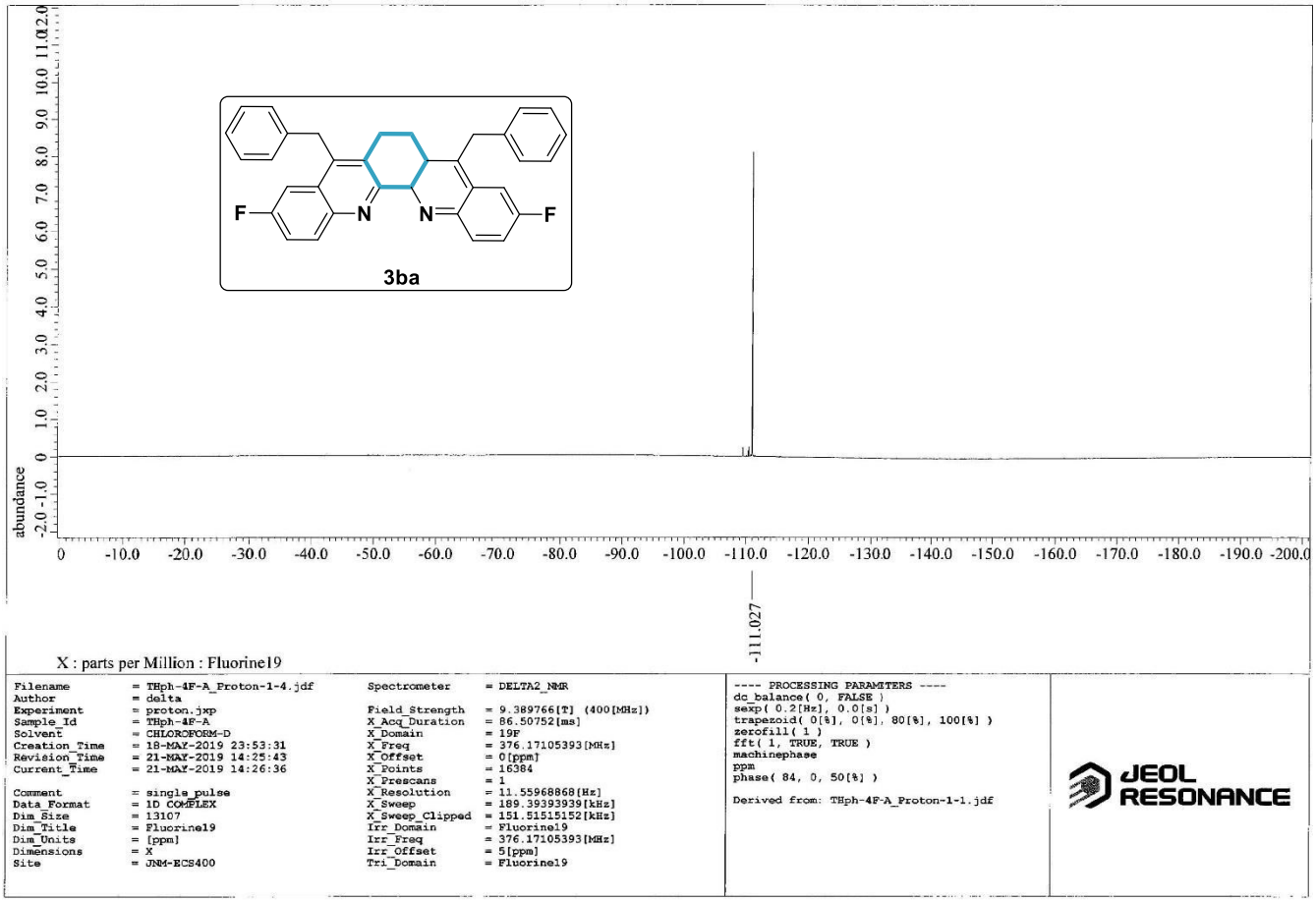

f1 (ppm)


Solvent CDCl3
Spectrometer Frequency 100.66
7qa
f1 (ppm)


Solvent CDCl3
Spectrometer Frequency 400.28
7ab
F
f1 (ppm)

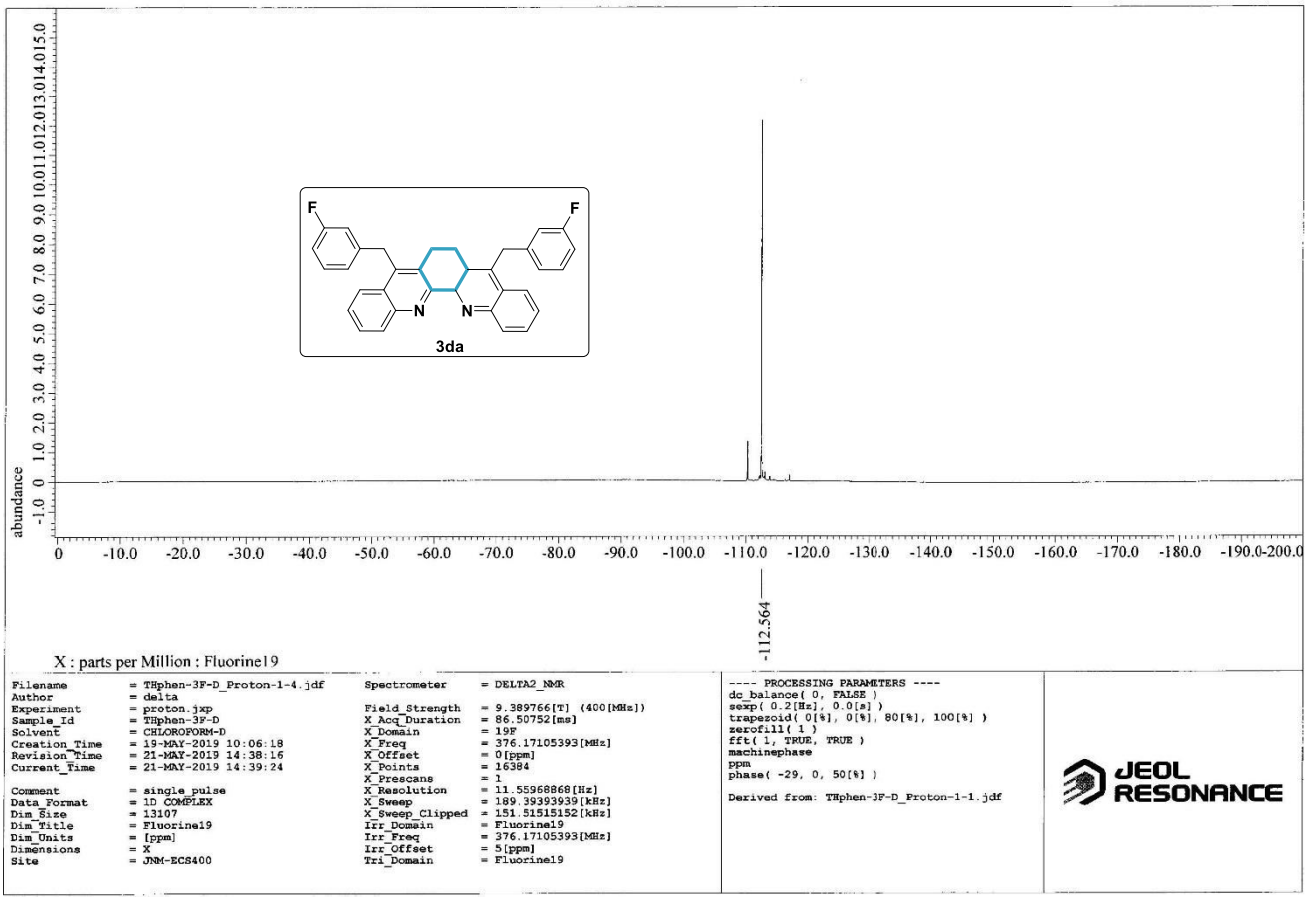

Solvent CDCl3
Spectrometer Frequency 100.66
7ab
F
f1 (ppm)


Solvent CDCl3
Spectrometer Frequency 400.40
7aa'
f1 (ppm)



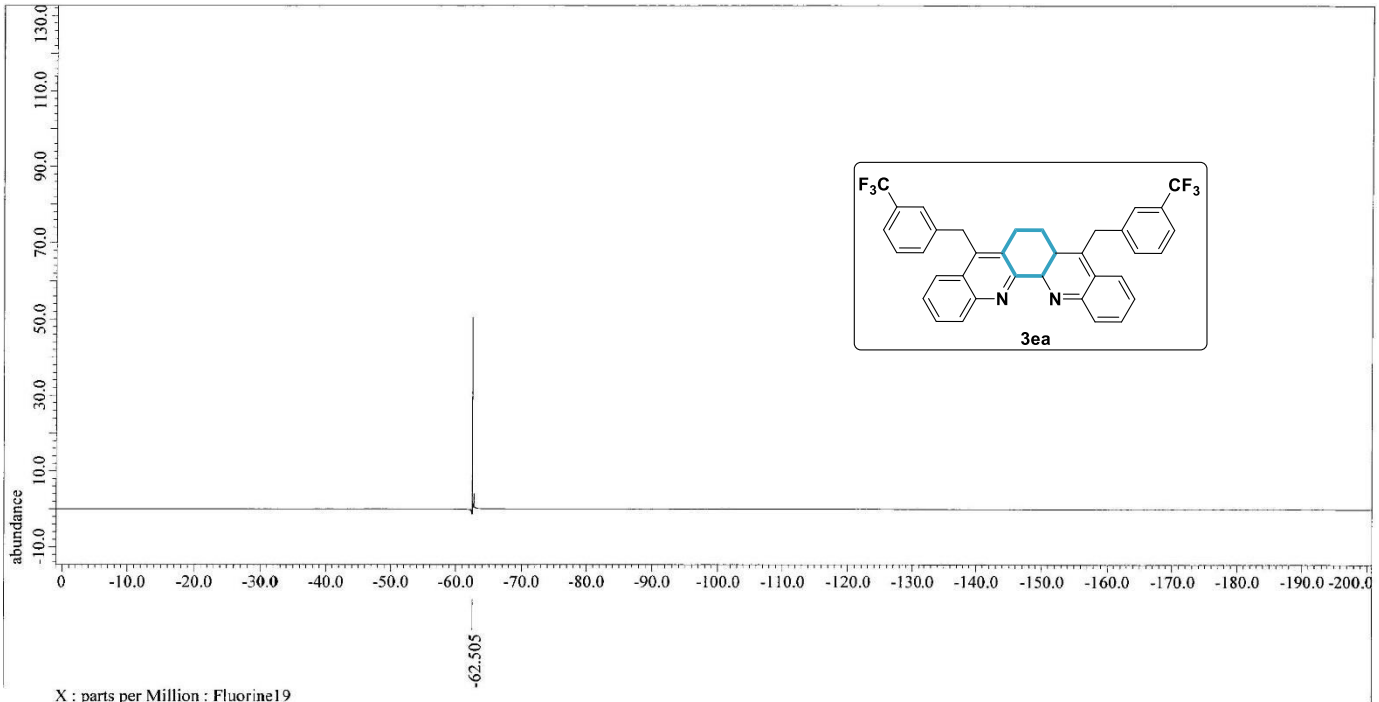
Filename	= THph-4F-A_Proton-1-4.jdf	Spectrometer	= DELTA2_NMR
Author	= delta		
Experiment	= proton.jxp	Field_Strength	= 9.389766[T] (400[MHz])
Sample_Id	= THph-4F-A	X_Acq_Duration	= 86.50752[ms]
Solvent	= CHLOROFORM-D	X_Domain	= 19F
Creation_Time	= 18-MAY-2019 23:53:31	X_Freq	= 376.17105393[MHz]
Revision_Time	= 21-MAY-2019 14:25:43	X_Offset	= 0[ppm]
Current_Time	= 21-MAY-2019 14:26:36	X_Points	= 16384
		X_Prescans	= 1
Comment	= single_pulse	X_Resolution	= 11.55968868[Hz]
Data_Format	= 1D COMPLEX	X_Sweep	= 189.39393939[kHz]
Dim_Size	= 13107	X_Sweep_Clipped	= 151.51515152[kHz]
Dim_Title	= Fluorine19	Irr_Domain	= Fluorine19
Dim_Units	= [ppm]	Irr_Freq	= 376.17105393[MHz]
Dimensions	= X	Irr_Offset	= 5[ppm]
Site	= JNM-ECS400	Tri_Domain	= Fluorine19

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 84, 0, 50[%] )

Derived from: THph-4F-A_Proton-1-1.jdf
```



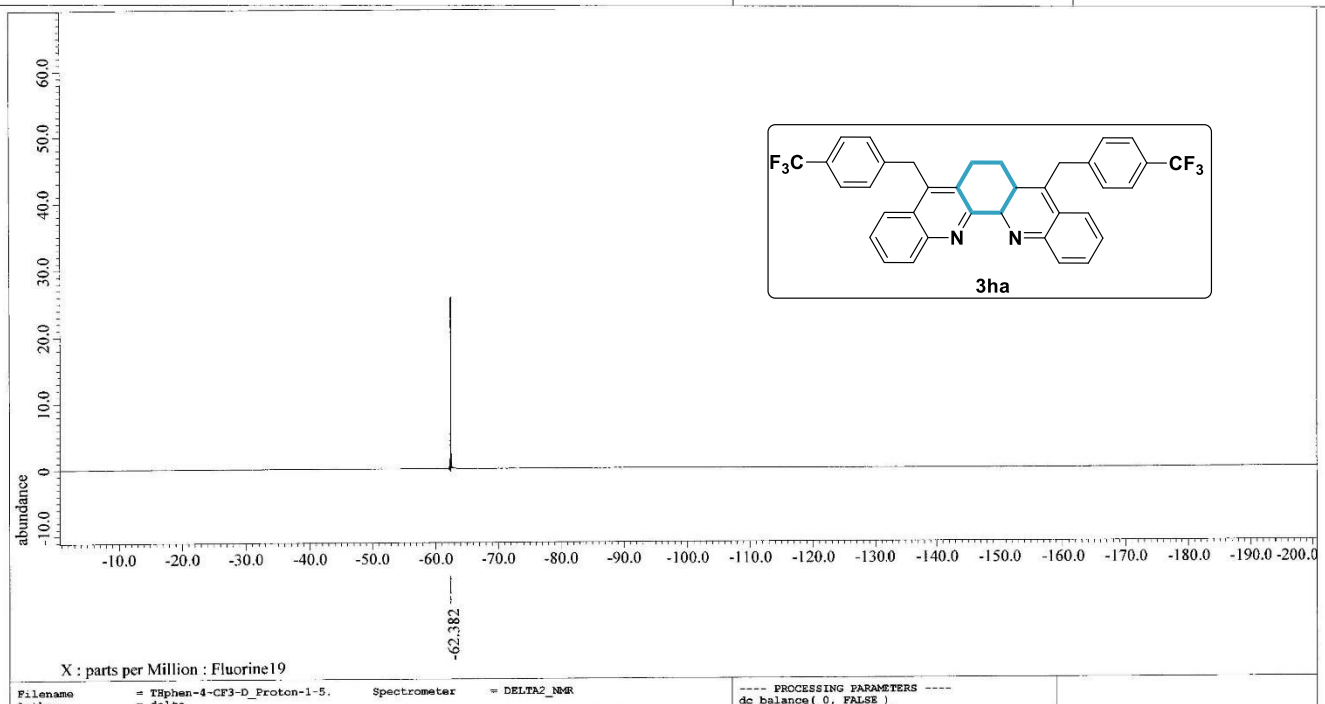
Filename	= THphen-3F-D_Proton-1-4.jdf	Spectrometer	= DELTA2_NMR
Author	= delta		
Experiment	= proton.jxp	Field_Strength	= 9.389766[T] (400[MHz])
Sample_Id	= THphen-3F-D	X_Acq_Duration	= 86.50752[ms]
Solvent	= CHLOROFORM-D	X_Domain	= 19F
Creation_Time	= 19-MAY-2019 10:06:18	X_Freq	= 376.17105393[MHz]
Revision_Time	= 21-MAY-2019 14:38:16	X_Offset	= 0[ppm]
Current_Time	= 21-MAY-2019 14:39:24	X_Points	= 16384
		X_Prescans	= 1
Comment	= single_pulse	X_Resolution	= 11.55968868[Hz]
Data_Format	= 1D COMPLEX	X_Sweep	= 189.39393939[kHz]
Dim_Size	= 13107	X_Sweep_Clipped	= 151.51515152[kHz]
Dim_Title	= Fluorine19	Irr_Domain	= Fluorine19
Dim_Units	= [ppm]	Irr_Freq	= 376.17105393[MHz]
Dimensions	= X	Irr_Offset	= 5[ppm]
Site	= JNM-ECS400	Tri_Domain	= Fluorine19

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -29, 0, 50[%] )

Derived from: THphen-3F-D_Proton-1-1.jdf
```



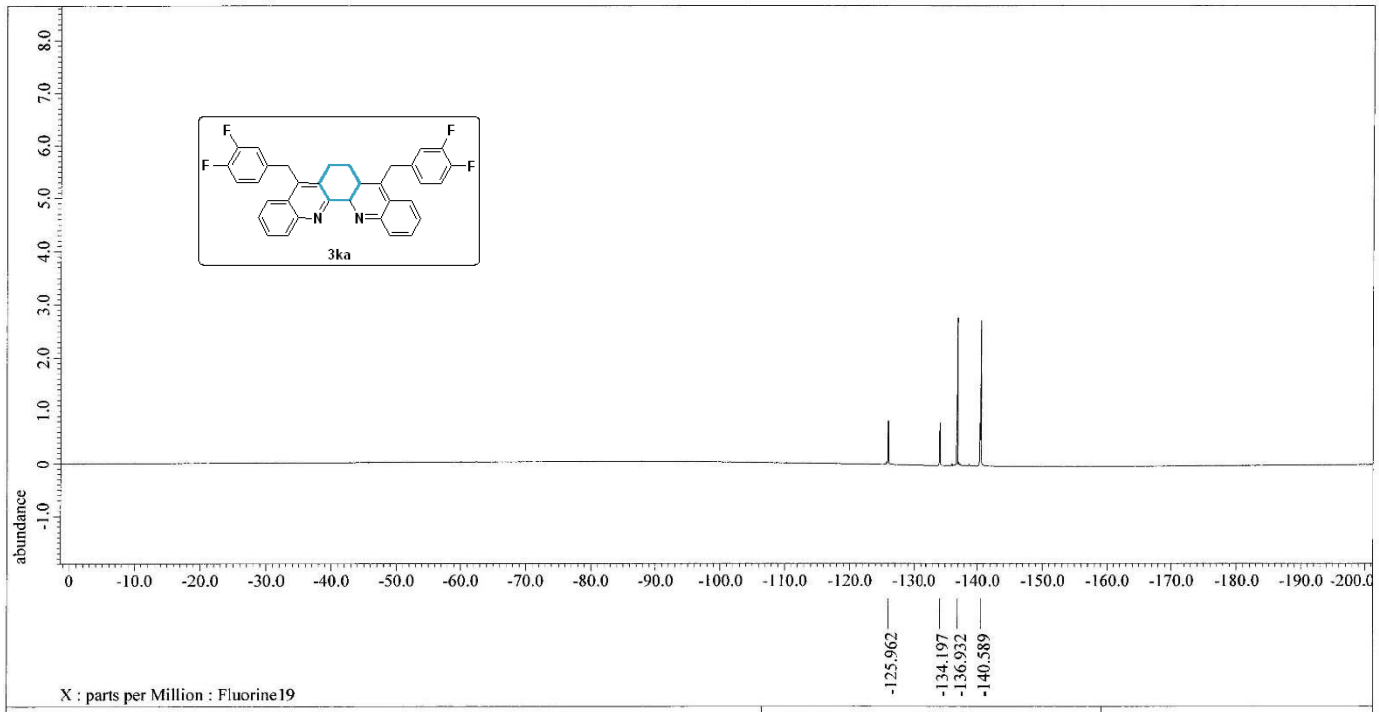

```
Filename          = THphen-3-CF3-D_Proton-1-6.
Author            = delta
Experiment        = proton.jxp
Sample_Id         = THphen-3-CF3-D
Solvent           = CHLOROFORM-D
Creation_Time     = 19-MAY-2019 10:39:34
Revision_Time     = 21-MAY-2019 14:36:22
Current_Time      = 21-MAY-2019 14:36:24

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Fluorine19
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400

Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 86.50752[ms]
X_Domain          = 19F
X_Freq            = 376.17105393[MHz]
X_Offset          = 0[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 11.55968868[Hz]
X_Sweep           = 189.39393939[kHz]
X_Sweep_Clipped   = 151.51515152[kHz]
Irr_Domain        = Fluorine19
Irr_Freq          = 376.17105393[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Fluorine19

---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 21, 0, 50[%] )

Derived from: THphen-3-CF3-D_Proton-1-1.jdf
```

JEOL RESONANCE



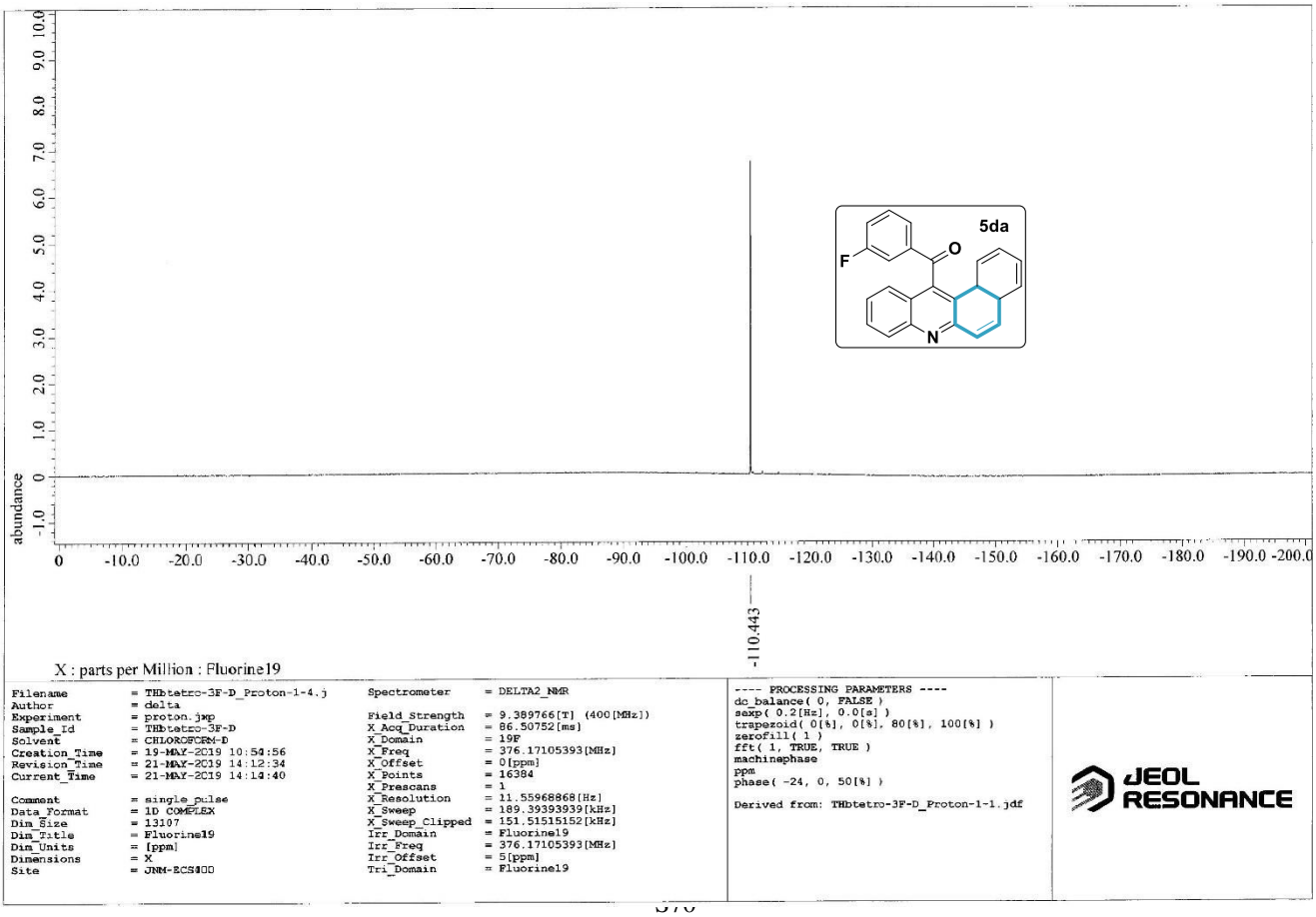

```
Filename          = THphen-4-CF3-D_Proton-1-5.
Author            = delta
Experiment        = proton.jxp
Sample_Id         = THphen-4-CF3-D
Solvent           = CHLOROFORM-D
Creation_Time     = 19-MAY-2019 10:27:03
Revision_Time     = 21-MAY-2019 14:41:58
Current_Time      = 21-MAY-2019 14:42:22

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Fluorine19
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400

Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 86.50752[ms]
X_Domain          = 19F
X_Freq            = 376.17105393[MHz]
X_Offset          = 0[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 11.55968868[Hz]
X_Sweep           = 189.39393939[kHz]
X_Sweep_Clipped   = 151.51515152[kHz]
Irr_Domain        = Fluorine19
Irr_Freq          = 376.17105393[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Fluorine19

---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 22, 0, 50[%] )

Derived from: THphen-4-CF3-D_Proton-1-1.jdf
```

JEOL RESONANCE



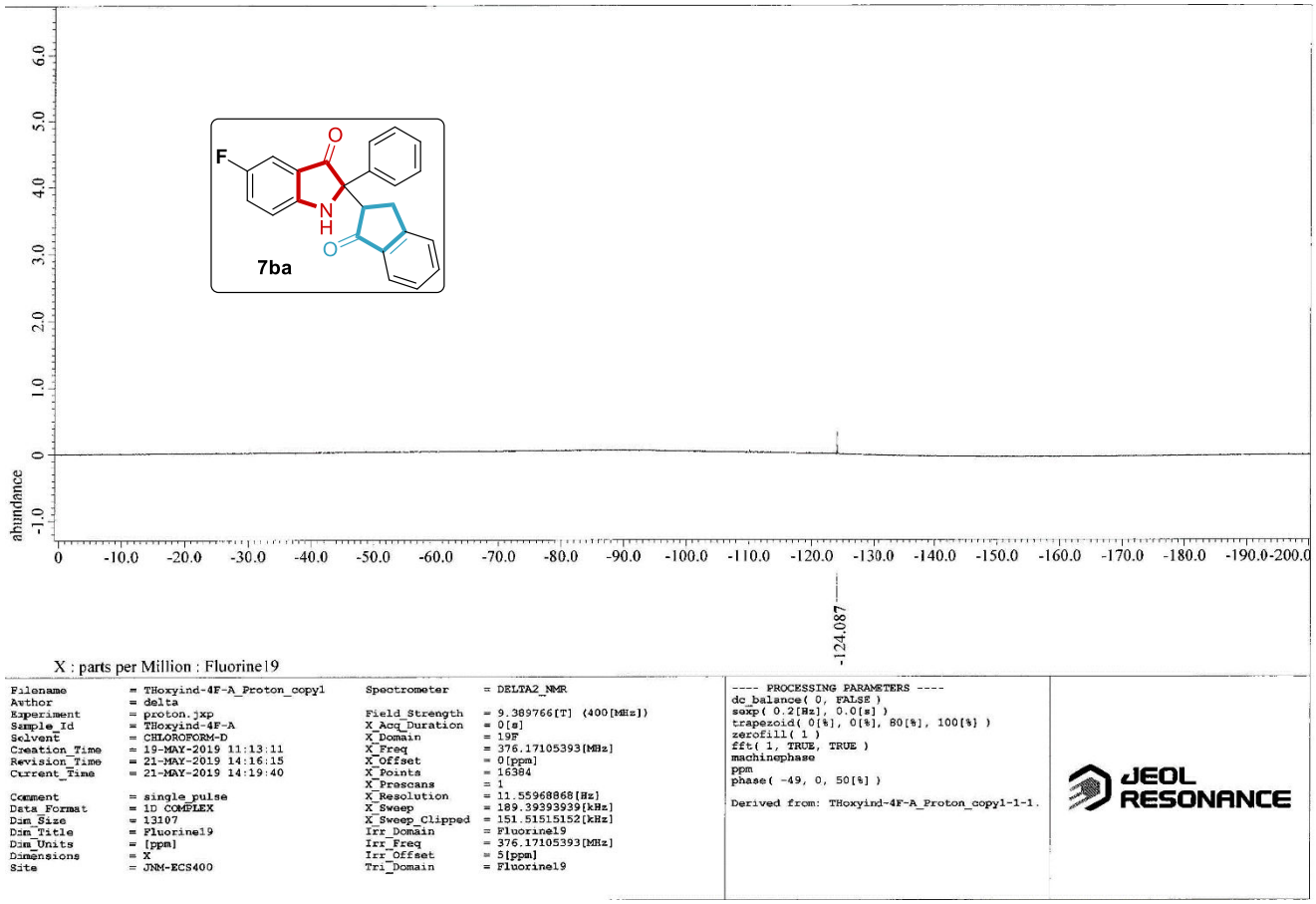
Filename	= TPphen-3,4-di-F_Proton-1-6	Spectrometer	= DELTA2_NMR
Author	= delta		
Experiment	= proton.jxp	Field_Strength	= 9.389766[T] (400[MHz])
Sample_Id	= TPphen-3,4-di-F	X_Acq_Duration	= 86.50752[ms]
Solvent	= CHLOROFORM-D	X_Domain	= 19F
Creation_Time	= 19-MAY-2019 09:21:40	X_Freq	= 376.17105393[MHz]
Revision_Time	= 21-MAY-2019 14:30:47	X_Offset	= 0[ppm]
Current_Time	= 21-MAY-2019 14:31:36	X_Points	= 16384
		X_Prescans	= 1
Comment	= single_pulse	X_Resolution	= 11.55968868[Hz]
Data_Format	= 1D COMPLEX	X_Sweep	= 189.39393939[kHz]
Dim_Size	= 13107	X_Sweep_Clipped	= 151.51515152[kHz]
Dim_Title	= Fluorine19	Irr_Domain	= Fluorine19
Dim_Units	= [ppm]	Irr_Freq	= 376.17105393[MHz]
Dimensions	= X	Irr_Offset	= 5[ppm]
Site	= JNM-ECS400	Tri_Domain	= Fluorine19

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -59, 0, 50[%] )

Derived from: TPphen-3,4-di-F_Proton-1-1.jdf
```



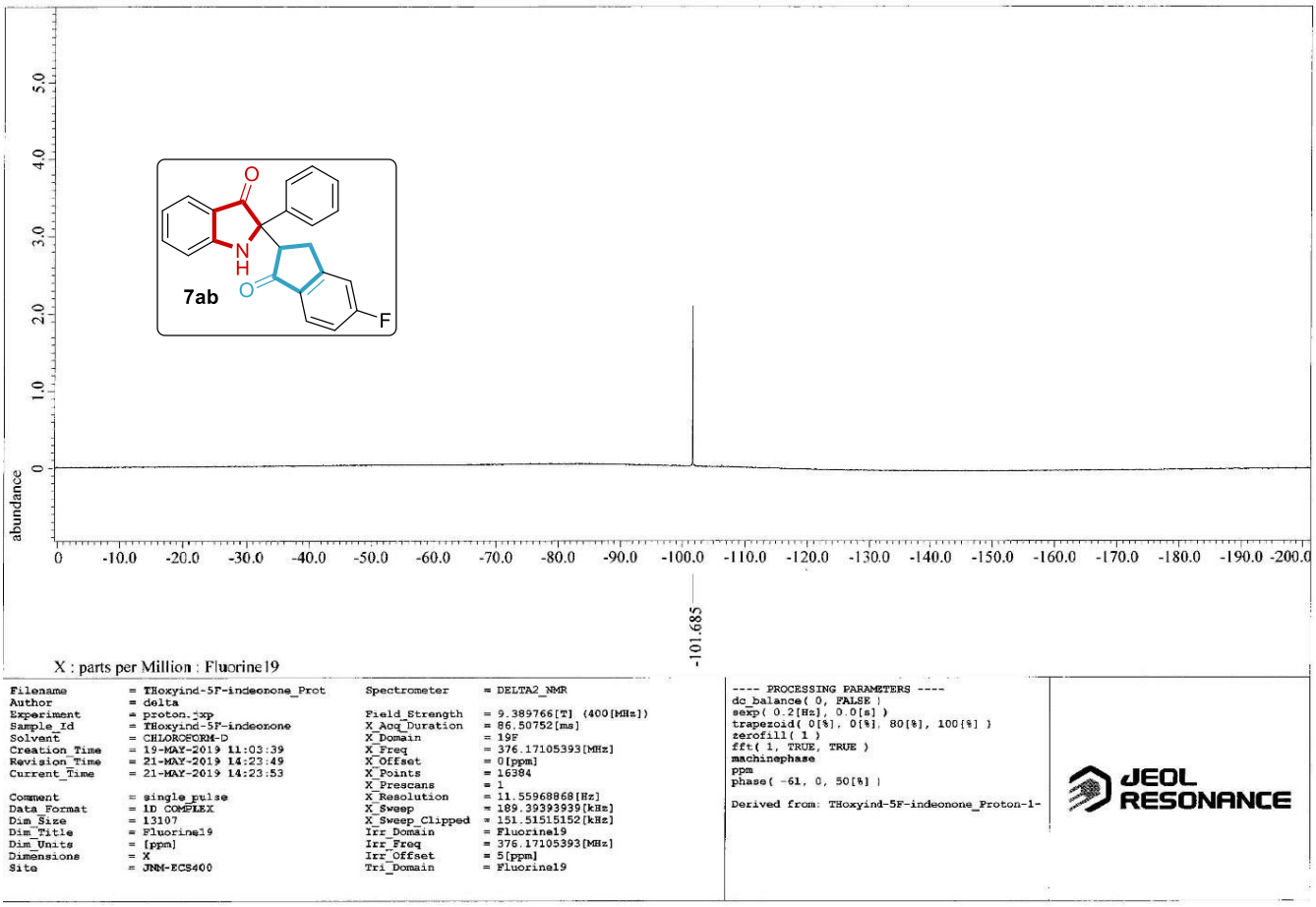
Filename	= THbtetro-3F-D_Proton-1-4.j	Spectrometer	= DELTA2_NMR
Author	= delta		
Experiment	= proton.jxp	Field_Strength	= 9.389766[T] (400[MHz])
Sample_Id	= THbtetro-3F-D	X_Acq_Duration	= 86.50752[ms]
Solvent	= CHLOROFORM-D	X_Domain	= 19F
Creation_Time	= 19-MAY-2019 10:54:56	X_Freq	= 376.17105393[MHz]
Revision_Time	= 21-MAY-2019 14:12:34	X_Offset	= 0[ppm]
Current_Time	= 21-MAY-2019 14:13:40	X_Points	= 16384
		X_Prescans	= 1
Comment	= single_pulse	X_Resolution	= 11.55968868[Hz]
Data_Format	= 1D COMPLEX	X_Sweep	= 189.39393939[kHz]
Dim_Size	= 13107	X_Sweep_Clipped	= 151.51515152[kHz]
Dim_Title	= Fluorine19	Irr_Domain	= Fluorine19
Dim_Units	= [ppm]	Irr_Freq	= 376.17105393[MHz]
Dimensions	= X	Irr_Offset	= 5[ppm]
Site	= JNM-ECS400	Tri_Domain	= Fluorine19

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -24, 0, 50[%] )

Derived from: THbtetro-3F-D_Proton-1-1.jdf
```



Filename	= THoxyind-4F-A_Proton_copy1	Spectrometer	= DELTA2_NMR
Author	= delta		
Experiment	= proton.jxp	Field_Strength	= 9.389766[T] (400[MHz])
Sample_Id	= THoxyind-4F-A	X_Acq_Duration	= 0[s]
Solvent	= CHLOROFORM-D	X_Domain	= 19F
Creation_Time	= 19-MAY-2019 11:13:11	X_Freq	= 376.17105393[MHz]
Revision_Time	= 21-MAY-2019 14:16:15	X_Offset	= 0[ppm]
Current_Time	= 21-MAY-2019 14:19:40	X_Points	= 16384
		X_Prescans	= 1
Comment	= single_pulse	X_Resolution	= 11.55968868[Hz]
Data_Format	= 1D COMPLEX	X_Sweep	= 189.39393939[kHz]
Dim_Size	= 13107	X_Sweep_Clipped	= 151.51515152[kHz]
Dim_Title	= Fluorine19	Irr_Domain	= Fluorine19
Dim_Units	= [ppm]	Irr_Freq	= 376.17105393[MHz]
Dimensions	= X	Irr_Offset	= 5[ppm]
Site	= JNM-ECS400	Tri_Domain	= Fluorine19

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -49, 0, 50[%] )

Derived from: THoxyind-4F-A_Proton_copy1-1-1.
```

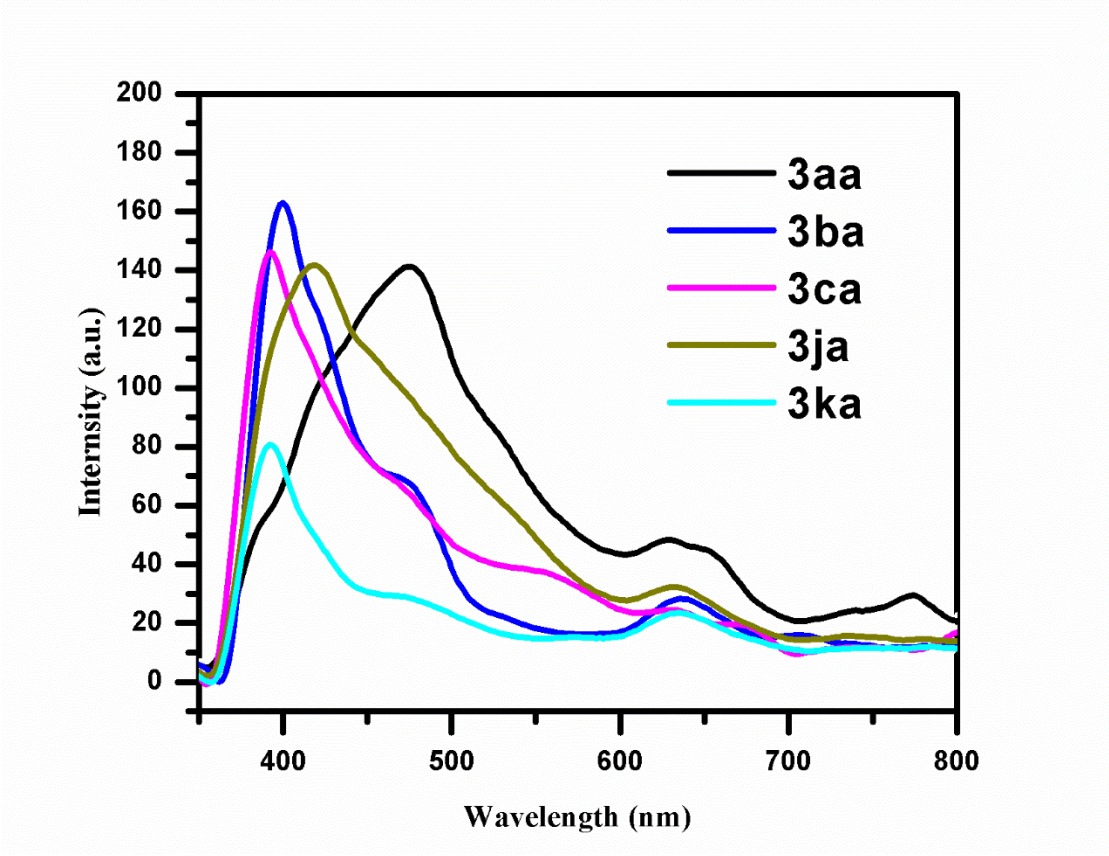


Filename	= THoxyind-5F-indeonone_Prot	Spectrometer	= DELTA2_NMR
Author	= delta		
Experiment	= proton.jxp	Field_Strength	= 9.389766[T] (400[MHz])
Sample_Id	= THoxyind-5F-indeonone	X_Acq_Duration	= 86.50752[ms]
Solvent	= CHLOROFORM-D	X_Domain	= 19F
Creation_Time	= 19-MAY-2019 11:03:39	X_Freq	= 376.17105393[MHz]
Revision_Time	= 21-MAY-2019 14:23:49	X_Offset	= 0[ppm]
Current_Time	= 21-MAY-2019 14:23:53	X_Points	= 16384
		X_Prescans	= 1
Comment	= single_pulse	X_Resolution	= 11.55968868[Hz]
Data_Format	= 1D COMPLEX	X_Sweep	= 189.39393939[kHz]
Dim_Size	= 13107	X_Sweep_Clipped	= 151.51515152[kHz]
Dim_Title	= Fluorine19	Irr_Domain	= Fluorine19
Dim_Units	= [ppm]	Irr_Freq	= 376.17105393[MHz]
Dimensions	= X	Irr_Offset	= 5[ppm]
Site	= JNM-ECS400	Tri_Domain	= Fluorine19

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -61, 0, 50[%] )

Derived from: THoxyind-5F-indeonone_Proton-1-
```

10) Uv-visible absorption and emission spectra of dibenzo[*b,j*][1,10]phenanthrolines

Figure. S3 Uv-visible absorption spectra of dibenzo[*b,j*][1,10]phenanthrolines compounds in chloroform .



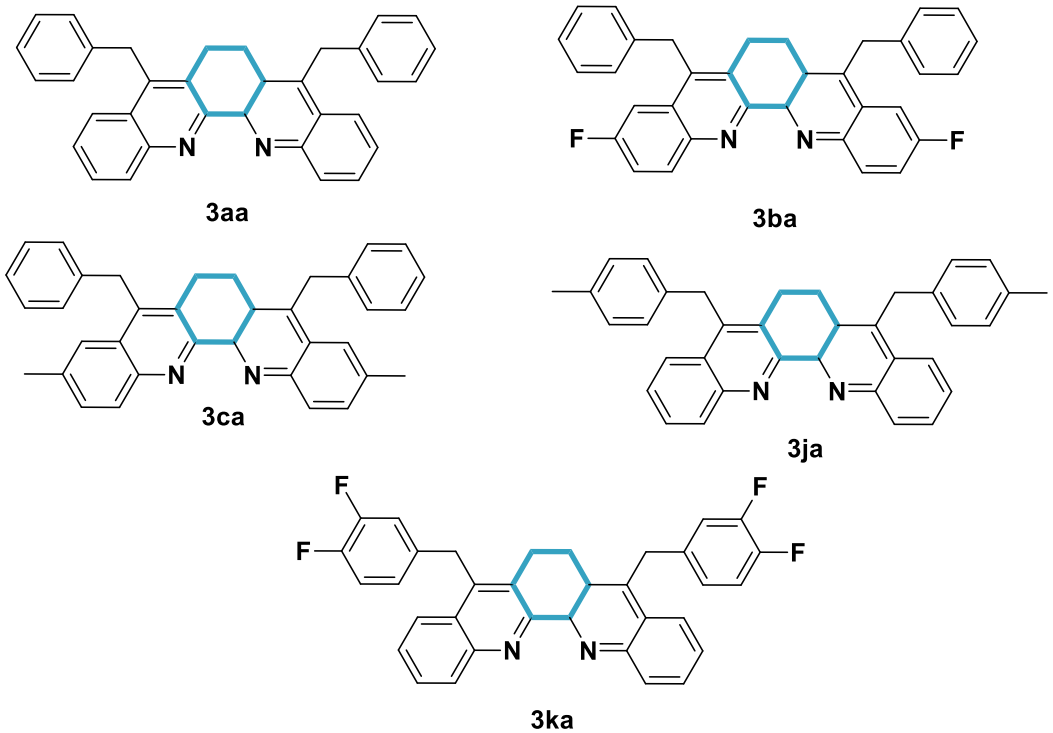


Figure. S4 Uv-visible emission spectra of dibenzo[*b,j*][1,10]phenanthrolines compounds in chloroform .