Supplementary Material

Total synthesis of structures proposed for quinocitrinines A and B and their analogs. Microwave energy as efficient tool for generating heterocycles

Victoria Machtey, Hugo E. Gottlieb and Gerardo Byk*

Laboratory of Nano-Biotechnology, Department of Chemistry
Bar Ilan University 52900-Ramat Gan, Israel
E-mail: bykger@mail.biu.ac.il

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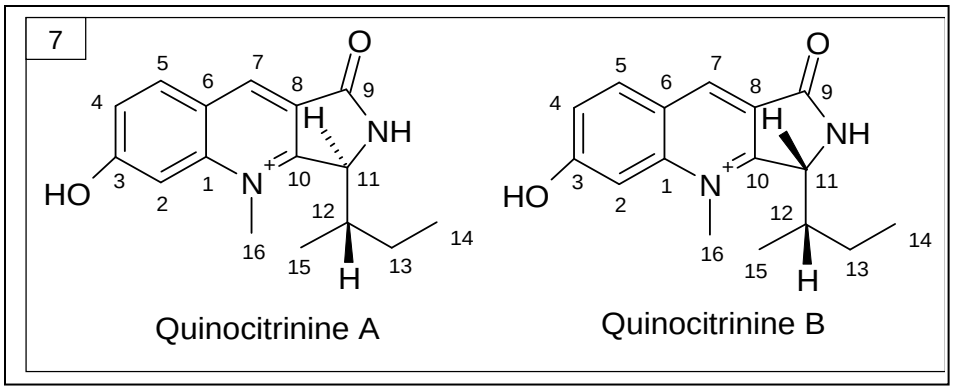


Figure S1. Arbitrary atom numbering of quinocitrinines 7 for NMR assignments.

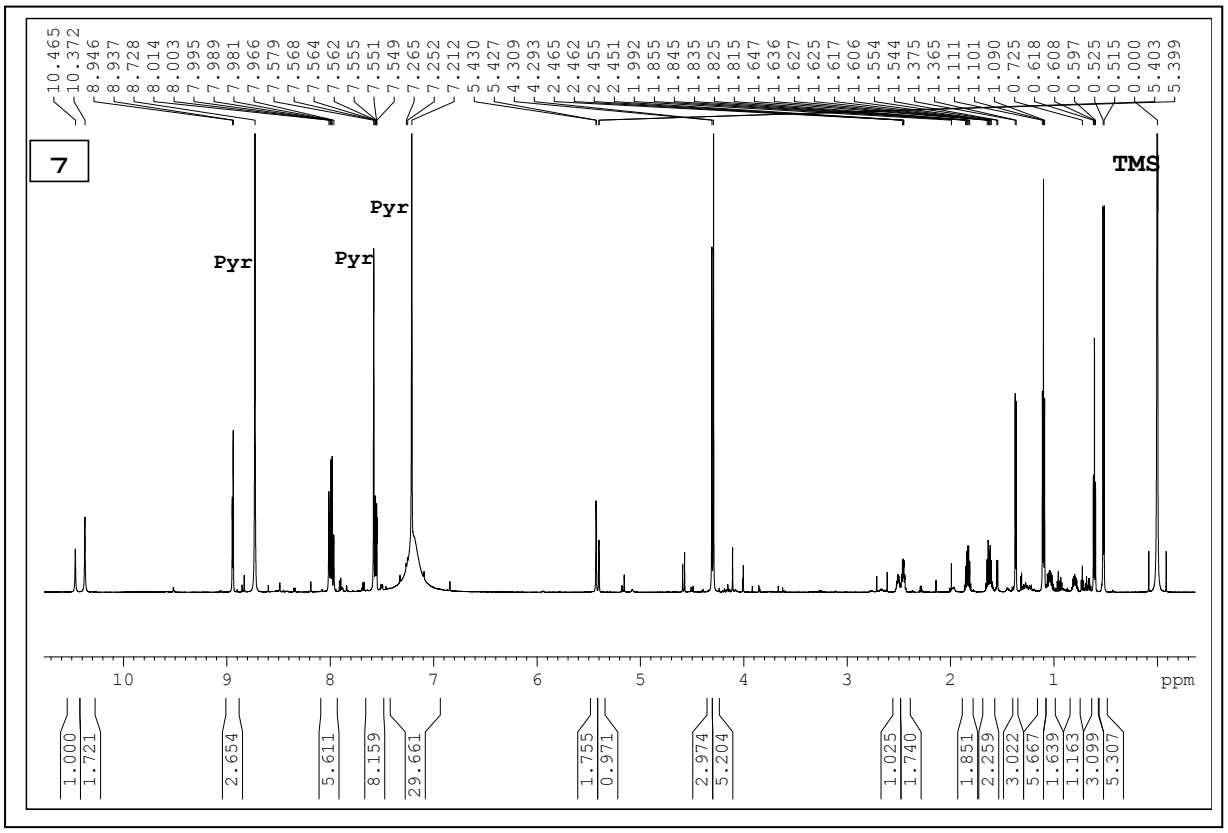


Figure S2. H-NMR of 7 in pyridine

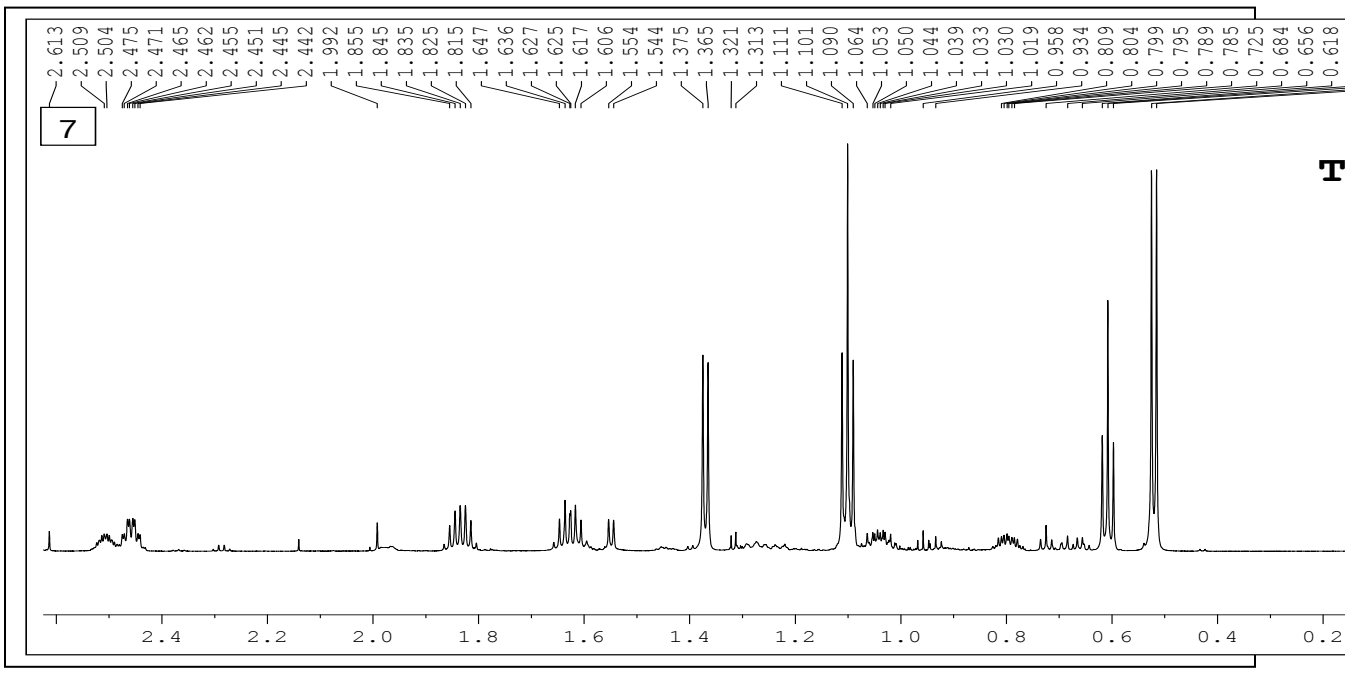


Figure S3. H-NMR of 7 in pyridine (enlarged)

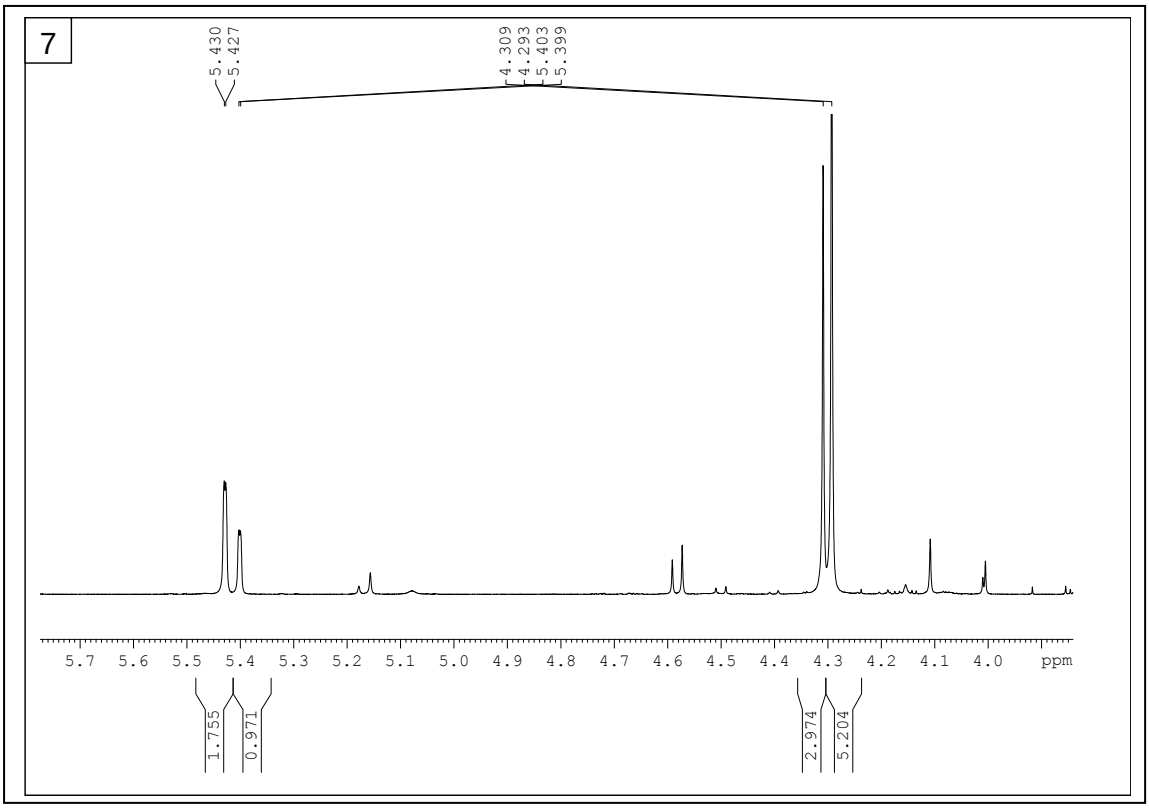


Figure S4. H-NMR of 7 in pyridine (enlarged)

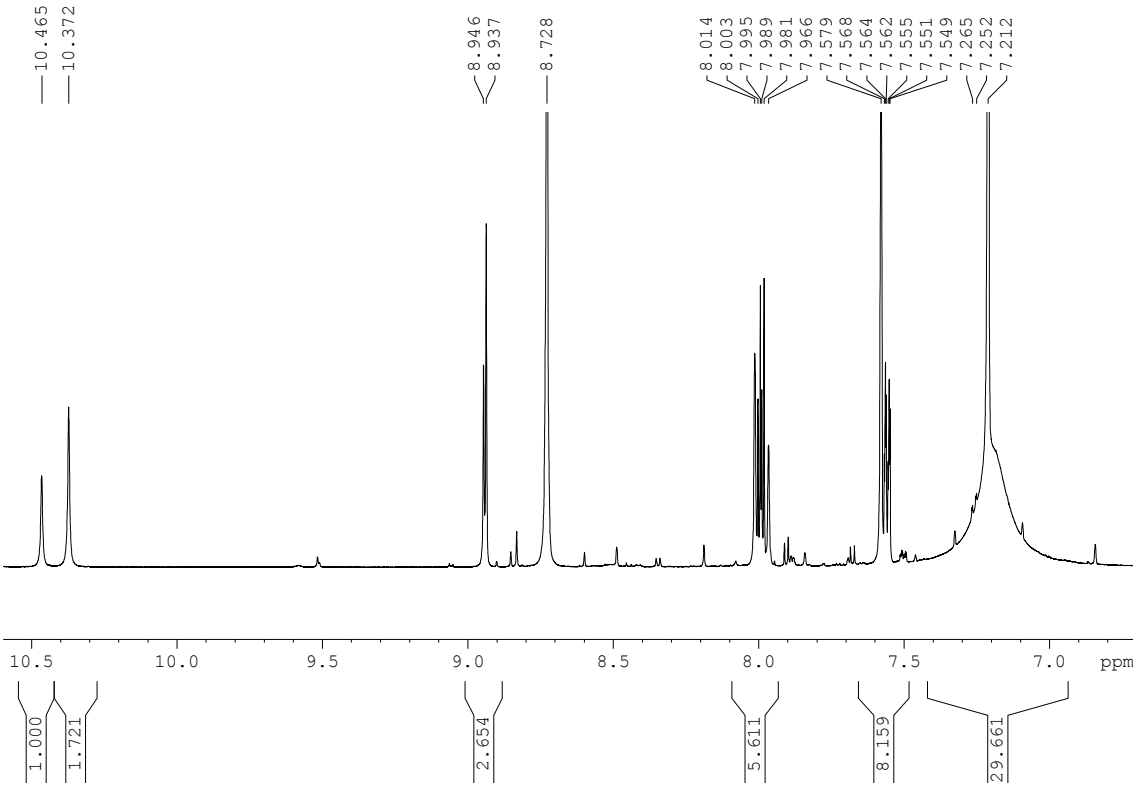


Figure S5. H-NMR of 7 in pyridine (enlarged)

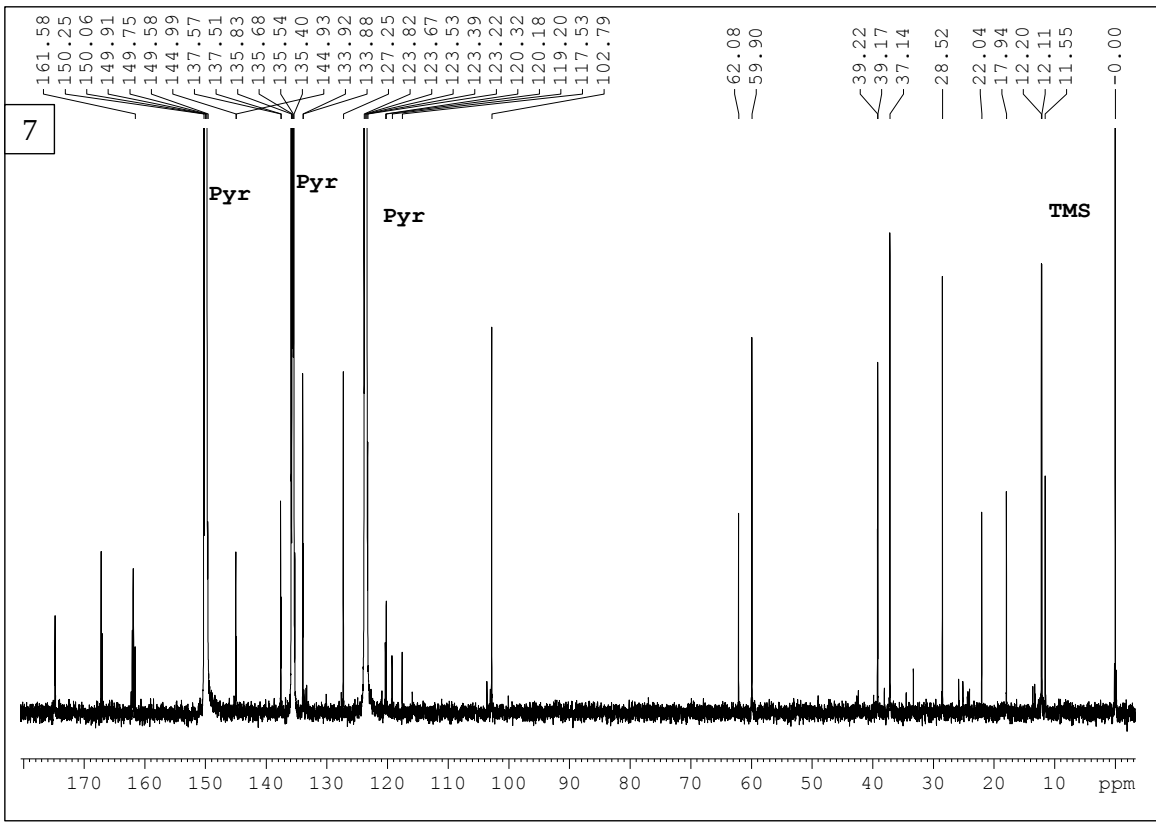


Figure S6. C-NMR of 7 in pyridine

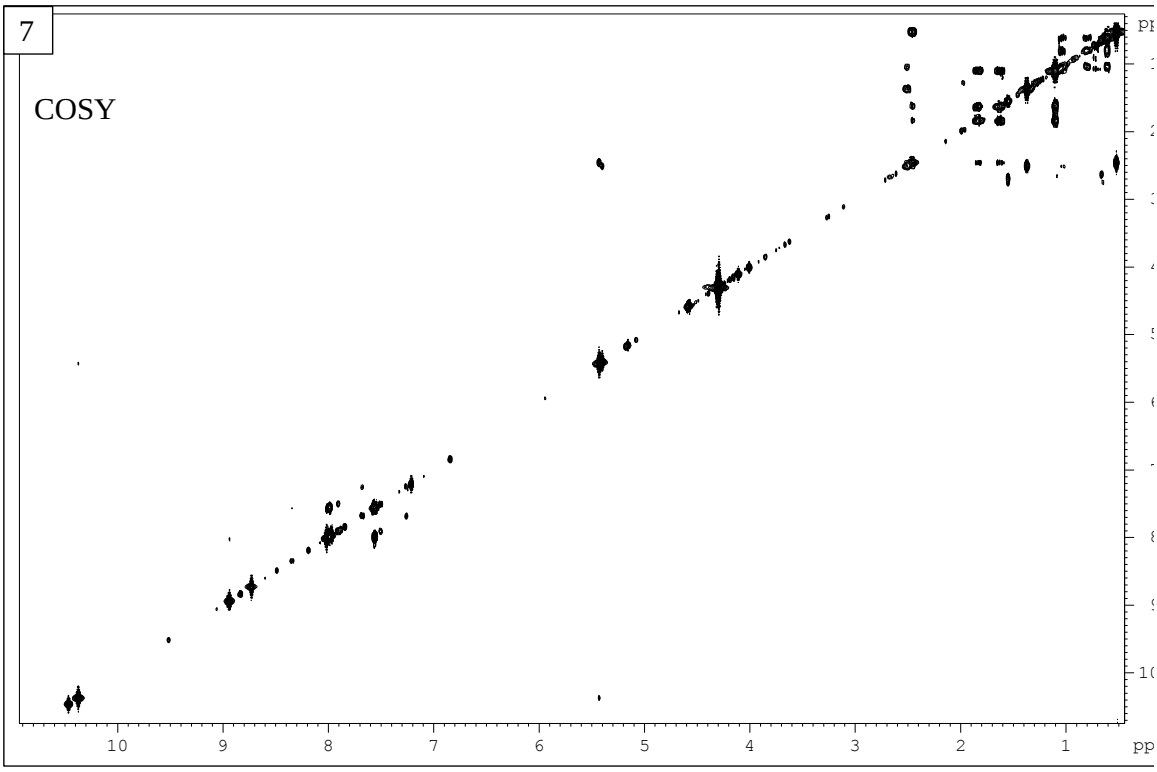


Figure S7. COSY spectrum of 7 in pyridine

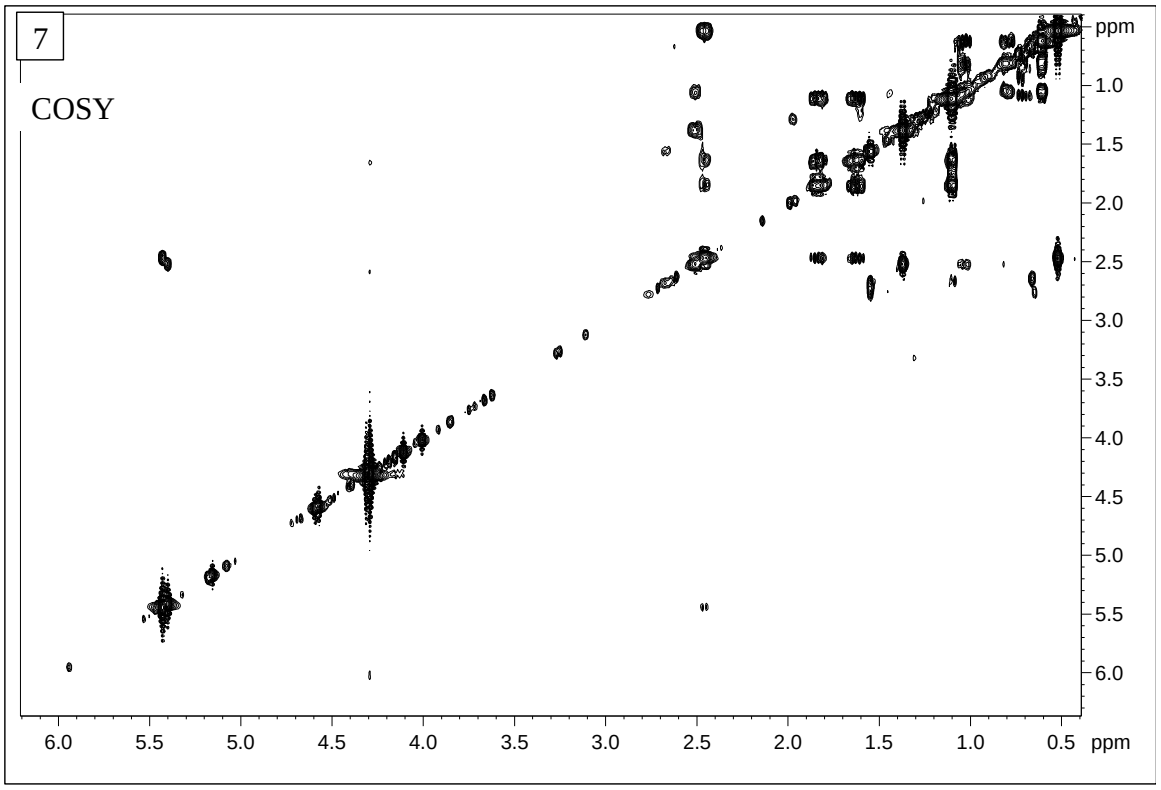


Figure S8. COSY spectrum of 7 in pyridine (enlarged)

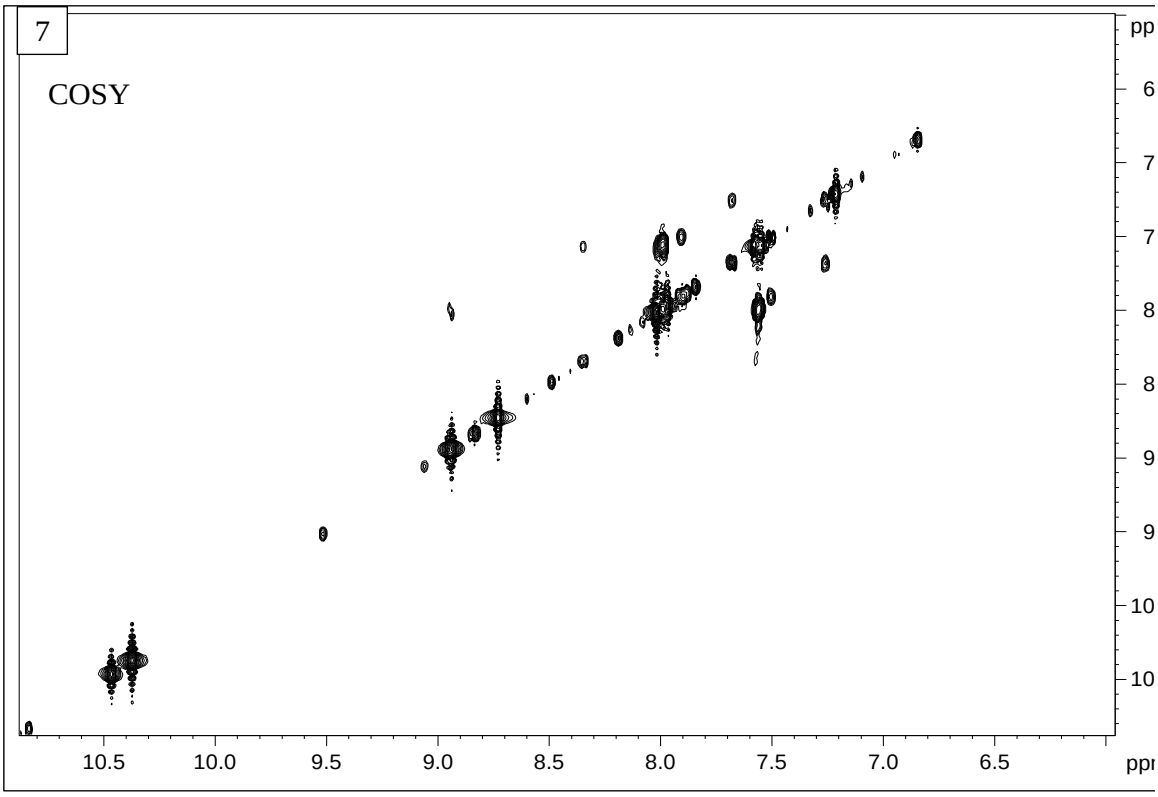


Figure S9. COSY spectrum of 7 in pyridine (enlarged)

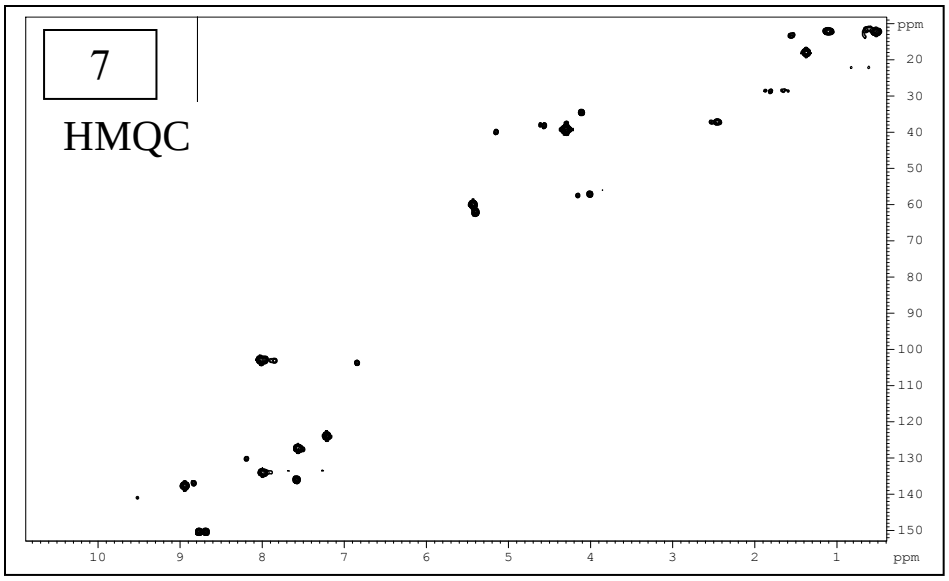


Figure S10. HMQC spectrum of 7 in pyridine

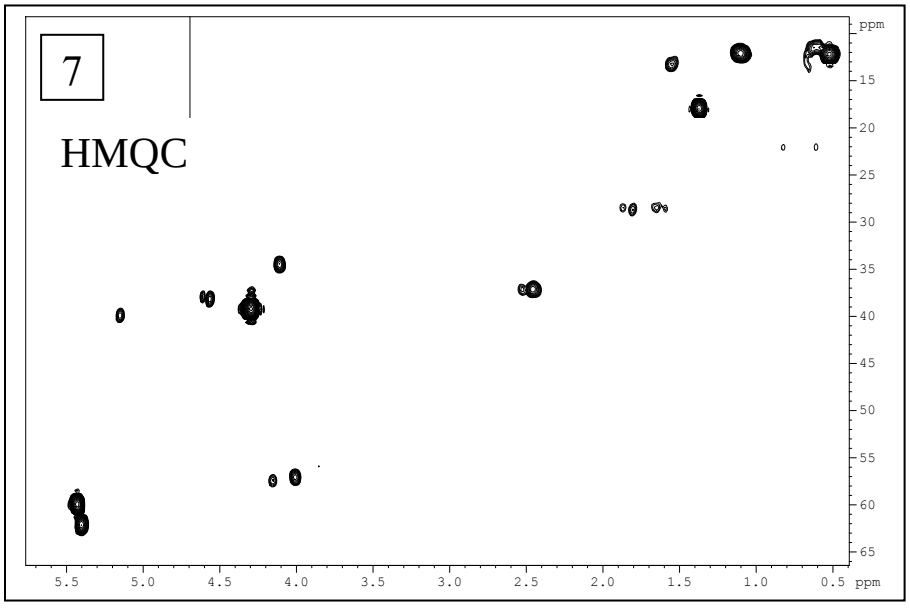


Figure S11. HMQC spectrum of 7 in pyridine (enlarged)

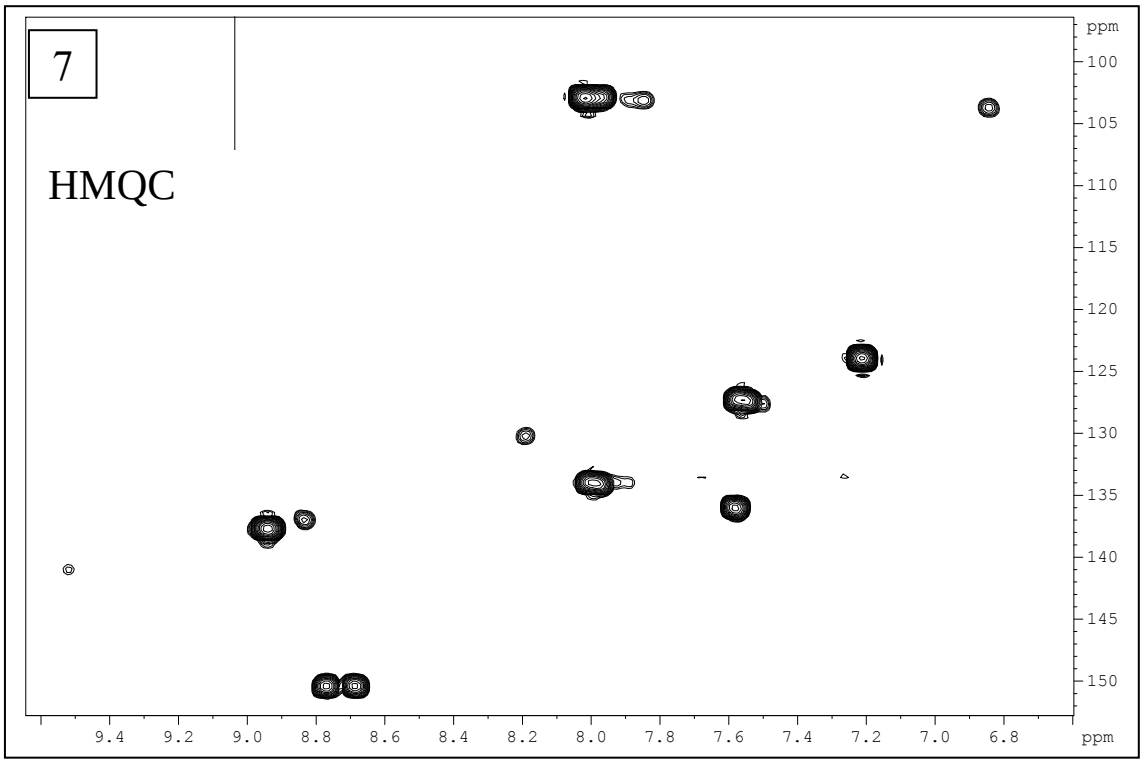


Figure S12. HMQC spectrum of 7 in pyridine (enlarged)

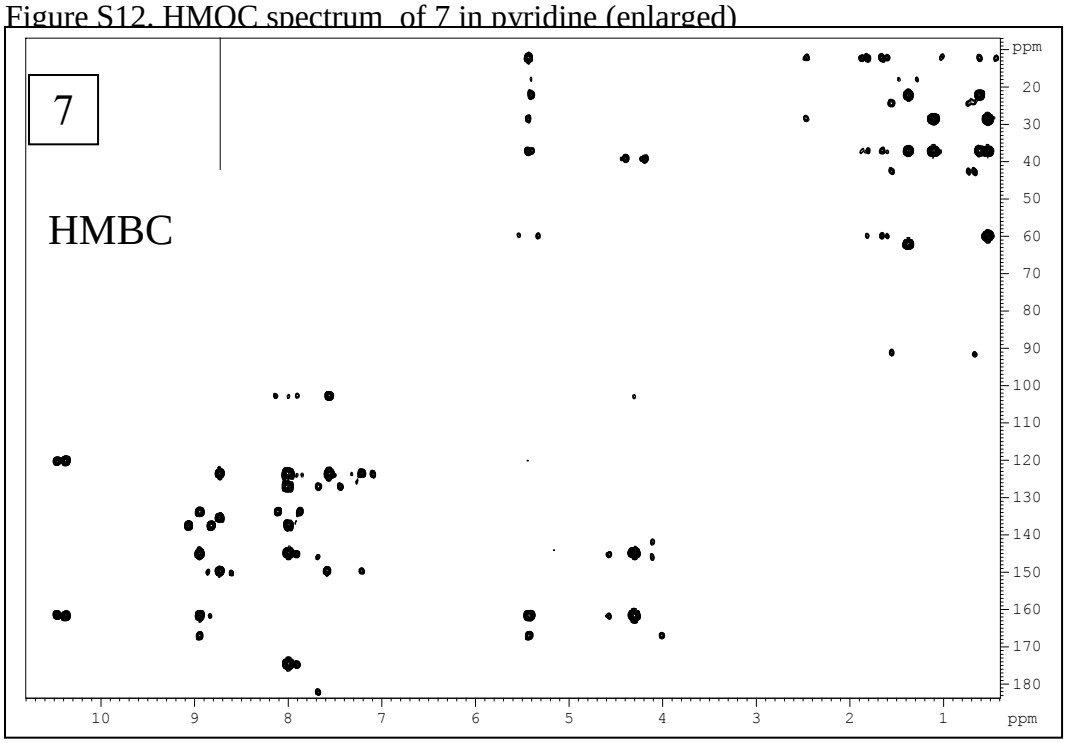


Figure S13. HMBC spectrum of 7 in pyridine

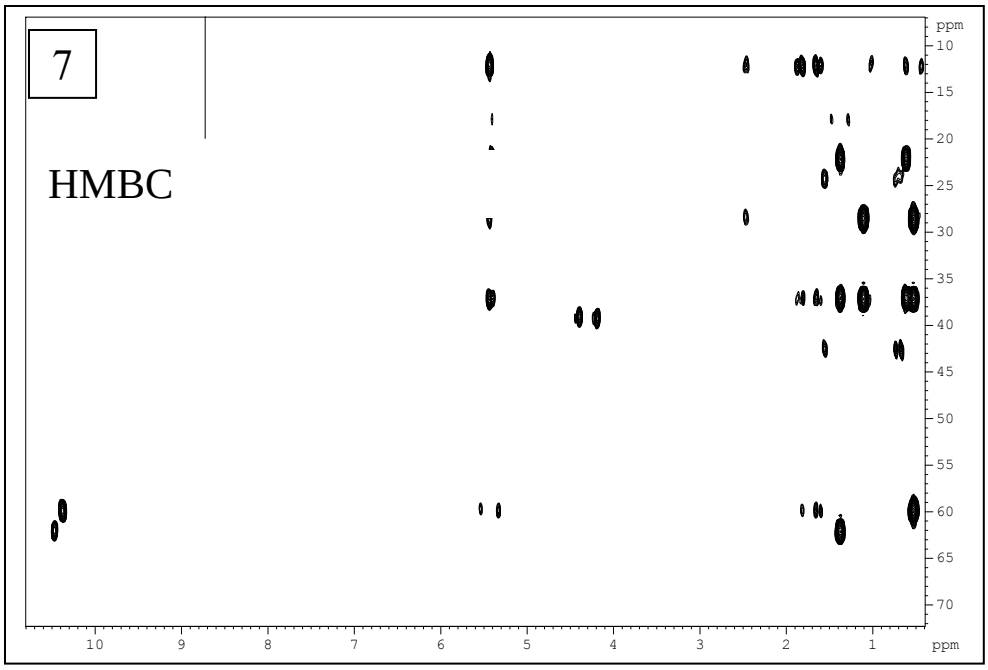


Figure S14. HMBC spectrum of 7 in pyridine (enlarged)

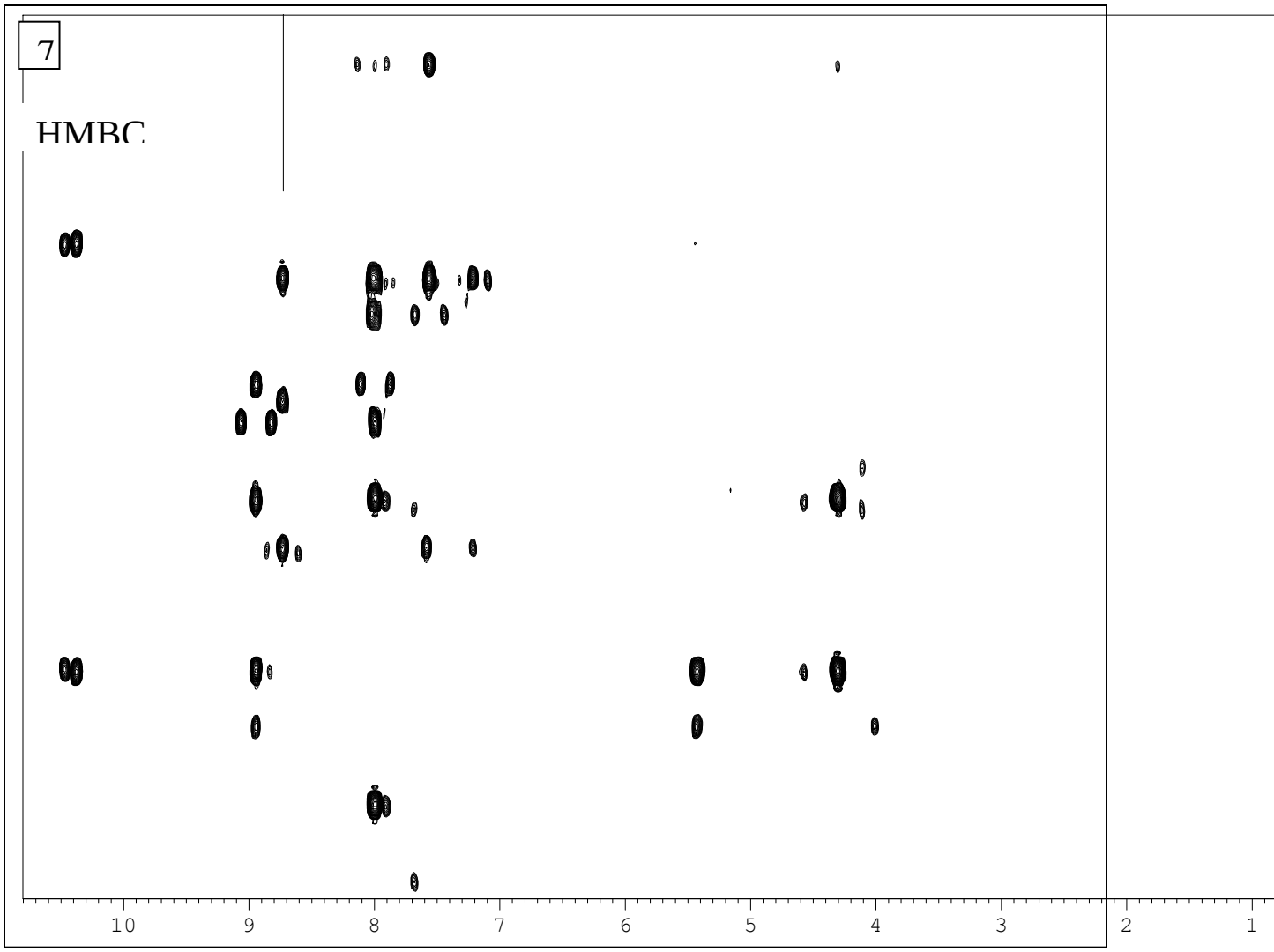


Figure S15. HMBC spectrum of 7 in pyridine (enlarged)

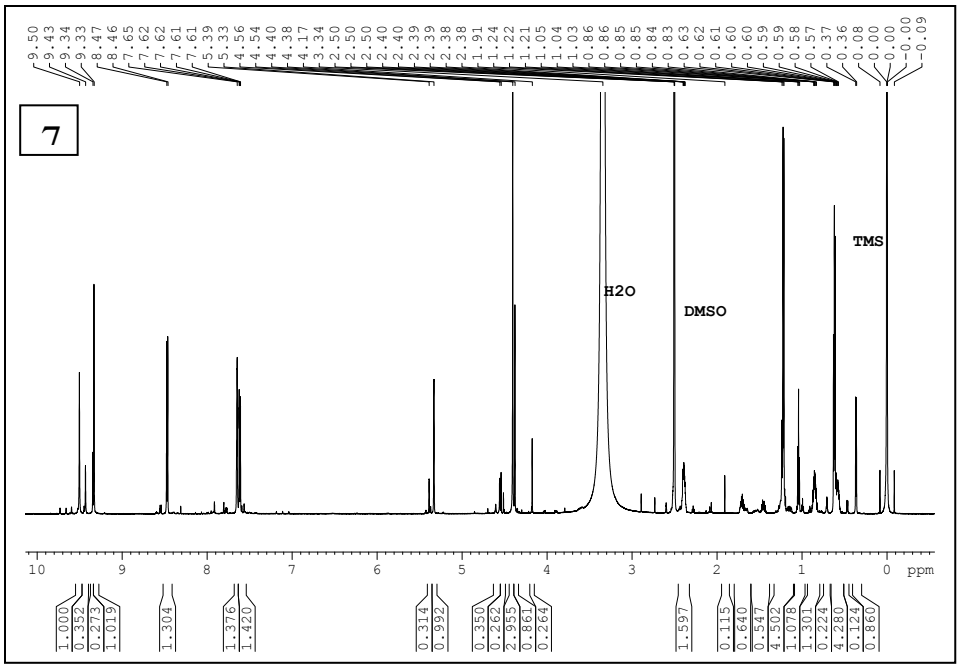


Figure S16. H-NMR of 7 in DMSO

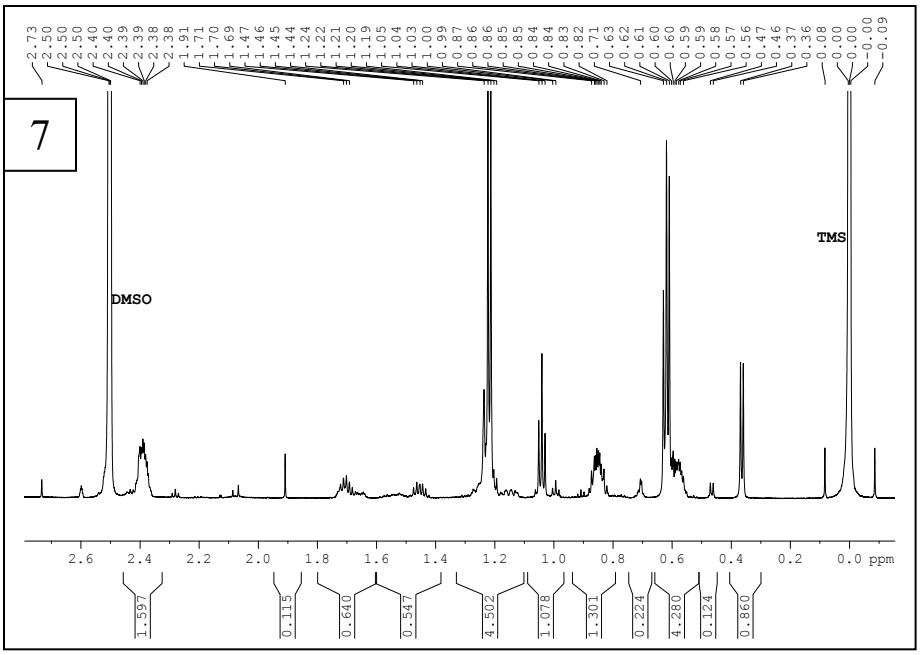


Figure S17. H-NMR of 7 in DMSO (enlarged)

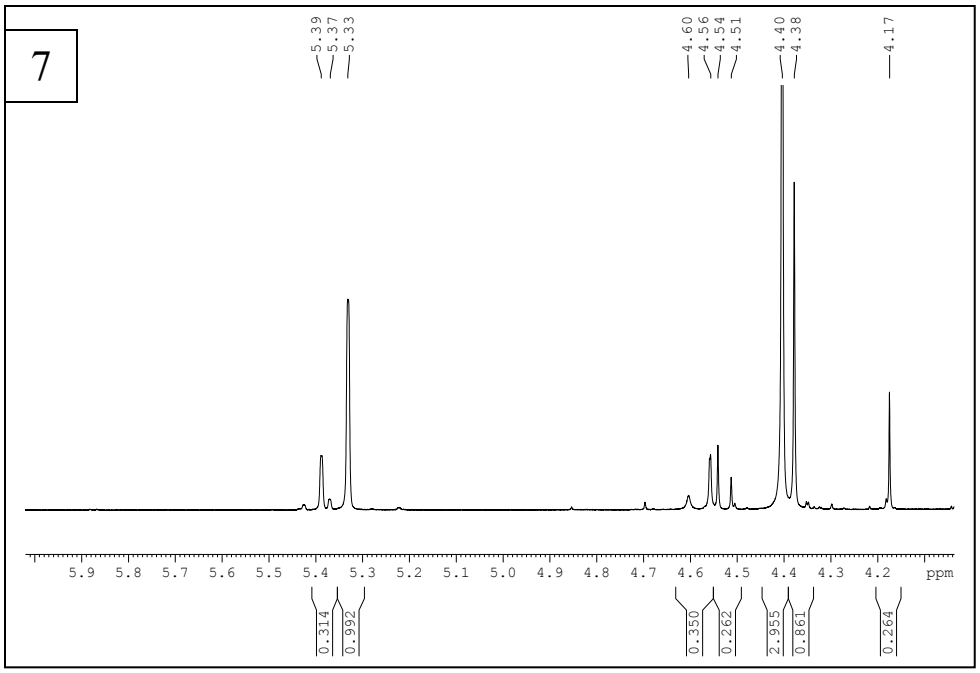


Figure S18. H-NMR of 7 in DMSO (enlarged)

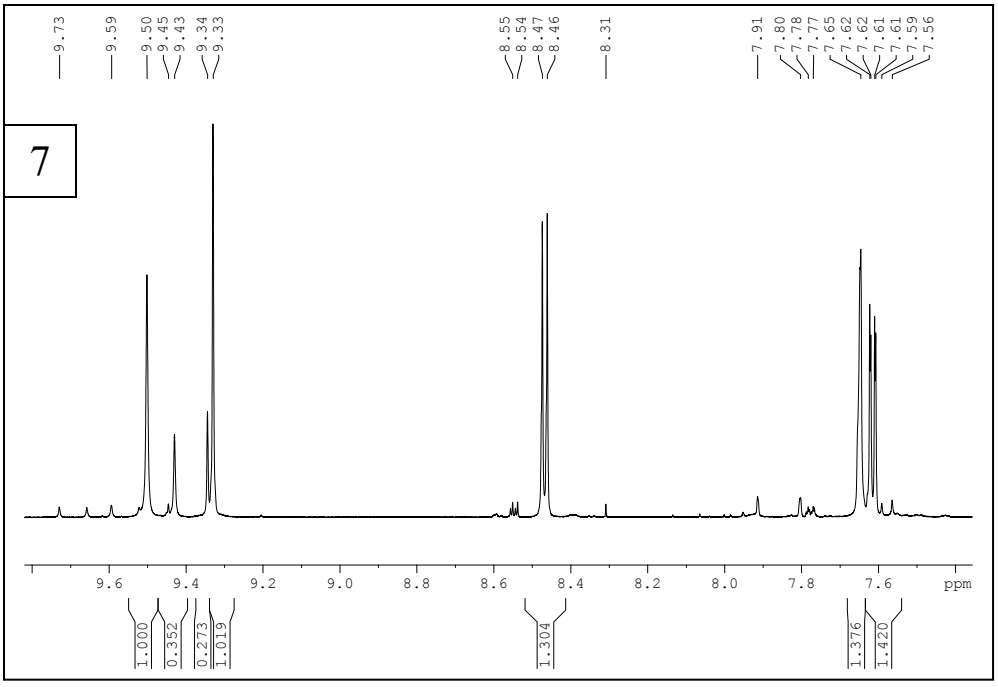


Figure S19. H-NMR of 7 in DMSO (enlarged)

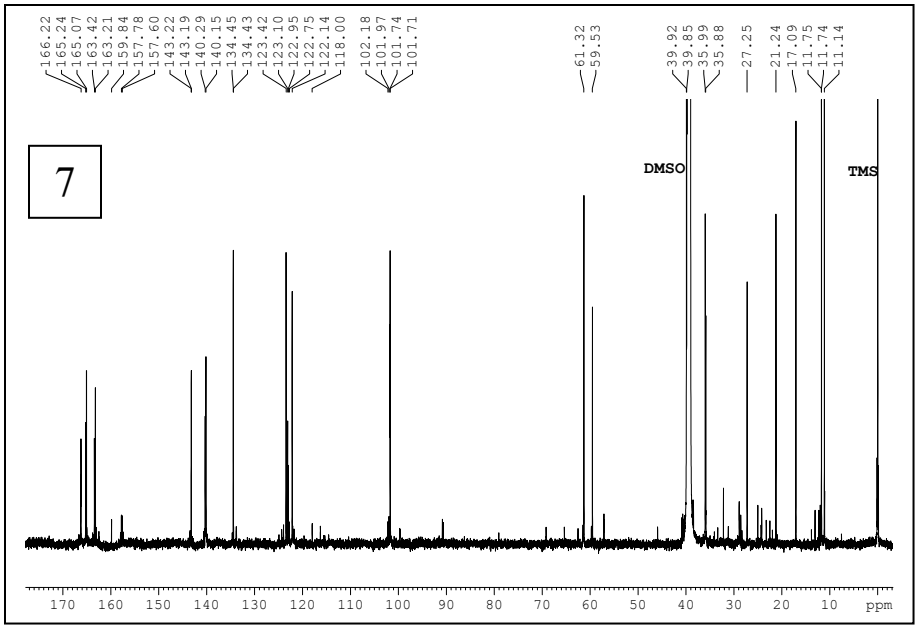
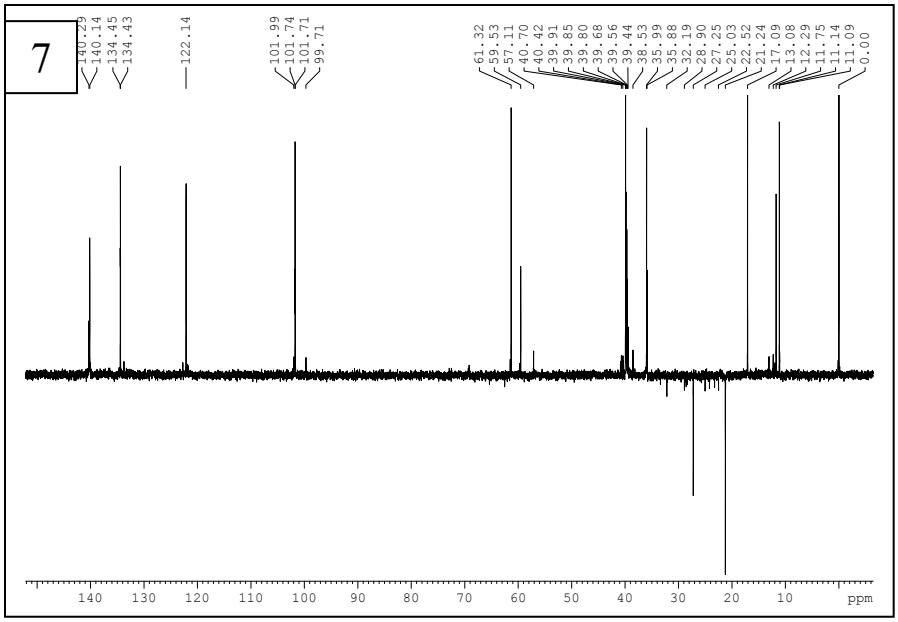


Figure S20. C-NMR of 7 in DMSO



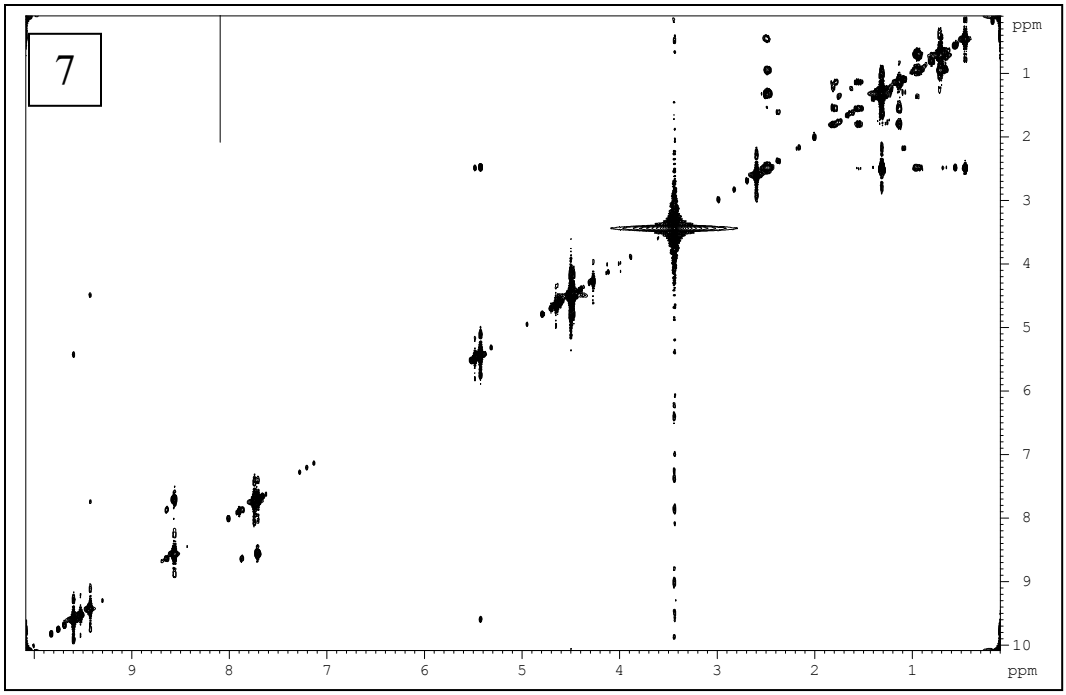


Figure S22. COSY spectrum of 7 in DMSO

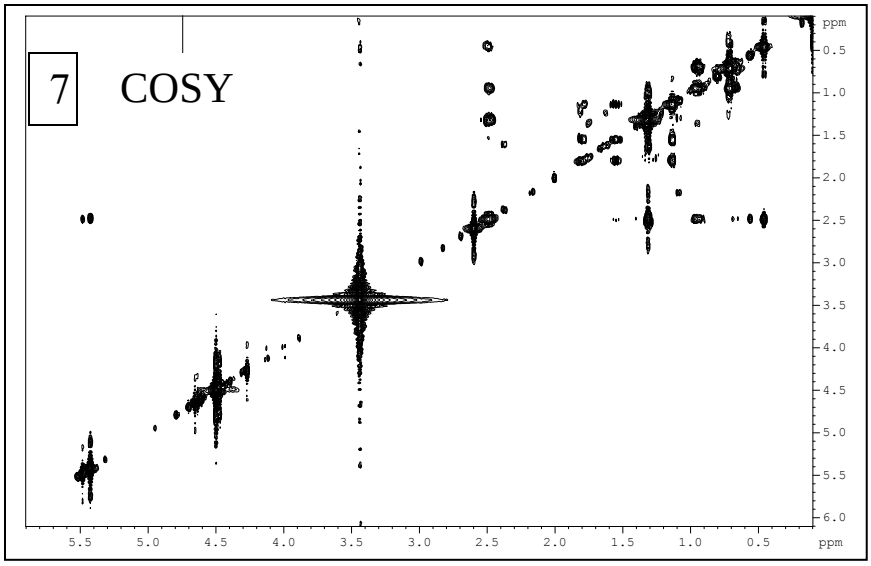


Figure S23. COSY spectrum of 7 in DMSO (enlarged)

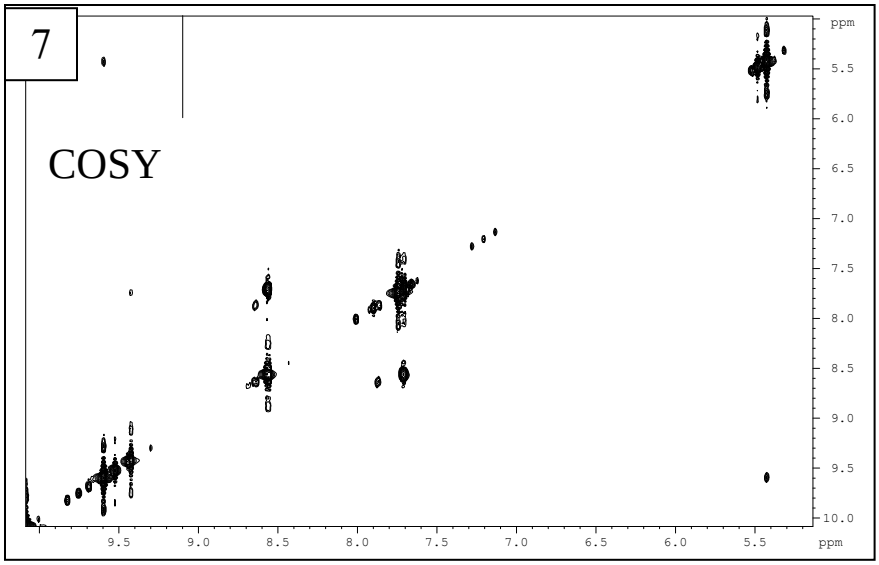


Figure S24. COSY spectrum of 7 in DMSO (enlarged)

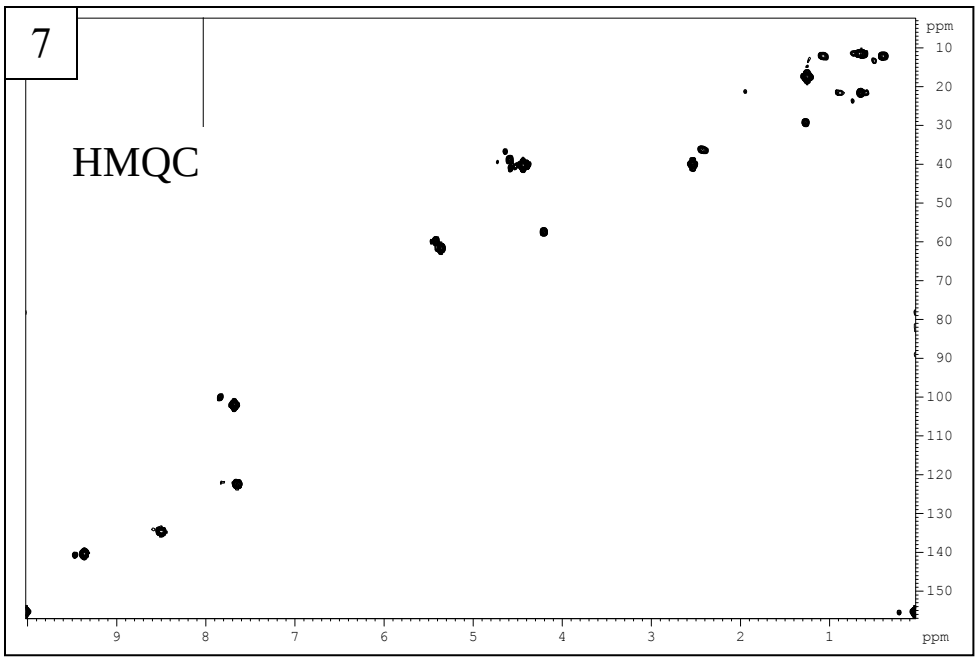


Figure S25. HMQC spectrum of 7 in DMSO (enlarged)

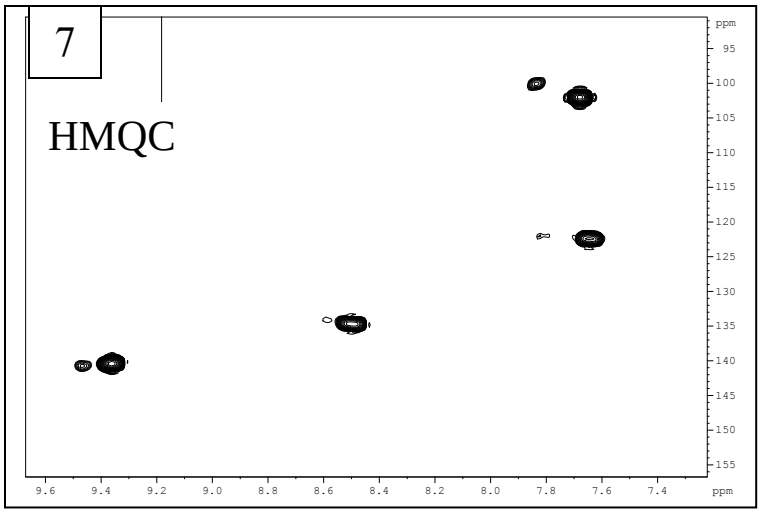


Figure S26. HMQC spectrum of 7 in DMSO (enlarged)

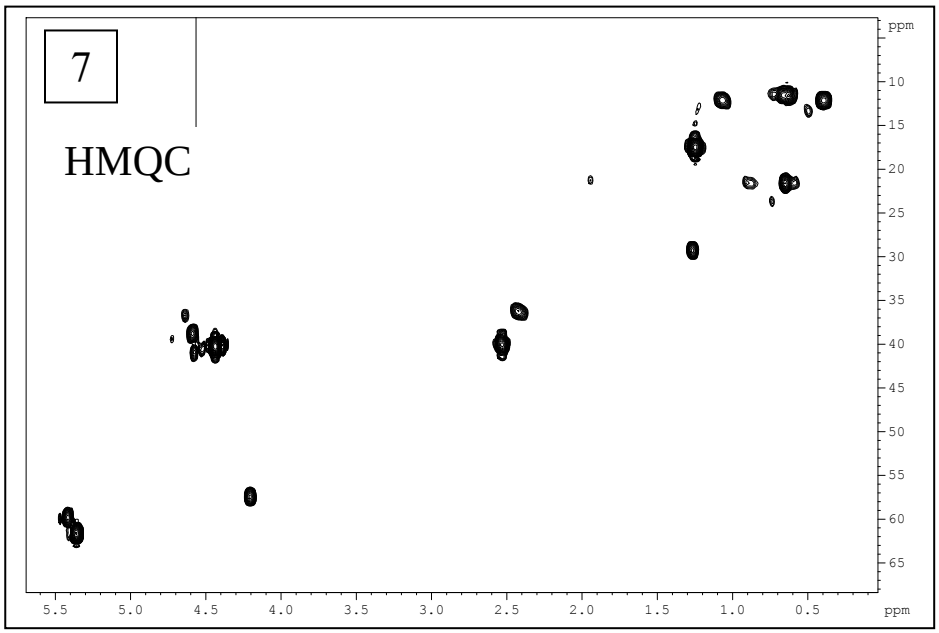


Figure S27. HMQC spectrum of 7 in DMSO (enlarged)

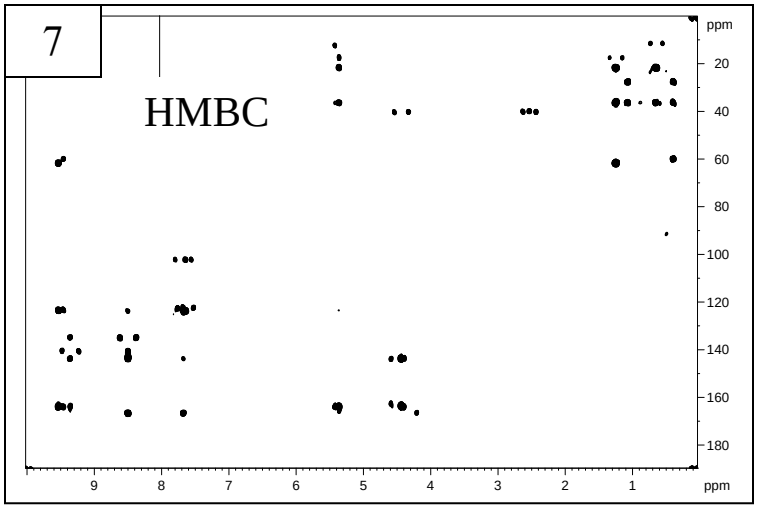


Figure S28. HMBC spectrum of 7 in DMSO

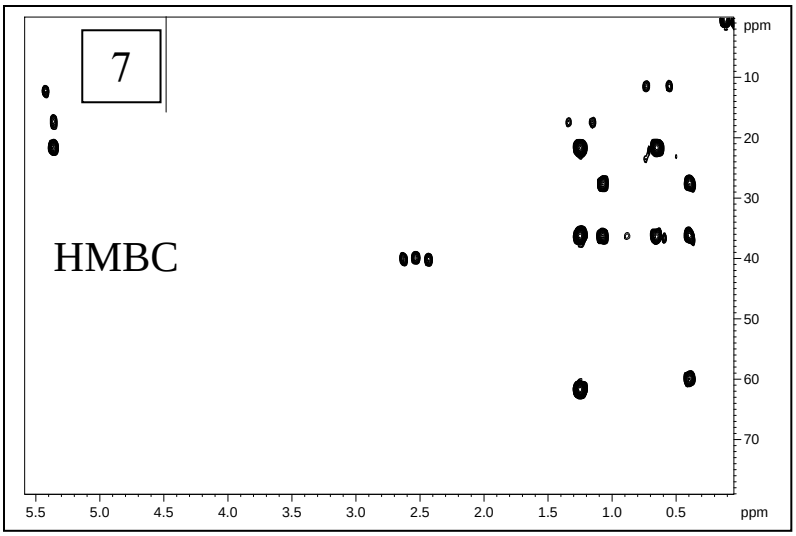


Figure S29. HMBC spectrum of 7 in DMSO (enlarged)

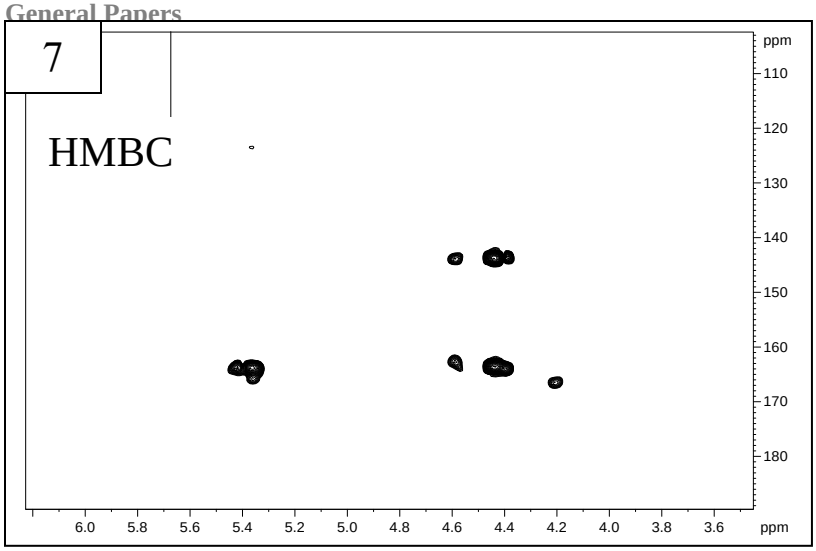


Figure S30. HMBC spectrum of 7 in DMSO (enlarged)

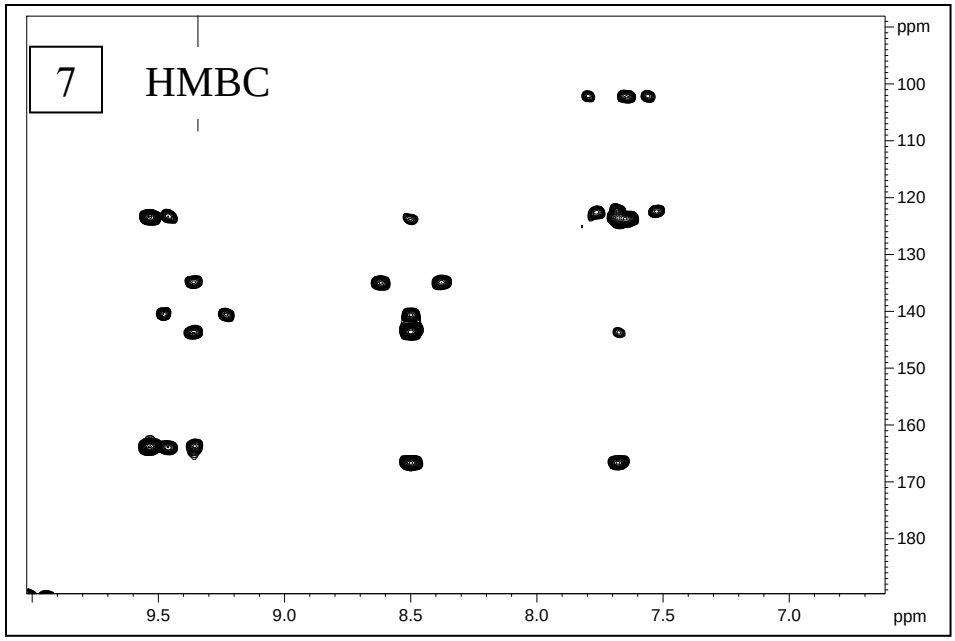


Figure S31. HMBC spectrum of 7 in DMSO (enlarged)

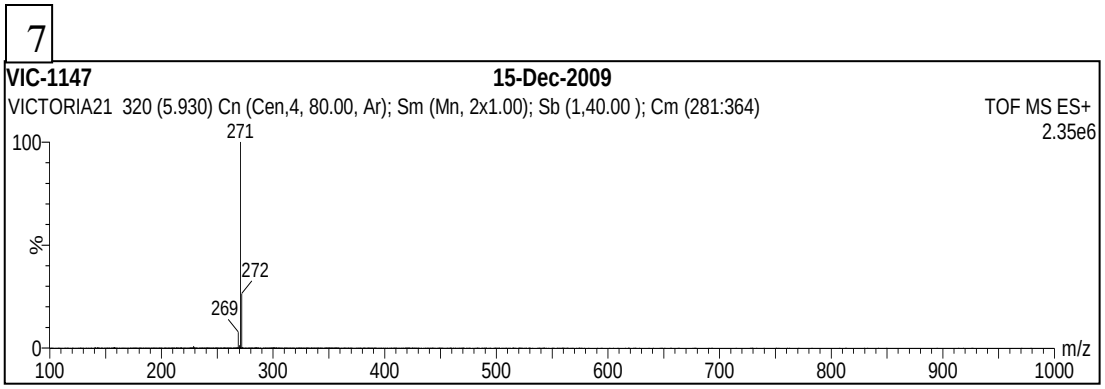


Figure S32. QTOF-MS of 7

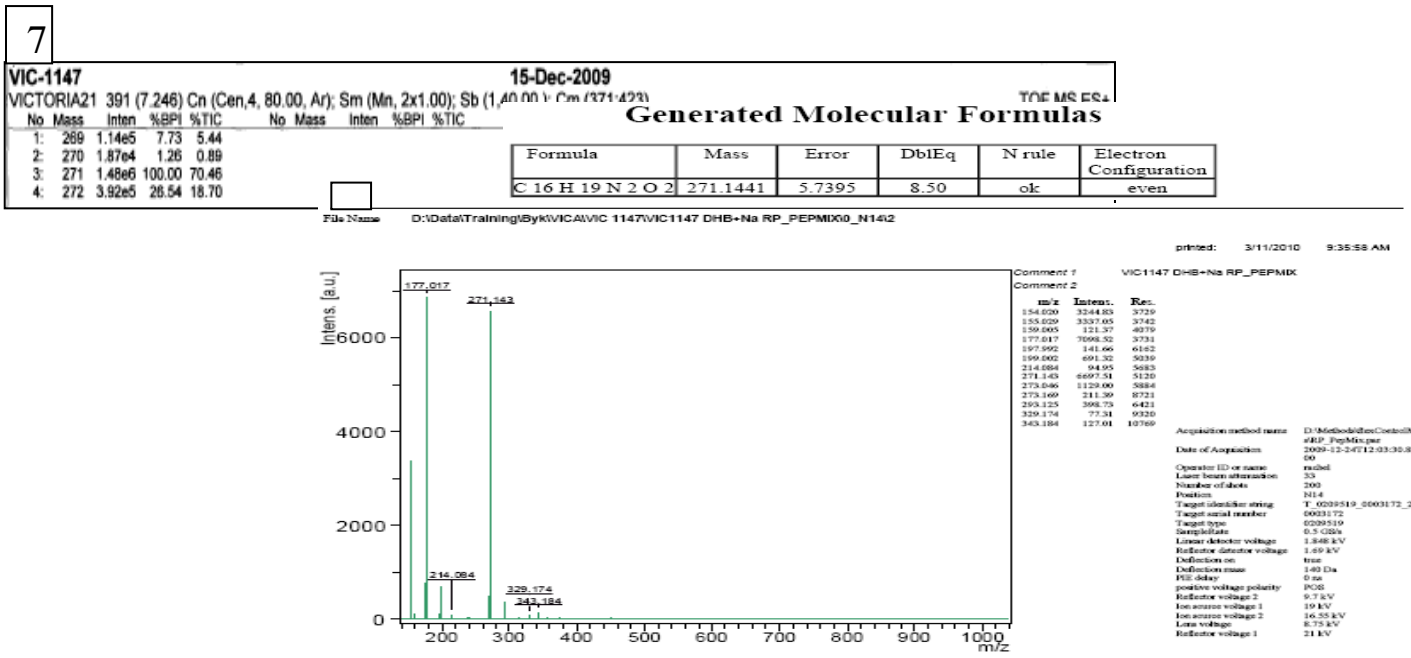
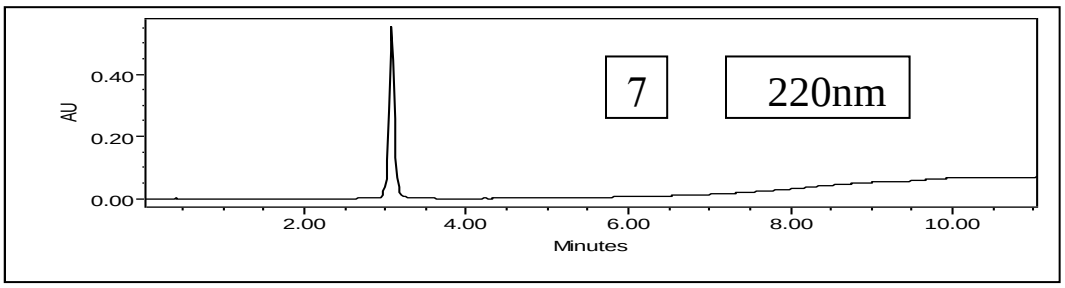


Figure S33. HR-MALDI-TOF MS of compound 7



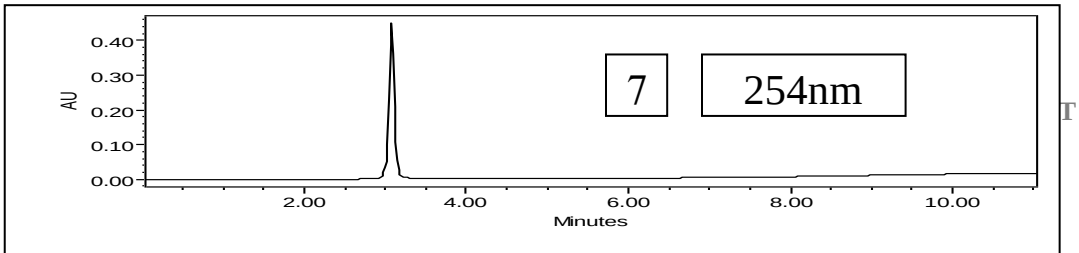


Figure S34. HPLC of compound 7

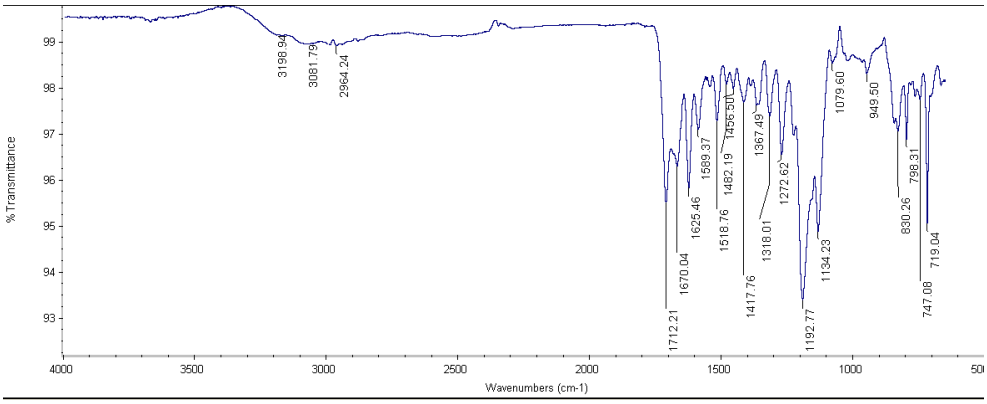


Figure S35. IR spectrum of compound 7

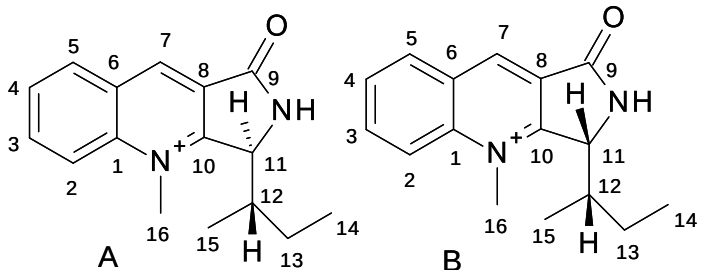
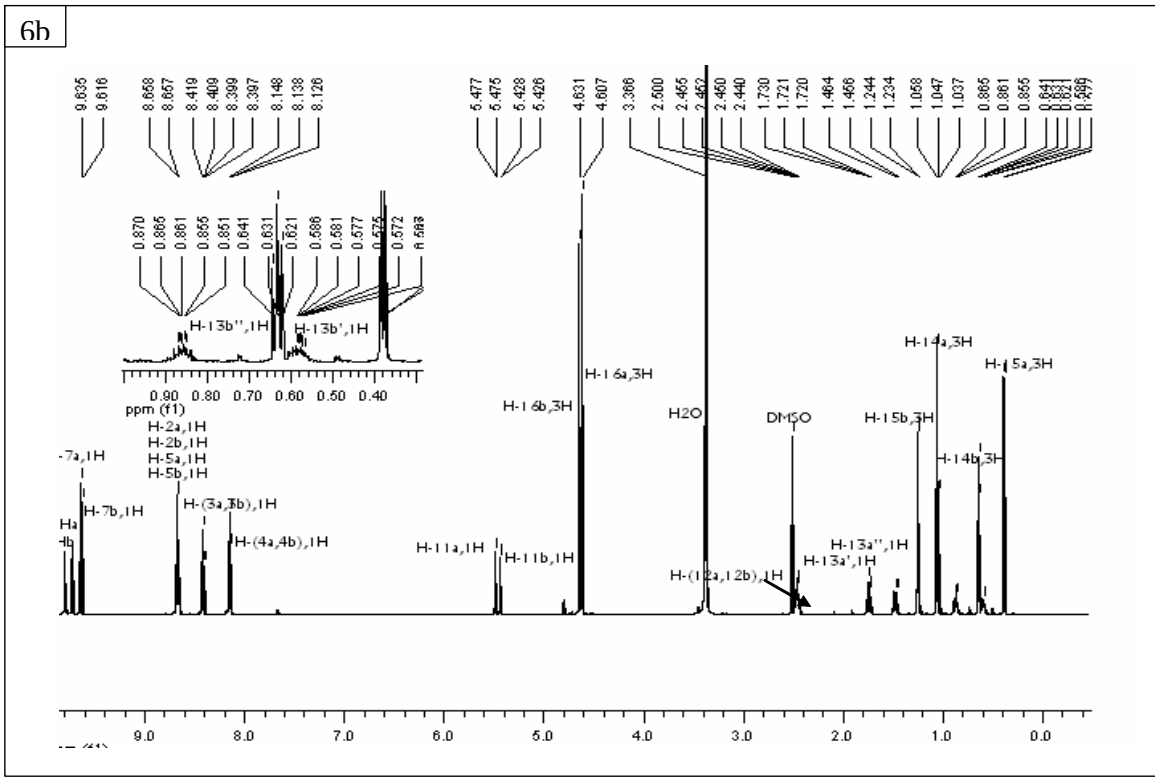


Figure S36. Arbitrary atom numbering of compound 6b for NMR assignments.



Fi S27. H NMR of and Cl in DMSO

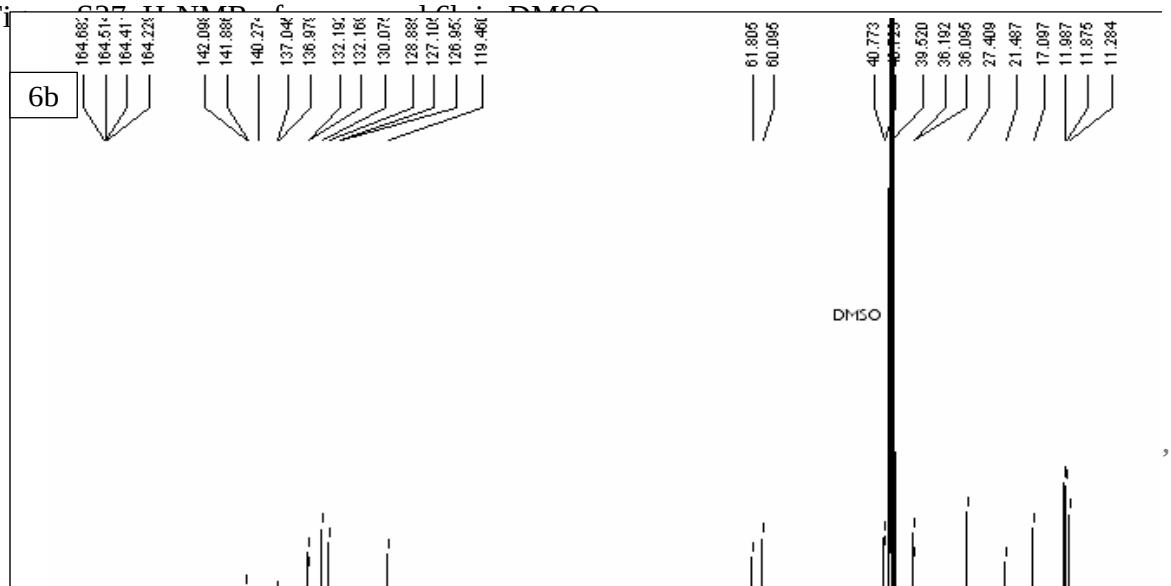
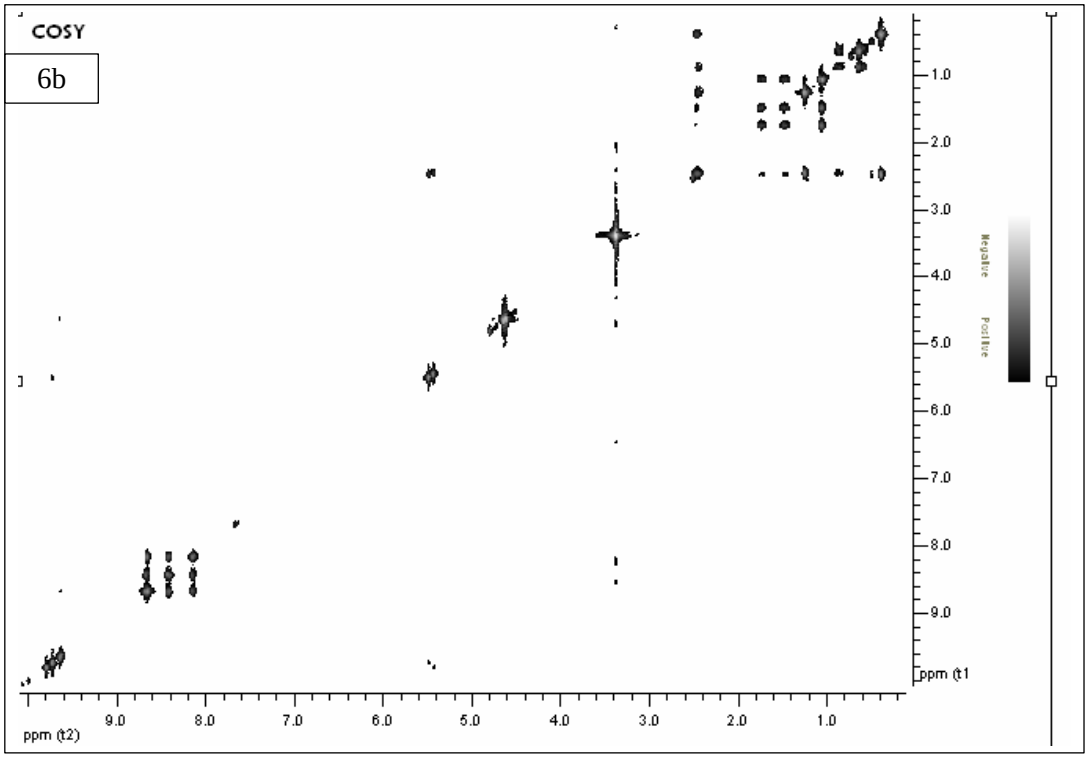


Figure S38. C-NMR of compound 6b in DMSO



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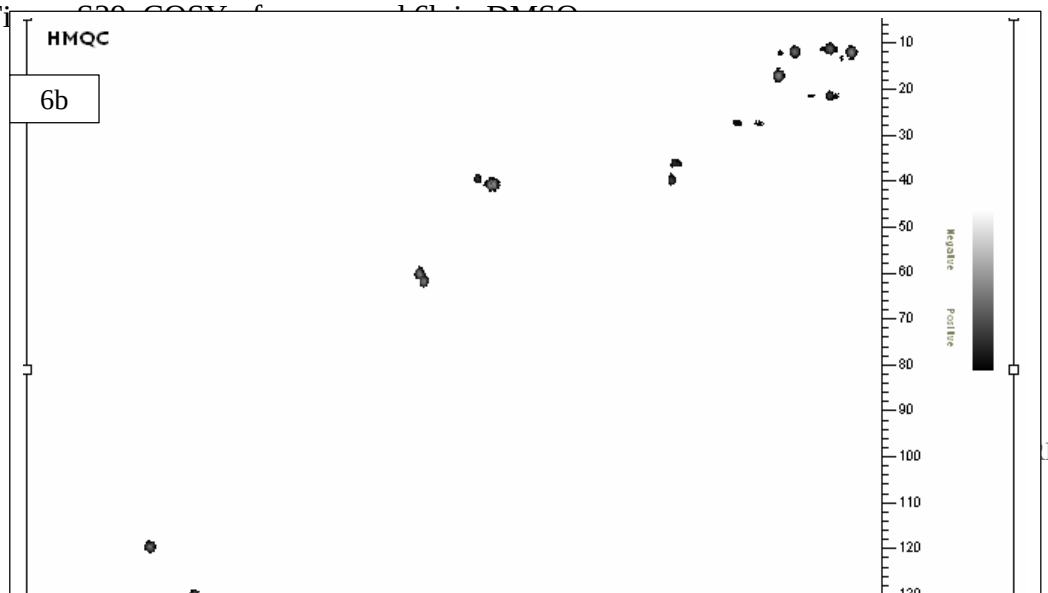


Figure S40. HMQC of compound 6b in DMSO

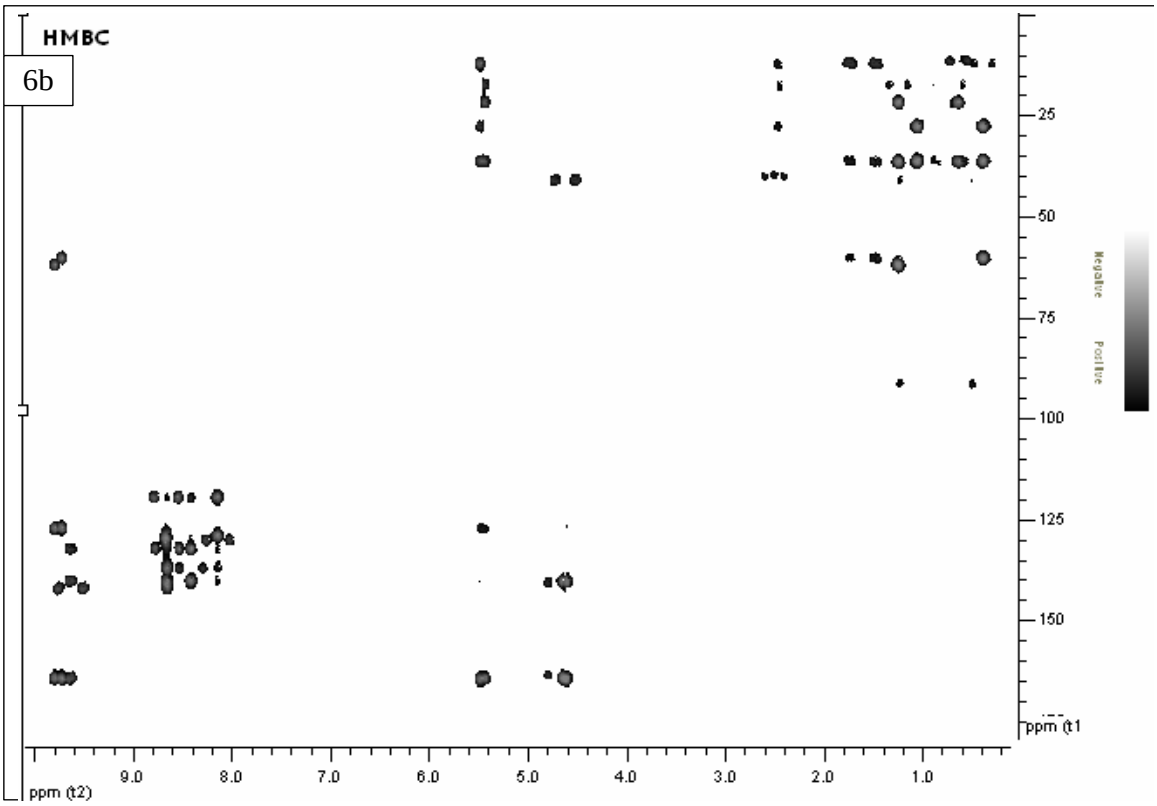
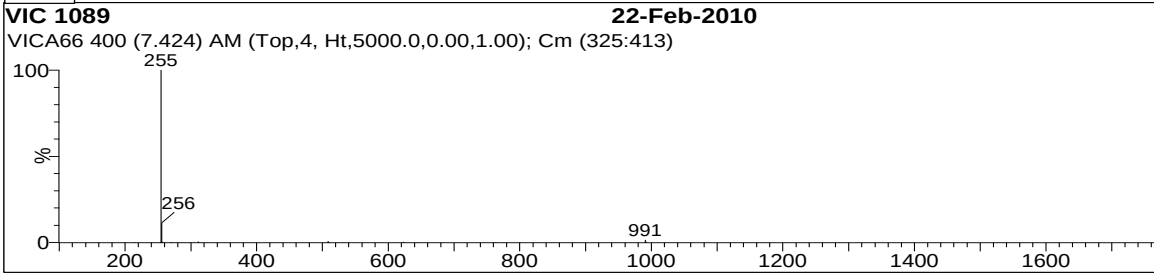


Figure S41. HMBC of compound 6b in DMSO

6b



6b

VIC 1089 22-Feb-2010

VICA66 400 (7.424) AM (Top,4, Ht,5000.0,0.00,1.00); Cm (325:414)

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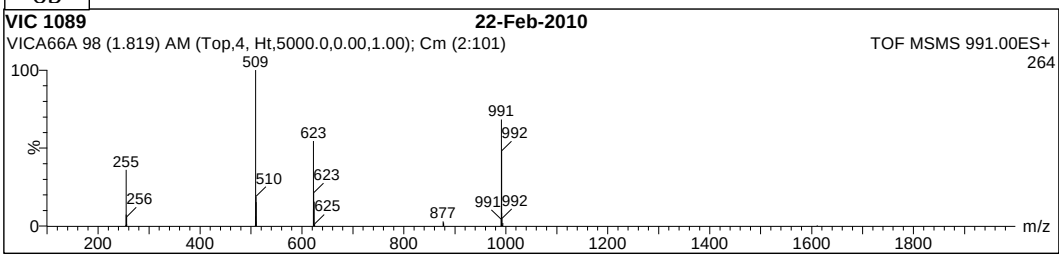
2:	255	1.98e2	0.20	0.17															
3:	255	2.76e2	0.28	0.24															
4:	255	9.84e4	100.00	85.32															
5:	255	1.74e2	0.18	0.15															
6:	256	1.11e4	11.28	9.63															
7:	257	2.09e2	0.21	0.18															
8:	311	5.10e2	0.52	0.44															
9:	509	7.45e2	0.76	0.65															

Figure S42. QTOF-MS of compound 6b

6b



6b

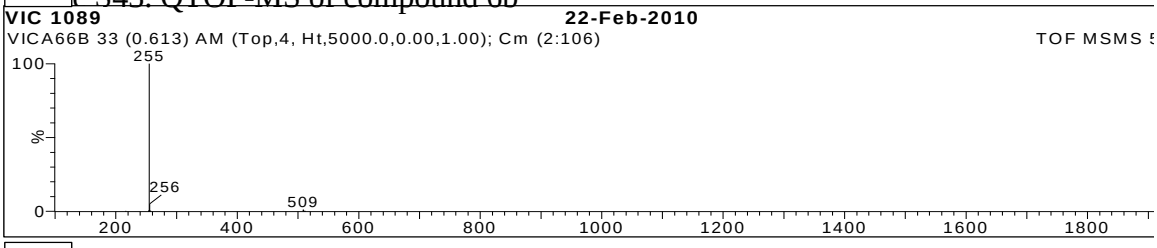
VIC 1089 22-Feb-2010

VICA66A 98 (1.819) AM (Top,4, Ht,5000.0,0.00,1.00); Cm (2:101) TOF MSMS 991.00ES+

No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Inten
1:	255	2.00e0	0.76	0.17																		
2:	255	9.50e1	35.98	7.84																		
3:	255	1.90e1	7.20	1.57																		
4:	256	1.50e1	5.68	1.24																		
5:	256	3.00e0	1.14	0.25																		
6:	509	2.00e0	0.76	0.17																		
7:	509	1.60e1	6.06	1.32																		
8:	509	2.64e2	100.00	21.80																		
9:	509	7.00e0	2.65	0.58																		
10:	510	8.00e0	3.03	0.66																		
11:	510	4.00e1	15.15	3.30																		
12:	510	5.00e1	18.94	4.13																		
13:	623	2.80e1	10.61	2.31																		
14:	623	1.44e2	54.55	11.89																		
15:	623	5.60e1	21.21	4.62																		
16:	623	7.00e0	2.65	0.58																		
17:	624	4.10e1	15.53	3.39																		
18:	625	3.00e0	1.14	0.25																		
19:	877	8.00e0	3.03	0.66																		
20:	878	3.00e0	1.14	0.25																		
21:	878	6.00e0	2.27	0.50																		
22:	991	1.10e1	4.17	0.91																		
23:	991	1.80e2	68.18	14.86																		
24:	991	3.20e1	12.12	2.64																		
25:	991	2.00e0	0.76	0.17																		
26:	992	2.20e1	8.33	1.82																		
27:	992	1.27e2	48.11	10.49																		
28:	992	1.20e1	4.55	0.99																		
29:	993	2.00e0	0.76	0.17																		
30:	994	5.00e0	1.89	0.41																		
31:	1611	1.00e0	0.38	0.08																		

6b

Figure S43. QTOF-MS of compound 6b



6b

VIC 1089 22-Feb-2010

VICA66B 33 (0.613) AM (Top,4, Ht,5000.0,0.00,1.00); Cm (2:106) TOF MSM

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1:	215	1.00e0	0.02	0.02																
2:	254	3.00e0	0.06	0.06																
3:	254	2.00e0	0.04	0.04																
4:	254	2.00e0	0.04	0.04																
5:	254	4.00e0	0.08	0.07																
6:	254	3.00e0	0.06	0.06																
7:	254	8.00e0	0.16	0.15																
8:	254	4.00e0	0.08	0.07																
9:	255	1.20e1	0.24	0.22																
10:	255	4.90e3	100.00	91.07																
11:	255	3.60e1	0.73	0.67																
12:	255	6.00e0	0.12	0.11																
13:	255	3.00e0	0.06	0.06																
14:	256	2.00e0	0.04	0.04																
15:	256	2.40e2	4.90	4.46																
16:	257	5.00e0	0.10	0.09																
17:	257	1.70e1	0.35	0.32																
18:	509	5.10e1	1.04	0.95																
19:	509	5.20e1	1.06	0.97																

Figure S44. QTOF-MS of compound 6b

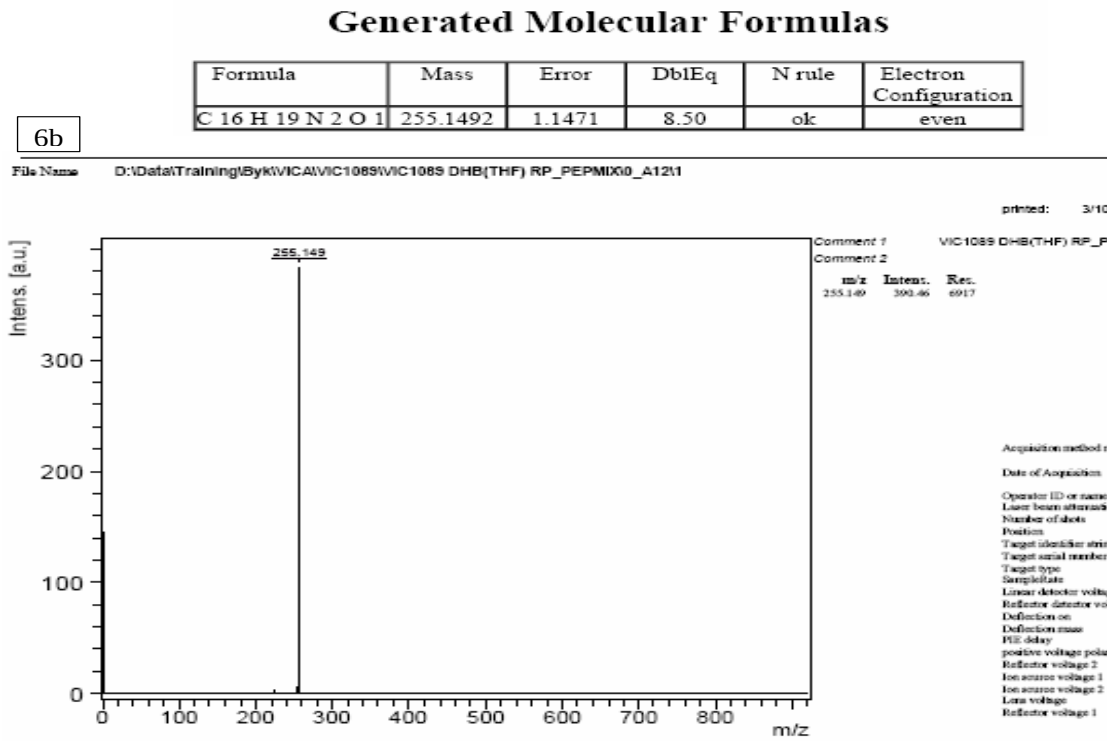


Figure S45. MALDI-TOF MS of compound 6b

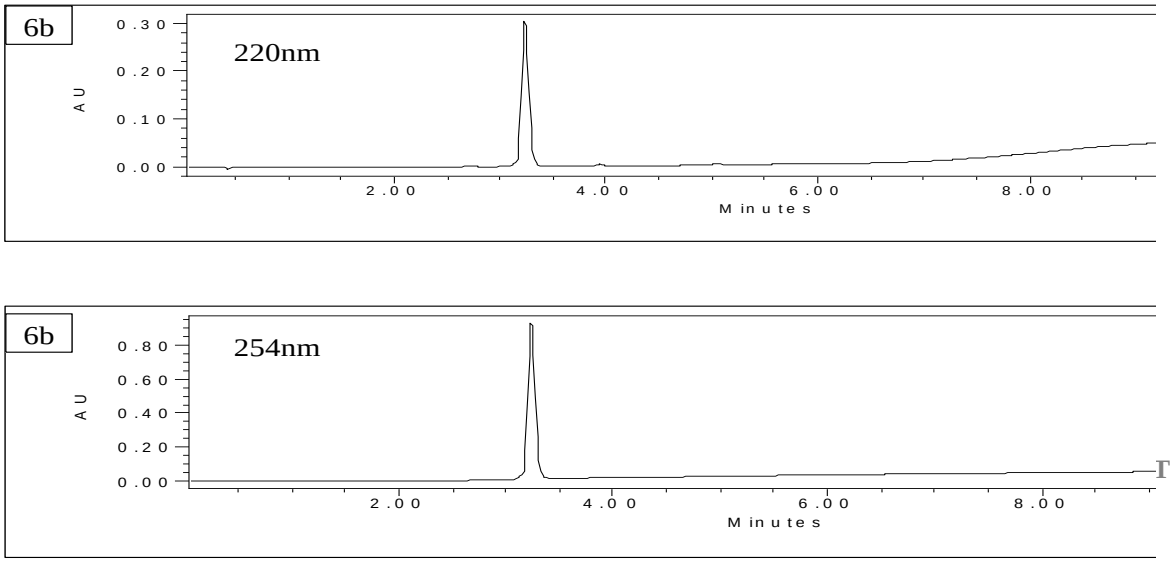


Figure S46. HPLC of compound 6b

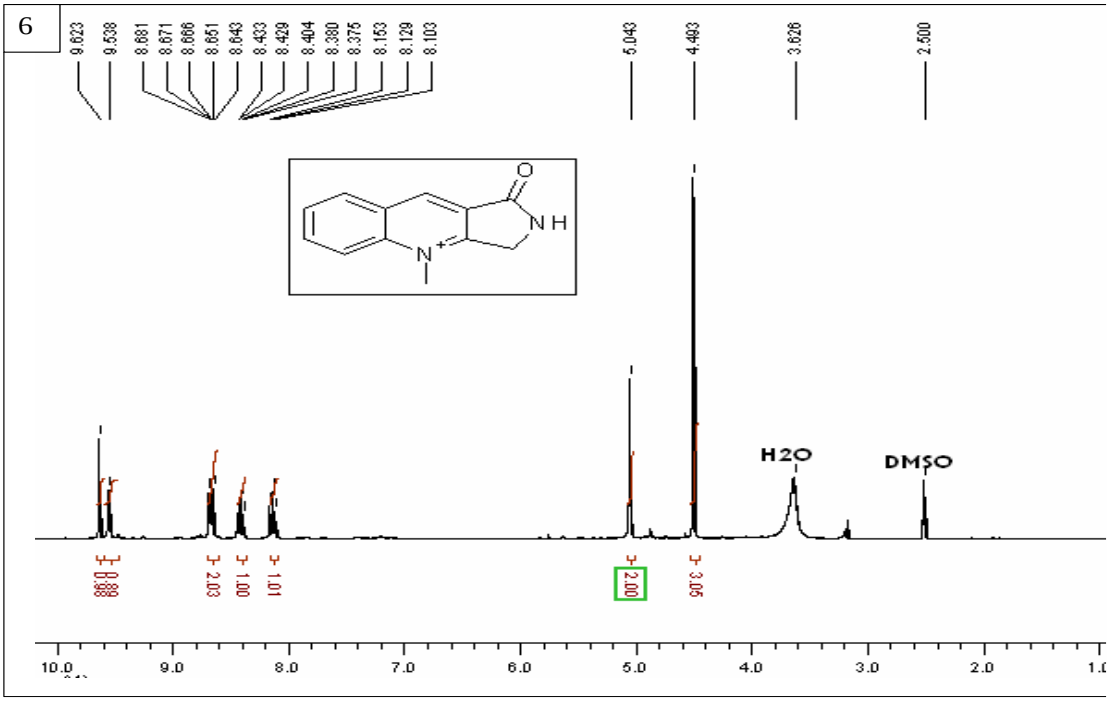


Figure S47. H-NMR of compound 6a in DMSO

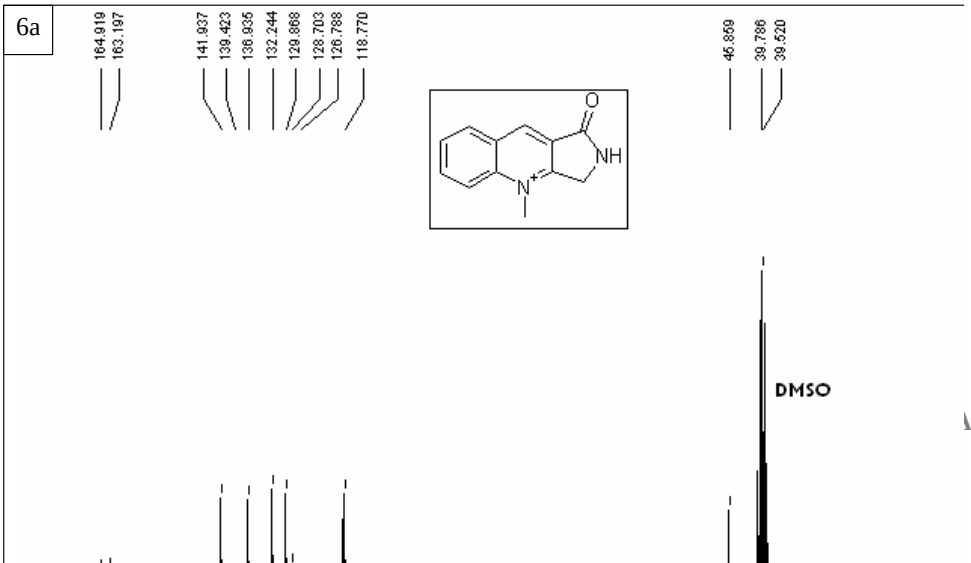
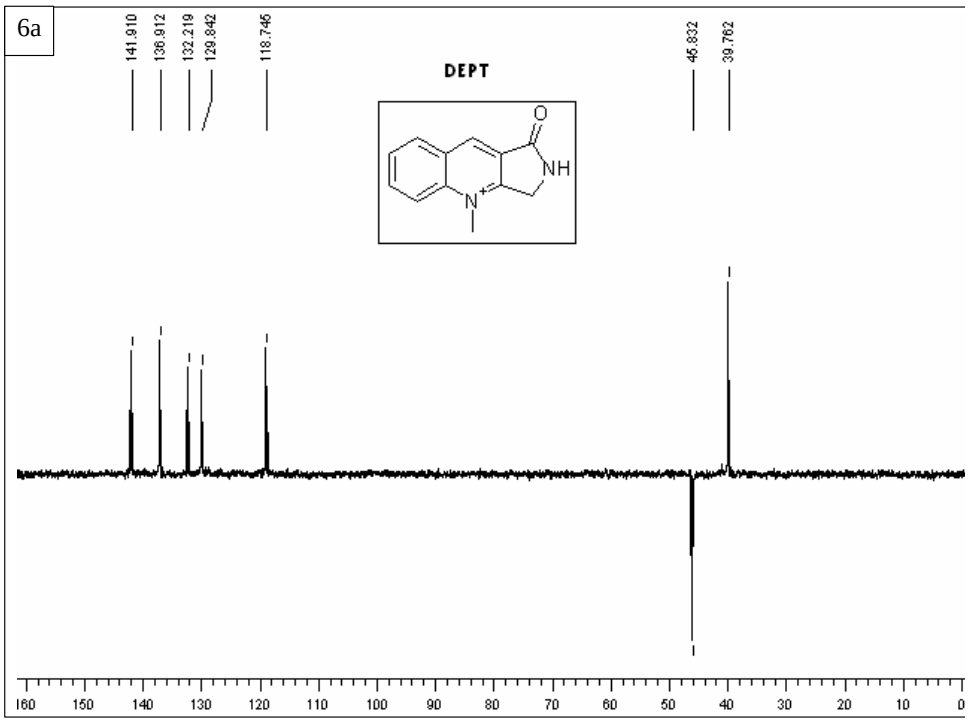
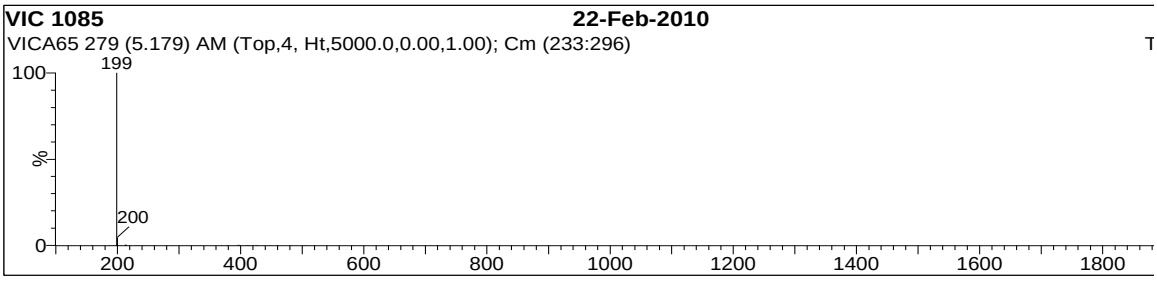


Figure S48. C-NMR of compound 6a in DMSO



6a

Figure S49. DEPT of compound 6a in DMSO



6a

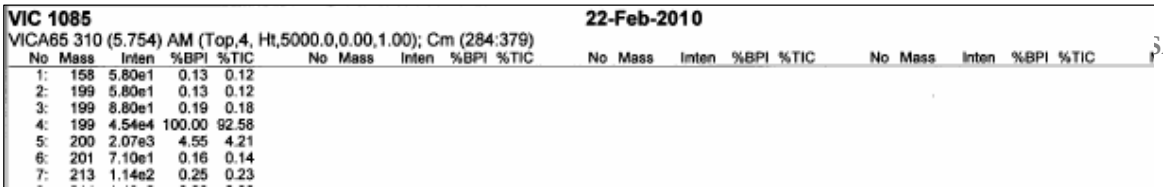


Figure S50. QTOF-MS of compound 6a

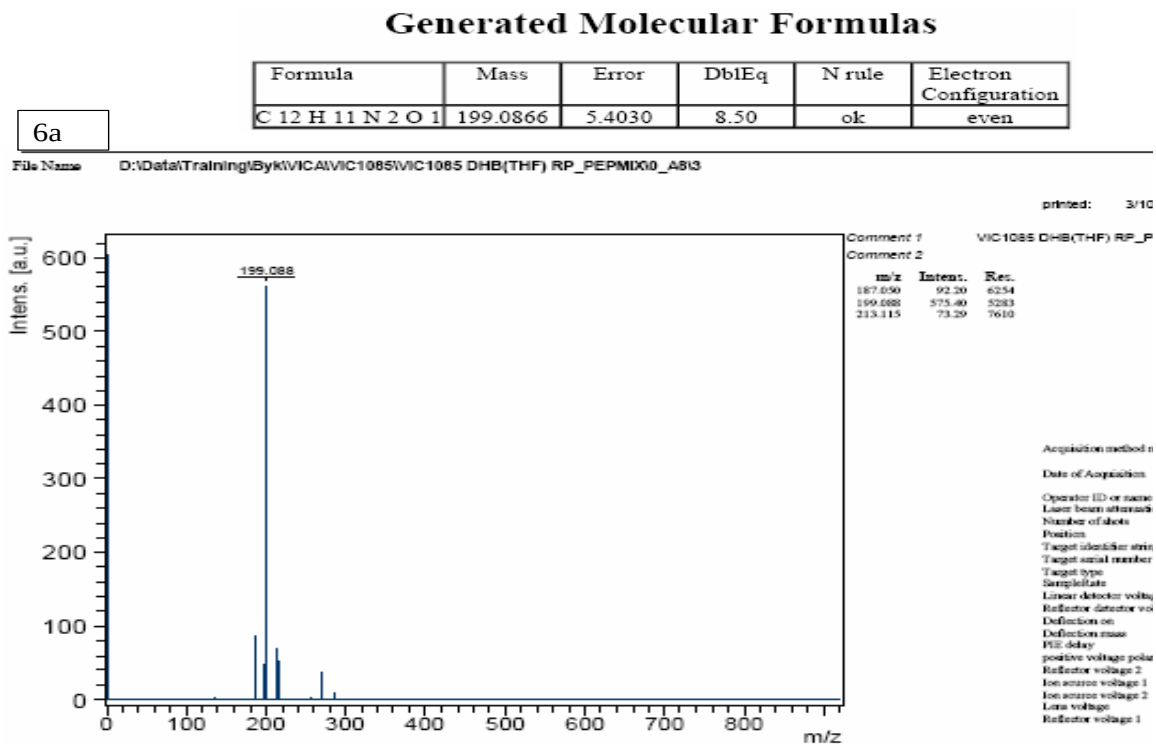


Figure S51. HR-MALDI-TOF MS of compound 6a

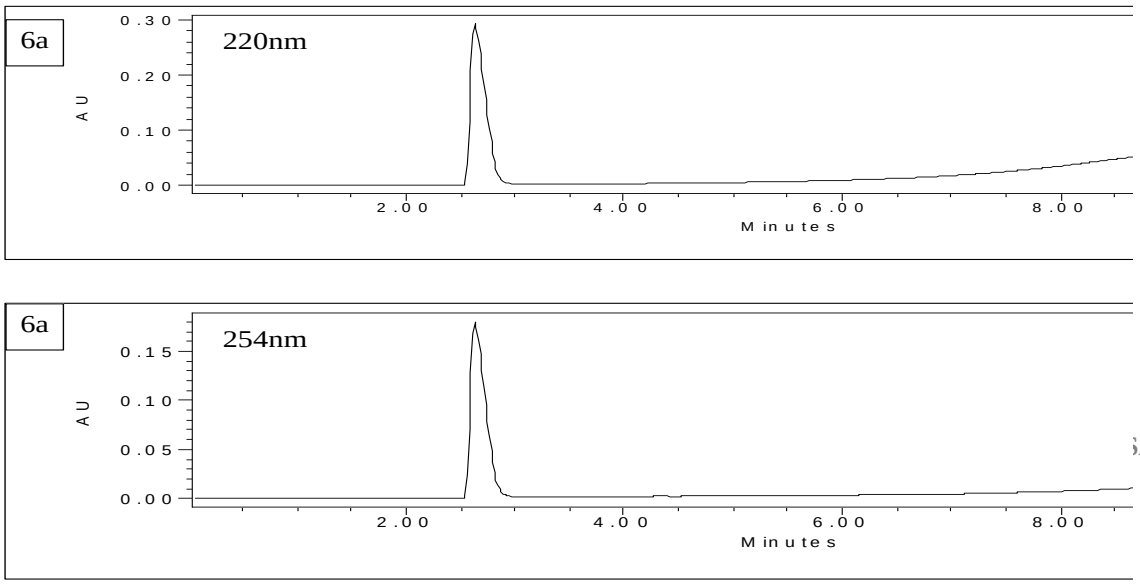


Figure S52. HPLC of compound 6a

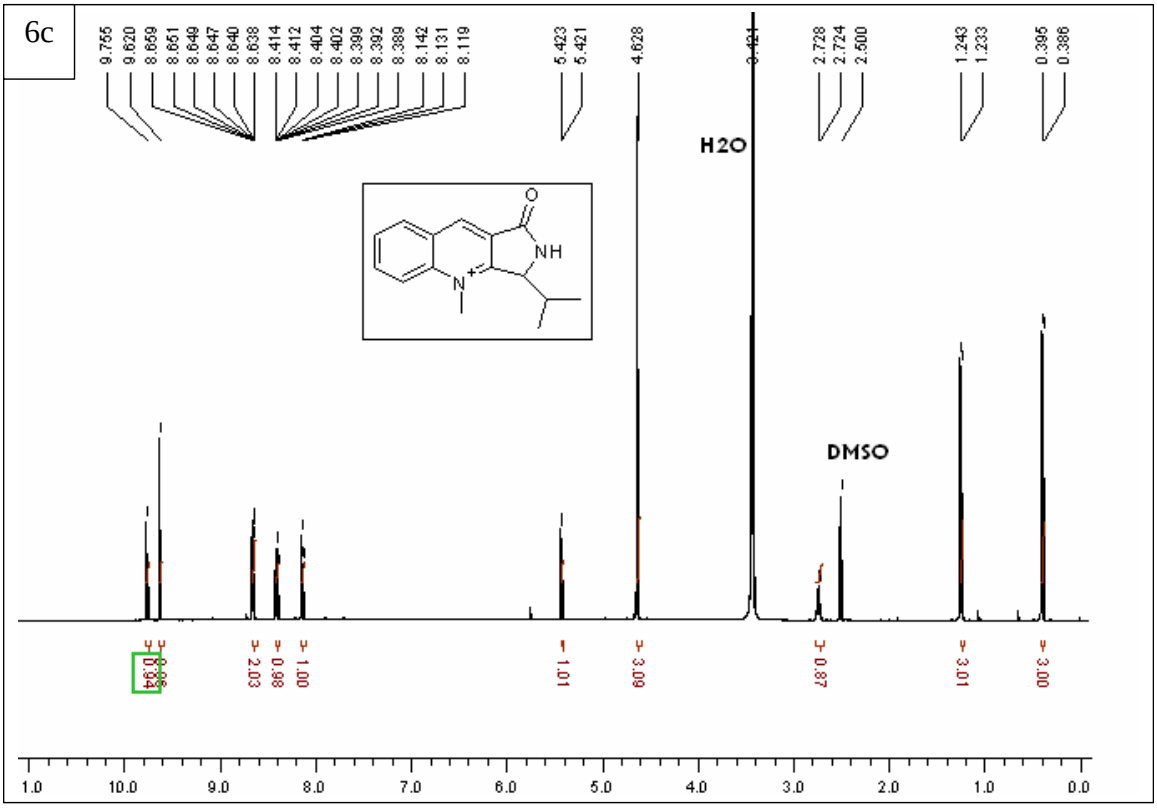


Figure S53. H-NMR of compound 6c in DMSO

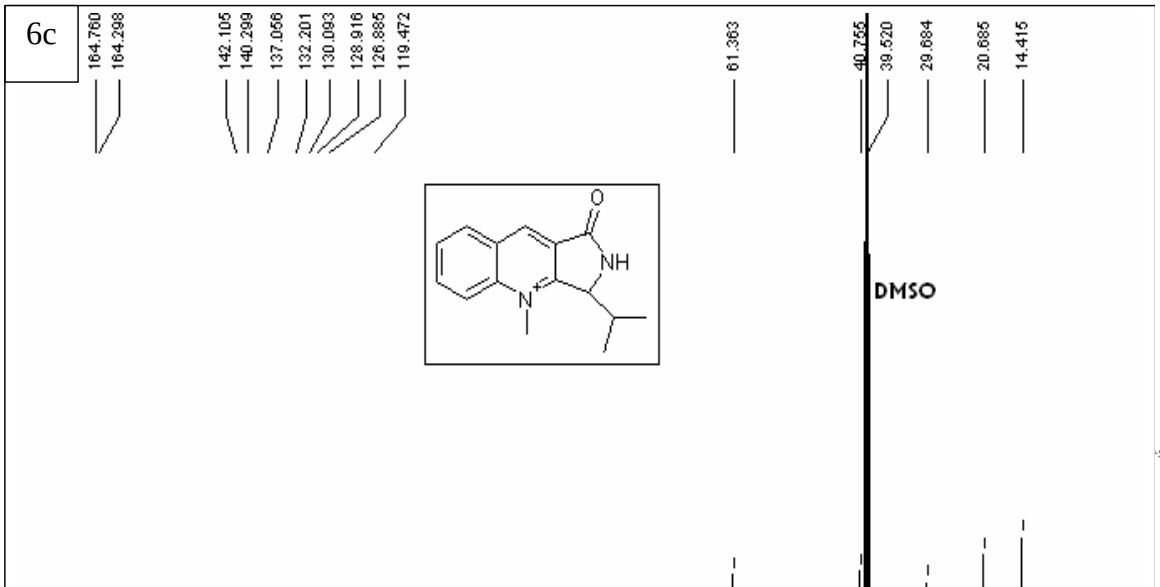
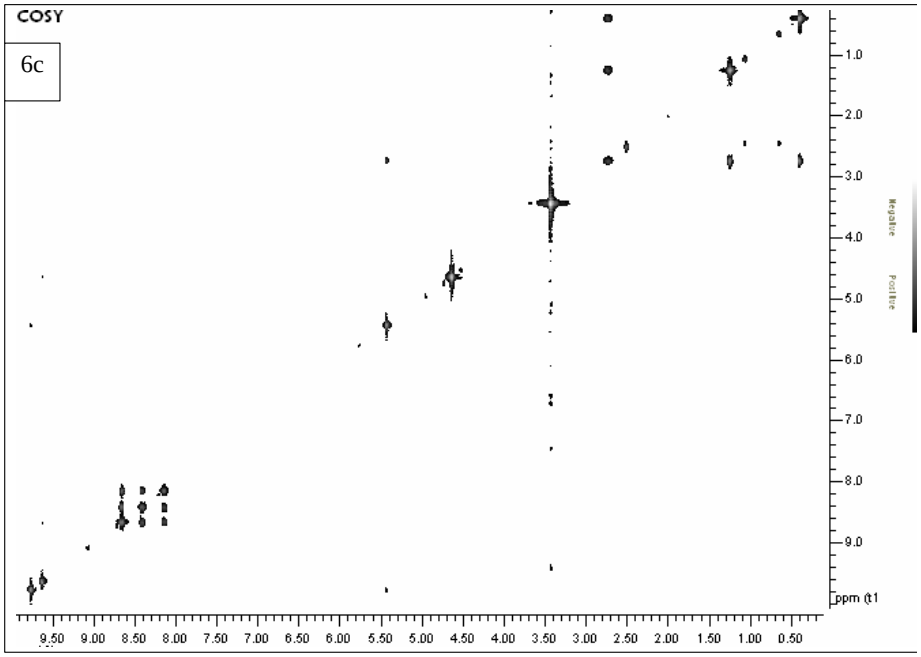


Figure S54. C-NMR of compound 6c in DMSO



Fi

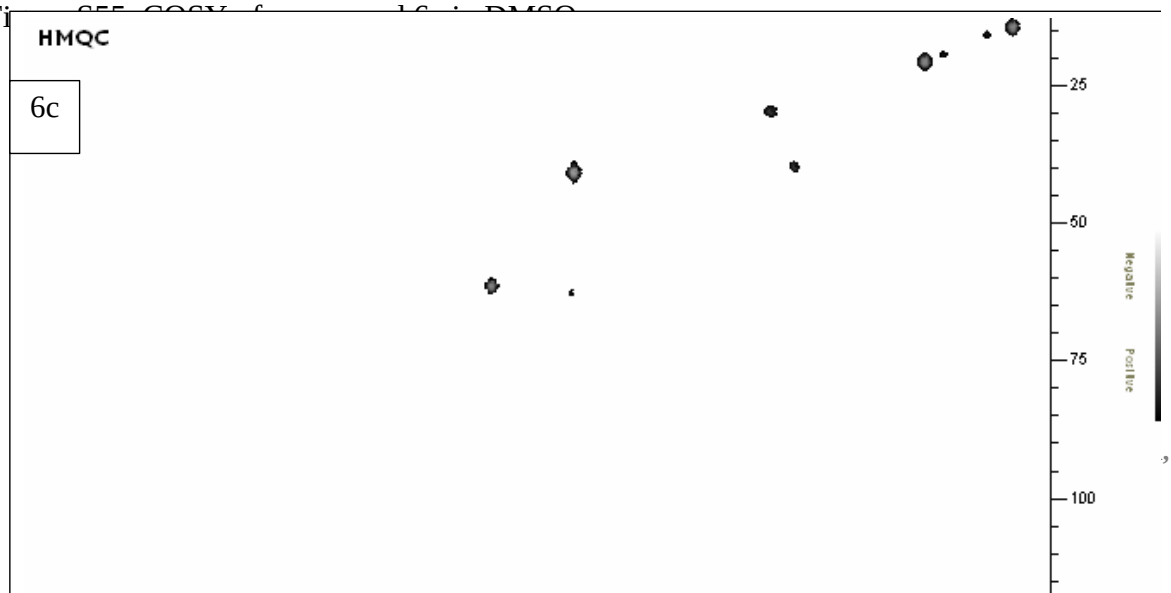


Figure S56. HMQC of compound 6c in DMSO

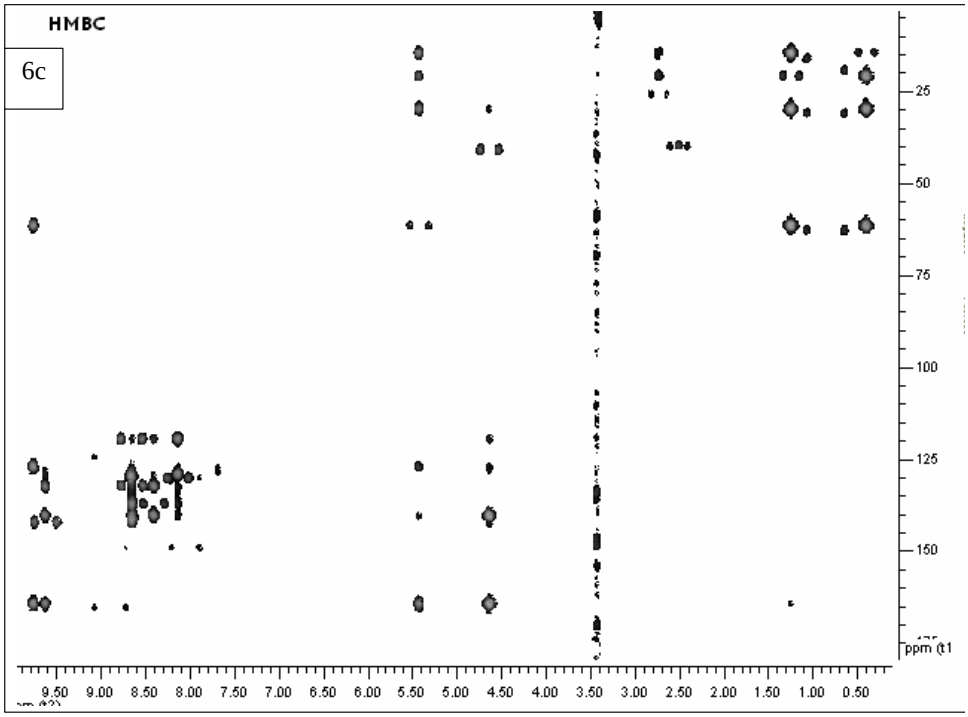


Figure S57. HMBC of compound 6c in DMSO

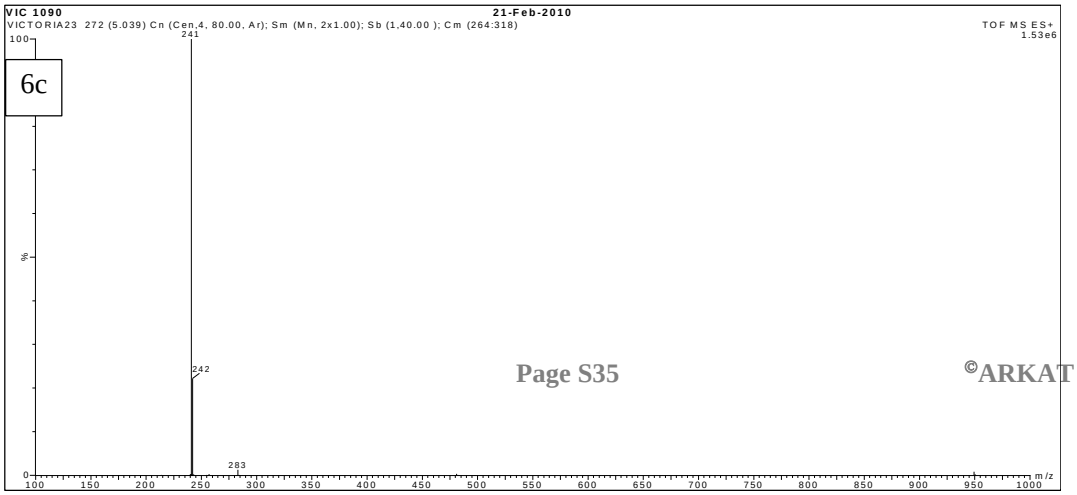
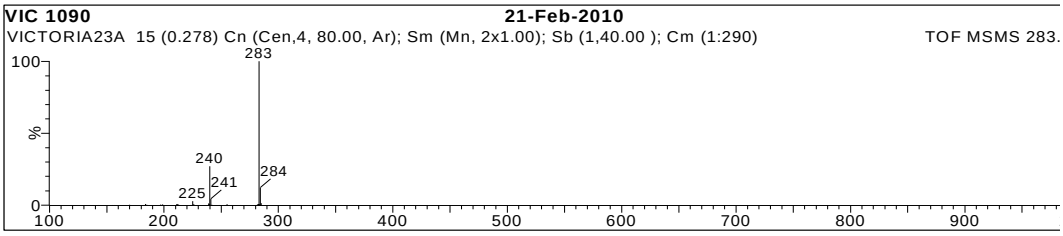


Figure S58. QTOF-MS of compound 6c

6c



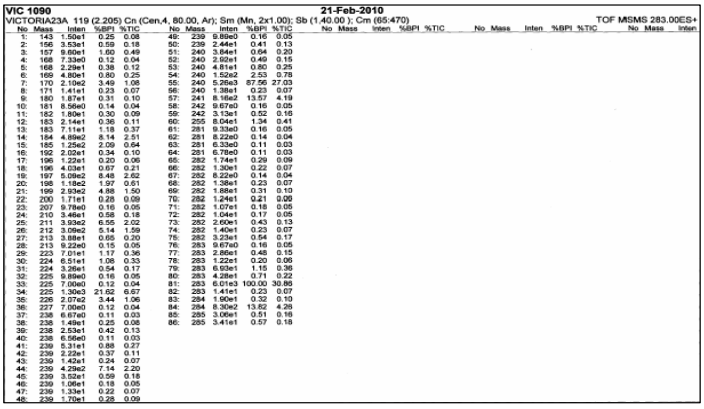


Figure S59. QTOF-M

Generated Molecular Formulas

Formula	Mass	Error	DblEq	N rule	Electron Configuration
C 15 H 17 N 2 O 1	241.1335	1.4888	8.50	ok	even

6c

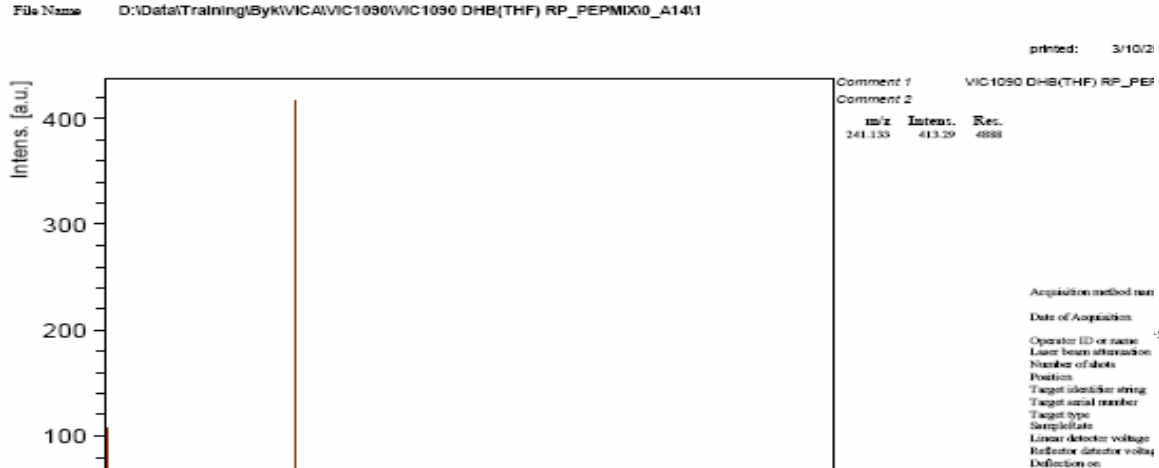


Figure S60. MALDI-TOF MS of compound 6c

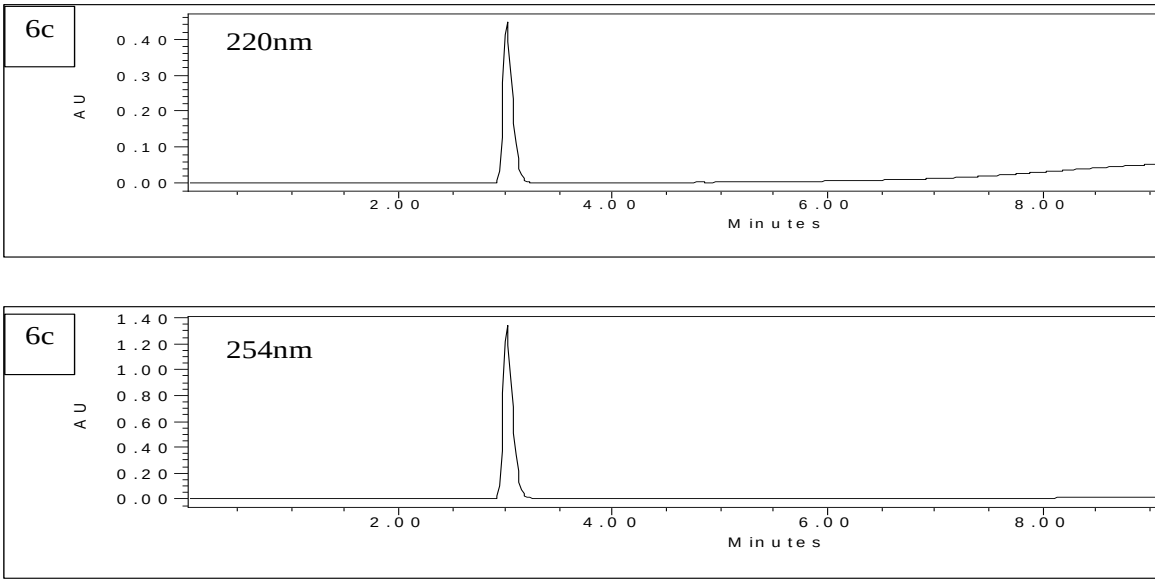


Figure S61. HPLC of compound 6c

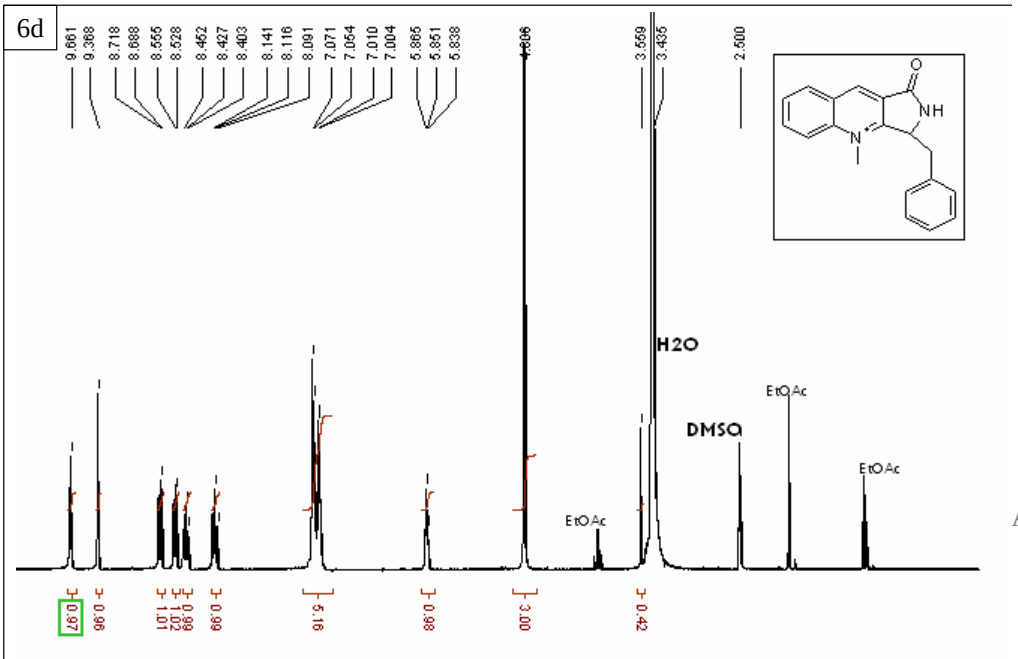


Figure S62. H-NMR of compound 6d in DMSO

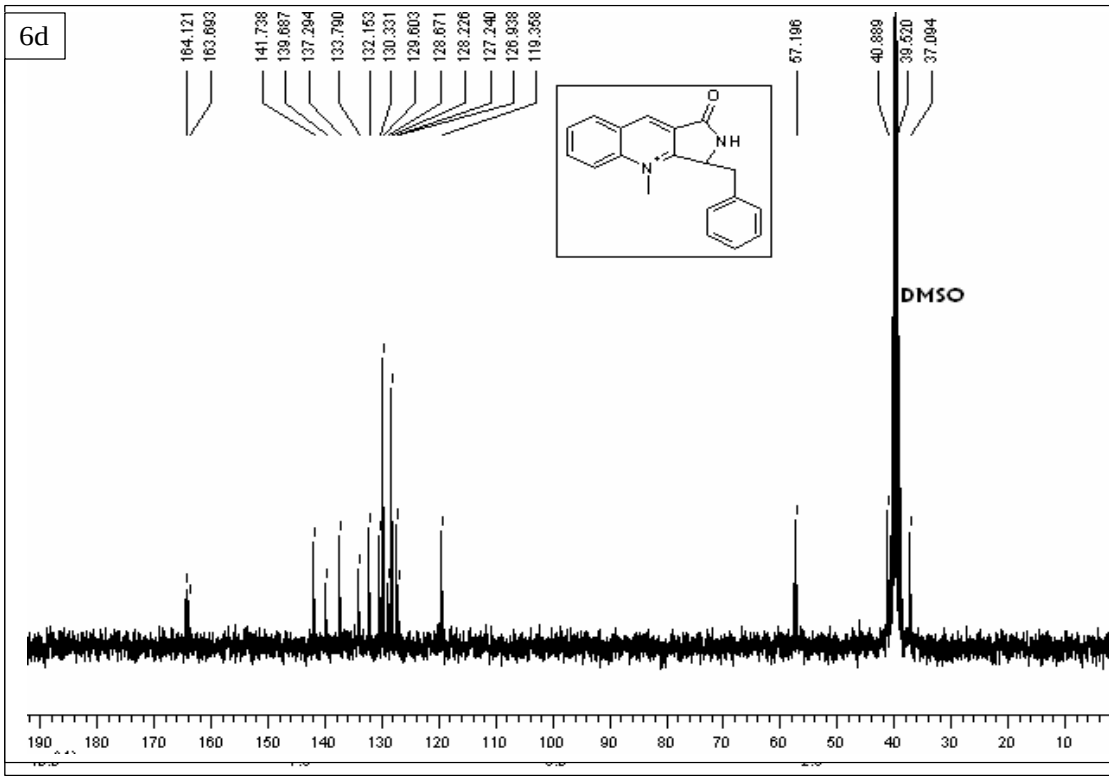


Figure S63. C-NMR of compound 6d in DMSO

6d

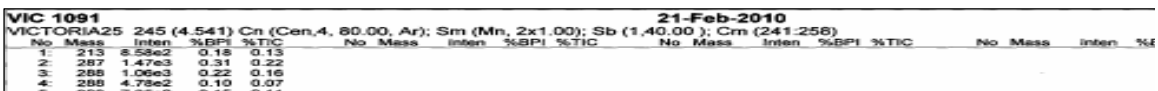


Figure S64. QTOF-MS of compound 6d

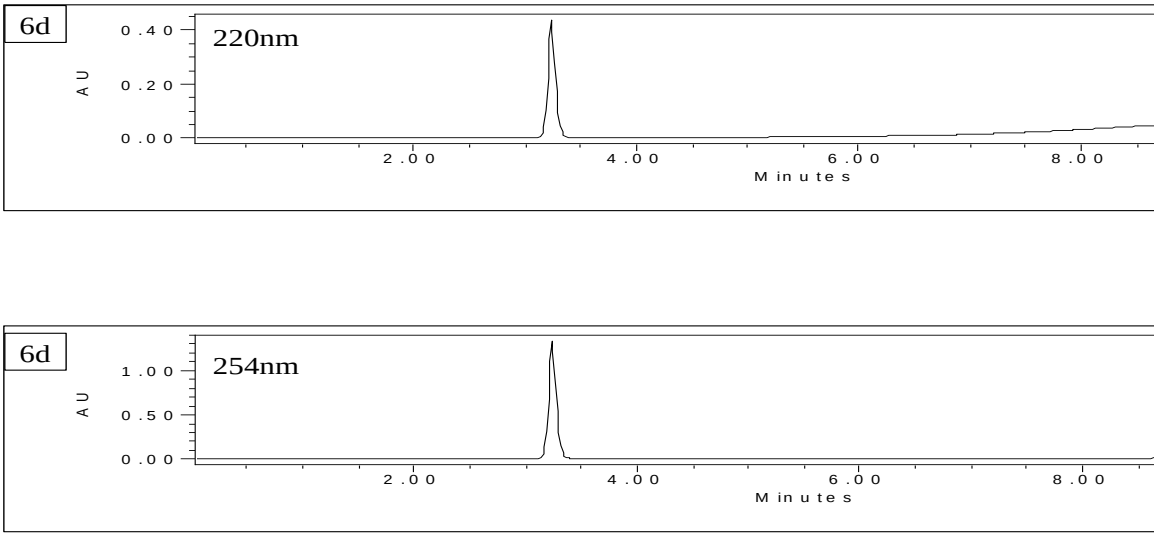


Figure S65. HPLC of compound 6d

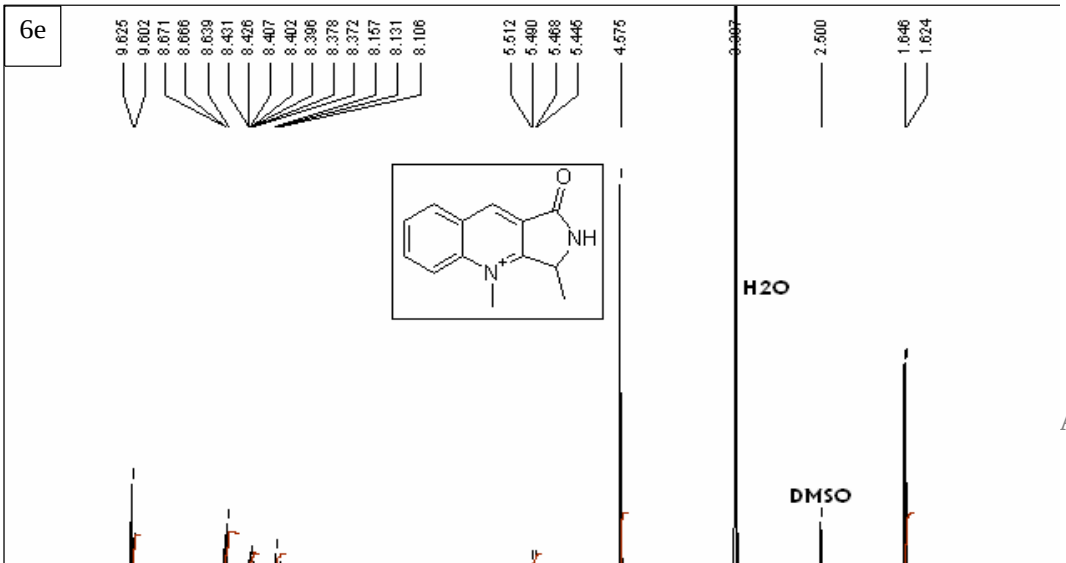


Figure S66. H-NMR of compound 6e in DMSO

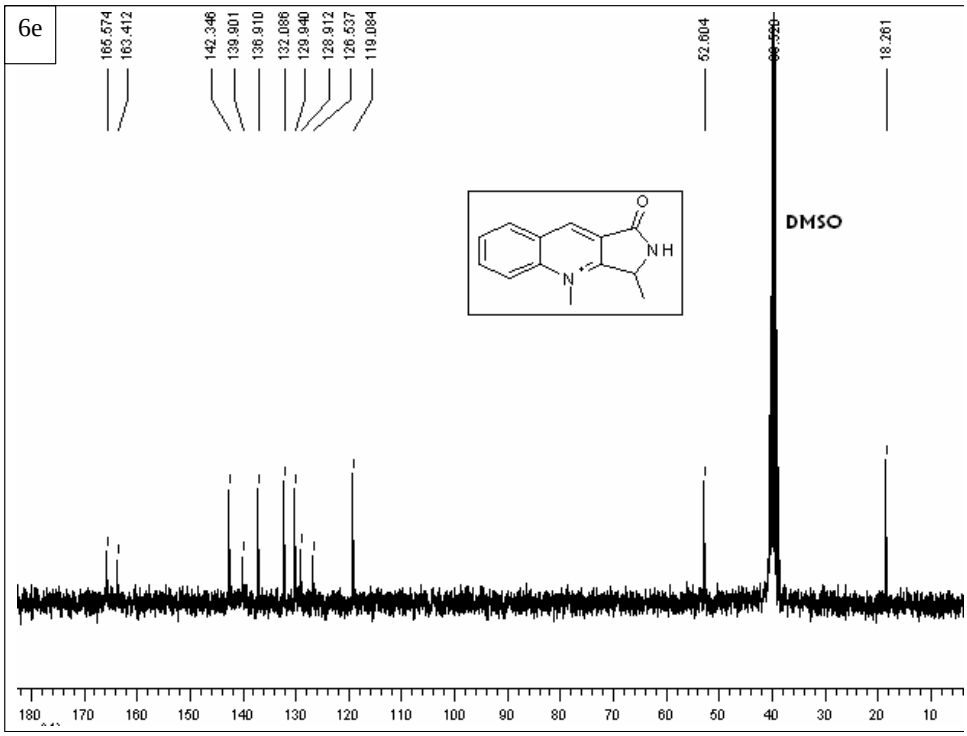


Figure S67. C-NMR of compound 6e in DMSO

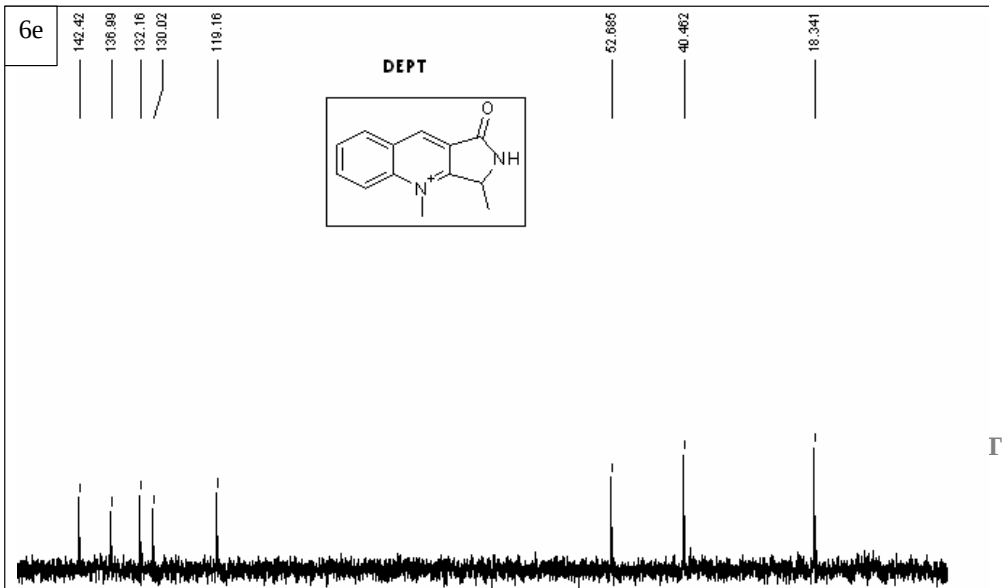
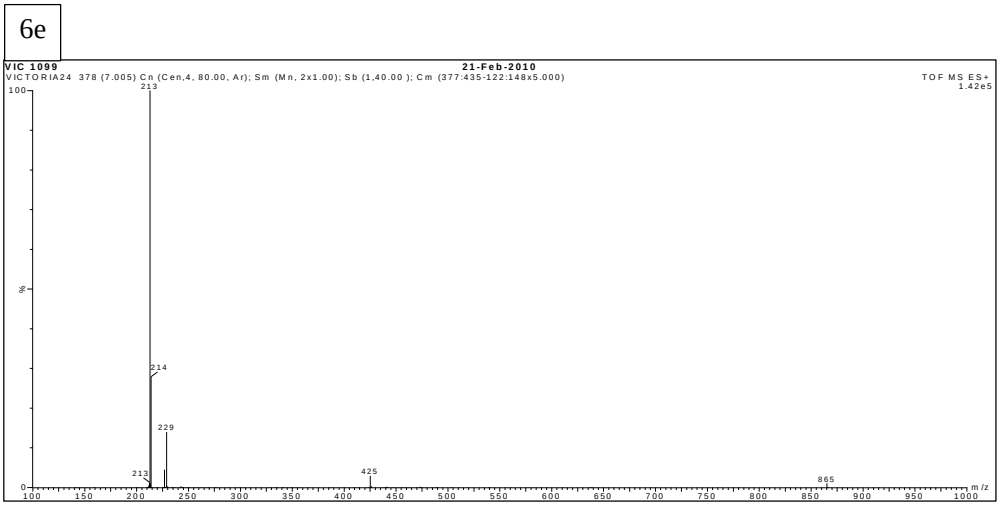


Figure S68. DEPT of compound 6e in DMSO



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VICTORIA24 262 (4.856) Cn (Cen,4, 80.00, Ar); Sm (Mn, 2x1.00); Sb (1,40.00); Cm (262:326-121:137x5.000) TOF MS ES+

No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Inten
1:	212	1.76e3	0.25	0.18													
2:	212	9.33e2	0.13	0.09													
3:	212	7.34e2	0.10	0.07													
4:	212	1.78e3	0.25	0.18													
5:	212	1.57e3	0.22	0.16													
6:	213	1.90e3	0.26	0.19													
7:	213	7.17e5	100.00	72.91													
8:	213	2.34e3	0.33	0.24													
9:	214	1.75e5	24.37	17.77													
10:	215	1.63e3	0.23	0.17													
11:	227	1.29e4	1.81	1.32													
12:	229	4.29e4	5.98	4.36													
13:	230	1.40e3	0.20	0.14													
14:	243	2.72e3	0.38	0.28													
15:	425	5.91e3	0.83	0.60													
16:	865	1.71e3	0.24	0.17													

Figure S69. QTOF-MS of compound 6e

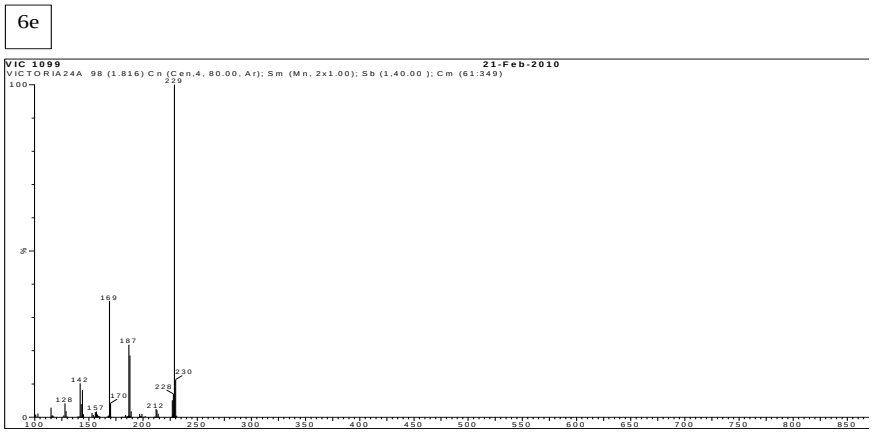

Figure S70. QTOF-MS of compound 6e

6e

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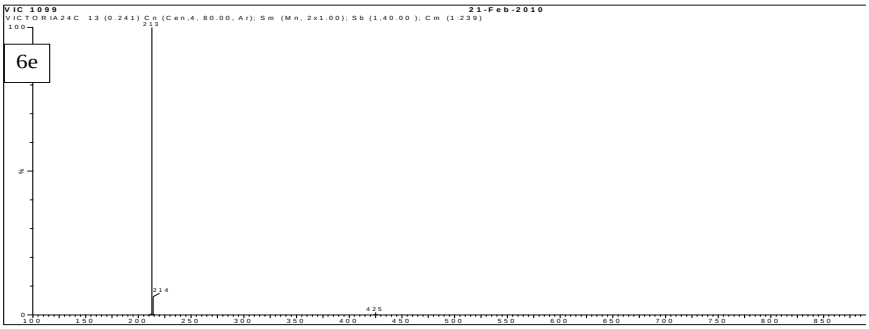
VICTORIA24A 98 (1.816) Cn (Cen,4, 80.00, Ar); Sm (Mn, 2x1.00); Sb (1,40.00); Cm (58:354)

No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TI
1:	101	9.20e1	0.77	0.28	49:	228	5.94e1	0.50	0.18					
2:	103	1.27e2	1.06	0.39	50:	229	8.47e1	0.71	0.26					
3:	115	3.40e2	2.84	1.04	51:	229	1.33e2	1.11	0.41					
4:	116	1.04e2	0.87	0.32	52:	229	1.20e4	100.00	36.54					
5:	117	5.10e1	0.43	0.16	53:	230	1.36e3	11.39	4.16					
6:	127	5.70e1	0.48	0.17	54:	231	4.93e1	0.41	0.15					
7:	128	4.88e2	4.07	1.49										
8:	129	2.13e2	1.78	0.65										
9:	142	1.19e3	9.95	3.64										
10:	143	4.67e2	3.90	1.42										
11:	144	9.50e2	7.94	2.90										
12:	145	9.81e1	0.82	0.30										
13:	153	1.48e2	1.24	0.45										
14:	154	8.30e1	0.69	0.25										
15:	156	1.77e2	1.48	0.54										
16:	157	1.94e2	1.62	0.59										
17:	158	9.40e1	0.79	0.29										
18:	158	7.73e1	0.65	0.24										
19:	160	3.60e1	0.30	0.11										
20:	168	5.52e1	0.46	0.17										
21:	168	3.76e1	0.31	0.11										
22:	169	4.01e1	0.34	0.12										
23:	169	4.06e3	33.95	12.40										
24:	170	4.86e2	4.06	1.48										
25:	184	7.90e1	0.66	0.24										
26:	187	4.66e1	0.39	0.14										
27:	187	2.52e3	21.09	7.71										
28:	188	4.49e1	0.38	0.14										
29:	188	2.17e3	18.11	6.62										
30:	189	2.06e2	1.72	0.63										
31:	197	1.11e2	0.93	0.34										
32:	199	1.12e2	0.94	0.34										
33:	212	2.90e2	2.43	0.89										
34:	213	2.44e2	2.04	0.75										
35:	214	1.22e2	1.02	0.37										
36:	227	6.07e2	5.07	1.85										
37:	228	3.87e1	0.32	0.12										
38:	228	3.71e1	0.31	0.11										
39:	228	6.79e1	0.57	0.21										
40:	228	4.69e1	0.39	0.14										
41:	228	5.73e1	0.48	0.18										
42:	228	8.18e2	6.83	2.50										
43:	228	4.50e1	0.38	0.14										
44:	228	5.32e1	0.44	0.16										
45:	228	4.43e1	0.37	0.14										
46:	228	3.61e1	0.30	0.11										
47:	229	5.11e1	0.43	0.16										
48:	229	1.27e2	1.06	0.39										

Figure S71. QTOF-MS of compound 6e



6e

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VICTORIA24C 13 (0.241) Cn (Cen,4, 80.00, Ar); Sm (Mn, 2x1.00); Sb (1,40.00); Cm (1:243)

No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC
1:	211	2.15e2	0.29	0.27										
2:	212	2.56e2	0.35	0.32										
3:	213	8.39e1	0.11	0.10										
4:	213	7.78e1	0.11	0.10										
5:	213	1.89e2	0.26	0.23										
6:	213	1.12e2	0.15	0.14										
7:	213	1.84e2	0.25	0.23										
8:	213	7.38e4	100.00	91.31										
9:	214	4.62e3	6.26	5.72										
10:	215	2.03e2	0.27	0.25										
11:	425	5.94e2	0.80	0.73										

Figure S72. QTOF-MS of compound 6e

Generated Molecular Formulas

Formula	Mass	Error	DblEq	N rule	Electron Configuration
C 13 H 13 N 2 O 1	213.1022	0.9076	8.50	ok	even

6e

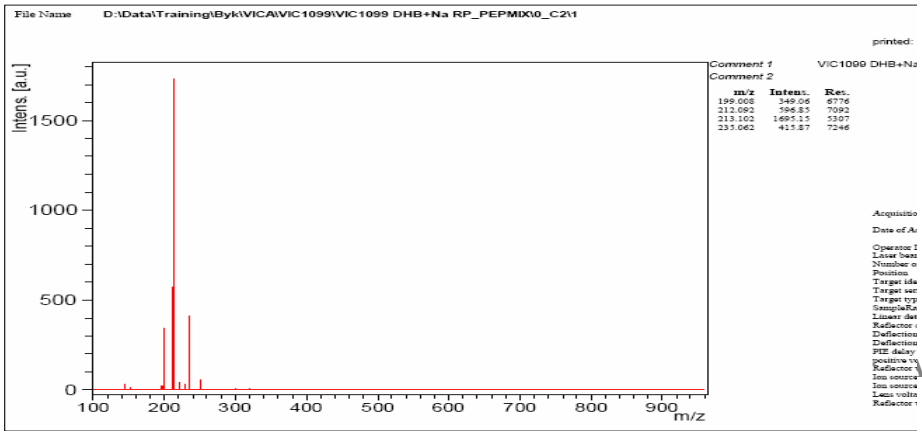


Figure S73. MALDI-TOF MS of compound 6e

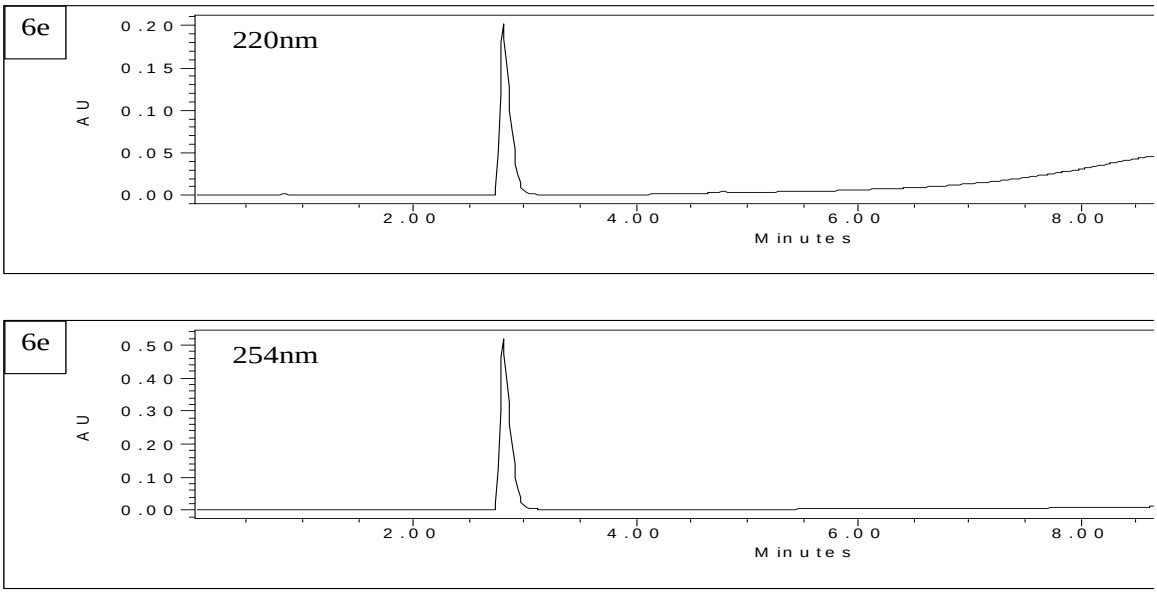


Figure S74. HPLC of compound 6e

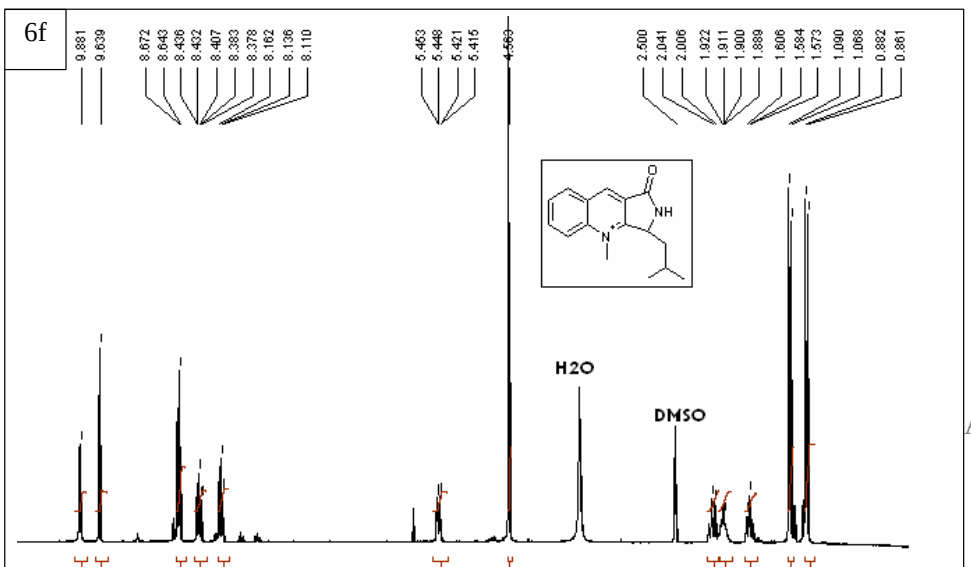


Figure S75. H-NMR of compound 6f in DMSO

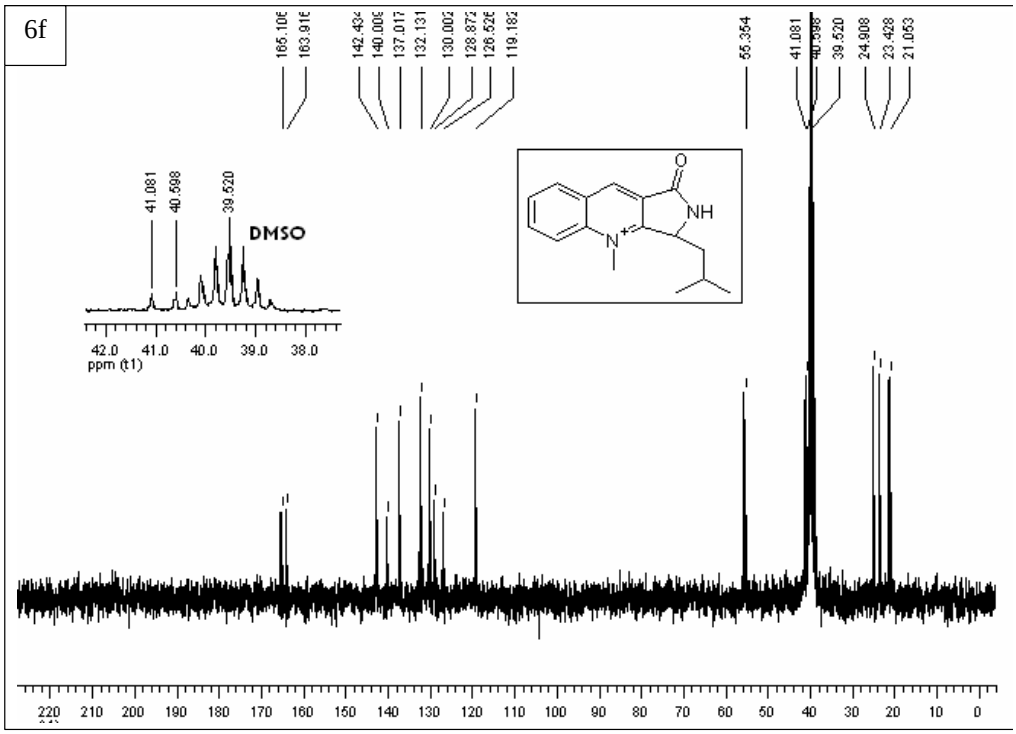
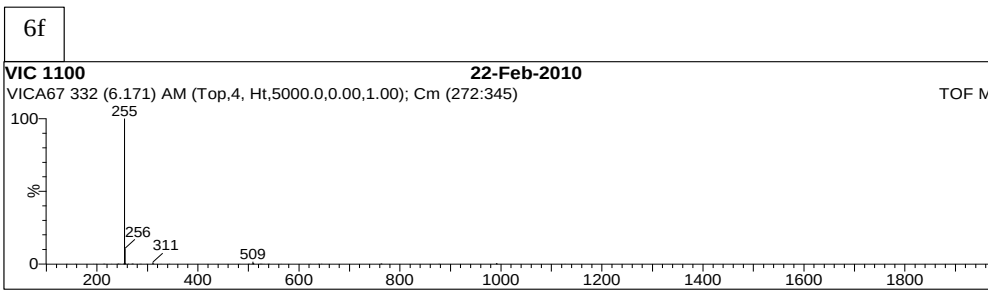
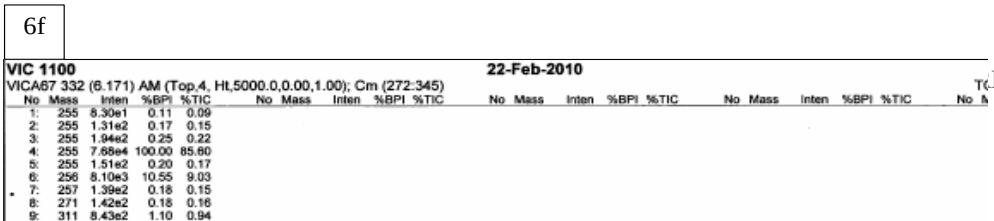


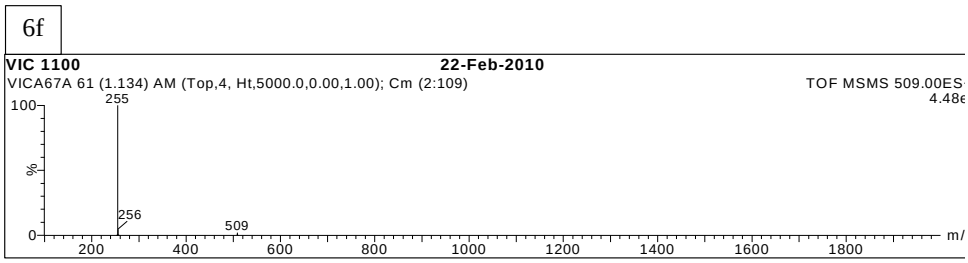
Figure S76. C-NMR of compound 6f in DMSO

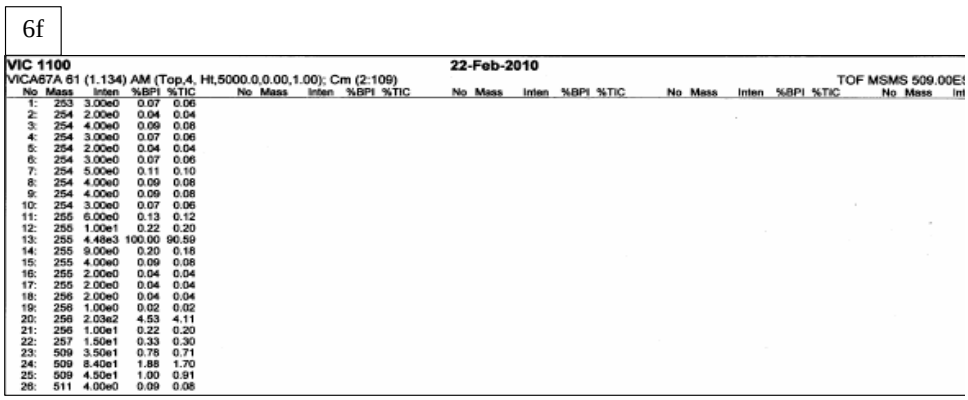




No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No
1:	255	8.30e1	0.11	0.09																
2:	255	1.31e2	0.17	0.15																
3:	255	1.94e2	0.25	0.22																
4:	255	7.68e4	100.00	85.80																
5:	255	1.51e2	0.20	0.17																
6:	256	8.10e3	10.55	9.03																
7:	257	1.39e2	0.18	0.15																
8:	271	1.42e2	0.18	0.16																
9:	311	8.43e2	1.10	0.94																

Figure S77. QTOF-MS of compound 6f



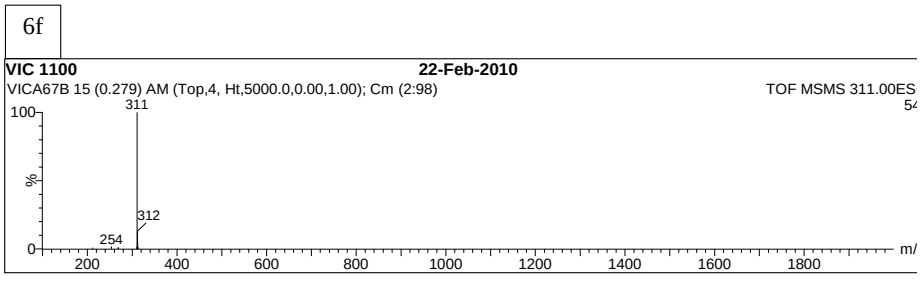

6f

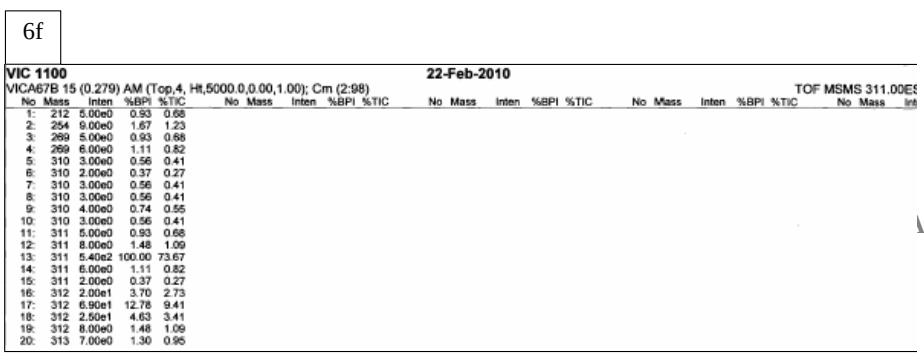
VIC 1100 22-Feb-2010

VICA67A 61 (1.134) AM (Top,4, Ht,5000.0,0.00,1.00); Cm (2:109) TOF MSMS 509.00ES

No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Int
1:	253	3.00e0	0.07	0.06																		
2:	254	2.00e0	0.04	0.04																		
3:	254	4.00e0	0.09	0.08																		
4:	254	3.00e0	0.07	0.06																		
5:	254	2.00e0	0.04	0.04																		
6:	254	3.00e0	0.07	0.06																		
7:	254	5.00e0	0.11	0.10																		
8:	254	4.00e0	0.09	0.08																		
9:	254	4.00e0	0.09	0.08																		
10:	254	3.00e0	0.07	0.06																		
11:	255	6.00e0	0.13	0.12																		
12:	255	1.00e1	0.22	0.20																		
13:	255	4.48e3	100.00	90.59																		
14:	255	9.00e0	0.20	0.18																		
15:	255	4.00e0	0.09	0.08																		
16:	255	2.00e0	0.04	0.04																		
17:	255	2.00e0	0.04	0.04																		
18:	256	2.00e0	0.04	0.04																		
19:	256	1.00e0	0.02	0.02																		
20:	256	2.03e2	4.53	4.11																		
21:	256	1.00e1	0.22	0.20																		
22:	257	1.50e1	0.33	0.30																		
23:	509	3.50e1	0.78	0.71																		
24:	509	8.40e1	1.88	1.70																		
25:	509	4.50e1	1.00	0.91																		
26:	511	4.00e0	0.09	0.08																		

Figure S78. QTOF-MS of compound 6f




6f

VIC 1100 22-Feb-2010

VICA67B 15 (0.279) AM (Top,4, Ht,5000.0,0.00,1.00); Cm (2:98) TOF MSMS 311.00ES

No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Int
1:	212	5.00e0	0.93	0.68													
2:	254	9.00e0	1.67	1.23													
3:	269	5.00e0	0.93	0.68													
4:	269	6.00e0	1.11	0.82													
5:	310	3.00e0	0.56	0.41													
6:	310	2.00e0	0.37	0.27													
7:	310	3.00e0	0.56	0.41													
8:	310	3.00e0	0.56	0.41													
9:	310	4.00e0	0.74	0.55													
10:	310	3.00e0	0.56	0.41													
11:	311	5.00e0	0.93	0.68													
12:	311	8.00e0	1.48	1.09													
13:	311	5.40e2	100.00	73.67													
14:	311	6.00e0	1.11	0.82													
15:	311	2.00e0	0.37	0.27													
16:	312	2.00e1	3.70	2.73													
17:	312	6.90e1	12.78	9.41													
18:	312	2.50e1	4.63	3.41													
19:	312	8.00e0	1.48	1.09													
20:	313	7.00e0	1.30	0.95													

Figure S79. QTOF-MS of compound 6f

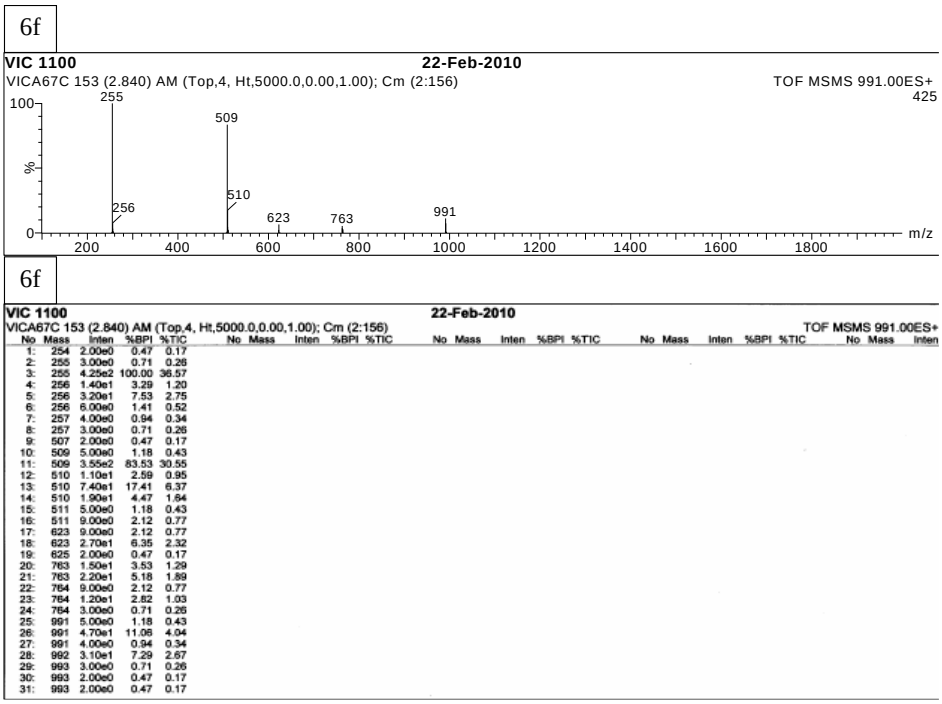


Figure S80. QTOF-MS of compound 6f

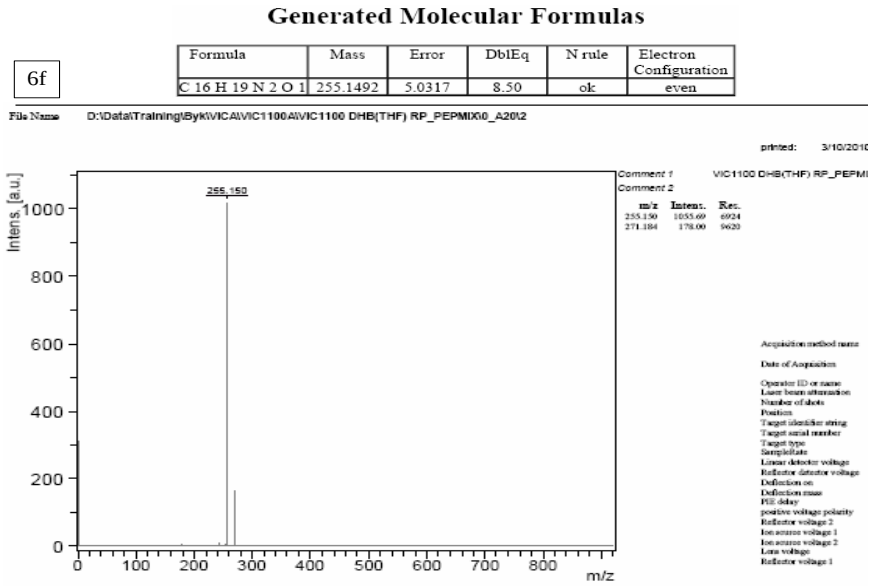


Figure S81. MALDI-TOF MS of compound 6f

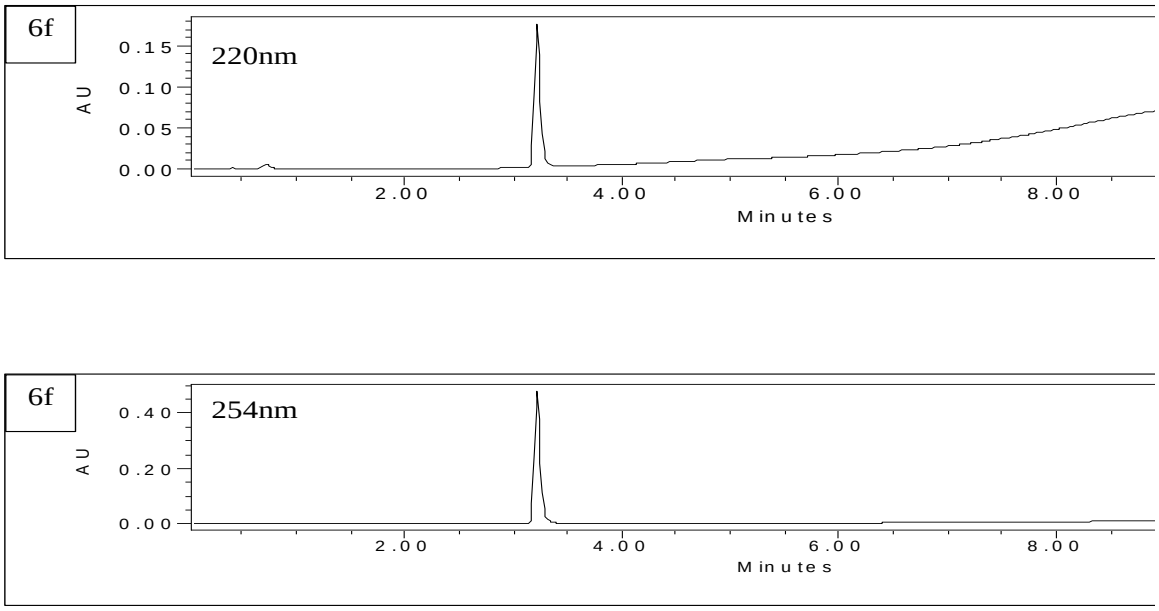


Figure S82. HPLC of compound 6f

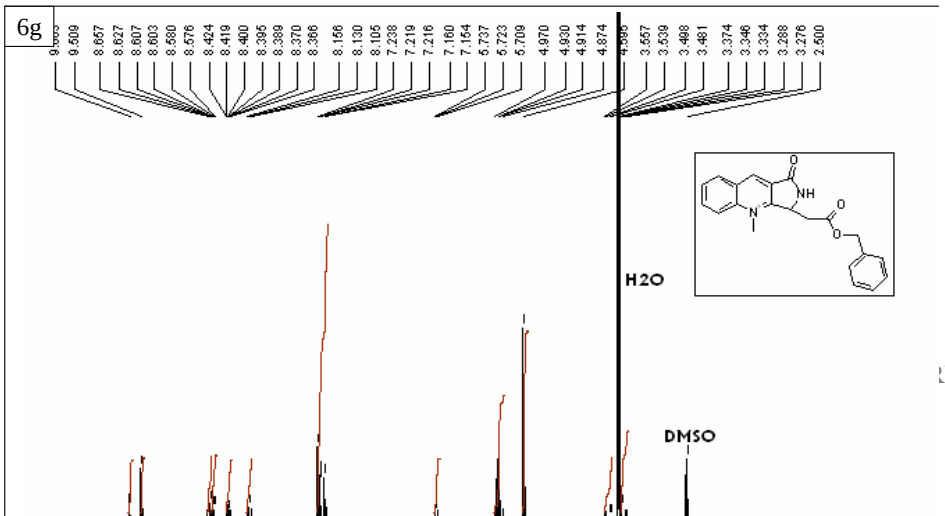


Figure S83. H-NMR of compound 6g in DMSO

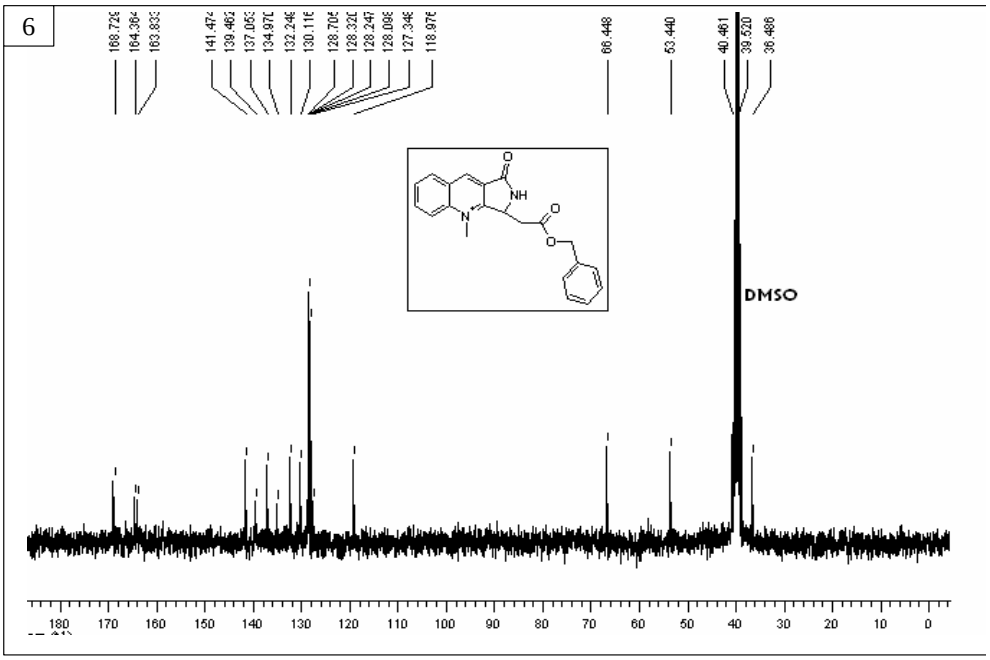
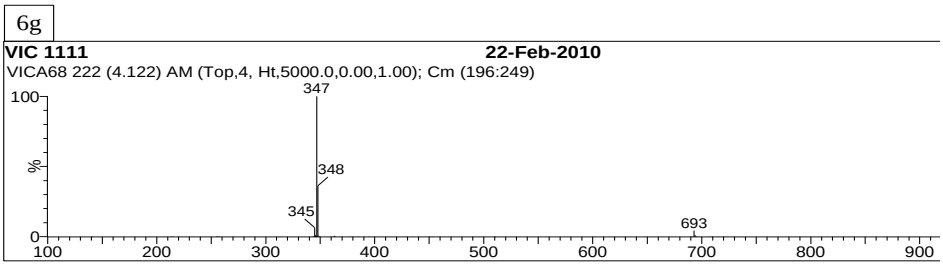


Figure S84. C-NMR of compound 6g in DMSO



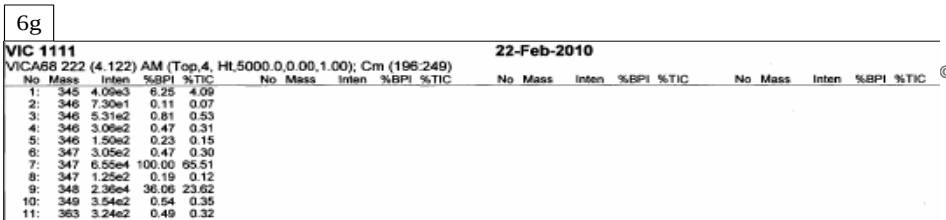


Figure S85. QTOF-MS of compound 6g

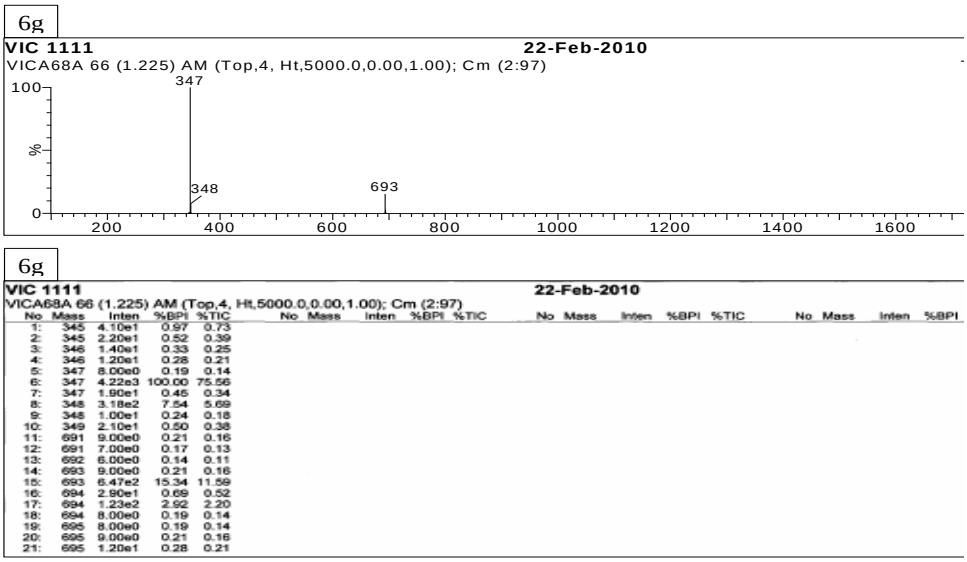


Figure S86. QTOF-MS of compound 6g

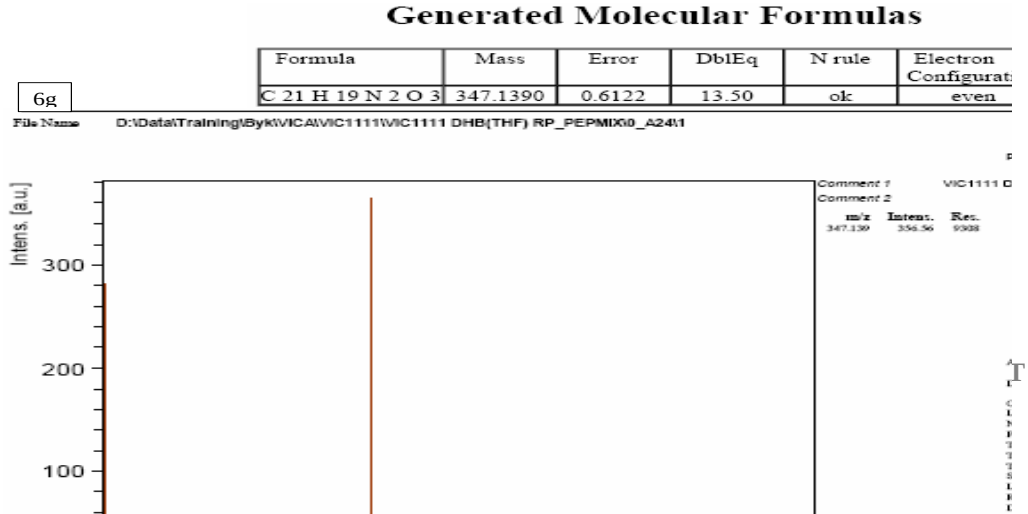


Figure S87. MALDI-TOF MS of compound 6f

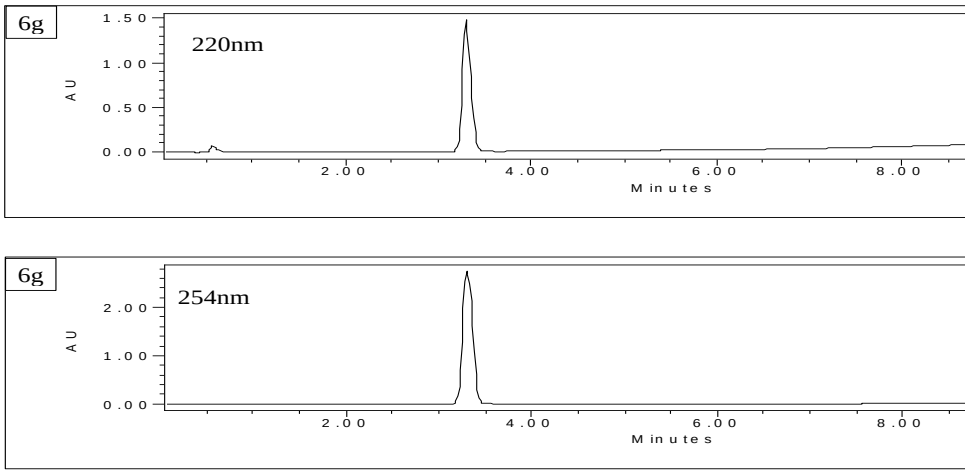
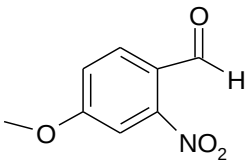


Figure S88. HPLC of compound 6g

4-methoxy-2-nitrobenzaldehyde



[1]HNMR: (300 MHz, DMSO-d_6), $\delta_{H(ppm)}$: 3.94 (s,3H), 7.42 (dd,1H,J=8.7Hz, J=2.4Hz), 7.62(d,1H, J=2.4Hz),7.96 (s,3H, J=8.7Hz), 10.04 (s,1H).

^{13}CNMR: (75 MHz, DMSO-d_6), $\delta_{C\ (ppm)}$:56.72, 109.8, 118.58, 122.05, 132.61, 151, 163.54, 188.22. (yield 11%)**MS:** MW=181 g/mol, MH^+ =182. **HPLC:** t_R =2.974 min**.**

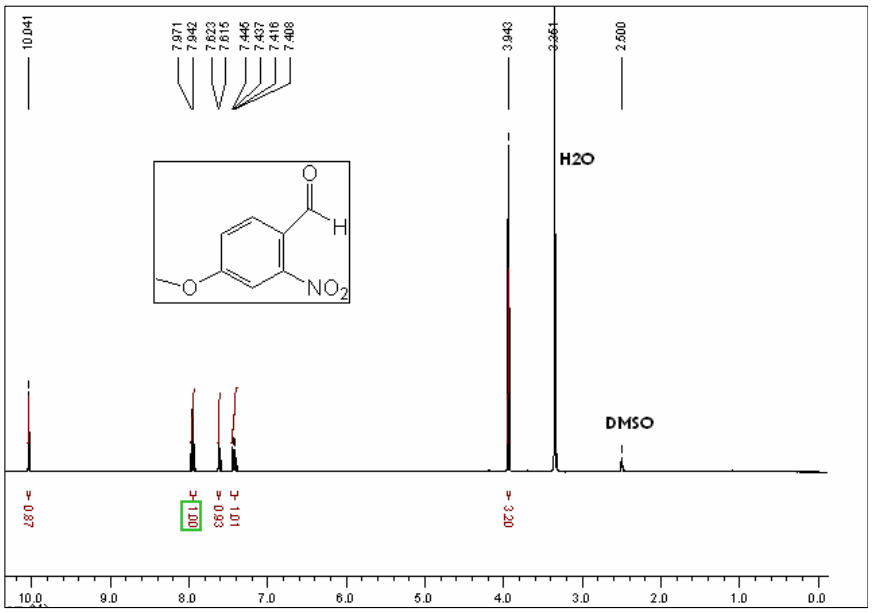


Figure S89. H-NMR of 4-methoxy-2-nitrobenzaldehyde in DMSO**.**

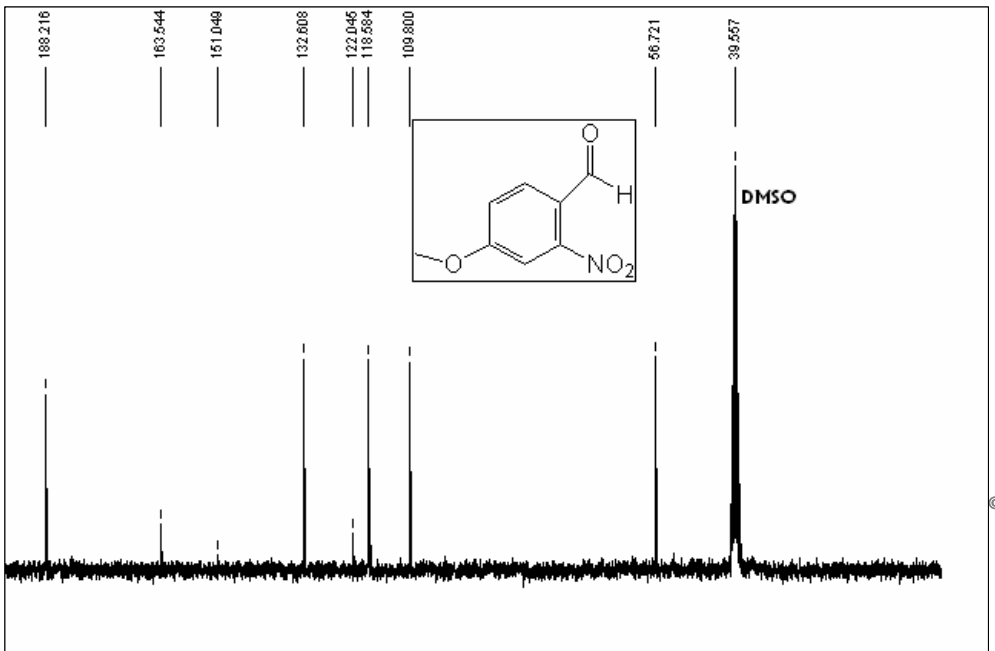


Figure S90. C-NMR of 4-methoxy-2-nitrobenzaldehyde in DMSO

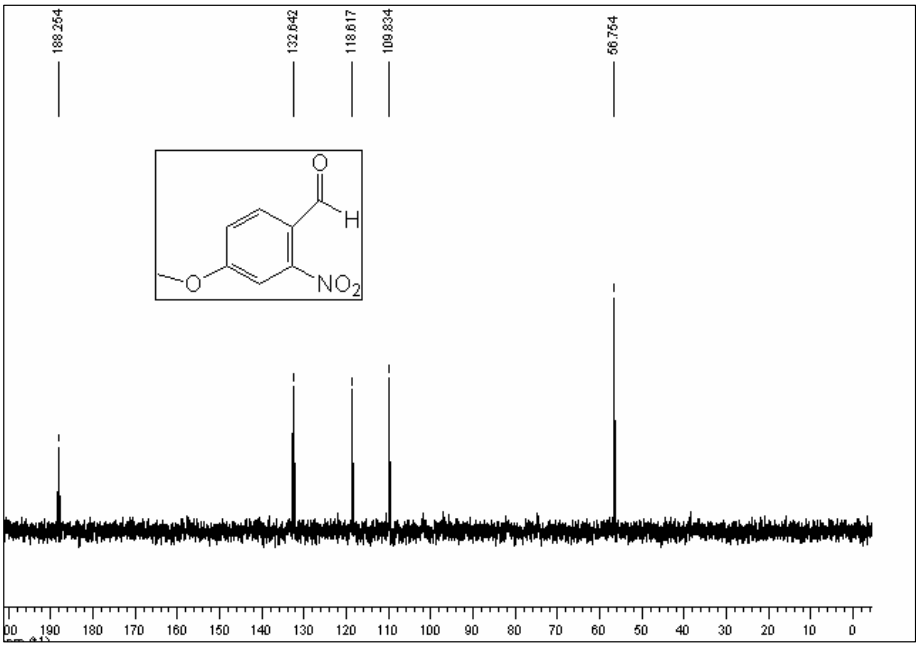
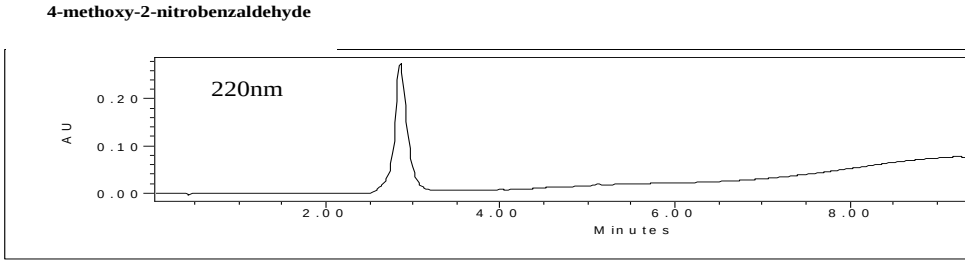


Figure S91. DEPT of 4-methoxy-2-nitrobenzaldehyde in DMSO



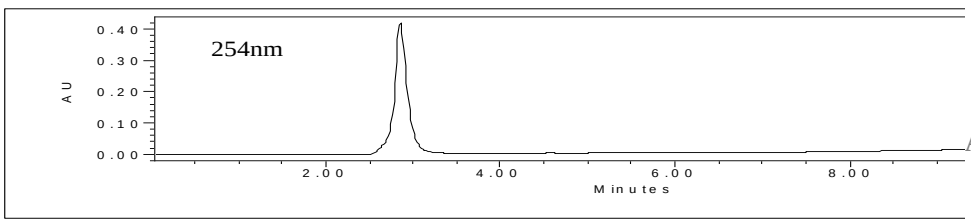
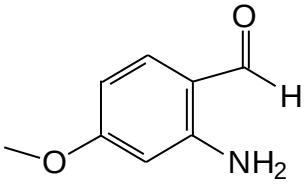


Figure S92. HPLC of 4-methoxy-2-nitrobenzaldehyde

2-amino-4-methoxybenzaldehyde



^{1}HNMR: (300 MHz, DMSO-d_6), $\delta_{H(ppm)}$: 3.75 (s,3H), 6.23 (m,2H), 7.19 (bs, NH), 7.42 (d,1H, J=9.3Hz), 9.63 (s,1H).

^{13}CNMR: (75 MHz, DMSO-d_6), $\delta_{C\ (ppm)}$: 55.17, 97.66, 104.30, 112.92, 137.61, 153.02, 164.71, 191.62. (yield 83%)**. MS:** MW=151.16 g/mol, MH^+ =152. **HPLC:** t_R =2.51 min**.**

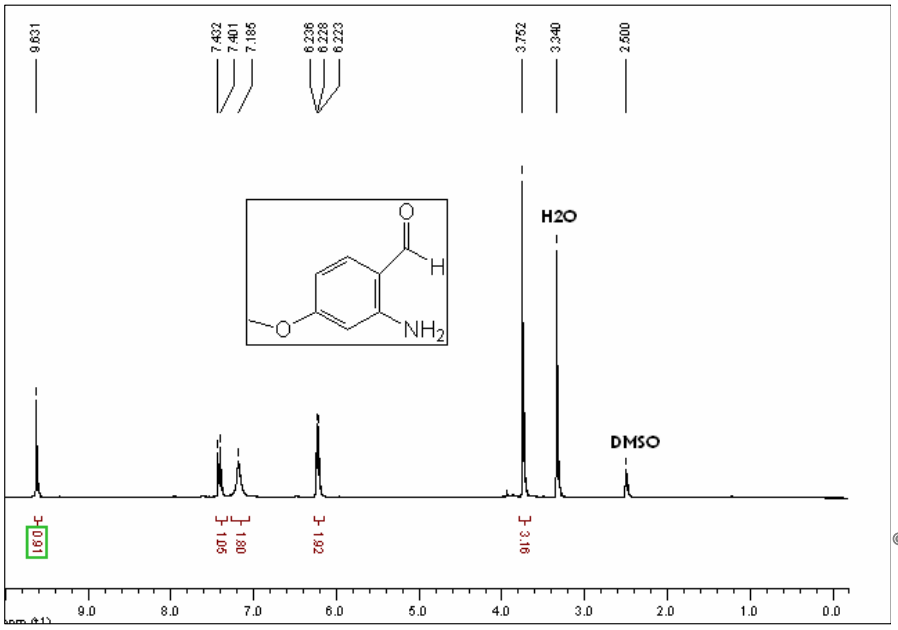


Figure S93. H-NMR of 4-methoxy-2-nitrobenzaldehyde in DMSO

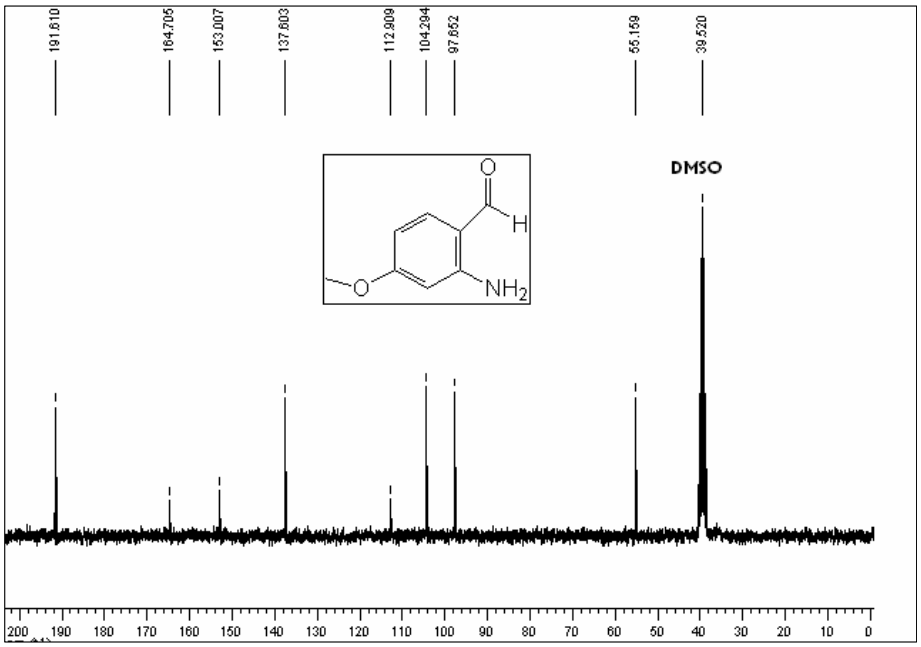


Figure S94. C-NMR of 4-methoxy-2-nitrobenzaldehyde in DMSO

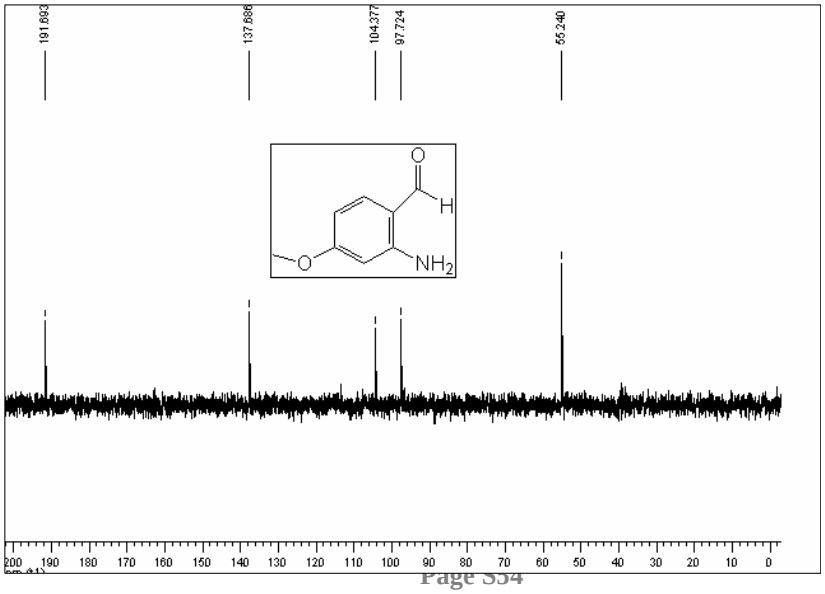
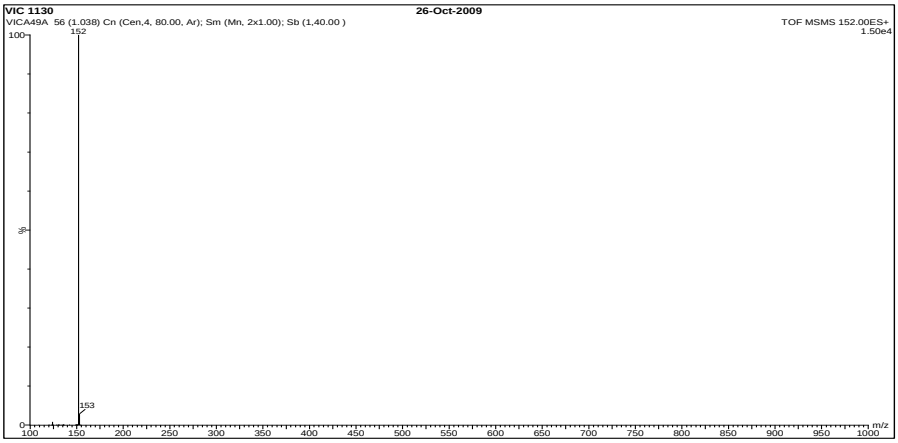


Figure S95. DEPT of 4-methoxy-2-aminobenzaldehyde in DMSO



VIC 1130 **26-Oct-2009**

VICA49A 56 (1.038) Cn (Cen,4, 80.00, Ar); Sm (Mn, 2x1.00); Sb (1,40.00)

No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%I
1:	124	1.20e2	0.80	0.77														
2:	150	3.07e1	0.20	0.20														
3:	152	1.50e4	100.00	96.20														
4:	153	4.19e2	2.79	2.69														

Figure S96. QTOF-MS of 4-methoxy-2-aminobenzaldehyde

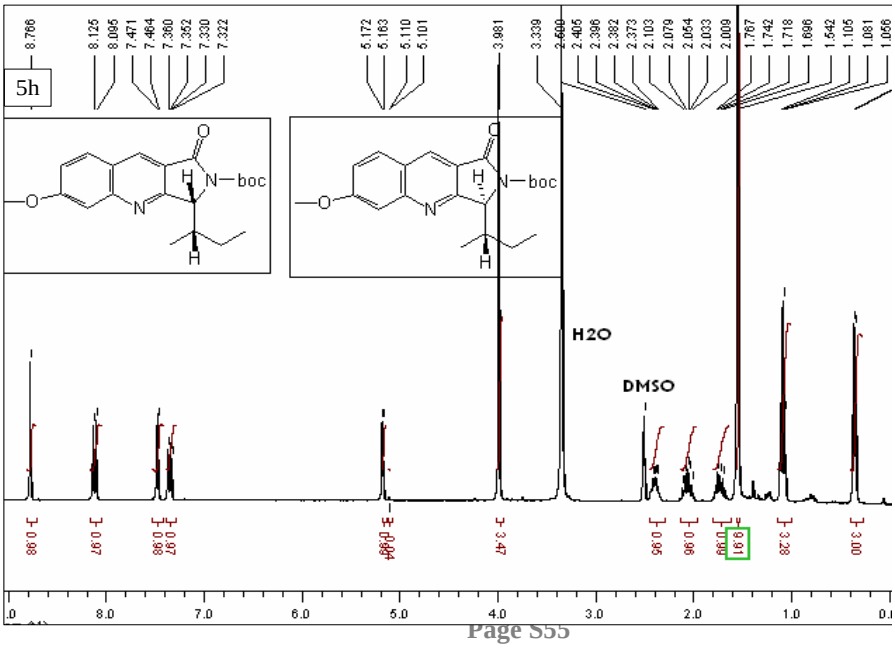


Figure S97. H-NMR of compound 5h in DMSO

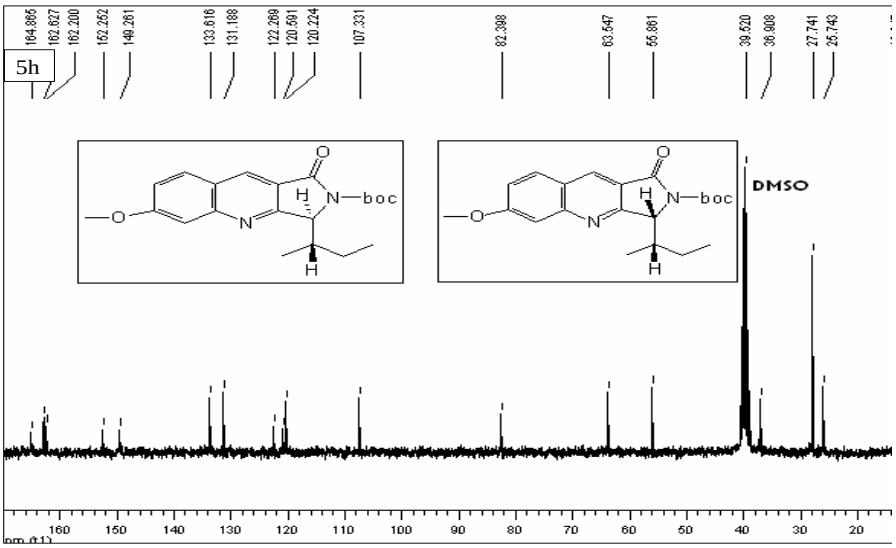


Figure S98. C-NMR of compound 5h in DMSO

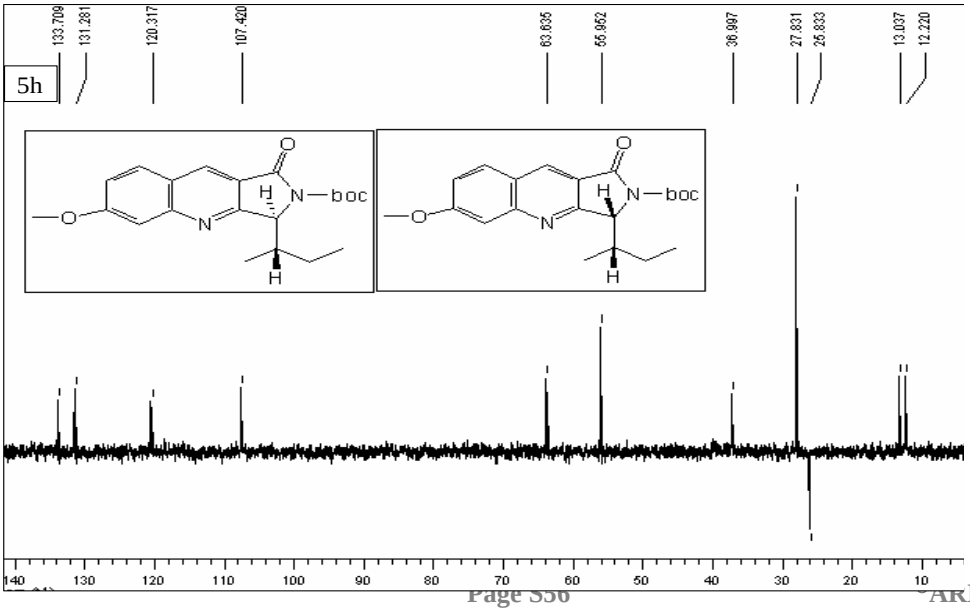


Figure S99. DEPT of compound 5h in DMSO

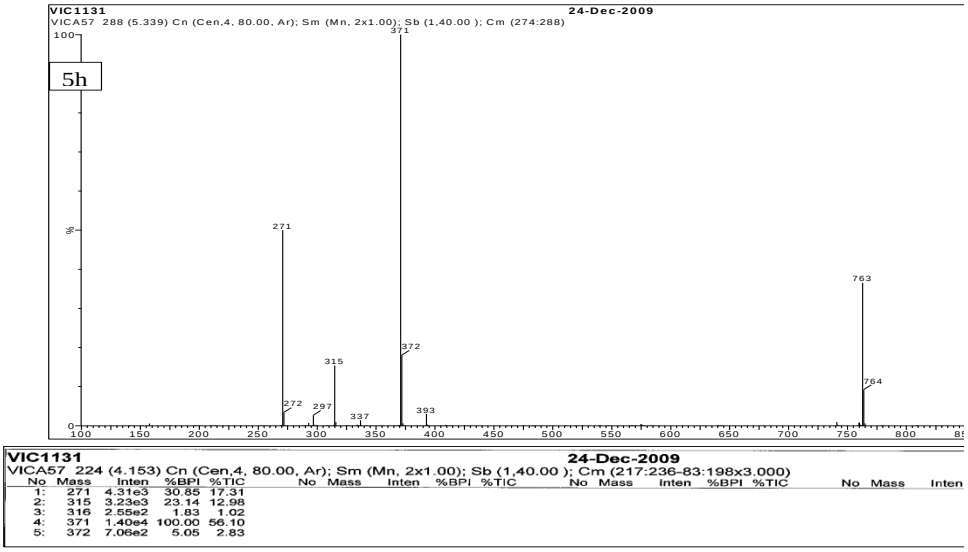
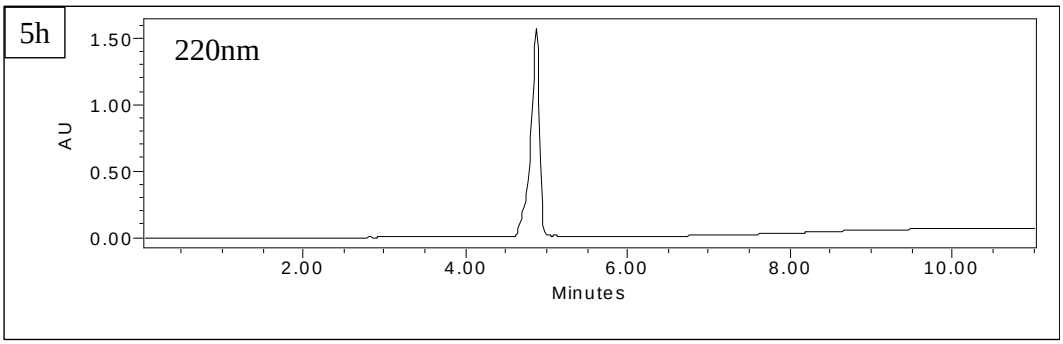


Figure S100. QTOF-MS of compound 5h



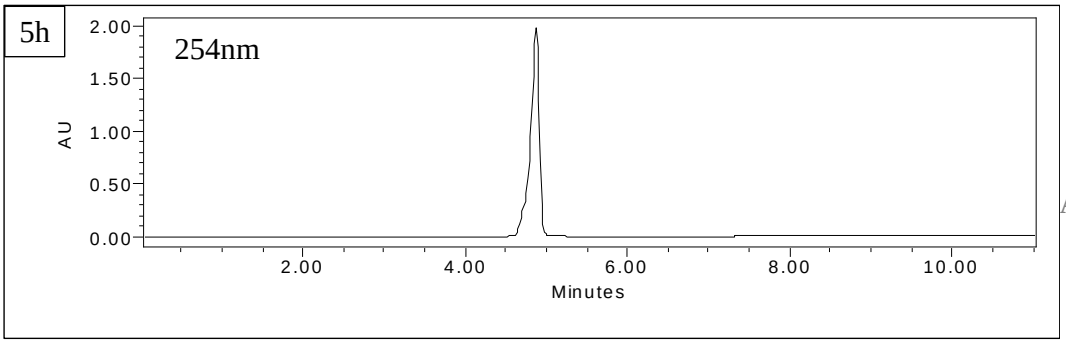


Figure S101. HPLC of compound 5h

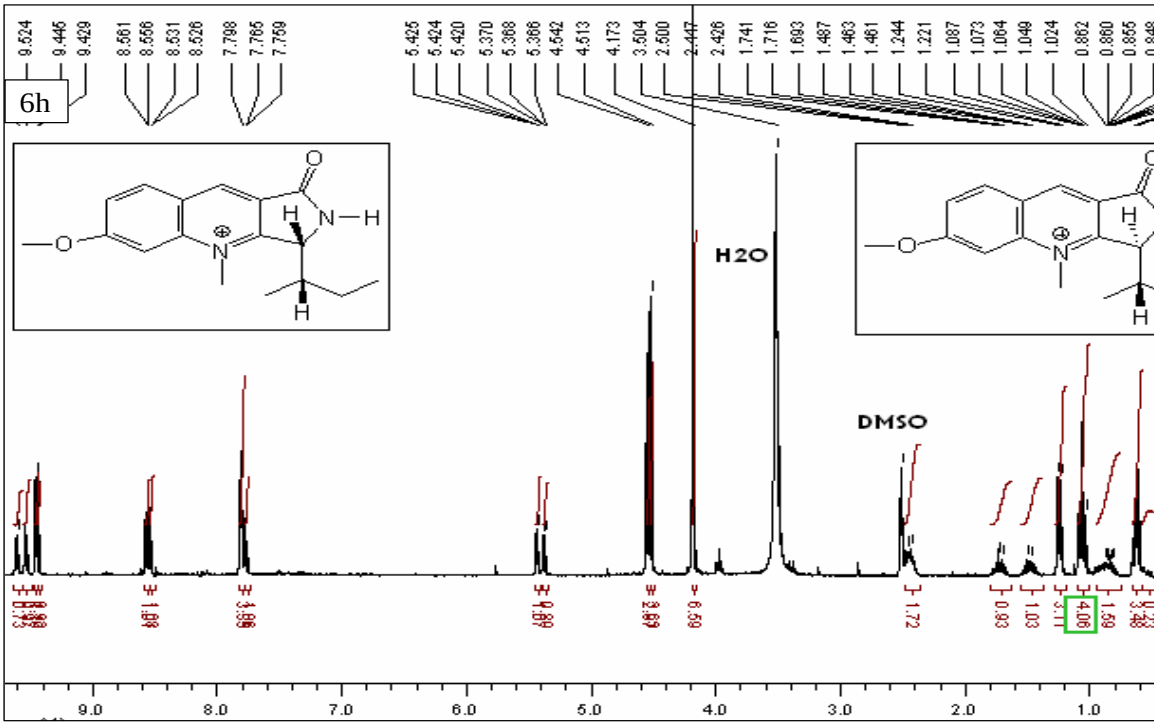


Figure S102. H-NMR of compound 6h in DMSO

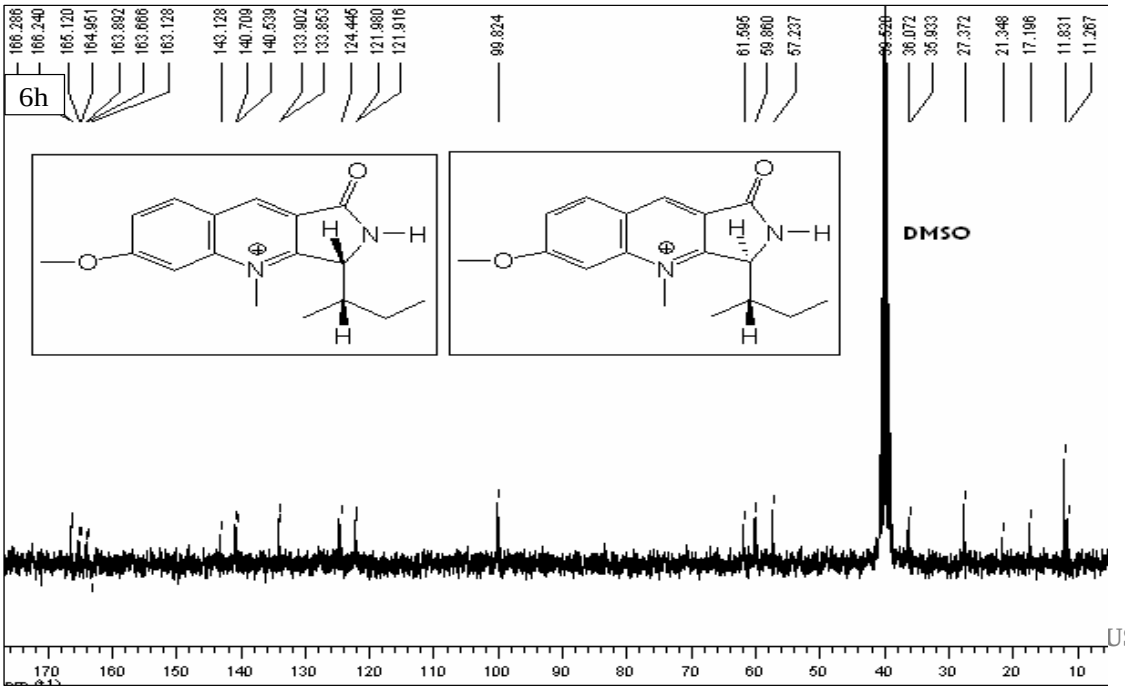


Figure S103. C-NMR of compound 6h in DMSO

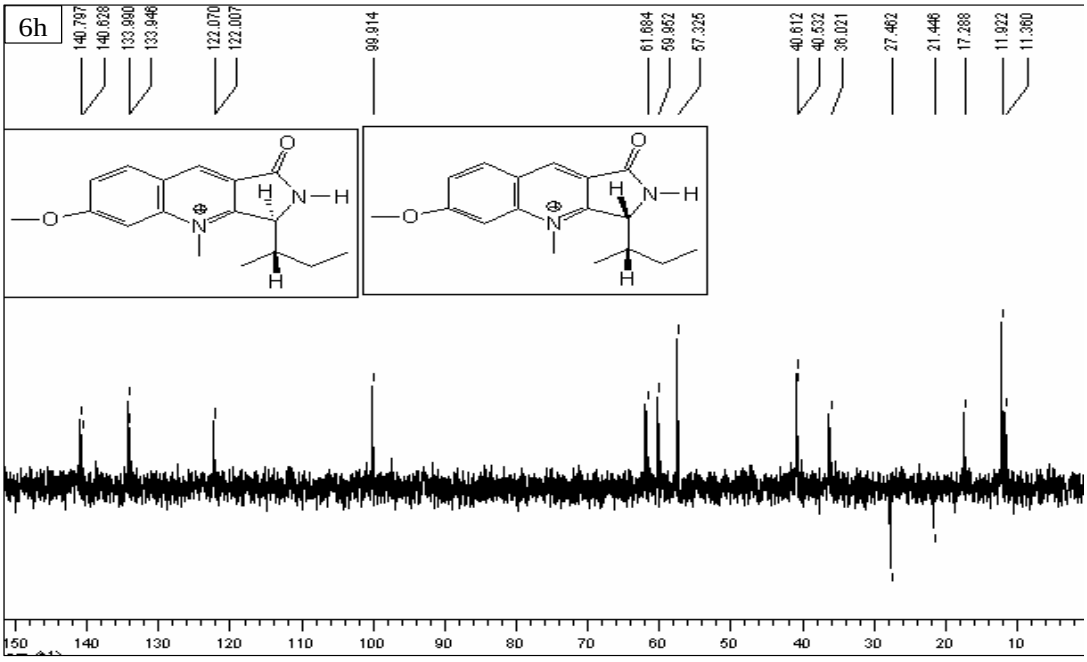
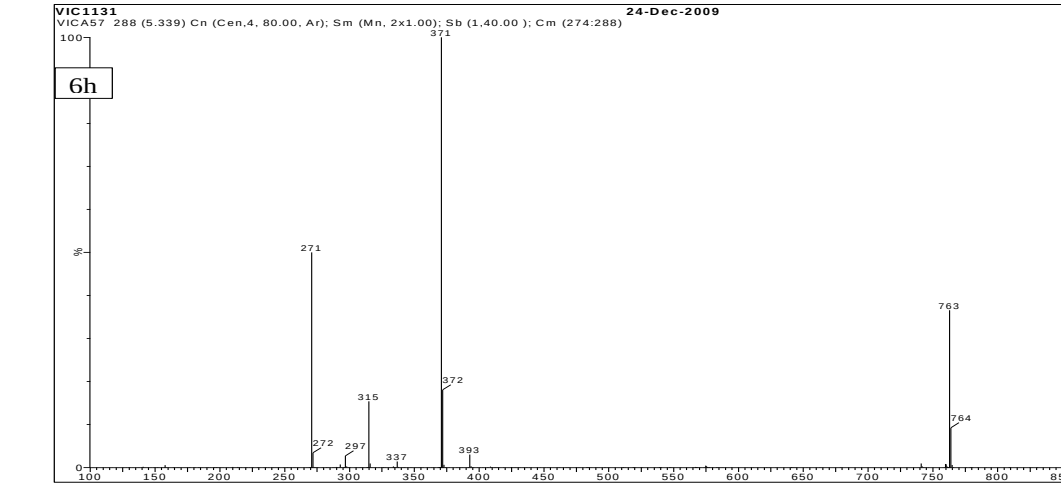


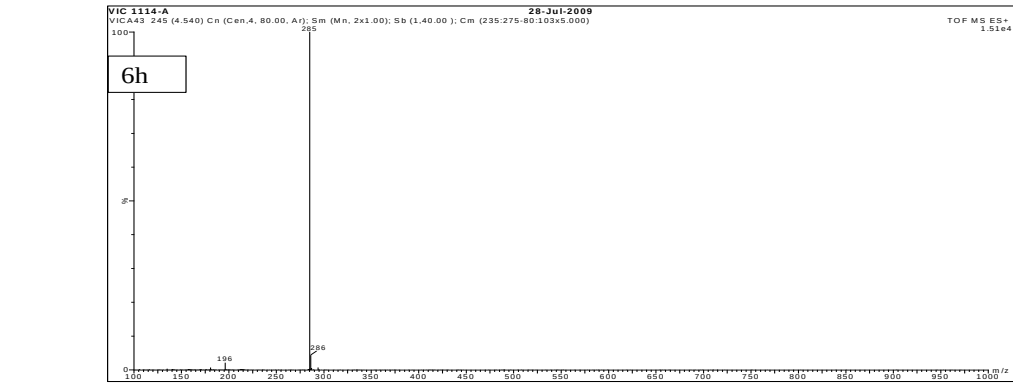
Figure S104. DEPT of compound 6h in DMSO



VIC1131 24-Dec-2009
VICA57 224 (4.153) Cn (Cen,4, 80.00, Ar); Sm (Mn, 2x1.00); Sb (1,40.00); Cm (217:236-83:198x3.000)

No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Inten
1:	271	4.31e3	30.85	17.31													
2:	315	3.23e3	23.14	12.98													
3:	316	2.55e2	1.83	1.02													
4:	371	1.40e4	100.00	56.10													
5:	372	7.06e2	5.05	2.83													

Figure S105. QTOF-MS of compound 6h



VIC 1114-A 28-Jul-2009

VICA43 184 (3.409) Cn (Cen,4, 80.00, Ar); Sm (Mn, 2x1.00); Sb (1,40.00); Cm (184:251-58:126)

No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TI
1:	141	3.19e3	11.00	4.51															
2:	158	1.12e4	38.80	15.93															
3:	159	6.61e2	2.28	0.94															
4:	160	4.52e2	1.56	0.64															
5:	196	7.24e2	2.50	1.02															
6:	214	1.33e4	45.90	18.84															
7:	215	1.24e3	4.26	1.75															
8:	216	5.77e2	1.99	0.82															
9:	236	5.20e2	1.79	0.74															
10:	285	2.90e4	100.00	41.04															
11:	286	3.39e3	11.71	4.80															
12:	287	4.04e2	1.39	0.57															

Figure S106. QTOF-MS of compound 6h

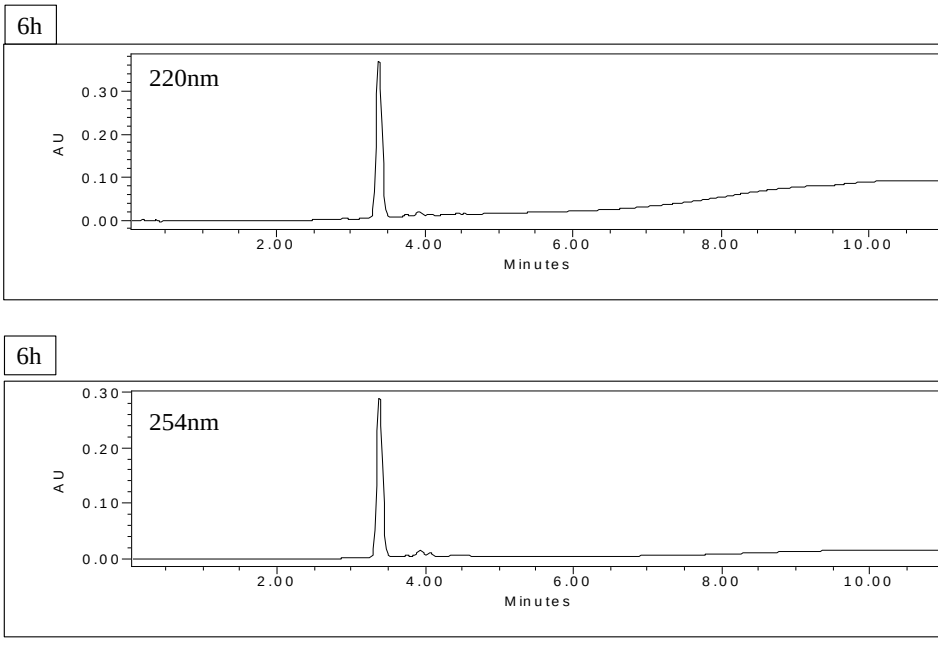
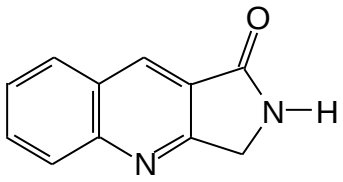


Figure S107. HPLC of compound 6h

5a



^{1}HNMR: (300 MHz, DMSO-d_6), $\delta_{H(ppm)}$: 4.56 (s,2H), 7.68 (t,1H,J=6.9Hz), 7.89 (t,1H,J=6.9Hz), 8.10 (d,1H,J=8.7Hz), 8.21 (d,1H,J=8.7Hz), 8.76 (s,1H), 8.94(bs,N-H) . **^{13}CNMR:** (75 MHz, DMSO-d_6, $\delta_{C(ppm)}$: 46.68, 124.08, 126.62, 127.02, 128.50, 129.87, 131.37, 132.24, 149.12, 163.14, 167.81.(yield 89%) **MS:** MW=184 g/mol, MH^+ =185. **HPLC:** t_R = 3.26min**.**

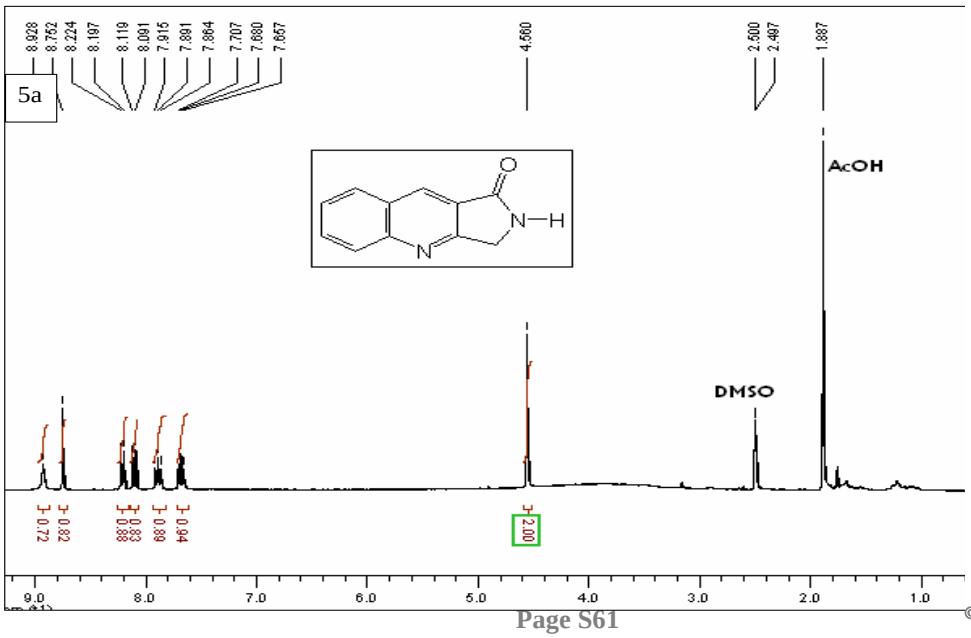


Figure S108. H-NMR of compound 5a in DMSO

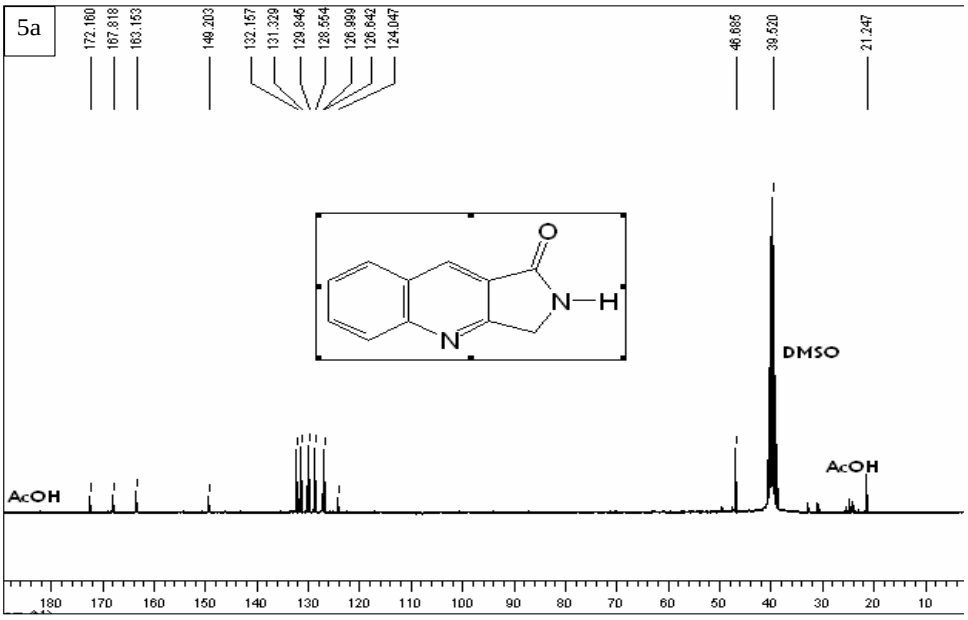


Figure S109. C-NMR of compound 5a in DMSO

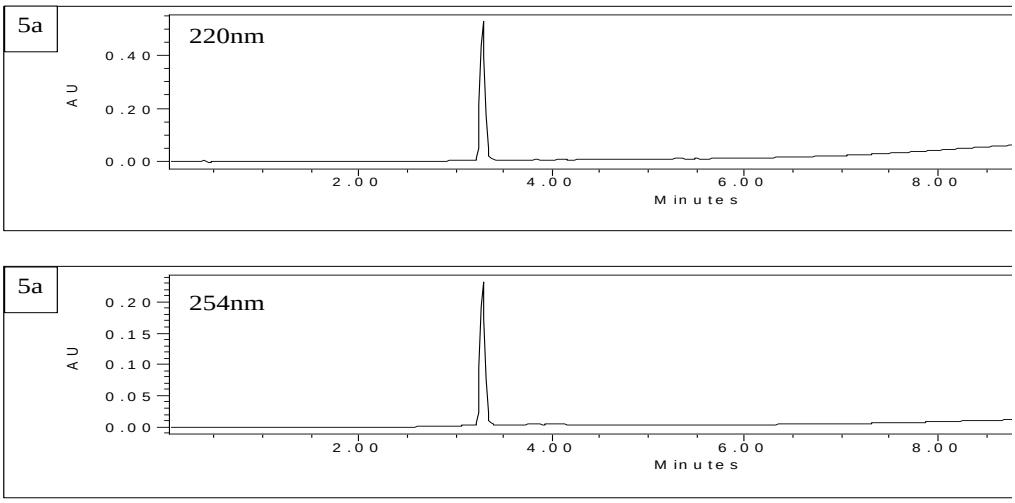


Figure S110. HPLC of compound 5a

5a

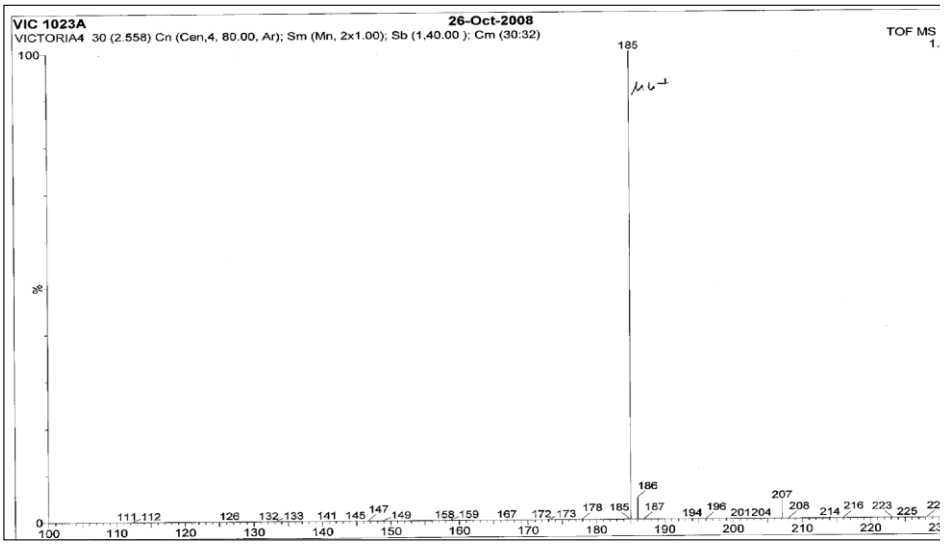


Figure S111. QTOF-MS of compound 5a

5a

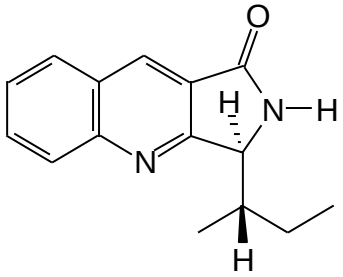
VIC 1023A 26-Oct-2008

VICTORIA4 30 (2.558) Cn (Cen,4, 80.00, Ar); Sm (Mn, 2x1.00); Sb (1,40.00); Cm (30:32) TOF MS

No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass
1:	111	1.00e0	0.00	0.00	49:	186	8.61e3	4.65	4.11												
2:	112	1.00e0	0.00	0.00	50:	187	1.25e2	0.07	0.06												
3:	126	1.00e0	0.00	0.00	51:	188	1.00e0	0.00	0.00												
4:	132	8.00e0	0.00	0.00	52:	190	7.00e0	0.00	0.00												
5:	132	1.00e0	0.00	0.00	53:	194	3.30e1	0.02	0.02												
6:	133	1.00e0	0.00	0.00	54:	195	1.00e0	0.00	0.00												
7:	136	1.00e0	0.00	0.00	55:	196	2.00e0	0.00	0.00												
8:	140	1.00e0	0.00	0.00	56:	199	1.00e0	0.00	0.00												
9:	141	7.00e0	0.00	0.00	57:	199	1.00e0	0.00	0.00												
10:	141	3.00e0	0.00	0.00	58:	201	1.10e1	0.01	0.01												
11:	145	2.00e0	0.00	0.00	59:	203	1.00e0	0.00	0.00												
12:	145	3.00e0	0.00	0.00	60:	204	2.20e1	0.01	0.01												
13:	147	1.00e0	0.00	0.00	61:	207	7.14e3	3.86	3.41												
14:	149	1.00e0	0.00	0.00	62:	208	1.63e2	0.09	0.08												
15:	153	1.00e0	0.00	0.00	63:	209	7.00e0	0.00	0.00												
16:	156	1.00e0	0.00	0.00	64:	212	7.00e0	0.00	0.00												
17:	158	5.10e1	0.03	0.02	65:	213	1.00e0	0.00	0.00												
18:	159	3.00e1	0.02	0.01	66:	214	8.89e-1	0.00	0.00												
19:	160	1.00e0	0.00	0.00	67:	214	2.06e2	0.11	0.10												
20:	161	1.00e0	0.00	0.00	68:	214	6.43e1	0.03	0.03												
21:	167	1.00e0	0.00	0.00	69:	215	3.00e0	0.00	0.00												
22:	172	4.00e0	0.00	0.00	70:	216	1.58e2	0.09	0.08												
23:	173	1.00e0	0.00	0.00	71:	216	2.00e0	0.00	0.00												
24:	173	2.00e0	0.00	0.00	72:	217	3.00e0	0.00	0.00												
25:	174	1.00e0	0.00	0.00	73:	217	2.00e0	0.00	0.00												
26:	178	2.00e0	0.00	0.00	74:	217	1.00e0	0.00	0.00												
27:	183	2.00e0	0.00	0.00	75:	219	1.00e0	0.00	0.00												
28:	184	1.22e0	0.00	0.00	76:	221	1.00e0	0.00	0.00												
29:	184	7.22e0	0.00	0.00	77:	222	1.00e0	0.00	0.00												
30:	184	4.00e0	0.00	0.00	78:	223	7.00e0	0.00	0.00												
31:	184	7.11e0	0.00	0.00	79:	224	1.00e0	0.00	0.00												
32:	184	2.00e0	0.00	0.00	80:	225	5.00e0	0.00	0.00												
33:	184	6.22e0	0.00	0.00	81:	225	2.00e0	0.00	0.00												
34:	184	5.67e0	0.00	0.00	82:	225	8.89e-1	0.00	0.00												
35:	184	3.78e0	0.00	0.00	83:	225	5.43e1	0.03	0.03												
36:	184	2.89e0	0.00	0.00	84:	226	1.01e1	0.01	0.00												
37:	184	1.69e1	0.01	0.01	85:	226	8.89e-1	0.00	0.00												
38:	184	3.78e0	0.00	0.00	86:	226	2.00e0	0.00	0.00												
39:	185	1.17e1	0.01	0.01	87:	227	1.30e1	0.01	0.01												
40:	185	2.33e1	0.01	0.01	88:	228	2.47e1	0.01	0.01												
41:	185	2.04e1	0.01	0.01	89:	228	2.62e1	0.01	0.01												
42:	185	1.20e1	0.01	0.01	90:	229	5.00e0	0.00	0.00												
43:	185	5.64e1	0.03	0.03	91:	229	1.00e0	0.00	0.00												
44:	185	3.53e1	0.02	0.02																	
45:	185	1.85e5	100.00	88.47																	
46:	185	1.00e0	0.00	0.00																	
47:	186	1.00e0	0.00	0.00																	
48:	186	1.00e0	0.00	0.00																	

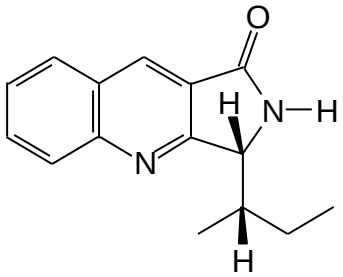
Figure S112. QTOF-MS of compound 5a

5b(major SS)



^{1}HNMR: (300 MHz, DMSO-d_6, $\delta_{H(ppm)}$: 0.49 (d,3H,J=6.6Hz), 0.98 (t,3H, J=7.5Hz), 1.33 (m,1H), 1.60 (m,1H), , 2.24 (m,1H), 4.76 (d,1H,J=2.1Hz), 7.67 (m,1H), 7.87 (m,1H), 8.11 (d,1H,J=8.1Hz), 8.19 (d,1H,J=8.1Hz), 8.71 (s,1H), 9.01 (bs,1H). **^{13}CNMR:** (75 MHz, DMSO-d_6, $\delta_{C(ppm)}$: 11.98, 12.64, 15.42, 26.35, 37.32, 60.82, 83.73, 124.45, 126.63, 128.69, 129.79, 131.28, 131.91, 149.19, 165.49, 167.80. (yield 50.8%) **MS:** MW=240 g/mol, MH^+ =241. **HPLC:** t_R = 3.88min**.**

5b(minor RS)



^{1}HNMR: (300 MHz, DMSO-d_6, $\delta_{H(ppm)}$: 0.77 (t,3H, J=7.5Hz), 0.93 (d,3H,J=7.2Hz), 0.92 (m,1H), 1.17 (m,1H), 2.16 (m,1H), 4.69 (d,1H,J=2.7Hz), 7.67 (m,1H), 7.87 (m,1H), 8.11

(d,1H,J=8.1Hz), 8.19 (d,1H,J=8.1Hz), 8.70 (s,1H), 9.04 (bs,1H). **^{13}CNMR:** (75 MHz, DMSO-d_6, $\delta_{C(ppm)}$: 11.79, 12.64, 15.42, 23.37, 37.55, 62.01, 83.73, 124.62, 127.09, 128.79, 129.79, 131.28, 131.84, 149.06, 165.09, 167.49. (yield 43.2%) **MS:** MW=240 g/mol, MH^+ =241. **HPLC:** t_R =3.88 min**.**

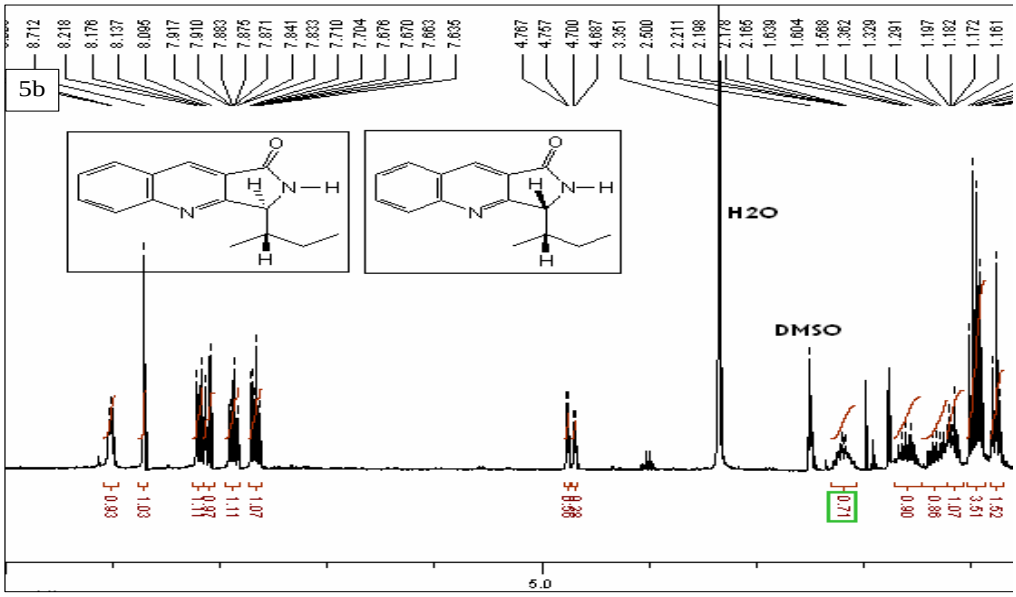


Figure S113. H-NMR of compound 5b in DMSO

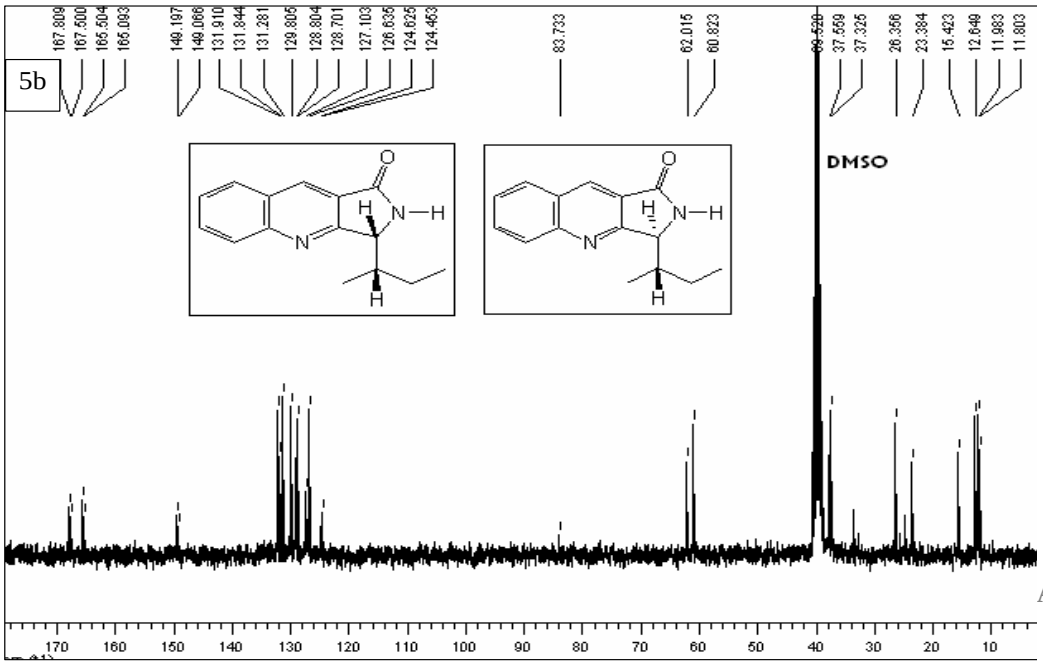


Figure S114. C-NMR of compound 5b in DMSO

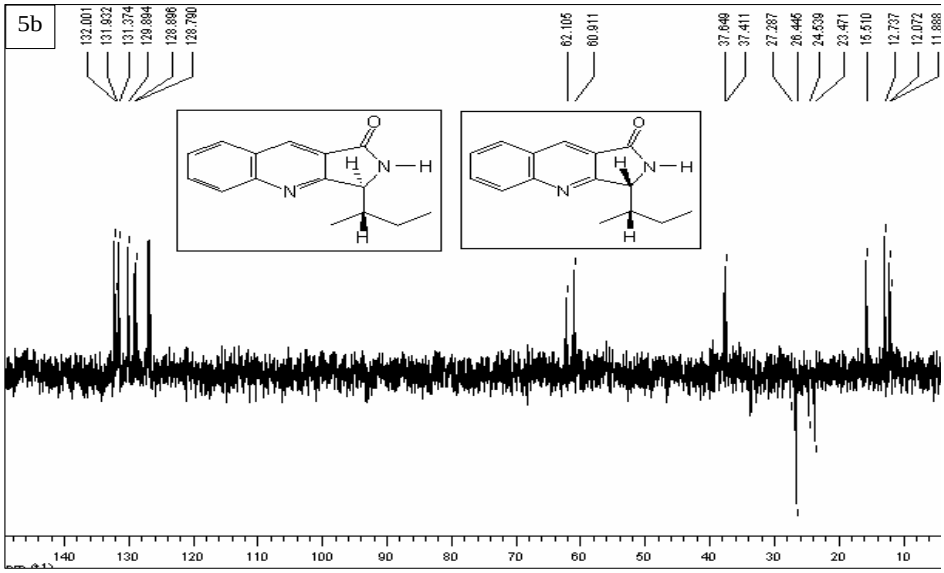
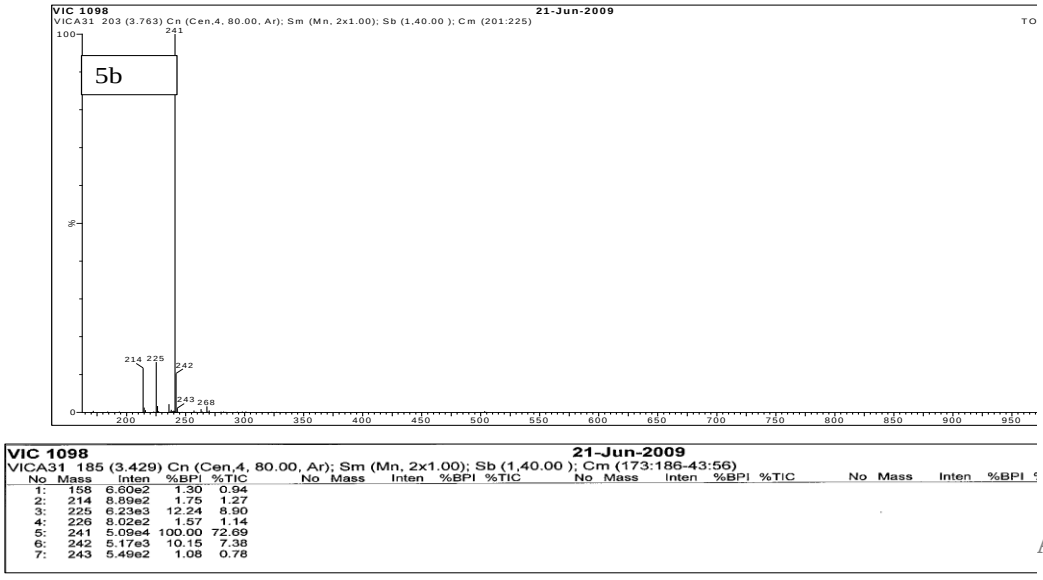


Figure S115. DEPT of compound 5b in DMSO



VIC 1098 21-Jun-2009
VICA31 185 (3.429) Cn (Cen,4, 80.00, Ar); Sm (Mn, 2x1.00); Sb (1,40.00); Cm (173:186-43:56)

No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI
1:	158	6.60e2	1.30	0.94														
2:	214	8.89e2	1.75	1.27														
3:	225	6.23e3	12.24	8.90														
4:	226	8.02e2	1.57	1.14														
5:	241	5.09e4	100.00	72.69														
6:	242	5.17e3	10.15	7.38														
7:	243	5.49e2	1.08	0.78														

Figure S116. QTOF-MS of compound 5b

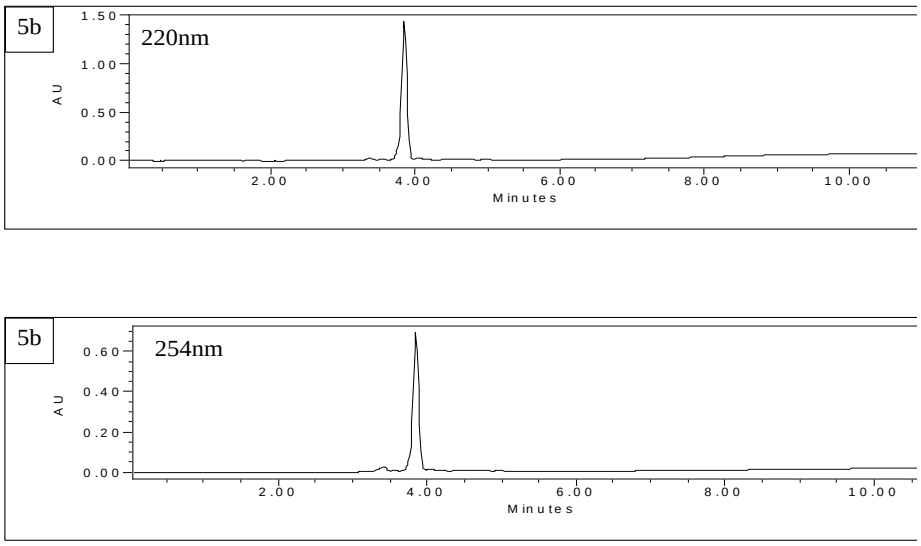
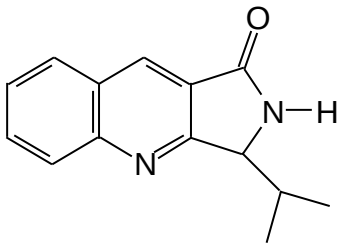


Figure S117. HPLC of compound 5b

5c



[1]HNMR: (300 MHz, DMSO-d_6), $\delta_{H(ppm)}$: 0.65 (d,3H, J=6.9Hz), 1.06 (d,3H,J=6.9Hz), 2.43 (m,1H), 4.65 (d,1H,J=3.3Hz), 7.67 (m,1H), 7.87 (m,1H), 8.11 (d,1H,J=8.4Hz), 8.19

(d,1H,J=8.1Hz), 8.72 (s,1H), 9.04 (bs,1H). **^{13}CNMR:** (75 MHz, DMSO-d_6, $\delta_{C(ppm)}$: 15.75, 19.21, 30.77, 62.48, 124.39, 126.65, 127.09, 128.76, 129.78, 131.26, 131.90, 149.12, 165.15, 167.68. (yield 63%) **MS:** MW=226 g/mol, MH^+ =227. **HPLC:** t_R =3.67 min**.**

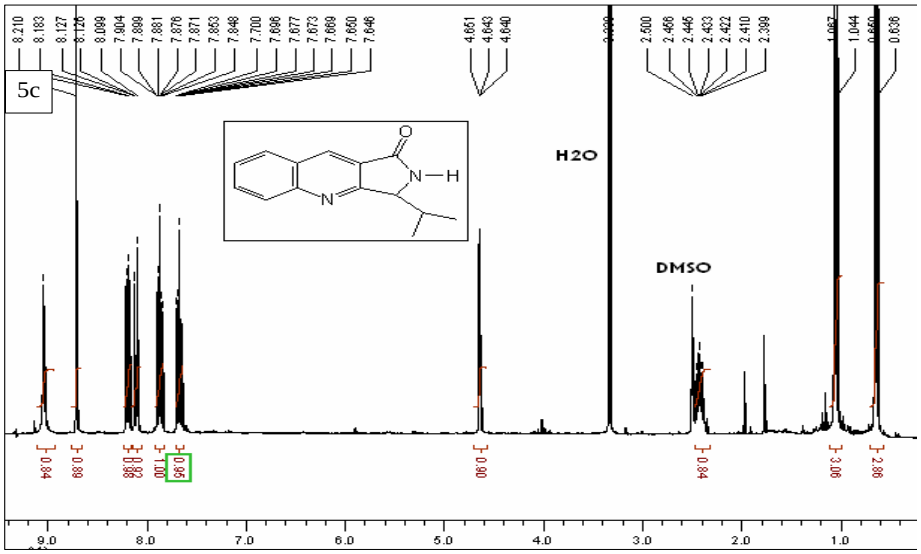


Figure S118. H-NMR of compound 5c in DMSO

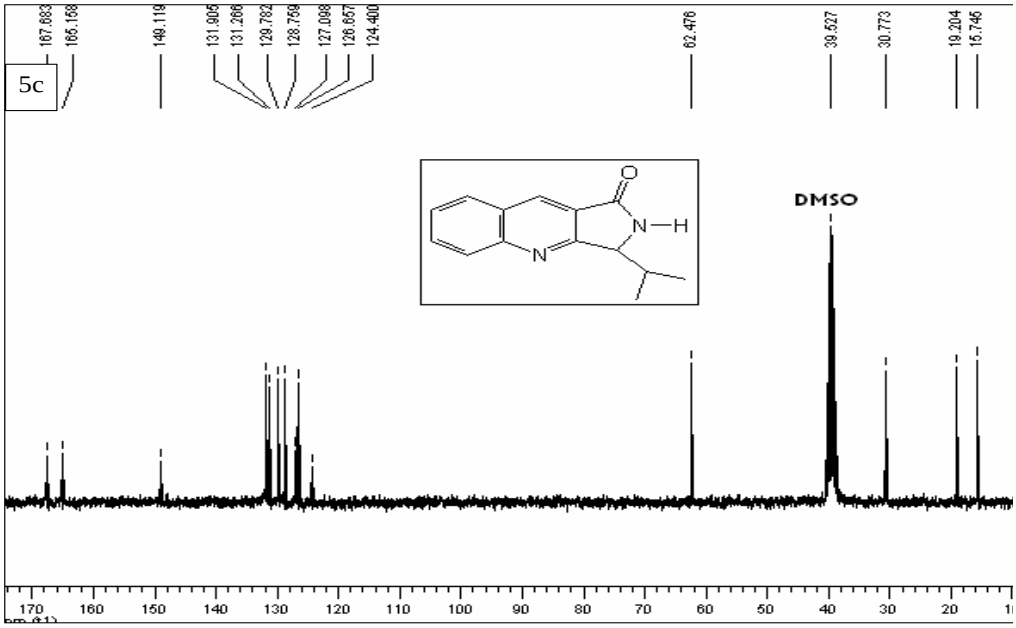


Figure S119. C-NMR of compound 5c in DMSO

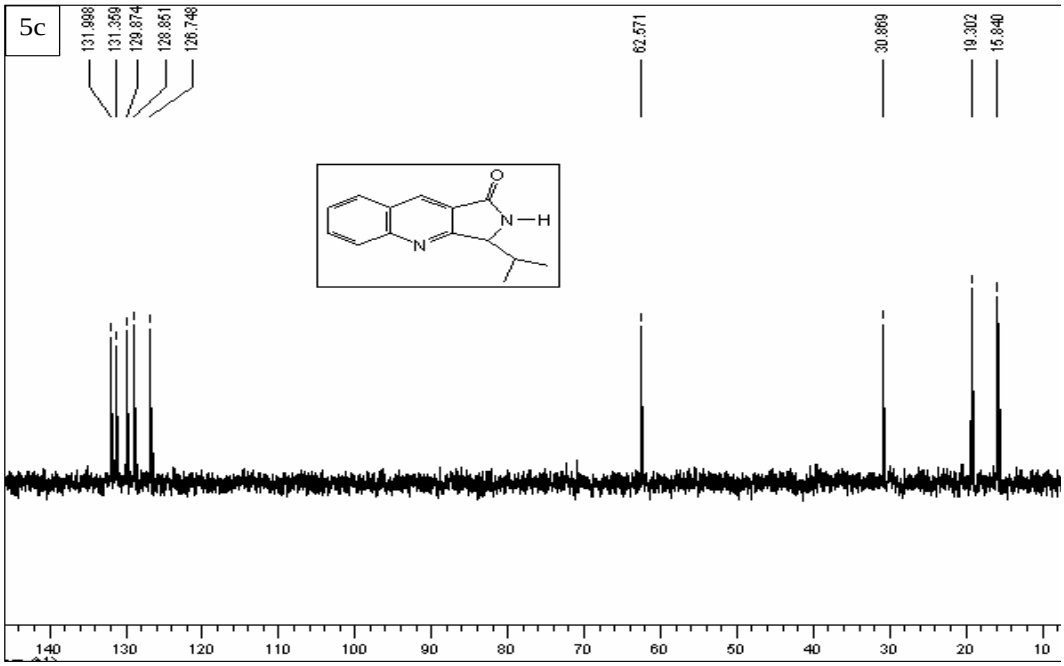
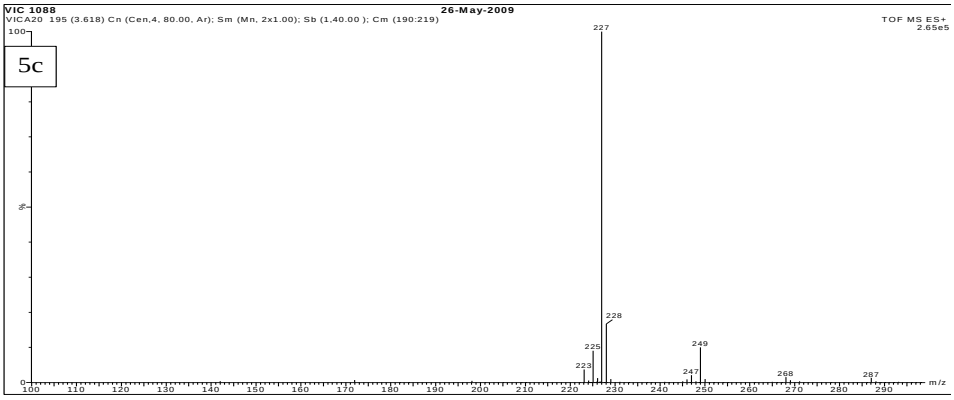


Figure S120. DEPT of compound 5c in DMSO



VIC 1088 **26-May-2009**

VICA20 157 (2.913) Cn (Cen,4, 80.00, Ar); Sm (Mn, 2x1.00); Sb (1,40.00); Cm (154:180-15:53)

No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%B
1:	223	7.74e3	2.97	1.66														
2:	225	1.87e4	7.19	4.02														
3:	227	2.60e5	100.00	55.99														
4:	228	4.34e4	16.68	9.34														
5:	247	4.83e3	1.86	1.04														
6:	249	2.08e4	7.98	4.47														

Figure S121. QTOF-MS of compound 5c

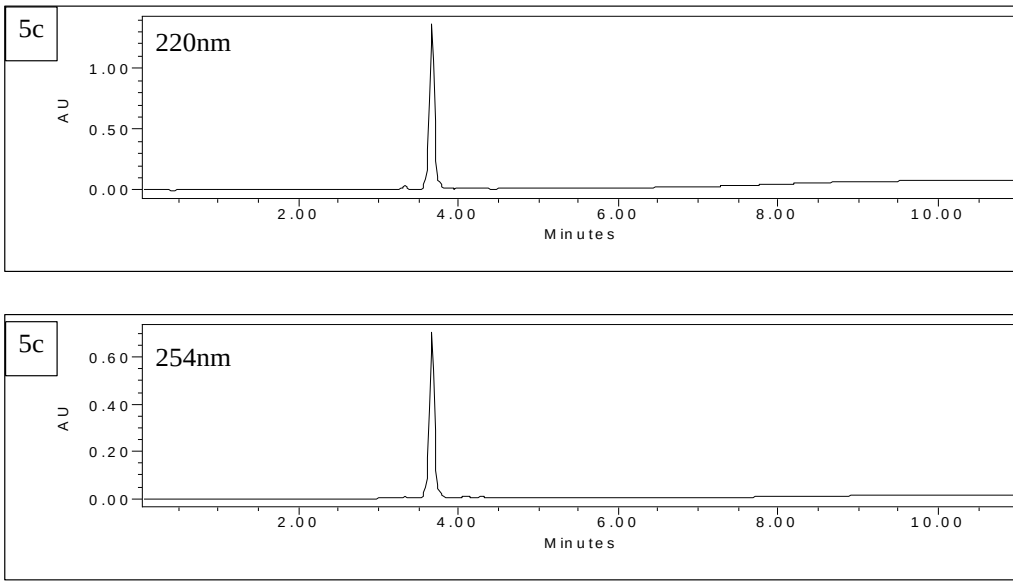
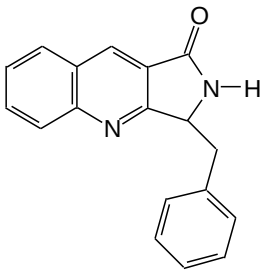


Figure S122. HPLC of compound 5c

5d



^{1}HNMR: (300 MHz, DMSO-d_6), $\delta_{H(ppm)}$: 3.25 (m,2H), 5.09 (t,1H,J=4.8Hz), 7.05 (m,5H),7.67 (m,1H), 7.92 (m,1H), 8.15 (d,1H,J=8.7Hz), 8.19 (m,1H,J=8.1Hz), 8.56 (s.1H), 9.01 (NH, bs) . **^{13}CNMR:** (75 MHz, DMSO-d_6, $\delta_{C\ (ppm)}$:38.07, 57.94, 124.21, 126.26,

126.71, 127.00, 127.72, 128.69, 129.76, 131.32, 131.88, 135.81, 149.04, 164.88, 166.96.

(yield 88.7%)**MS:** MW=274 g/mol, MH^+ =275.

HPLC: t_R = 3.97 min**.**

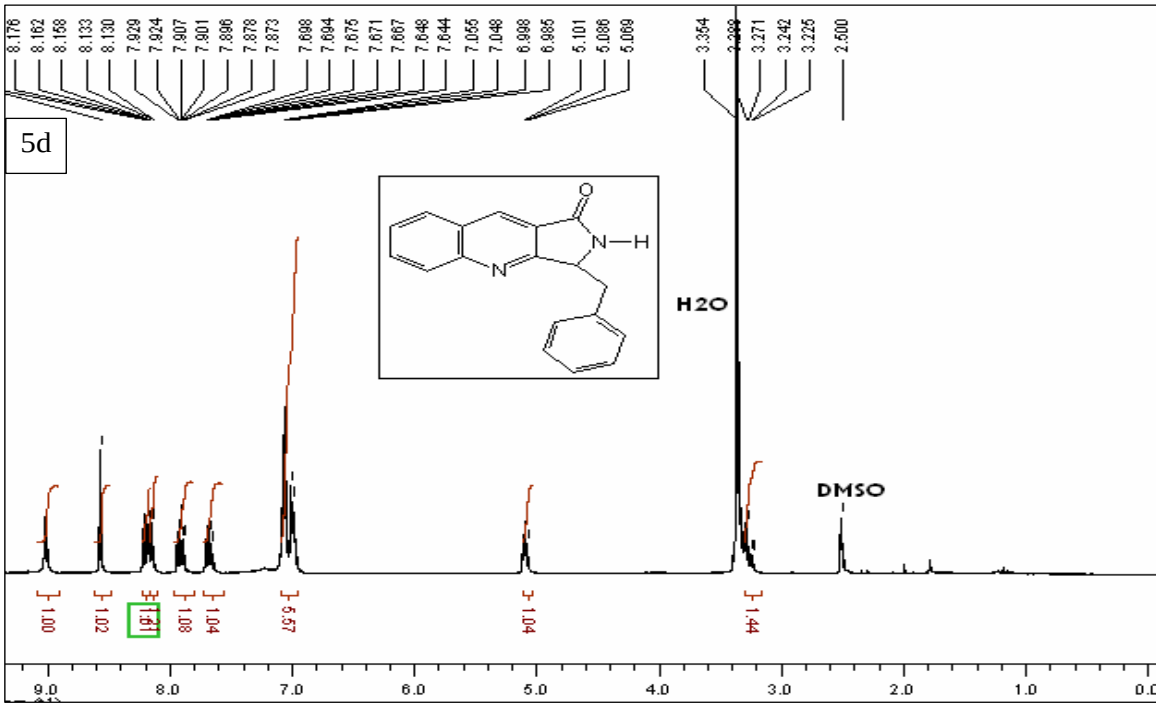


Figure S123. H-NMR of compound 5d in DMSO

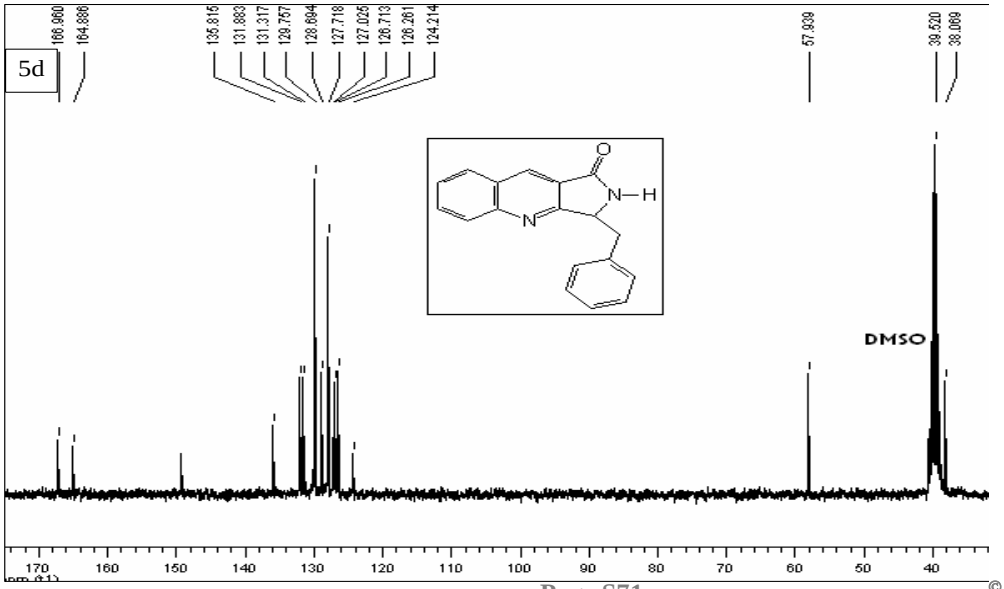


Figure S124. C-NMR of compound 5d in DMSO

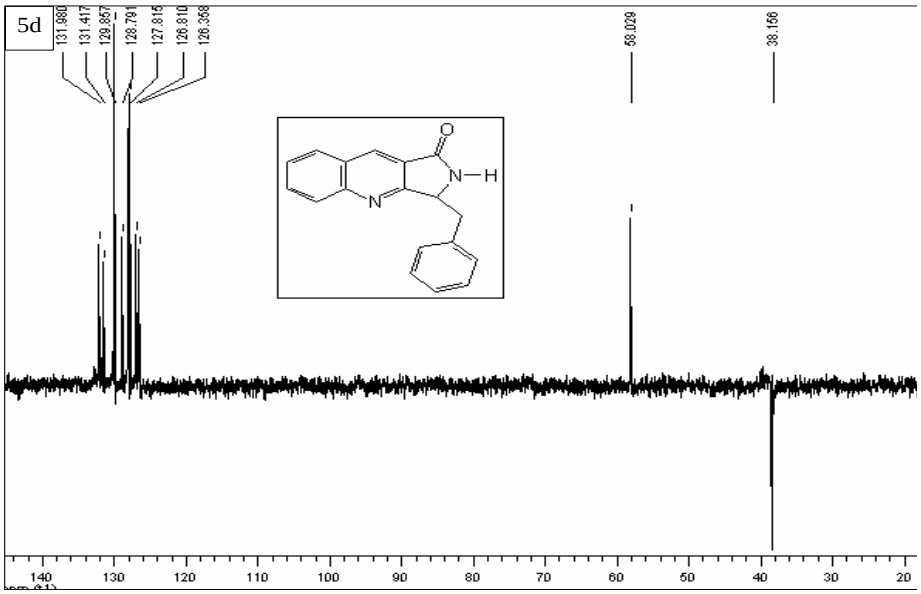


Figure S125. DEPT of compound 5d in DMSO

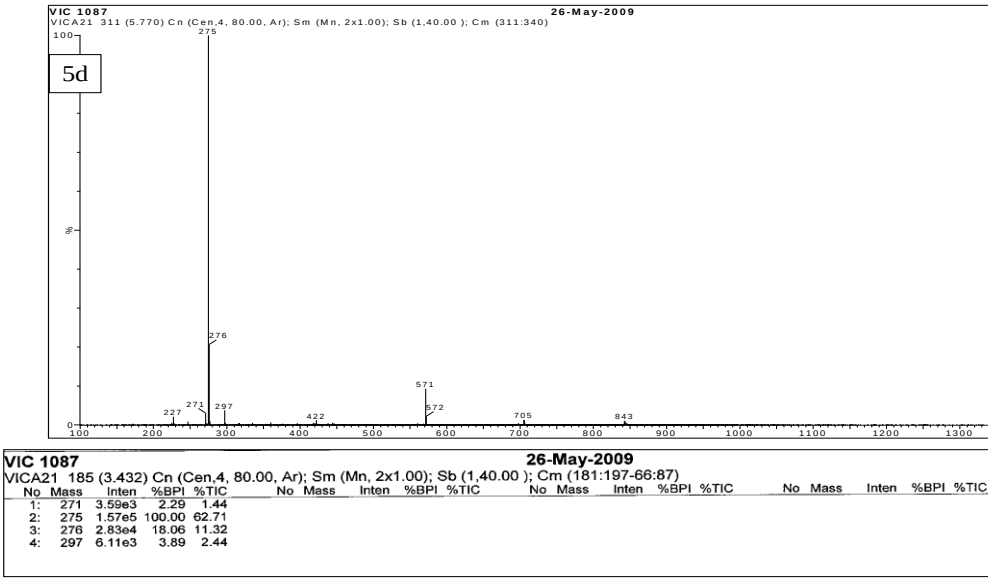


Figure S126. QTOF-MS of compound 5d

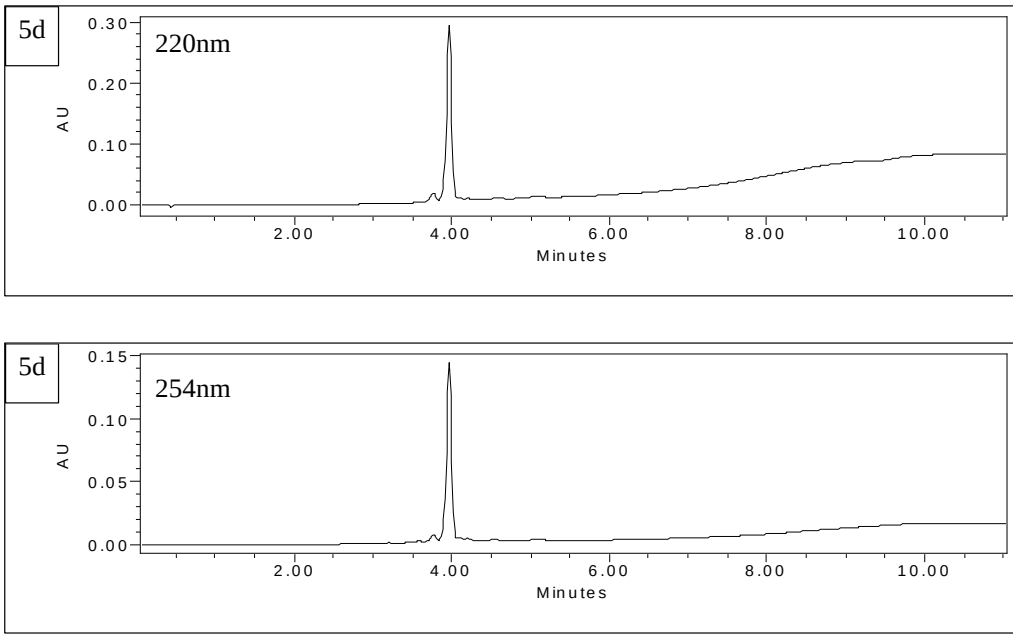
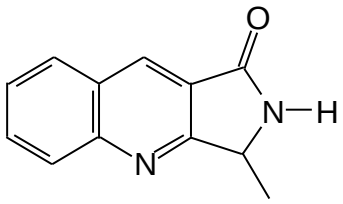


Figure S127. HPLC of compound 5d

5e



^{1}HNMR: (300 MHz, DMSO-d_6), $\delta_{H(ppm)}$: 1.49 (d,3H, J=6.6Hz), 4.78 (q,1H, J=6.6Hz), 7.67 (m,1H), 7.88 (m,1H), 8.12 (d, 1H,J=8.1Hz), 8.19 (d, 1H,J=8.1Hz), 8.73 (s, 1H), 9.02 (bs,1H).

^{13}CNMR: (75 MHz, DMSO-d_6, $\delta_{C\ (ppm)}$:19.25, 53.35, 123.46, 126.67, 127.15, 128.64, 129.79, 131.29, 132.33, 149.16, 166.72. (yield 54.7%)**MS:** MW=198 g/mol, MH^+ =199. **HPLC:** t_R = 3.29 min**.**

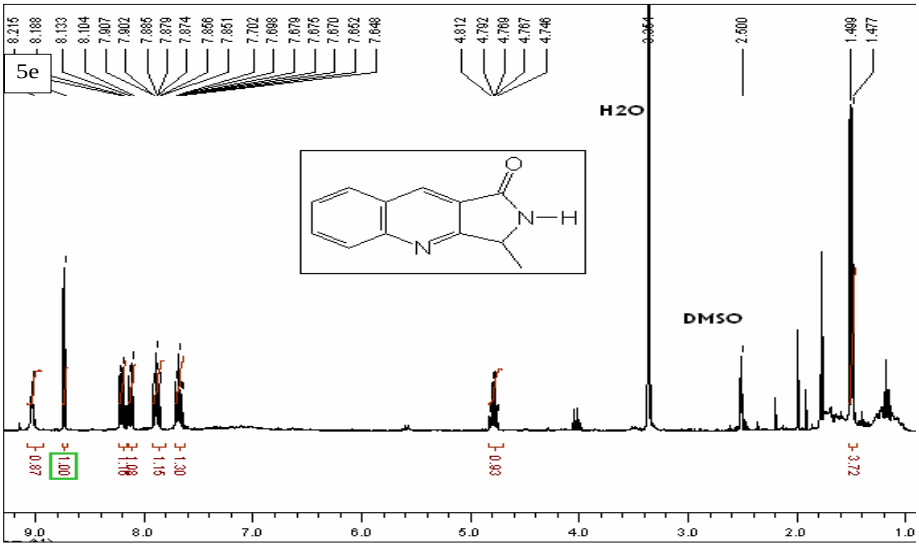


Figure S128. H-NMR of compound 5e in DMSO

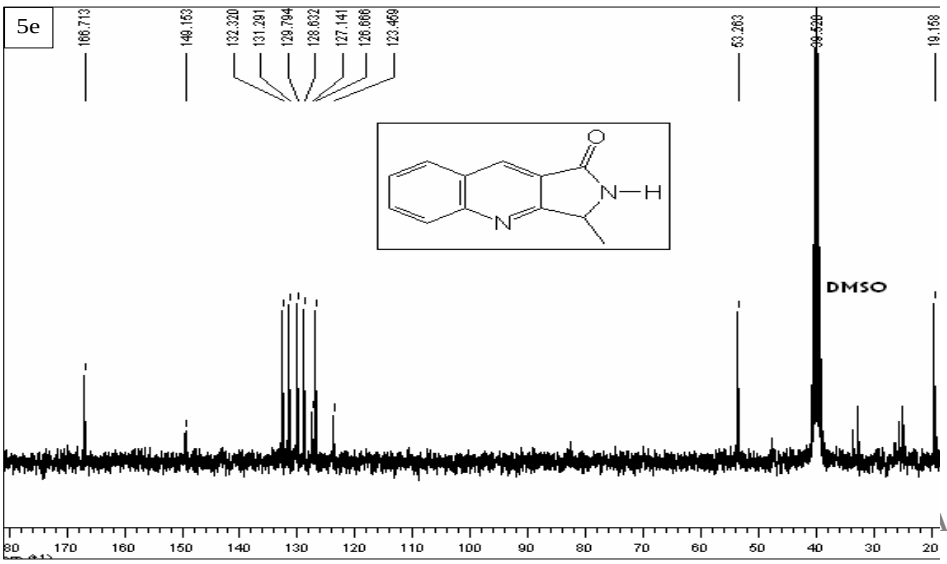


Figure S129. C-NMR of compound 5e in DMSO

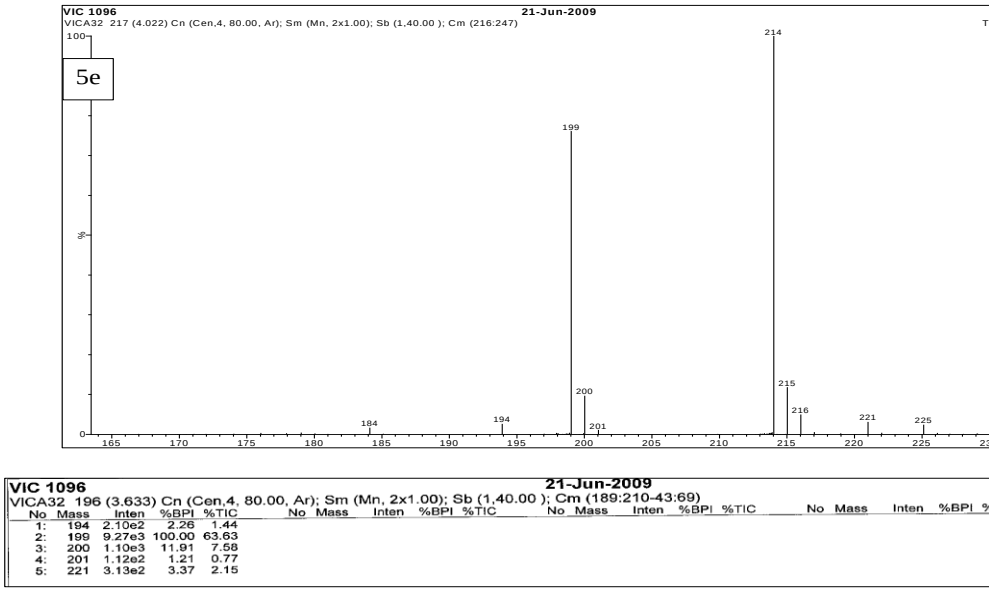


Figure S130. QTOF-MS of compound 5e

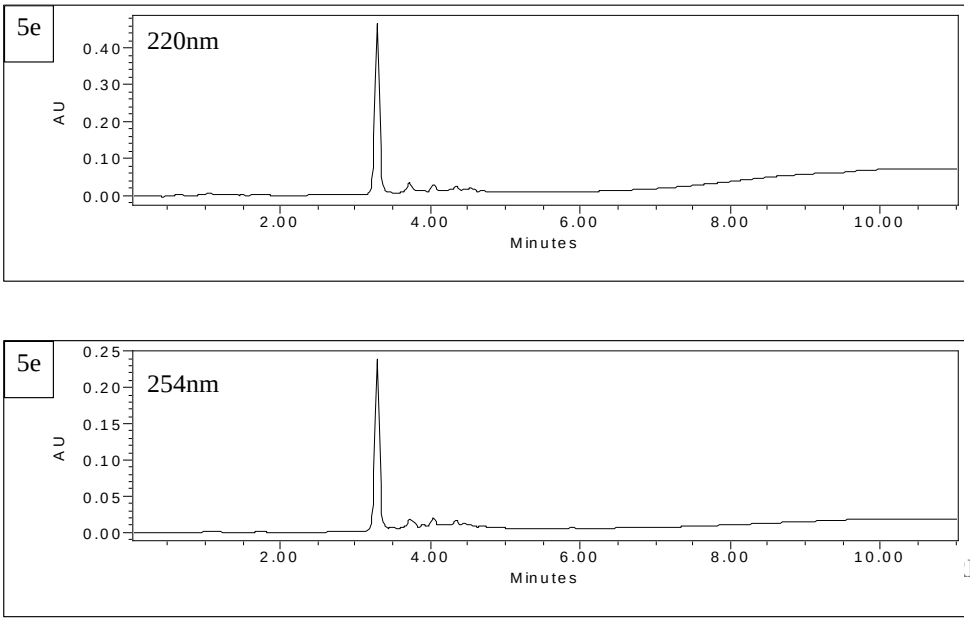
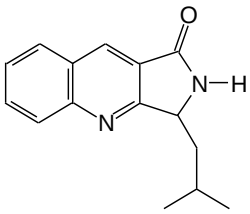


Figure S131. HPLC of compound 5e

5f



^{1}HNMR: (300 MHz, DMSO-d_6), $\delta_{H(ppm)}$: 0.89 (d,3H,J=6.6Hz), 1.01 (d,3H, J=6.6Hz), 1.46 (m,1H), 1.88 (m,1H), 2.20 (m,1H), 4.73 (dd,1H, J=8.7Hz,J=3.9Hz), 7.67(m,1H), 7.87 (m, 1H), 8.12 (d,1H,J=8.1Hz), 8.19 (d,1H,J=8.1Hz), 8.72 (s,1H), 9.16 (bs,1H, NH). **^{13}CNMR:** (75 MHz, DMSO-d_6, $\delta_{C(ppm)}$:21.81, 23.47, 24.53, 39.55, 43.10, 55.90, 123.69, 126.65, 127.17, 128.69, 129.80, 131.29, 132.25, 149.16, 166.37, 167.19. (yield 95.8%) **MS:** MW=240 g/mol, MH^+ =241. **HPLC:** t_R = 3.91 min**.**

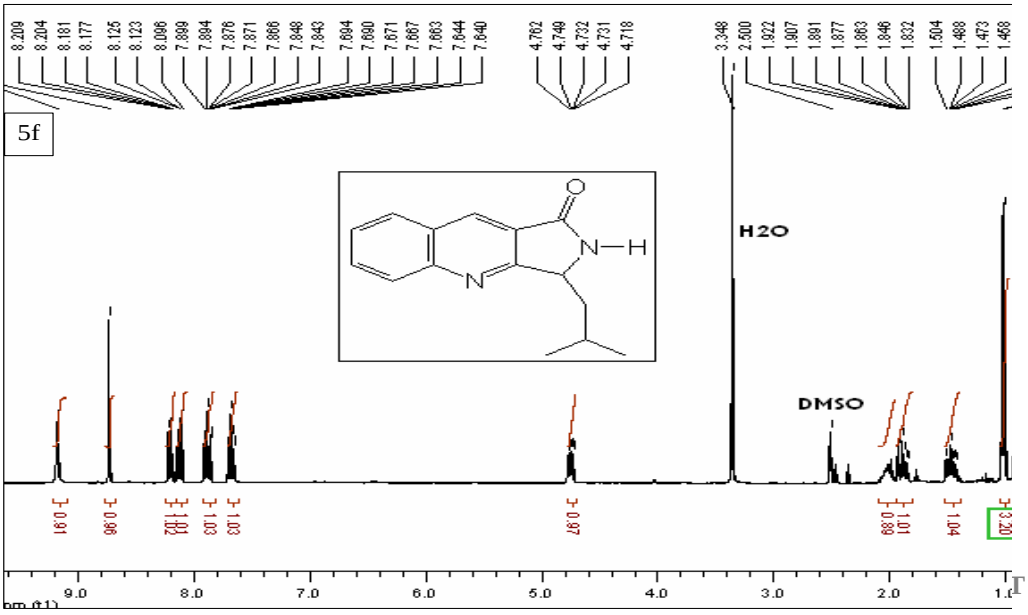


Figure S132. H-NMR of compound 5f in DMSO

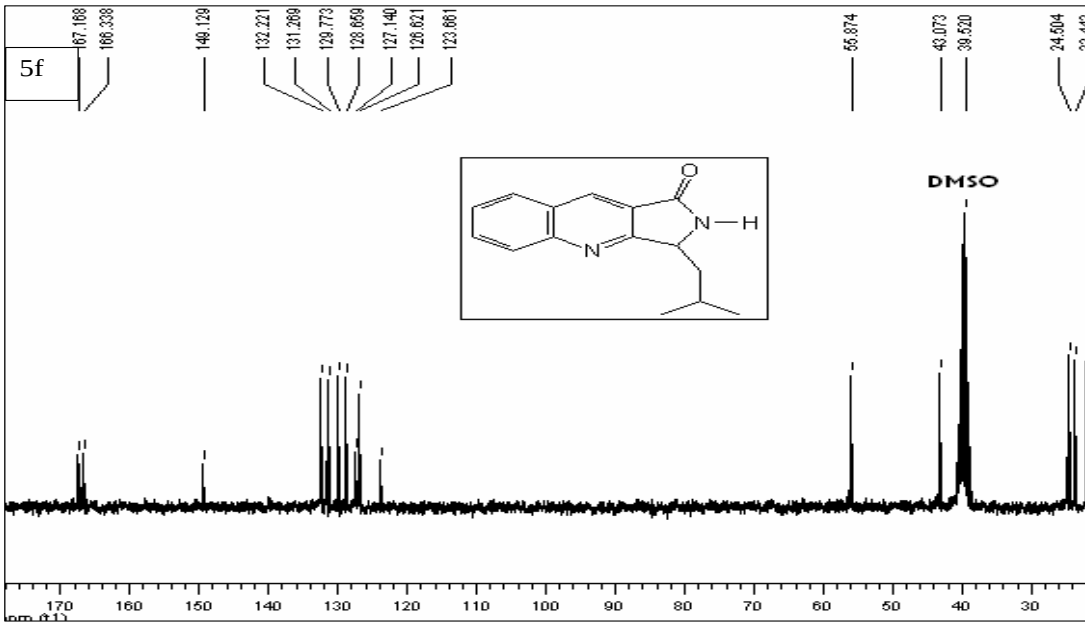


Figure S133. C-NMR of compound 5f in DMSO

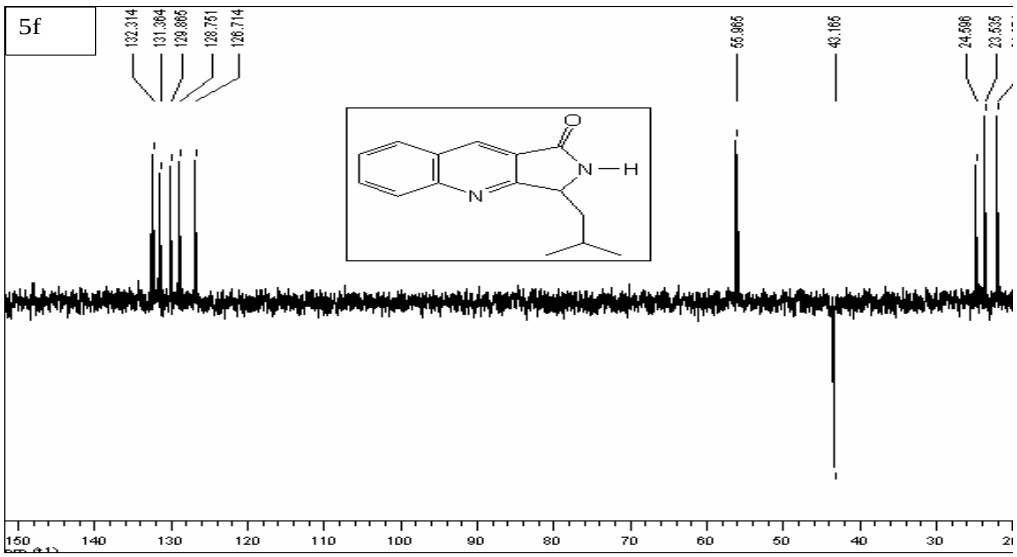
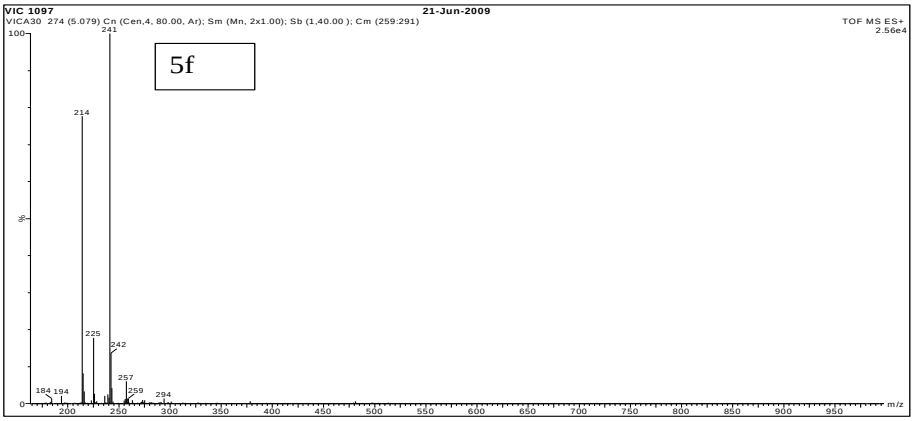


Figure S134. DEPT of compound 5f in DMSO



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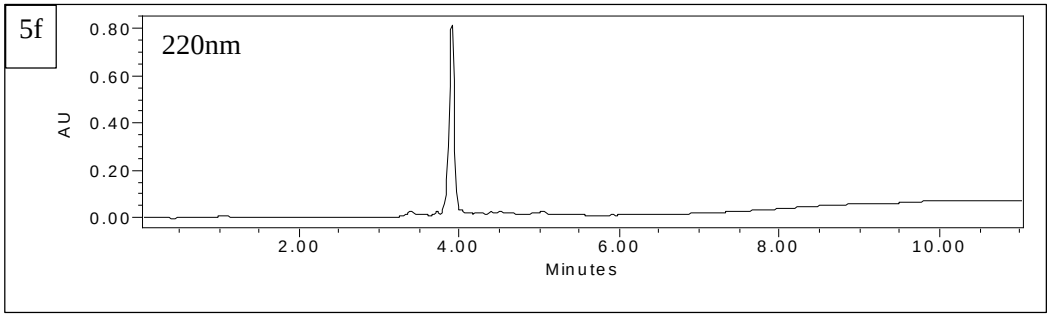
VICA30 274 (5.079) Cn (Cen,4, 80.00, Ar); Sm (Mn, 2x1.00); Sb (1,40.00); Cm (233:298-32:76)

No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TIC	No	Mass	Inten	%BPI	%TI
1:	141	6.17e3	12.98	5.42															
2:	158	2.05e4	43.11	18.01															
3:	159	1.35e3	2.84	1.19															
4:	160	9.53e2	2.00	0.84															
5:	214	1.12e4	23.44	9.79															
6:	215	1.49e3	3.13	1.31															
7:	216	8.59e2	1.81	0.75															
8:	225	1.73e3	3.65	1.52															
9:	236	5.94e2	1.25	0.52															
10:	239	6.21e2	1.31	0.55															
11:	241	4.76e4	100.00	41.78															
12:	242	6.08e3	12.79	5.34															
13:	243	1.43e3	3.00	1.25															
14:	257	2.64e3	5.55	2.32															
15:	294	6.93e2	1.46	0.61															

Figure S135. QTOF-MS of compound 5f



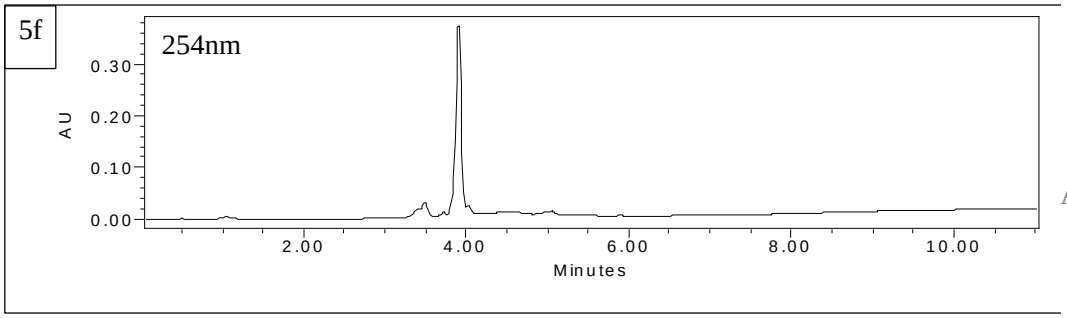
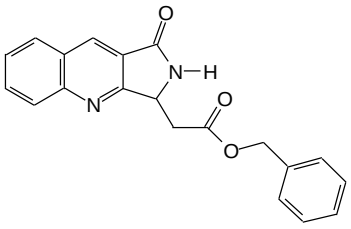


Figure S136. HPLC of compound 5f

5g



[1]HNMR: (300 MHz, DMSO-d_6), $\delta_{H(ppm)}$: 3.00 (dd,1H,J=6.6Hz, J=16.2Hz), 3.13 (dd,1H,J=5.1Hz, J=16.2Hz), 5.04 (d, 2H, J=1.8Hz), 5.08 (d,1H, J=5.7Hz), 7.26 (m,5H), 7.69 (m,1H), 7.89 (m, 1H), 8.09 (d,1H, J=8.4Hz), 8.20 (dd,1H, J=1.5Hz, J=8.4Hz), 8.71 (s,1H), 9.06 (bs, NH). **[13]CNMR:** (75 MHz, DMSO-d_6), $\delta_{C(ppm)}$:37.35, 54.15, 65.73, 123.91, 126.73, 127.21, 127.81, 127.90, 128.25, 128.59, 129.82, 131.31, 132.18, 135.73, 149.04, 164.74, 167.19, 169.55. (yield 66%) **MS:** MW=332 g/mol, MH^+ =332. **HPLC:** t_R = 4.25 min.

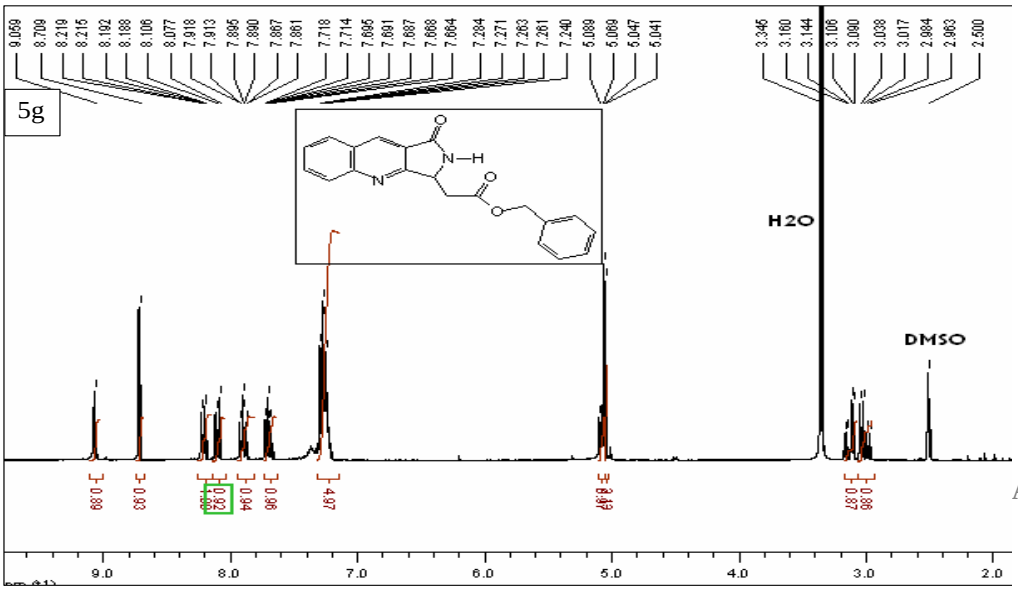


Figure S137. H-NMR of compound 5g in DMSO

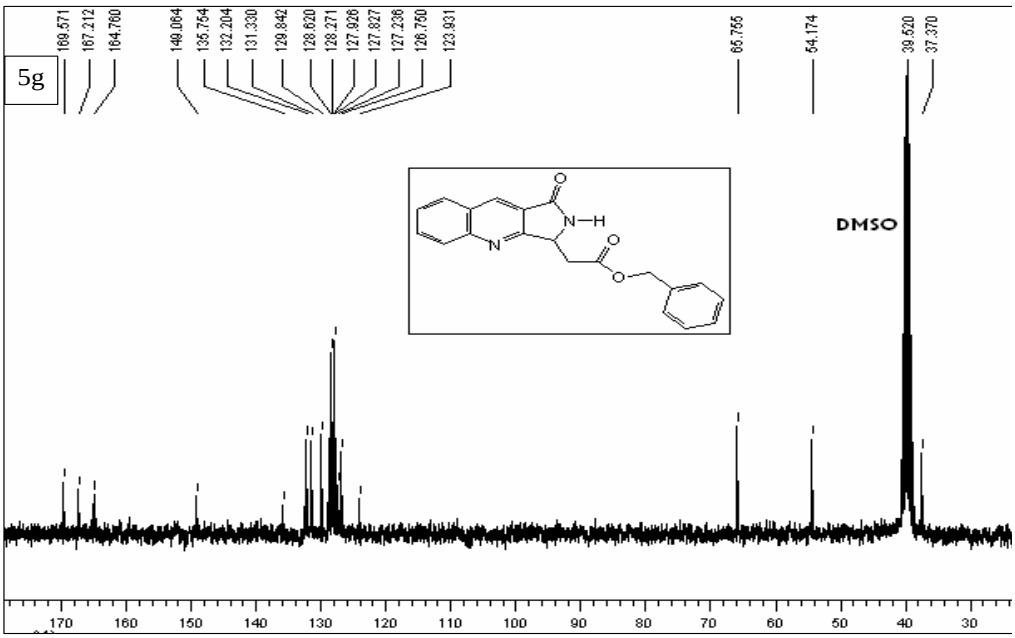


Figure S138. C-NMR of compound 5g in DMSO

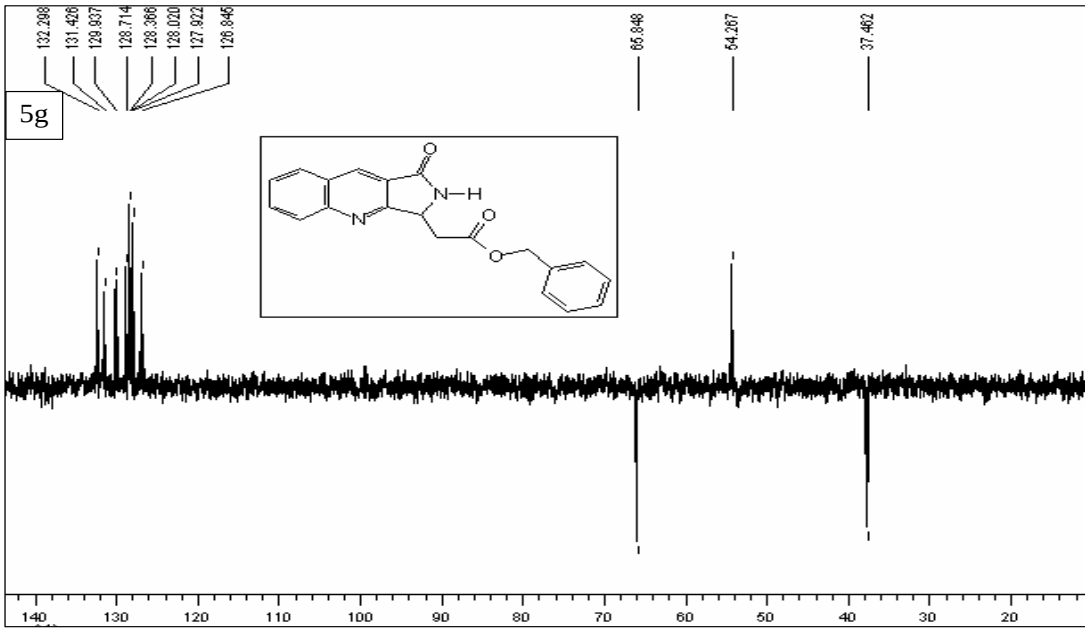


Figure S139. DEPT of compound 5g in DMSO

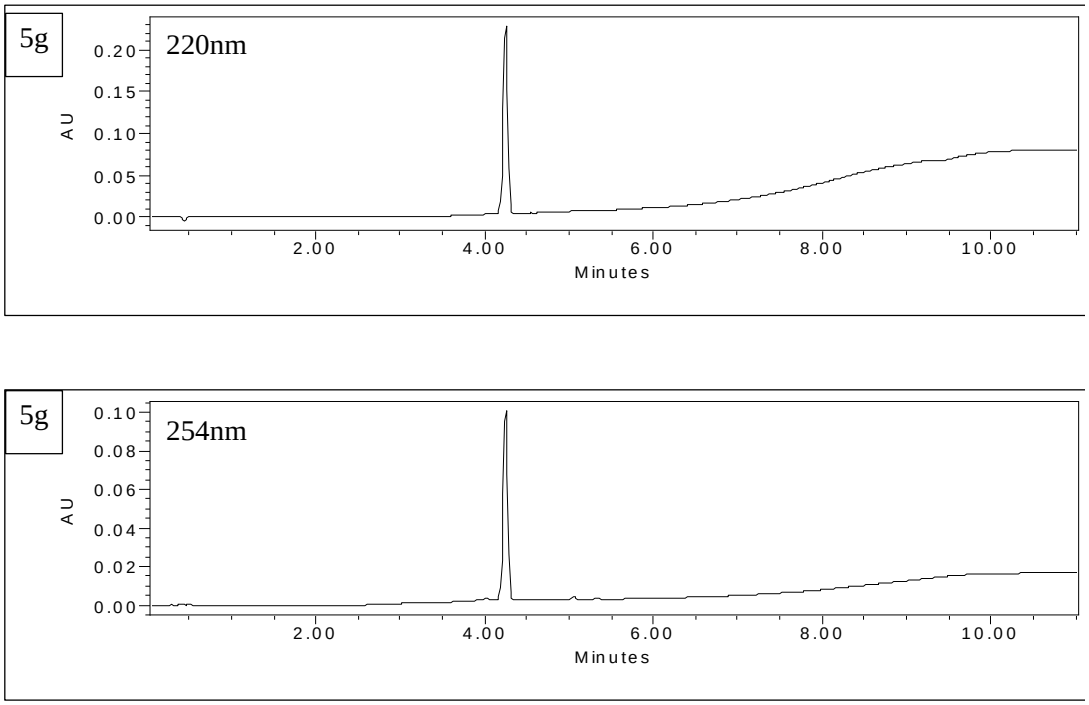


Figure S140. HPLC of compound 5g

