Electronic Supplementary Material (ESI) for ChemComm.

Supplementary information

Copper-Promoted [2+2+2] Annulation of 1,*n*-Enynes through Decomposition of Azobis(alkyl nitrile)s

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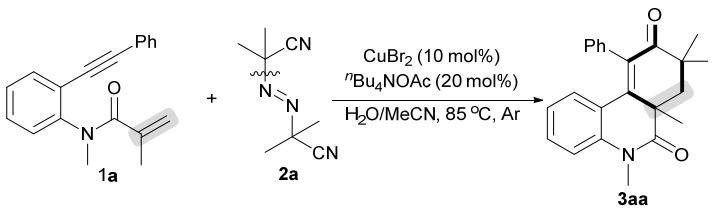
(A) Typical experimental procedure

(a) Preparation of Substrates 1:

Substrates **1** were prepared according to literature procedures.[1]

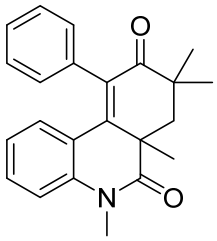
(b) General Procedures for the [2+2+2] Annulation of 1,*n*-Enynes with Azobis(alkyl nitrile)s:



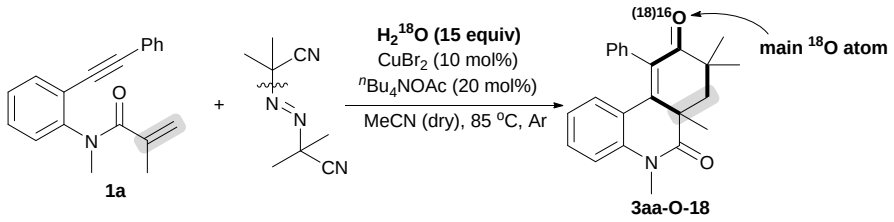
To a Schlenk tube were added 1,7-enyne **1a** (0.1 mmol), AIBN **2a** (5 equiv), $CuBr_2$ (10 mol%), *n*-Bu_4NOAc (20 mol%), and H_2O/MeCN (v/v=3/0.4; 3.4 mL). The mixture was stirred at 65-85 °C under argon atmosphere for 24 h until complete consumption of startingmaterial as monitored by TLC and/or GC-MS analysis. After the reaction was finished, the reaction mixture was cooled to room temperature, diluted with ethyl acetate, and washed with 10% (w/w) H_2SO_4 and saturated $NaHCO_3$. The aqueous phase was extracted with ethyl acetate and washed with saturated $NaHCO_3$. The combined organic phases were dried over anhydrous Na_2SO_4 and concentrated, then purified by column chromatography (petroleum ether/ethyl acetate, v/v=10/1) to afford the target product **3aa**.

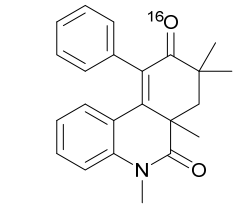
(B) Analytical data

5,6a,8,8-Tetramethyl-10-phenyl-7,8-dihydrophenanthridine-6,9(5*H*,6a*H*)-dione (3aa)



White solid, mp 159.5 - 160.7 °C (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 – 7.20 (m, 4H), 7.02 – 6.99 (m, 3H), 6.68 (t, *J* = 7.6 Hz, 1H), 6.61 (d, *J* = 7.6 Hz, 1H), 3.44 (s, 3H), 2.89 (d, *J* = 14.8 Hz, 1H), 2.04 (d, *J* = 14.8 Hz, 1H), 1.33 (s, 3H), 1.32 (s, 3H), 1.24 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.5, 172.5, 148.2, 139.2, 135.4, 134.4, 130.6, 130.3, 129.7, 127.9, 127.3, 123.4, 122.3, 114.2, 43.1, 42.3, 40.8, 30.5, 27.3, 26.2, 25.8; LRMS (EI, 70 eV) *m/z* (%): 345 (M^+, 100), 330 (34), 302 (31), 289 (58), 274 (61), 260 (80); HRMS (ESI): calculated for [$C_{23}H_{24}NO_2^+$] 346.1802, found 346.1798.





Chemical Formula: $C_{23}H_{23}N^{16}O_2$
Molecular Weight: 345

[MS Spectrum] # of Peaks 327

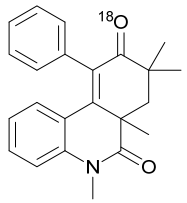
Raw Spectrum 18.525 (scan : 3006) Base Peak m/z 344.90 (Inten : 1,174,894)

Background 18.245 (scan : 2950)

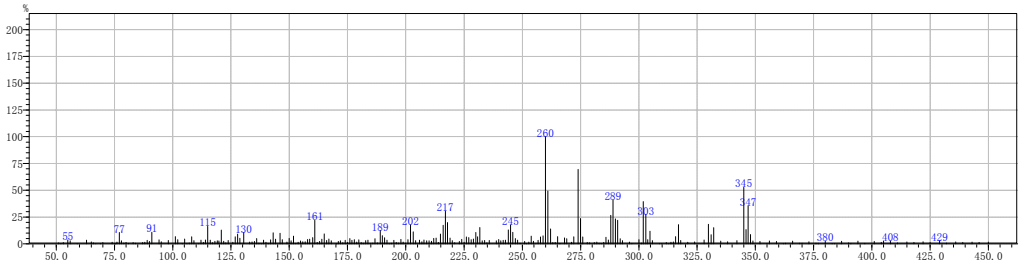
m/zAbsolute Intensity Relative Intensity

151.90	40598	3.46	165.95	15798	1.34	176.95	22462	1.91
157.05	36145	3.08	166.95	28882	2.46	177.95	34651	2.95
158.00	48871	4.16	168.00	18766	1.60	180.95	16184	1.38
158.95	26421	2.25	172.10	45276	3.85	181.95	25869	2.20
160.00	30018	2.55	173.05	22915	1.95	182.95	17095	1.46
160.95	129716	11.04	173.90	13782	1.17	183.95	37663	3.21
164.95	66076	5.62	175.95	35228	3.00	184.95	18583	1.58

185.95	16337	1.39	227.90	49083	4.18	274.90	198815	16.92
186.90	23834	2.03	228.90	27154	2.31	275.85	38709	3.29
187.95	14705	1.25	229.90	73204	6.23	285.90	46473	3.96
188.95	57513	4.90	230.90	74543	6.34	286.95	57771	4.92
189.90	42523	3.62	231.90	99036	8.43	287.90	374649	31.89
190.95	30008	2.55	232.85	24766	2.11	288.90	684703	58.28
191.95	12015	1.02	233.90	48522	4.13	289.90	148159	12.61
193.00	14161	1.21	234.85	13308	1.13	290.85	18564	1.58
196.00	19403	1.65	235.90	23944	2.04	299.95	16752	1.43
196.95	12042	1.02	239.90	43460	3.70	300.95	18014	1.53
197.95	33494	2.85	240.90	31488	2.68	301.90	366080	31.16
199.90	19019	1.62	241.90	30297	2.58	302.90	316250	26.92
200.95	26964	2.30	242.85	26531	2.26	303.90	61578	5.24
201.90	82695	7.04	243.90	73360	6.24	311.90	12471	1.06
202.95	63963	5.44	244.85	144628	12.31	313.90	33674	2.87
203.95	52645	4.48	245.90	84944	7.23	314.90	42627	3.63
209.90	24155	2.06	246.90	34118	2.90	315.95	65687	5.59
210.90	39329	3.35	257.85	46140	3.93	316.95	151821	12.92
211.90	39092	3.33	258.95	75393	6.42	317.95	35910	3.06
214.90	47587	4.05	259.90	939443	79.96	327.95	12280	1.05
215.90	98012	8.34	260.85	368905	31.40	329.90	401760	34.20
216.90	203745	17.34	261.90	135030	11.49	330.90	98545	8.39
217.90	133796	11.39	262.90	53112	4.52	343.95	23247	1.98
218.90	39284	3.34	263.85	13909	1.18	**344.90**	**1174894**	**100.00**
223.95	36632	3.12	267.90	28869	2.46	345.90	306620	26.10
224.90	13643	1.16	271.90	34950	2.97	**346.90**	**41804**	**3.56**
225.90	52048	4.43	272.95	31316	2.67	347.85	5372	0.46
226.90	31975	2.72	273.90	710807	60.50	350.90	1036	0.09



Chemical Formula: $C_{23}H_{23}NO^{18}O$
Molecular Weight: 347



[MS Spectrum] # of Peaks 273

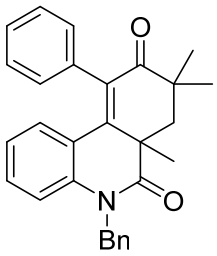
Raw Spectrum 18.645 (scan : 3030) Base Peak m/z 259.90 (Inten : 11,000)

Background 18.485 (scan : 2998)

m/z	Absolute Intensity	Relative Intensity	m/z	Absolute Intensity	Relative Intensity	m/z	Absolute Intensity	Relative Intensity
150.00	554	5.04	167.00	514	4.67	190.90	627	5.70
150.80	313	2.85	168.00	308	2.80	191.90	378	3.44
151.80	789	7.17	171.00	239	2.17	194.90	365	3.32
155.80	220	2.00	172.00	313	2.85	197.90	480	4.36
156.80	229	2.08	174.00	362	3.29	200.90	441	4.01
157.80	458	4.16	176.00	543	4.94	202.00	1962	17.84
158.80	487	4.43	176.90	360	3.27	203.20	1242	11.29
160.05	638	5.80	177.90	445	4.05	204.20	339	3.08
161.00	2433	22.12	179.80	427	3.88	205.90	385	3.50
162.00	153	1.39	182.80	334	3.04	206.90	224	2.04
163.00	243	2.21	183.80	377	3.43	207.80	402	3.65
164.00	488	4.44	186.80	519	4.72	208.95	306	2.78
165.00	1028	9.35	189.05	1374	12.49	210.00	319	2.90
166.00	341	3.10	189.90	830	7.55	211.00	263	2.39

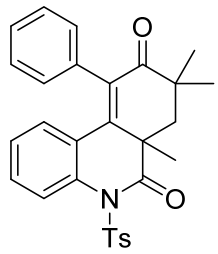
212.00	557	5.06	245.00	1935	17.59	293.00	327	2.97
213.00	596	5.42	247.00	549	4.99	296.00	197	1.79
214.00	161	1.46	248.00	377	3.43	300.00	438	3.98
214.85	1007	9.15	252.80	194	1.76	301.95	4358	39.62
216.00	1899	17.26	253.80	788	7.16	303.00	3019	27.45
217.00	3358	30.53	254.80	258	2.35	303.80	433	3.94
217.95	2175	19.77	256.80	365	3.32	304.75	1312	11.93
219.00	600	5.45	257.80	719	6.54	305.80	333	3.03
220.00	327	2.97	258.90	822	7.47	315.80	748	6.80
223.00	246	2.24	259.90	11000	100.00	317.00	1973	17.94
224.00	465	4.23	260.95	5438	49.44	318.00	346	3.15
226.00	719	6.54	262.10	1538	13.98	321.00	167	1.52
227.00	642	5.84	265.10	757	6.88	324.00	203	1.85
228.00	428	3.89	268.10	608	5.53	328.00	394	3.58
229.00	499	4.54	269.10	527	4.79	329.85	2004	18.22
230.80	738	6.71	271.10	174	1.58	331.00	936	8.51
231.80	1674	15.22	272.10	740	6.73	332.10	1657	15.06
232.80	310	2.82	273.90	7643	69.48	335.10	279	2.54
233.80	350	3.18	274.95	2610	23.73	338.10	226	2.05
234.80	122	1.11	275.90	716	6.51	342.10	334	3.04
235.80	346	3.15	285.90	687	6.25	**345.00**	**5797**	**52.70**
238.80	315	2.86	286.90	404	3.67	345.95	1467	13.34
239.80	456	4.15	287.90	2933	26.66	**346.95**	**3901**	**35.46**
240.80	322	2.93	288.95	4517	41.06	347.95	957	8.70
241.80	374	3.40	290.00	2575	23.41	**349.00**	**297**	**2.70**
242.80	392	3.56	290.95	2436	22.15			
244.05	1428	12.98	292.00	517	4.70			

5-Benzyl-6a,8,8-trimethyl-10-phenyl-7,8-dihydrophenanthridine-6,9(5*H*,6a*H*)-dione (3ba)



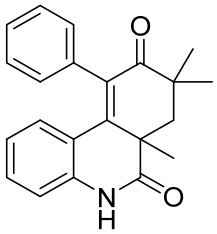
White solid, mp 178.3 – 180.1 ºC (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 (t, *J* = 7.6 Hz, 2H), 7.28 – 7.22(m, 6H), 7.08 – 7.04 (m, 1H), 6.98 (s, 2H), 6.91 (d, *J* = 8.0 Hz, 1H), 6.65 – 6.60 (m, 2H), 5.38 (d, *J* = 16.0 Hz, 1H), 5.09 (d, *J* = 16.0 Hz, 1H), 2.97 (d, *J* = 14.8 Hz, 1H), 2.07 (d, *J* = 14.8 Hz, 1H), 1.46 (s, 3H), 1.34 (s, 3H), 1.27 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.5, 172.7, 148.0, 138.5, 136.7, 135.4, 134.7, 130.7, 130.3, 129.7, 128.8, 128.0, 127.4, 127.3, 126.2, 123.8, 122.4, 115.2, 46.7, 43.3, 42.3, 40.9, 27.4, 26.2, 25.9; LRMS (EI, 70 eV) *m/z* (%): 421 (M^+, 68), 379 (11), 350 (15), 330 (21), 91 (100); HRMS (ESI): calculated for [$C_{29}H_{28}NO_2^+$] 422.2115, found 422.2111.

6a,8,8-Trimethyl-10-phenyl-5-tosyl-7,8-dihydrophenanthridine-6,9(5*H*,6a*H*)-dione (3ca)



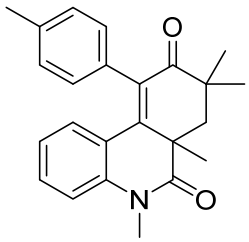
White solid, mp 191.5 – 192.9 ºC (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.4 Hz, 2H), 7.75 (d, *J* = 8.4 Hz, 1H), 7.33 – 7.30 (m, 6H), 7.05 – 7.03 (m, 2H), 6.93 (t, *J* = 7.6 Hz, 1H), 6.57 (d, *J* = 8.0 Hz, 1H), 2.77 (d, *J* = 14.8 Hz, 1H), 2.40 (s, 3H), 1.81 (d, *J* = 14.8 Hz, 1H), 1.11 (s, 3H), 1.09 (s, 3H), 0.48 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.6, 171.5, 145.6, 145.5, 135.6, 135.0, 134.0, 133.9, 130.4, 129.4, 129.3, 129.1, 128.9, 128.0, 127.8, 127.7, 125.9, 123.6, 45.4, 42.3, 40.5, 27.1, 24.8, 24.7, 21.6; LRMS (EI, 70 eV) *m/z* (%): 485 (M^+, 17), 421 (9), 330 (100), 302 (48); HRMS (ESI): calculated for [$C_{29}H_{28}NO_4S^+$] 486.1734, found 486.1728.

6a,8,8-Trimethyl-10-phenyl-7,8-dihydrophenanthridine-6,9(5*H*,6a*H*)-dione (3da)



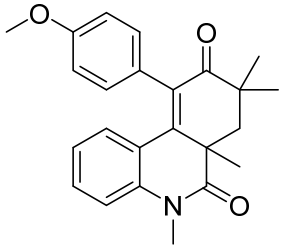
White solid, mp 210.2 – 211.8 ºC (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.03 (s, 1H), 7.26 (s, 3H), 7.15 (t, *J* = 7.6 Hz, 1H), 6.99 (s, 2H), 6.86 (d, *J* = 8.0 Hz, 1H), 6.66 – 6.62 (m, 1H), 6.57 (d, *J* = 8.0 Hz, 1H), 2.92 (d, *J* = 14.8 Hz, 1H), 2.04 (d, *J* = 14.8 Hz, 1H), 1.44 (s, 3H), 1.34 (s, 3H), 1.28 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.5, 174.3, 147.9, 136.2, 135.5, 135.3, 130.5, 130.4, 129.9, 128.0, 127.4, 122.4, 121.7, 115.2, 42.9, 41.4, 40.7, 27.6, 26.3, 25.7; LRMS (EI, 70 eV) *m/z* (%): 331 (M^+, 94), 316 (15), 288 (45), 275 (100), 246 (67); HRMS (ESI): calculated for [$C_{22}H_{22}NO_2^+$] 332.1645, found 332.1650.

5,6a,8,8-Tetramethyl-10-(*p*-tolyl)-7,8-dihydrophenanthridine-6,9(5*H*,6a*H*)-dione (3ea)



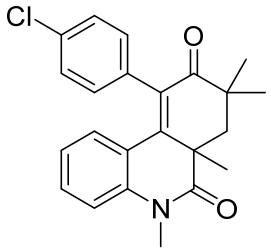
White solid, mp 132.5 – 134.3 ºC (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22 (t, *J* = 7.6 Hz, 1H), 7.05 (d, *J* = 7.6 Hz, 2H), 7.00 (d, *J* = 8.0 Hz, 1H), 6.87 (d, *J* = 7.6 Hz, 2H), 6.72 – 6.65 (m, 2H), 3.43 (s, 3H), 2.88 (d, *J* = 14.8 Hz, 1H), 2.31 (s, 3H), 2.03 (d, *J* = 14.8 Hz, 1H), 1.32 (s, 6H), 1.23 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.7, 172.6, 147.7, 139.2, 137.0, 134.4, 132.3, 130.5, 130.3, 129.6, 128.7, 123.6, 122.3, 114.1, 43.1, 42.4, 40.8, 30.4, 27.2, 26.2, 25.9, 21.2; LRMS (EI, 70 eV) *m/z* (%): 359 (M^+, 100), 344 (27), 303 (58), 288 (58), 274 (66); HRMS (ESI): calculated for [$C_{24}H_{26}NO_2^+$] 360.1958, found 360.1963.

10-(4-Methoxyphenyl)-5,6a,8,8-tetramethyl-7,8-dihydrophenanthridine-6,9(5*H*,6a*H*)-dione (3fa)



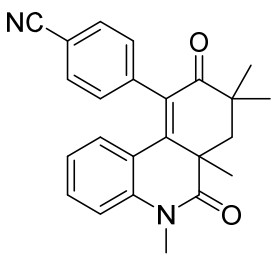
White solid, mp 126.7 – 128.1 ºC (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22 (t, *J* = 7.6 Hz, 1H), 7.01 (d, *J* = 8.4 Hz, 1H), 6.90 (d, *J* = 7.6 Hz, 2H), 6.78 (d, *J* = 8.0 Hz, 2H), 6.74 – 6.66 (m, 2H), 3.78 (s, 3H), 3.43 (s, 3H), 2.86 (d, *J* = 14.8 Hz, 1H), 2.03 (d, *J* = 14.8 Hz, 1H), 1.32 (s, 6H), 1.23 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.8, 172.6, 158.8, 147.7, 139.3, 133.9, 131.9, 130.3, 129.6, 127.4, 123.6, 122.4, 114.2, 113.5, 55.1, 43.1, 42.4, 40.8, 30.4, 27.1, 26.2, 25.9; LRMS (EI, 70 eV) *m/z* (%): 375 (M^+, 100), 360 (17), 332 (35) 304 (54), 290 (76); HRMS (ESI): calculated for [$C_{24}H_{26}NO_3^+$] 376.1907, found 376.1907.

10-(4-Chlorophenyl)-5,6a,8,8-tetramethyl-7,8-dihydrophenanthridine-6,9(5*H*,6a*H*)-dione (3ga)



White solid, mp 162.7 – 164.3 ºC (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 – 7.21 (m, 3H), 7.02 (d, *J* = 8.4 Hz, 1H), 6.93 (d, *J* = 7.6 Hz, 2H), 6.74 (t, *J* = 7.6 Hz, 1H), 6.62 (d, *J* = 8.0 Hz, 1H), 3.44 (s, 3H), 2.87 (d, *J* = 14.8 Hz, 1H), 2.04 (d, *J* = 14.8 Hz, 1H), 1.33 (s, 3H), 1.31 (s, 3H), 1.23 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.3, 172.3, 149.0, 139.3, 133.9, 133.4, 133.2, 132.1, 130.2, 130.0, 128.2, 123.1, 122.5, 114.3, 43.2, 42.3, 40.9, 30.5, 27.2, 26.2, 25.9; LRMS (EI, 70 eV) *m/z* (%): 381 (M^++2, 36), 379 (M^+, 100), 364 (32), 336 (34), 308 (64), 294 (72); HRMS (ESI): calculated for [$C_{23}H_{23}ClNO_2^+$] 380.1412, found 380.1398.

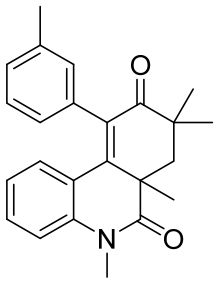
4-(5,6a,8,8-Tetramethyl-6,9-dioxo-5,6,6a,7,8,9-hexahydrophenanthridin-10-yl)benzonitrile (3ha)



White solid, mp 145.0 – 146.3 ºC (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 (d, *J* = 7.6 Hz, 2H), 7.30 – 7.26 (m, 1H), 7.12 (d, *J* = 6.8 Hz, 2H), 7.04 (d, *J* = 8.0 Hz, 1H), 6.72 (t, *J* = 7.6 Hz, 1H), 6.51 (d, *J* = 7.6 Hz, 1H), 3.44 (s, 3H), 2.88 (d, *J* = 14.8 Hz, 1H), 2.06 (d, *J* = 14.8 Hz, 1H), 1.35 (s, 3H), 1.32 (s, 3H), 1.23 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.7, 172.0,

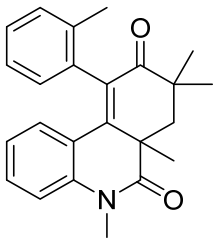
150.3, 140.8, 139.3, 132.7, 131.7, 131.6, 130.5, 130.0, 122.5, 122.5, 118.8, 114.5, 111.1, 43.4, 42.2, 40.9, 30.5, 27.3, 26.2, 25.8; LRMS (EI, 70 eV) *m/z* (%): 370 (M^+, 100), 355 (49), 327 (38), 314 (49), 299 (73), 285 (89); HRMS (ESI): calculated for [$C_{24}H_{23}N_2O_2^+$] 371.1754, found 371.1750.

5,6a,8,8-Tetramethyl-10-(*m*-tolyl)-7,8-dihydrophenanthridine-6,9(5*H*,6a*H*)-dione (3ia)



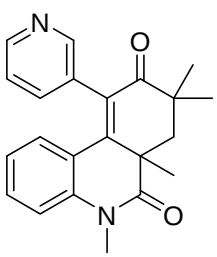
White solid, mp 162.1 – 163.8 ºC (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22 (t, *J* = 7.6 Hz, 1H), 7.11 (t, *J* = 7.6 Hz, 1H), 7.06 – 6.99 (m, 2H), 6.83 (s, 1H), 6.74 – 6.63 (m, 3H), 3.44 (s, 3H), 2.89 (d, *J* = 14.8 Hz, 1H), 2.27 (s, 3H), 2.03 (d, *J* = 14.8 Hz, 1H), 1.32 (s, 6H), 1.23 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.7, 172.6, 147.9, 139.2, 137.4, 135.3, 134.6, 131.2, 130.3, 129.7, 128.1, 127.8, 127.6, 123.5, 122.3, 114.1, 43.1, 42.4, 40.8, 30.5, 27.4, 26.2, 25.8, 21.4; LRMS (EI, 70 eV) *m/z* (%): 359 (M^+, 100), 344 (29), 303 (52), 288 (61), 274 (66); HRMS (ESI): calculated for [$C_{24}H_{26}NO_2^+$] 360.1958, found 360.1963.

5,6a,8,8-Tetramethyl-10-(*o*-tolyl)-7,8-dihydrophenanthridine-6,9(5*H*,6a*H*)-dione (3ja)



White solid, mp 155.4 – 156.8 ºC (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 – 7.14 (m, 7H), 7.00 – 6.90 (m, 4H), 6.70 – 6.61 (m, 3H), 6.51 (d, *J* = 8.0 Hz, 2H), 3.43 (s, 6H), 3.02 (d, *J* = 14.4 Hz, 1H), 2.88 (d, *J* = 14.4 Hz, 1H), 2.28 (s, 3H), 2.06 (d, *J* = 14.4 Hz, 1H), 1.99 (d, *J* = 14.4 Hz, 1H), 1.56 (s, 3H), 1.38 (s, 3H), 1.34 (s, 3H), 1.31 (s, 3H), 1.29 (s, 3H), 1.24 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.7, 201.9, 172.6, 172.5, 148.7, 148.6, 139.1, 138.6, 138.1, 136.0, 135.1, 135.0, 134.1, 133.8, 131.8, 130.4, 129.8, 129.8, 129.6, 129.5, 129.3, 128.9, 127.7, 127.7, 126.0, 125.2, 124.0, 123.3, 122.4, 122.4, 114.2, 114.0, 43.3, 43.0, 42.5, 42.2, 41.0, 40.8, 30.5, 30.4, 28.3, 27.6, 26.4, 26.2, 26.0, 25.5, 20.0, 18.9; LRMS (EI, 70 eV) *m/z* (%): 359 (M^+, 100), 344 (41), 303 (30), 286 (54), 260 (36); HRMS (ESI): calculated for [$C_{24}H_{26}NO_2^+$] 360.1958, found 360.1961.

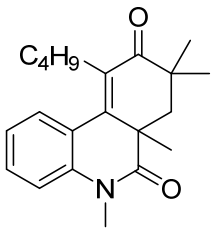
5,6a,8,8-Tetramethyl-10-(pyridin-3-yl)-7,8-dihydrophenanthridine-6,9(5*H*,6a*H*)-dione (3ka)



White solid, mp 149.6 – 151.1 ºC (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.48 (s, 1H), 8.14 (s, 1H), 7.43 (d, *J* = 6.4 Hz, 1H), 7.27 – 7.24 (m, 2H), 7.03 (d, *J* = 8.0 Hz, 1H), 6.72 (t, *J* = 7.6 Hz, 1H), 6.55 (d, *J* = 7.6 Hz, 1H), 3.45 (s, 3H), 2.90 (d, *J* = 14.8 Hz, 1H), 2.07 (d, *J* = 14.8 Hz, 1H), 1.35 (s, 3H), 1.33 (s, 3H), 1.24 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.0, 172.1, 151.1, 150.4, 148.2, 139.4, 138.5,

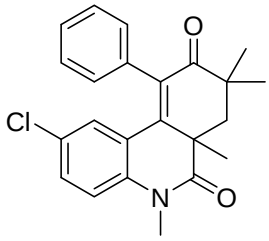
130.8, 130.4, 130.1, 122.7, 122.6, 114.5, 43.4, 42.2, 40.9, 30.5, 27.2, 26.2, 25.9; LRMS (EI, 70 eV) *m/z* (%): 346 (M^+, 100), 331 (48), 303 (34), 289 (43), 261 (88); HRMS (ESI): calculated for [$C_{22}H_{23}N_2O_2^+$] 347.1754, found 347.1749.

10-Butyl-5,6a,8,8-tetramethyl-7,8-dihydrophenanthridine-6,9(5*H*,6a*H*)-dione (3la)



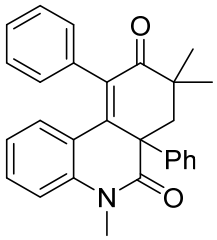
Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.38 (m, 2H), 7.16 (t, *J* = 7.6 Hz, 1H), 7.06 (d, *J* = 8.4 Hz, 1H), 3.38 (s, 3H), 2.81 (d, *J* = 14.4 Hz, 1H), 2.53 – 2.30 (m, 2H) 1.86 (d, *J* = 14.4 Hz, 1H), 1.55 – 1.47 (m, 1H), 1.35 – 1.26 (m, 3H), 1.20 (s, 3H), 1.17 (s, 3H), 1.13 (s, 3H), 0.87 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.2, 173.1, 146.1, 139.0, 134.6, 129.7, 128.3, 124.4, 122.7, 114.4, 42.9, 42.4, 40.6, 32.2, 30.4, 27.3, 27.0, 26.1, 25.6, 22.8, 13.8; LRMS (EI, 70 eV) *m/z* (%): 325 (M^+, 98), 310 (100), 282 (35), 269 (39), 254 (38); HRMS (ESI): calculated for [$C_{21}H_{28}NO_2^+$] 326.2115, found 326.2122.

2-Chloro-5,6a,8,8-tetramethyl-10-phenyl-7,8-dihydrophenanthridine-6,9(5*H*,6a*H*)-dione (3ma)



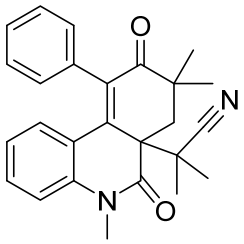
White solid, mp 150.3 – 152.0 ºC (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 – 7.28 (m, 3H), 7.17 (d, *J* = 8.4 Hz, 1H), 6.97 – 6.92 (m, 3H), 6.53 (s, 1H), 3.42 (s, 3H), 2.90 (d, *J* = 14.8 Hz, 1H), 2.03 (d, *J* = 14.8 Hz, 1H), 1.33 (s, 3H), 1.31 (s, 3H), 1.23 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.2, 172.2, 146.5, 137.8, 135.3, 134.7, 130.3, 130.0, 129.5, 128.1, 127.7, 127.7, 124.8, 115.4, 42.9, 42.3, 40.8, 30.6, 27.3, 26.1, 25.7; LRMS (EI, 70 eV) *m/z* (%): 381 (M^++2, 33), 379 (M^+, 100), 364 (31), 336 (34), 308 (62), 294 (82); HRMS (ESI): calculated for [$C_{23}H_{23}ClNO_2^+$] 380.1412, found 380.1390.

5,8,8-Trimethyl-6a,10-diphenyl-7,8-dihydrophenanthridine-6,9(5*H*,6a*H*)-dione (3na)



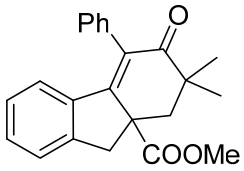
White solid, mp 209.1 – 210.4 ºC (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 – 7.24 (m, 5H), 7.17 – 7.02 (m, 6H), 6.78 (d, *J* = 8.0 Hz, 1H), 6.71 (d, *J* = 7.6 Hz, 1H), 6.59 (t, *J* = 7.6 Hz, 1H), 3.43 (s, 3H), 3.13 (d, *J* = 14.4Hz, 1H), 2.37 (d, *J* = 14.4 Hz, 1H), 1.26 (s, 3H), 0.98 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.3, 170.0, 145.9, 141.7, 138.8, 137.6, 135.2, 130.8, 130.6, 129.6, 128.4, 128.0, 127.6, 127.1, 127.1, 124.7, 122.3, 114.6, 52.3, 46.0, 41.2, 30.8, 26.6, 24.9; LRMS (EI, 70 eV) *m/z* (%): 407 (M^+, 100), 379 (20), 351 (20), 336 (99), 323 (31), 260 (74); HRMS (ESI): calculated for [$C_{28}H_{26}NO_2^+$] 408.1958, found 408.1956.

5,8,8-Trimethyl-10-phenyl-7,8-dihydrophenanthridine-6,9(5*H*,6a*H*)-dione (3oa)



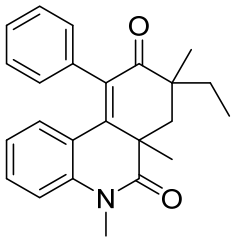
White solid, mp 181.7 – 183.6 ºC (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 – 7.26(m, 4H), 7.00 – 6.98 (m, *J* = 8.2 Hz, 3H), 6.74 (t, *J* = 7.6 Hz, 1H), 6.59 (d, *J* = 7.6 Hz, 1H), 3.51 (s, 3H), 3.16 (d, *J* = 14.4 Hz, 1H), 1.89 (d, *J* = 14.4 Hz, 1H), 1.36 (s, 3H), 1.25 (s, 3H),1.23 (s, 3H), 1.22 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.5, 168.4, 143.1, 140.5, 138.5, 134.3, 130.6, 129.7, 129.3, 128.1, 127.5, 123.0, 122.3, 122.1, 114.3, 50.5, 41.2, 39.6, 37.0, 30.9, 25.3, 24.3, 23.9, 23.3; LRMS (EI, 70 eV) *m/z* (%): 331 (71), 316 (15), 288 (100), 260 (24), 245 (10); HRMS (ESI): calculated for [$C_{26}H_{27}N_2O_2^+$] 399.2067, found 399.2063.

Methyl 7,7-dimethyl-6-oxo-5-phenyl-6,7,8,9-tetrahydro-8a*H*-fluorene-8a-carboxylate (3qa)



White solid, mp 142.4 – 144.3 ºC (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 – 7.37(m, 3H), 7.22 – 7.19 (m, 4H), 6.94 – 6.90 (m, 1H), 6.45 (d, *J* = 7.6 Hz, 1H), 3.65 (s, 3H), 3.49 (d, *J* = 16.0 Hz, 1H), 3.16 (d, *J* = 16.0 Hz, 1H), 2.81 (d, *J* = 13.6 Hz, 1H), 2.30 (d, *J* = 13.6 Hz, 1H), 1.23 (s, 3H), 1.18 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.1, 176.2, 156.5, 144.9, 138.6, 135.2, 133.0, 130.5, 129.6, 128.6, 127.7, 127.0, 126.4, 124.8, 53.6, 52.8, 46.2, 45.3, 40.8, 29.0, 24.8; LRMS (EI, 70 eV) *m/z* (%): 346 (24), 290 (100), 258 (85), 231 (41), 202 (60); HRMS (ESI): calculated for [$C_{23}H_{23}O_3^+$] 347.1642, found 347.1644.

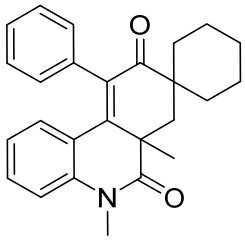
8-Ethyl-5,6a,8-trimethyl-10-phenyl-7,8-dihydrophenanthridine-6,9(5*H*,6a*H*)-dione (3ab)



Yellow oil; d.r. = 1.1:1, as determined by the crude H NMR spectroscopy

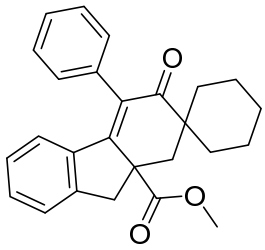
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 – 7.19 (m, 8H), 7.01 – 6.96 (m, 6H), 6.69 – 6.59 (m, 4H), 3.44 (s, 3H), 3.43 (s, 3H), 3.06 (d, *J* = 14.8 Hz, 1H), 2.74 (d, *J* = 14.8 Hz, 1H), 2.14 (d, *J* = 14.8 Hz, 1H), 1.94 (d, *J* = 14.8 Hz, 1H), 1.80 – 1.57 (m, 4H), 1.35 (s, 3H), 1.29 (s, 3H), 1.23 (s, 3H), 1.18 (s, 3H), 1.00 (t, *J* = 7.6 Hz, 3H), 0.77 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.5, 202.2, 172.7, 172.6, 147.9, 147.7, 139.2, 139.2, 135.7, 135.2, 134.8, 134.5, 130.7, 130.5, 130.3, 130.2, 129.7, 129.6, 127.9, 127.2, 123.3, 123.3, 122.2, 114.1, 114.1, 44.1, 44.1, 43.0, 42.9, 40.5, 39.0, 31.7, 30.5, 30.4, 28.0, 26.9, 22.9, 22.4, 8.7, 8.2; LRMS (EI, 70 eV) *m/z* (%): 359 (M^+, 75), 330 (54), 316 (44), 289 (60), 260 (100); HRMS (ESI): calculated for [$C_{24}H_{26}NO_2^+$] 360.1958, found 360.1964.

5',6a'-Dimethyl-10'-phenyl-6a',7'-dihydro-6'*H*-spiro[cyclohexane-1,8'-phenanthridine]-6',9'(5'*H*)-dione (3ac)



White solid, mp 201.8 – 203.1 ºC (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 – 7.19 (m, 4H), 7.00 – 6.98 (m, 3H), 6.67 (t, *J* = 7.6 Hz, 1H), 6.60 (d, *J* = 7.6 Hz, 1H), 3.43 (s, 3H), 2.94 (d, *J* = 14.8 Hz, 1H), 2.05 (d, *J* = 14.8 Hz, 1H), 1.98 – 1.92 (m, 1H), 1.87 – 1.82 (m, 1H), 1.69 – 1.40 (m, 8H), 1.31 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.4, 172.6, 146.7, 139.2, 135.6, 134.8, 130.5, 130.2, 129.6, 128.0, 127.3, 123.3, 122.3, 114.1, 44.1, 43.2, 39.2, 34.4, 33.9, 30.5, 27.5, 26.0, 22.1, 21.9; LRMS (EI, 70 eV) *m/z* (%): 385 (M^+, 20), 370 (100), 314 (23), 260 (20); HRMS (ESI): calculated for [$C_{26}H_{28}NO_2^+$] 386.2115, found 386.2123.

Methyl 3'-oxo-4'-phenyl-3',9'-dihydrospiro[cyclohexane-1,2'-fluorene]-9a'(1'*H*)-carboxylate (3qc)



White solid, mp 119.4 – 121.0 ºC (uncorrected); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 – 7.37 (m, 3H), 7.25 – 7.19 (m, 4H), 6.92 (t, *J* = 7.2 Hz, 1H), 6.45 (d, *J* = 7.6 Hz, 1H), 3.62 (s, 3H), 3.53 (d, *J* = 16.4 Hz, 1H), 3.25 (d, *J* = 13.6 Hz, 1H), 3.18 (d, *J* = 16.4 Hz, 1H), 2.00 (d, *J* = 13.6 Hz, 1H), 1.93 (td, *J* = 12.4, 3.2 Hz, 1H), 1.71 – 1.34 (m, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.2, 176.2, 155.3, 144.9, 138.5, 135.4, 133.5, 130.5, 129.6, 128.5, 127.7, 127.0, 126.3, 124.8, 53.2, 52.6, 45.3, 44.5, 40.1, 37.5, 31.0, 25.5, 21.1; LRMS (EI, 70 eV) *m/z* (%): 386 (M^+, 44), 331 (32), 290 (100), 262 (41), 258 (80); HRMS (ESI): calculated for [$C_{26}H_{27}O_3^+$] 387.1955, found 387.1963.

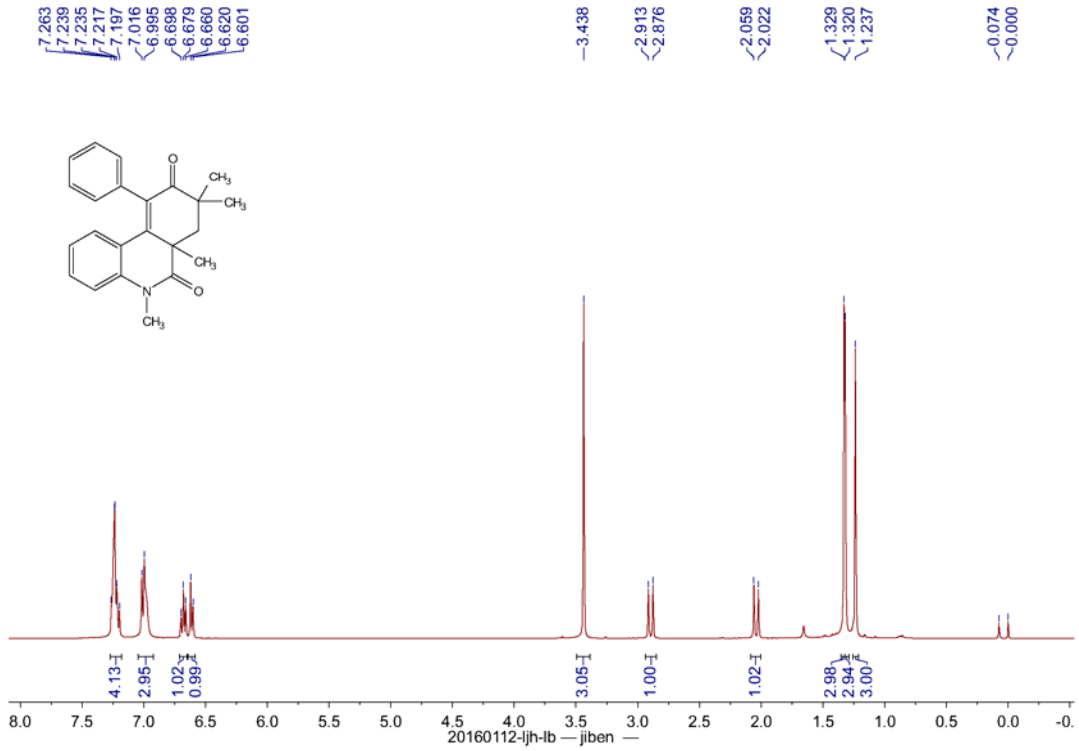
(C) References

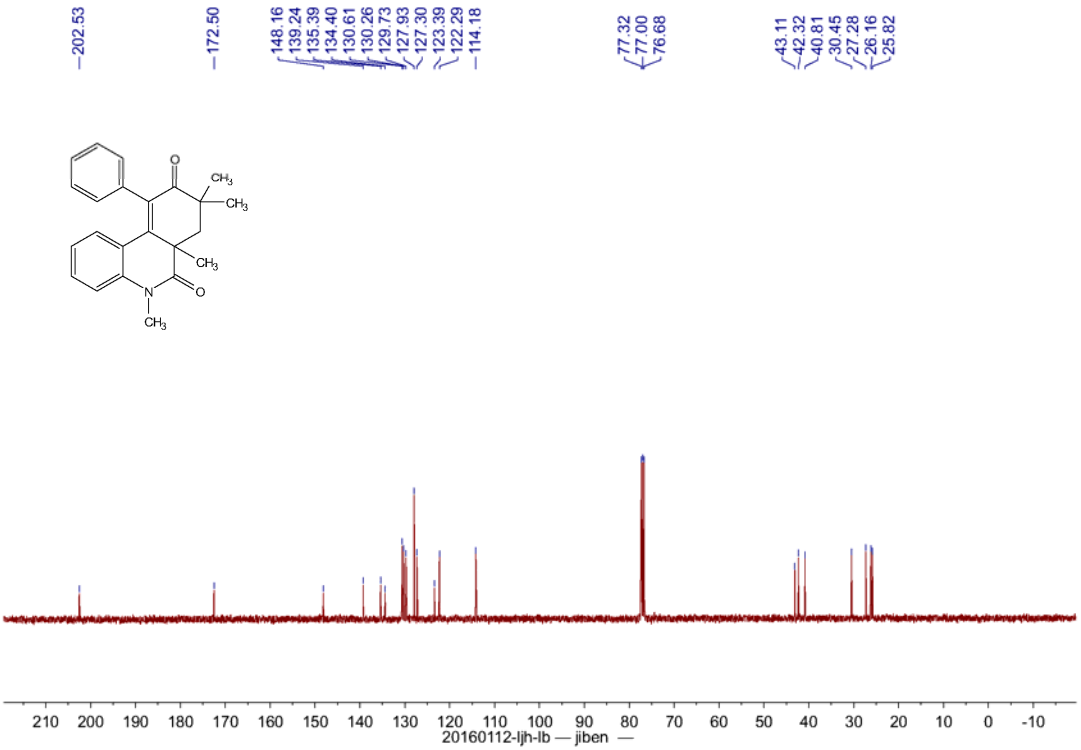
[1] a) X. Mu, T. Wu, H.-Y. Wang, Y.-L. Guo, G. Liu *J. Am. Chem. Soc.* **2012**, *134*, 878;

b) N. Sakiyama, K. Noguchi, K. Tanaka, *Angew. Chem. Int. Ed.* **2012**, *51*, 5976.

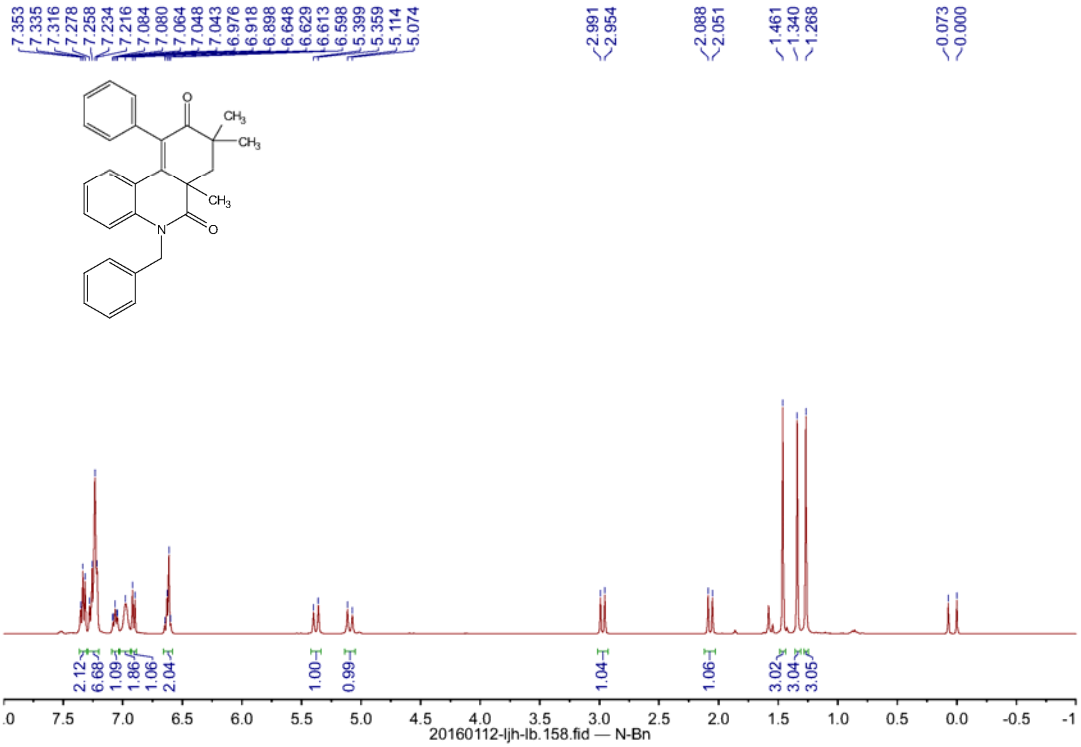
(D) Spectra

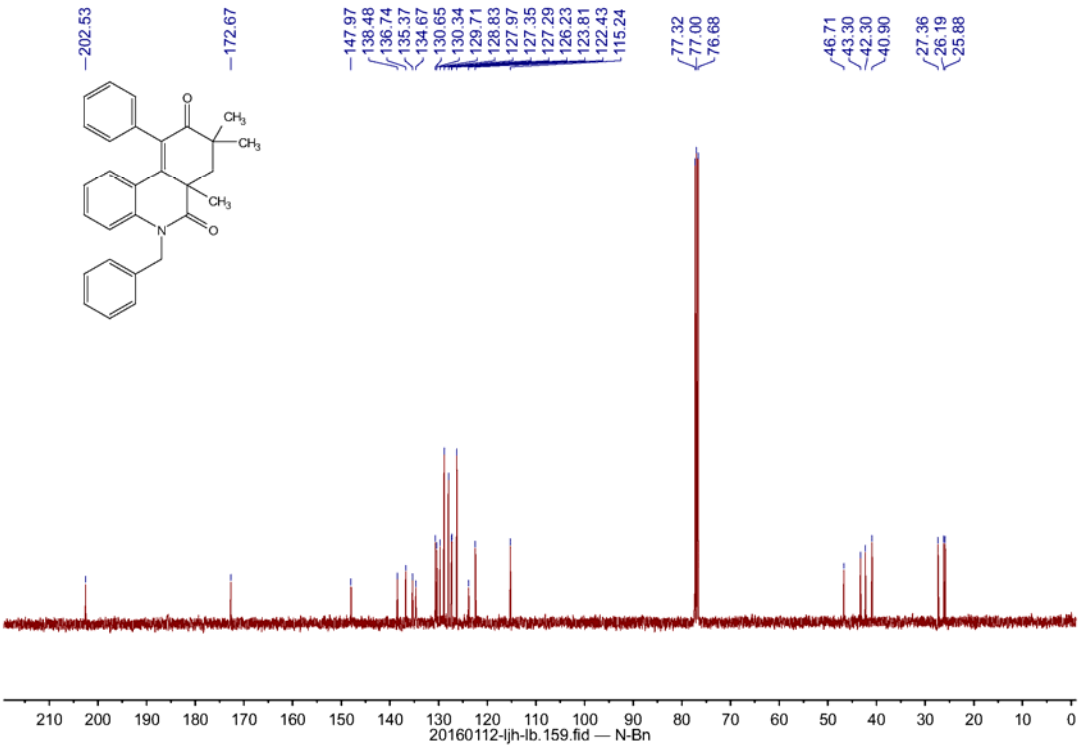
5,6a,8,8-Tetramethyl-10-phenyl-7,8-dihydrophenanthridine-6,9(5*H*,6a*H*)-dione (**3aa**)



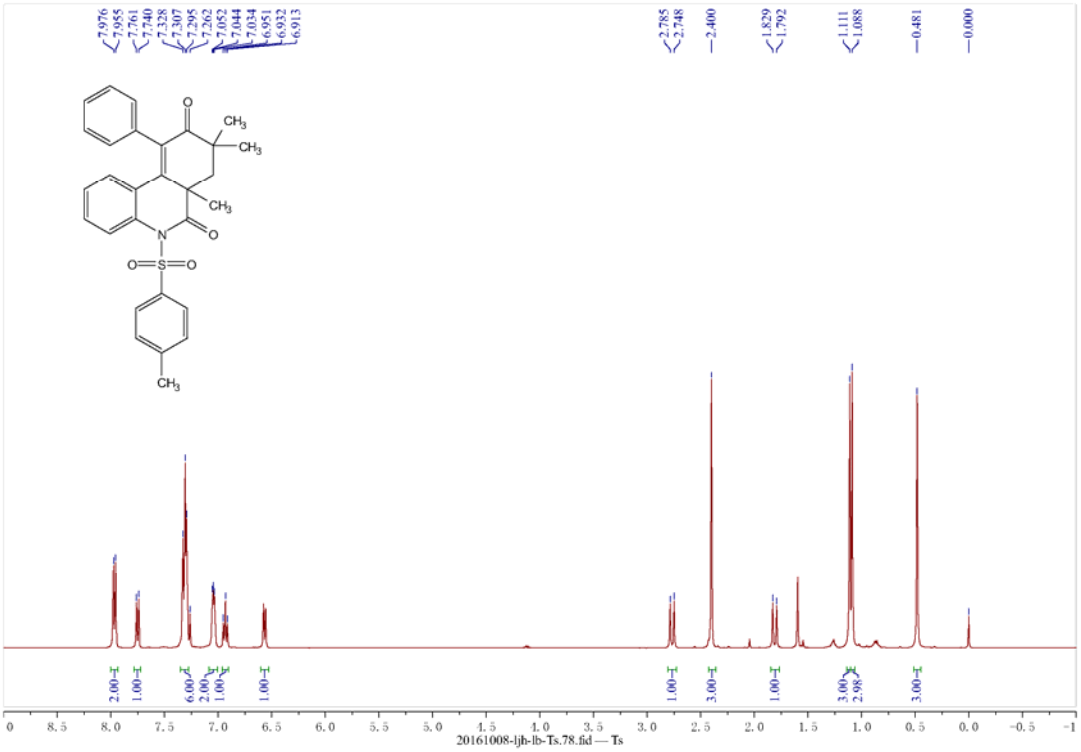


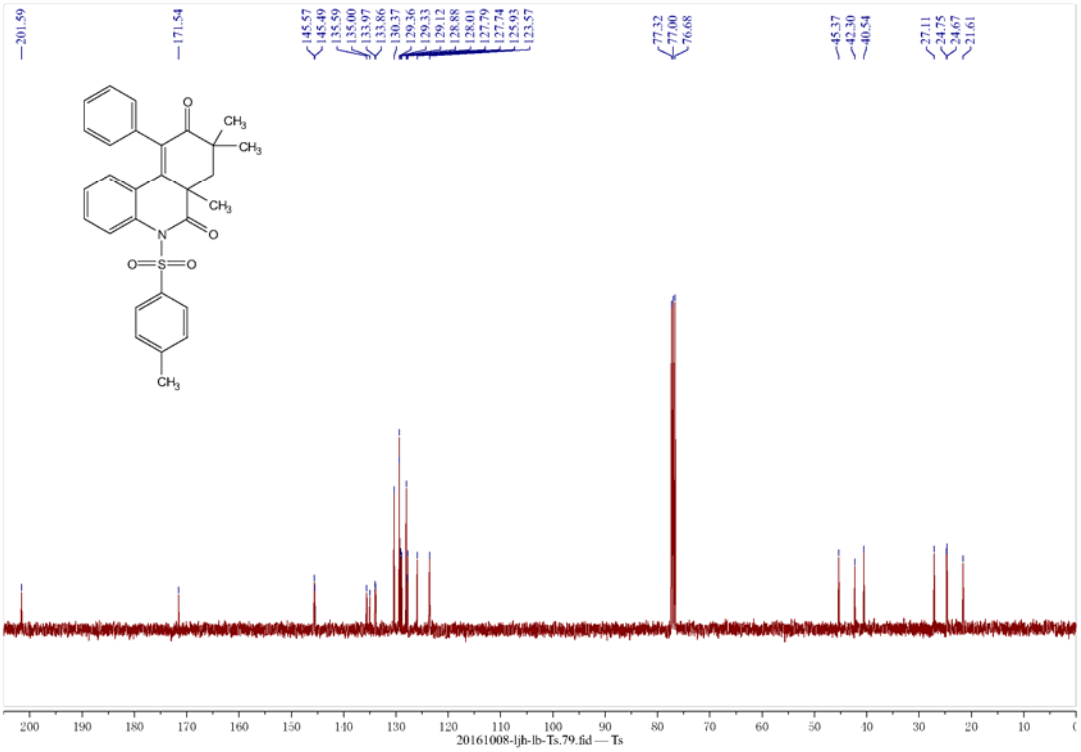
5-Benzyl-6a,8,8-trimethyl-10-phenyl-7,8-dihydrophenanthridine-6,9(5*H*,6a*H*)-dione (**3ba**)



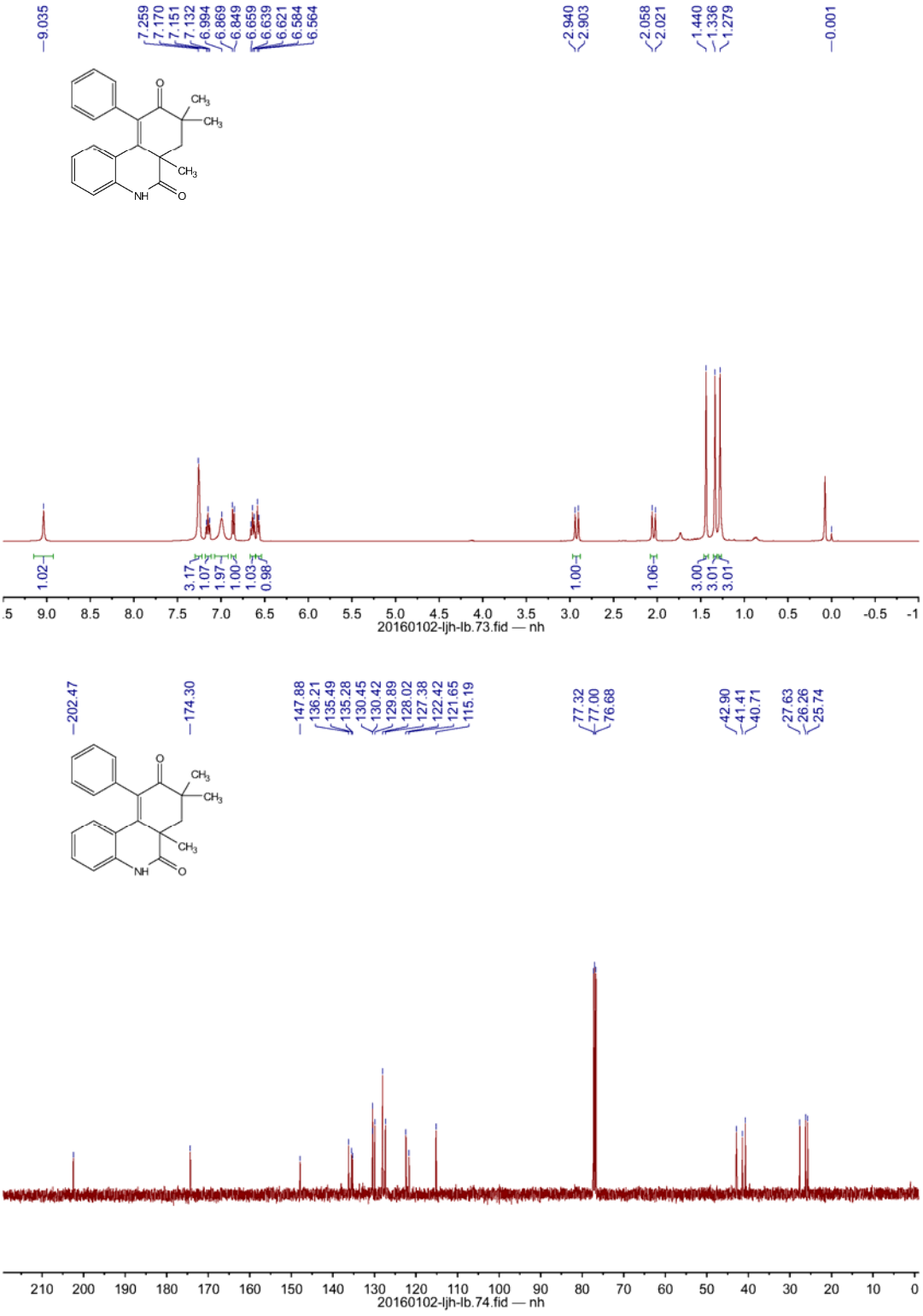


6a,8,8-Trimethyl-10-phenyl-5-tosyl-7,8-dihydrophenanthridine-6,9(5*H*,6a*H*)-dione (**3ca**)

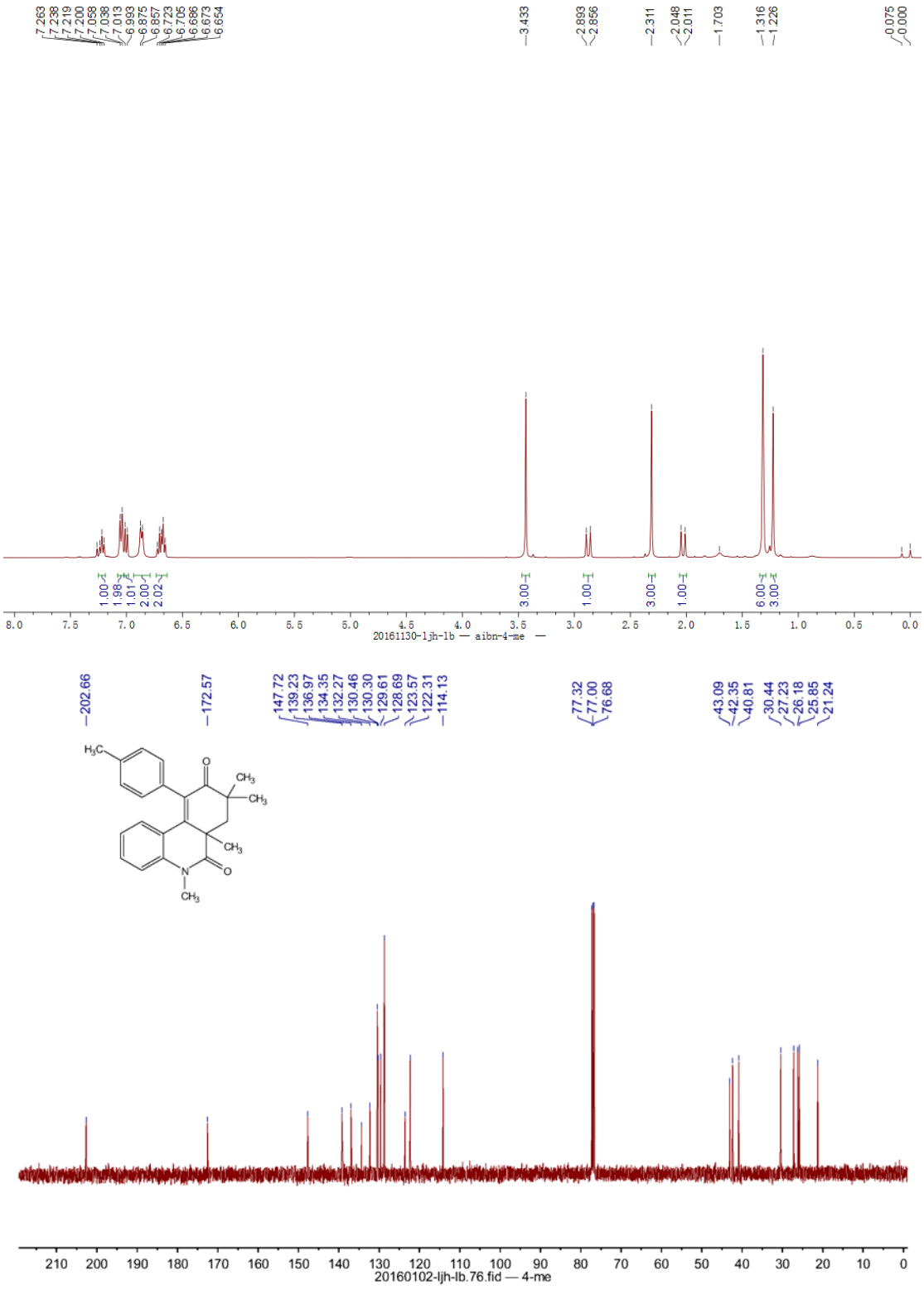




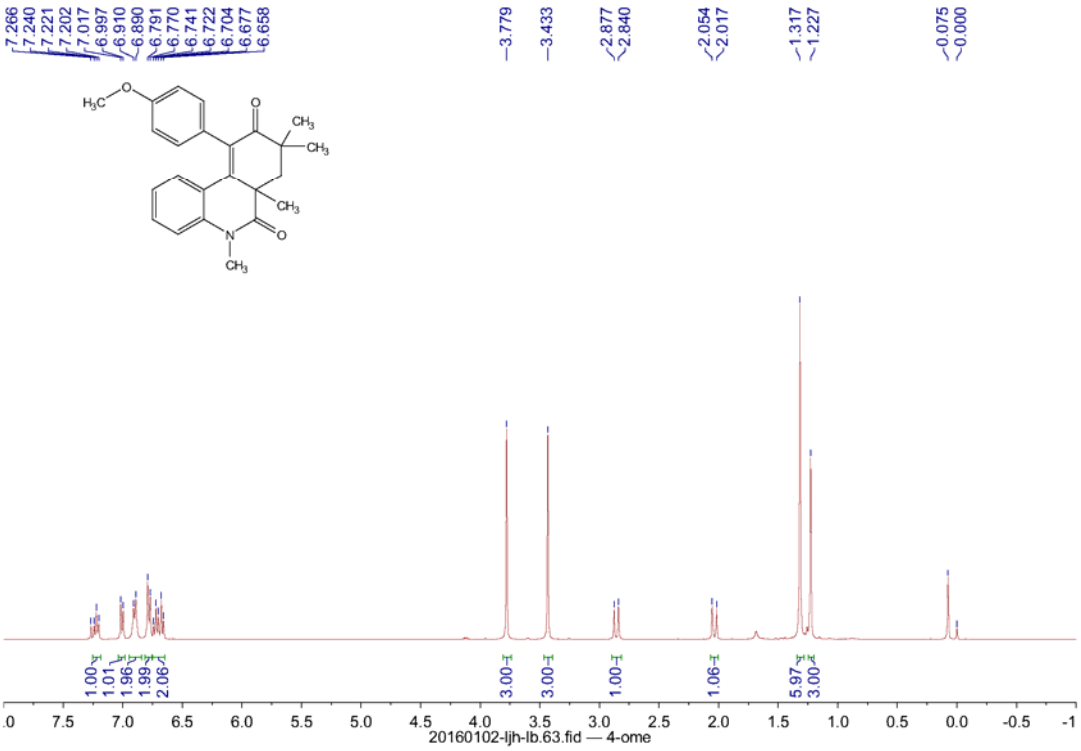
6a,8,8-Trimethyl-10-phenyl-7,8-dihydrophenanthridine-6,9(5*H*,6a*H*)-dione (**3da**)

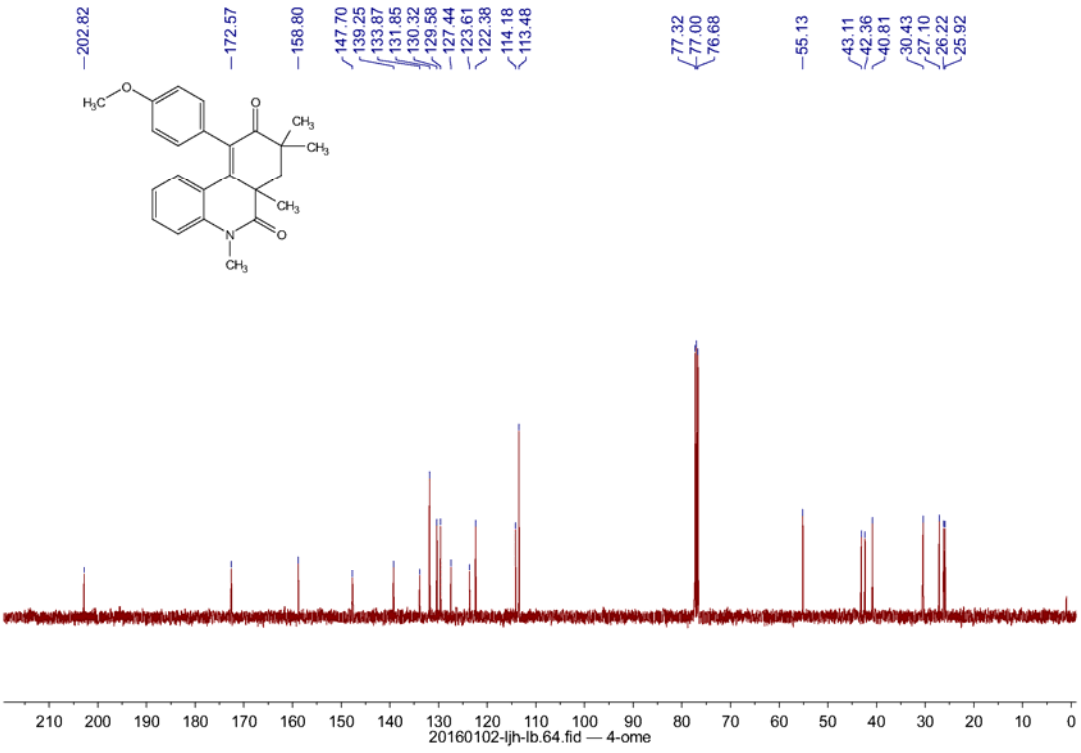


5,6a,8,8-Tetramethyl-10-(*p*-tolyl)-7,8-dihydrophenanthridine-6,9(5*H*,6a*H*)-dione (**3ea**)

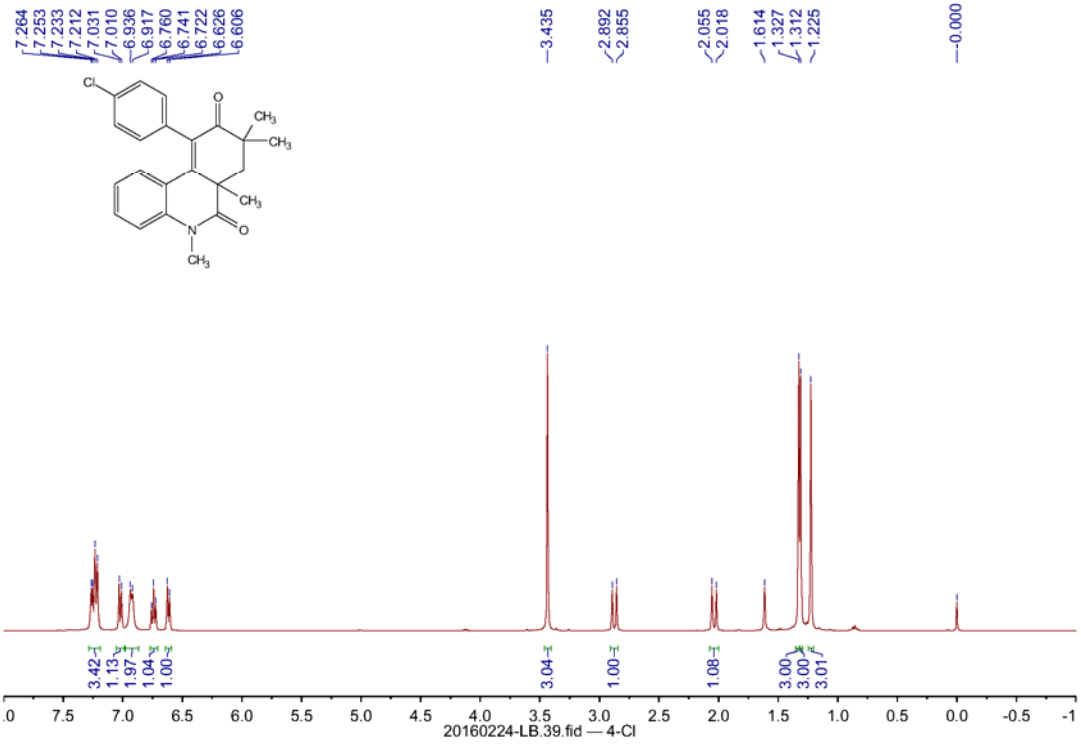


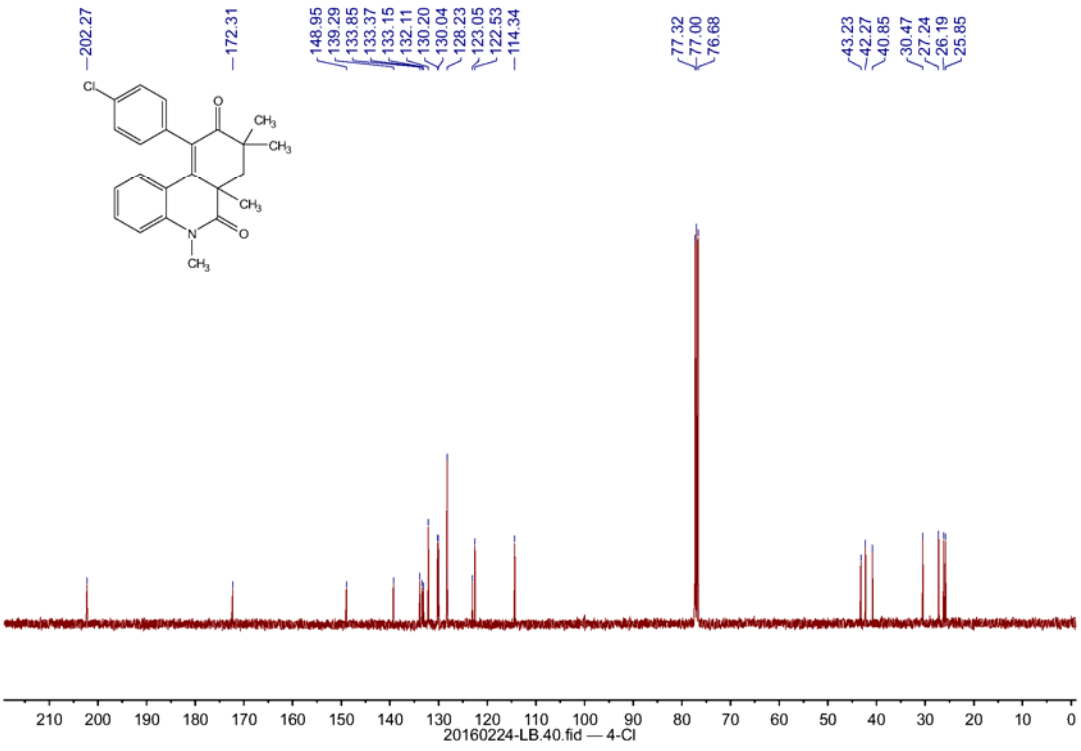
10-(4-Methoxyphenyl)-5,6a,8,8-tetramethyl-7,8-dihydrophenanthridine-6,9(5*H*,6a*H*)-dione (**3fa**)



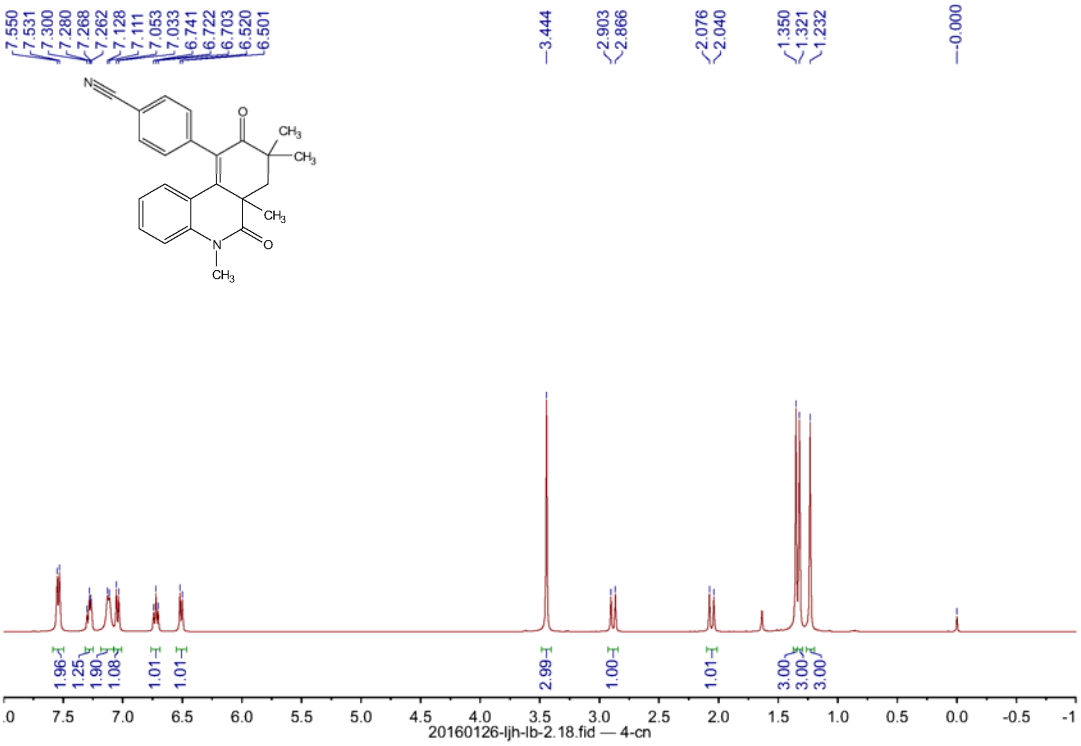


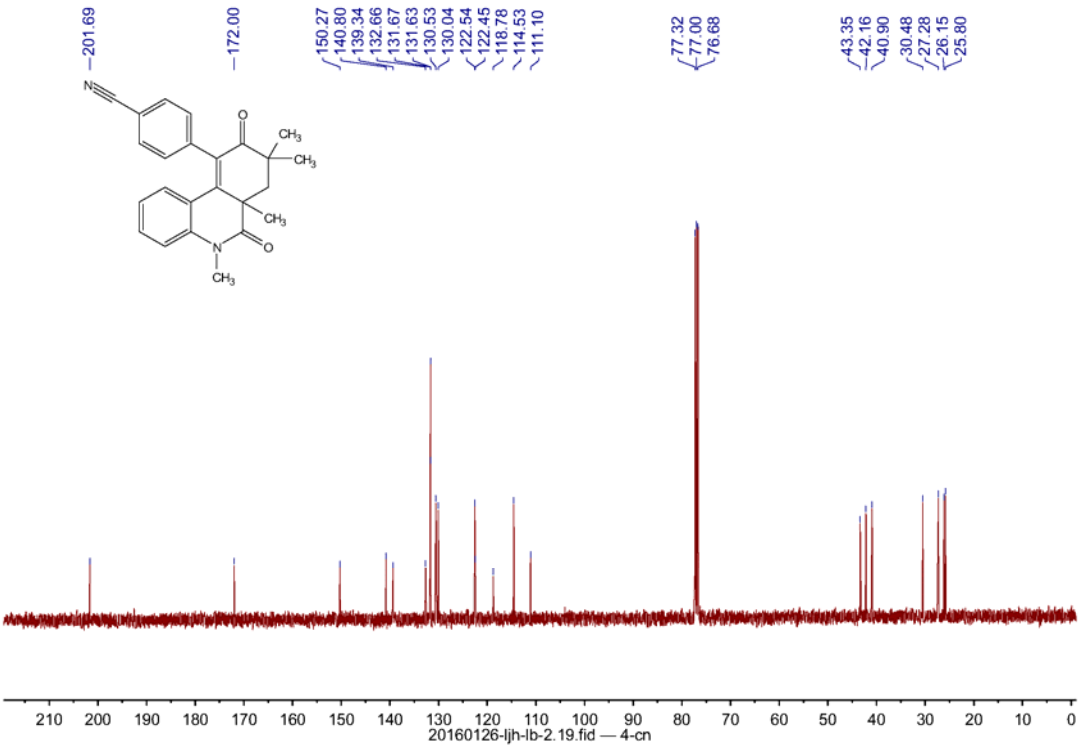
10-(4-Chlorophenyl)-5,6a,8,8-tetramethyl-7,8-dihydrophenanthridine-6,9(5*H*,6a*H*)-dione (**3ga**)



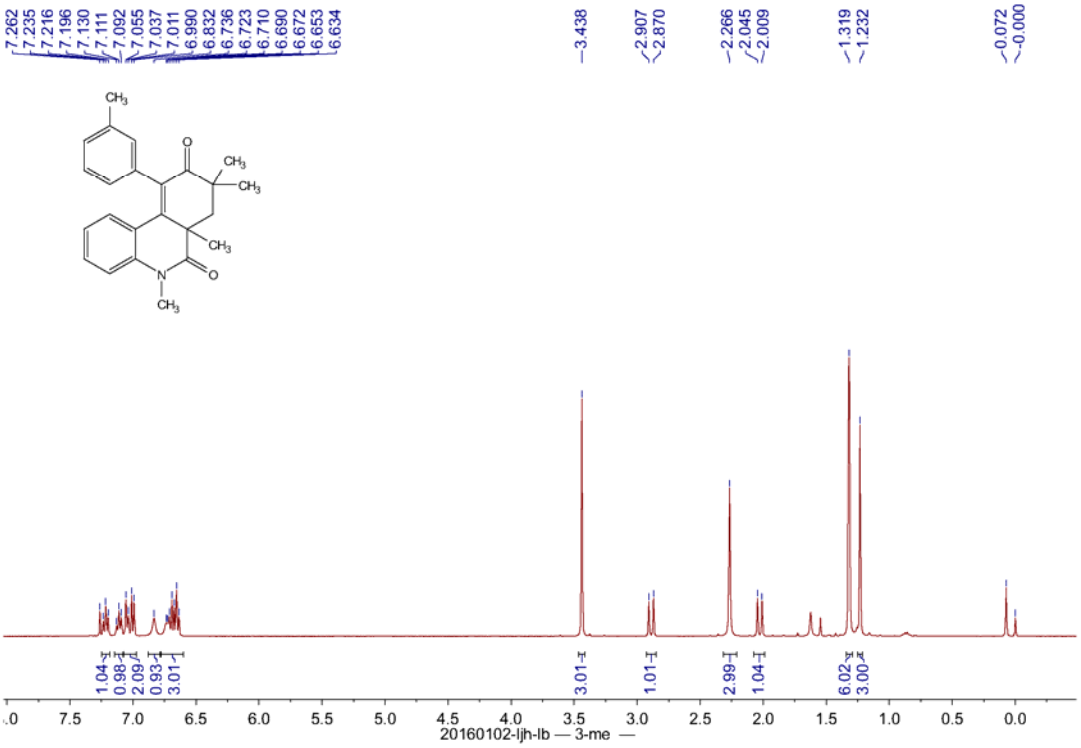


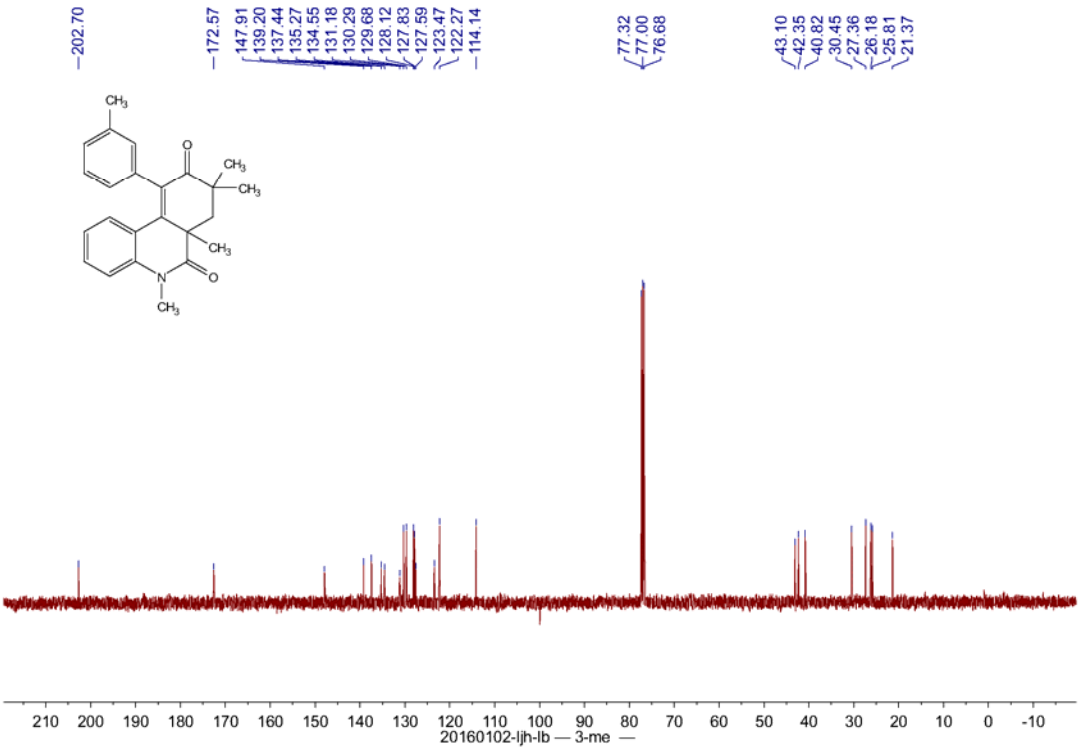
4-(5,6a,8,8-Tetramethyl-6,9-dioxo-5,6,6a,7,8,9-hexahydrophenanthridin-10-yl)benzonitrile (**3ha**)



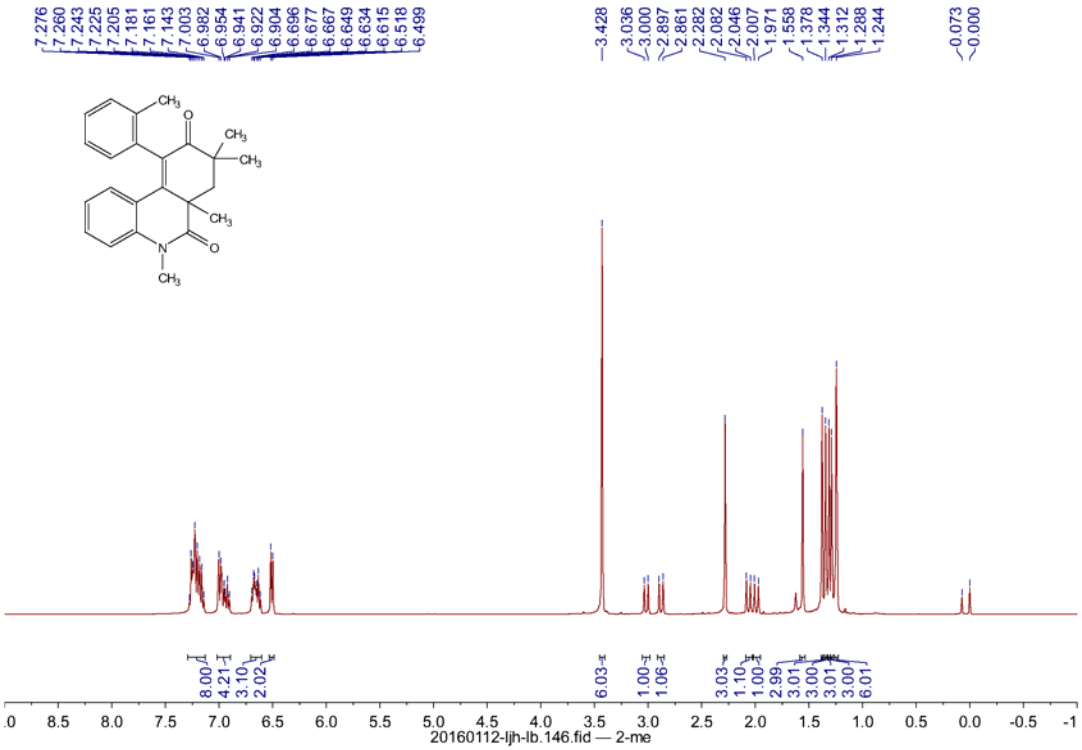


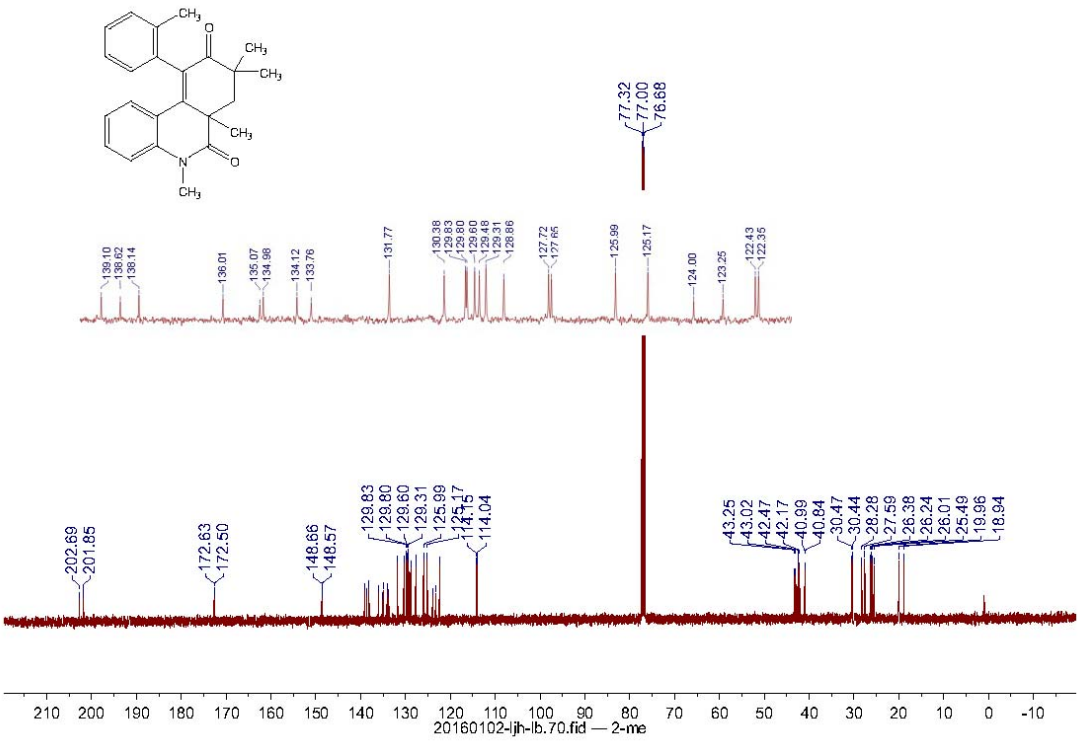
5,6a,8,8-Tetramethyl-10-(*m*-tolyl)-7,8-dihydrophenanthridine-6,9(5*H*,6a*H*)-dione (**3ia**)



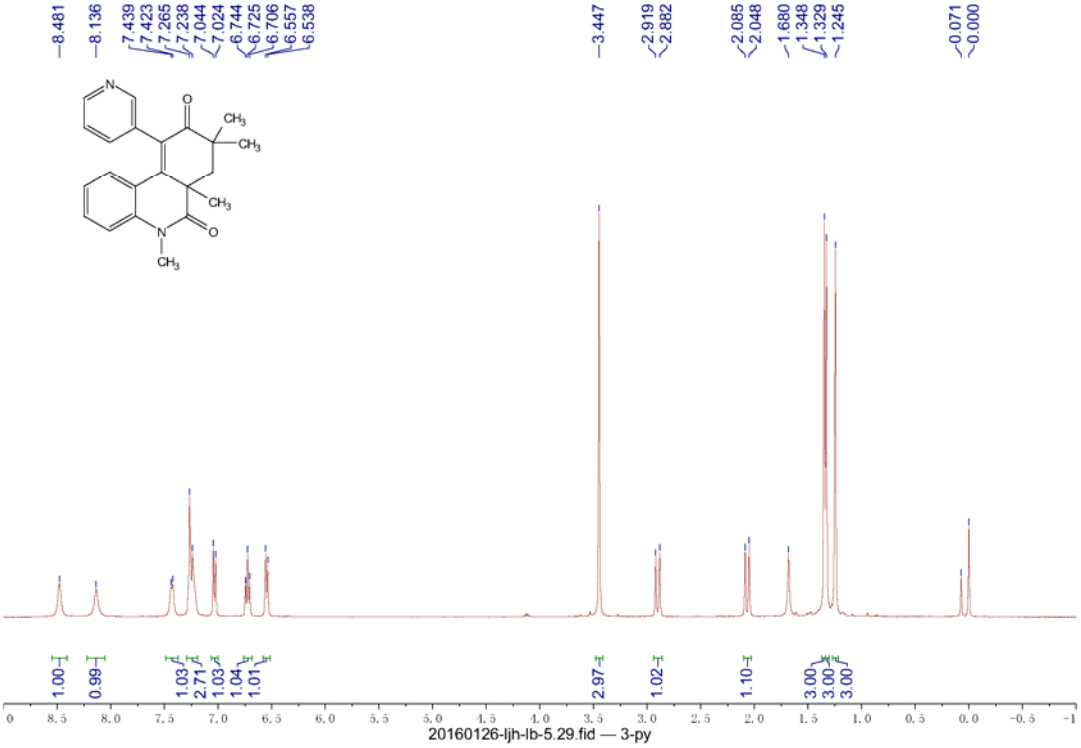


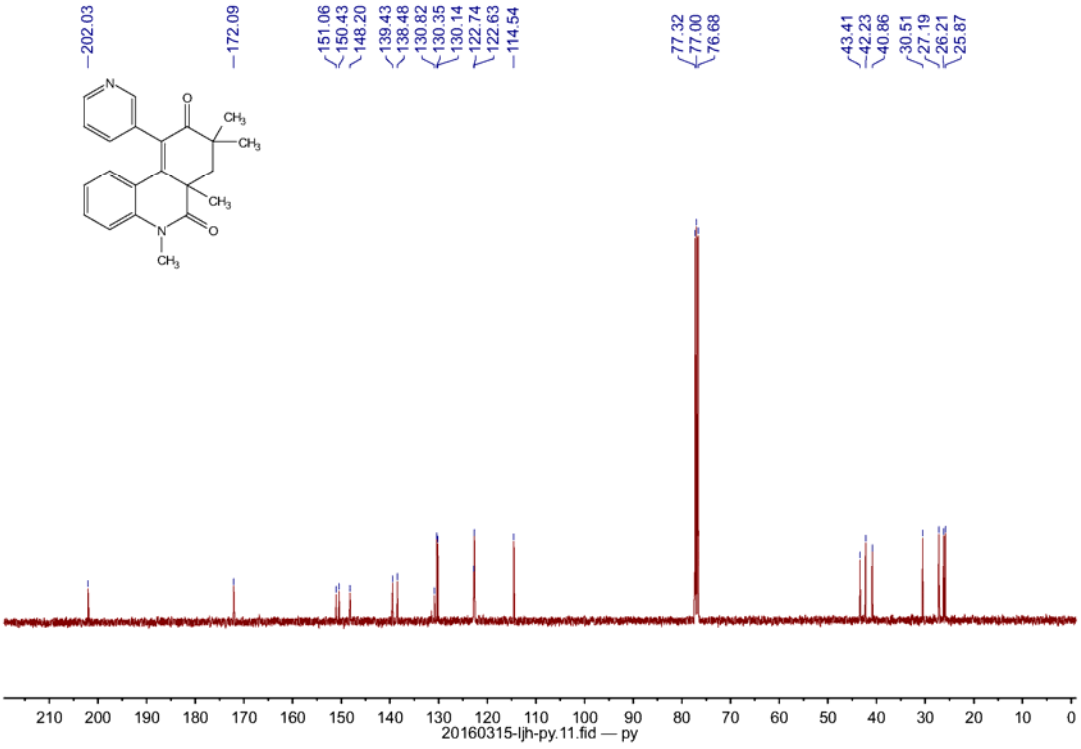
5,6a,8,8-Tetramethyl-10-(*o*-tolyl)-7,8-dihydrophenanthridine-6,9(5*H*,6a*H*)-dione (**3ja**)



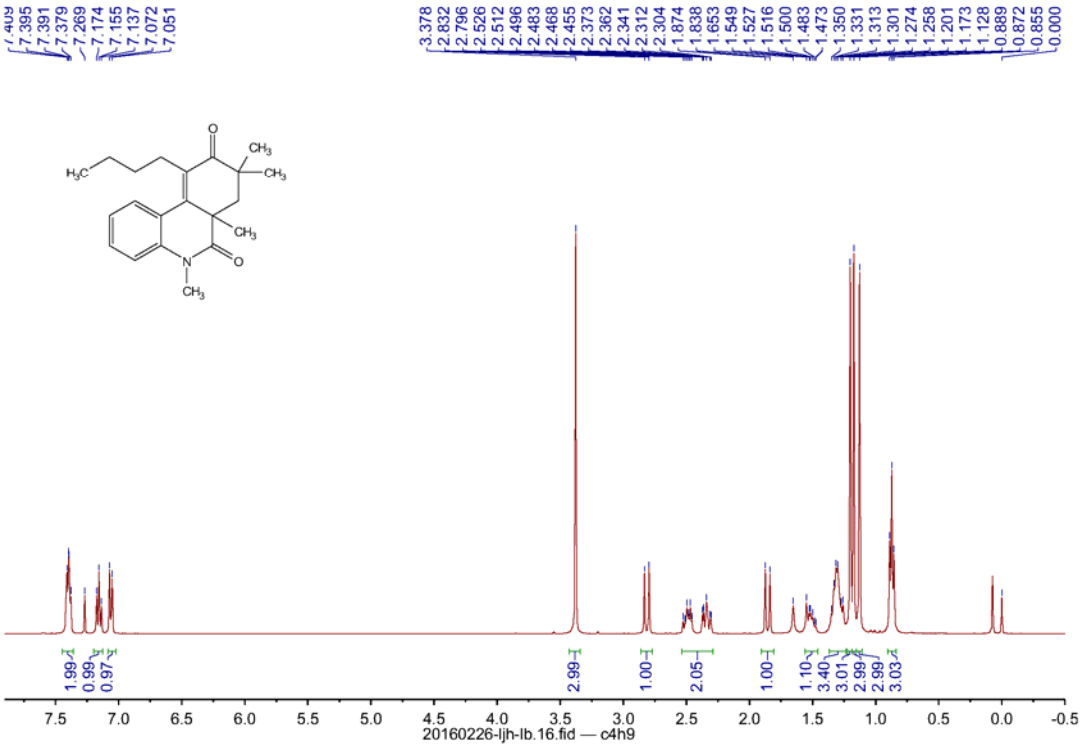


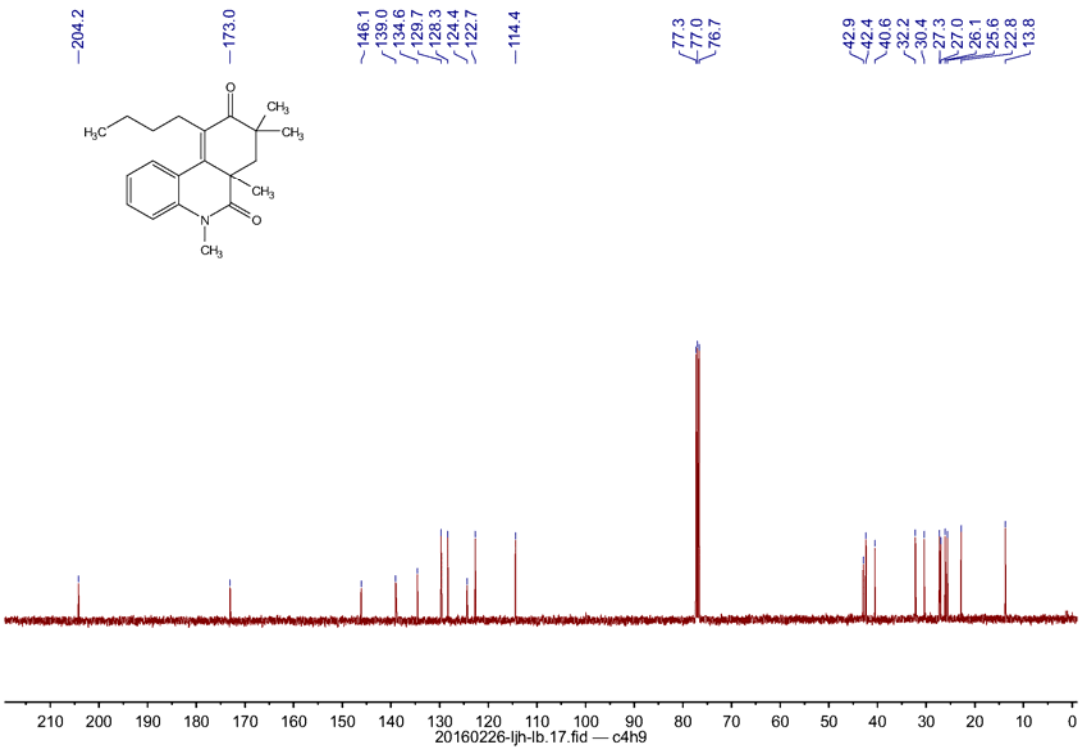
5,6a,8,8-Tetramethyl-10-(pyridin-3-yl)-7,8-dihydrophenanthridine-6,9(5*H*,6a*H*)-dione (**3ka**)



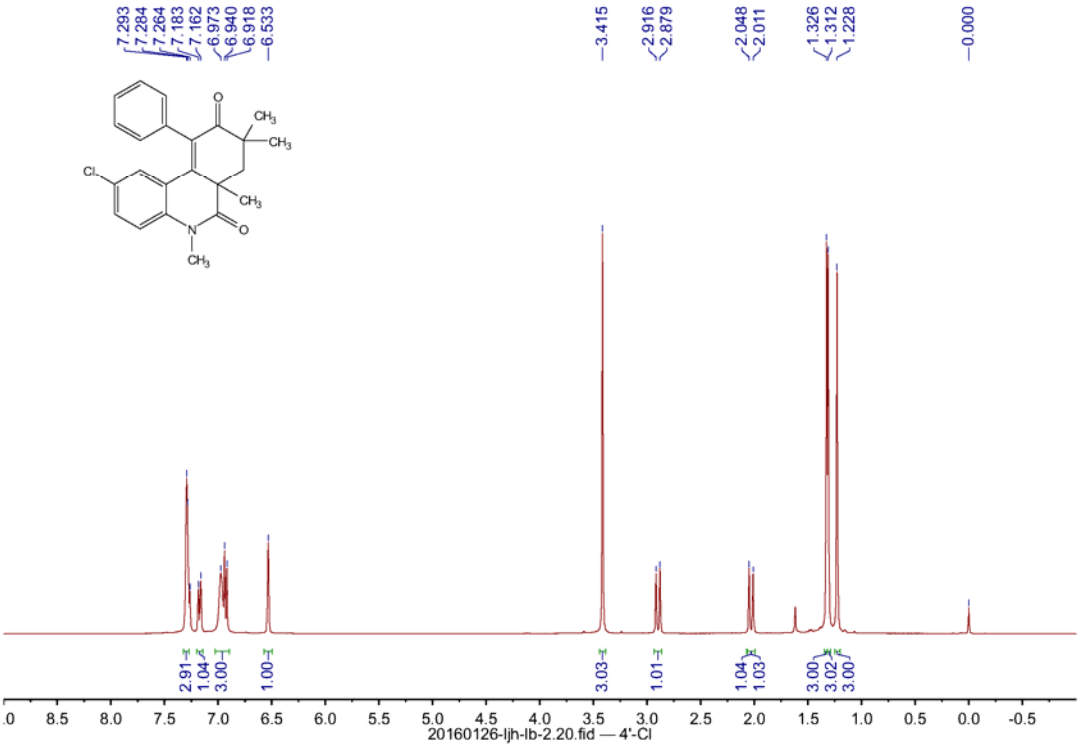


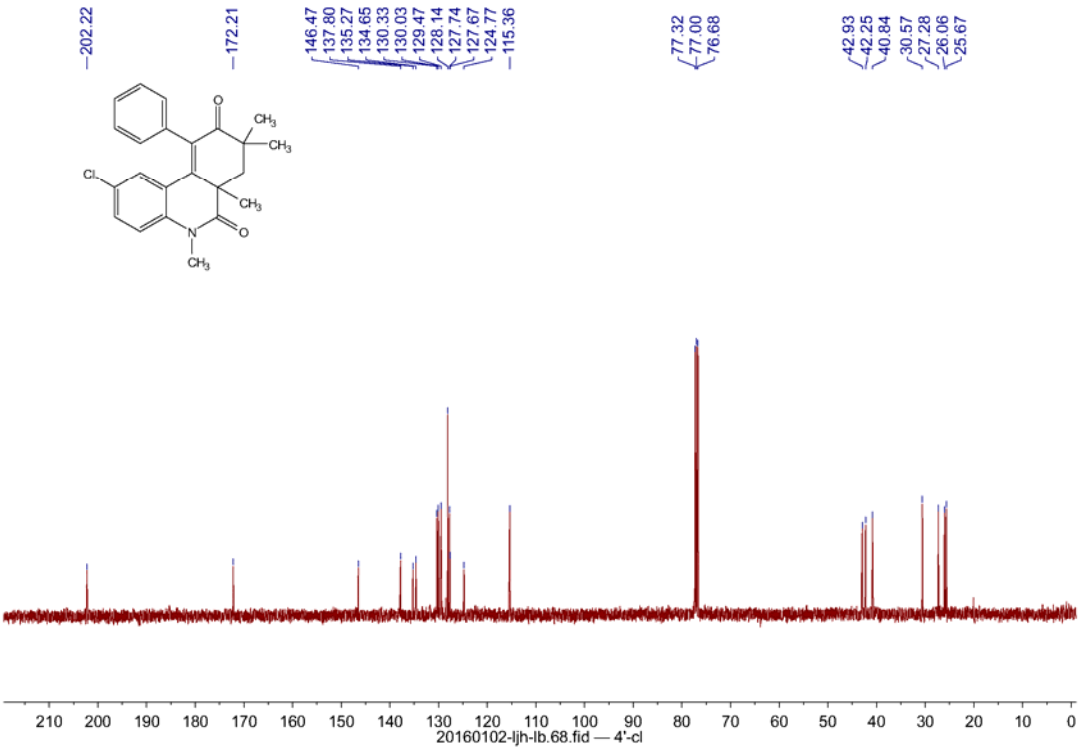
10-Butyl-5,6a,8,8-tetramethyl-7,8-dihydrophenanthridine-6,9(5*H*,6a*H*)-dione (**3la**)



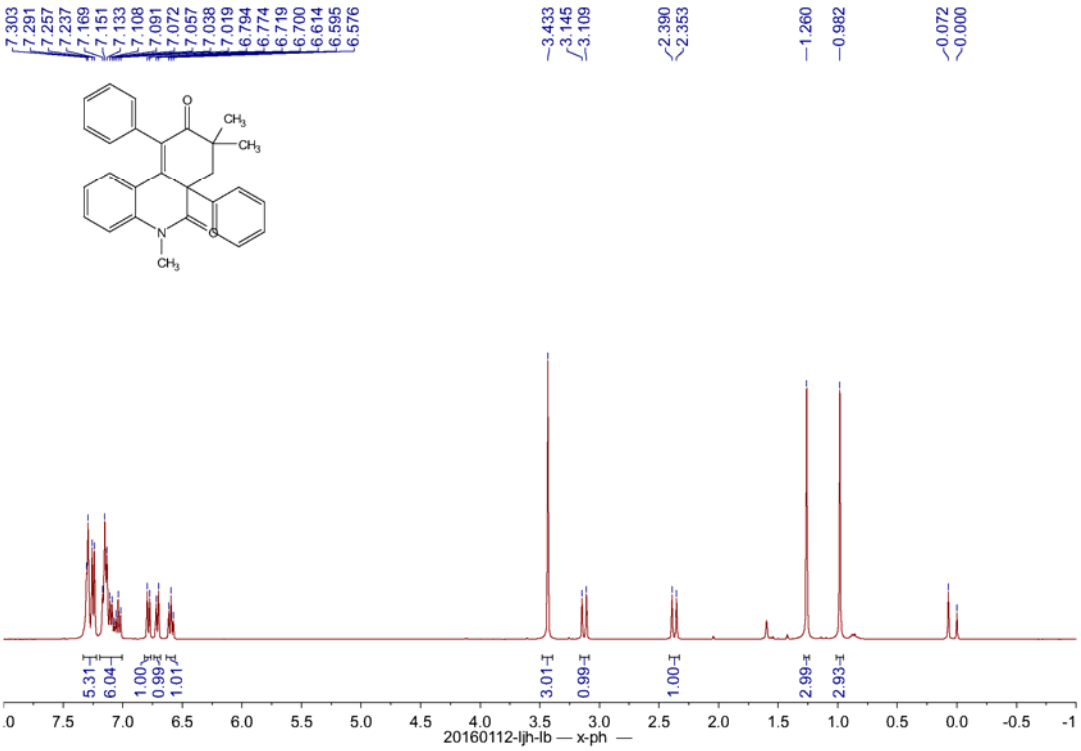


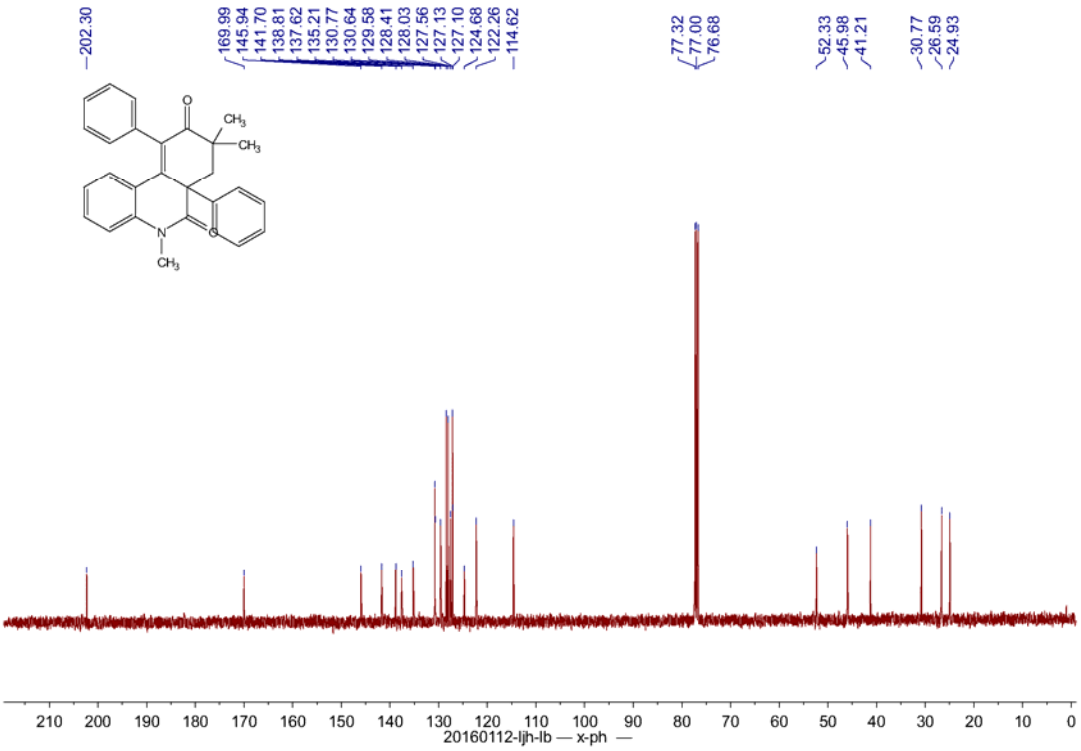
2-Chloro-5,6a,8,8-tetramethyl-10-phenyl-7,8-dihydrophenanthridine-6,9(5*H*,6a*H*)-dione (**3ma**)



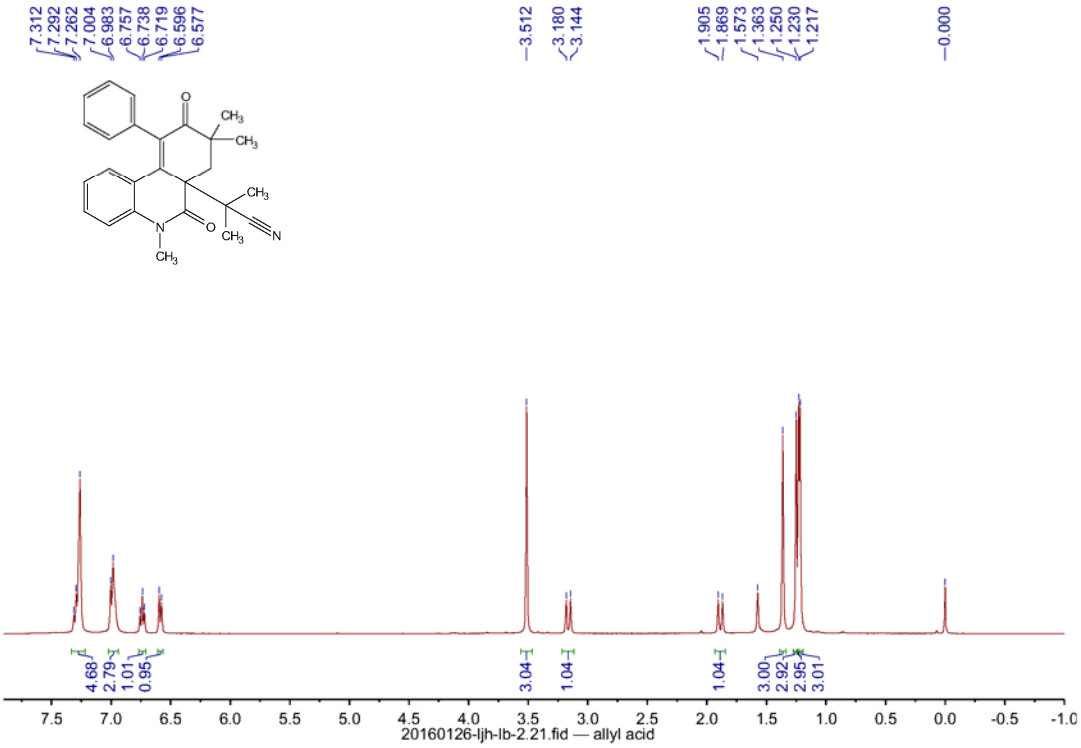


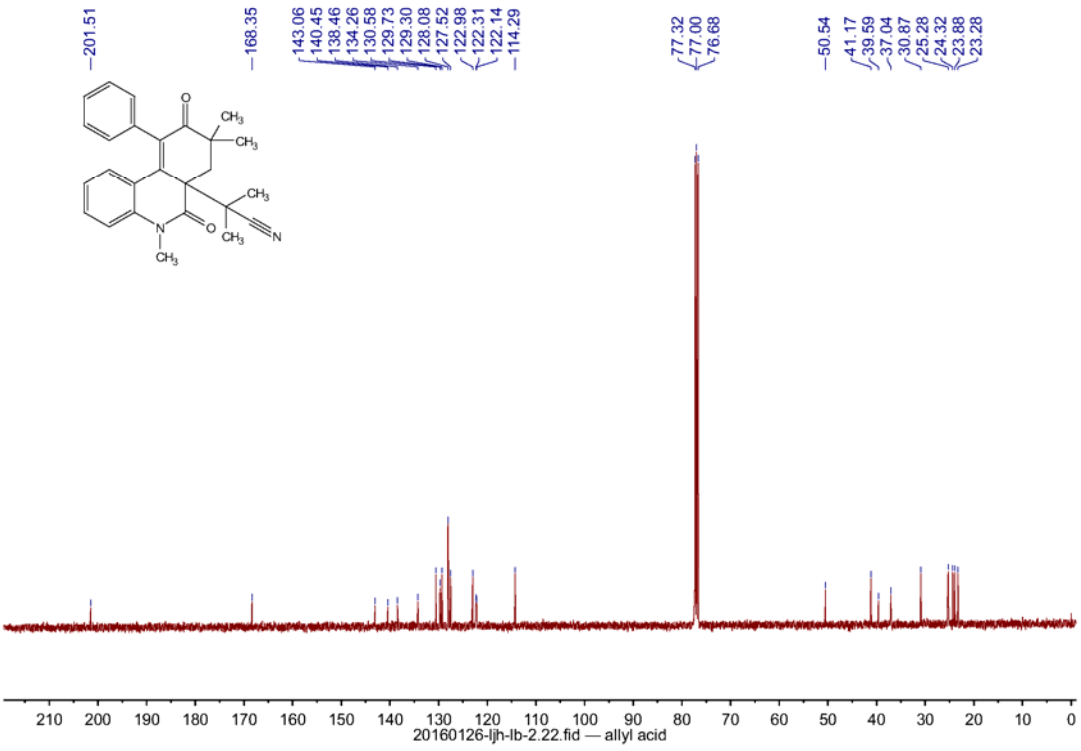
5,8,8-Trimethyl-6a,10-diphenyl-7,8-dihydrophenanthridine-6,9(5*H*,6a*H*)-dione (**3na**)



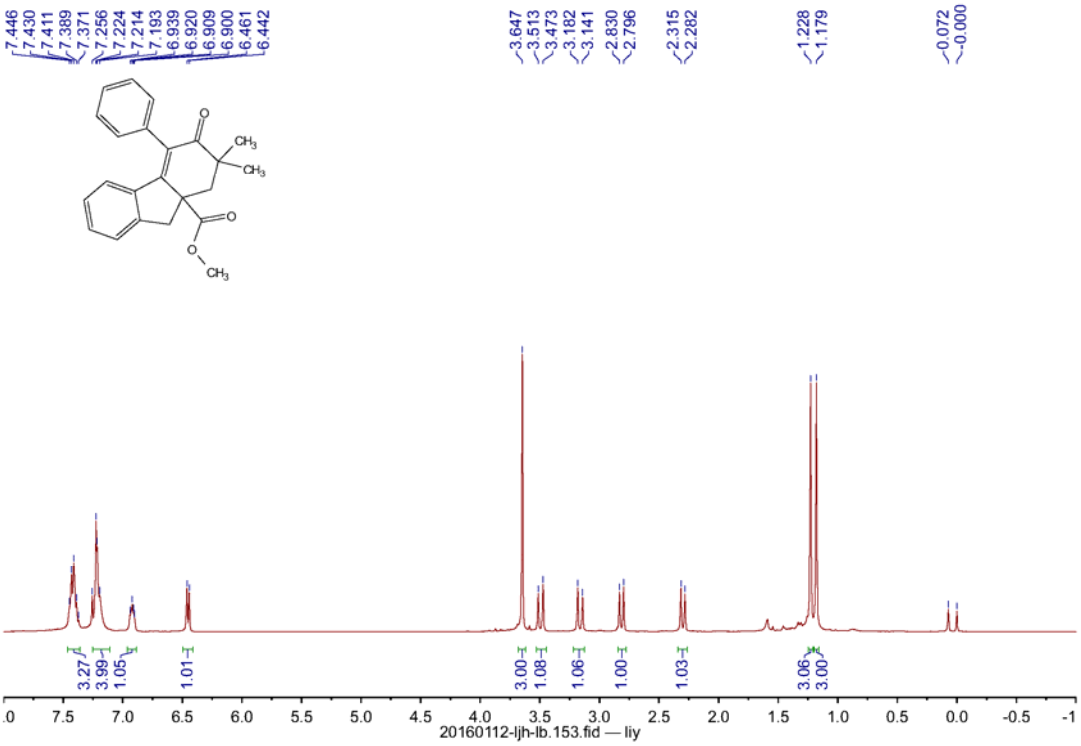


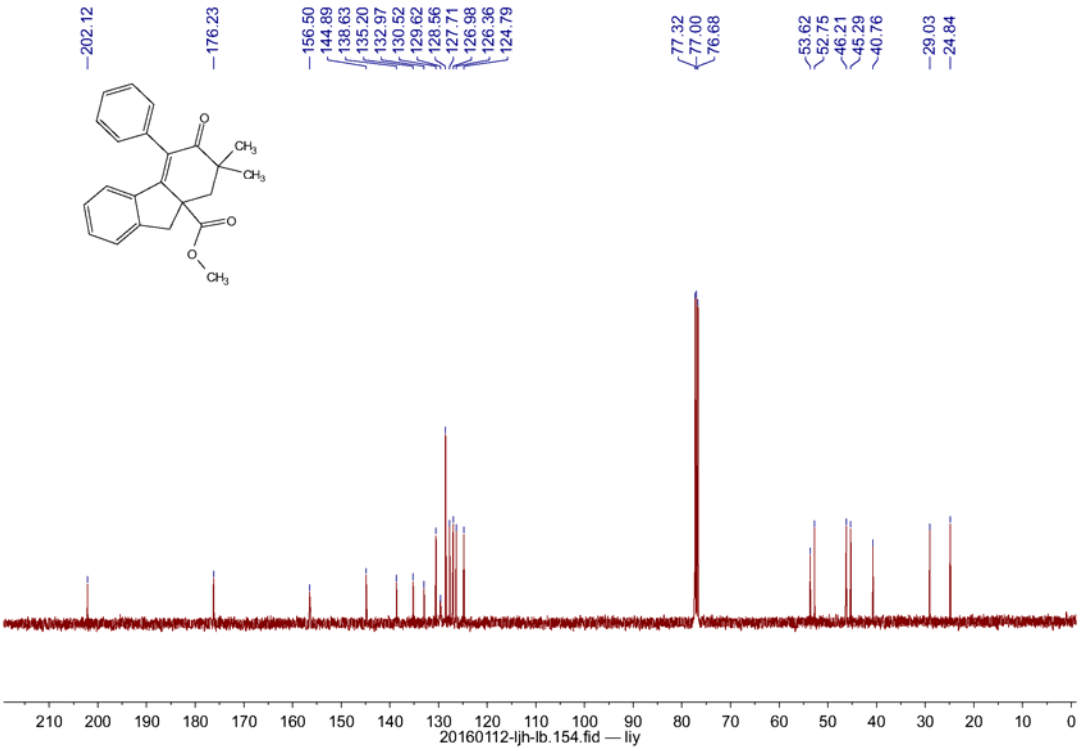
5,8,8-Trimethyl-10-phenyl-7,8-dihydrophenanthridine-6,9(5*H*,6a*H*)-dione (**3oa**)



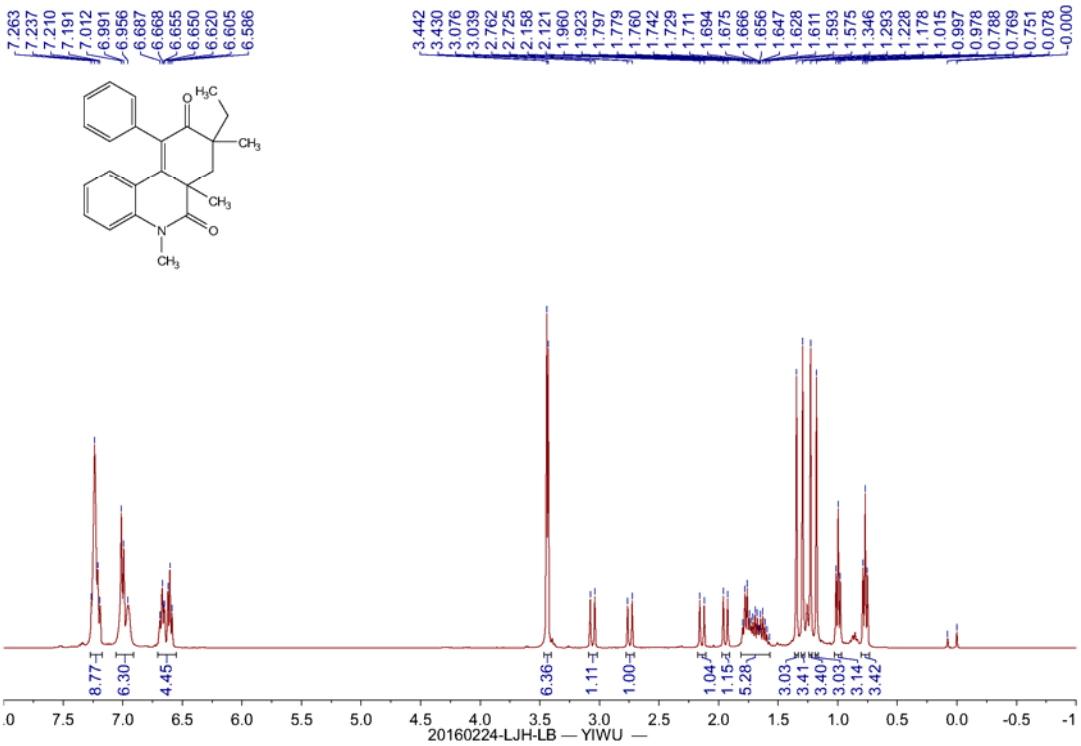


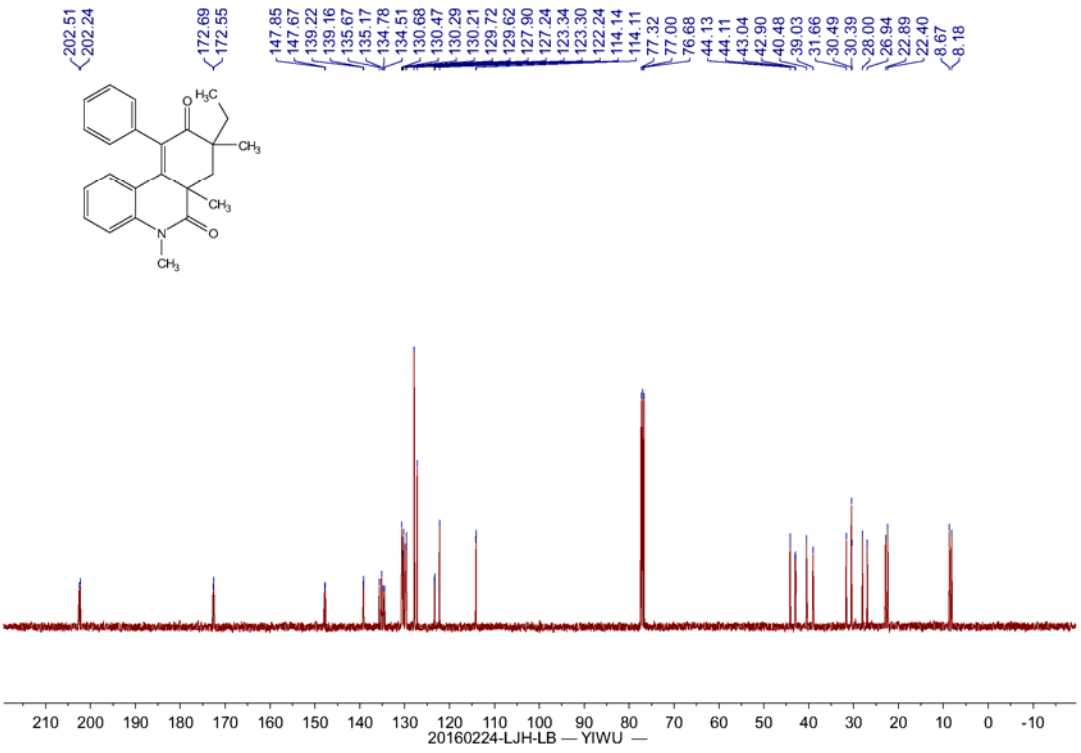
Methyl 7,7-dimethyl-6-oxo-5-phenyl-6,7,8,9-tetrahydro-8a*H*-fluorene-8a-carboxylate (**3qa**)



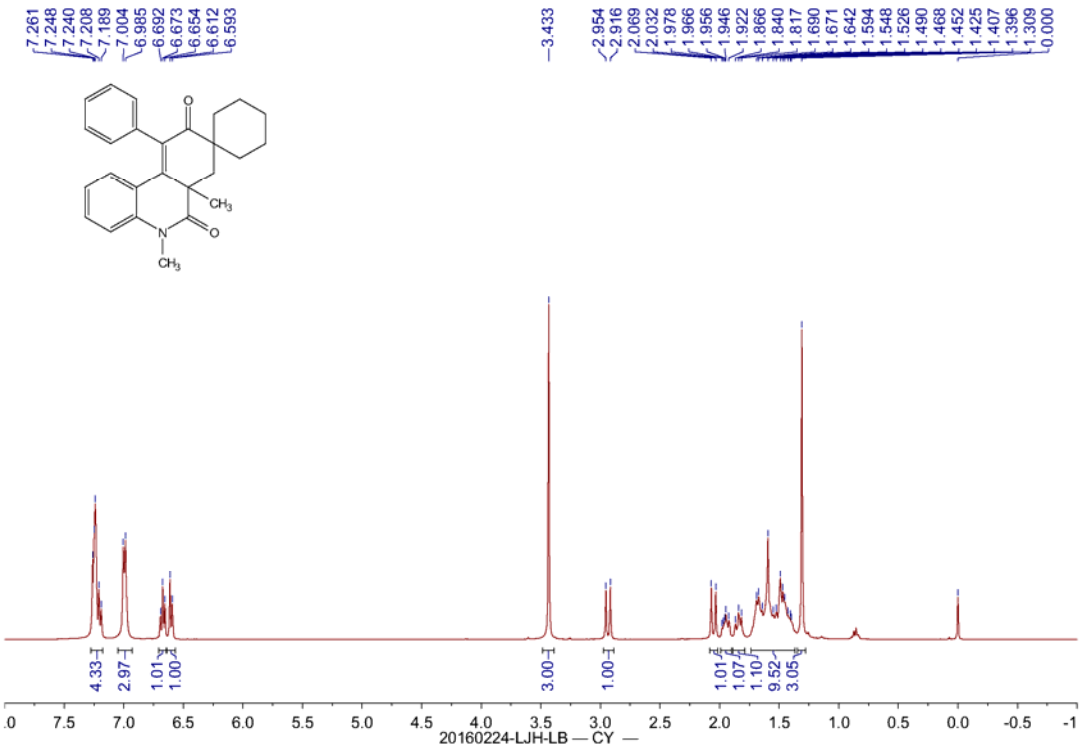


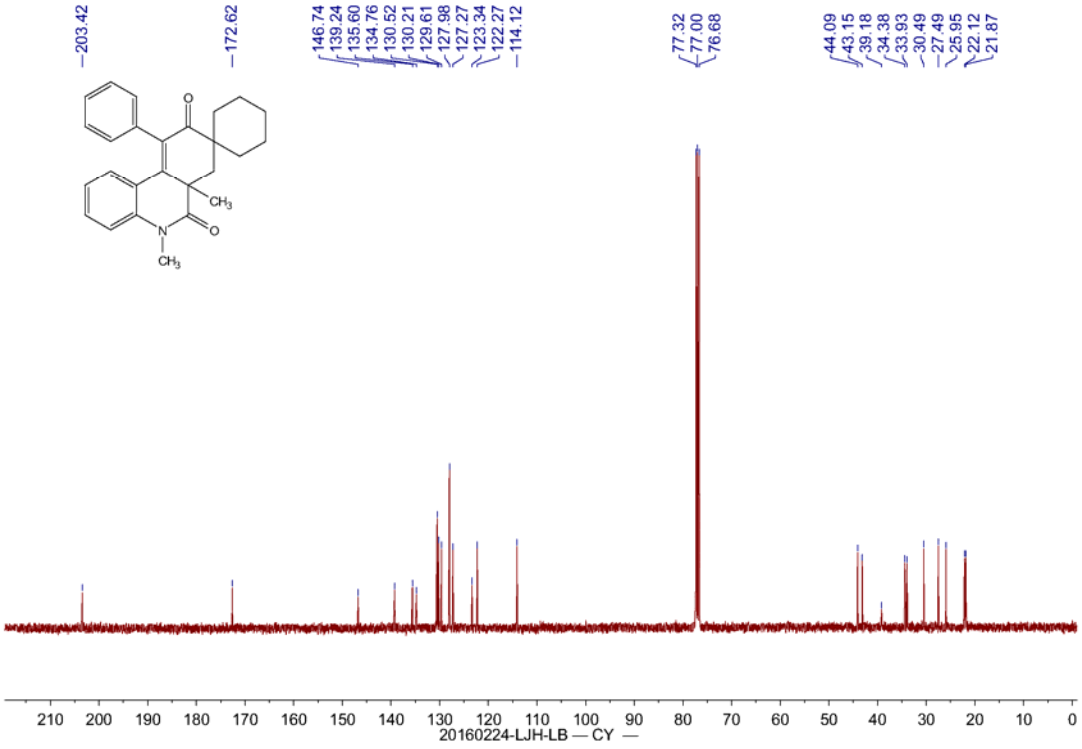
8-Ethyl-5,6a,8-trimethyl-10-phenyl-7,8-dihydrophenanthridine-6,9(5*H*,6a*H*)-dione (**3ab**)



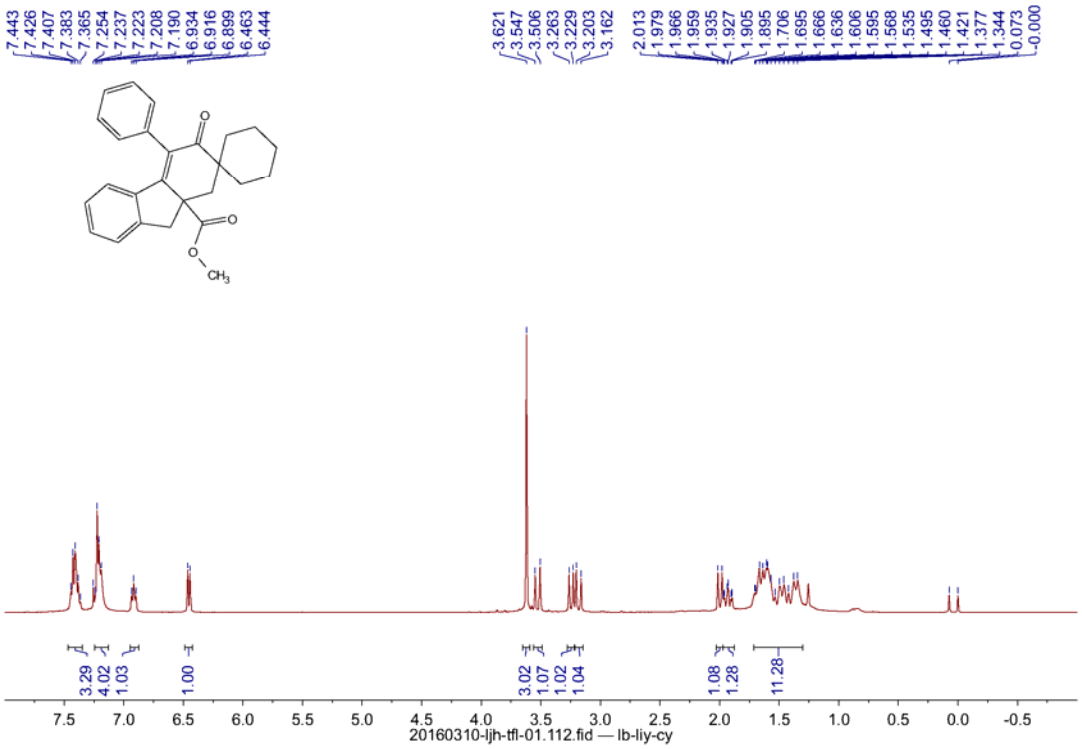


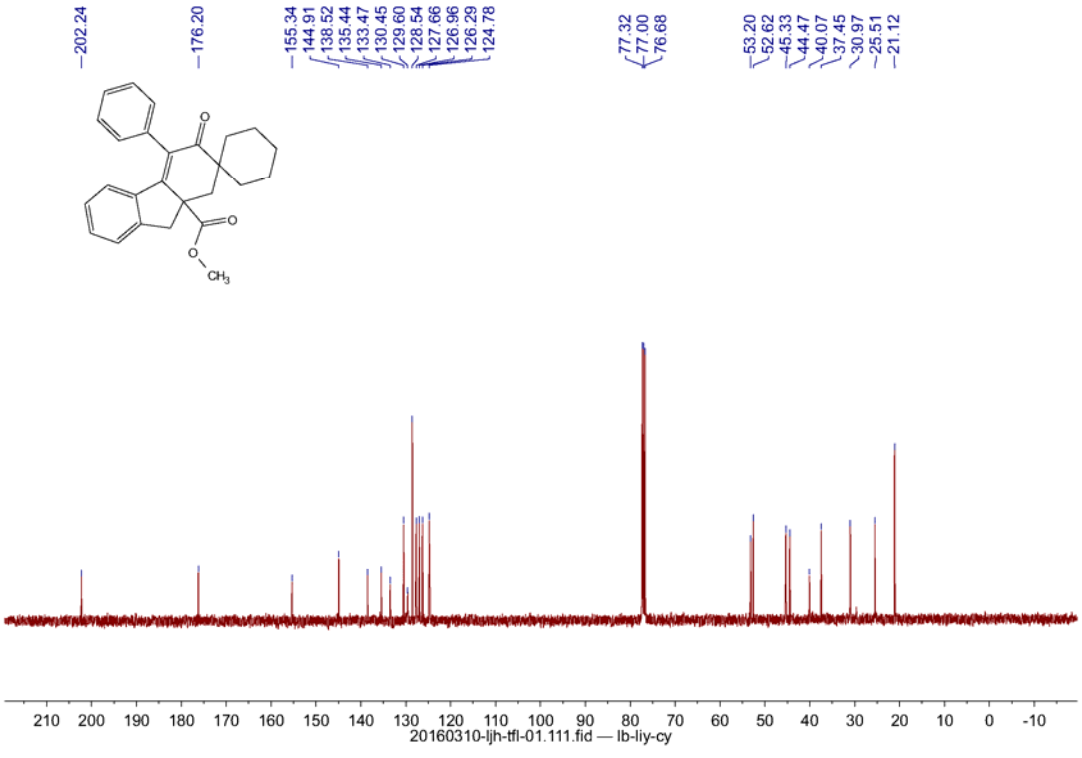
5',6a'-Dimethyl-10'-phenyl-6a',7'-dihydro-6'*H*-spiro[cyclohexane-1,8'-phenanthridine]-6',9'(5'*H*)-dione (**3ac**)



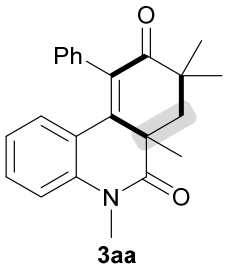


Methyl 3'-oxo-4'-phenyl-3',9'-dihydrospiro[cyclohexane-1,2'-fluorene]-9a'(1'*H*)-carboxylate (**3qc**)





(E) The X-ray single-crystal diffraction analysis of 3aa



3aa

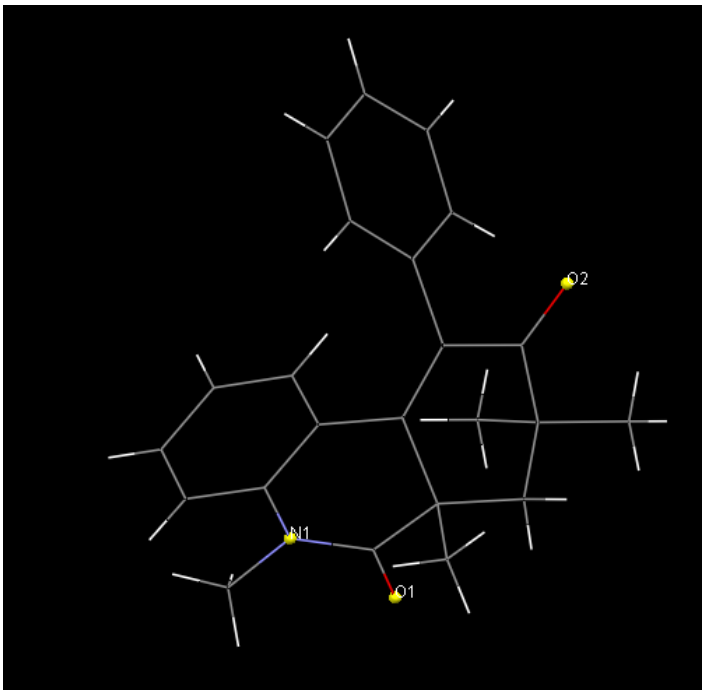


Table 1. Crystal data and structure refinement for ljh039_0m.

Identification code	ljh039_0m
Empirical formula	C23 H23 N O2
Formula weight	345.42
Temperature	296(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P2(1)/c
Unit cell dimensions	a = 10.9689(7) A alpha = 90 deg.
	b = 11.2631(8) A beta = 104.2180(10) deg.
	c = 15.5168(10) A gamma = 90 deg.
Volume	1858.3(2) A^3
Z, Calculated density	4, 1.235 Mg/m^3
Absorption coefficient	0.078 mm^-1
F(000)	736
Crystal size	0.23 x 0.21 x 0.20 mm

Theta range for data collection	2.63 to 27.53 deg.
Limiting indices	-14<=h<=14, -14<=k<=14, -20<=l<=20
Reflections collected / unique	22432 / 4272 [R(int) = 0.0307]
Completeness to theta = 27.53	99.6 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9845 and 0.9822
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4272 / 0 / 236
Goodness-of-fit on F^2	1.048
Final R indices [I>2sigma(I)]	R1 = 0.0466, wR2 = 0.1250
R indices (all data)	R1 = 0.0664, wR2 = 0.1379
Extinction coefficient	0.0044(12)
Largest diff. peak and hole	0.319 and -0.264 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for ljh039_0m. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
N(1)	3783(1)	8188(1)	8419(1)	41(1)
C(1)	3248(1)	7901(1)	9101(1)	37(1)
O(1)	3667(1)	7108(1)	9626(1)	51(1)
O(2)	1634(1)	11419(1)	10719(1)	61(1)
C(2)	2052(1)	8585(1)	9138(1)	34(1)
C(3)	1769(1)	8477(1)	10050(1)	38(1)
C(4)	2375(1)	9419(1)	10728(1)	38(1)
C(5)	1967(1)	10616(1)	10308(1)	37(1)
C(6)	2067(1)	10796(1)	9374(1)	34(1)

C(7)	2175(1)	9862(1)	8857(1)	33(1)
C(8)	2460(1)	9935(1)	7975(1)	37(1)
C(9)	1955(2)	10775(2)	7329(1)	46(1)
C(10)	2226(2)	10759(2)	6504(1)	58(1)
C(11)	3015(2)	9896(2)	6319(1)	61(1)
C(12)	3531(2)	9052(2)	6944(1)	53(1)
C(13)	3258(1)	9055(1)	7775(1)	39(1)
C(14)	4864(2)	7476(2)	8327(1)	56(1)
C(15)	969(2)	7960(2)	8452(1)	45(1)
C(16)	1926(2)	9275(2)	11580(1)	55(1)
C(17)	3816(2)	9373(2)	10954(1)	46(1)
C(18)	2162(1)	12072(1)	9133(1)	35(1)
C(19)	1117(2)	12802(2)	8948(1)	52(1)
C(20)	1231(2)	13997(2)	8794(1)	61(1)
C(21)	2386(2)	14483(2)	8832(1)	57(1)
C(22)	3427(2)	13776(2)	9025(1)	61(1)
C(23)	3320(2)	12578(2)	9175(1)	49(1)

Table 3. Selected bond lengths [A] and angles [deg] for ljh039_0m.

Symmetry transformations used to generate equivalent atoms:

Table 4. Bond lengths [A] and angles [deg] for ljh039_0m.

N(1)-C(1)	1.369(2)
N(1)-C(13)	1.414(2)
N(1)-C(14)	1.468(2)
C(1)-O(1)	1.2196(18)
C(1)-C(2)	1.535(2)
O(2)-C(5)	1.2138(18)

C(2)-C(7)	1.519(2)
C(2)-C(3)	1.527(2)
C(2)-C(15)	1.555(2)
C(3)-C(4)	1.527(2)
C(3)-H(3A)	0.9700
C(3)-H(3B)	0.9700
C(4)-C(5)	1.516(2)
C(4)-C(16)	1.529(2)
C(4)-C(17)	1.534(2)
C(5)-C(6)	1.495(2)
C(6)-C(7)	1.346(2)
C(6)-C(18)	1.494(2)
C(7)-C(8)	1.479(2)
C(8)-C(9)	1.390(2)
C(8)-C(13)	1.407(2)
C(9)-C(10)	1.384(2)
C(9)-H(9)	0.9300
C(10)-C(11)	1.378(3)
C(10)-H(10)	0.9300
C(11)-C(12)	1.377(3)
C(11)-H(11)	0.9300
C(12)-C(13)	1.393(2)
C(12)-H(12)	0.9300
C(14)-H(14A)	0.9600
C(14)-H(14B)	0.9600
C(14)-H(14C)	0.9600
C(15)-H(15A)	0.9600
C(15)-H(15B)	0.9600
C(15)-H(15C)	0.9600
C(16)-H(16A)	0.9600

C(16)-H(16B)	0.9600
C(16)-H(16C)	0.9600
C(17)-H(17A)	0.9600
C(17)-H(17B)	0.9600
C(17)-H(17C)	0.9600
C(18)-C(23)	1.380(2)
C(18)-C(19)	1.383(2)
C(19)-C(20)	1.377(3)
C(19)-H(19)	0.9300
C(20)-C(21)	1.368(3)
C(20)-H(20)	0.9300
C(21)-C(22)	1.364(3)
C(21)-H(21)	0.9300
C(22)-C(23)	1.379(3)
C(22)-H(22)	0.9300
C(23)-H(23)	0.9300

C(1)-N(1)-C(13)	122.15(13)
C(1)-N(1)-C(14)	117.13(14)
C(13)-N(1)-C(14)	120.46(14)
O(1)-C(1)-N(1)	121.74(14)
O(1)-C(1)-C(2)	121.89(14)
N(1)-C(1)-C(2)	116.29(13)
C(7)-C(2)-C(3)	113.21(12)
C(7)-C(2)-C(1)	109.10(12)
C(3)-C(2)-C(1)	111.38(12)
C(7)-C(2)-C(15)	109.83(12)
C(3)-C(2)-C(15)	107.67(12)
C(1)-C(2)-C(15)	105.34(12)
C(4)-C(3)-C(2)	115.46(12)

C(4)-C(3)-H(3A)	108.4
C(2)-C(3)-H(3A)	108.4
C(4)-C(3)-H(3B)	108.4
C(2)-C(3)-H(3B)	108.4
H(3A)-C(3)-H(3B)	107.5
C(5)-C(4)-C(3)	106.82(12)
C(5)-C(4)-C(16)	110.01(13)
C(3)-C(4)-C(16)	110.09(13)
C(5)-C(4)-C(17)	107.93(13)
C(3)-C(4)-C(17)	112.59(13)
C(16)-C(4)-C(17)	109.34(14)
O(2)-C(5)-C(6)	120.75(14)
O(2)-C(5)-C(4)	122.19(14)
C(6)-C(5)-C(4)	116.95(12)
C(7)-C(6)-C(18)	125.52(13)
C(7)-C(6)-C(5)	120.66(13)
C(18)-C(6)-C(5)	113.55(12)
C(6)-C(7)-C(8)	125.28(13)
C(6)-C(7)-C(2)	122.87(13)
C(8)-C(7)-C(2)	111.83(12)
C(9)-C(8)-C(13)	118.68(14)
C(9)-C(8)-C(7)	124.56(14)
C(13)-C(8)-C(7)	116.71(13)
C(10)-C(9)-C(8)	121.34(16)
C(10)-C(9)-H(9)	119.3
C(8)-C(9)-H(9)	119.3
C(11)-C(10)-C(9)	119.36(17)
C(11)-C(10)-H(10)	120.3
C(9)-C(10)-H(10)	120.3
C(12)-C(11)-C(10)	120.71(17)

C(12)-C(11)-H(11)	119.6
C(10)-C(11)-H(11)	119.6
C(11)-C(12)-C(13)	120.39(17)
C(11)-C(12)-H(12)	119.8
C(13)-C(12)-H(12)	119.8
C(12)-C(13)-C(8)	119.52(15)
C(12)-C(13)-N(1)	120.64(15)
C(8)-C(13)-N(1)	119.84(14)
N(1)-C(14)-H(14A)	109.5
N(1)-C(14)-H(14B)	109.5
H(14A)-C(14)-H(14B)	109.5
N(1)-C(14)-H(14C)	109.5
H(14A)-C(14)-H(14C)	109.5
H(14B)-C(14)-H(14C)	109.5
C(2)-C(15)-H(15A)	109.5
C(2)-C(15)-H(15B)	109.5
H(15A)-C(15)-H(15B)	109.5
C(2)-C(15)-H(15C)	109.5
H(15A)-C(15)-H(15C)	109.5
H(15B)-C(15)-H(15C)	109.5
C(4)-C(16)-H(16A)	109.5
C(4)-C(16)-H(16B)	109.5
H(16A)-C(16)-H(16B)	109.5
C(4)-C(16)-H(16C)	109.5
H(16A)-C(16)-H(16C)	109.5
H(16B)-C(16)-H(16C)	109.5
C(4)-C(17)-H(17A)	109.5
C(4)-C(17)-H(17B)	109.5
H(17A)-C(17)-H(17B)	109.5
C(4)-C(17)-H(17C)	109.5

H(17A)-C(17)-H(17C)	109.5
H(17B)-C(17)-H(17C)	109.5
C(23)-C(18)-C(19)	118.07(15)
C(23)-C(18)-C(6)	120.35(14)
C(19)-C(18)-C(6)	121.28(14)
C(20)-C(19)-C(18)	120.75(17)
C(20)-C(19)-H(19)	119.6
C(18)-C(19)-H(19)	119.6
C(21)-C(20)-C(19)	120.42(18)
C(21)-C(20)-H(20)	119.8
C(19)-C(20)-H(20)	119.8
C(22)-C(21)-C(20)	119.45(17)
C(22)-C(21)-H(21)	120.3
C(20)-C(21)-H(21)	120.3
C(21)-C(22)-C(23)	120.49(18)
C(21)-C(22)-H(22)	119.8
C(23)-C(22)-H(22)	119.8
C(22)-C(23)-C(18)	120.80(17)
C(22)-C(23)-H(23)	119.6
C(18)-C(23)-H(23)	119.6

Symmetry transformations used to generate equivalent atoms:

Table 5. Anisotropic displacement parameters (A^2 x 10^3) for ljh039_0m.

The anisotropic displacement factor exponent takes the form:-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
N(1)	43(1)	35(1)	47(1)	-3(1)	12(1)	6(1)
C(1)	40(1)	25(1)	43(1)	-4(1)	4(1)	0(1)
O(1)	55(1)	35(1)	60(1)	7(1)	9(1)	9(1)

O(2)	93(1)	44(1)	55(1)	-6(1)	34(1)	12(1)
C(2)	35(1)	27(1)	37(1)	-2(1)	5(1)	-1(1)
C(3)	40(1)	31(1)	42(1)	3(1)	10(1)	-4(1)
C(4)	42(1)	34(1)	36(1)	1(1)	10(1)	-1(1)
C(5)	38(1)	34(1)	40(1)	-3(1)	10(1)	-1(1)
C(6)	34(1)	29(1)	37(1)	0(1)	7(1)	2(1)
C(7)	32(1)	30(1)	35(1)	1(1)	4(1)	1(1)
C(8)	43(1)	31(1)	35(1)	-3(1)	8(1)	-2(1)
C(9)	60(1)	35(1)	41(1)	2(1)	7(1)	1(1)
C(10)	84(1)	48(1)	41(1)	8(1)	11(1)	-7(1)
C(11)	89(2)	56(1)	44(1)	-3(1)	28(1)	-12(1)
C(12)	65(1)	48(1)	52(1)	-10(1)	26(1)	-5(1)
C(13)	42(1)	34(1)	41(1)	-6(1)	10(1)	-4(1)
C(14)	51(1)	53(1)	65(1)	-9(1)	17(1)	14(1)
C(15)	41(1)	40(1)	50(1)	-7(1)	4(1)	-6(1)
C(16)	71(1)	52(1)	45(1)	4(1)	22(1)	-4(1)
C(17)	44(1)	44(1)	46(1)	1(1)	2(1)	-1(1)
C(18)	41(1)	28(1)	35(1)	-2(1)	8(1)	2(1)
C(19)	42(1)	37(1)	71(1)	3(1)	5(1)	5(1)
C(20)	71(1)	35(1)	70(1)	5(1)	4(1)	16(1)
C(21)	91(2)	30(1)	51(1)	1(1)	17(1)	-6(1)
C(22)	66(1)	47(1)	73(1)	-1(1)	25(1)	-18(1)
C(23)	44(1)	42(1)	63(1)	0(1)	16(1)	0(1)

Table 6. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for ljh039_0m.

	x	y	z	U(eq)
H(3A)	865	8512	9972	45
H(3B)	2047	7701	10293	45

H(9)	1426	11359	7455	55
H(10)	1878	11325	6078	70
H(11)	3201	9883	5765	73
H(12)	4065	8477	6811	64
H(14A)	5157	7759	7830	84
H(14B)	4616	6659	8232	84
H(14C)	5528	7542	8859	84
H(15A)	893	7154	8633	68
H(15B)	1151	7967	7877	68
H(15C)	194	8373	8421	68
H(16A)	2186	8514	11840	82
H(16B)	1025	9329	11442	82
H(16C)	2285	9891	11992	82
H(17A)	4098	8620	11221	70
H(17B)	4152	9999	11363	70
H(17C)	4100	9469	10420	70
H(19)	328	12484	8928	62
H(20)	518	14475	8663	73
H(21)	2460	15290	8727	69
H(22)	4215	14104	9056	73
H(23)	4038	12106	9307	59

Table 7. Selected torsion angles [deg] for ljh039_0m.

Symmetry transformations used to generate equivalent atoms:

Table 8. Torsion angles [deg] for ljh039_0m.

C(13)-N(1)-C(1)-O(1)	175.93(14)
C(14)-N(1)-C(1)-O(1)	1.8(2)

C(13)-N(1)-C(1)-C(2)	-0.8(2)
C(14)-N(1)-C(1)-C(2)	-174.90(13)
O(1)-C(1)-C(2)-C(7)	146.19(14)
N(1)-C(1)-C(2)-C(7)	-37.12(17)
O(1)-C(1)-C(2)-C(3)	20.49(19)
N(1)-C(1)-C(2)-C(3)	-162.82(13)
O(1)-C(1)-C(2)-C(15)	-95.96(17)
N(1)-C(1)-C(2)-C(15)	80.73(16)
C(7)-C(2)-C(3)-C(4)	-34.38(17)
C(1)-C(2)-C(3)-C(4)	89.01(15)
C(15)-C(2)-C(3)-C(4)	-155.98(13)
C(2)-C(3)-C(4)-C(5)	55.64(16)
C(2)-C(3)-C(4)-C(16)	175.05(13)
C(2)-C(3)-C(4)-C(17)	-62.67(17)
C(3)-C(4)-C(5)-O(2)	136.75(16)
C(16)-C(4)-C(5)-O(2)	17.3(2)
C(17)-C(4)-C(5)-O(2)	-101.95(18)
C(3)-C(4)-C(5)-C(6)	-47.06(17)
C(16)-C(4)-C(5)-C(6)	-166.54(13)
C(17)-C(4)-C(5)-C(6)	74.24(16)
O(2)-C(5)-C(6)-C(7)	-165.97(15)
C(4)-C(5)-C(6)-C(7)	17.8(2)
O(2)-C(5)-C(6)-C(18)	19.7(2)
C(4)-C(5)-C(6)-C(18)	-156.56(13)
C(18)-C(6)-C(7)-C(8)	2.8(2)
C(5)-C(6)-C(7)-C(8)	-170.86(13)
C(18)-C(6)-C(7)-C(2)	-179.16(13)
C(5)-C(6)-C(7)-C(2)	7.2(2)
C(3)-C(2)-C(7)-C(6)	1.2(2)
C(1)-C(2)-C(7)-C(6)	-123.45(15)

C(15)-C(2)-C(7)-C(6)	121.57(15)
C(3)-C(2)-C(7)-C(8)	179.49(12)
C(1)-C(2)-C(7)-C(8)	54.86(15)
C(15)-C(2)-C(7)-C(8)	-60.13(16)
C(6)-C(7)-C(8)-C(9)	-40.9(2)
C(2)-C(7)-C(8)-C(9)	140.84(15)
C(6)-C(7)-C(8)-C(13)	141.83(16)
C(2)-C(7)-C(8)-C(13)	-36.42(17)
C(13)-C(8)-C(9)-C(10)	-0.1(2)
C(7)-C(8)-C(9)-C(10)	-177.33(16)
C(8)-C(9)-C(10)-C(11)	-0.3(3)
C(9)-C(10)-C(11)-C(12)	0.2(3)
C(10)-C(11)-C(12)-C(13)	0.3(3)
C(11)-C(12)-C(13)-C(8)	-0.7(3)
C(11)-C(12)-C(13)-N(1)	179.84(16)
C(9)-C(8)-C(13)-C(12)	0.6(2)
C(7)-C(8)-C(13)-C(12)	178.01(14)
C(9)-C(8)-C(13)-N(1)	-179.94(14)
C(7)-C(8)-C(13)-N(1)	-2.5(2)
C(1)-N(1)-C(13)-C(12)	-157.73(15)
C(14)-N(1)-C(13)-C(12)	16.2(2)
C(1)-N(1)-C(13)-C(8)	22.8(2)
C(14)-N(1)-C(13)-C(8)	-163.26(15)
C(7)-C(6)-C(18)-C(23)	-77.2(2)
C(5)-C(6)-C(18)-C(23)	96.85(17)
C(7)-C(6)-C(18)-C(19)	109.29(19)
C(5)-C(6)-C(18)-C(19)	-76.68(19)
C(23)-C(18)-C(19)-C(20)	1.3(3)
C(6)-C(18)-C(19)-C(20)	174.97(17)
C(18)-C(19)-C(20)-C(21)	-0.8(3)

C(19)-C(20)-C(21)-C(22)	-0.1(3)
C(20)-C(21)-C(22)-C(23)	0.4(3)
C(21)-C(22)-C(23)-C(18)	0.1(3)
C(19)-C(18)-C(23)-C(22)	-0.9(3)
C(6)-C(18)-C(23)-C(22)	-174.67(17)

Symmetry transformations used to generate equivalent atoms:

Table 9. Hydrogen bonds for ljh039_0m [A and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)