Supplementary data

Pd-catalysed intramolecular transformations of indolylbenzenesulfonamides: *ortho*-sulfonamido-bi(hetero)aryls *via* C2-arylation and polycyclic sultams *via* C3 arylation

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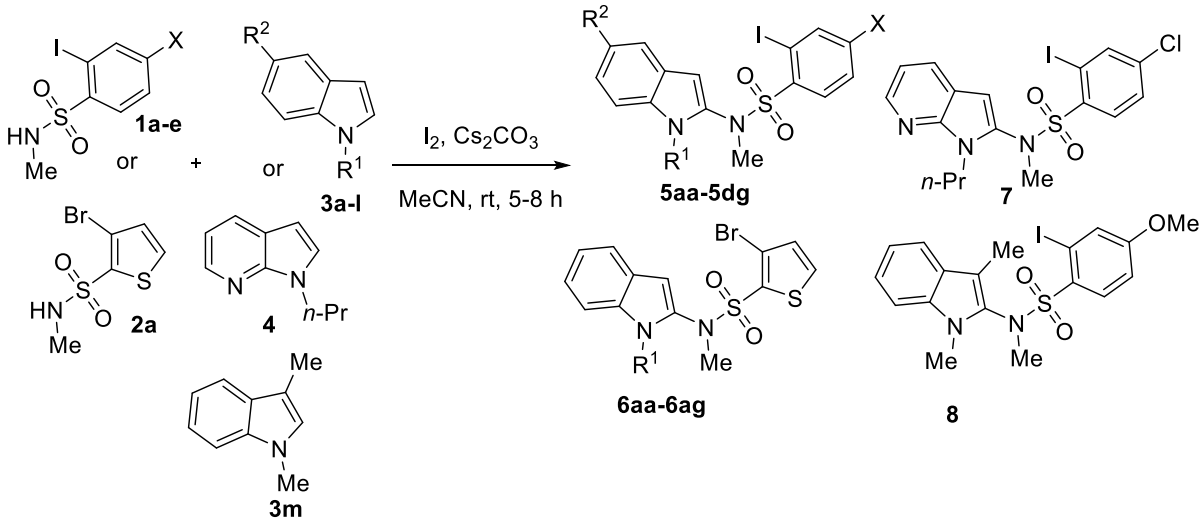
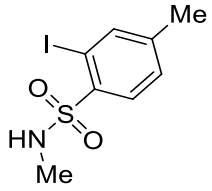
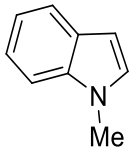
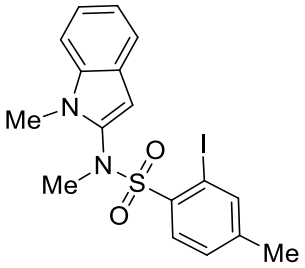
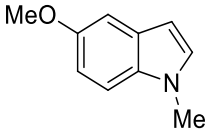
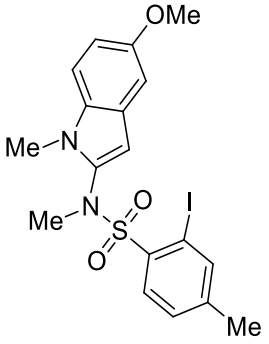
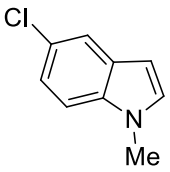
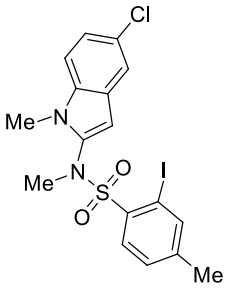
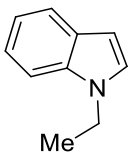
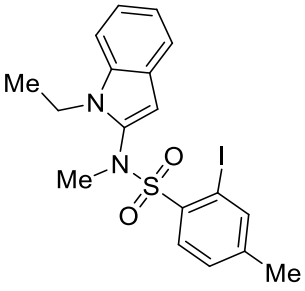
Table S1: *Iodine mediated synthesis* of *Indolylbenzene/thiophene sulfonamides* ***(5), (6), (7) and (8).***[a]

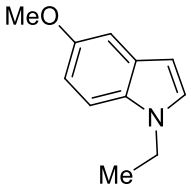
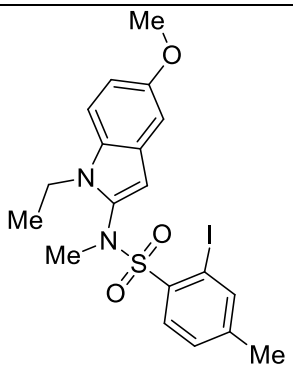
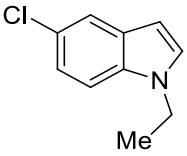
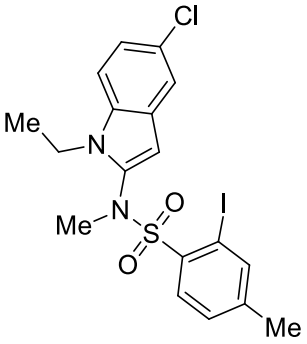
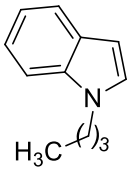
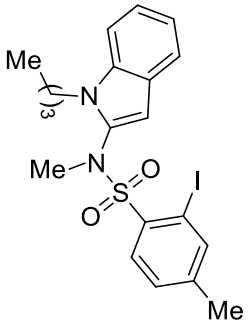
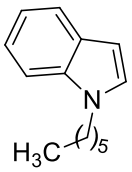
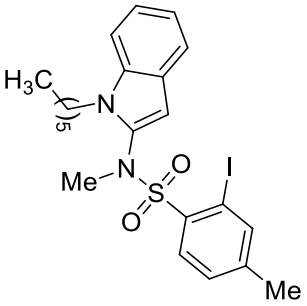
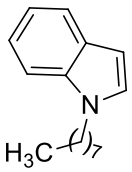
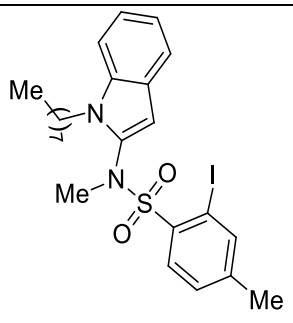


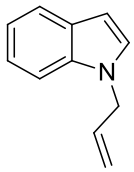
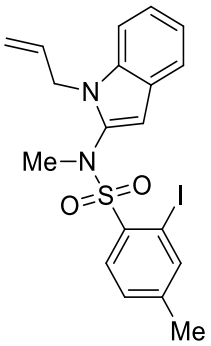
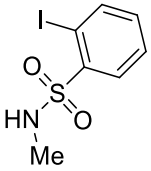
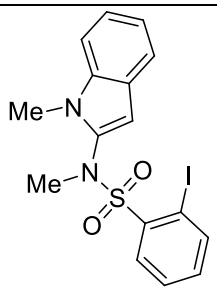
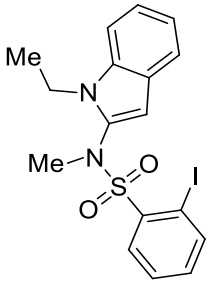
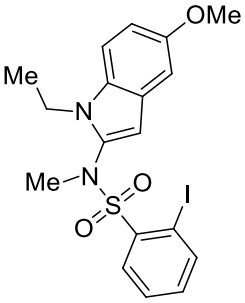
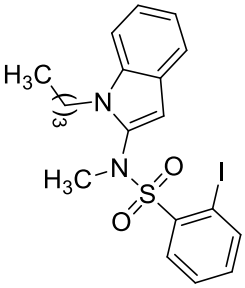
(i) *General Procedure for the preparation of* ***1, 2a, 3, and 4:*** 2-Iodo substituted sulfonamides **1** were prepared according to the procedure described in literature.[1] The compound 3-bromo-*N*-methylthiophene-2-sulfonamide **2a** was prepared according to a procedure described in the literature.[2] Other starting materials **3 & 4** were prepared *via* alkylation, benzylation and allylation of the corresponding indoles according to a known procedure.[3]

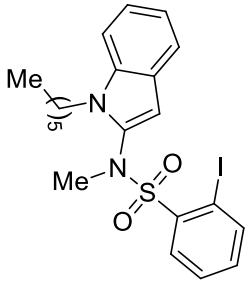
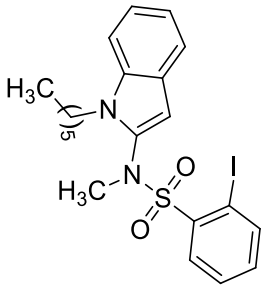
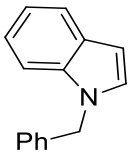
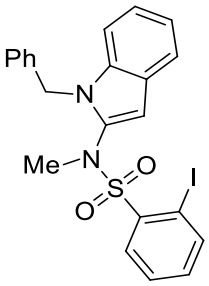
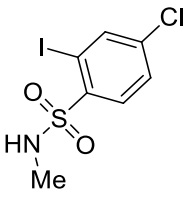
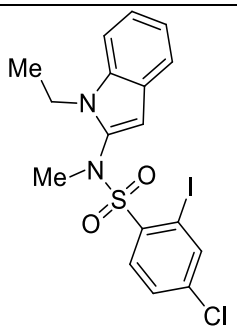
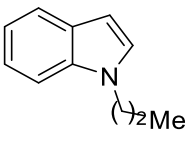
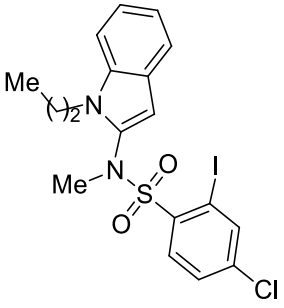
(ii) *General procedure for the preparation of iodo-substituted indolylbenzene/thiophene sulfonamides* ***5aa-5dg, 6aa-6ag, 7, and 8:*** An oven dried 25 mL round-bottomed flask was charged with sulfonamide **1** (0.32 mmol), Cs_2CO_3 (0.48 mmol) and I_2 (0.32 mmol) in acetonitrile (2.5 mL) added indole **3** (0.38 mmol). Then the mixture was stirred at rt (25 ^oC) under nitrogen for 5-8 h. After completion of the reaction (TLC), the reaction was quenched with a saturated solution of $Na_2S_2O_3$ (10 mL) and the mixture was treated with ethyl acetate (20 mL). The resulting solution was washed with water and the aqueous part extracted with ethyl acetate (2 x 20 mL). The combined organic layer was washed with saturated brine solution (2 × 20 mL), dried over anhydrous Na_2SO_4, and concentrated in vacuum. The

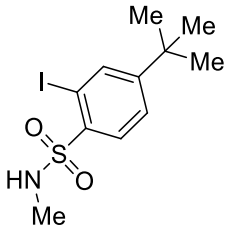
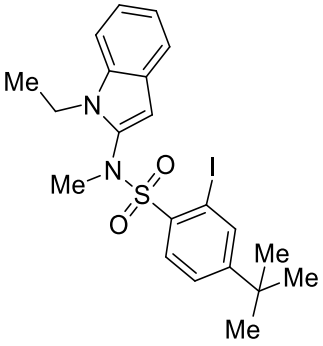
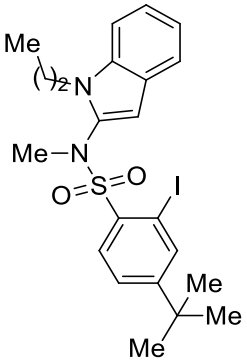
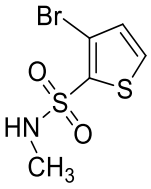
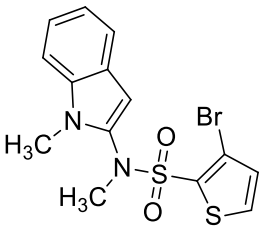
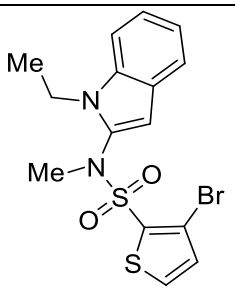
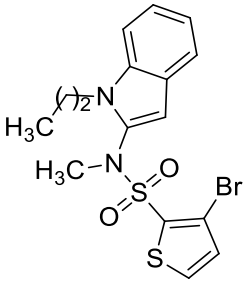
residue was then purified by using silica gel column chromatography using hexane-ethyl acetate (9:1) as eluent to afford the pure desired compounds **5, 6, 7 and 8**. Compounds **5aa-5dg, 6aa-6ag, 7** and **8** were prepared from the appropriate sulfonamide **1a-e, 2a** and indole **3, 4** by using the same procedure and the same molar quantities

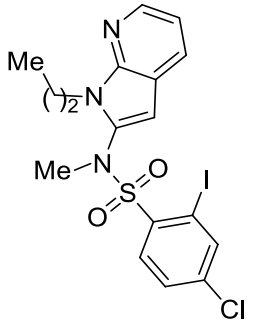
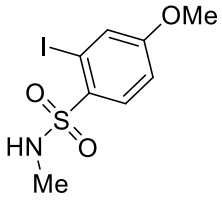
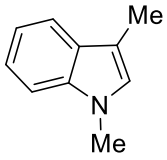
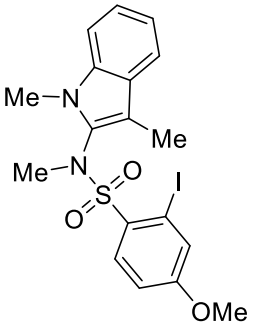
Entry	Sulfonamide	Indole	Time/ h	Product	Yield[b] (%)
1	 **1a**	 **3a**	5	 **5aa**	77
2	**1a**	 **3b**	6	 **5ab**	67
3	**1a**	 **3c**	5	 **5ac**	70
4	**1a**	 **3d**	7	 **5ad**	72

5	**1a**	 **3e**	6	 **5ae**	70
6	**1a**	 **3f**	5	 **5af**	67
7	**1a**	 **3h**	7	 **5ah**	82
8	**1a**	 **3i**	6	 **5ai**	81
9	**1a**	 **3j**	6	 **5aj**	77

10	**1a**	 **3l**	7	 **5al**	62
11	 **1b**	**3a**	7	 **5ba**	83
12	**1b**	**3d**	7	 **5bd**	71
13	**1b**	**3e**	6	 **5be**	77
14	**1b**	**3h**	6	 **5bh**	83

15	**1b**	**3i**	7	 **5bi**	79
16	**1b**	**3j**	6	 **5bj**	76
17	**1b**	 **3k**	5	 **5bk**	70
18	 **1c**	**3d**	6	 **5cd**	72
19	**1c**	 **3g**	7	 **5cg**	67

20	 **1d**	**3d**	7	 **5dd**	48
21	**1d**	**3g**	7	 **5dg**	60
22	 **2a**	**3a**		 **6aa**	82
23	**2a**	**3d**	8	 **6ad**	73
24	**2a**	**3g**	8	 **6ag**	80

25	**1c**	**4**	8	 **7**	68
26	 **1e**	 **3m**	8	 8	70

[a]All the reactions were carried out using **1** (0.32 mmol), **2** (0.38 mmol), I_2 (0.32 mmol) and Cs_2CO_3 (0.48 mmol) in acetonitrile (5.0 mL), at rt (25 ^oC) under nitrogen atmosphere. [b]Isolated yield.

1. X-ray data collection, solution, refinement and the ORTEPs/crystal data:

Single crystal X-ray data for crystals of compounds **5aa**, **9ba**, **9bh**, **10al**, **10bk** and **12ag** were collected on an X-ray diffractometer using Mo-K_α (λ = 0.71073 Å) radiation after mounting on glass fibers inside a brass pin in open air. The structures were solved by direct methods and refined by full-matrix least squares method using standard procedures; absorption corrections were done using SADABS program, where applicable [(a) Sheldrick, G. M. *SADABS, Siemens Area Detector Absorption Correction*, University of Gottingen, Germany, **1996**. (b) Sheldrick, G. M. SHELX-97-*A program for crystal structure solution and refinement*, University of Gottingen, **1997**. (c) Sheldrick, G. M. *SHELXTL NT Crystal Structure Analysis Package*, Bruker AXS, Analytical X-ray System, WI, USA, **1999**, version 5.10]. In general, all non-hydrogen atoms were refined anisotropically; hydrogen atoms were fixed by geometry or located by a Difference Fourier map and refined isotropically.

ORTEPs and crystal data of* 5aa, 9ba**, **9bh**, **10al**, **10bk** and **12ag** *(Figures S1-S6)

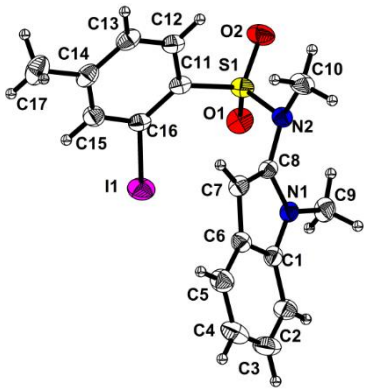


Figure S1. ORTEP of **5aa** with 30% probability of ellipsoids: ***Crystal data***: $C_{17}H_{17}IN_2O_2S$, *M* = 440.29, Monoclinic, Space group $P2_1/c$, *a* = 6.2737(4), *b* = 18.0774(6), *c* = 16.6031(8) Å, *V* = 1734.91(15) $Å^3$, β = 112.875(8)^o, *Z* = 4, μ = 1.976 mm^{-1}, data/restraints/parameters: 2513/0/212, R indices (I> 2σ\(I)): R1 = 0.0405, *w*R2 (all data) = 0.1204. CCDC No: 2202000

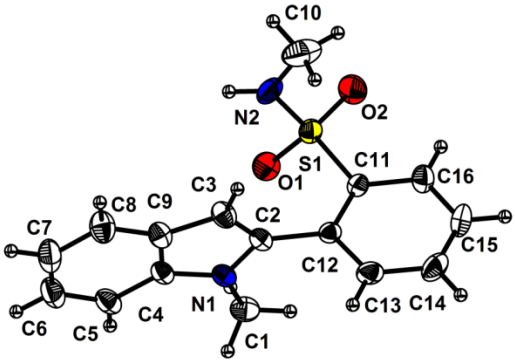


Figure S2. ORTEP of **9ba** with 30% probability of ellipsoids: ***Crystal data***: $C_{16}H_{16}N_2O_2S$, *M* = 300.37, Monoclinic, Space group *P21/n*, *a* = 11.6333(5), *b* = 11.3408(5), *c* = 11.7715(5) Å, *V* = 1523.13(11) $Å^3$, β = 101.2610(19)^o, *Z* = 4, μ = 0.218 mm^{-1}, data/restraints/parameters: 2683/0/193, R indices (I> 2σ\(I)): R1 = 0.0464, *w*R2 (all data) = 0.1128. CCDC No: 2202001

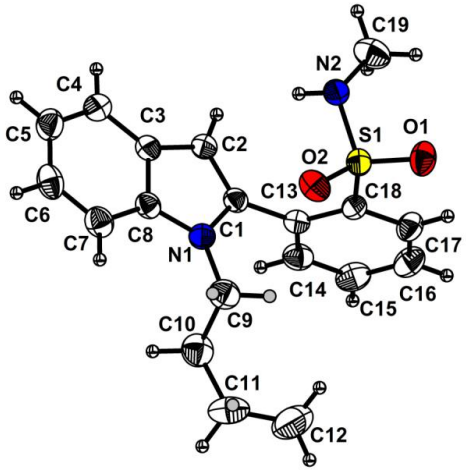


Figure S3. ORTEP of **9bh** with 30% probability of ellipsoids: ***Crystal data***: $C_{19}H_{22}N_2O_2S$, *M* = 342.44, Triclinic, Space group *P-1*, *a* = 8.4303(4), *b* = 10.1352(5), *c* = 11.2583(4) Å, *V* = 899.57(7) $Å^3$, α = 80.979(4)°, β = 71.424(4)°, γ = 89.642(4)°, *Z* = 2, μ = 0.193 mm^{-1}, data/restraints/parameters: 3131/0/224, R indices (I> 2σ\(I)): R1 = 0.0532, *w*R2 (all data) = 0.1620. CCDC No: 2202002

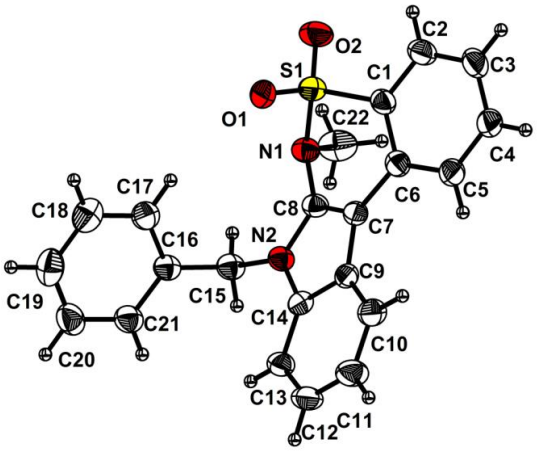


Figure S4. ORTEP of **10al** with 30% probability of ellipsoids: ***Crystal data***: $C_{22}H_{18}N_2O_2S$, *M* = 374.44, Monoclinic, Space group *P121/n1*, *a* = 9.2441(3), *b* = 9.3416(3), *c* = 21.8748(6) Å, *V* = 1884.83(10) $Å^3$, β = 93.805(3)°, *Z* = 4, μ = 0.191 mm^{-1}, data/restraints/parameters: 3312/0/246, R indices (I> 2σ\(I)): R1 = 0.0535, *w*R2 (all data) = 0.1430. CCDC No: 2202003

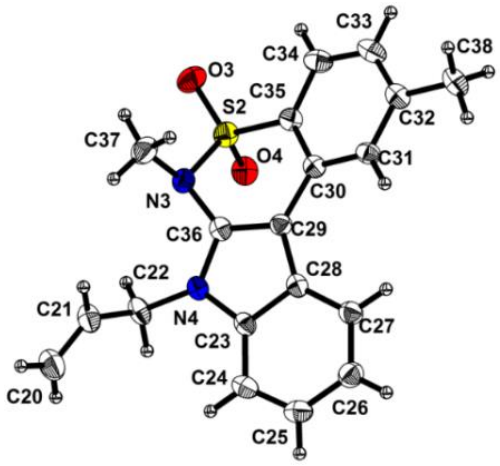


Figure S5. ORTEP of **10bk** with 30% probability of ellipsoids: ***Crystal data***: $C_{19}H_{18}N_2O_2S$, *M* = 338.41, Orthorhombic, Space group *Pna2(1)*, *a* = 37.1188(12), *b* = 7.4217(2), *c* = 11.9822(5) Å, *V* = 3300.9(2) $Å^3$, *Z* = 8, μ = 0.210 mm^{-1}, data/restraints/parameters: 5529/1/438, R indices (I> 2σ\(I)): R1 = 0.0578, *w*R2 (all data) = 0.1464. CCDC No: 2202004

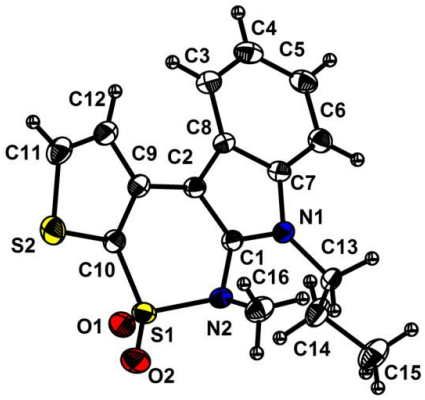


Figure S6. ORTEP view of **12ag** with 30% probability of ellipsoids. ***Crystal data***: $C_{16}H_{16}N_2O_2S_2$, *M* = 332.43, Monoclinic, Space group *P121/n1*, *a* = 10.3674(4), *b* = 7.8969(3), *c* = 19.6001(8) Å, *V* = 1568.26(11) $Å^3$, α = 90°, β = 102.228(4)°, γ = 90°, *Z* = 4, μ = 0.347 mm^{-1}, data/restraints/parameters: 2772/0/202, R indices (I> 2σ\(I)): R1 = 0.0492, *w*R2 (all data) = 0.1387. CCDC No: 2202005.

2. ^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of all new compounds

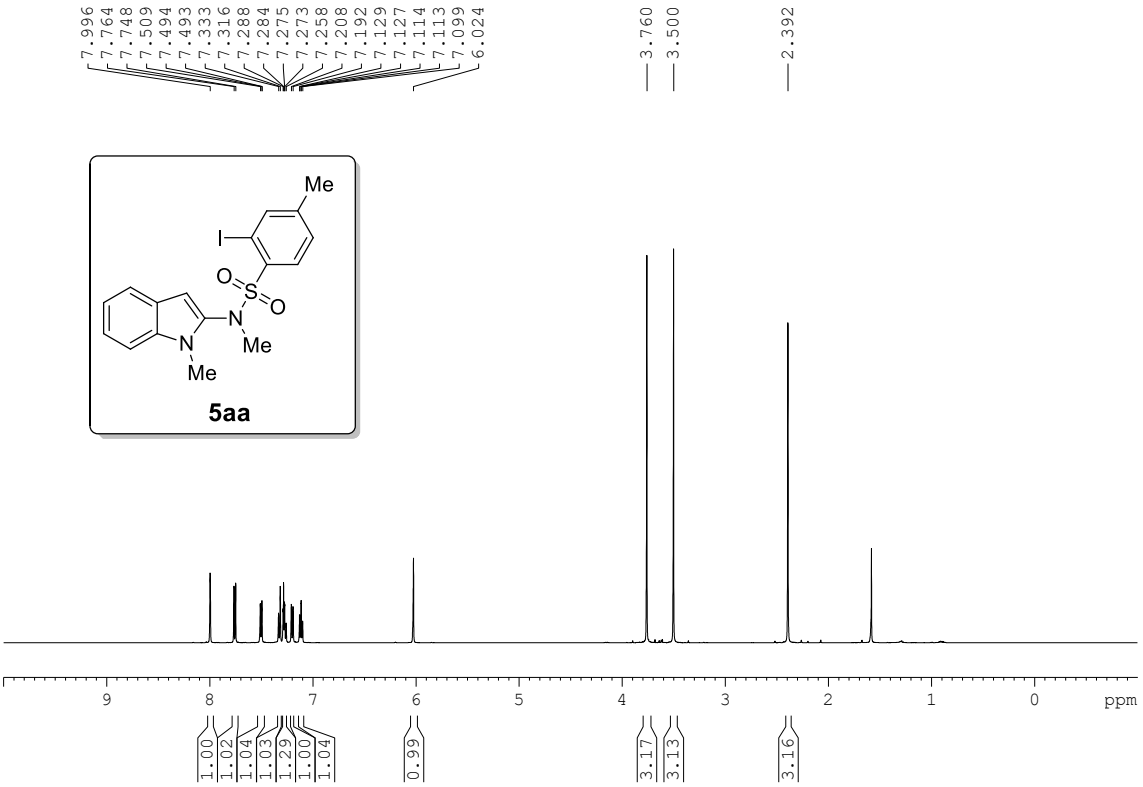


Figure S7. ^{1}H NMR spectrum of compound **5aa**

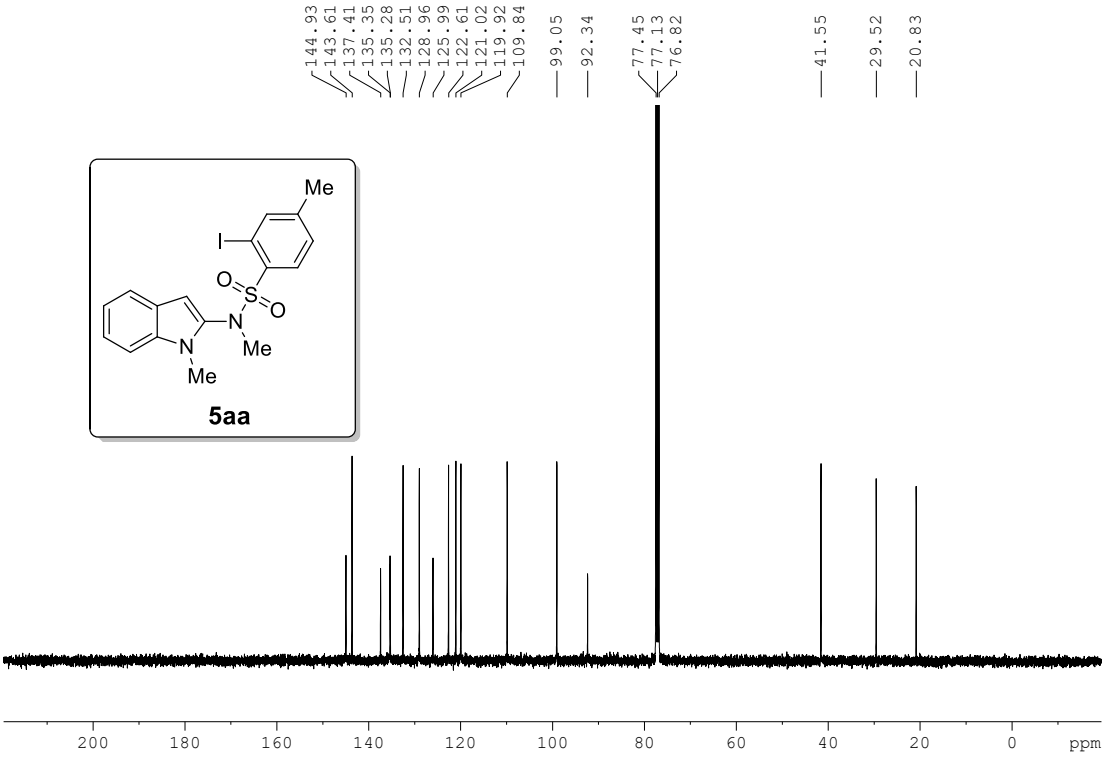


Figure S8. $^{13}C\{^{1}H\}$ NMR spectrum of compound **5aa**

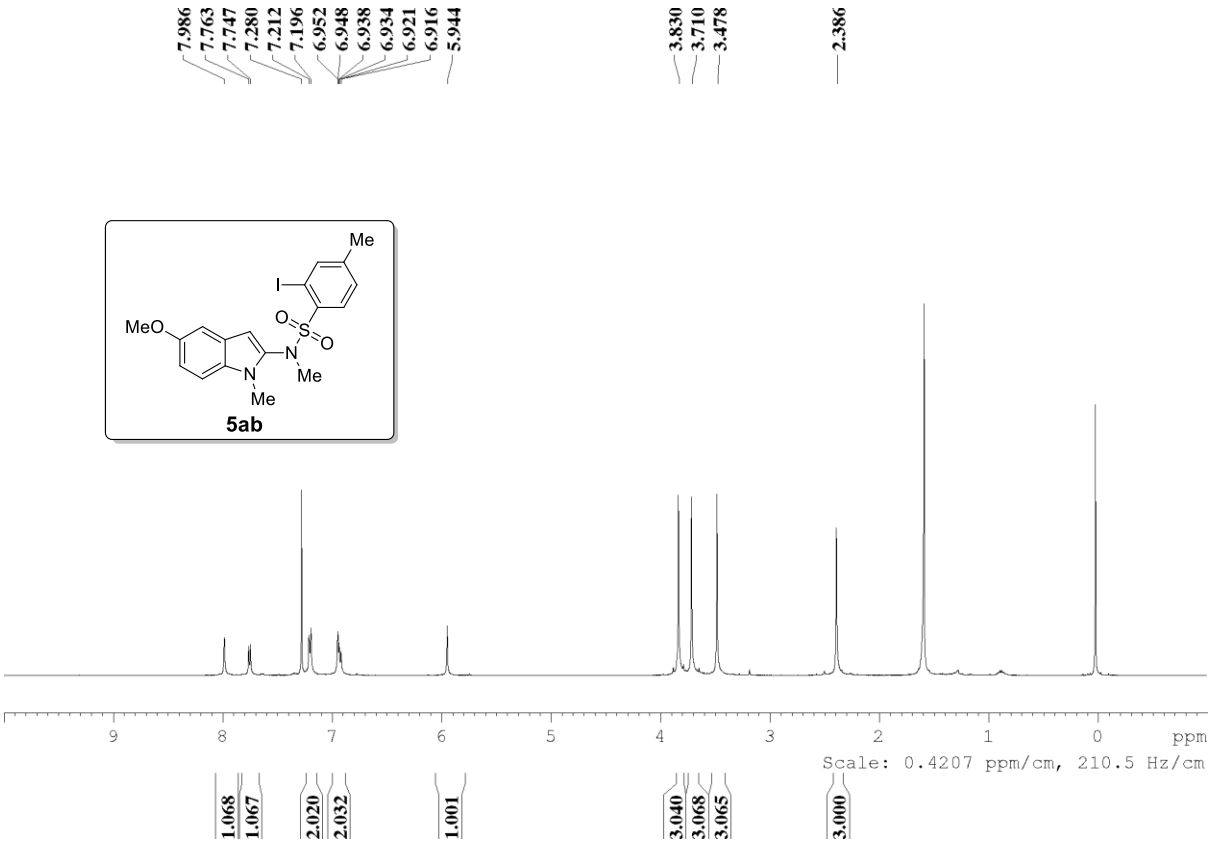


Figure S9. ^{1}H NMR spectrum of compound **5ab**

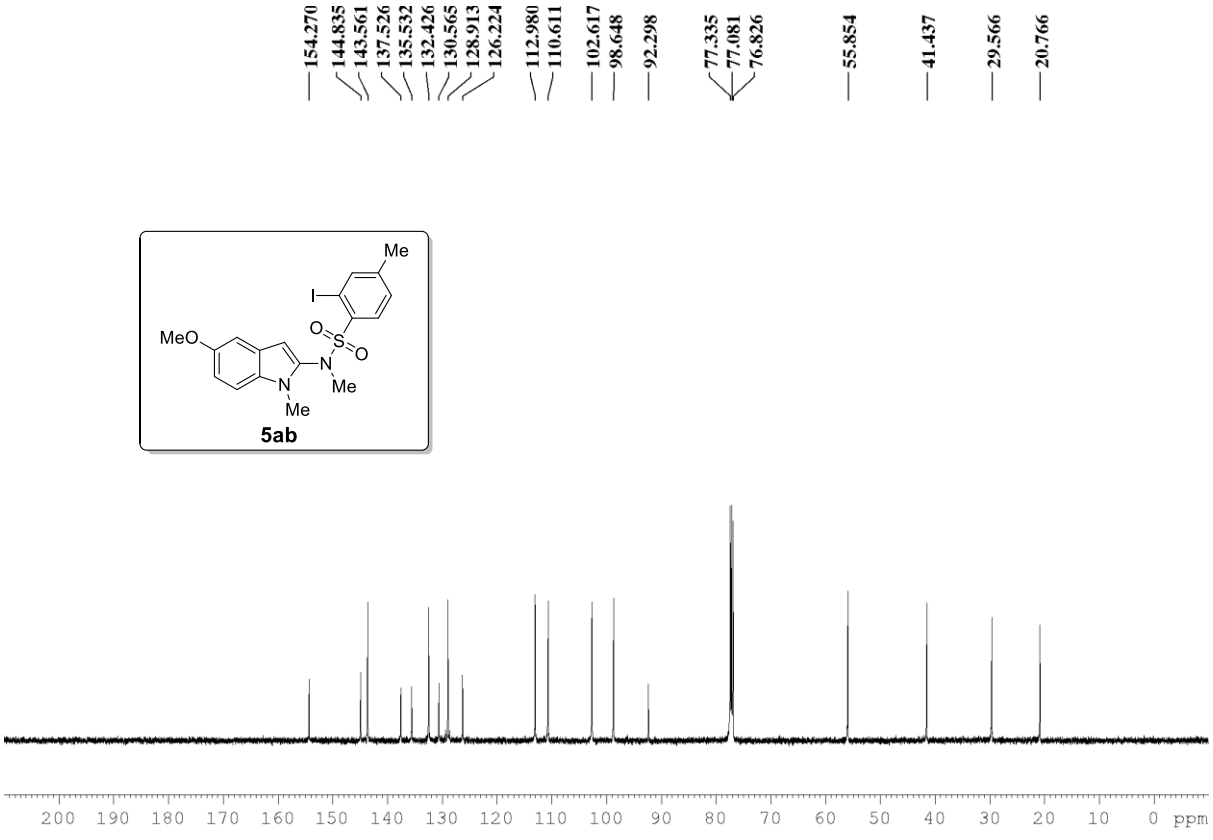


Figure S10. ^{13}C{^{1}H} NMR spectrum of compound **5ab**

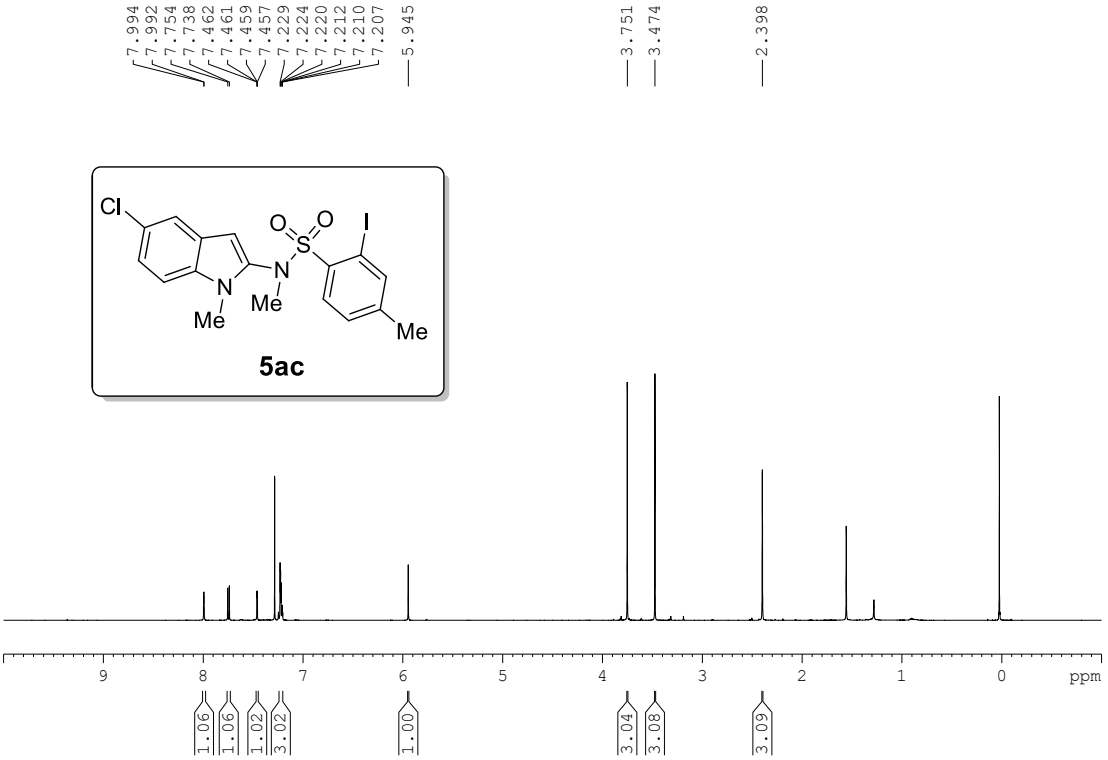


Figure S11. ^{1}H NMR spectrum of compound **5ac**

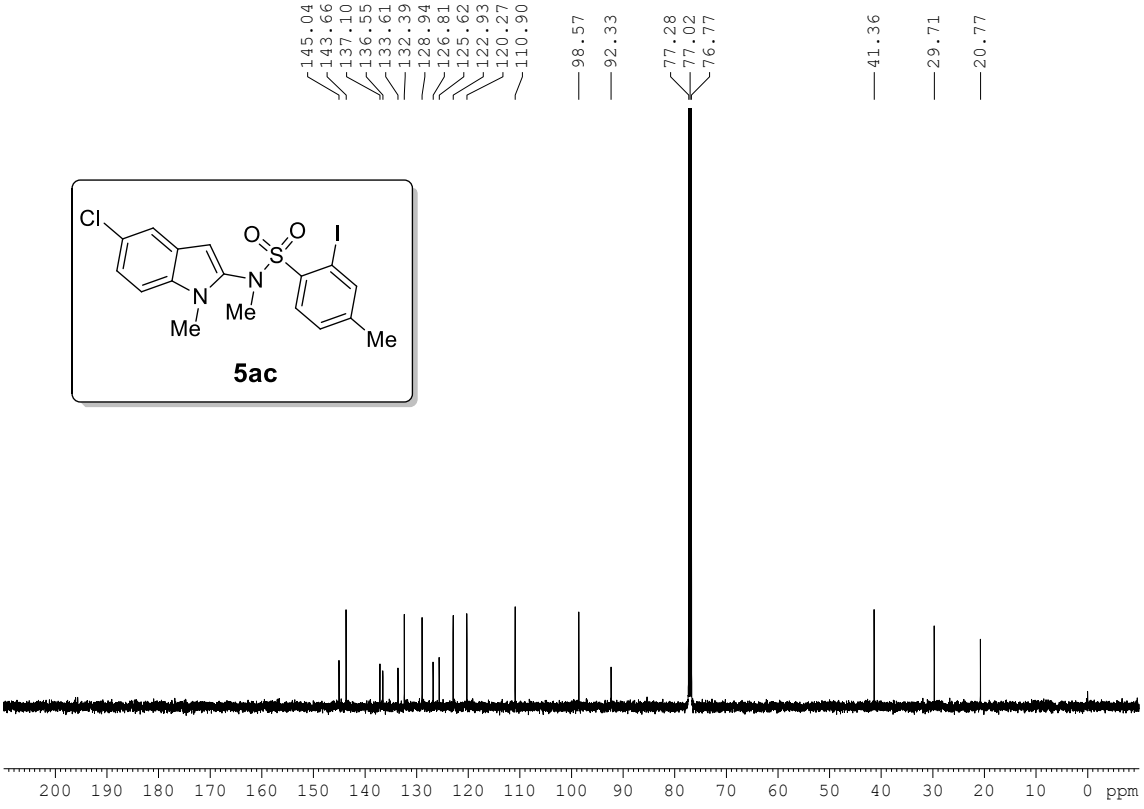


Figure S12. ^{13}C{^{1}H} NMR spectrum of compound **5ac**

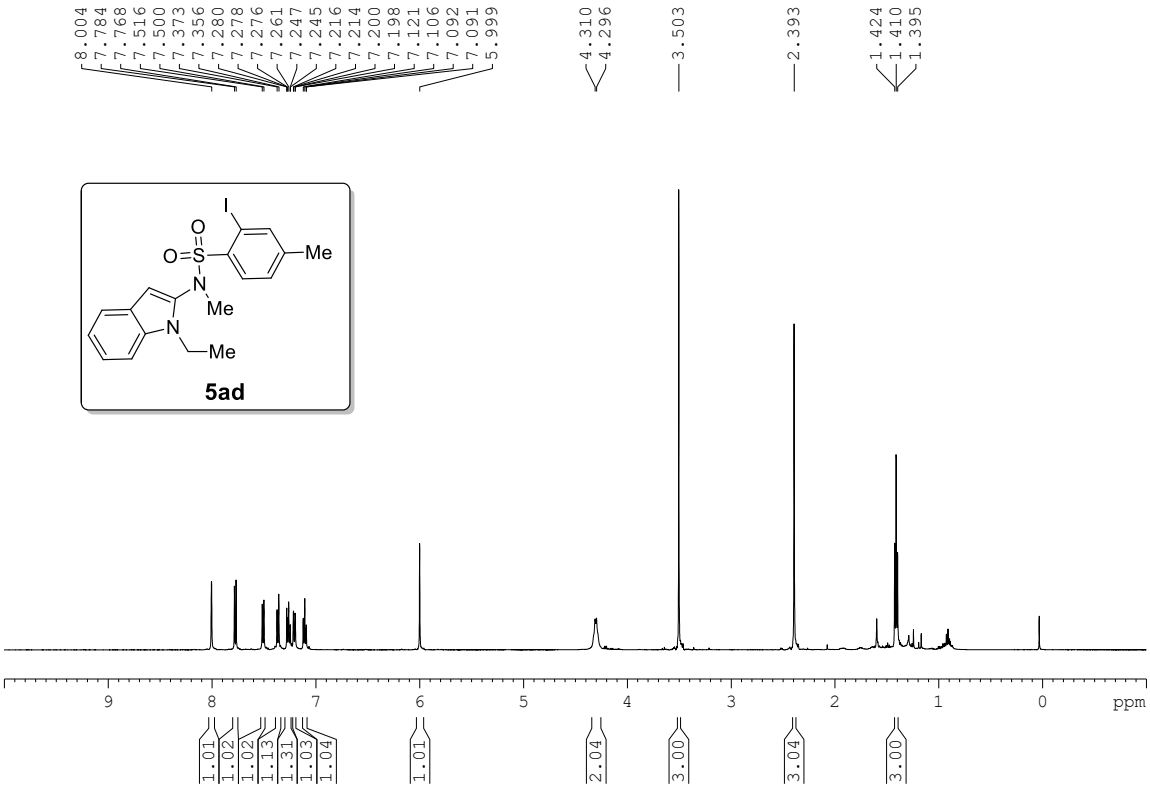


Figure S13. ^{1}H NMR spectrum of compound **5ad**

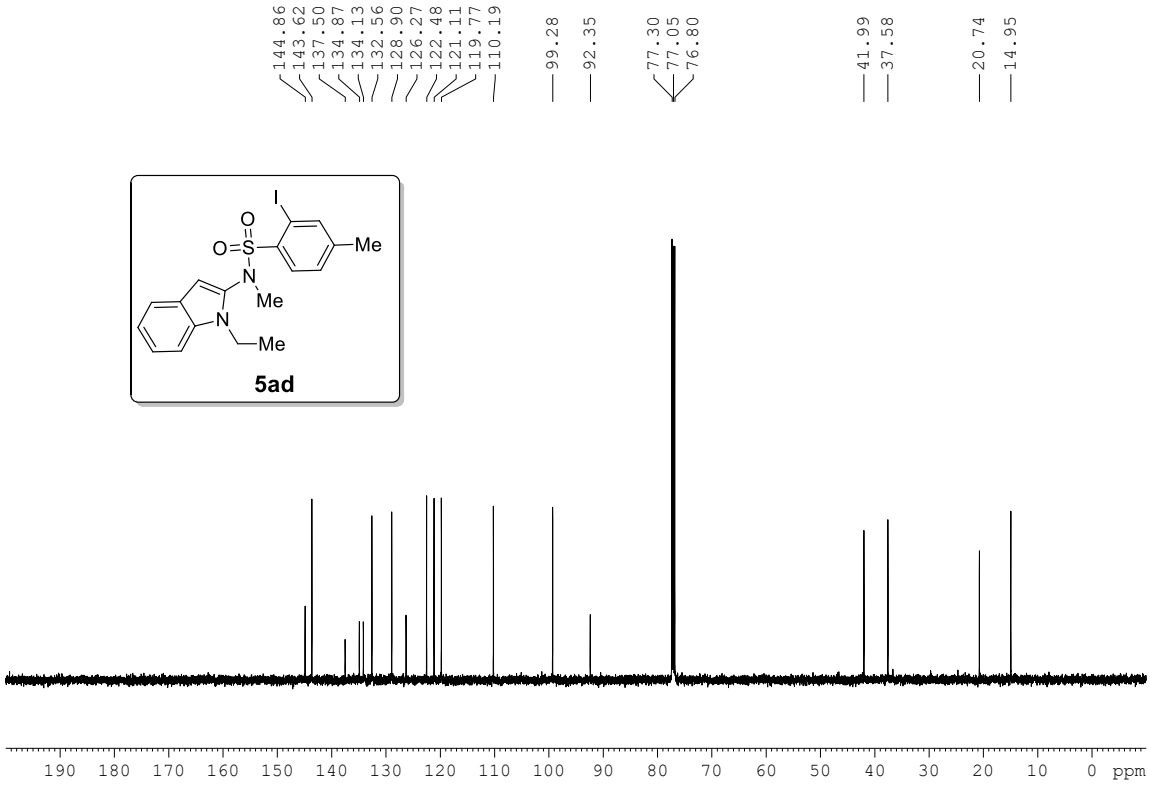


Figure S14. ^{13}C{^{1}H} NMR spectrum of compound **5ad**

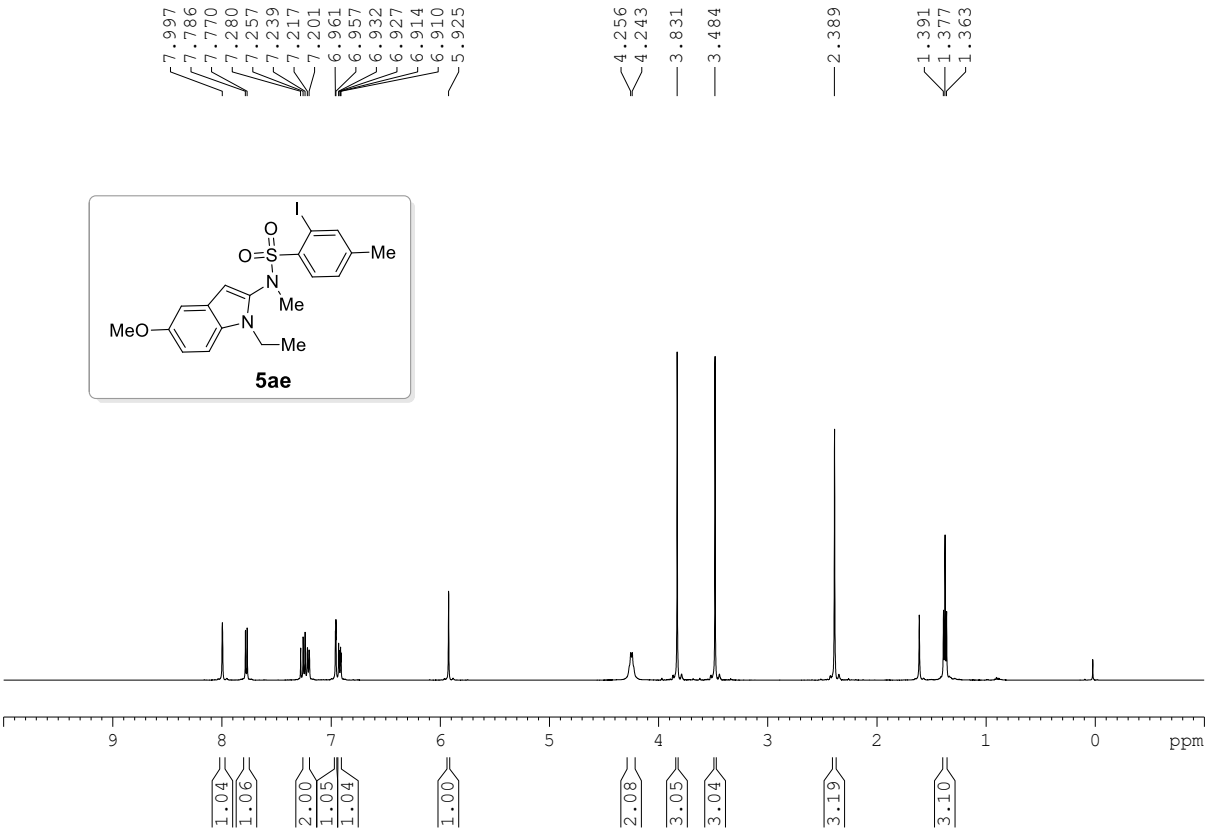


Figure S15. ^{1}H NMR spectrum of compound **5ae**

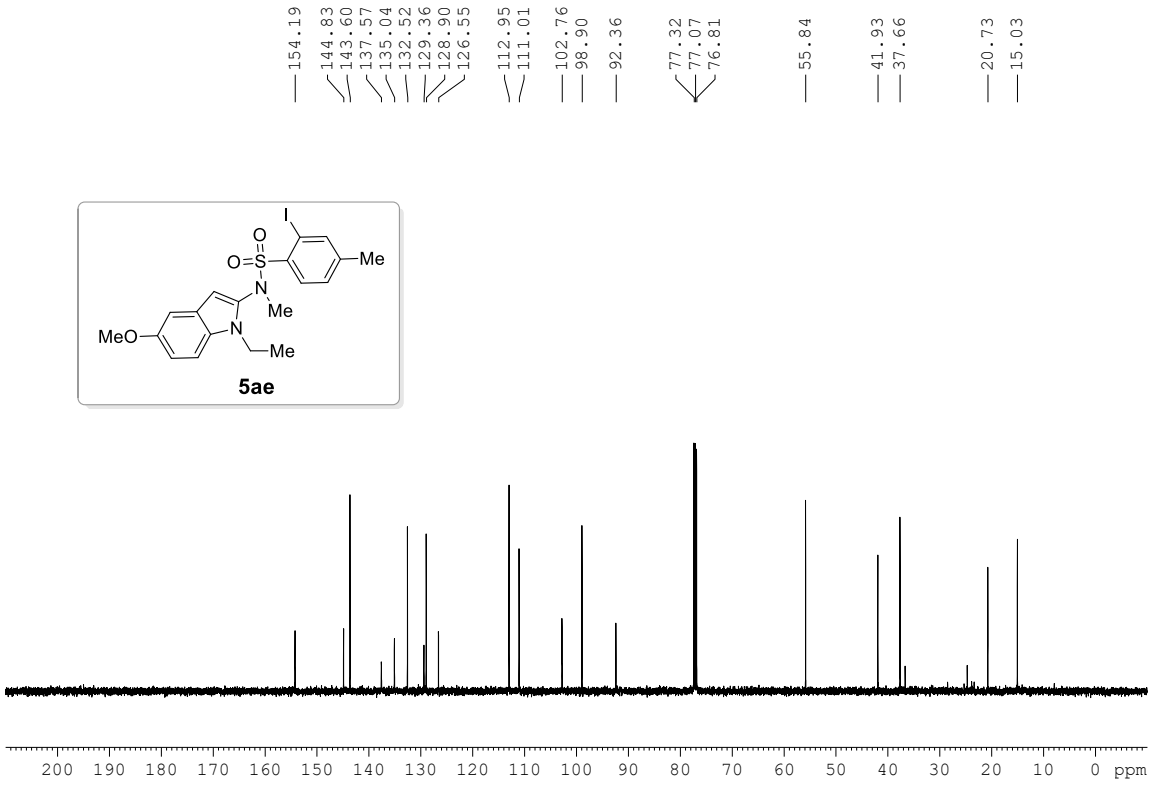


Figure S16. ^{13}C{^{1}H} NMR spectrum of compound **5ae**

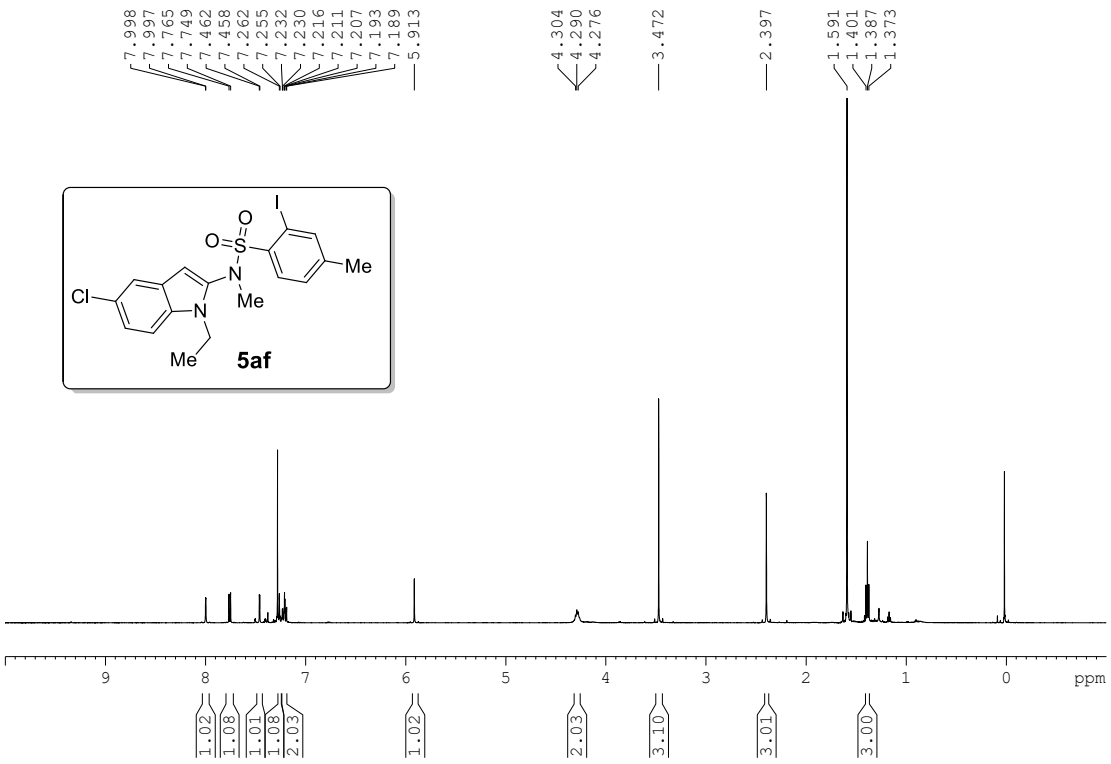


Figure S17. ^{1}H NMR spectrum of compound **5af**

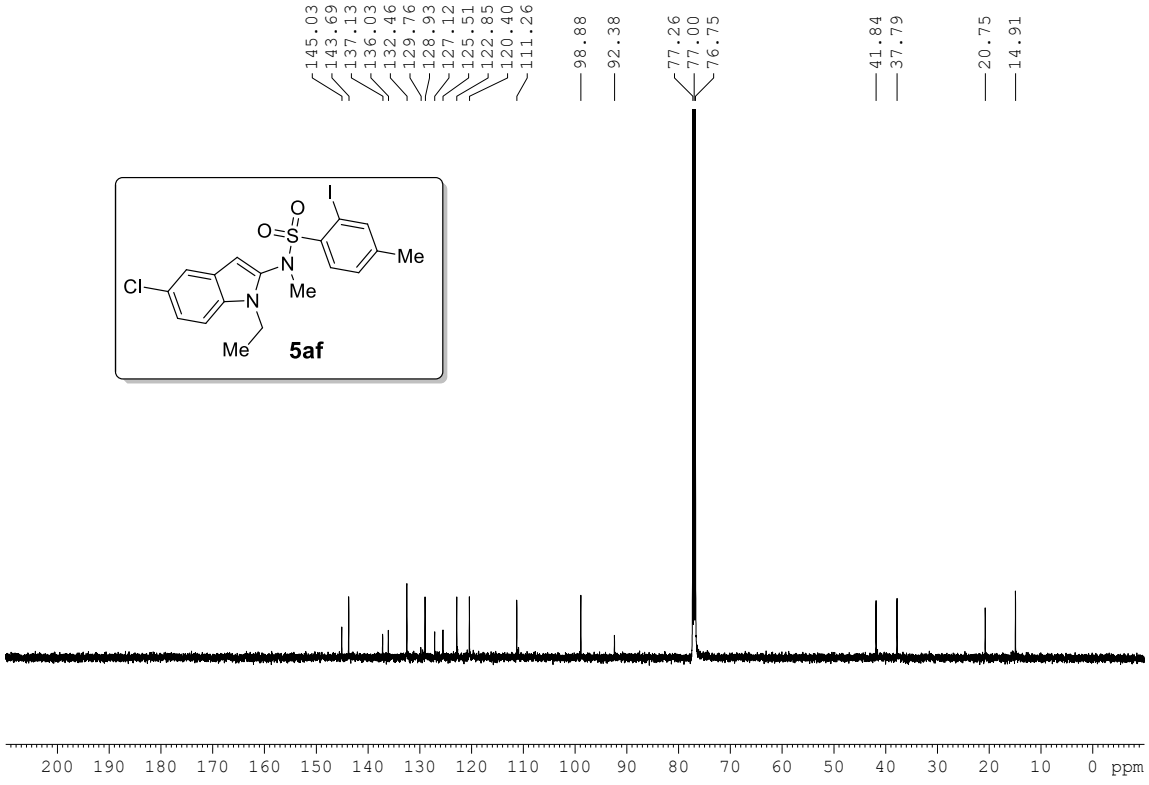


Figure S18. ^{13}C{^{1}H} NMR spectrum of compound **5af**

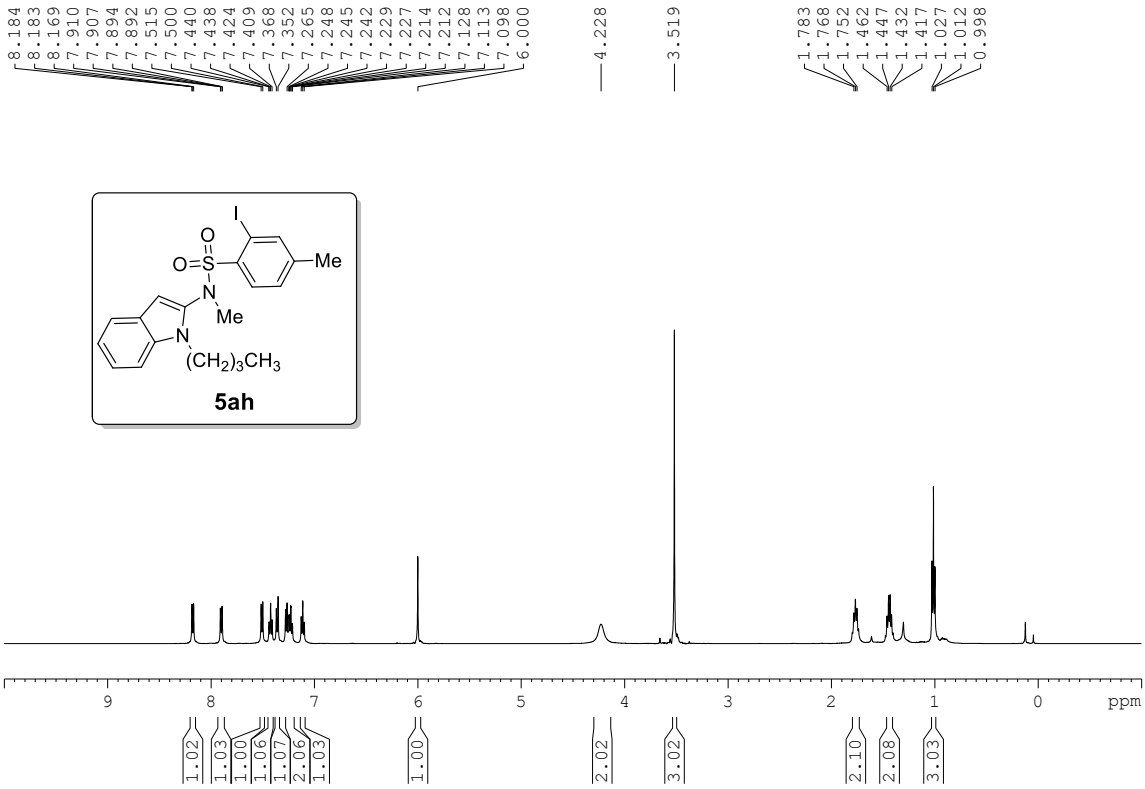


Figure S19. ^{1}H NMR spectrum of compound **5ah**

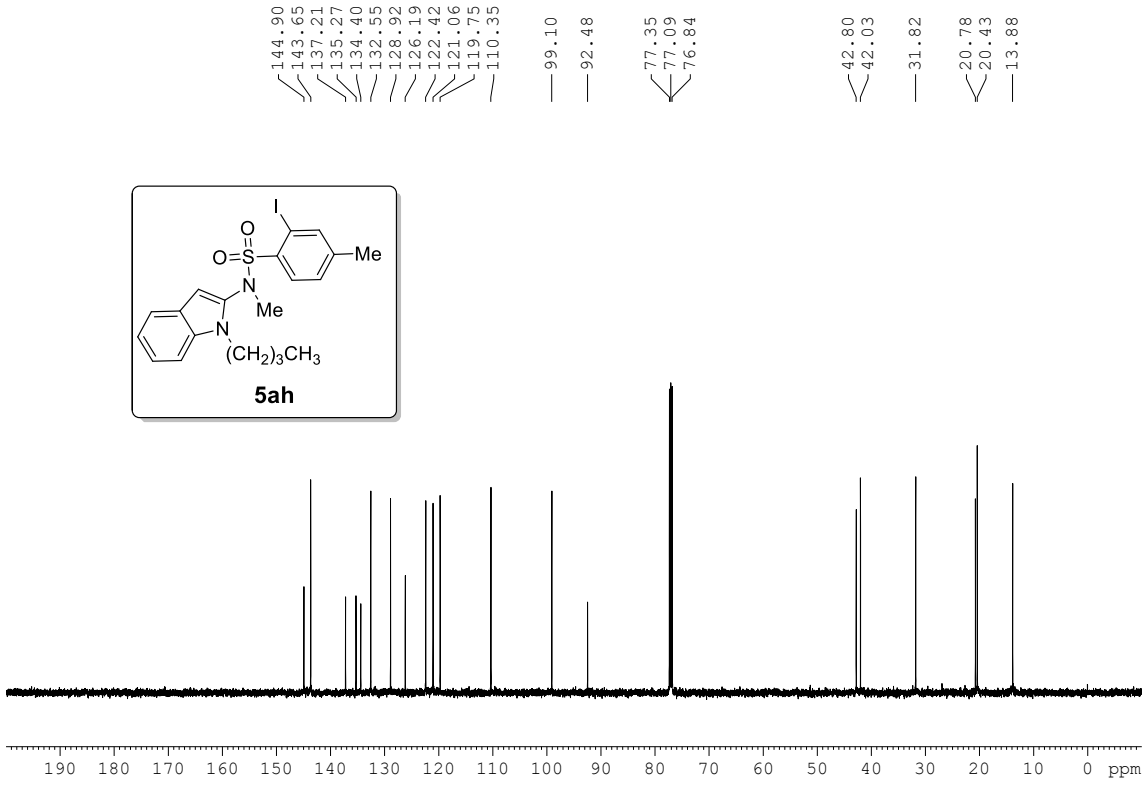


Figure S20. ^{13}C{^{1}H} NMR spectrum of compound **5ah**

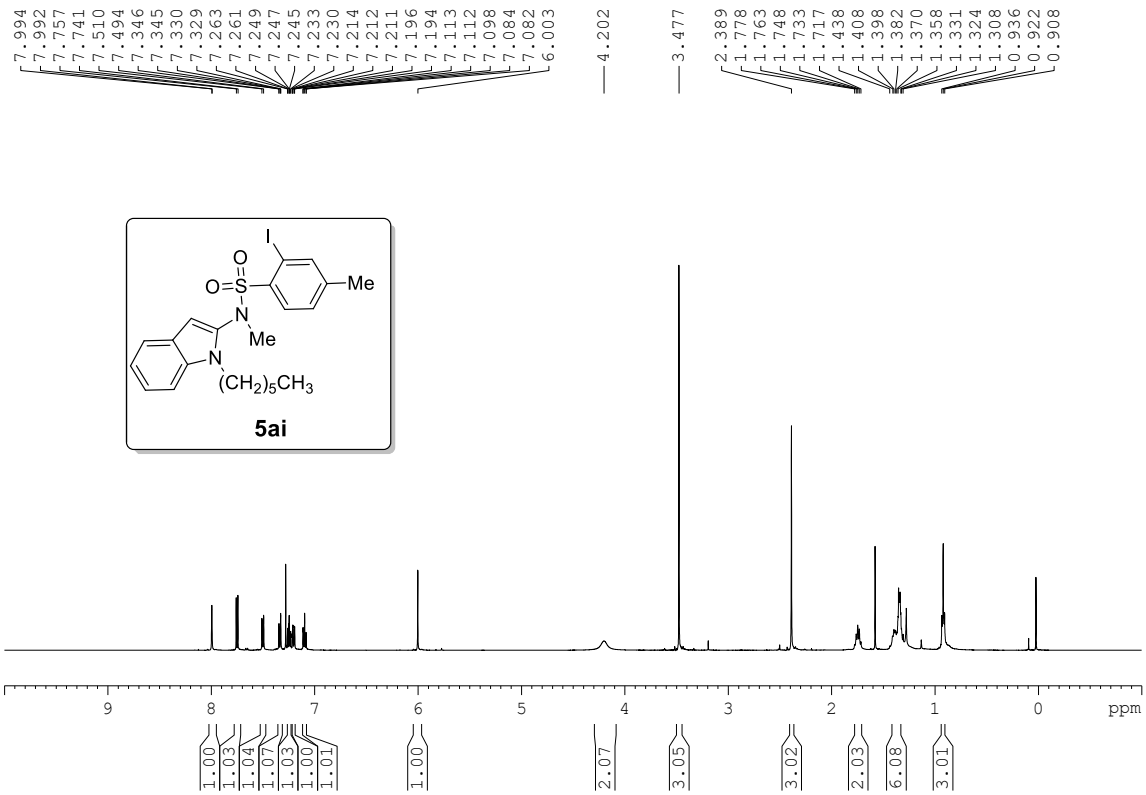


Figure S21. ^{1}H NMR spectrum of compound **5ai**

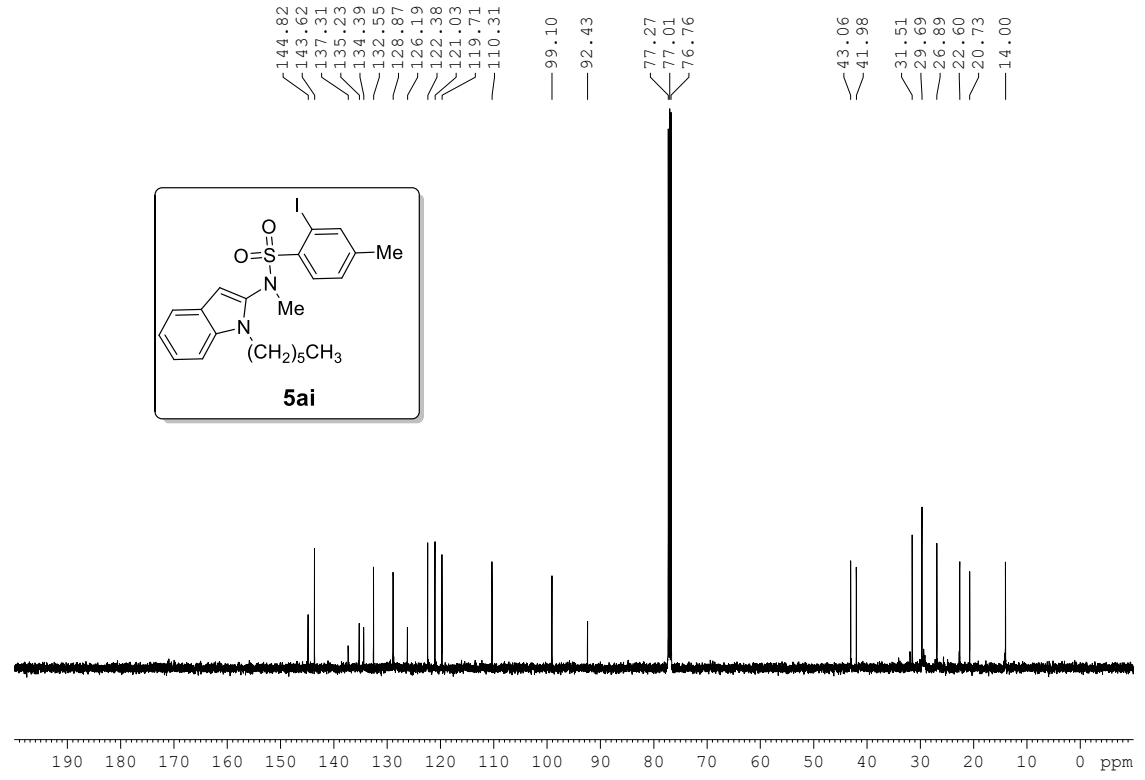


Figure S22. ^{13}C{^{1}H} NMR spectrum of compound **5ai**

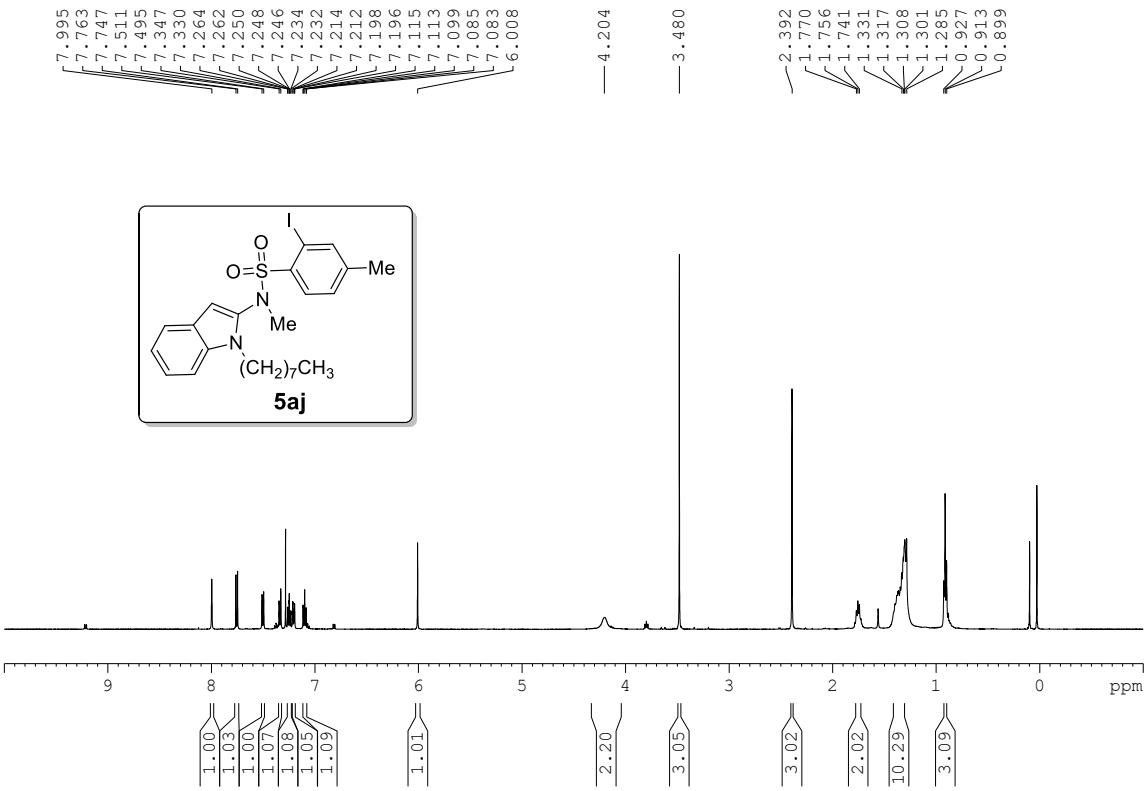


Figure S23. ^{1}H NMR spectrum of compound **5aj**

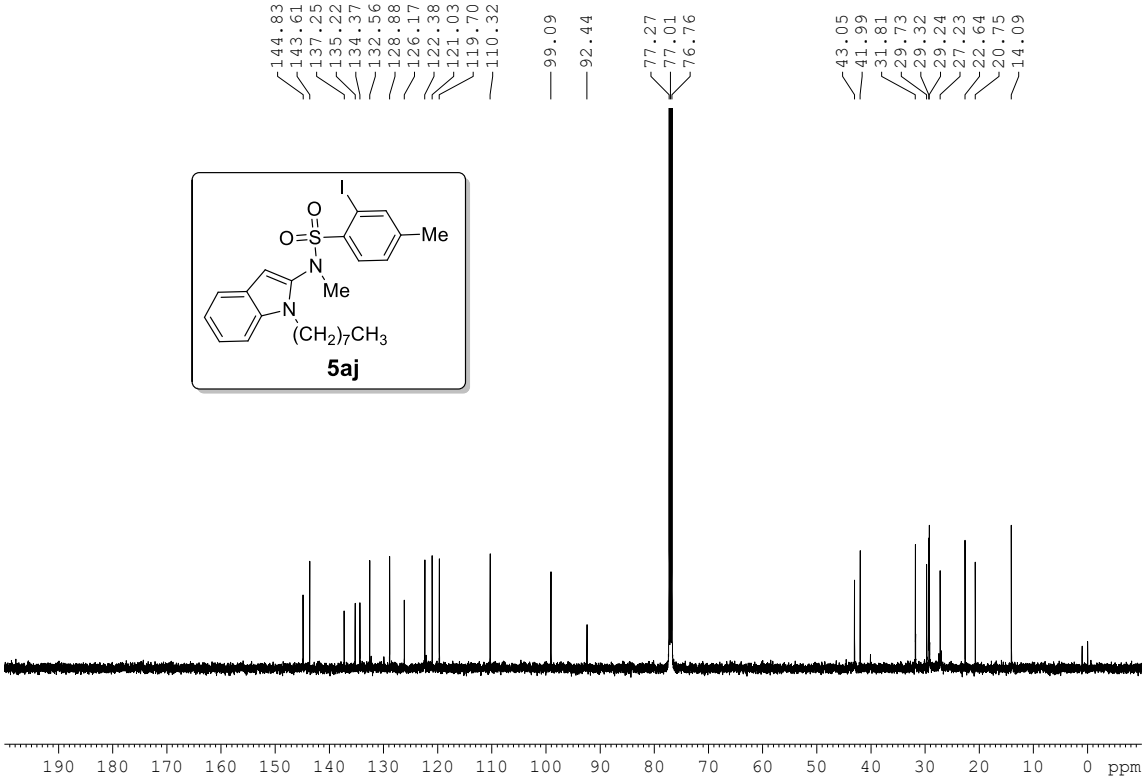


Figure S24. ^{13}C{^{1}H} NMR spectrum of compound **5aj**

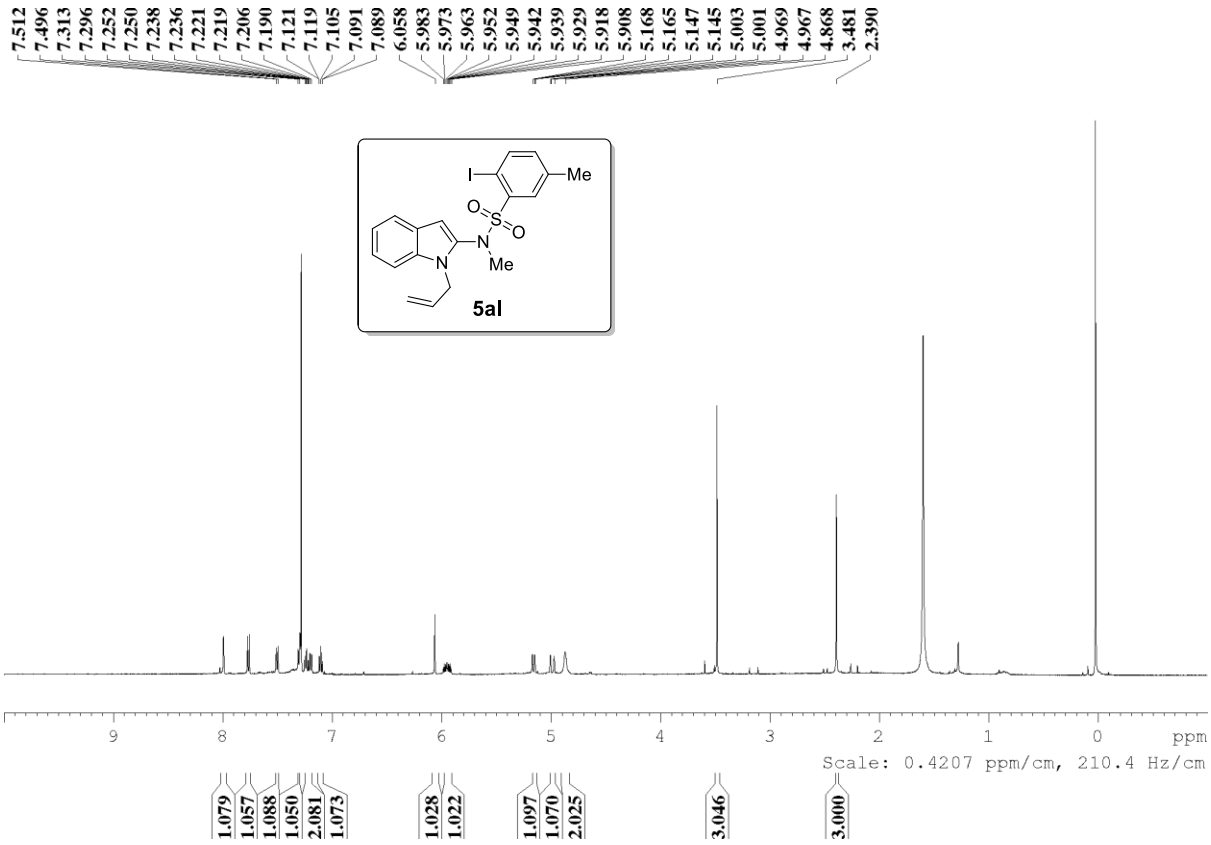


Figure S25. ^{1}H NMR spectrum of compound **5al**

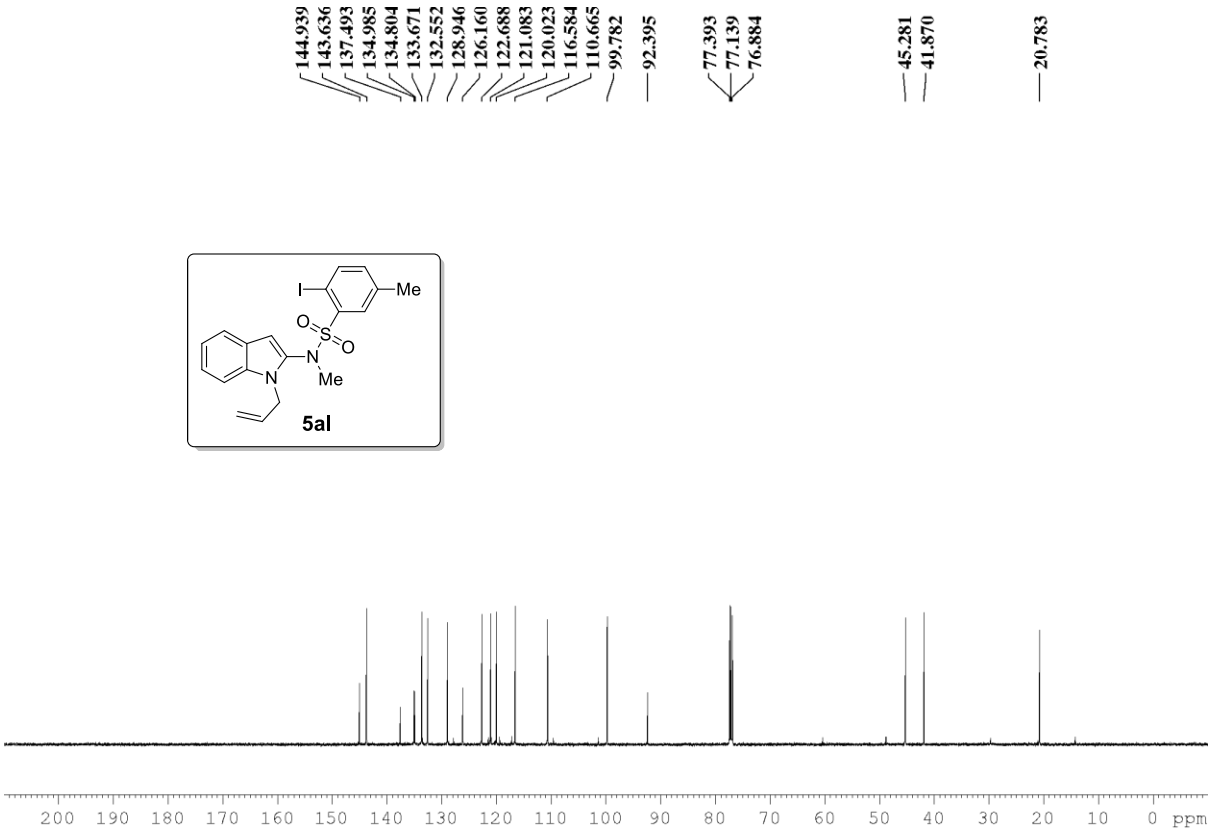


Figure S26. ^{13}C{^{1}H} NMR spectrum of compound **5al**

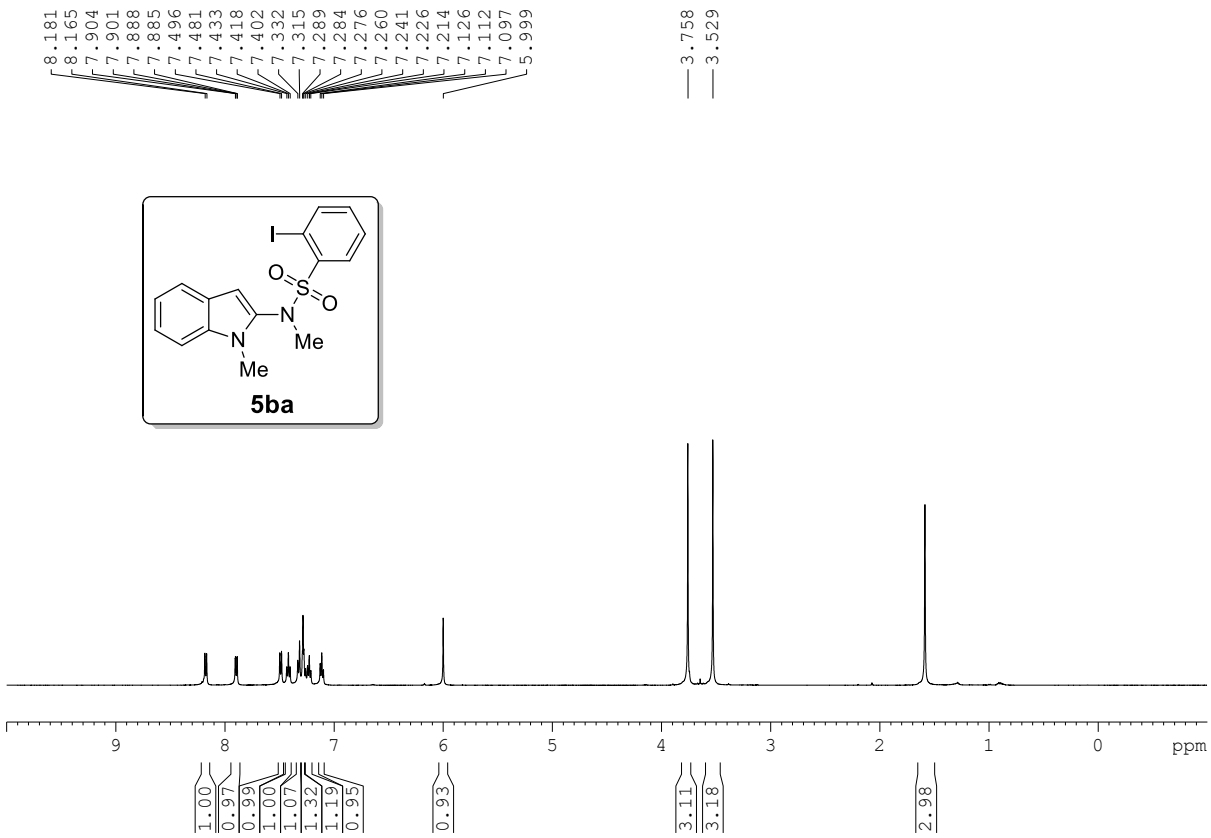


Figure S27. ^{1}H NMR spectrum of compound **5ba**

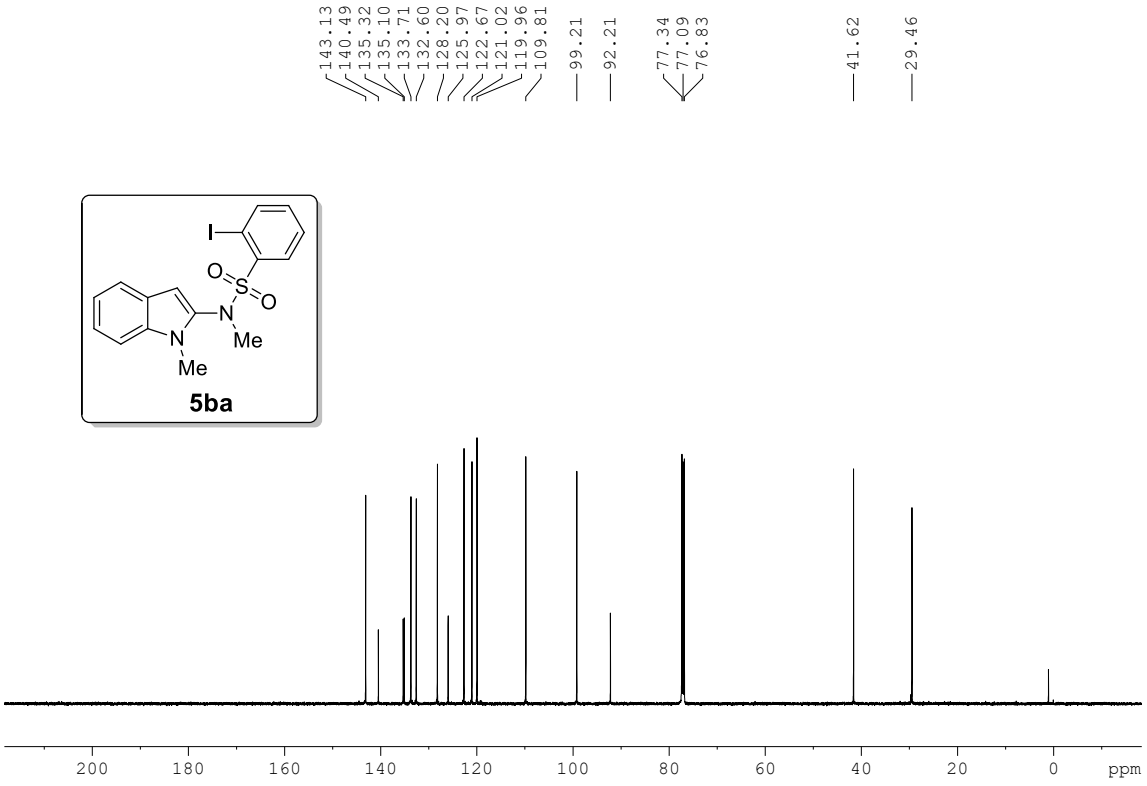


Figure S28. ^{13}C{^{1}H} NMR spectrum of compound **5ba**

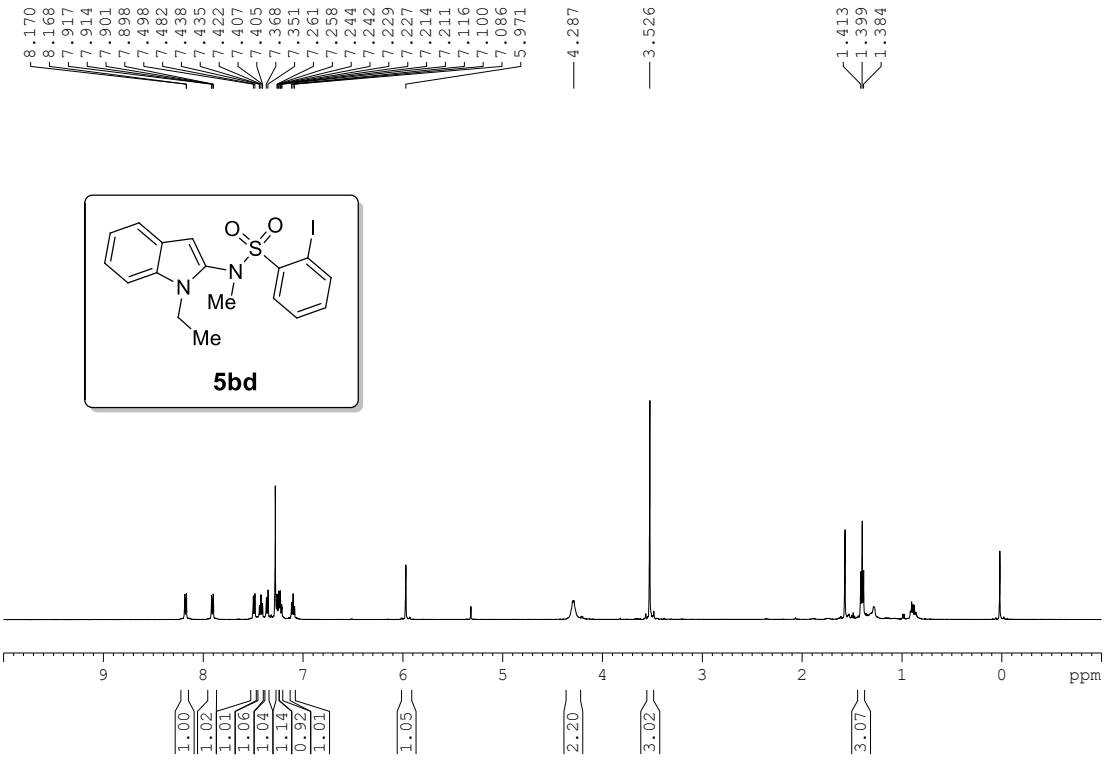


Figure S29. ^{1}H NMR spectrum of compound **5bd**

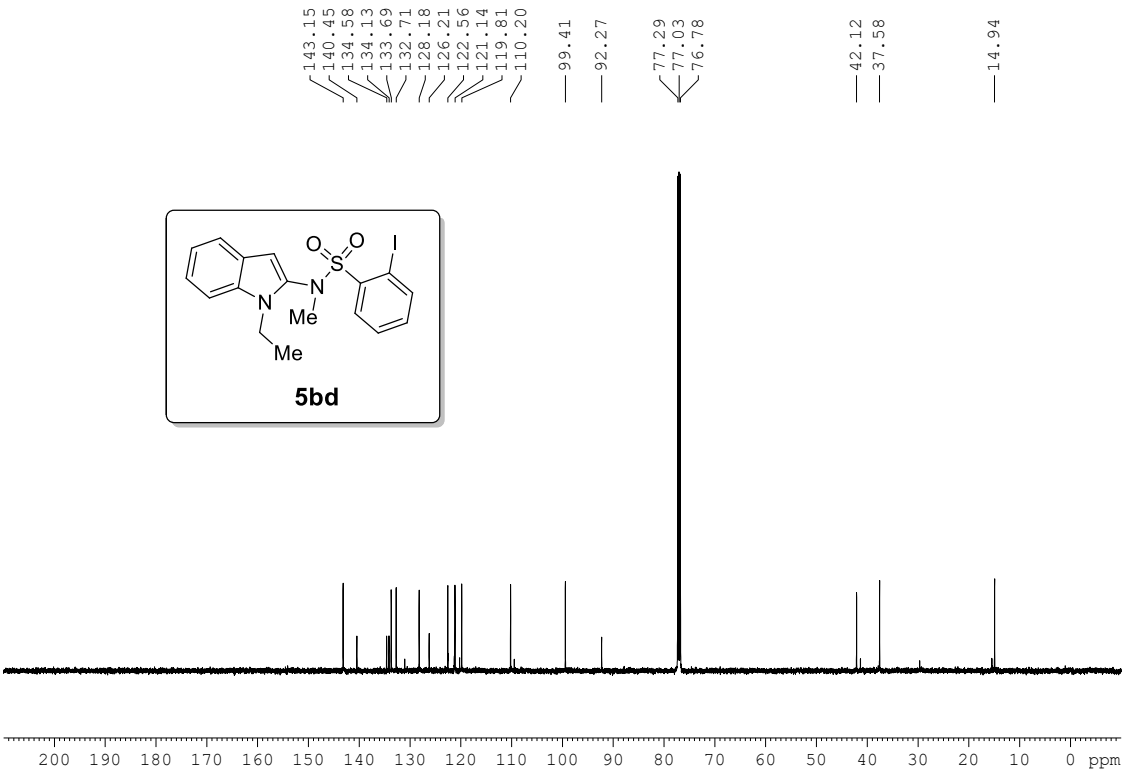


Figure S30. ^{13}C{^{1}H} NMR spectrum of compound **5bd**

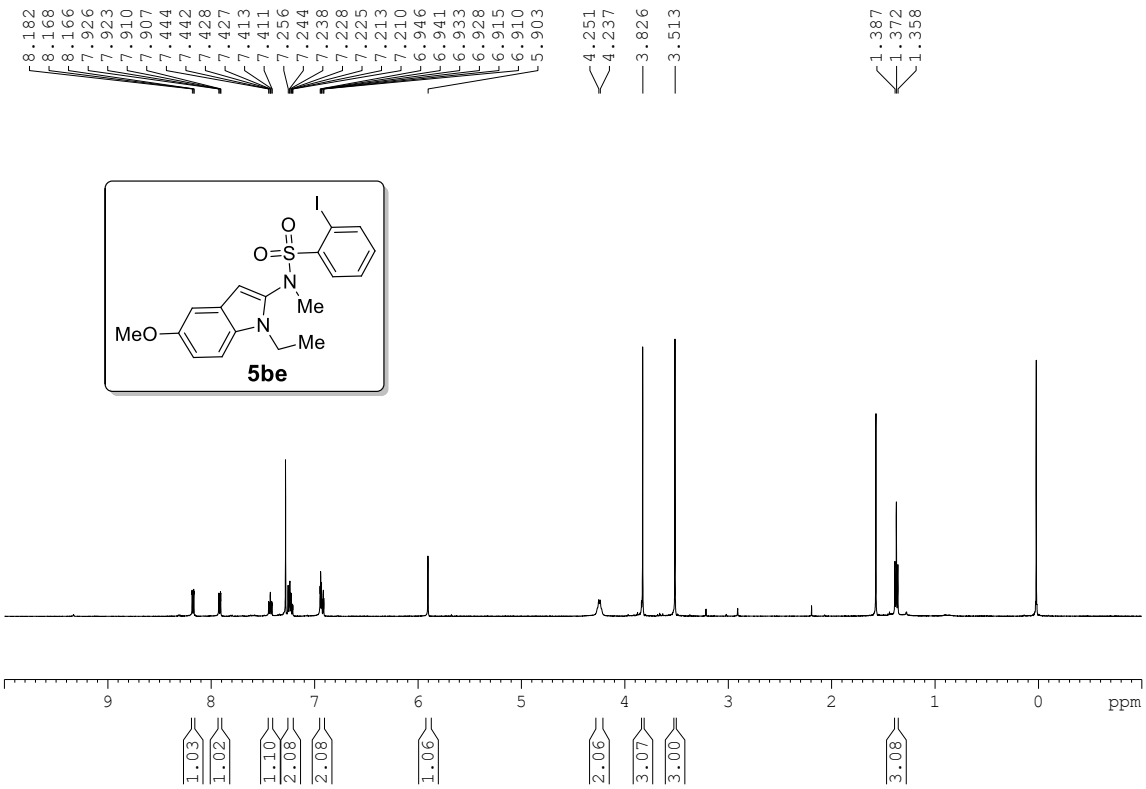


Figure S31. ^{1}H NMR spectrum of compound **5be**

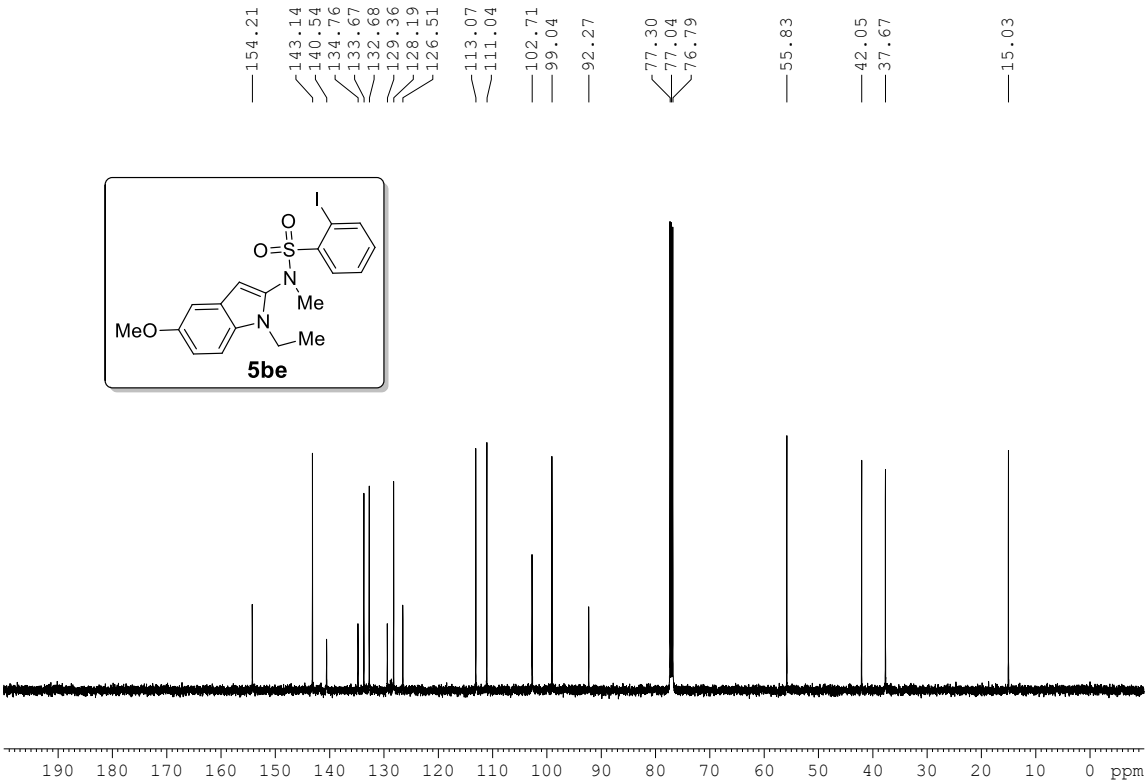


Figure S32. ^{13}C{^{1}H} NMR spectrum of compound **5be**

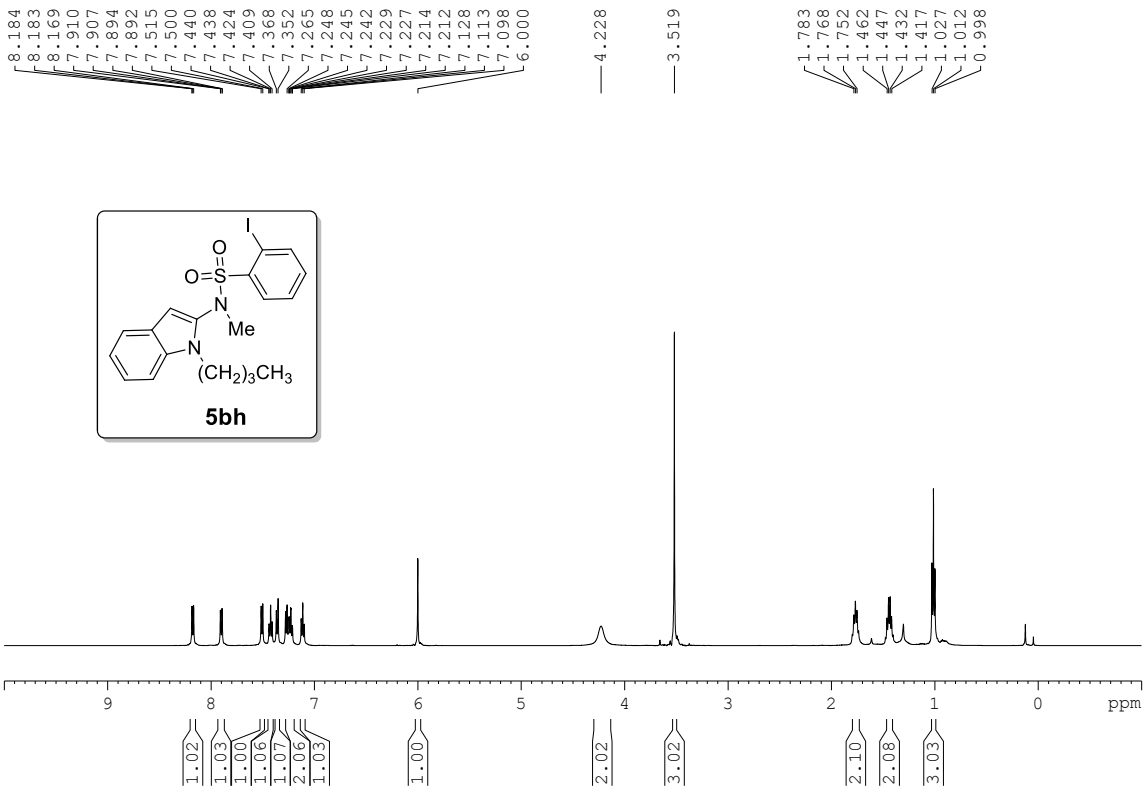


Figure S33. ^{1}H NMR spectrum of compound **5bh**

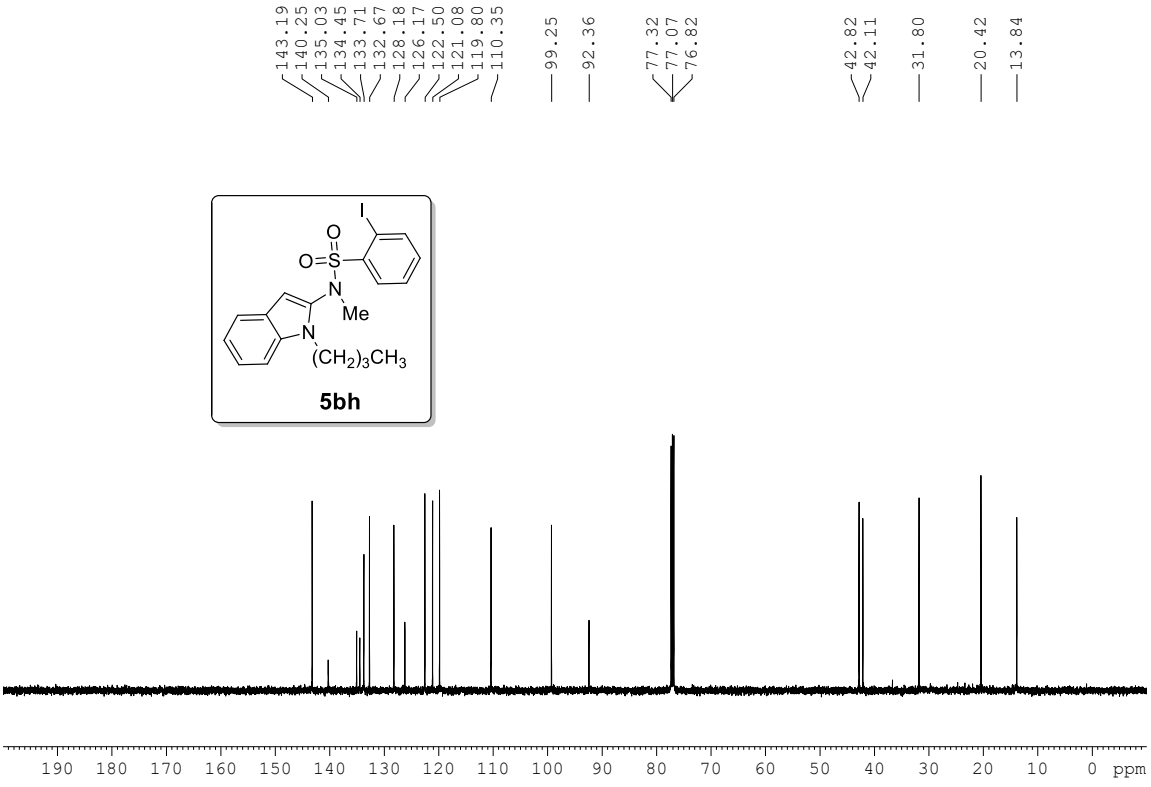


Figure S34. ^{13}C{^{1}H} NMR spectrum of compound **5bh**

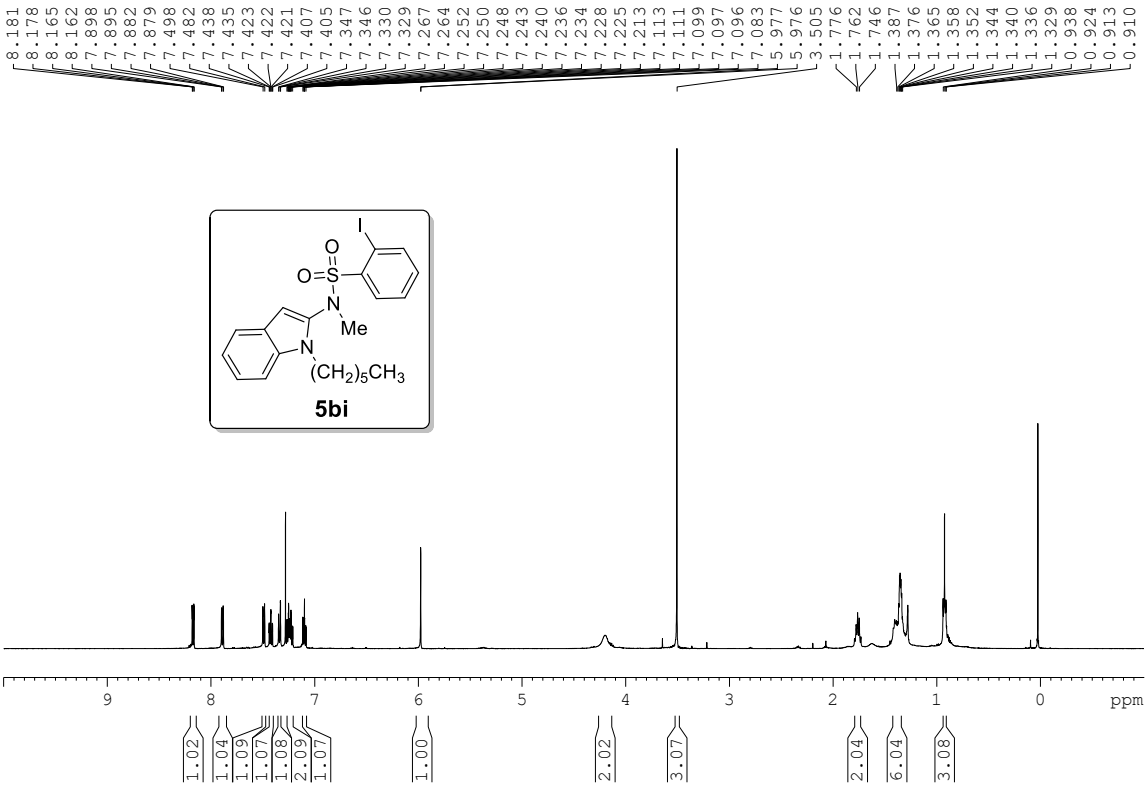


Figure S35. ^{1}H NMR spectrum of compound **5bi**

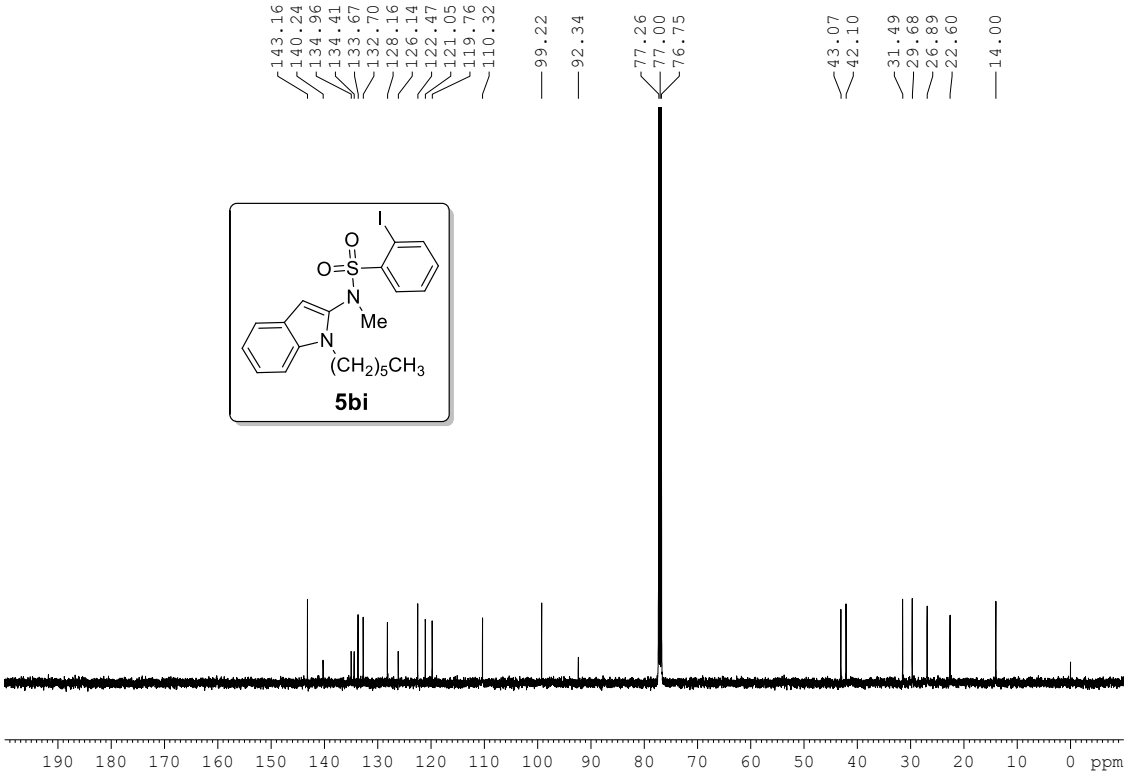


Figure S36. ^{13}C{^{1}H} NMR spectrum of compound **5bi**

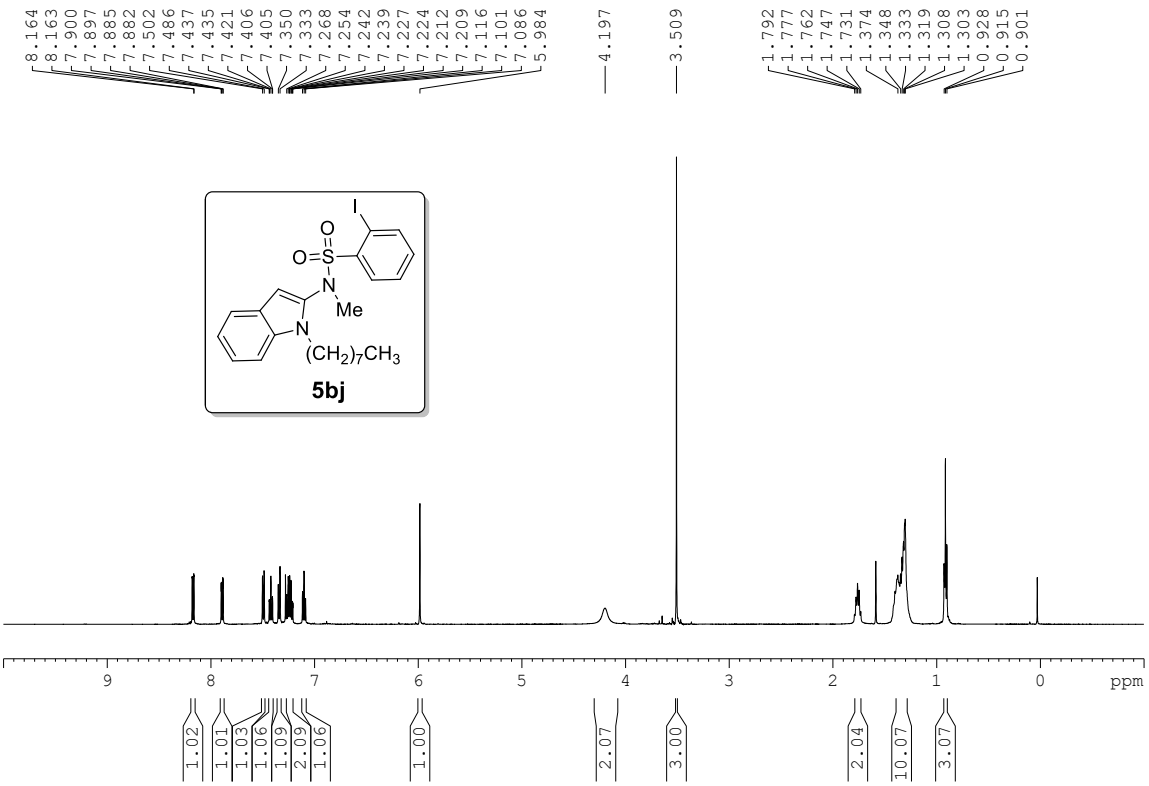


Figure S37. ^{1}H NMR spectrum of compound **5bj**

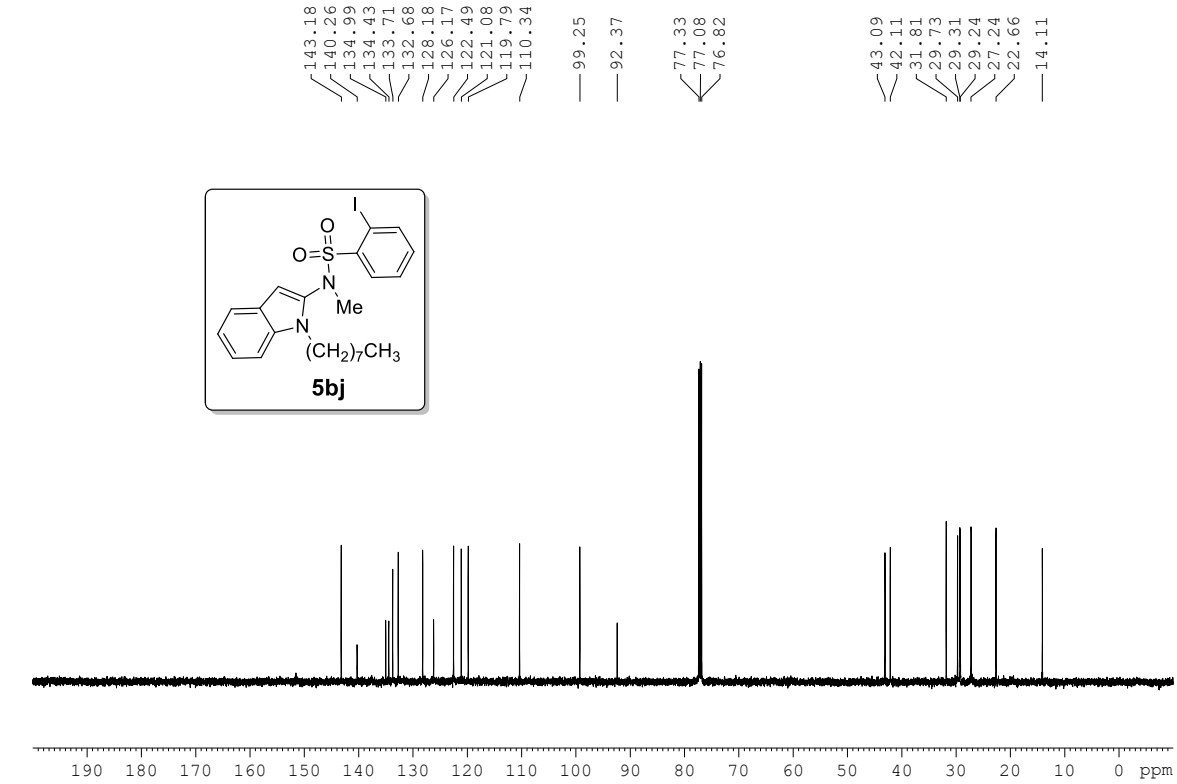


Figure S38. ^{13}C{^{1}H} NMR spectrum of compound **5bj**

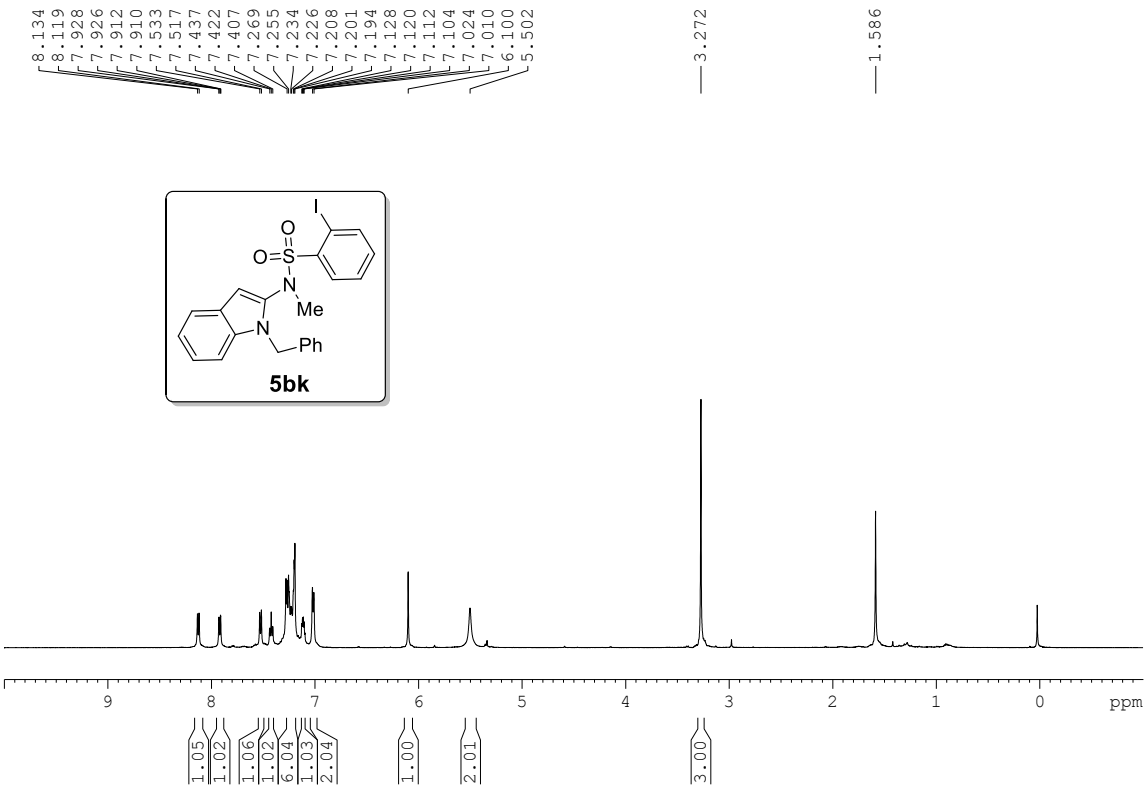


Figure S39. ^{1}H NMR spectrum of compound **5bk**

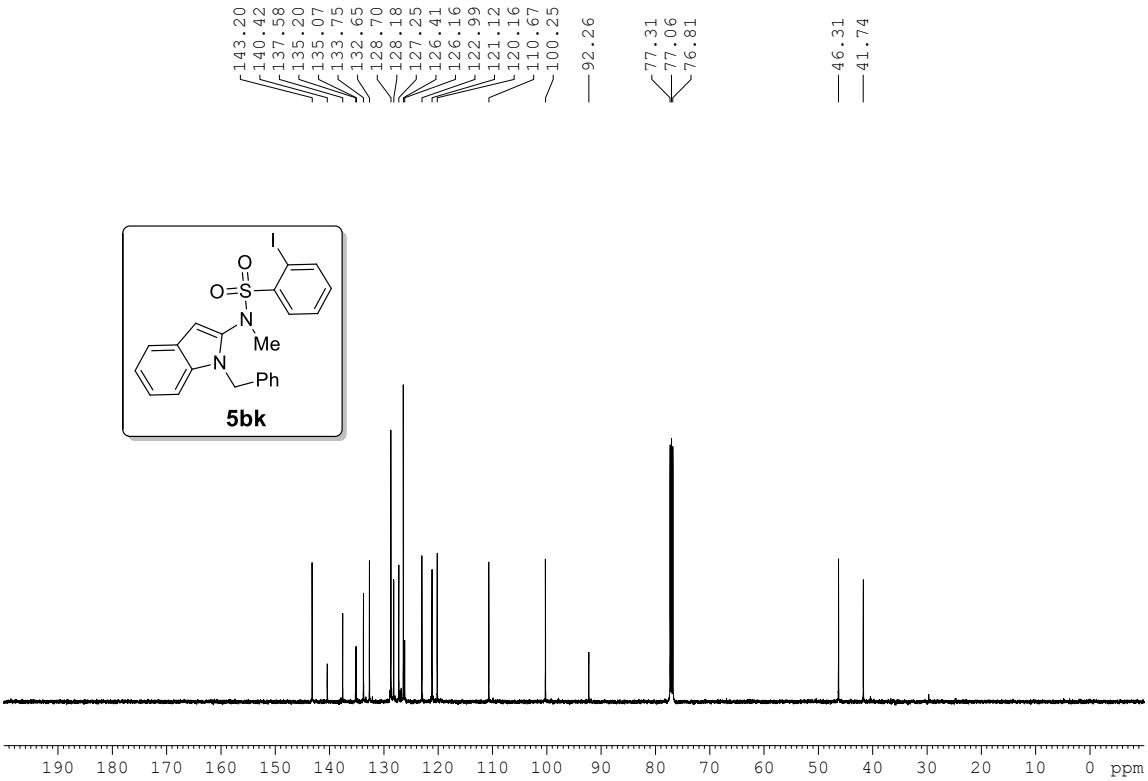


Figure S40. ^{13}C{^{1}H} NMR spectrum of compound **5bk**

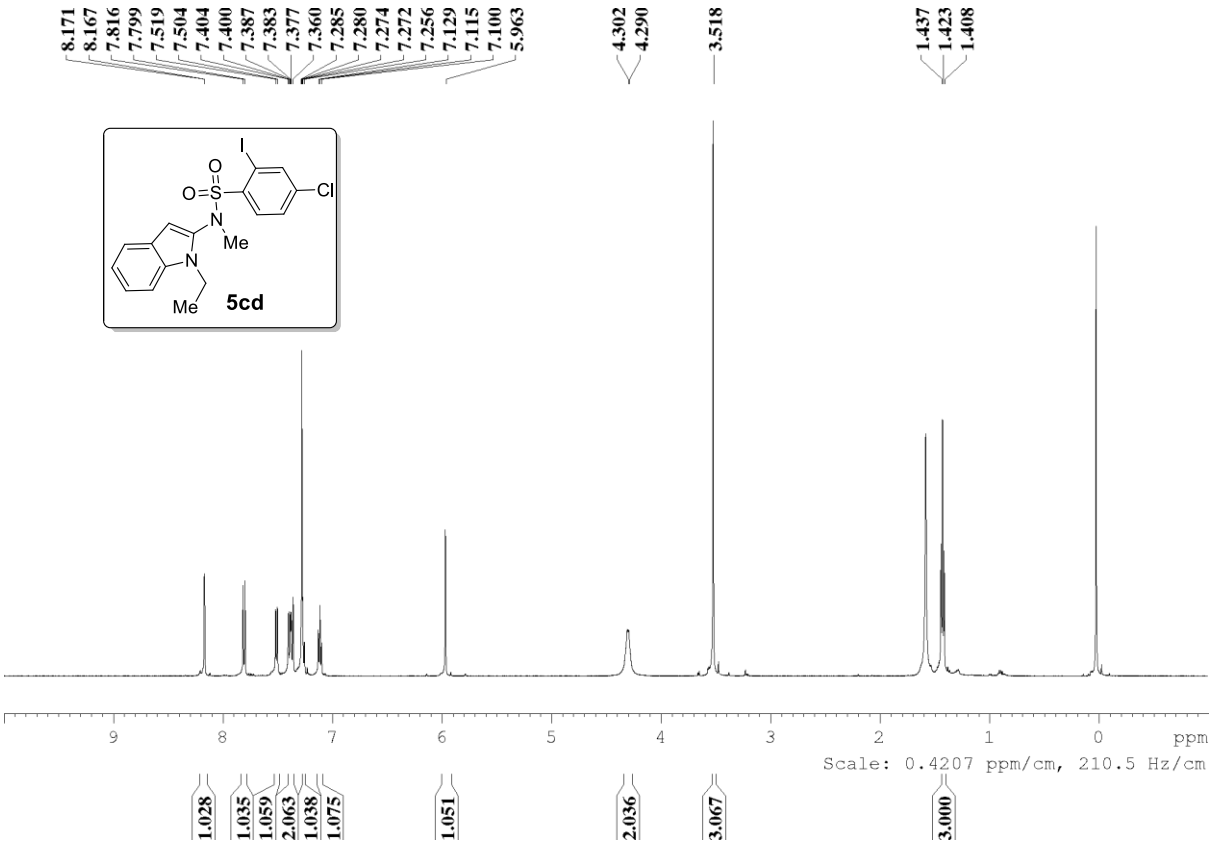


Figure S41. ^{1}H NMR spectrum of compound **5cd**

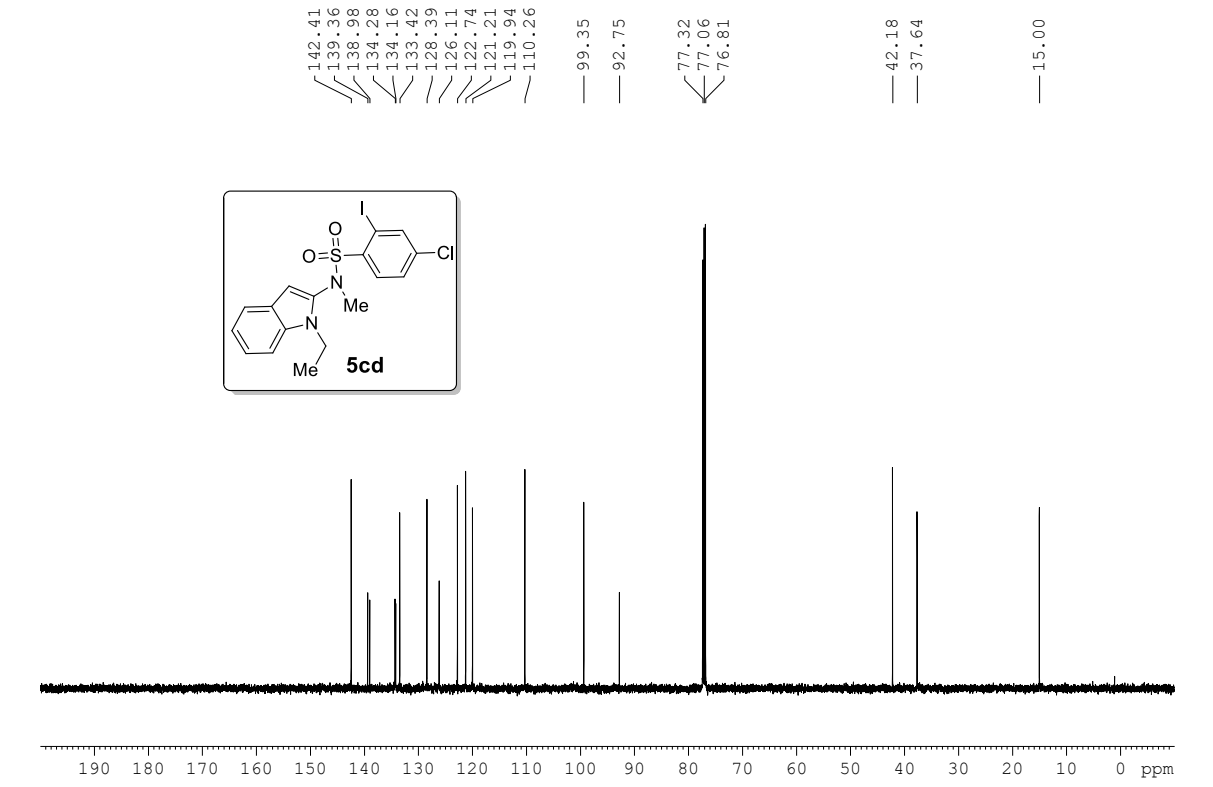


Figure S42. ^{13}C{^{1}H} NMR spectrum of compound **5cd**

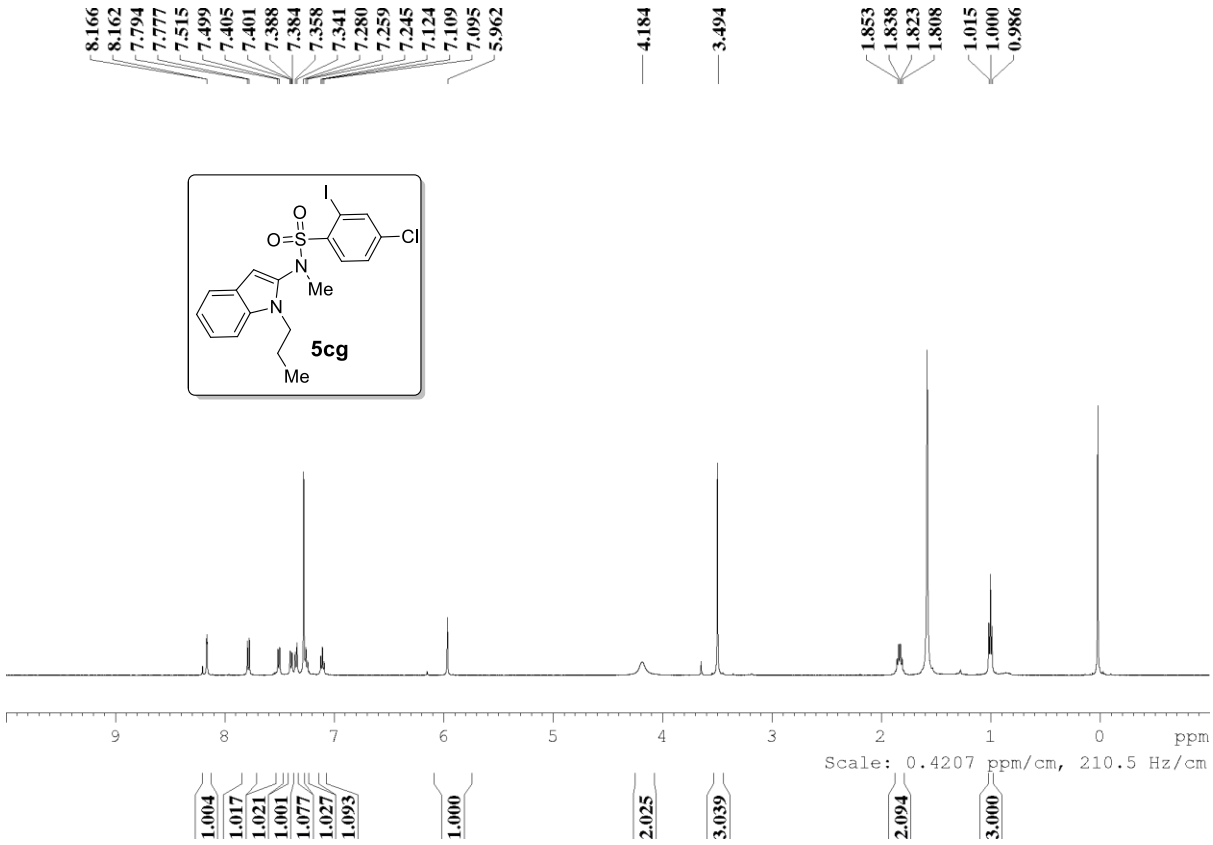


Figure S43. ^{1}H NMR spectrum of compound **5cg**

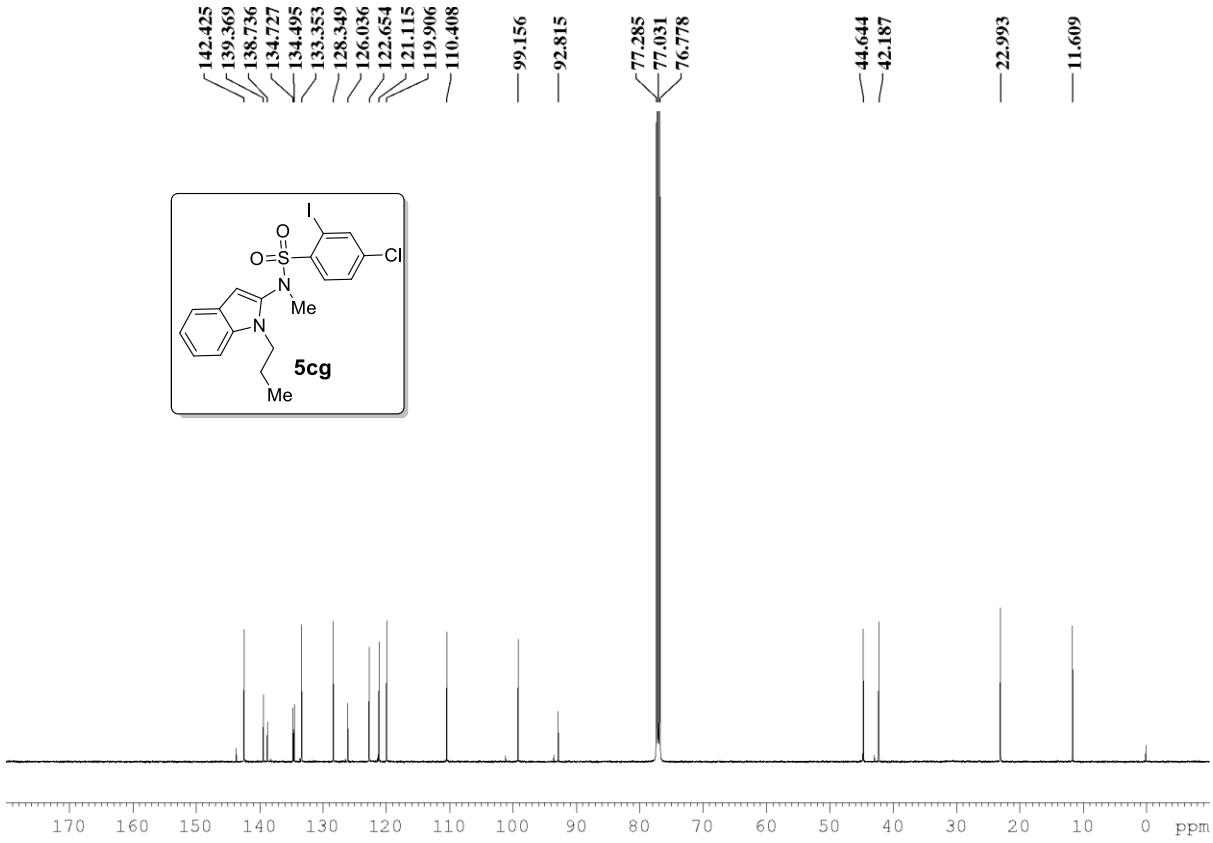


Figure S44. ^{13}C{^{1}H} NMR spectrum of compound **5cg**

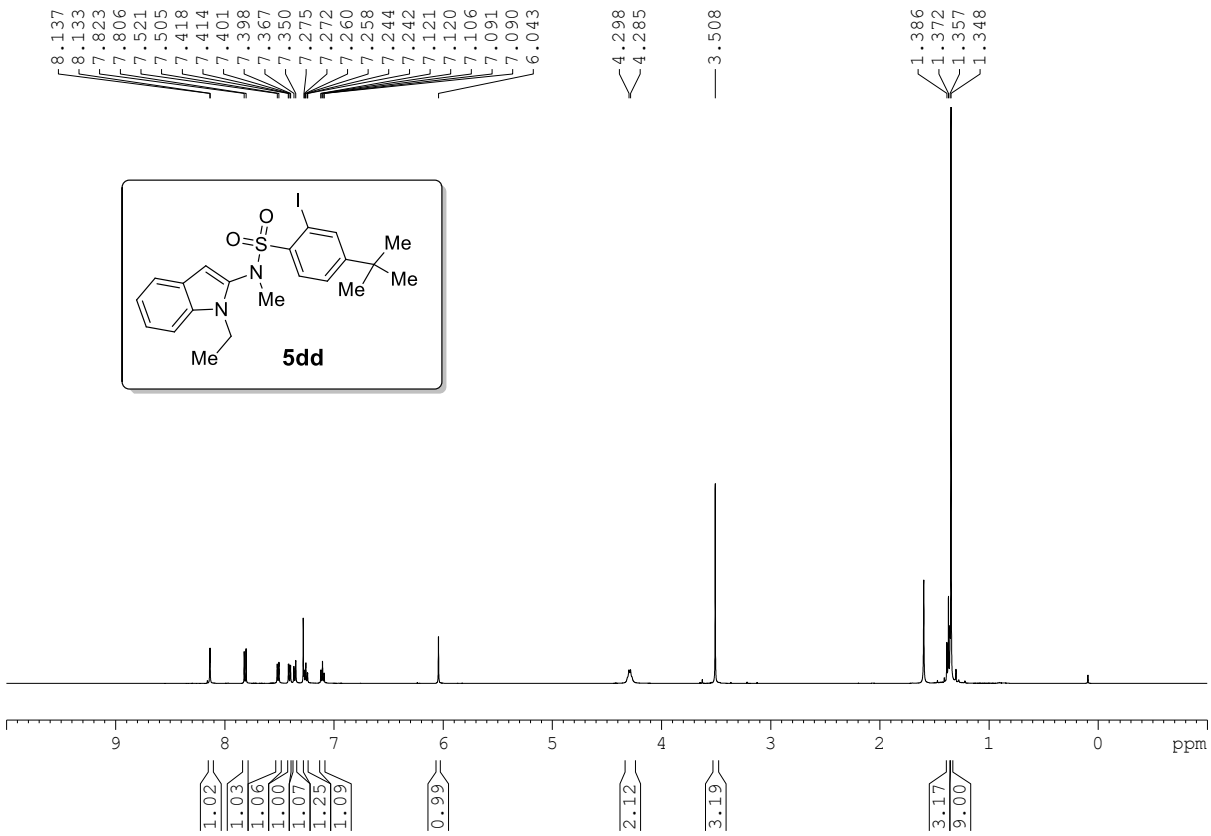


Figure S45. ^{1}H NMR spectrum of compound **5dd**

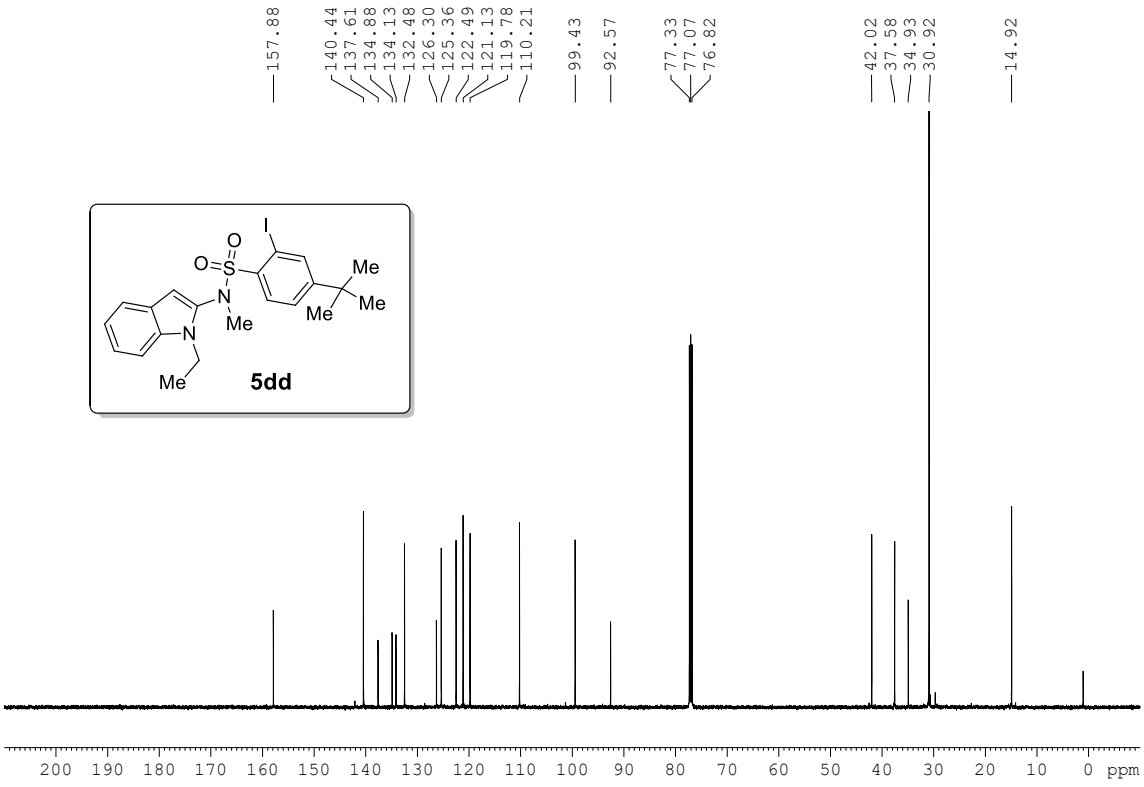


Figure S46. ^{13}C{^{1}H} NMR spectrum of compound **5dd**

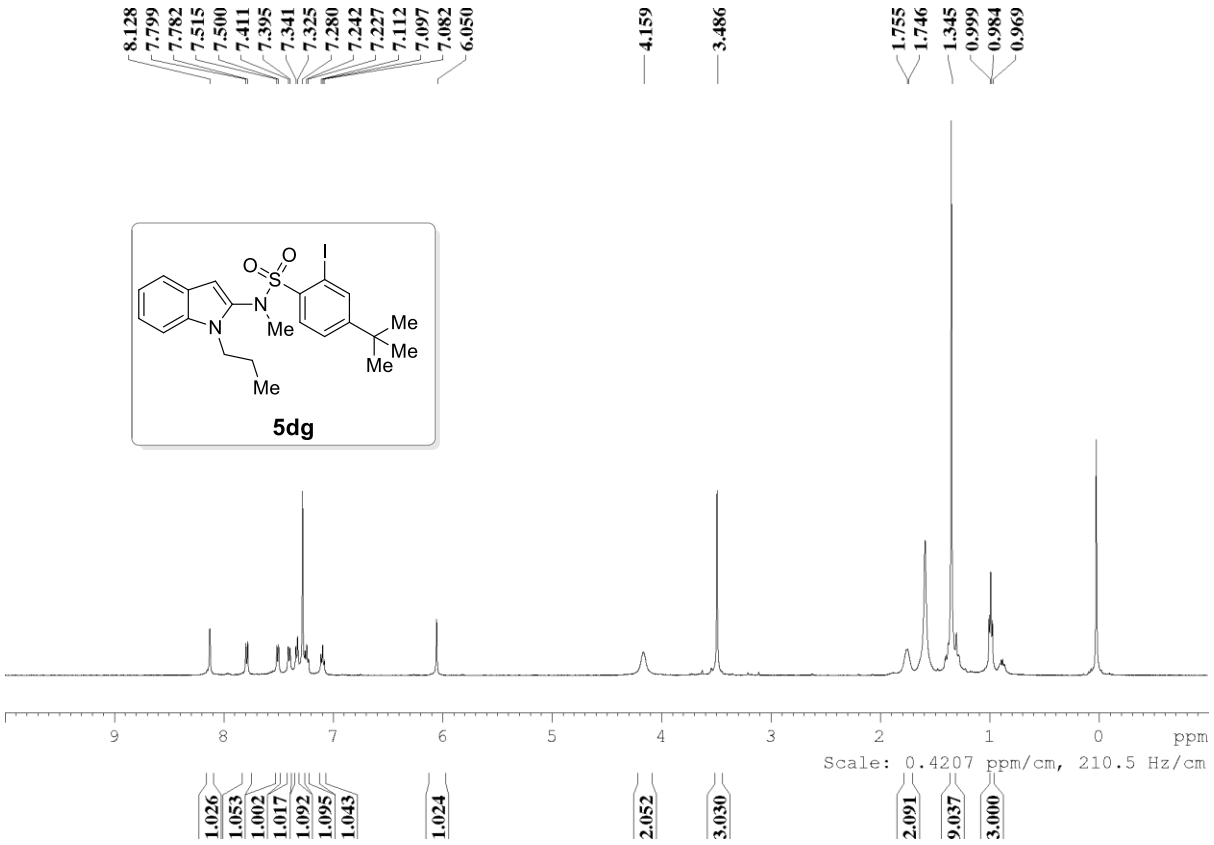


Figure S47. ^{1}H NMR spectrum of compound **5dg**

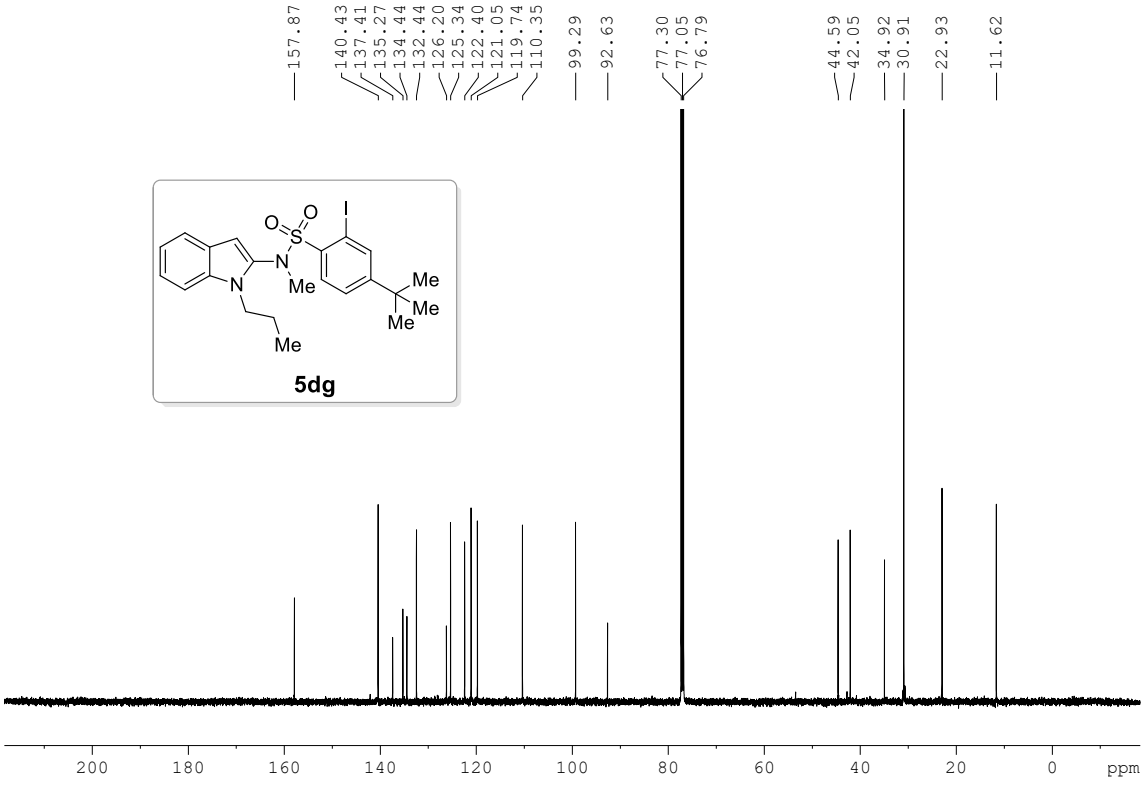


Figure S48. ^{13}C{^{1}H} NMR spectrum of compound **5dg**

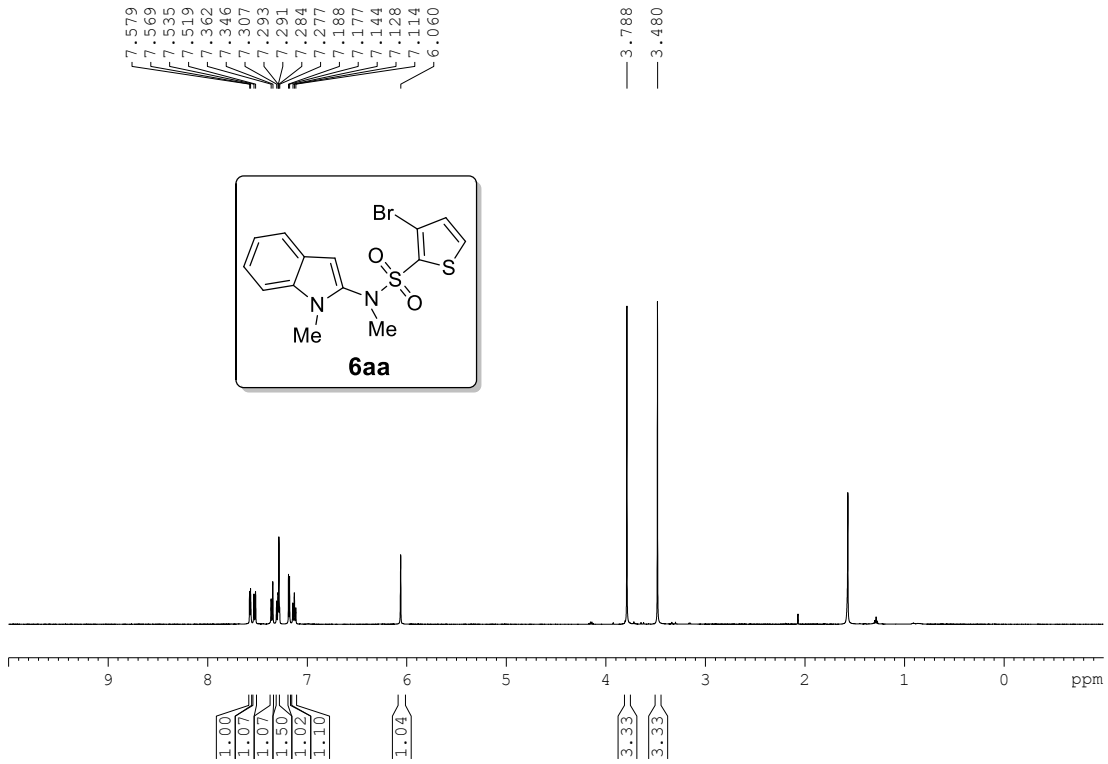


Figure S49. ^{1}H NMR spectrum of compound **6aa**

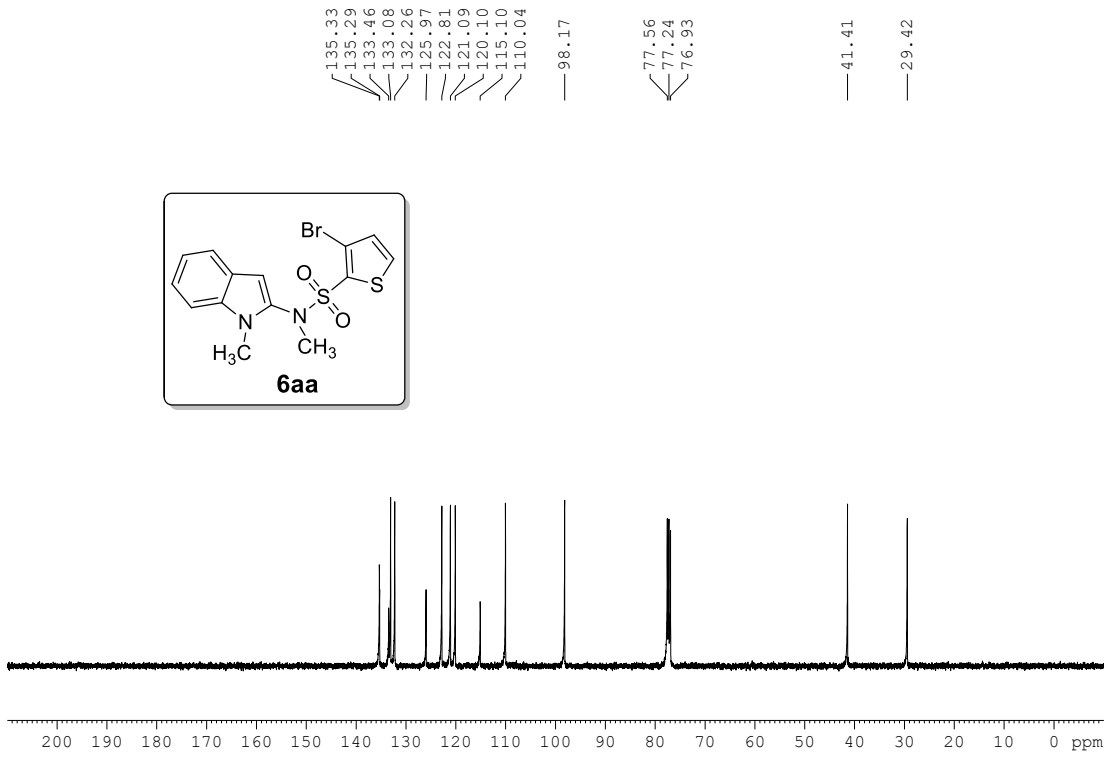


Figure S50. ^{13}C{^{1}H} NMR spectrum of compound **6aa**

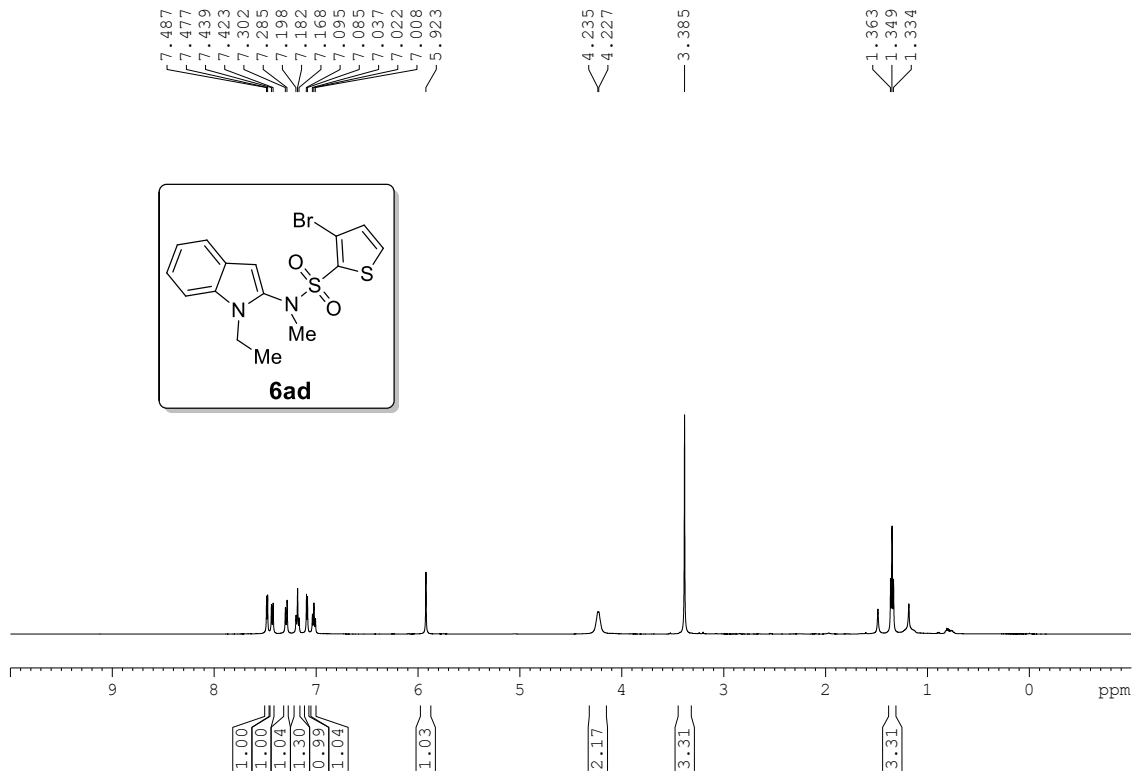


Figure S51. ^{1}H NMR spectrum of compound **6ad**

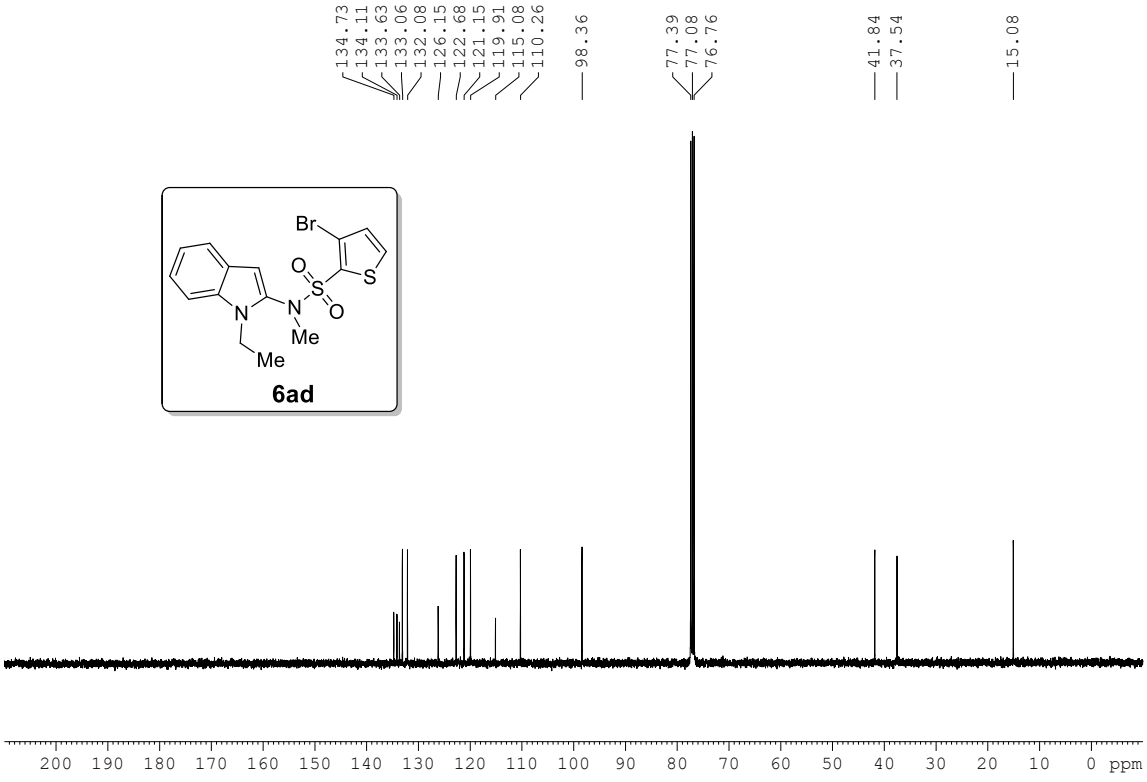


Figure S52. ^{13}C{^{1}H} NMR spectrum of compound **6ad**

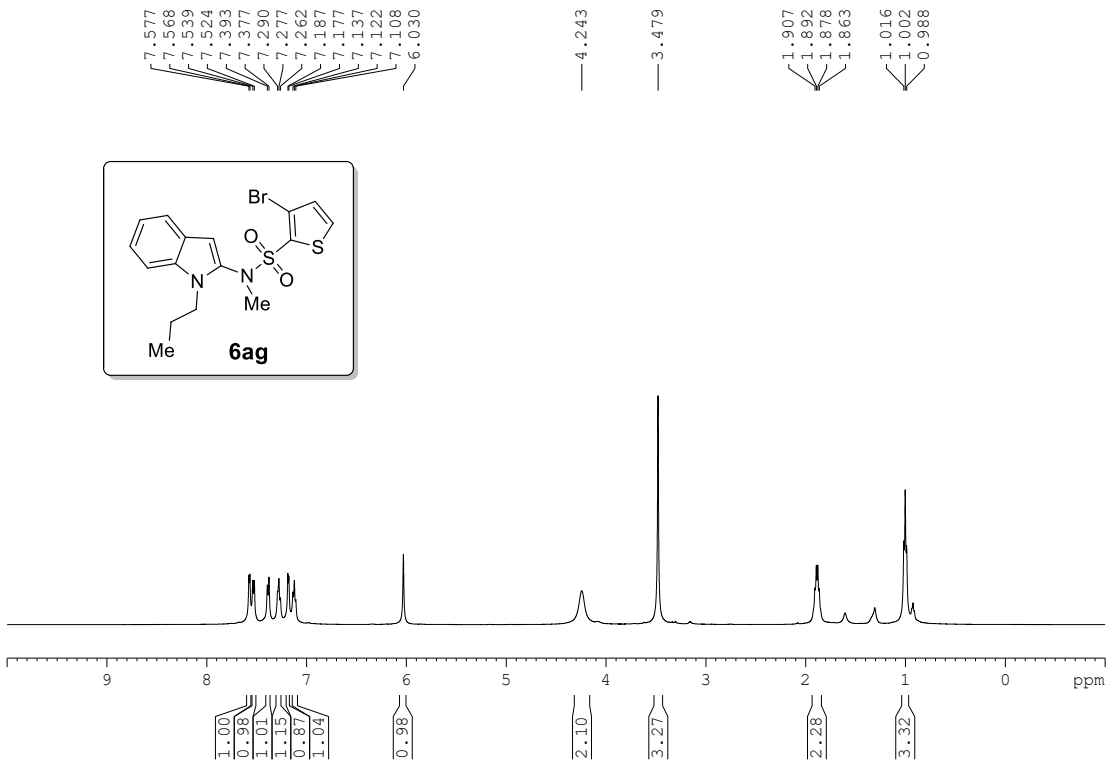


Figure S53. ^{1}H NMR spectrum of compound **6ag**

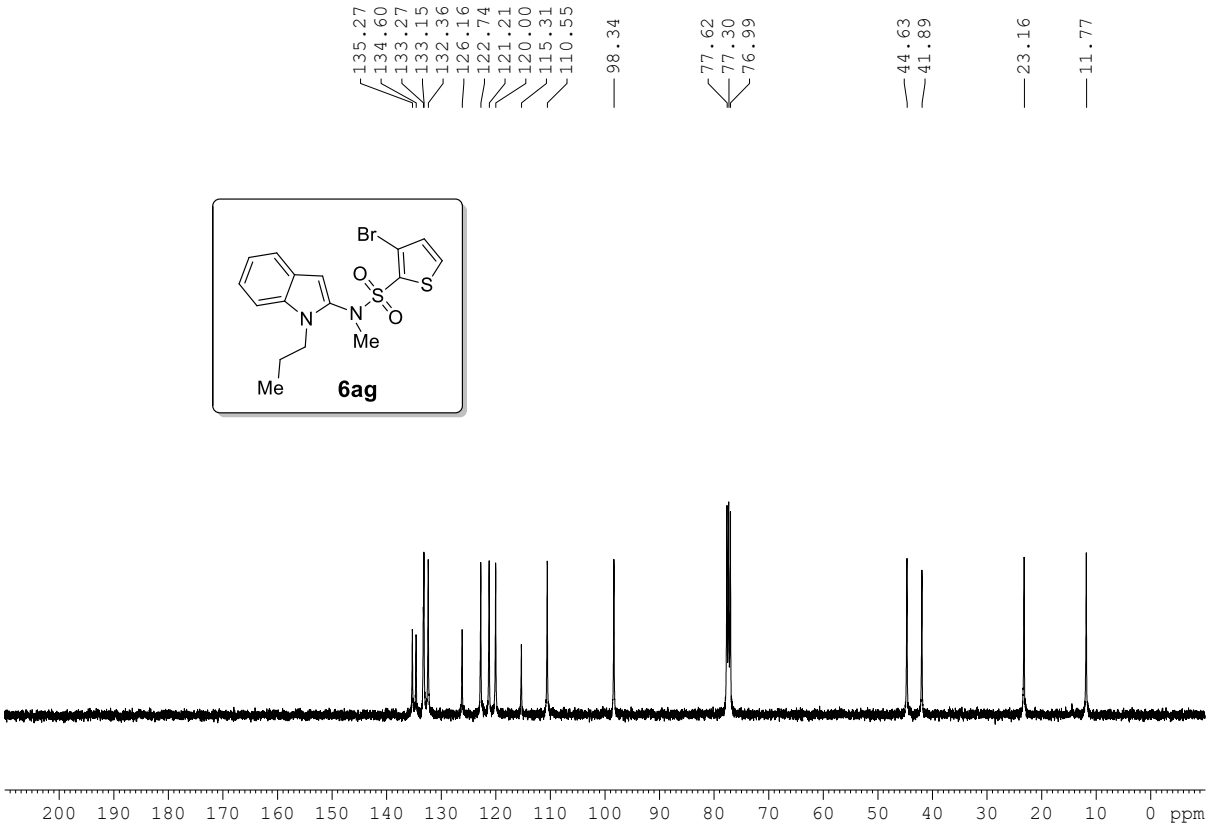


Figure S54. ^{13}C{^{1}H} NMR spectrum of compound **6ag**

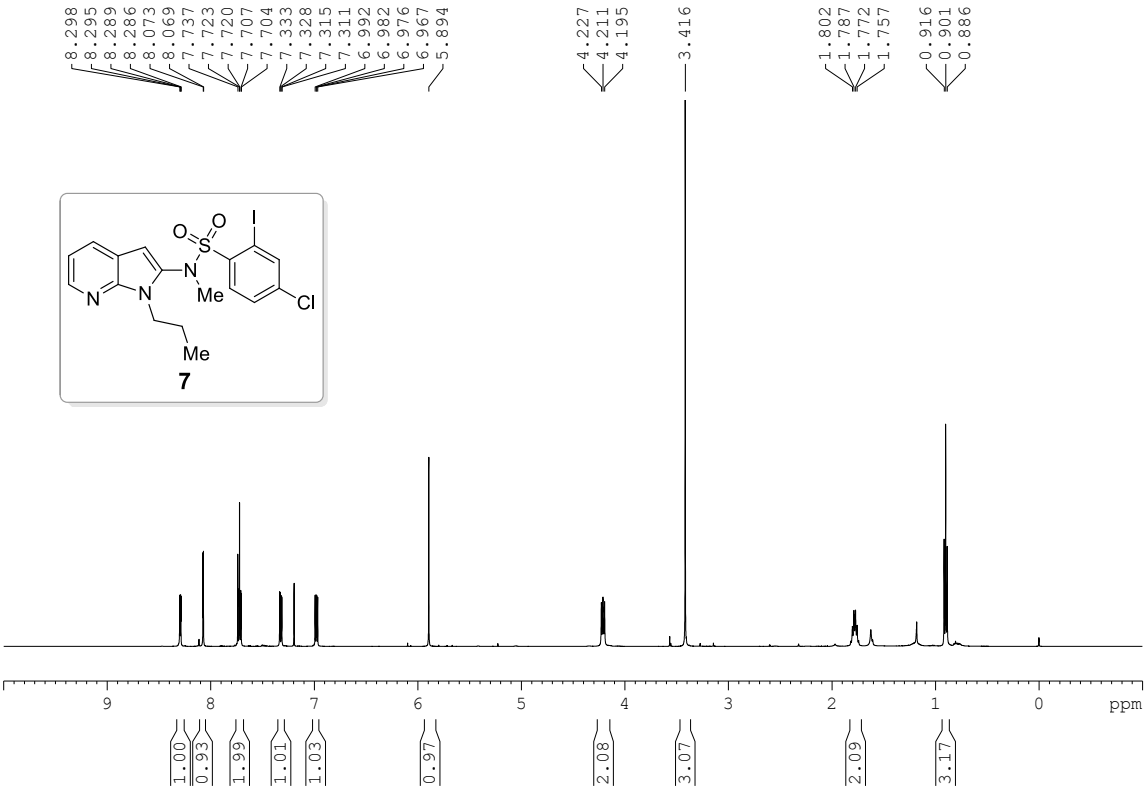


Figure S55. ^{1}H NMR spectrum of compound **7**

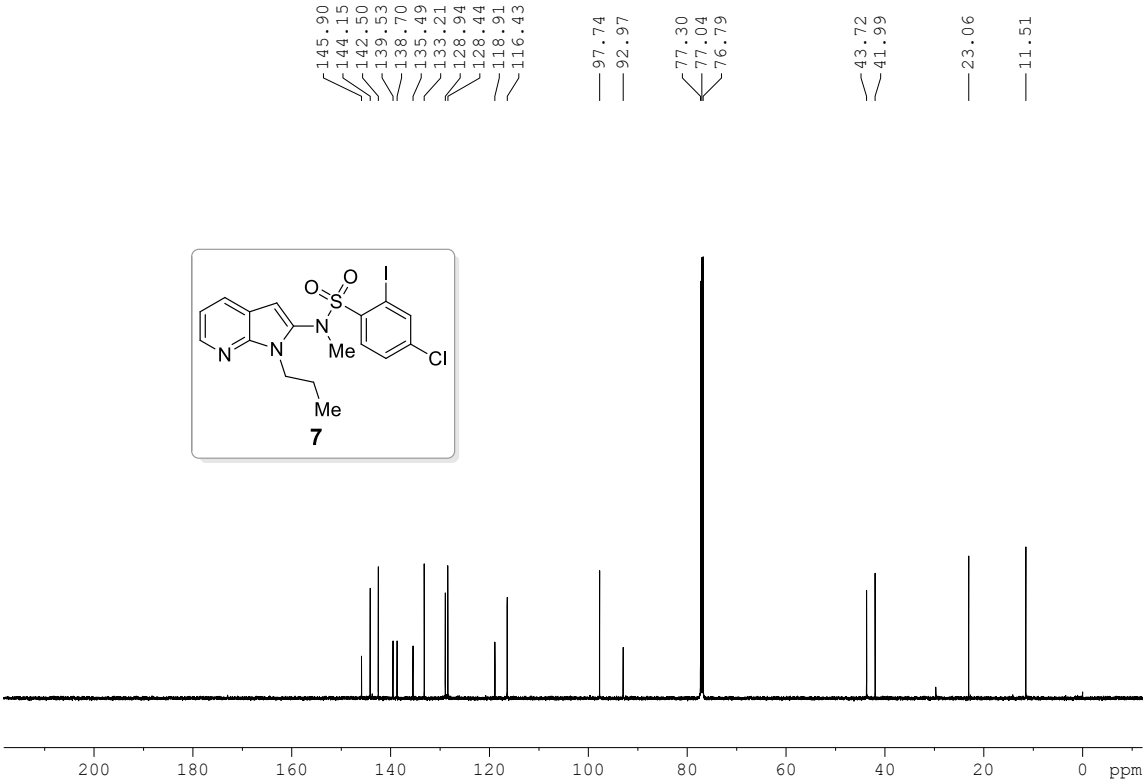


Figure S56. ^{13}C{^{1}H} NMR spectrum of compound **7**

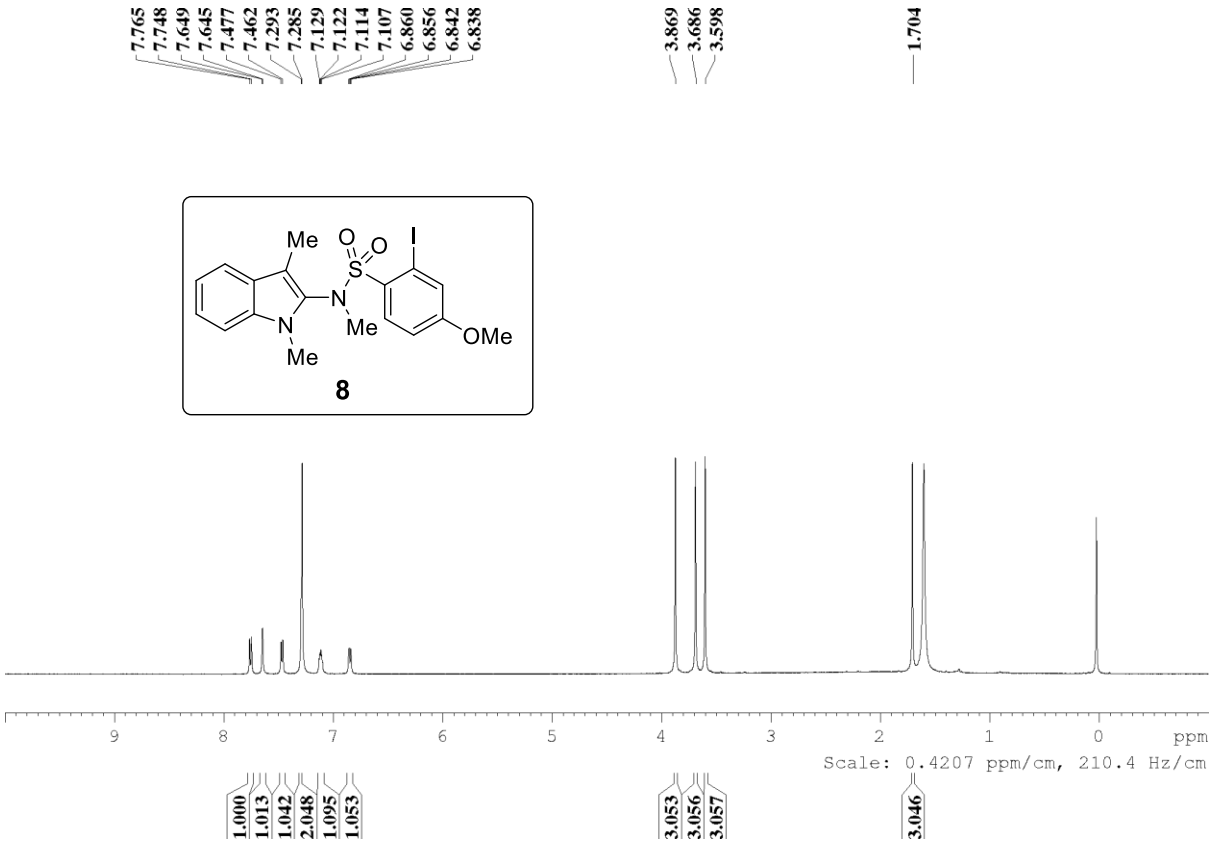


Figure S57. ^{1}H NMR spectrum of compound **8**

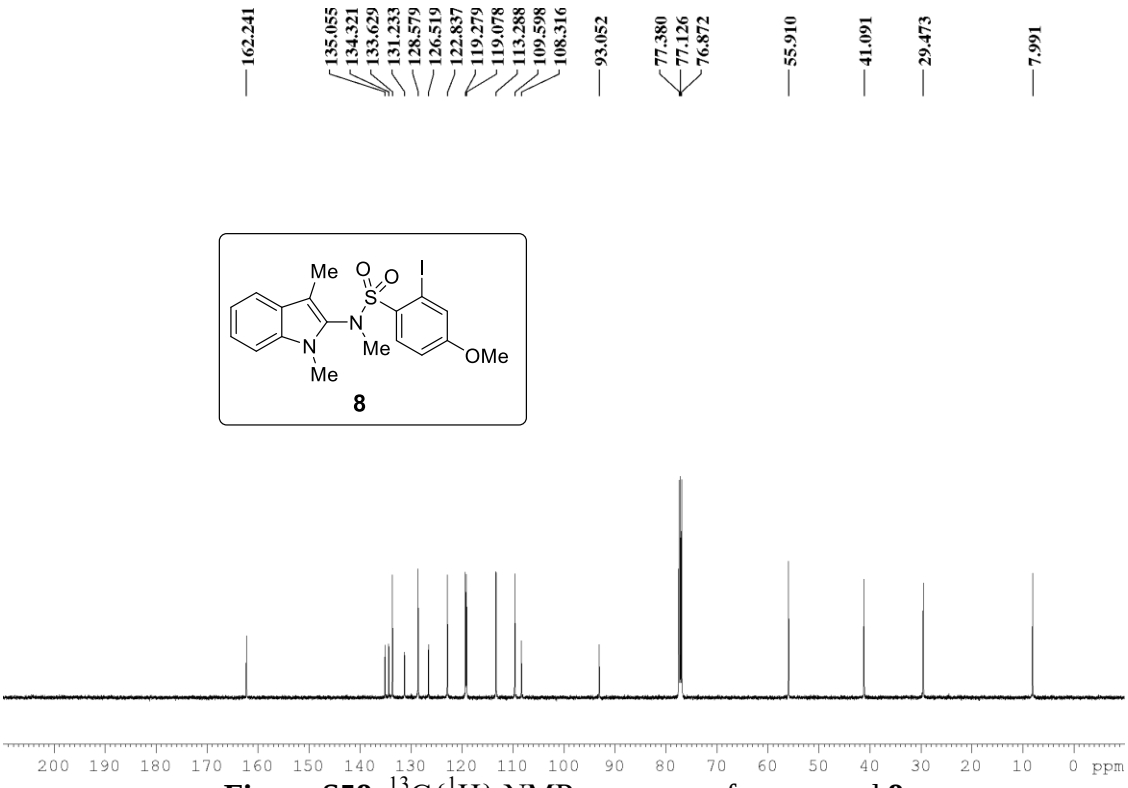


Figure S58. ^{13}C{^{1}H} NMR spectrum of compound **8**

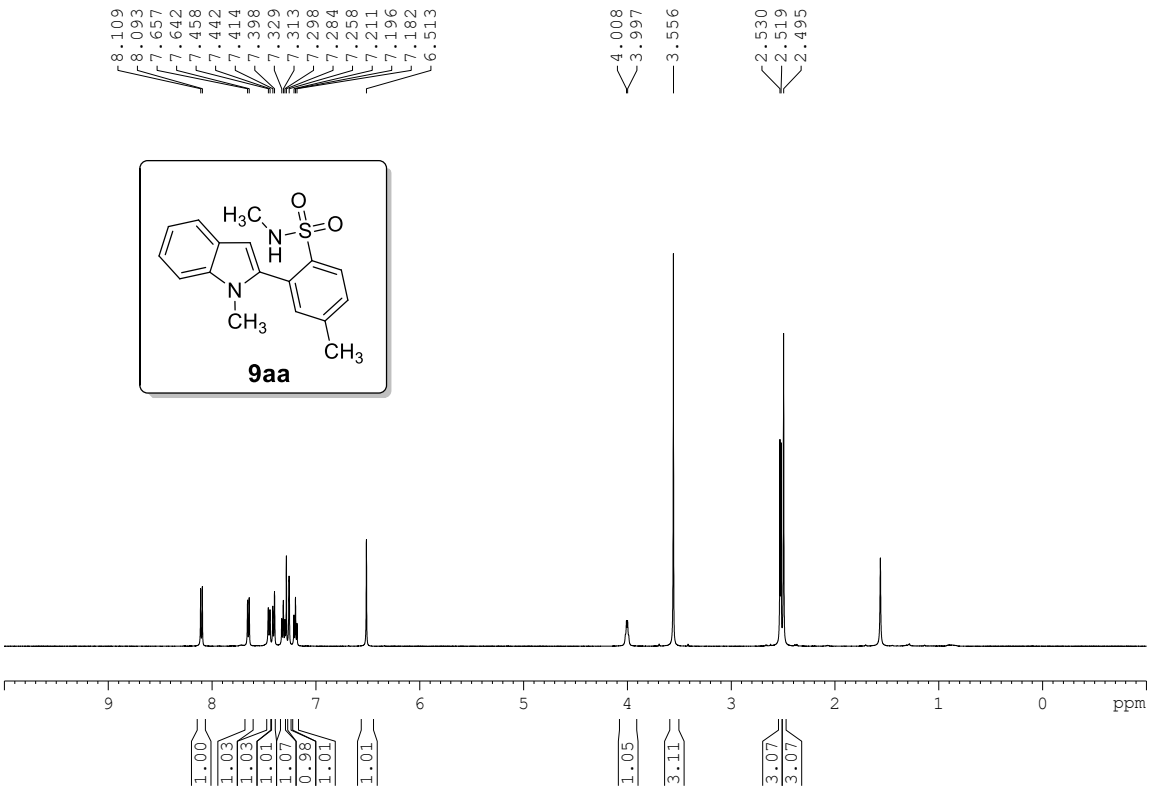


Figure S59. ^{1}H NMR spectrum of compound **9aa**

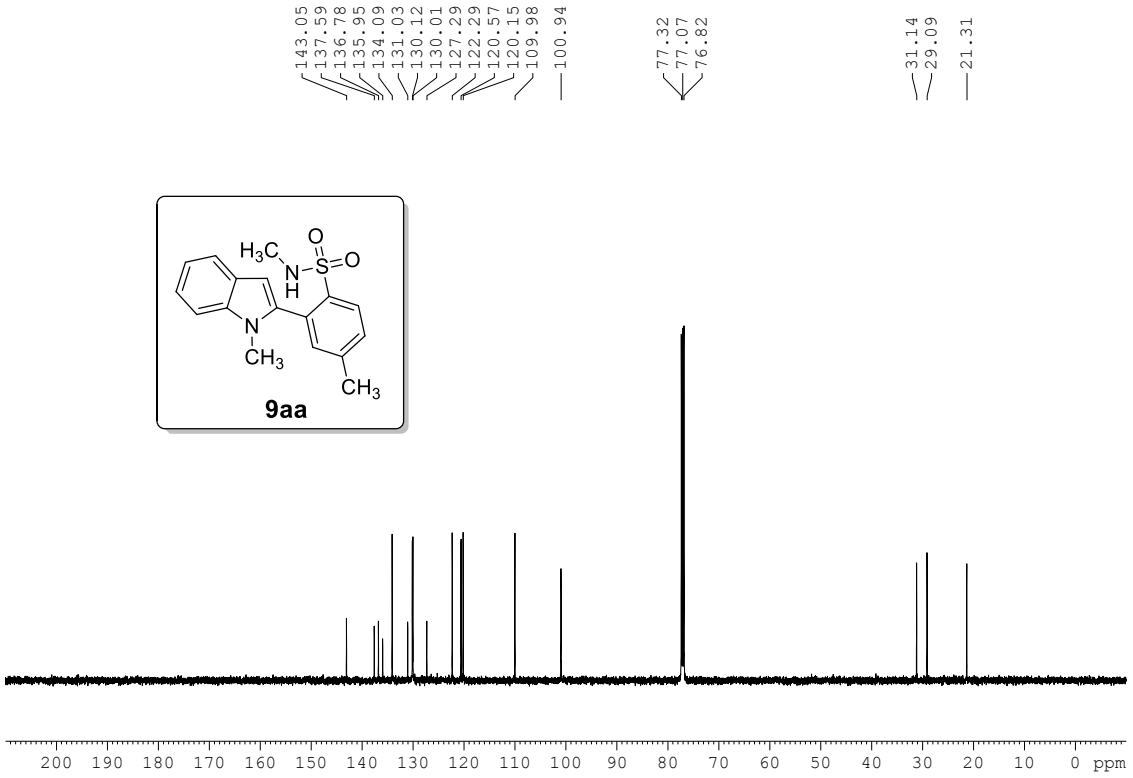


Figure S60. ^{13}C{^{1}H} NMR spectrum of compound **9aa**

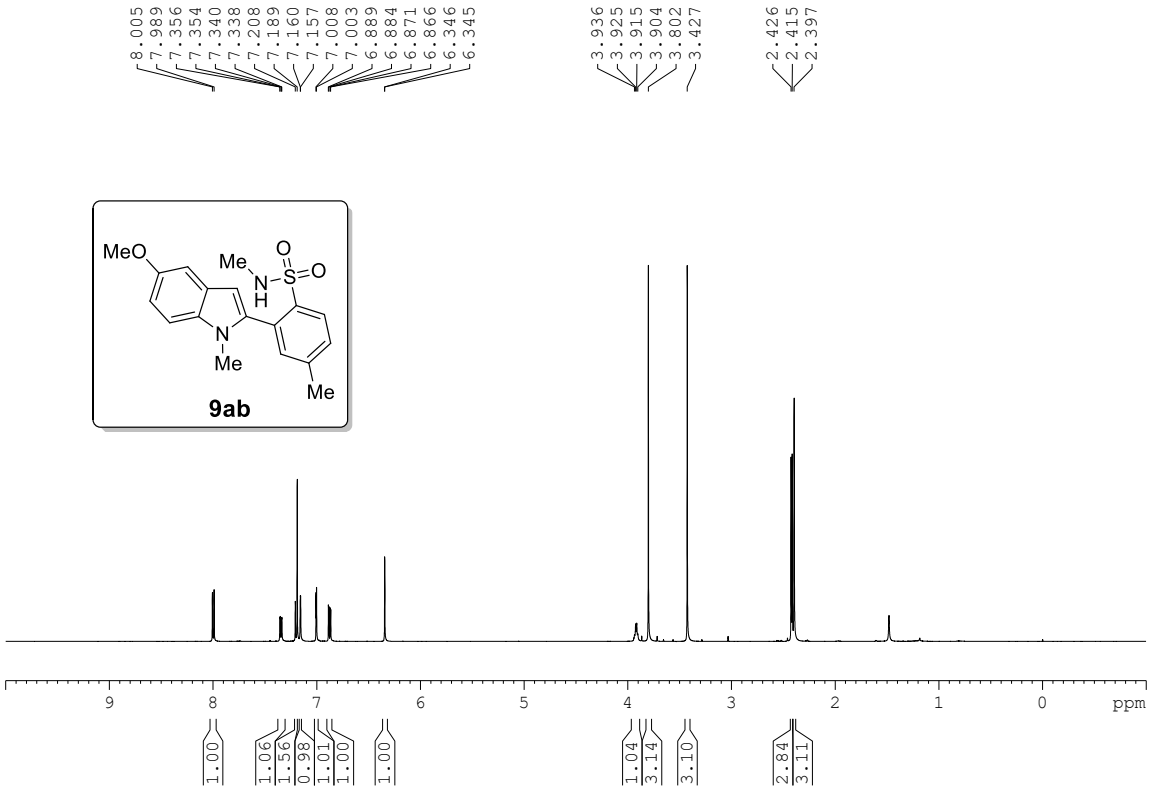


Figure S61. ^{1}H NMR spectrum of compound **9ab**

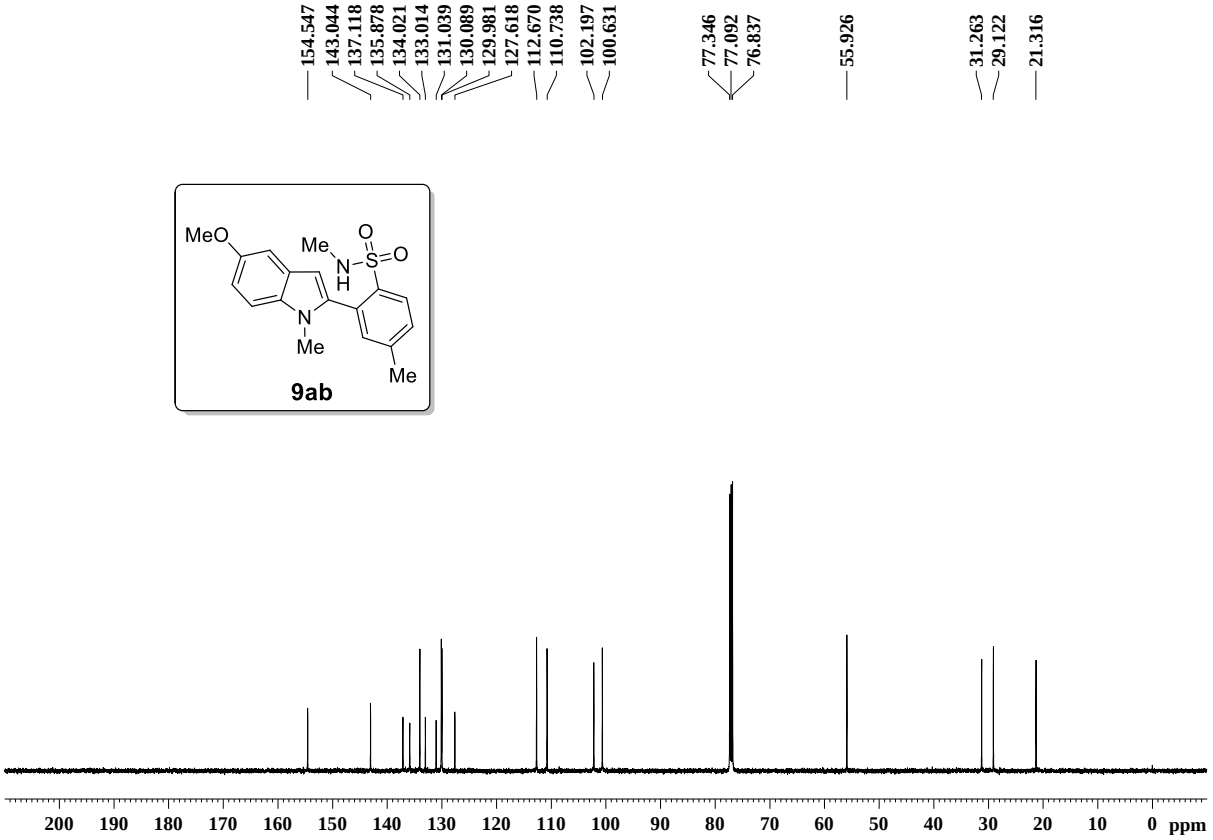


Figure S62. ^{13}C{^{1}H} NMR spectrum of compound **9ab**

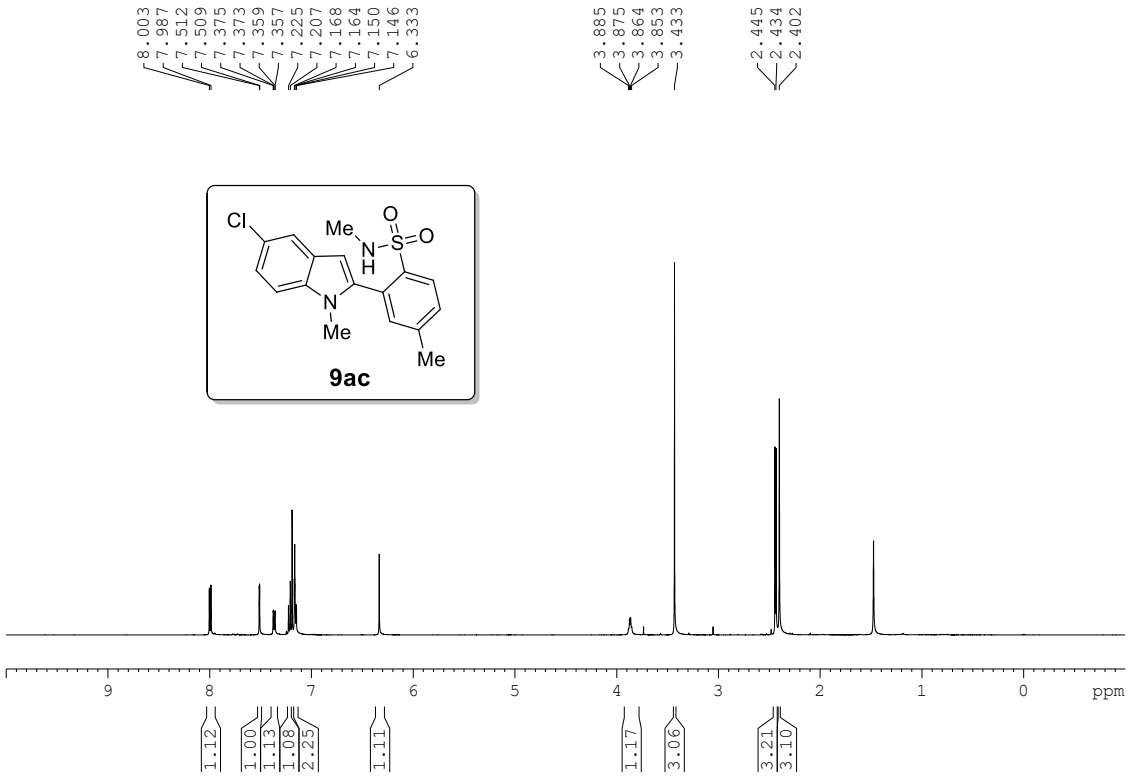


Figure S63. ^{1}H NMR spectrum of compound **9ac**

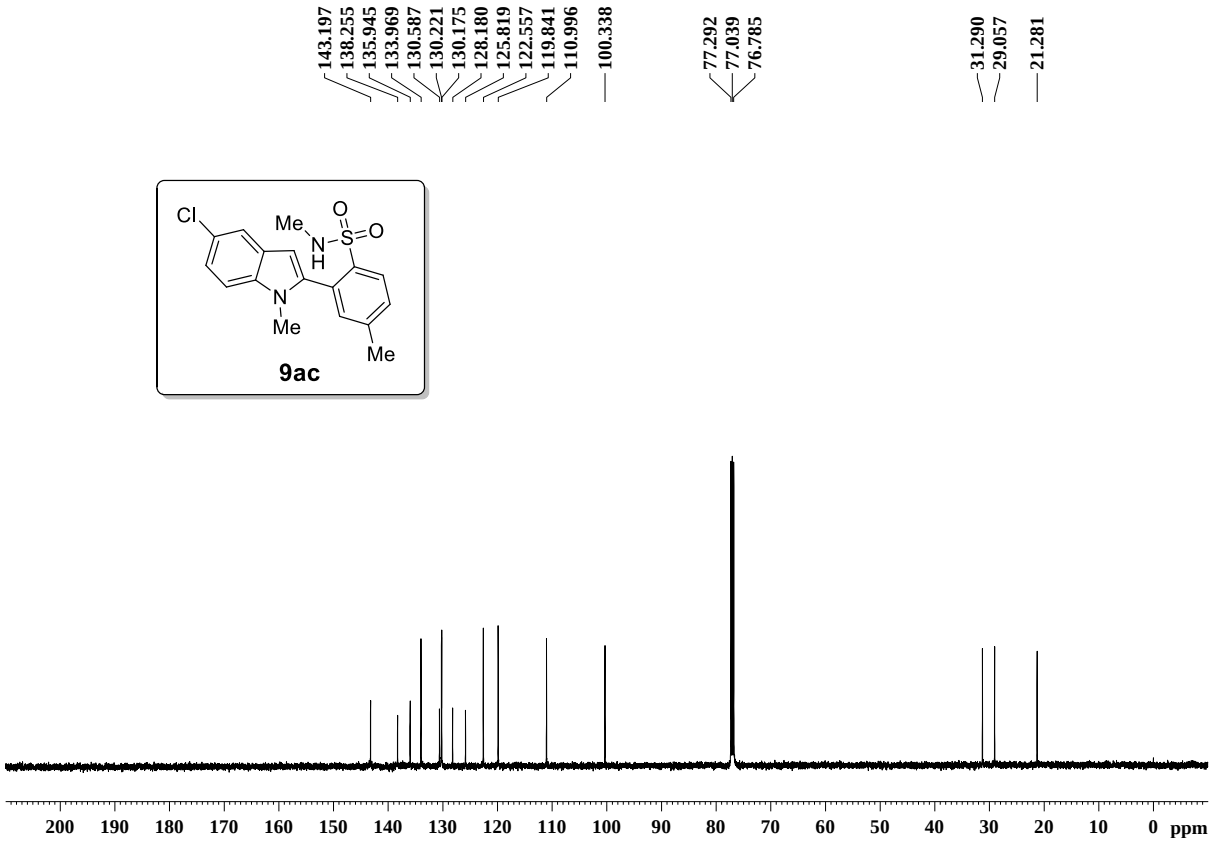


Figure S64. ^{13}C{^{1}H} NMR spectrum of compound **9ac**

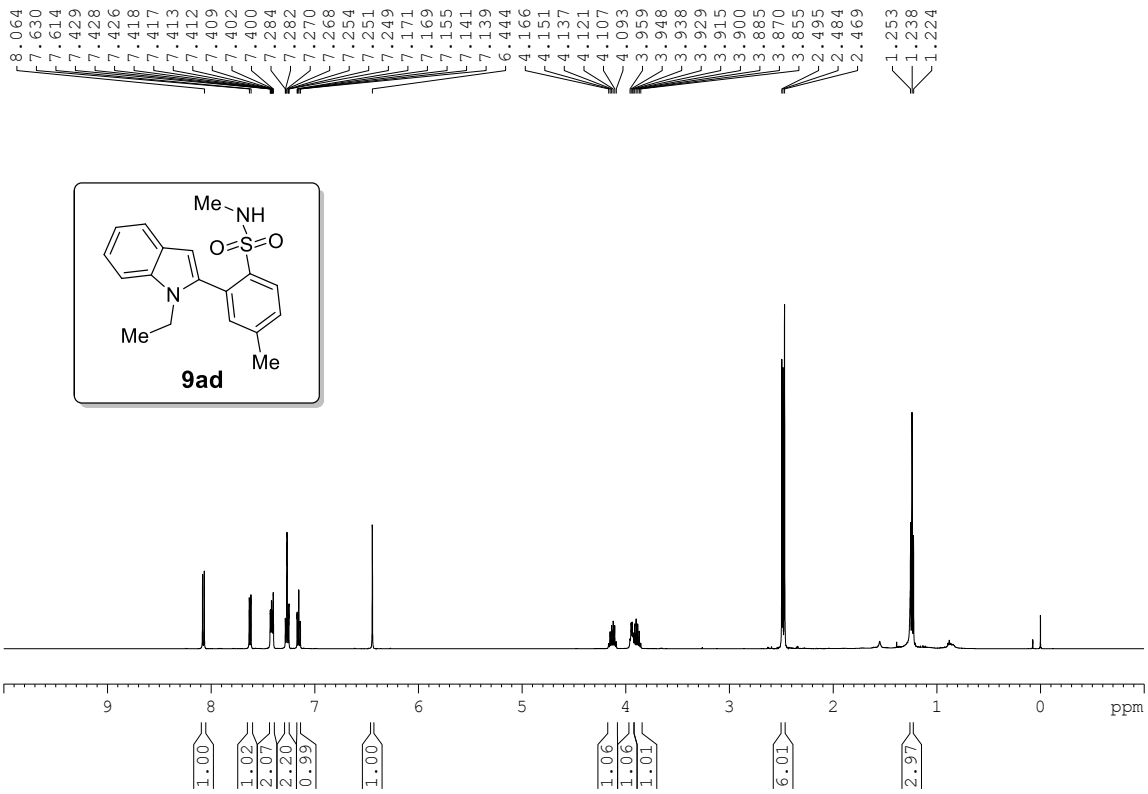


Figure S65. ^{1}H NMR spectrum of compound **9ad**

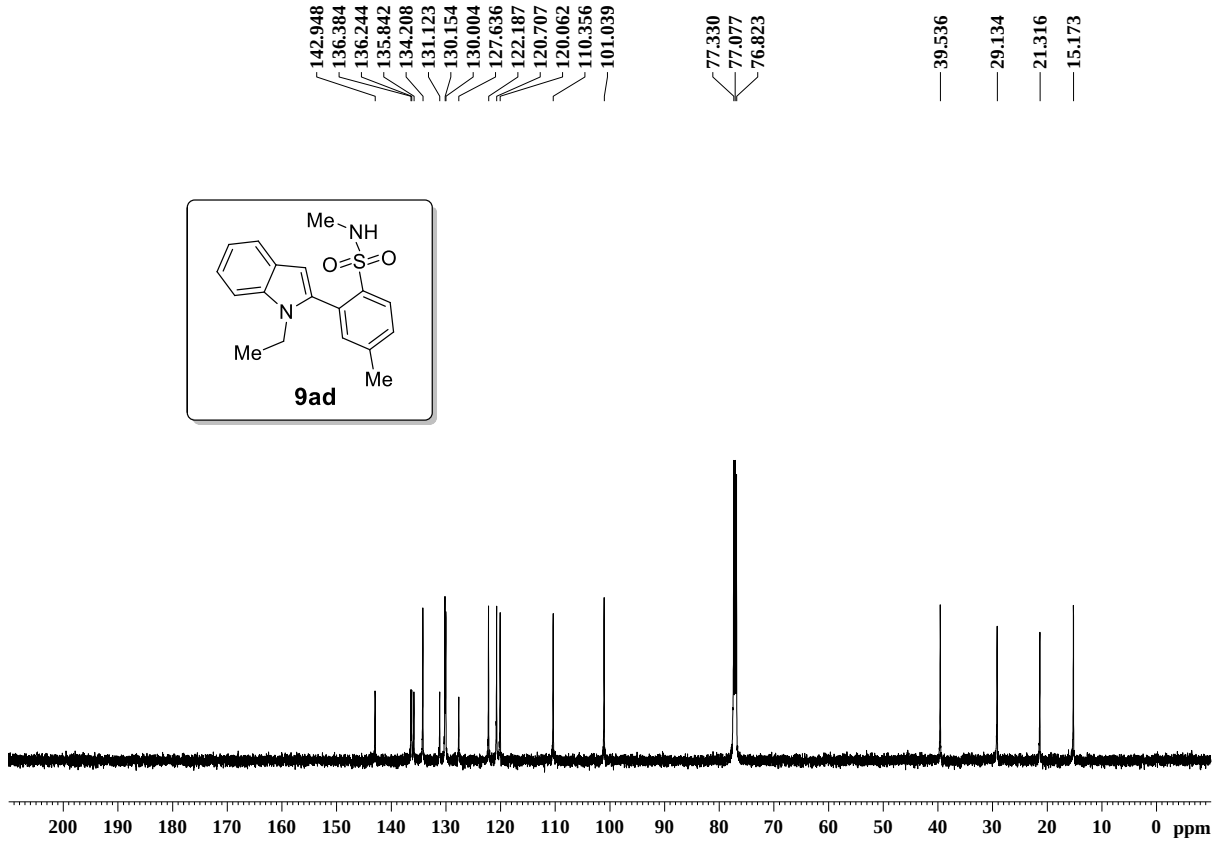


Figure S66. ^{13}C{^{1}H} NMR spectrum of compound **9ad**

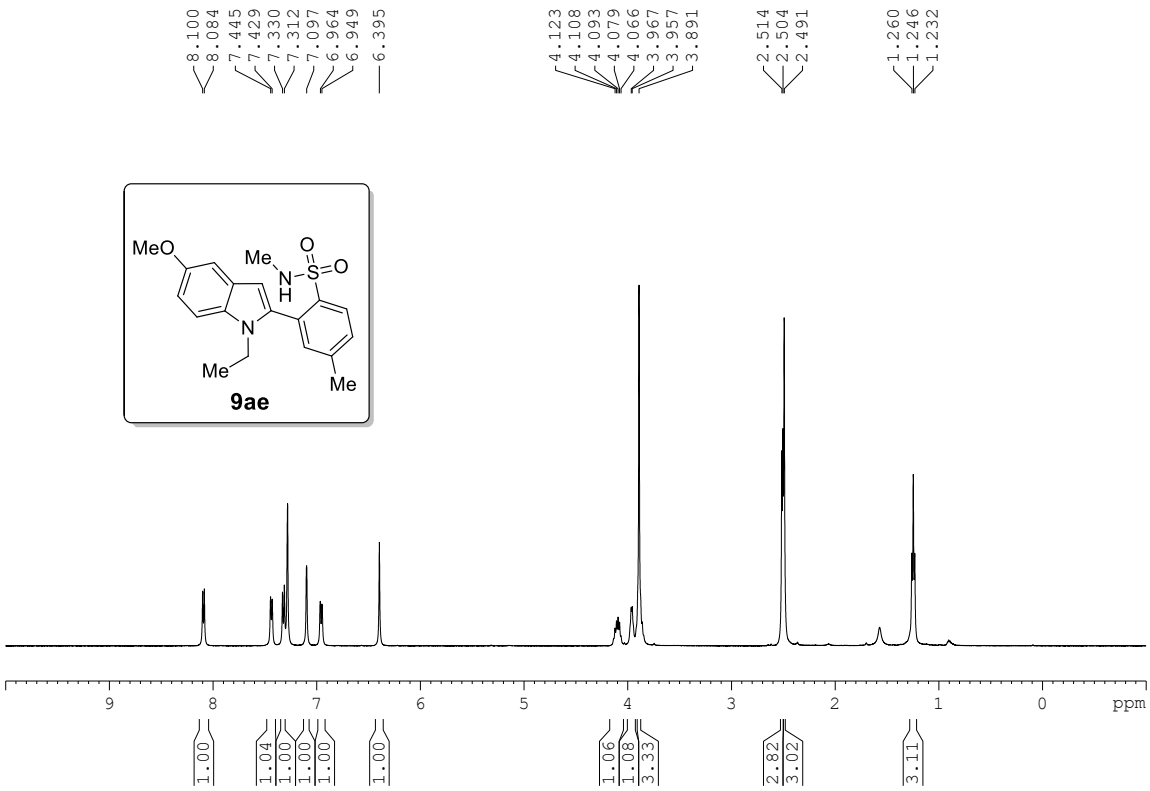


Figure S67. ^{1}H NMR spectrum of compound **9ae**

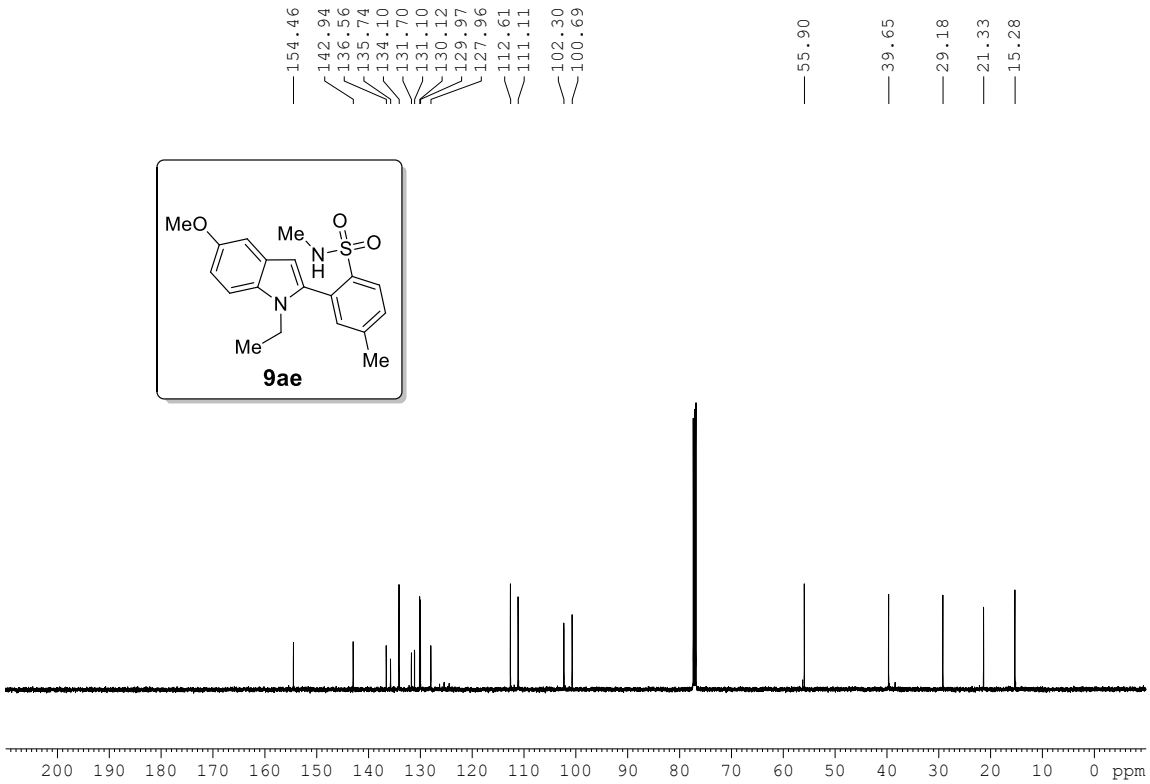


Figure S68. ^{13}C{^{1}H} NMR spectrum of compound **9ae**

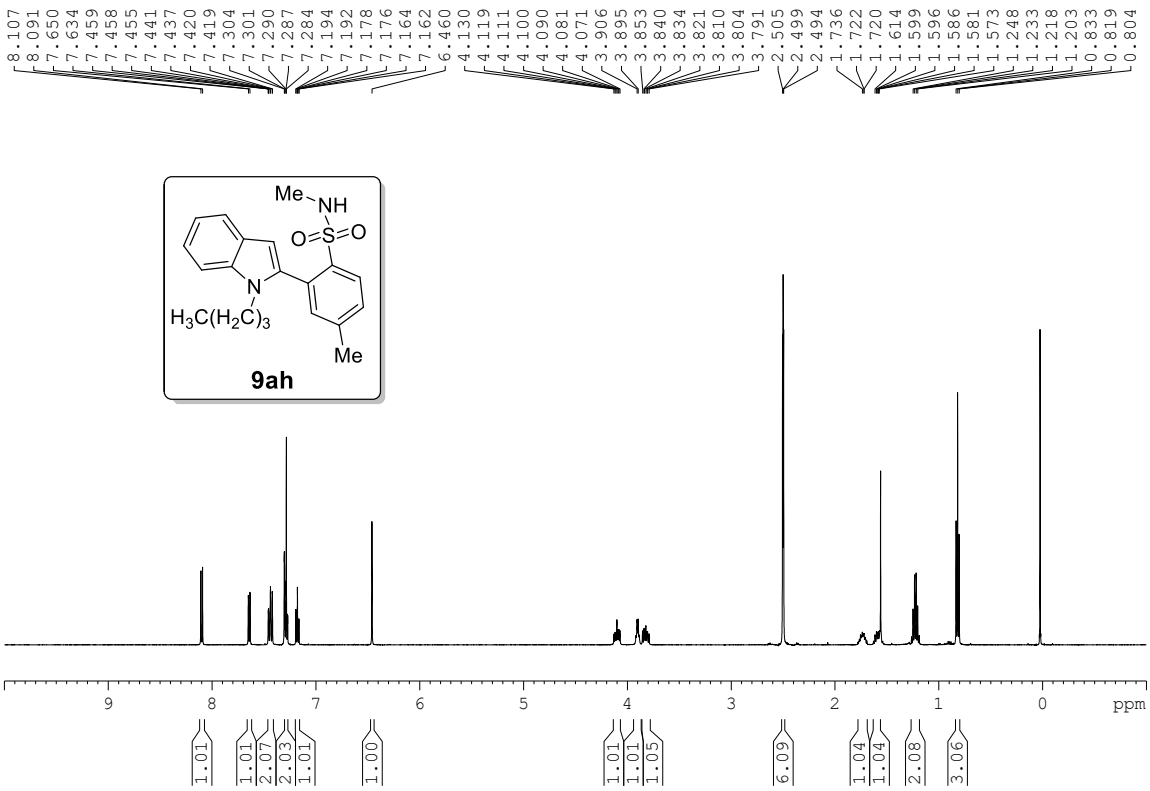


Figure S69. ^{1}H NMR spectrum of compound **9ah**

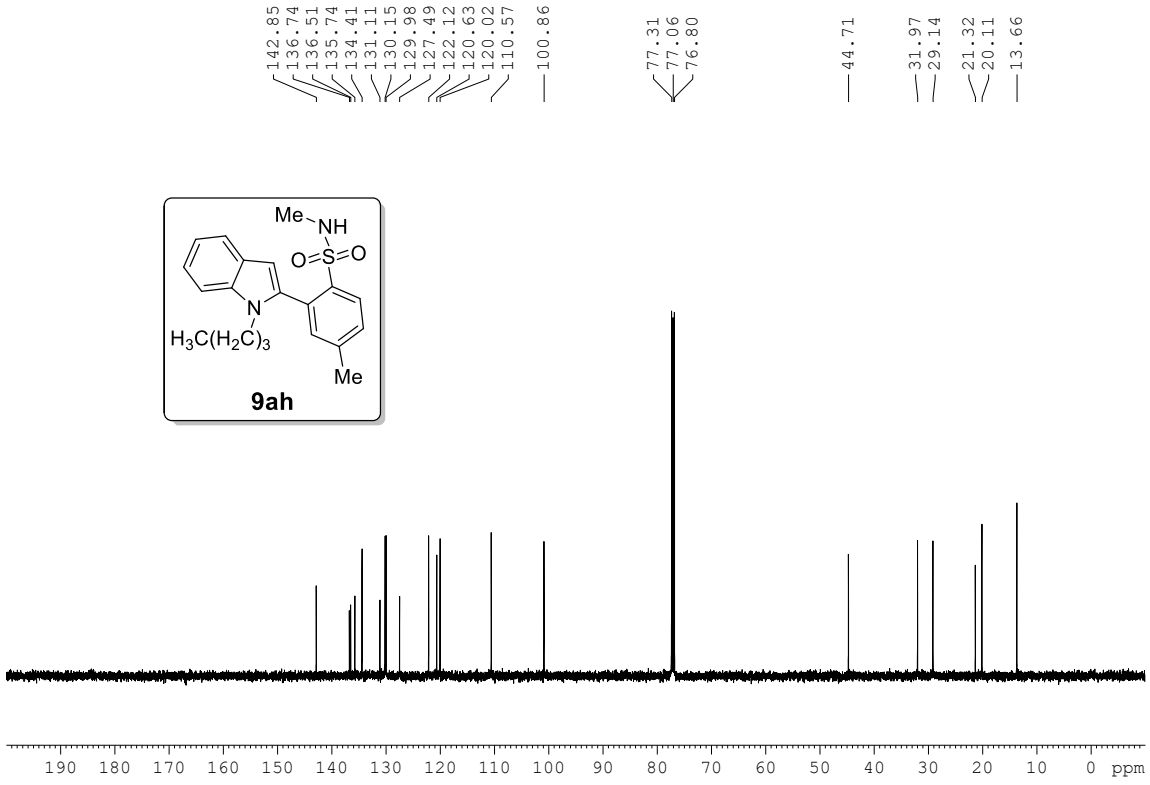


Figure S70. ^{13}C{^{1}H} NMR spectrum of compound **9ah**

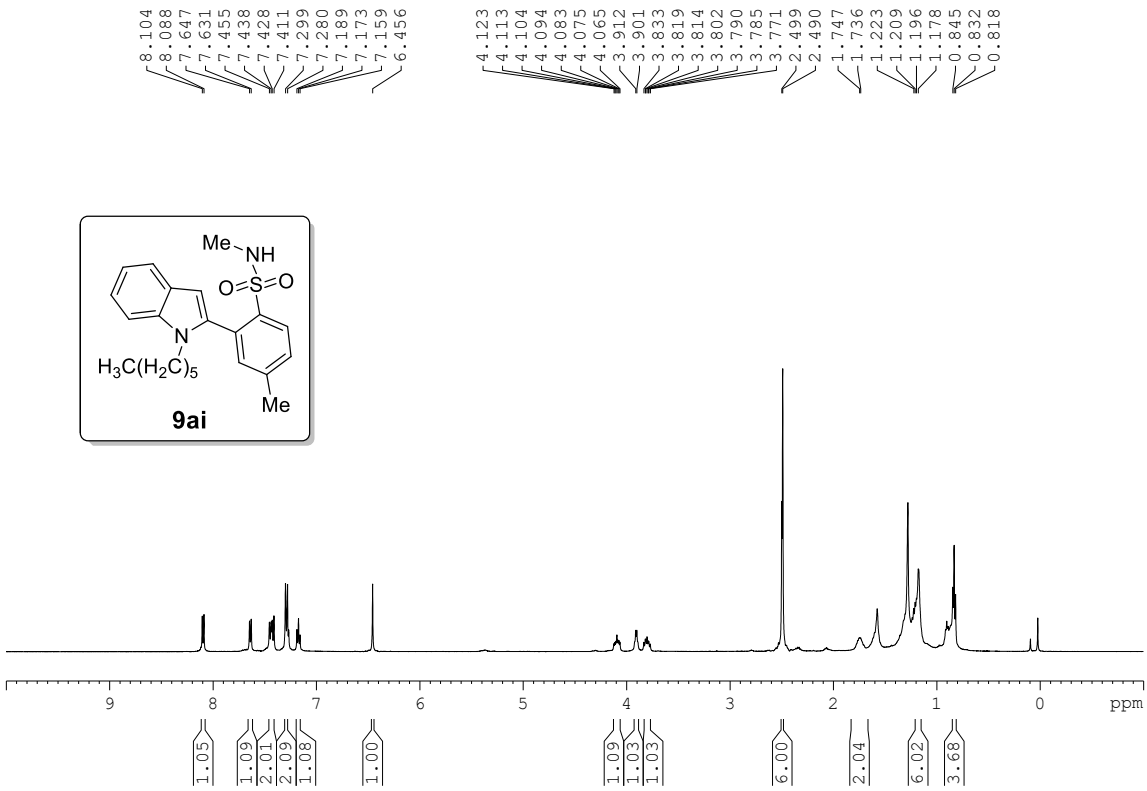


Figure S71. ^{1}H NMR spectrum of compound **9ai**

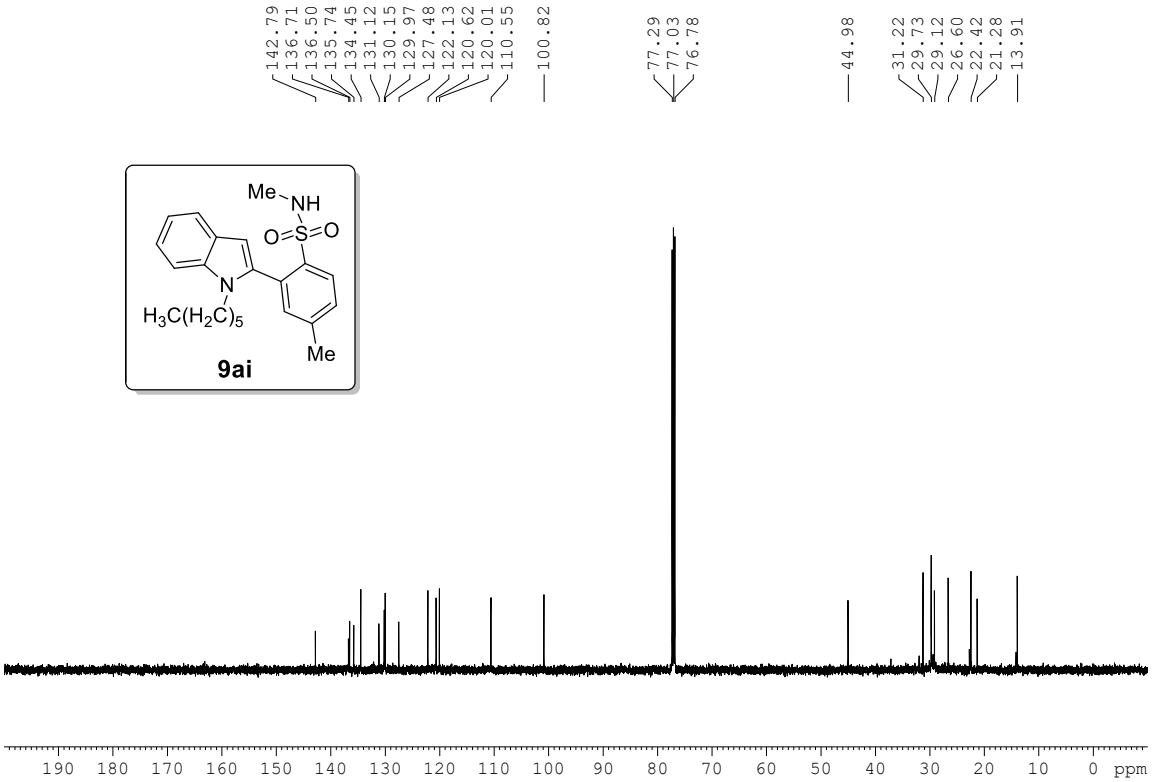


Figure S72. ^{13}C{^{1}H} NMR spectrum of compound **9ai**

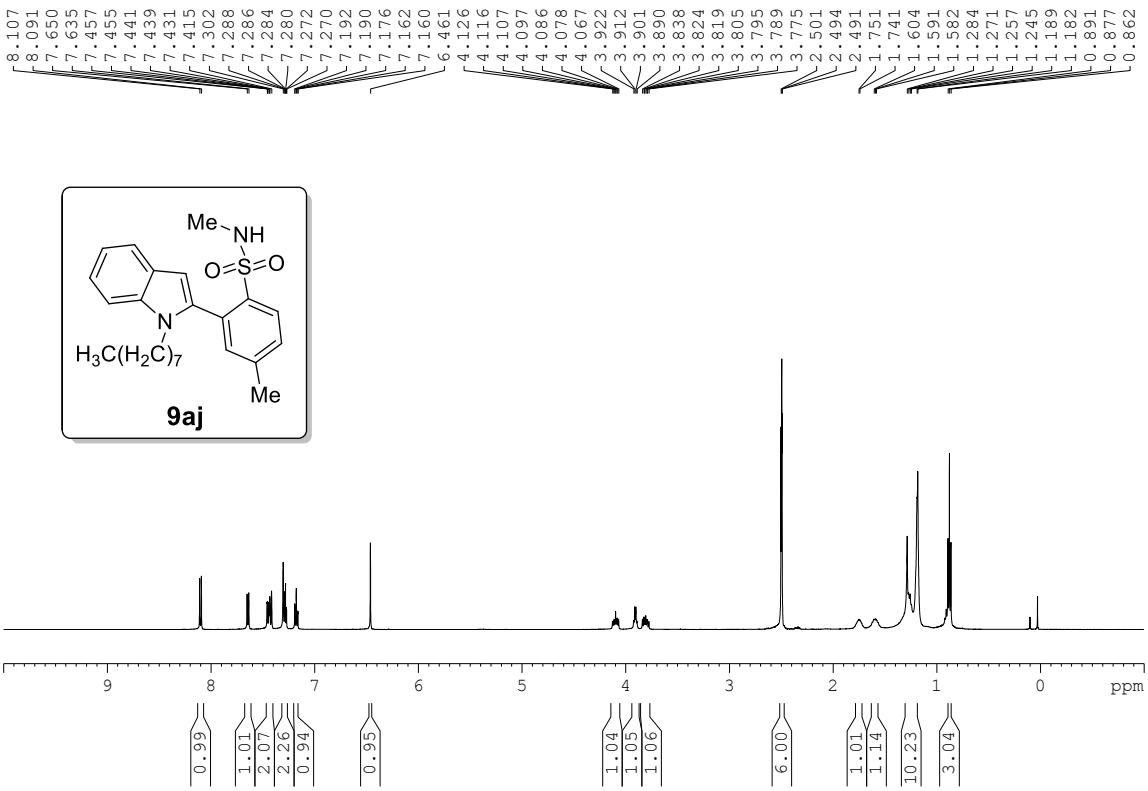


Figure S73. ^{1}H NMR spectrum of compound **9aj**

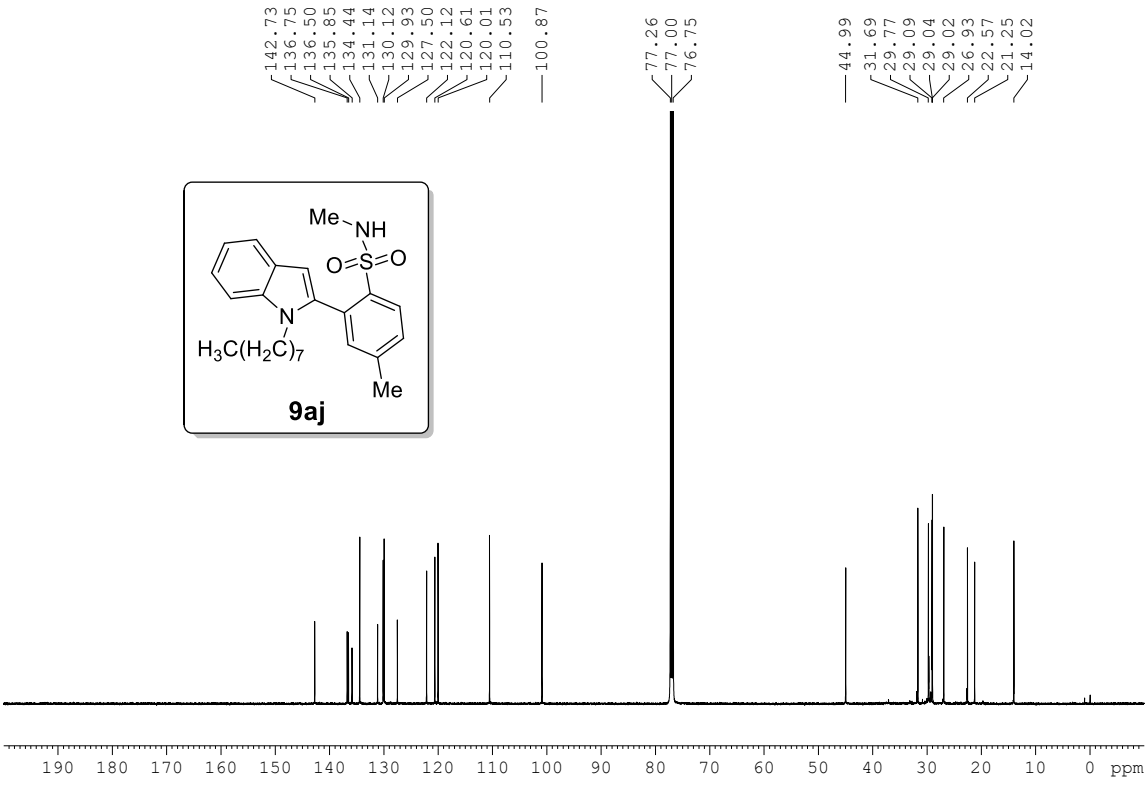


Figure S74. ^{13}C{^{1}H} NMR spectrum of compound **9aj**

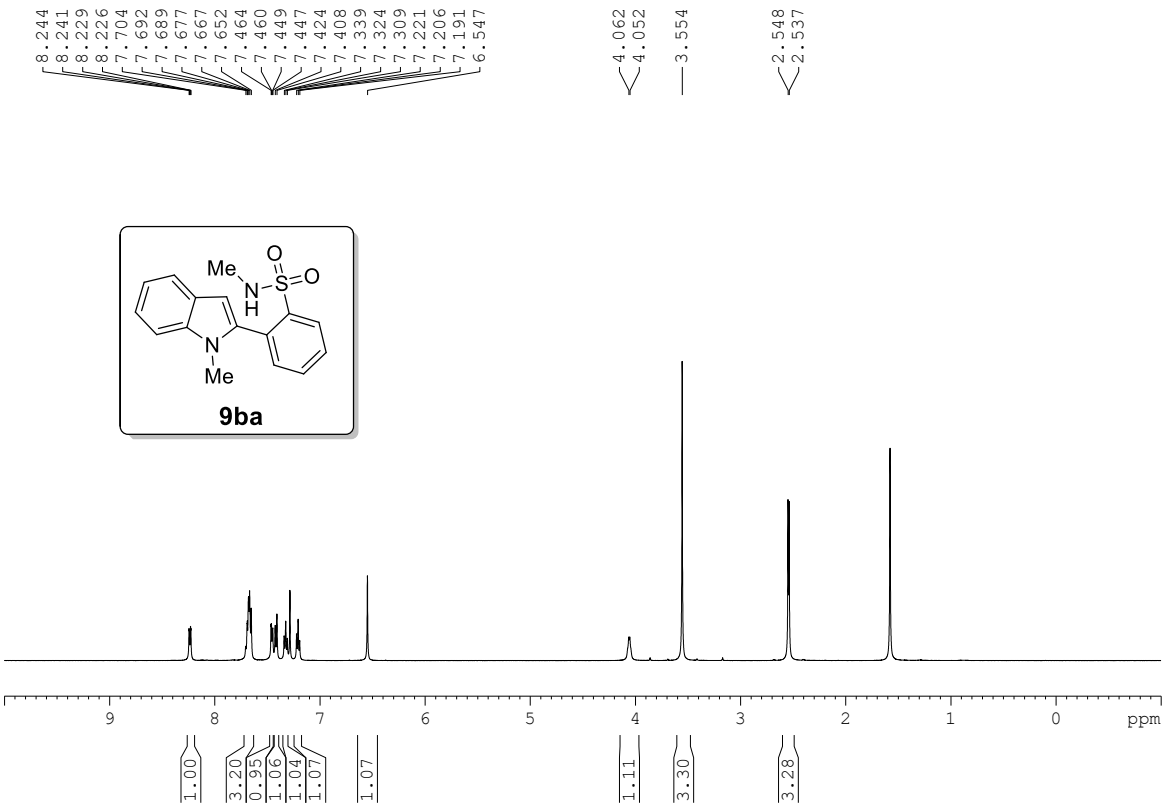


Figure S75. ^{1}H NMR spectrum of compound **9ba**

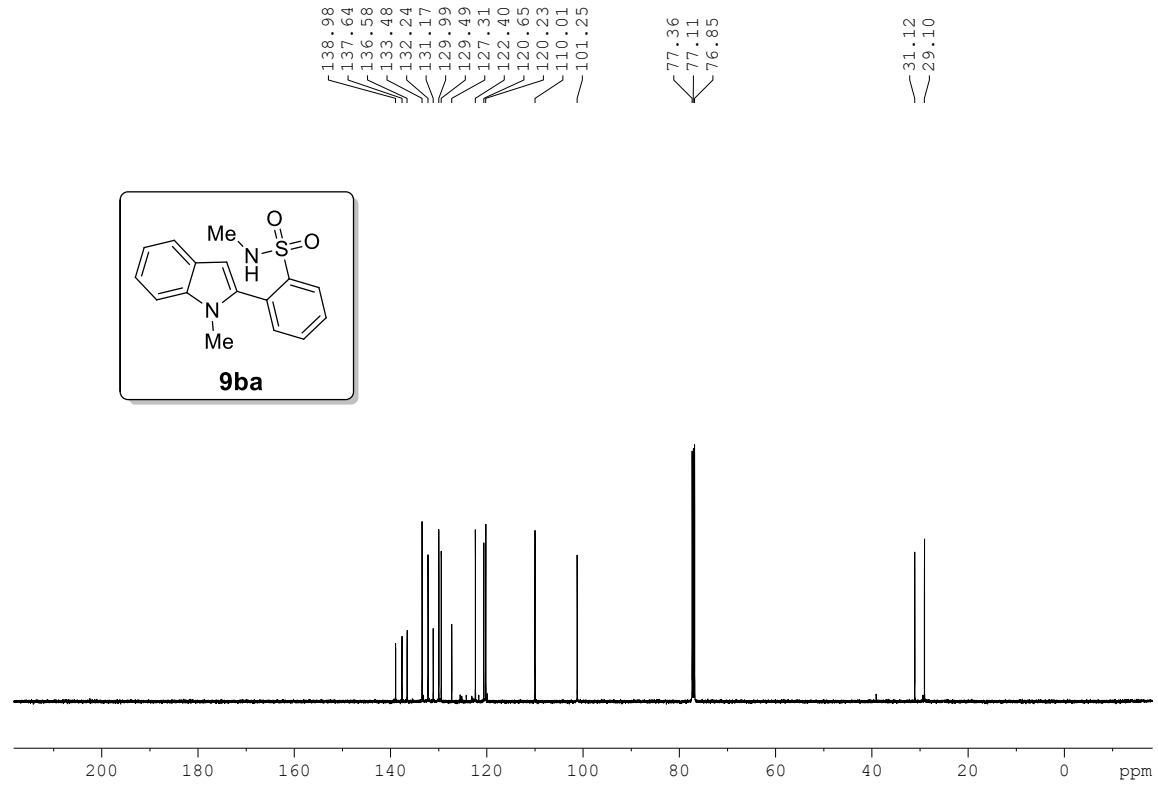


Figure S76. ^{13}C{^{1}H} NMR spectrum of compound **9ba**

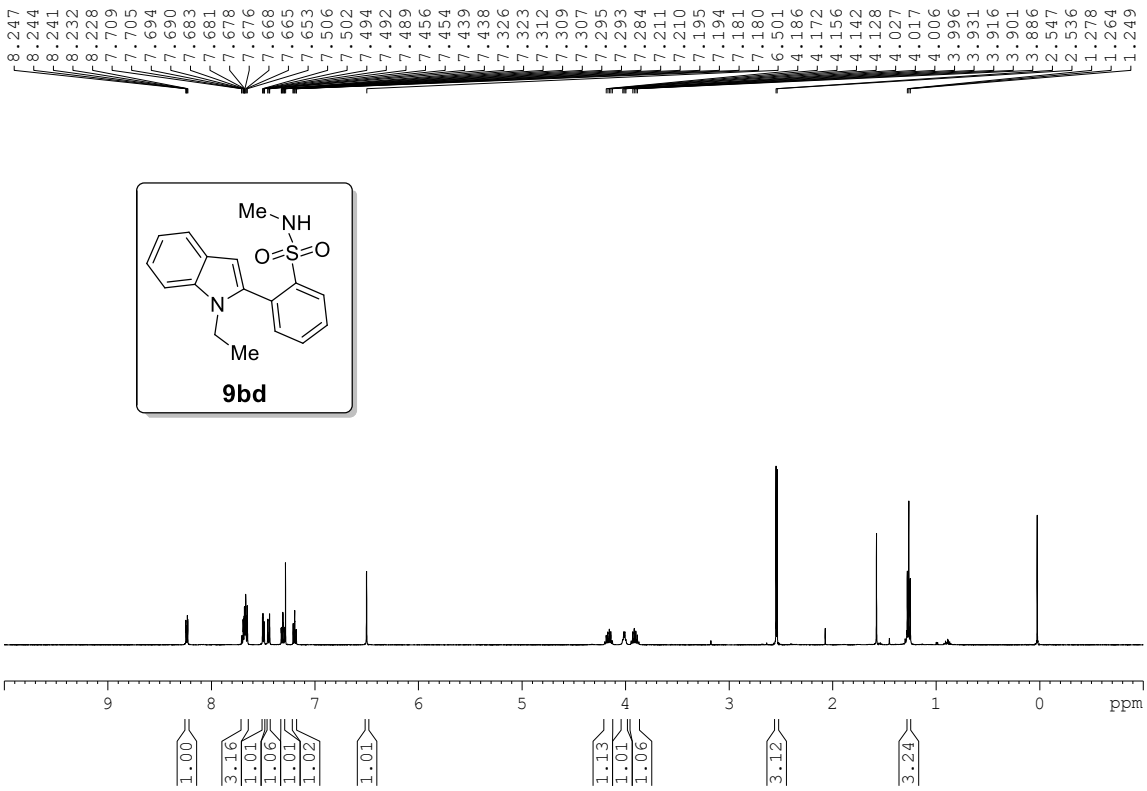


Figure S77. ^{1}H NMR spectrum of compound **9bd**

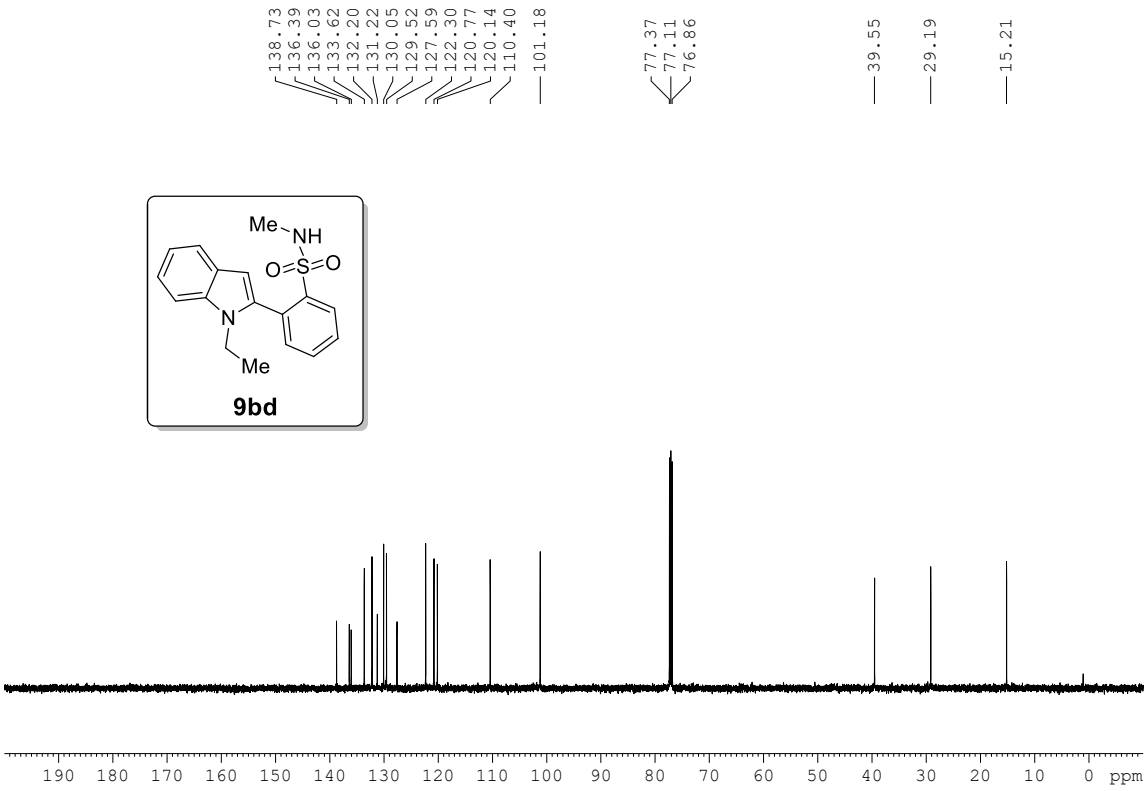


Figure S78. ^{13}C{^{1}H} NMR spectrum of compound **9bd**

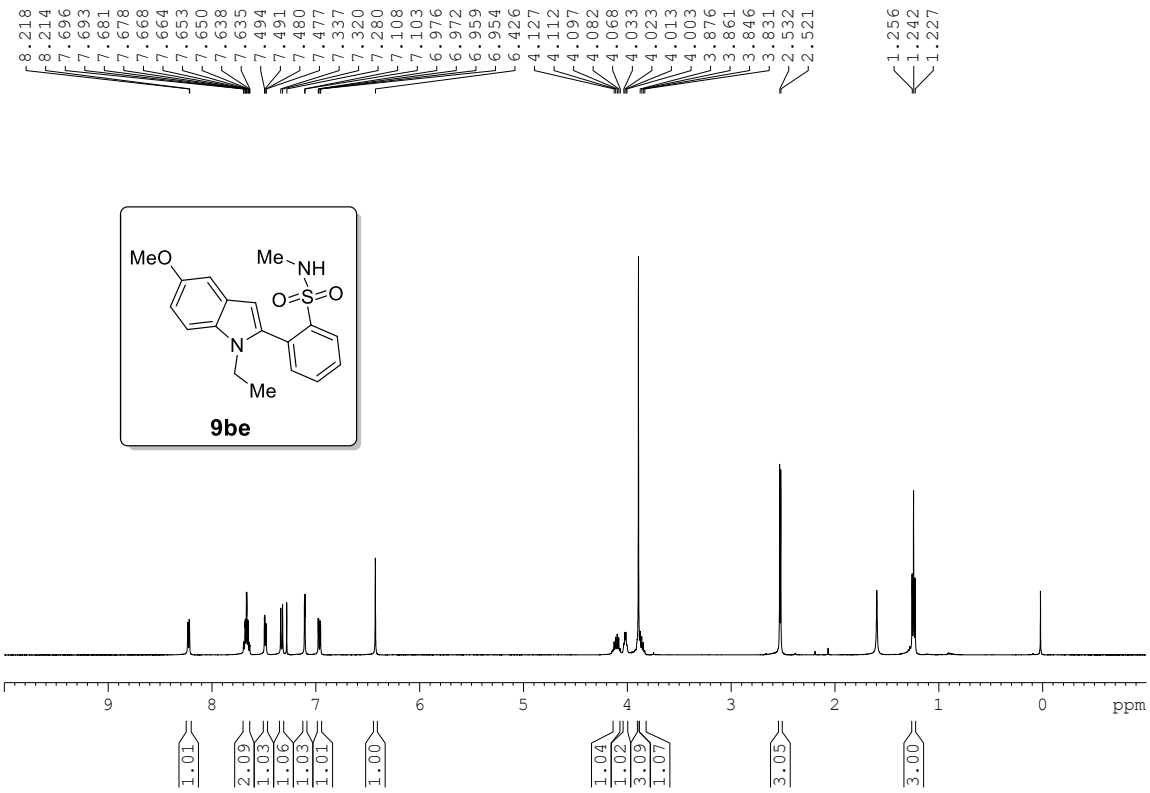


Figure S79. ^{1}H NMR spectrum of compound **9be**

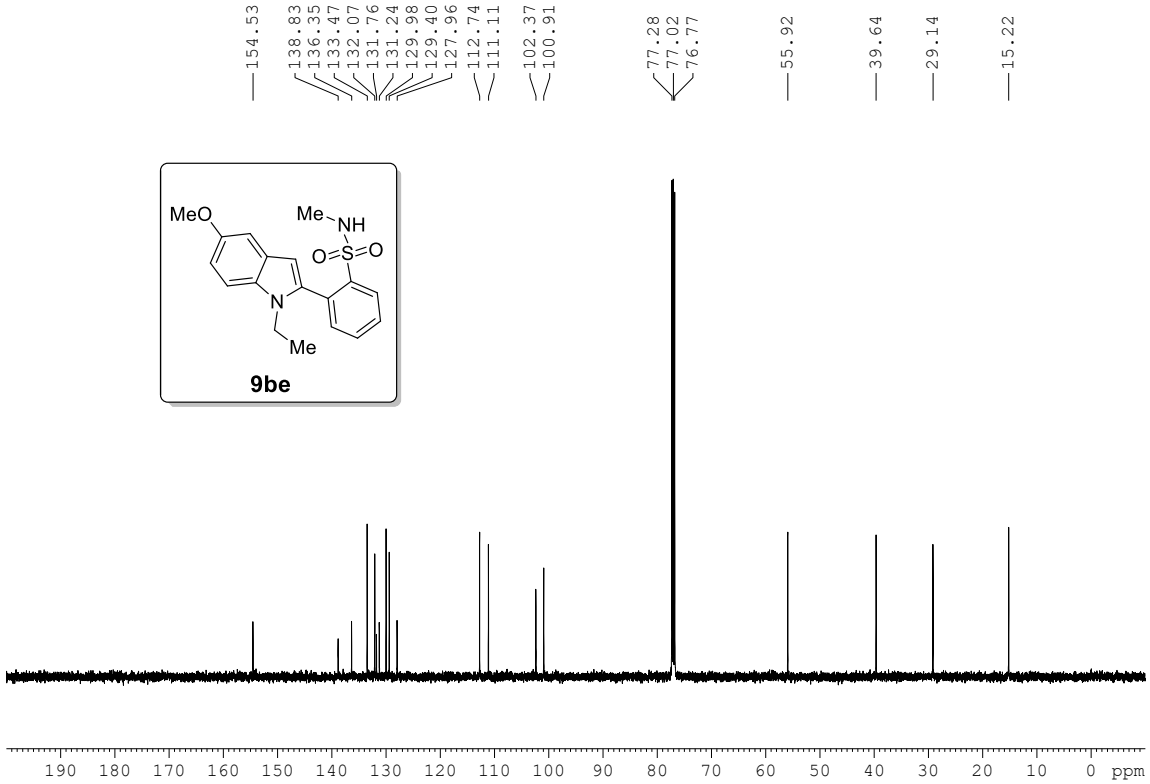


Figure S80. ^{13}C{^{1}H} NMR spectrum of compound **9be**

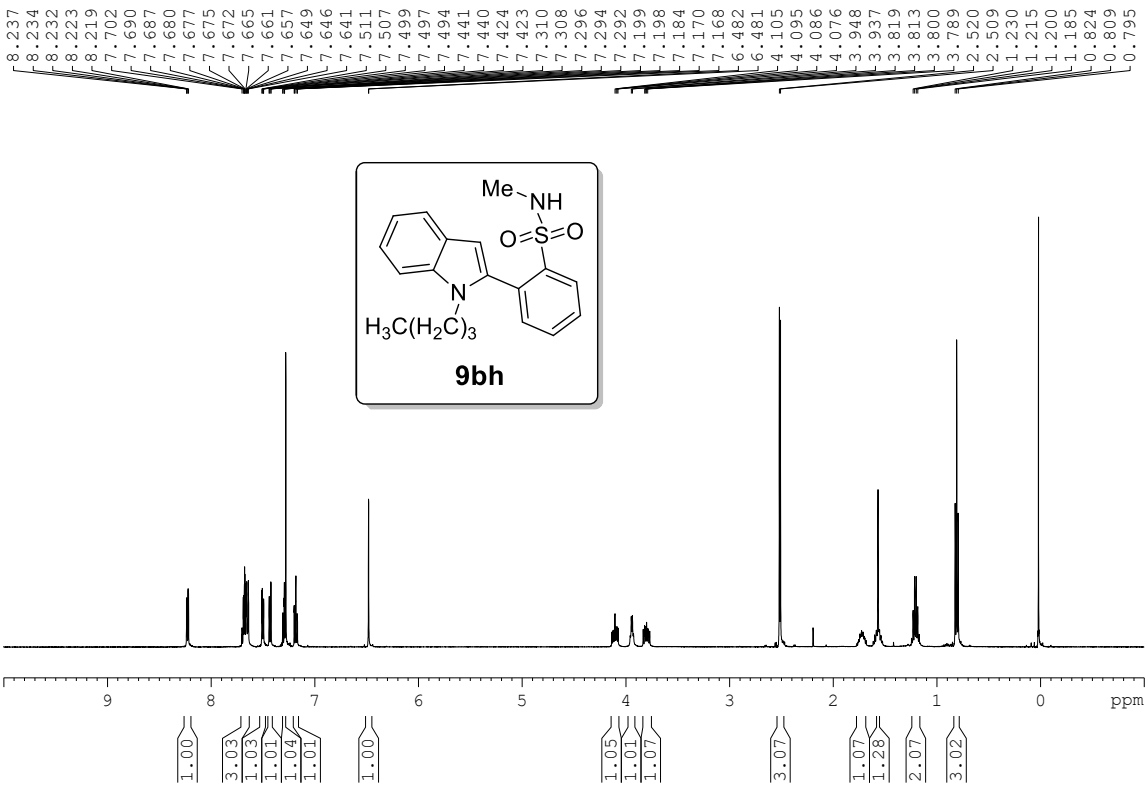


Figure S81. ^{1}H NMR spectrum of compound **9bh**

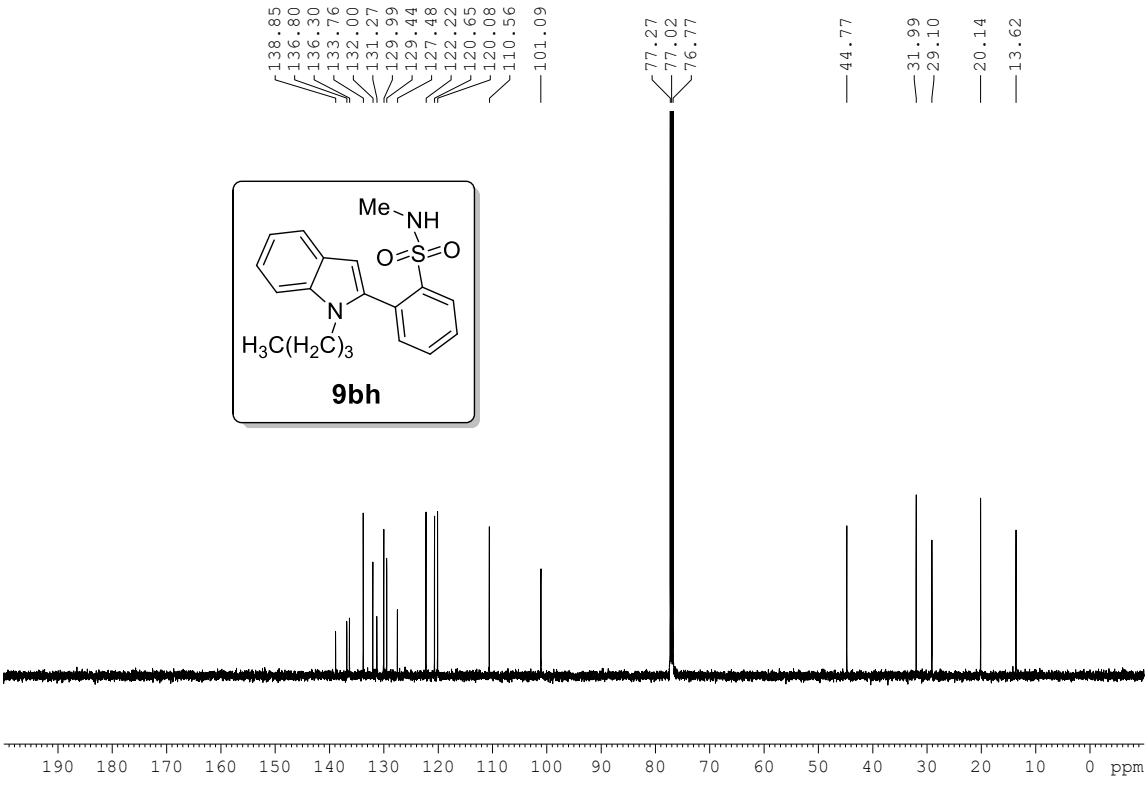


Figure S82. ^{13}C{^{1}H} NMR spectrum of compound **9bh**

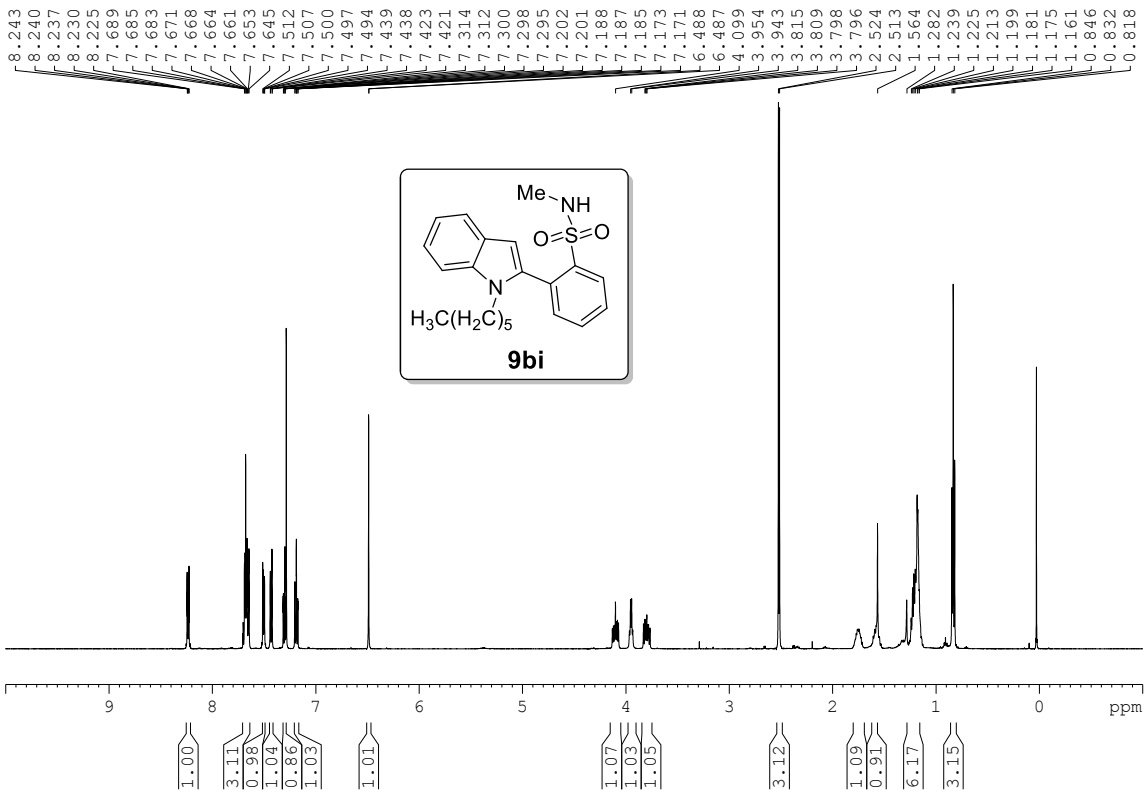


Figure S83. ^{1}H NMR spectrum of compound **9bi**

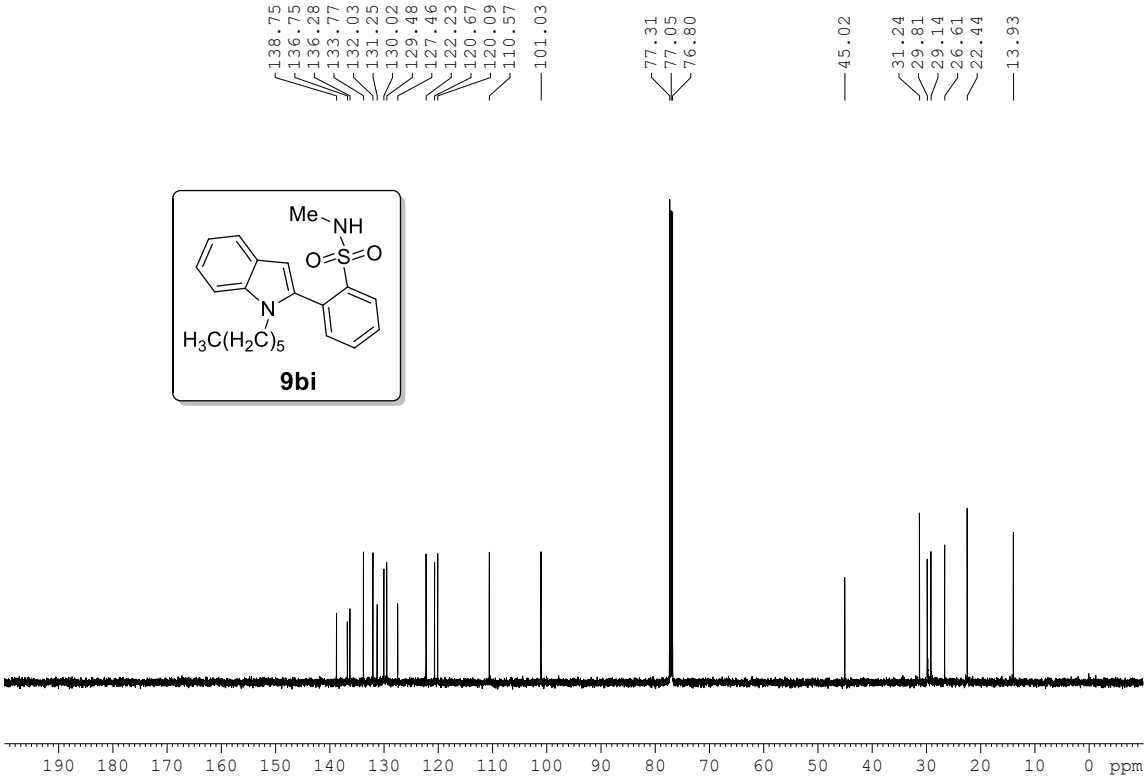


Figure S84. ^{13}C{^{1}H} NMR spectrum of compound **9bi**

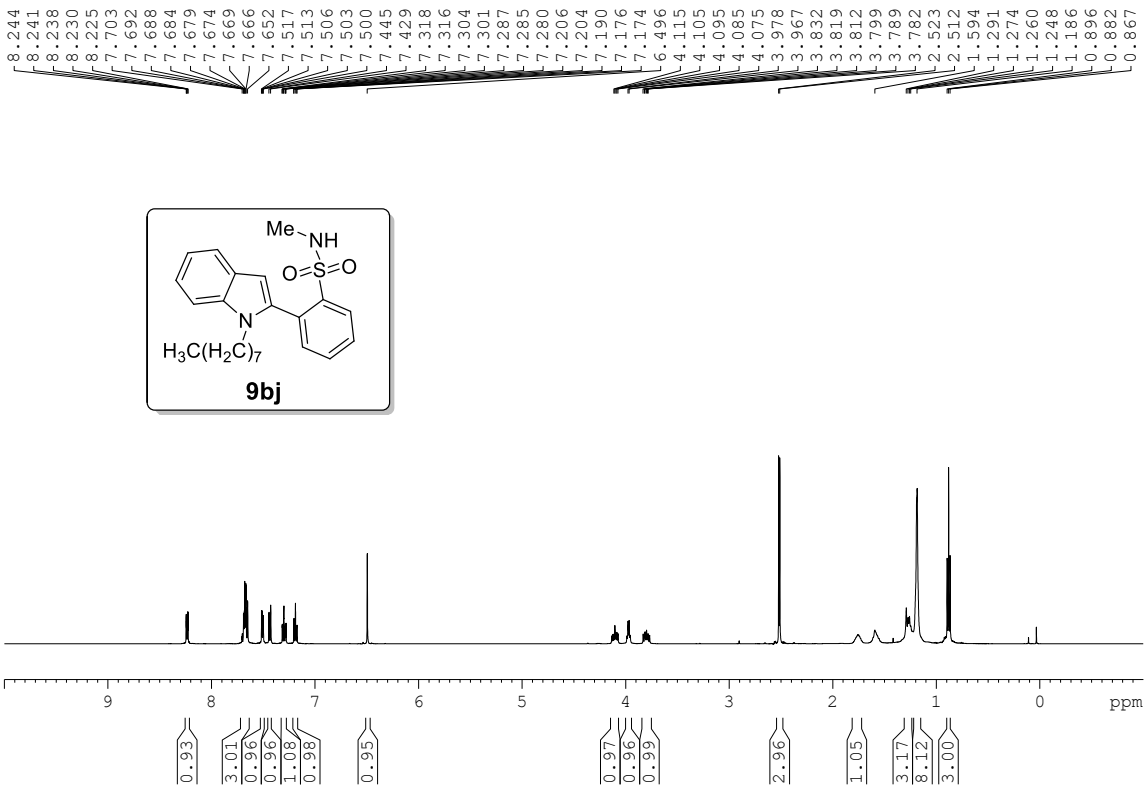


Figure S85. ^{1}H NMR spectrum of compound **9bj**

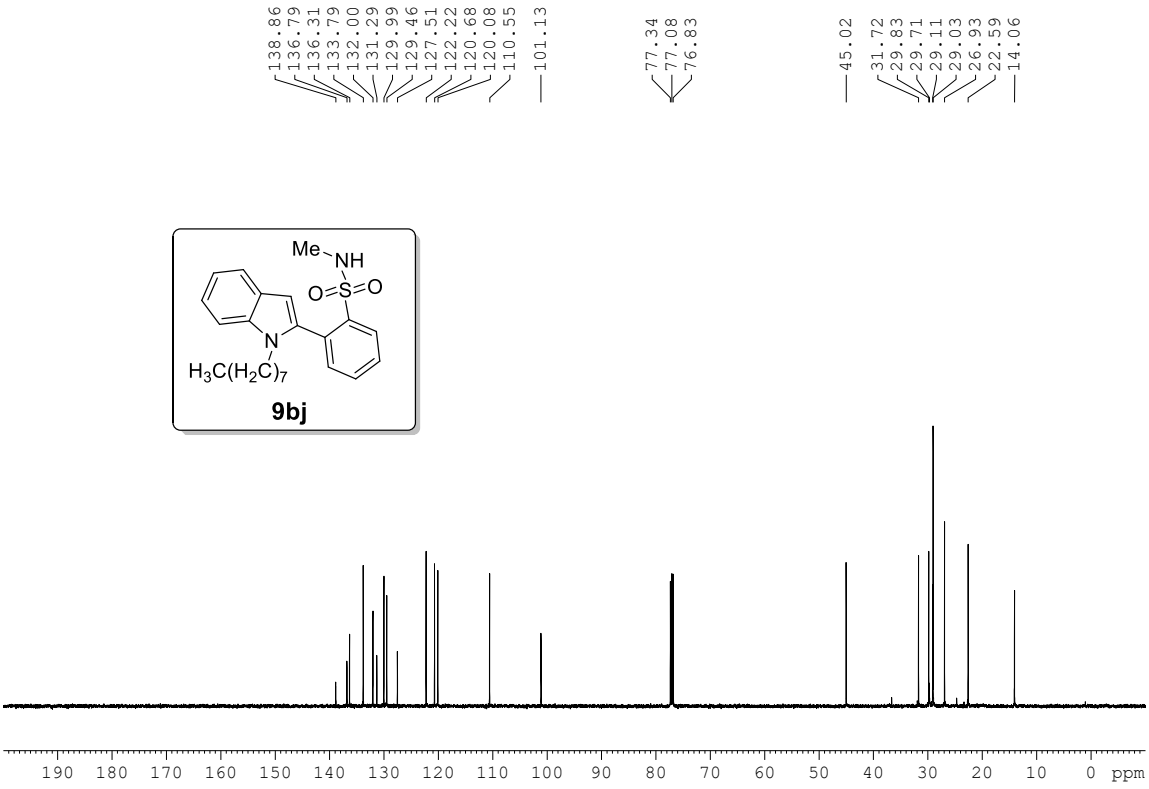


Figure S86. ^{13}C{^{1}H} NMR spectrum of compound **9bj**

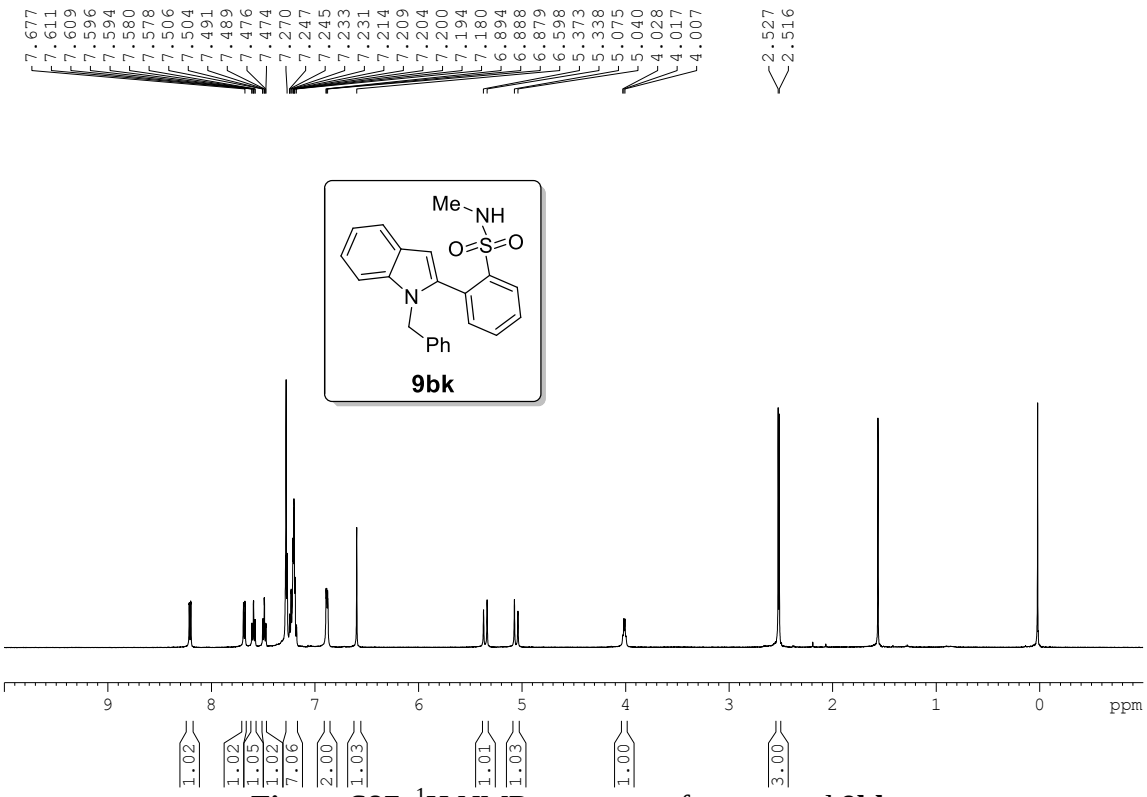


Figure S87. ^{1}H NMR spectrum of compound **9bk**

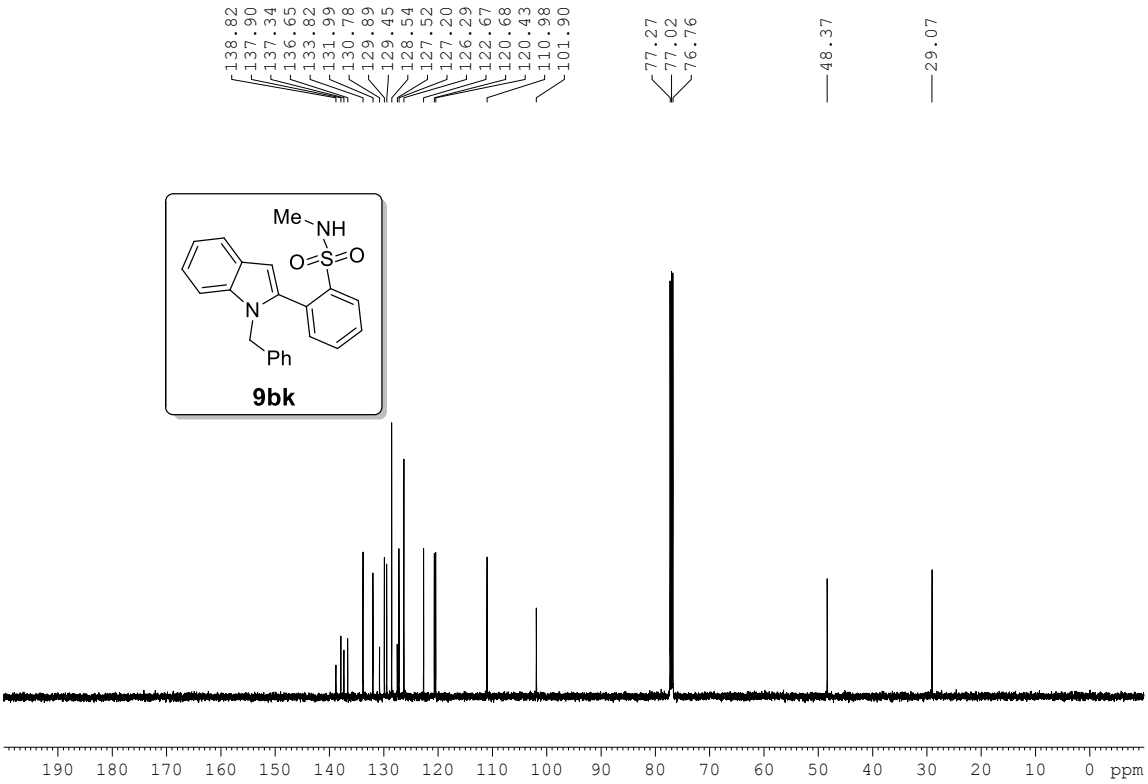


Figure S88. ^{13}C{^{1}H} NMR spectrum of compound **9bk**

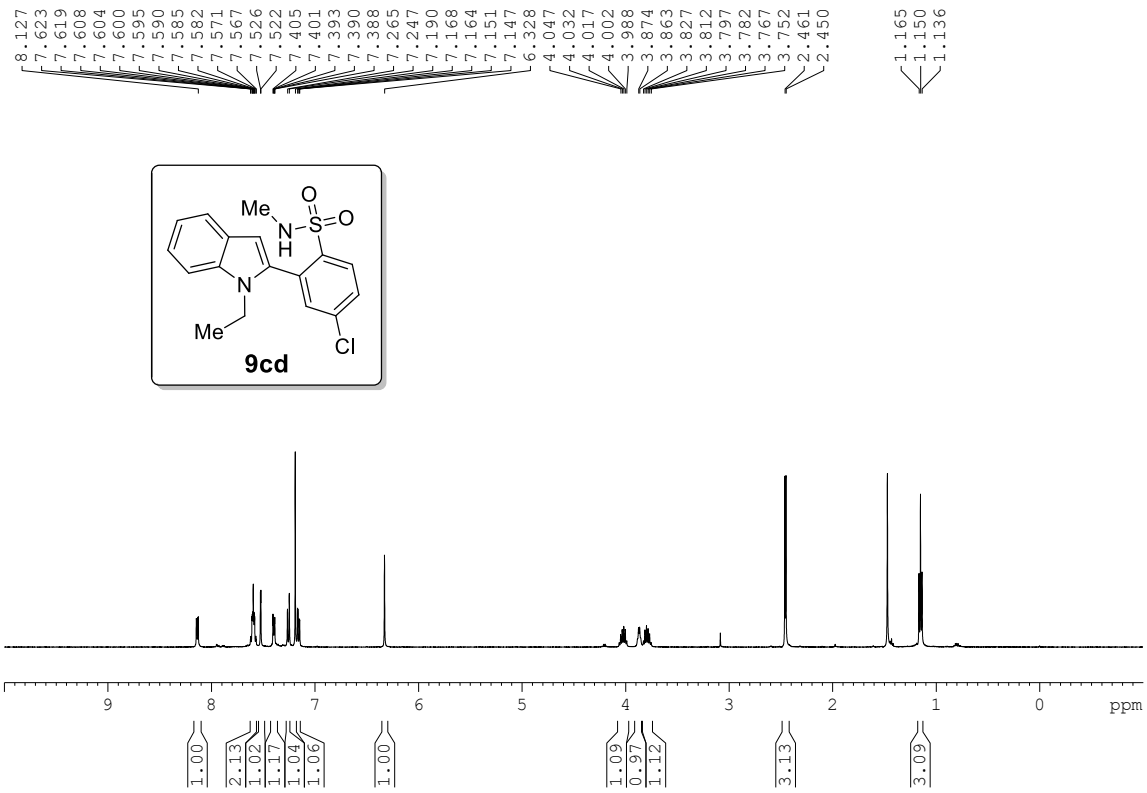


Figure S89. ^{1}H NMR spectrum of compound **9cd**

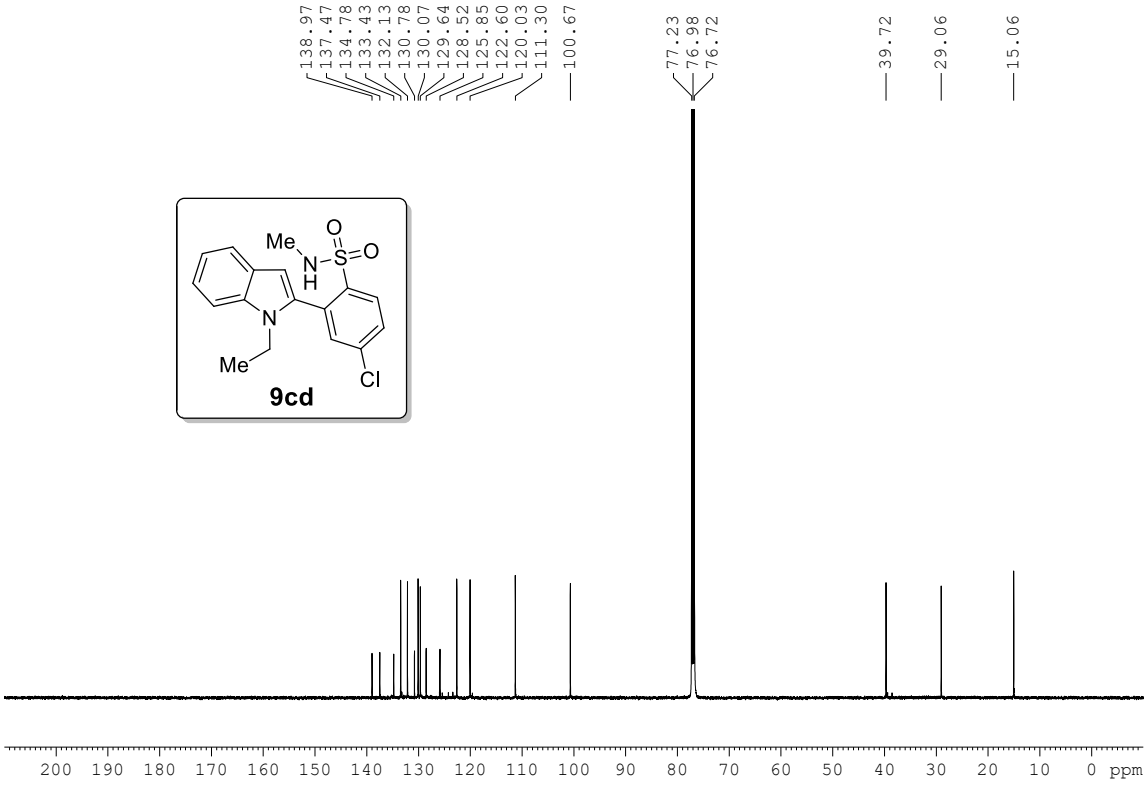


Figure S90. ^{13}C{^{1}H} NMR spectrum of compound **9cd**

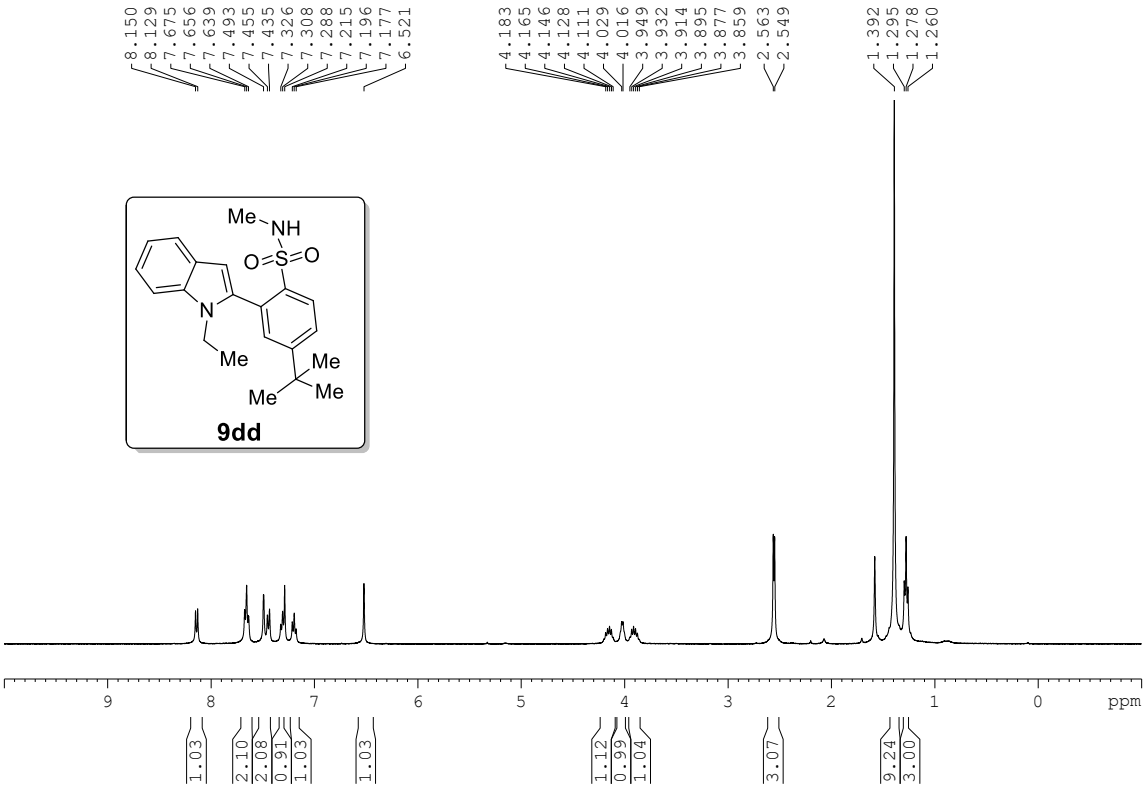


Figure S91. ^{1}H NMR spectrum of compound **9dd**

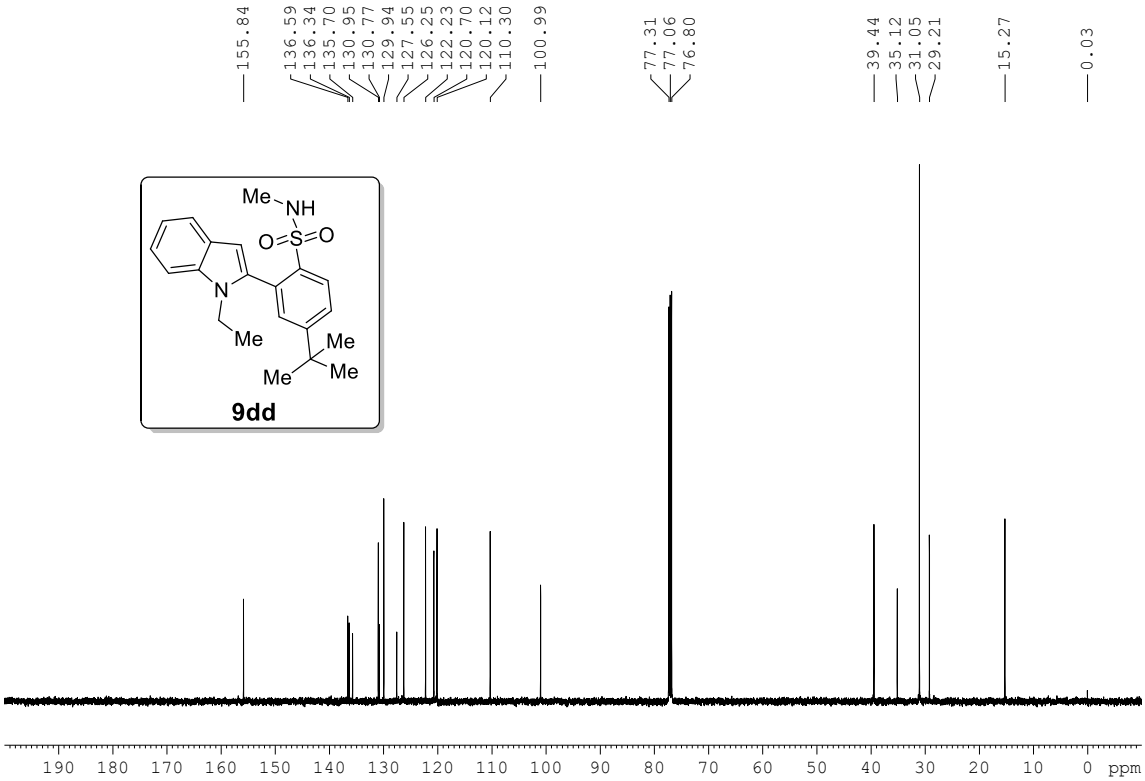


Figure S92. ^{13}C{^{1}H} NMR spectrum of compound **9dd**

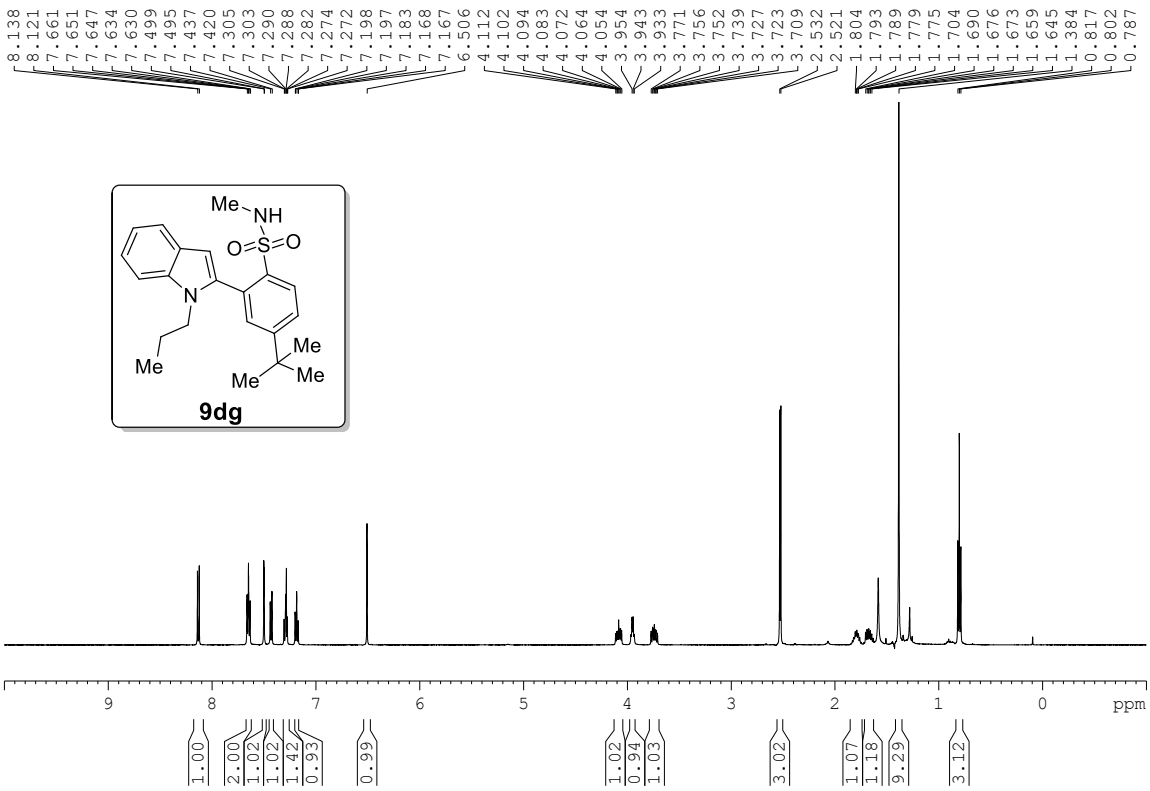


Figure S93. ^{1}H NMR spectrum of compound **9dg**

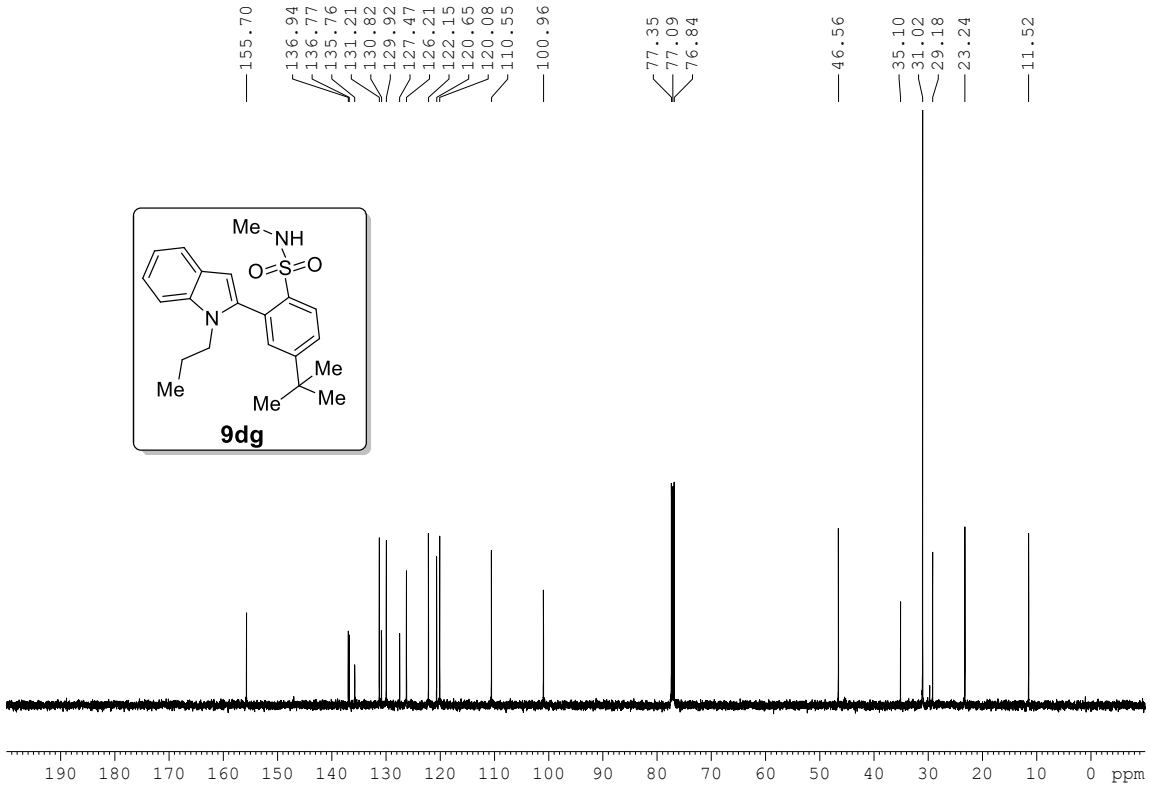


Figure S94. ^{13}C{^{1}H} NMR spectrum of compound **9dg**

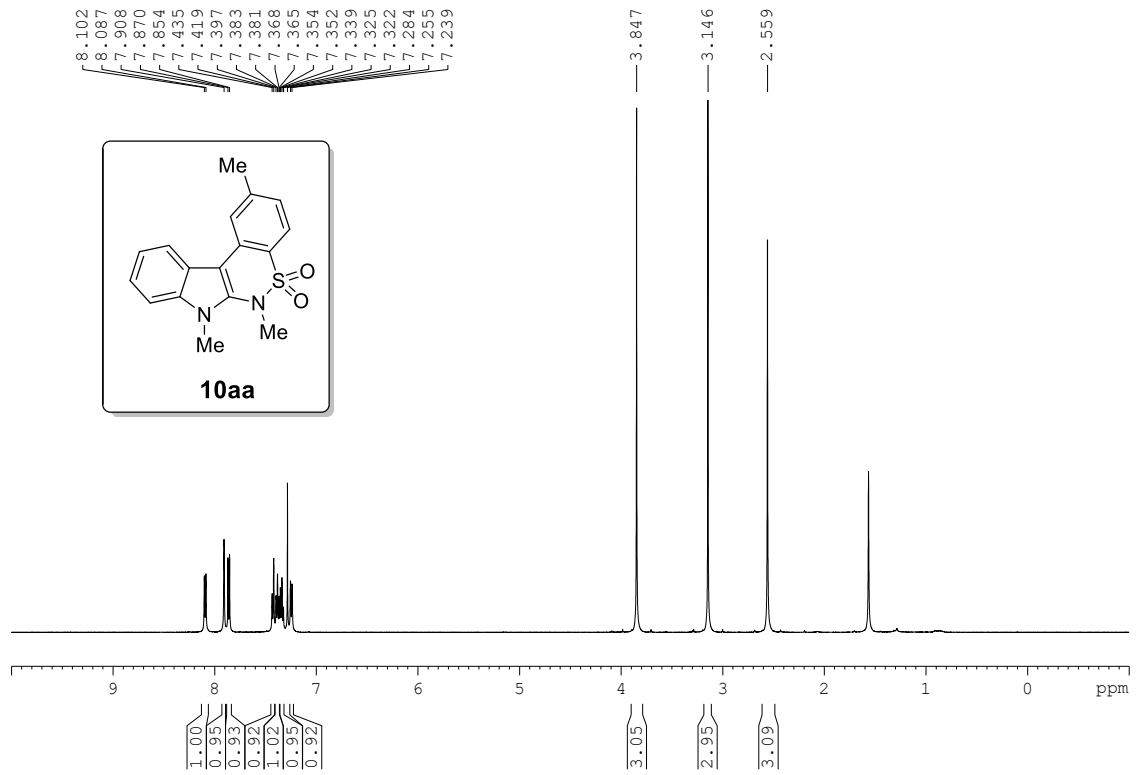


Figure S95. ^{1}H NMR spectrum of compound **10aa**

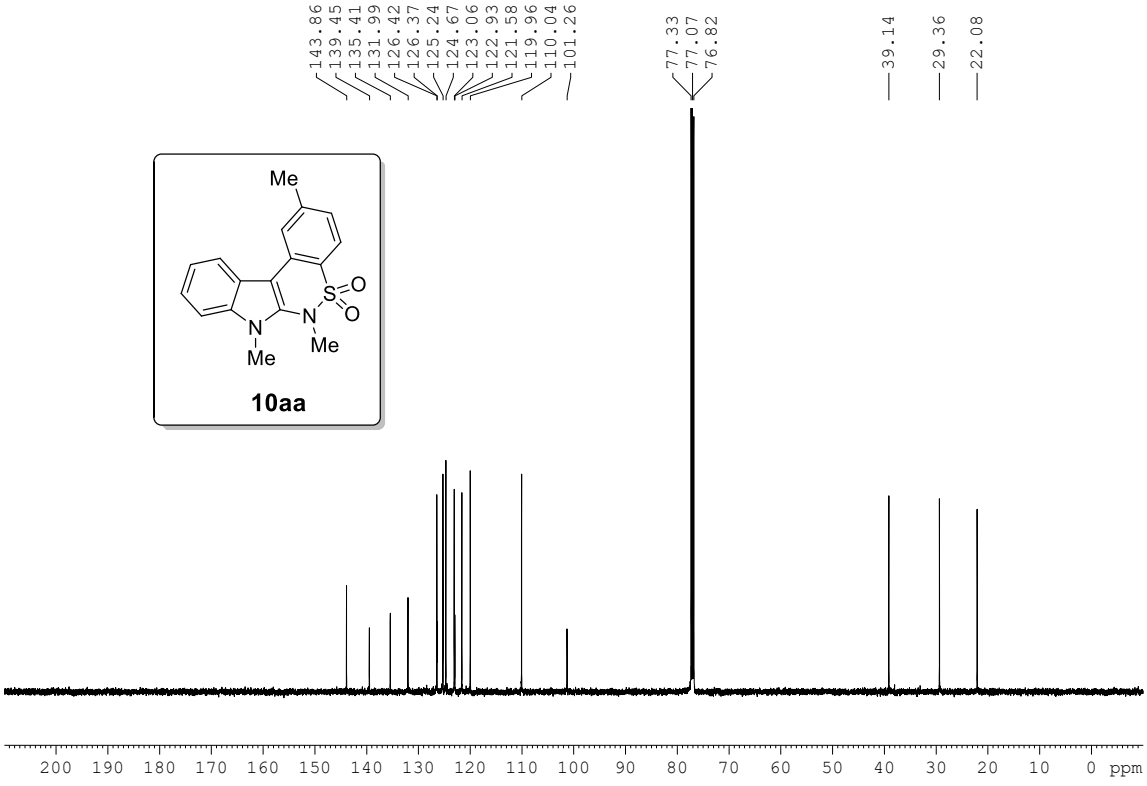


Figure S96. ^{13}C{^{1}H} NMR spectrum of compound **10aa**

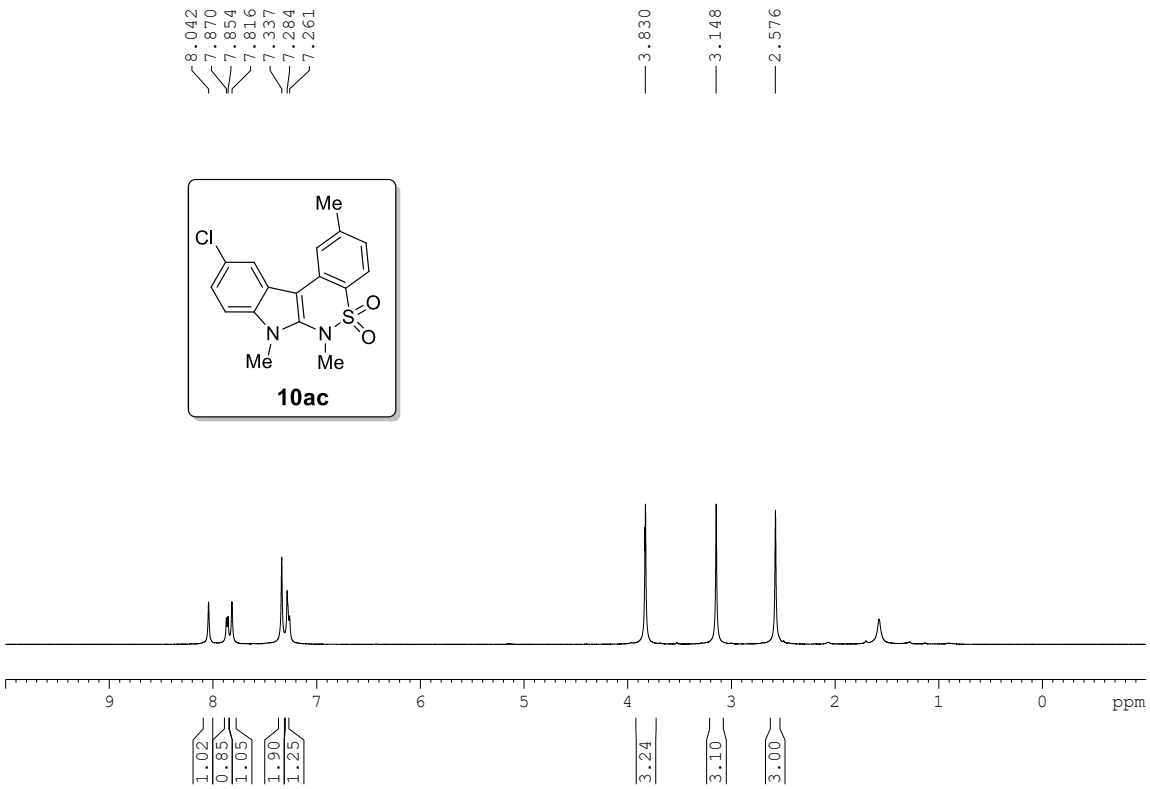


Figure S97. ^{1}H NMR spectrum of compound **10ac**

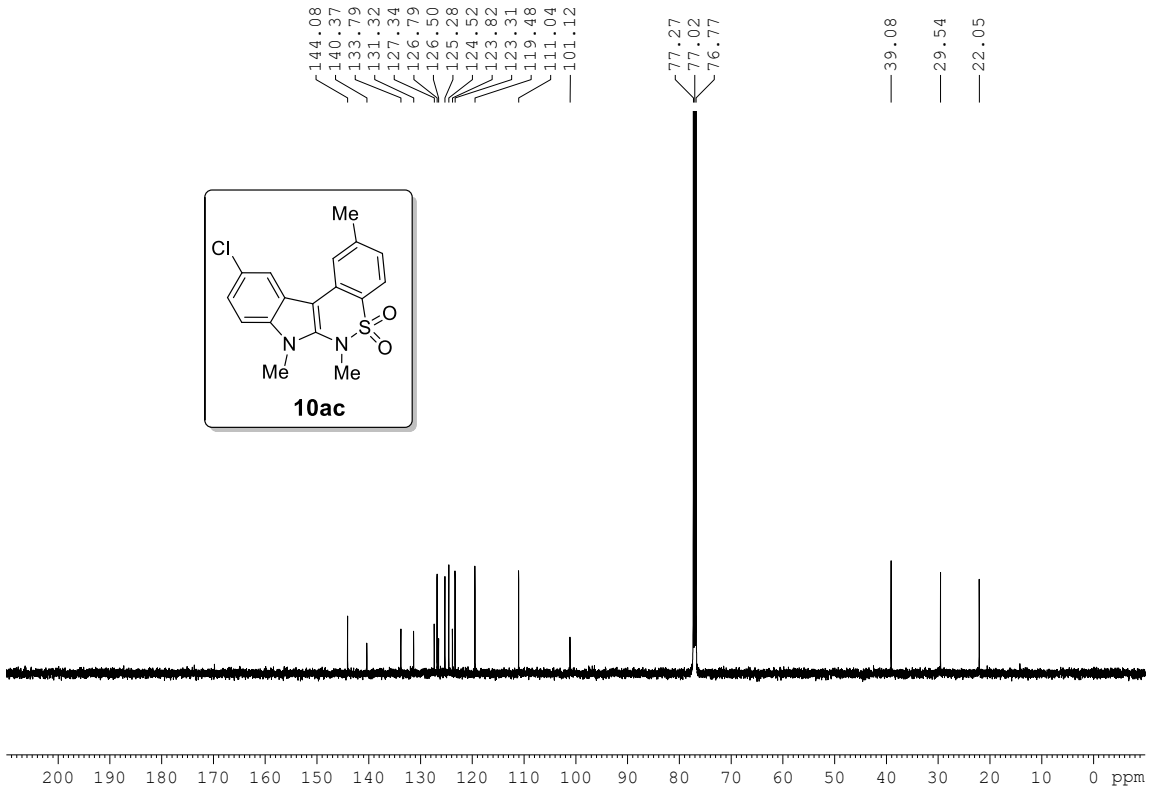


Figure S98. ^{13}C{^{1}H} NMR spectrum of compound **10ac**

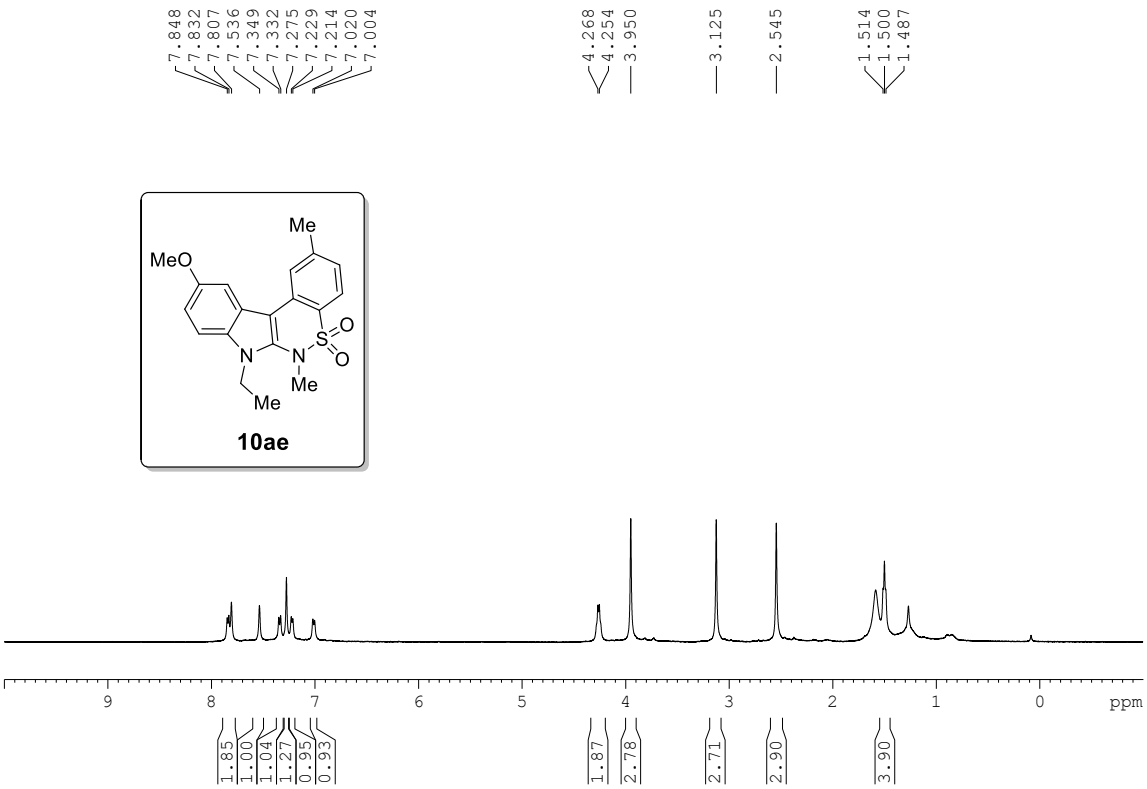


Figure S99. ^{1}H NMR spectrum of compound **10ae**

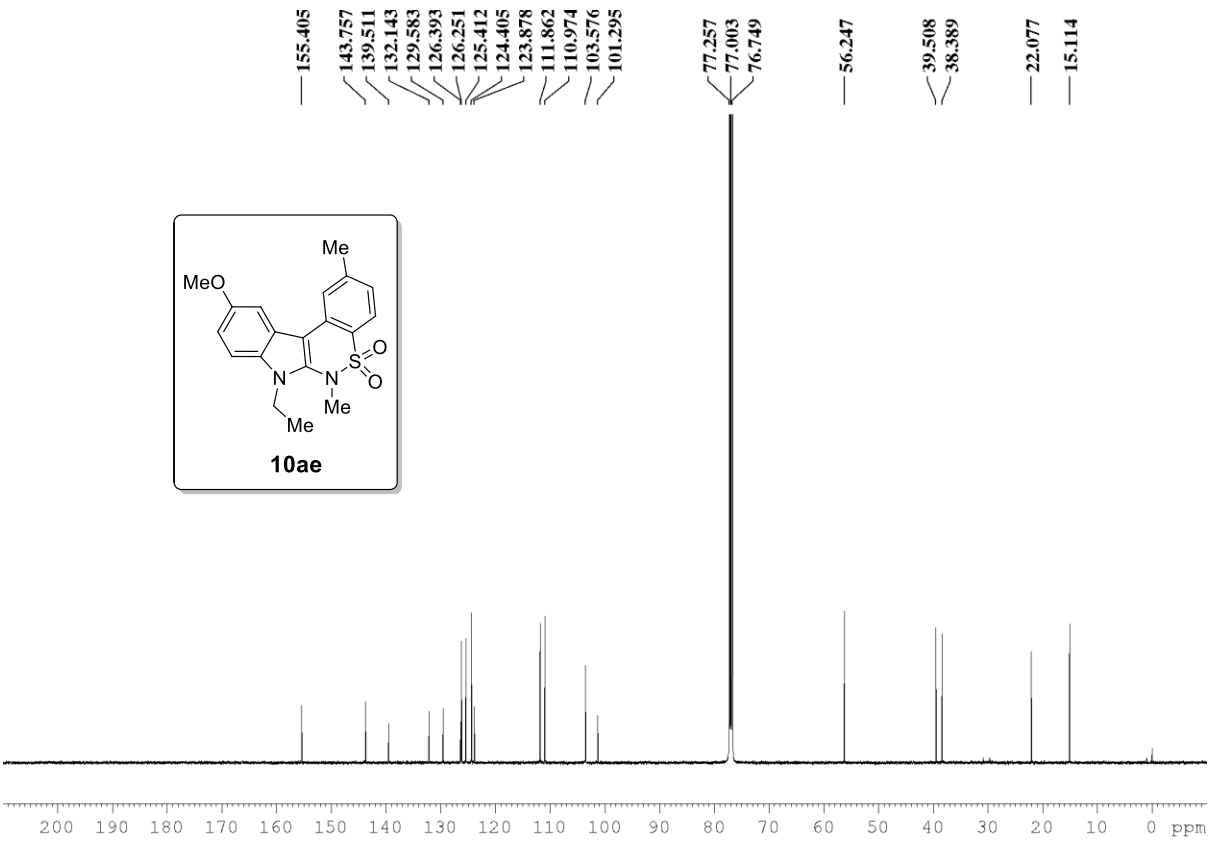


Figure S100. ^{13}C{^{1}H} NMR spectrum of compound **10ae**

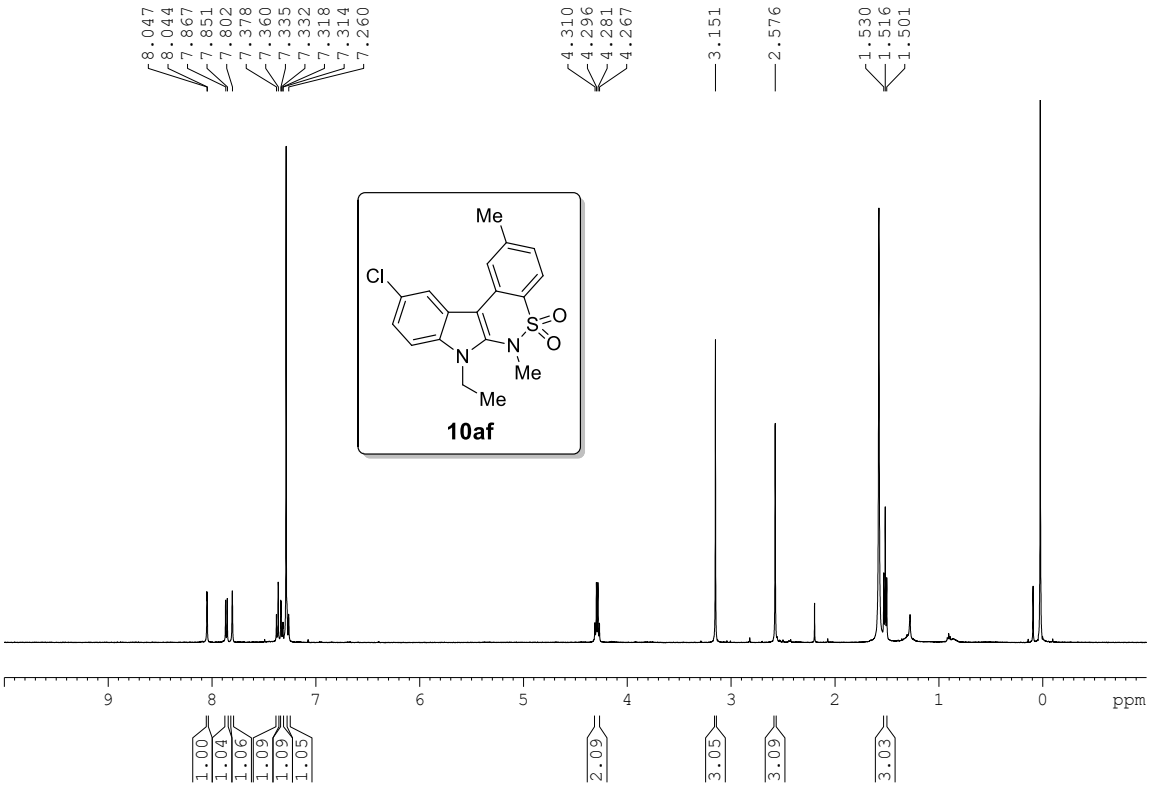


Figure S101. ^{1}H NMR spectrum of compound **10af**

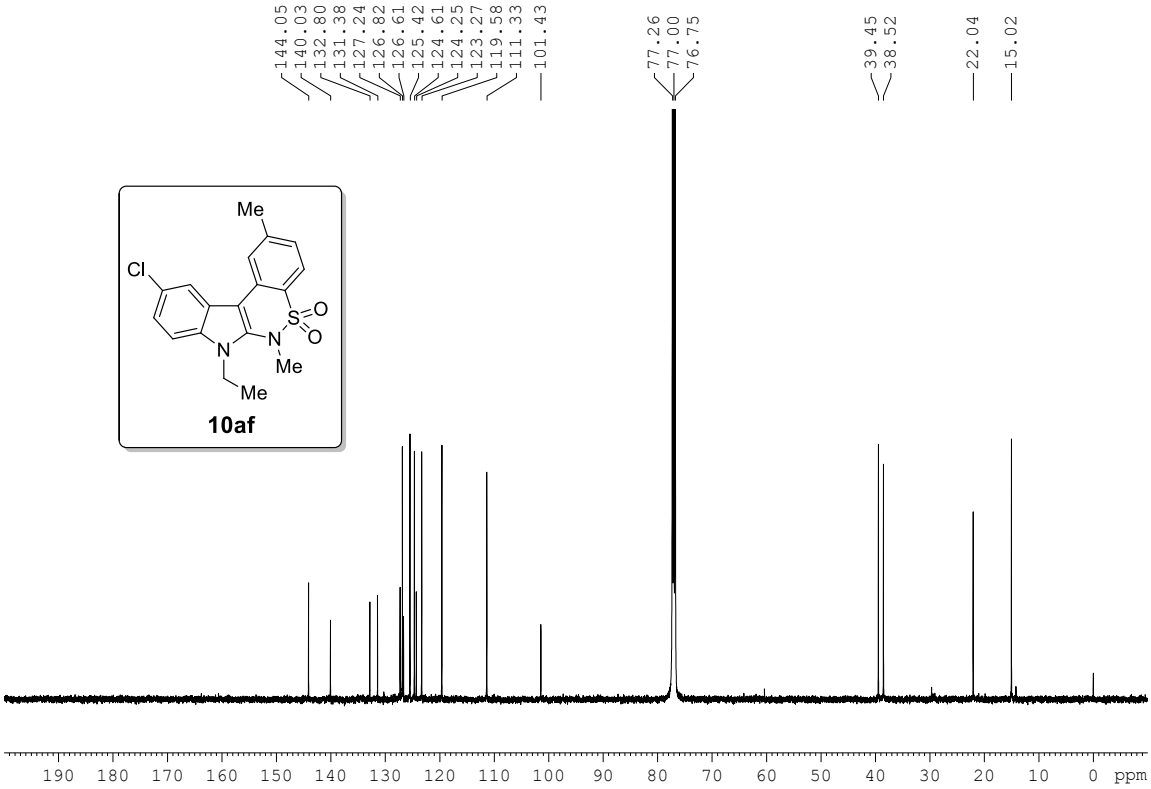


Figure S102. ^{13}C{^{1}H} NMR spectrum of compound **10af**

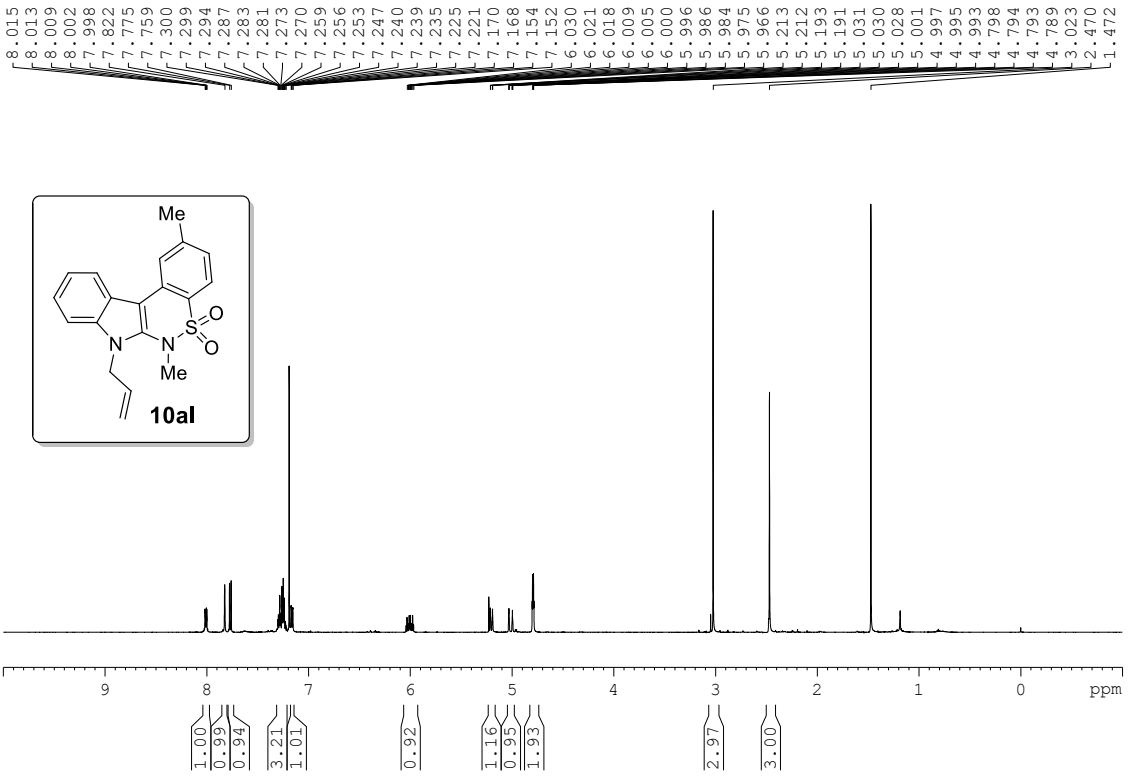


Figure S103. ^{1}H NMR spectrum of compound **10al**

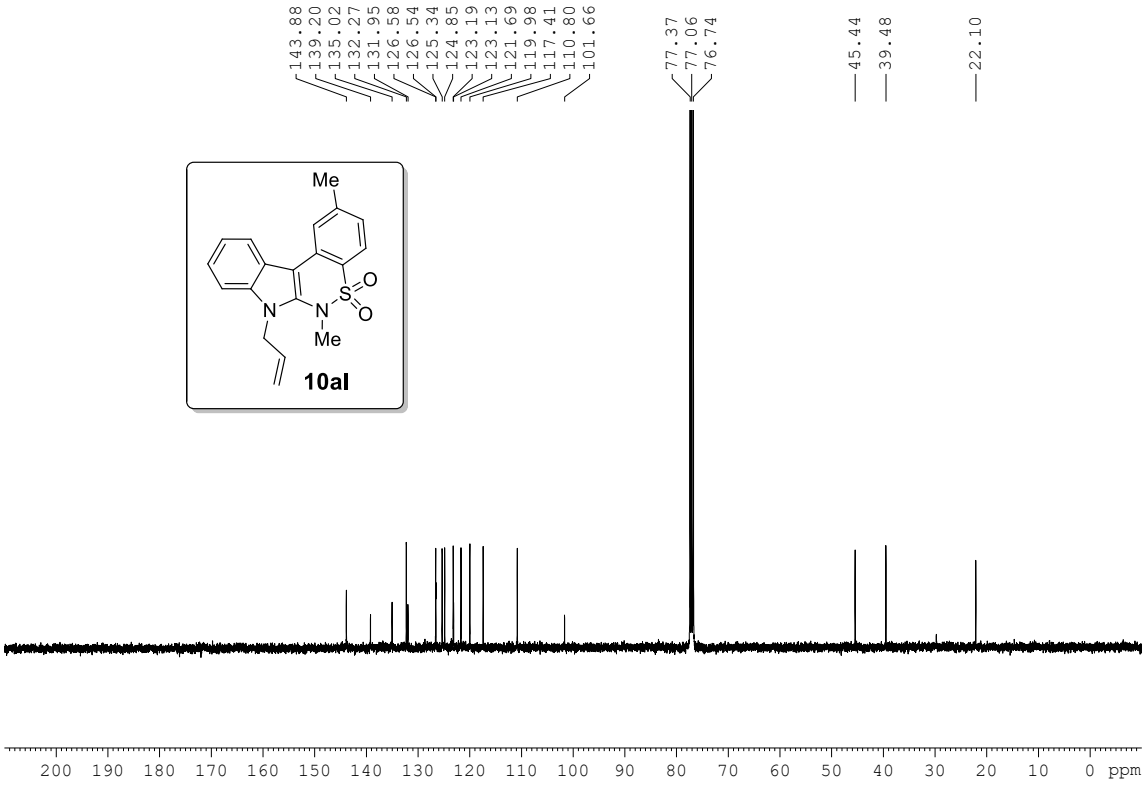


Figure S104. ^{13}C{^{1}H} NMR spectrum of compound **10al**

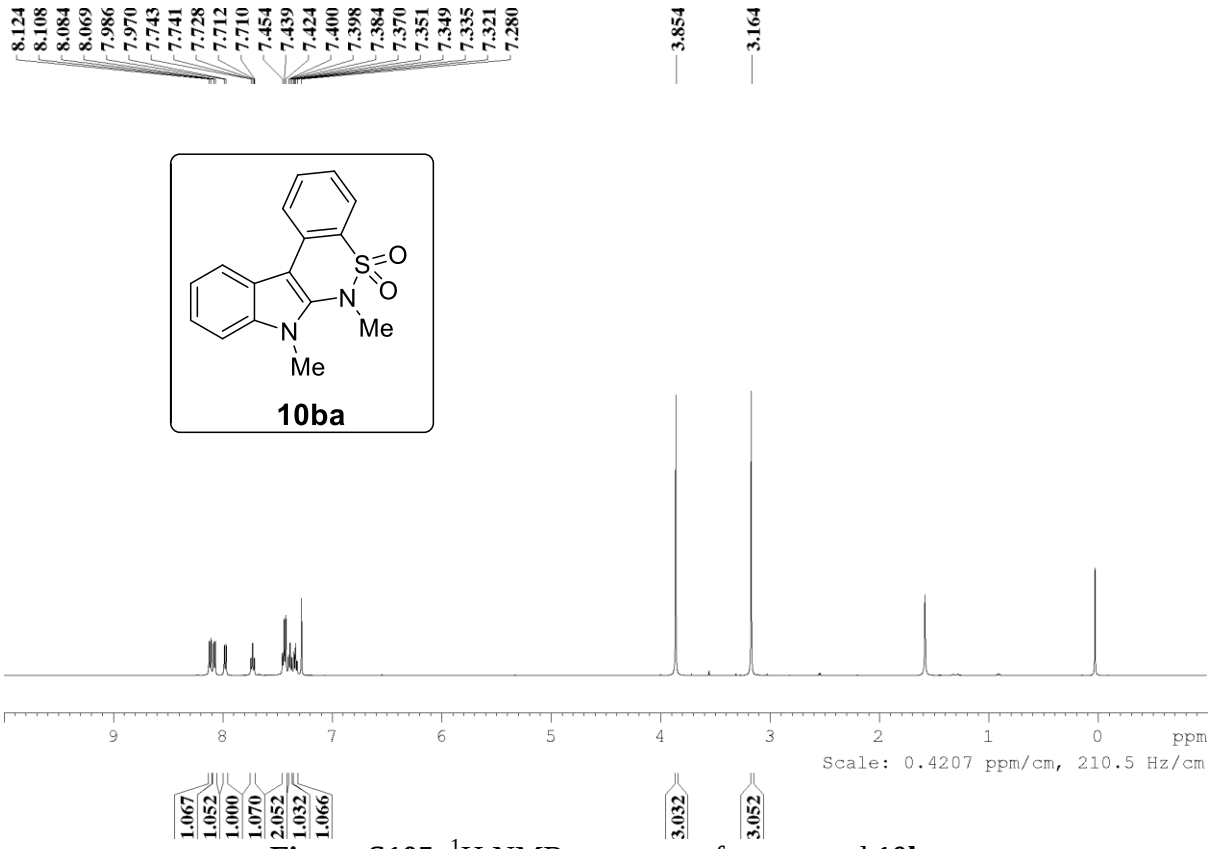


Figure S105. ^{1}H NMR spectrum of compound **10ba**

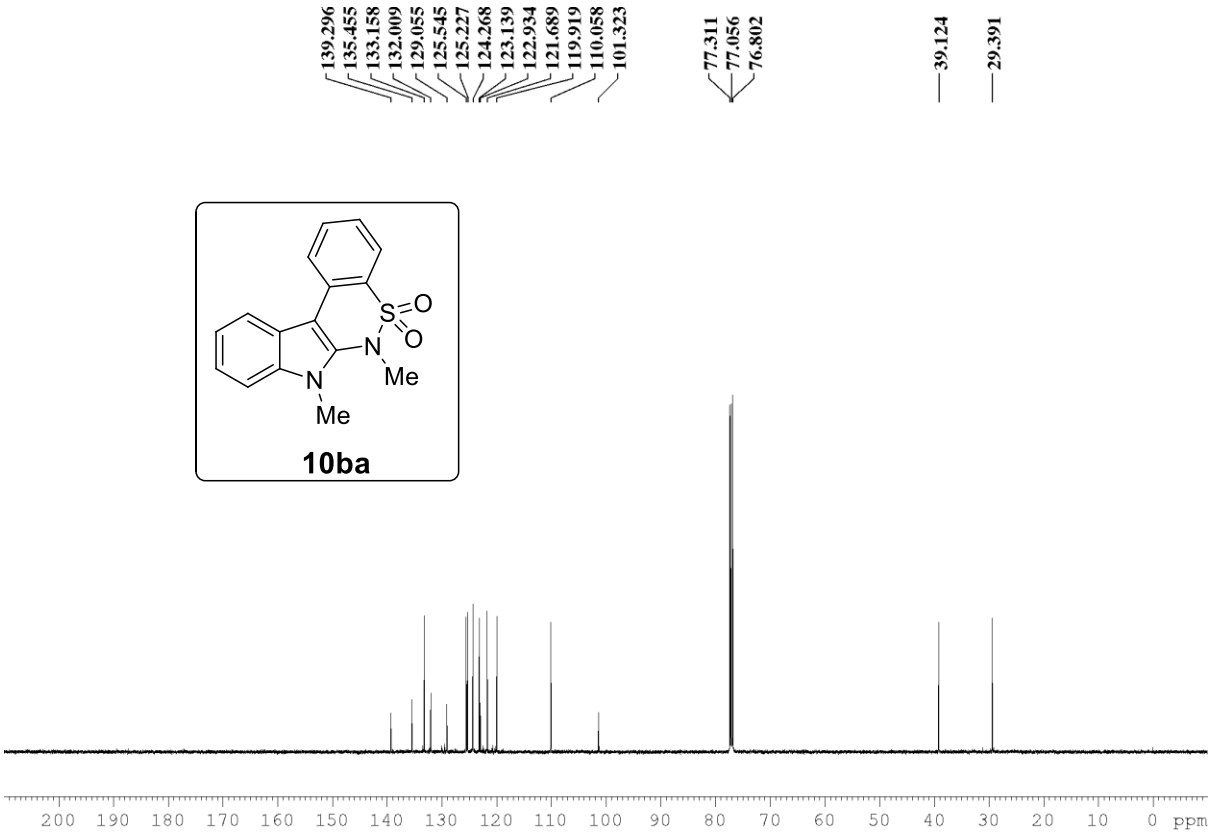


Figure S106. ^{13}C{^{1}H} NMR spectrum of compound **10ba**

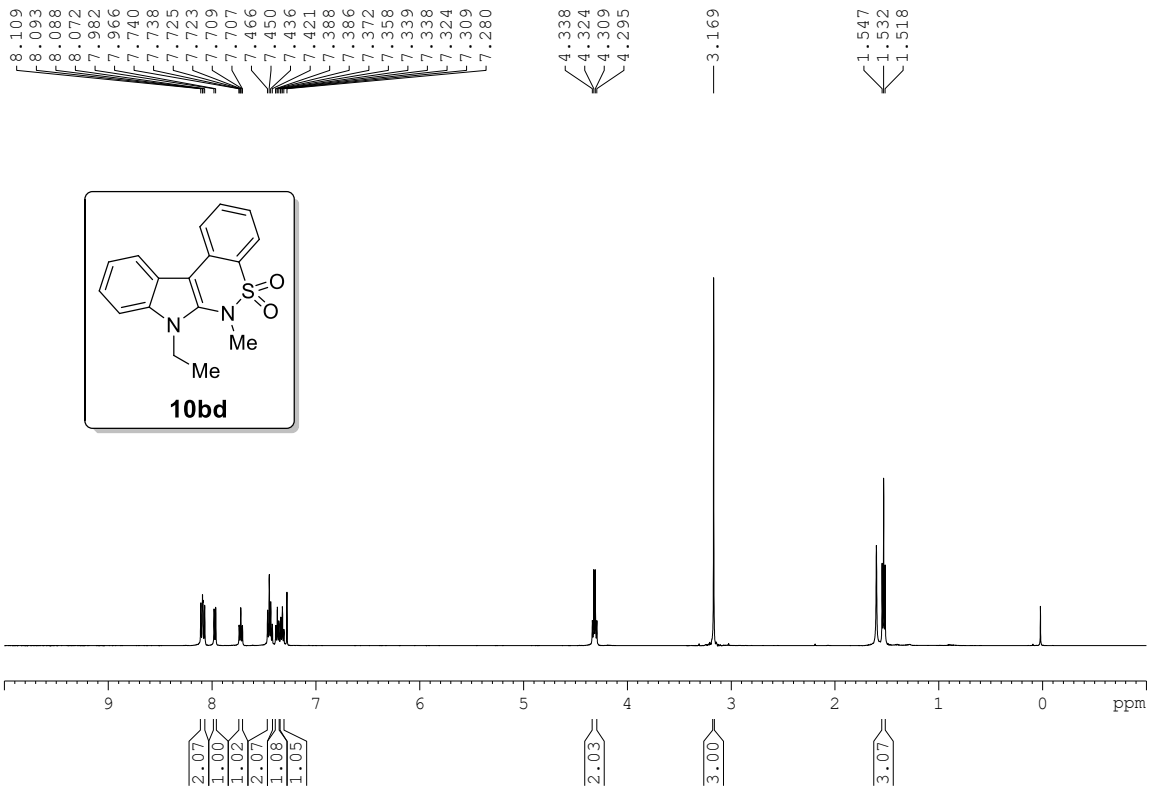


Figure S107. ^{1}H NMR spectrum of compound **10bd**

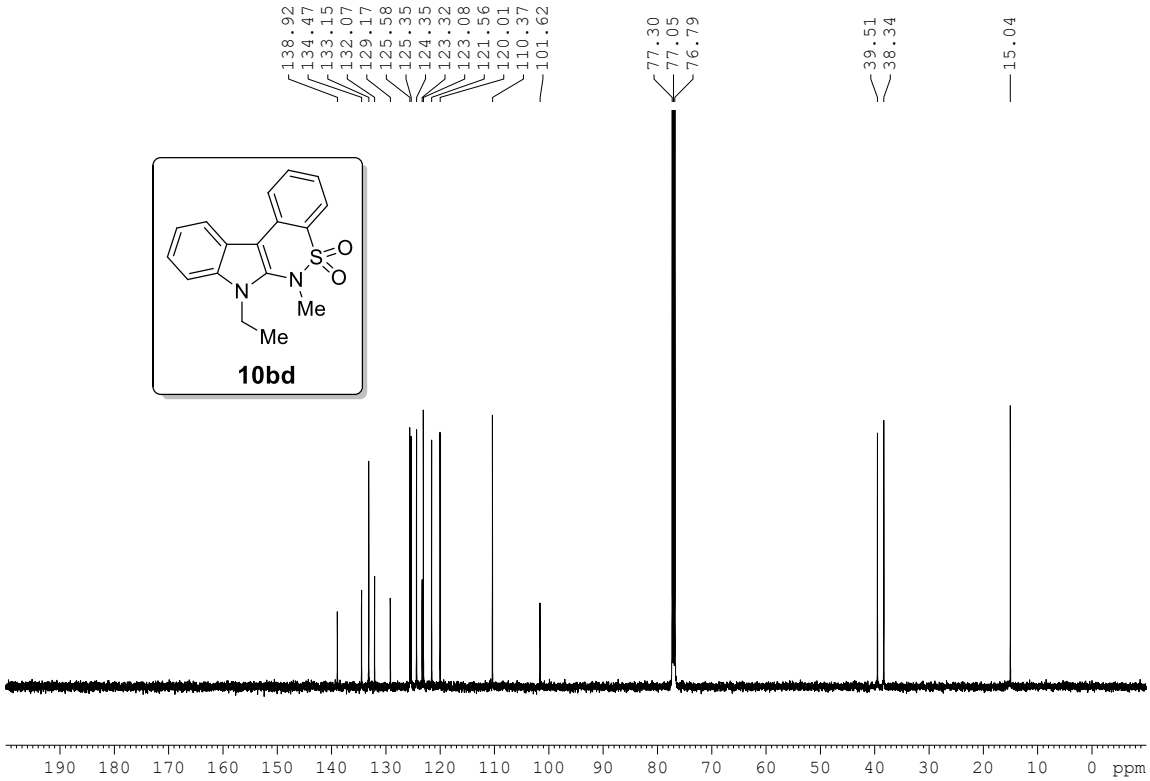


Figure S108. ^{13}C{^{1}H} NMR spectrum of compound **10bd**

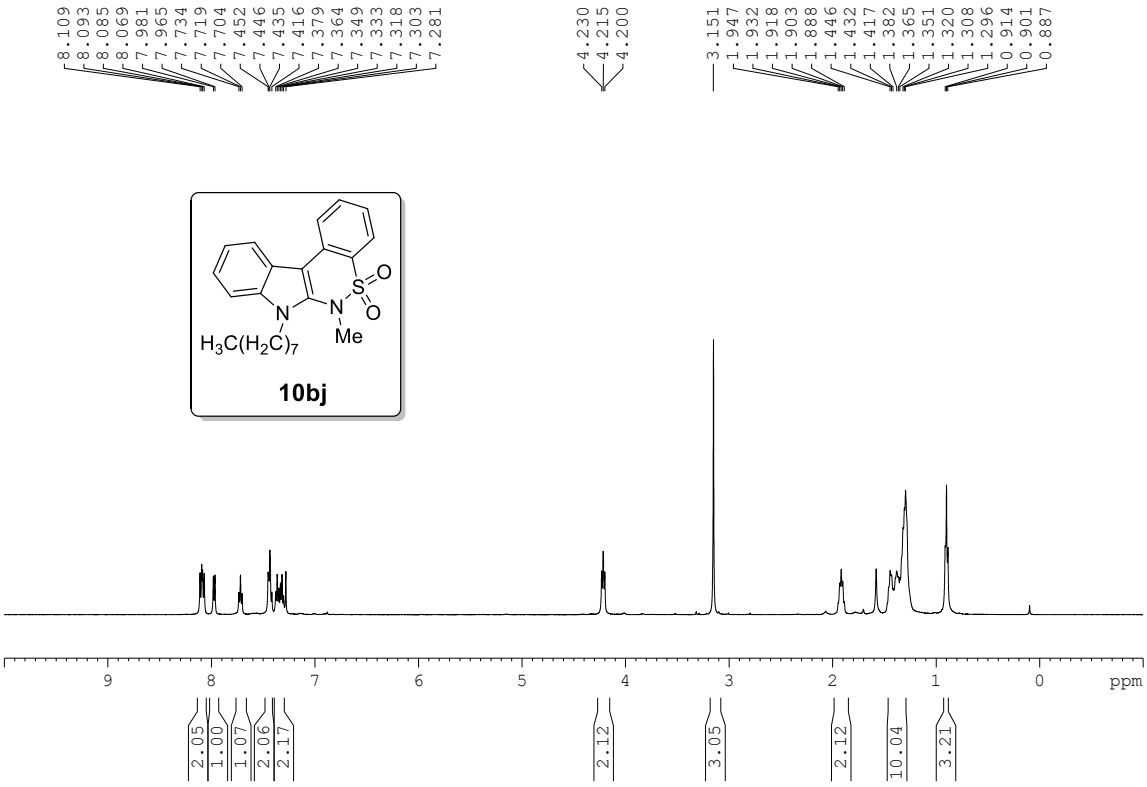


Figure S109. ^{1}H NMR spectrum of compound **10bj**

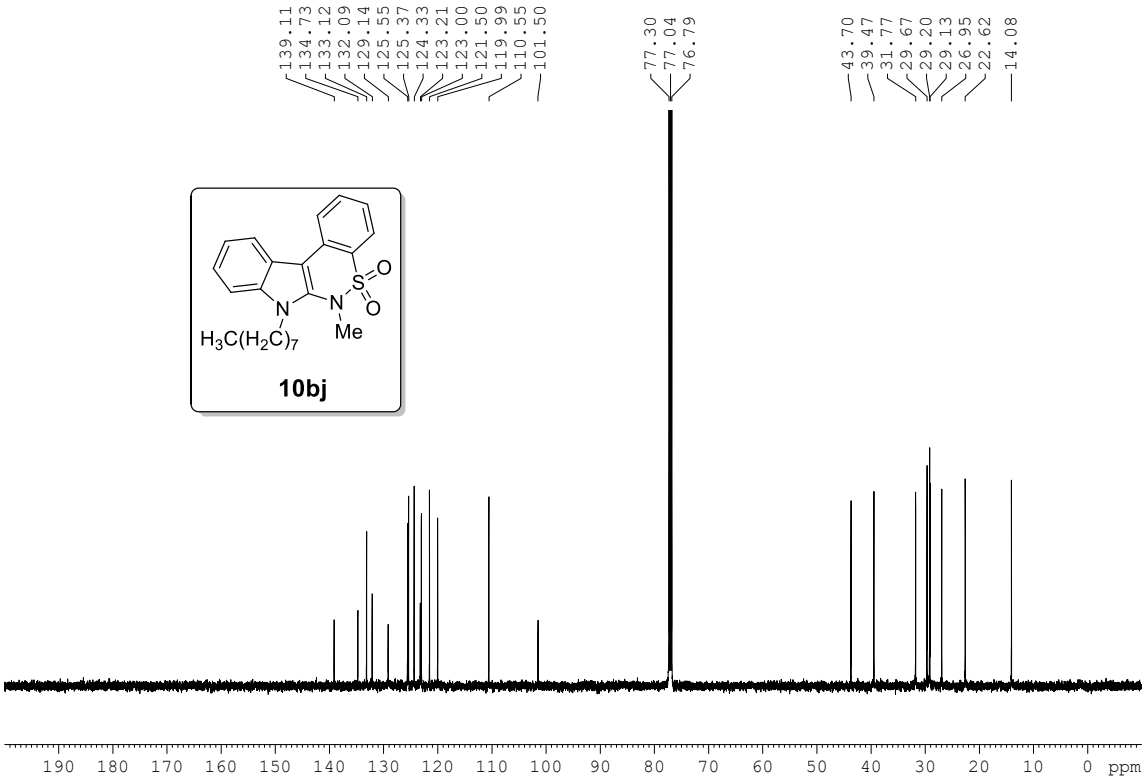


Figure S110. ^{13}C{^{1}H} NMR spectrum of compound **10bj**

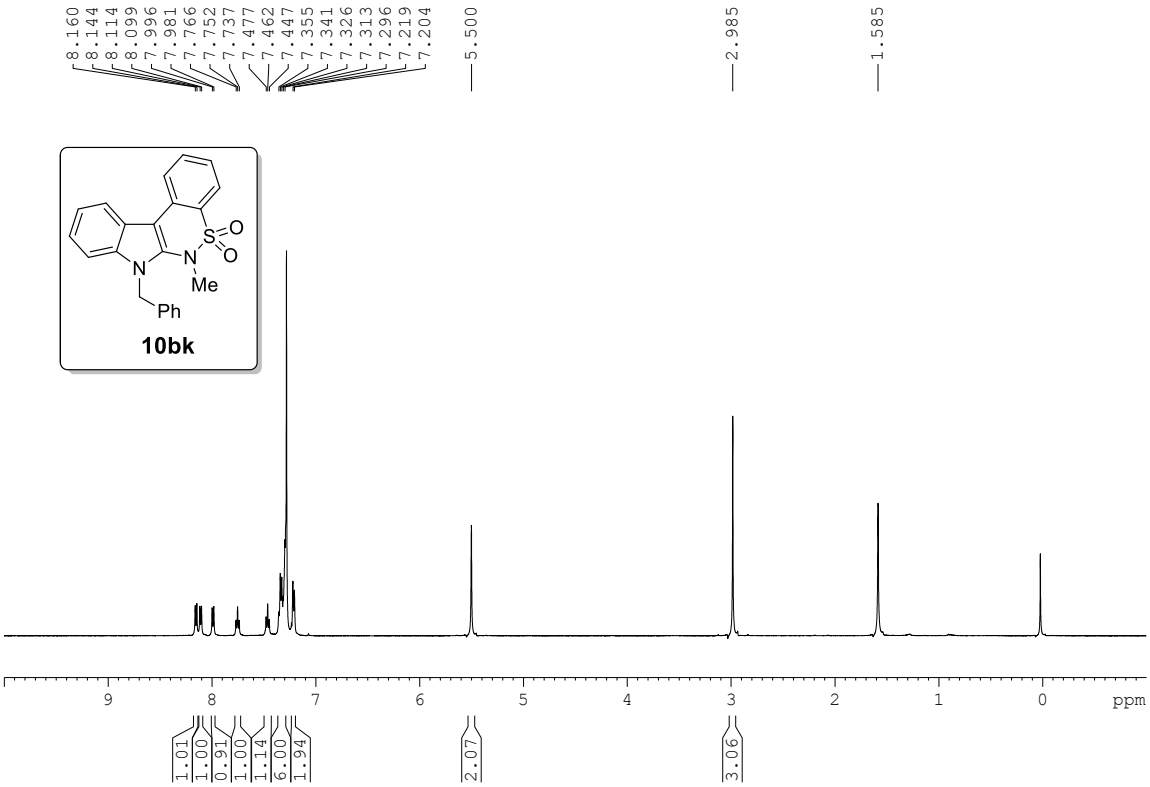


Figure S111. ^{1}H NMR spectrum of compound **10bk**

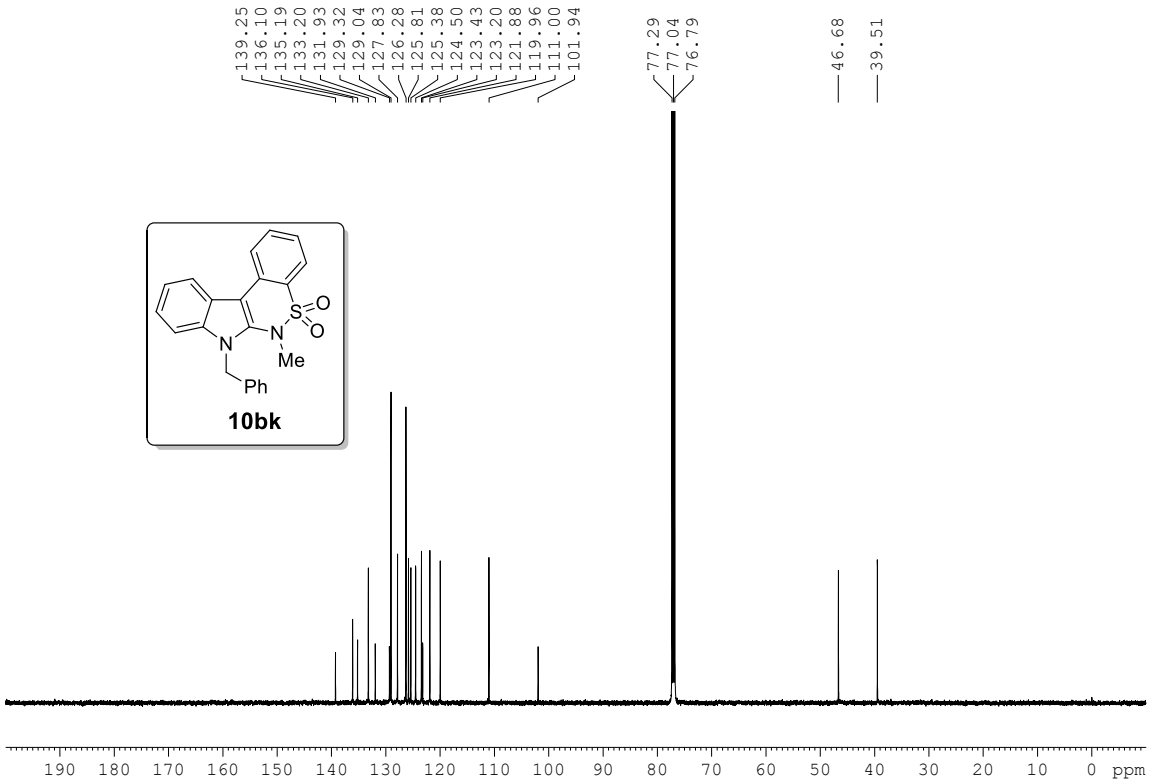


Figure S112. ^{13}C{^{1}H} NMR spectrum of compound **10bk**

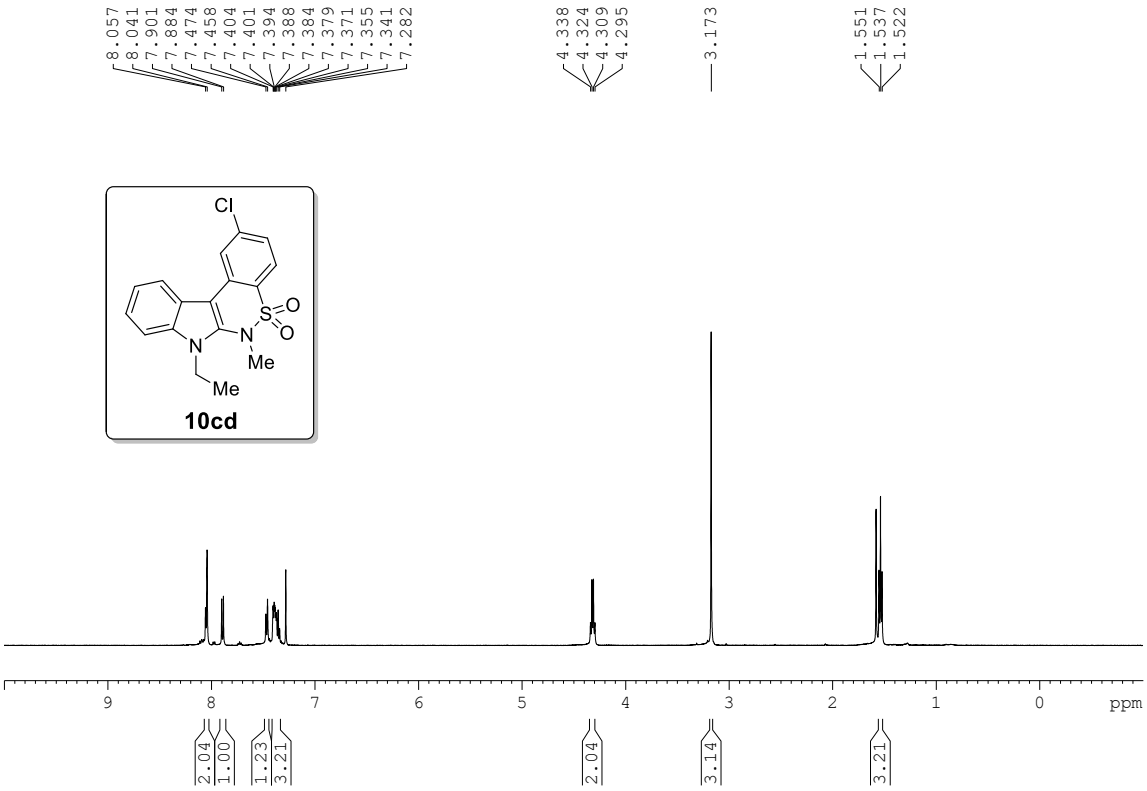


Figure S113. ^{1}H NMR spectrum of compound **10cd**

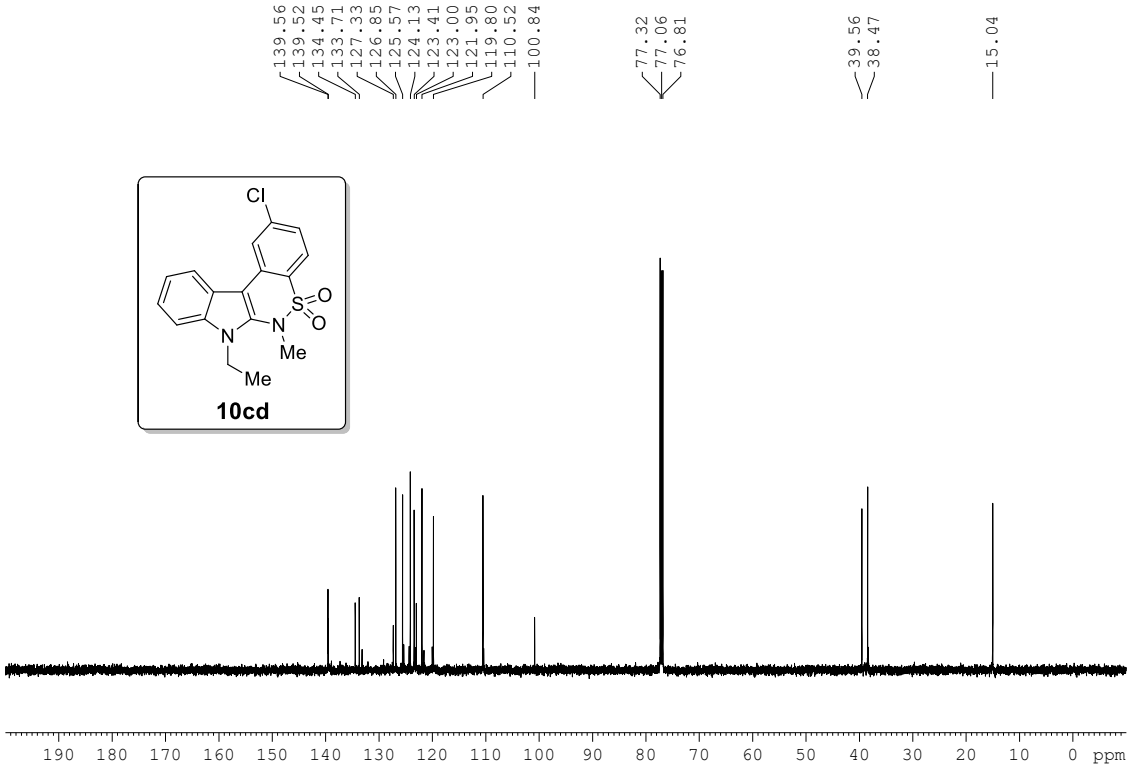


Figure S114. ^{13}C{^{1}H} NMR spectrum of compound **10cd**

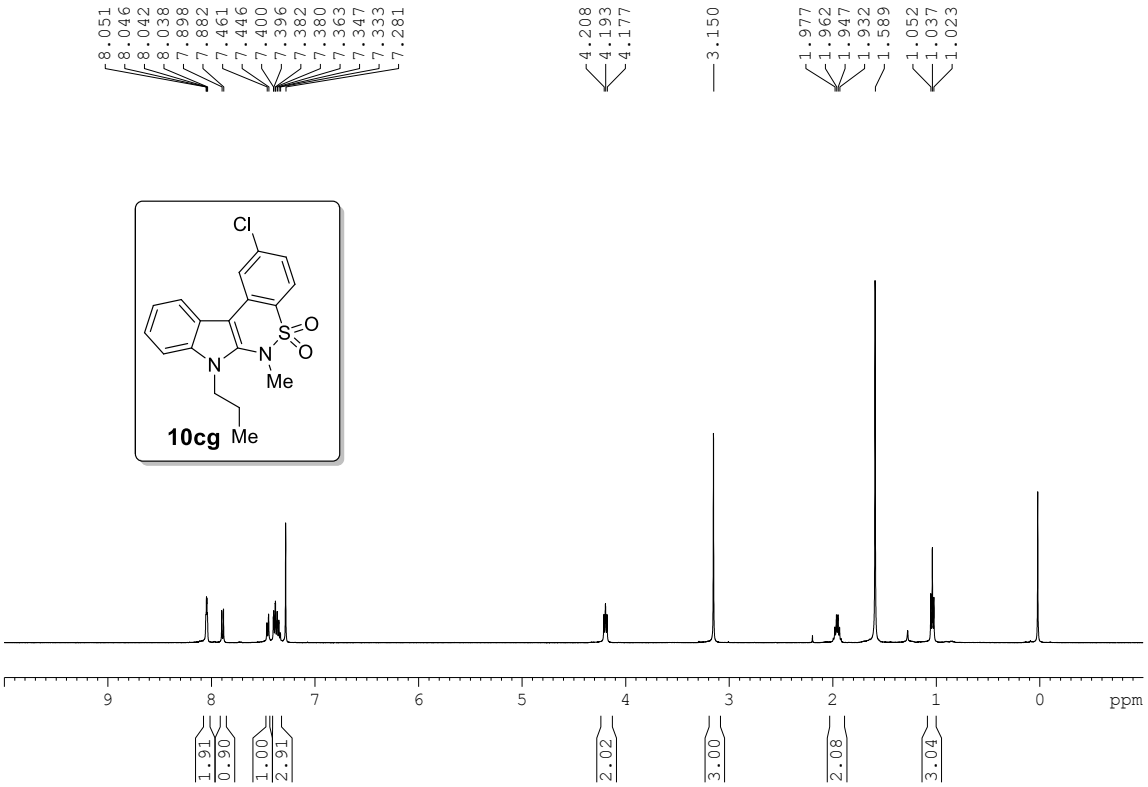


Figure S115. ^{1}H NMR spectrum of compound **10cg**

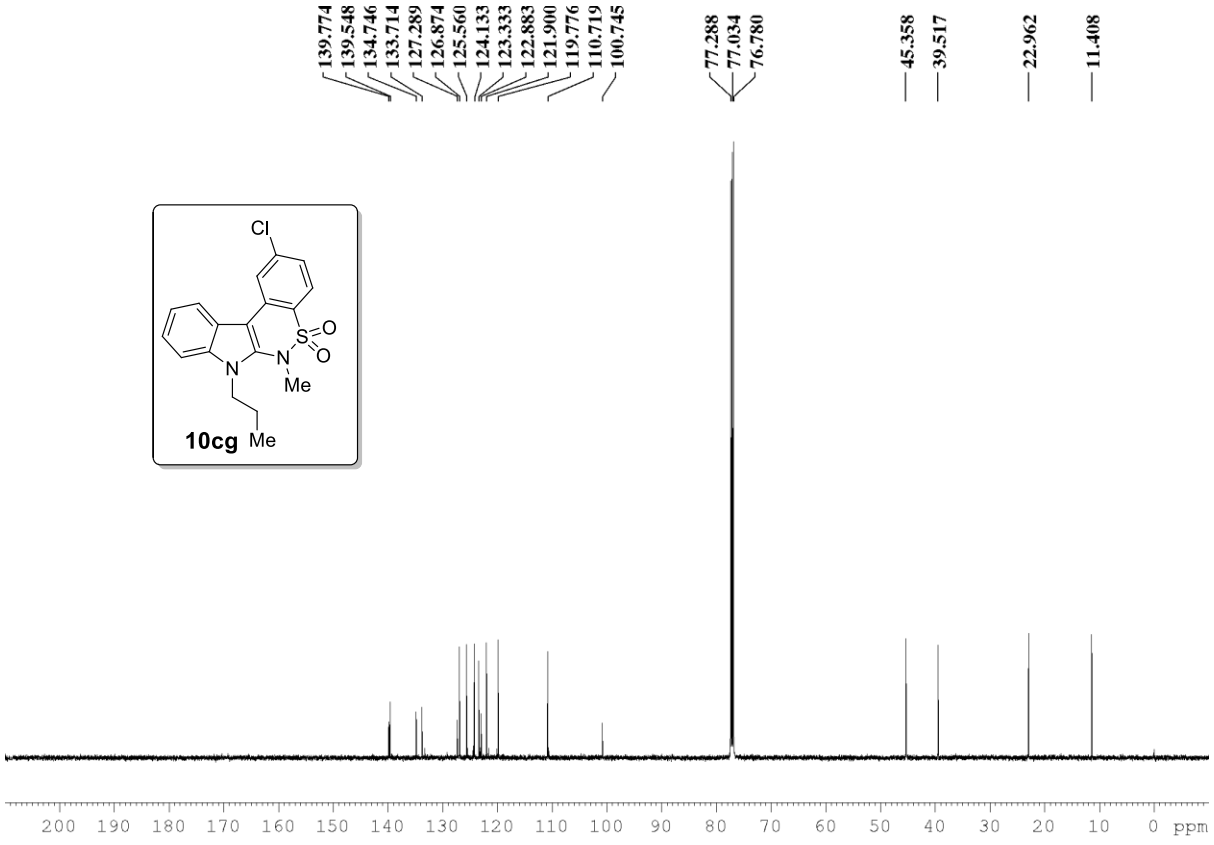


Figure S116. ^{13}C{^{1}H} NMR spectrum of compound **10cg**

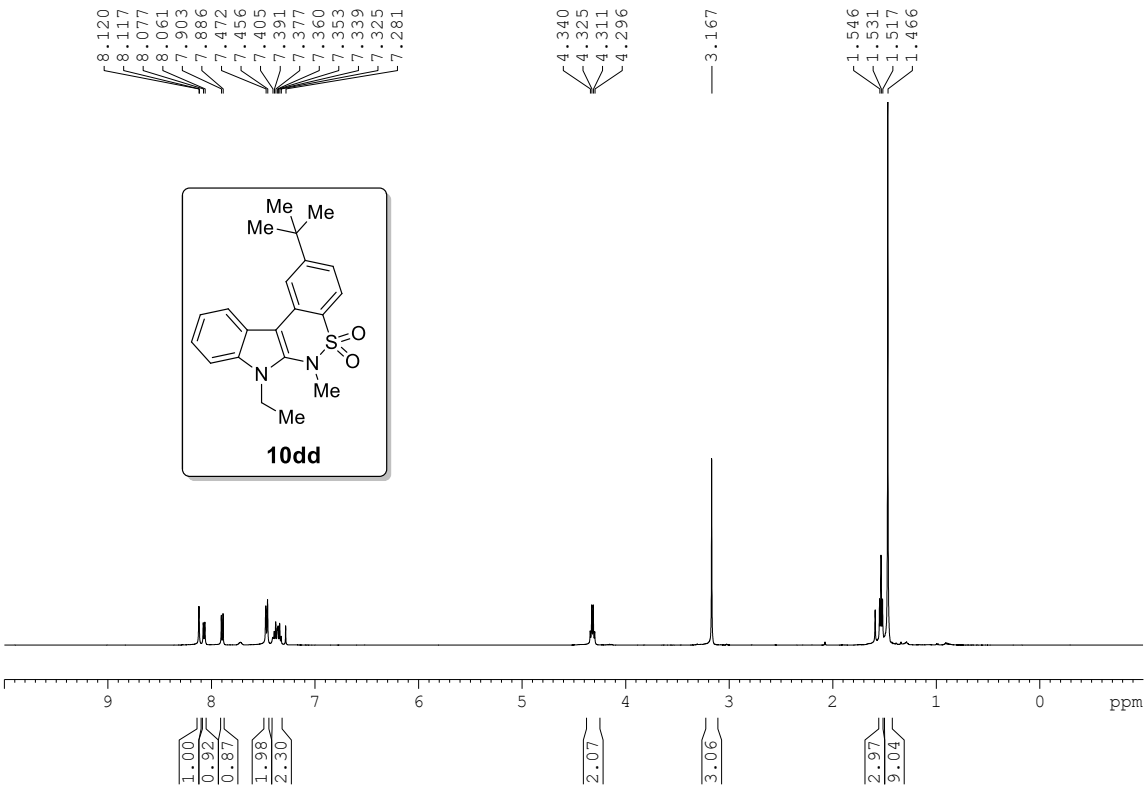


Figure S117. ^{1}H NMR spectrum of compound **10dd**

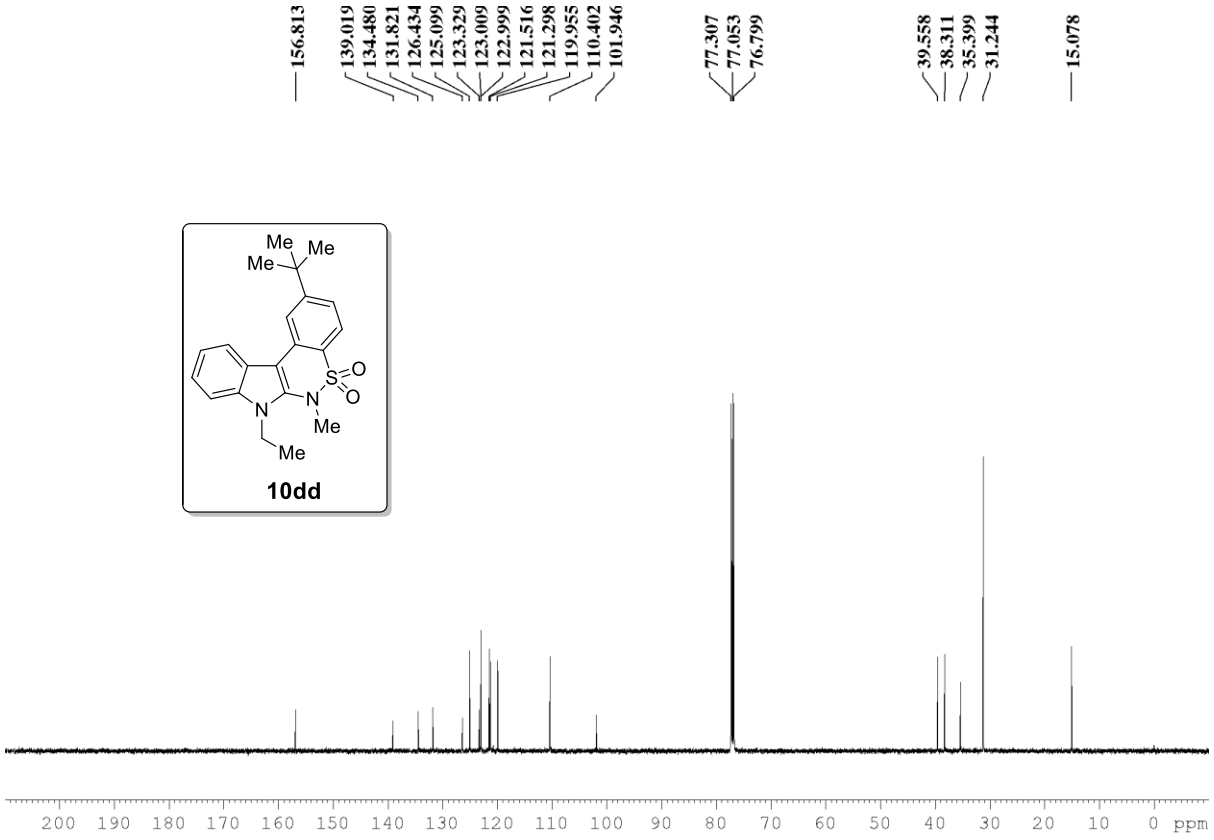


Figure S118. ^{13}C{^{1}H} NMR spectrum of compound **10dd**

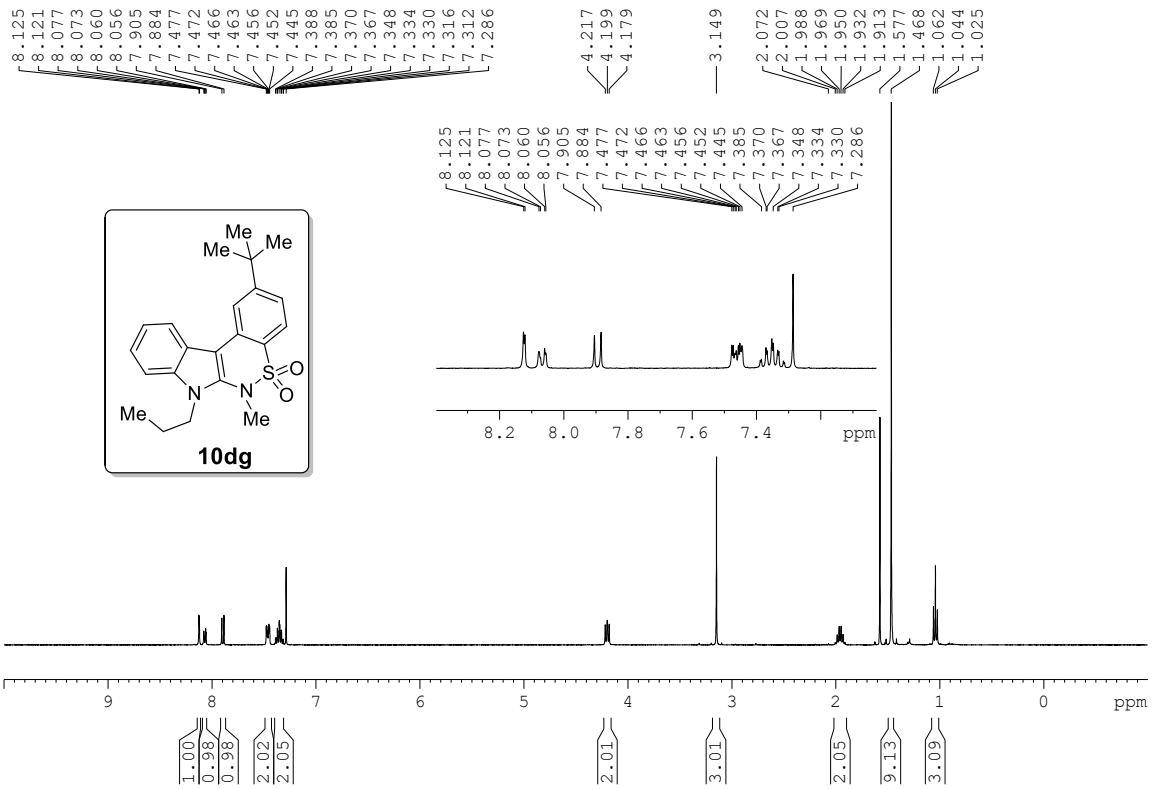


Figure S119. ^{1}H NMR spectrum of compound **10dg**

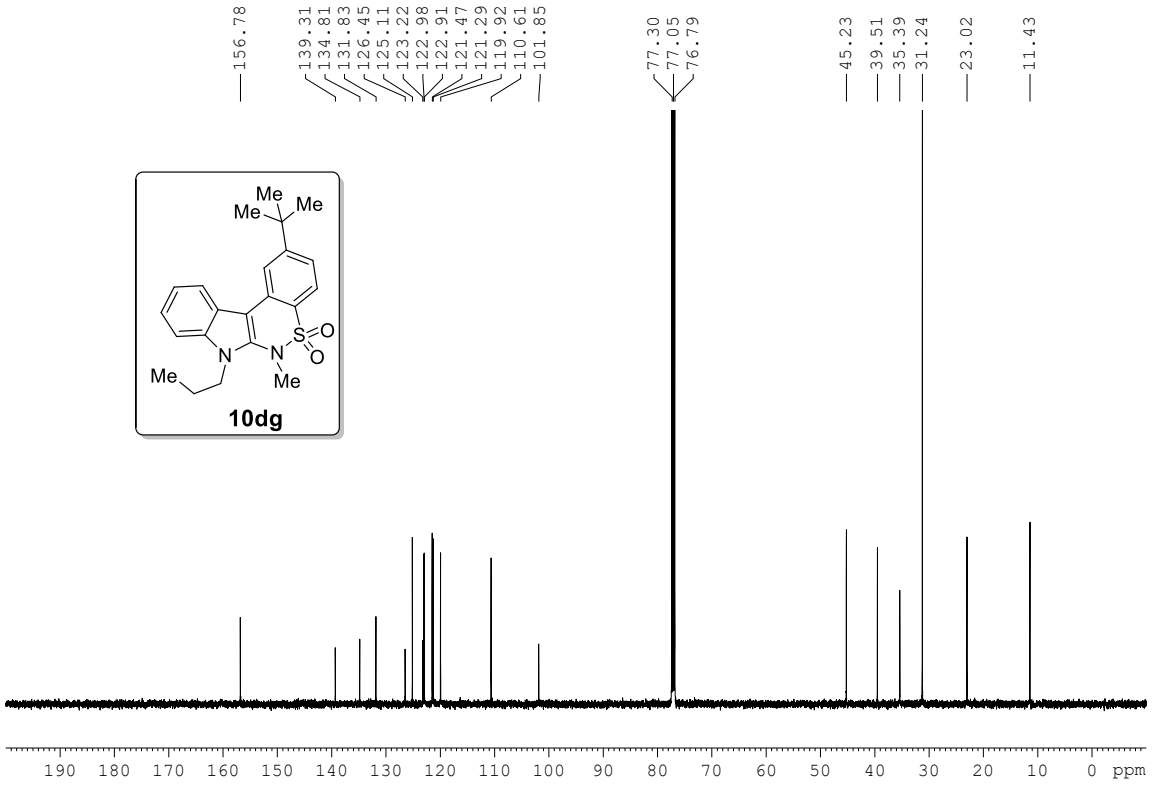


Figure S120. ^{13}C{^{1}H} NMR spectrum of compound **10dg**

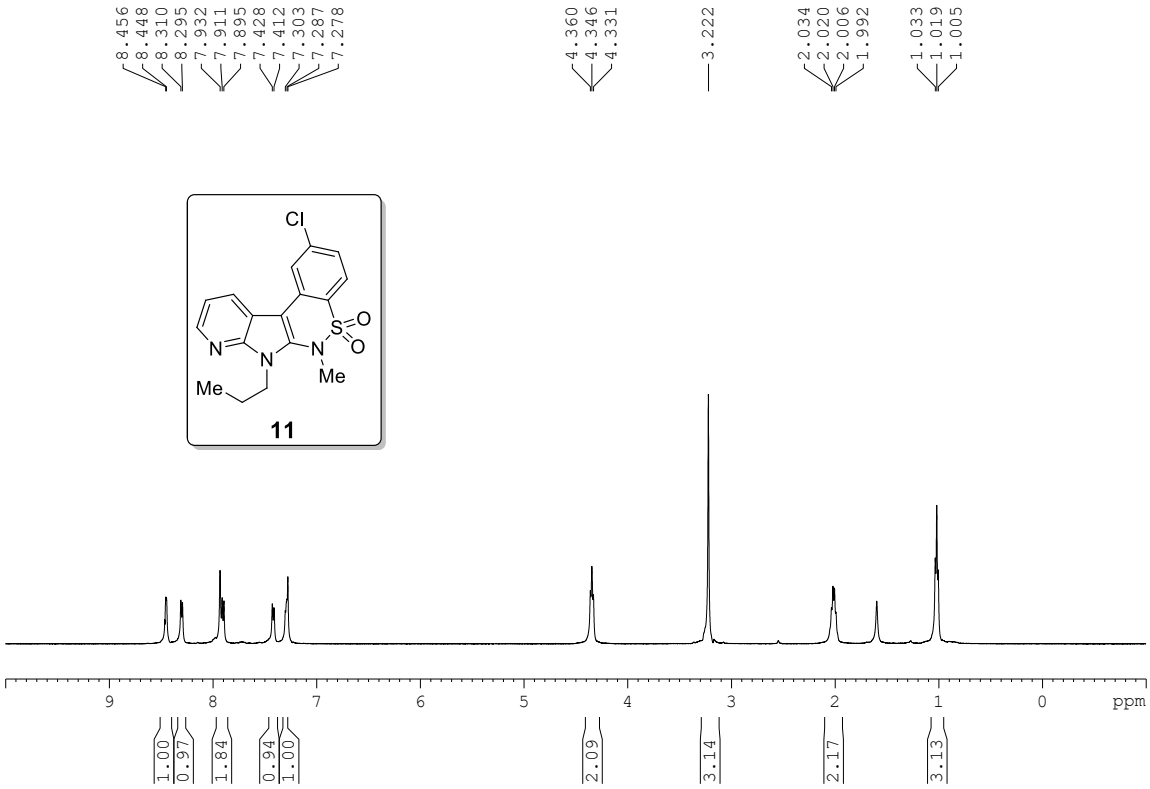


Figure S121. ^{1}H NMR spectrum of compound **11**

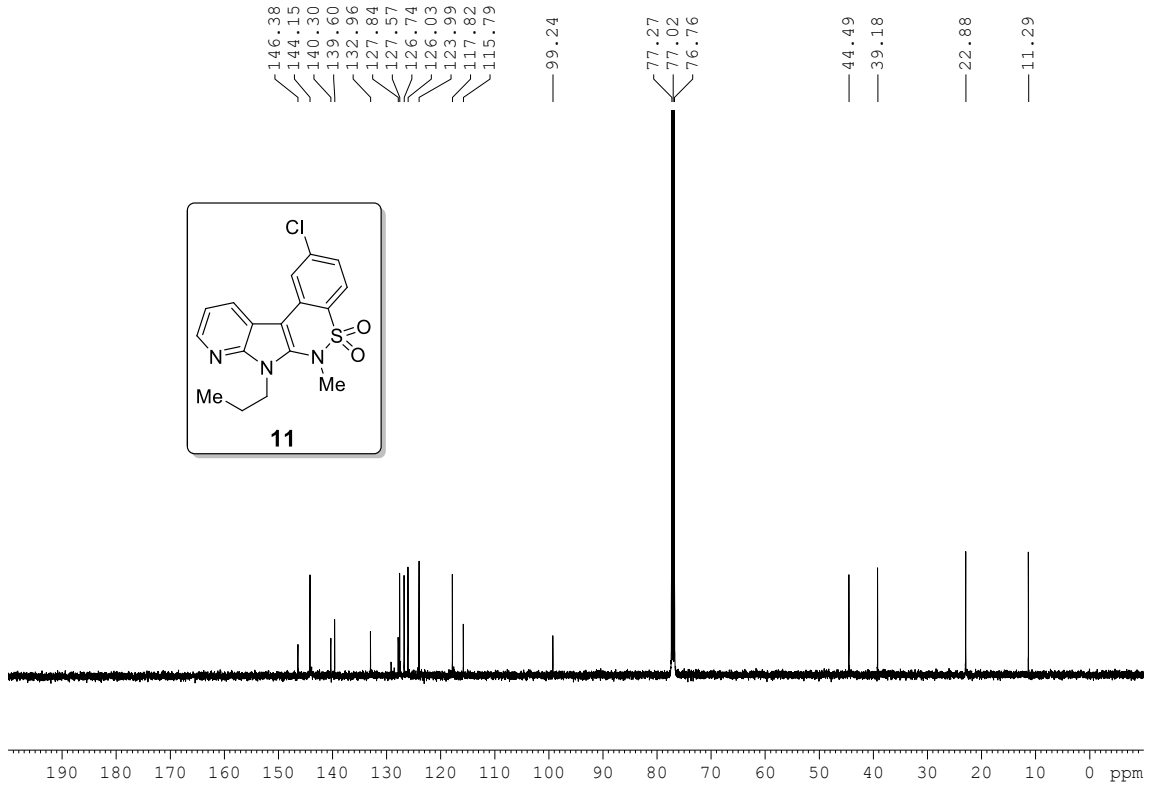


Figure S122. ^{13}C{^{1}H} NMR spectrum of compound **11**

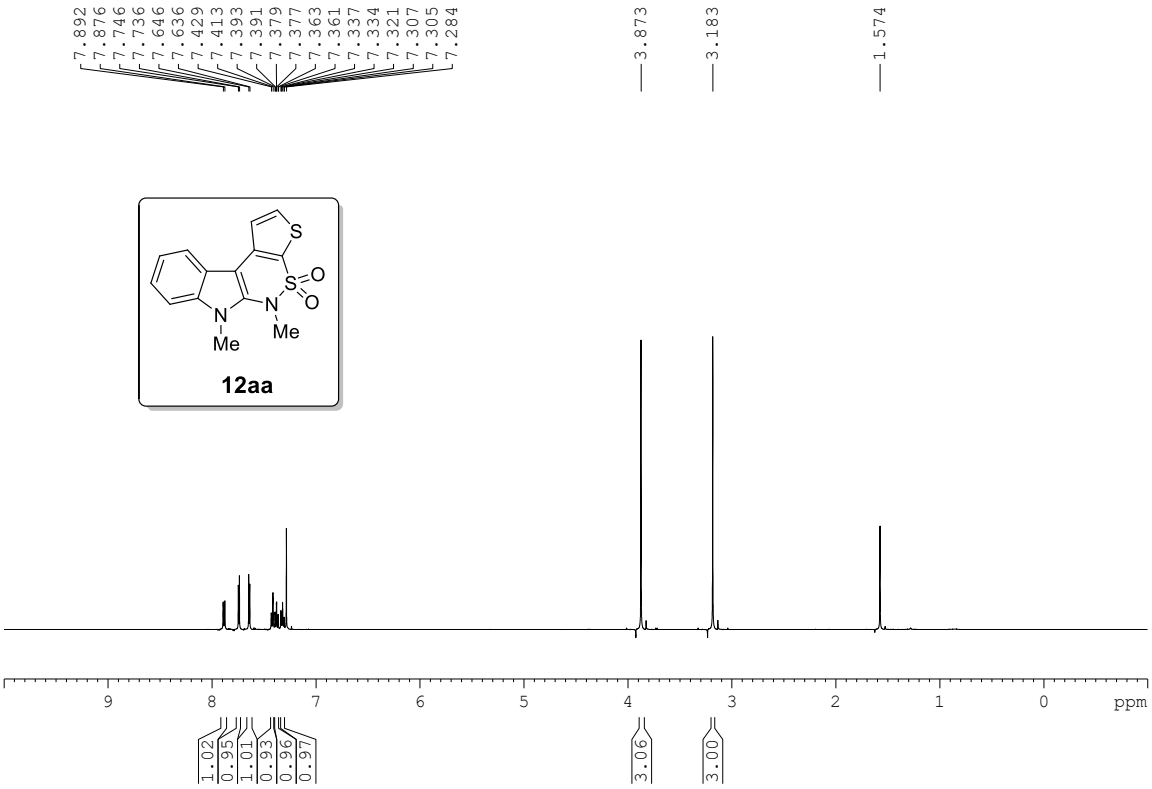


Figure S123. ^{1}H NMR spectrum of compound **12aa**

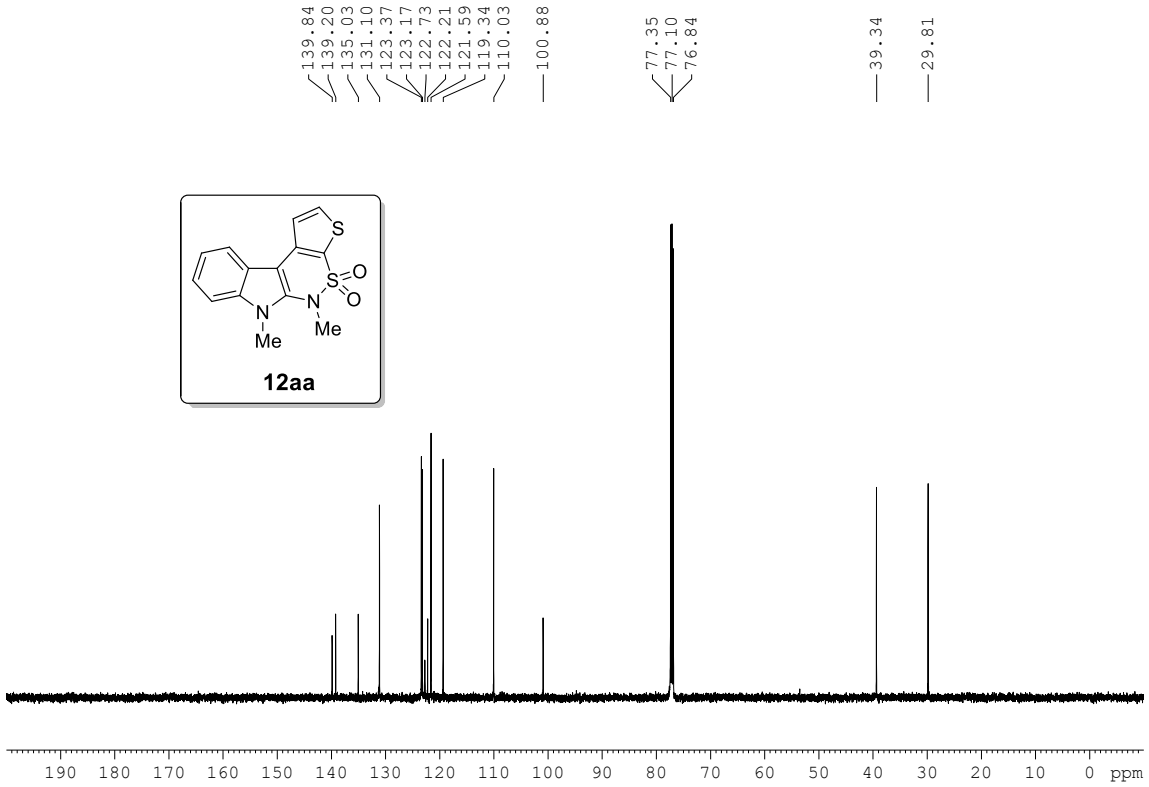


Figure S124. ^{13}C{^{1}H} NMR spectrum of compound **12aa**

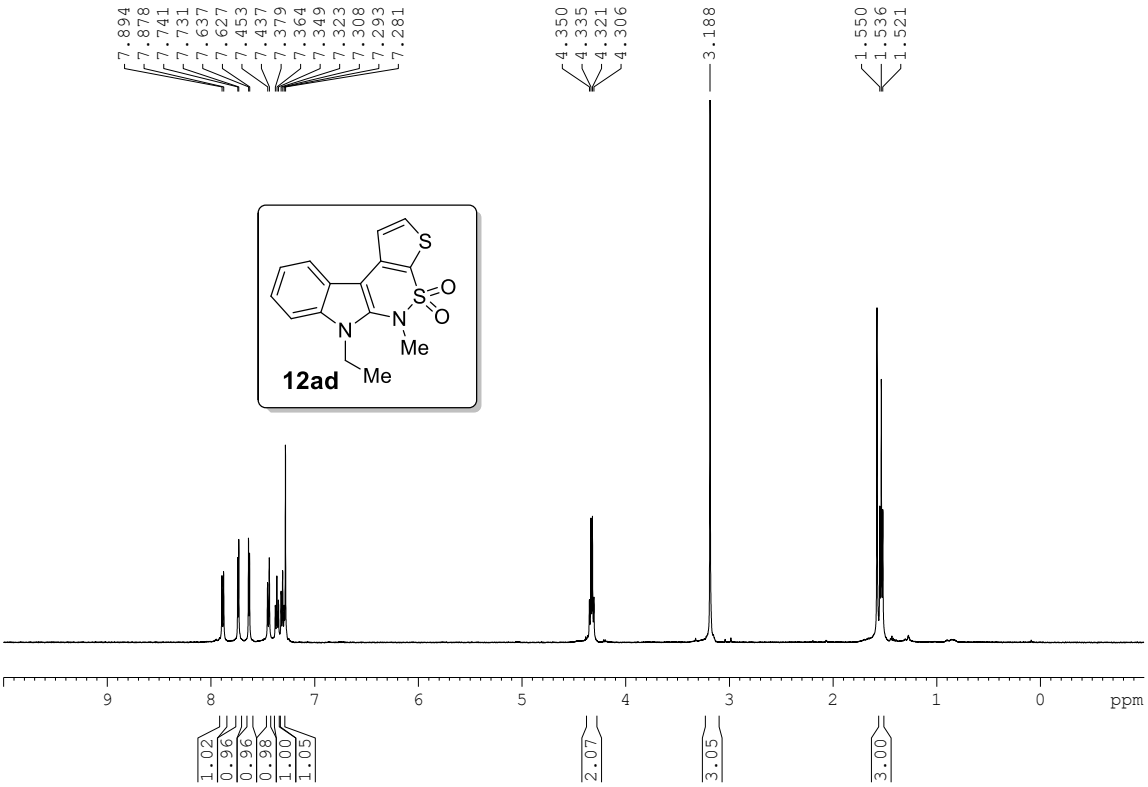


Figure S125. ^{1}H NMR spectrum of compound **12ad**

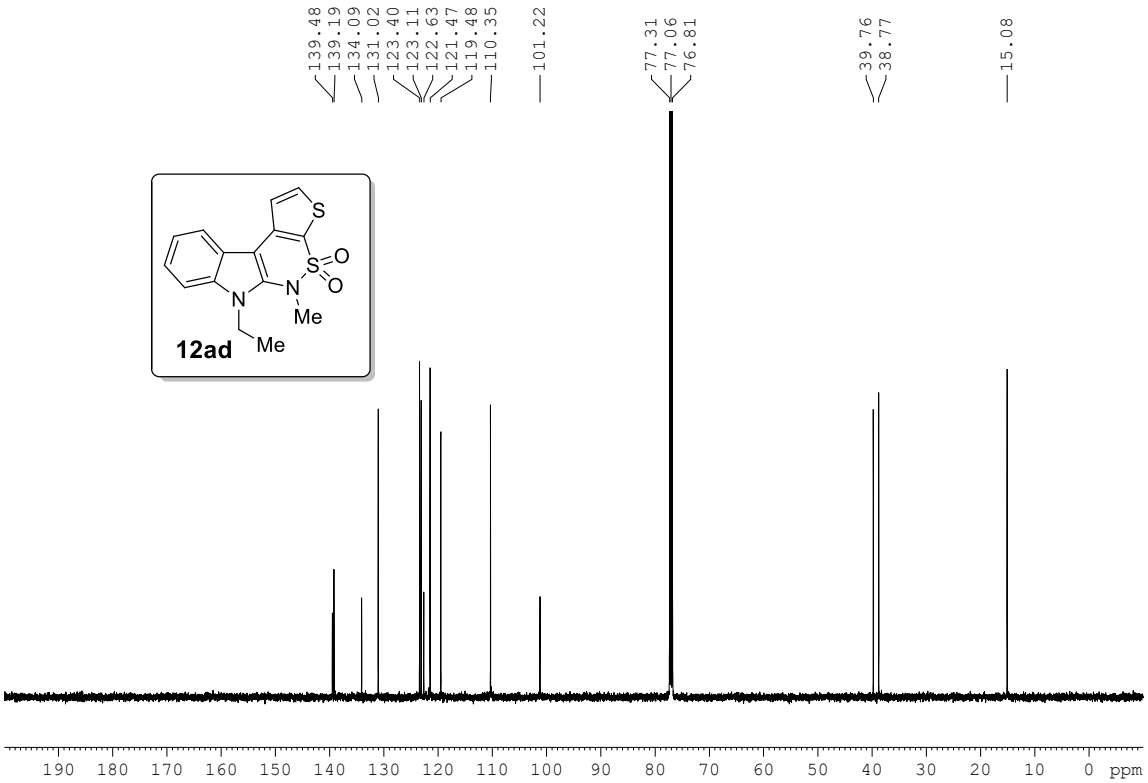


Figure S126. ^{13}C{^{1}H} NMR spectrum of compound **12ad**

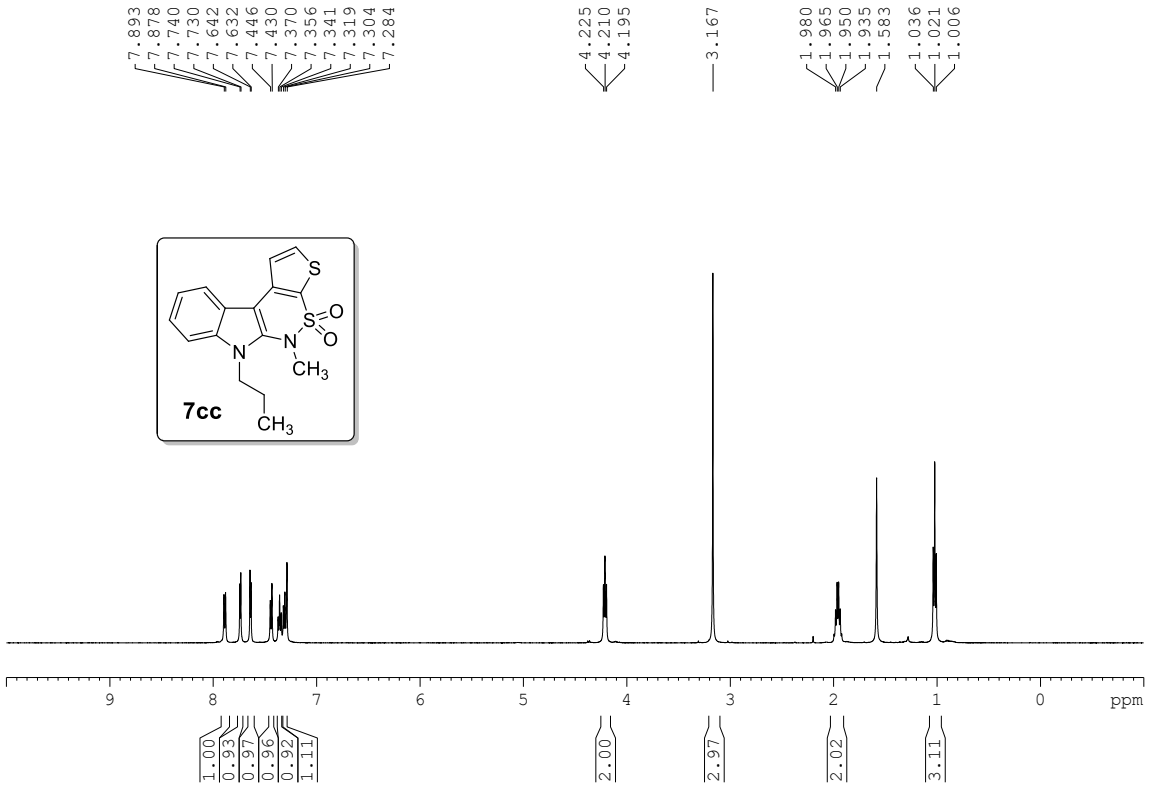


Figure S127. ^{1}H NMR spectrum of compound **12ag**

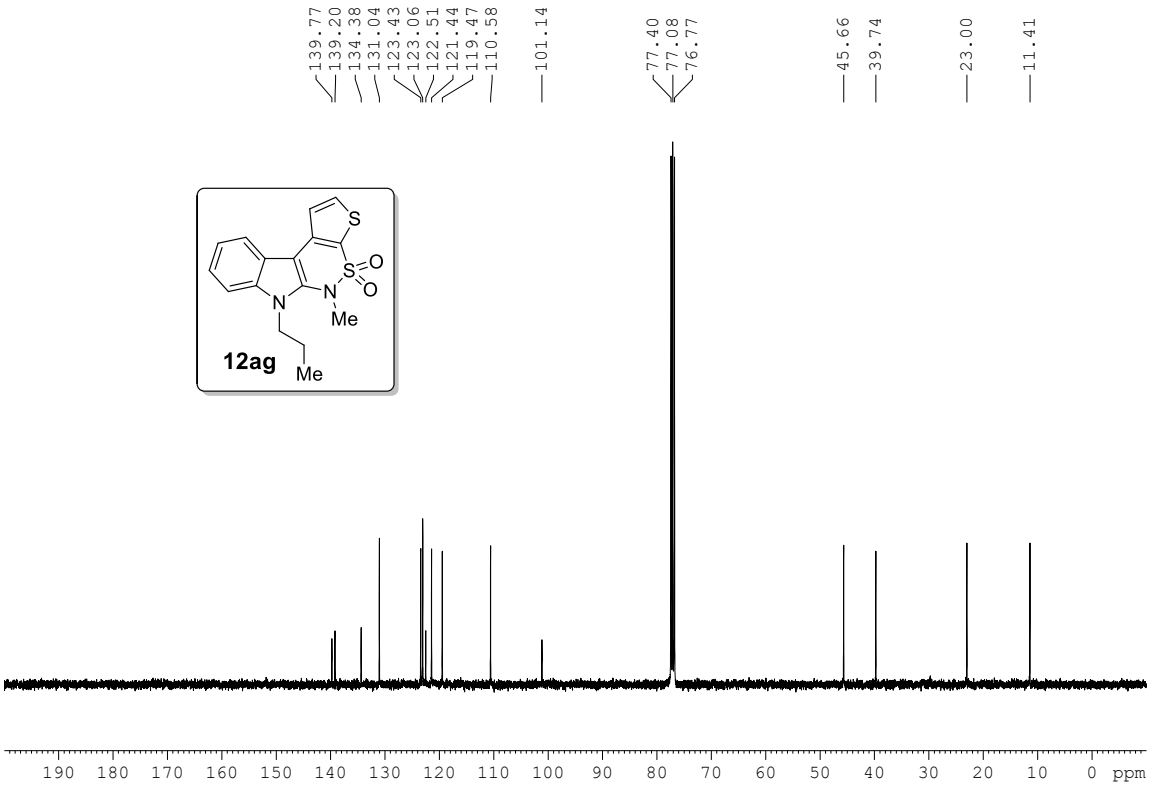


Figure S128. $^{13}C\{^{1}H\}$ NMR spectrum of compound **12ag**

Display Report

Analysis Info

Analysis Name	E:\2021-data\PROF.KCK\SEPT\SAK-4.d	Acquisition Date	14-09-2021 13:01:44
Method	tune_low.m	Operator	BDAL
Sample Name	SAK-4	Instrument	maXis 255552.10138
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	1500 m/z	Set Charging Voltage	0 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C

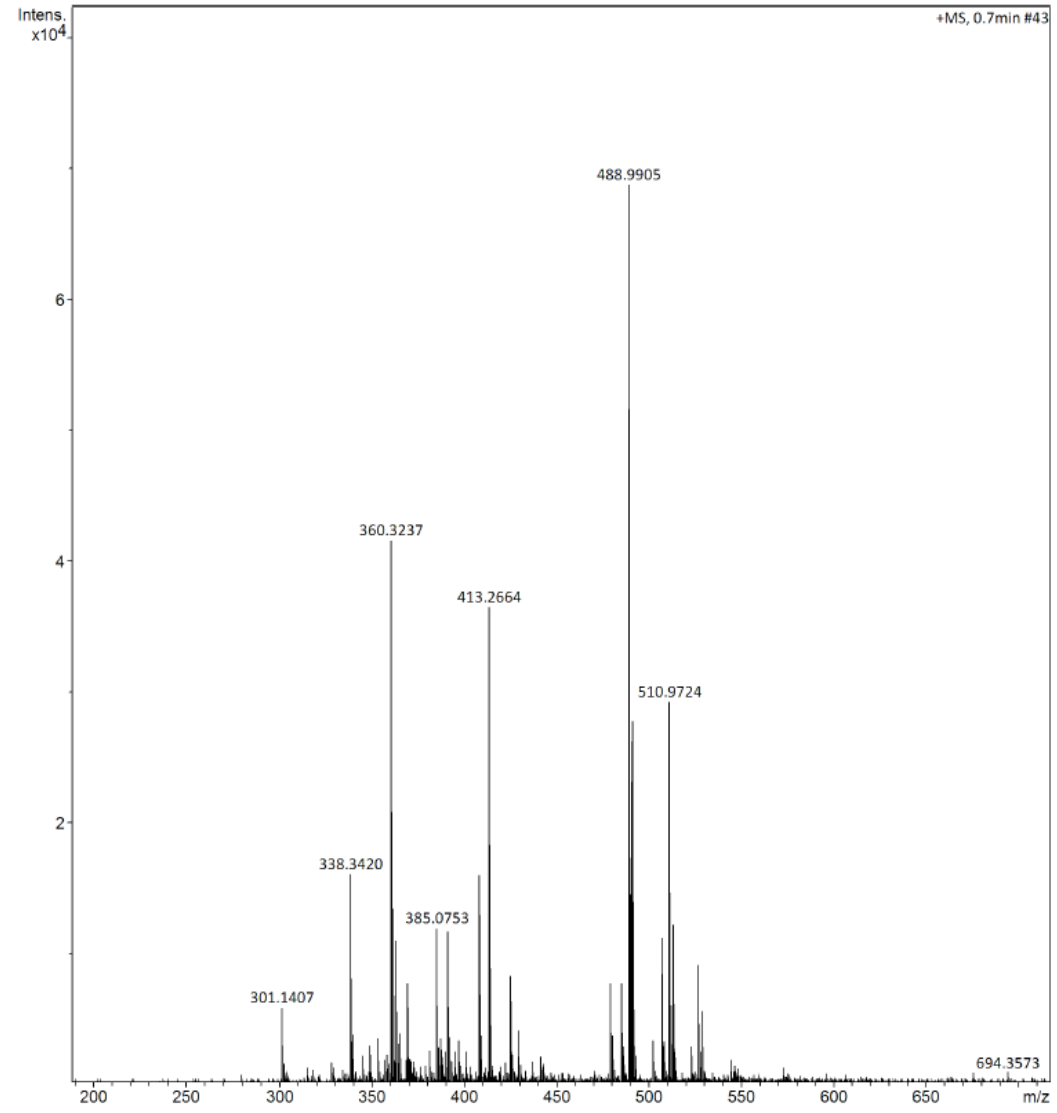


Figure S129. HRMS (ESI) of compound **5af**

Display Report

Analysis Info

Analysis Name	E:\2021-data\PROF.KCK\JULY-2021\SAK-286R.d	Acquisition Date	03-07-2021 11:47:34
Method	tune_low_PosR.m	Operator	BDAL
Sample Name	SAK-286R	Instrument	maXis 255552.10138
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
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Scan Begin	300 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	2500 m/z	Set Charging Voltage	0 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C

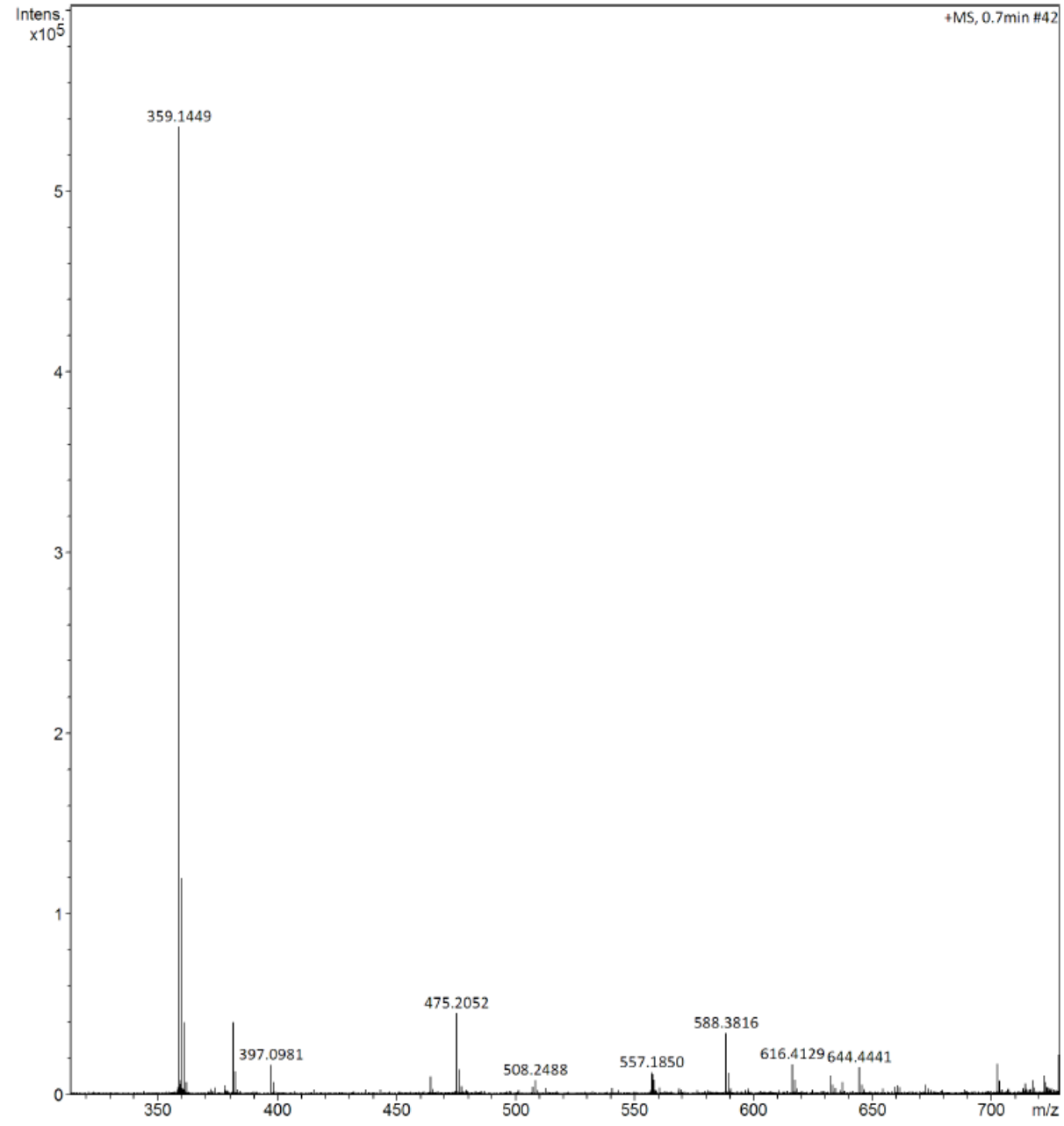

Figure S130. HRMS (ESI) of compound **9ae**

Display Report

Analysis Info

Analysis Name	E:\2021-data\PROF.KCK\JULY-2021\RAJ-176.d	Acquisition Date	03-07-2021 12:00:14
Method	tune_low_Pos-R2.m	Operator	BDAL
Sample Name	RAJ-176	Instrument	maXis 255552.10138
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.3 Bar
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Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	2580 m/z	Set Charging Voltage	0 V	Set Divert Valve	Waste
		Set Corona	0 nA	Set APCI Heater	0 °C

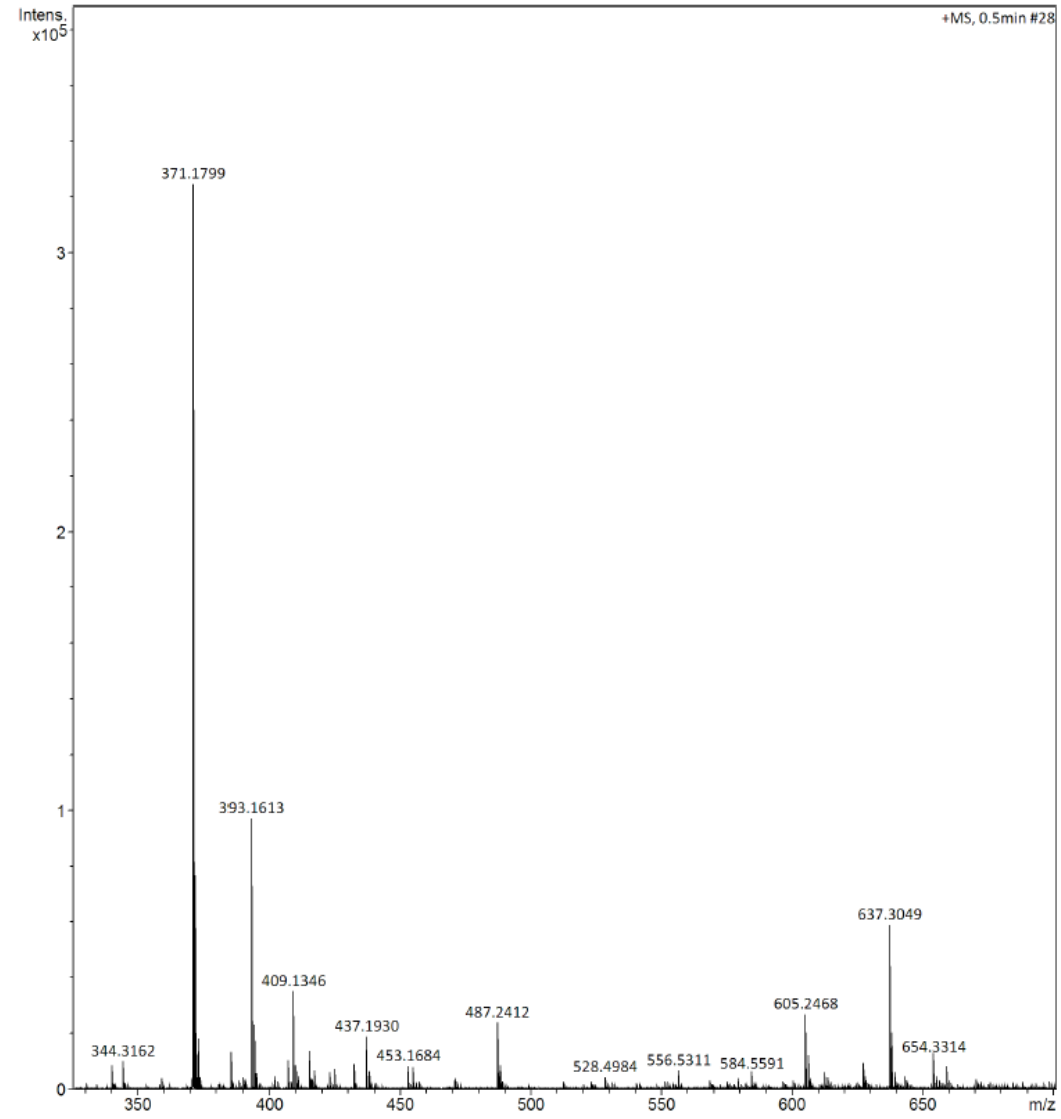

Figure S131. HRMS (ESI) of compound **9bi**

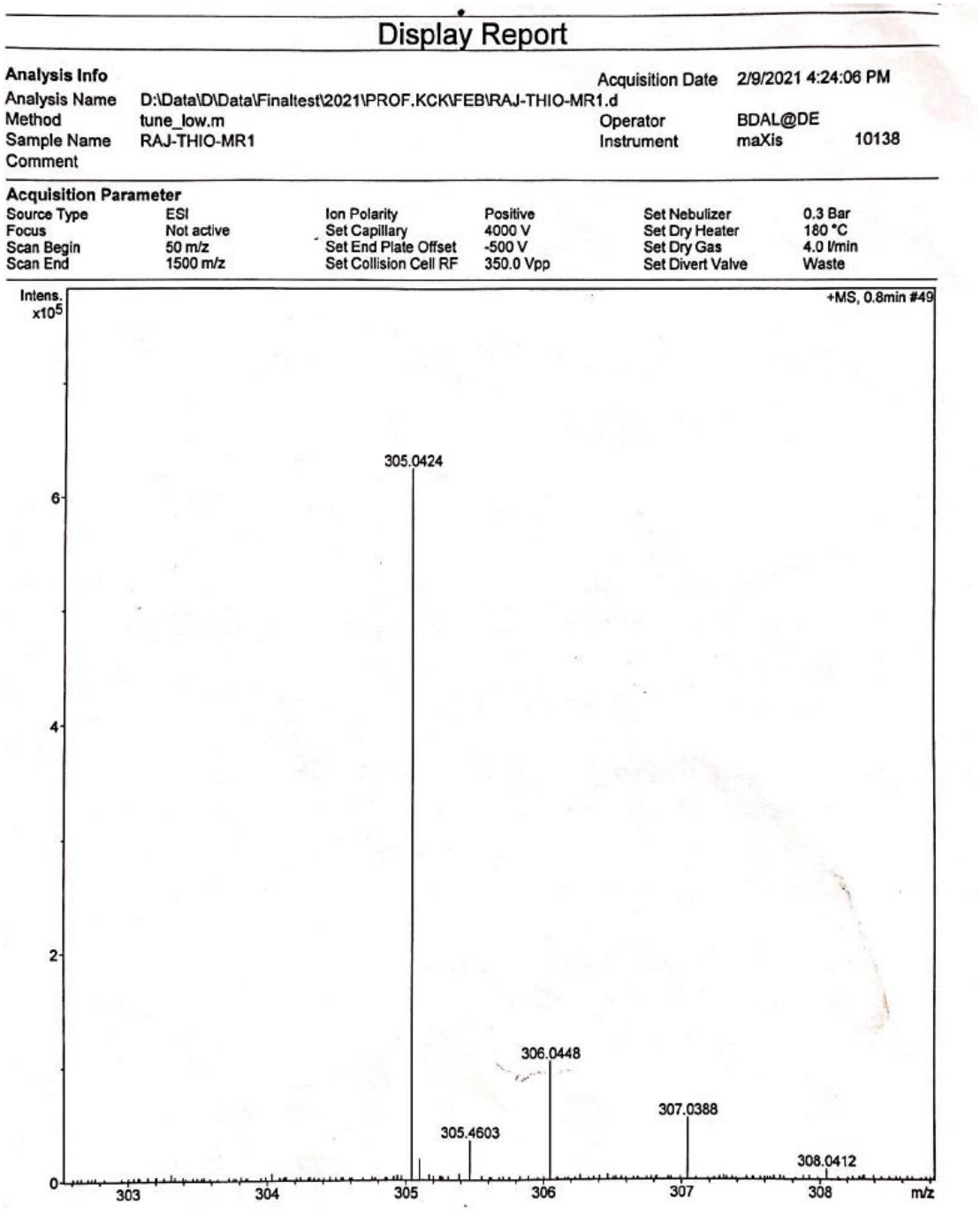

Figure S132. HRMS (ESI) of compound **12aa**

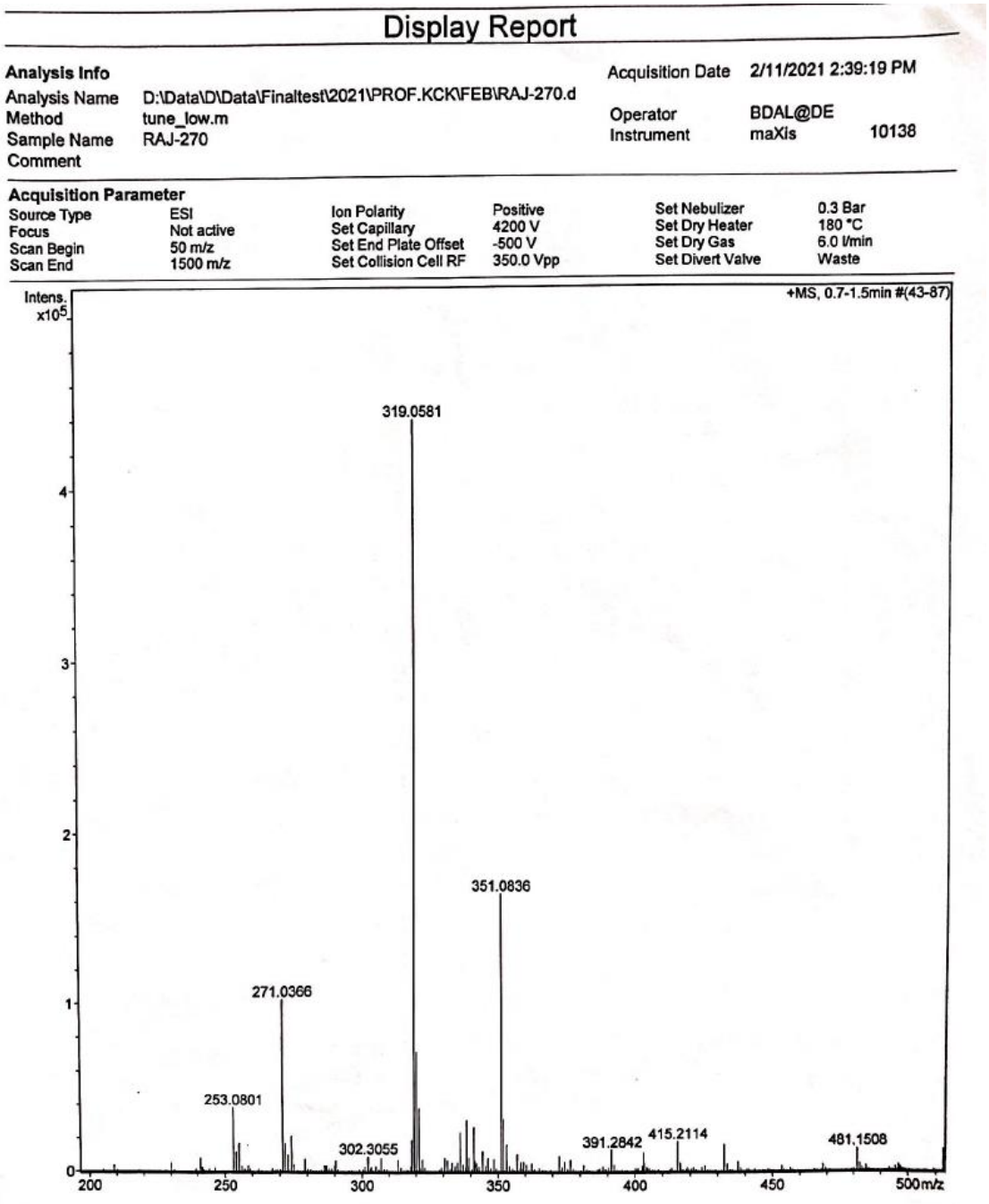

Figure S133. HRMS (ESI) of compound **12ad**

References

(1) Sandeep, K.; Reddy, A. S.; Swamy, K. C. K. Cu(I) catalysed annulation of isothiocyanates/isocyanates with 2-iodo-sulfonamides: synthesis of benzodithiazines, benzothiadiazinones, benzothiazinylidene-anilines and benzothiazolylidene-anilines. *Org. Biomol. Chem.* **2019**, *17*, 6880 (and references cited therein).

(2) Barange, D. K.; Kavala, V.; Kuo, C. -W.; Wang, C.-C.; Rajawinslin, R. R.; Donala, J.; Yao, C. -Fa. Regioselective synthesis of thiophene fused sultam derivatives via iodocyclization approach and their application towards triazole linker. *Tetrahedron Lett*. **2014**, *70*, 7598.

(3) Merlic, C. A.; You, Y.; Mclnnes, D. M.; Zechman, A. L.; Miller, M. M.; Deng, Q. Benzannulation reactions of Fischer carbene complexes for the synthesis of indolocarbazole. *Tetrahedron* **2001**, *57*, 5199.