Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Supporting Information

Visible-light-mediated Radical Cascade Alkylation/Cyclization: Access to alkylated Indolo/Benzoimidazo[2,1-*a*]isoquinolin-6(5*H*)-ones

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1. General Information

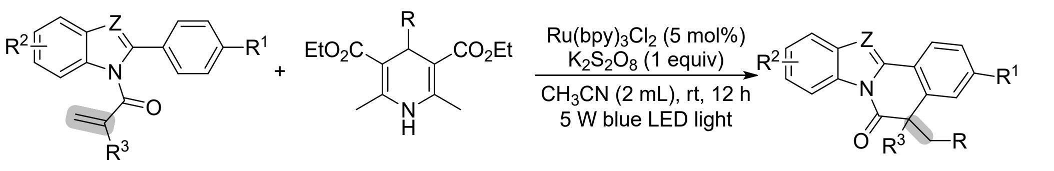
Unless otherwise stated, all commercial reagents were used as received. Propiophenone, aldehydes (Innochem, >98%), *o*-Phenylenediamine and Phenylhydrazine hydrochloride were used without further treatment. All reagents and solvents were commercially available and used without any further purification unless specified. All solvents were dried and distilled according to standard procedures. Flash column chromatography was performed using silica gel (0.25mm, 300-400 mesh). Analytical thin-layer chromatography was performed using glass plates pre-coated with 0.25mm 300-400 mesh silica gel impregnated with a fluorescent indicator (254 nm). All reactions were carried out with magnetic stirring and in dried glassware.Nuclear magnetic resonance (NMR) spectra are recorded in parts per million from internal tetramethylsilane on the δ scale. ^{1}H NMR, ^{19}F NMR and ^{13}C NMR spectra were recorded in $CDCl_3$ on a Bruker DRX-400 spectrometer operating at 400 MHz, 376 MHz and 100 MHz, respectively. All chemical shift values are quoted in ppm and coupling constants quated in Hz. The solvent peak was used as a reference value, for ^{1}H NMR: TMS = 0.00 ppm, for ^{13}C NMR: $CDCl_3$ = 77.00 ppm. The following abbreviations were used to explain multiplicities: s = singlet, d = doublet, dd = doublet of doublet, t = triplet, td = triplet of doublet, q = quartet, m = multiplet, and br = broad. High-resolution mass spectra (HRMS) were obtained on an Agilent mass spectrometer using ESI-TOF (electrospray ionization-time of flight).

2. Experiment Section

2.1 General Procedure for the Synthesis of Substrates

All 2-aryl indoles **1**[1] and 4-alkylated Hantzsch esters **2**[2-3] were synthesized according to the known methods.

2.2 Typical Experimental Procedure

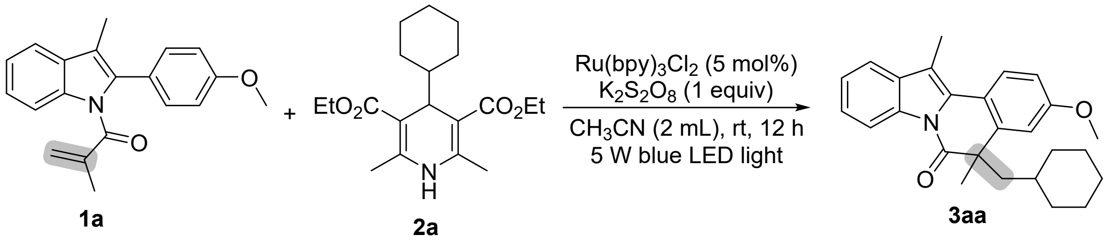


1 **2** **3**

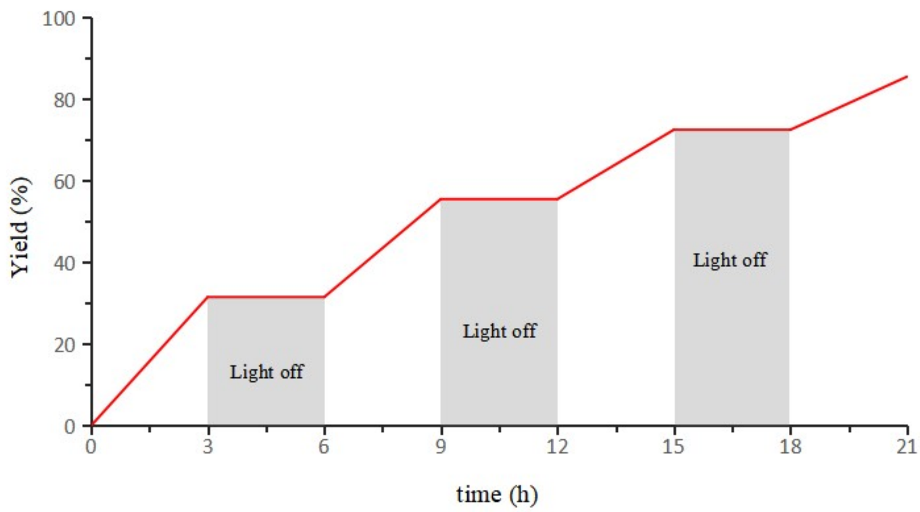
To a Schlenk tube were added 2-aryl indoles **1** (0.2 mmol), alkyl Hantzsch esters **2** (2 equiv, 0.4 mmol), $Ru(bpy)_2Cl_2$ (5 mol%, 0.01 mmol), $K_2S_2O_8$ (1 equiv, 0.2 mmol) and CH_3CN (2 mL). Then the tube was stirred at room temperature in Ar atmosphere for the indicated time until complete consumption of starting material as monitored by TLC analysis. The residue was purified by silica gel flash column chromatography (petroleum ether/ethyl acetate = 30 : 1) to afford the desired products **3**.

2.3 The Light on/off Experiments

Figure S1 The Light on/off Experiments



Time/h	0	3 (blue)	6 (dark)	9 (blue)	12 (dark)	15(blue)	18(dark)	21(blue)
Yield/%	0	31.5	31.5	55.5	55.5	72.5	72.5	85.5



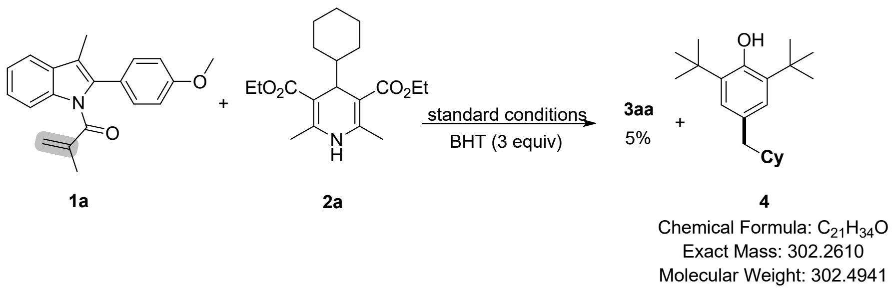
2.4 Details of Visible-Light Source

The light source bought from SANYI (https://item.taobao.com/item.htm?spm=a1z09.2.0.0.42672e8dv2Chsz&id=35497290577&_u=j35sh1qt9325), 5 W blue LED light bulb (E27). The wavelength was about 460-470 nm and the wavelength of peak intensity was about 467.5 nm. The pictures of the visible-light source (Figure S1) was shown as follow:

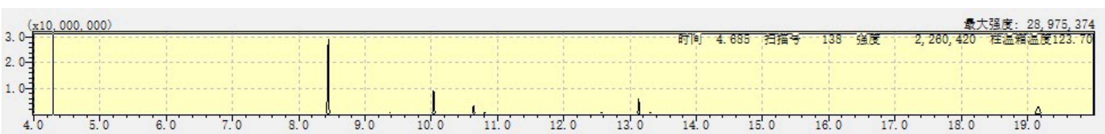
Figure S2. Pictures of Visible-Light Source. Reproduced from [Liu, Y.; Wang, Q.-L.; Chen, Z.; Zhou, Q.; Li, H.; Zhou, C.-S.; Xiong, B.-Q.; Zhang, P.-L.; and Tang, K.-W.; Visible-Light-Catalyzed C−C Bond Difunctionalization of Methylenecyclopropanes with Sulfonyl Chlorides for the Synthesis of 3-Sulfonyl-1,2-dihydronaphthalenes, *J. Org. Chem.* **2019**, *84*, 2829-2839]. Copyright [2019] American Chemical Society

2.5 Control Experiments

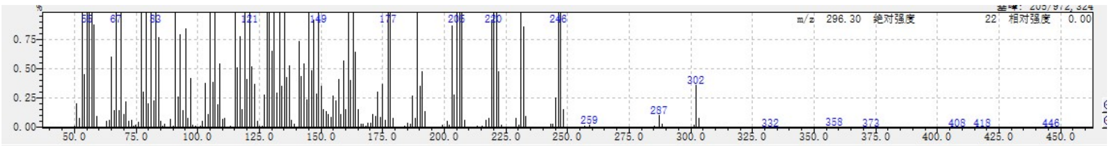
2.5.1 GC-MS Analysis of Raw Reaction Mixture by Using BHT as Radical Inhibitor



Spectra of GC-MS



MS spectra of the peak at 10.640 min



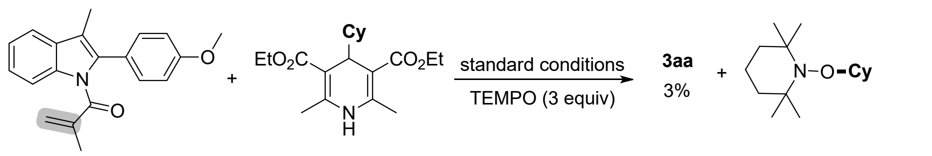
[MS Spectrum]
# of Peaks313
Raw Spectrum 10.640 (scan : 1329)
Background 10.605 (scan : 1322)
Base Peakm/z 205.15 (Inten : 972,324)
Event# 1

m/z	Absolute Intensity	Relative Intensity
50.00	158	0.02
51.00	1995	0.21
52.00	849	0.09
53.05	11406	1.17
54.05	4421	0.45
55.05	247612	25.47
56.05	11913	1.23
57.05	177837	18.29
58.05	8604	0.88
59.10	990	0.10
60.10	16	0.00
61.10	21	0.00
62.00	46	0.00
63.05	580	0.06
64.05	672	0.07
65.05	5943	0.61
66.00	1449	0.15
67.00	14942	1.54
68.00	1493	0.15
69.05	13052	1.34
70.00	1105	0.11
71.00	2165	0.22
72.15	572	0.06
73.20	672	0.07
73.90	154	0.02
74.95	243	0.02
76.00	464	0.05
77.00	12401	1.28
78.00	2960	0.30
79.00	14787	1.52
80.05	2017	0.21
81.05	14494	1.49
82.15	2280	0.23
83.05	105424	10.84
84.05	7499	0.77
85.20	555	0.06
85.80	150	0.02
86.75	322	0.03
87.80	39	0.00
89.05	729	0.07
89.95	197	0.02
91.05	23762	2.44
92.10	2555	0.26
93.05	7805	0.80
94.10	1456	0.15
95.05	8238	0.85
96.05	815	0.08
97.05	4137	0.43
98.10	254	0.03
99.10	139	0.01
100.10	5	0.00
101.00	202	0.02
102.05	568	0.06
103.00	3723	0.38
104.05	1100	0.11
105.05	32846	3.38
106.05	3834	0.39
107.05	9542	0.98
108.05	1953	0.20
109.05	5303	0.55
110.10	730	0.08
111.10	809	0.08
112.10	19	0.00
113.10	128	0.01
114.10	61	0.01
115.05	9761	1.00
116.05	5008	0.52
117.05	7638	0.79
118.05	1572	0.16
119.10	23670	2.43
120.10	4062	0.42
121.10	38157	3.92
122.10	5132	0.53
123.10	3610	0.37
124.10	550	0.06
125.10	160	0.02
126.10	149	0.02
127.15	2731	0.28
128.10	9770	1.00
129.10	10686	1.10
130.10	6405	0.66
131.10	12507	1.29

132.15	2914	0.30	176.15	654	0.07	225.10	14	0.00
133.15	13761	1.42	177.10	60605	6.23	226.10	24	0.00
134.15	3516	0.36	178.10	9545	0.98	227.10	54	0.01
135.15	16703	1.72	179.10	802	0.08	228.20	26	0.00
136.10	4197	0.43	180.10	66	0.01	229.15	803	0.08
137.05	5177	0.53	181.10	37	0.00	230.25	280	0.03
138.10	628	0.06	182.10	70	0.01	231.15	48640	5.00
139.10	299	0.03	183.10	113	0.01	232.15	8411	0.87
140.10	73	0.01	184.10	148	0.02	233.10	978	0.10
141.10	7168	0.74	185.10	431	0.04	234.10	107	0.01
142.10	4286	0.44	186.15	337	0.03	235.10	5	0.00
143.10	5337	0.55	187.10	2710	0.28	236.10	24	0.00
144.15	2312	0.24	188.15	842	0.09	238.10	20	0.00
145.10	28033	2.88	189.10	18840	1.94	239.10	29	0.00
146.10	4786	0.49	190.10	3472	0.36	240.10	76	0.01
147.10	8986	0.92	191.10	4680	0.48	241.10	92	0.01
148.10	2803	0.29	192.10	1410	0.15	242.10	62	0.01
149.10	32844	3.38	193.10	69	0.01	243.25	303	0.03
150.10	3450	0.35	197.10	41	0.00	244.20	324	0.03
151.15	1564	0.16	198.10	46	0.00	245.20	2542	0.26
152.05	1371	0.14	199.10	206	0.02	246.20	82533	8.49
153.10	1133	0.12	200.10	102	0.01	247.20	14987	1.54
154.05	853	0.09	201.15	606	0.06	248.15	1517	0.16
155.05	2674	0.28	202.10	214	0.02	249.10	120	0.01
156.10	2284	0.23	203.15	8482	0.87	250.10	53	0.01
157.15	4019	0.41	204.15	2750	0.28	251.10	19	0.00
158.15	1172	0.12	205.15	972324	100.00	253.10	39	0.00
159.10	5570	0.57	206.15	150404	15.47	255.10	31	0.00
160.15	1504	0.15	207.15	13515	1.39	256.10	18	0.00
161.10	22328	2.30	208.20	650	0.07	257.10	167	0.02
162.10	3960	0.41	209.20	18	0.00	258.10	44	0.00
163.10	16903	1.74	210.20	22	0.00	259.10	234	0.02
164.10	6327	0.65	212.20	31	0.00	260.10	86	0.01
165.10	1542	0.16	213.20	147	0.02	262.10	107	0.01
166.10	312	0.03	214.20	108	0.01	266.10	14	0.00
167.05	350	0.04	215.20	181	0.02	267.10	24	0.00
168.10	122	0.01	216.00	82	0.01	269.10	11	0.00
169.05	420	0.04	217.05	614	0.06	270.10	24	0.00
170.10	389	0.04	218.15	826	0.08	271.10	95	0.01
171.10	1101	0.11	219.15	9021	0.93	272.10	49	0.01
172.05	1011	0.10	220.15	344455	35.43	273.10	2	0.00
173.10	2978	0.31	221.15	55632	5.72	276.10	3	0.00
174.05	891	0.09	222.15	4733	0.49	277.10	8	0.00
175.10	3680	0.38	223.10	235	0.02	278.10	23	0.00

280.10	25	0.00	291.30	5	0.00	**301.20**	**254**	**0.03**
282.10	19	0.00	292.30	3	0.00	**302.25**	**3558**	**0.37**
283.10	42	0.00	294.30	15	0.00	**303.25**	**817**	**0.08**
285.10	133	0.01	295.30	29	0.00	304.20	55	0.01
286.10	56	0.01	296.30	22	0.00	305.20	37	0.00
287.30	1015	0.10	297.30	23	0.00	306.20	21	0.00
288.30	318	0.03	298.30	31	0.00	307.20	4	0.00
289.30	36	0.00	300.30	74	0.01	309.20	21	0.00

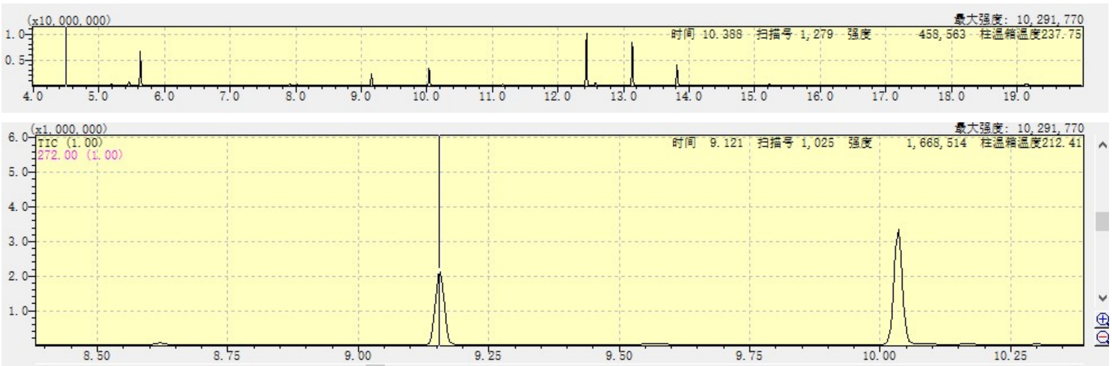
2.5.2 GC-MS Analysis of Raw Reaction Mixture by Using TEMPO as Radical Inhibitor



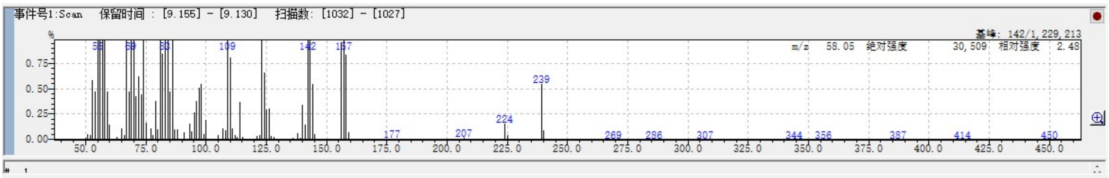
1a **2a** **5**

Chemical Formula: $C_{15}H_{29}NO$
Exact Mass: 239.2249
Molecular Weight: 239.3969

Spectra of GC-MS



MS spectra of the peak at 9.155min



[MS Spectrum]
\# of Peaks399
Raw Spectrum 9.155 (scan : 1032)
Background No
Background Spectrum

Base Peakm/z 142.15 (Inten : 1,231,943)
Event# 1
m/z Absolute Intensity Relative Intensity

50.00	242	0.02
51.00	860	0.07
52.00	639	0.05
53.00	7292	0.59
54.05	5920	0.48
55.05	130942	10.63
56.05	44819	3.64

57.05	15049	1.22
58.05	30675	2.49
59.05	5865	0.48
60.05	1823	0.15
61.10	220	0.02
62.10	132	0.01
63.20	399	0.03
64.00	202	0.02
65.00	1444	0.12
66.05	670	0.05
67.00	13551	1.10
68.05	5939	0.48
69.05	69815	5.67
70.05	15448	1.25
71.05	5382	0.44
72.05	7840	0.64
73.05	5805	0.47
74.05	59610	4.84
75.05	2302	0.19
76.00	174	0.01
77.05	1569	0.13
78.10	703	0.06
79.05	4892	0.40
80.05	1324	0.11
81.05	16481	1.34
82.05	10609	0.86
83.05	35680	2.90
84.05	15883	1.29
85.05	5858	0.48
86.00	13225	1.07
87.05	1299	0.11
88.05	1345	0.11
89.00	135	0.01
89.90	27	0.00
90.95	1193	0.10
91.90	242	0.02
93.10	1983	0.16
94.10	1103	0.09
95.05	3479	0.28
96.05	4839	0.39
97.10	6441	0.52
98.10	6897	0.56
99.05	769	0.06
100.10	2428	0.20
101.10	114	0.01
102.10	150	0.01
103.10	70	0.01
104.10	36	0.00
105.05	613	0.05
106.10	114	0.01
107.15	1538	0.12
108.05	1220	0.10
109.10	89060	7.23
110.10	10090	0.82
111.10	1377	0.11
112.15	642	0.05
113.05	394	0.03
114.15	4667	0.38
115.05	450	0.04
116.10	135	0.01
117.10	122	0.01
118.10	30	0.00
119.10	161	0.01
120.10	81	0.01
121.25	480	0.04
122.15	638	0.05
123.15	14567	1.18
124.15	8236	0.67
125.15	3729	0.30
126.15	3931	0.32
127.10	527	0.04
128.10	340	0.03
129.10	159	0.01
130.10	52	0.00
131.10	89	0.01
132.10	90	0.01
133.10	116	0.01
134.10	74	0.01
135.10	522	0.04
136.10	300	0.02
137.10	74	0.01
138.15	788	0.06
139.20	329	0.03
140.15	4333	0.35
141.15	1869	0.15
142.15	1231943	100.00
143.15	111090	9.02
144.15	6873	0.56
145.10	759	0.06
146.10	71	0.01
147.10	94	0.01
148.10	76	0.01
149.10	82	0.01
150.10	24	0.00
151.10	19	0.00
152.10	21	0.00
153.10	34	0.00
154.10	52	0.00
155.10	178	0.01
156.15	11898	0.97
157.15	102844	8.35
158.15	10452	0.85
159.15	893	0.07
160.10	60	0.00
161.10	68	0.01
162.10	50	0.00
163.10	8	0.00
164.10	70	0.01
165.10	55	0.00
166.10	39	0.00
167.10	33	0.00
168.10	46	0.00
169.10	5	0.00
170.10	19	0.00
171.10	21	0.00
172.10	21	0.00
173.10	78	0.01
174.10	66	0.01
175.10	57	0.00
176.10	86	0.01
177.10	90	0.01
178.10	63	0.01
179.10	22	0.00
181.10	31	0.00
182.10	50	0.00
183.10	33	0.00
184.10	27	0.00
185.10	46	0.00
186.10	6	0.00
187.10	19	0.00
188.10	47	0.00
189.10	30	0.00

190.10	54	0.00	220.00	27	0.00	250.20	47	0.00
191.10	68	0.01	221.00	36	0.00	251.20	21	0.00
192.10	47	0.00	222.00	33	0.00	252.20	26	0.00
193.10	73	0.01	223.00	36	0.00	253.20	44	0.00
194.10	31	0.00	224.15	2057	0.17	254.20	38	0.00
195.10	50	0.00	225.30	564	0.05	255.20	27	0.00
196.10	42	0.00	226.30	30	0.00	256.20	26	0.00
197.10	27	0.00	227.30	33	0.00	257.20	24	0.00
198.10	24	0.00	228.30	36	0.00	258.20	49	0.00
199.10	36	0.00	229.30	58	0.00	259.20	14	0.00
200.10	24	0.00	230.30	39	0.00	260.20	26	0.00
201.10	41	0.00	231.30	11	0.00	261.20	31	0.00
202.10	65	0.01	232.30	34	0.00	262.20	22	0.00
203.10	22	0.00	233.30	70	0.01	263.20	36	0.00
204.10	65	0.01	234.30	27	0.00	264.20	31	0.00
205.10	30	0.00	235.30	30	0.00	265.20	50	0.00
206.10	24	0.00	236.30	30	0.00	266.20	44	0.00
207.00	431	0.03	237.30	22	0.00	267.20	63	0.01
208.00	70	0.01	**238.20**	**33**	**0.00**	268.20	36	0.00
209.00	79	0.01	**239.20**	**6830**	**0.55**	269.20	49	0.00
210.00	22	0.00	**240.20**	**1167**	**0.09**	270.20	33	0.00
211.00	47	0.00	**241.20**	**143**	**0.01**	271.20	42	0.00
212.00	34	0.00	242.20	71	0.01	272.20	29	0.00
213.00	33	0.00	243.20	36	0.00	273.20	54	0.00
214.00	24	0.00	244.20	29	0.00	275.20	26	0.00
215.00	29	0.00	245.20	33	0.00	276.20	29	0.00
216.00	24	0.00	246.20	21	0.00	277.20	47	0.00
217.00	74	0.01	247.20	46	0.00	278.20	8	0.00
218.00	42	0.00	248.20	18	0.00	279.20	31	0.00
219.00	118	0.01	249.20	16	0.00	280.20	14	0.00

3. References

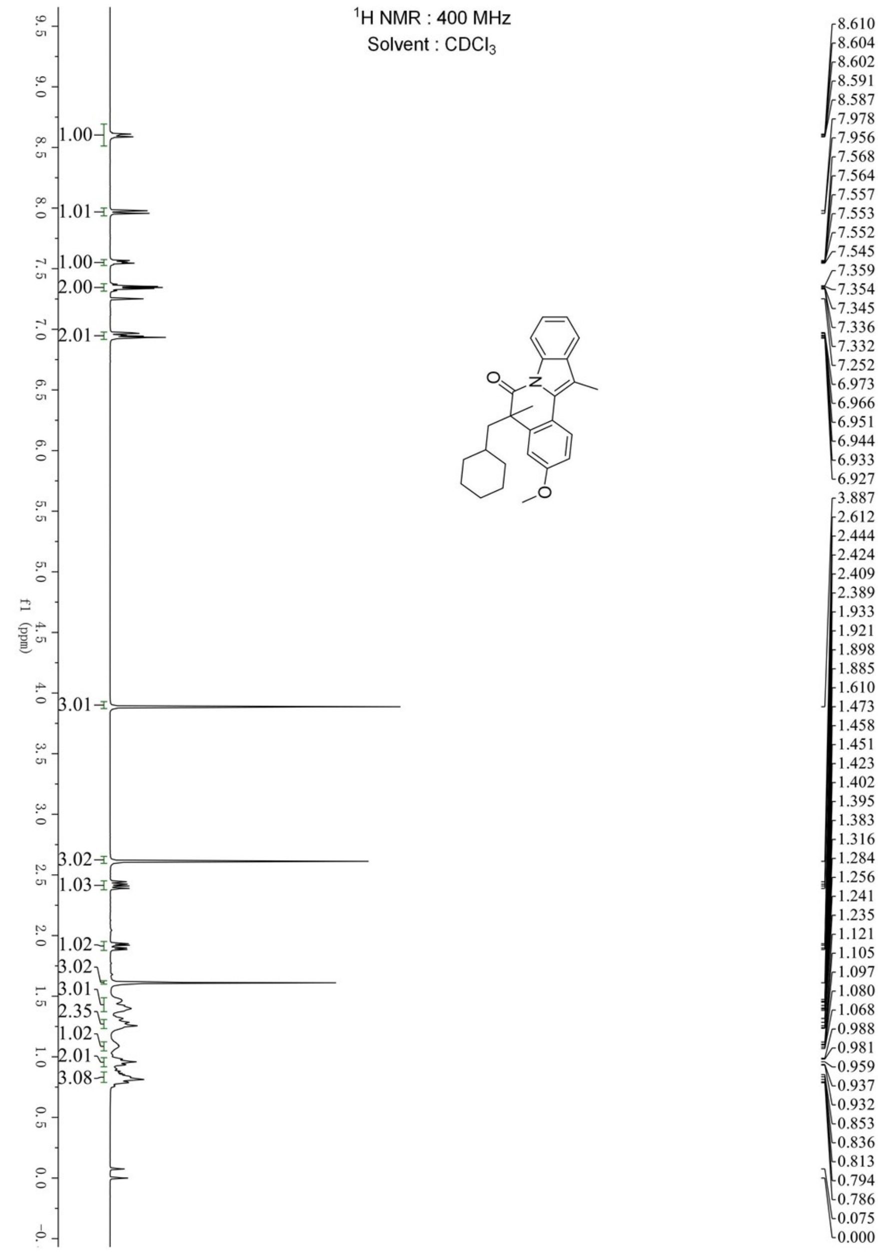
[1] Wei,Y.-L.; Chen, J. Q.; Sun, B.; Xu, P. F. *Chem. Commun*. **2019**, *55*, 5922.

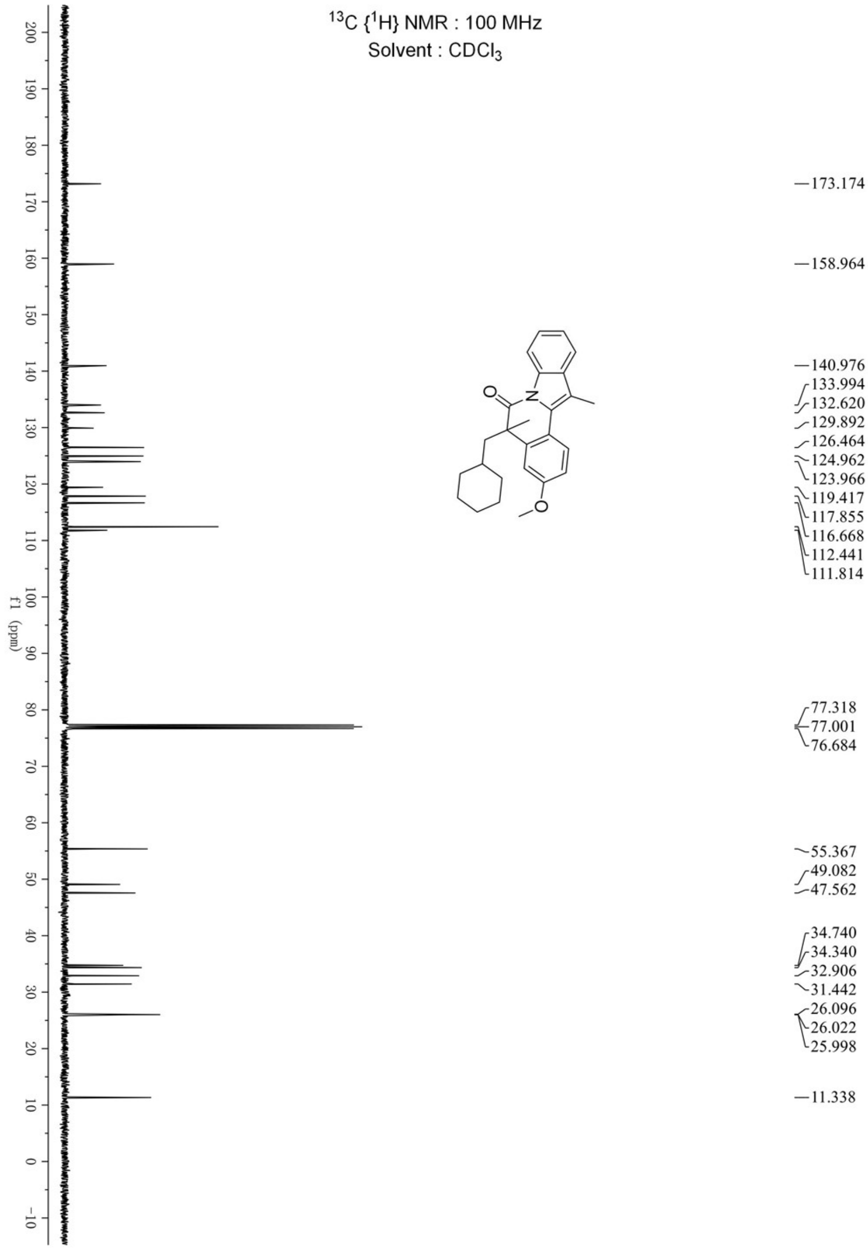
[2] Gu, F.; Huang, W.; Liu, X. Chen W.; Cheng, X. *Adv. Synth. Catal.*, **2018**, *360*, 925.

[3] Wu, Q. Y.; Min, Q. Q.; Ao, G. Z.; Liu, F. *Org. Biomol. Chem.,* **2018**, *16*, 6391.

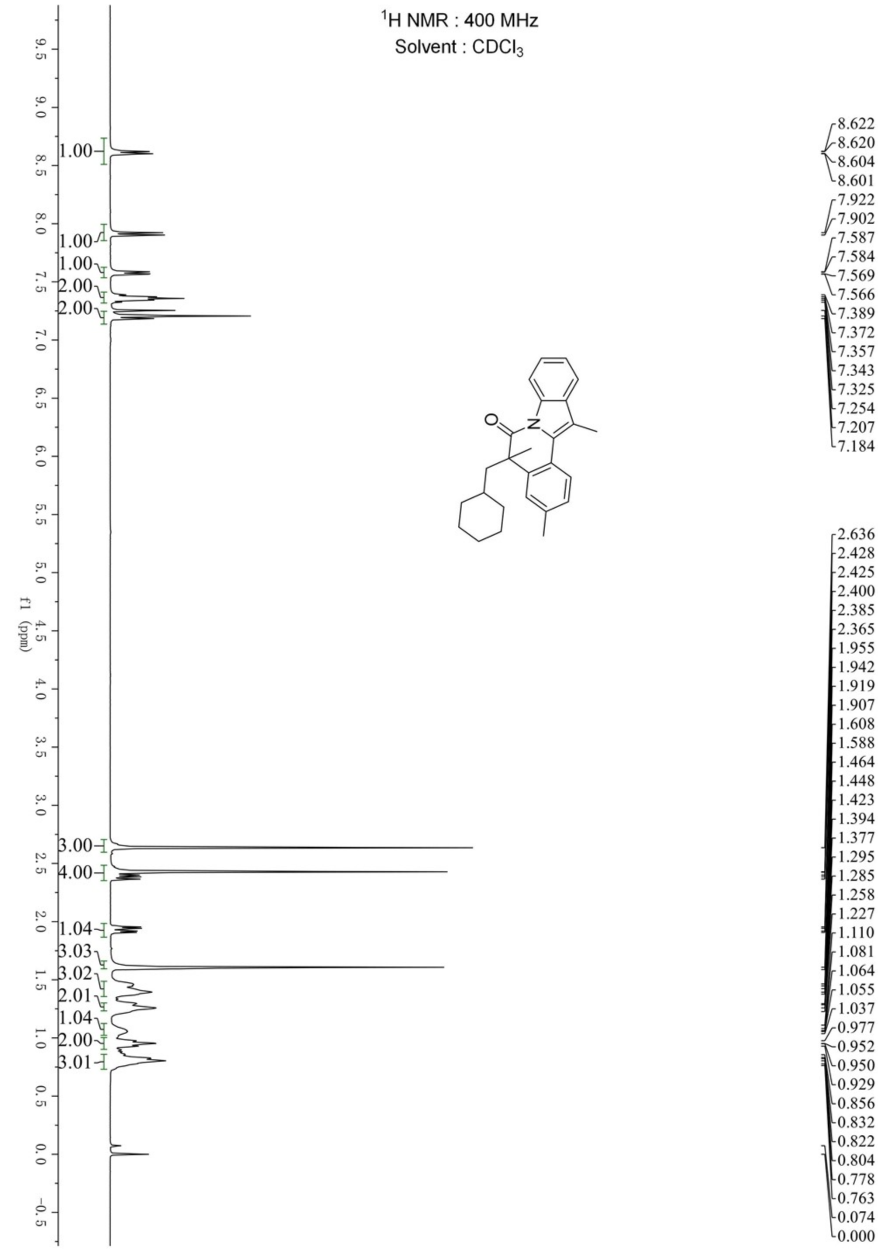
4. ^{1}H and ^{13}C spectra

5-(Cyclohexy

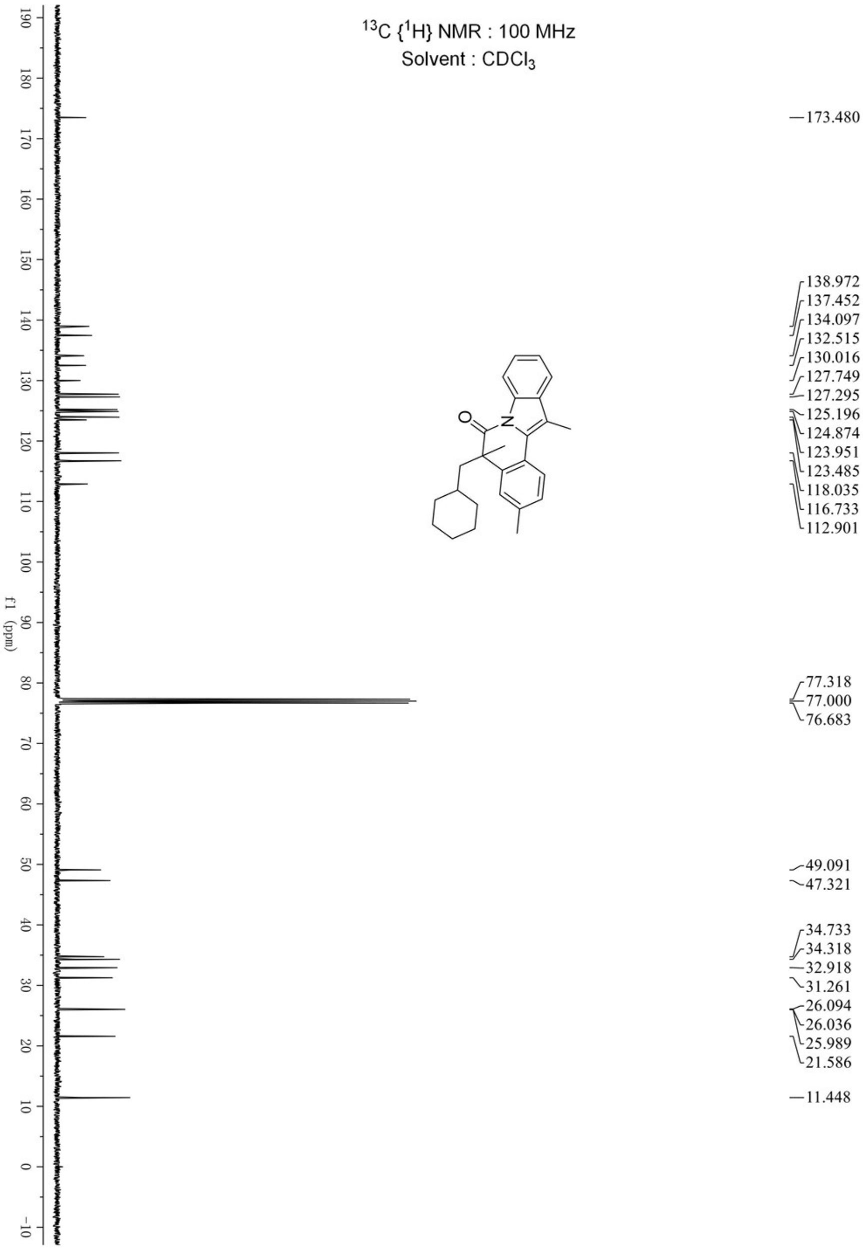



$^{13}C\ \{^1H\}$ NMR : 100 MHz
Solvent : $CDCl_3$
173.174
158.964
140.976
133.994
132.620
129.892
126.464
124.962
123.966
119.417
117.855
116.668
112.441
111.814
77.318
77.001
76.684
55.367
49.082
47.562
34.740
34.340
32.906
31.442
26.096
26.022
25.998
11.338
f1 (ppm)

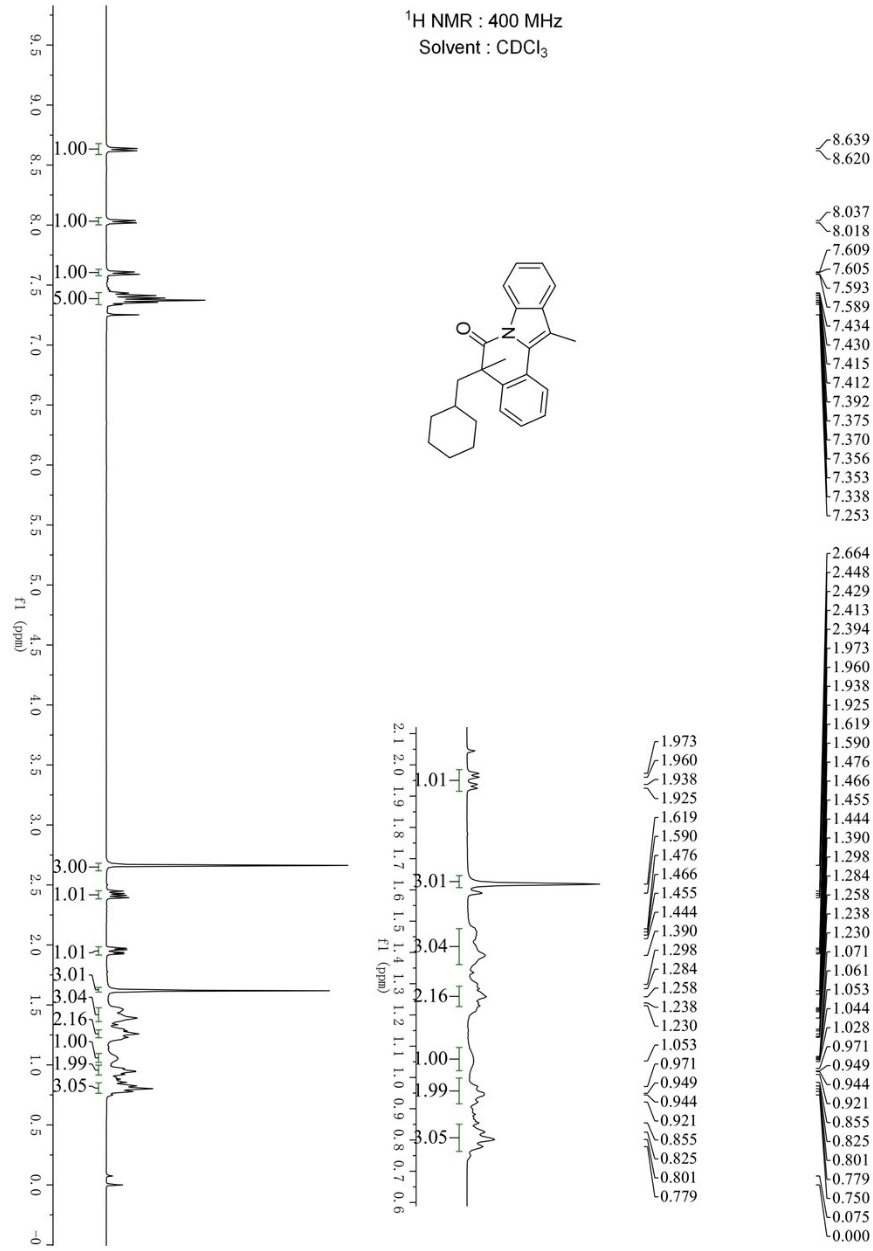
5-(Cyclohexy

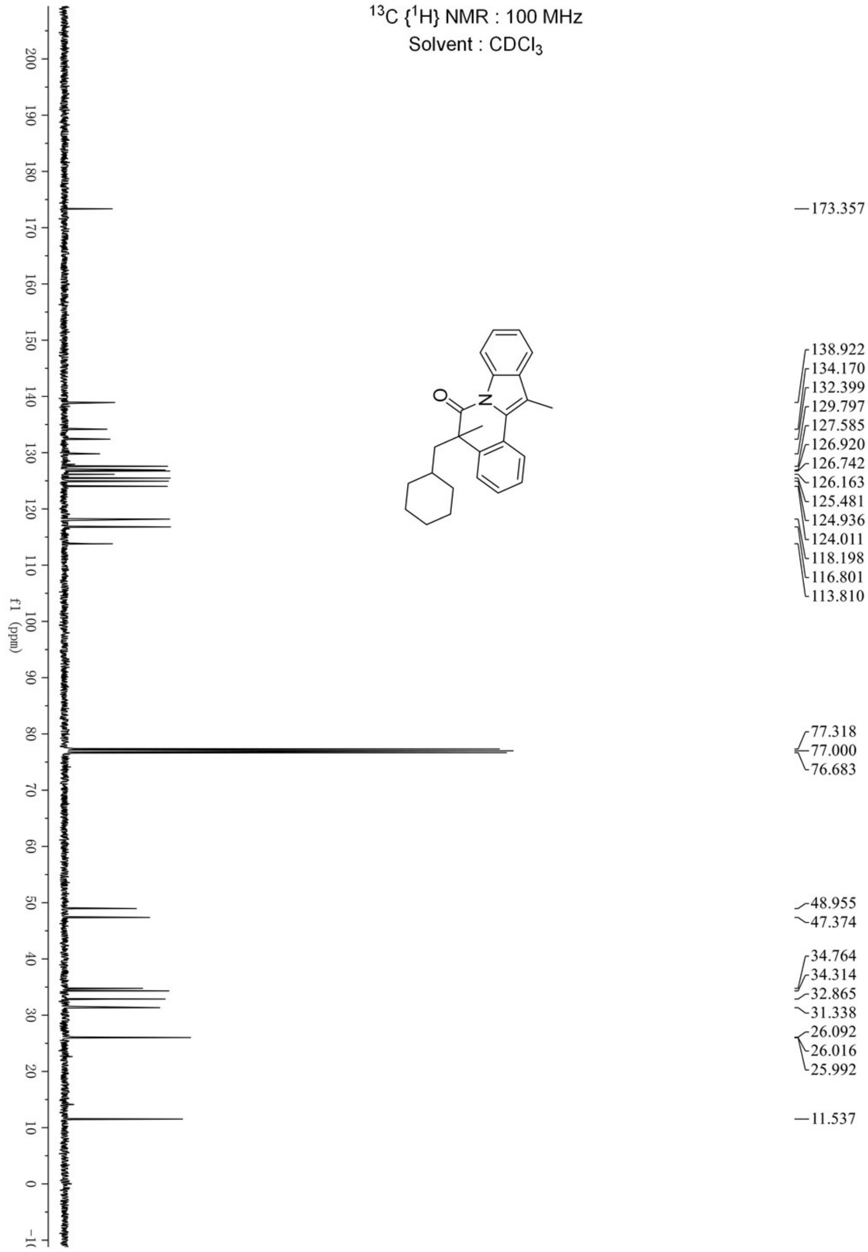


1H NMR : 400 MHz
Solvent : CDCl3
8.622
8.620
8.604
8.601
7.922
7.902
7.587
7.584
7.569
7.566
7.389
7.372
7.357
7.343
7.325
7.254
7.207
7.184
2.636
2.428
2.425
2.400
2.385
2.365
1.955
1.942
1.919
1.907
1.608
1.588
1.464
1.448
1.423
1.394
1.377
1.295
1.285
1.258
1.227
1.110
1.081
1.064
1.055
1.037
0.977
0.952
0.950
0.929
0.856
0.832
0.822
0.804
0.778
0.763
0.074
0.000

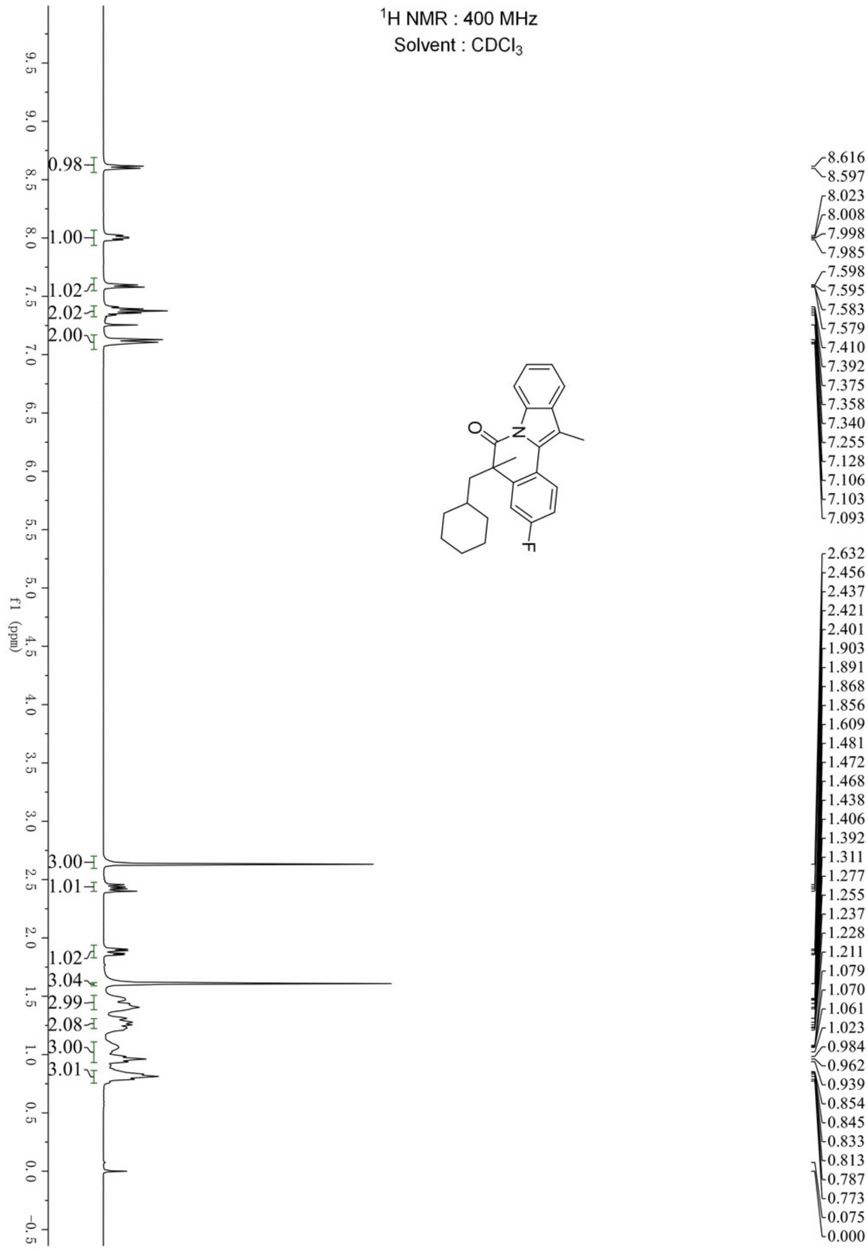

13C {1H} NMR : 100 MHz
Solvent : CDCl3
173.480
138.972
137.452
134.097
132.515
130.016
127.749
127.295
125.196
124.874
123.951
123.485
118.035
116.733
112.901
77.318
77.000
76.683
49.091
47.321
34.733
34.318
32.918
31.261
26.094
26.036
25.989
21.586
11.448
f1 (ppm)
N
O

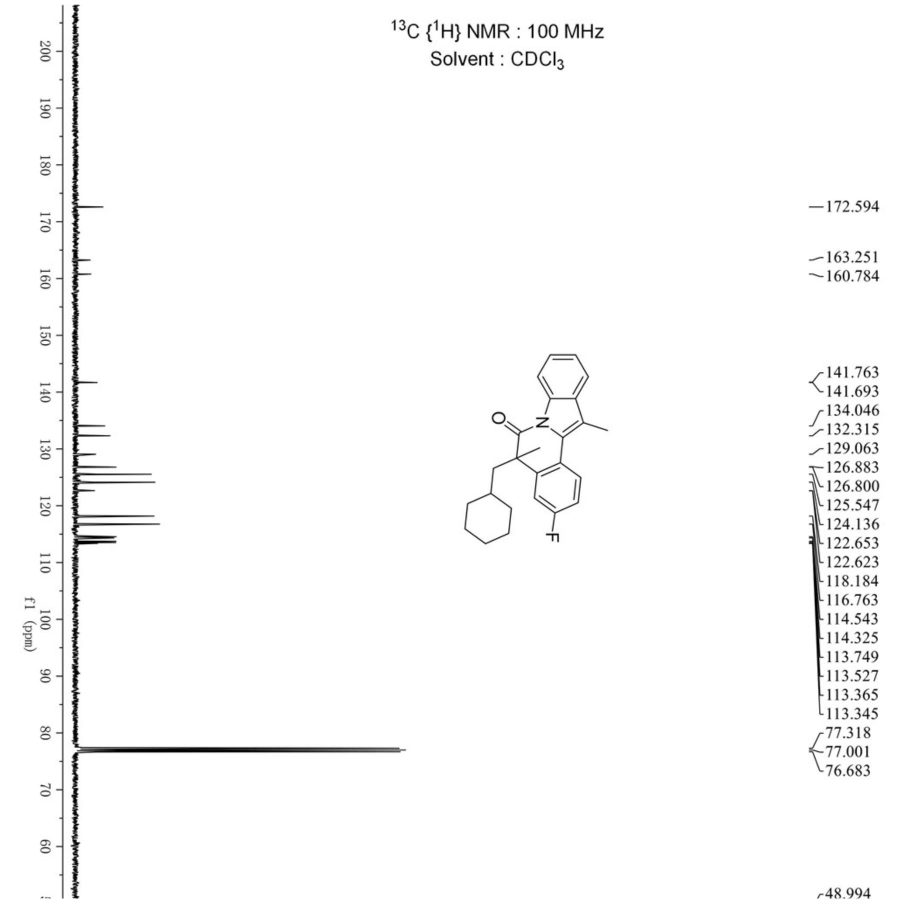
5-(Cyclohe

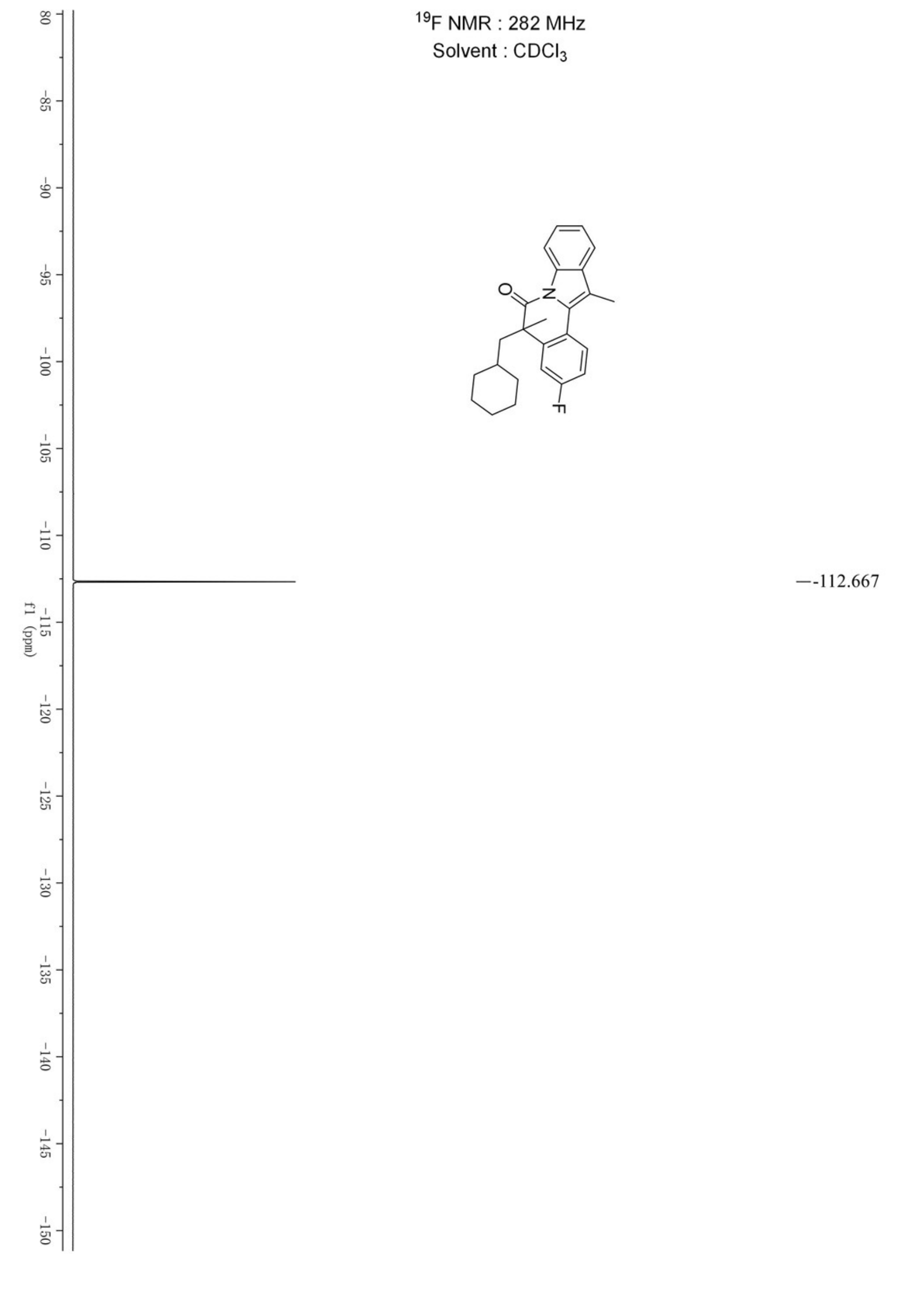



$^{13}C\ \{^1H\}$ NMR : 100 MHz
Solvent : $CDCl_3$
173.357
138.922
134.170
132.399
129.797
127.585
126.920
126.742
126.163
125.481
124.936
124.011
118.198
116.801
113.810
77.318
77.000
76.683
48.955
47.374
34.764
34.314
32.865
31.338
26.092
26.016
25.992
11.537
f1 (ppm)

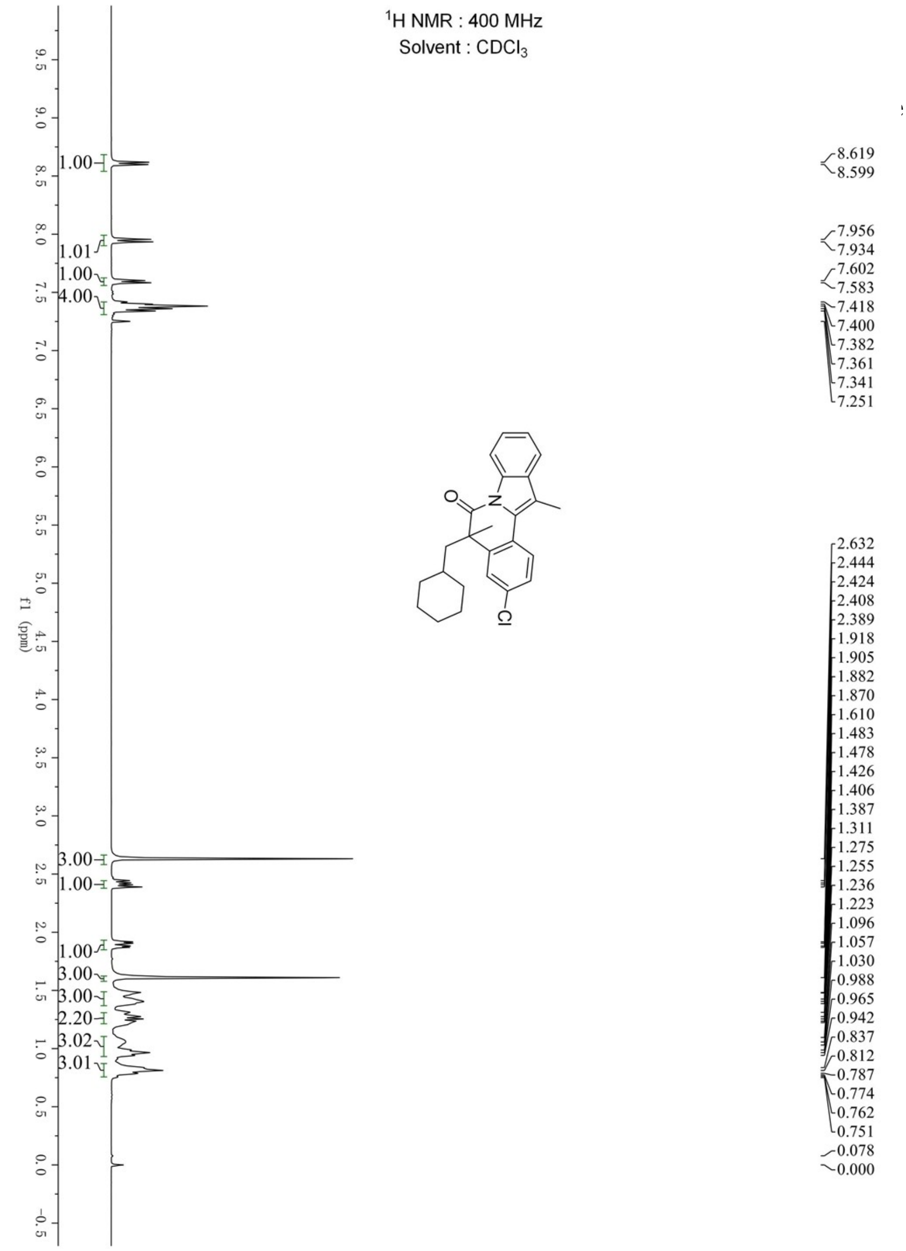
5-(Cyclohexy


^{1}H NMR : 400 MHz
Solvent : CDCl$_3$
f1 (ppm)
0.98
1.00
1.02
2.02
2.00
3.00
1.01
1.02
3.04
2.99
2.08
3.00
3.01
8.616
8.597
8.023
8.008
7.998
7.985
7.598
7.595
7.583
7.579
7.410
7.392
7.375
7.358
7.340
7.255
7.128
7.106
7.103
7.093
2.632
2.456
2.437
2.421
2.401
1.903
1.891
1.868
1.856
1.609
1.481
1.472
1.468
1.438
1.406
1.392
1.311
1.277
1.255
1.237
1.228
1.211
1.079
1.070
1.061
1.023
0.984
0.962
0.939
0.854
0.845
0.833
0.813
0.787
0.773
0.075
0.000

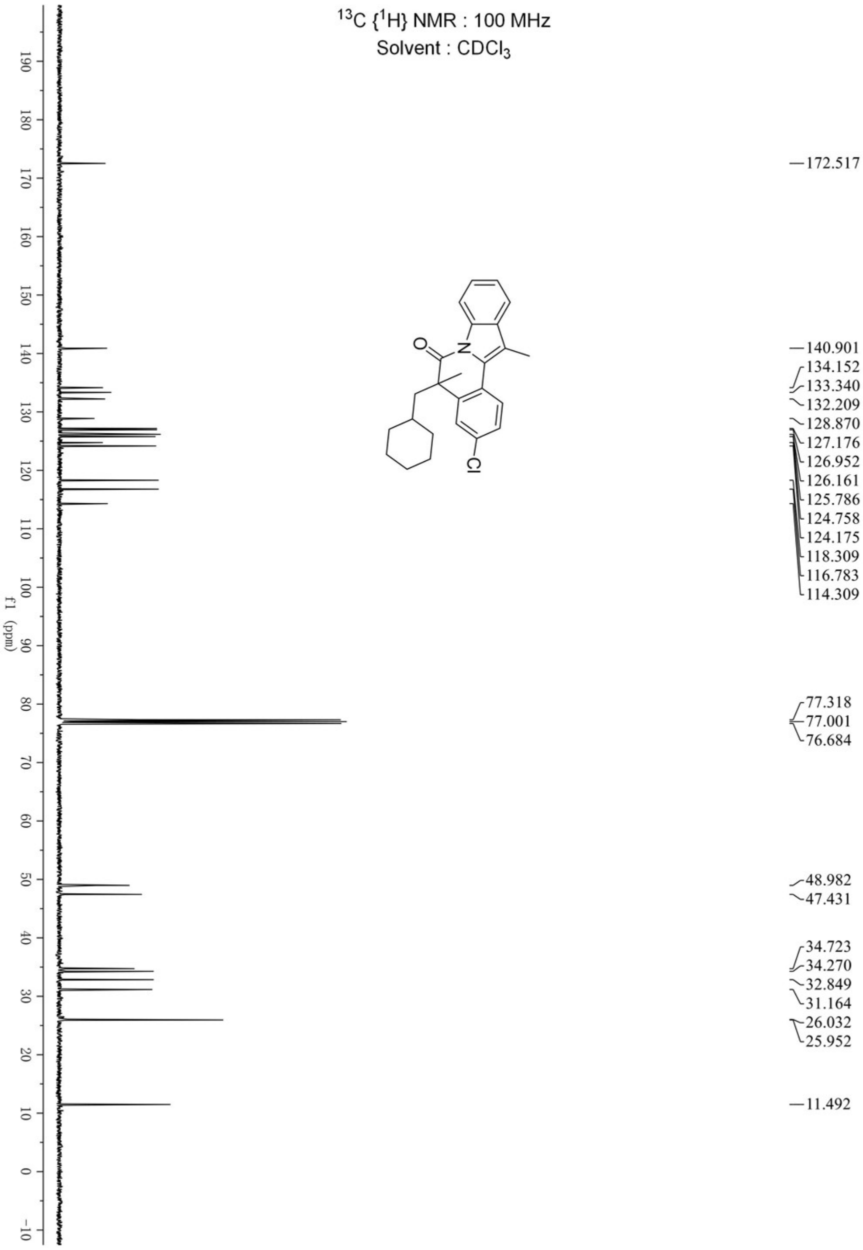

^{13}C {^{1}H} NMR : 100 MHz
Solvent : $CDCl_3$
172.594
163.251
160.784
141.763
141.693
134.046
132.315
129.063
126.883
126.800
125.547
124.136
122.653
122.623
118.184
116.763
114.543
114.325
113.749
113.527
113.365
113.345
77.318
77.001
76.683
48.994
f1 (ppm)
O
N
F


^{19}F NMR : 282 MHz
Solvent : $CDCl_3$
-112.667
f1 (ppm)
O
N
F

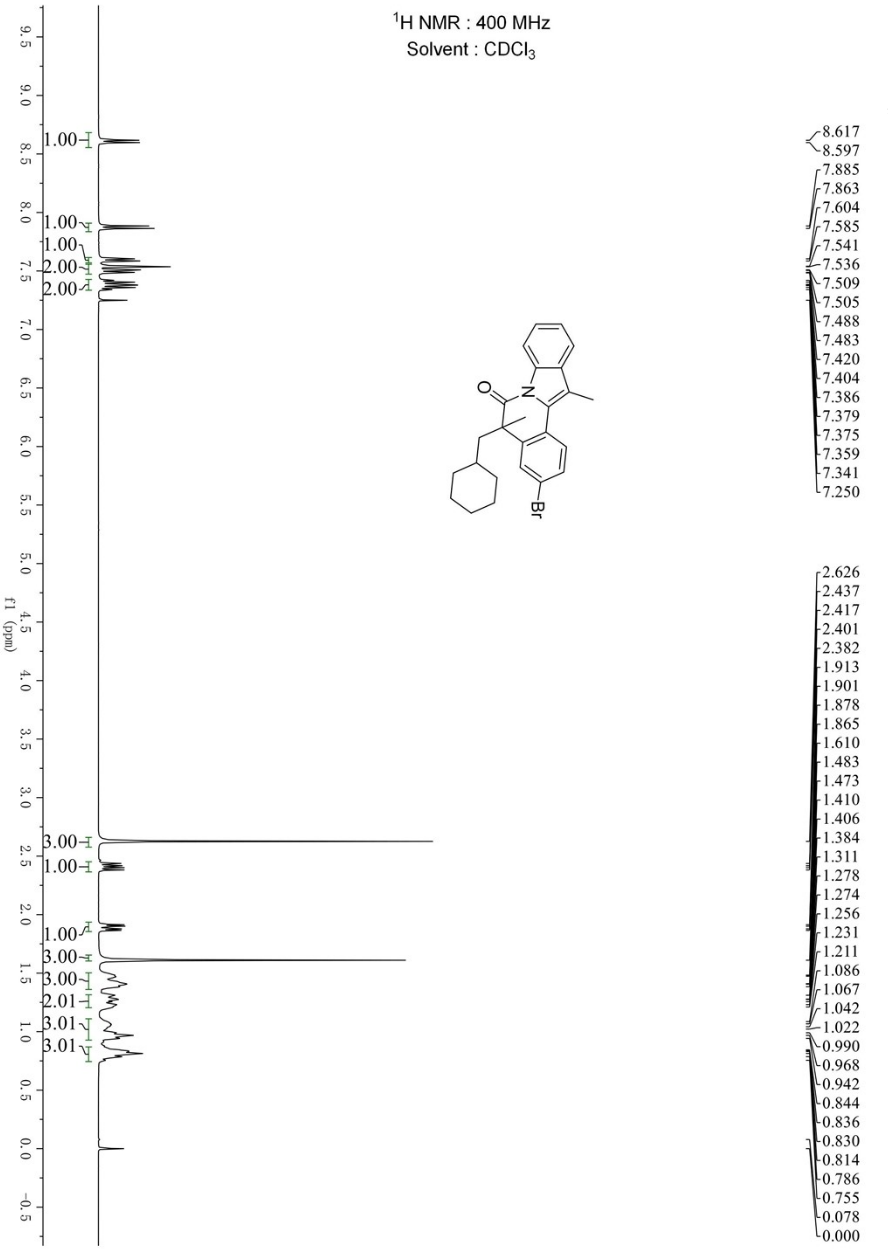
3-Chloro-5-(



1H NMR : 400 MHz
Solvent : CDCl3
8.619
8.599
7.956
7.934
7.602
7.583
7.418
7.400
7.382
7.361
7.341
7.251
2.632
2.444
2.424
2.408
2.389
1.918
1.905
1.882
1.870
1.610
1.483
1.478
1.426
1.406
1.387
1.311
1.275
1.255
1.236
1.223
1.096
1.057
1.030
0.988
0.965
0.942
0.837
0.812
0.787
0.774
0.762
0.751
0.078
0.000
f1 (ppm)

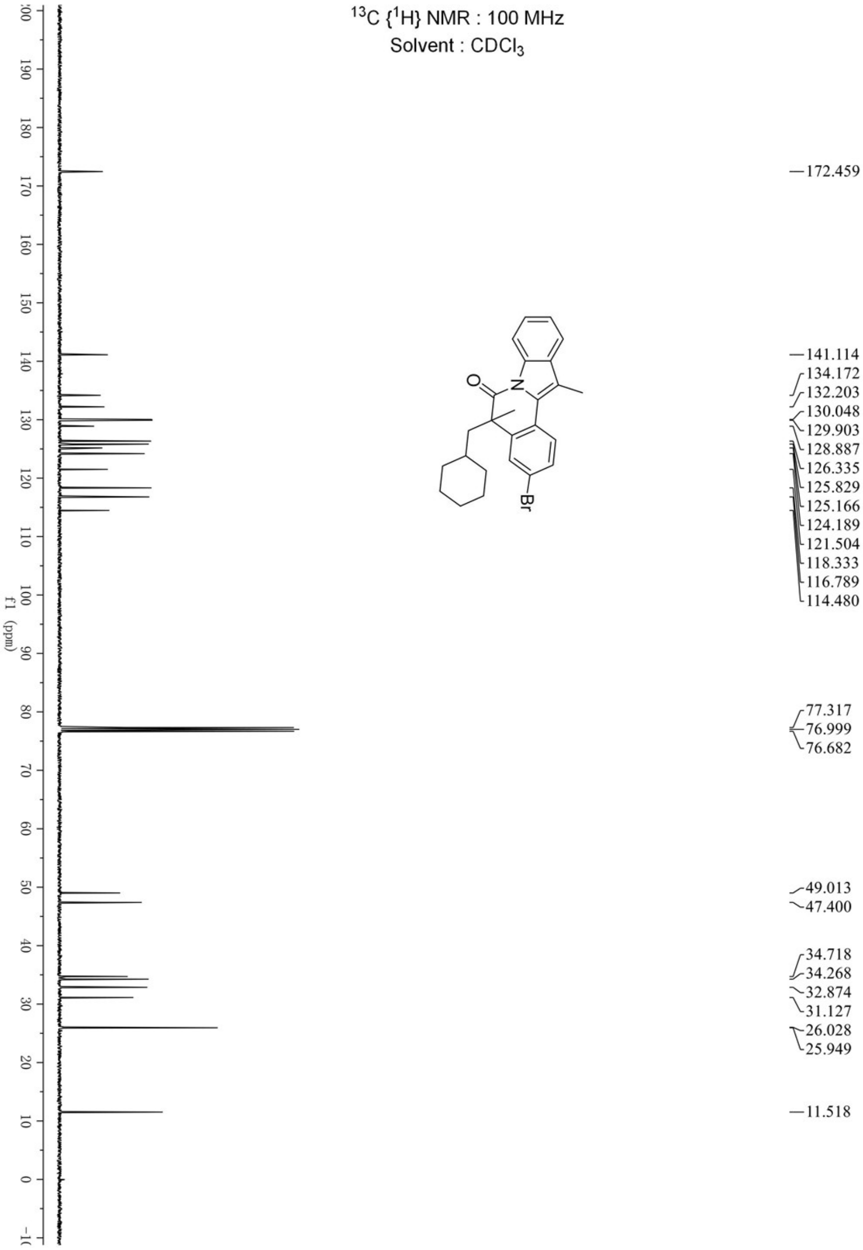

$^{13}C\ \{^1H\}$ NMR : 100 MHz
Solvent : $CDCl_3$
172.517
140.901
134.152
133.340
132.209
128.870
127.176
126.952
126.161
125.786
124.758
124.175
118.309
116.783
114.309
77.318
77.001
76.684
48.982
47.431
34.723
34.270
32.849
31.164
26.032
25.952
11.492
f1 (ppm)
N
O
Cl

3-Bromo-5-(

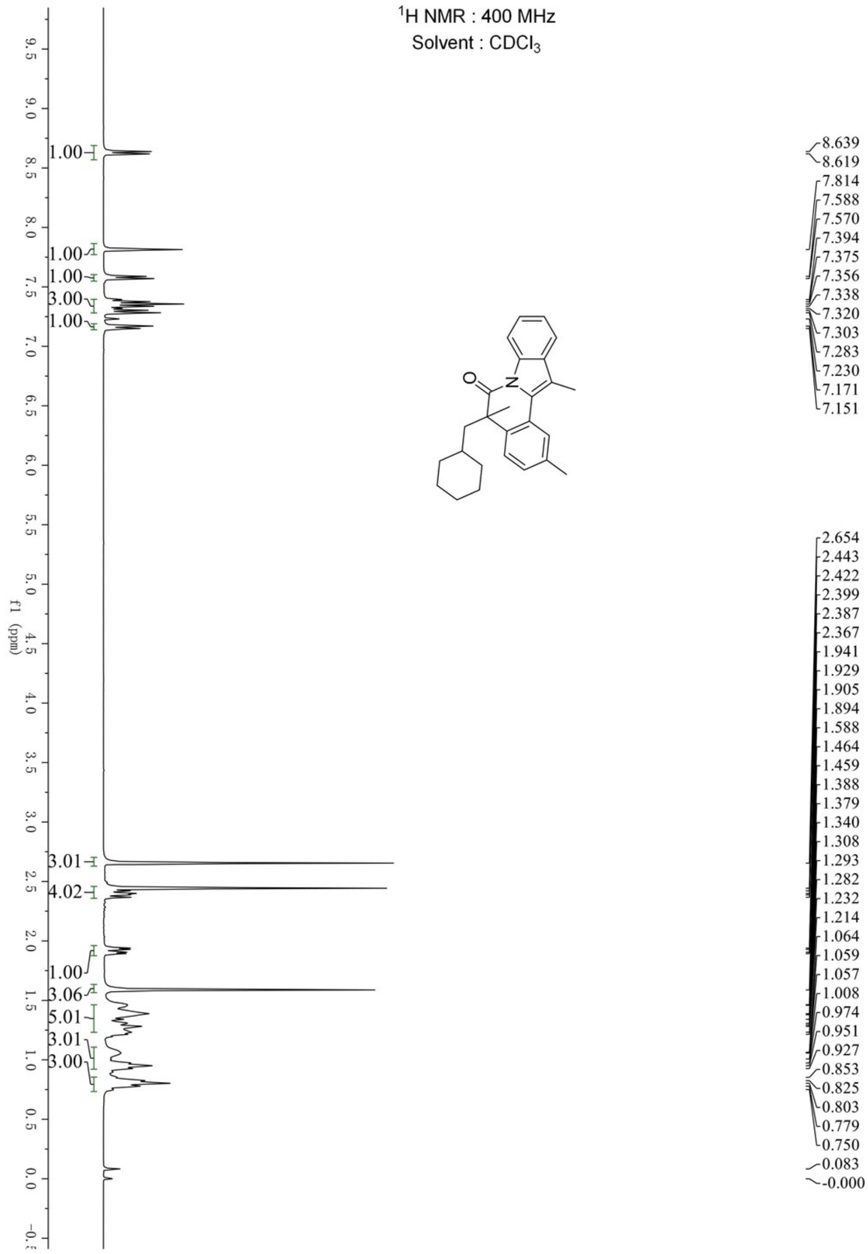


1H NMR : 400 MHz
Solvent : CDCl3
f1 (ppm)
O
N
Br
8.617
8.597
7.885
7.863
7.604
7.585
7.541
7.536
7.509
7.505
7.488
7.483
7.420
7.404
7.386
7.379
7.375
7.359
7.341
7.250
2.626
2.437
2.417
2.401
2.382
1.913
1.901
1.878
1.865
1.610
1.483
1.473
1.410
1.406
1.384
1.311
1.278
1.274
1.256
1.231
1.211
1.086
1.067
1.042
1.022
0.990
0.968
0.942
0.844
0.836
0.830
0.814
0.786
0.755
0.078
0.000

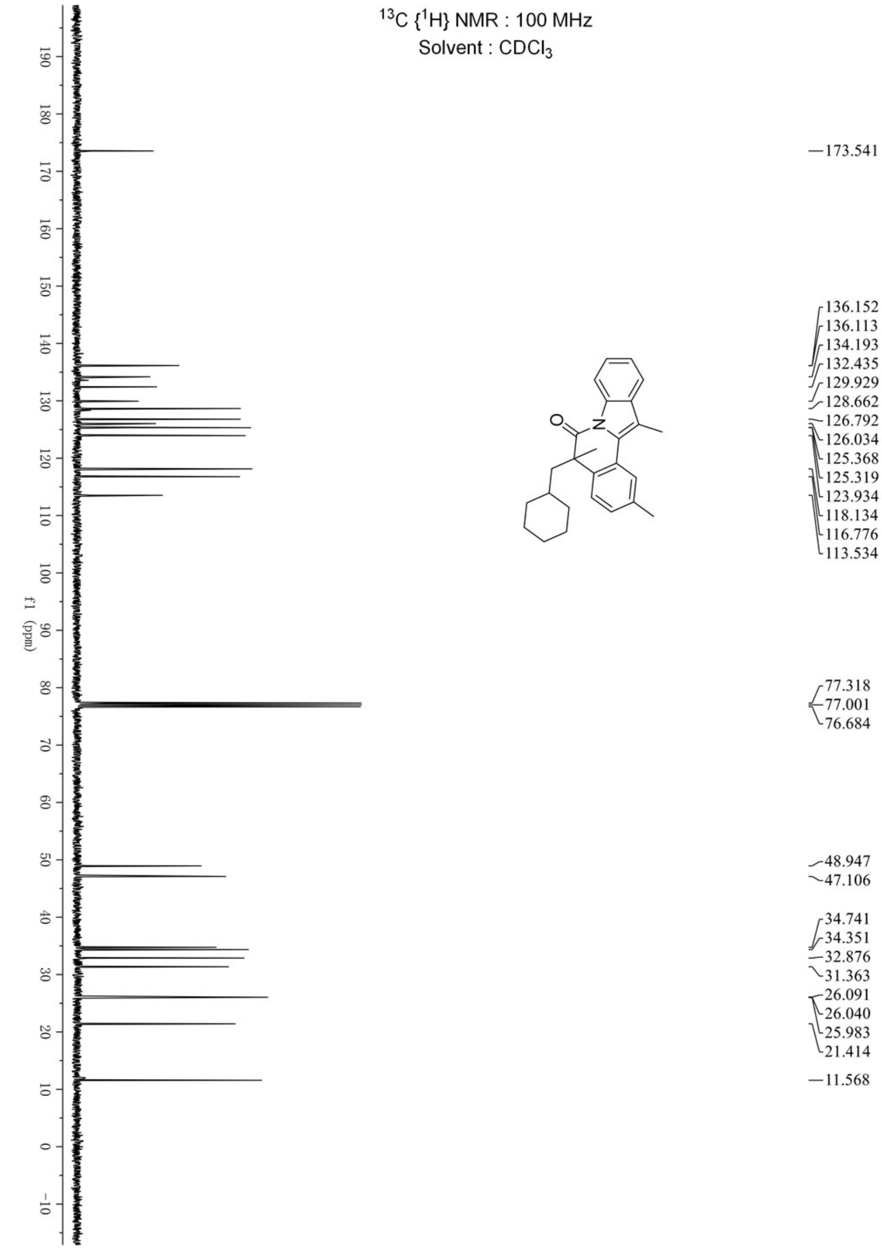
^{13}C {^{1}H} NMR : 100 MHz
Solvent : $CDCl_3$



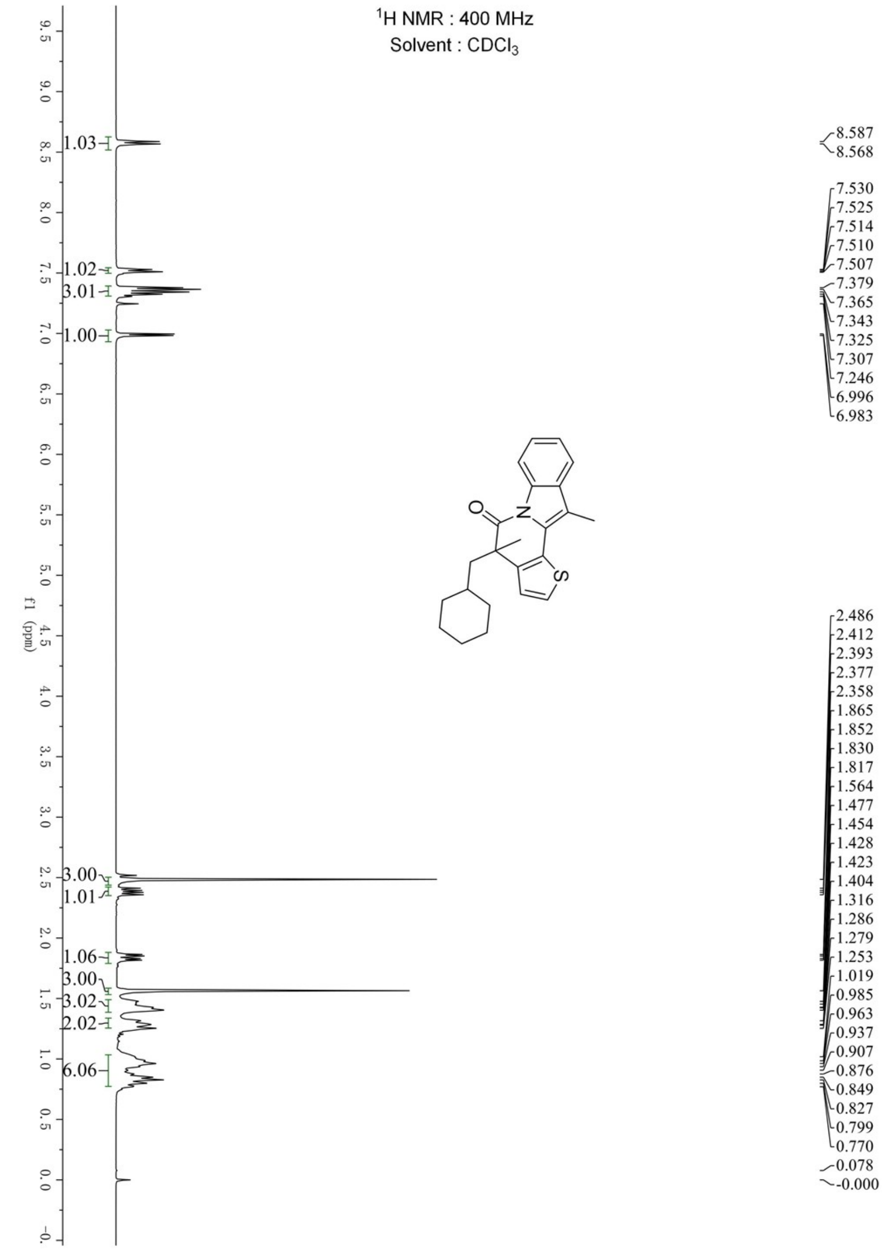
5-(Cyclohexy

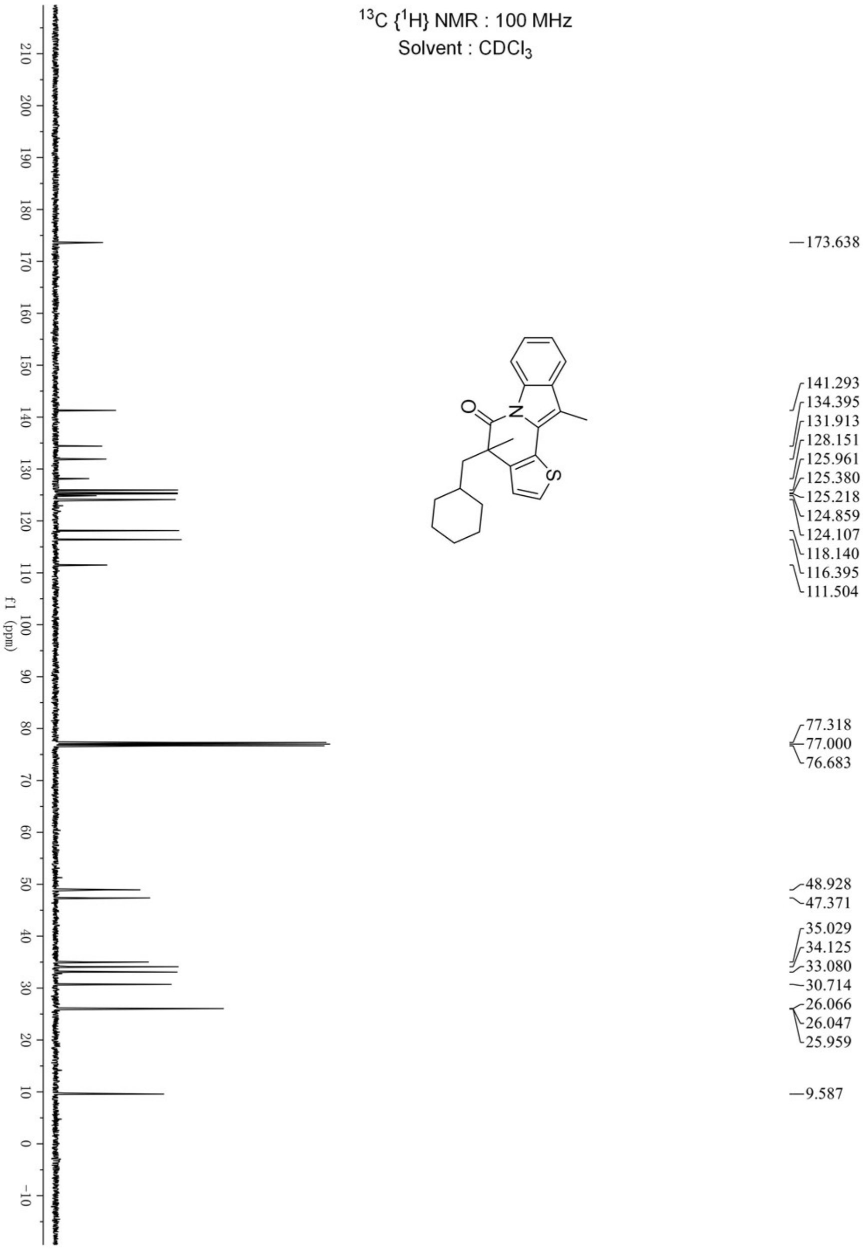


^{1}H NMR : 400 MHz
Solvent : $CDCl_3$
f1 (ppm)
8.639
8.619
7.814
7.588
7.570
7.394
7.375
7.356
7.338
7.320
7.303
7.283
7.230
7.171
7.151
2.654
2.443
2.422
2.399
2.387
2.367
1.941
1.929
1.905
1.894
1.588
1.464
1.459
1.388
1.379
1.340
1.308
1.293
1.282
1.232
1.214
1.064
1.059
1.057
1.008
0.974
0.951
0.927
0.853
0.825
0.803
0.779
0.750
0.083
-0.000

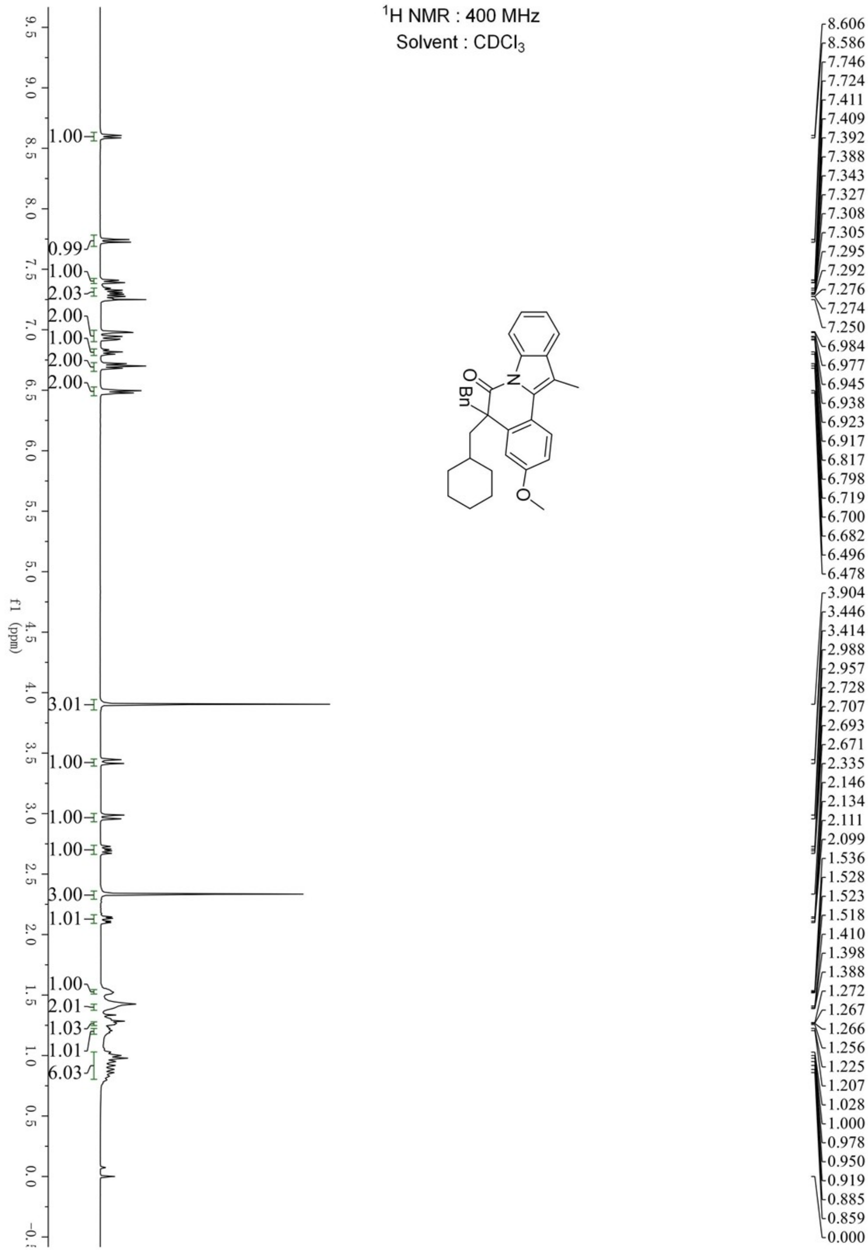

$^{13}C\ \{^{1}H\}$ NMR : 100 MHz
Solvent : $CDCl_3$
173.541
136.152
136.113
134.193
132.435
129.929
128.662
126.792
126.034
125.368
125.319
123.934
118.134
116.776
113.534
77.318
77.001
76.684
48.947
47.106
34.741
34.351
32.876
31.363
26.091
26.040
25.983
21.414
11.568
f1 (ppm)

4-(Cyclohexyl ... **e**

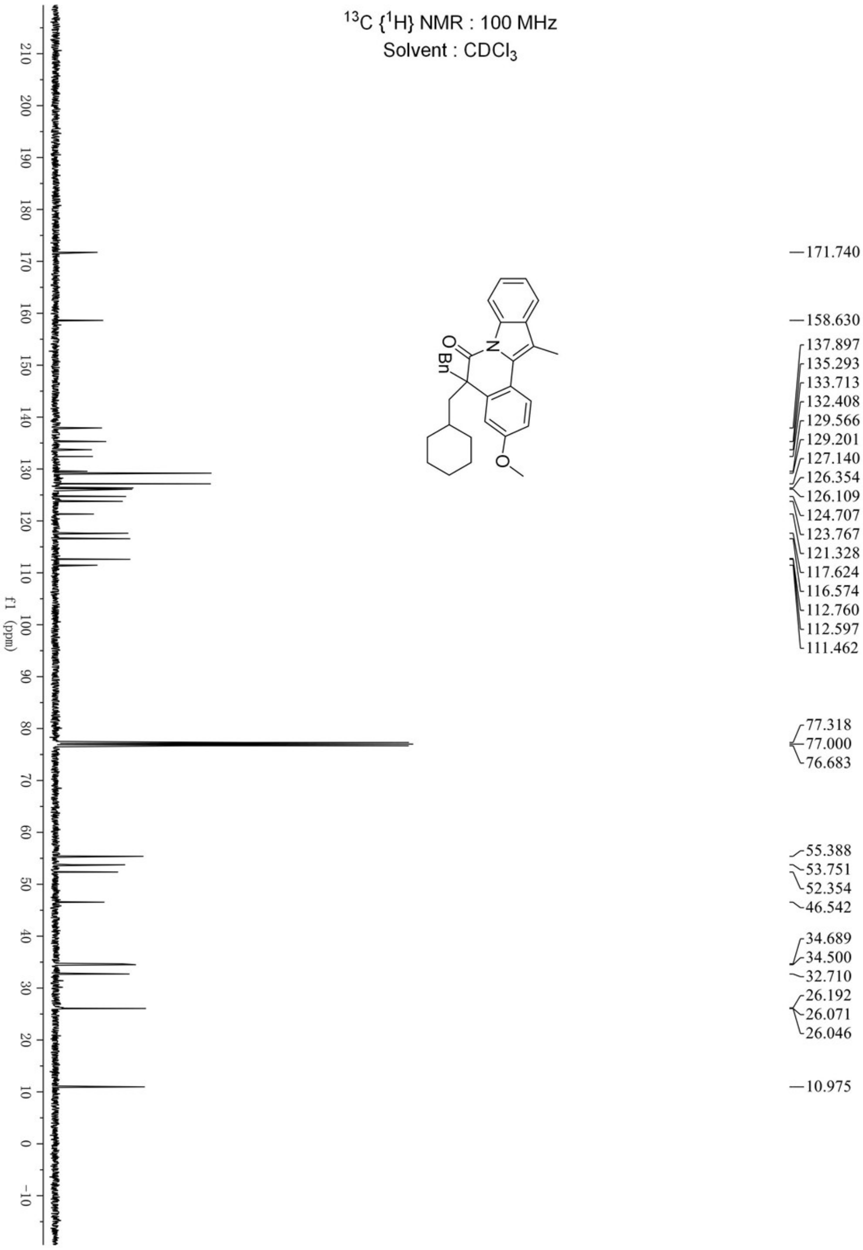



$^{13}C\ \{^{1}H\}$ NMR : 100 MHz
Solvent : $CDCl_3$
173.638
141.293
134.395
131.913
128.151
125.961
125.380
125.218
124.859
124.107
118.140
116.395
111.504
77.318
77.000
76.683
48.928
47.371
35.029
34.125
33.080
30.714
26.066
26.047
25.959
9.587
f1 (ppm)
O
N
S

5-Benzyl-5-

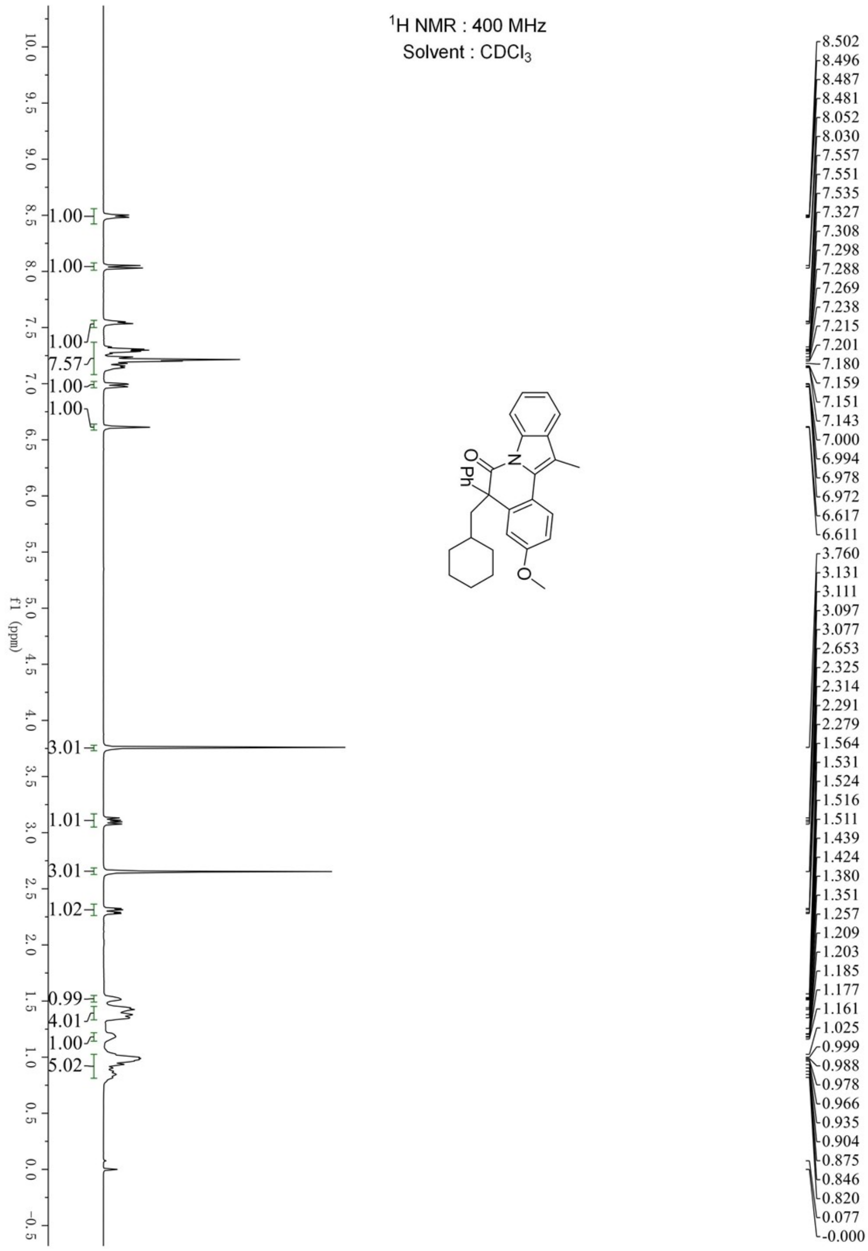


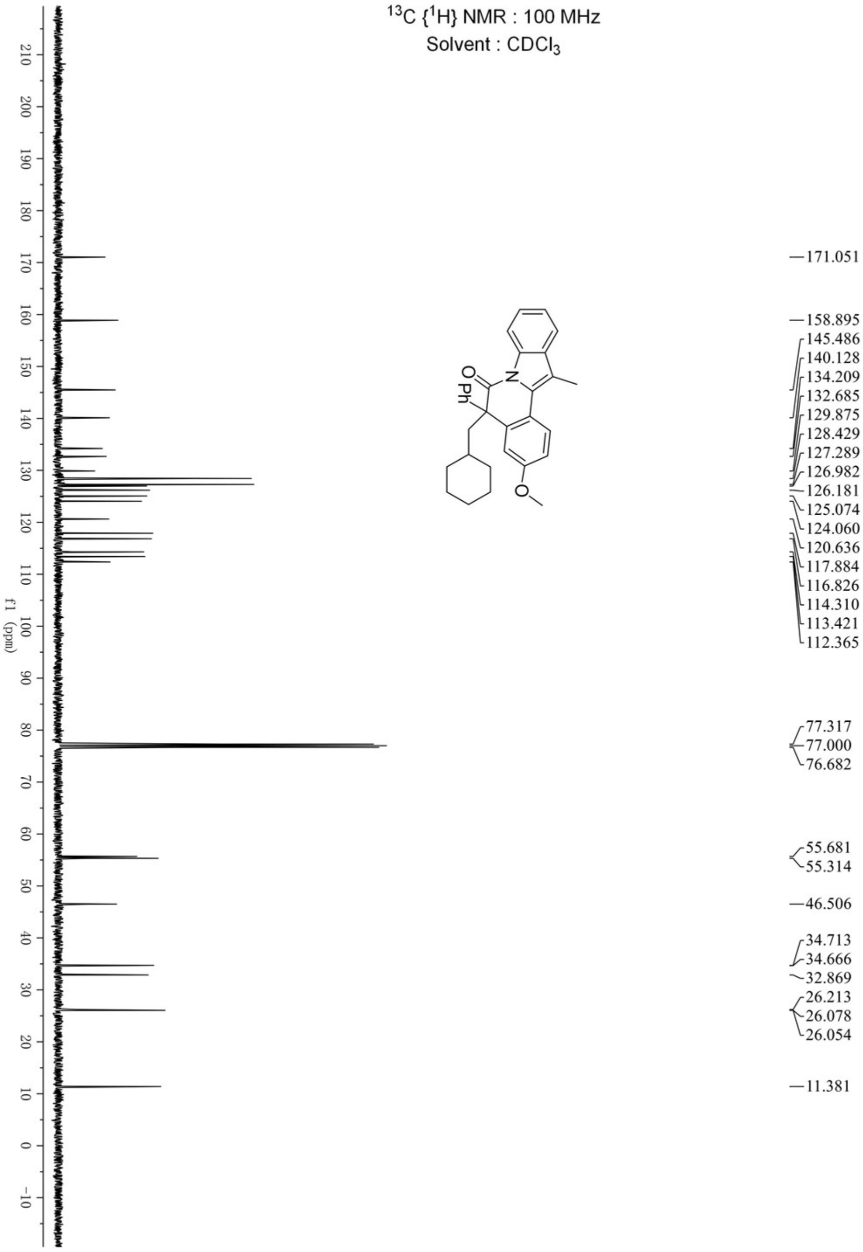
^{1}H NMR : 400 MHz
Solvent : $CDCl_3$
OBn
8.606
8.586
7.746
7.724
7.411
7.409
7.392
7.388
7.343
7.327
7.308
7.305
7.295
7.292
7.276
7.274
7.250
6.984
6.977
6.945
6.938
6.923
6.917
6.817
6.798
6.719
6.700
6.682
6.496
6.478
3.904
3.446
3.414
2.988
2.957
2.728
2.707
2.693
2.671
2.335
2.146
2.134
2.111
2.099
1.536
1.528
1.523
1.518
1.410
1.398
1.388
1.272
1.267
1.266
1.256
1.225
1.207
1.028
1.000
0.978
0.950
0.919
0.885
0.859
0.000
f1 (ppm)



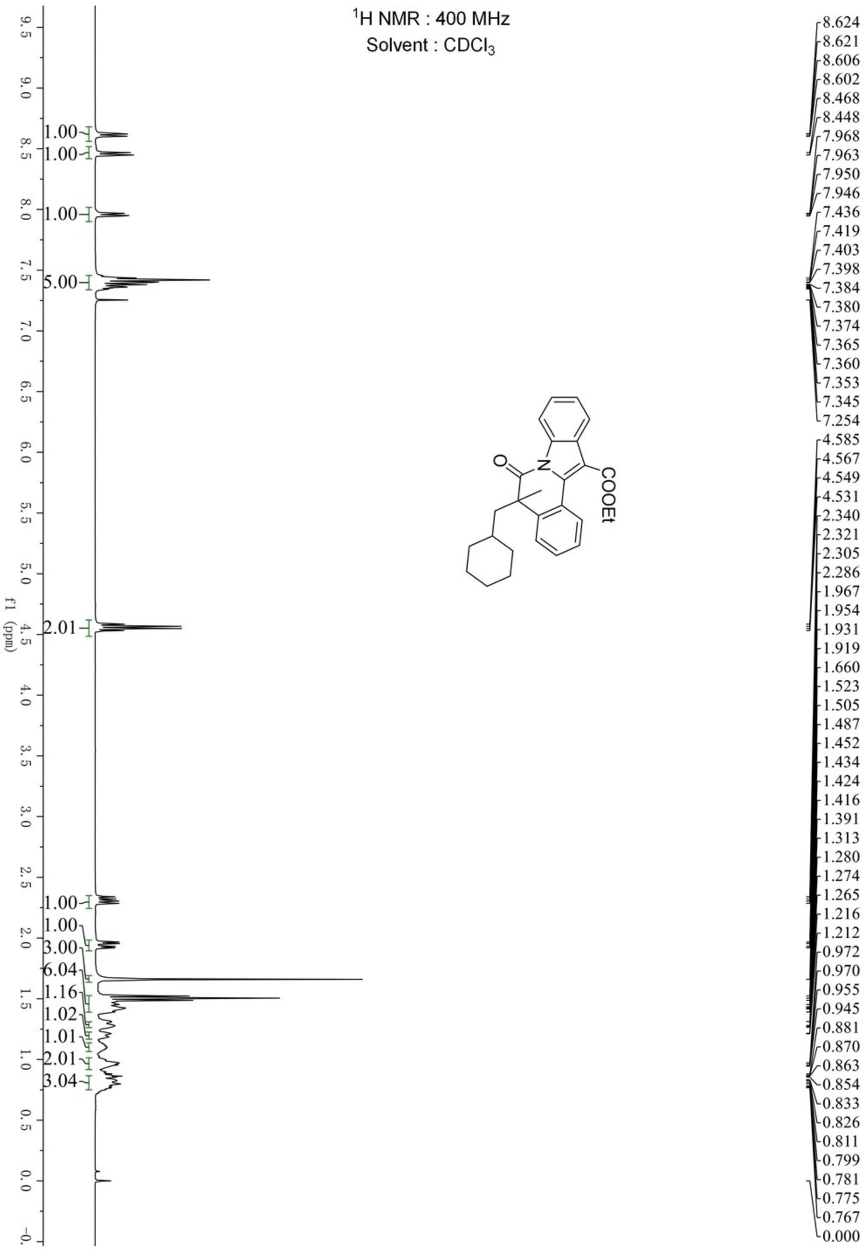
$^{13}C\ \{^1H\}$ NMR : 100 MHz
Solvent : $CDCl_3$
O
N
OBn
O
171.740
158.630
137.897
135.293
133.713
132.408
129.566
129.201
127.140
126.354
126.109
124.707
123.767
121.328
117.624
116.574
112.760
112.597
111.462
77.318
77.000
76.683
55.388
53.751
52.354
46.542
34.689
34.500
32.710
26.192
26.071
26.046
10.975
f1 (ppm)

5-(Cyclohex

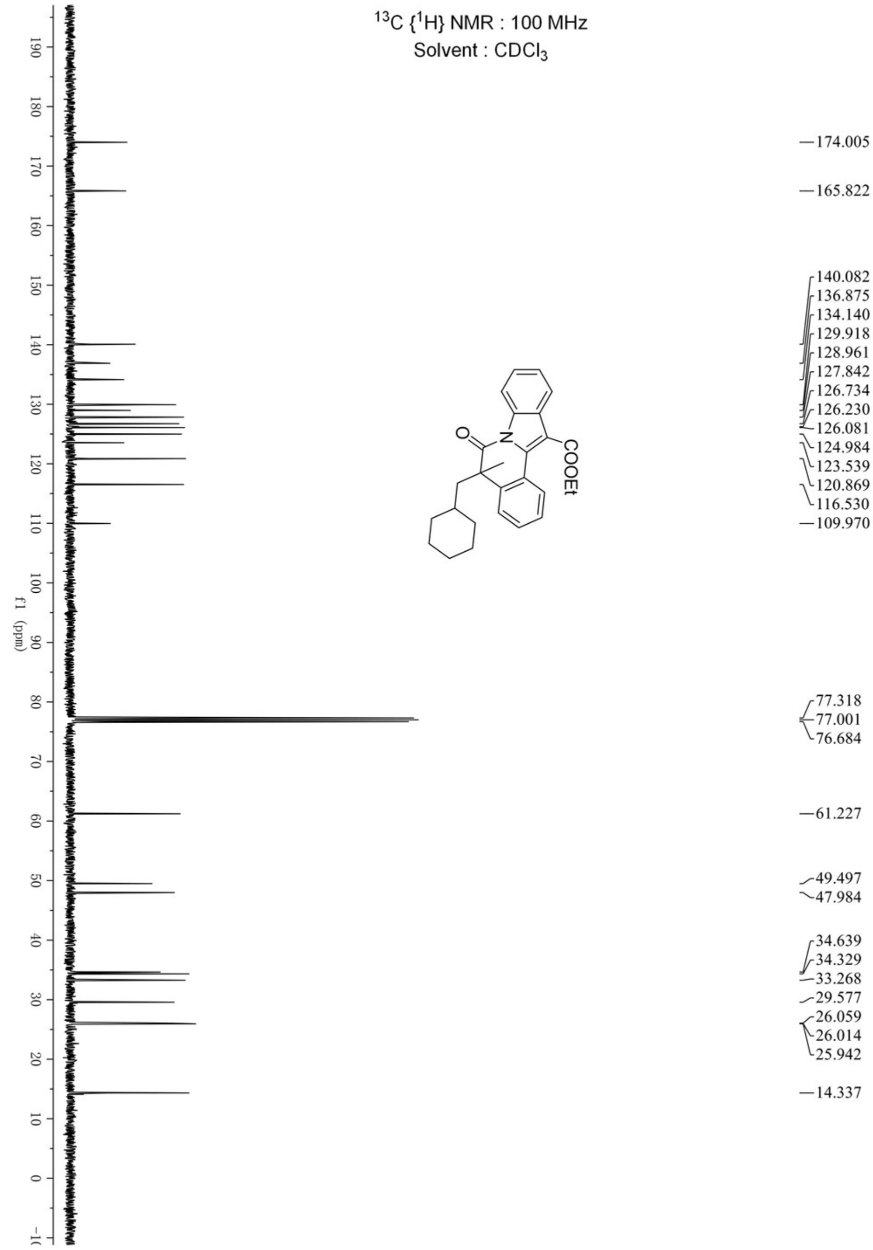



^{13}C {^1H} NMR : 100 MHz
Solvent : $CDCl_3$
171.051
158.895
145.486
140.128
134.209
132.685
129.875
128.429
127.289
126.982
126.181
125.074
124.060
120.636
117.884
116.826
114.310
113.421
112.365
77.317
77.000
76.682
55.681
55.314
46.506
34.713
34.666
32.869
26.213
26.078
26.054
11.381
f1 (ppm)
O
N
Ph
O

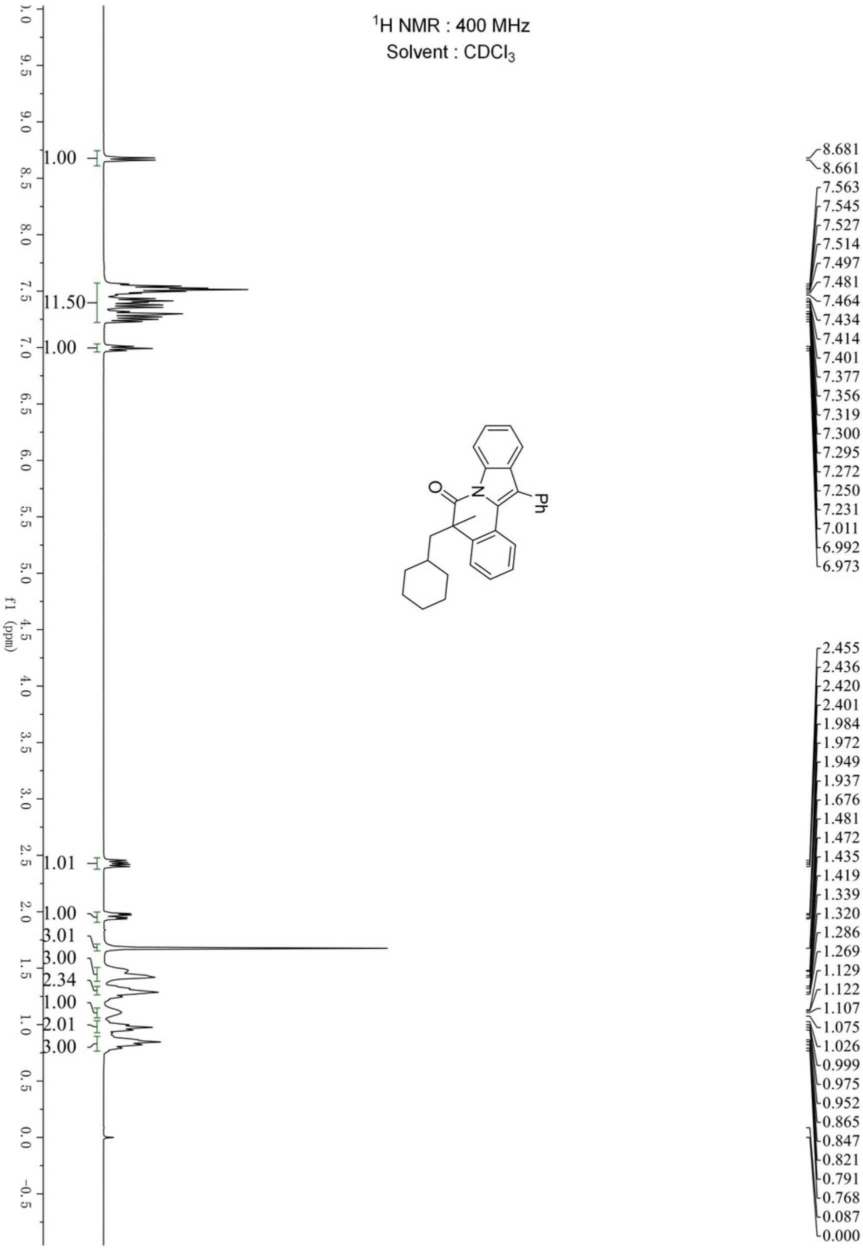
5-(Cyclohexy



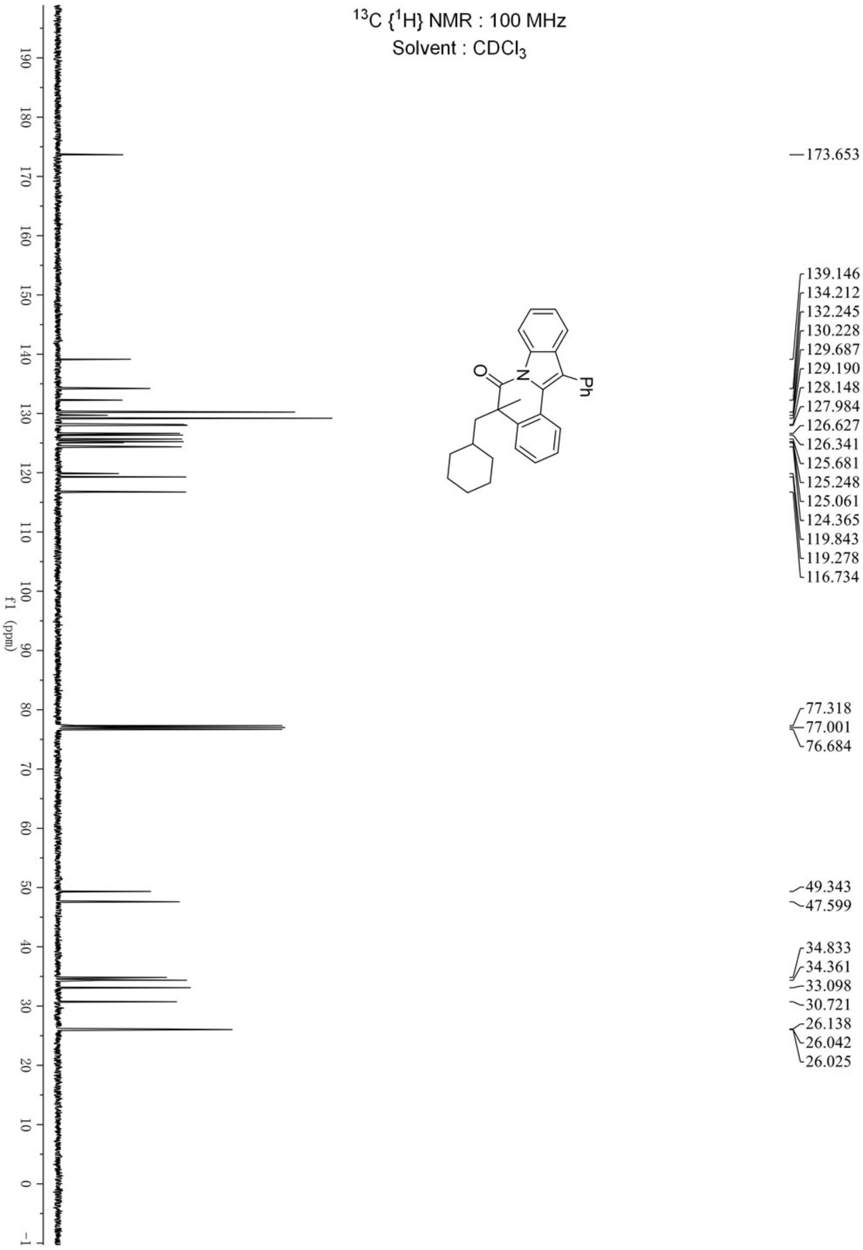
1H NMR : 400 MHz
Solvent : CDCl3
COOEt
8.624
8.621
8.606
8.602
8.468
8.448
7.968
7.963
7.950
7.946
7.436
7.419
7.403
7.398
7.384
7.380
7.374
7.365
7.360
7.353
7.345
7.254
4.585
4.567
4.549
4.531
2.340
2.321
2.305
2.286
1.967
1.954
1.931
1.919
1.660
1.523
1.505
1.487
1.452
1.434
1.424
1.416
1.391
1.313
1.280
1.274
1.265
1.216
1.212
0.972
0.970
0.955
0.945
0.881
0.870
0.863
0.854
0.833
0.826
0.811
0.799
0.781
0.775
0.767
0.000


$^{13}C\{^{1}H\}$ NMR : 100 MHz
Solvent : $CDCl_3$
174.005
165.822
140.082
136.875
134.140
129.918
128.961
127.842
126.734
126.230
126.081
124.984
123.539
120.869
116.530
109.970
77.318
77.001
76.684
61.227
49.497
47.984
34.639
34.329
33.268
29.577
26.059
26.014
25.942
14.337
COOEt
N
O
f1 (ppm)

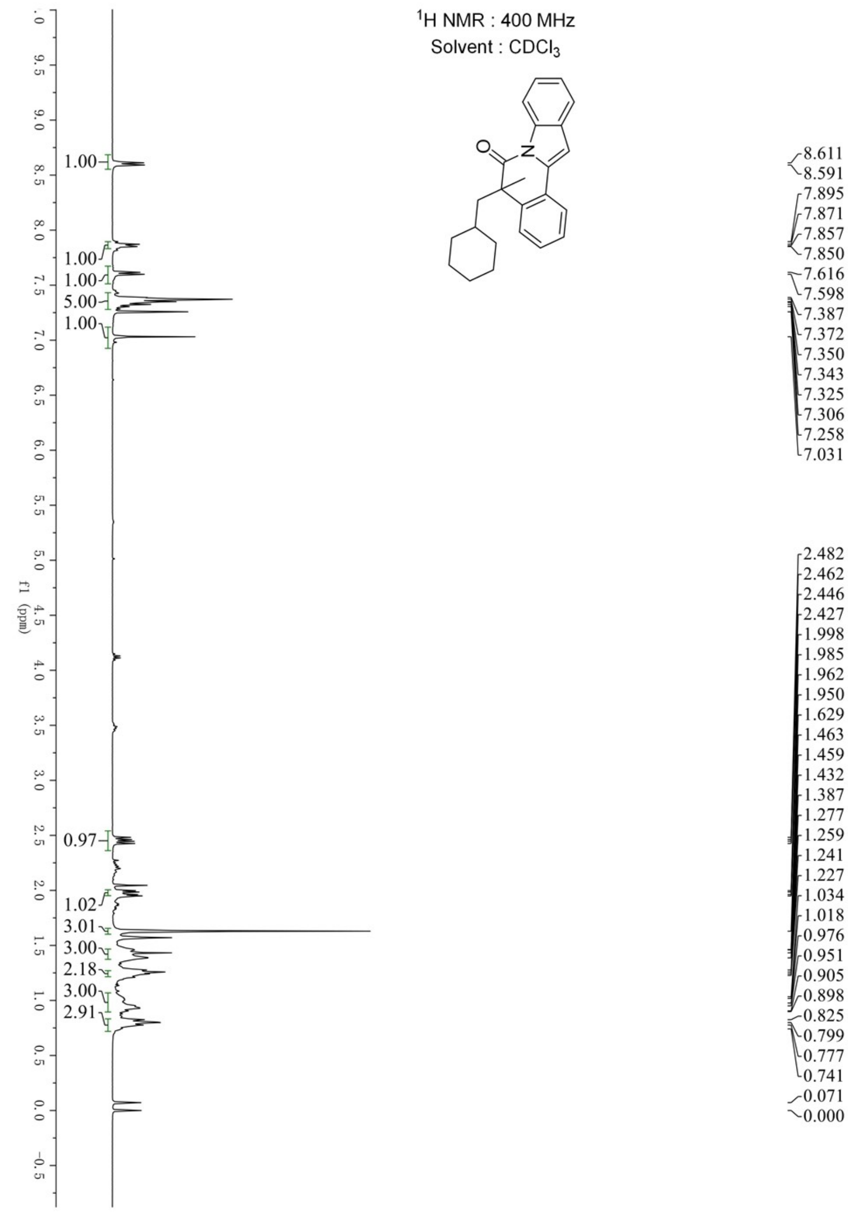
5-(Cyclohe

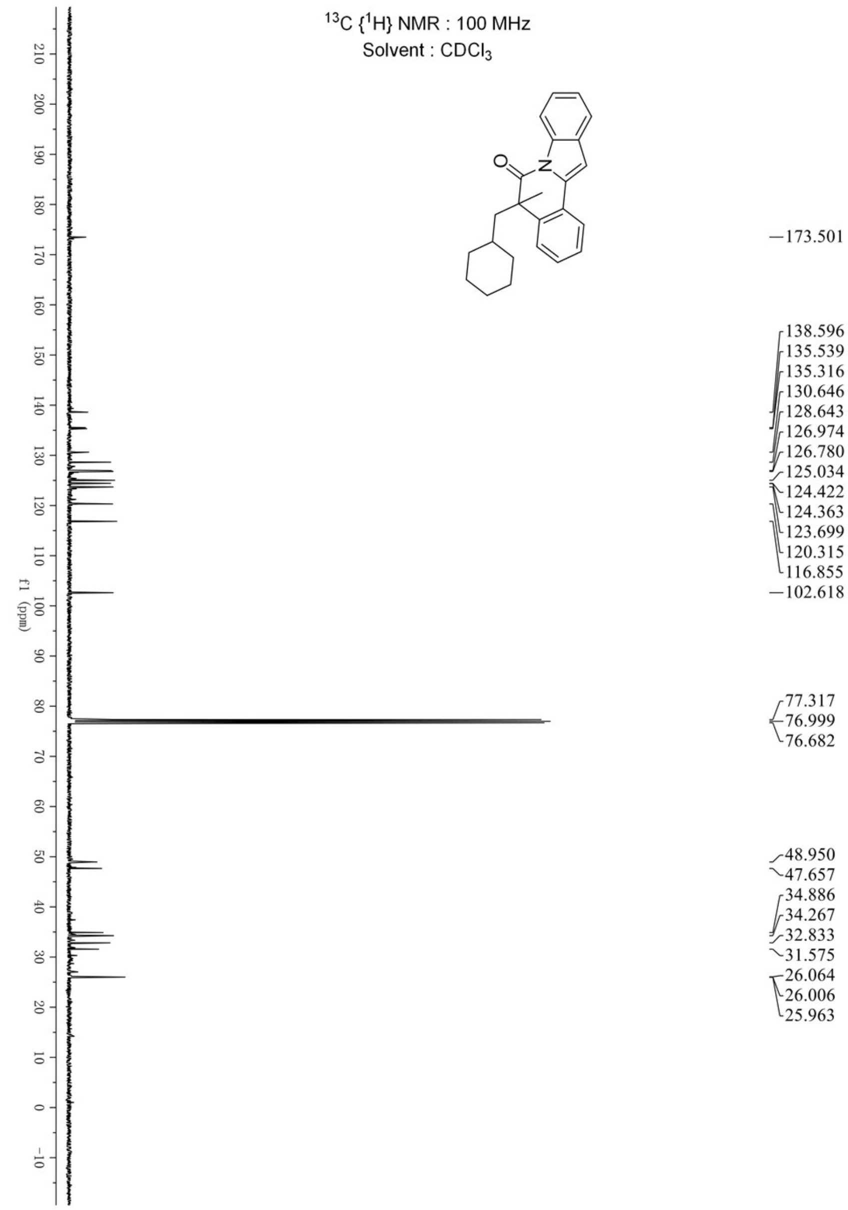


^{1}H NMR : 400 MHz
Solvent : $CDCl_3$
Ph
O
N
f1 (ppm)
8.681
8.661
7.563
7.545
7.527
7.514
7.497
7.481
7.464
7.434
7.414
7.401
7.377
7.356
7.319
7.300
7.295
7.272
7.250
7.231
7.011
6.992
6.973
2.455
2.436
2.420
2.401
1.984
1.972
1.949
1.937
1.676
1.481
1.472
1.435
1.419
1.339
1.320
1.286
1.269
1.129
1.122
1.107
1.075
1.026
0.999
0.975
0.952
0.865
0.847
0.821
0.791
0.768
0.087
0.000
1.00
11.50
1.00
1.01
1.00
3.01
3.00
2.34
1.00
2.01
3.00

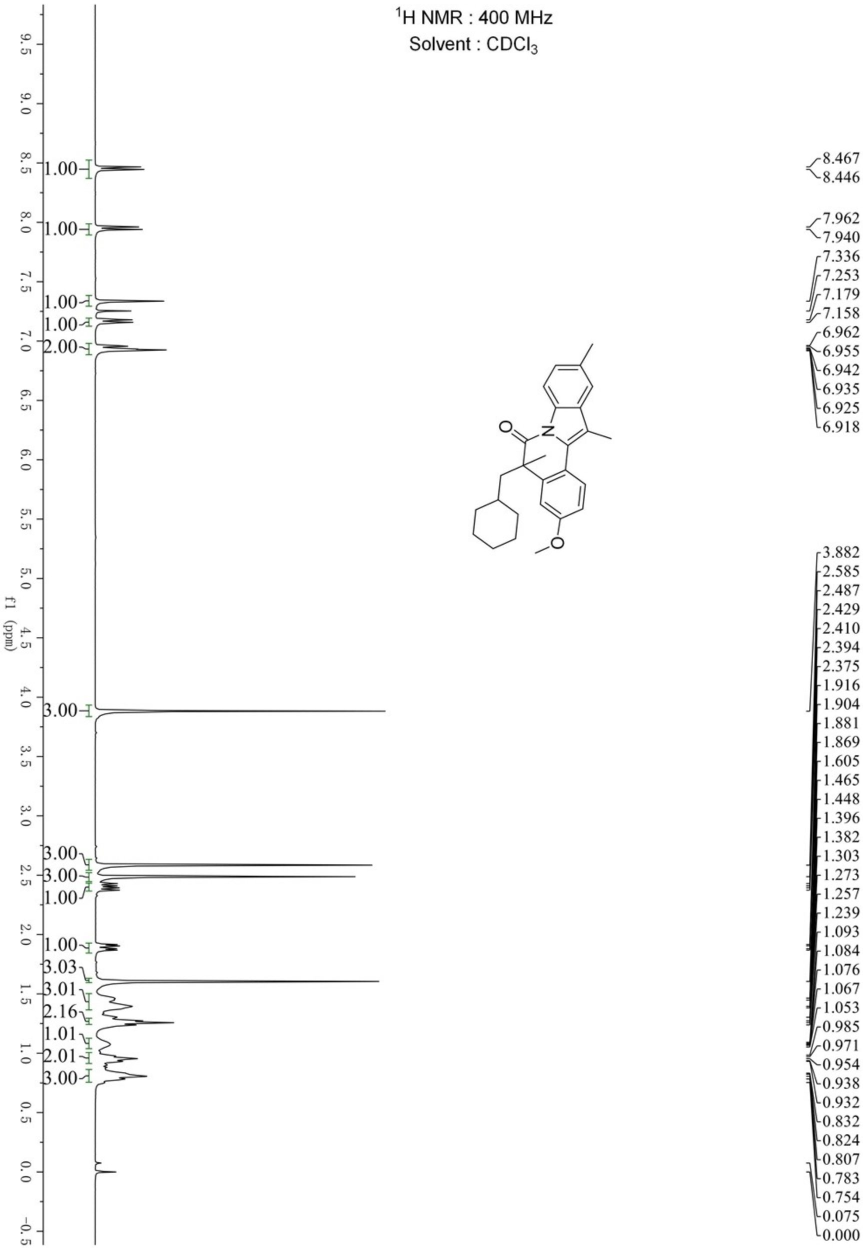

^{13}C {^{1}H} NMR : 100 MHz
Solvent : $CDCl_3$
Ph
O
N
f1 (ppm)
173.653
139.146
134.212
132.245
130.228
129.687
129.190
128.148
127.984
126.627
126.341
125.681
125.248
125.061
124.365
119.843
119.278
116.734
77.318
77.001
76.684
49.343
47.599
34.833
34.361
33.098
30.721
26.138
26.042
26.025

5-(Cyclo

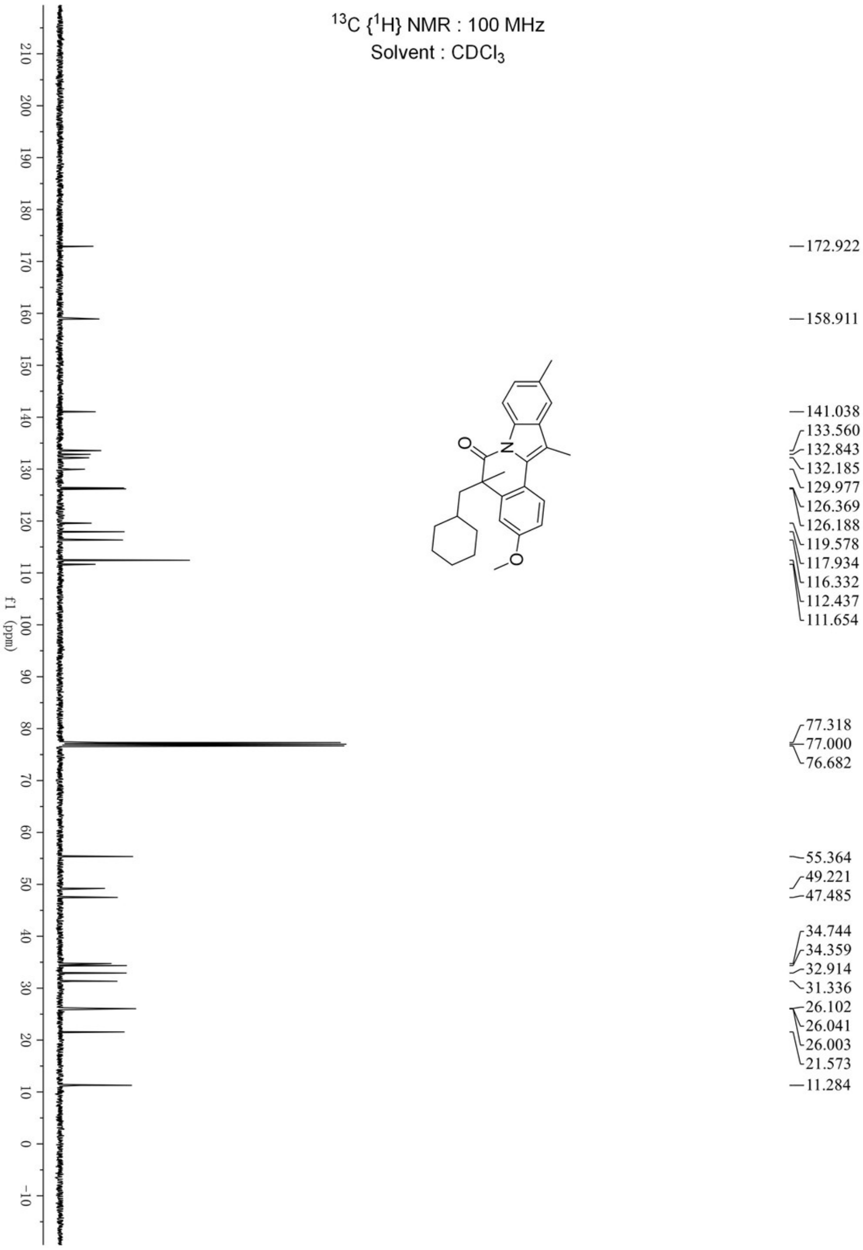

1H NMR : 400 MHz
Solvent : CDCl3
f1 (ppm)
8.611
8.591
7.895
7.871
7.857
7.850
7.616
7.598
7.387
7.372
7.350
7.343
7.325
7.306
7.258
7.031
2.482
2.462
2.446
2.427
1.998
1.985
1.962
1.950
1.629
1.463
1.459
1.432
1.387
1.277
1.259
1.241
1.227
1.034
1.018
0.976
0.951
0.905
0.898
0.825
0.799
0.777
0.741
0.071
0.000



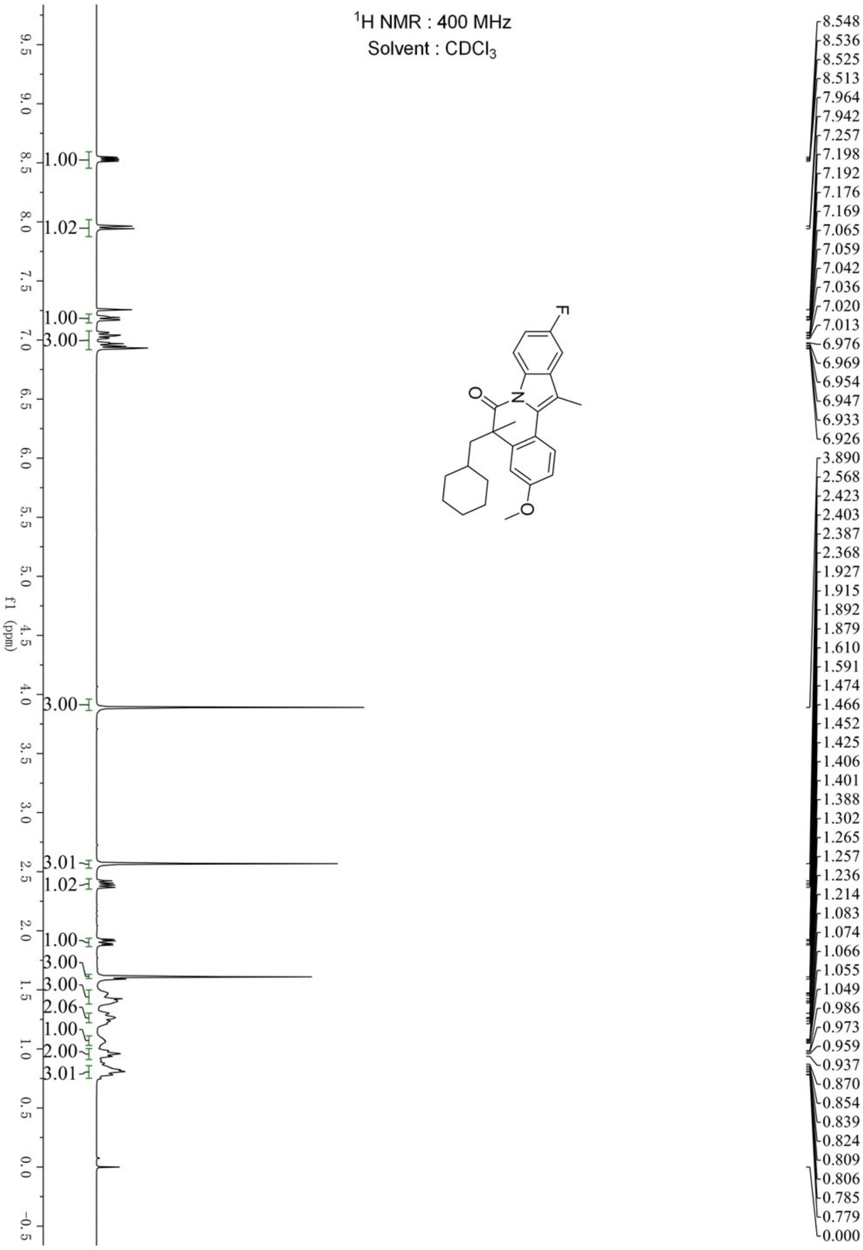
5-(Cyclohexylmethyl)-3-methoxy-5,10,12-trimethylindolo[2,1-*a*]isoquinolin-6(5*H*)-one (3pa)


^{1}H NMR : 400 MHz
Solvent : $CDCl_3$
8.467
8.446
7.962
7.940
7.336
7.253
7.179
7.158
6.962
6.955
6.942
6.935
6.925
6.918
3.882
2.585
2.487
2.429
2.410
2.394
2.375
1.916
1.904
1.881
1.869
1.605
1.465
1.448
1.396
1.382
1.303
1.273
1.257
1.239
1.093
1.084
1.076
1.067
1.053
0.985
0.971
0.954
0.938
0.932
0.832
0.824
0.807
0.783
0.754
0.075
0.000
f1 (ppm)

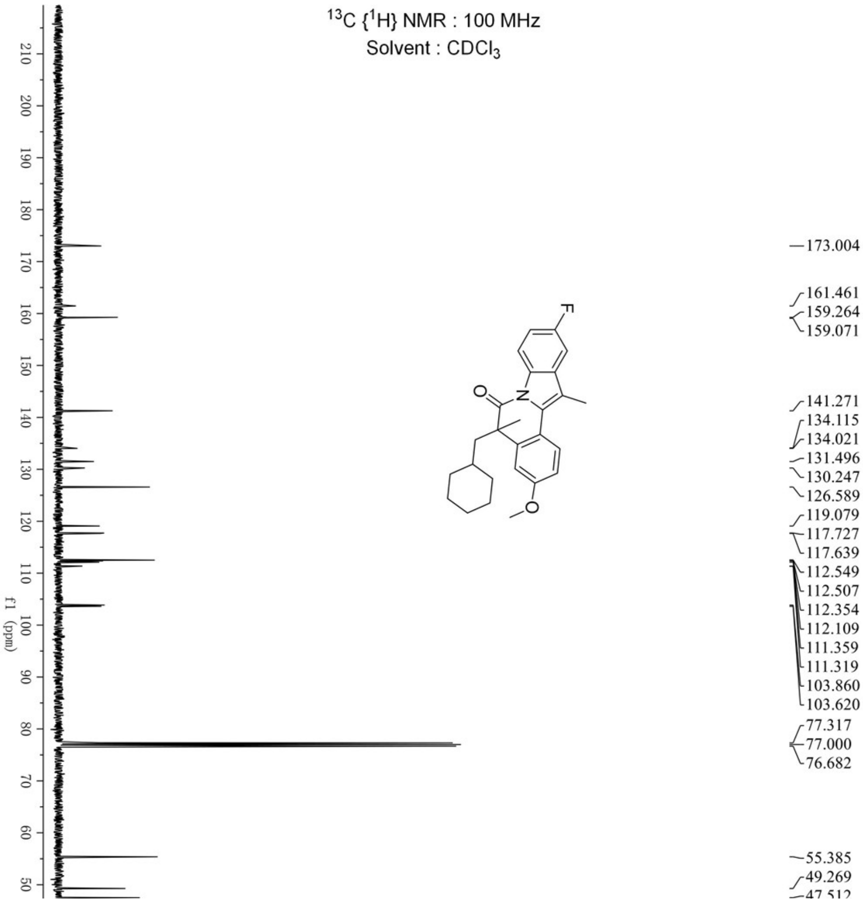
$^{13}C \{^1H\}$ NMR : 100 MHz
Solvent : $CDCl_3$

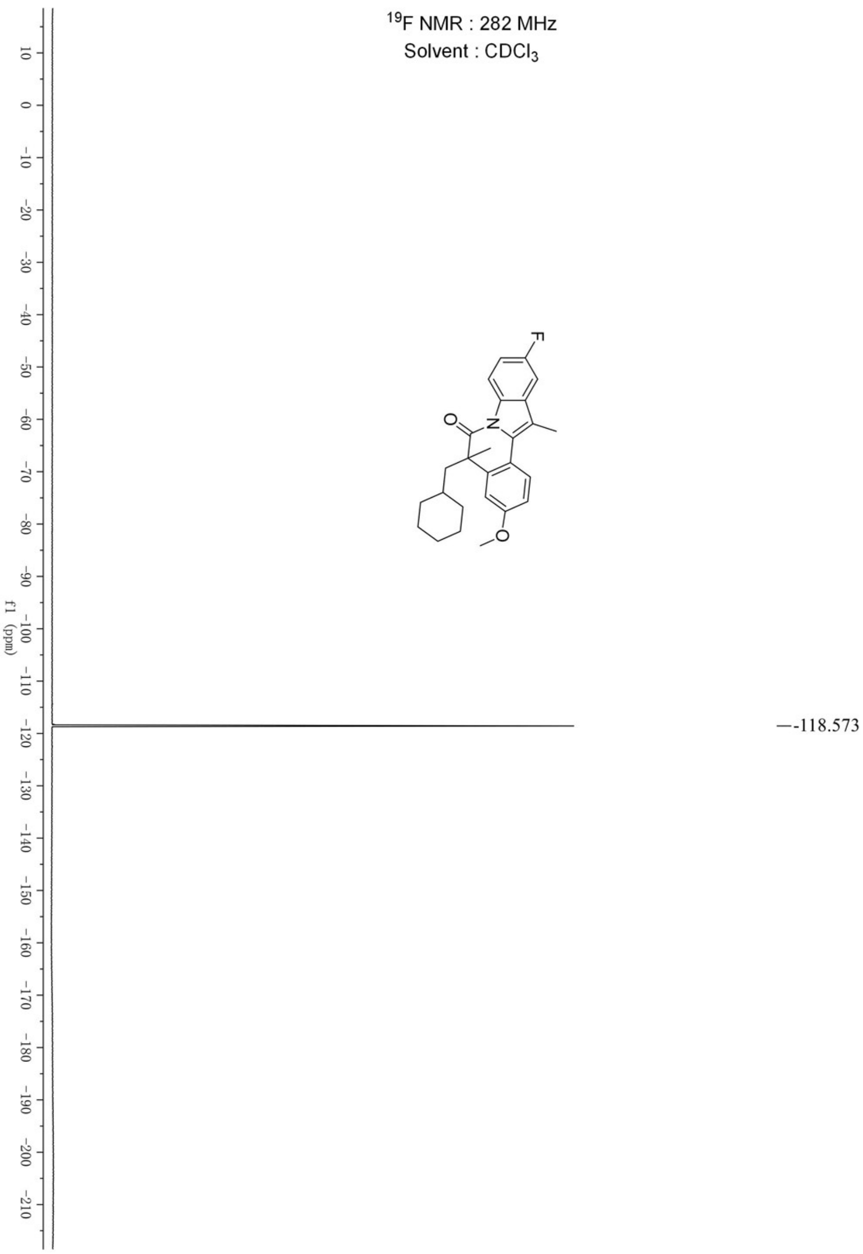


5-(Cycl

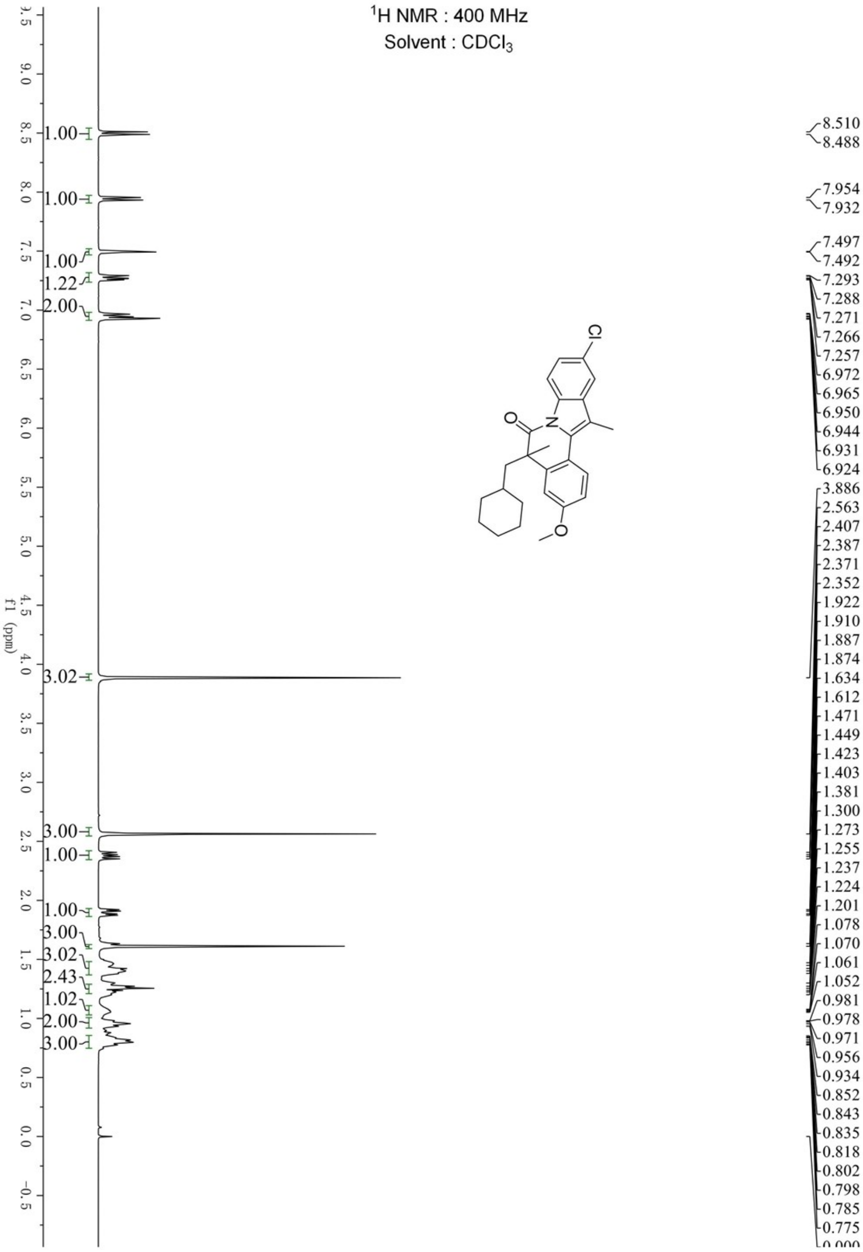


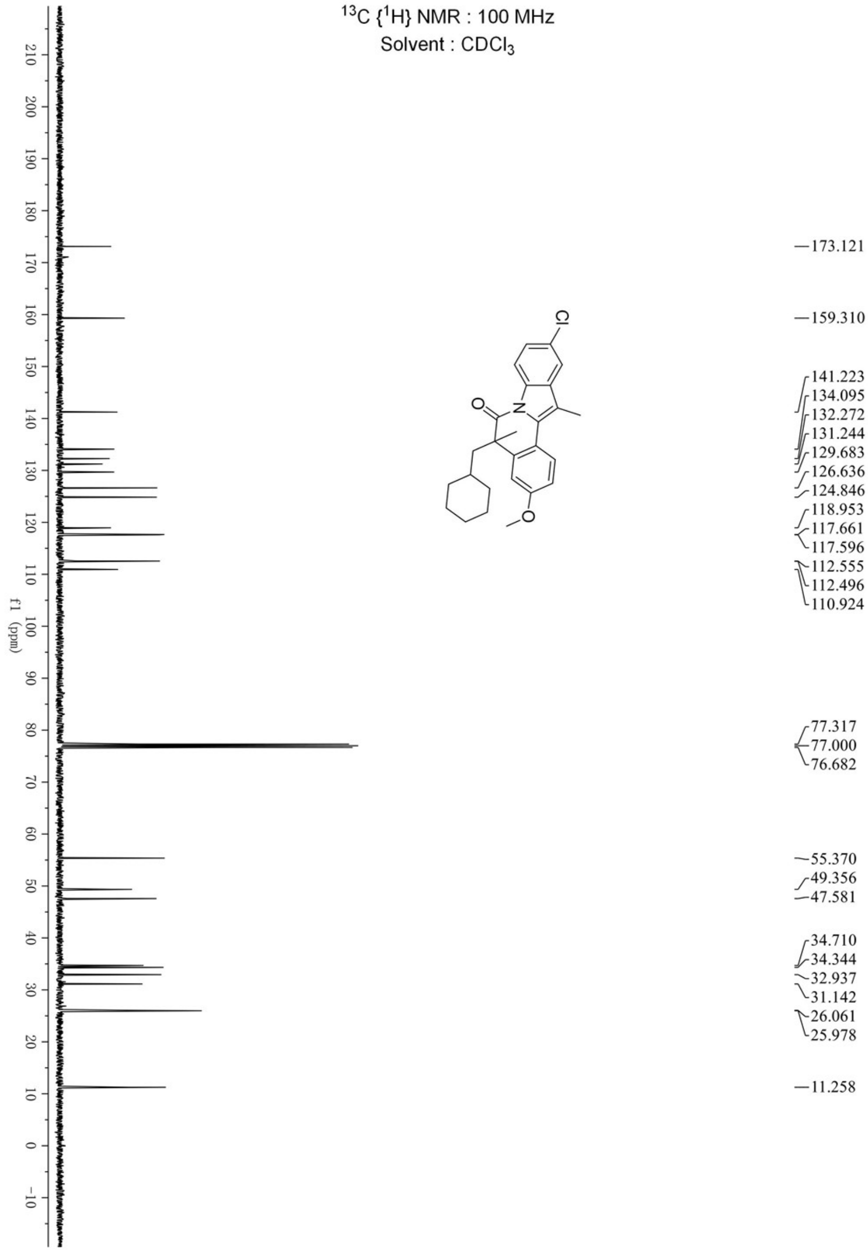
^{1}H NMR : 400 MHz
Solvent : CDCl$_3$
8.548
8.536
8.525
8.513
7.964
7.942
7.257
7.198
7.192
7.176
7.169
7.065
7.059
7.042
7.036
7.020
7.013
6.976
6.969
6.954
6.947
6.933
6.926
3.890
2.568
2.423
2.403
2.387
2.368
1.927
1.915
1.892
1.879
1.610
1.591
1.474
1.466
1.452
1.425
1.406
1.401
1.388
1.302
1.265
1.257
1.236
1.214
1.083
1.074
1.066
1.055
1.049
0.986
0.973
0.959
0.937
0.870
0.854
0.839
0.824
0.809
0.806
0.785
0.779
0.000
f1 (ppm)


13C {1H} NMR : 100 MHz
Solvent : CDCl3
f1 (ppm)
173.004
161.461
159.264
159.071
141.271
134.115
134.021
131.496
130.247
126.589
119.079
117.727
117.639
112.549
112.507
112.354
112.109
111.359
111.319
103.860
103.620
77.317
77.000
76.682
55.385
49.269
F
O
N


19F NMR : 282 MHz
Solvent : CDCl3
f1 (ppm)
-118.573
F
O
N

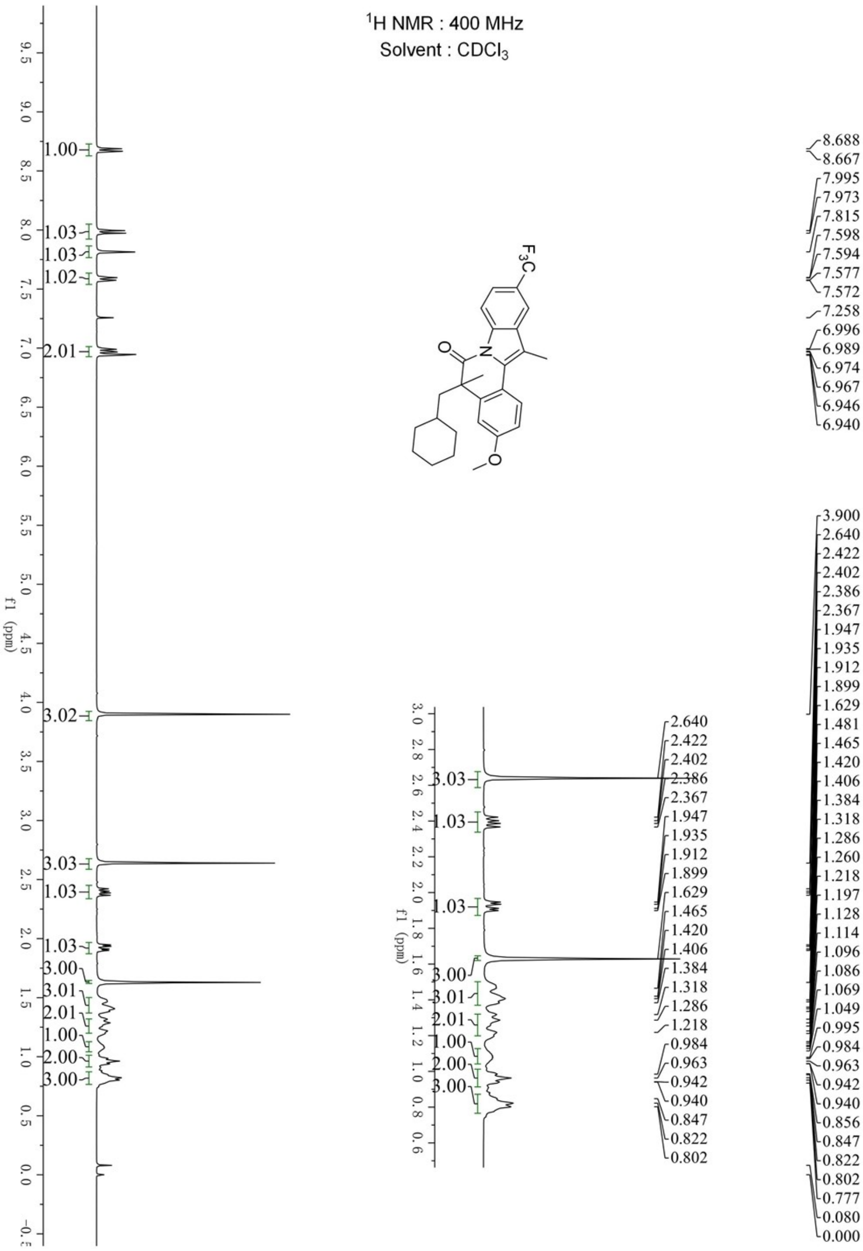
10-Chl


^{1}H NMR : 400 MHz
Solvent : $CDCl_3$
8.510
8.488
7.954
7.932
7.497
7.492
7.293
7.288
7.271
7.266
7.257
6.972
6.965
6.950
6.944
6.931
6.924
3.886
2.563
2.407
2.387
2.371
2.352
1.922
1.910
1.887
1.874
1.634
1.612
1.471
1.449
1.423
1.403
1.381
1.300
1.273
1.255
1.237
1.224
1.201
1.078
1.070
1.061
1.052
0.981
0.978
0.971
0.956
0.934
0.852
0.843
0.835
0.818
0.802
0.798
0.785
0.775
f1 (ppm)



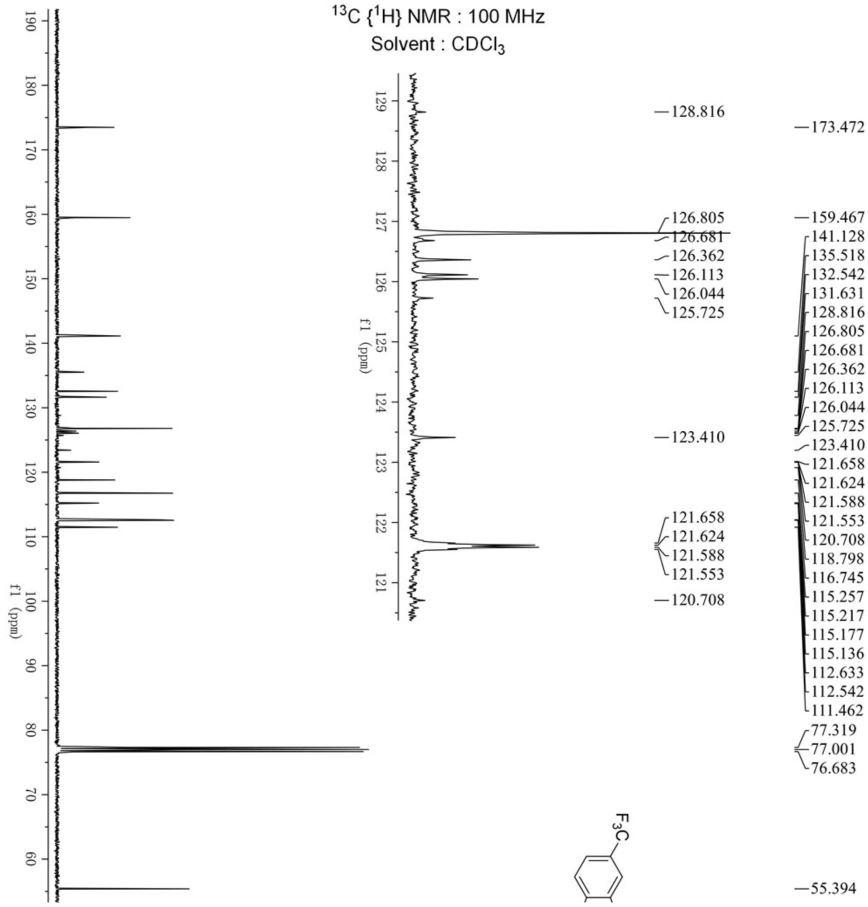
13C {1H} NMR : 100 MHz
Solvent : CDCl3
173.121
159.310
141.223
134.095
132.272
131.244
129.683
126.636
124.846
118.953
117.661
117.596
112.555
112.496
110.924
77.317
77.000
76.682
55.370
49.356
47.581
34.710
34.344
32.937
31.142
26.061
25.978
11.258
Cl
N
O
f1 (ppm)

5-(Cyclohexy

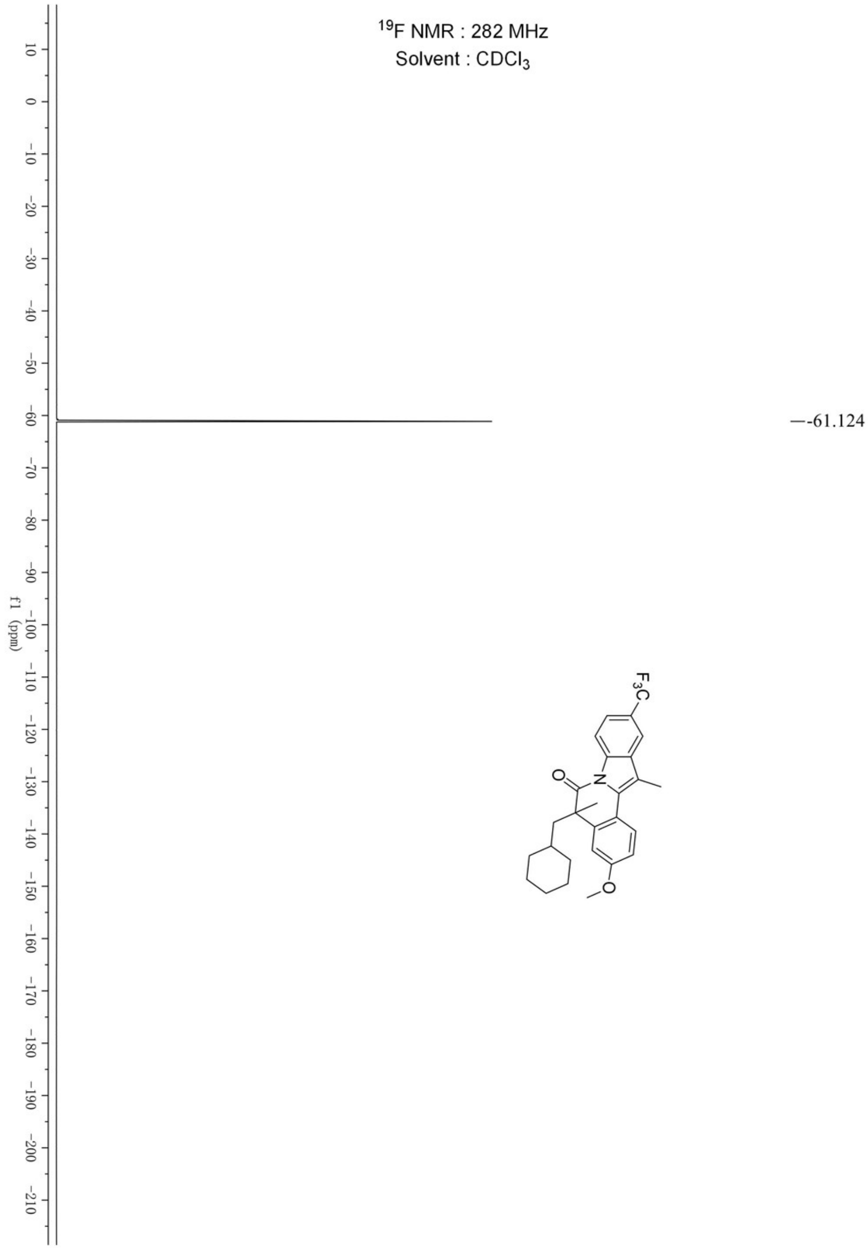


1H NMR : 400 MHz
Solvent : CDCl3
F3C
N
O
f1 (ppm)
8.688
8.667
7.995
7.973
7.815
7.598
7.594
7.577
7.572
7.258
6.996
6.989
6.974
6.967
6.946
6.940
3.900
2.640
2.422
2.402
2.386
2.367
1.947
1.935
1.912
1.899
1.629
1.481
1.465
1.420
1.406
1.384
1.318
1.286
1.260
1.218
1.197
1.128
1.114
1.096
1.086
1.069
1.049
0.995
0.984
0.963
0.942
0.940
0.856
0.847
0.822
0.802
0.777
0.080
0.000

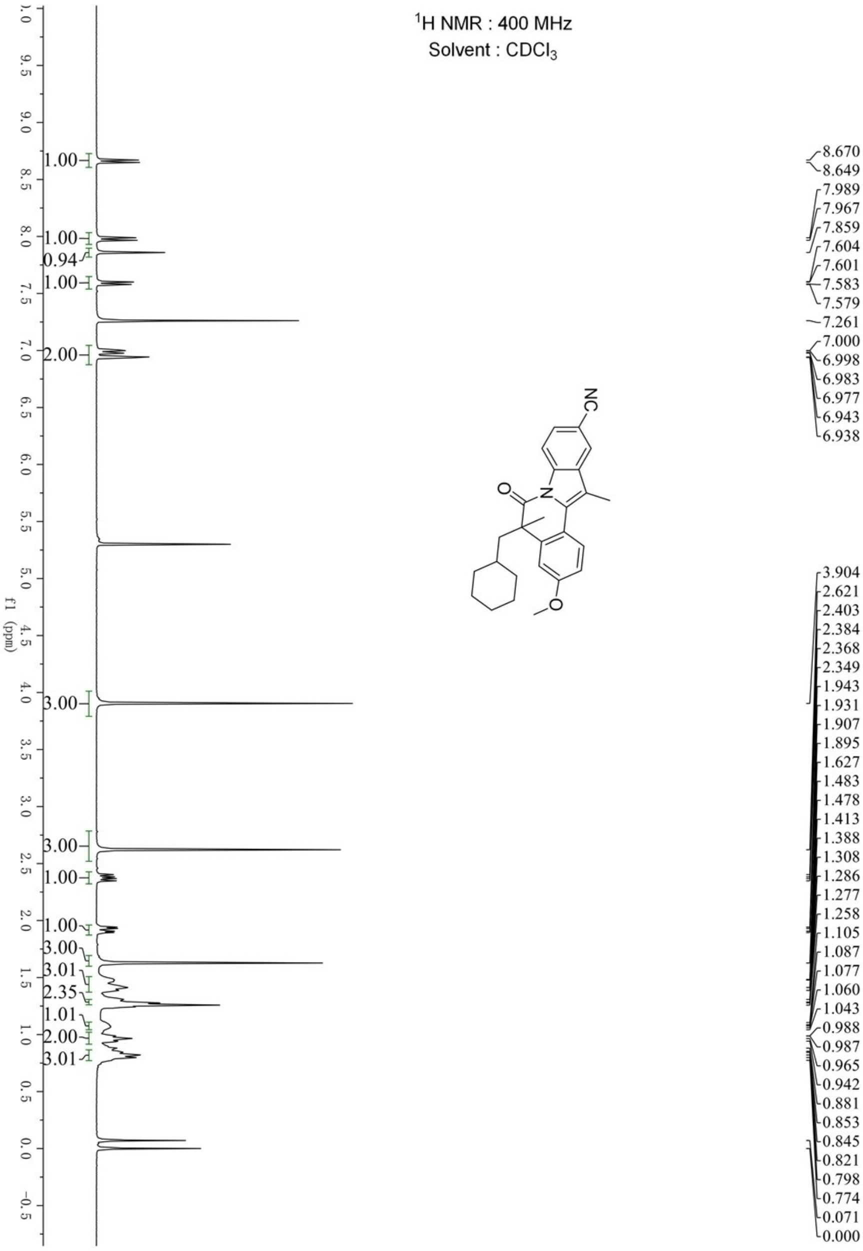
$^{13}C \{^{1}H\}$ NMR : 100 MHz
Solvent : $CDCl_3$



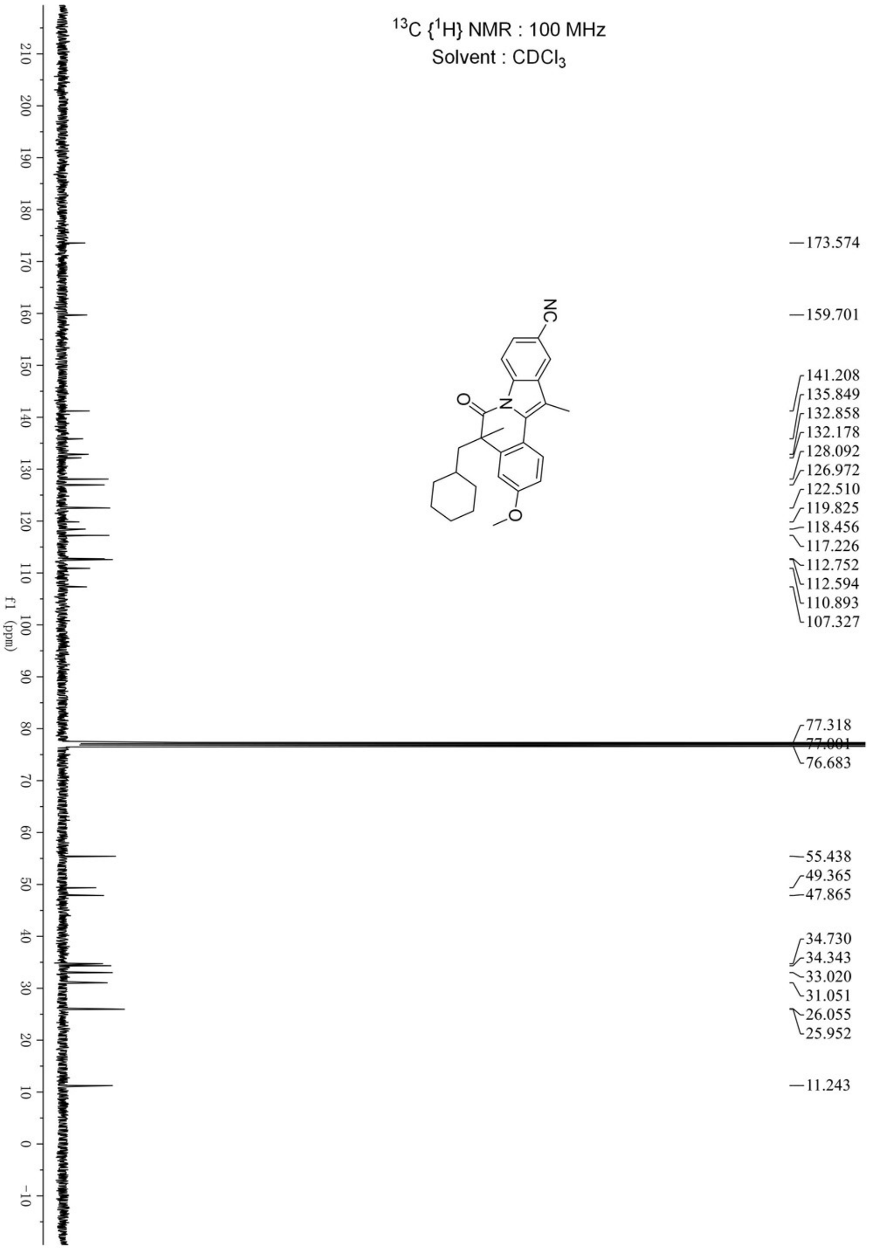
^{19}F NMR : 282 MHz
Solvent : $CDCl_3$



5-(Cyclohex

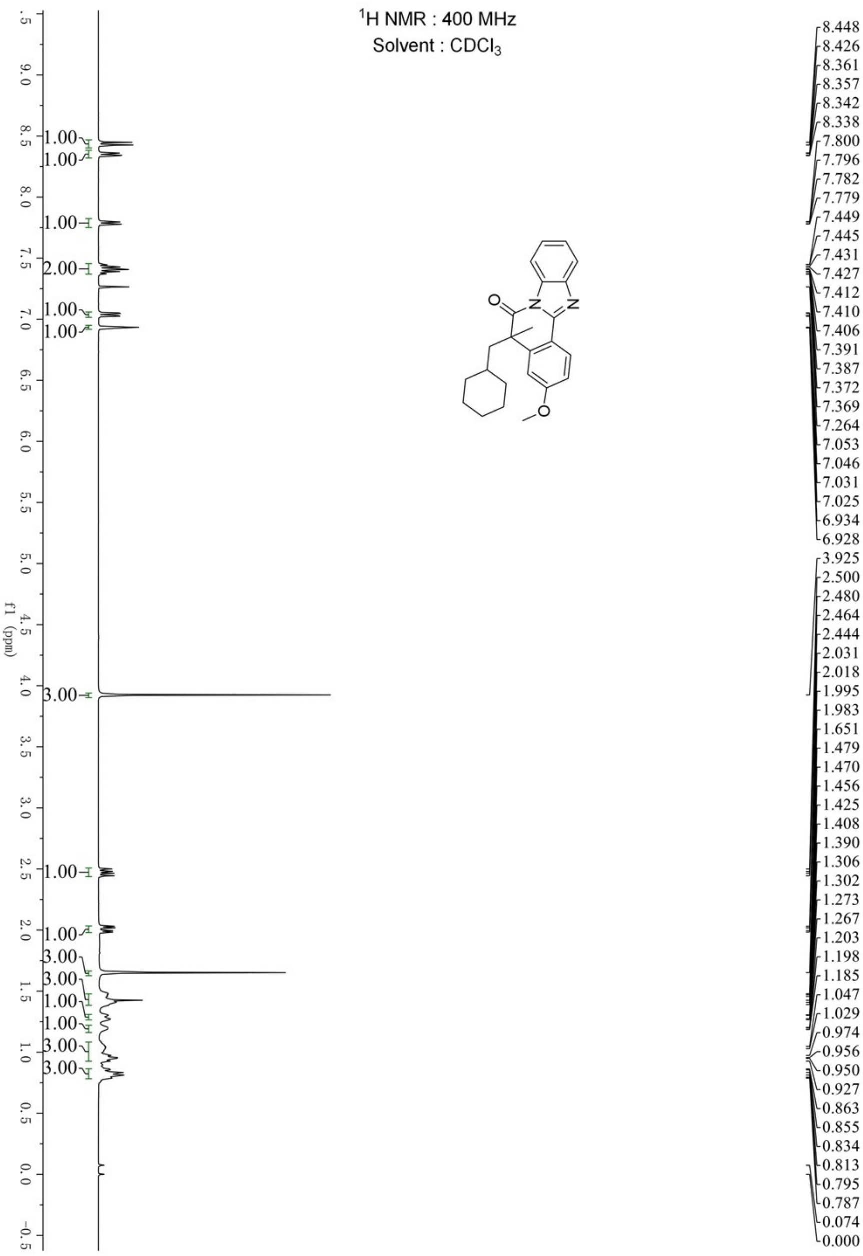


^{1}H NMR : 400 MHz
Solvent : $CDCl_3$
NC
O
N
O
f1 (ppm)
8.670
8.649
7.989
7.967
7.859
7.604
7.601
7.583
7.579
7.261
7.000
6.998
6.983
6.977
6.943
6.938
3.904
2.621
2.403
2.384
2.368
2.349
1.943
1.931
1.907
1.895
1.627
1.483
1.478
1.413
1.388
1.308
1.286
1.277
1.258
1.105
1.087
1.077
1.060
1.043
0.988
0.987
0.965
0.942
0.881
0.853
0.845
0.821
0.798
0.774
0.071
0.000

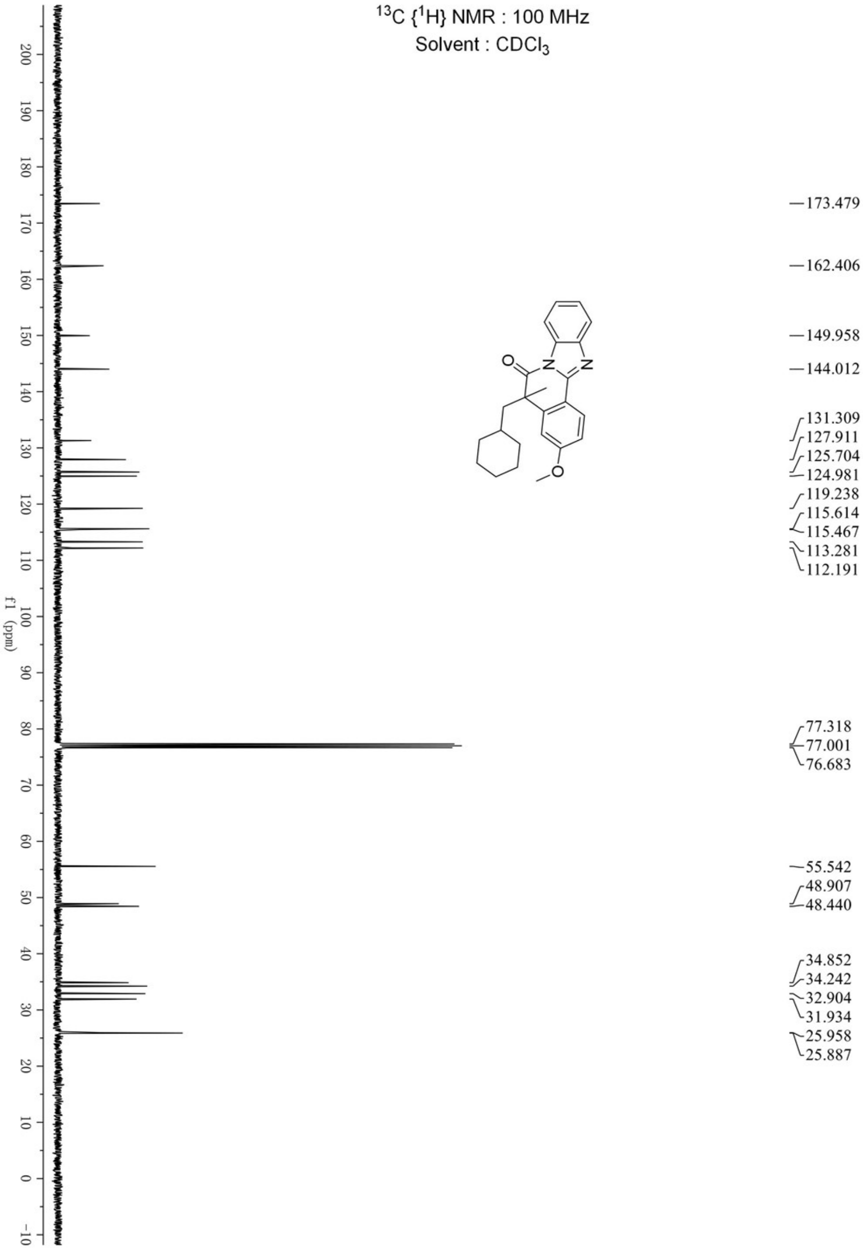


$^{13}C\ \{^1H\}$ NMR : 100 MHz
Solvent : $CDCl_3$
NC
N
O
O
173.574
159.701
141.208
135.849
132.858
132.178
128.092
126.972
122.510
119.825
118.456
117.226
112.752
112.594
110.893
107.327
77.318
77.001
76.683
55.438
49.365
47.865
34.730
34.343
33.020
31.051
26.055
25.952
11.243
f1 (ppm)

5-(Cyclohexy

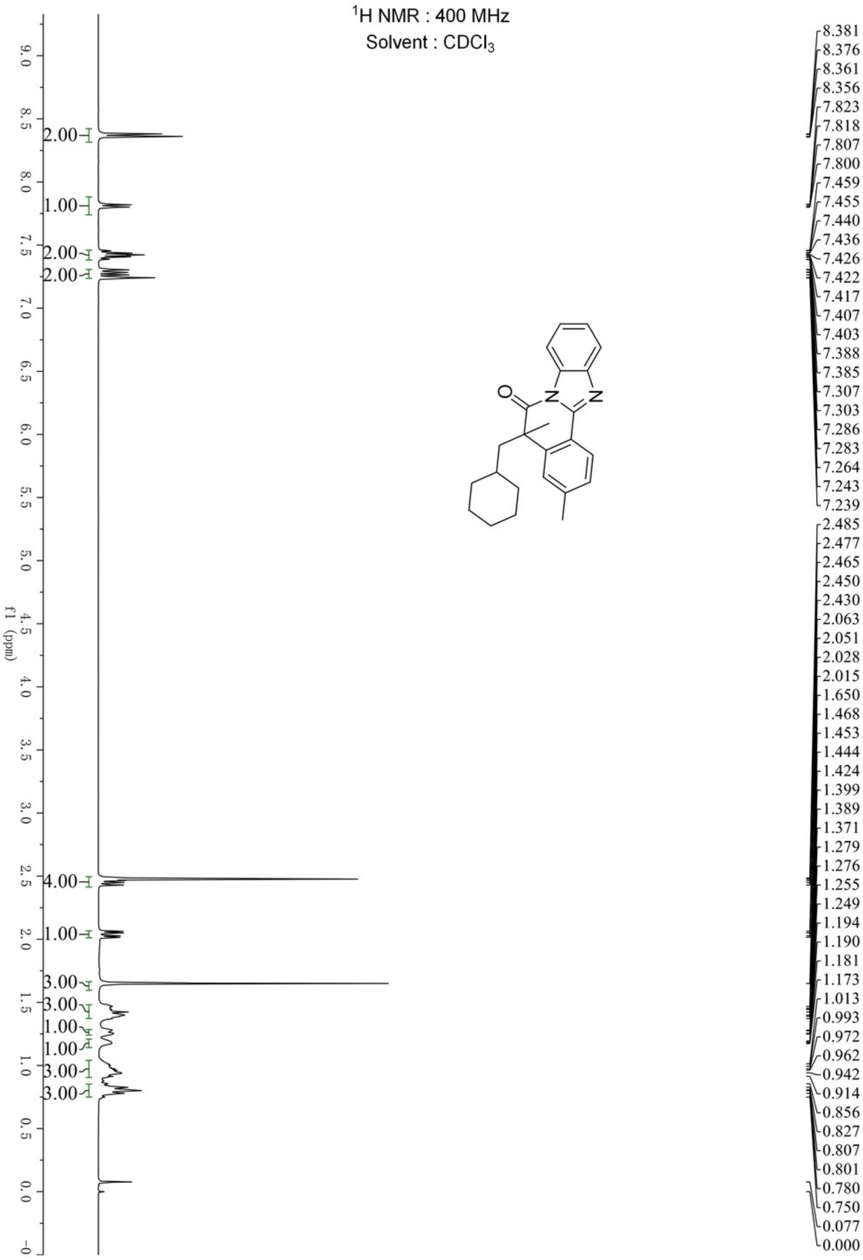


1H NMR : 400 MHz
Solvent : CDCl3
8.448
8.426
8.361
8.357
8.342
8.338
7.800
7.796
7.782
7.779
7.449
7.445
7.431
7.427
7.412
7.410
7.406
7.391
7.387
7.372
7.369
7.264
7.053
7.046
7.031
7.025
6.934
6.928
3.925
2.500
2.480
2.464
2.444
2.031
2.018
1.995
1.983
1.651
1.479
1.470
1.456
1.425
1.408
1.390
1.306
1.302
1.273
1.267
1.203
1.198
1.185
1.047
1.029
0.974
0.956
0.950
0.927
0.863
0.855
0.834
0.813
0.795
0.787
0.074
0.000
f1 (ppm)



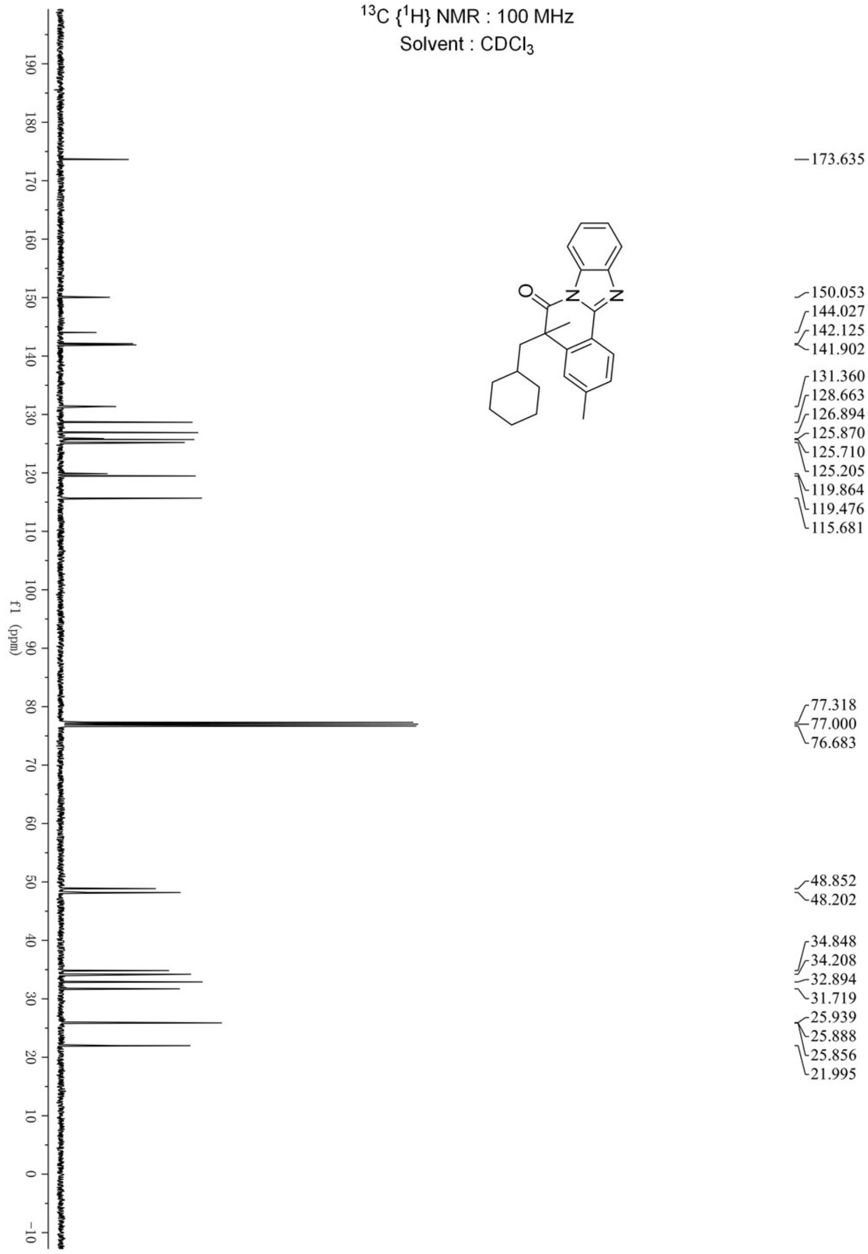
$^{13}C\{^{1}H\}$ NMR : 100 MHz
Solvent : $CDCl_3$
173.479
162.406
149.958
144.012
131.309
127.911
125.704
124.981
119.238
115.614
115.467
113.281
112.191
77.318
77.001
76.683
55.542
48.907
48.440
34.852
34.242
32.904
31.934
25.958
25.887
f1 (ppm)

5-(Cyclohex

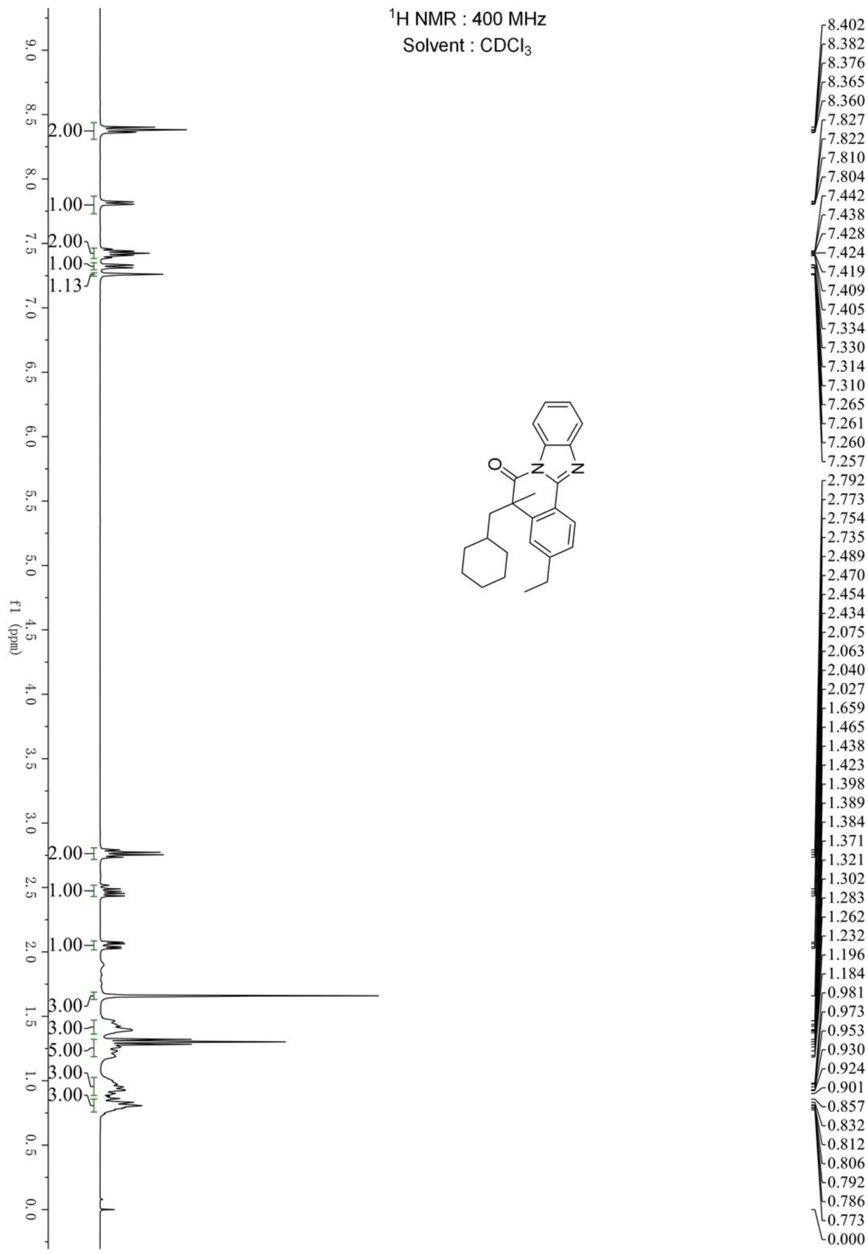


1H NMR : 400 MHz
Solvent : CDCl3
f1 (ppm)
8.381
8.376
8.361
8.356
7.823
7.818
7.807
7.800
7.459
7.455
7.440
7.436
7.426
7.422
7.417
7.407
7.403
7.388
7.385
7.307
7.303
7.286
7.283
7.264
7.243
7.239
2.485
2.477
2.465
2.450
2.430
2.063
2.051
2.028
2.015
1.650
1.468
1.453
1.444
1.424
1.399
1.389
1.371
1.279
1.276
1.255
1.249
1.194
1.190
1.181
1.173
1.013
0.993
0.972
0.962
0.942
0.914
0.856
0.827
0.807
0.801
0.780
0.750
0.077
0.000

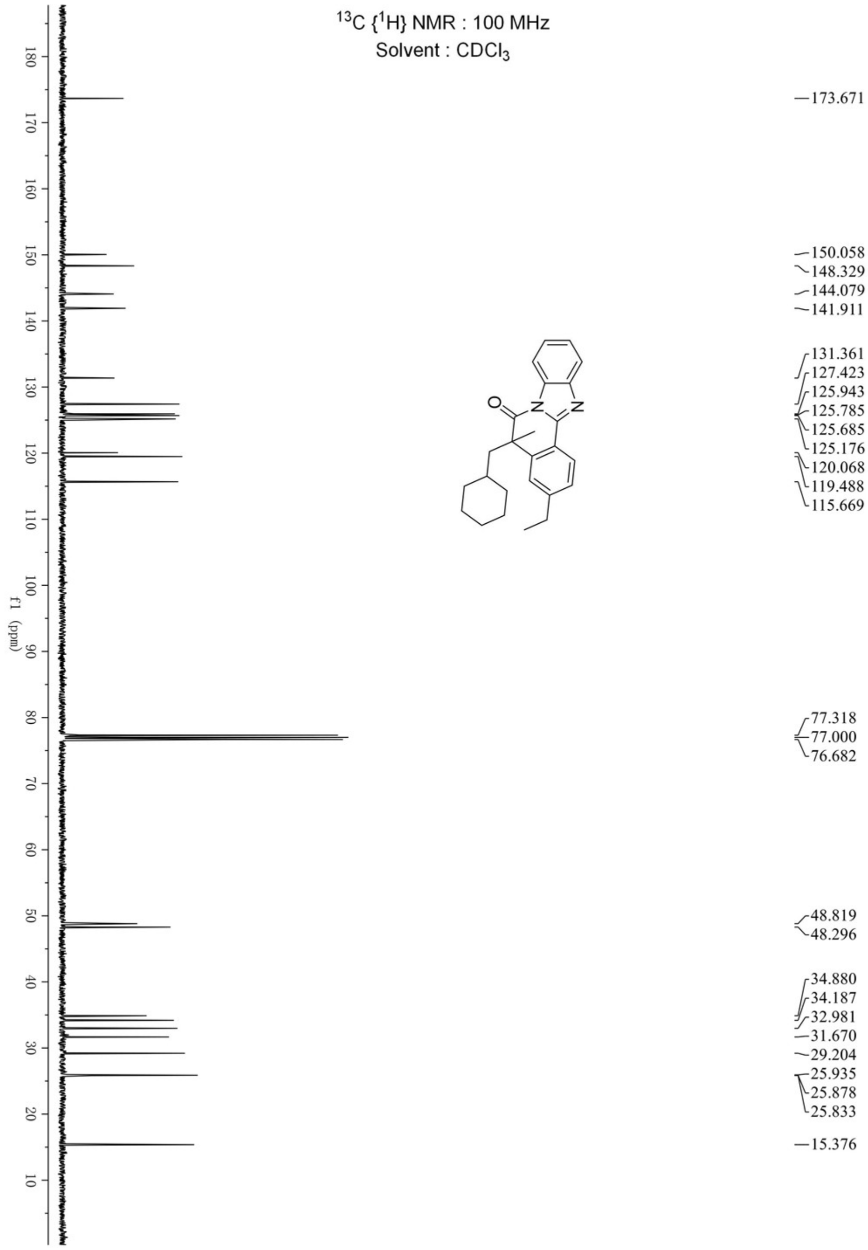
$^{13}C\{^1H\}$ NMR : 100 MHz
Solvent : $CDCl_3$



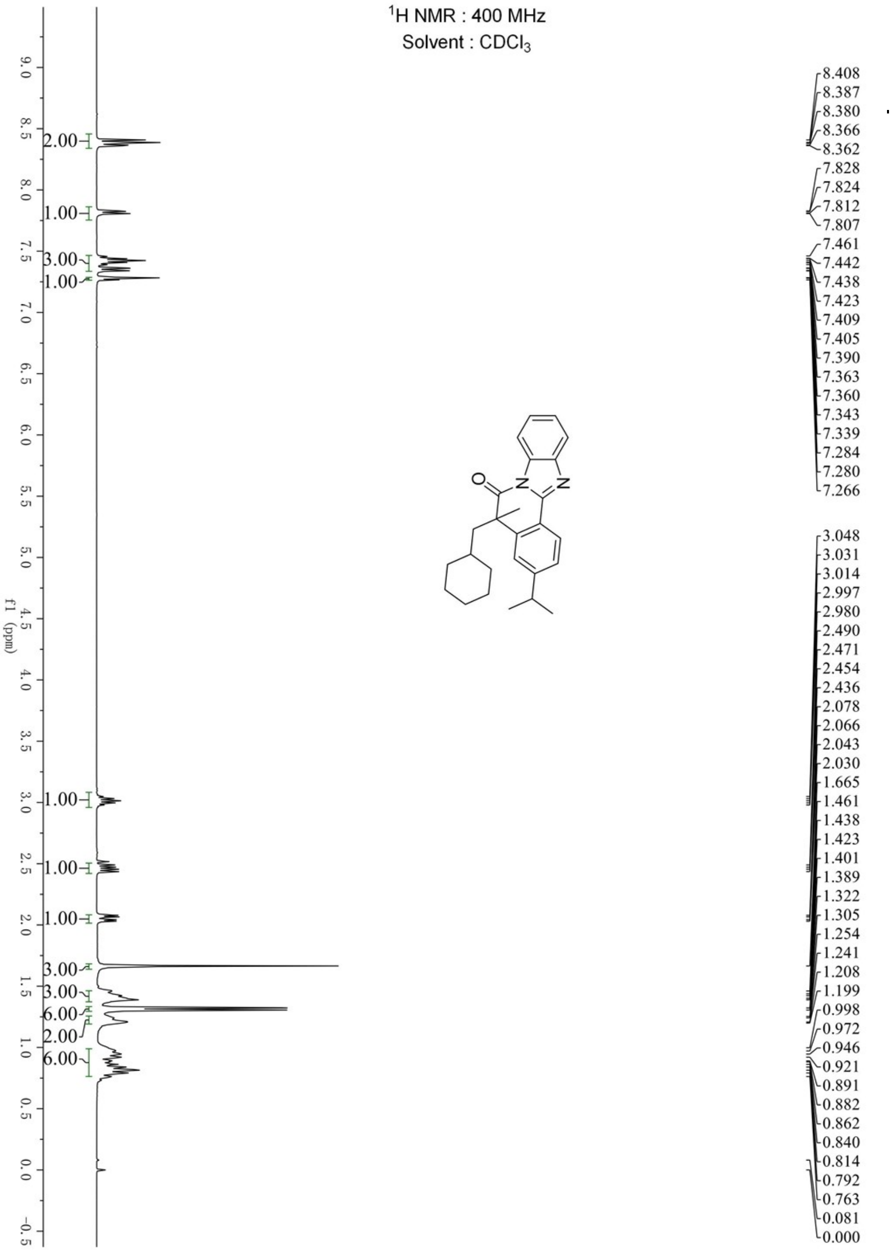
5-(Cyclohe



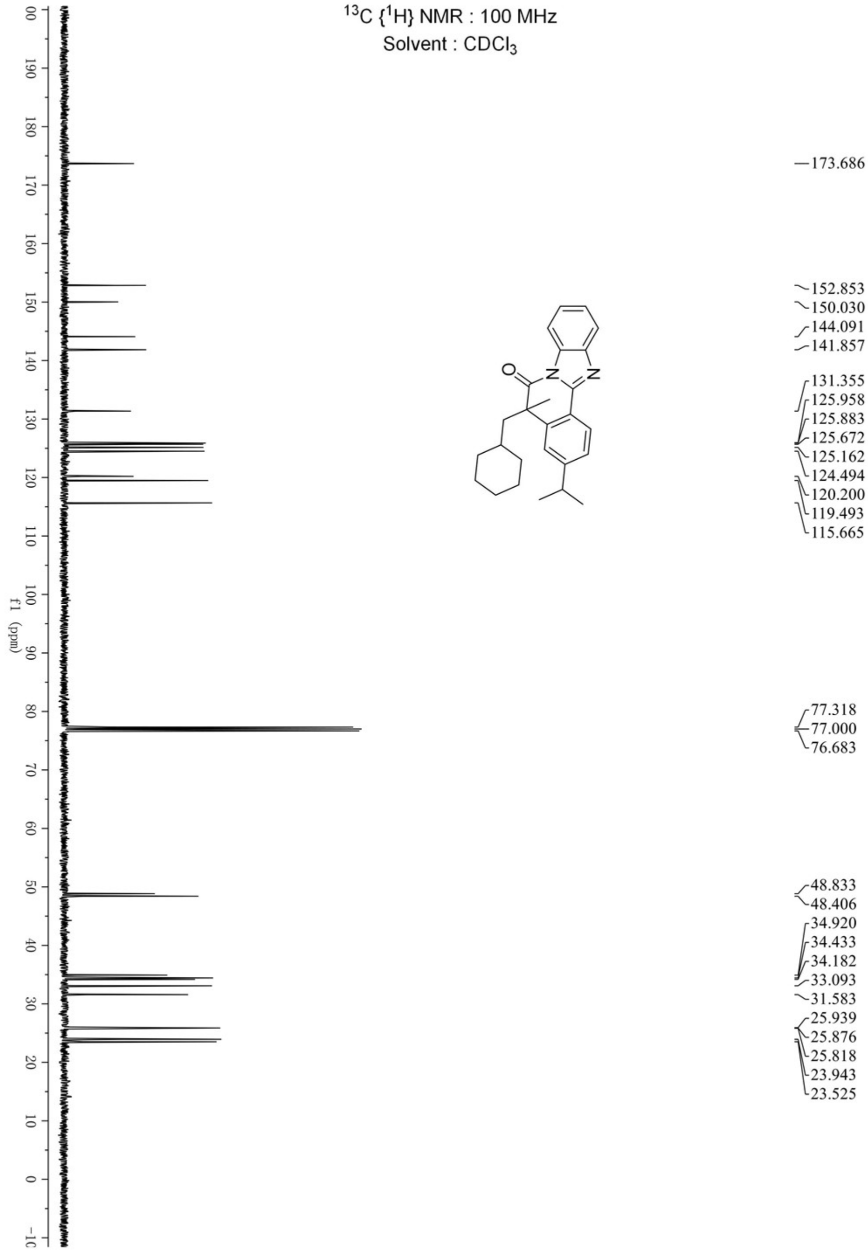
1H NMR : 400 MHz
Solvent : CDCl3
8.402
8.382
8.376
8.365
8.360
7.827
7.822
7.810
7.804
7.442
7.438
7.428
7.424
7.419
7.409
7.405
7.334
7.330
7.314
7.310
7.265
7.261
7.260
7.257
2.792
2.773
2.754
2.735
2.489
2.470
2.454
2.434
2.075
2.063
2.040
2.027
1.659
1.465
1.438
1.423
1.398
1.389
1.384
1.371
1.321
1.302
1.283
1.262
1.232
1.196
1.184
0.981
0.973
0.953
0.930
0.924
0.901
0.857
0.832
0.812
0.806
0.792
0.786
0.773
0.000


13C {1H} NMR : 100 MHz
Solvent : CDCl3
173.671
150.058
148.329
144.079
141.911
131.361
127.423
125.943
125.785
125.685
125.176
120.068
119.488
115.669
77.318
77.000
76.682
48.819
48.296
34.880
34.187
32.981
31.670
29.204
25.935
25.878
25.833
15.376
f1 (ppm)

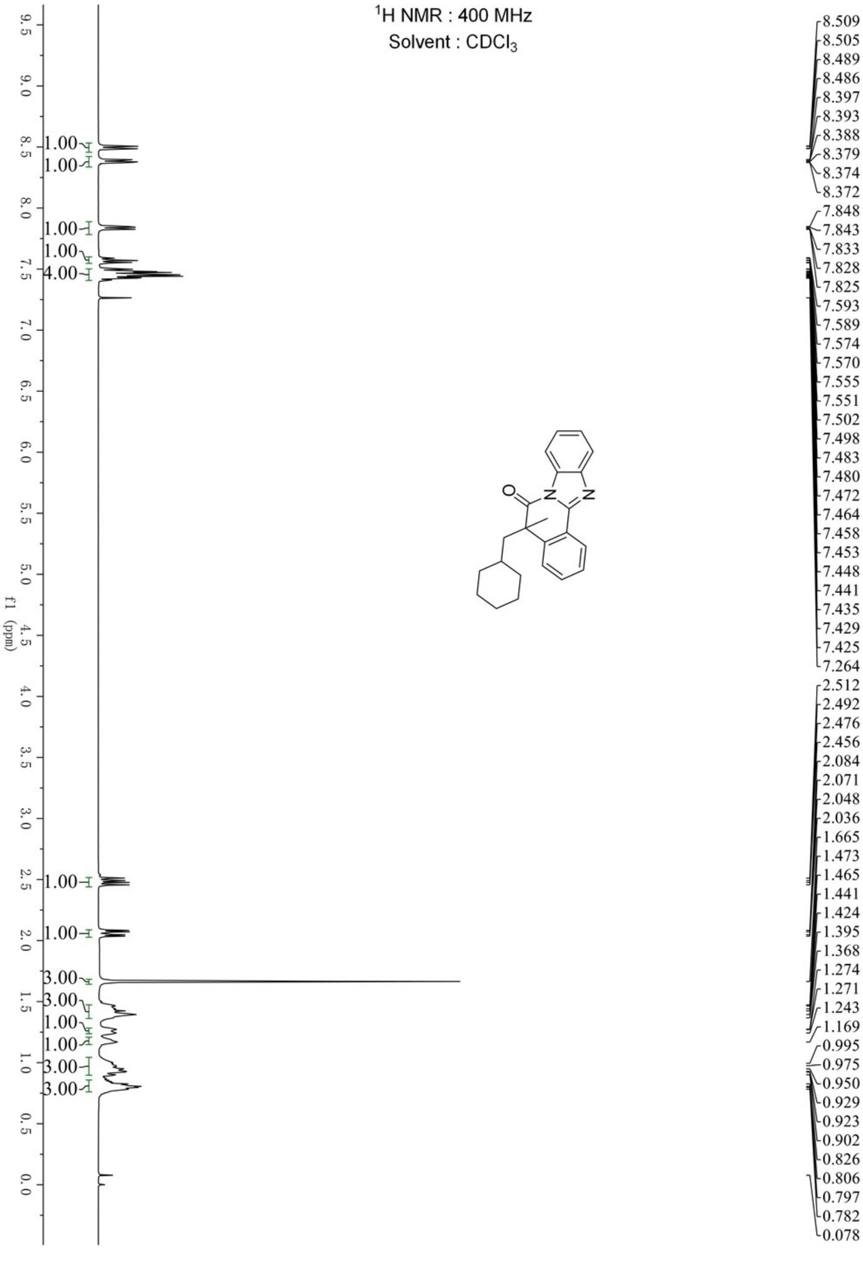
5-(Cyclohexy



1H NMR : 400 MHz
Solvent : CDCl3
8.408
8.387
8.380
8.366
8.362
7.828
7.824
7.812
7.807
7.461
7.442
7.438
7.423
7.409
7.405
7.390
7.363
7.360
7.343
7.339
7.284
7.280
7.266
3.048
3.031
3.014
2.997
2.980
2.490
2.471
2.454
2.436
2.078
2.066
2.043
2.030
1.665
1.461
1.438
1.423
1.401
1.389
1.322
1.305
1.254
1.241
1.208
1.199
0.998
0.972
0.946
0.921
0.891
0.882
0.862
0.840
0.814
0.792
0.763
0.081
0.000
f1 (ppm)


13C {1H} NMR : 100 MHz
Solvent : CDCl3
173.686
152.853
150.030
144.091
141.857
131.355
125.958
125.883
125.672
125.162
124.494
120.200
119.493
115.665
77.318
77.000
76.683
48.833
48.406
34.920
34.433
34.182
33.093
31.583
25.939
25.876
25.818
23.943
23.525
f1 (ppm)

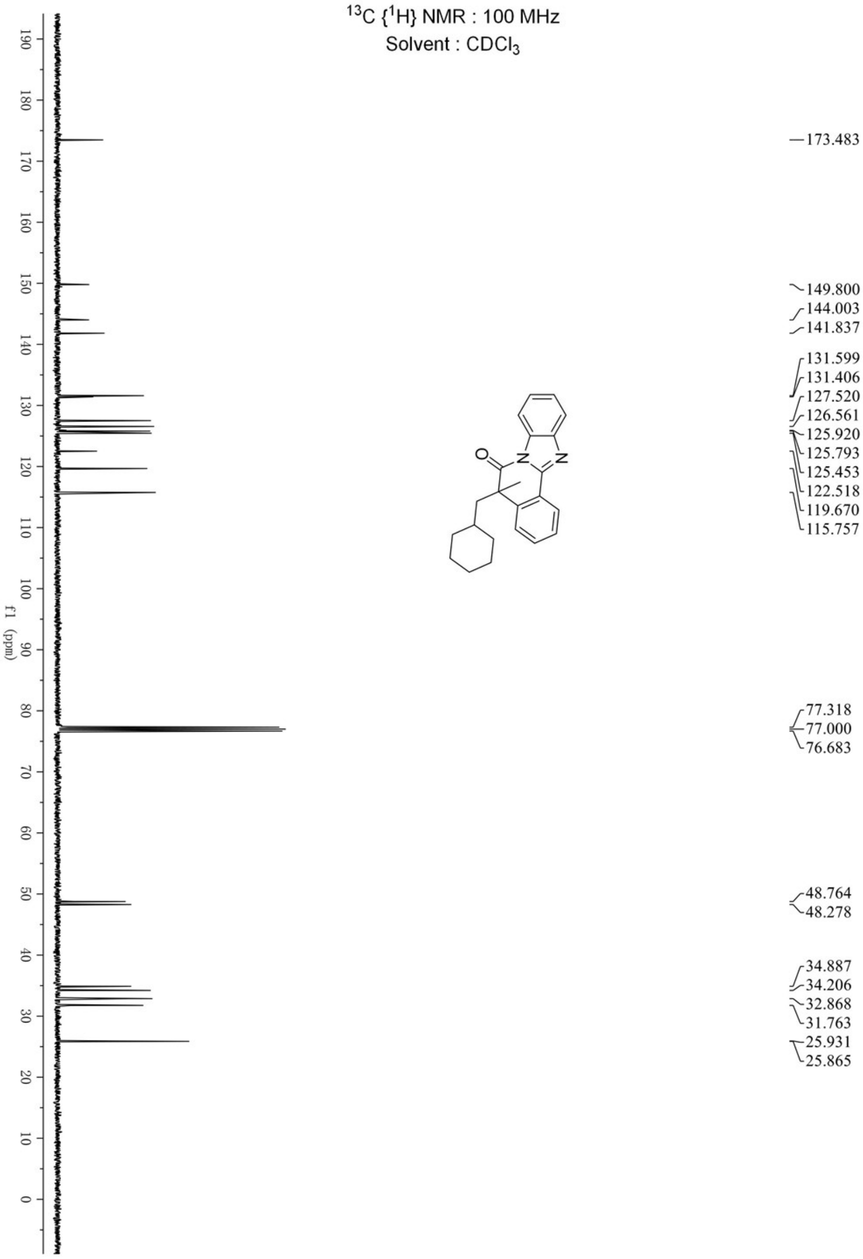
5-(Cyclohex



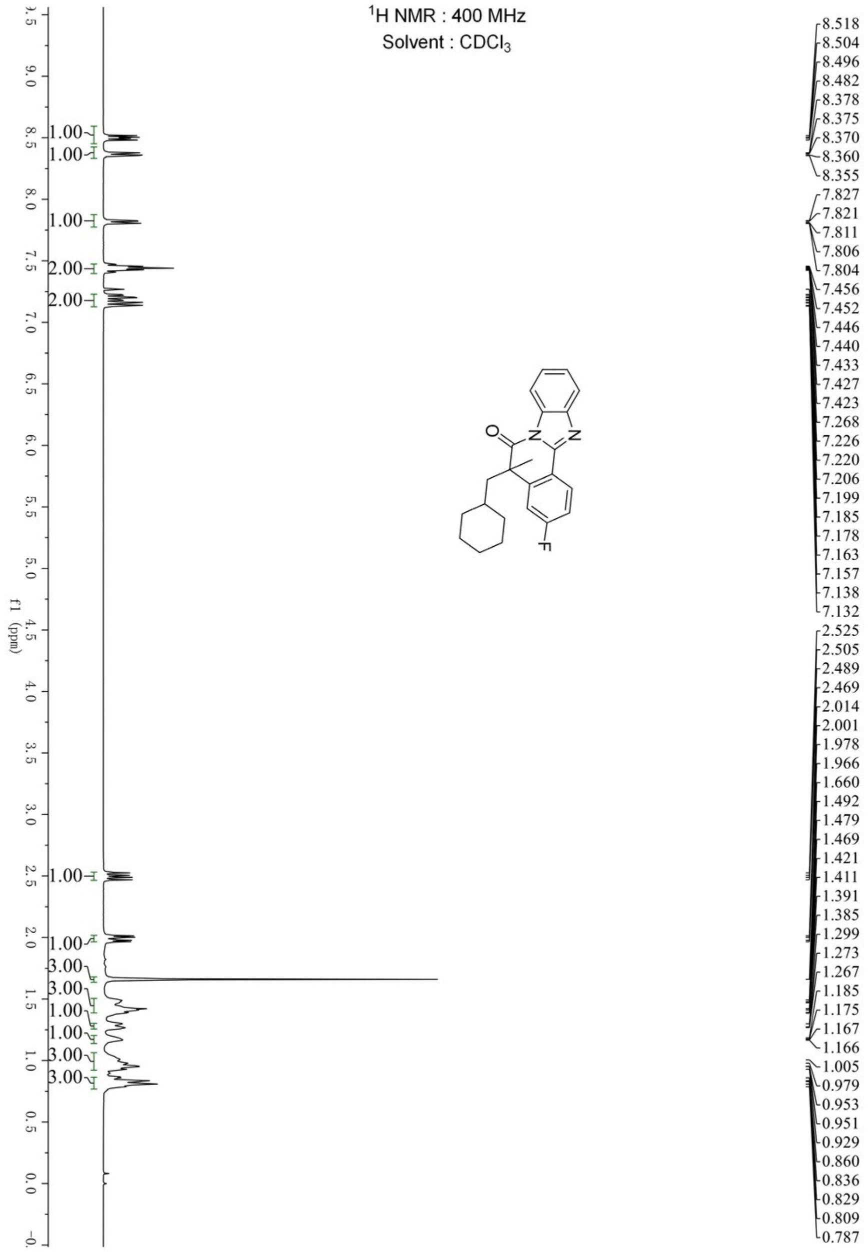
^{1}H NMR : 400 MHz
Solvent : $CDCl_3$
8.509
8.505
8.489
8.486
8.397
8.393
8.388
8.379
8.374
8.372
7.848
7.843
7.833
7.828
7.825
7.593
7.589
7.574
7.570
7.555
7.551
7.502
7.498
7.483
7.480
7.472
7.464
7.458
7.453
7.448
7.441
7.435
7.429
7.425
7.264
2.512
2.492
2.476
2.456
2.084
2.071
2.048
2.036
1.665
1.473
1.465
1.441
1.424
1.395
1.368
1.274
1.271
1.243
1.169
0.995
0.975
0.950
0.929
0.923
0.902
0.826
0.806
0.797
0.782
0.078
f1 (ppm)

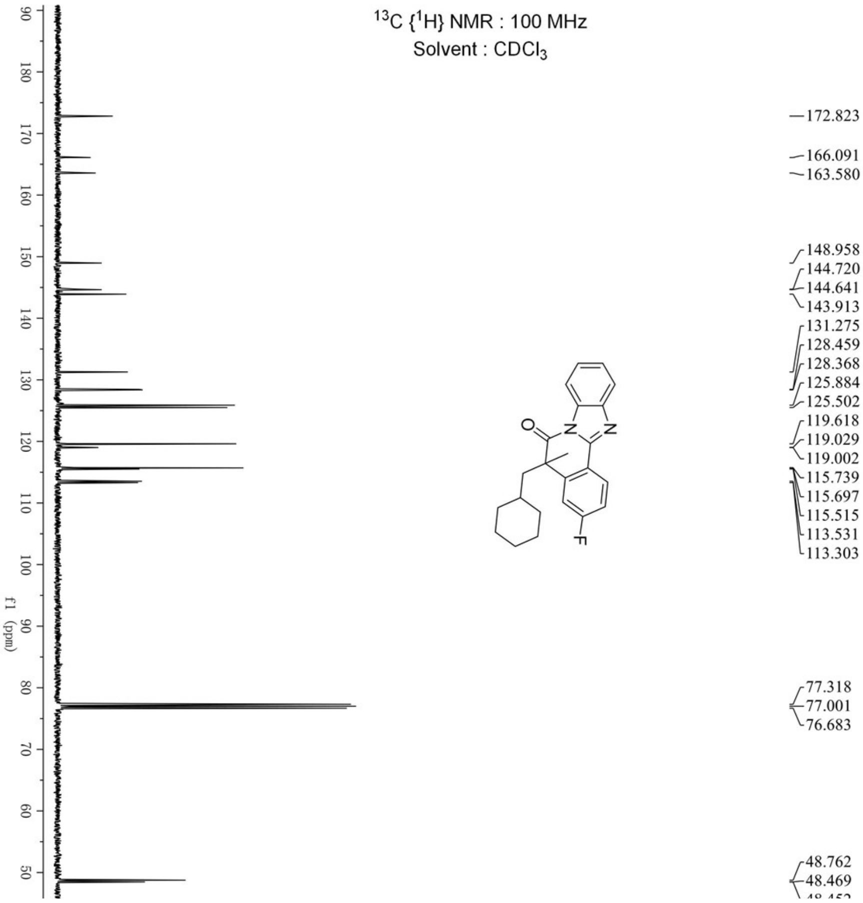
^{13}C {^{1}H} NMR : 100 MHz

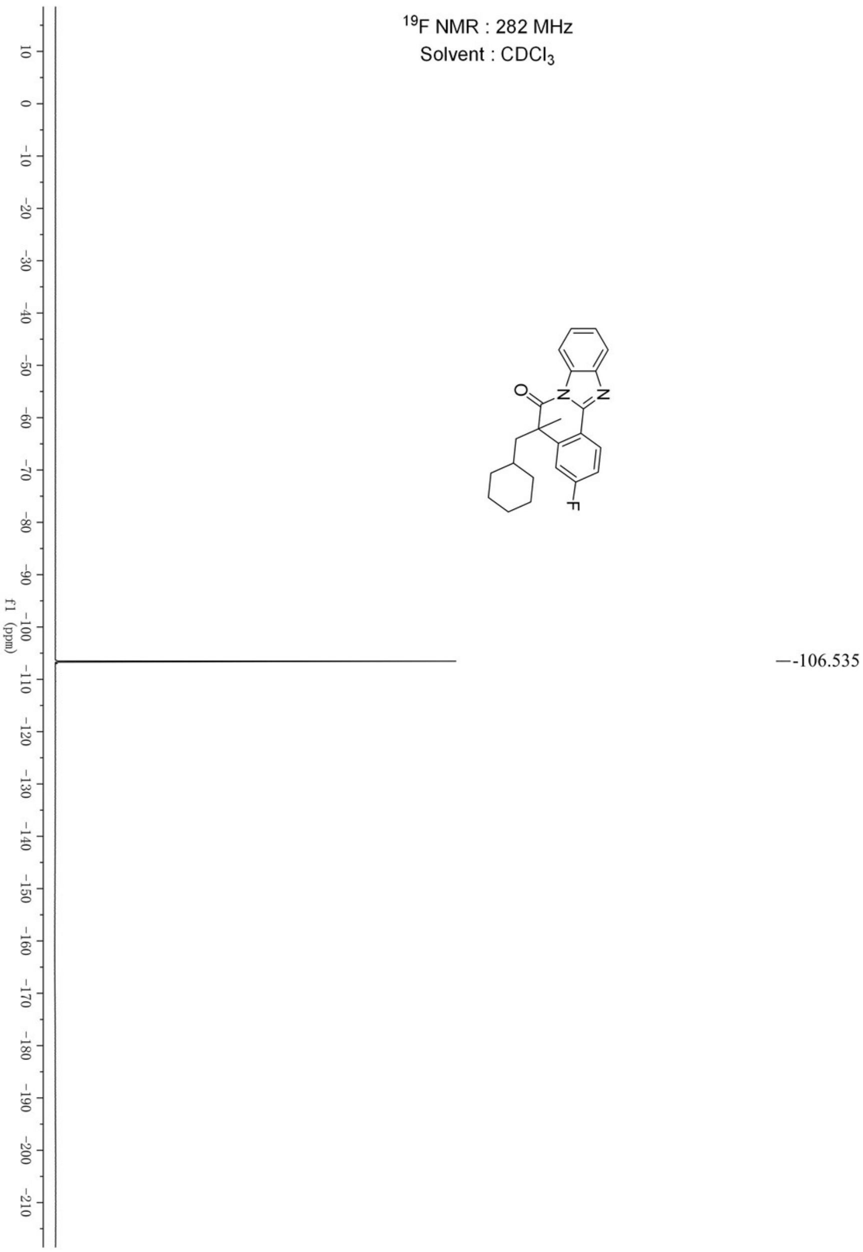
Solvent : $CDCl_3$



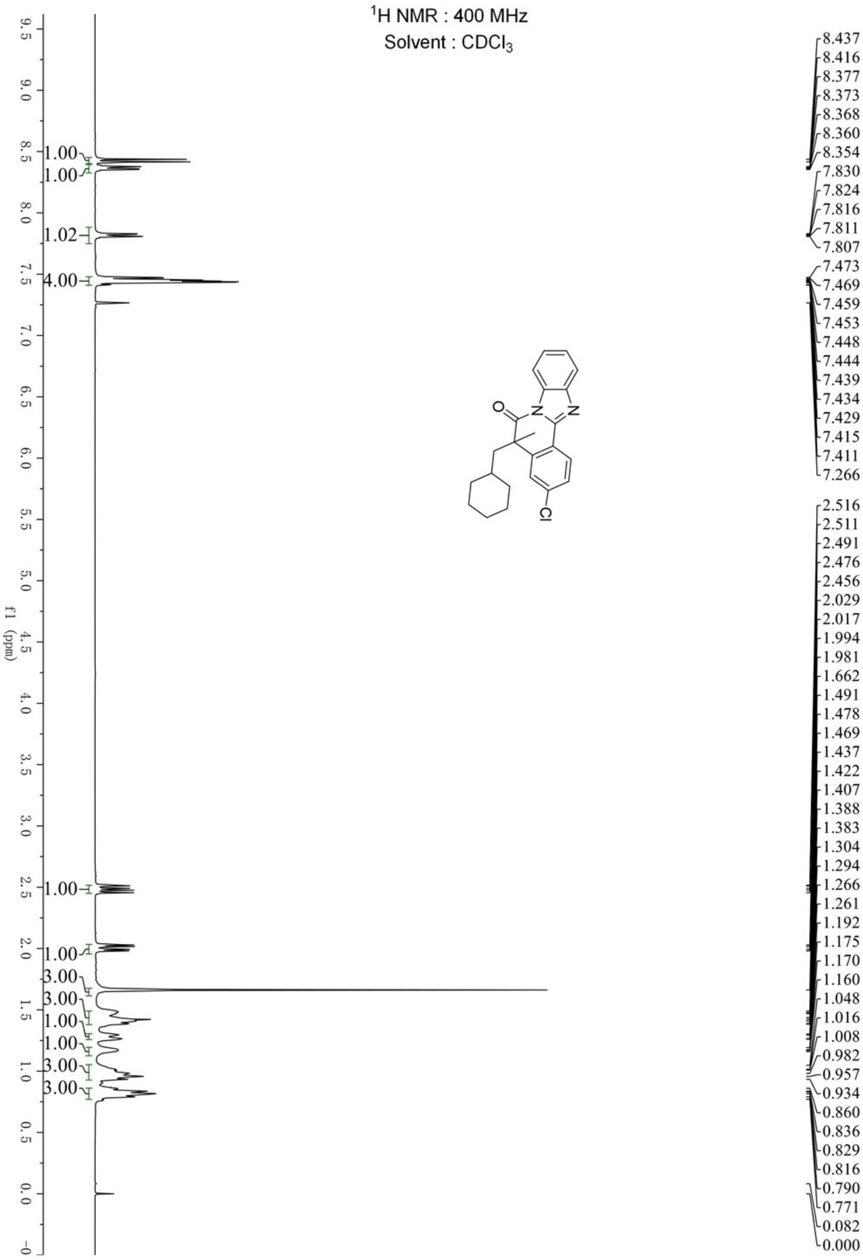
5-(Cyclohex


^{1}H NMR : 400 MHz
Solvent : $CDCl_3$
8.518
8.504
8.496
8.482
8.378
8.375
8.370
8.360
8.355
7.827
7.821
7.811
7.806
7.804
7.456
7.452
7.446
7.440
7.433
7.427
7.423
7.268
7.226
7.220
7.206
7.199
7.185
7.178
7.163
7.157
7.138
7.132
2.525
2.505
2.489
2.469
2.014
2.001
1.978
1.966
1.660
1.492
1.479
1.469
1.421
1.411
1.391
1.385
1.299
1.273
1.267
1.185
1.175
1.167
1.166
1.005
0.979
0.953
0.951
0.929
0.860
0.836
0.829
0.809
0.787
f1 (ppm)
N
N
O
F

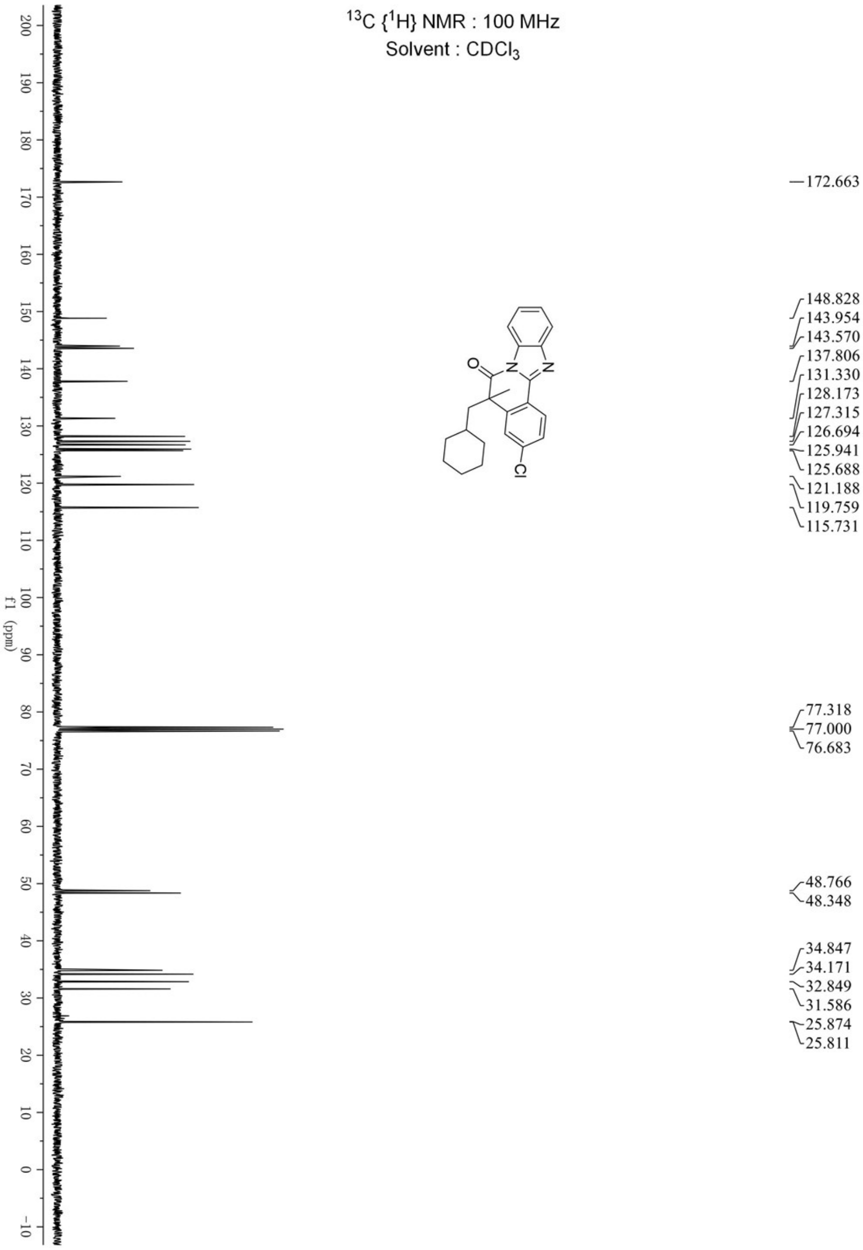

^{13}C {^{1}H} NMR : 100 MHz
Solvent : $CDCl_3$
172.823
166.091
163.580
148.958
144.720
144.641
143.913
131.275
128.459
128.368
125.884
125.502
119.618
119.029
119.002
115.739
115.697
115.515
113.531
113.303
77.318
77.001
76.683
48.762
48.469
f1 (ppm)
O
N
N
F


^{19}F NMR : 282 MHz
Solvent : $CDCl_3$
-106.535
f1 (ppm)
O
N
N
F

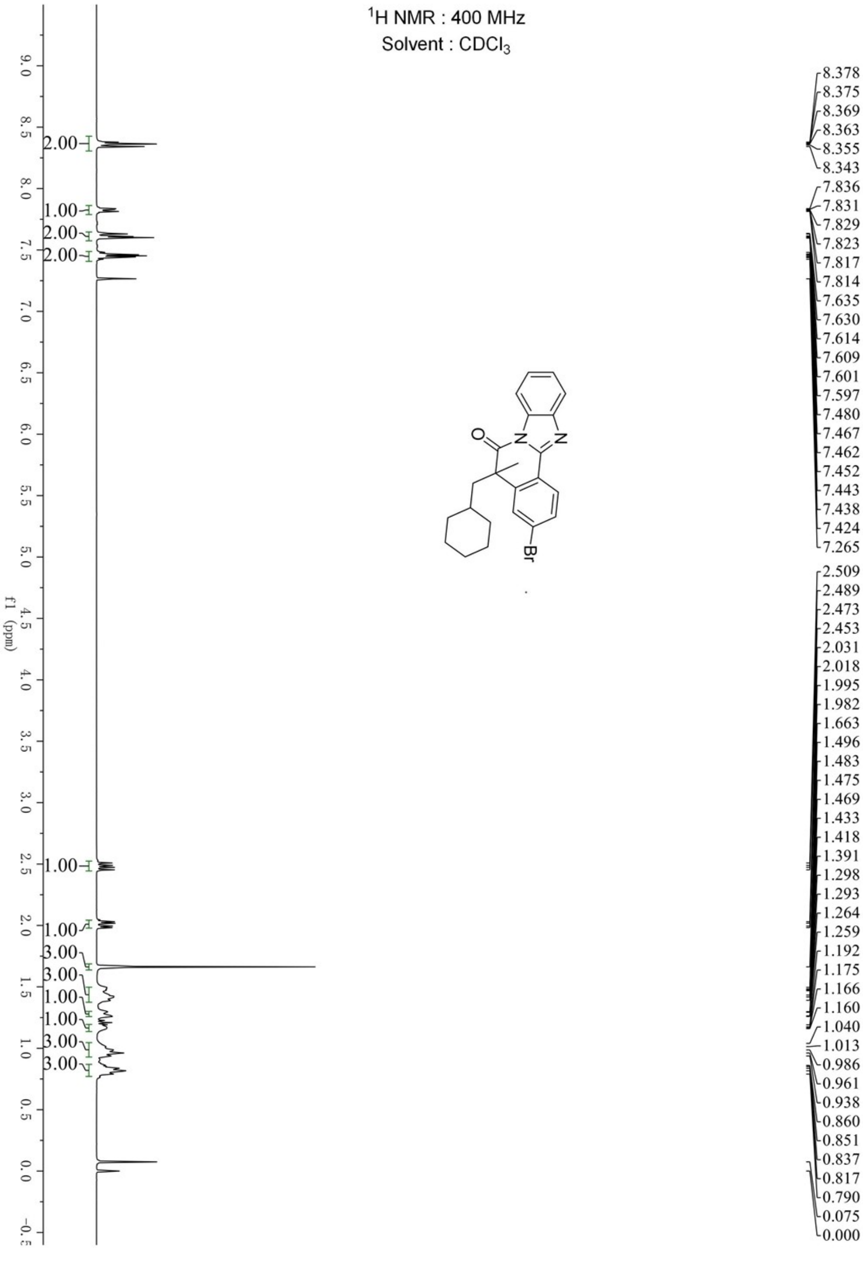
3-Chloro-5-

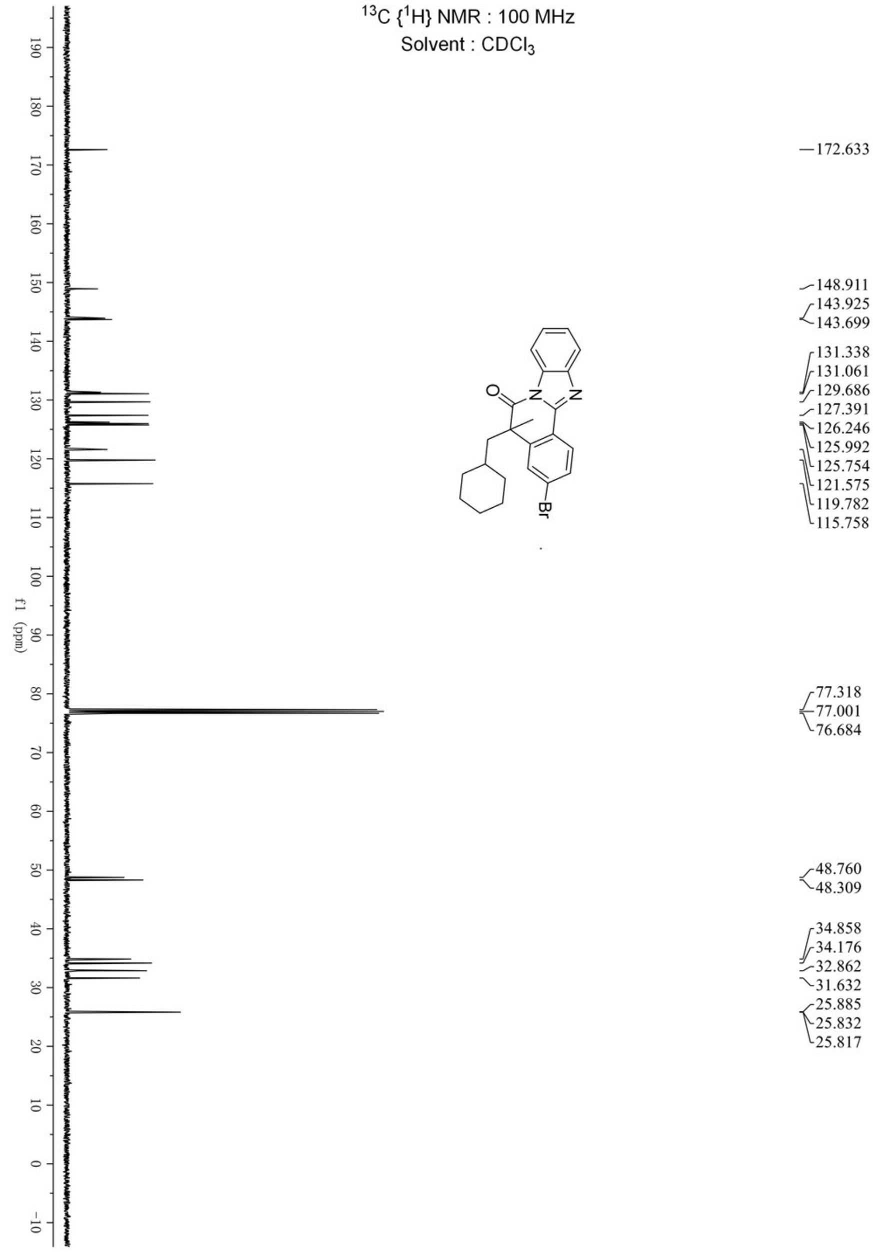


1H NMR : 400 MHz
Solvent : CDCl3
8.437
8.416
8.377
8.373
8.368
8.360
8.354
7.830
7.824
7.816
7.811
7.807
7.473
7.469
7.459
7.453
7.448
7.444
7.439
7.434
7.429
7.415
7.411
7.266
2.516
2.511
2.491
2.476
2.456
2.029
2.017
1.994
1.981
1.662
1.491
1.478
1.469
1.437
1.422
1.407
1.388
1.383
1.304
1.294
1.266
1.261
1.192
1.175
1.170
1.160
1.048
1.016
1.008
0.982
0.957
0.934
0.860
0.836
0.829
0.816
0.790
0.771
0.082
0.000
1.00
1.00
1.02
4.00
1.00
1.00
3.00
3.00
1.00
1.00
3.00
3.00
f1 (ppm)
Cl
O
N

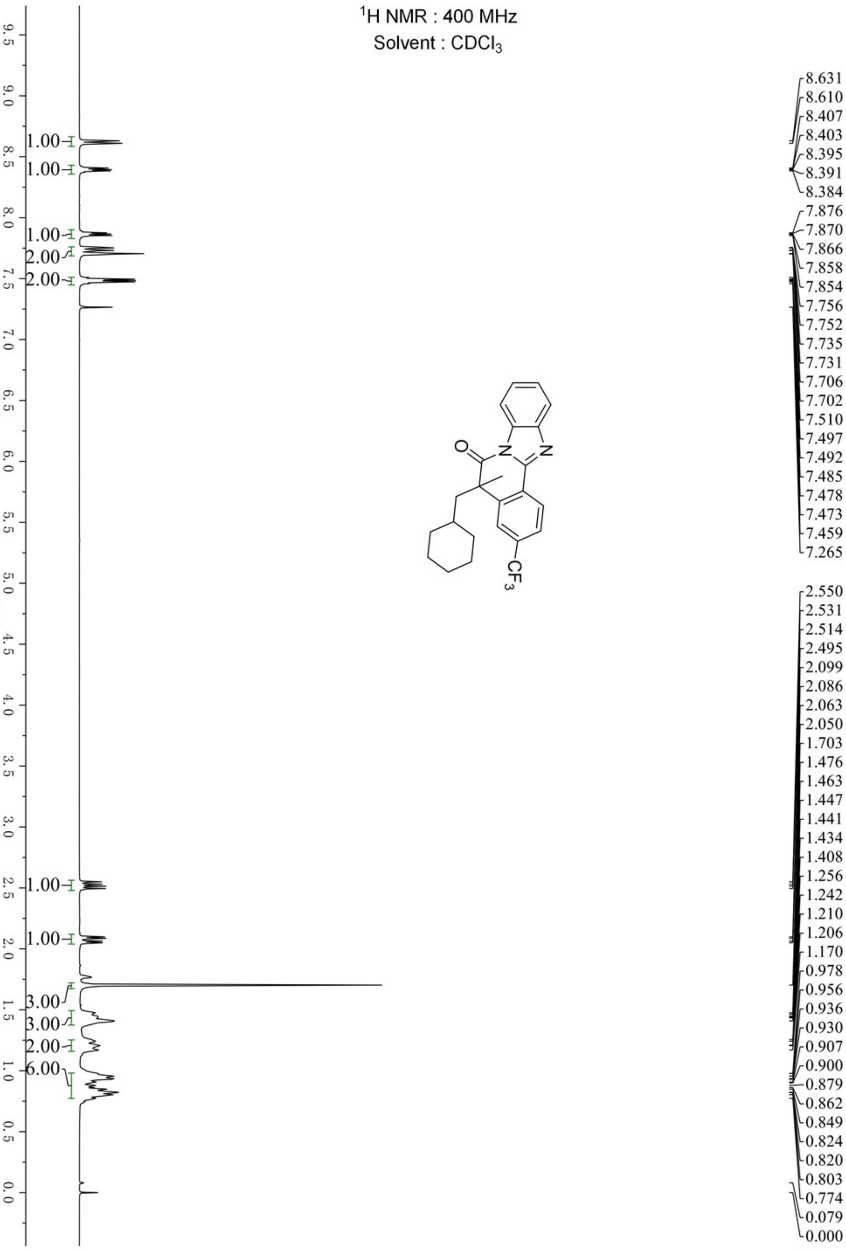

^{13}C {^{1}H} NMR : 100 MHz
Solvent : $CDCl_3$
172.663
148.828
143.954
143.570
137.806
131.330
128.173
127.315
126.694
125.941
125.688
121.188
119.759
115.731
77.318
77.000
76.683
48.766
48.348
34.847
34.171
32.849
31.586
25.874
25.811
O
N
N
Cl
f1 (ppm)

3-Bromo-5-

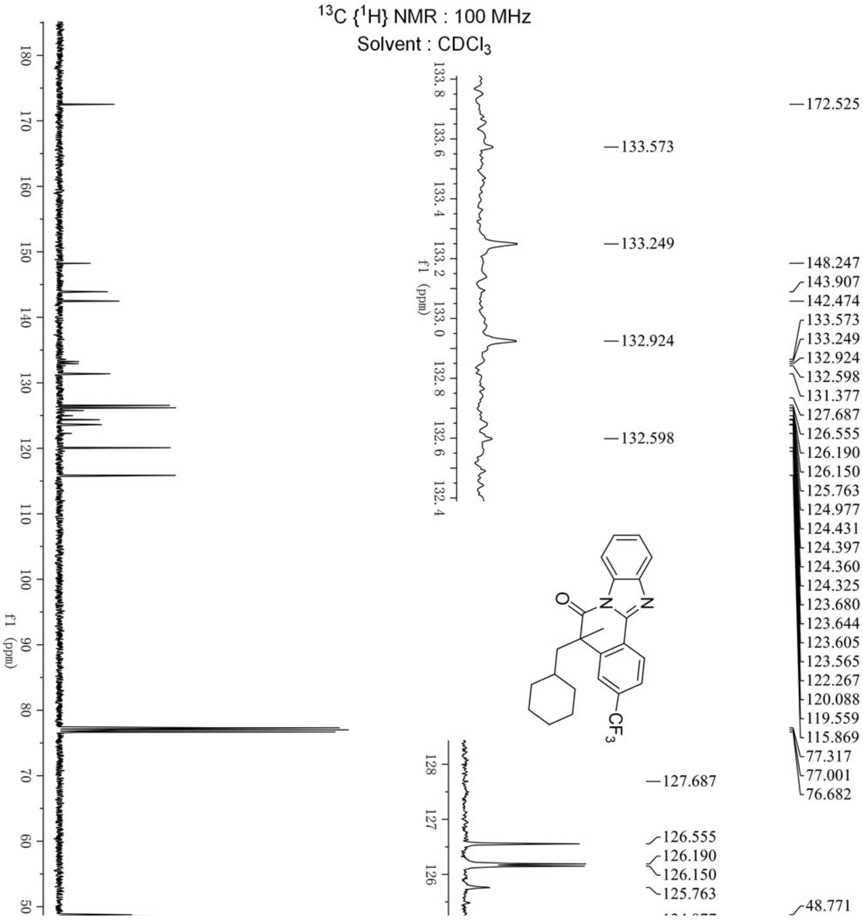

^{1}H NMR : 400 MHz
Solvent : $CDCl_3$
8.378
8.375
8.369
8.363
8.355
8.343
7.836
7.831
7.829
7.823
7.817
7.814
7.635
7.630
7.614
7.609
7.601
7.597
7.480
7.467
7.462
7.452
7.443
7.438
7.424
7.265
2.509
2.489
2.473
2.453
2.031
2.018
1.995
1.982
1.663
1.496
1.483
1.475
1.469
1.433
1.418
1.391
1.298
1.293
1.264
1.259
1.192
1.175
1.166
1.160
1.040
1.013
0.986
0.961
0.938
0.860
0.851
0.837
0.817
0.790
0.075
0.000
O
N
N
Br
f1 (ppm)

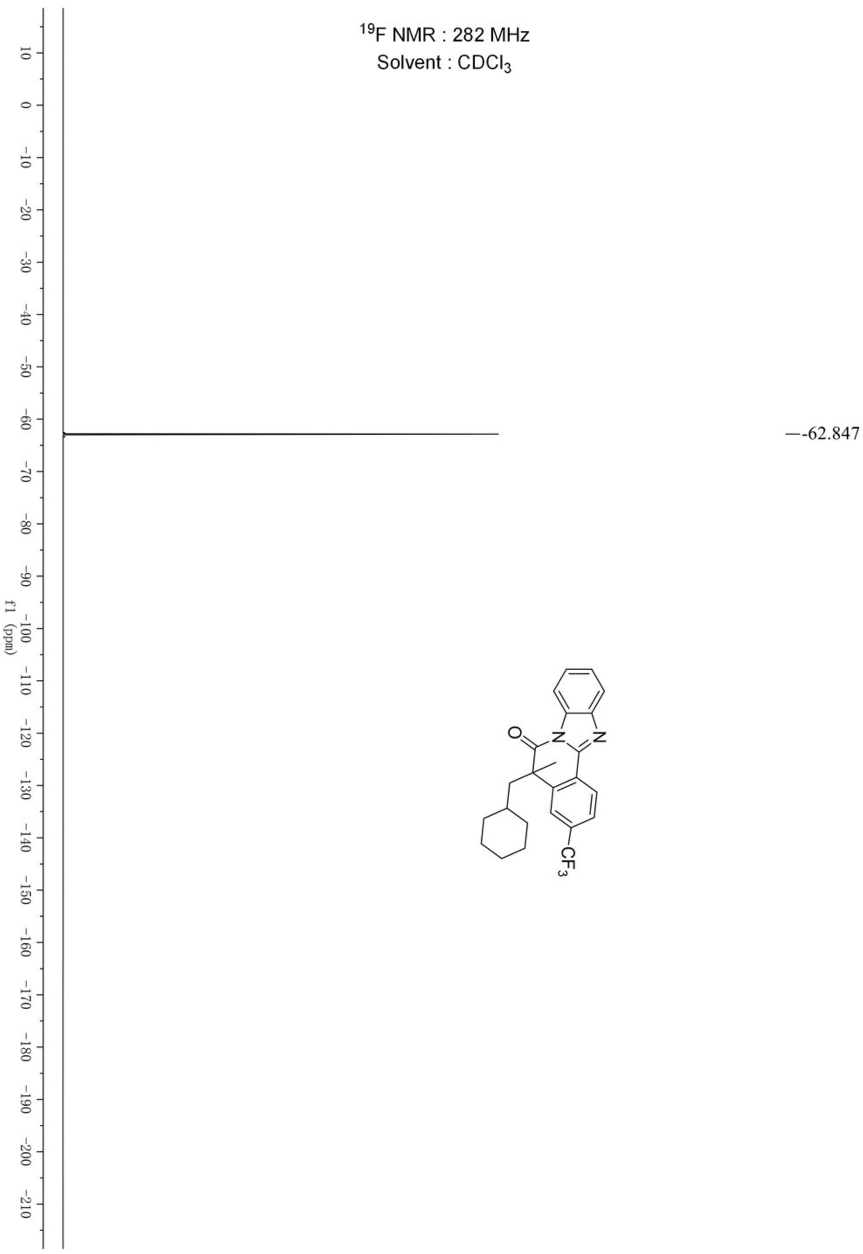

^{13}C {^{1}H} NMR : 100 MHz
Solvent : $CDCl_3$
f1 (ppm)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
172.633
148.911
143.925
143.699
131.338
131.061
129.686
127.391
126.246
125.992
125.754
121.575
119.782
115.758
77.318
77.001
76.684
48.760
48.309
34.858
34.176
32.862
31.632
25.885
25.832
25.817
O
N
N
Br

5-(Cycloh

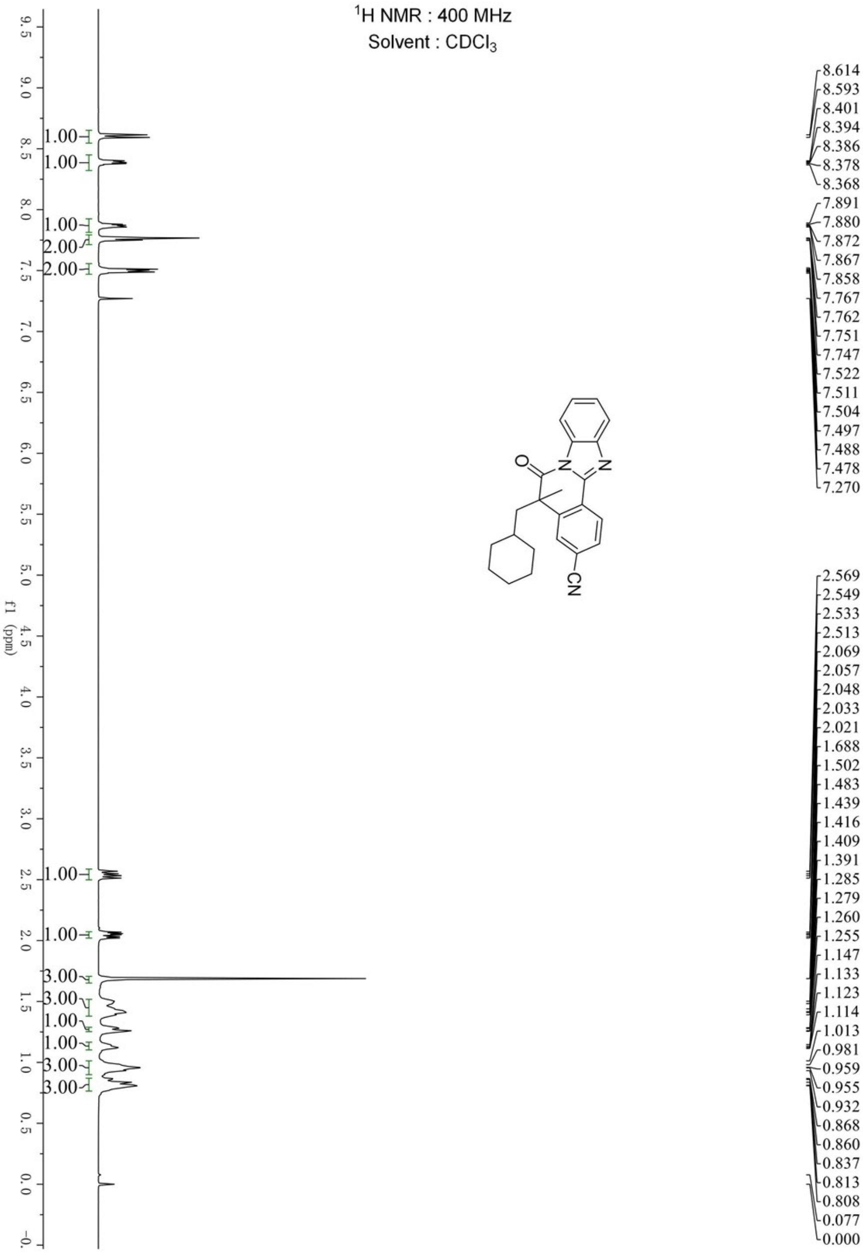


^{1}H NMR : 400 MHz
Solvent : CDCl$_3$
8.631
8.610
8.407
8.403
8.395
8.391
8.384
7.876
7.870
7.866
7.858
7.854
7.756
7.752
7.735
7.731
7.706
7.702
7.510
7.497
7.492
7.485
7.478
7.473
7.459
7.265
2.550
2.531
2.514
2.495
2.099
2.086
2.063
2.050
1.703
1.476
1.463
1.447
1.441
1.434
1.408
1.256
1.242
1.210
1.206
1.170
0.978
0.956
0.936
0.930
0.907
0.900
0.879
0.862
0.849
0.824
0.820
0.803
0.774
0.079
0.000
f1 (ppm)

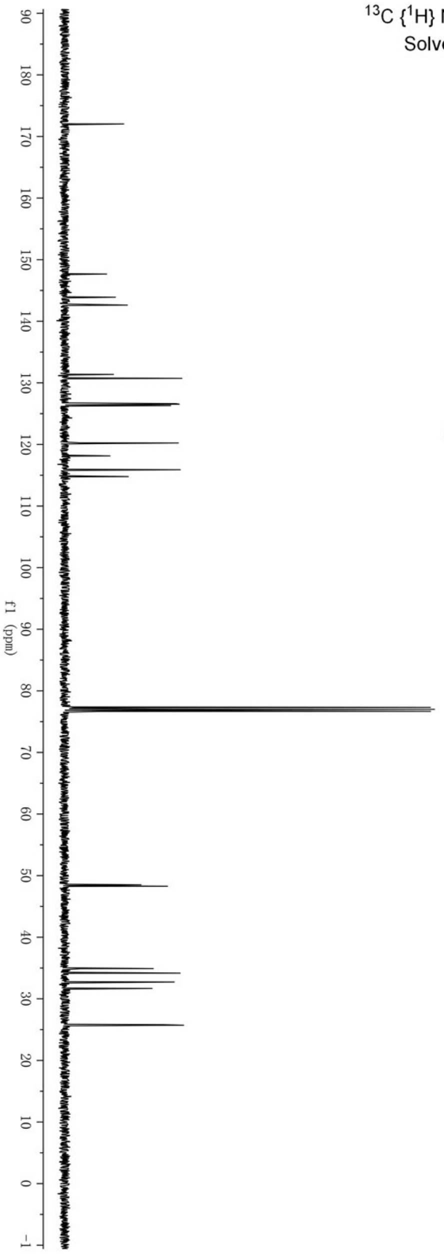

13C {1H} NMR : 100 MHz
Solvent : CDCl3
172.525
148.247
143.907
142.474
133.573
133.249
132.924
132.598
131.377
127.687
126.555
126.190
126.150
125.763
124.977
124.431
124.397
124.360
124.325
123.680
123.644
123.605
123.565
122.267
120.088
119.559
115.869
77.317
77.001
76.682
48.771
f1 (ppm)
N
N
O
CF3

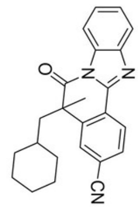

19F NMR : 282 MHz
Solvent : CDCl3
-62.847
f1 (ppm)
N
N
O
CF3

5-(Cycloh


^{1}H NMR : 400 MHz
Solvent : $CDCl_3$
8.614
8.593
8.401
8.394
8.386
8.378
8.368
7.891
7.880
7.872
7.867
7.858
7.767
7.762
7.751
7.747
7.522
7.511
7.504
7.497
7.488
7.478
7.270
2.569
2.549
2.533
2.513
2.069
2.057
2.048
2.033
2.021
1.688
1.502
1.483
1.439
1.416
1.409
1.391
1.285
1.279
1.260
1.255
1.147
1.133
1.123
1.114
1.013
0.981
0.959
0.955
0.932
0.868
0.860
0.837
0.813
0.808
0.077
0.000
CN
f1 (ppm)

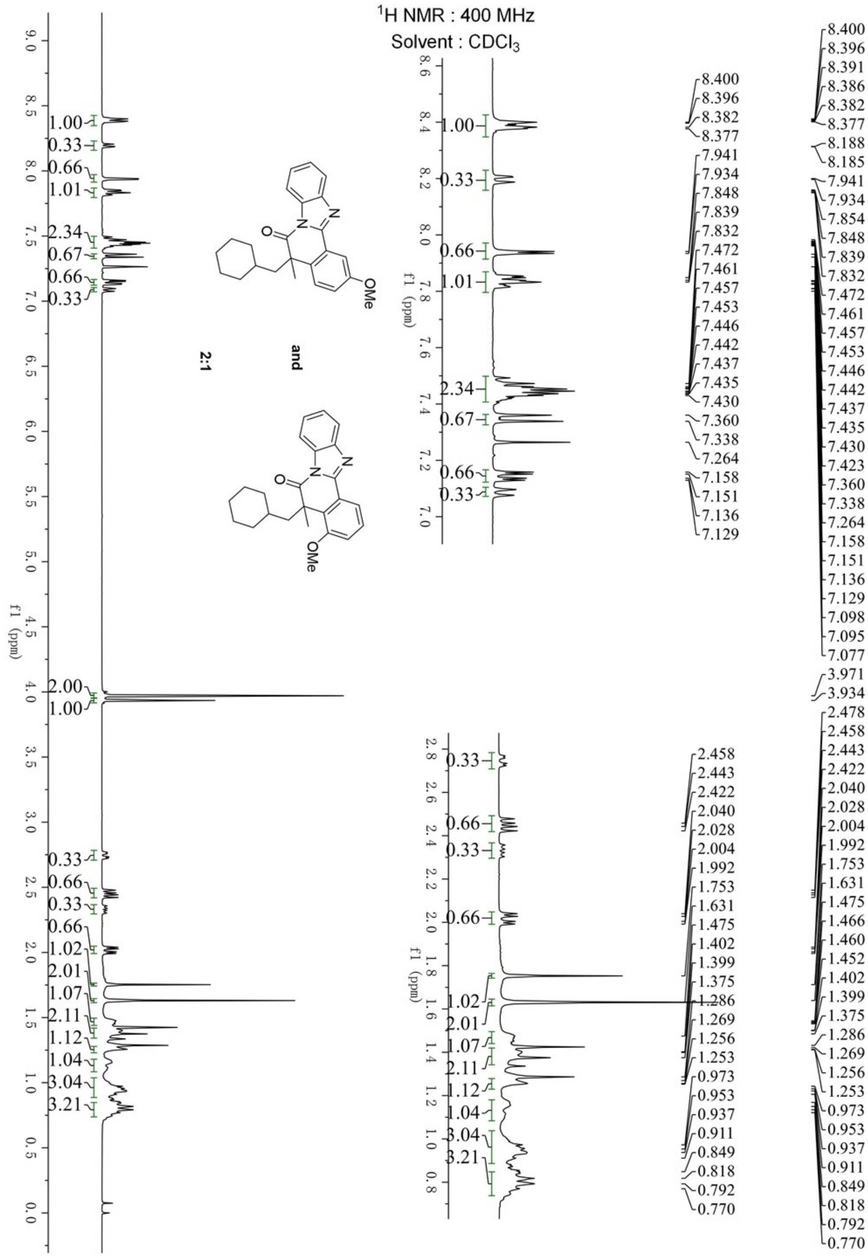
$^{13}C\{^1H\}$ NMR : 100 MHz
Solvent : $CDCl_3$



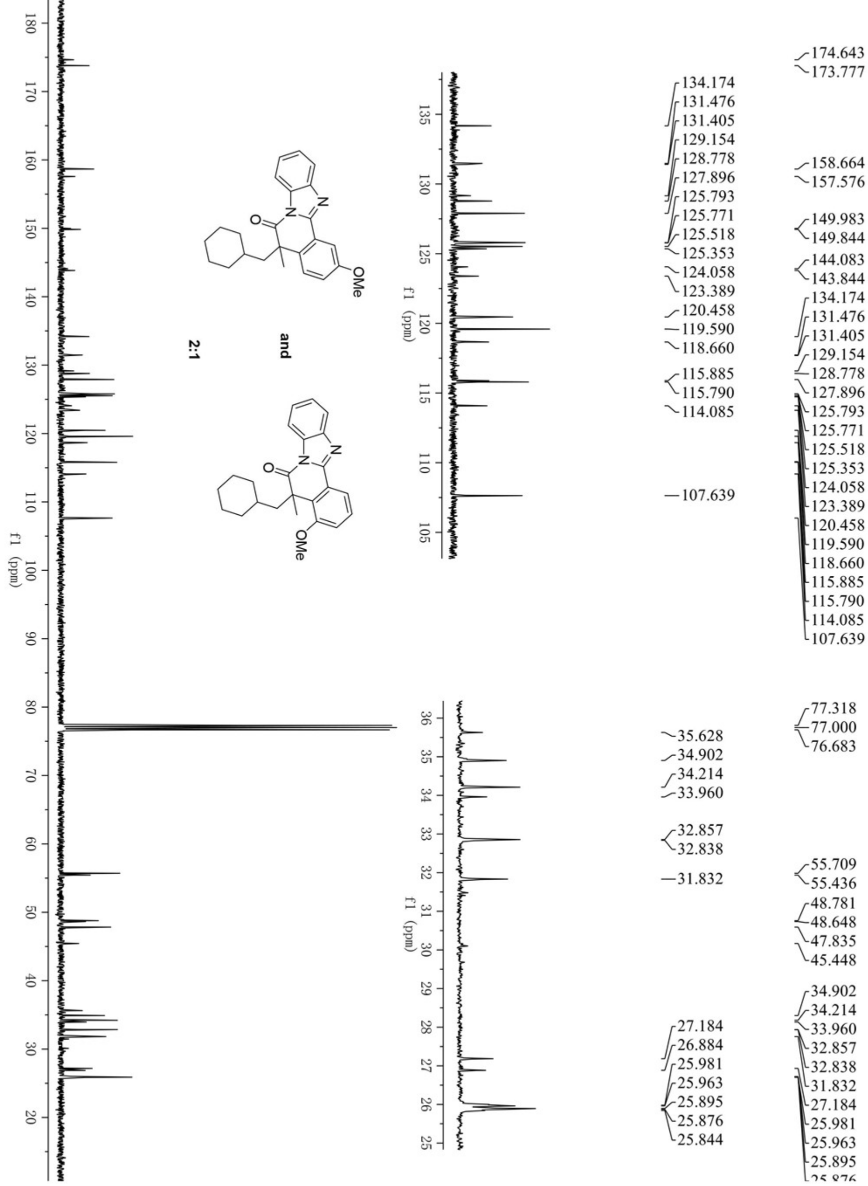


5-(Cyclohexylmethyl)-2-methoxy-5-methylbenzo[4,5]imidazo[2,1-*a*]isoquinolin-6(5*H*)-one (3aea)

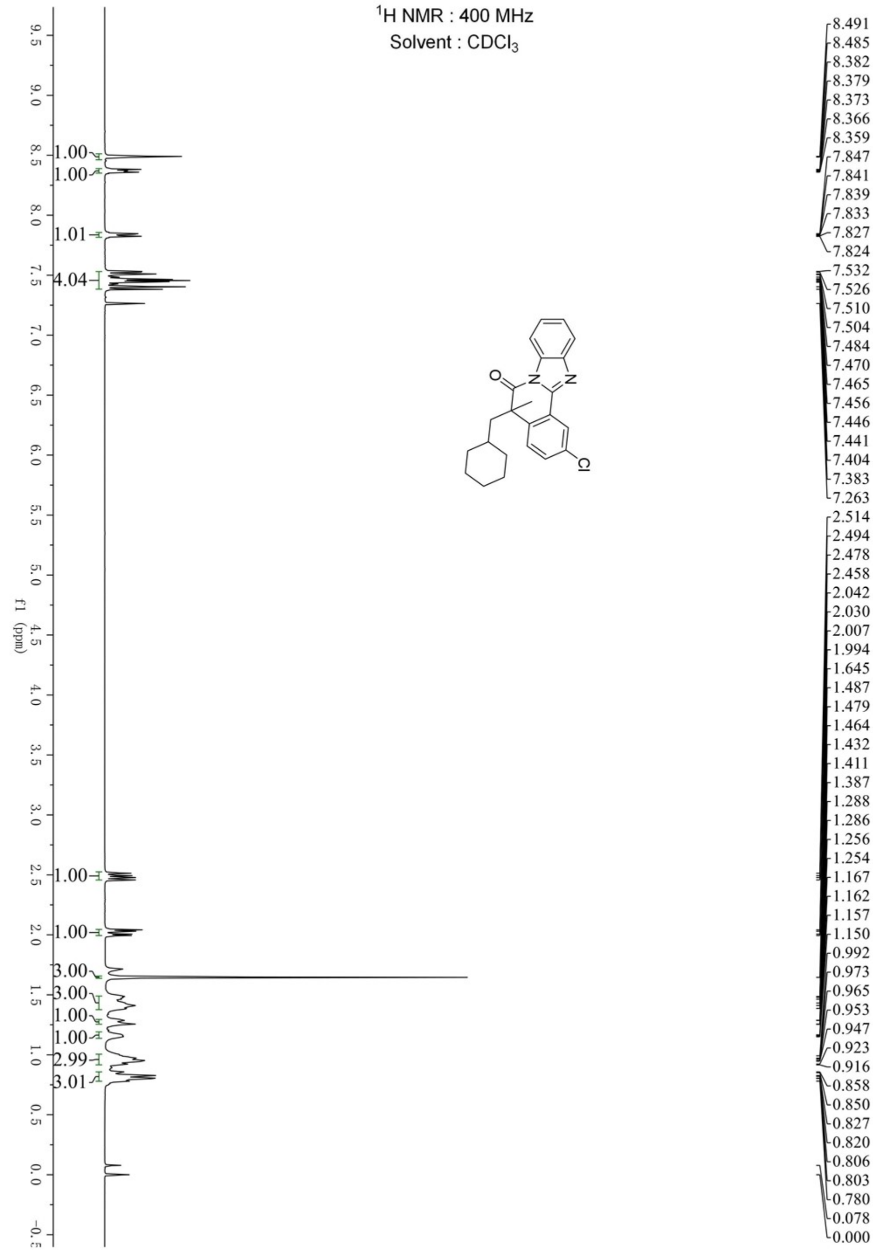
5-(Cyclohexy



$^{13}C\ \{^1H\}$ NMR : 100 MHz
Solvent : $CDCl_3$

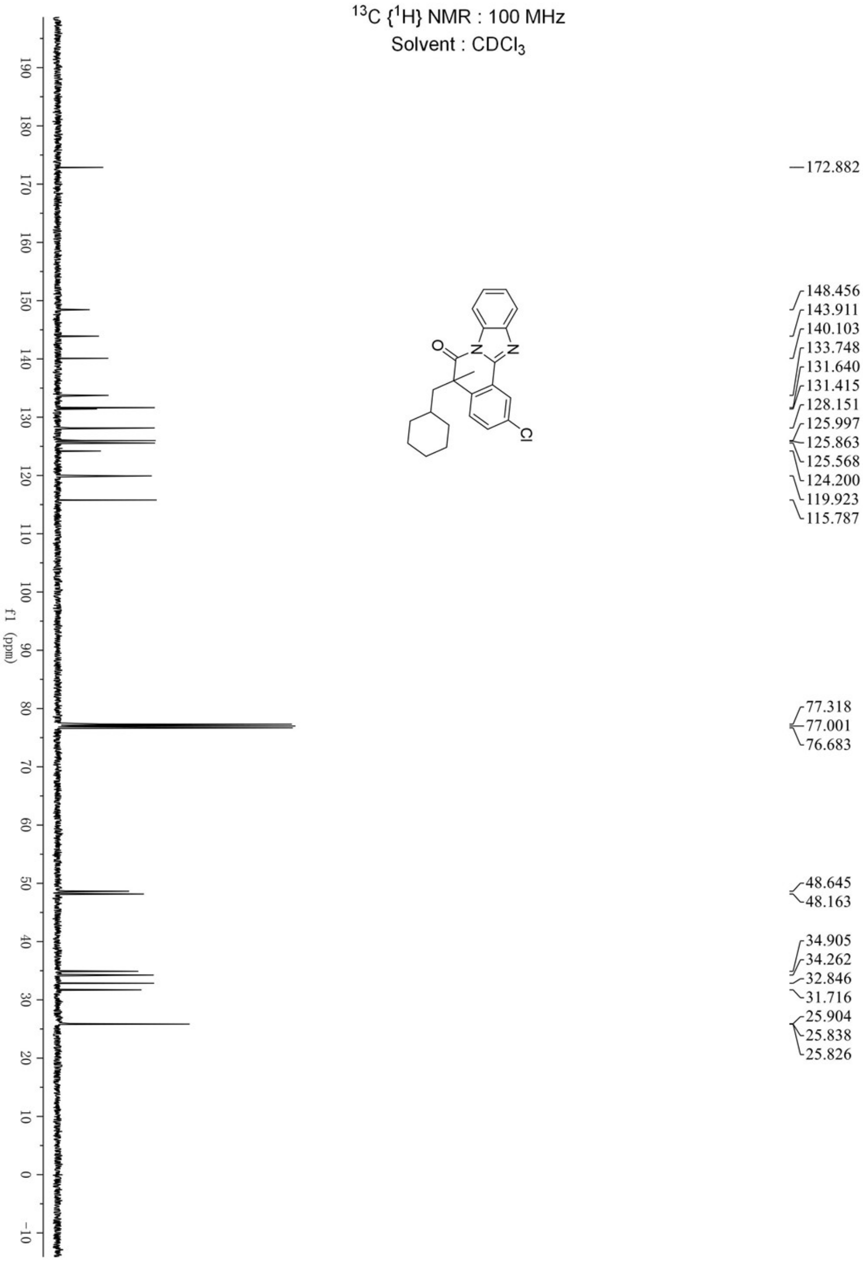


2-Chloro-5-

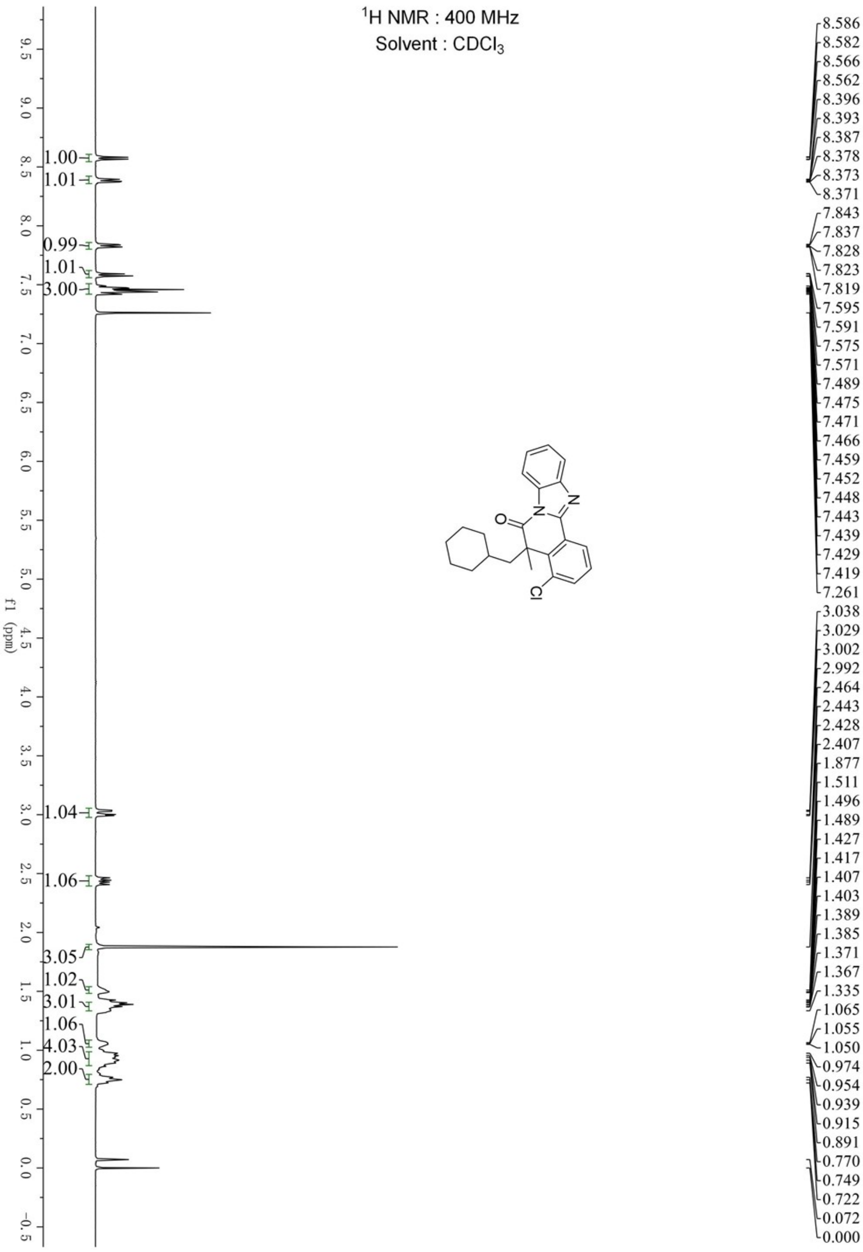


1H NMR : 400 MHz
Solvent : CDCl3
8.491
8.485
8.382
8.379
8.373
8.366
8.359
7.847
7.841
7.839
7.833
7.827
7.824
7.532
7.526
7.510
7.504
7.484
7.470
7.465
7.456
7.446
7.441
7.404
7.383
7.263
2.514
2.494
2.478
2.458
2.042
2.030
2.007
1.994
1.645
1.487
1.479
1.464
1.432
1.411
1.387
1.288
1.286
1.256
1.254
1.167
1.162
1.157
1.150
0.992
0.973
0.965
0.953
0.947
0.923
0.916
0.858
0.850
0.827
0.820
0.806
0.803
0.780
0.078
0.000
f1 (ppm)

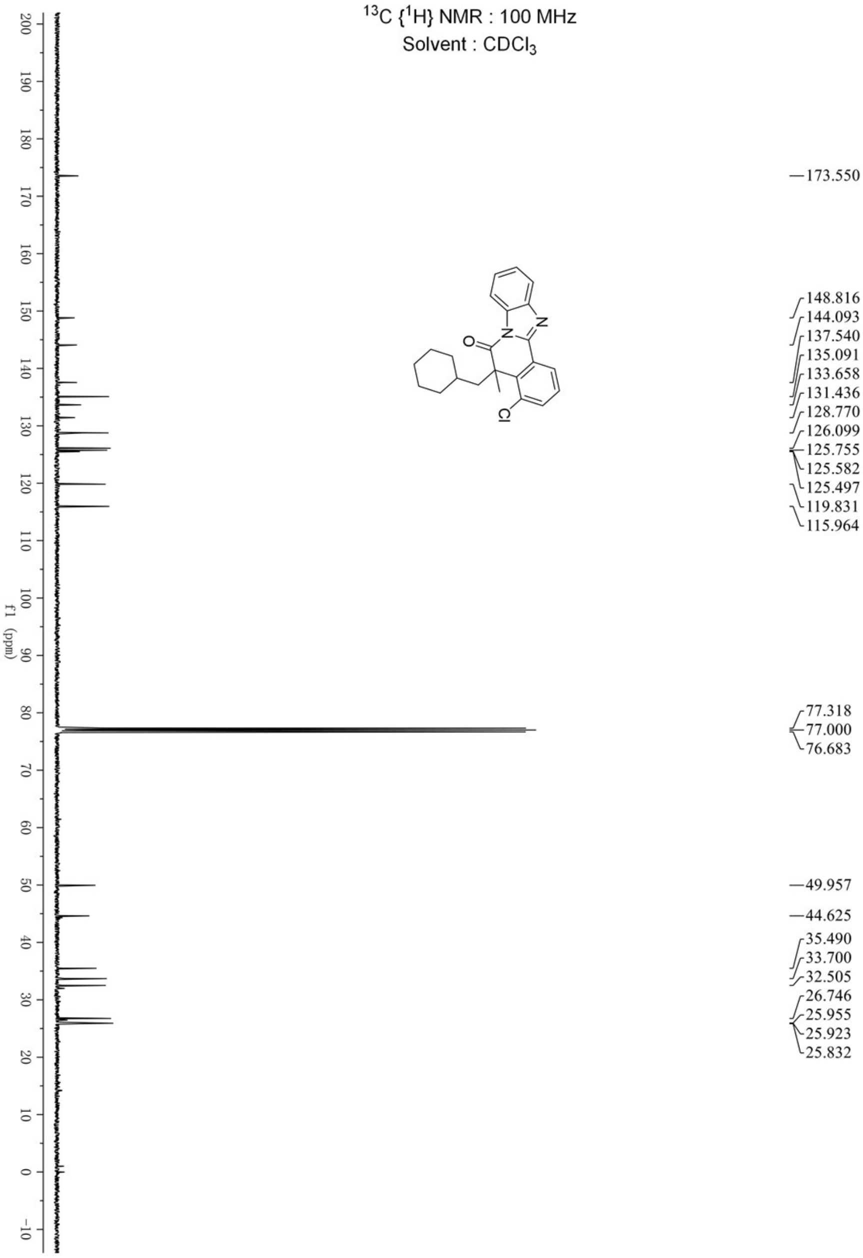
$^{13}C\{^{1}H\}$ NMR : 100 MHz
Solvent : $CDCl_3$



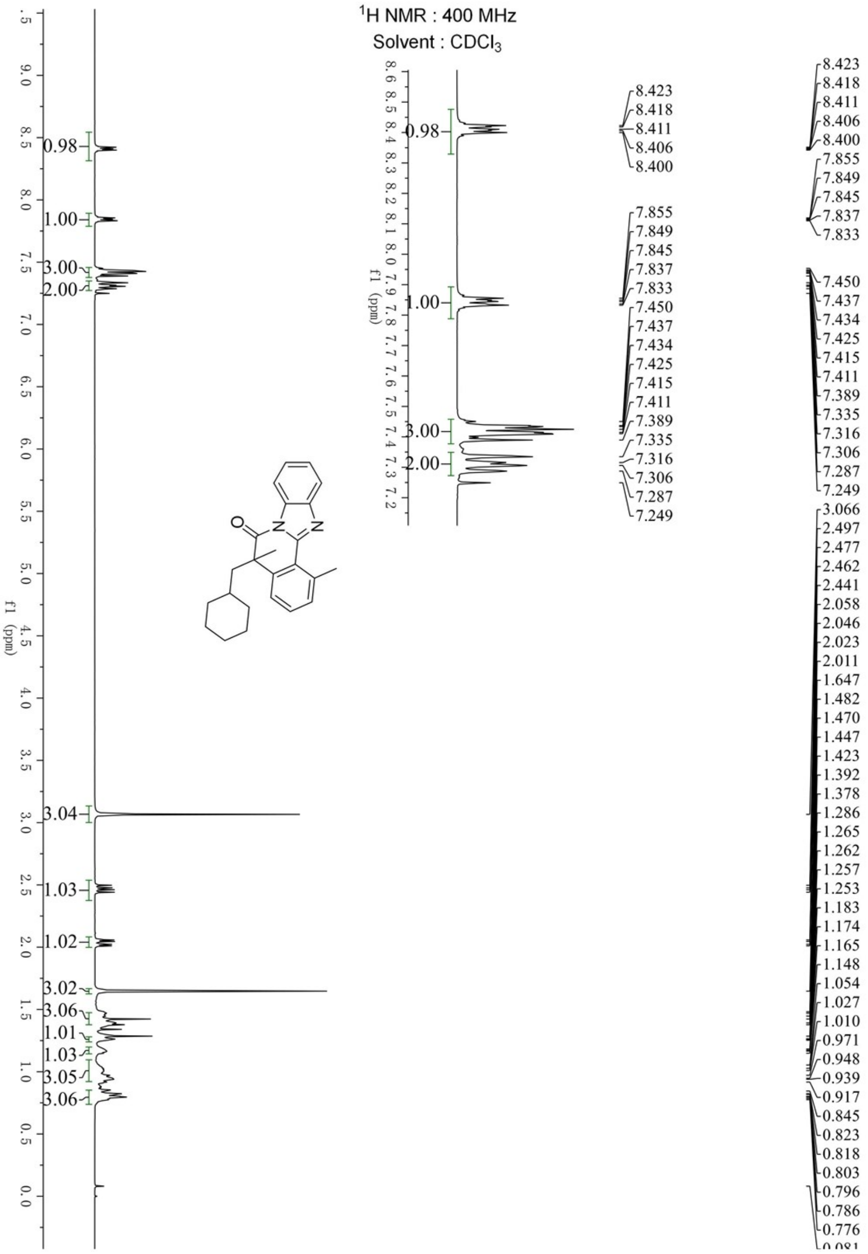
4-Chloro-5-



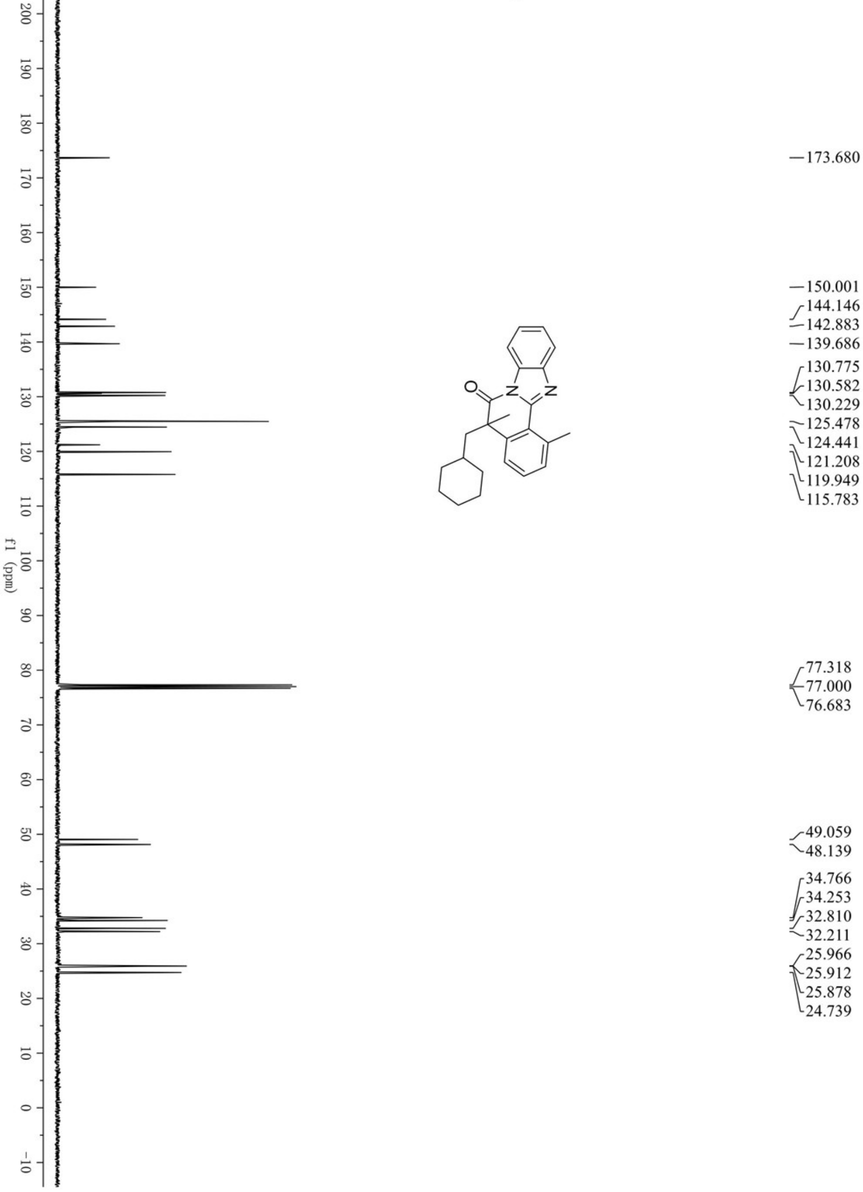
1H NMR : 400 MHz
Solvent : CDCl3
f1 (ppm)
Cl
O
N
N
8.586
8.582
8.566
8.562
8.396
8.393
8.387
8.378
8.373
8.371
7.843
7.837
7.828
7.823
7.819
7.595
7.591
7.575
7.571
7.489
7.475
7.471
7.466
7.459
7.452
7.448
7.443
7.439
7.429
7.419
7.261
3.038
3.029
3.002
2.992
2.464
2.443
2.428
2.407
1.877
1.511
1.496
1.489
1.427
1.417
1.407
1.403
1.389
1.385
1.371
1.367
1.335
1.065
1.055
1.050
0.974
0.954
0.939
0.915
0.891
0.770
0.749
0.722
0.072
0.000


$^{13}C\{^1H\}$ NMR : 100 MHz
Solvent : $CDCl_3$
173.550
148.816
144.093
137.540
135.091
133.658
131.436
128.770
126.099
125.755
125.582
125.497
119.831
115.964
77.318
77.000
76.683
49.957
44.625
35.490
33.700
32.505
26.746
25.955
25.923
25.832
f1 (ppm)
N
N
O
Cl

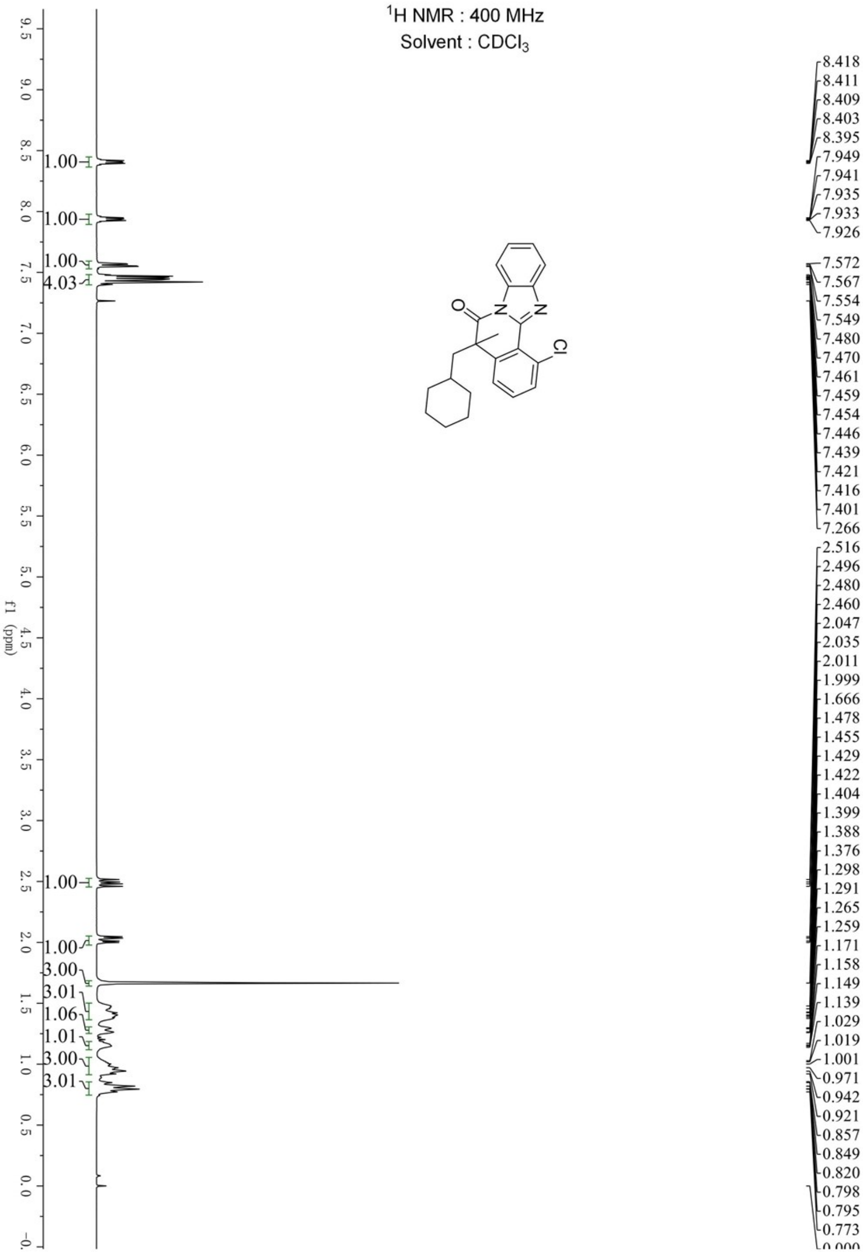
5-(Cyclohex



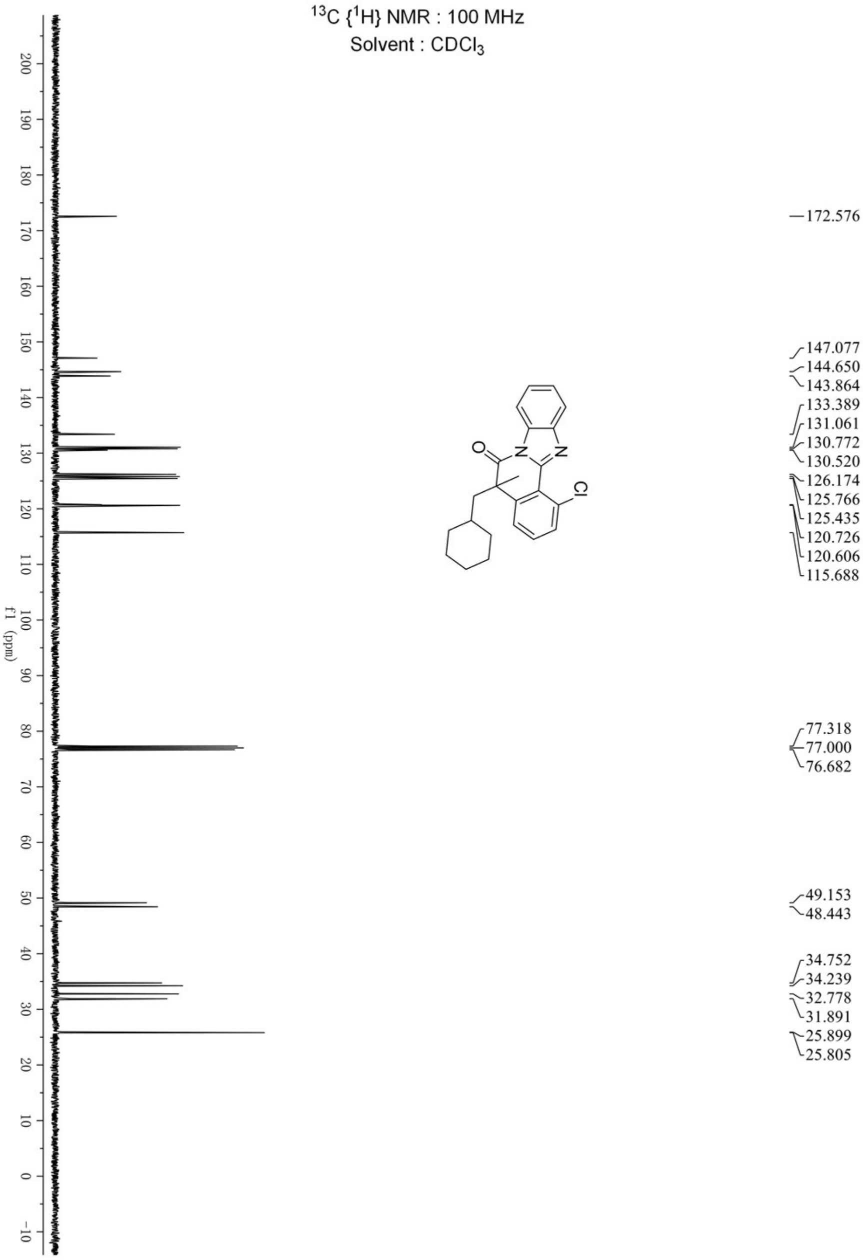
^{13}C {^{1}H} NMR : 100 MHz
Solvent : $CDCl_3$



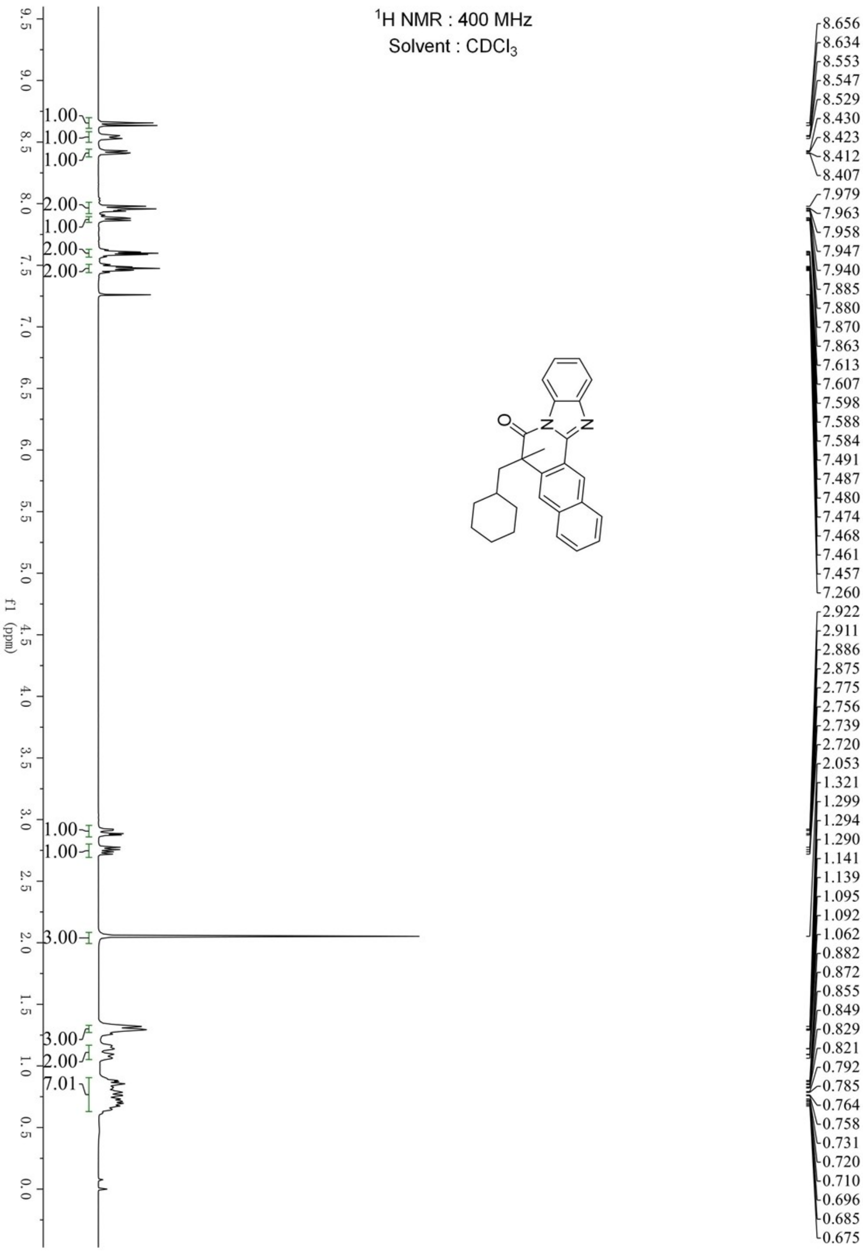
1-Chloro-5-



$^{13}C\{^1H\}$ NMR : 100 MHz
Solvent : $CDCl_3$

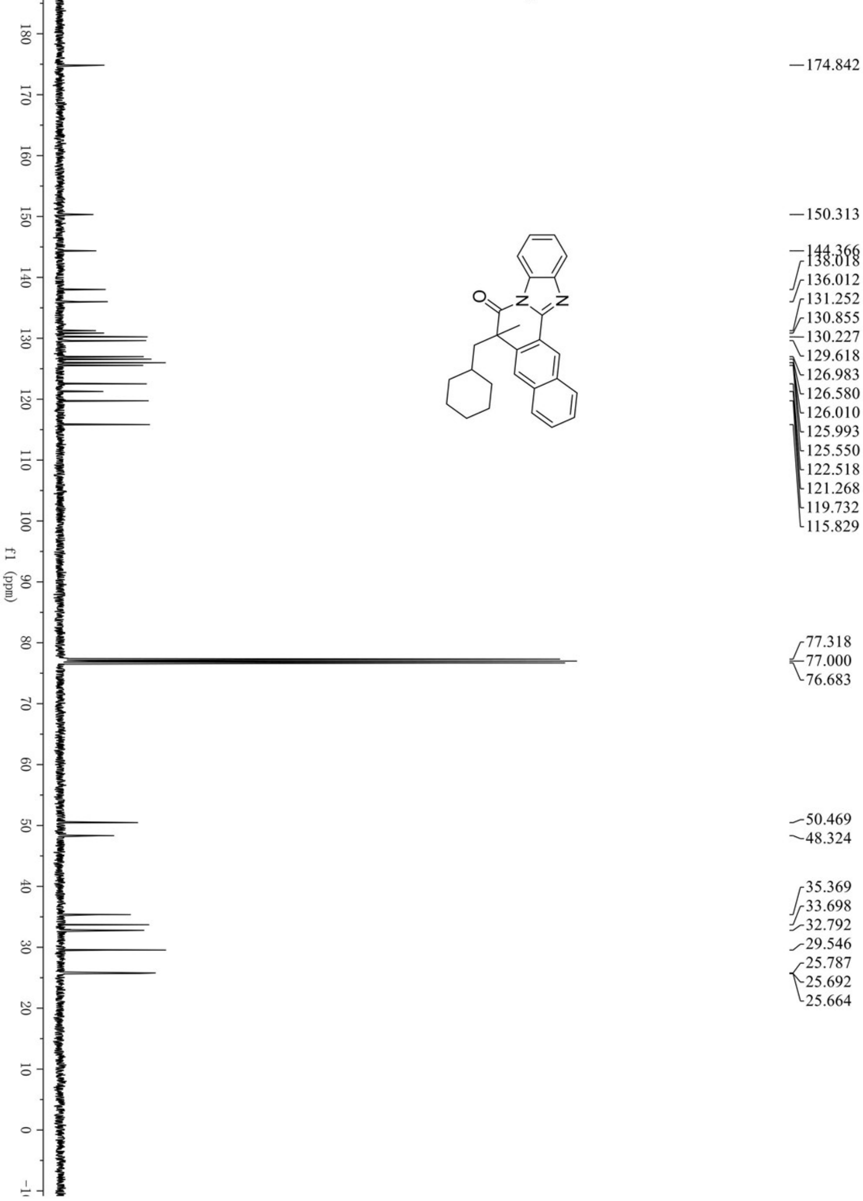


7-(Cyclohe

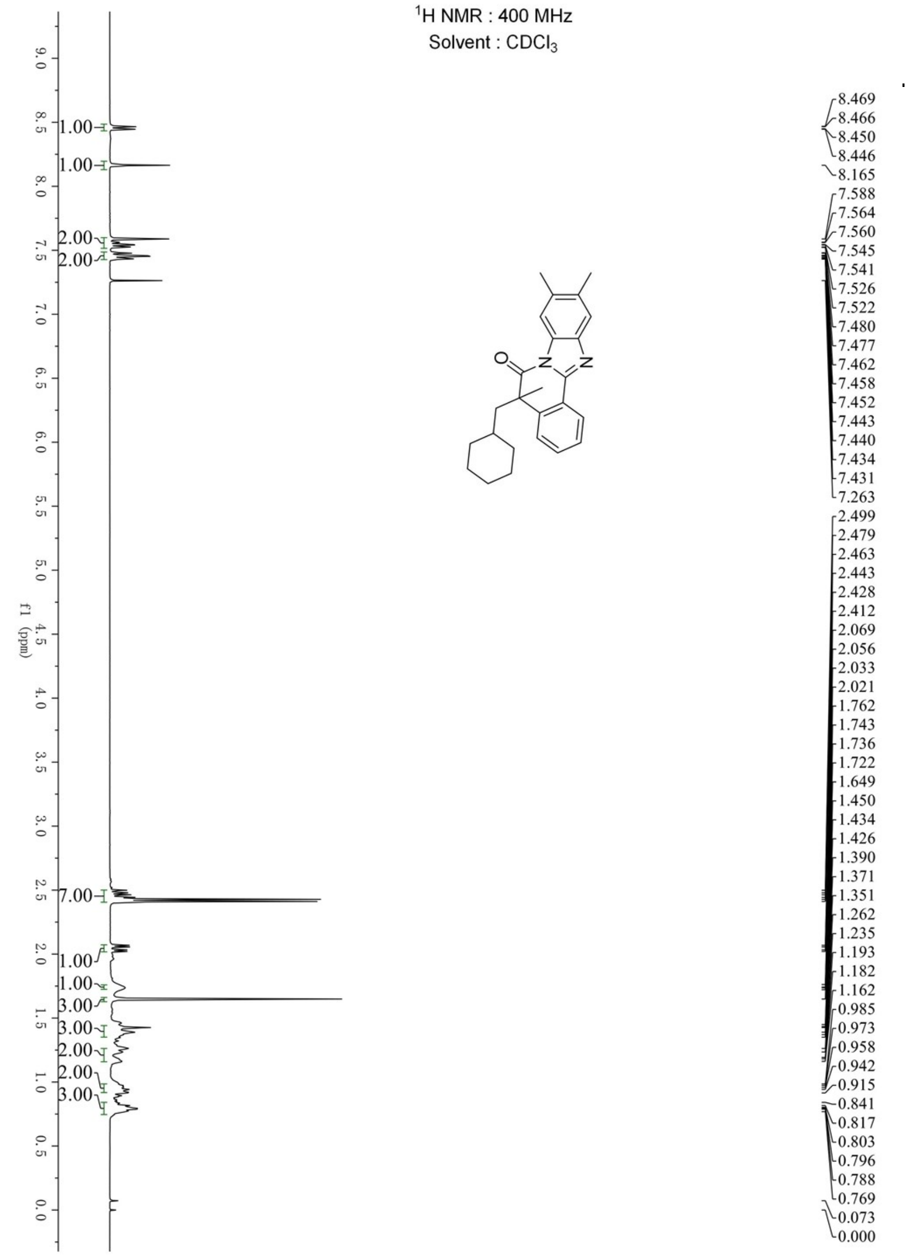


1H NMR : 400 MHz
Solvent : CDCl3
8.656
8.634
8.553
8.547
8.529
8.430
8.423
8.412
8.407
7.979
7.963
7.958
7.947
7.940
7.885
7.880
7.870
7.863
7.613
7.607
7.598
7.588
7.584
7.491
7.487
7.480
7.474
7.468
7.461
7.457
7.260
2.922
2.911
2.886
2.875
2.775
2.756
2.739
2.720
2.053
1.321
1.299
1.294
1.290
1.141
1.139
1.095
1.092
1.062
0.882
0.872
0.855
0.849
0.829
0.821
0.792
0.785
0.764
0.758
0.731
0.720
0.710
0.696
0.685
0.675
f1 (ppm)

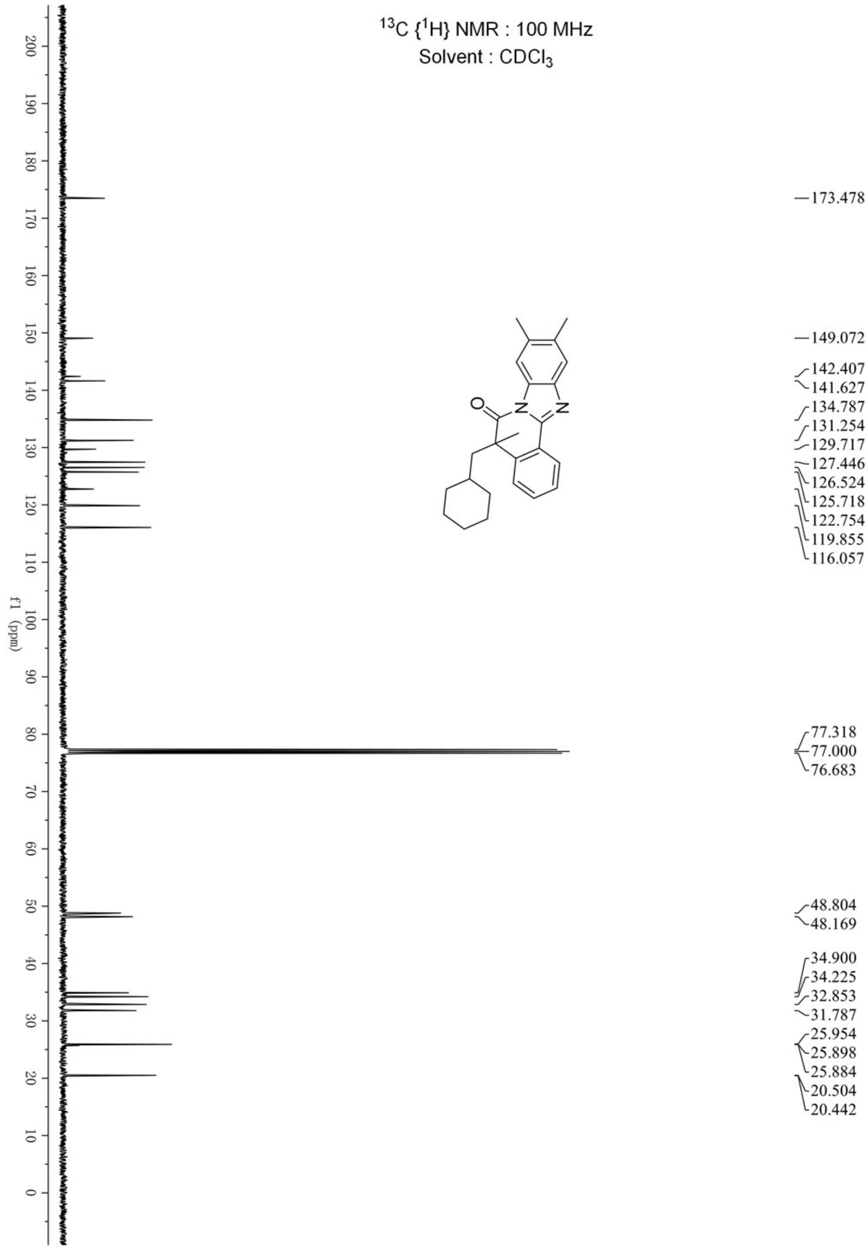
$^{13}C\{^1H\}$ NMR : 100 MHz
Solvent : $CDCl_3$



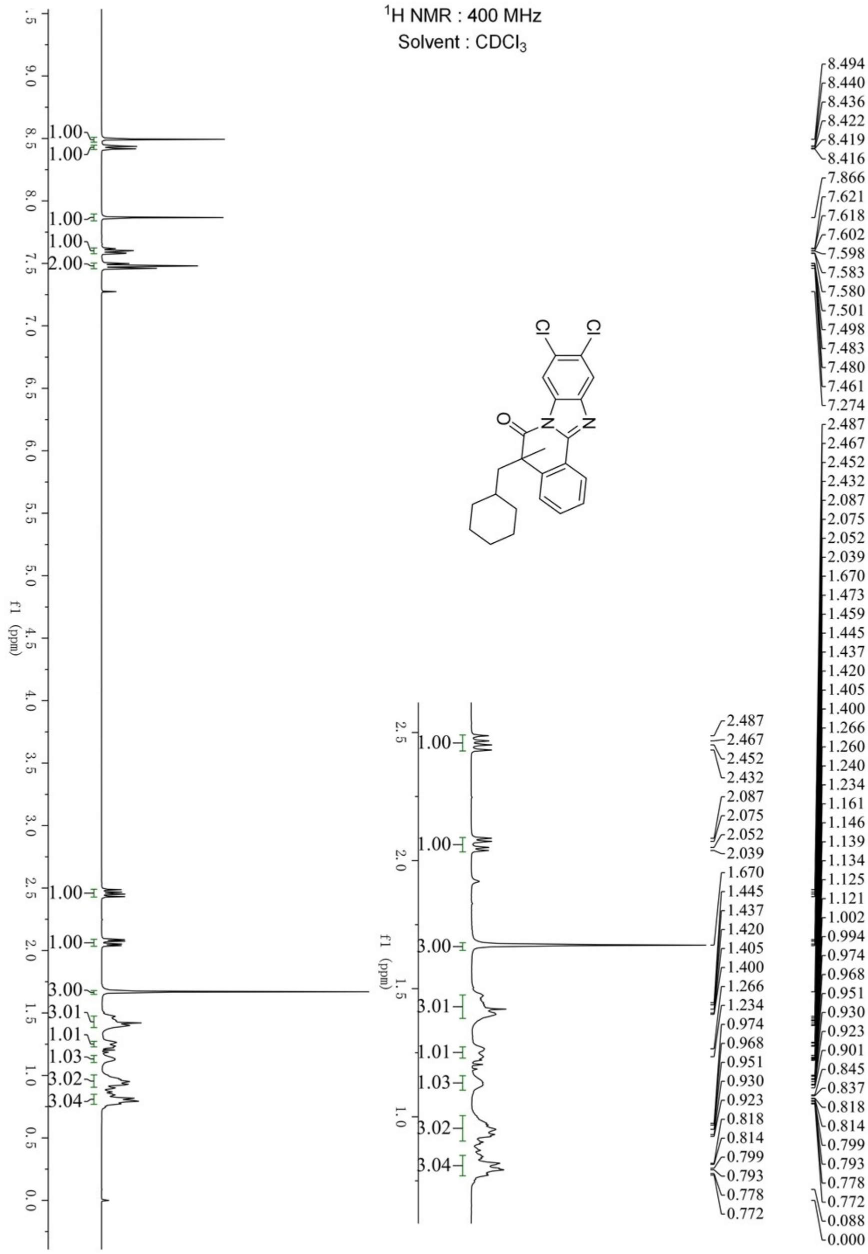
5-(Cyclohexy



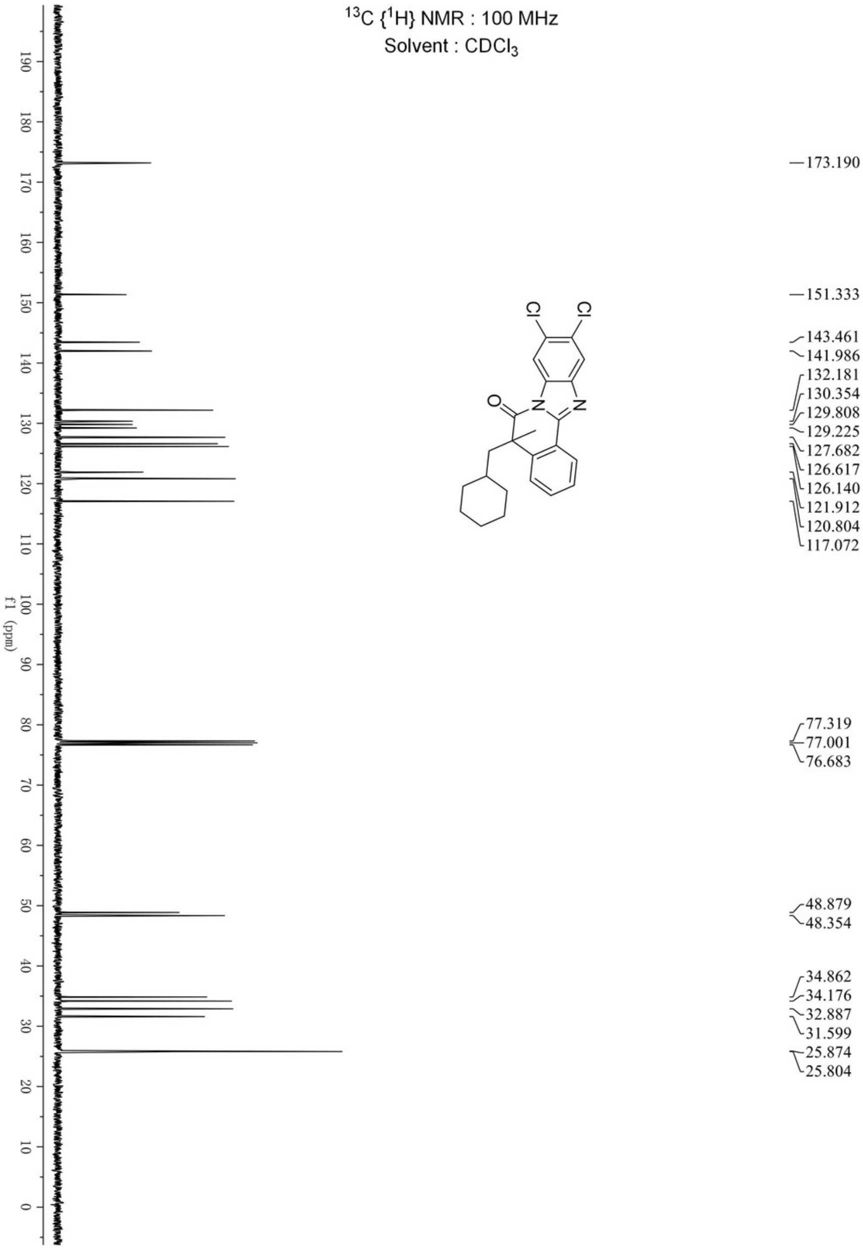
^{1}H NMR : 400 MHz
Solvent : $CDCl_3$
f1 (ppm)
8.469
8.466
8.450
8.446
8.165
7.588
7.564
7.560
7.545
7.541
7.526
7.522
7.480
7.477
7.462
7.458
7.452
7.443
7.440
7.434
7.431
7.263
2.499
2.479
2.463
2.443
2.428
2.412
2.069
2.056
2.033
2.021
1.762
1.743
1.736
1.722
1.649
1.450
1.434
1.426
1.390
1.371
1.351
1.262
1.235
1.193
1.182
1.162
0.985
0.973
0.958
0.942
0.915
0.841
0.817
0.803
0.796
0.788
0.769
0.073
0.000


^{13}C {^{1}H} NMR : 100 MHz
Solvent : $CDCl_3$
173.478
149.072
142.407
141.627
134.787
131.254
129.717
127.446
126.524
125.718
122.754
119.855
116.057
77.318
77.000
76.683
48.804
48.169
34.900
34.225
32.853
31.787
25.954
25.898
25.884
20.504
20.442
f1 (ppm)

9,10-Di

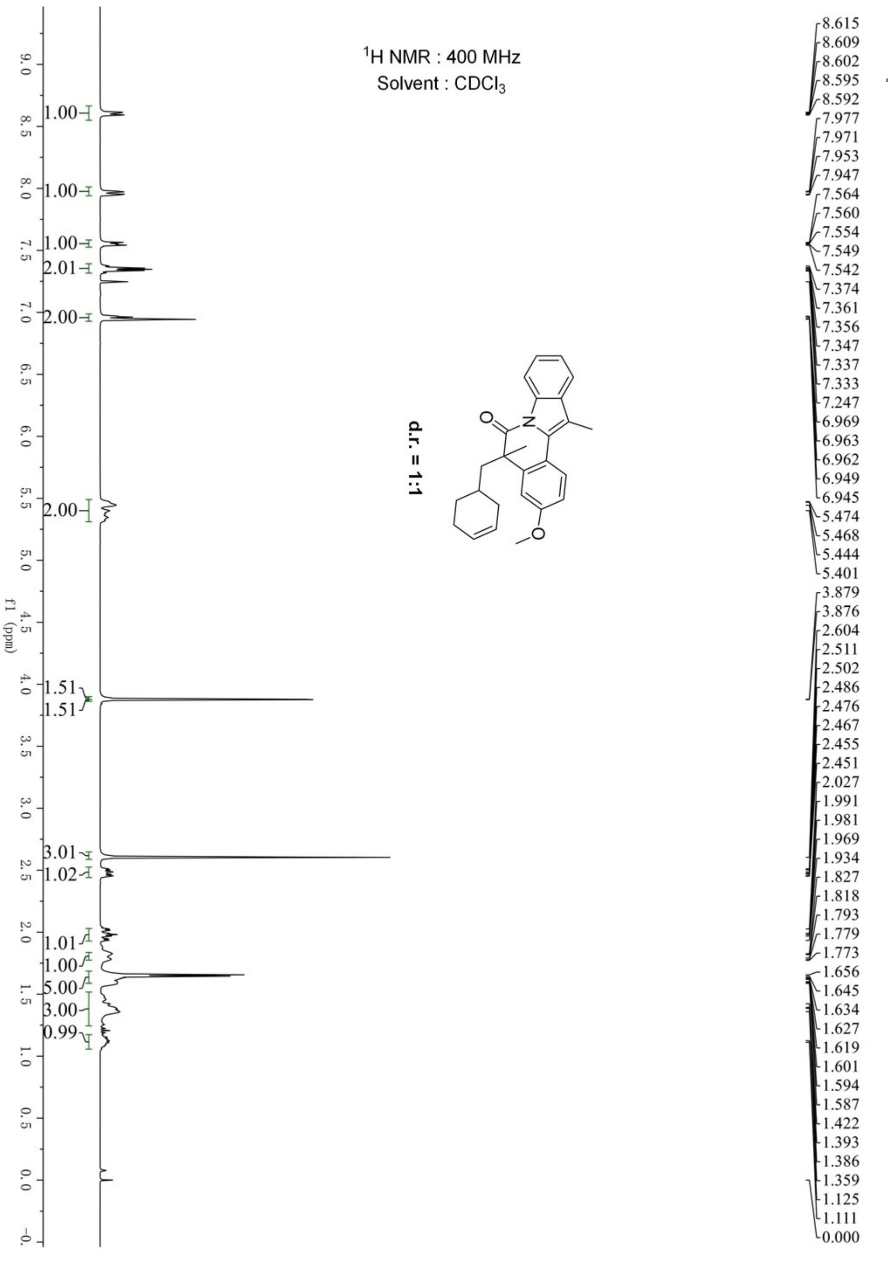


^{1}H NMR : 400 MHz
Solvent : $CDCl_3$
8.494 8.440 8.436 8.422 8.419 8.416 7.866 7.621 7.618 7.602 7.598 7.583 7.580 7.501 7.498 7.483 7.480 7.461 7.274 2.487 2.467 2.452 2.432 2.087 2.075 2.052 2.039 1.670 1.473 1.459 1.445 1.437 1.420 1.405 1.400 1.266 1.260 1.240 1.234 1.161 1.146 1.139 1.134 1.125 1.121 1.002 0.994 0.974 0.968 0.951 0.930 0.923 0.901 0.845 0.837 0.818 0.814 0.799 0.793 0.778 0.772 0.088 0.000

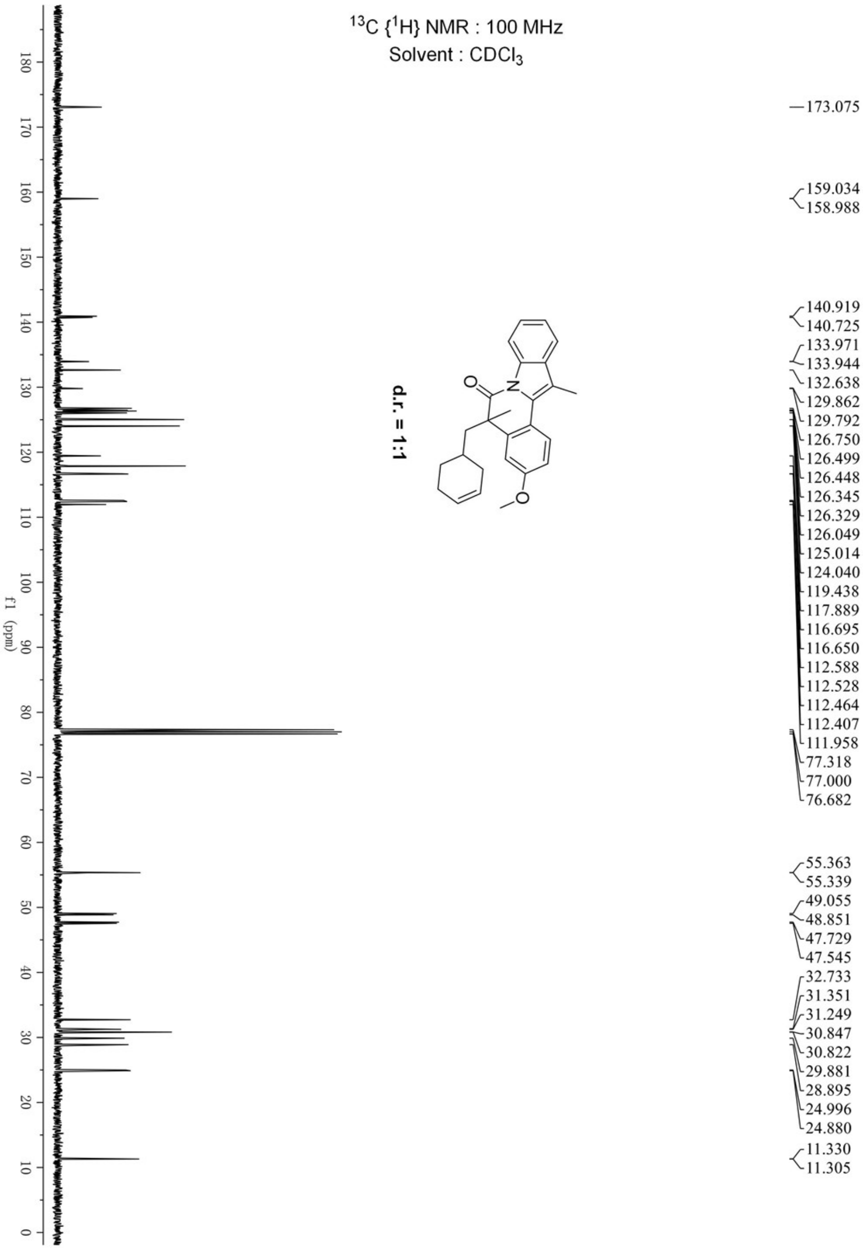


^{13}C {^{1}H} NMR : 100 MHz
Solvent : $CDCl_3$
Cl
Cl
O
N
N
f1 (ppm)
173.190
151.333
143.461
141.986
132.181
130.354
129.808
129.225
127.682
126.617
126.140
121.912
120.804
117.072
77.319
77.001
76.683
48.879
48.354
34.862
34.176
32.887
31.599
25.874
25.804

5-(Cyclohex-3

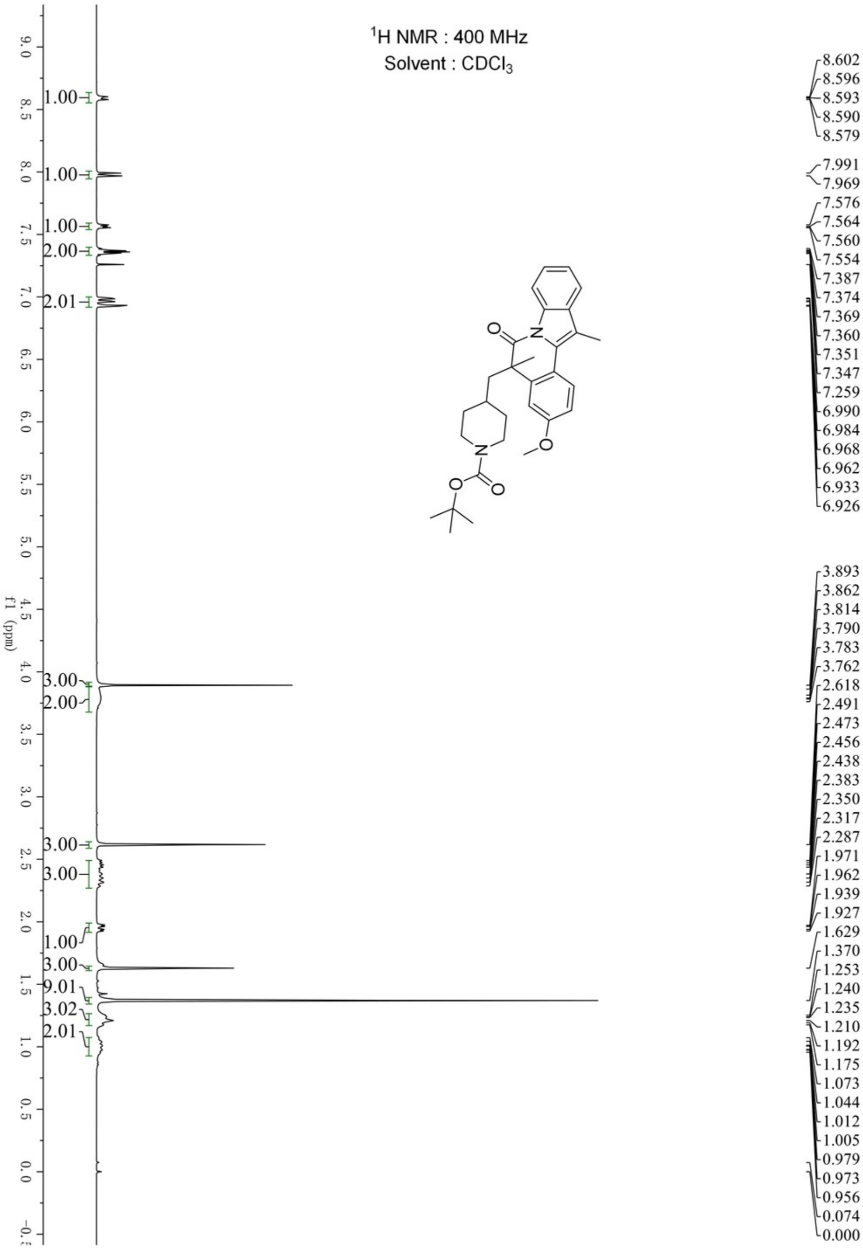


1H NMR : 400 MHz
Solvent : CDCl3
d.r. = 1:1
8.615
8.609
8.602
8.595
8.592
7.977
7.971
7.953
7.947
7.564
7.560
7.554
7.549
7.542
7.374
7.361
7.356
7.347
7.337
7.333
7.247
6.969
6.963
6.962
6.949
6.945
5.474
5.468
5.444
5.401
3.879
3.876
2.604
2.511
2.502
2.486
2.476
2.467
2.455
2.451
2.027
1.991
1.981
1.969
1.934
1.827
1.818
1.793
1.779
1.773
1.656
1.645
1.634
1.627
1.619
1.601
1.594
1.587
1.422
1.393
1.386
1.359
1.125
1.111
0.000
f1 (ppm)

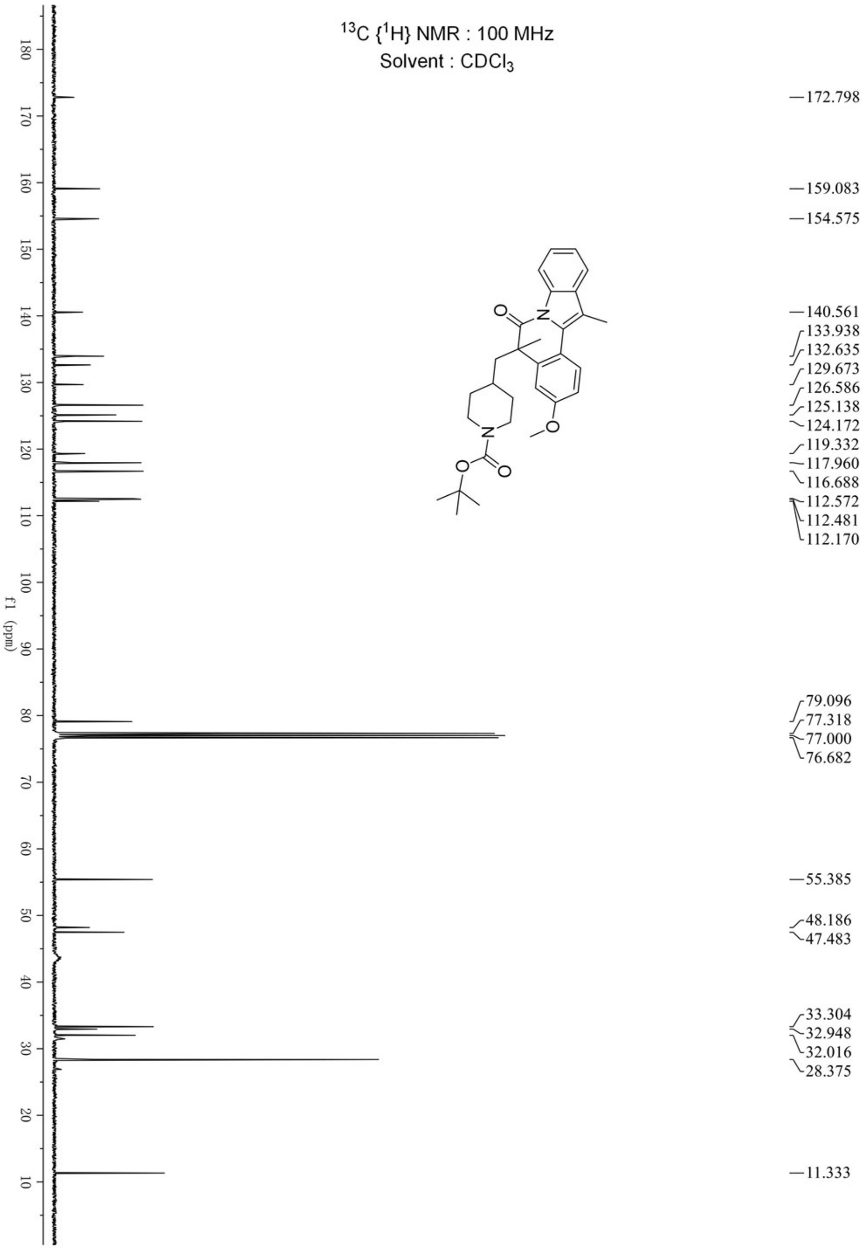

^{13}C {^{1}H} NMR : 100 MHz
Solvent : $CDCl_3$
d.r. = 1:1
173.075
159.034
158.988
140.919
140.725
133.971
133.944
132.638
129.862
129.792
126.750
126.499
126.448
126.345
126.329
126.049
125.014
124.040
119.438
117.889
116.695
116.650
112.588
112.528
112.464
112.407
111.958
77.318
77.000
76.682
55.363
55.339
49.055
48.851
47.729
47.545
32.733
31.351
31.249
30.847
30.822
29.881
28.895
24.996
24.880
11.330
11.305
f1 (ppm)

***tert*-Bu**

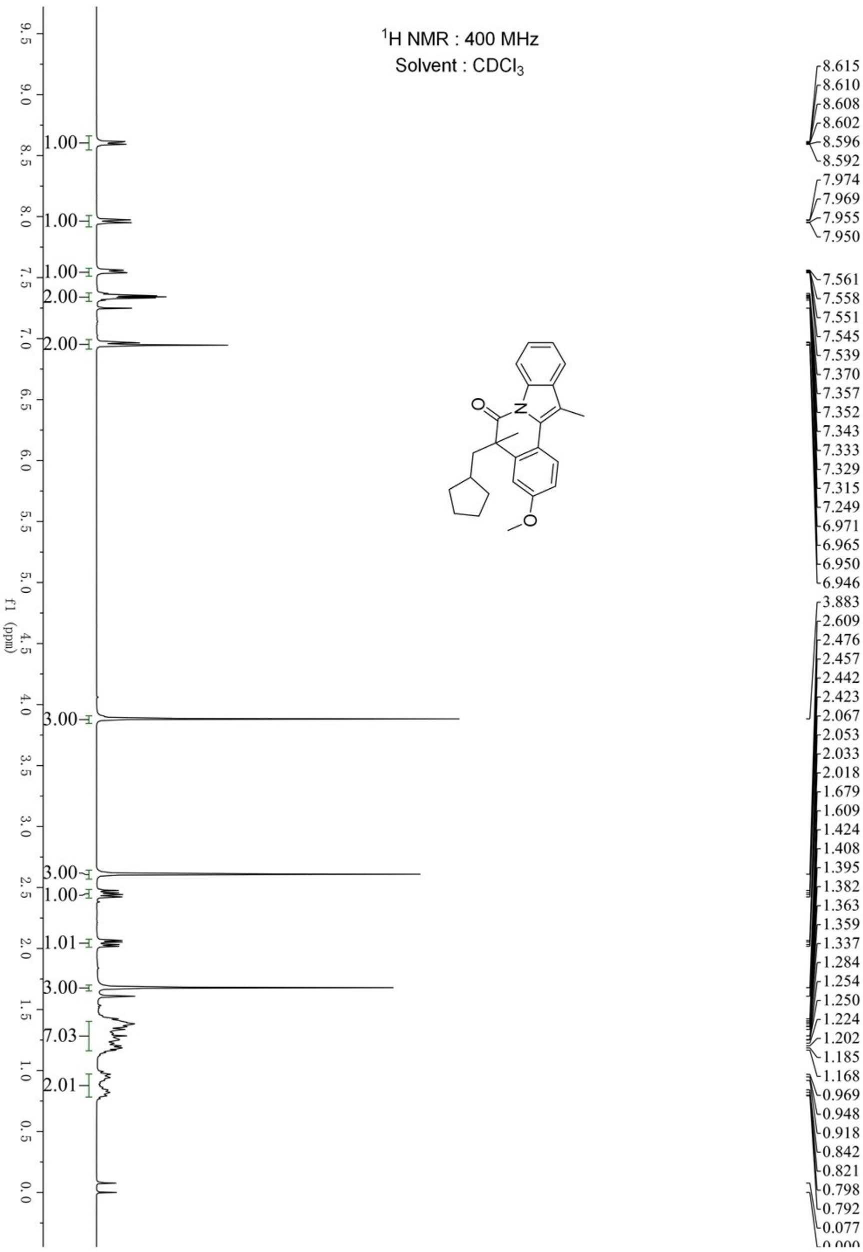
a



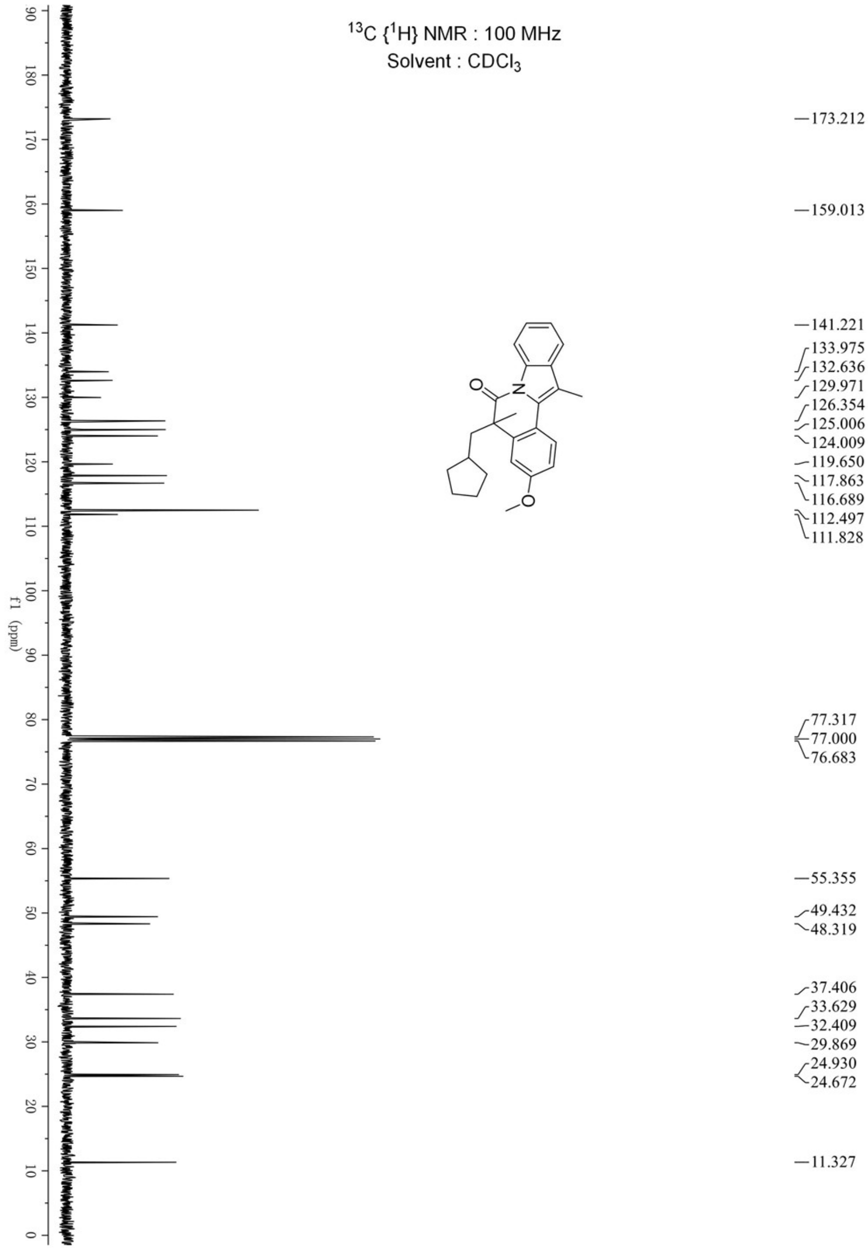
¹H NMR : 400 MHz
Solvent : CDCl3
f1 (ppm)
8.602
8.596
8.593
8.590
8.579
7.991
7.969
7.576
7.564
7.560
7.554
7.387
7.374
7.369
7.360
7.351
7.347
7.259
6.990
6.984
6.968
6.962
6.933
6.926
3.893
3.862
3.814
3.790
3.783
3.762
2.618
2.491
2.473
2.456
2.438
2.383
2.350
2.317
2.287
1.971
1.962
1.939
1.927
1.629
1.370
1.253
1.240
1.235
1.210
1.192
1.175
1.073
1.044
1.012
1.005
0.979
0.973
0.956
0.074
0.000


$^{13}C\ \{^{1}H\}$ NMR : 100 MHz
Solvent : $CDCl_3$
172.798
159.083
154.575
140.561
133.938
132.635
129.673
126.586
125.138
124.172
119.332
117.960
116.688
112.572
112.481
112.170
79.096
77.318
77.000
76.682
55.385
48.186
47.483
33.304
32.948
32.016
28.375
11.333
f1 (ppm)

5-(Cyclopent

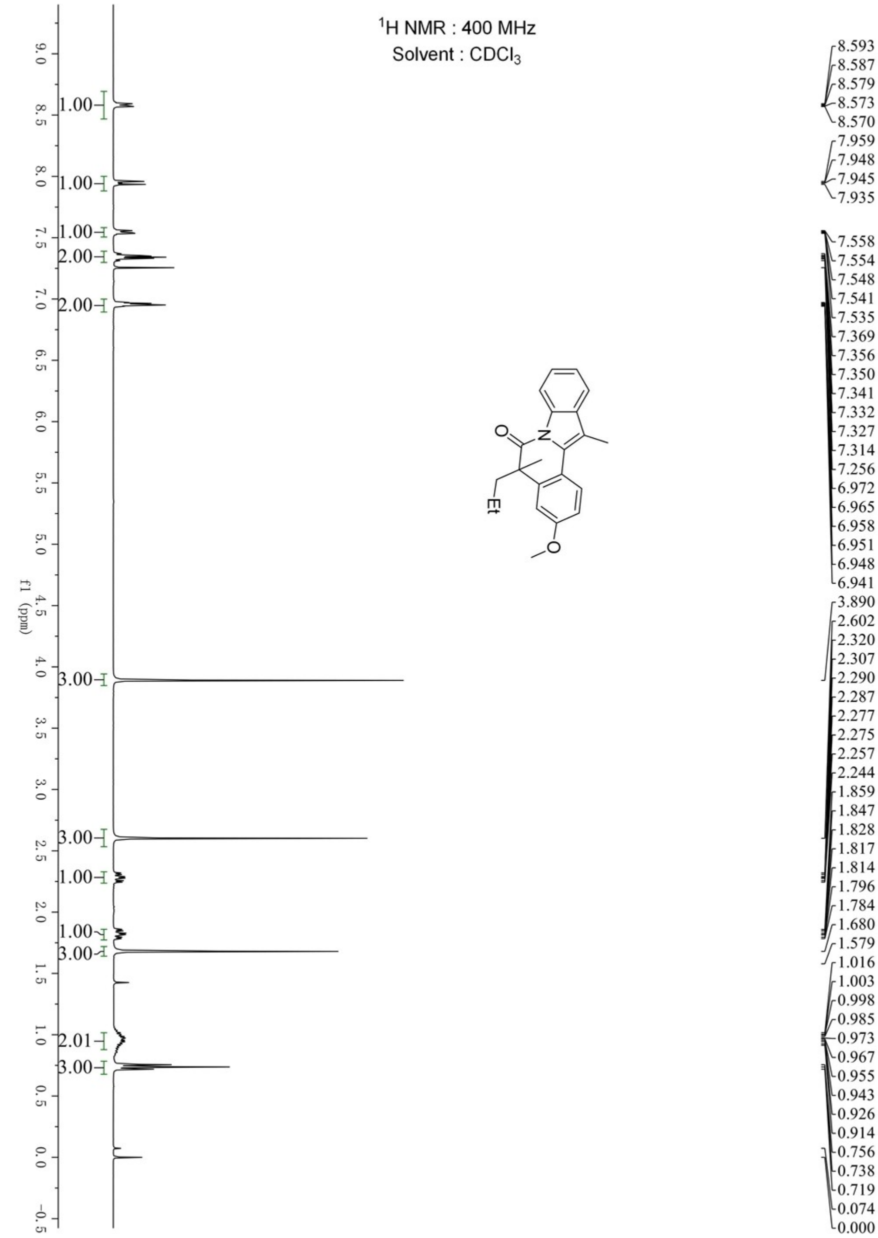


1H NMR : 400 MHz
Solvent : CDCl3
8.615
8.610
8.608
8.602
8.596
8.592
7.974
7.969
7.955
7.950
7.561
7.558
7.551
7.545
7.539
7.370
7.357
7.352
7.343
7.333
7.329
7.315
7.249
6.971
6.965
6.950
6.946
3.883
2.609
2.476
2.457
2.442
2.423
2.067
2.053
2.033
2.018
1.679
1.609
1.424
1.408
1.395
1.382
1.363
1.359
1.337
1.284
1.254
1.250
1.224
1.202
1.185
1.168
0.969
0.948
0.918
0.842
0.821
0.798
0.792
0.077
1.00
1.00
1.00
2.00
2.00
3.00
3.00
1.00
1.01
3.00
7.03
2.01
f1 (ppm)

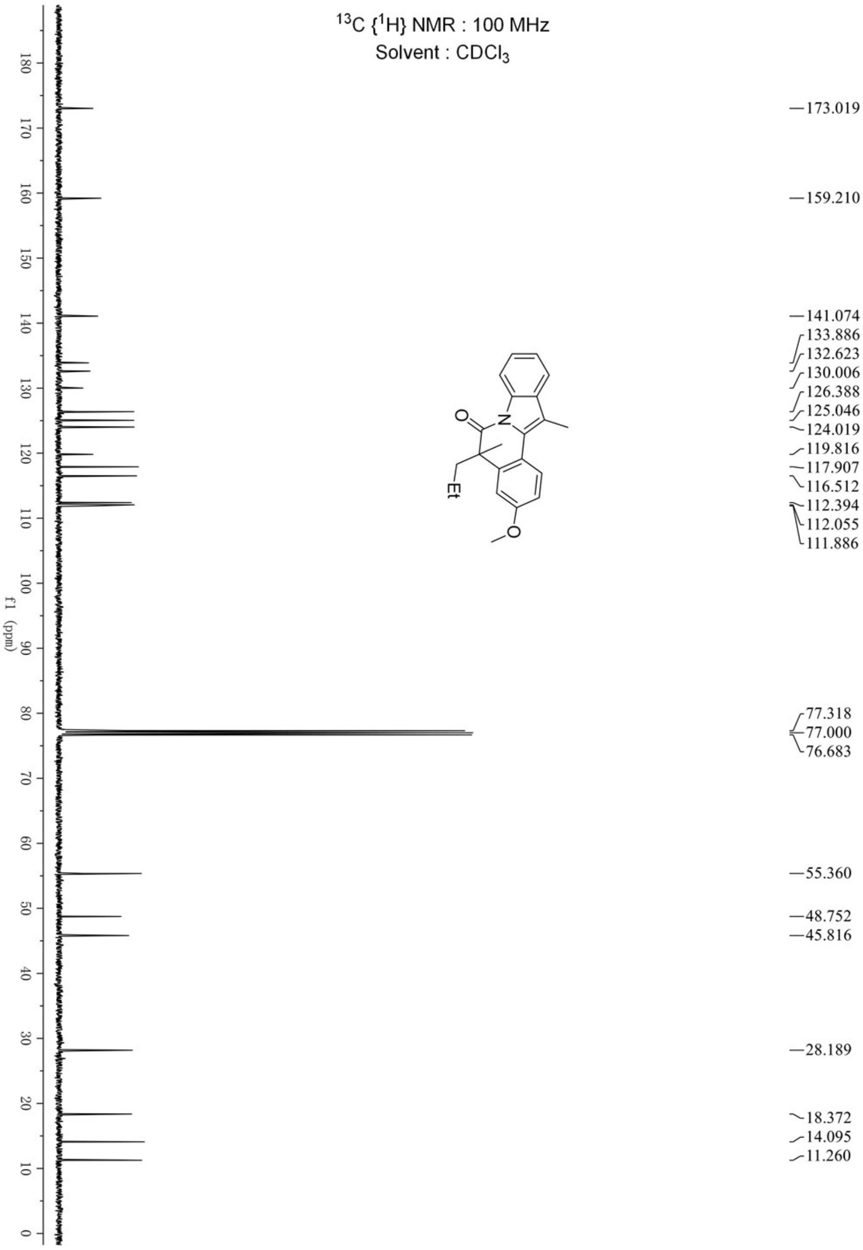


$^{13}C\{^1H\}$ NMR : 100 MHz
Solvent : $CDCl_3$
173.212
159.013
141.221
133.975
132.636
129.971
126.354
125.006
124.009
119.650
117.863
116.689
112.497
111.828
77.317
77.000
76.683
55.355
49.432
48.319
37.406
33.629
32.409
29.869
24.930
24.672
11.327
f1 (ppm)
O
N
O

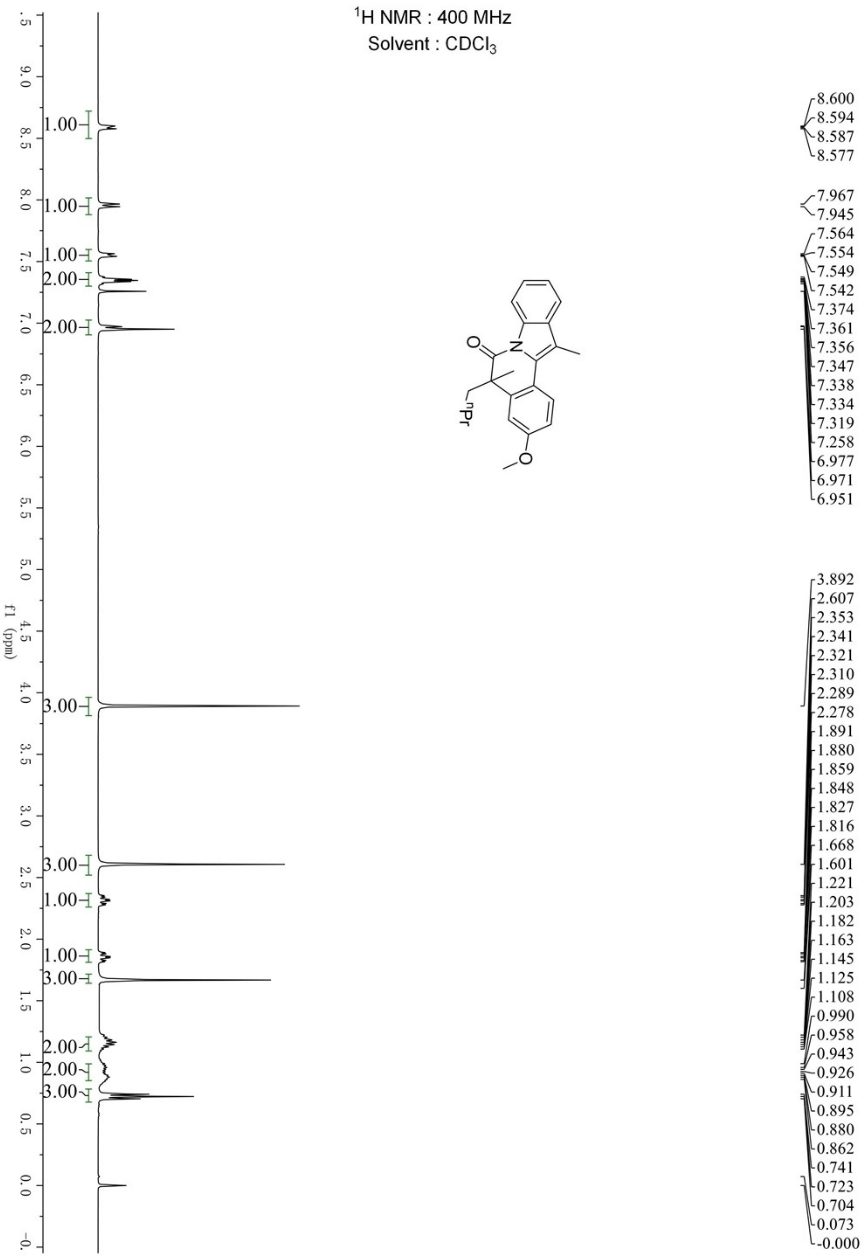
3-Methoxy



^{1}H NMR : 400 MHz
Solvent : $CDCl_3$
8.593
8.587
8.579
8.573
8.570
7.959
7.948
7.945
7.935
7.558
7.554
7.548
7.541
7.535
7.369
7.356
7.350
7.341
7.332
7.327
7.314
7.256
6.972
6.965
6.958
6.951
6.948
6.941
3.890
2.602
2.320
2.307
2.290
2.287
2.277
2.275
2.257
2.244
1.859
1.847
1.828
1.817
1.814
1.796
1.784
1.680
1.579
1.016
1.003
0.998
0.985
0.973
0.967
0.955
0.943
0.926
0.914
0.756
0.738
0.719
0.074
0.000
1.00
1.00
1.00
2.00
2.00
3.00
3.00
1.00
1.00
3.00
2.01
3.00
f1 (ppm)
N
O
Et

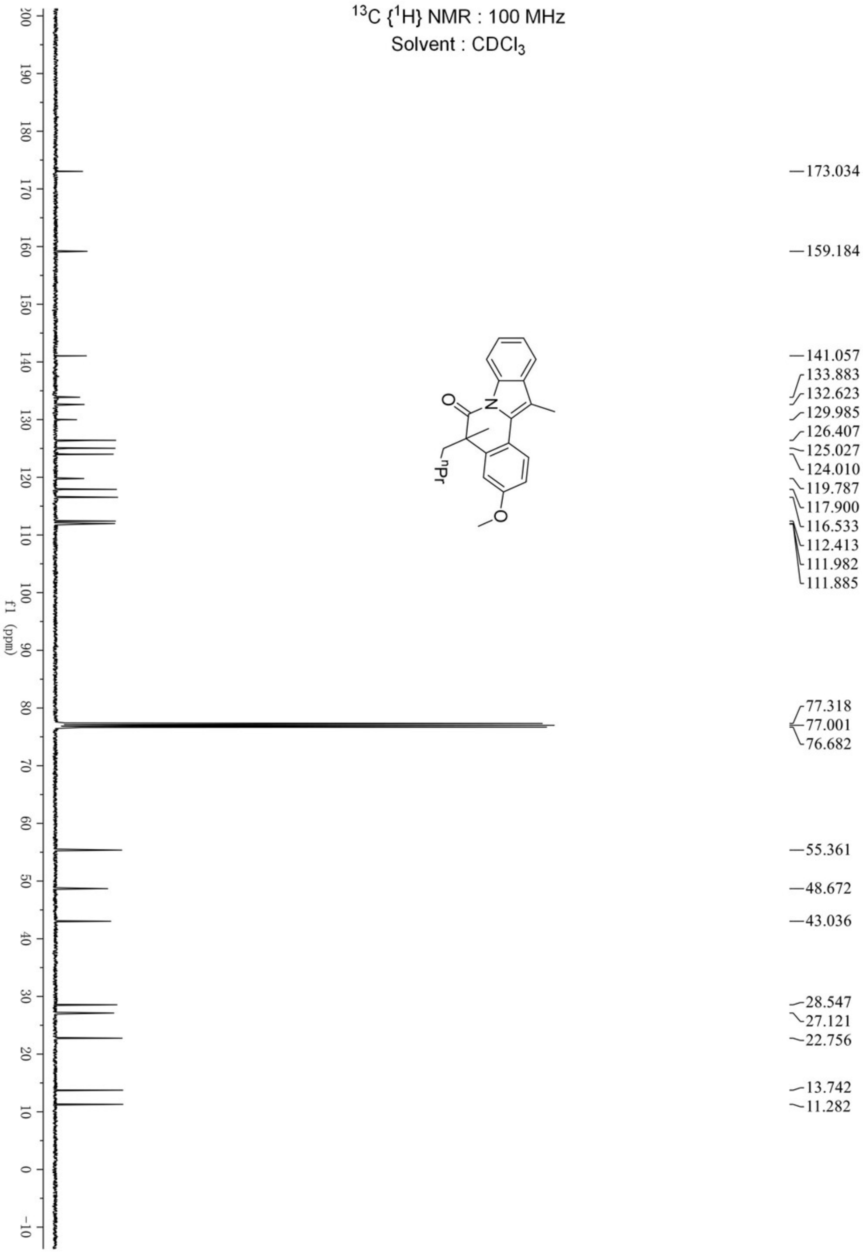

$^{13}C\{^1H\}$ NMR : 100 MHz
Solvent : $CDCl_3$
173.019
159.210
141.074
133.886
132.623
130.006
126.388
125.046
124.019
119.816
117.907
116.512
112.394
112.055
111.886
77.318
77.000
76.683
55.360
48.752
45.816
28.189
18.372
14.095
11.260
f1 (ppm)
N
O
Et
O

5-Butyl-3-

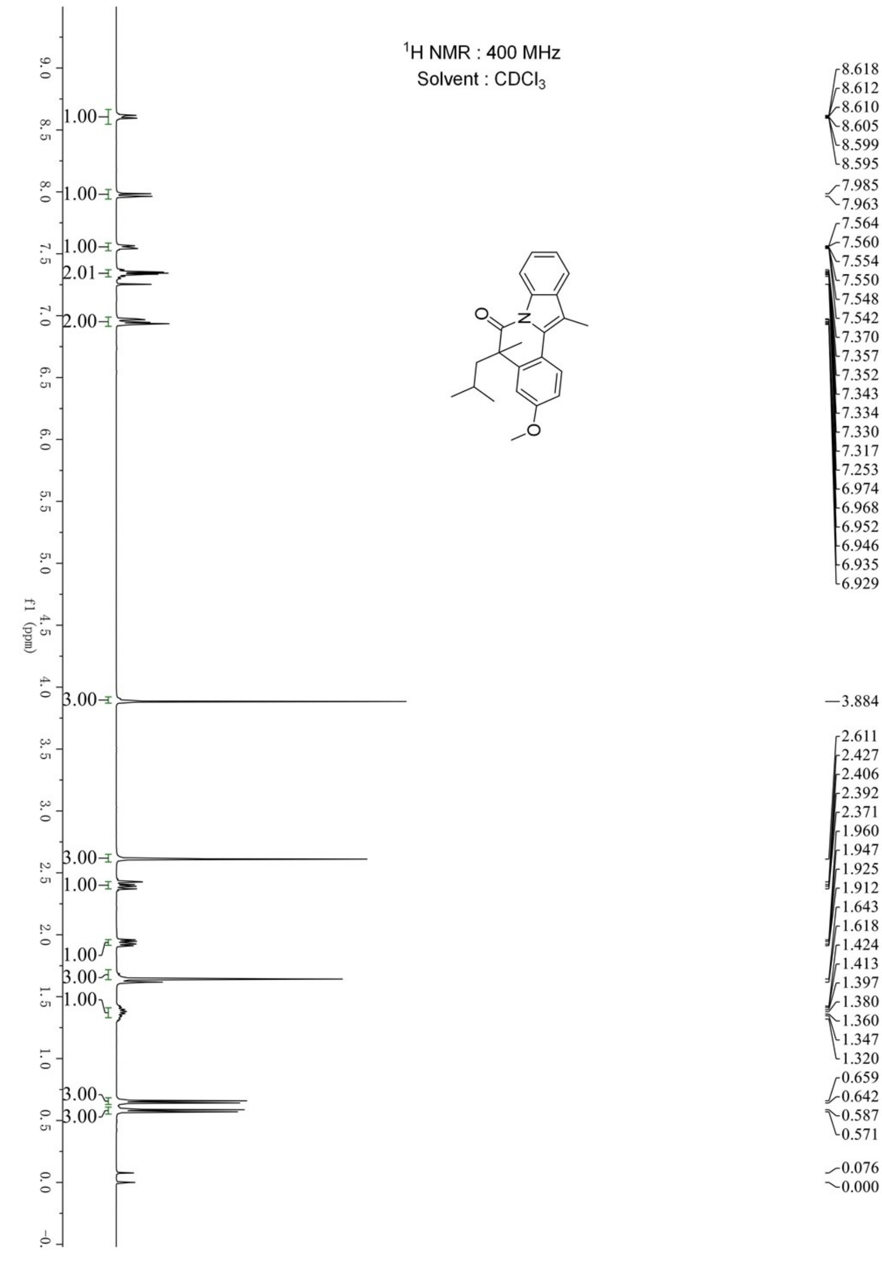


^{1}H NMR : 400 MHz
Solvent : CDCl$_3$
8.600
8.594
8.587
8.577
7.967
7.945
7.564
7.554
7.549
7.542
7.374
7.361
7.356
7.347
7.338
7.334
7.319
7.258
6.977
6.971
6.951
3.892
2.607
2.353
2.341
2.321
2.310
2.289
2.278
1.891
1.880
1.859
1.848
1.827
1.816
1.668
1.601
1.221
1.203
1.182
1.163
1.145
1.125
1.108
0.990
0.958
0.943
0.926
0.911
0.895
0.880
0.862
0.741
0.723
0.704
0.073
-0.000
1.00
1.00
1.00
2.00
2.00
3.00
3.00
1.00
1.00
3.00
2.00
2.00
3.00
f1 (ppm)
N
O
^{n}Pr

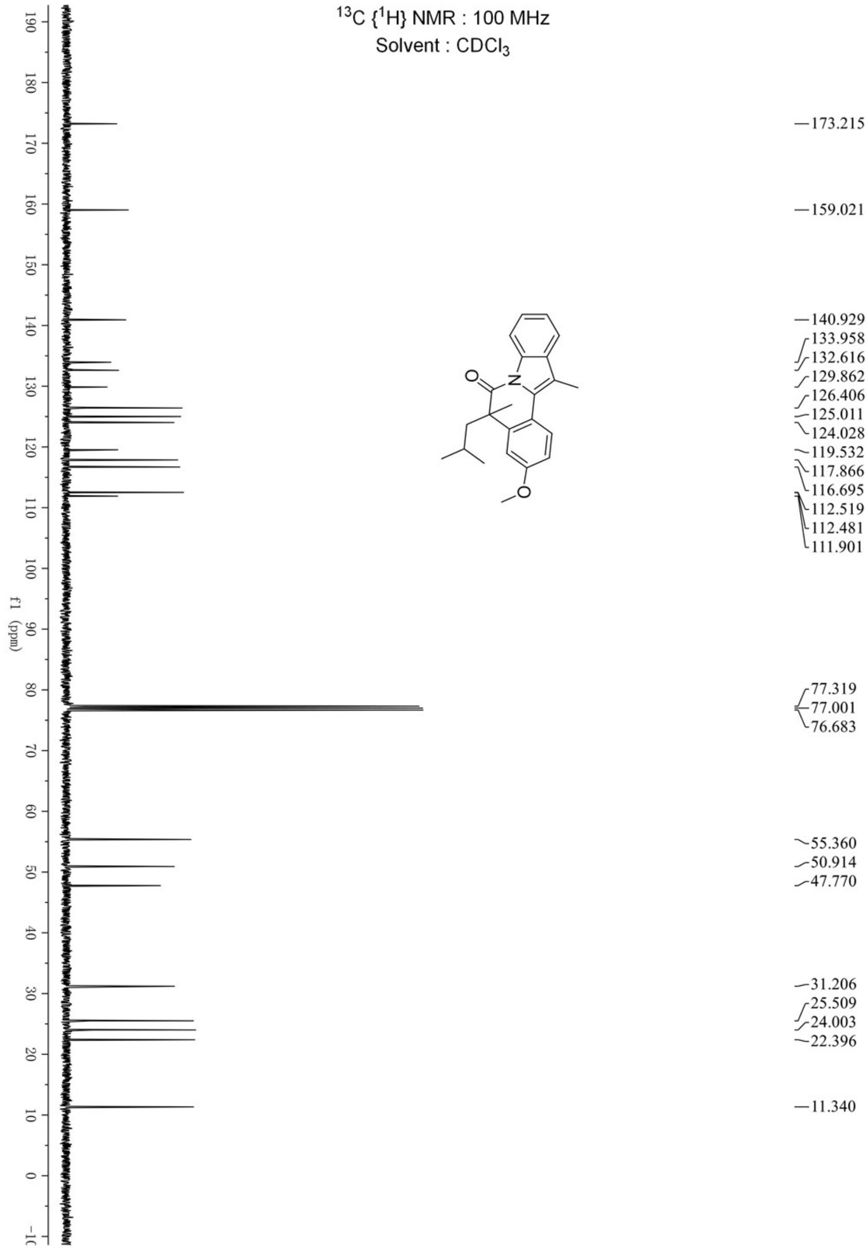
$^{13}C\ \{^{1}H\}$ NMR : 100 MHz
Solvent : $CDCl_3$



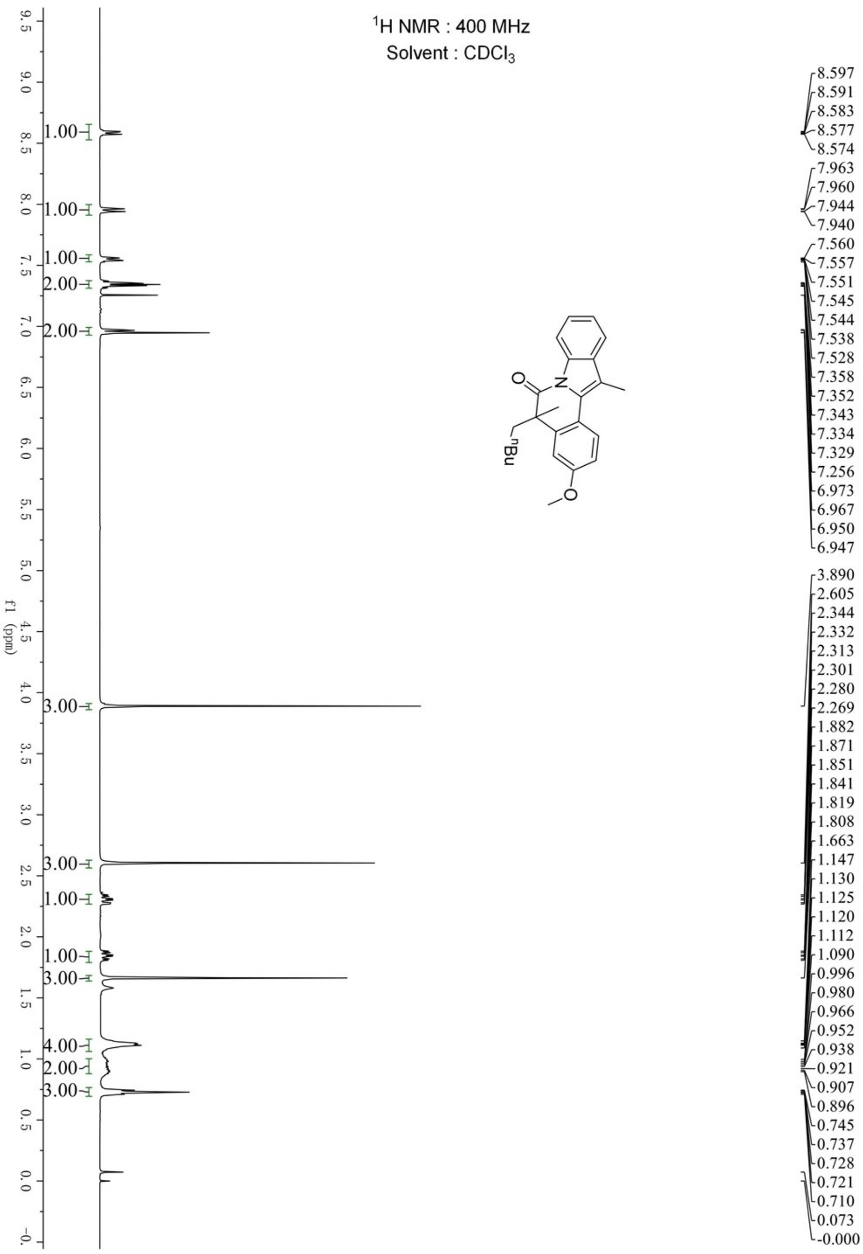
5-Isobutyl-



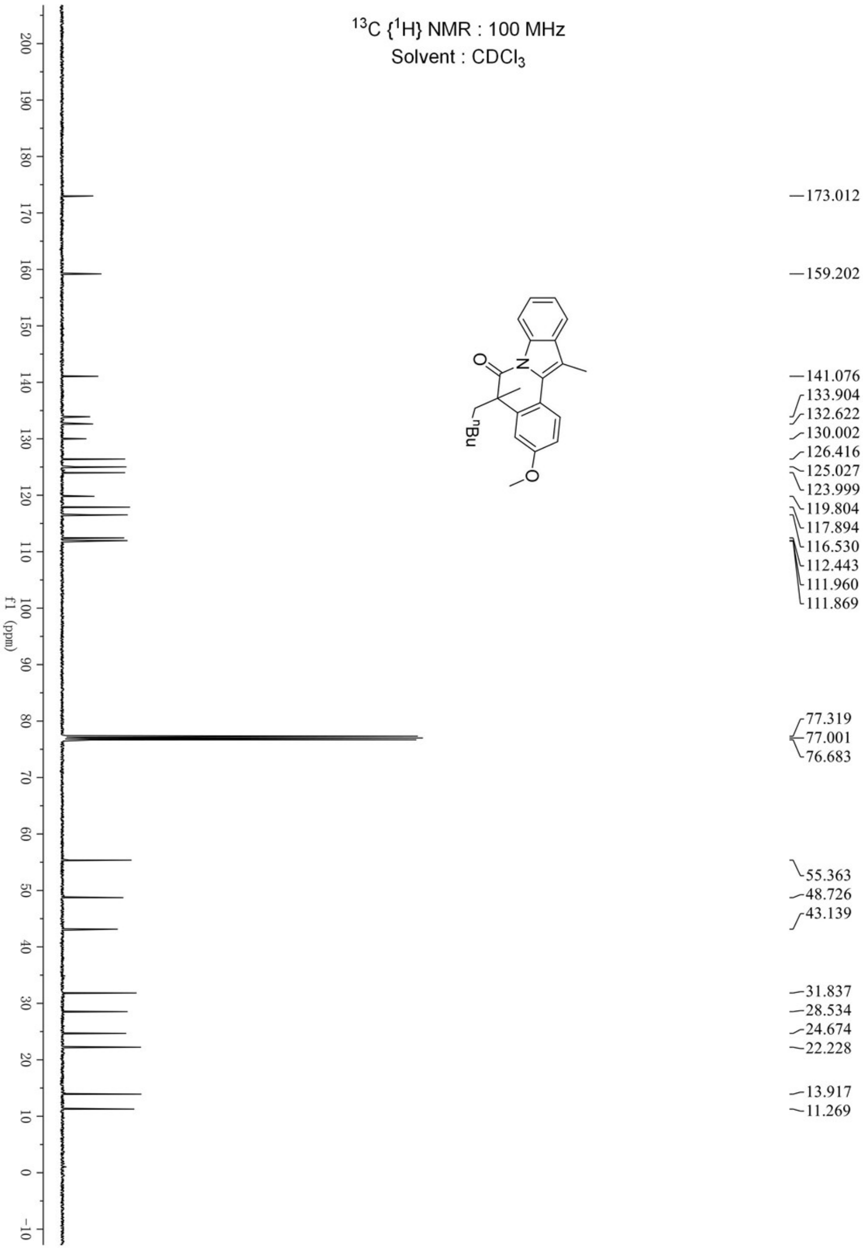
1H NMR : 400 MHz
Solvent : CDCl3
8.618
8.612
8.610
8.605
8.599
8.595
7.985
7.963
7.564
7.560
7.554
7.550
7.548
7.542
7.370
7.357
7.352
7.343
7.334
7.330
7.317
7.253
6.974
6.968
6.952
6.946
6.935
6.929
3.884
2.611
2.427
2.406
2.392
2.371
1.960
1.947
1.925
1.912
1.643
1.618
1.424
1.413
1.397
1.380
1.360
1.347
1.320
0.659
0.642
0.587
0.571
0.076
0.000
f1 (ppm)


$^{13}C\ \{^1H\}$ NMR : 100 MHz
Solvent : $CDCl_3$
f1 (ppm)
173.215
159.021
140.929
133.958
132.616
129.862
126.406
125.011
124.028
119.532
117.866
116.695
112.519
112.481
111.901
77.319
77.001
76.683
55.360
50.914
47.770
31.206
25.509
24.003
22.396
11.340
O
N
O

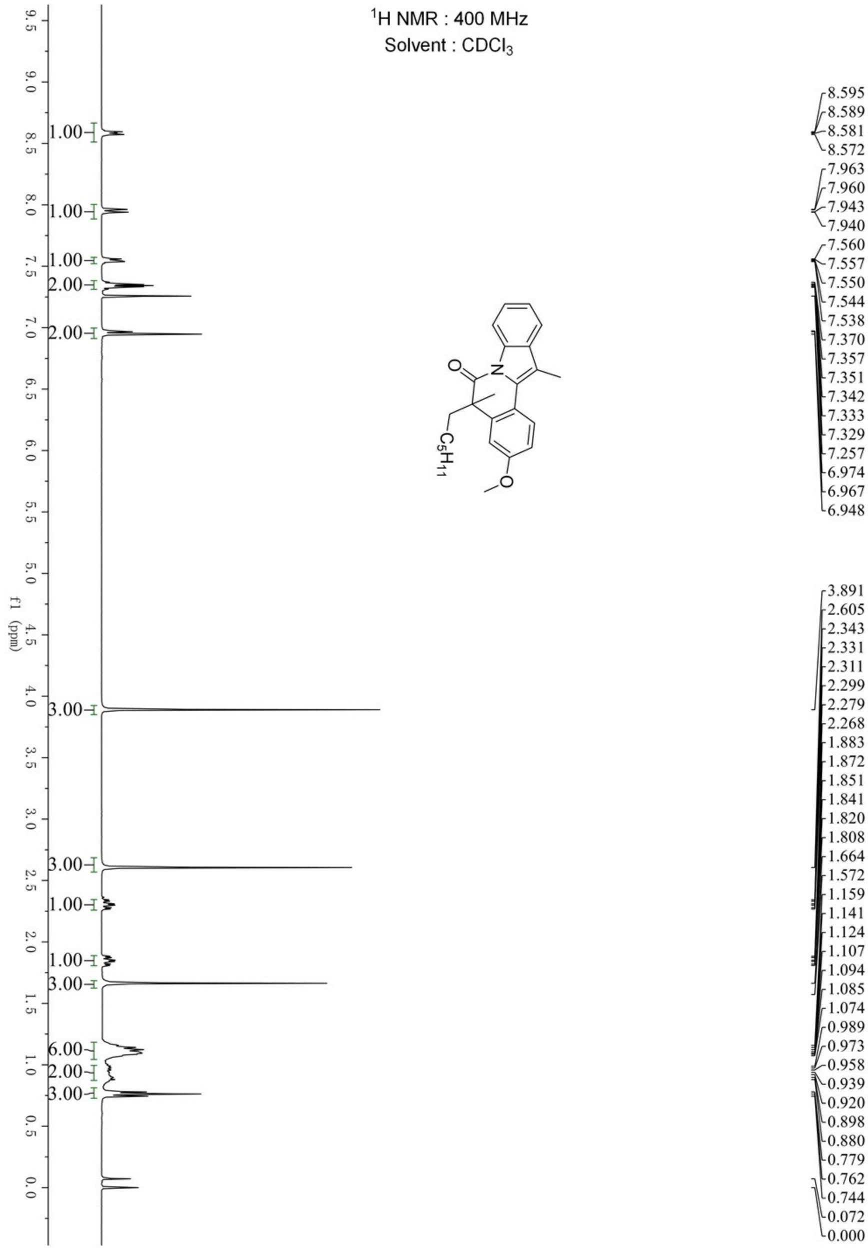
3-Methoxy



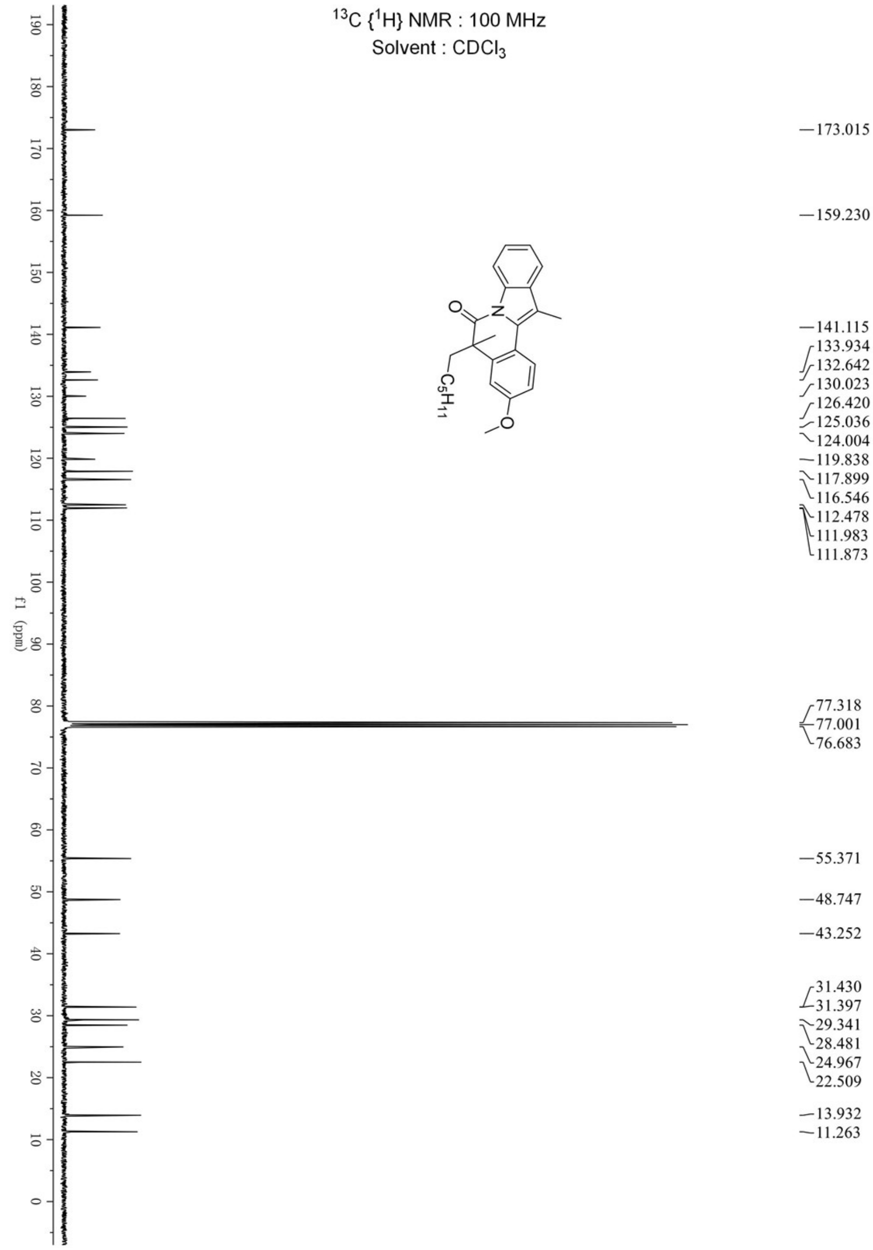
1H NMR : 400 MHz
Solvent : CDCl3
8.597
8.591
8.583
8.577
8.574
7.963
7.960
7.944
7.940
7.560
7.557
7.551
7.545
7.544
7.538
7.528
7.358
7.352
7.343
7.334
7.329
7.256
6.973
6.967
6.950
6.947
3.890
2.605
2.344
2.332
2.313
2.301
2.280
2.269
1.882
1.871
1.851
1.841
1.819
1.808
1.663
1.147
1.130
1.125
1.120
1.112
1.090
0.996
0.980
0.966
0.952
0.938
0.921
0.907
0.896
0.745
0.737
0.728
0.721
0.710
0.073
-0.000
nBu
N
O
f1 (ppm)


^{13}C {^{1}H} NMR : 100 MHz
Solvent : $CDCl_3$
O
N
^{n}Bu
O
173.012
159.202
141.076
133.904
132.622
130.002
126.416
125.027
123.999
119.804
117.894
116.530
112.443
111.960
111.869
77.319
77.001
76.683
55.363
48.726
43.139
31.837
28.534
24.674
22.228
13.917
11.269
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

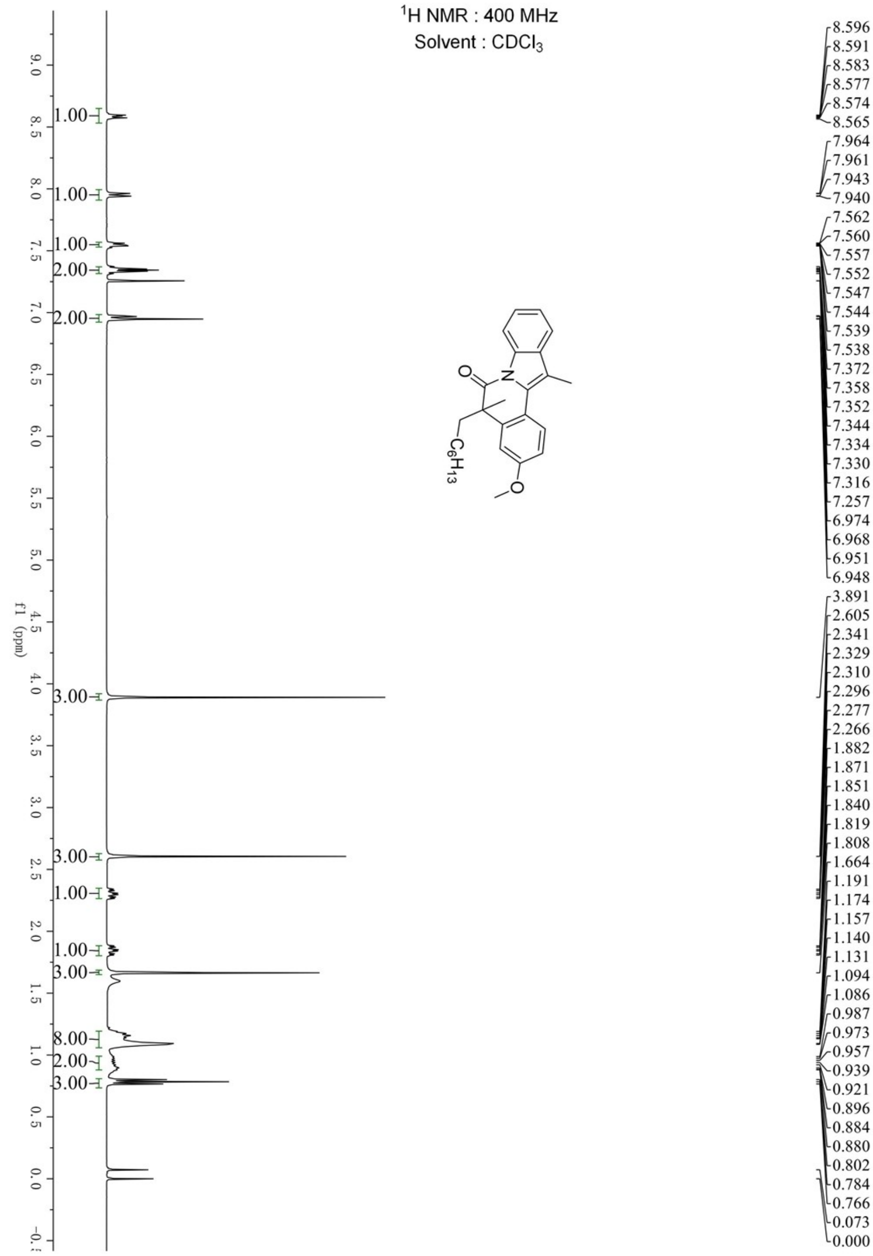
5-Hexyl-3-



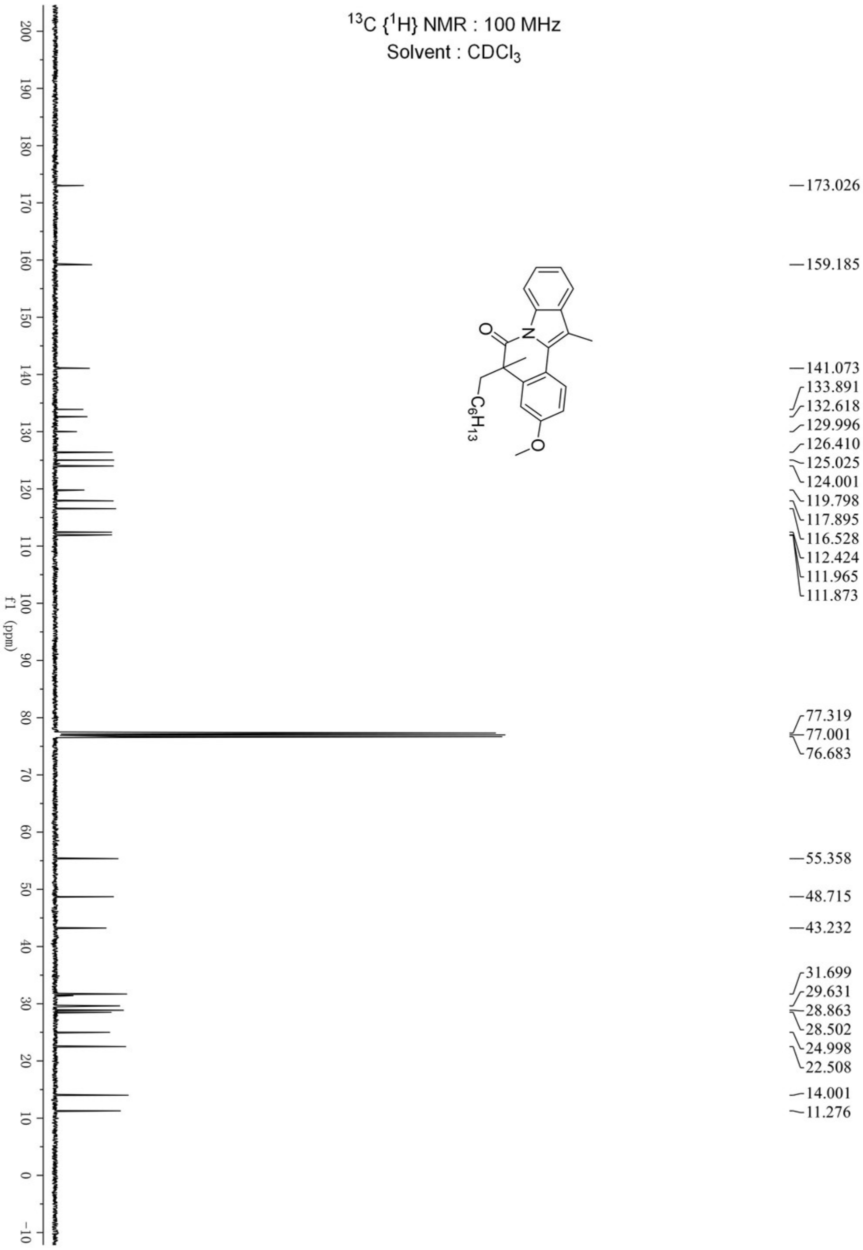
^{1}H NMR : 400 MHz
Solvent : $CDCl_3$
8.595
8.589
8.581
8.572
7.963
7.960
7.943
7.940
7.560
7.557
7.550
7.544
7.538
7.370
7.357
7.351
7.342
7.333
7.329
7.257
6.974
6.967
6.948
3.891
2.605
2.343
2.331
2.311
2.299
2.279
2.268
1.883
1.872
1.851
1.841
1.820
1.808
1.664
1.572
1.159
1.141
1.124
1.107
1.094
1.085
1.074
0.989
0.973
0.958
0.939
0.920
0.898
0.880
0.779
0.762
0.744
0.072
0.000
f1 (ppm)


13C {1H} NMR : 100 MHz
Solvent : CDCl3
173.015
159.230
141.115
133.934
132.642
130.023
126.420
125.036
124.004
119.838
117.899
116.546
112.478
111.983
111.873
77.318
77.001
76.683
55.371
48.747
43.252
31.430
31.397
29.341
28.481
24.967
22.509
13.932
11.263
f1 (ppm)
O
N
C5H11
O

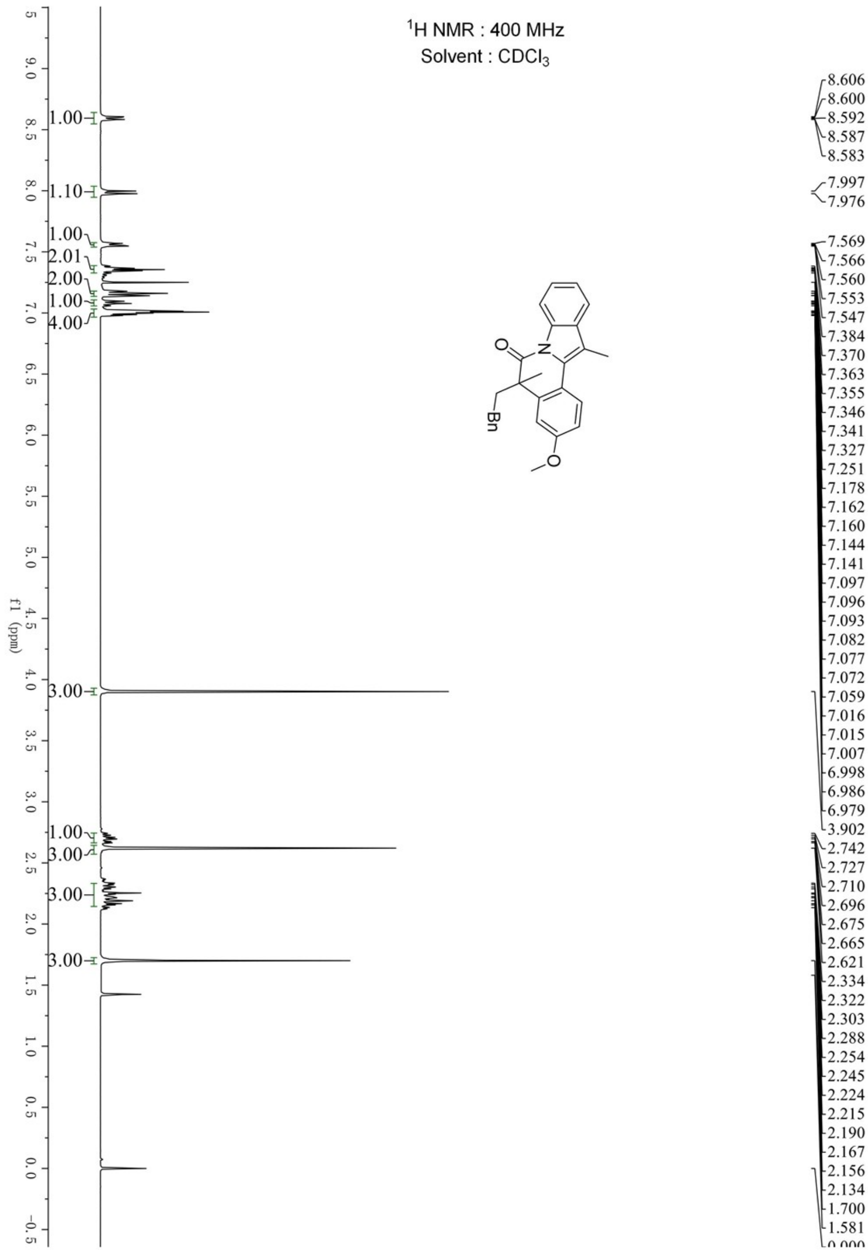
5-Heptyl-3



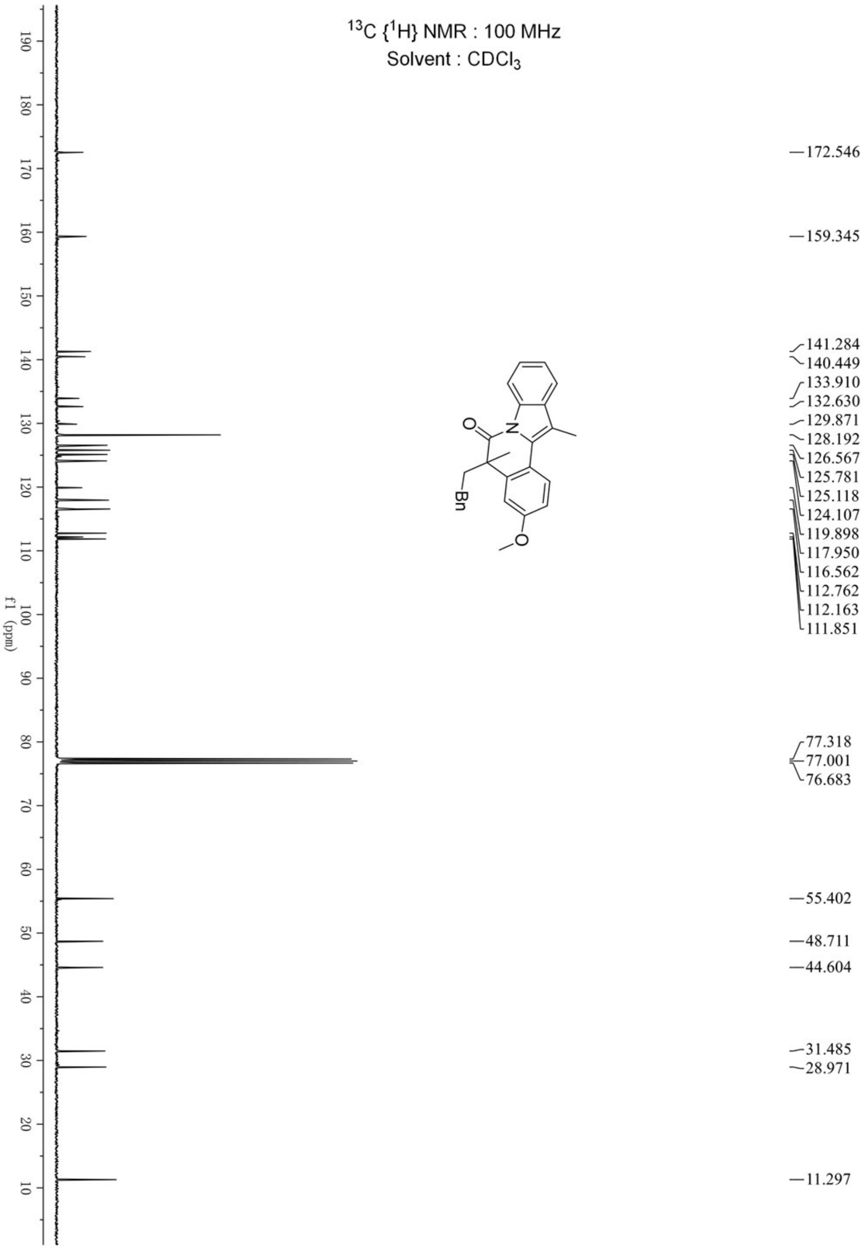
1H NMR : 400 MHz
Solvent : CDCl3
f1 (ppm)
1.00
1.00
1.00
2.00
2.00
3.00
3.00
1.00
1.00
3.00
8.00
2.00
3.00
N
O
C6H13
8.596
8.591
8.583
8.577
8.574
8.565
7.964
7.961
7.943
7.940
7.562
7.560
7.557
7.552
7.547
7.544
7.539
7.538
7.372
7.358
7.352
7.344
7.334
7.330
7.316
7.257
6.974
6.968
6.951
6.948
3.891
2.605
2.341
2.329
2.310
2.296
2.277
2.266
1.882
1.871
1.851
1.840
1.819
1.808
1.664
1.191
1.174
1.157
1.140
1.131
1.094
1.086
0.987
0.973
0.957
0.939
0.921
0.896
0.884
0.880
0.802
0.784
0.766
0.073
0.000


13C {1H} NMR : 100 MHz
Solvent : CDCl3
173.026
159.185
141.073
133.891
132.618
129.996
126.410
125.025
124.001
119.798
117.895
116.528
112.424
111.965
111.873
77.319
77.001
76.683
55.358
48.715
43.232
31.699
29.631
28.863
28.502
24.998
22.508
14.001
11.276
f1 (ppm)
N
O
C6H13

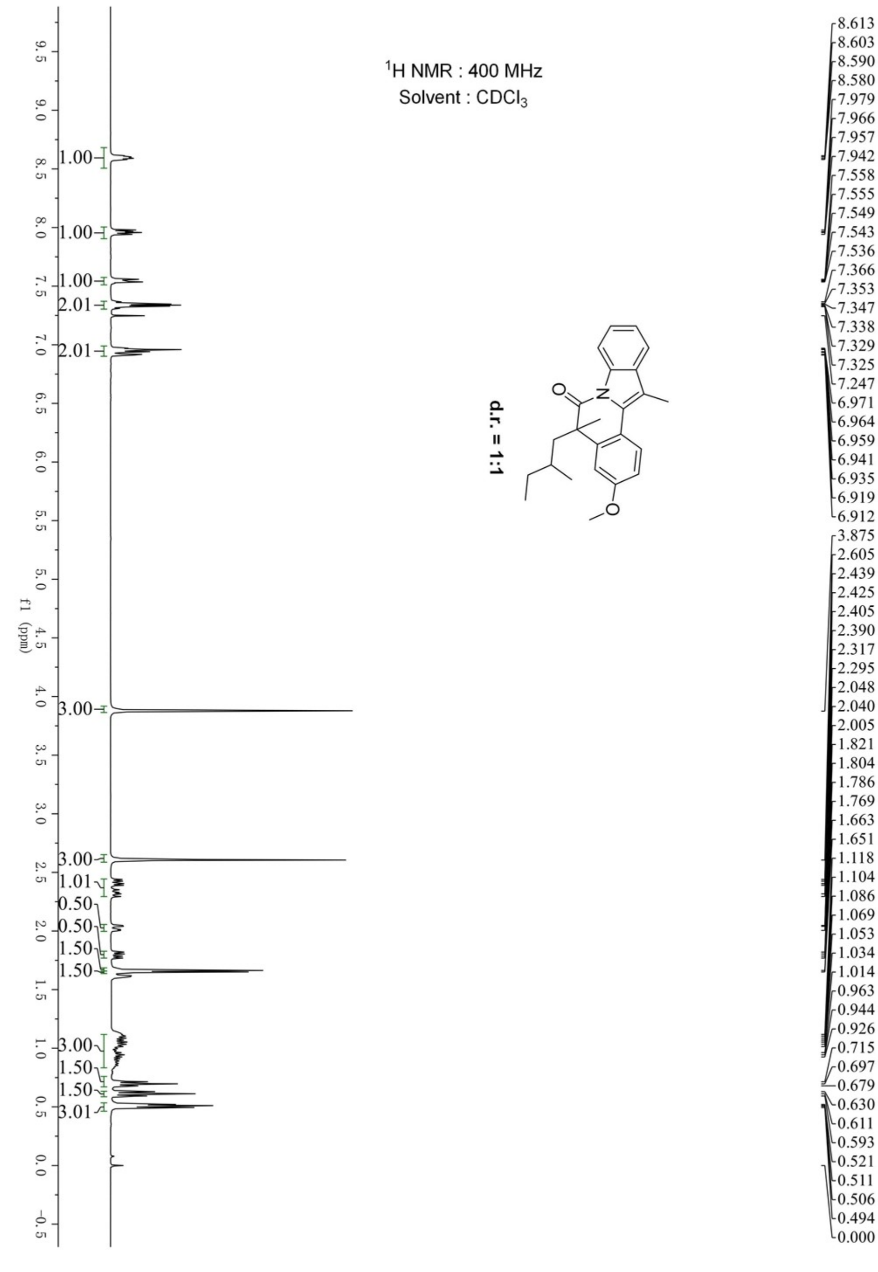
3-Methoxy-5



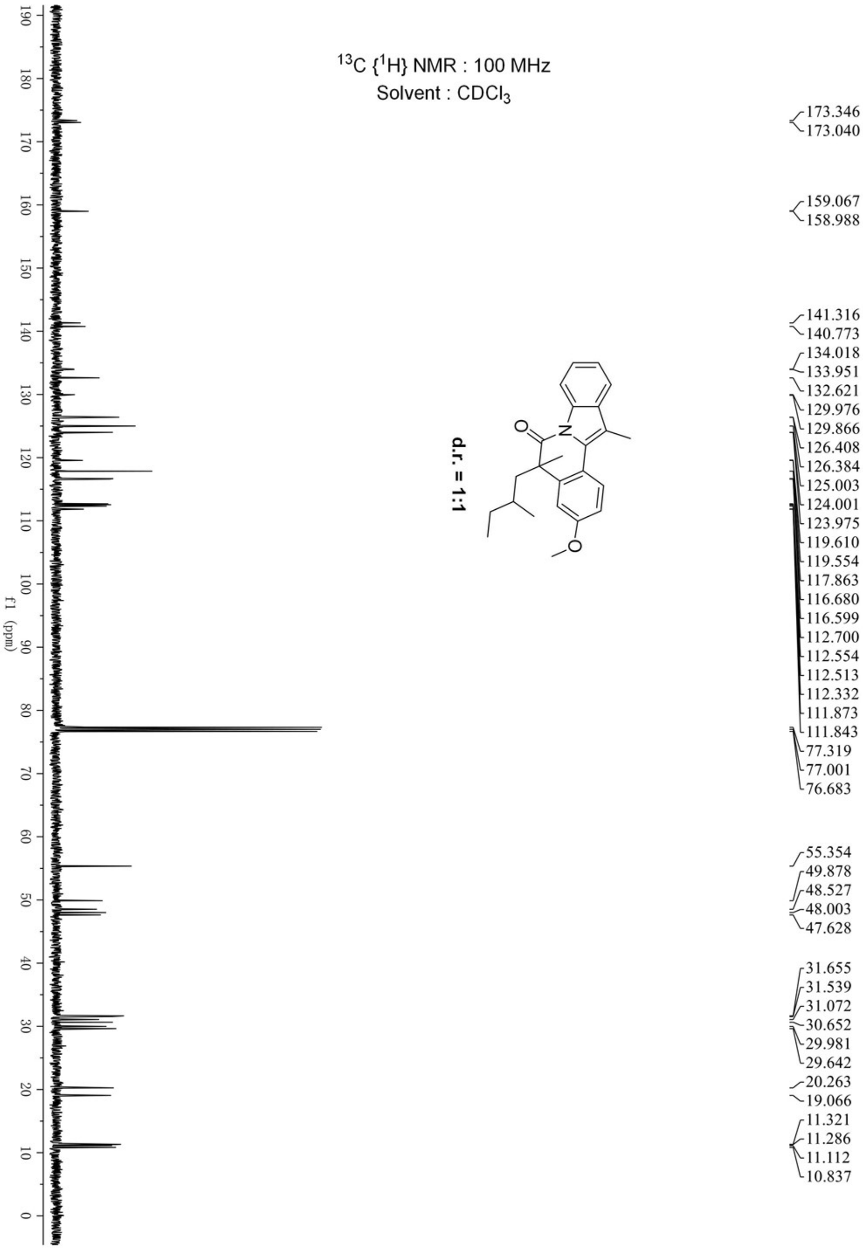
1H NMR : 400 MHz
Solvent : CDCl3
Bn
N
O
f1 (ppm)
8.606
8.600
8.592
8.587
8.583
7.997
7.976
7.569
7.566
7.560
7.553
7.547
7.384
7.370
7.363
7.355
7.346
7.341
7.327
7.251
7.178
7.162
7.160
7.144
7.141
7.097
7.096
7.093
7.082
7.077
7.072
7.059
7.016
7.015
7.007
6.998
6.986
6.979
3.902
2.742
2.727
2.710
2.696
2.675
2.665
2.621
2.334
2.322
2.303
2.288
2.254
2.245
2.224
2.215
2.190
2.167
2.156
2.134
1.700
1.581


$^{13}C\ \{^1H\}$ NMR : 100 MHz
Solvent : $CDCl_3$
172.546
159.345
141.284
140.449
133.910
132.630
129.871
128.192
126.567
125.781
125.118
124.107
119.898
117.950
116.562
112.762
112.163
111.851
77.318
77.001
76.683
55.402
48.711
44.604
31.485
28.971
11.297
O
N
Bn
O
f1 (ppm)

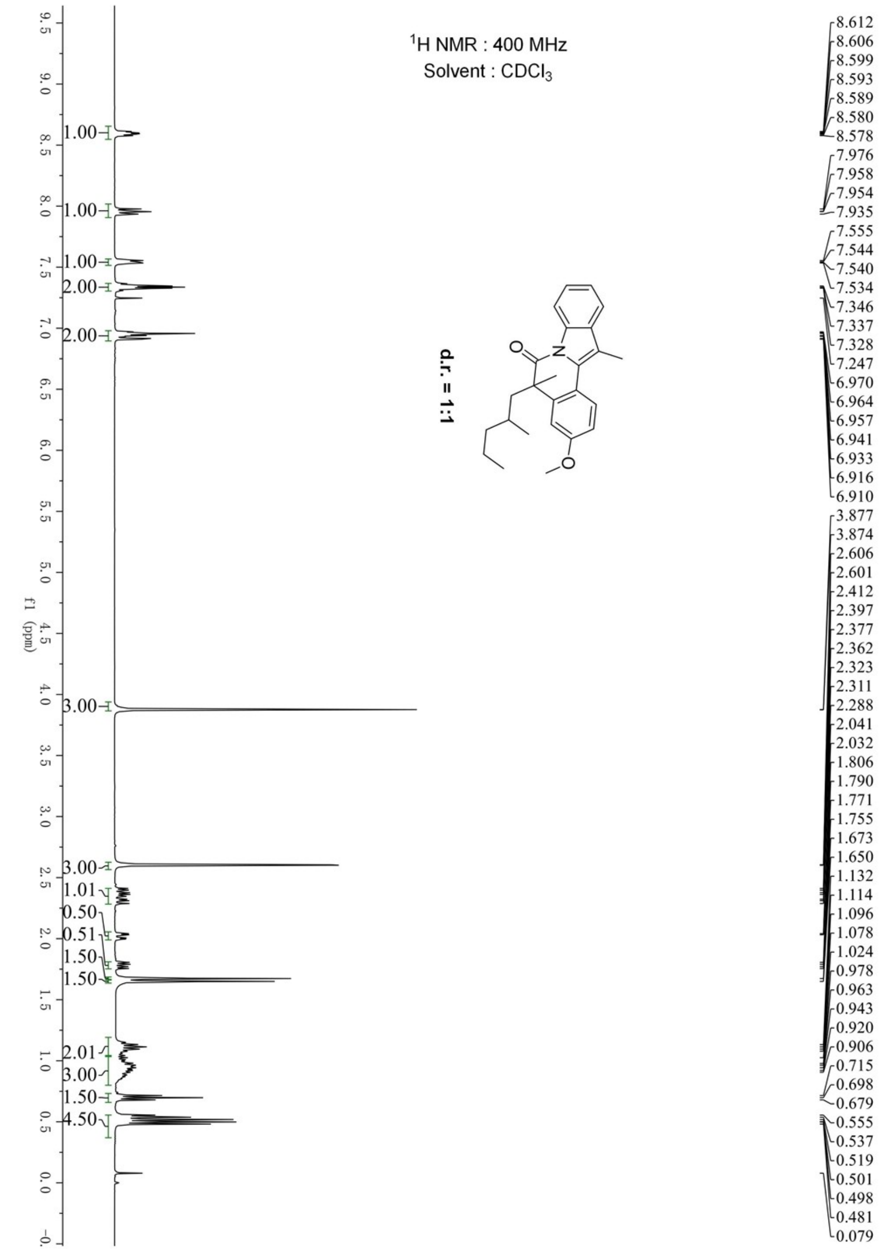
3-Methoxy-5

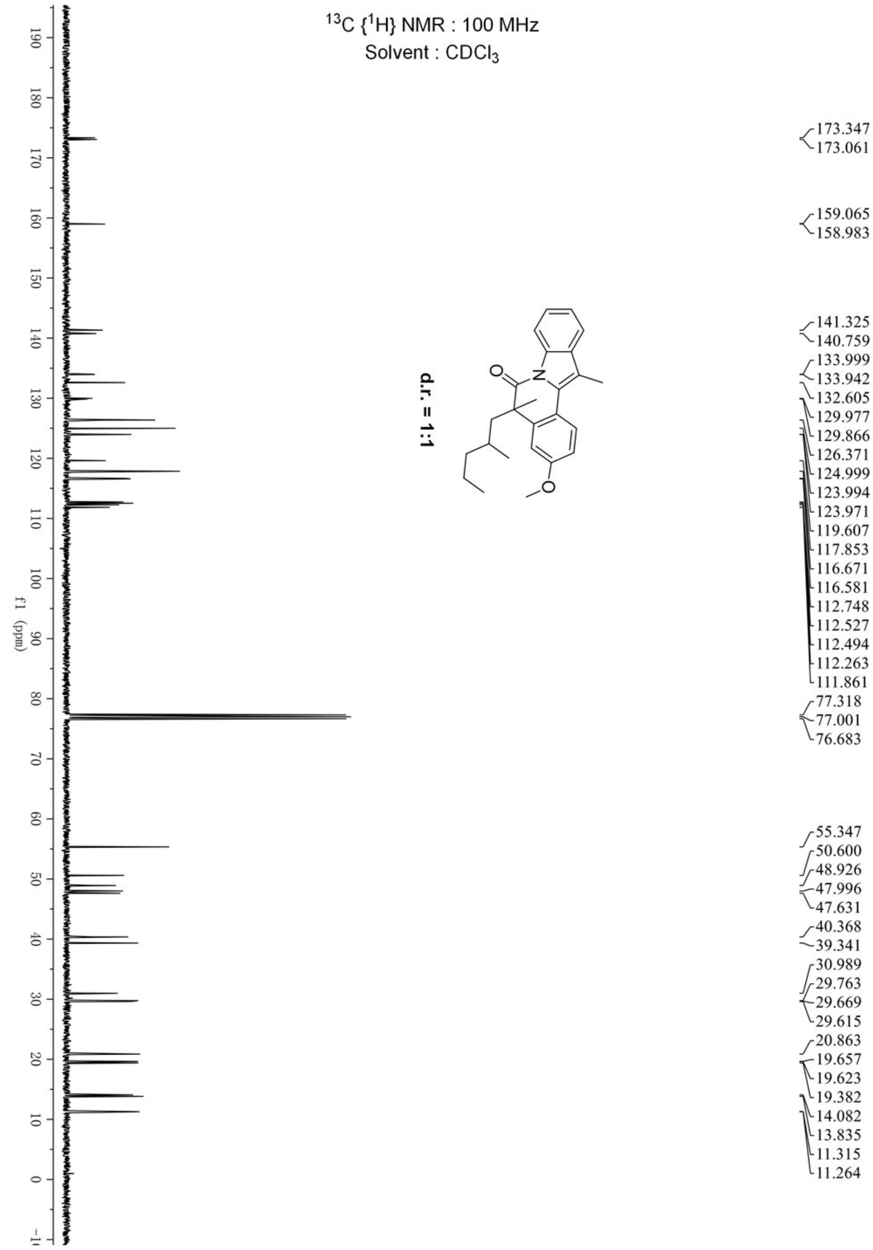


^{1}H NMR : 400 MHz
Solvent : CDCl$_3$
d.r. = 1:1
8.613
8.603
8.590
8.580
7.979
7.966
7.957
7.942
7.558
7.555
7.549
7.543
7.536
7.366
7.353
7.347
7.338
7.329
7.325
7.247
6.971
6.964
6.959
6.941
6.935
6.919
6.912
3.875
2.605
2.439
2.425
2.405
2.390
2.317
2.295
2.048
2.040
2.005
1.821
1.804
1.786
1.769
1.663
1.651
1.118
1.104
1.086
1.069
1.053
1.034
1.014
0.963
0.944
0.926
0.715
0.697
0.679
0.630
0.611
0.593
0.521
0.511
0.506
0.494
0.000
f1 (ppm)


^{13}C {^{1}H} NMR : 100 MHz
Solvent : CDCl$_3$
d.r. = 1:1
173.346
173.040
159.067
158.988
141.316
140.773
134.018
133.951
132.621
129.976
129.866
126.408
126.384
125.003
124.001
123.975
119.610
119.554
117.863
116.680
116.599
112.700
112.554
112.513
112.332
111.873
111.843
77.319
77.001
76.683
55.354
49.878
48.527
48.003
47.628
31.655
31.539
31.072
30.652
29.981
29.642
20.263
19.066
11.321
11.286
11.112
10.837
f1 (ppm)

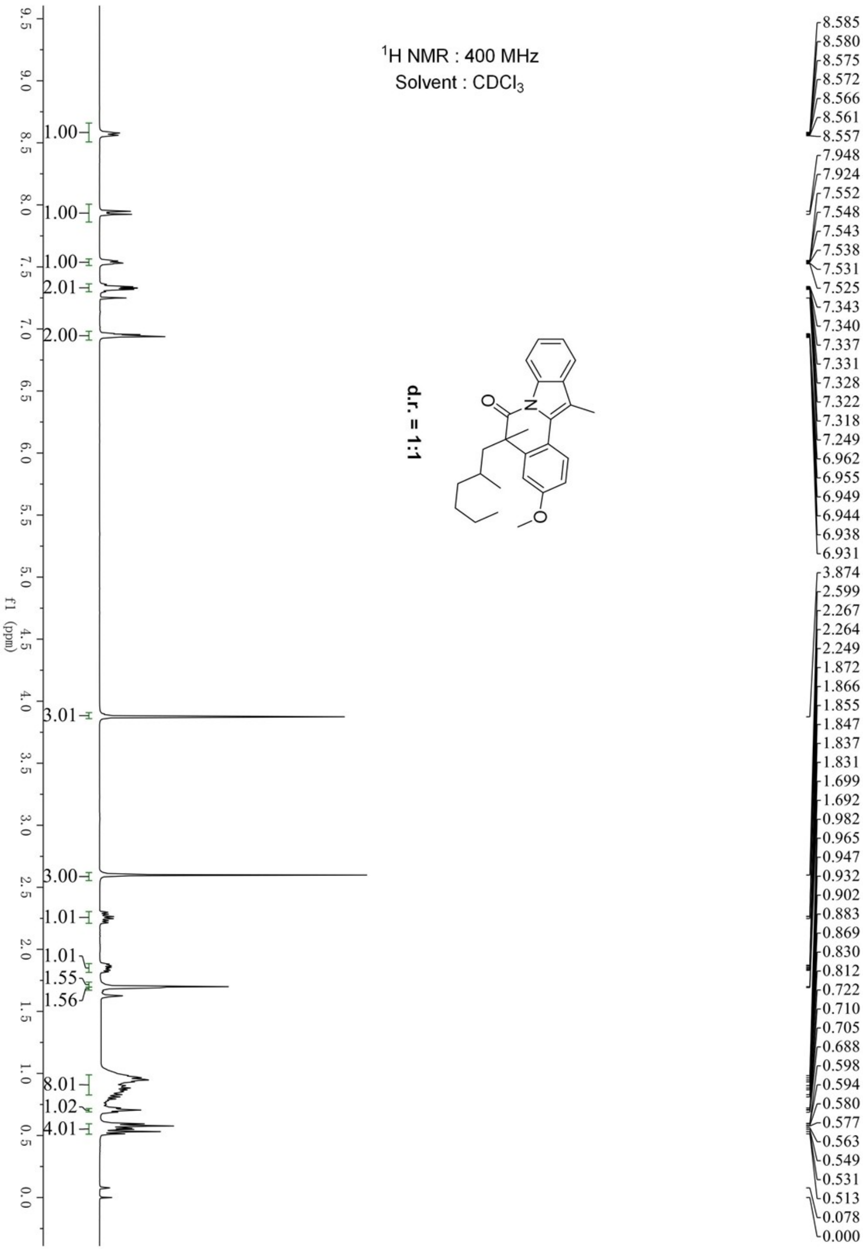
3-Methoxy-5



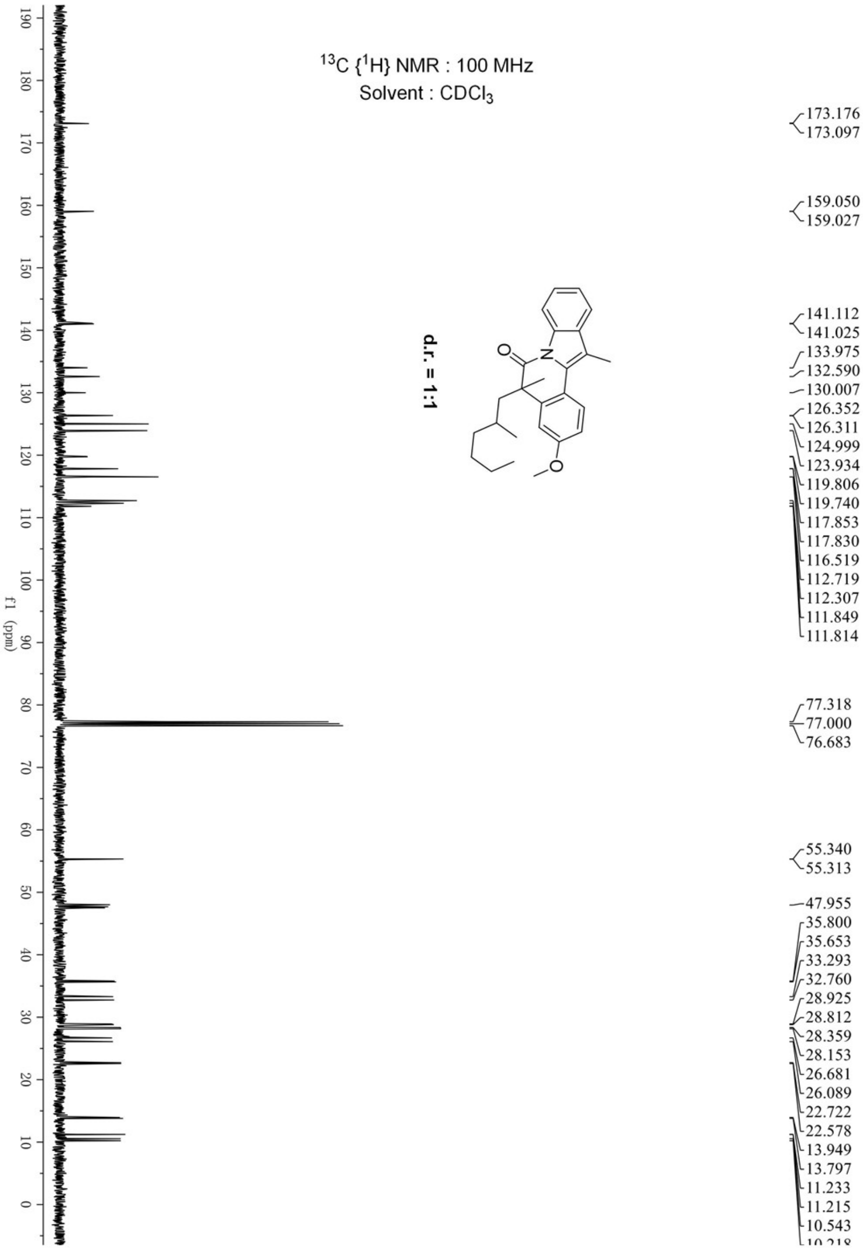


^{13}C {^{1}H} NMR : 100 MHz
Solvent : $CDCl_3$
d.r. = 1:1
173.347
173.061
159.065
158.983
141.325
140.759
133.999
133.942
132.605
129.977
129.866
126.371
124.999
123.994
123.971
119.607
117.853
116.671
116.581
112.748
112.527
112.494
112.263
111.861
77.318
77.001
76.683
55.347
50.600
48.926
47.996
47.631
40.368
39.341
30.989
29.763
29.669
29.615
20.863
19.657
19.623
19.382
14.082
13.835
11.315
11.264
f1 (ppm)

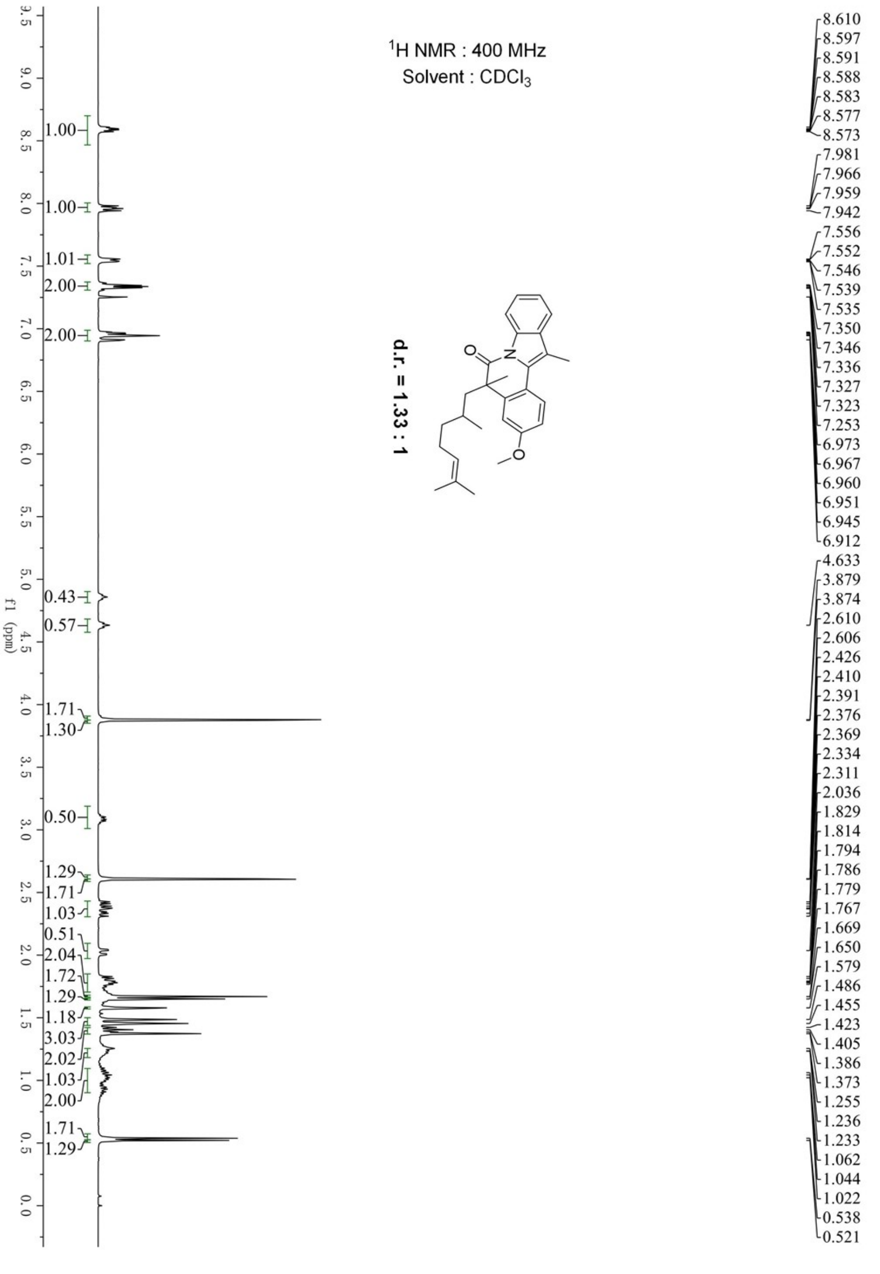
3-Methoxy-5

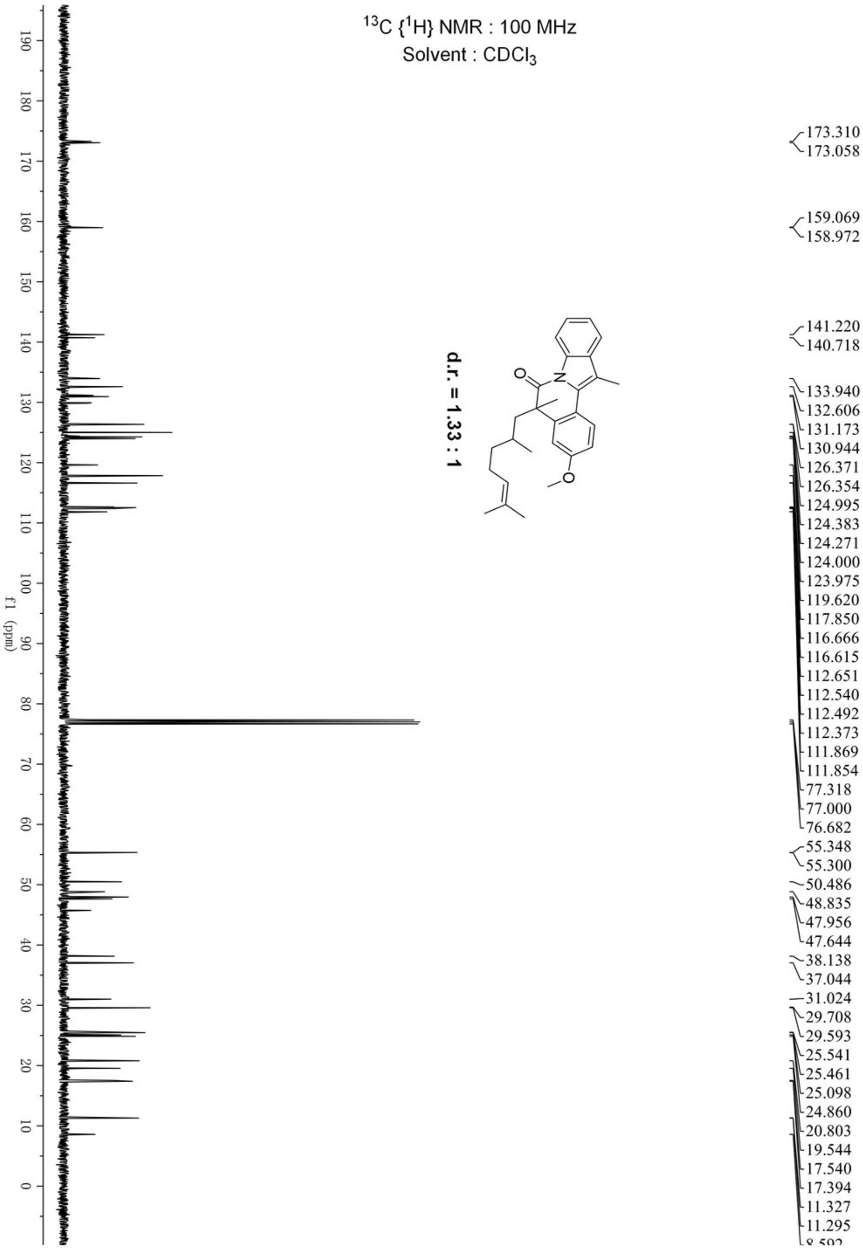


^{1}H NMR : 400 MHz
Solvent : $CDCl_3$
d.r. = 1:1
f1 (ppm)
8.585 8.580 8.575 8.572 8.566 8.561 8.557
7.948 7.924 7.552 7.548 7.543 7.538 7.531 7.525
7.343 7.340 7.337 7.331 7.328 7.322 7.318 7.249
6.962 6.955 6.949 6.944 6.938 6.931
3.874 2.599 2.267 2.264 2.249
1.872 1.866 1.855 1.847 1.837 1.831 1.699 1.692
0.982 0.965 0.947 0.932 0.902 0.883 0.869 0.830 0.812
0.722 0.710 0.705 0.688 0.598 0.594 0.580 0.577 0.563 0.549 0.531 0.513
0.078 0.000
1.00 1.00 1.00 2.01 2.00 3.01 3.00 1.01 1.01 1.55 1.56 8.01 1.02 4.01

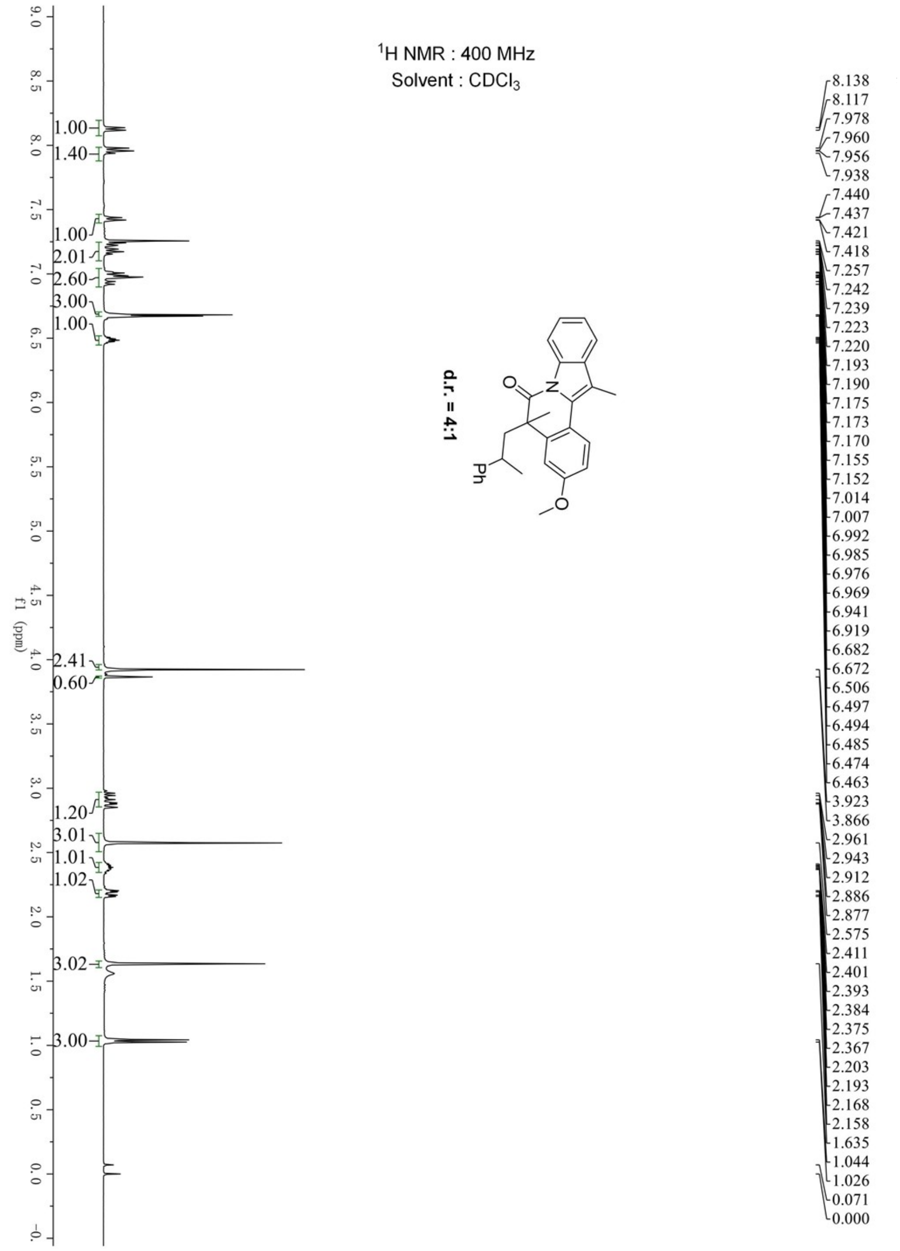

^{13}C {^{1}H} NMR : 100 MHz
Solvent : $CDCl_3$
d.r. = 1:1
173.176
173.097
159.050
159.027
141.112
141.025
133.975
132.590
130.007
126.352
126.311
124.999
123.934
119.806
119.740
117.853
117.830
116.519
112.719
112.307
111.849
111.814
77.318
77.000
76.683
55.340
55.313
47.955
35.800
35.653
33.293
32.760
28.925
28.812
28.359
28.153
26.681
26.089
22.722
22.578
13.949
13.797
11.233
11.215
10.543
f1 (ppm)

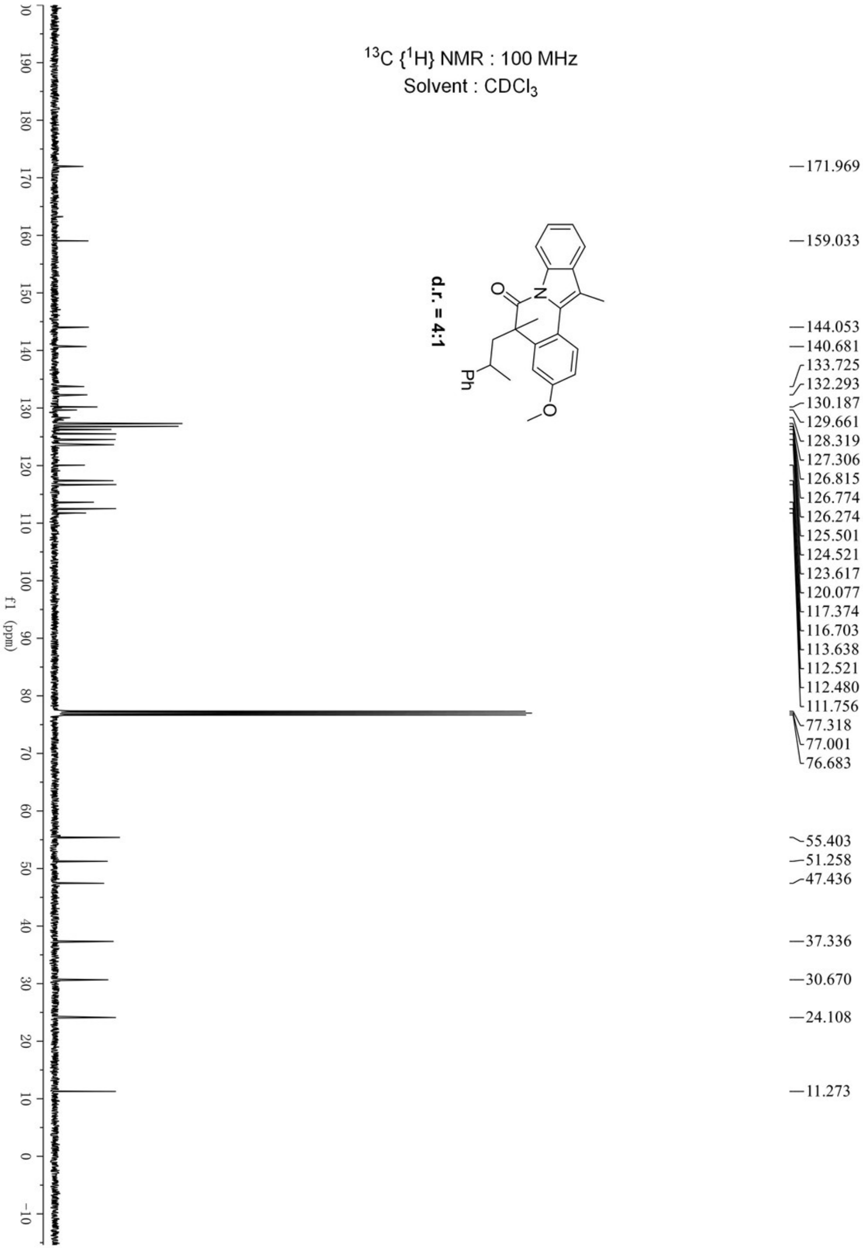
5-(2,6-Dimeth -




$^{13}C\ \{^{1}H\}$ NMR : 100 MHz
Solvent : $CDCl_3$
d.r. = 1.33 : 1
f1 (ppm)
173.310
173.058
159.069
158.972
141.220
140.718
133.940
132.606
131.173
130.944
126.371
126.354
124.995
124.383
124.271
124.000
123.975
119.620
117.850
116.666
116.615
112.651
112.540
112.492
112.373
111.869
111.854
77.318
77.000
76.682
55.348
55.300
50.486
48.835
47.956
47.644
38.138
37.044
31.024
29.708
29.593
25.541
25.461
25.098
24.860
20.803
19.544
17.540
17.394
11.327
11.295

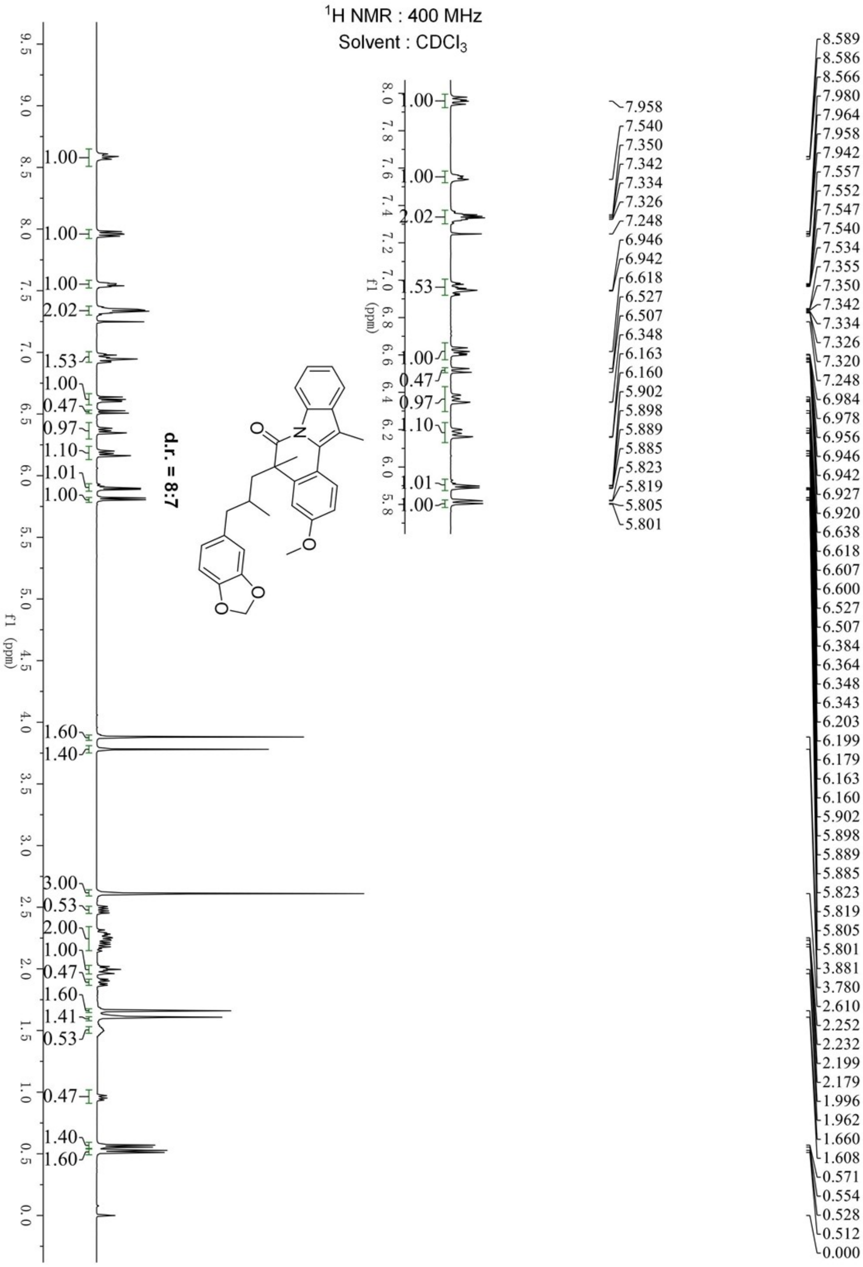
3-Methoxy-5


^{1}H NMR : 400 MHz
Solvent : $CDCl_3$
d.r. = 4:1
Ph
N
O
f1 (ppm)
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
1.00
1.40
1.00
2.01
2.60
3.00
1.00
2.41
0.60
1.20
3.01
1.01
1.02
3.02
3.00
8.138
8.117
7.978
7.960
7.956
7.938
7.440
7.437
7.421
7.418
7.257
7.242
7.239
7.223
7.220
7.193
7.190
7.175
7.173
7.170
7.155
7.152
7.014
7.007
6.992
6.985
6.976
6.969
6.941
6.919
6.682
6.672
6.506
6.497
6.494
6.485
6.474
6.463
3.923
3.866
2.961
2.943
2.912
2.886
2.877
2.575
2.411
2.401
2.393
2.384
2.375
2.367
2.203
2.193
2.168
2.158
1.635
1.044
1.026
0.071
0.000



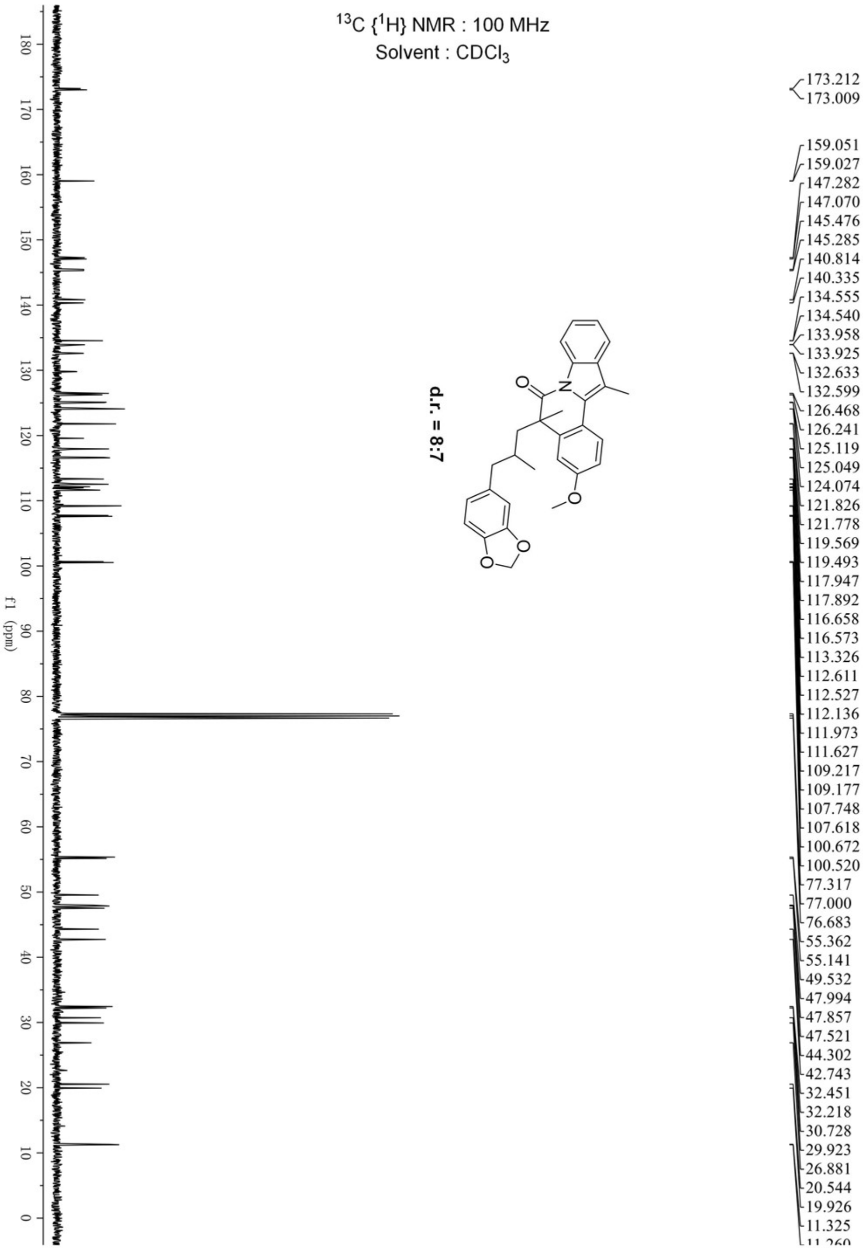
^{13}C {^{1}H} NMR : 100 MHz
Solvent : $CDCl_3$
d.r. = 4:1
Ph
N
O
171.969
159.033
144.053
140.681
133.725
132.293
130.187
129.661
128.319
127.306
126.815
126.774
126.274
125.501
124.521
123.617
120.077
117.374
116.703
113.638
112.521
112.480
111.756
77.318
77.001
76.683
55.403
51.258
47.436
37.336
30.670
24.108
11.273
f1 (ppm)

5-(3-(

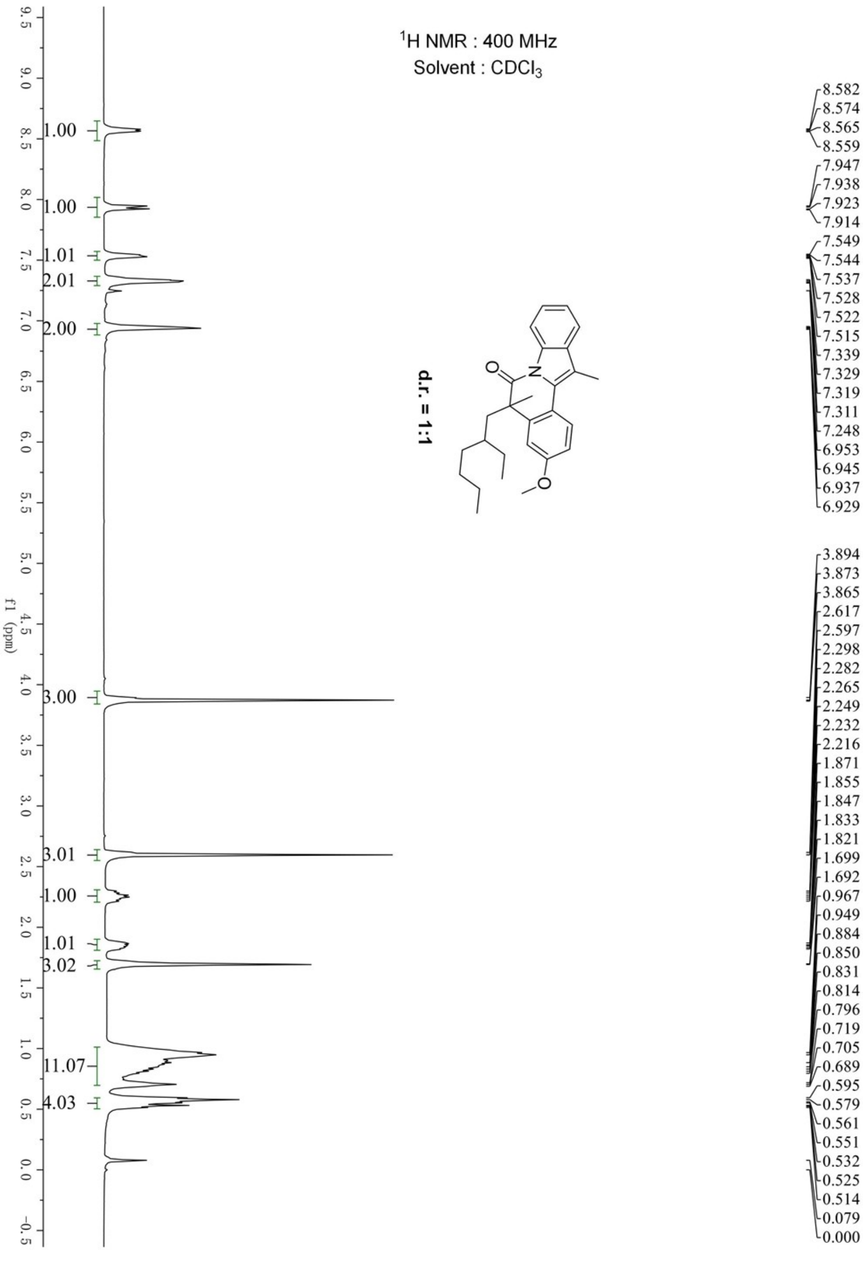


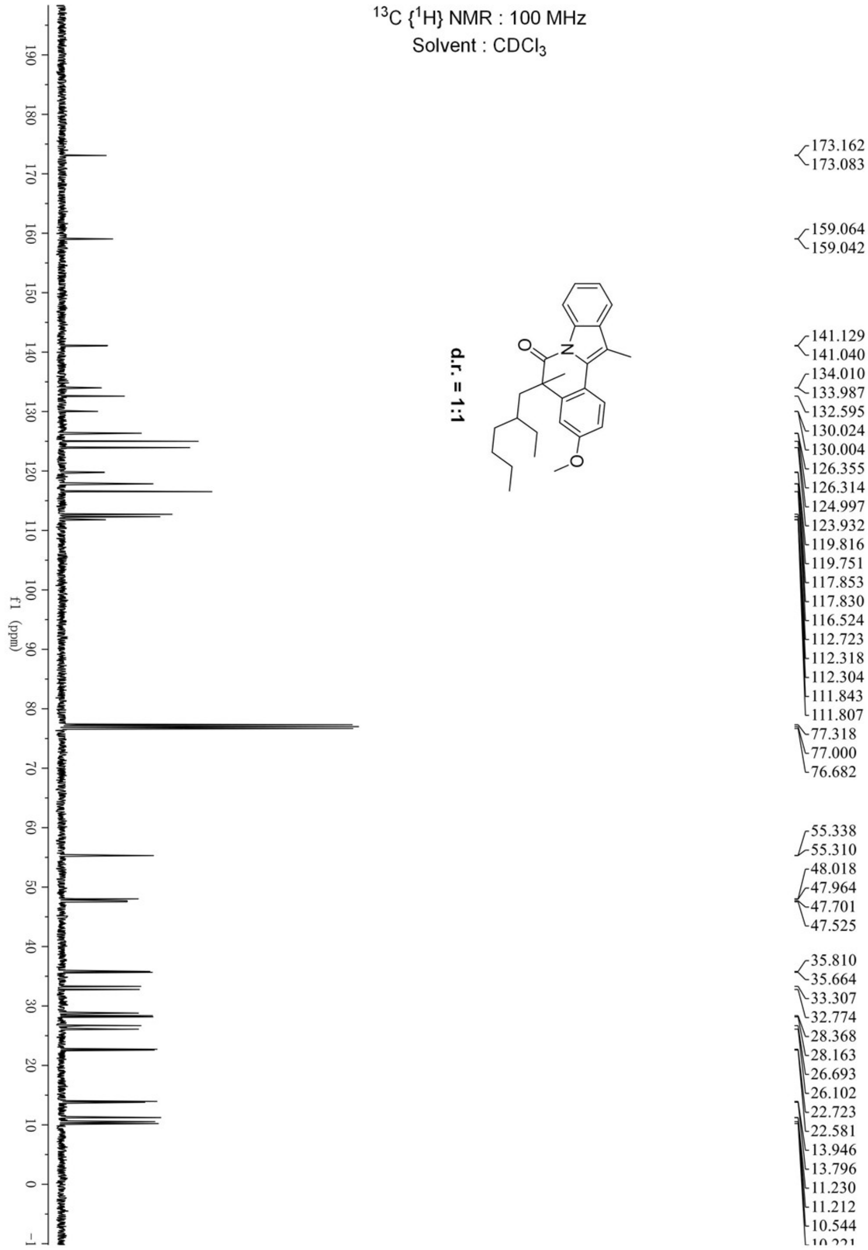
$^{13}C\{^{1}H\}$ NMR : 100 MHz

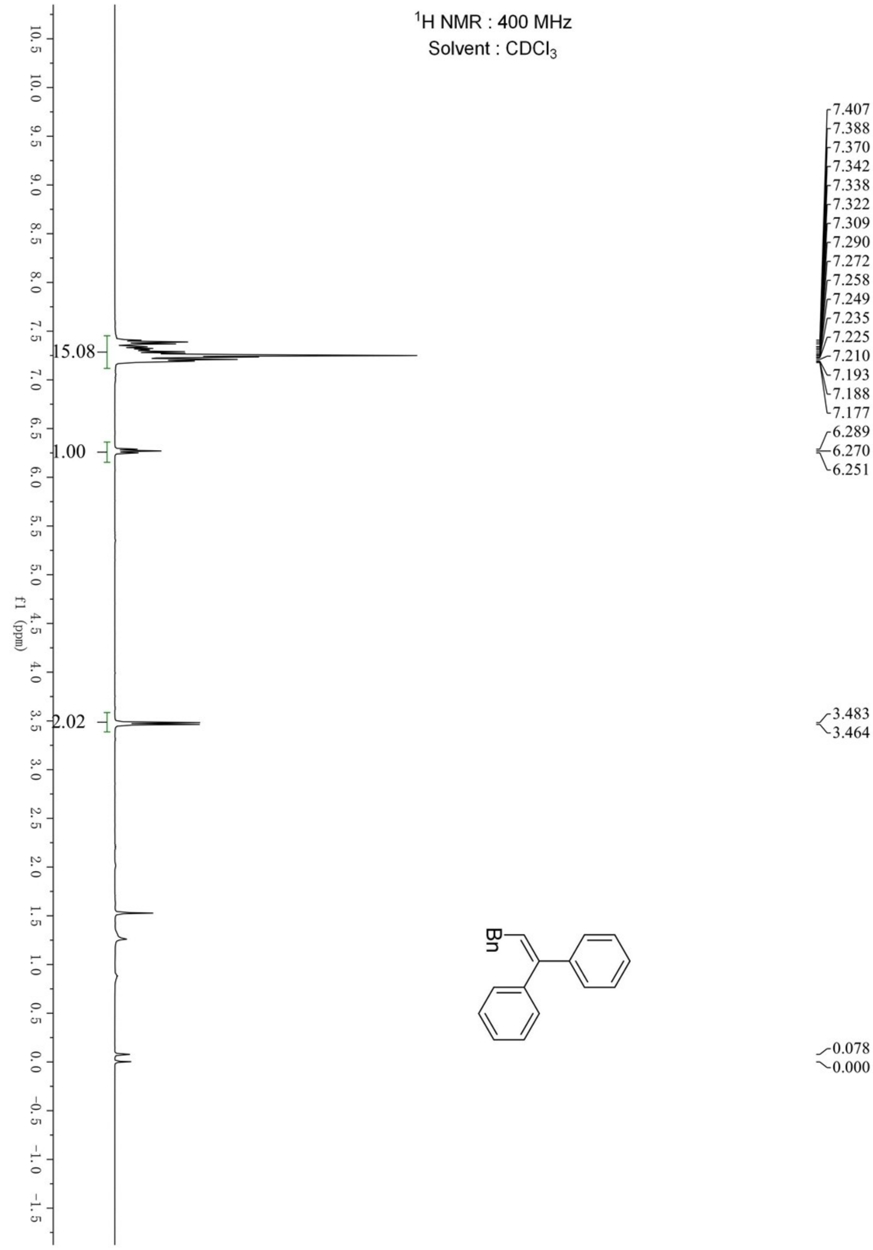
Solvent : $CDCl_3$



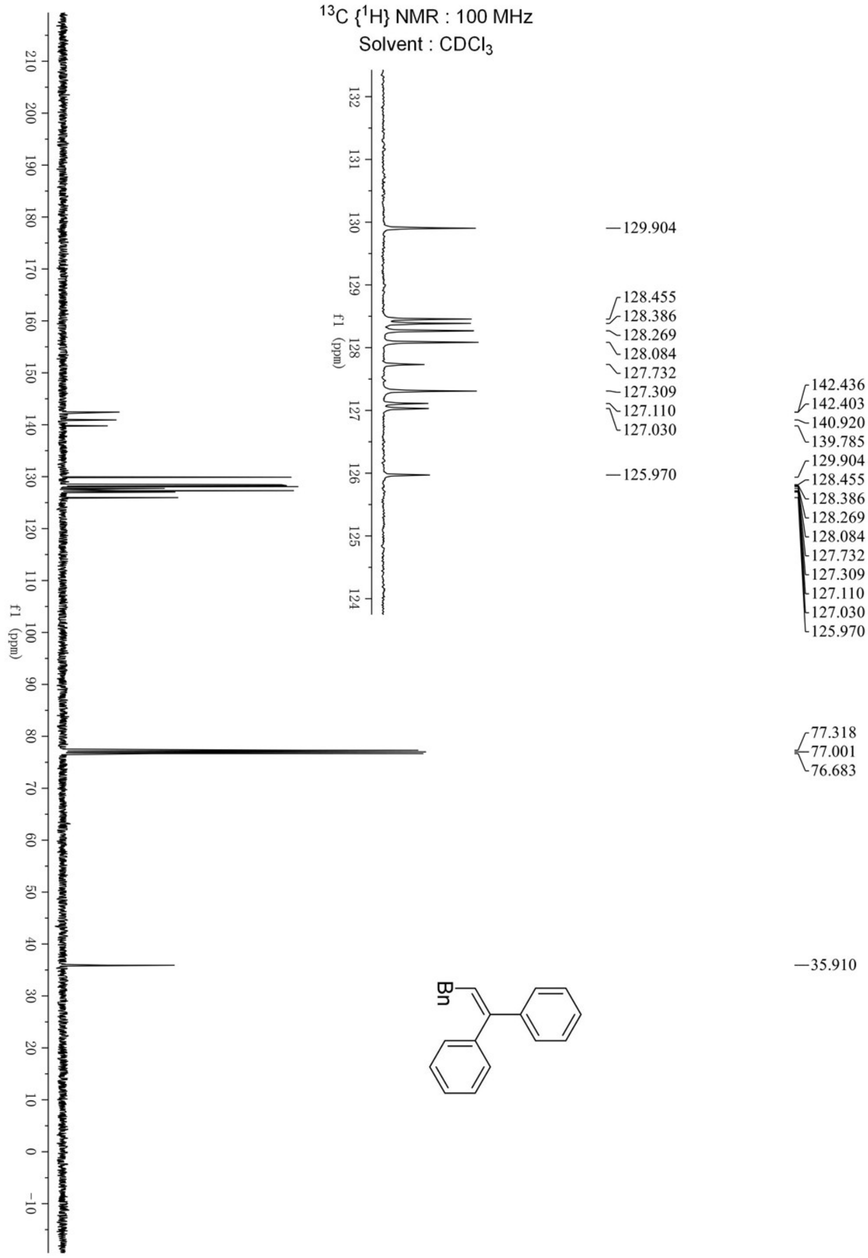
5-(2-Ethylh


1H NMR : 400 MHz
Solvent : CDCl3
d.r. = 1:1
8.582
8.574
8.565
8.559
7.947
7.938
7.923
7.914
7.549
7.544
7.537
7.528
7.522
7.515
7.339
7.329
7.319
7.311
7.248
6.953
6.945
6.937
6.929
3.894
3.873
3.865
2.617
2.597
2.298
2.282
2.265
2.249
2.232
2.216
1.871
1.855
1.847
1.833
1.821
1.699
1.692
0.967
0.949
0.884
0.850
0.831
0.814
0.796
0.719
0.705
0.689
0.595
0.579
0.561
0.551
0.532
0.525
0.514
0.079
0.000
f1 (ppm)


13C {1H} NMR : 100 MHz
Solvent : CDCl3
d.r. = 1:1
f1 (ppm)
173.162
173.083
159.064
159.042
141.129
141.040
134.010
133.987
132.595
130.024
130.004
126.355
126.314
124.997
123.932
119.816
119.751
117.853
117.830
116.524
112.723
112.318
112.304
111.843
111.807
77.318
77.000
76.682
55.338
55.310
48.018
47.964
47.701
47.525
35.810
35.664
33.307
32.774
28.368
28.163
26.693
26.102
22.723
22.581
13.946
13.796
11.230
11.212
10.544



^{1}H NMR : 400 MHz
Solvent : $CDCl_3$
7.407
7.388
7.370
7.342
7.338
7.322
7.309
7.290
7.272
7.258
7.249
7.235
7.225
7.210
7.193
7.188
7.177
6.289
6.270
6.251
3.483
3.464
0.078
0.000
15.08
1.00
2.02
f1 (ppm)
Bn



13C {1H} NMR : 100 MHz
Solvent : CDCl3
129.904
128.455
128.386
128.269
128.084
127.732
127.309
127.110
127.030
125.970
142.436
142.403
140.920
139.785
129.904
128.455
128.386
128.269
128.084
127.732
127.309
127.110
127.030
125.970
77.318
77.001
76.683
35.910
Bn
f1 (ppm)