Supporting information

Enantioselective assembly of functionalized carbocyclic spirooxindoles using L-proline derived thiourea organocatalyst

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1. X-ray Crystallographic data for compound (6aa)

CCDC 985882 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

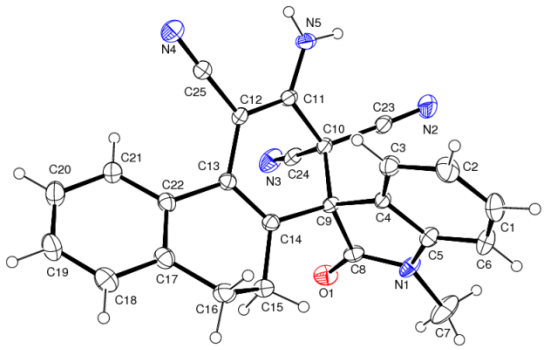


Table 1. Crystal data and structure refinement for Gv-23-19.

Identification code	shelxl
Empirical formula	C25 H17 N5 O
Formula weight	403.44
Temperature	296(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P21/n
Unit cell dimensions	a = 7.4484(2) A alpha = 90 deg. b = 13.8881(5) A beta = 98.3180(10) deg. c = 19.3487(7) A gamma = 90 deg.
Volume	1980.45(11) A^3
Z, Calculated density	4, 1.353 Mg/m^3
Absorption coefficient	0.087 mm^-1
F(000)	840

Crystal size	0.32 x 0.30 x 0.25 mm
Theta range for data collection	1.81 to 26.00 deg.
Limiting indices	-9<=h<=9, -17<=k<=17, -23<=l<=23
Reflections collected / unique	33764 / 33764 [R(int) = 0.0000]
Completeness to theta = 26.00	99.7 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9792 and 0.9717
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	33764 / 4 / 291
Goodness-of-fit on F^2	1.085
Final R indices [I>2sigma(I)]	R1 = 0.0568, wR2 = 0.1395
R indices (all data)	R1 = 0.0786, wR2 = 0.1503
Extinction coefficient	0.0075(4)
Largest diff. peak and hole	0.319 and -0.259 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for gv2319.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
C(1)	2614(2)	6693(1)	663(1)	48(1)
C(2)	2023(2)	7276(1)	1158(1)	44(1)
C(3)	1255(1)	6891(1)	1708(1)	34(1)
C(4)	1107(1)	5907(1)	1748(1)	25(1)
C(5)	1748(1)	5326(1)	1252(1)	30(1)
C(6)	2496(2)	5702(1)	698(1)	44(1)
C(7)	2175(2)	3545(1)	1036(1)	66(1)
C(8)	836(1)	4247(1)	2020(1)	29(1)
C(9)	327(1)	5270(1)	2265(1)	24(1)
C(10)	-1806(1)	5353(1)	2172(1)	26(1)
C(11)	-2402(1)	6252(1)	2548(1)	26(1)
C(12)	-1442(1)	6465(1)	3179(1)	26(1)
C(13)	272(1)	5965(1)	3446(1)	25(1)
C(14)	1136(1)	5442(1)	3015(1)	26(1)
C(15)	3035(1)	5085(1)	3239(1)	35(1)
C(16)	4069(2)	5811(1)	3736(1)	38(1)
C(17)	3024(2)	6022(1)	4327(1)	34(1)
C(18)	3871(2)	6153(1)	5006(1)	52(1)
C(19)	2872(2)	6320(1)	5538(1)	58(1)
C(20)	1010(2)	6344(1)	5409(1)	49(1)
C(21)	142(2)	6230(1)	4730(1)	36(1)
C(22)	1128(1)	6090(1)	4183(1)	28(1)
C(23)	-2514(1)	5389(1)	1417(1)	34(1)
C(24)	-2658(1)	4513(1)	2476(1)	31(1)
C(25)	-2018(1)	7273(1)	3548(1)	30(1)
N(1)	1544(1)	4349(1)	1422(1)	36(1)
N(2)	-3075(1)	5448(1)	844(1)	57(1)
N(3)	-3423(1)	3902(1)	2696(1)	49(1)
N(4)	-2515(1)	7920(1)	3833(1)	51(1)
N(5)	-3887(1)	6714(1)	2238(1)	38(1)
O(1)	638(1)	3499(1)	2330(1)	40(1)

Table 3. Bond lengths [A] and angles [deg] for gv2319.

C(1)-C(2)	1.3760(15)
C(1)-C(6)	1.3811(16)
C(1)-H(1)	0.9300
C(2)-C(3)	1.3849(14)
C(2)-H(2)	0.9300
C(3)-C(4)	1.3753(13)
C(3)-H(3)	0.9300
C(4)-C(5)	1.3902(13)
C(4)-C(9)	1.5132(13)
C(5)-C(6)	1.3792(14)
C(5)-N(1)	1.4092(13)
C(6)-H(6)	0.9300
C(7)-N(1)	1.4593(12)
C(7)-H(7A)	0.9600
C(7)-H(7B)	0.9600
C(7)-H(7C)	0.9600
C(8)-O(1)	1.2193(11)
C(8)-N(1)	1.3464(12)
C(8)-C(9)	1.5621(13)
C(9)-C(14)	1.5077(13)
C(9)-C(10)	1.5767(13)
C(10)-C(23)	1.4804(14)
C(10)-C(24)	1.4905(14)
C(10)-C(11)	1.5433(13)
C(11)-N(5)	1.3421(13)
C(11)-C(12)	1.3547(13)
C(12)-C(25)	1.4299(13)
C(12)-C(13)	1.4798(13)
C(13)-C(14)	1.3383(13)
C(13)-C(22)	1.4859(13)
C(14)-C(15)	1.5024(13)
C(15)-C(16)	1.5232(14)
C(15)-H(15A)	0.9700
C(15)-H(15B)	0.9700
C(16)-C(17)	1.5018(14)
C(16)-H(16A)	0.9700
C(16)-H(16B)	0.9700
C(17)-C(18)	1.3849(15)
C(17)-C(22)	1.4023(14)
C(18)-C(19)	1.3742(17)
C(18)-H(18)	0.9300
C(19)-C(20)	1.3739(17)
C(19)-H(19)	0.9300
C(20)-C(21)	1.3863(15)
C(20)-H(20)	0.9300
C(21)-C(22)	1.3862(14)
C(21)-H(21)	0.9300
C(23)-N(2)	1.1297(13)
C(24)-N(3)	1.1377(13)
C(25)-N(4)	1.1431(12)
N(5)-H(5A)	0.879(10)

N(5)-H(5B)	0.855(10)
C(2)-C(1)-C(6)	121.52(11)
C(2)-C(1)-H(1)	119.2
C(6)-C(1)-H(1)	119.2
C(1)-C(2)-C(3)	121.15(11)
C(1)-C(2)-H(2)	119.4
C(3)-C(2)-H(2)	119.4
C(4)-C(3)-C(2)	118.17(10)
C(4)-C(3)-H(3)	120.9
C(2)-C(3)-H(3)	120.9
C(3)-C(4)-C(5)	119.99(9)
C(3)-C(4)-C(9)	131.28(9)
C(5)-C(4)-C(9)	108.73(8)
C(6)-C(5)-C(4)	122.28(10)
C(6)-C(5)-N(1)	127.99(10)
C(4)-C(5)-N(1)	109.71(9)
C(5)-C(6)-C(1)	116.86(10)
C(5)-C(6)-H(6)	121.6
C(1)-C(6)-H(6)	121.6
N(1)-C(7)-H(7A)	109.5
N(1)-C(7)-H(7B)	109.5
H(7A)-C(7)-H(7B)	109.5
N(1)-C(7)-H(7C)	109.5
H(7A)-C(7)-H(7C)	109.5
H(7B)-C(7)-H(7C)	109.5
O(1)-C(8)-N(1)	127.01(9)
O(1)-C(8)-C(9)	125.07(9)
N(1)-C(8)-C(9)	107.91(8)
C(14)-C(9)-C(4)	113.99(8)
C(14)-C(9)-C(8)	110.82(8)
C(4)-C(9)-C(8)	101.36(8)
C(14)-C(9)-C(10)	110.78(8)
C(4)-C(9)-C(10)	110.98(8)
C(8)-C(9)-C(10)	108.44(8)
C(23)-C(10)-C(24)	107.80(9)
C(23)-C(10)-C(11)	110.61(8)
C(24)-C(10)-C(11)	106.20(8)
C(23)-C(10)-C(9)	108.98(8)
C(24)-C(10)-C(9)	112.17(8)
C(11)-C(10)-C(9)	111.02(8)
N(5)-C(11)-C(12)	126.24(9)
N(5)-C(11)-C(10)	116.84(9)
C(12)-C(11)-C(10)	116.80(9)
C(11)-C(12)-C(25)	117.67(9)
C(11)-C(12)-C(13)	121.96(9)
C(25)-C(12)-C(13)	119.97(9)
C(14)-C(13)-C(12)	120.32(9)
C(14)-C(13)-C(22)	118.88(9)
C(12)-C(13)-C(22)	120.67(8)
C(13)-C(14)-C(15)	121.14(9)
C(13)-C(14)-C(9)	121.62(9)
C(15)-C(14)-C(9)	116.97(8)
C(14)-C(15)-C(16)	109.40(8)

C(14)-C(15)-H(15A)	109.8
C(16)-C(15)-H(15A)	109.8
C(14)-C(15)-H(15B)	109.8
C(16)-C(15)-H(15B)	109.8
H(15A)-C(15)-H(15B)	108.2
C(17)-C(16)-C(15)	109.86(9)
C(17)-C(16)-H(16A)	109.7
C(15)-C(16)-H(16A)	109.7
C(17)-C(16)-H(16B)	109.7
C(15)-C(16)-H(16B)	109.7
H(16A)-C(16)-H(16B)	108.2
C(18)-C(17)-C(22)	119.13(11)
C(18)-C(17)-C(16)	122.20(11)
C(22)-C(17)-C(16)	118.67(9)
C(19)-C(18)-C(17)	120.77(12)
C(19)-C(18)-H(18)	119.6
C(17)-C(18)-H(18)	119.6
C(20)-C(19)-C(18)	120.60(11)
C(20)-C(19)-H(19)	119.7
C(18)-C(19)-H(19)	119.7
C(19)-C(20)-C(21)	119.34(11)
C(19)-C(20)-H(20)	120.3
C(21)-C(20)-H(20)	120.3
C(22)-C(21)-C(20)	120.88(11)
C(22)-C(21)-H(21)	119.6
C(20)-C(21)-H(21)	119.6
C(21)-C(22)-C(17)	119.17(10)
C(21)-C(22)-C(13)	123.23(10)
C(17)-C(22)-C(13)	117.55(9)
N(2)-C(23)-C(10)	177.63(12)
N(3)-C(24)-C(10)	175.22(12)
N(4)-C(25)-C(12)	178.43(12)
C(8)-N(1)-C(5)	111.81(8)
C(8)-N(1)-C(7)	123.66(9)
C(5)-N(1)-C(7)	124.30(9)
C(11)-N(5)-H(5A)	121.4(7)
C(11)-N(5)-H(5B)	120.8(8)
H(5A)-N(5)-H(5B)	117.2(10)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for gv2319. 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
C(1)	57(1)	54(1)	35(1)	11(1)	17(1)	-7(1)
C(2)	58(1)	31(1)	45(1)	9(1)	11(1)	-2(1)
C(3)	39(1)	29(1)	35(1)	0(1)	10(1)	3(1)
C(4)	24(1)	28(1)	23(1)	-2(1)	5(1)	2(1)
C(5)	31(1)	29(1)	32(1)	-5(1)	8(1)	-3(1)
C(6)	49(1)	52(1)	34(1)	-7(1)	21(1)	-6(1)
C(7)	85(1)	38(1)	85(1)	-28(1)	46(1)	1(1)
C(8)	26(1)	23(1)	39(1)	-5(1)	5(1)	1(1)
C(9)	25(1)	21(1)	27(1)	-3(1)	7(1)	2(1)
C(10)	26(1)	28(1)	24(1)	-3(1)	6(1)	2(1)
C(11)	25(1)	26(1)	28(1)	-1(1)	8(1)	3(1)
C(12)	28(1)	27(1)	24(1)	-3(1)	7(1)	2(1)
C(13)	26(1)	25(1)	26(1)	1(1)	4(1)	0(1)
C(14)	28(1)	24(1)	28(1)	2(1)	6(1)	2(1)
C(15)	31(1)	36(1)	37(1)	1(1)	6(1)	7(1)
C(16)	27(1)	44(1)	42(1)	2(1)	4(1)	3(1)
C(17)	38(1)	31(1)	32(1)	-1(1)	0(1)	-2(1)
C(18)	44(1)	66(1)	41(1)	-4(1)	-8(1)	4(1)
C(19)	70(1)	70(1)	29(1)	-7(1)	-8(1)	12(1)
C(20)	65(1)	53(1)	29(1)	1(1)	10(1)	8(1)
C(21)	41(1)	38(1)	30(1)	1(1)	7(1)	5(1)
C(22)	34(1)	23(1)	26(1)	2(1)	2(1)	0(1)
C(23)	30(1)	42(1)	29(1)	-9(1)	5(1)	6(1)
C(24)	27(1)	33(1)	35(1)	-9(1)	11(1)	1(1)
C(25)	33(1)	31(1)	28(1)	-1(1)	4(1)	4(1)
N(1)	41(1)	26(1)	45(1)	-12(1)	19(1)	1(1)
N(2)	54(1)	84(1)	32(1)	-10(1)	3(1)	16(1)
N(3)	53(1)	40(1)	61(1)	-6(1)	28(1)	-7(1)
N(4)	64(1)	45(1)	43(1)	-11(1)	7(1)	17(1)
N(5)	37(1)	42(1)	33(1)	-9(1)	-3(1)	16(1)
O(1)	42(1)	25(1)	55(1)	3(1)	8(1)	-2(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for gv2319.

	x	y	z	U(eq)
H(1)	3105	6972	295	57
H(2)	2140	7940	1124	53
H(3)	851	7288	2040	41
H(6)	2902	5307	365	52
H(7A)	2066	2957	1288	99
H(7B)	3422	3645	982	99
H(7C)	1451	3502	584	99
H(15A)	3645	5000	2833	41
H(15B)	3001	4467	3470	41
H(16A)	5252	5553	3920	45
H(16B)	4245	6401	3486	45
H(18)	5130	6128	5103	62
H(19)	3463	6417	5990	69
H(20)	338	6435	5773	58
H(21)	-1118	6249	4640	44
H(5A)	-4433(14)	6543(8)	1823(5)	47(4)
H(5B)	-4242(16)	7235(8)	2411(6)	61(4)

Table 6. Torsion angles [deg] for gv2319.

C(6)-C(1)-C(2)-C(3)	-1.10(19)
C(1)-C(2)-C(3)-C(4)	0.33(17)
C(2)-C(3)-C(4)-C(5)	1.13(15)
C(2)-C(3)-C(4)-C(9)	-179.07(10)
C(3)-C(4)-C(5)-C(6)	-1.90(16)
C(9)-C(4)-C(5)-C(6)	178.25(10)
C(3)-C(4)-C(5)-N(1)	176.65(9)
C(9)-C(4)-C(5)-N(1)	-3.19(11)
C(4)-C(5)-C(6)-C(1)	1.12(17)
N(1)-C(5)-C(6)-C(1)	-177.15(10)
C(2)-C(1)-C(6)-C(5)	0.37(18)
C(3)-C(4)-C(9)-C(14)	-54.82(14)
C(5)-C(4)-C(9)-C(14)	125.00(9)
C(3)-C(4)-C(9)-C(8)	-173.91(10)
C(5)-C(4)-C(9)-C(8)	5.91(10)
C(3)-C(4)-C(9)-C(10)	71.10(13)
C(5)-C(4)-C(9)-C(10)	-109.08(9)
O(1)-C(8)-C(9)-C(14)	50.93(13)
N(1)-C(8)-C(9)-C(14)	-128.17(9)
O(1)-C(8)-C(9)-C(4)	172.27(10)
N(1)-C(8)-C(9)-C(4)	-6.83(10)
O(1)-C(8)-C(9)-C(10)	-70.86(12)
N(1)-C(8)-C(9)-C(10)	110.04(9)
C(14)-C(9)-C(10)-C(23)	167.98(8)
C(4)-C(9)-C(10)-C(23)	40.30(11)
C(8)-C(9)-C(10)-C(23)	-70.20(10)
C(14)-C(9)-C(10)-C(24)	-72.73(10)
C(4)-C(9)-C(10)-C(24)	159.59(8)
C(8)-C(9)-C(10)-C(24)	49.09(11)
C(14)-C(9)-C(10)-C(11)	45.92(10)
C(4)-C(9)-C(10)-C(11)	-81.77(10)
C(8)-C(9)-C(10)-C(11)	167.73(8)
C(23)-C(10)-C(11)-N(5)	23.08(13)
C(24)-C(10)-C(11)-N(5)	-93.62(11)
C(9)-C(10)-C(11)-N(5)	144.20(9)
C(23)-C(10)-C(11)-C(12)	-160.79(9)
C(24)-C(10)-C(11)-C(12)	82.52(11)
C(9)-C(10)-C(11)-C(12)	-39.67(12)
N(5)-C(11)-C(12)-C(25)	-1.79(16)
C(10)-C(11)-C(12)-C(25)	-177.52(9)
N(5)-C(11)-C(12)-C(13)	-174.49(10)
C(10)-C(11)-C(12)-C(13)	9.79(14)
C(11)-C(12)-C(13)-C(14)	14.87(15)
C(25)-C(12)-C(13)-C(14)	-157.66(10)
C(11)-C(12)-C(13)-C(22)	-169.40(9)
C(25)-C(12)-C(13)-C(22)	18.06(14)
C(12)-C(13)-C(14)-C(15)	168.32(9)
C(22)-C(13)-C(14)-C(15)	-7.48(14)
C(12)-C(13)-C(14)-C(9)	-5.46(15)
C(22)-C(13)-C(14)-C(9)	178.74(8)
C(4)-C(9)-C(14)-C(13)	100.90(11)

C(8)-C(9)-C(14)-C(13)	-145.53(9)
C(10)-C(9)-C(14)-C(13)	-25.12(13)
C(4)-C(9)-C(14)-C(15)	-73.13(11)
C(8)-C(9)-C(14)-C(15)	40.44(12)
C(10)-C(9)-C(14)-C(15)	160.84(8)
C(13)-C(14)-C(15)-C(16)	-32.65(13)
C(9)-C(14)-C(15)-C(16)	141.41(9)
C(14)-C(15)-C(16)-C(17)	53.49(11)
C(15)-C(16)-C(17)-C(18)	141.71(11)
C(15)-C(16)-C(17)-C(22)	-38.45(13)
C(22)-C(17)-C(18)-C(19)	1.91(18)
C(16)-C(17)-C(18)-C(19)	-178.25(11)
C(17)-C(18)-C(19)-C(20)	1.1(2)
C(18)-C(19)-C(20)-C(21)	-2.2(2)
C(19)-C(20)-C(21)-C(22)	0.28(18)
C(20)-C(21)-C(22)-C(17)	2.68(16)
C(20)-C(21)-C(22)-C(13)	-179.92(10)
C(18)-C(17)-C(22)-C(21)	-3.74(15)
C(16)-C(17)-C(22)-C(21)	176.42(9)
C(18)-C(17)-C(22)-C(13)	178.71(10)
C(16)-C(17)-C(22)-C(13)	-1.13(14)
C(14)-C(13)-C(22)-C(21)	-151.32(10)
C(12)-C(13)-C(22)-C(21)	32.89(14)
C(14)-C(13)-C(22)-C(17)	26.12(14)
C(12)-C(13)-C(22)-C(17)	-149.66(9)
C(24)-C(10)-C(23)-N(2)	123(3)
C(11)-C(10)-C(23)-N(2)	7(3)
C(9)-C(10)-C(23)-N(2)	-115(3)
C(23)-C(10)-C(24)-N(3)	-61.8(14)
C(11)-C(10)-C(24)-N(3)	56.8(14)
C(9)-C(10)-C(24)-N(3)	178(100)
C(11)-C(12)-C(25)-N(4)	10(5)
C(13)-C(12)-C(25)-N(4)	-178(100)
O(1)-C(8)-N(1)-C(5)	-173.61(10)
C(9)-C(8)-N(1)-C(5)	5.46(11)
O(1)-C(8)-N(1)-C(7)	1.09(18)
C(9)-C(8)-N(1)-C(7)	-179.84(10)
C(6)-C(5)-N(1)-C(8)	176.88(11)
C(4)-C(5)-N(1)-C(8)	-1.57(12)
C(6)-C(5)-N(1)-C(7)	2.21(18)
C(4)-C(5)-N(1)-C(7)	-176.24(10)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for gv2319 [A and deg.].

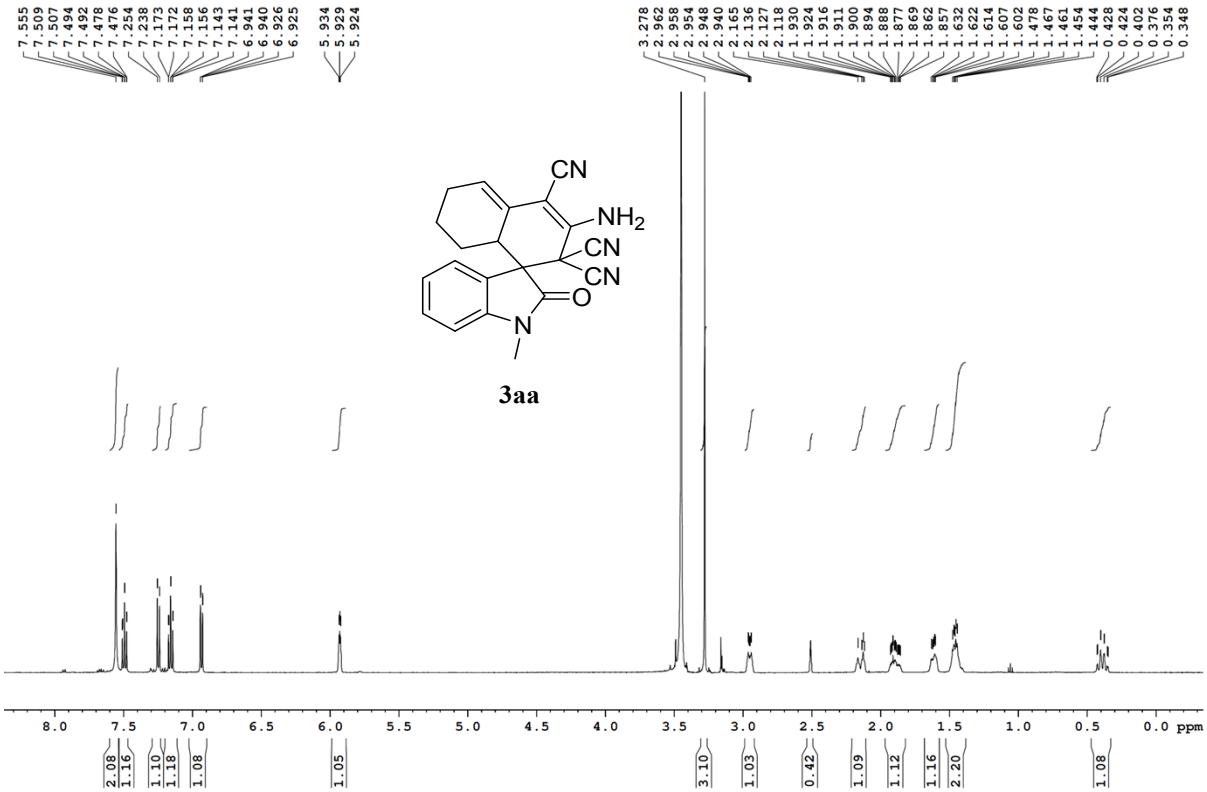
D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
C(6)-H(6)...N(2)#1	0.93	2.58	3.4659(15)	158.6
N(5)-H(5B)...O(1)#2	0.855(10)	2.136(11)	2.9759(12)	167.4(11)

Symmetry transformations used to generate equivalent atoms:
#1 -x,-y+1,-z #2 -x-1/2,y+1/2,-z+1/2

2. ^{1}H NMR and ^{13}C NMR spectra for new compounds

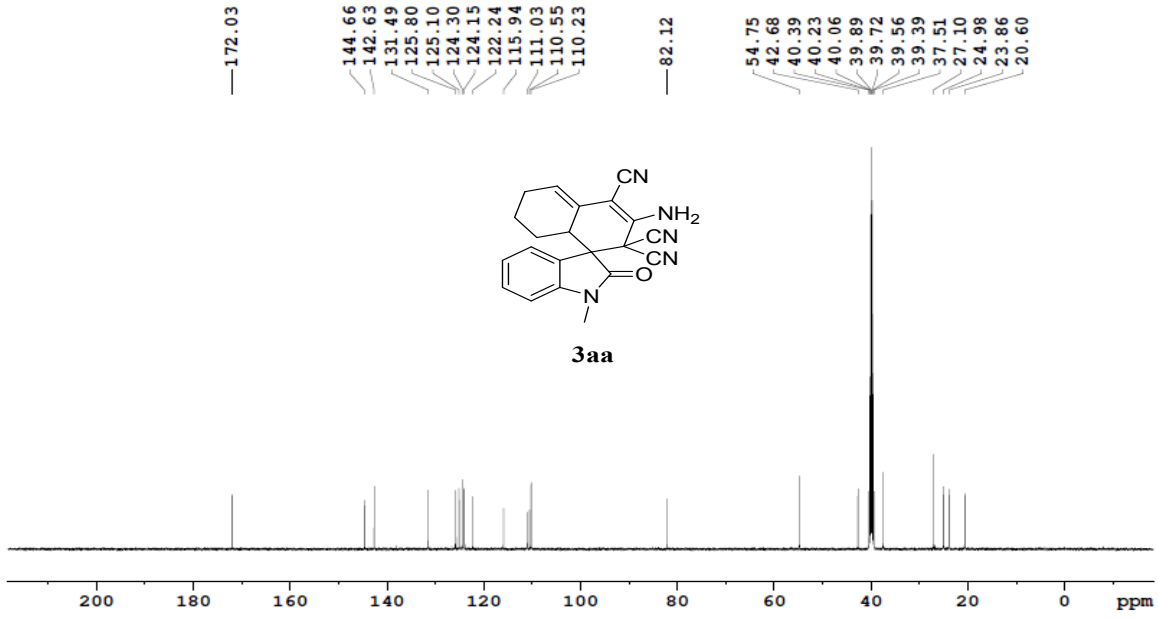
^{1}H NMR and ^{13}C NMR of compound (3aa):

GV-20-79.....Pradhap,



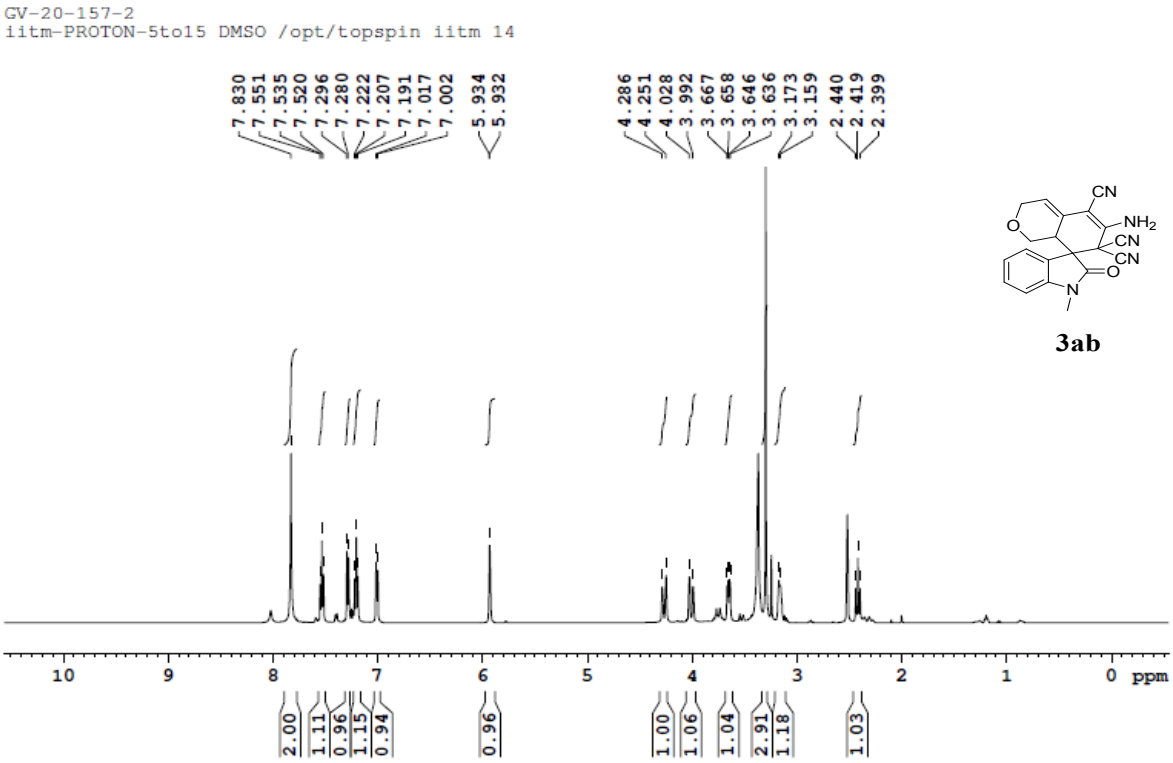
3aa

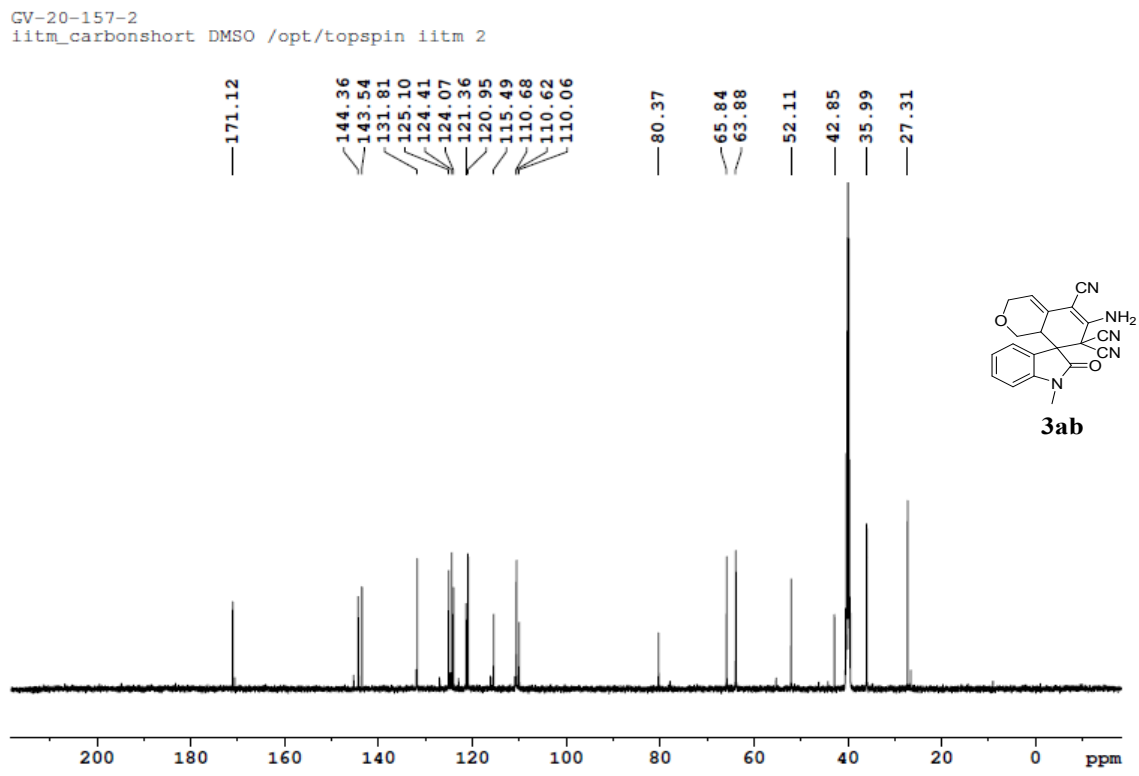
GV-20-79.....Pradhap,



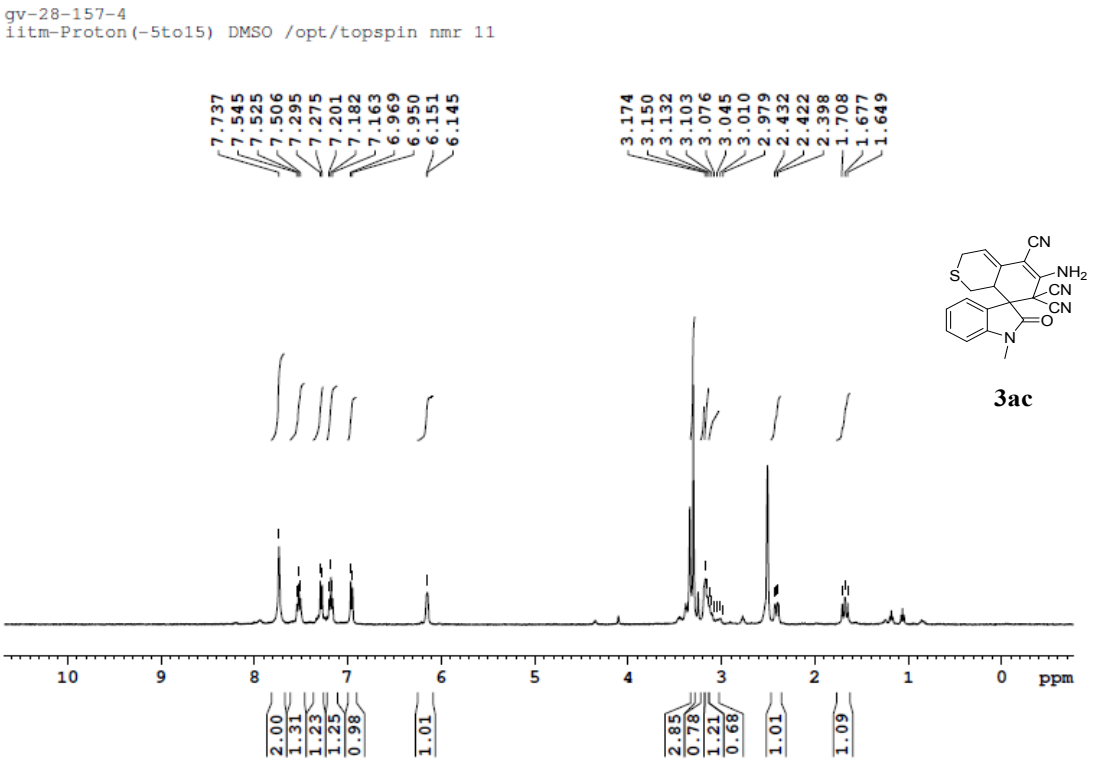
3aa

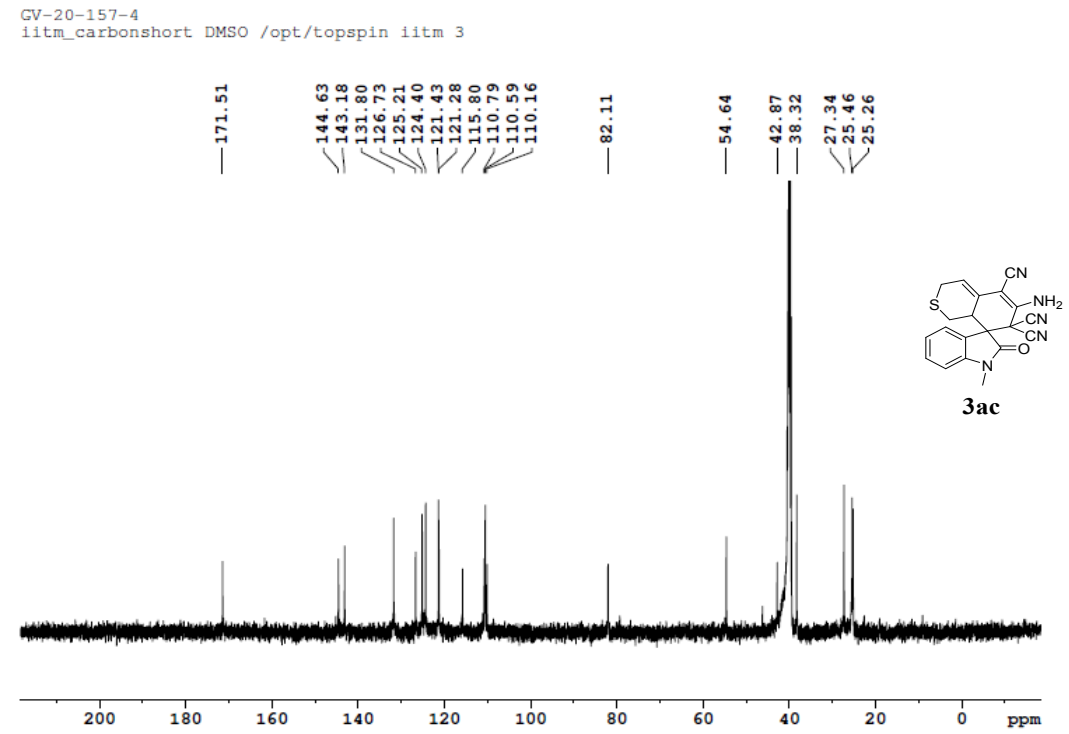
^{1}H NMR and ^{13}C NMR of compound (3ab):





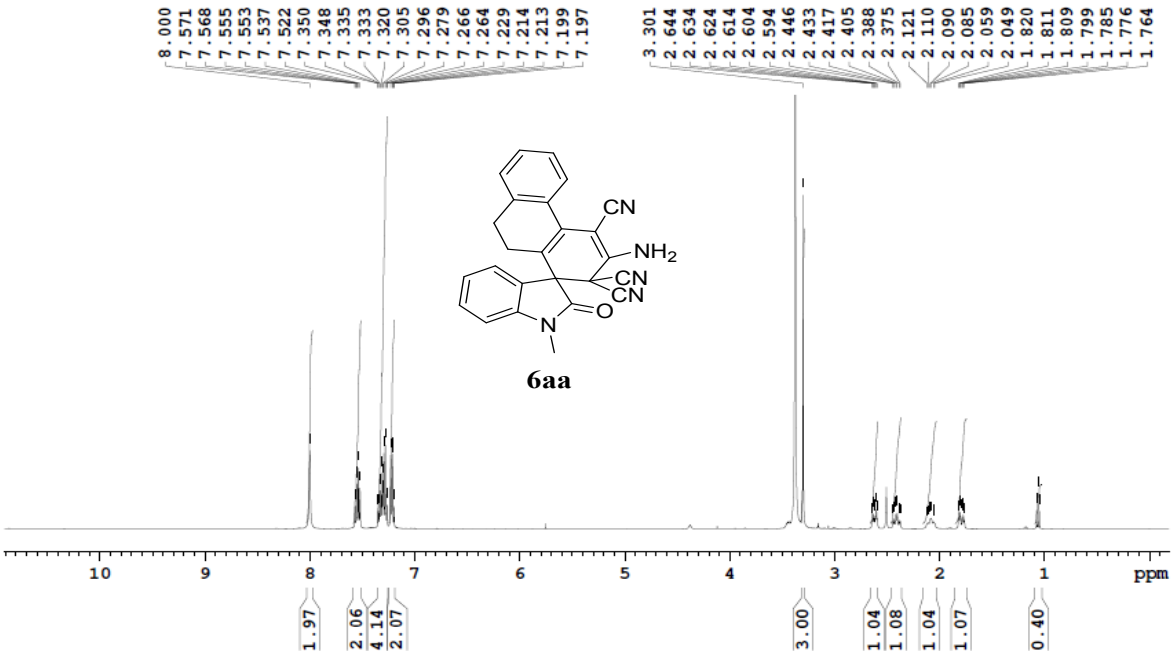
^{1}H NMR and ^{13}C NMR of compound (3ac):





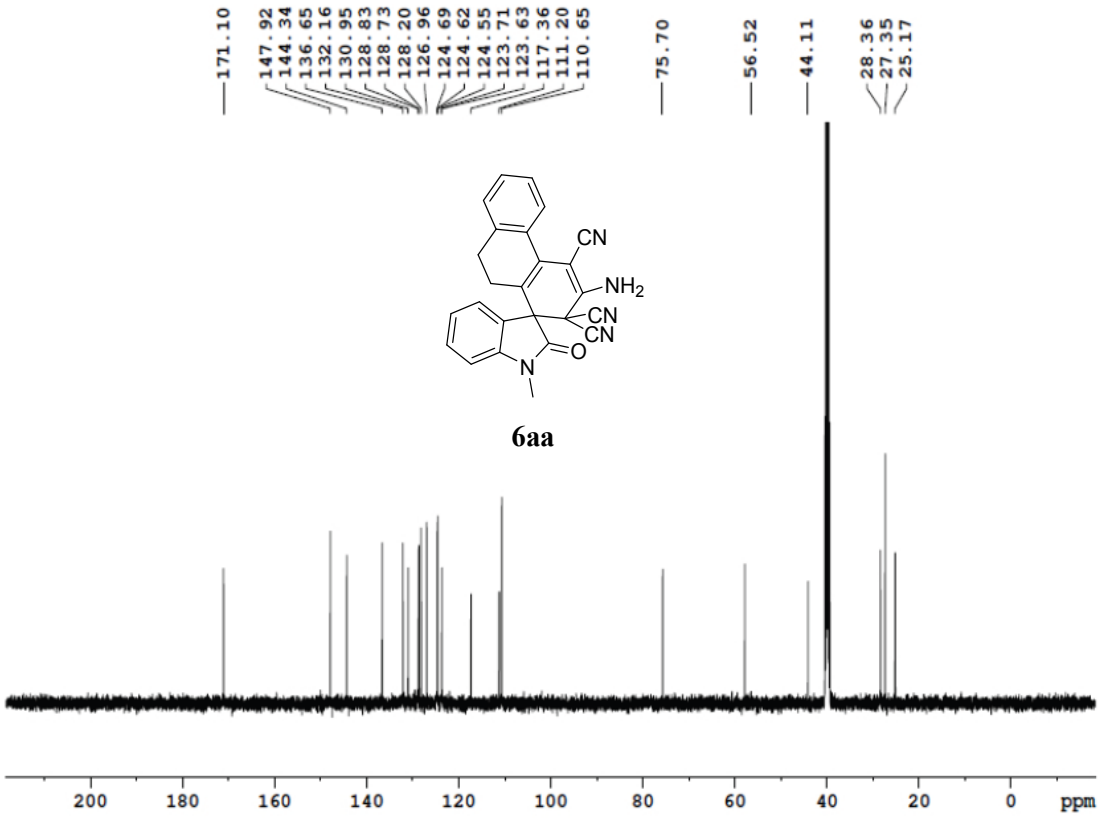
^{1}H NMR and ^{13}C NMR of compound (6aa):

GV-20-157-6 PROTON pratap



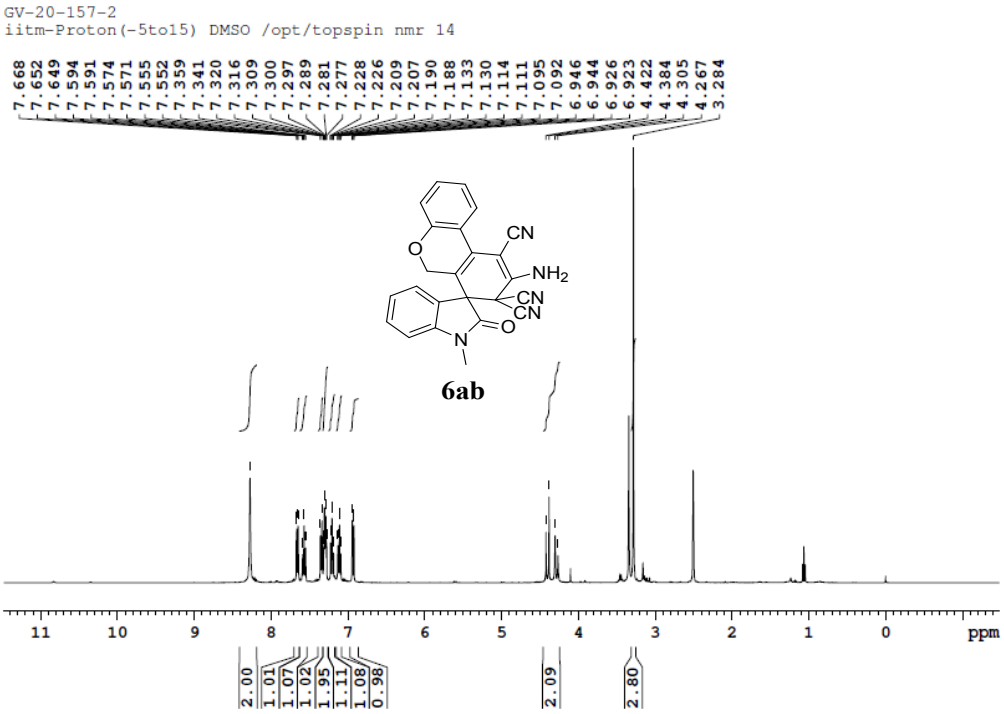
6aa

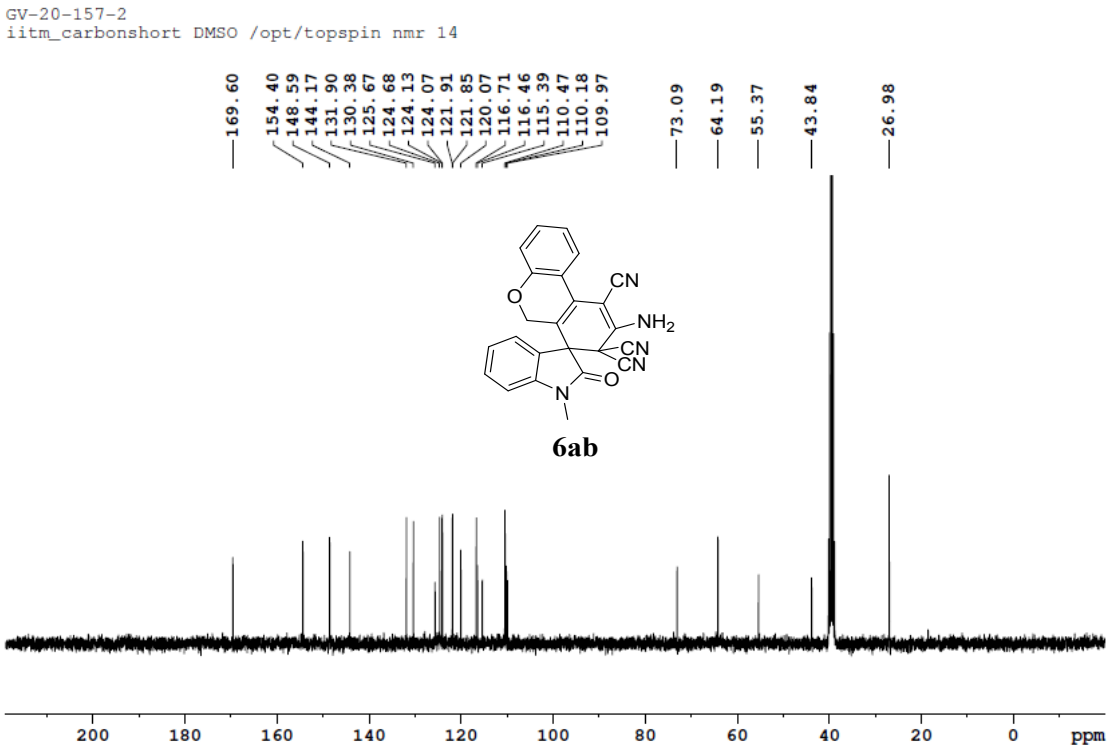
GV-23-19.....Pradhap,



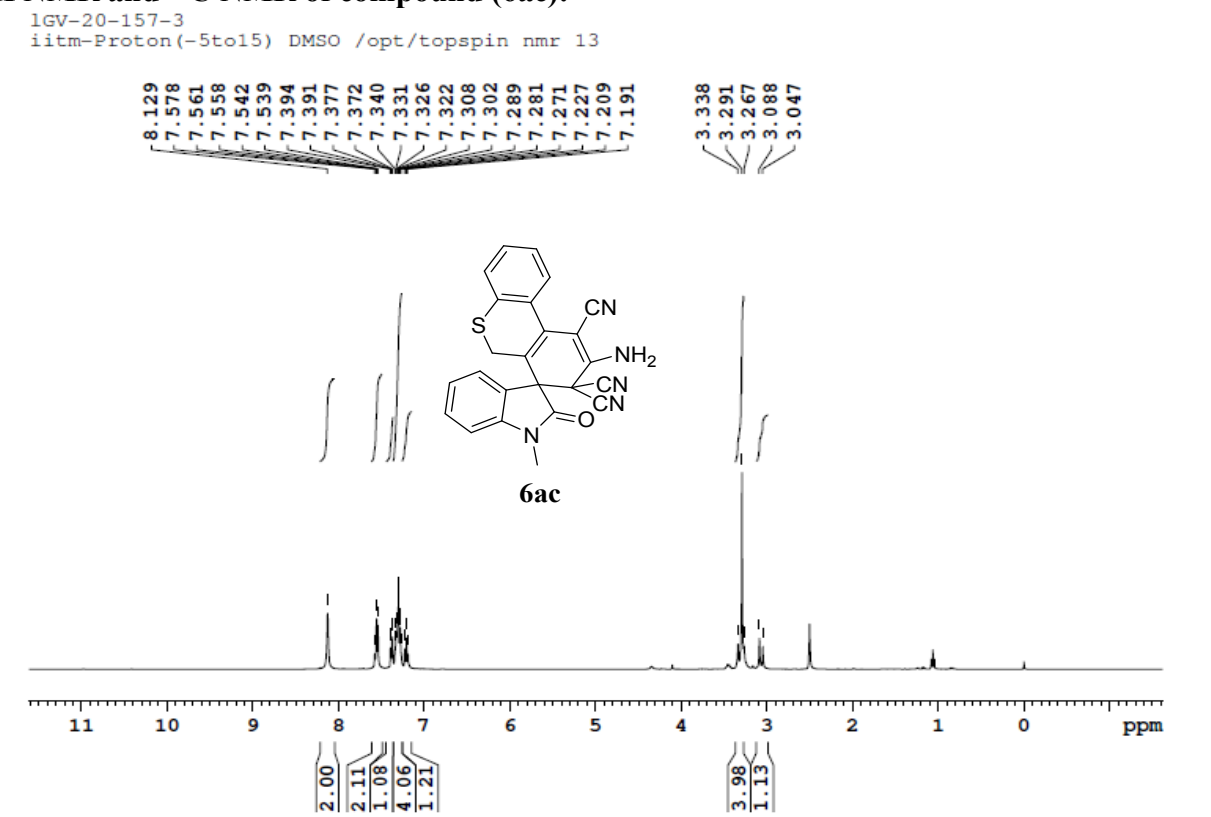
6aa

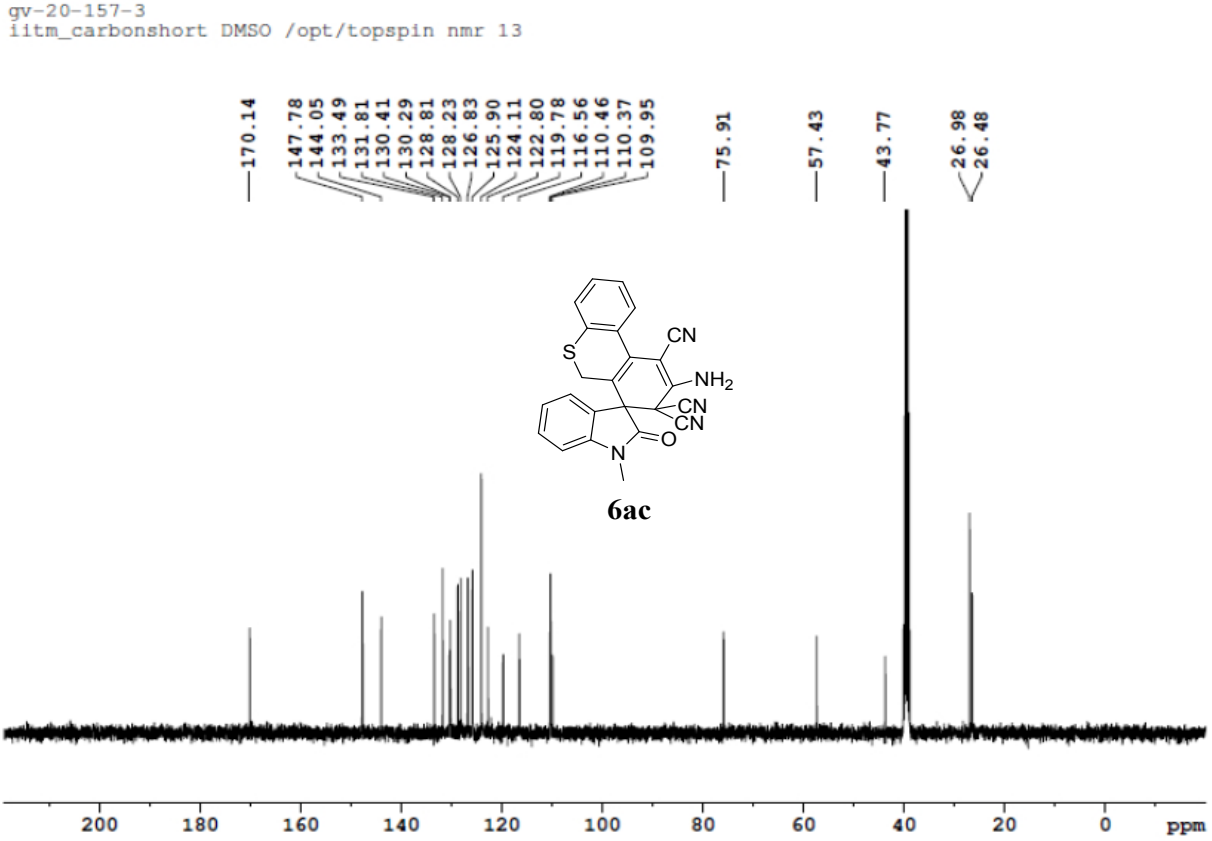
^{1}H NMR and ^{13}C NMR of compound (6ab):



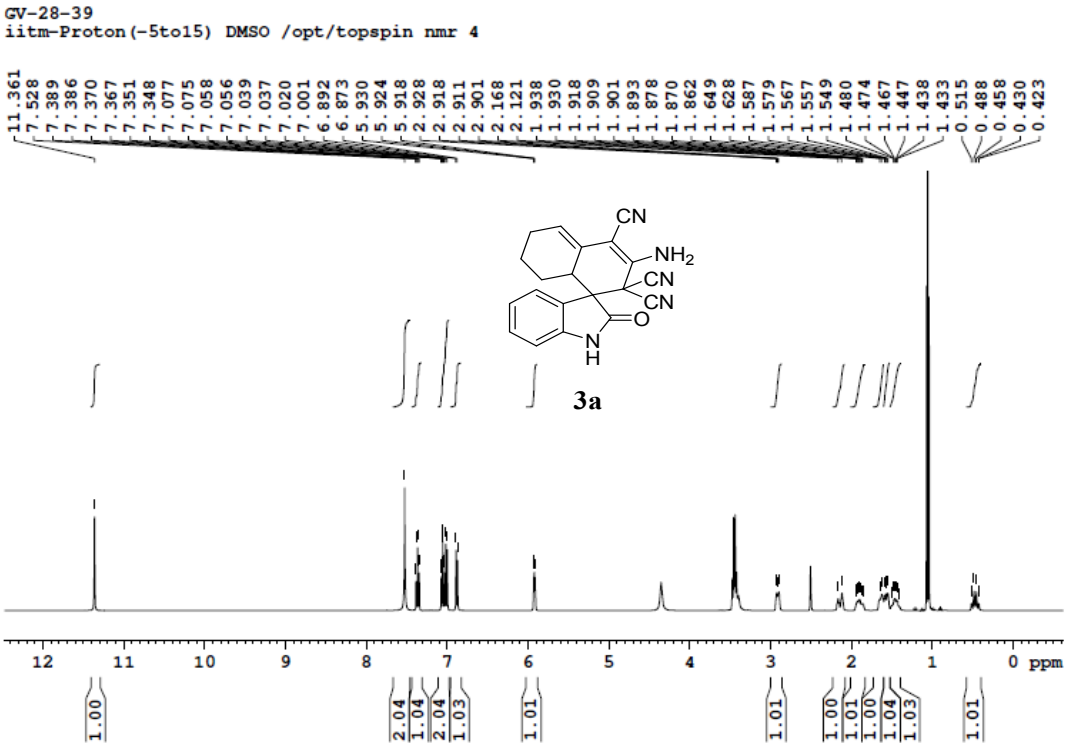


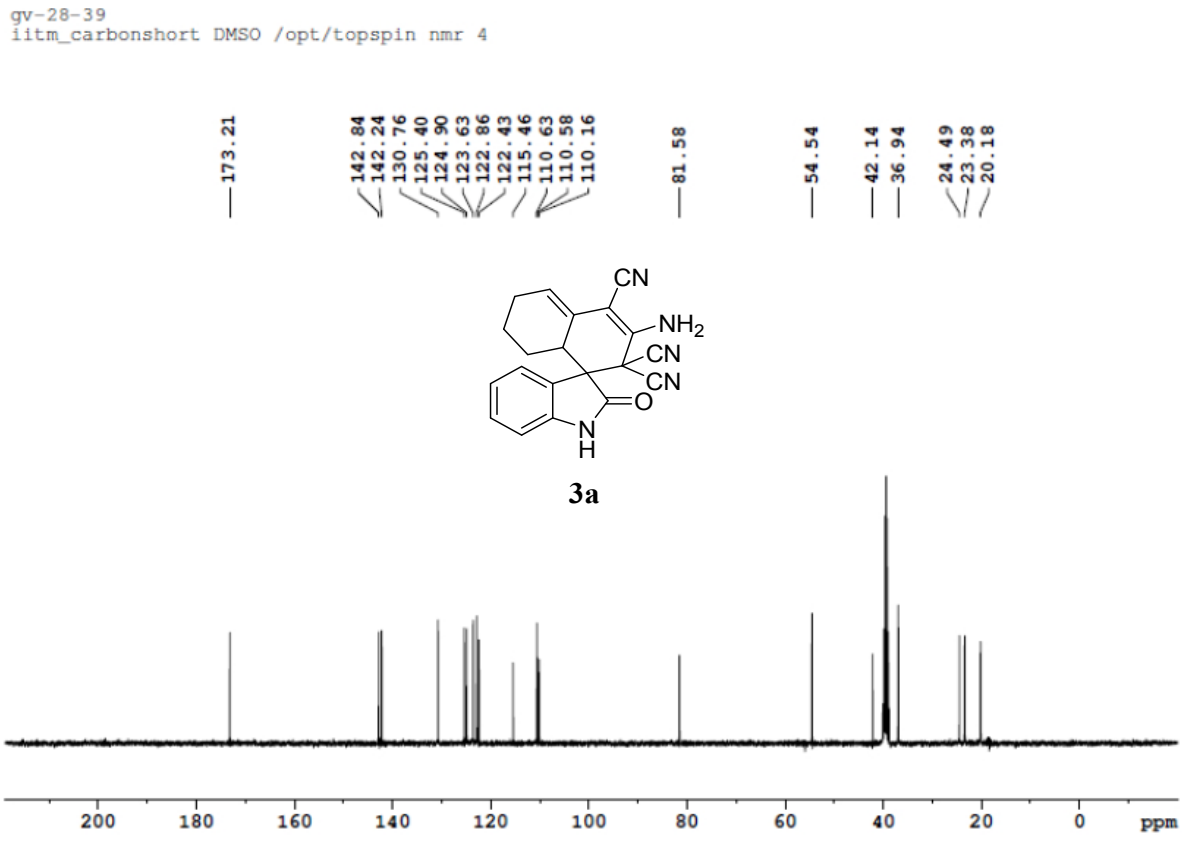
^{1}H NMR and ^{13}C NMR of compound (6ac):



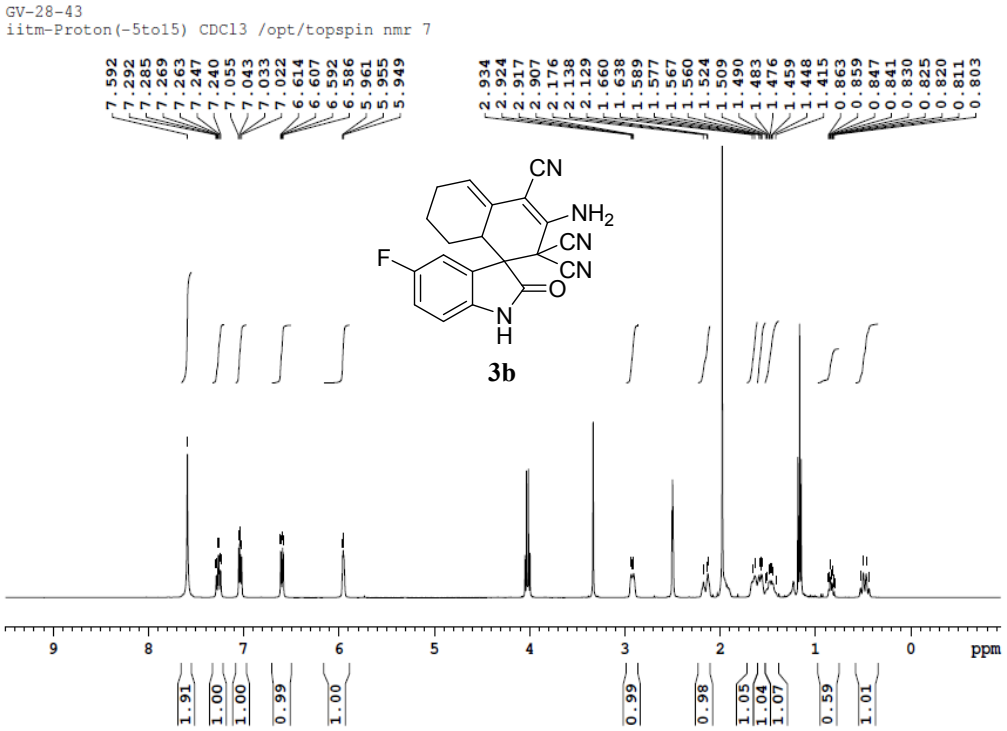


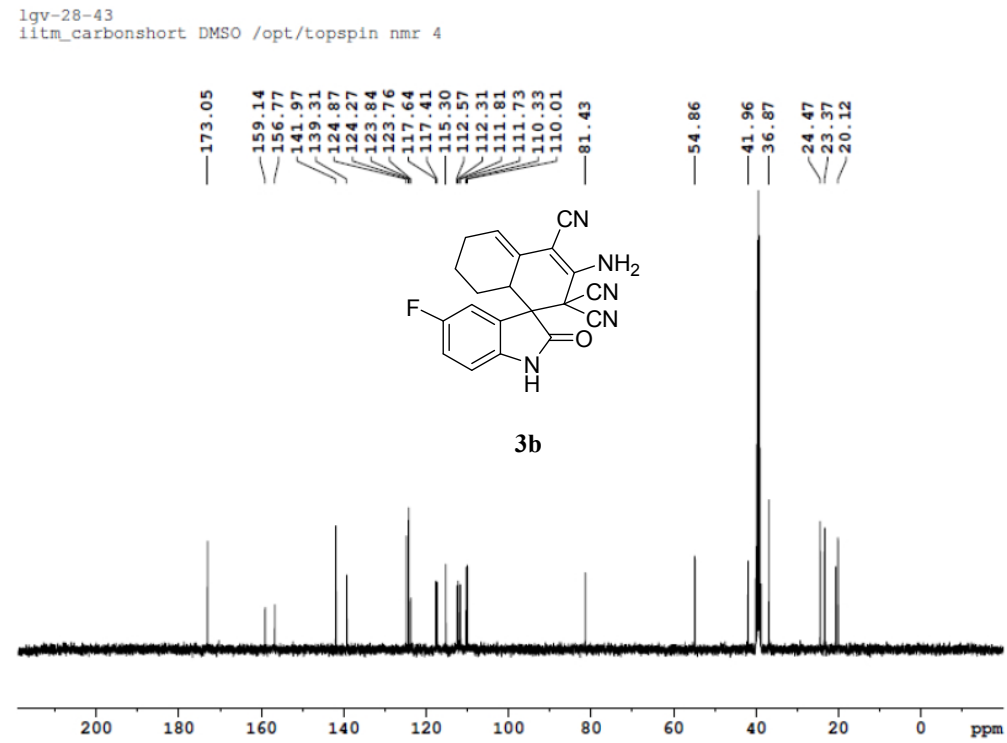
^{1}H NMR and ^{13}C NMR of compound (3a):



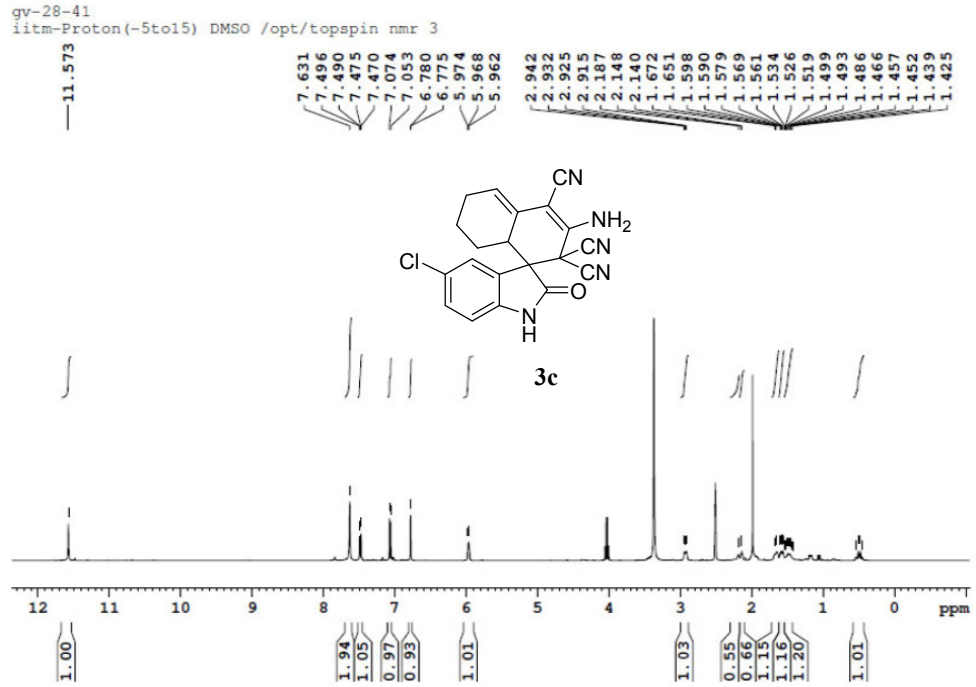


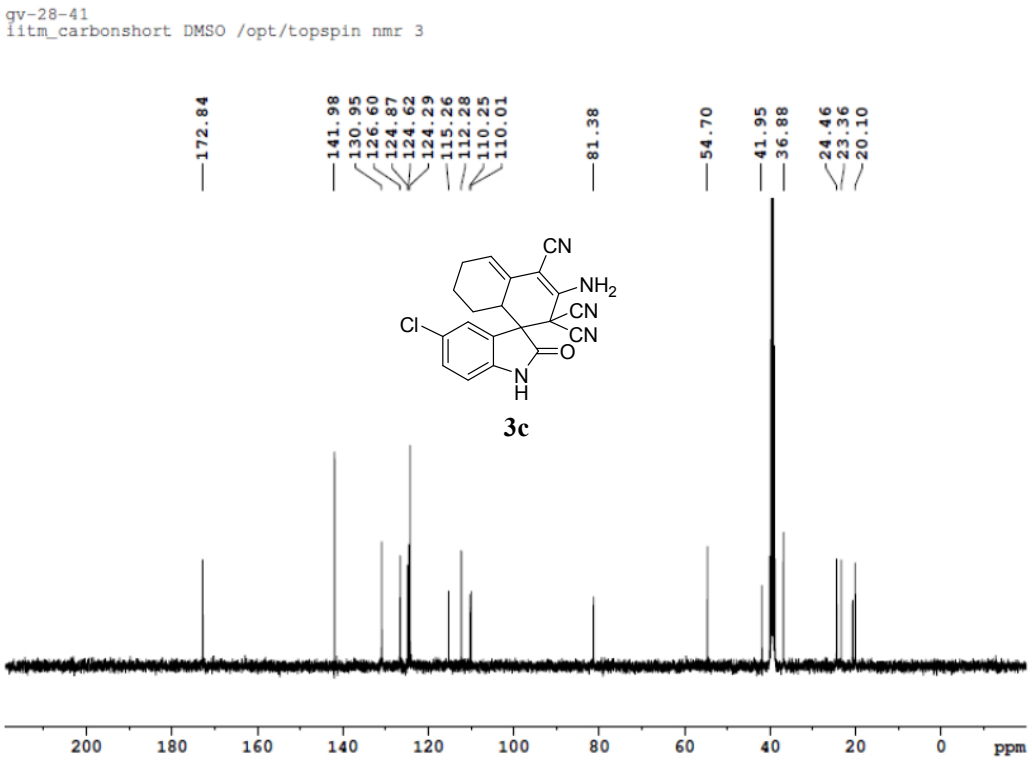
^{1}H NMR and ^{13}C NMR of compound (3b):



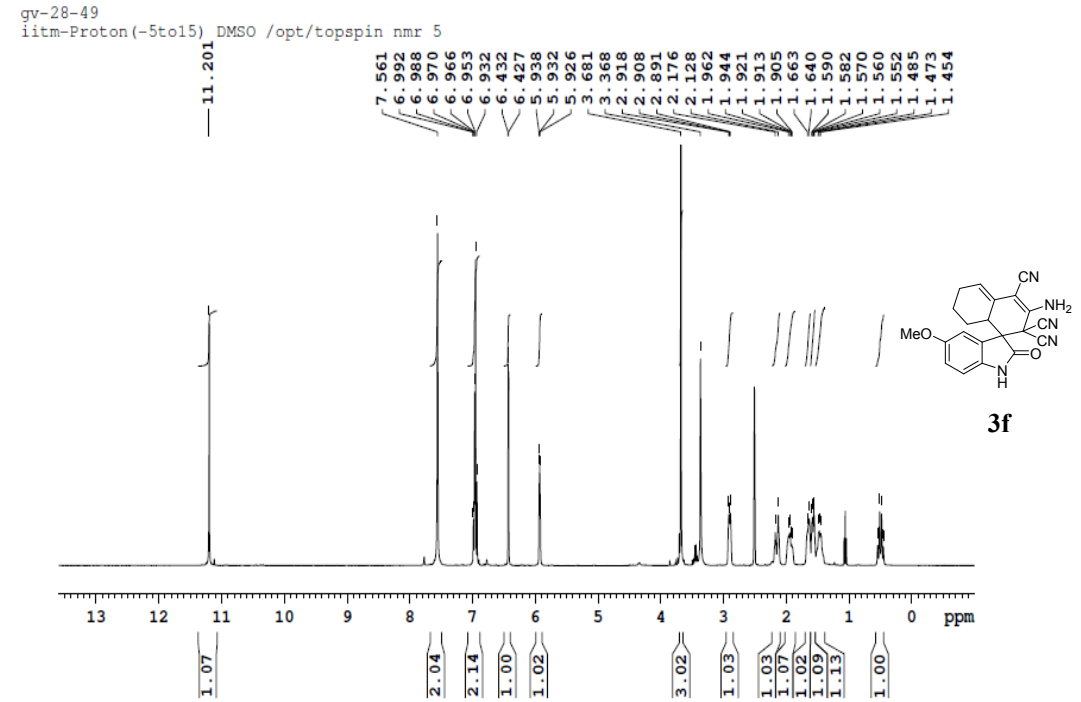


^{1}H NMR and ^{13}C NMR of compound (3c):

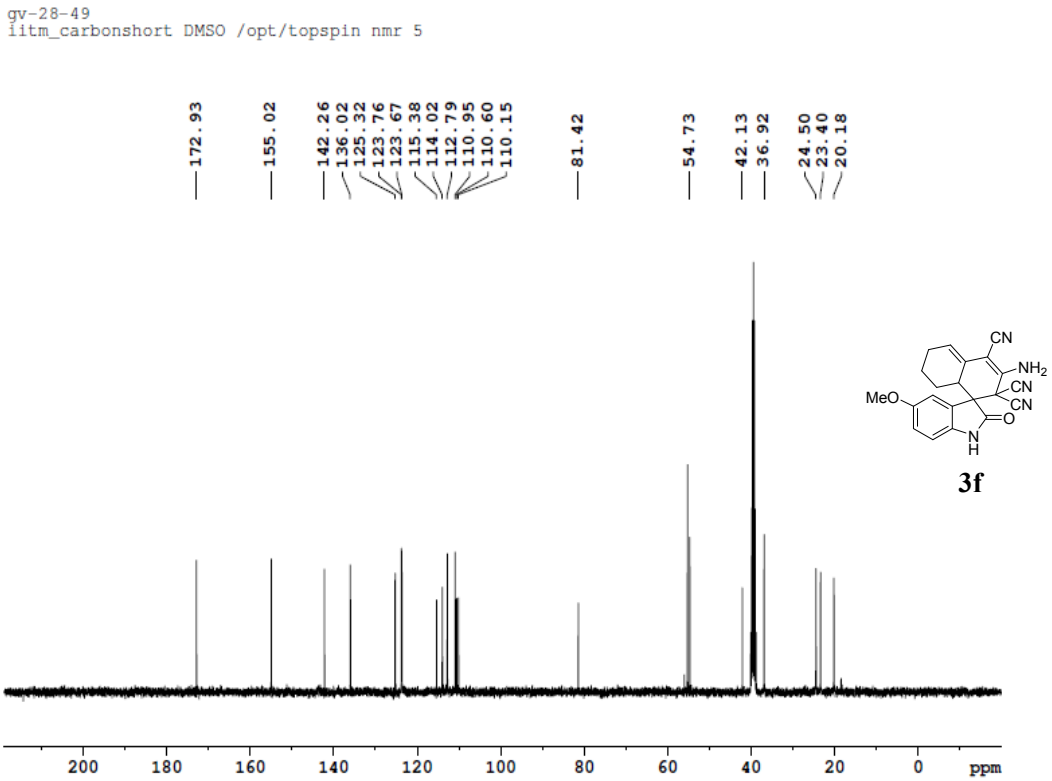




^{1}H NMR and ^{13}C NMR of compound (3f):

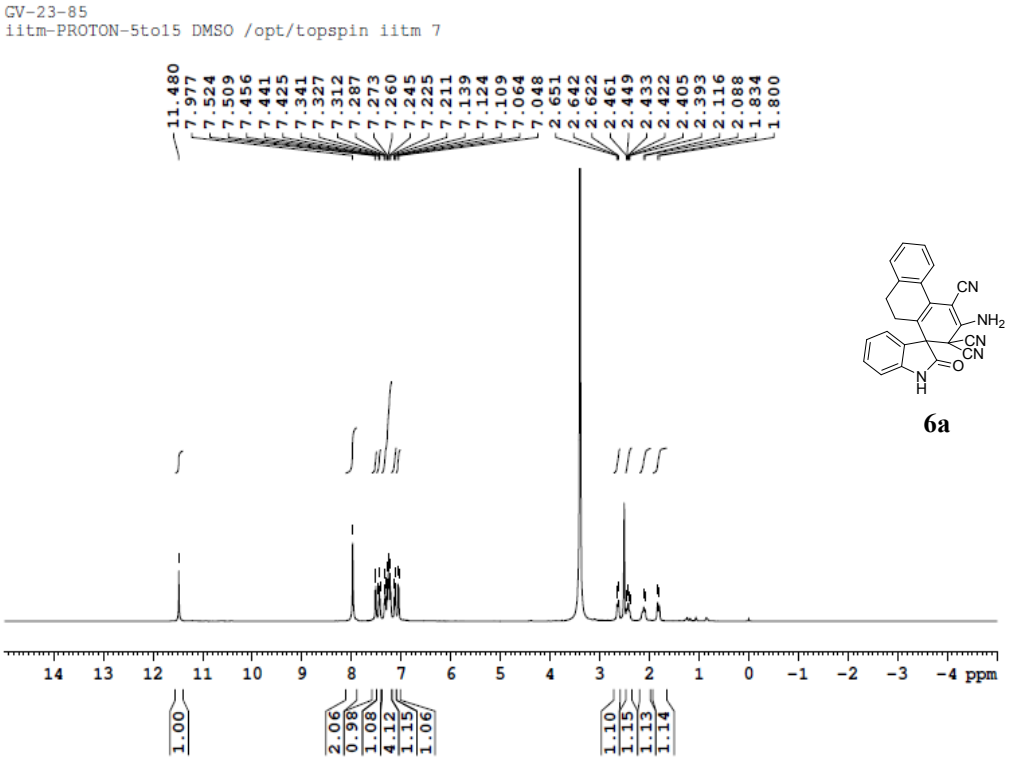


3f

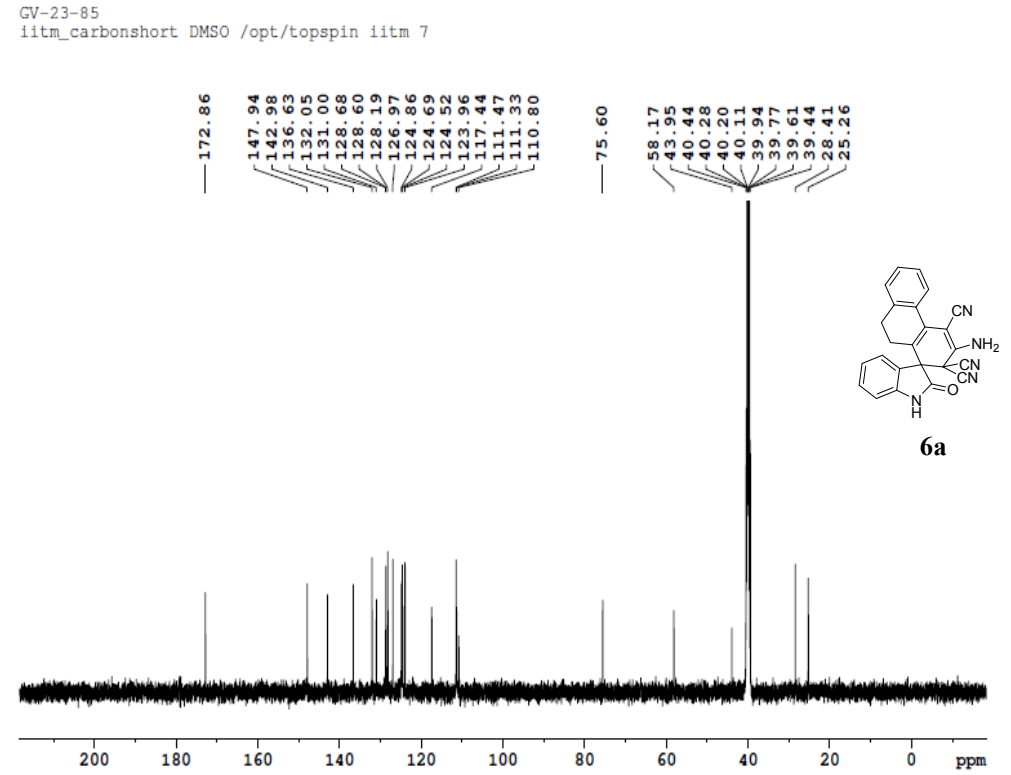


3f

^{1}H NMR and ^{13}C NMR of compound (6a):

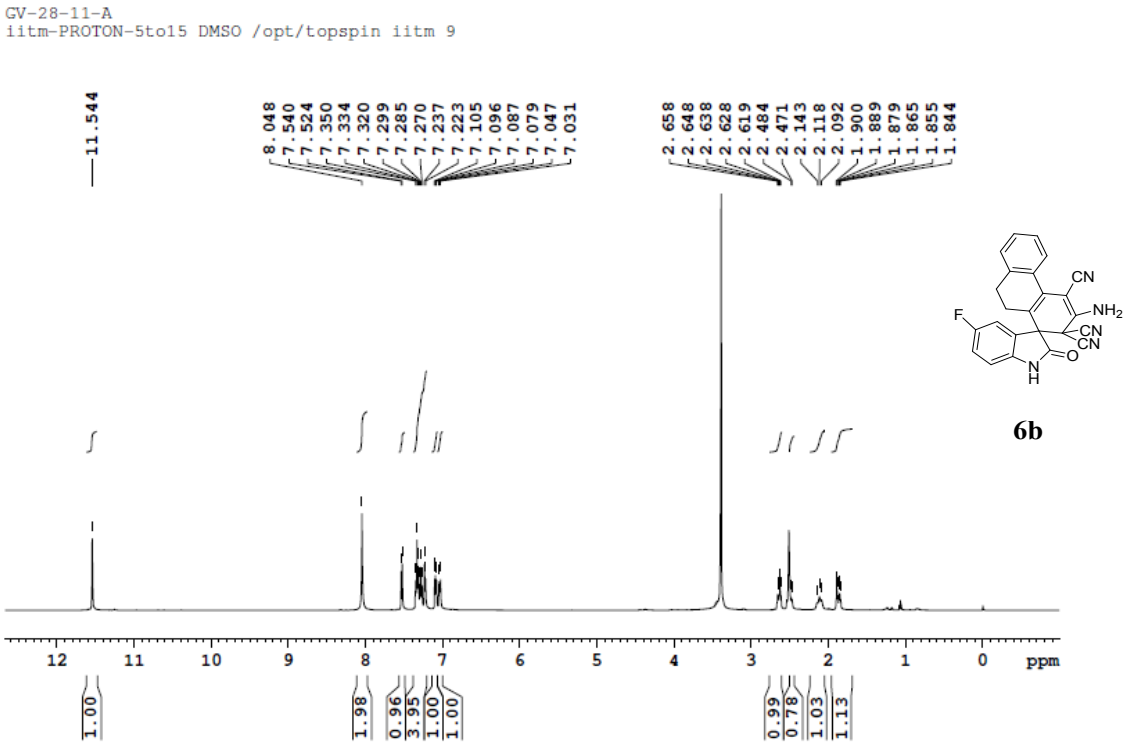


6a

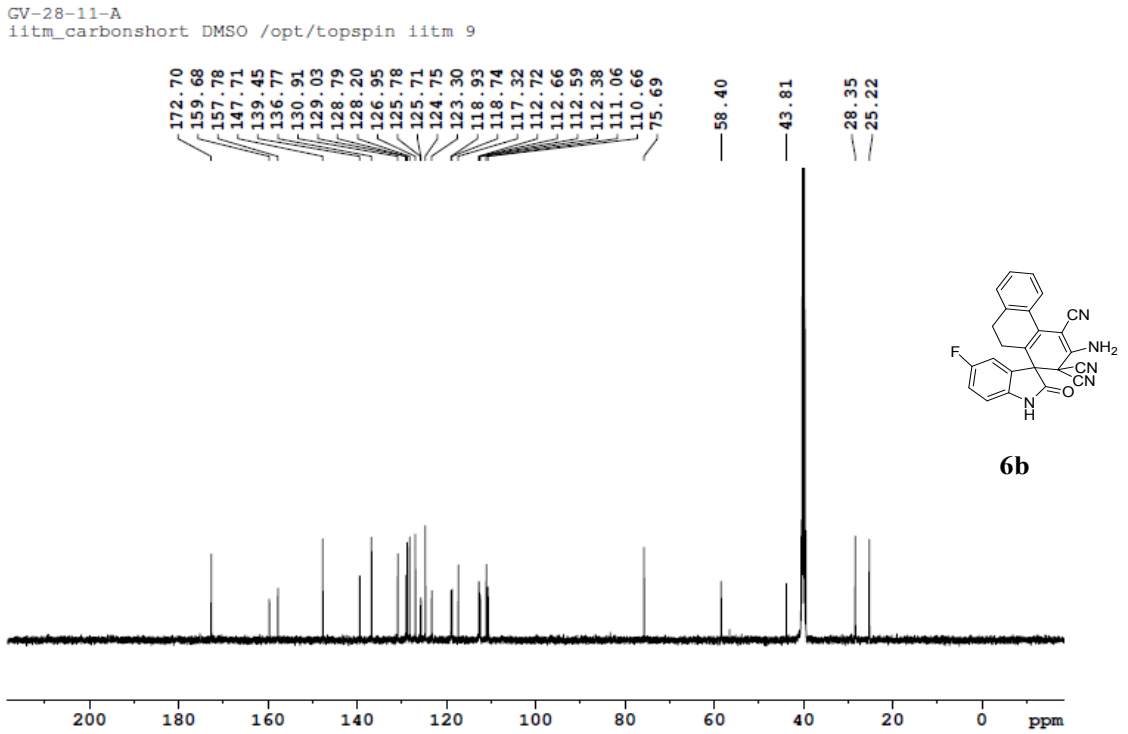


6a

^{1}H NMR and ^{13}C NMR of compound (6b)

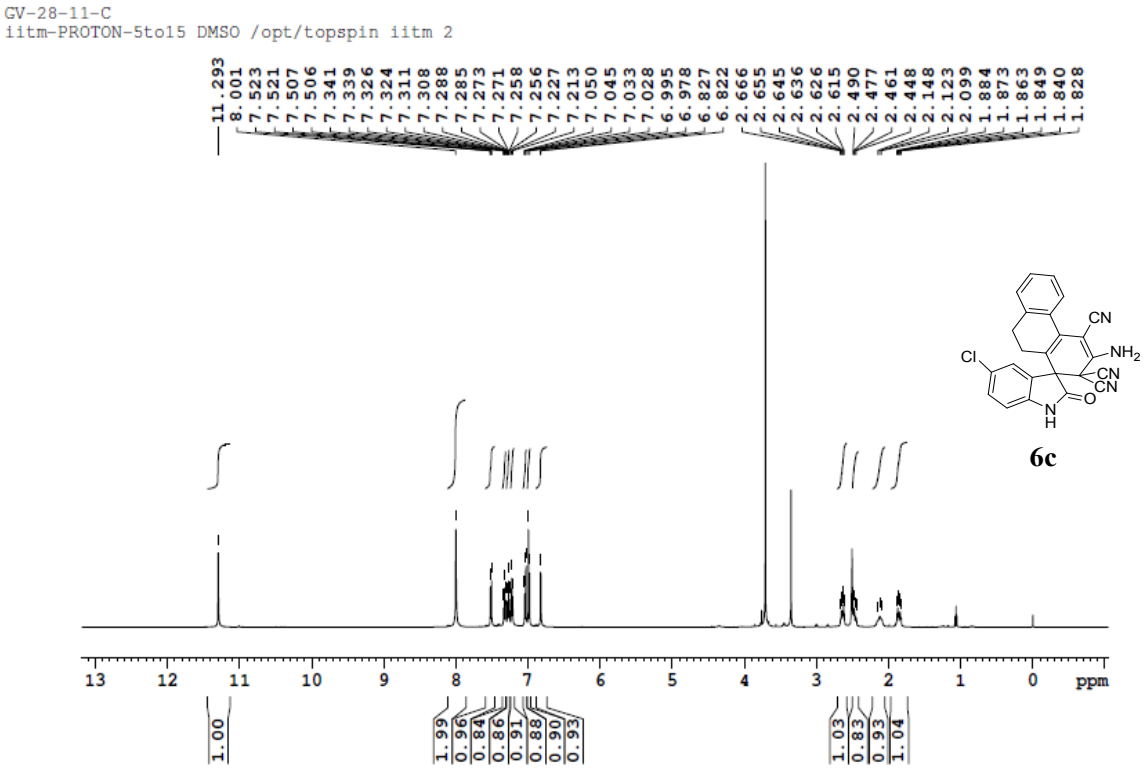


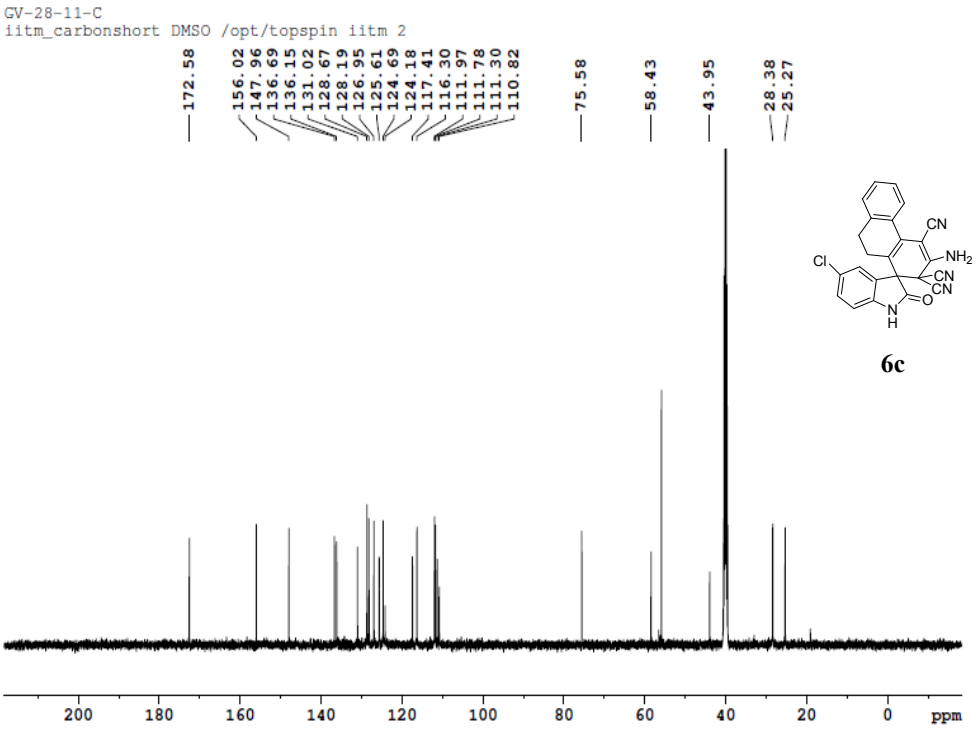
6b



6b

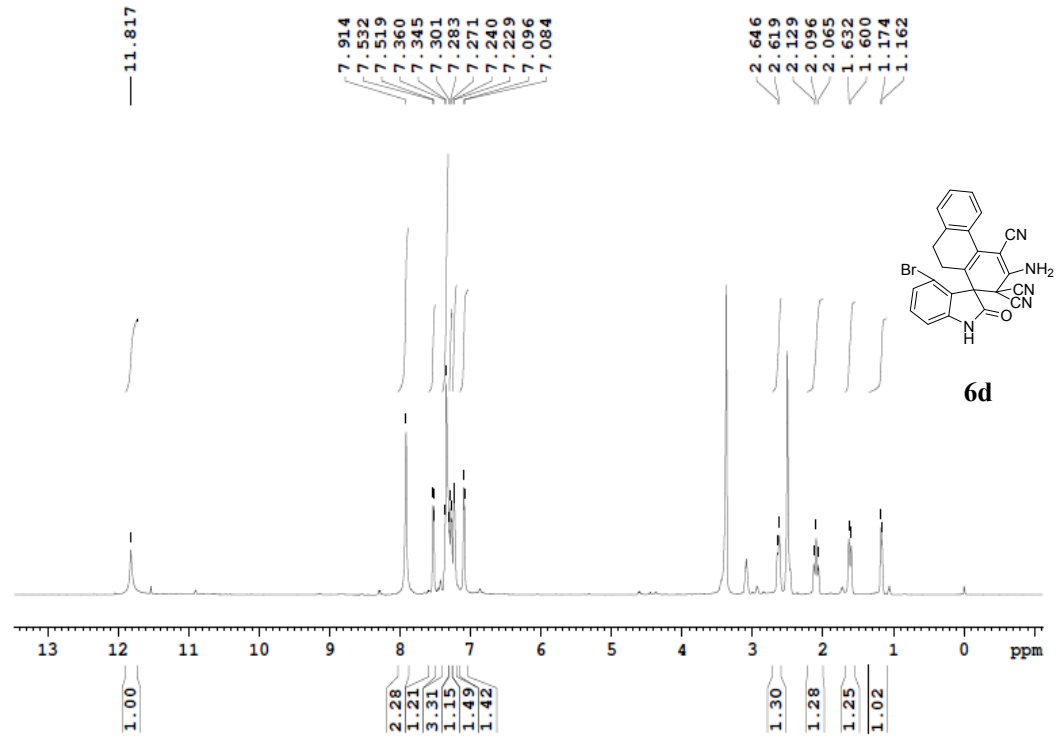
^{1}H NMR and ^{13}C NMR of compound (6c)





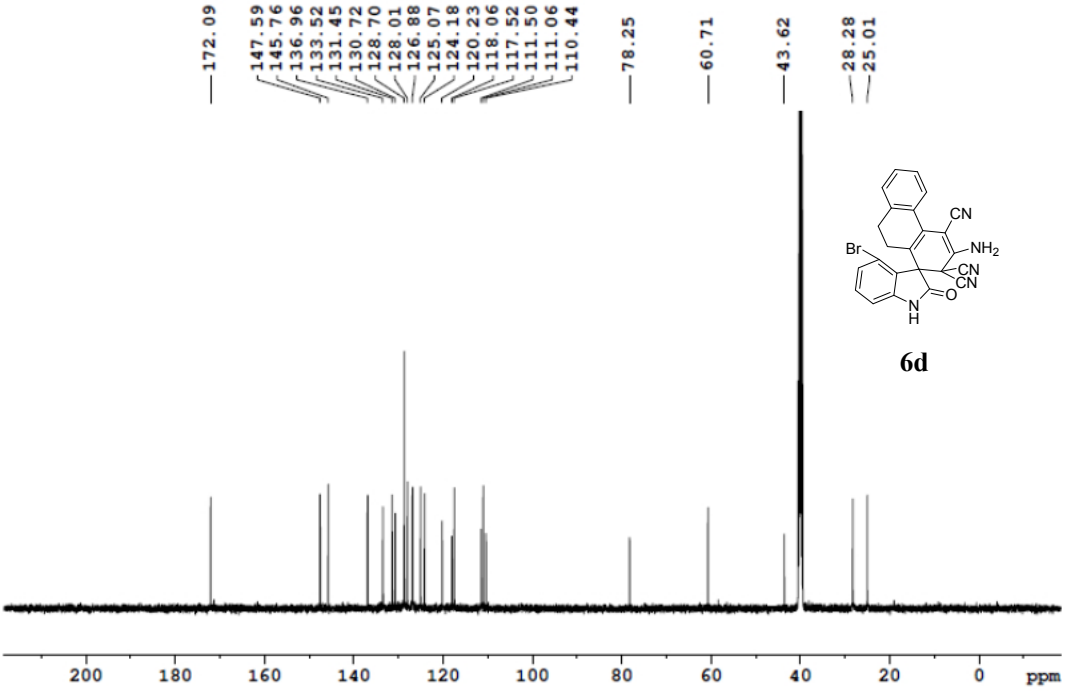
^{1}H NMR and ^{13}C NMR of compound (6d)

GV-28-91.....



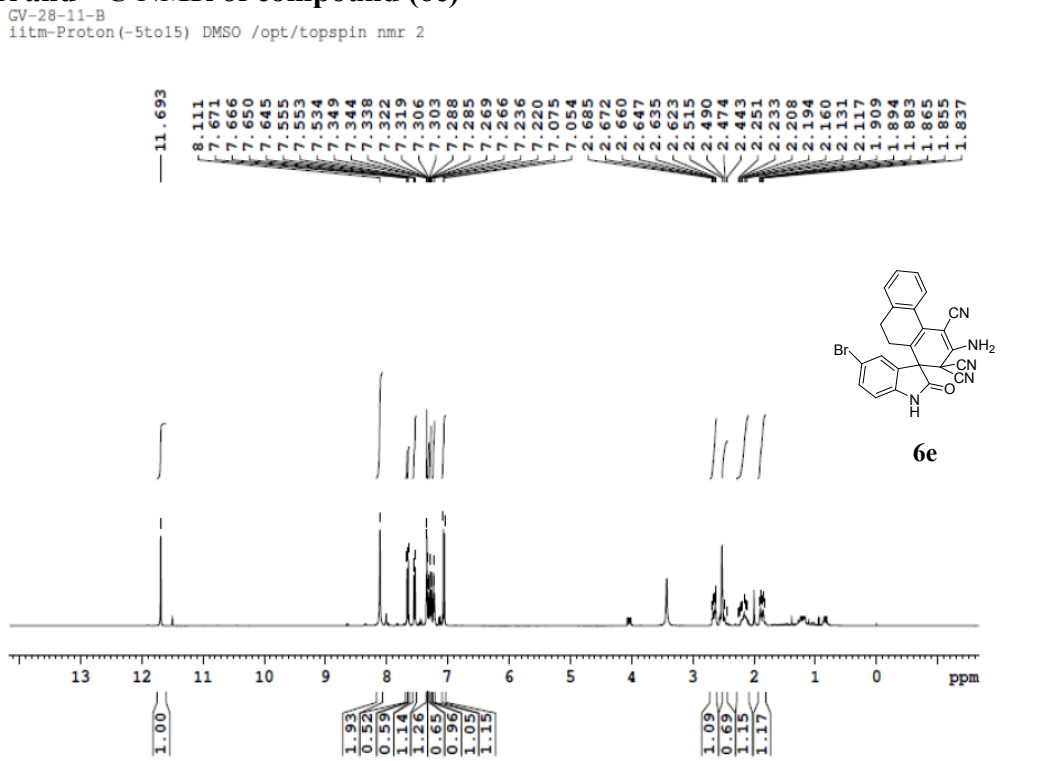
6d

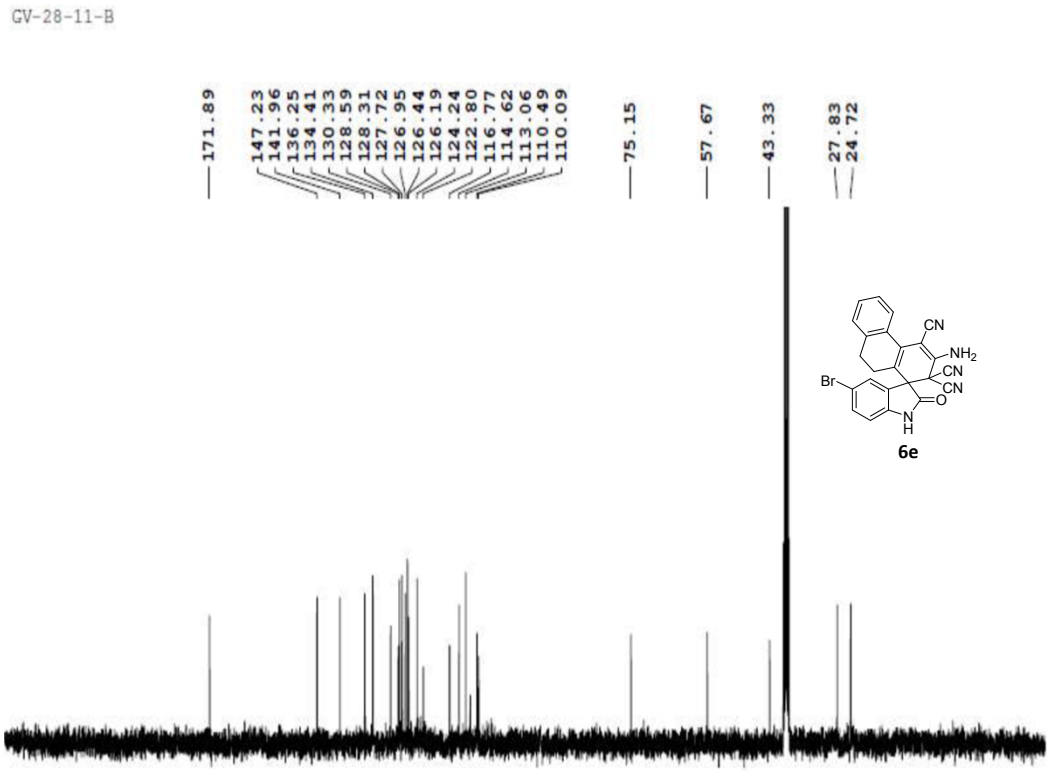
GV-28-91.....



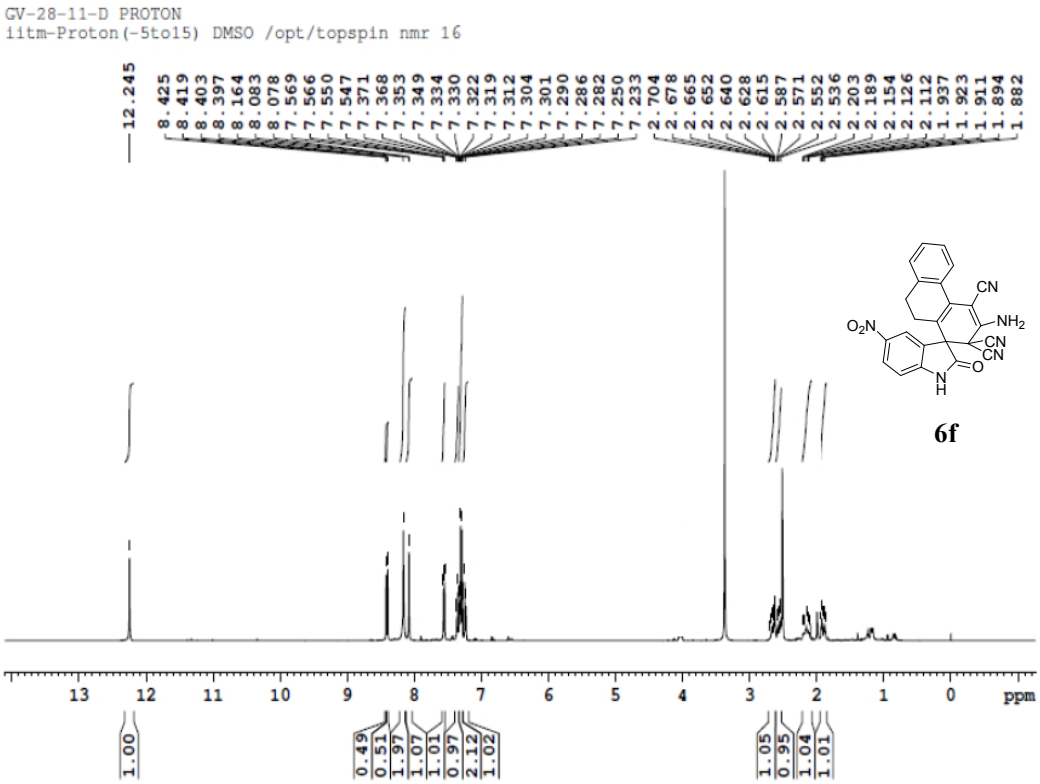
6d

^{1}H NMR and ^{13}C NMR of compound (6e)

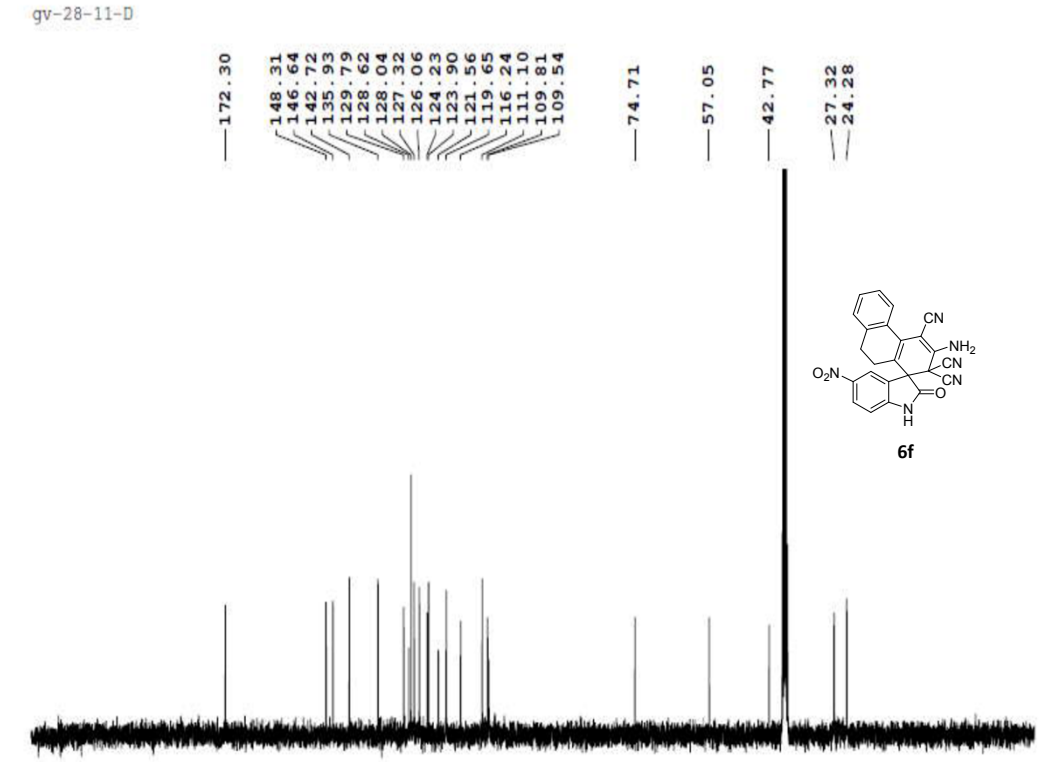




^{1}H NMR and ^{13}C NMR of compound (6f)

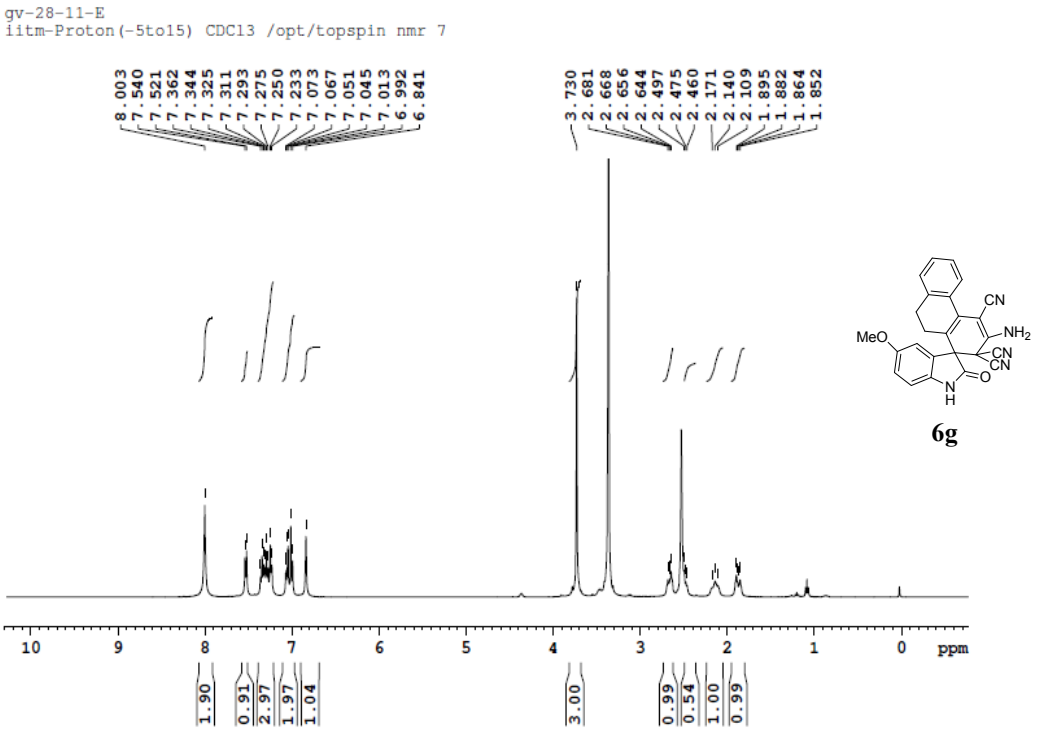


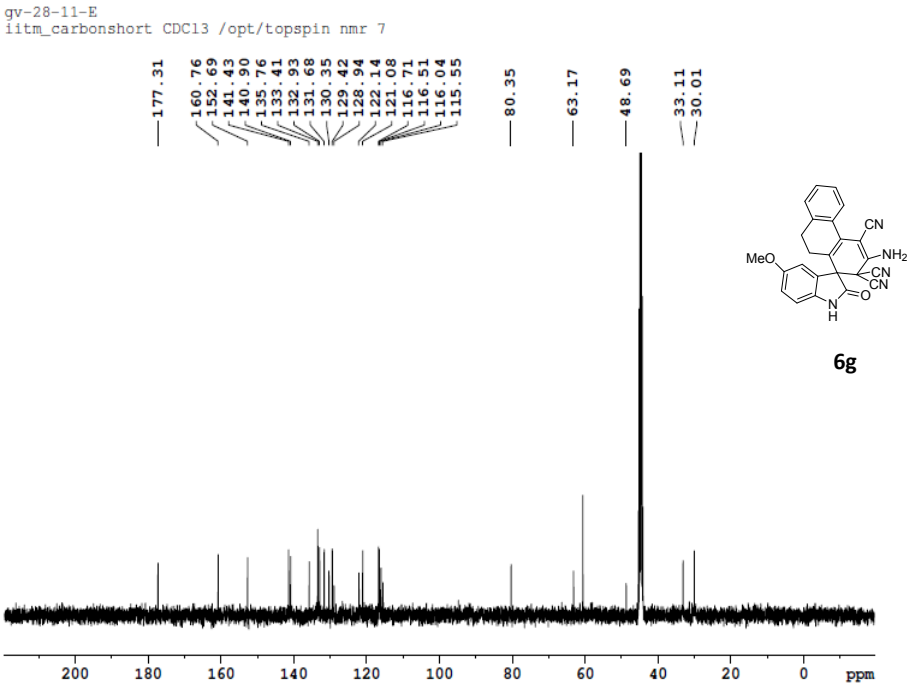
6f



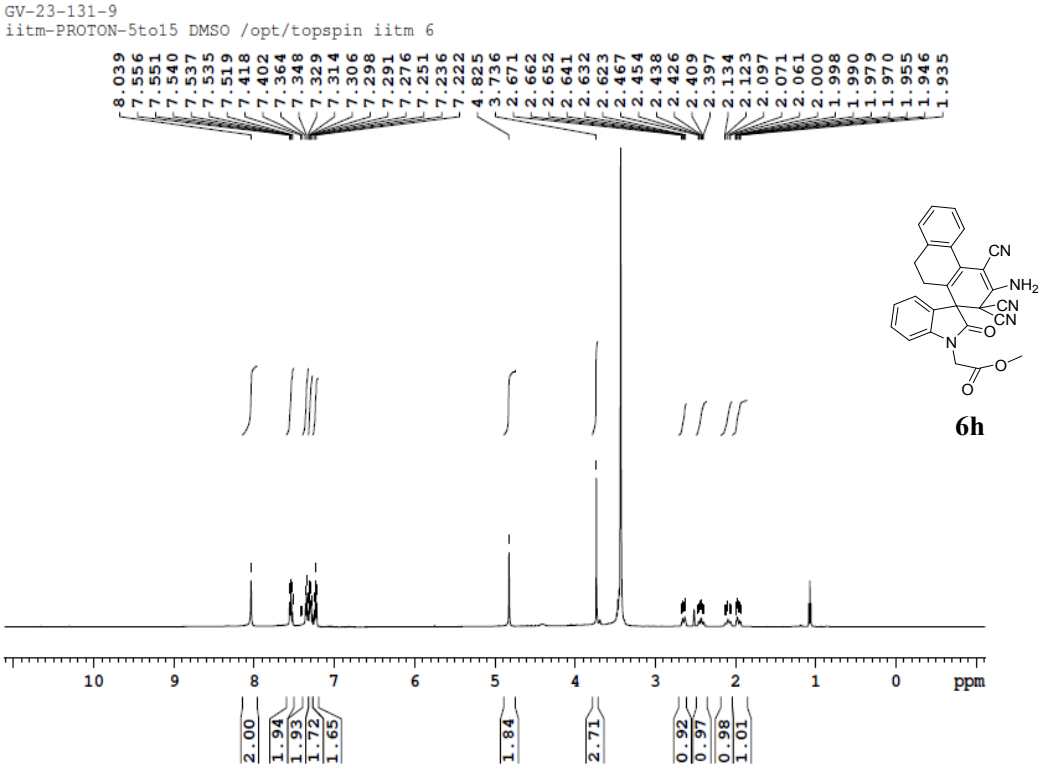
6f

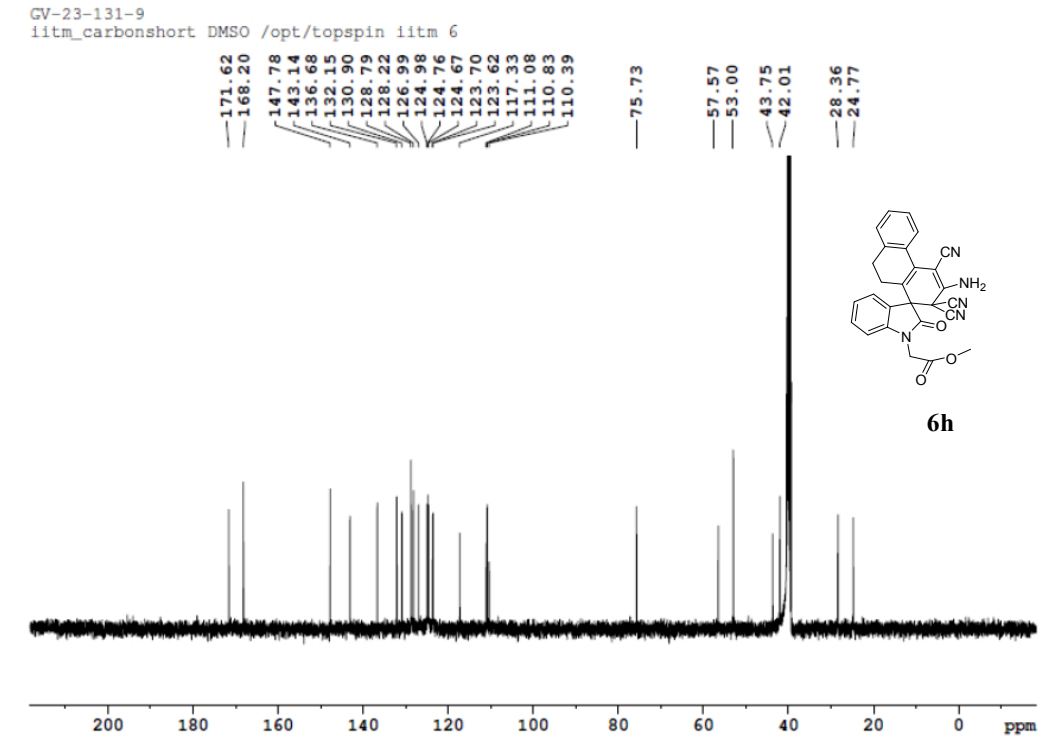
^{1}H NMR and ^{13}C NMR of compound (6g)



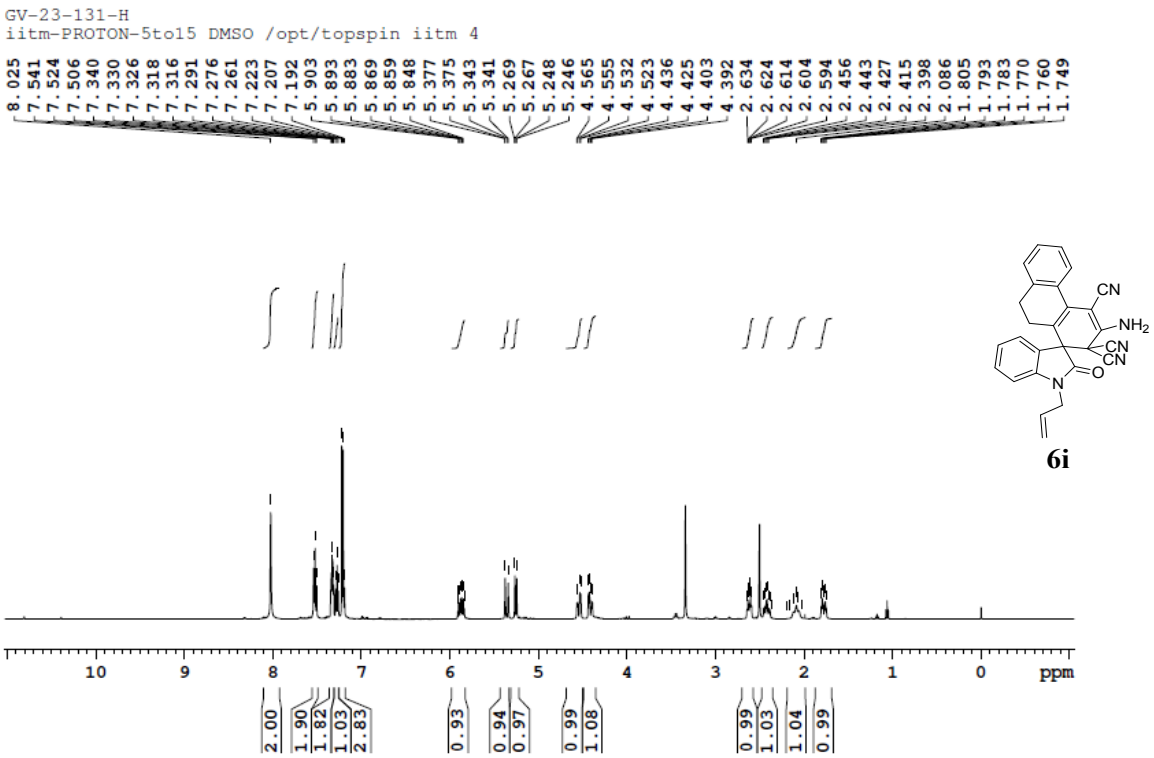


^{1}H NMR and ^{13}C NMR of compound (6h)

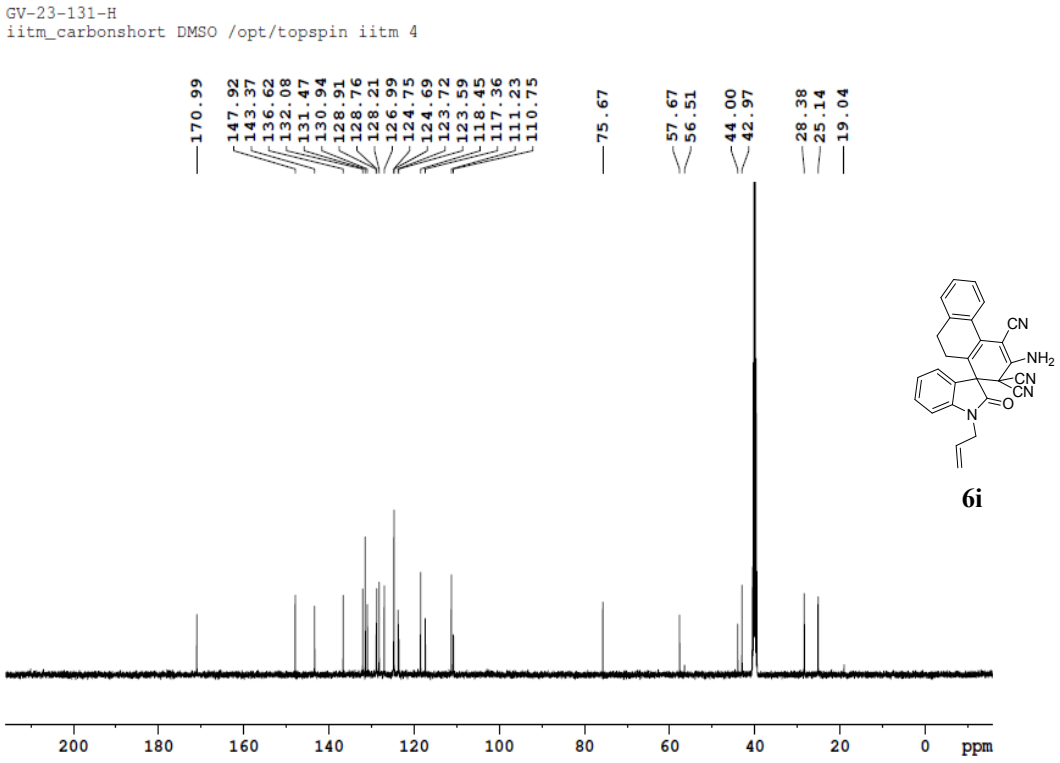




^{1}H NMR and ^{13}C NMR of compound (6i)

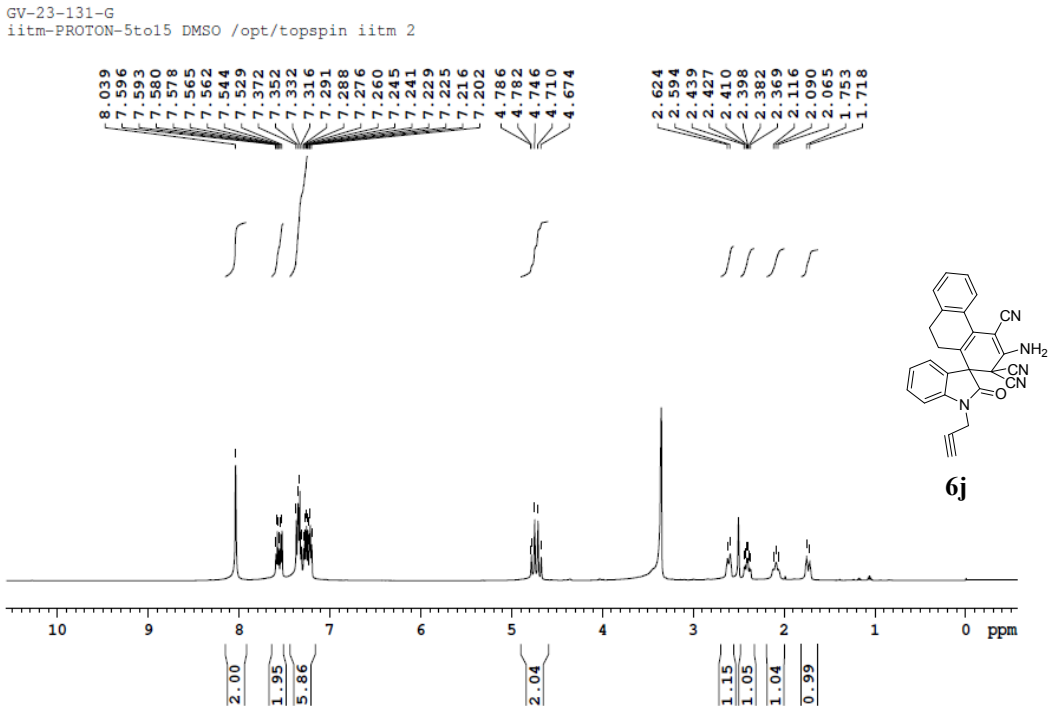


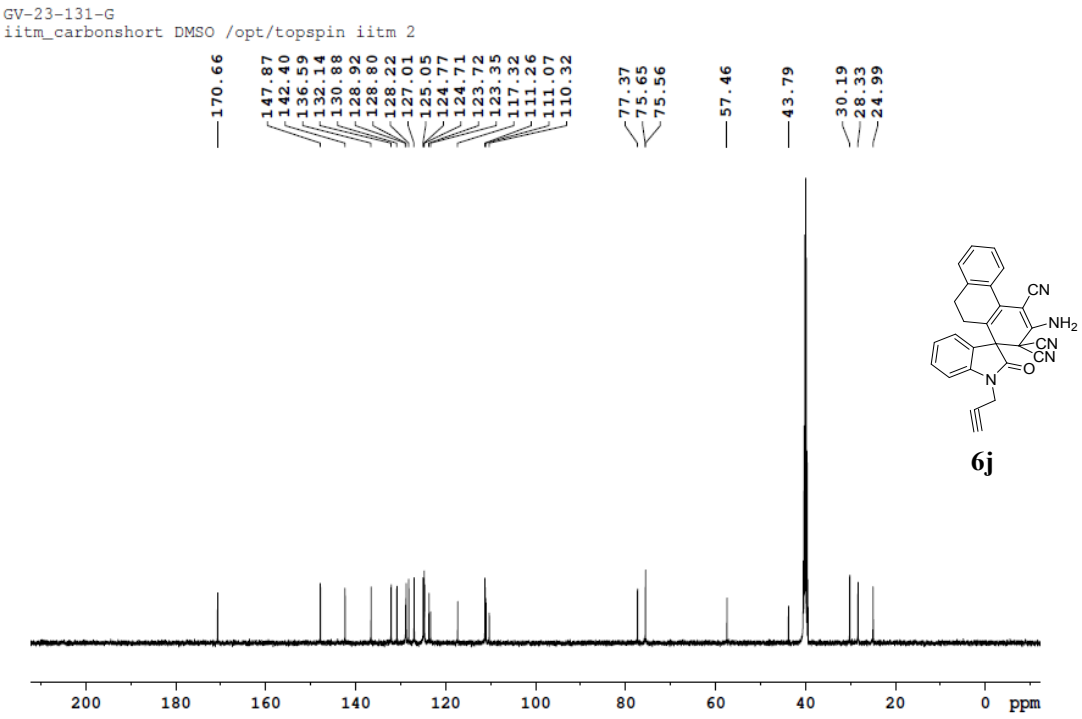
6i



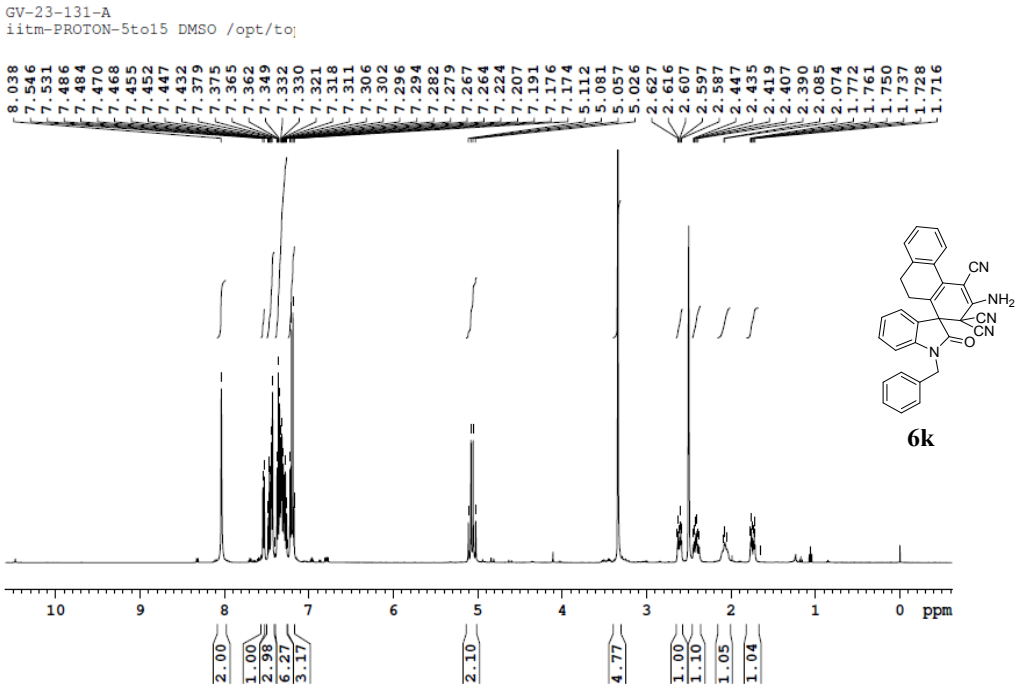
6i

^{1}H NMR and ^{13}C NMR of compound (6j)

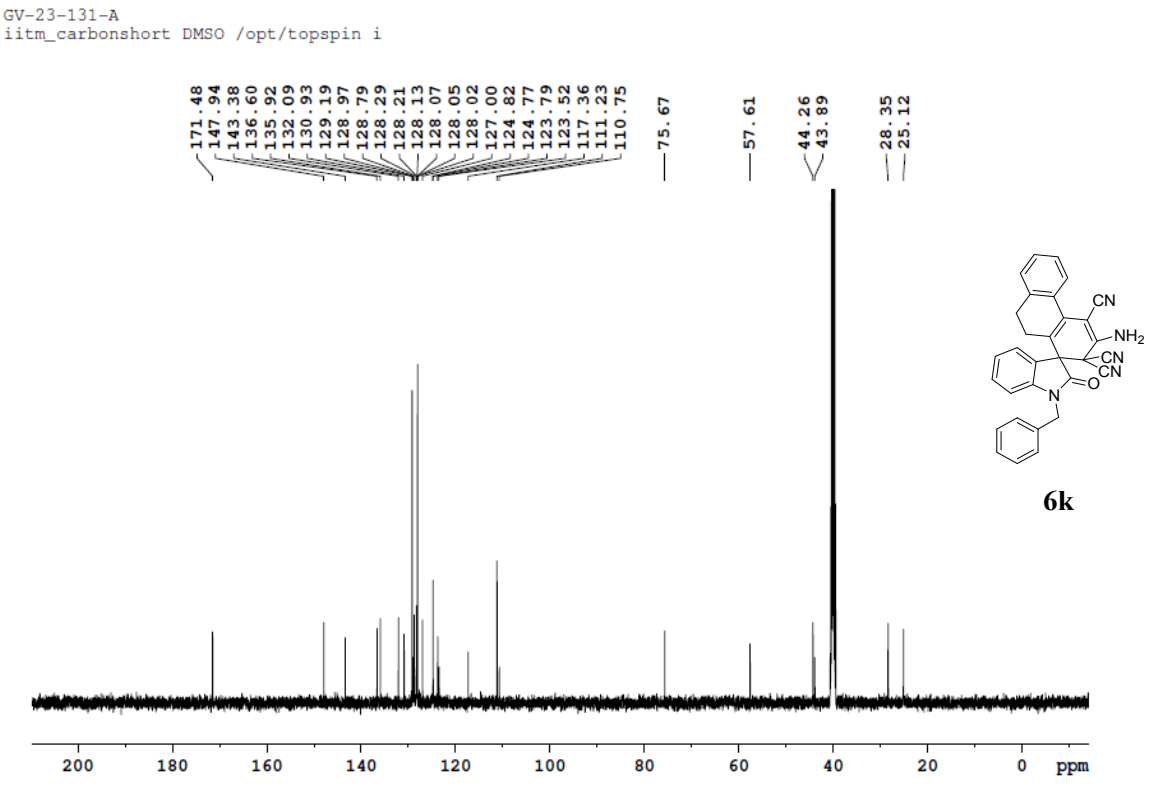




^{1}H NMR and ^{13}C NMR of compound (6k)



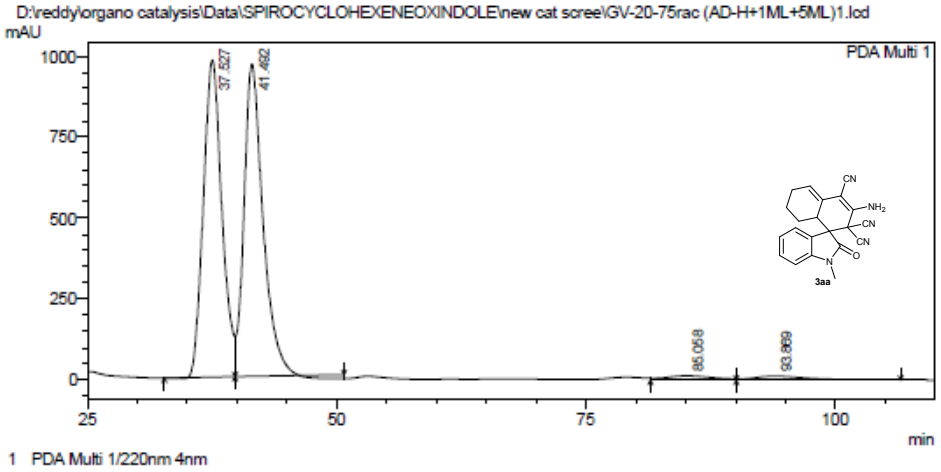
6k



6k

3. HPLC profile

HPLC profile for table 3, entry 1

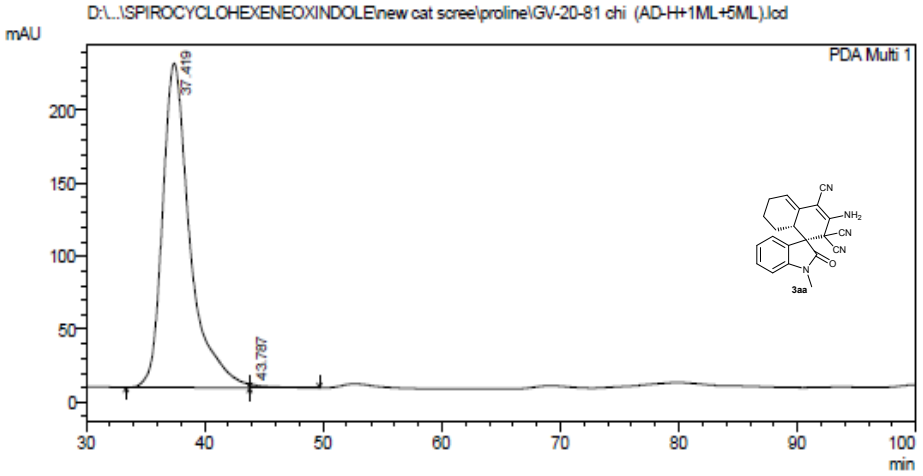


1 PDA Multi 1/220nm 4nm

PeakTable

PDA Ch1 220nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	37.527	126132325	978118	48.358	49.697
2	41.492	125731028	964300	48.205	48.995
3	85.058	4307427	13397	1.651	0.681
4	93.869	4656848	12361	1.785	0.628
Total		260827628	1968175	100.000	100.000



1 PDA Multi 1/220nm 4nm

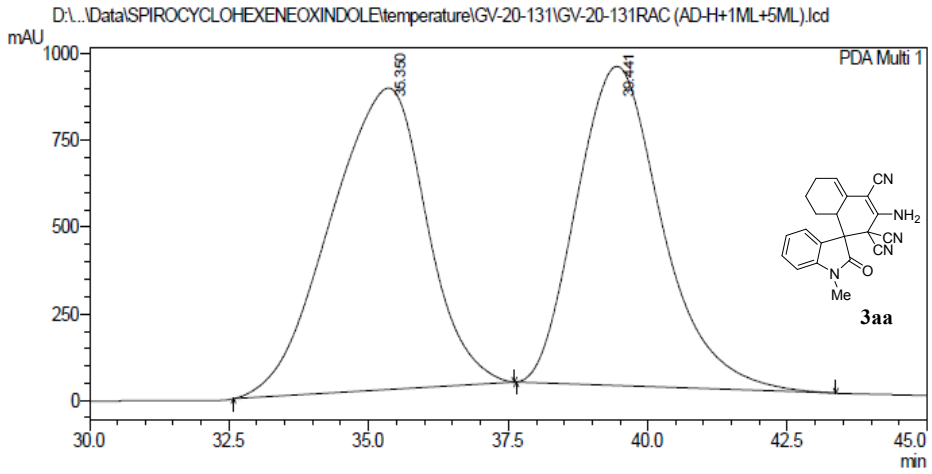
PeakTable

PDA Ch1 220nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	37.419	35721034	222040	99.398	98.942
2	43.787	216302	2375	0.602	1.058
Total		35937335	224415	100.000	100.000

HPLC profile for table 3, entry 1

<Chromatogram>



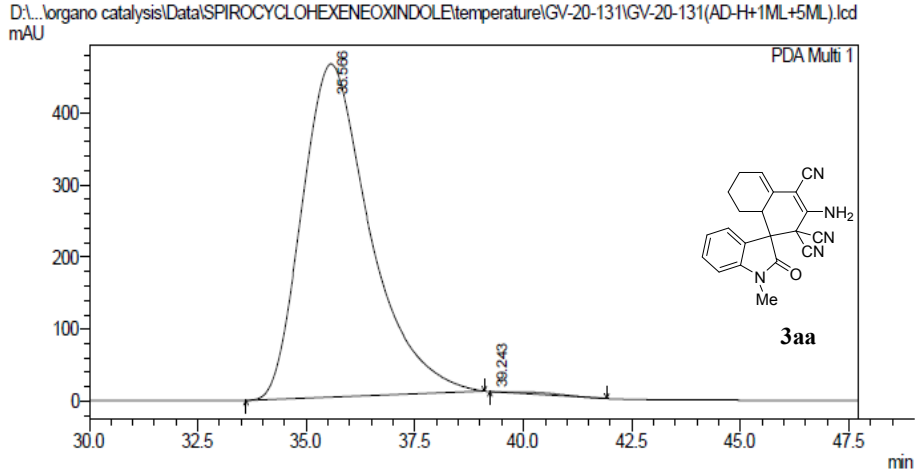
1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	35.350	104540713	869064	51.625	48.580
2	39.441	97958286	919854	48.375	51.420
Total		202498998	1788918	100.000	100.000

<Chromatogram>



1 PDA Multi 1/254nm 4nm

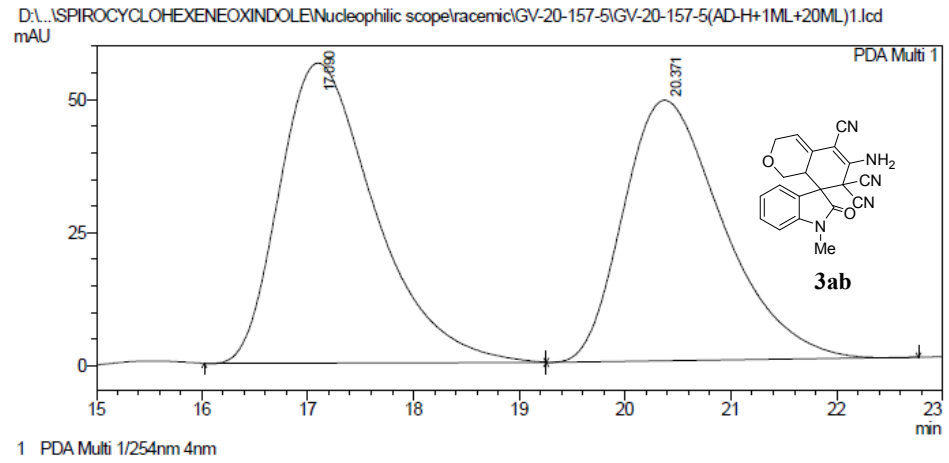
PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	35.566	50278209	462402	99.624	100.005
2	39.243	189844	-22	0.376	-0.005
Total		50468053	462380	100.000	100.000

HPLC profile for table 3, entry 2

<Chromatogram>



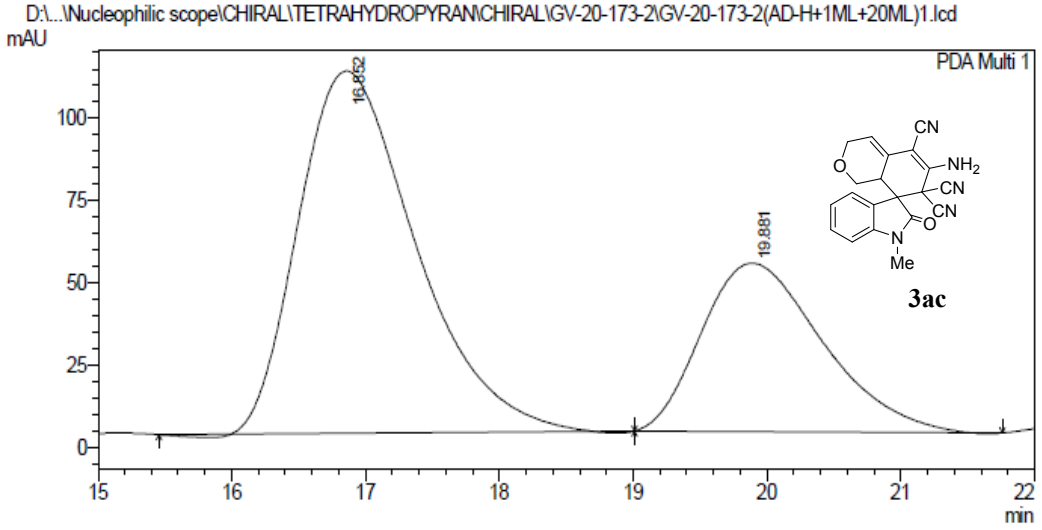
1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	17.090	3535664	56317	52.456	53.521
2	20.371	3204562	48907	47.544	46.479
Total		6740227	105224	100.000	100.000

<Chromatogram>



1 PDA Multi 1/254nm 4nm

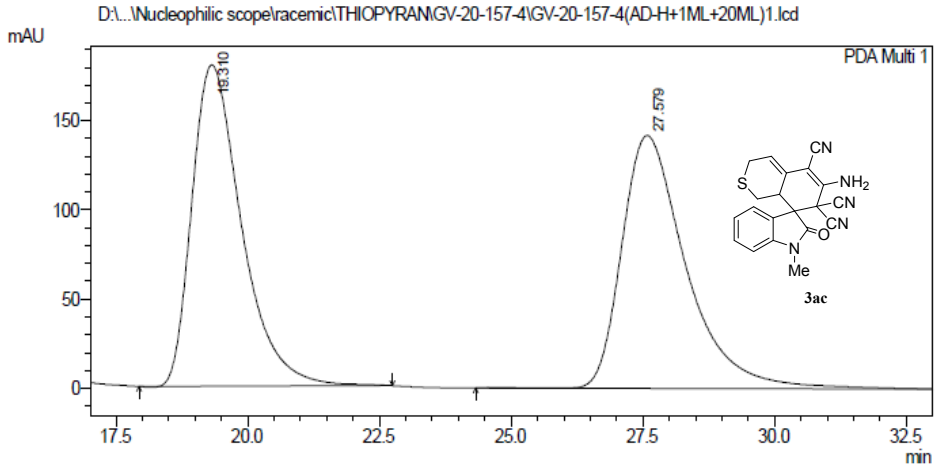
PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	16.852	6669528	109936	67.465	68.254
2	19.881	3216347	51133	32.535	31.746
Total		9885875	161069	100.000	100.000

HPLC profile for table 3, entry 3

<Chromatogram>



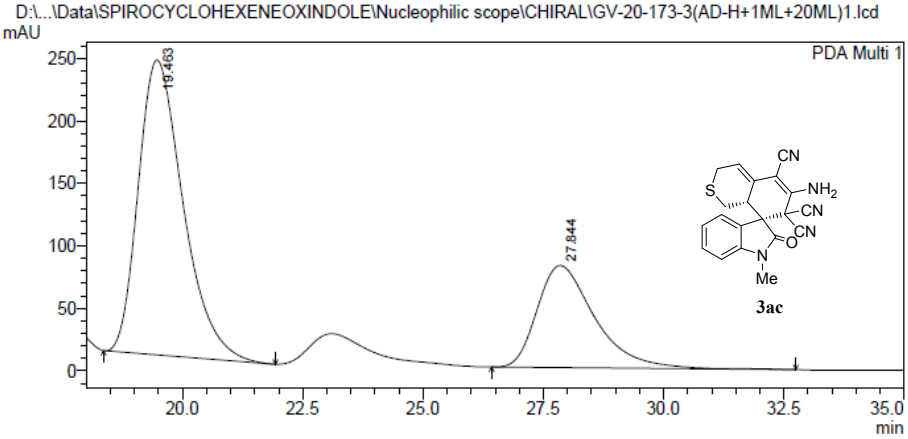
1 PDA Multi 1/220nm 4nm

PeakTable

PDA Ch1 220nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	19.310	11836014	180404	49.569	55.993
2	27.579	12041932	141784	50.431	44.007
Total		23877946	322188	100.000	100.000

<Chromatogram>



1 PDA Multi 1/254nm 4nm

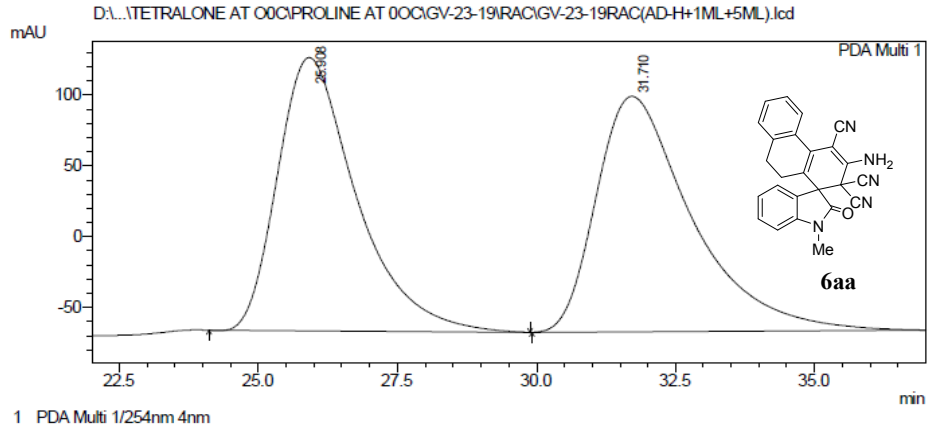
PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	19.463	15284941	235804	69.160	74.269
2	27.844	6816038	81696	30.840	25.731
Total		22100979	317500	100.000	100.000

HPLC profile for table 3, entry 4

<Chromatogram>



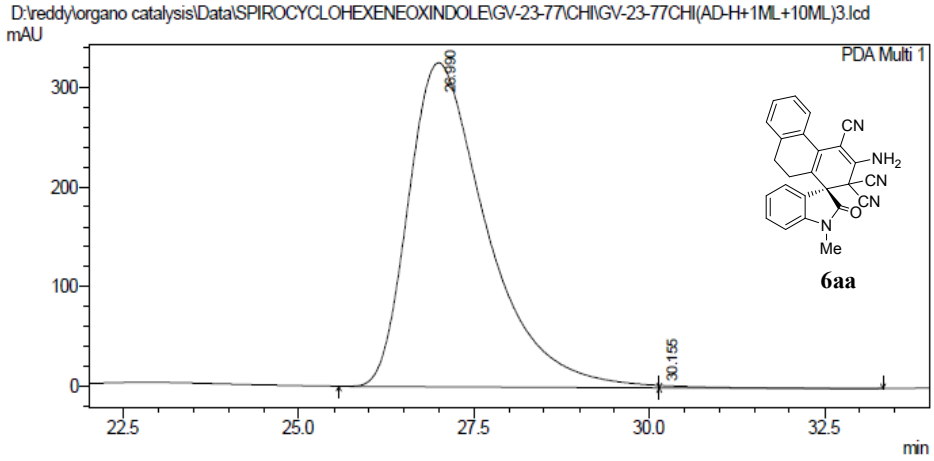
1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	25.908	19025829	192975	48.855	53.726
2	31.710	19917717	166212	51.145	46.274
Total		38943547	359187	100.000	100.000

<Chromatogram>



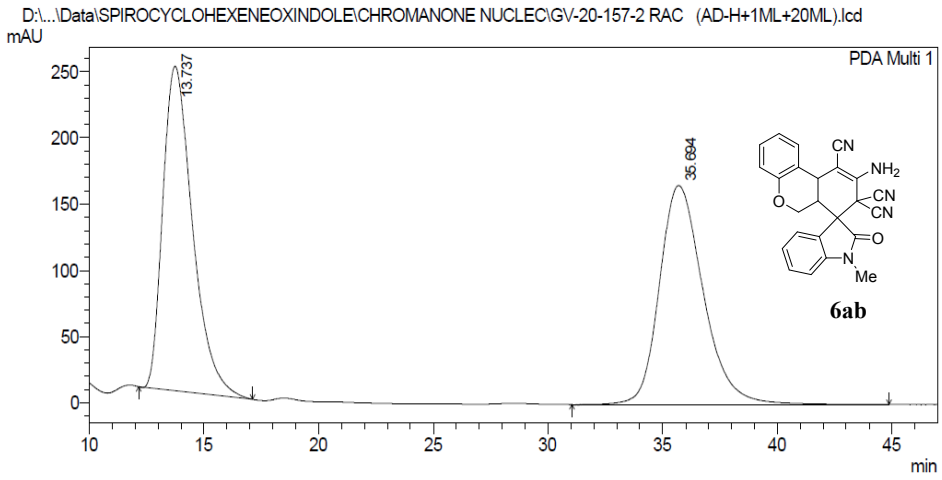
1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	26.990	25271488	324851	99.808	99.328
2	30.155	48688	2197	0.192	0.672
Total		25320176	327048	100.000	100.000

HPLC profile for table 3, entry 5



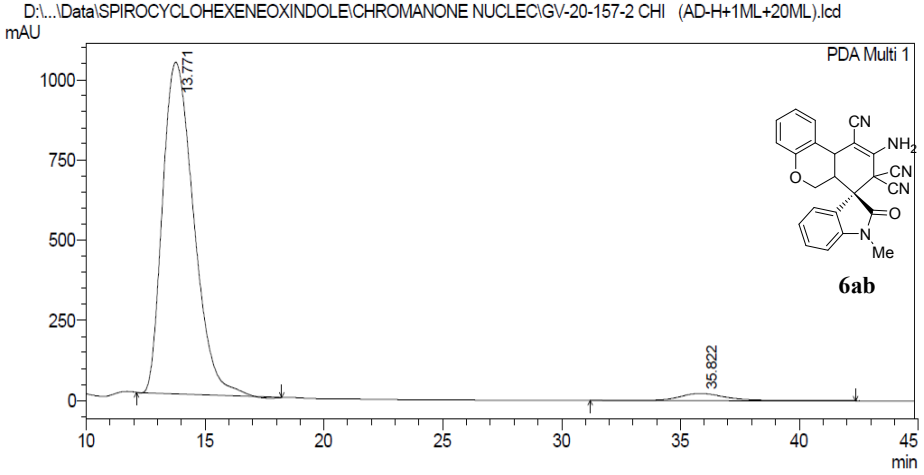
1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.737	22049259	244827	50.013	59.681
2	35.694	22038080	165396	49.987	40.319
Total		44087339	410224	100.000	100.000

<Chromatogram>



1 PDA Multi 1/254nm 4nm

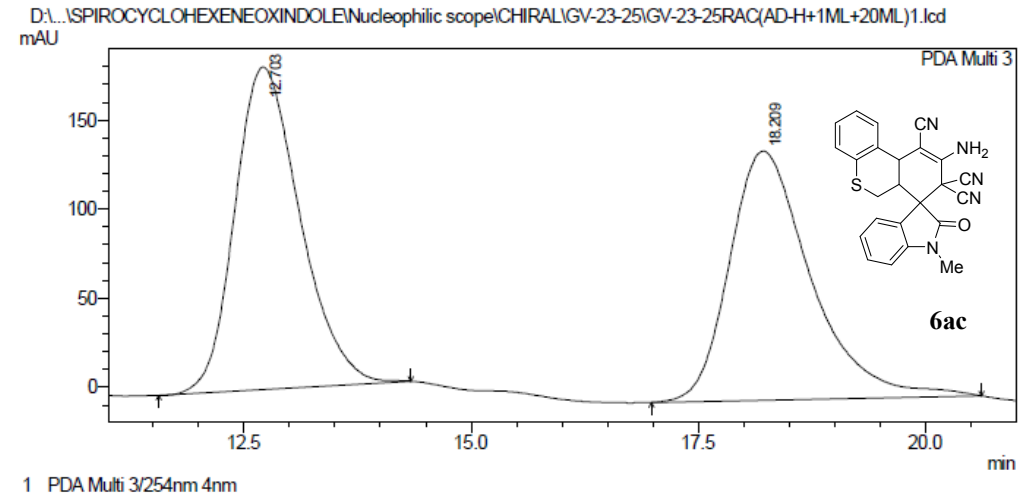
PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.771	93968016	1032900	96.998	97.921
2	35.822	2908598	21931	3.002	2.079
Total		96876614	1054830	100.000	100.000

HPLC profile for table 3, entry 6

<Chromatogram>



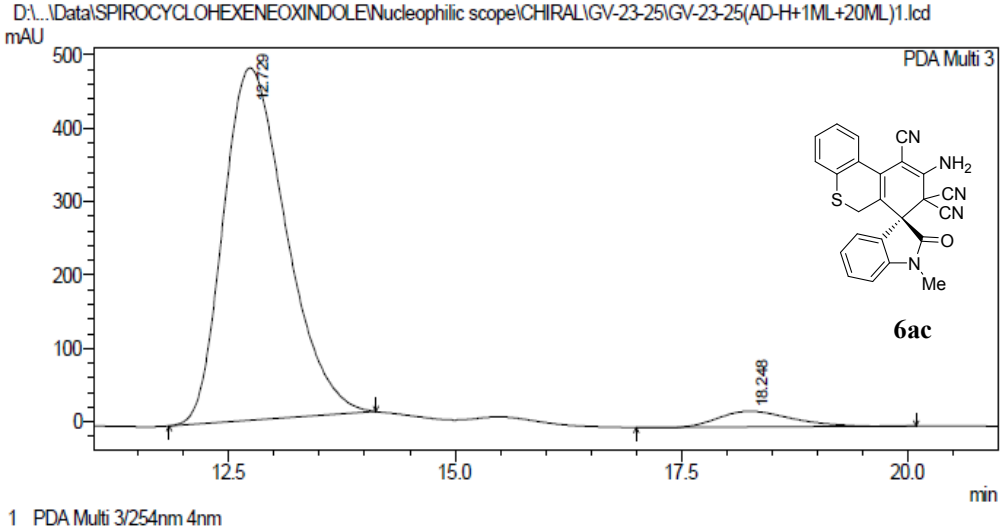
1 PDA Multi 3/254nm 4nm

PeakTable

PDA Ch3 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	12.703	9053326	180819	50.918	56.406
2	18.209	8726904	139746	49.082	43.594
Total		17780230	320565	100.000	100.000

<Chromatogram>



1 PDA Multi 3/254nm 4nm

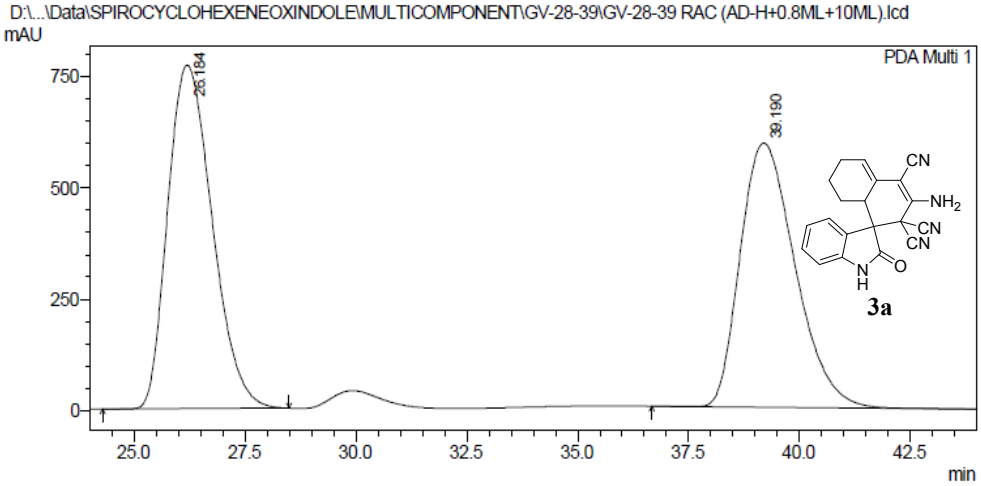
PeakTable

PDA Ch3 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	12.729	23274726	480499	95.173	95.749
2	18.248	1180501	21332	4.827	4.251
Total		24455227	501831	100.000	100.000

HPLC profile for table 4, entry 1

<Chromatogram>



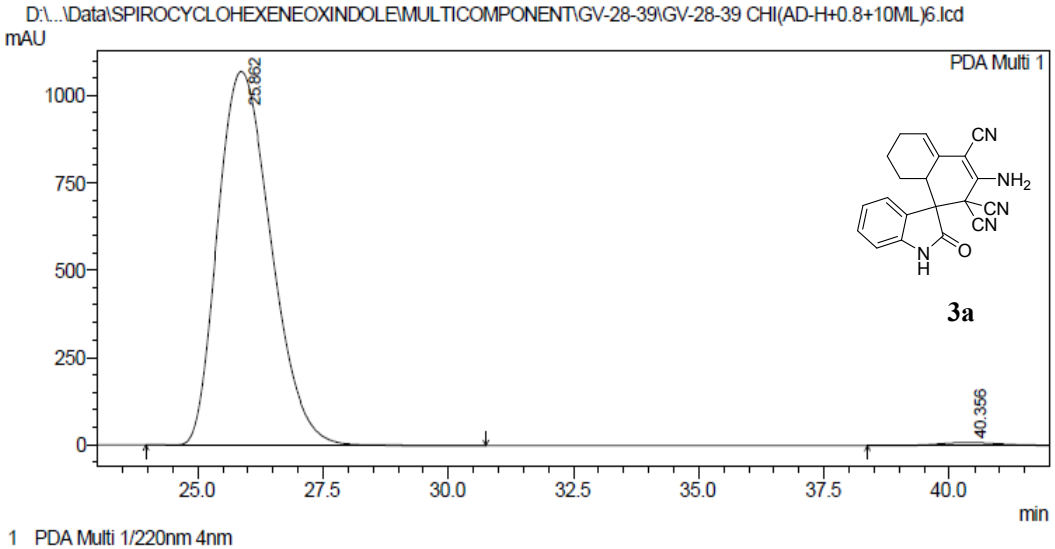
1 PDA Multi 1/220nm 4nm

PeakTable

PDA Ch1 220nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	26.184	53834765	770057	51.370	56.535
2	39.190	50963493	592035	48.630	43.465
Total		104798258	1362092	100.000	100.000

<Chromatogram>



1 PDA Multi 1/220nm 4nm

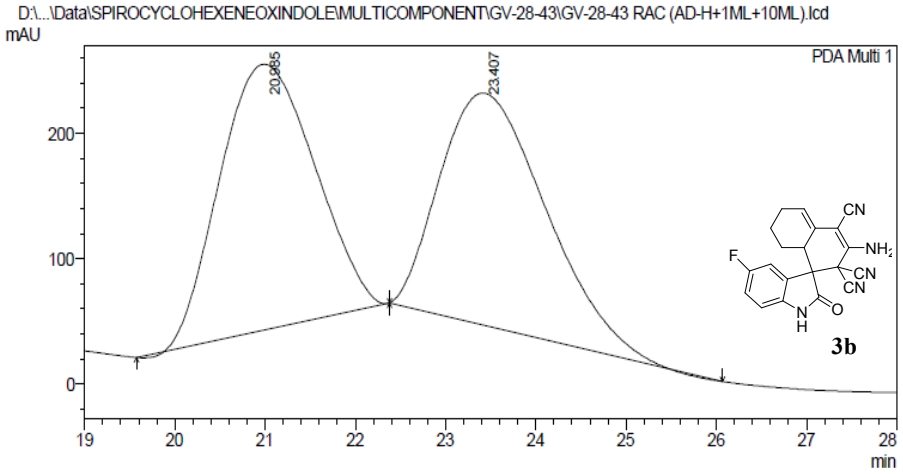
PeakTable

PDA Ch1 220nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	25.862	79156056	1070465	99.051	99.180
2	40.356	758749	8852	0.949	0.820
Total		79914806	1079317	100.000	100.000

HPLC profile for table 4, entry 2

<Chromatogram>



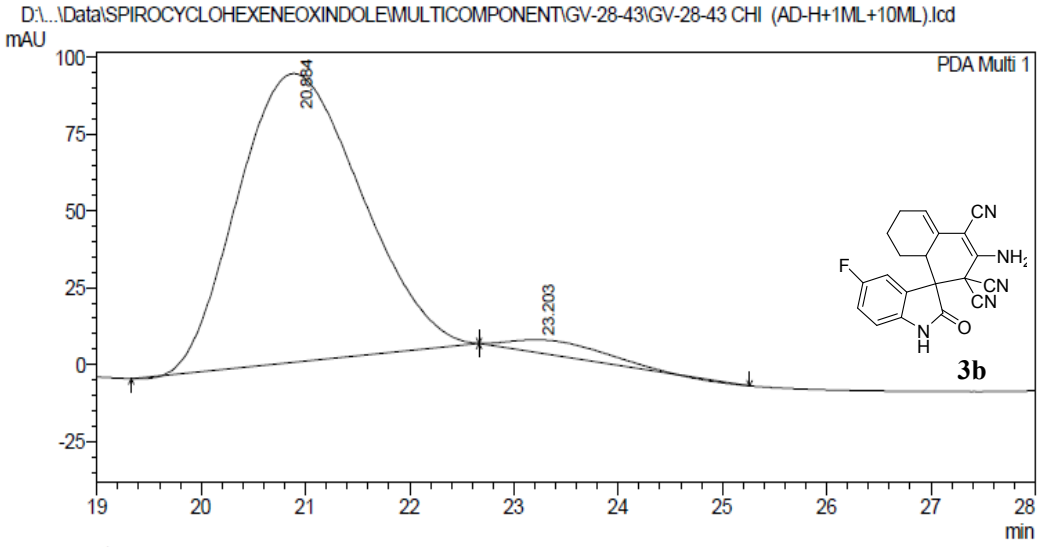
1 PDA Multi 1/220nm 4nm

PeakTable

PDA Ch1 220nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	20.985	15457491	212066	50.455	53.430
2	23.407	15178935	184837	49.545	46.570
Total		30636426	396904	100.000	100.000

<Chromatogram>



1 PDA Multi 1/220nm 4nm

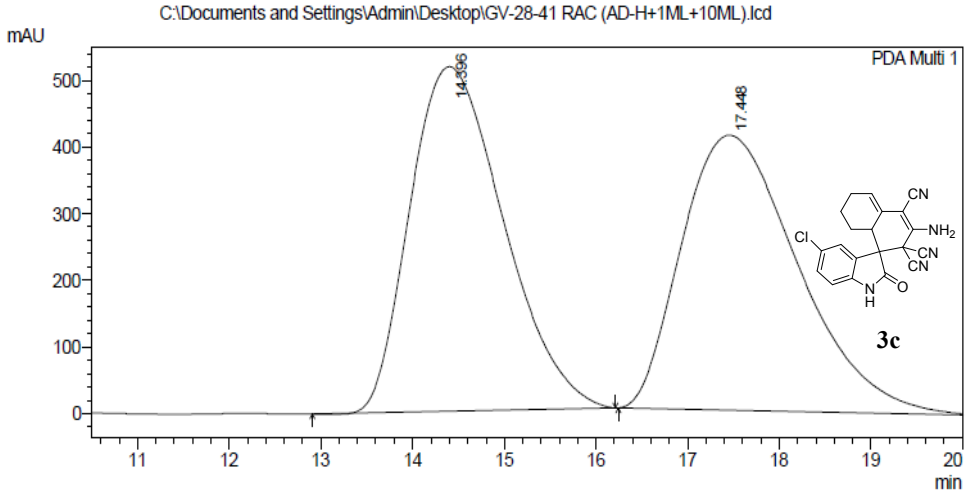
PeakTable

PDA Ch1 220nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	20.884	7694062	93850	96.561	95.881
2	23.203	274042	4032	3.439	4.119
Total		7968104	97882	100.000	100.000

HPLC profile for table 4, entry 3

<Chromatogram>



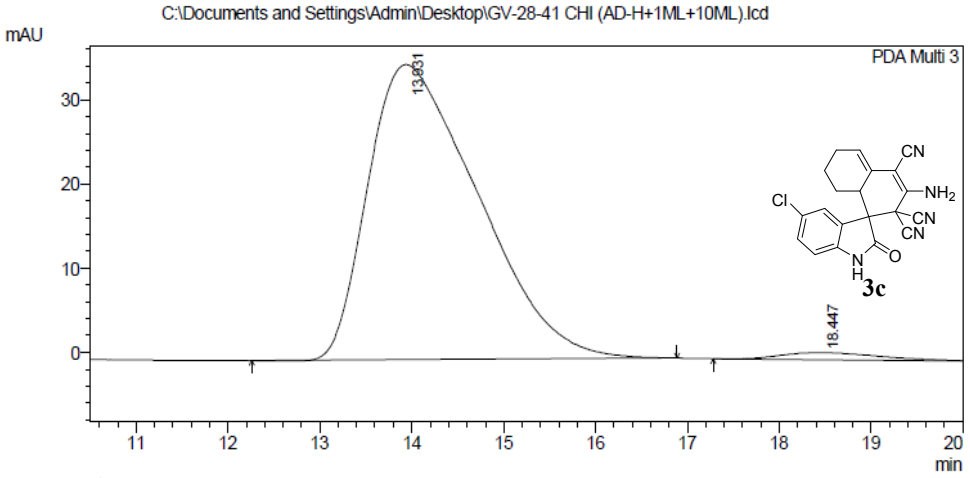
1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	14.396	36738169	517389	50.348	55.634
2	17.448	36229930	412599	49.652	44.366
Total		72968100	929988	100.000	100.000

<Chromatogram>



1 PDA Multi 3/254nm 4nm

PeakTable

PDA Ch3 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.931	2914010	35039	97.908	97.659
2	18.447	62262	840	2.092	2.341
Total		2976272	35879	100.000	100.000

HPLC profile for table 4, entry 4

<Chromatogram>

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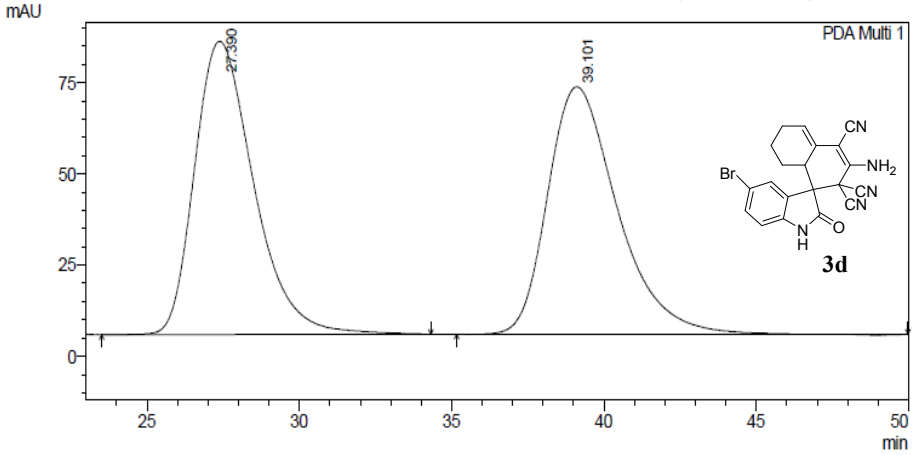


1 PDA Multi 1/220nm 4nm

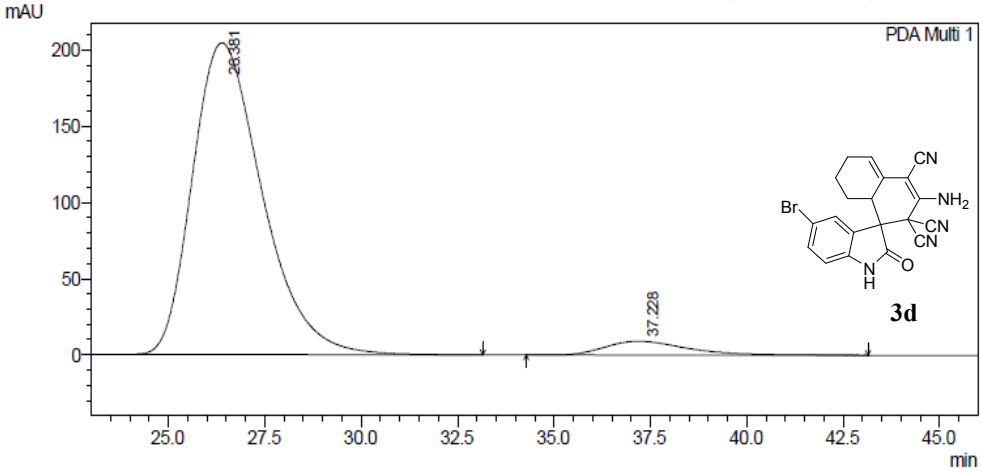
PeakTable

PDA Ch1 220nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	27.390	10858802	80373	50.122	54.238
2	39.101	10805864	67813	49.878	45.762
Total		21664666	148186	100.000	100.000

<Chromatogram>

D:\...\Data\SPIROCYCLOHEXENEOXINDOLE\MULTICOMPONENT\GV-28-45\GV-28-45 CHI (AD-H+1ML+10ML).lcd



1 PDA Multi 1/220nm 4nm

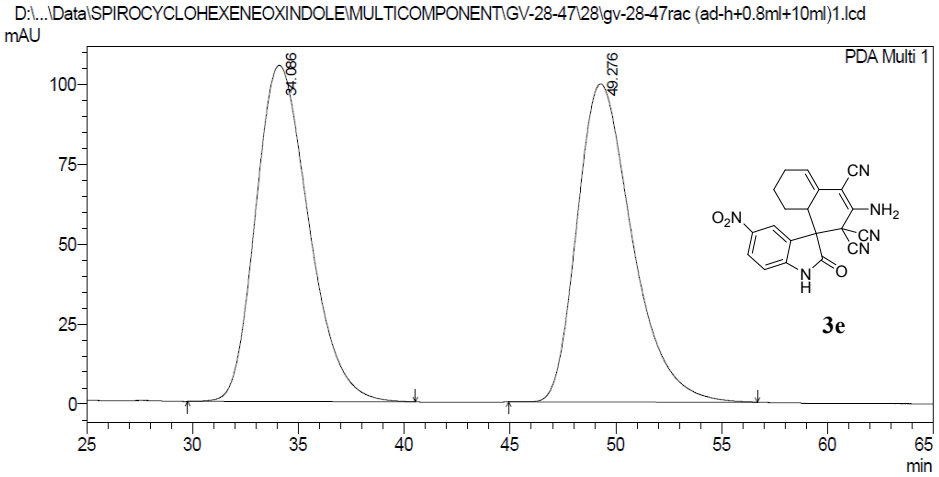
PeakTable

PDA Ch1 220nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	26.381	25749610	204497	95.049	95.731
2	37.228	1341406	9119	4.951	4.269
Total		27091016	213616	100.000	100.000

HPLC profile for table 4, entry 5

<Chromatogram>



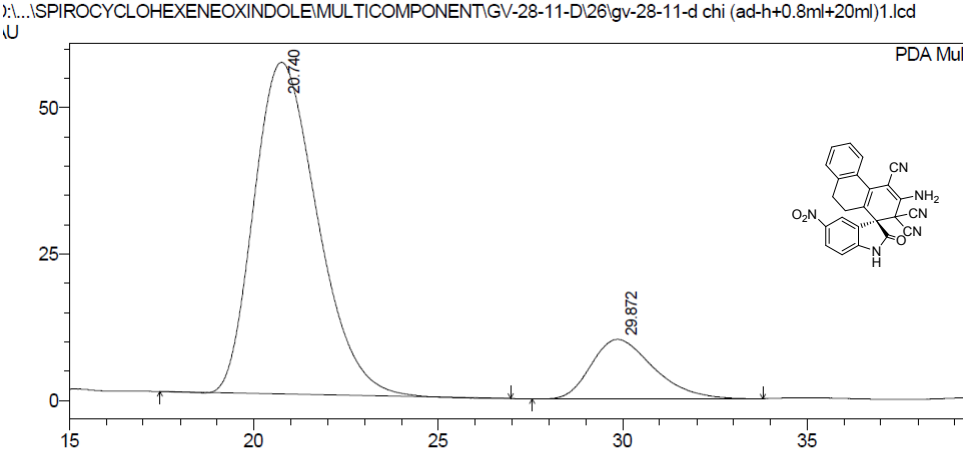
1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	34.086	18365656	105228	50.421	51.396
2	49.276	18059000	99511	49.579	48.604
Total		36424656	204739	100.000	100.000

romatogram>



PDA Multi 1/254nm 4nm

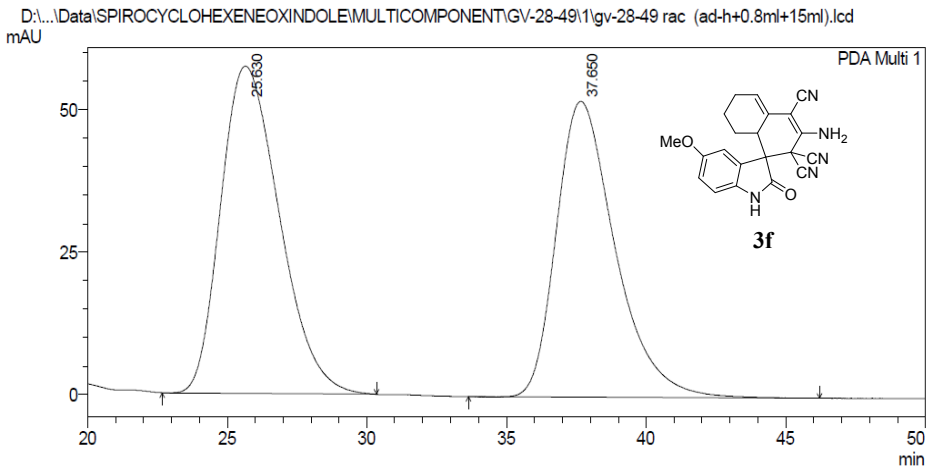
PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	20.740	6773208	56621	85.065	84.815
2	29.872	1189197	10137	14.935	15.185
Total		7962405	66758	100.000	100.000

HPLC profile for table 4, entry 6

<Chromatogram>

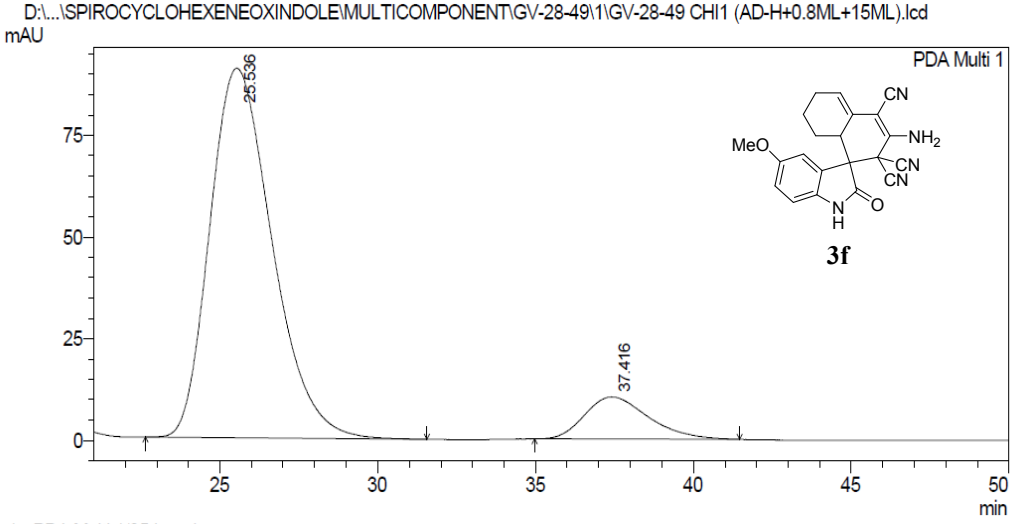


PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	25.630	8387900	57362	52.451	52.508
2	37.650	7603951	51881	47.549	47.492
Total		15991851	109243	100.000	100.000

<Chromatogram>

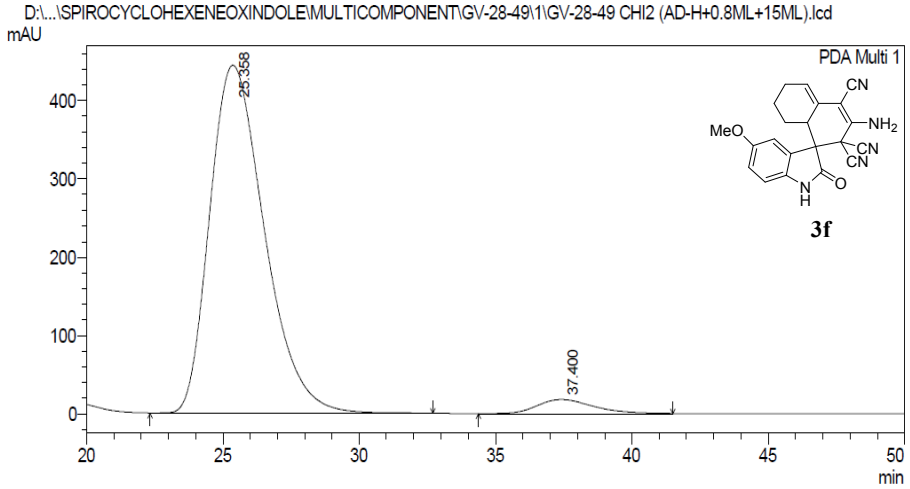


PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	25.536	12482481	90869	89.490	89.751
2	37.416	1465946	10376	10.510	10.249
Total		13948427	101245	100.000	100.000

HPLC profile for table 4, entry 6 (at -10°c)



1 PDA Multi 1/254nm 4nm

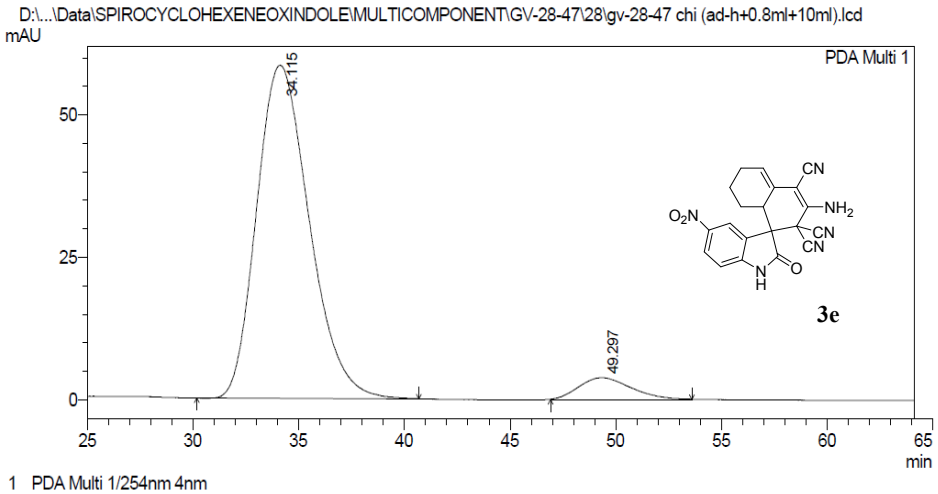
PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	25.358	61884989	443964	96.013	96.092
2	37.400	2569935	18056	3.987	3.908
Total		64454924	462020	100.000	100.000

HPLC profile for table 4, entry 5 at -10°c

<Chromatogram>



1 PDA Multi 1/254nm 4nm

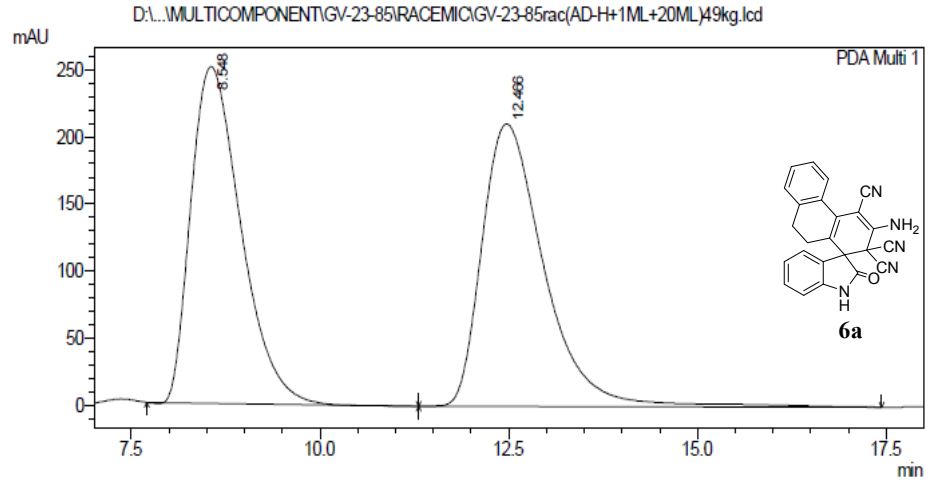
PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	34.115	10121196	58455	94.041	93.928
2	49.297	641392	3779	5.959	6.072
Total		10762589	62233	100.000	100.000

HPLC profile for table 4, entry 7

<Chromatogram>



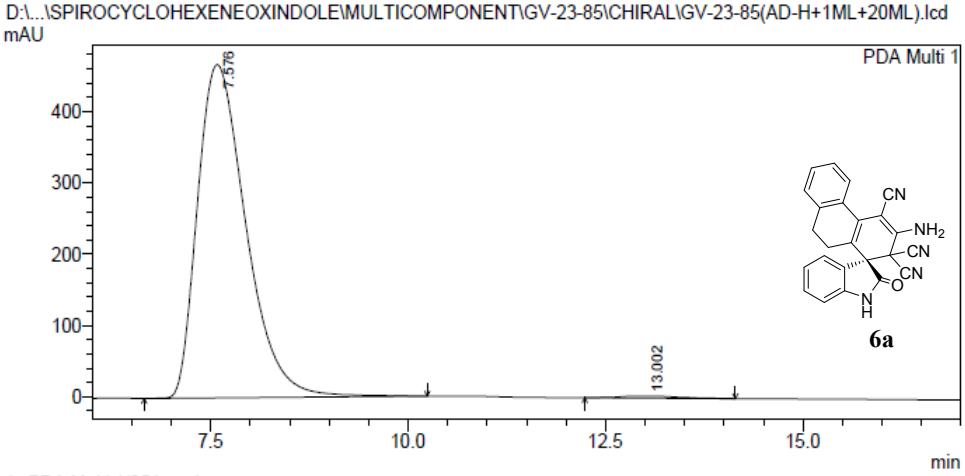
1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.548	11534641	250696	48.884	54.381
2	12.466	12061234	210305	51.116	45.619
Total		23595875	461001	100.000	100.000

<Chromatogram>



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.576	19939714	467542	99.286	99.404
2	13.002	143425	2803	0.714	0.596
Total		20083139	470346	100.000	100.000

HPLC profile for table 4, entry 8

<Chromatogram>

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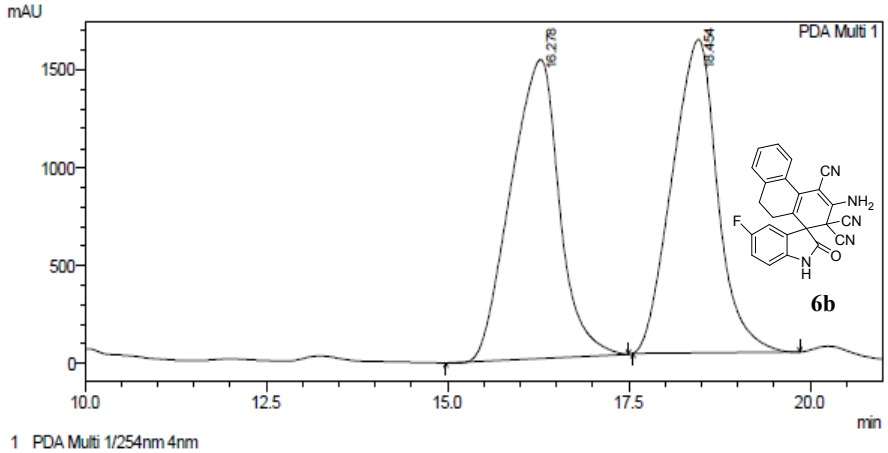


1 PDA Multi 1/254nm 4nm

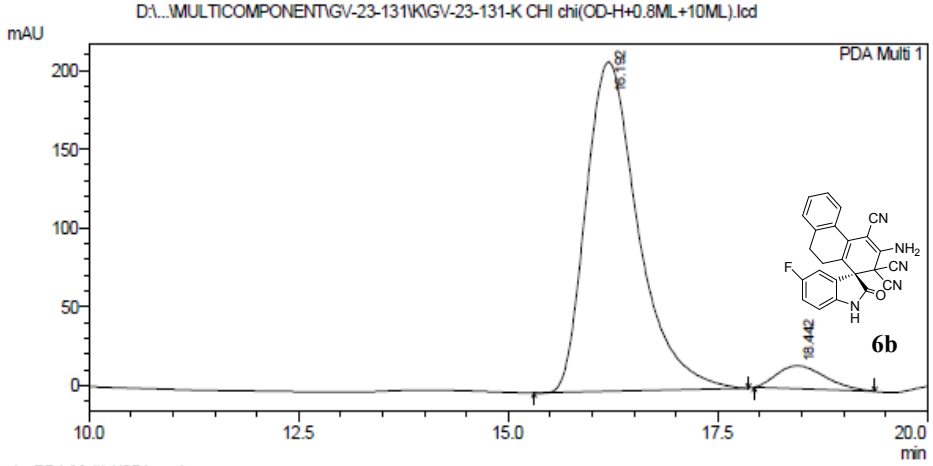
PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	16.278	71308376	1526252	50.447	48.816
2	18.454	70044524	1600295	49.553	51.184
Total		141352900	3126547	100.000	100.000

<Chromatogram>

D:\...\MULTICOMPONENT\GV-23-131\K\GV-23-131-K CHI chi(OD-H+0.8ML+10ML).lcd



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	16.192	9063823	208976	93.901	93.485
2	18.442	588727	14565	6.099	6.515
Total		9652551	223540	100.000	100.000

HPLC profile for table 4, entry 9

<Chromatogram>

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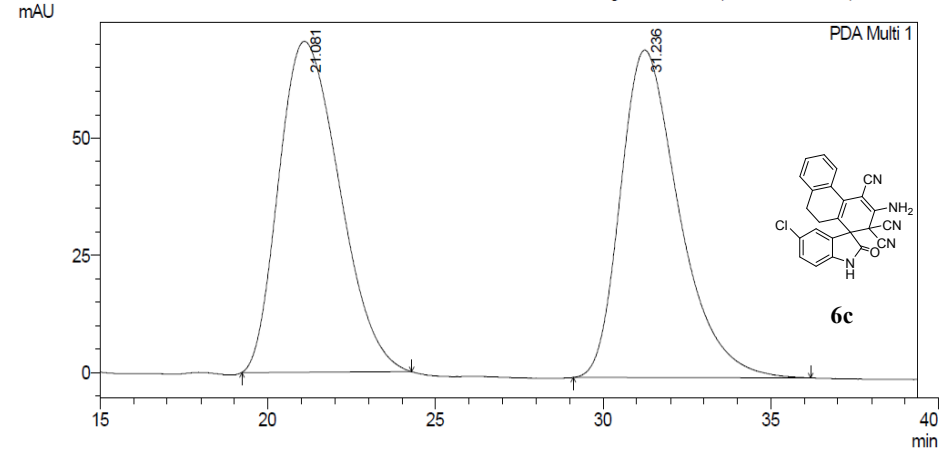


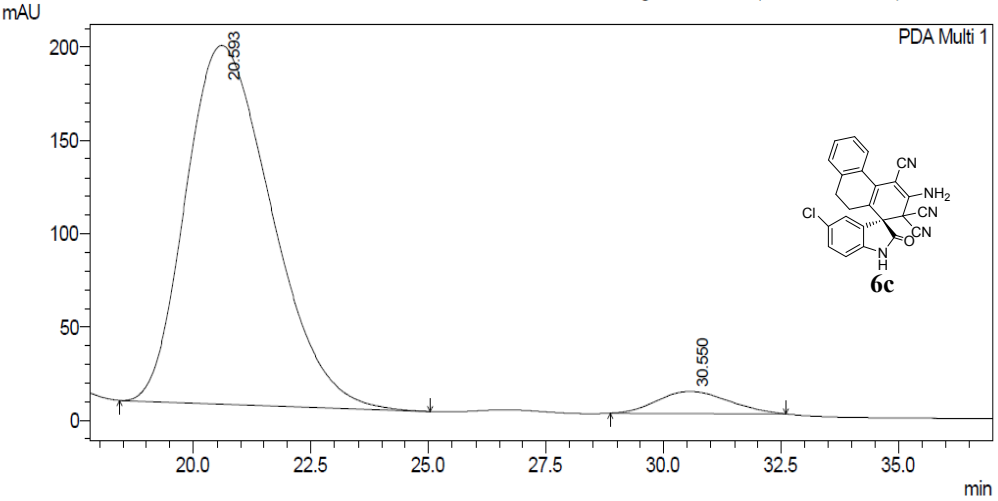
1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	21.081	8897565	70583	51.101	50.258
2	31.236	8513996	69857	48.899	49.742
Total		17411561	140440	100.000	100.000

D:\...\Data\SPIROCYCLOHEXENEOXINDOLE\MULTICOMPONENT\GV-28-11-C\1\gv-28-11-c chi (ad-h+0.8ml+20ml).lcd



1 PDA Multi 1/254nm 4nm

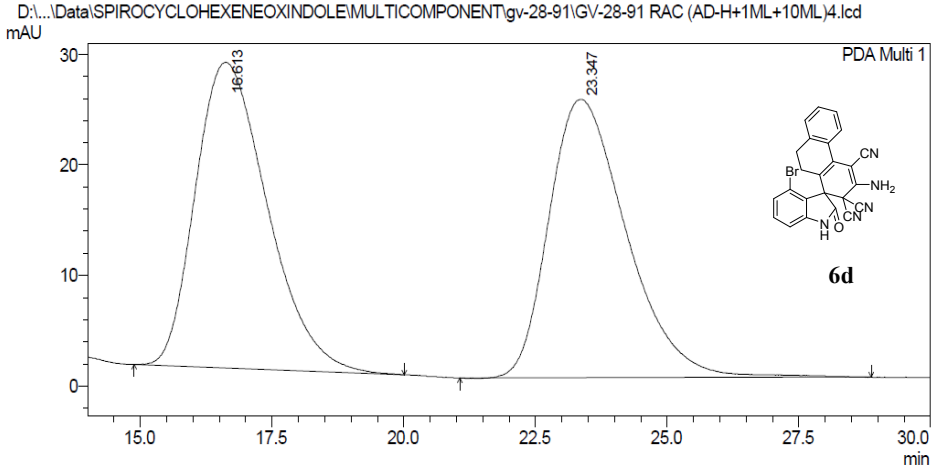
PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	20.593	24353023	192066	95.078	94.175
2	30.550	1260814	11881	4.922	5.825
Total		25613837	203947	100.000	100.000

HPLC profile for table 4, entry 10

<Chromatogram>

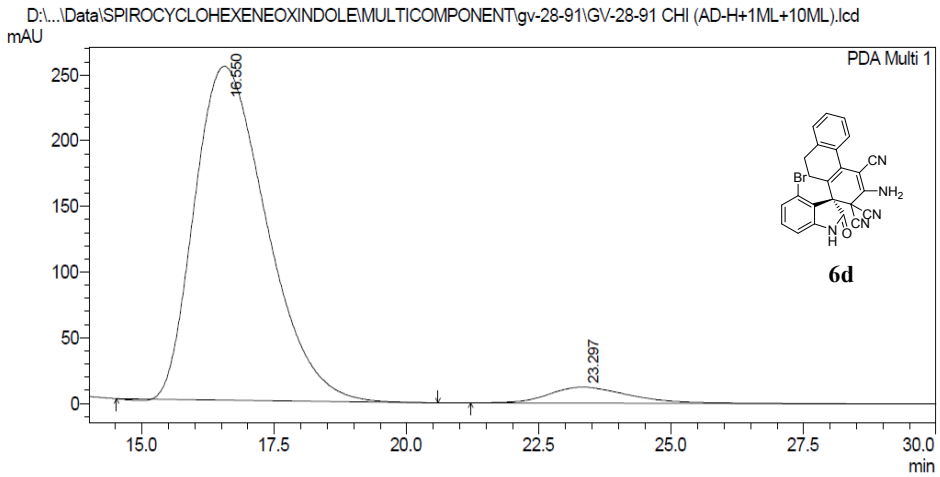


PeakTable

PDA Ch1 220nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	16.613	2632034	27682	50.399	52.312
2	23.347	2590333	25235	49.601	47.688
Total		5222366	52917	100.000	100.000

<Chromatogram>



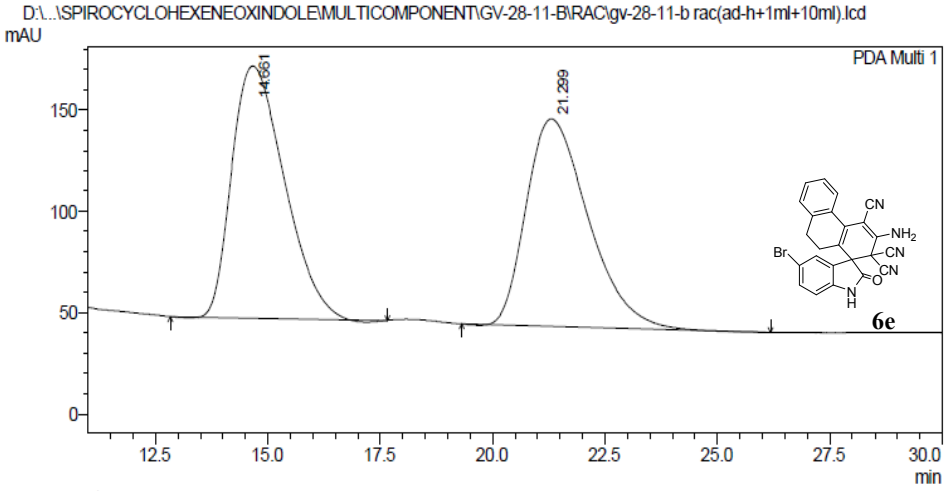
PeakTable

PDA Ch1 220nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	16.550	23982987	253763	95.088	95.441
2	23.297	1238973	12123	4.912	4.559
Total		25221961	265886	100.000	100.000

HPLC profile for table 4, entry 11

<Chromatogram>



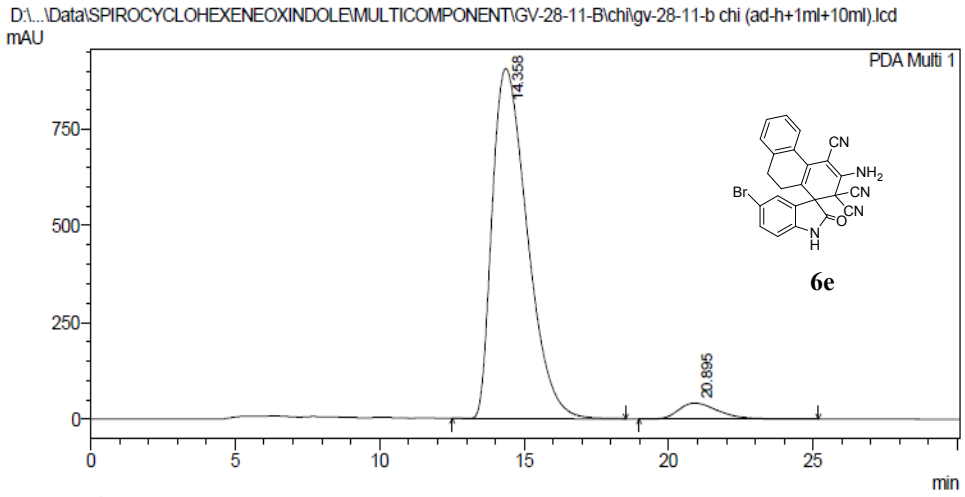
1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	14.661	10096335	124382	50.682	54.851
2	21.299	9824503	102380	49.318	45.149
Total		19920838	226761	100.000	100.000

<Chromatogram>



1 PDA Multi 1/254nm 4nm

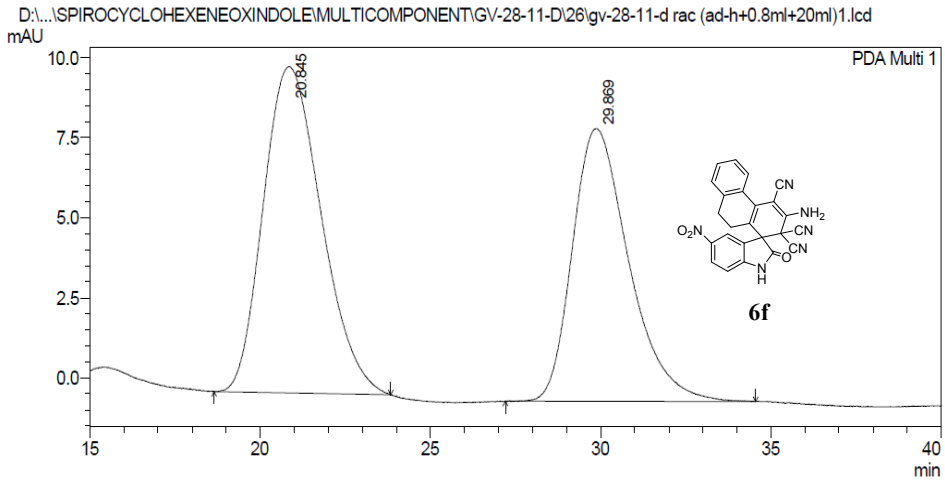
PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	14.358	76296081	904633	95.134	95.650
2	20.895	3902816	41145	4.866	4.350
Total		80198898	945777	100.000	100.000

HPLC profile for table 4, entry 12

<Chromatogram>



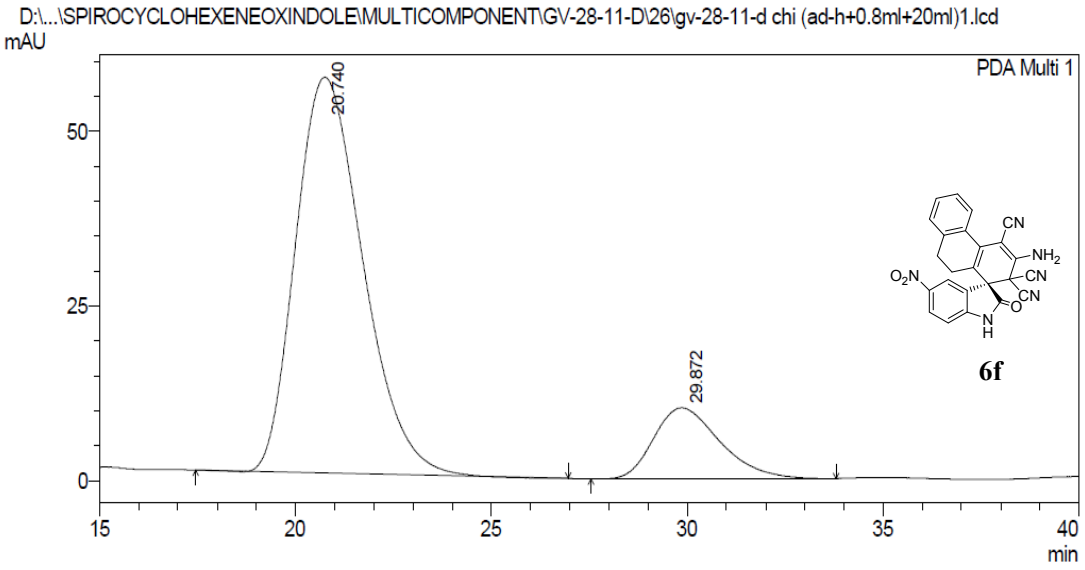
1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	20.845	1209511	10198	55.234	54.487
2	29.869	980276	8518	44.766	45.513
Total		2189787	18716	100.000	100.000

<Chromatogram>



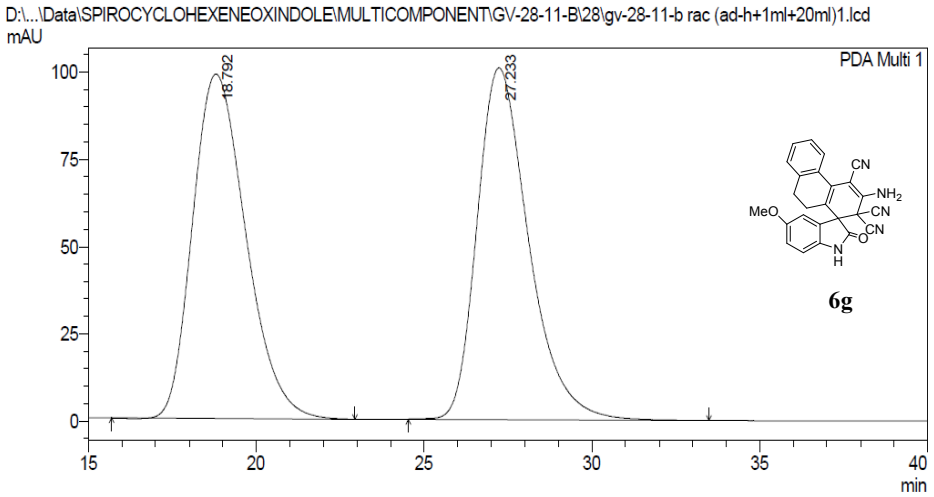
1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	20.740	6773208	56621	85.065	84.815
2	29.872	1189197	10137	14.935	15.185
Total		7962405	66758	100.000	100.000

HPLC profile for table 4, entry 13



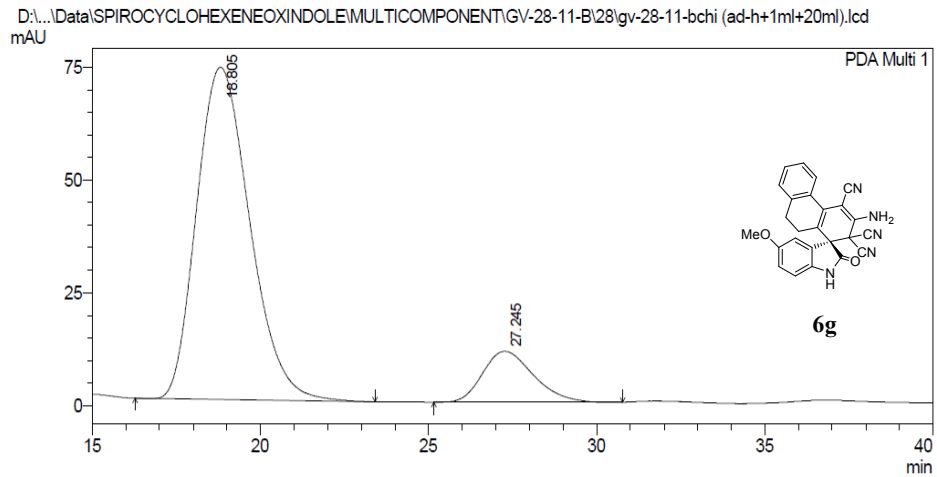
1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	18.792	10863724	98604	50.233	49.482
2	27.233	10762853	100670	49.767	50.518
Total		21626577	199274	100.000	100.000

<Chromatogram>



1 PDA Multi 1/254nm 4nm

PeakTable

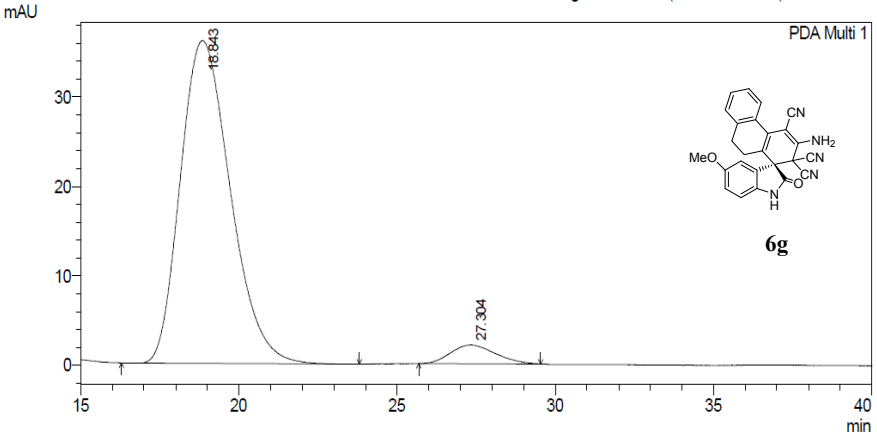
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	18.805	8100354	73694	87.289	86.713
2	27.245	1179561	11292	12.711	13.287
Total		9279916	84987	100.000	100.000

HPLC profile for table 4, entry 13 (at -10 °C)

<Chromatogram>

D:\...\Data\SPIROCYCLOHEXENEOXINDOLE\MULTICOMPONENT\GV-28-11-B\28\gv-28-11-bchi (ad-h+1ml+20ml)1.lcd



6g

1 PDA Multi 1/254nm 4nm

PeakTable

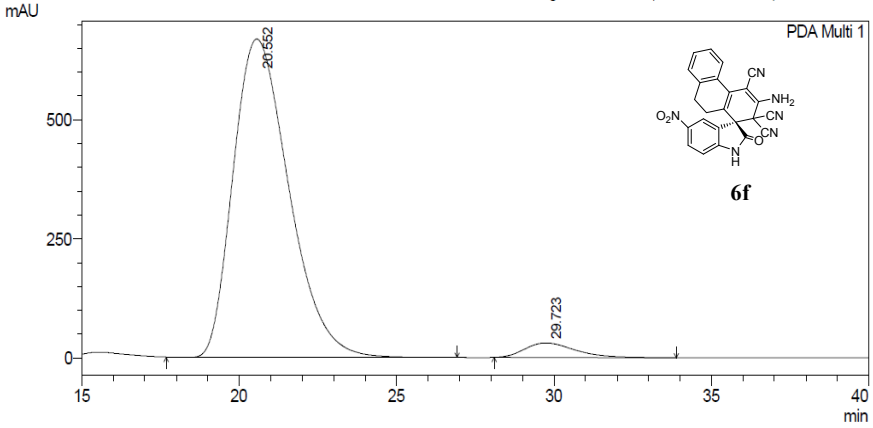
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	18.843	3955908	36092	95.045	94.516
2	27.304	206214	2094	4.955	5.484
Total		4162122	38186	100.000	100.000

HPLC profile for table 4, entry 12 (at -10 °C)

<Chromatogram>

D:\...\SPIROCYCLOHEXENEOXINDOLE\MULTICOMPONENT\GV-28-11-D\26\gv-28-11-d chi (ad-h+0.8ml+20ml).lcd



6f

1 PDA Multi 1/254nm 4nm

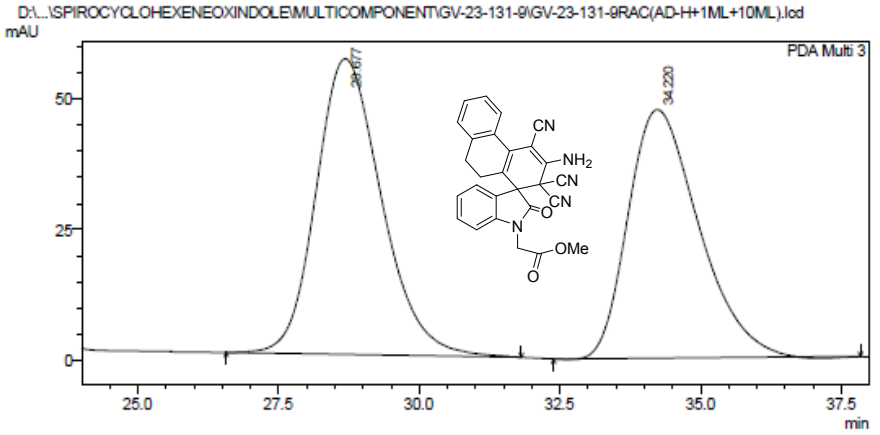
PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	20.552	82257289	668265	95.960	95.575
2	29.723	3462990	30937	4.040	4.425
Total		85720280	699202	100.000	100.000

HPLC profile for table 4, entry 14

<Chromatogram>



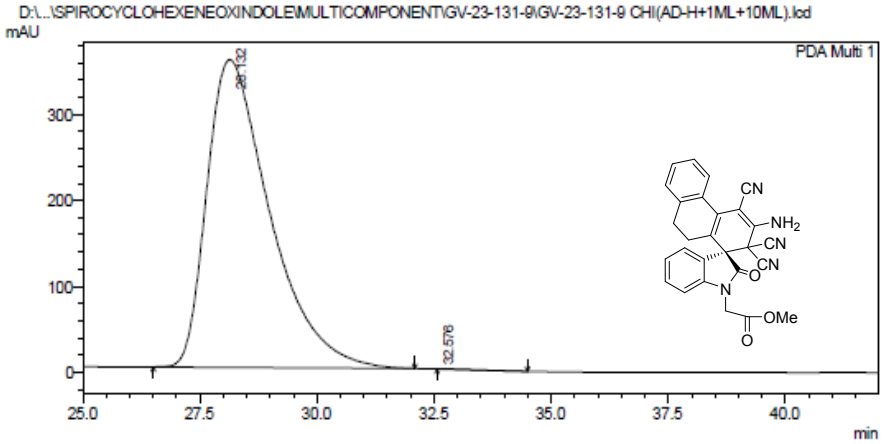
1 PDA Multi 3/254nm 4nm

PeakTable

PDA Ch3 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	28.677	4588752	56674	52.499	54.327
2	34.220	4151824	47646	47.501	45.673
Total		8740576	104320	100.000	100.000

<Chromatogram>



1 PDA Multi 1/254nm 4nm

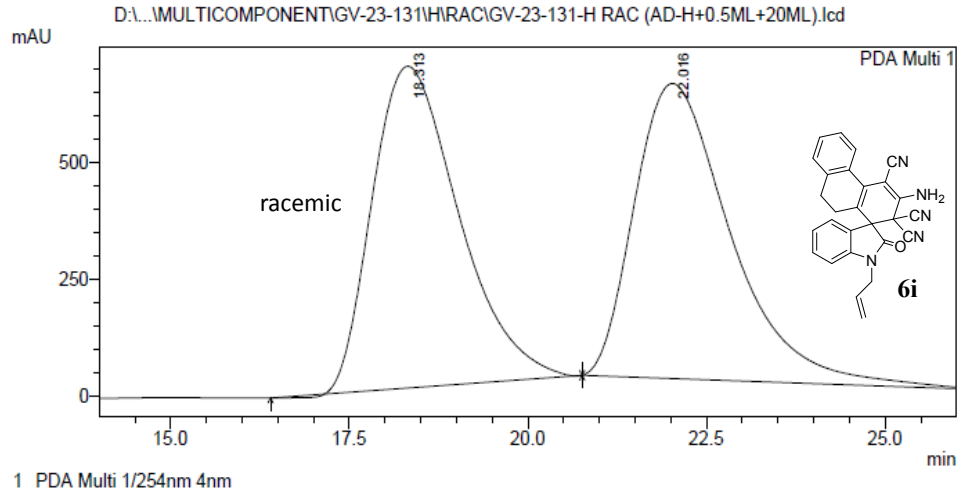
PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	28.132	33460094	357836	99.987	99.999
2	32.576	4332	2	0.013	0.001
Total		33464425	357838	100.000	100.000

HPLC profile for table 4, entry 15

<Chromatogram>



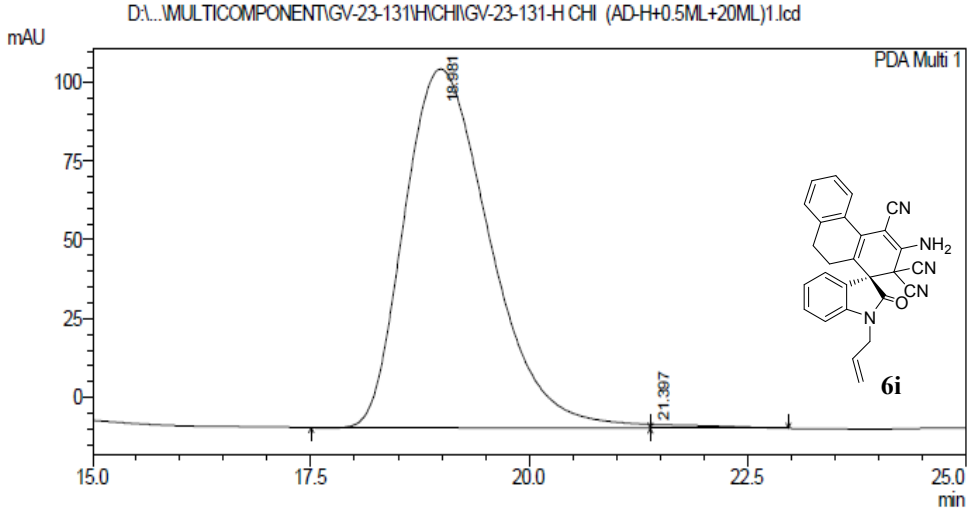
1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	18.313	57897135	688572	49.430	52.164
2	22.016	59232636	631446	50.570	47.836
Total		117129771	1320018	100.000	100.000

<Chromatogram>



1 PDA Multi 1/254nm 4nm

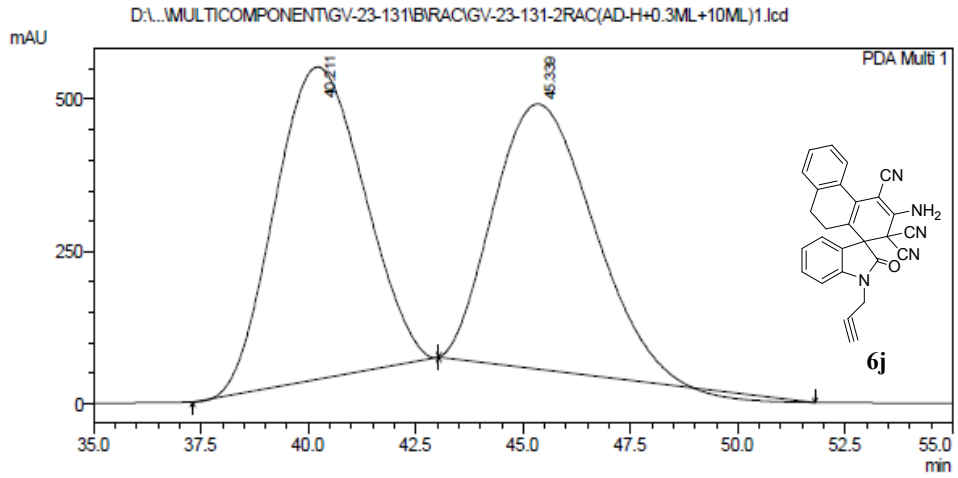
PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	18.981	7788876	114102	99.263	98.956
2	21.397	57853	1204	0.737	1.044
Total		7846729	115307	100.000	100.000

HPLC profile for table 4, entry 16

<Chromatogram>



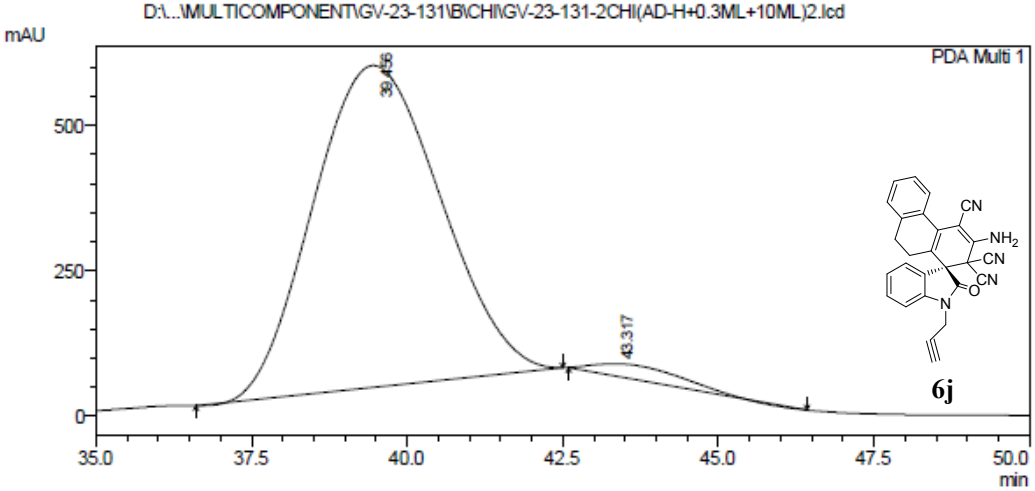
1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	40.211	73949904	512570	51.443	54.091
2	45.339	69802482	435036	48.557	45.909
Total		143752386	947606	100.000	100.000

<Chromatogram>



1 PDA Multi 1/254nm 4nm

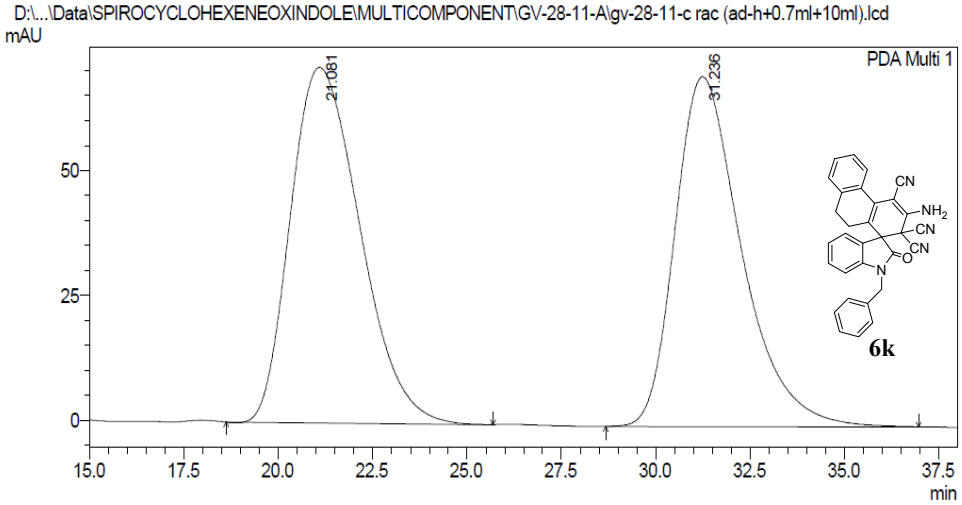
PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	39.456	78567105	554122	97.176	96.469
2	43.317	2283133	20283	2.824	3.531
Total		80850238	574405	100.000	100.000

HPLC profile for table 4, entry 17

<Chromatogram>



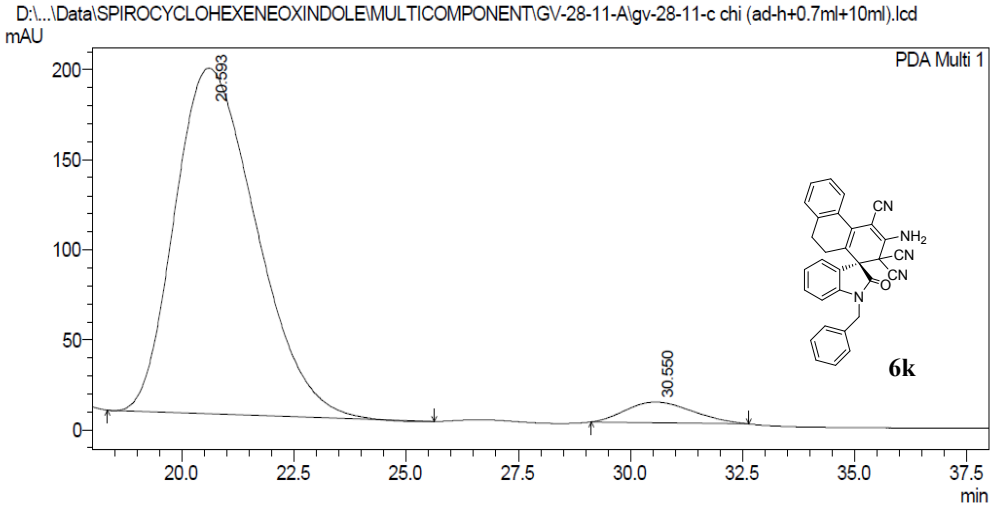
1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	21.081	9129654	71219	51.515	50.419
2	31.236	8592820	70037	48.485	49.581
Total		17722474	141256	100.000	100.000

<Chromatogram>



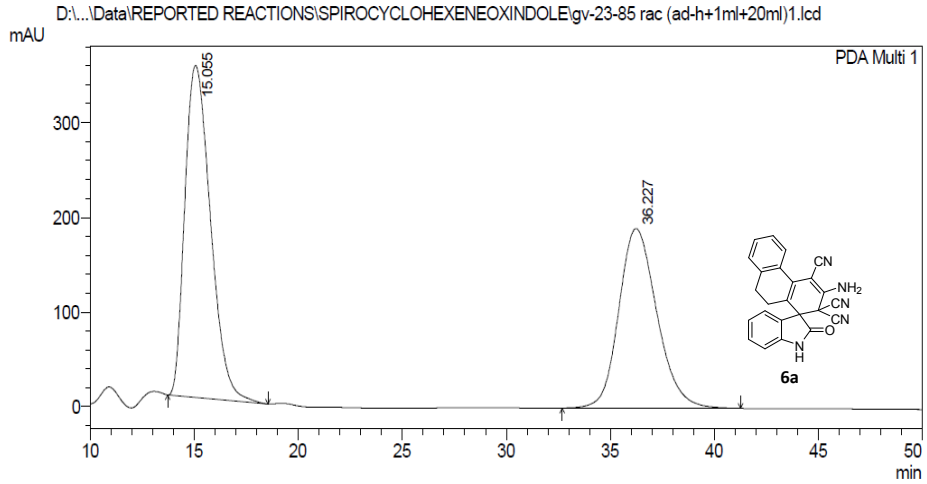
1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	20.593	24227282	191825	95.299	94.337
2	30.550	1195176	11515	4.701	5.663
Total		25422458	203340	100.000	100.000

HPLC profile for table 4, entry 18



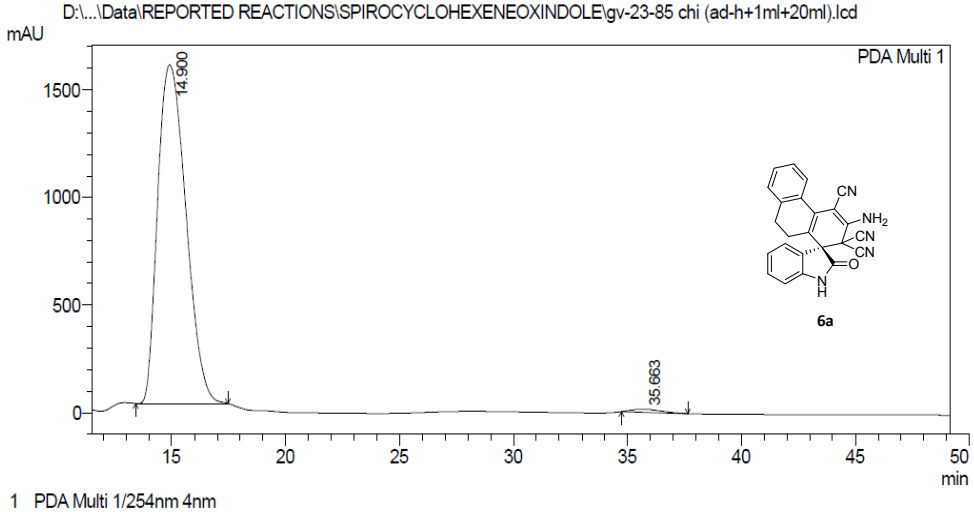
1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	15.055	30068090	350426	55.402	64.885
2	36.227	24204297	189648	44.598	35.115
Total		54272387	540074	100.000	100.000

<Chromatogram>



1 PDA Multi 1/254nm 4nm

PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	14.900	135721342	1574352	99.082	99.135
2	35.663	1257736	13736	0.918	0.865
Total		136979078	1588088	100.000	100.000