

Electronic Supplementary Information

**Palladium-catalysed cyclisation of ynamides and propargyl tethered iodosulfonamides
with boronic acids leading to benzosultams**

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1	X-ray data for Compounds 3aa, 3ba, 3ad, 3jb and 5ah	S2-S4
2	Copies of $^1\text{H}/^{13}\text{C}\{^1\text{H}\}$ NMR spectra for compounds 3aa-3ob and 5aa-5cb	S5-S50

X-ray data:

Crystallographic data were collected on a Bruker APX-II-SMART or Oxford-Rigaku using Mo- K_{α} radiation ($\lambda = 0.71073 \text{ \AA}$) and have been deposited at the Cambridge Crystallographic Data Centre (CCDC). The structures were solved and refined by standard methods.^[1] Copies of the information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1 EZ, UK (Fax: +44-1223-336033; email: deposit@ccdc.cam.ac.uk or www.ccdc.cam.ac.uk).

(1) ORTEPs and crystal data for compounds 3aa, 3ba, 3ad, 3jb and 5ah

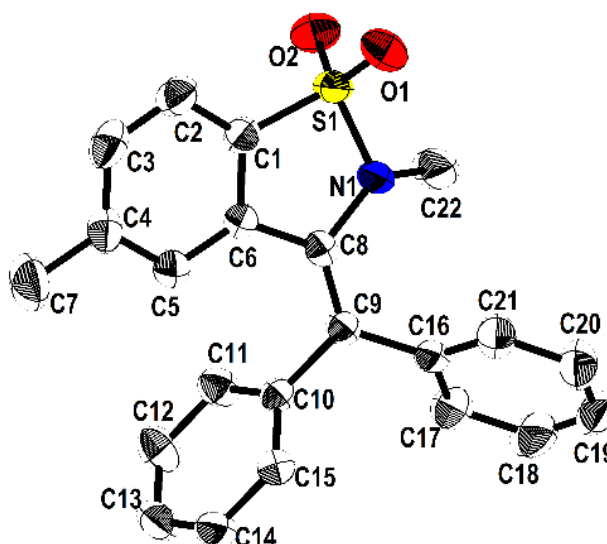


Figure S1. ORTEP (ellipsoid contour probability 50%) of compound **3aa** (CCDC no. 2082515). Colourless block, $C_{22}H_{19}NO_2S$, $M_r = 361.44$, Monoclinic, space group $P2(1)/n$, $a = 11.613(2)$, $b = 10.494(2)$, $c = 15.562(3) \text{ \AA}$, $\beta = 91.952(6)$, $V = 1895.2(6) \text{ \AA}^3$, $Z = 4$, $\mu = 0.186 \text{ mm}^{-1}$, Data/restraints/parameters: 3331/0/238. R indices ($I > 2\sigma(I)$): $R_1 = 0.0436$, wR_2 (all data) = 0.1138. Selected bond lengths [\AA] with esds in parentheses: C(6)-C(8) 1.489(3), C(8)-C(9) 1.348(3), C(9)-C(10) 1.490(3), C(9)-C(16) 1.489(3).

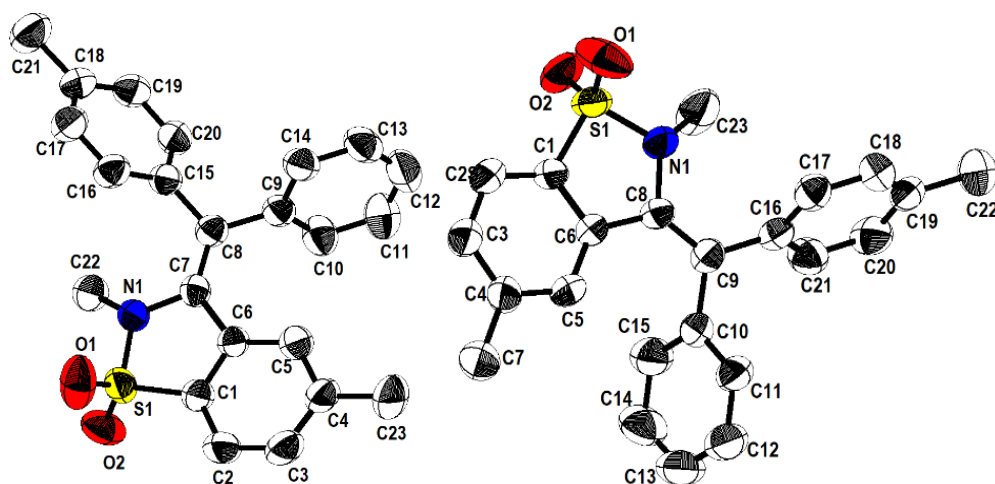


Figure S2. ORTEP (ellipsoid contour probability 50%) of compound **3ba** (CCDC no. 2082516). Colourless block, $C_{16}H_{18}N_2O_4S_2$, $M_r = 395.88$, Triclinic, space group $P-1$, $a = 9.9875(2)$, $b = 14.1732(3)$, $c = 14.8341(3)$ Å, $\alpha = 83.860(2)$, $\beta = 80.349(2)$, $\gamma = 74.882(2)$, $V = 1994.06(7)$ Å³, $Z = 2$, $\mu = 0.179$ mm⁻¹, Data/restraints/parameters: 7013/0/494. R indices ($I > 2\sigma(I)$): R1 = 0.0543, wR2 (all data) = 0.1546. Selected bond lengths [Å] with esds in parentheses: C(6)-C(7) 1.488(3), C(7)-C(8) 1.344(3), C(8)-C(9) 1.487(3), C(8)-C(15) 1.488(3), C(29)-C(30) 1.485(3), C(30)-C(31) 1.343(3), C(31)-C(32) 1.481(3), C(31)-C(39) 1.495(3).

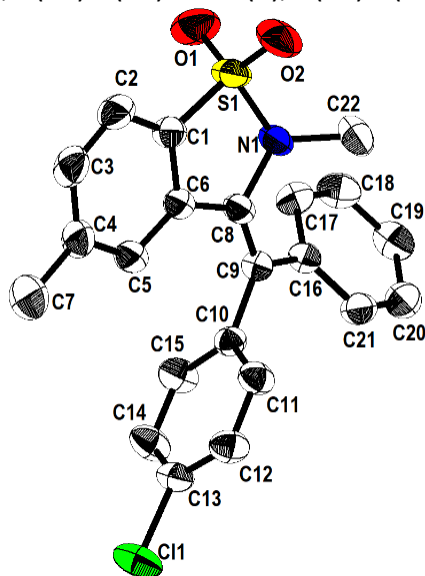


Figure S3. ORTEP (ellipsoid contour probability 50%) of compound **3ad** (CCDC no. 2082517). Colourless block, $C_{22}H_{18}ClNO_2S$, $M_r = 395.88$, Triclinic, space group $P-1$, $a = 10.0494(11)$, $b = 10.1081(10)$, $c = 10.9713(10)$ Å, $\alpha = 104.997(3)$, $\beta = 112.432(3)$, $\gamma = 95.785(4)$, $V = 969.88(17)$ Å³, $Z = 2$, $\mu = 0.321$ mm⁻¹, Data/restraints/parameters: 3388/0/247. R indices ($I > 2\sigma(I)$): R1 = 0.0343, wR2 (all data) = 0.0918. Selected bond lengths [Å] with esds in parentheses: C(6)-C(8) 1.481(2), C(8)-C(9) 1.353(2), C(9)-C(10) 1.494(2), C(9)-C(16) 1.484(2).

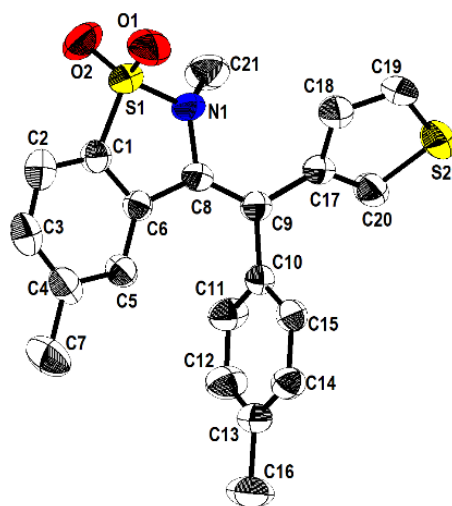


Figure S4. ORTEP (ellipsoid contour probability 50%) of compound **3jb** (CCDC no. 2082518).. Colourless block, $C_{21}H_{19}NO_2S_2$, $M_r = 381.49$, Triclinic, space group $P-1$, $a = 9.8974(2)$, $b = 9.9589(3)$, $c = 11.7524(3)$ Å, $\alpha = 90.477(2)$, $\beta = 106.658(2)$, $\gamma = 116.051(2)$, $V = 984.84(4)$ Å³, $Z = 2$, $\mu = 0.285$ mm⁻¹, Data/restraints/parameters: 3470/0/239. R indices ($I > 2\sigma(I)$): $R_1 = 0.0457$, wR_2 (all data) = 0.1339. Selected bond lengths [Å] with esds in parentheses: C(6)-C(8) 1.479(3), C(8)-C(9) 1.348(3), C(9)-C(10) 1.487(3), C(9)-C(17) 1.472(3).

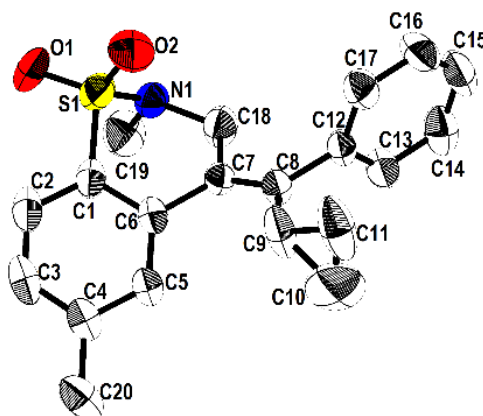


Figure S5. ORTEP (ellipsoid contour probability 50%) of compound **5ah** (CCDC no. 2082519). Colourless block, $C_{20}H_{21}NO_2S$, $M_r = 339.44$, Monoclinic, space group $P12(1)/c1$, $a = 20.5737(8)$, $b = 6.0343(2)$, $c = 14.7085(8)$ Å, $\beta = 104.444(4)$, $V = 1768.31(13)$ Å³, $Z = 4$, $\mu = 0.194$ mm⁻¹, Data/restraints/parameters: 3112/0/220. R indices ($I > 2\sigma(I)$): $R_1 = 0.0555$, wR_2 (all data) = 0.1692. Selected bond lengths [Å] with esds in parentheses: C(8)-C(9) 1.472(4), C(7)-C(18) 1.528(4), C(7)-C(8) 1.336(3), C(8)-C(12) 1.496(3).

- (1) (a) G. M. Sheldrick, *SADABS, Siemens Area Detector Absorption Correction*; University of Göttingen: Göttingen, Germany, 1996; b) Sheldrick, G. M. *SHELX-97, A Program for Crystal Structure Solution and Refinement*; University of Göttingen, *SHELXTL NT Crystal Structure Analysis Package*, version 5.10; Bruker AXS: Madison, WI, 1999.

(2) Copies of $^1\text{H}/^{13}\text{C}$ $\{^1\text{H}\}/^{19}\text{F}$ NMR spectra for compounds 3aa-3la and 5aa-5cb

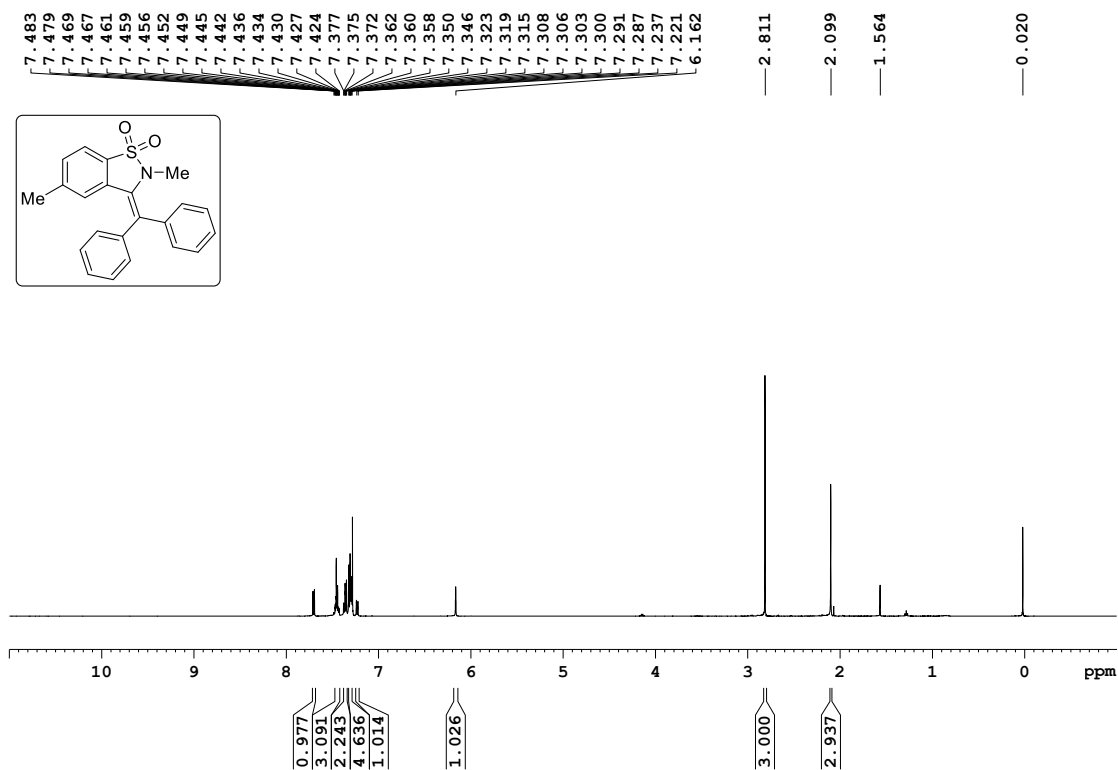


Figure S6. ^1H NMR spectrum of compound 3aa

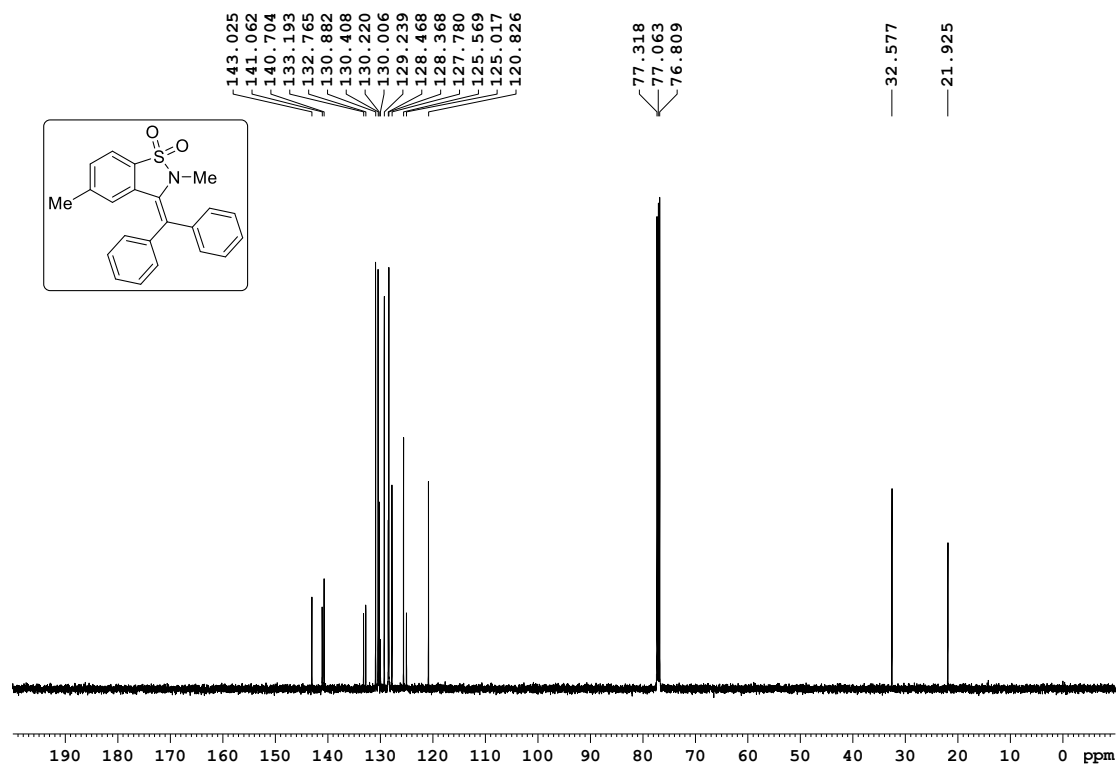


Figure S7. ^{13}C NMR spectrum of compound 3aa

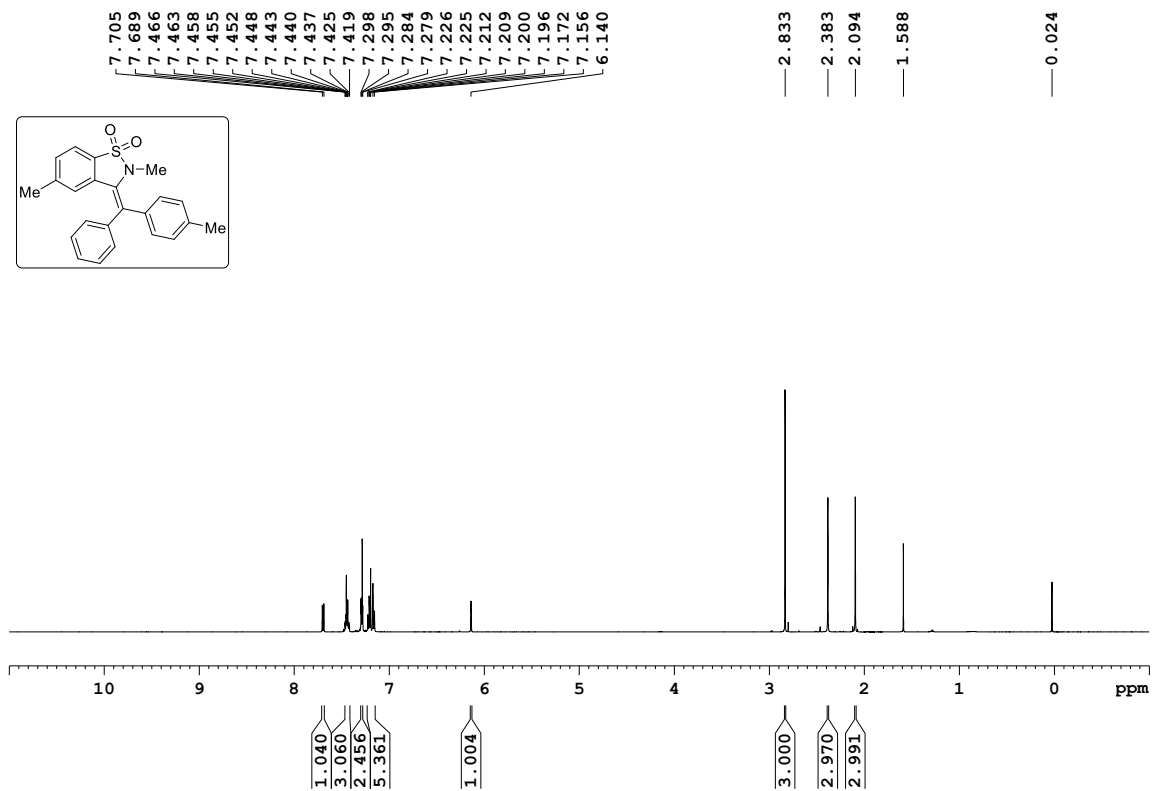


Figure S8. ¹H NMR spectrum of compound 3ba

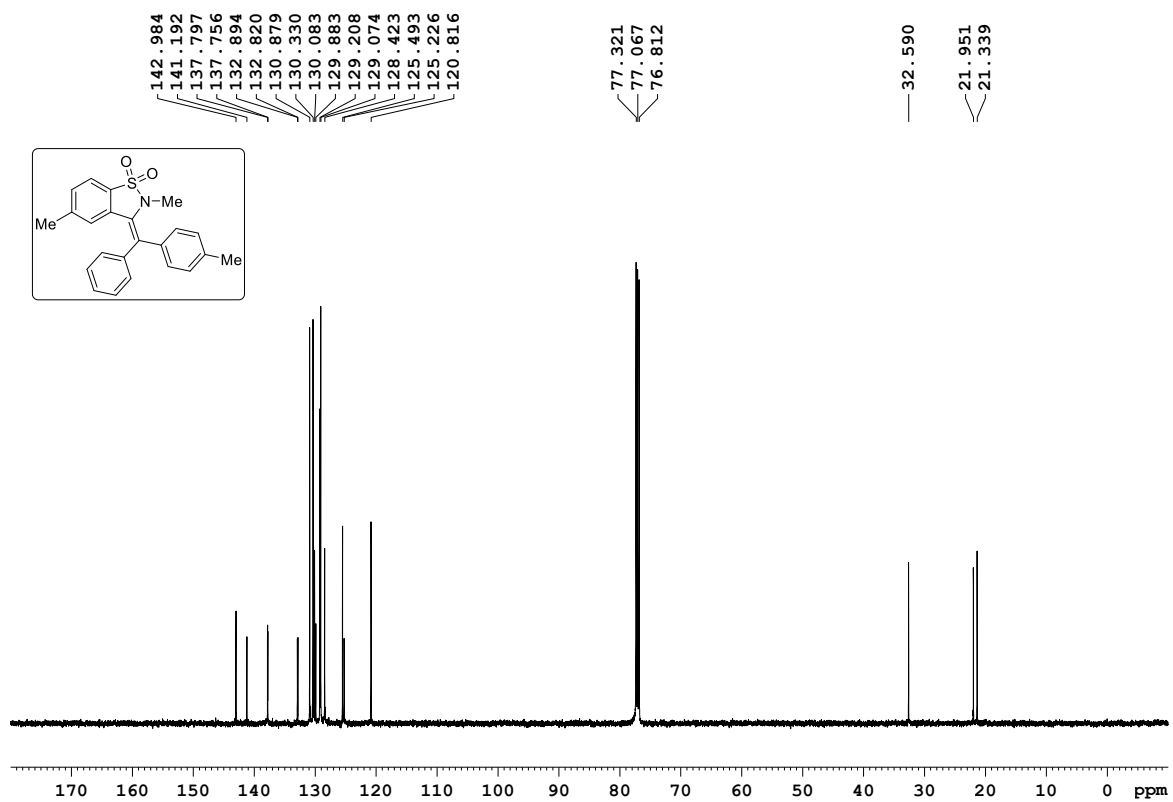


Figure S9. ¹³C NMR spectrum of compound 3ba

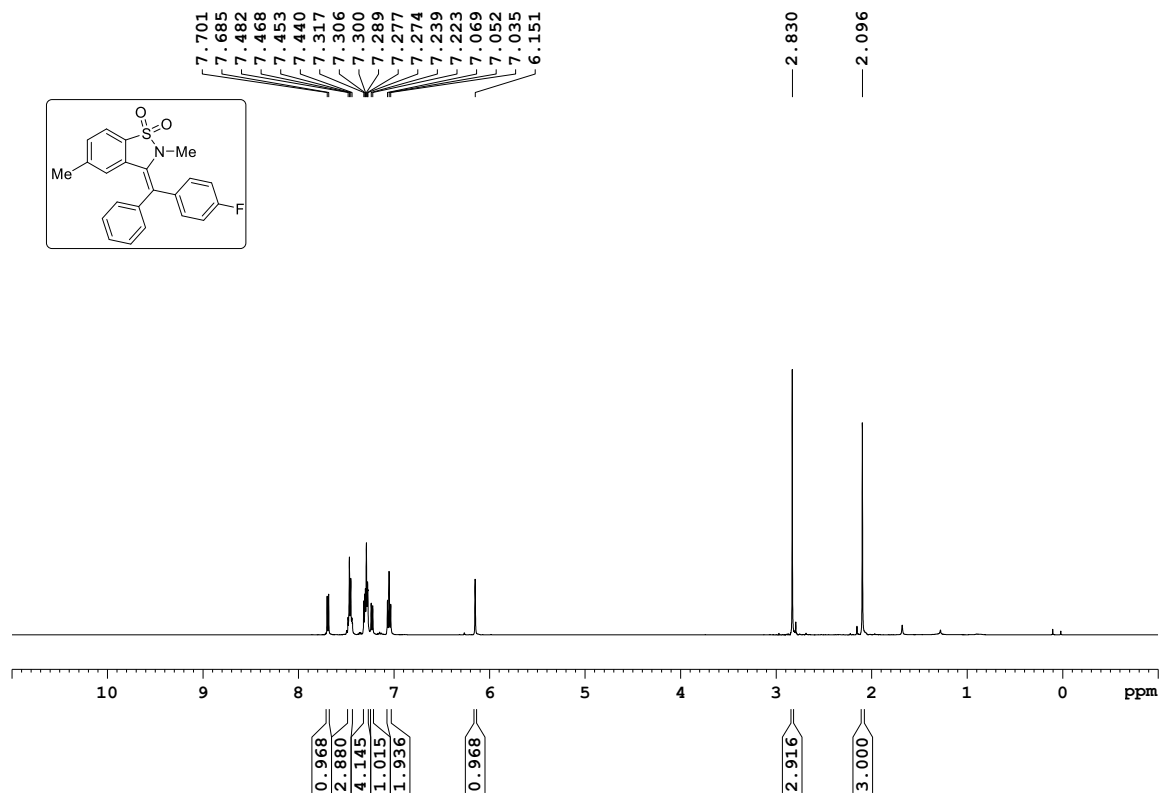


Figure S10. ¹H NMR spectrum of compound 3da

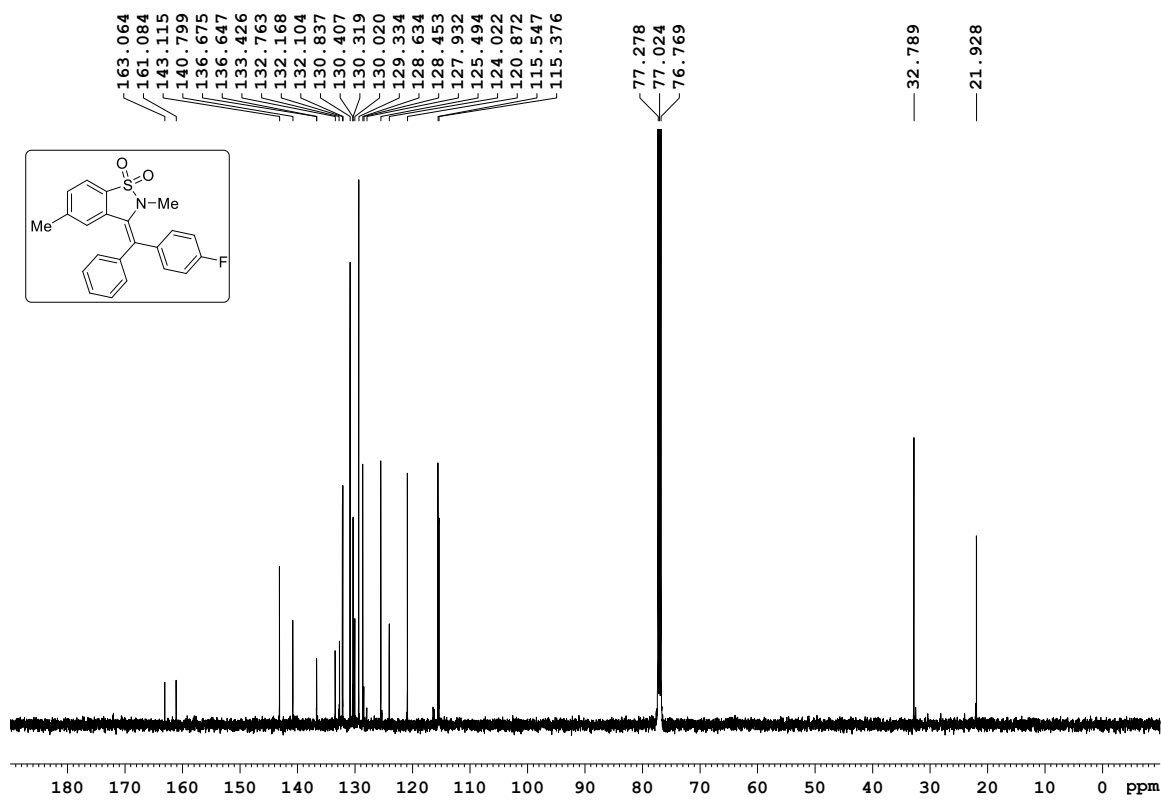


Figure S11. ¹³C NMR spectrum of compound 3da

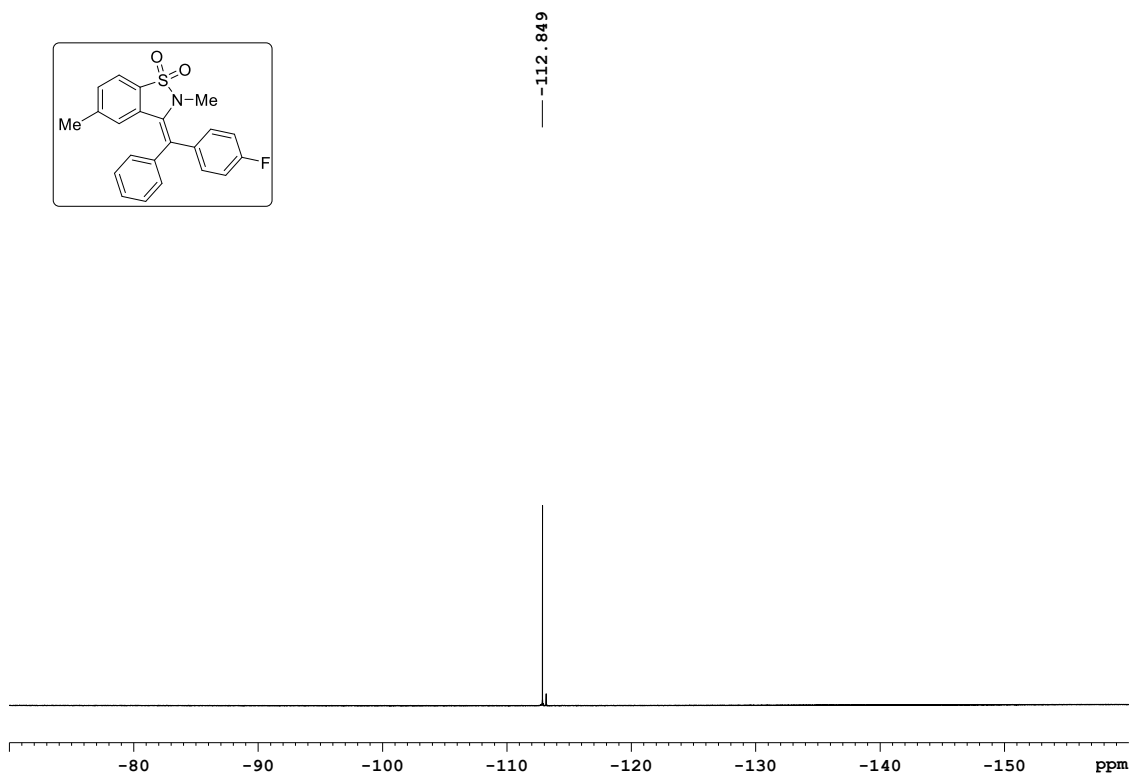


Figure S12. ^{19}F NMR spectrum of compound 3da

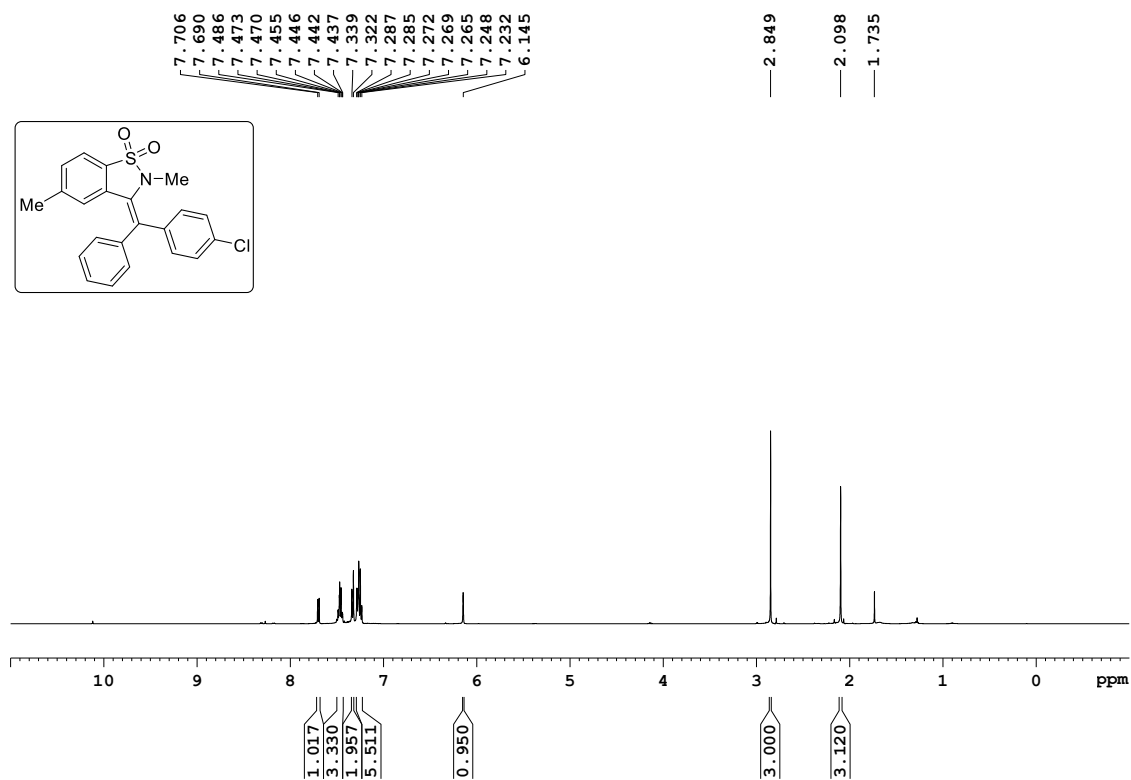


Figure S13. ¹H NMR spectrum of compound 3ea

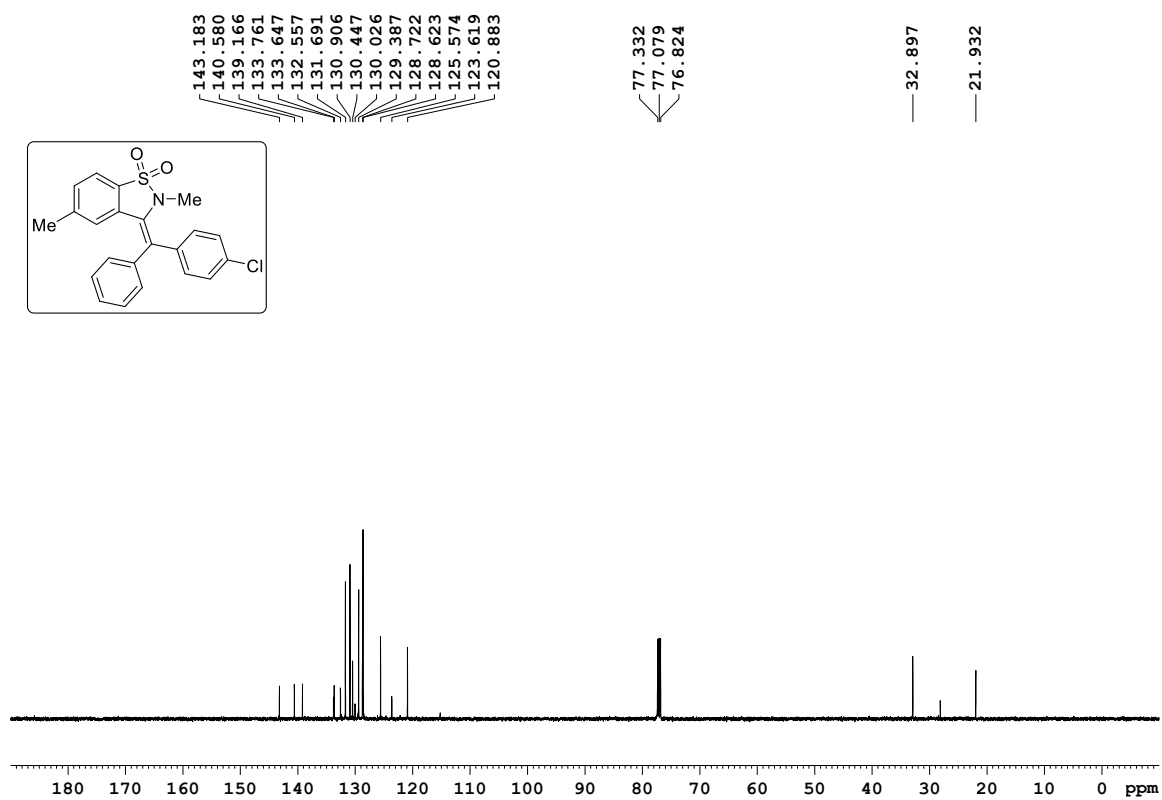


Figure S14. ¹³C NMR spectrum of compound 3ea

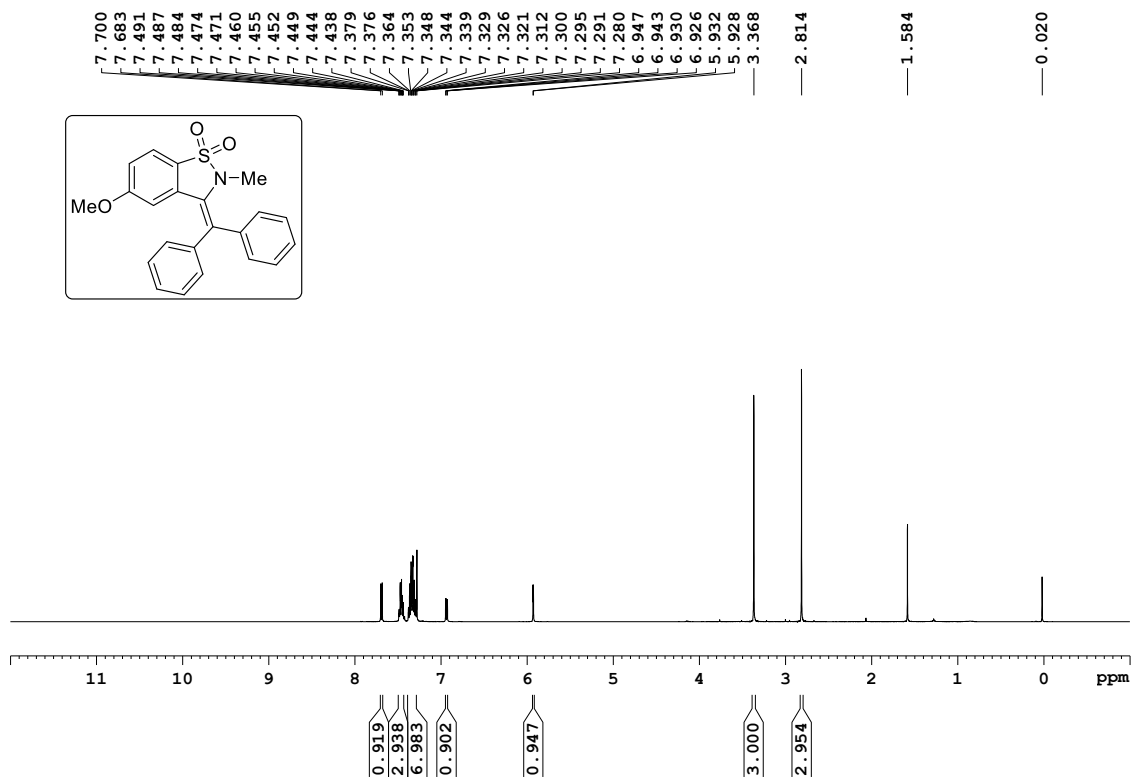


Figure S15. ^1H NMR spectrum of compound 3fa

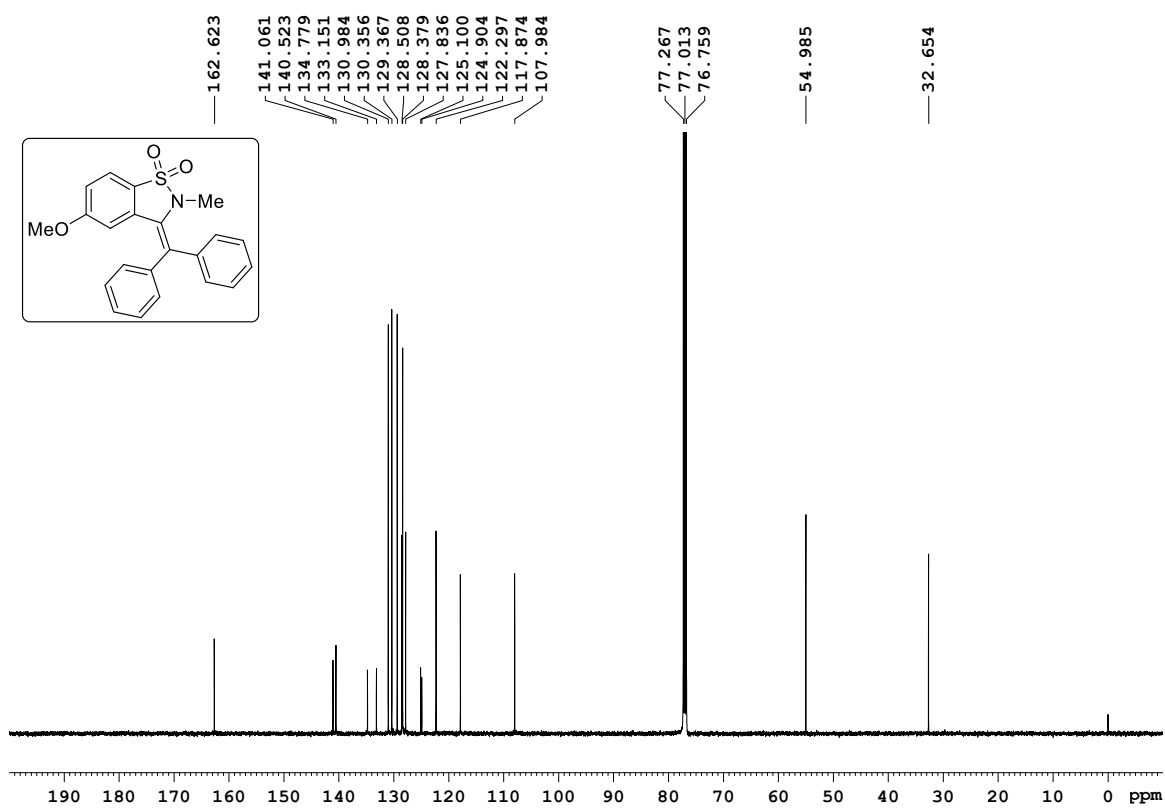


Figure S16. ^{13}C NMR spectrum of compound 3fa

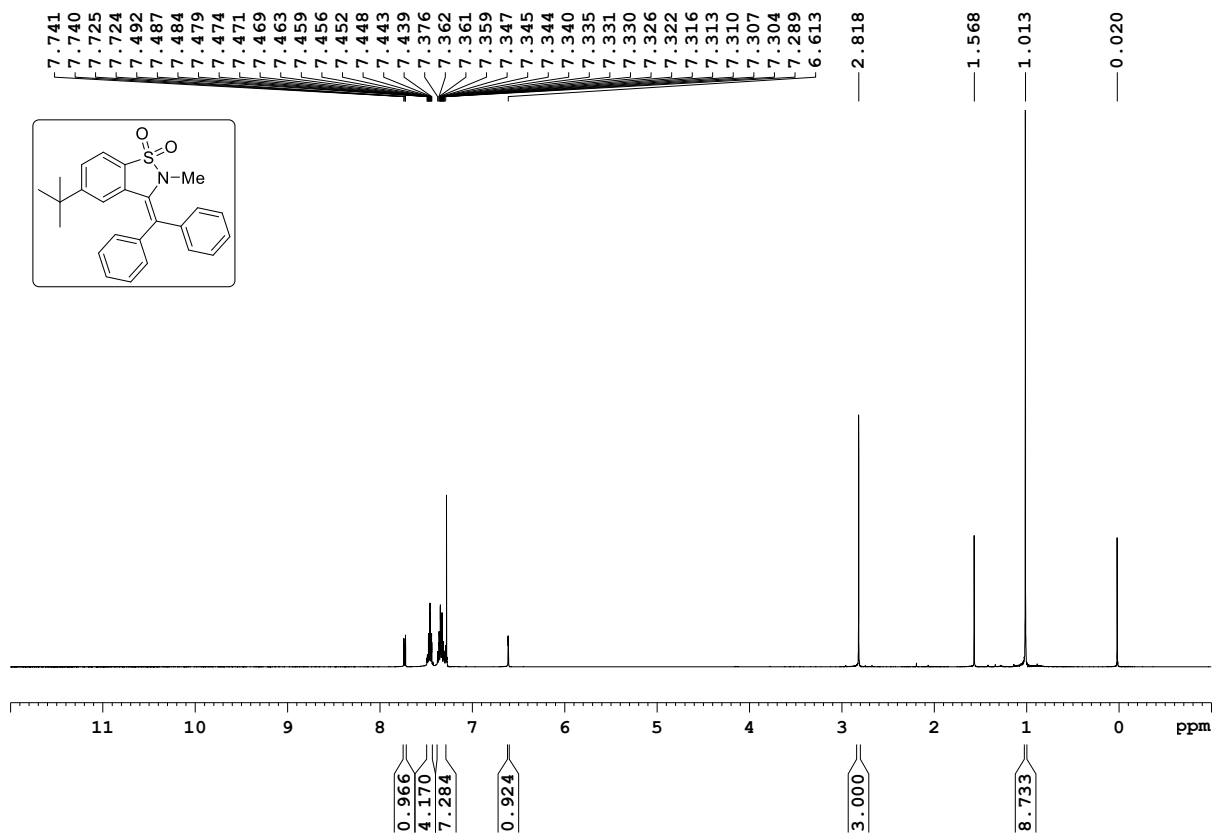


Figure S17. ¹H NMR spectrum of compound 3ga

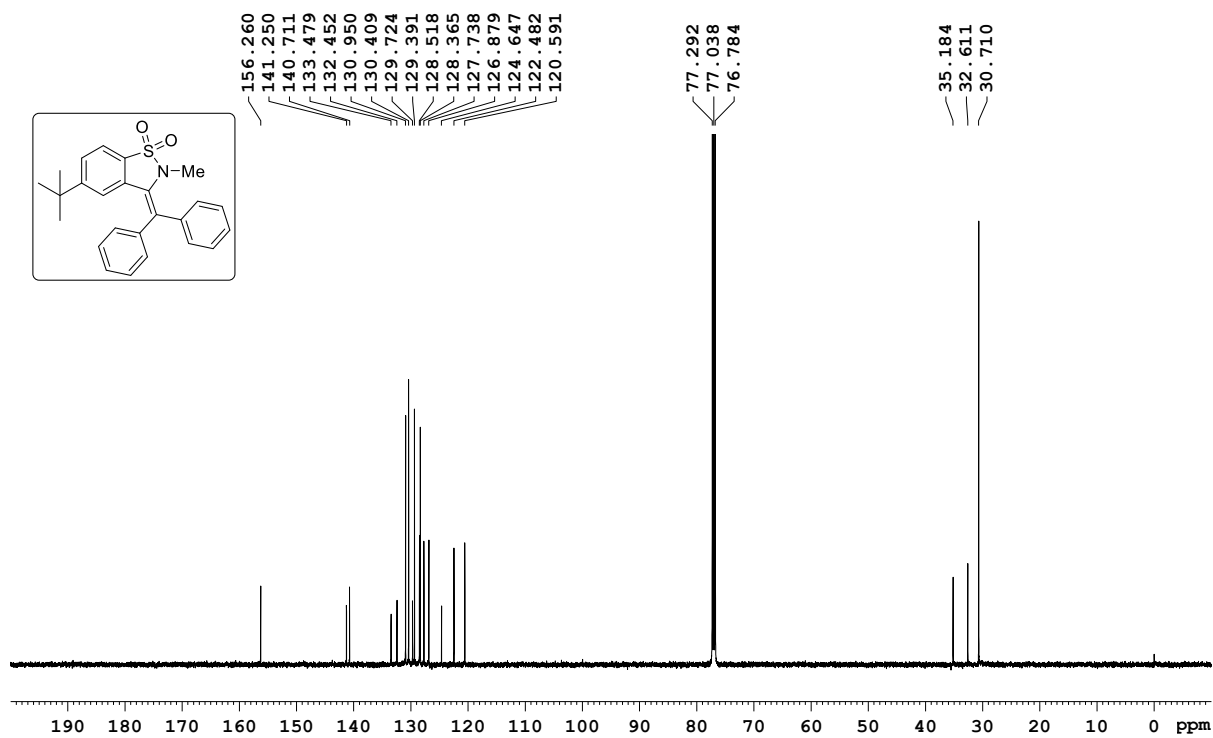


Figure S18. ¹³C NMR spectrum of compound 3ga

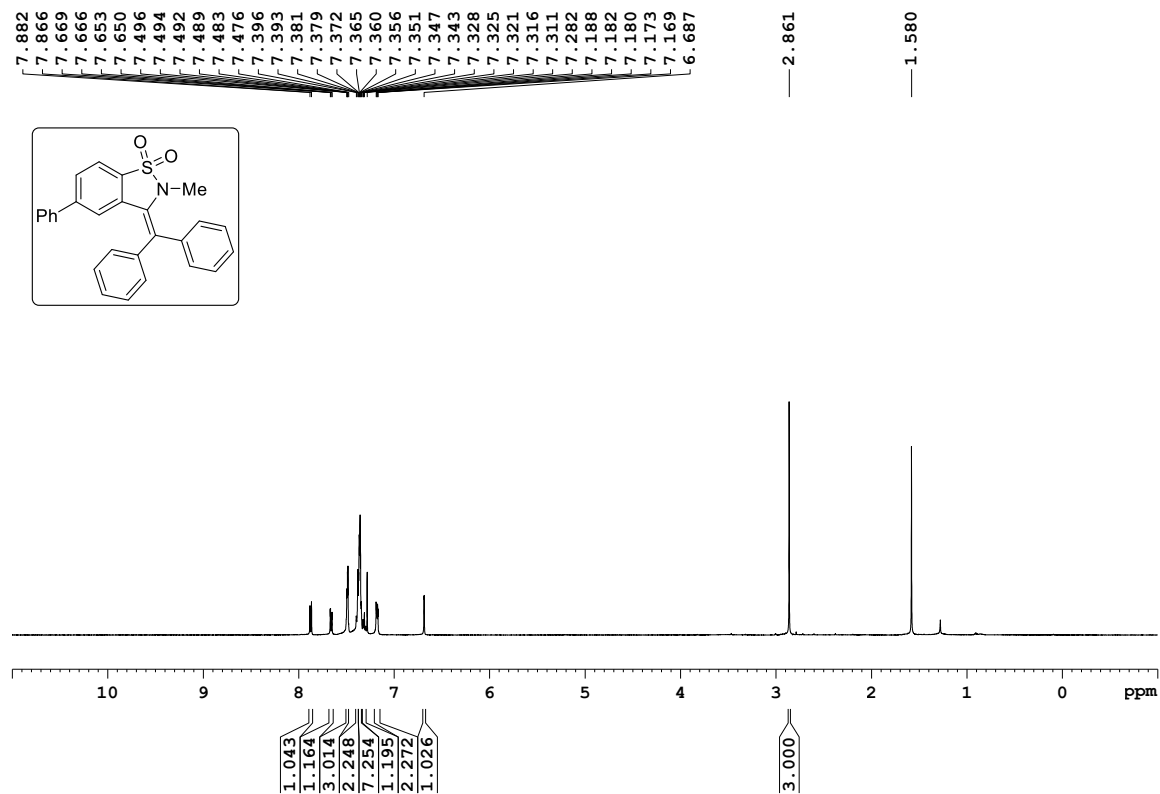


Figure S19. ^1H NMR spectrum of compound 3ha

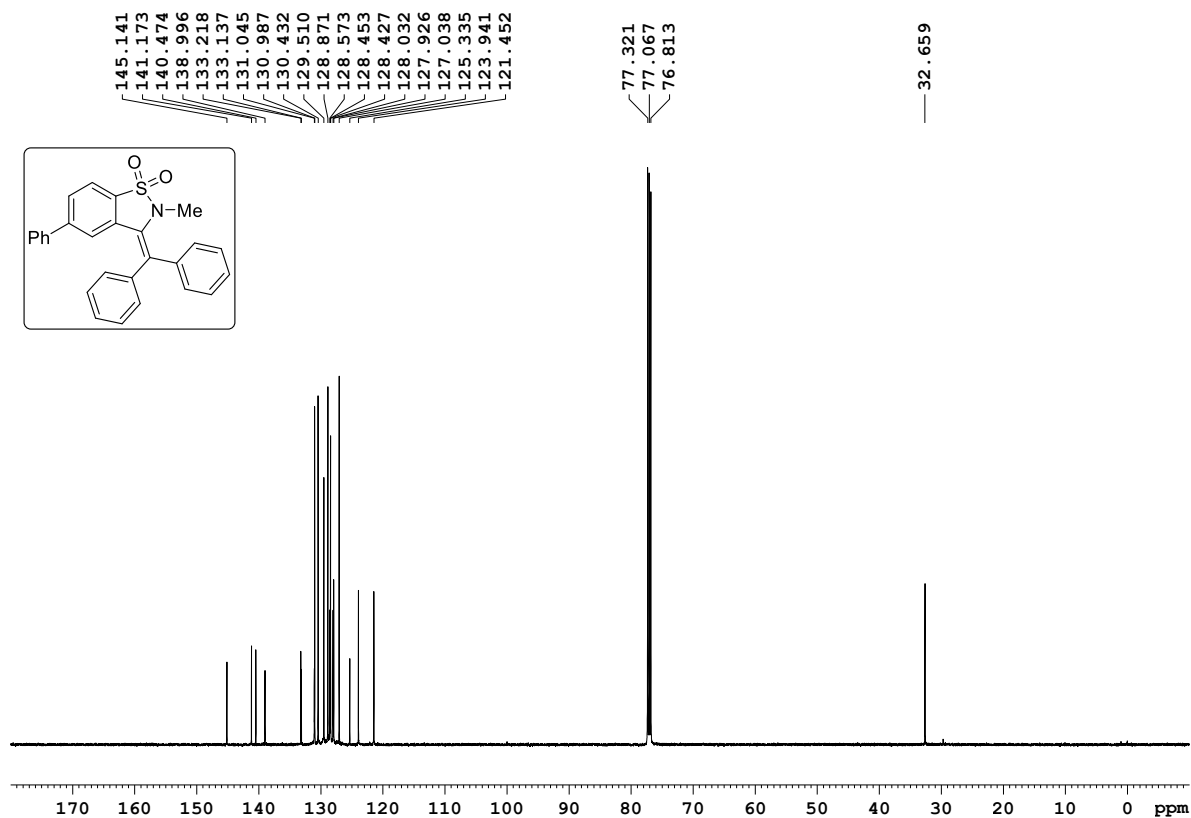


Figure S20. ^{13}C NMR spectrum of compound 3ha

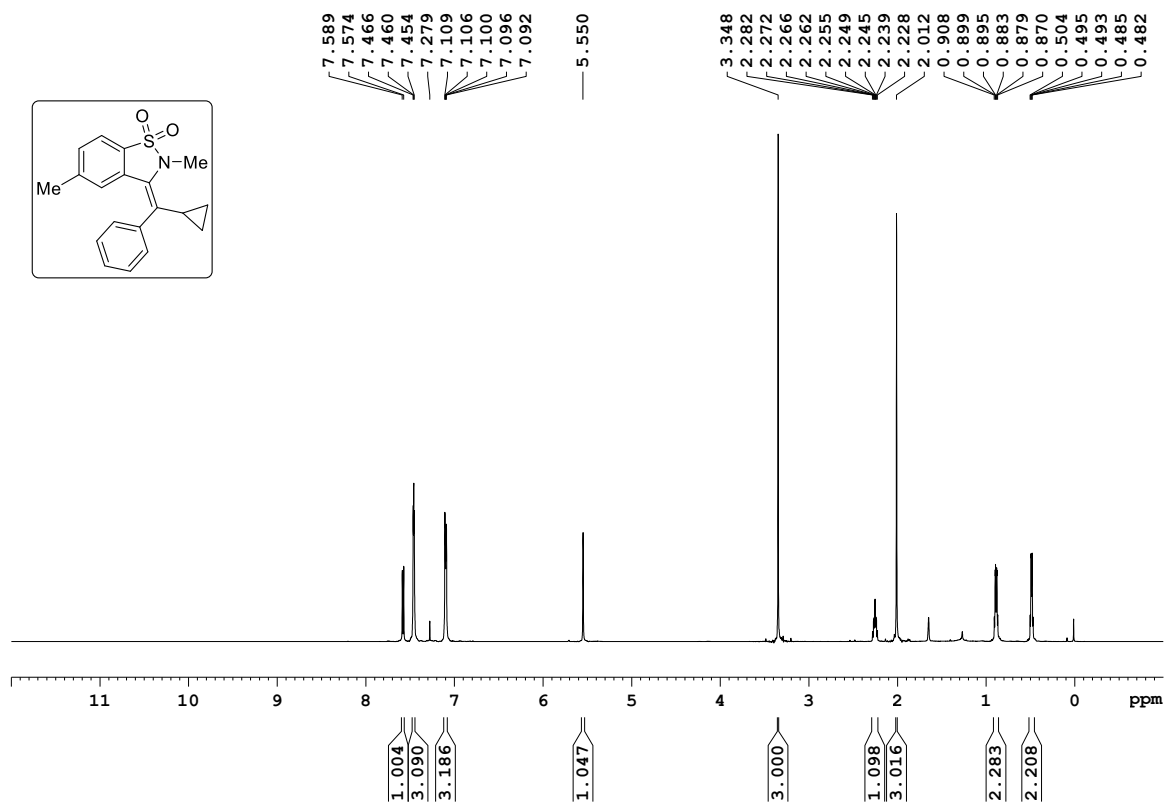


Figure S21. ¹H NMR spectrum of compound **3ia**

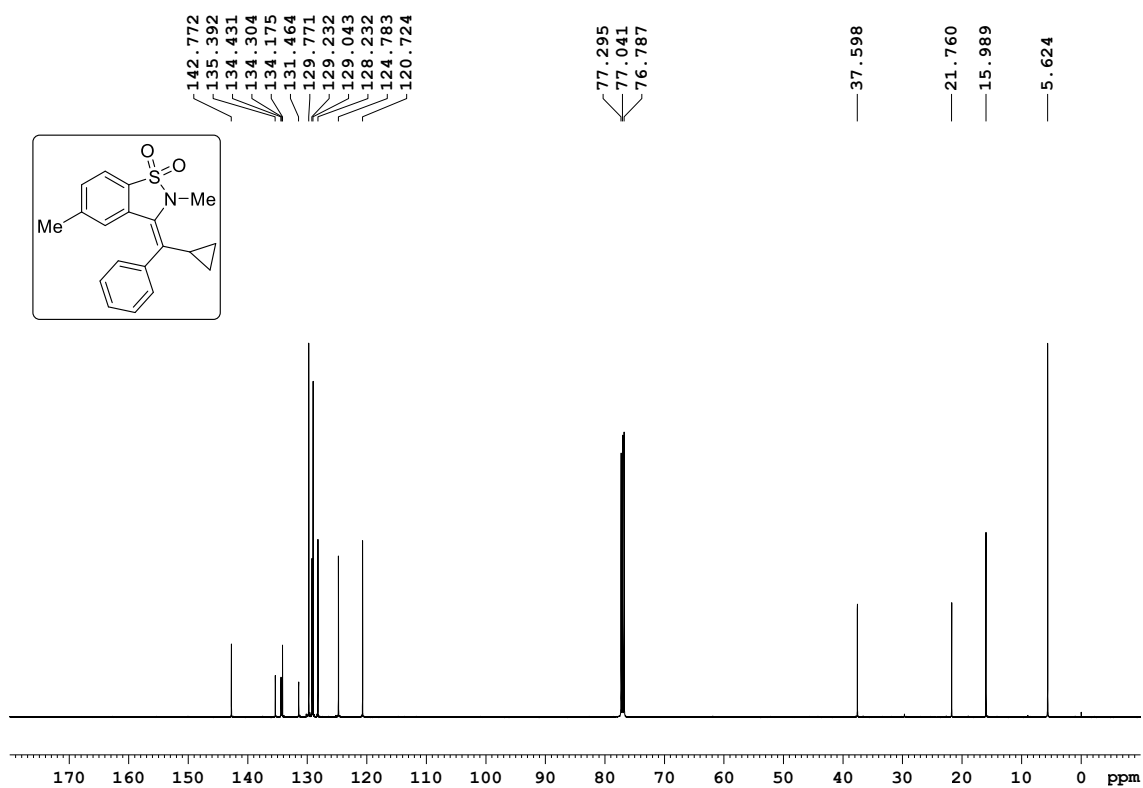


Figure S22. ¹³C NMR spectrum of compound **3ia**

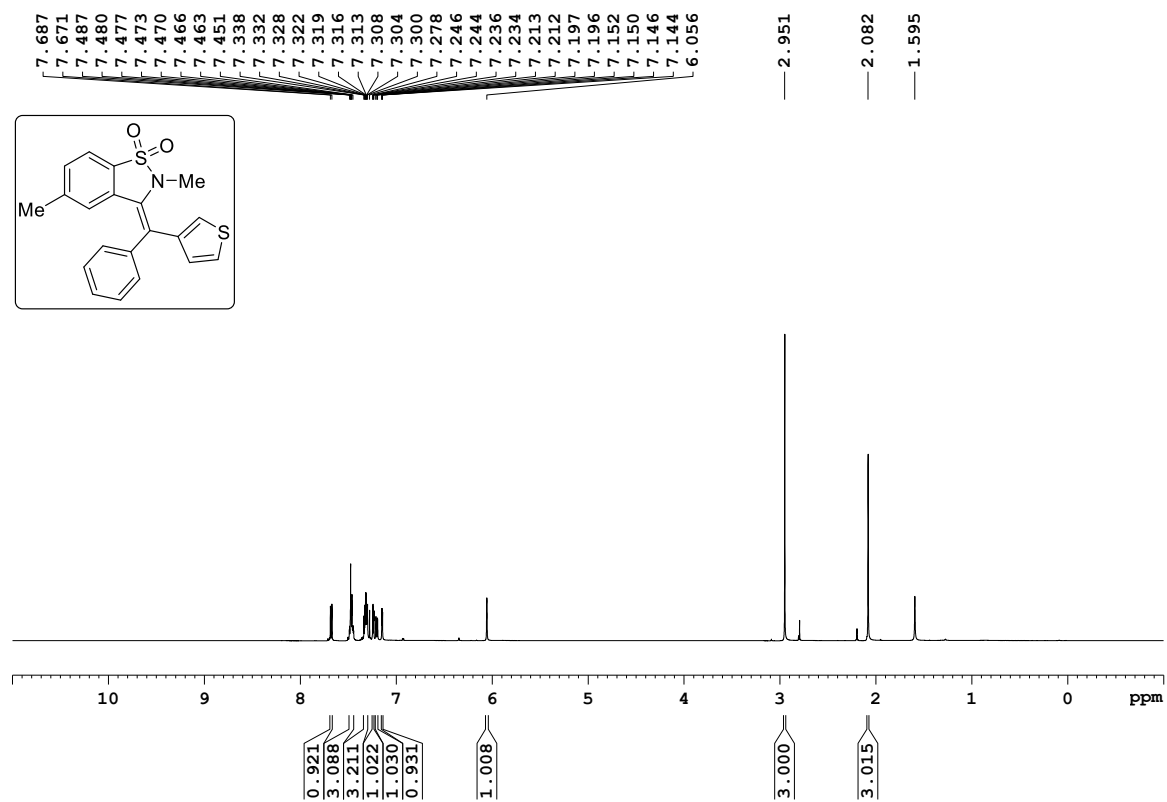


Figure S23. ^1H NMR spectrum of compound **3ja** (purity >96%)

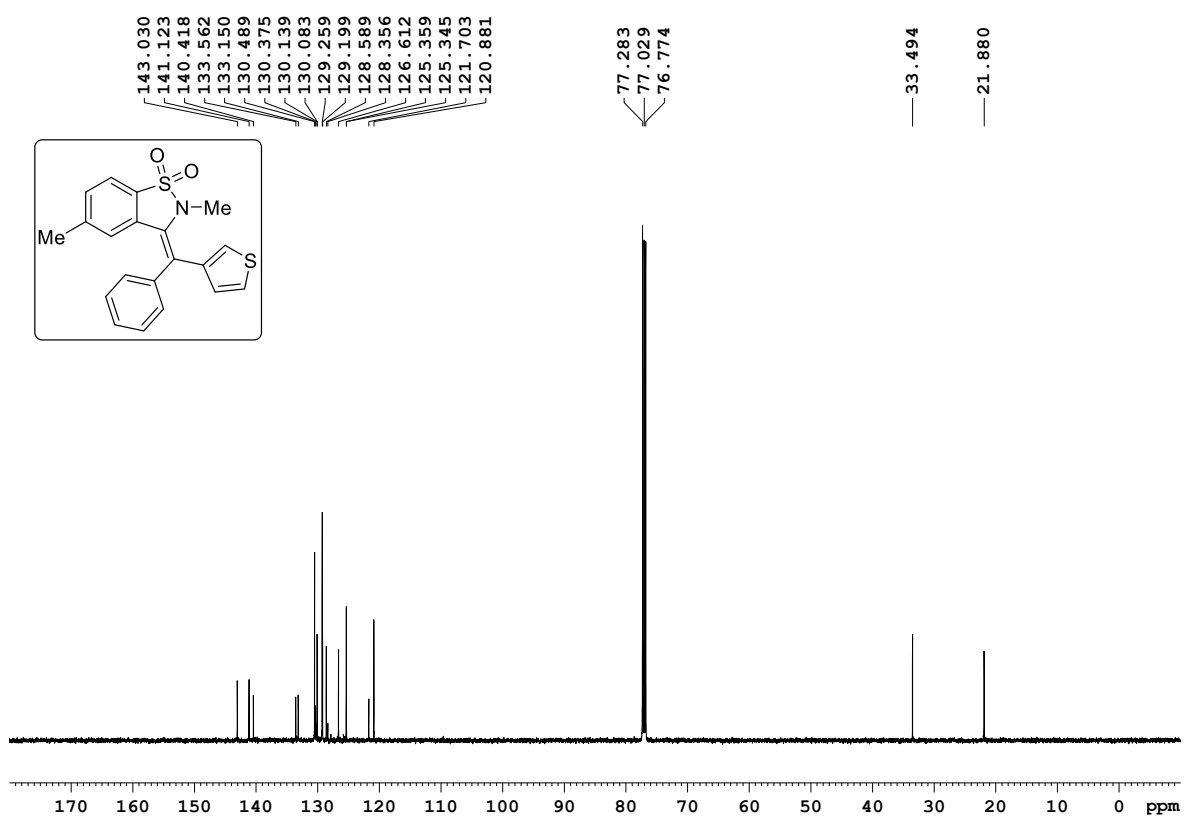


Figure S24. ^{13}C NMR spectrum of compound **3ja** (purity >96%)

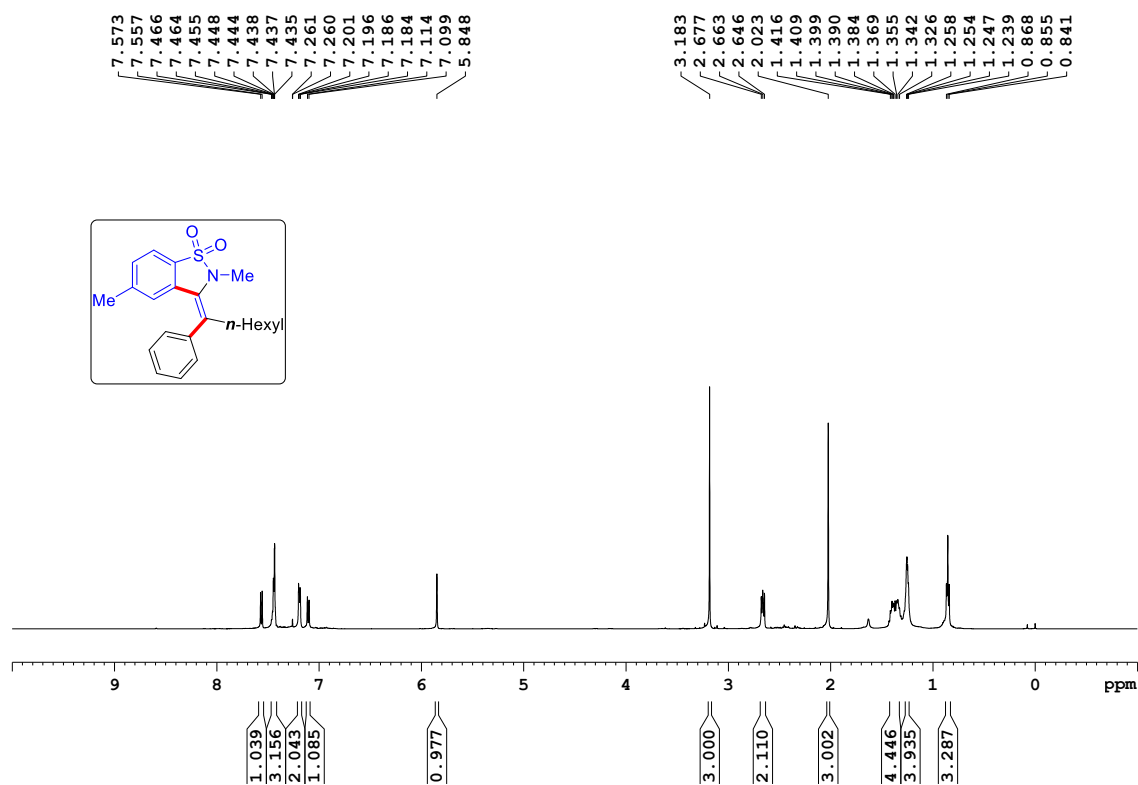


Figure S25. ¹H NMR spectrum of compound 3ka

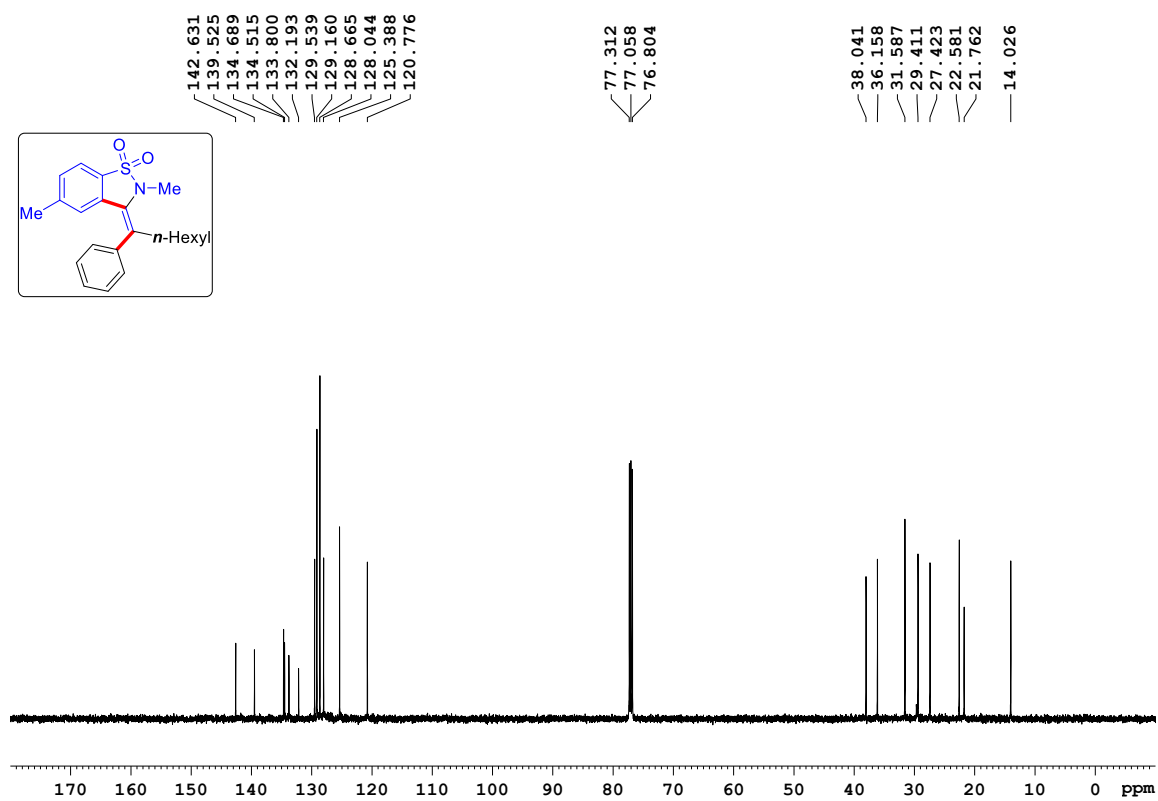


Figure S26. ¹³C NMR spectrum of compound 3ka

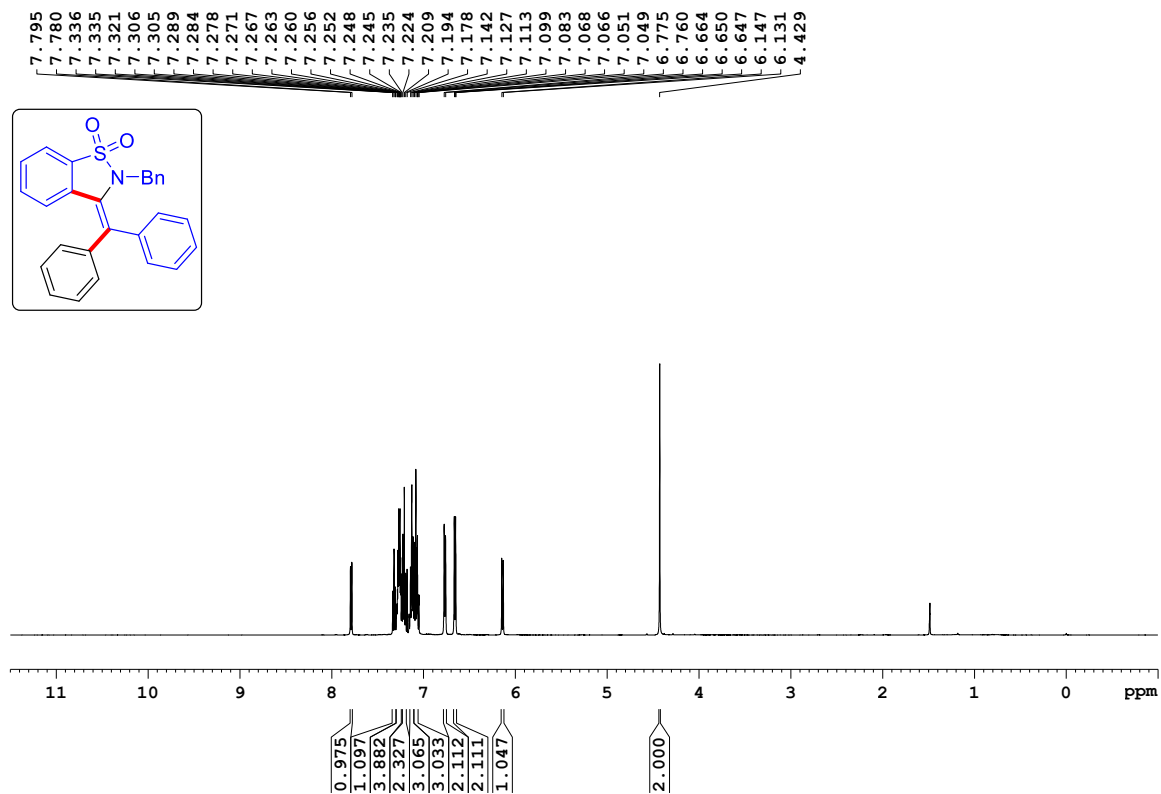


Figure S27. ¹H NMR spectrum of compound 3ma

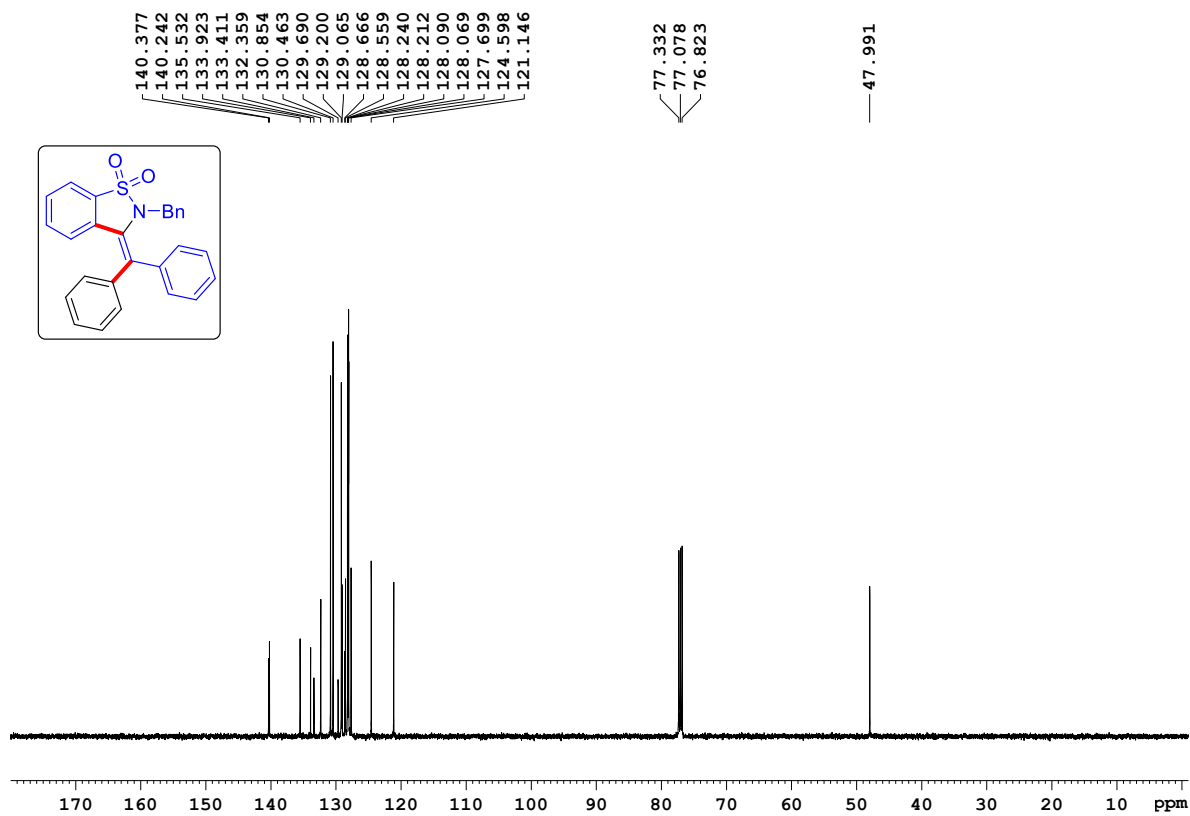


Figure S28. ¹³C NMR spectrum of compound 3ma

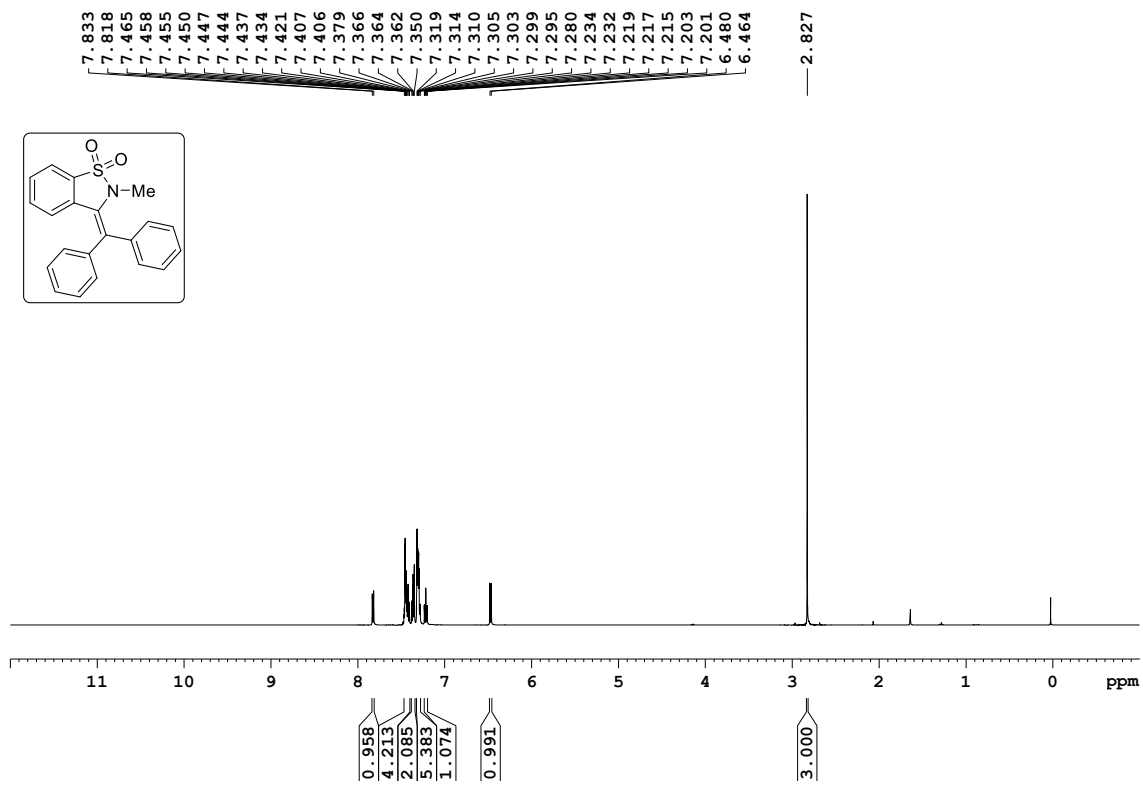


Figure S29. ¹H NMR spectrum of compound 3na

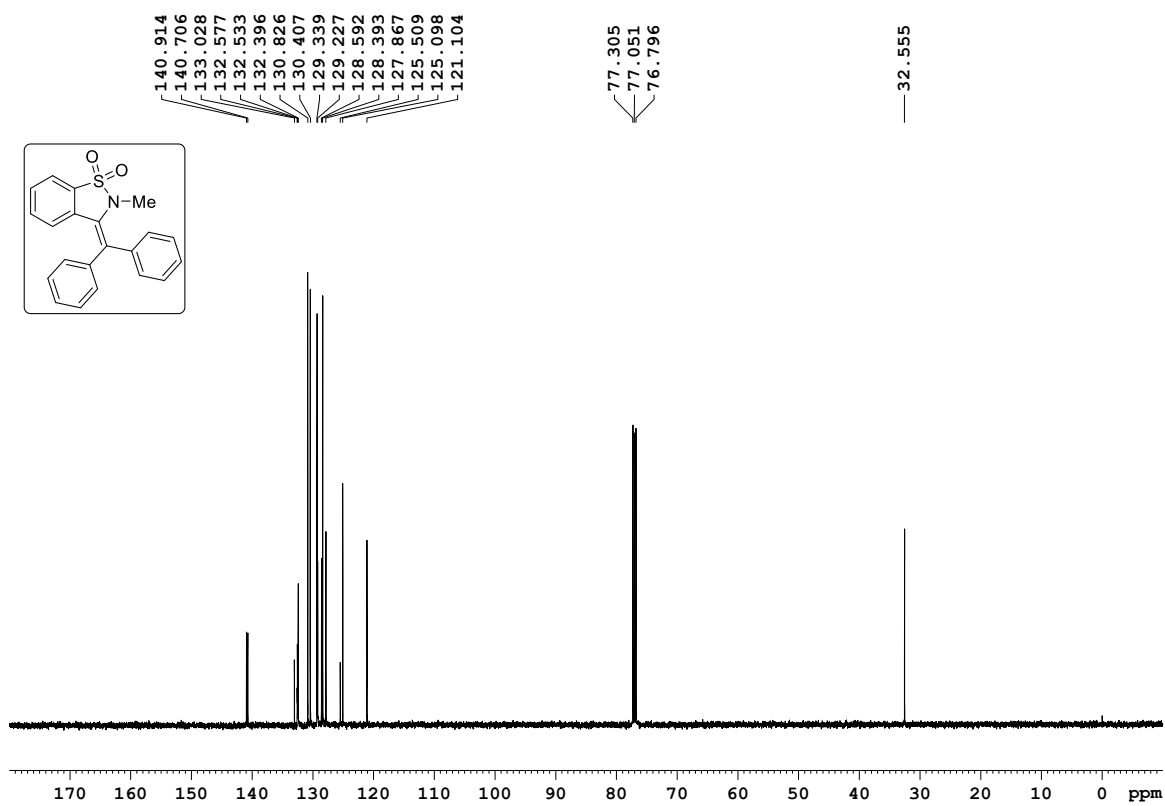


Figure S30. ¹³C NMR spectrum of compound 3na

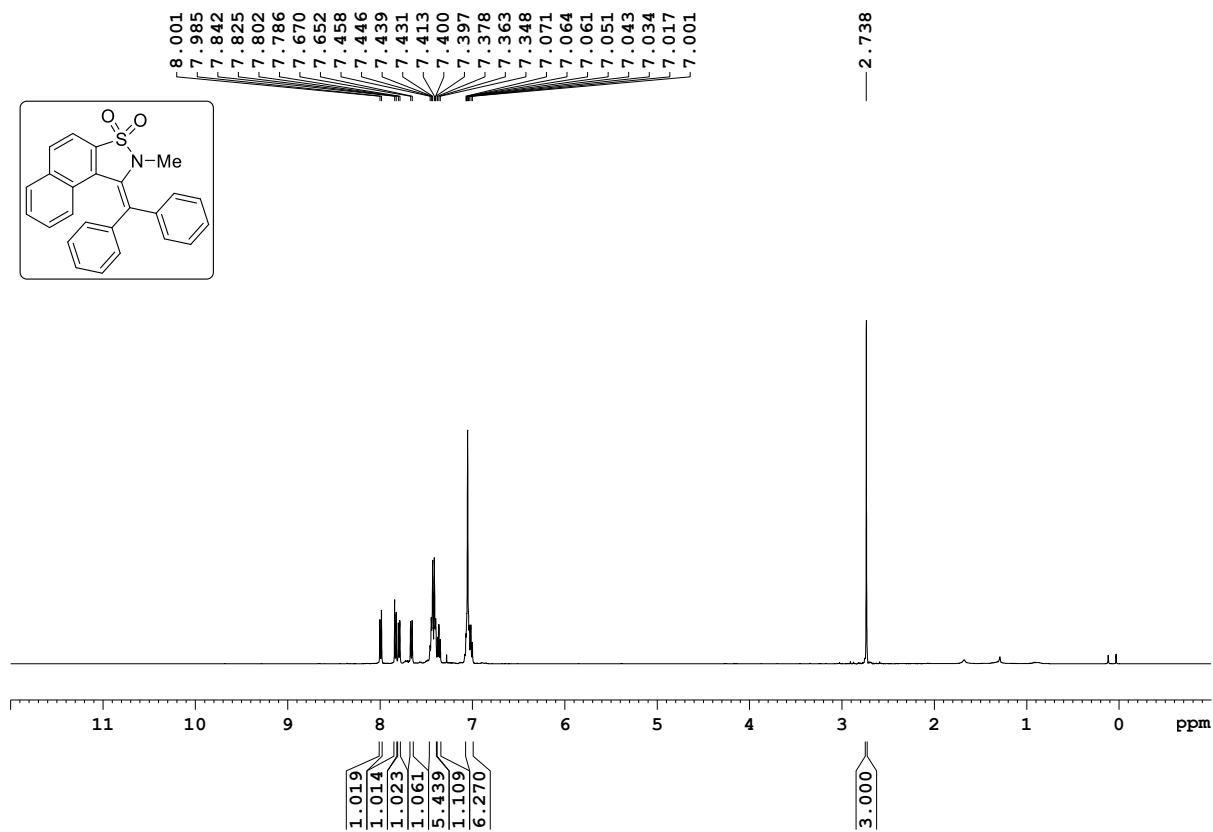


Figure S31. ¹H NMR spectrum of compound 30a

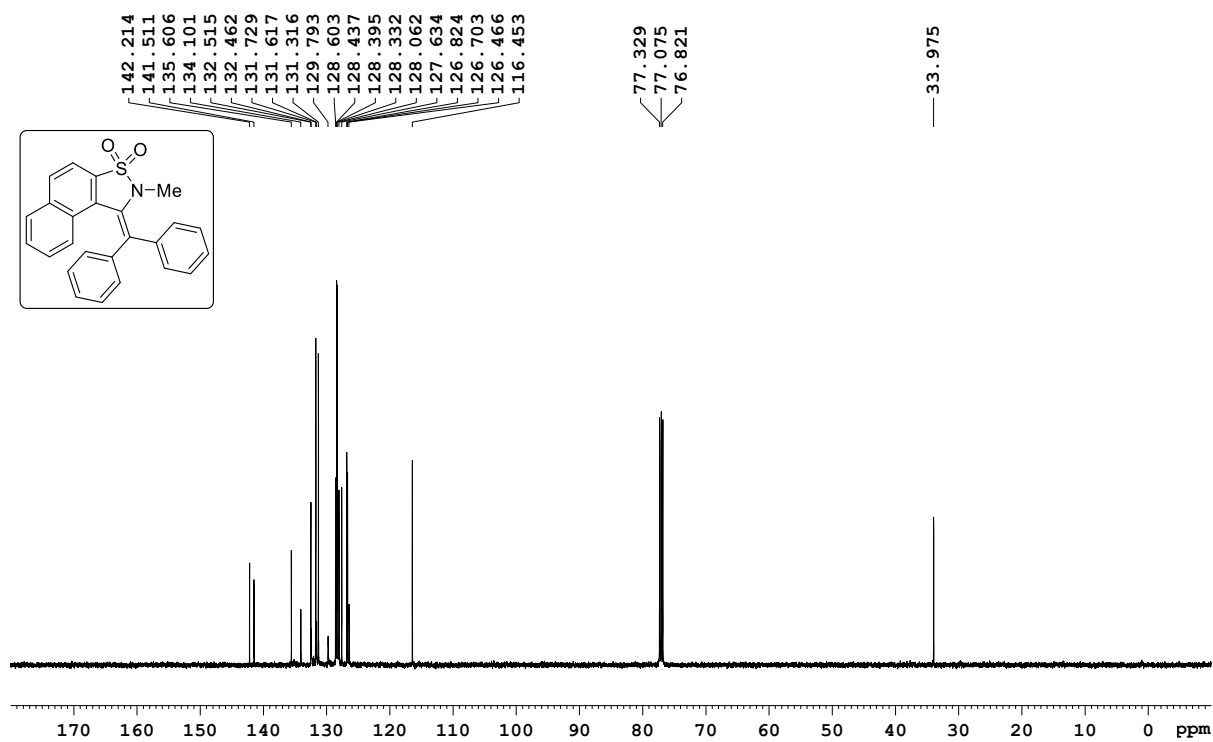


Figure S32. ¹³C NMR spectrum of compound 30a

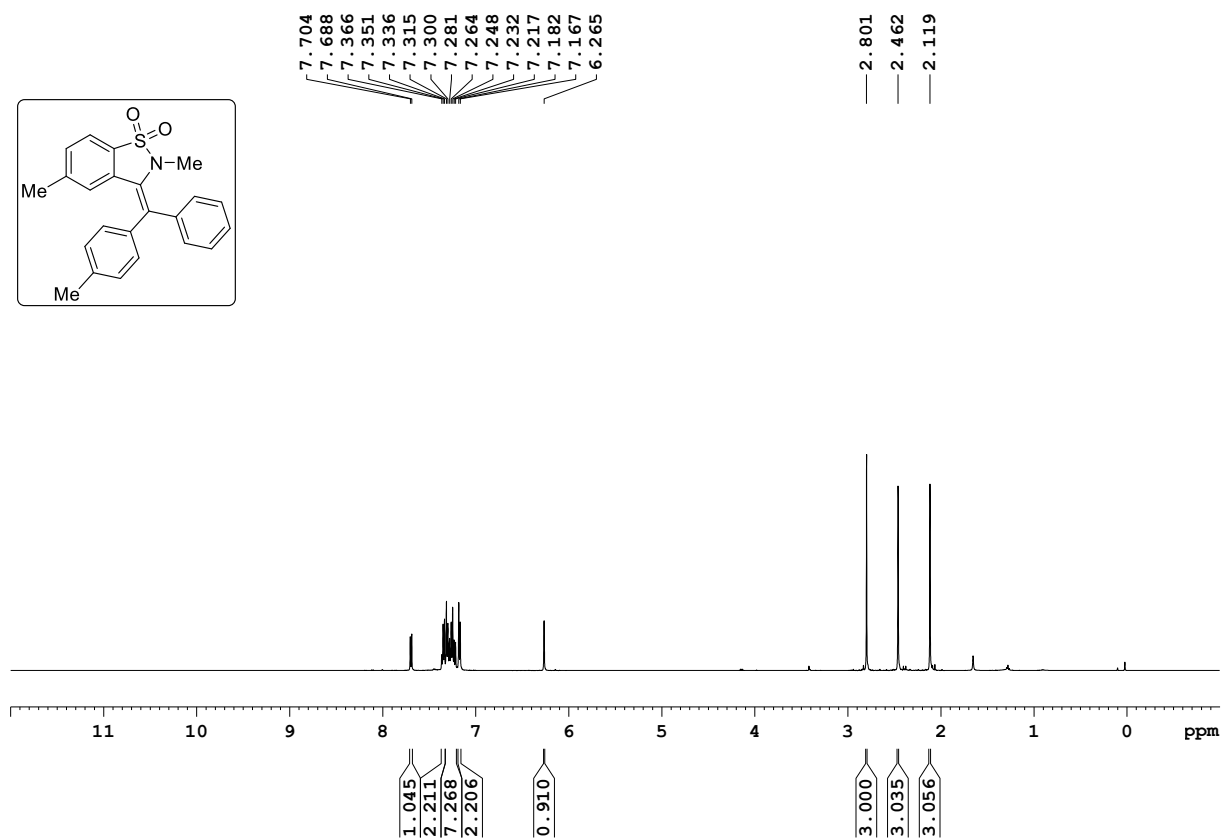


Figure S33. ^1H NMR spectrum of compound **3ab**

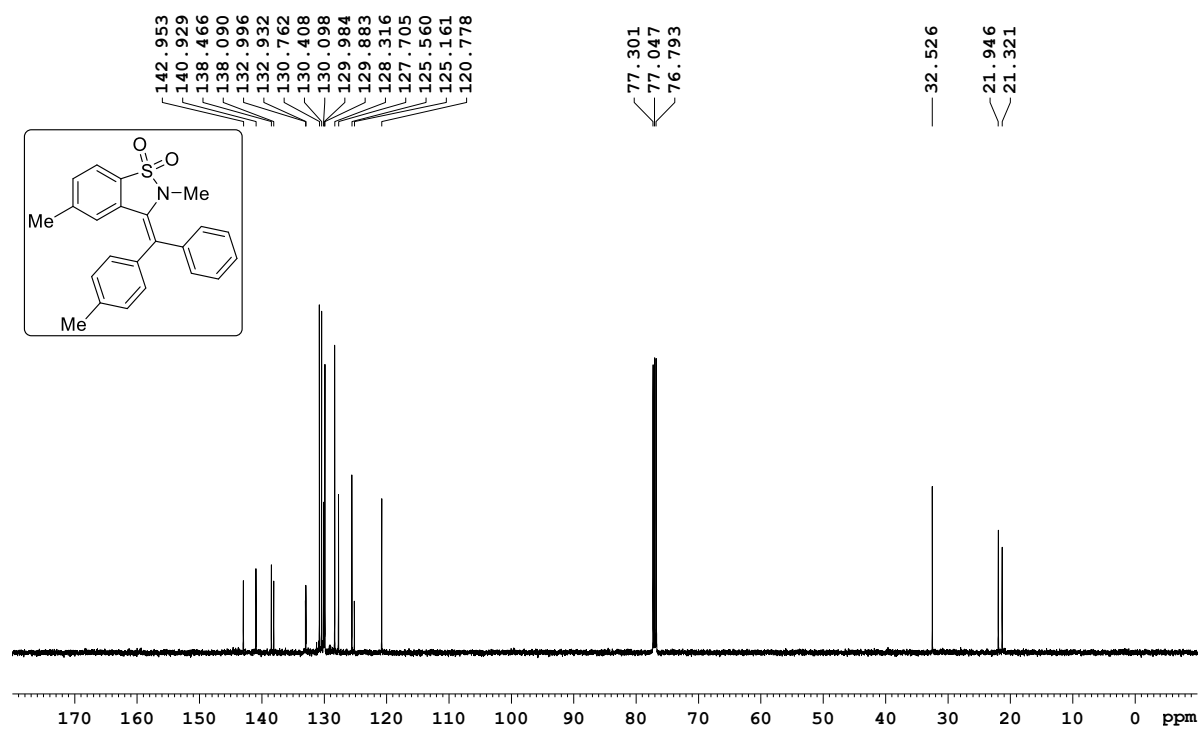


Figure S34. ^{13}C NMR spectrum of compound **3ab**

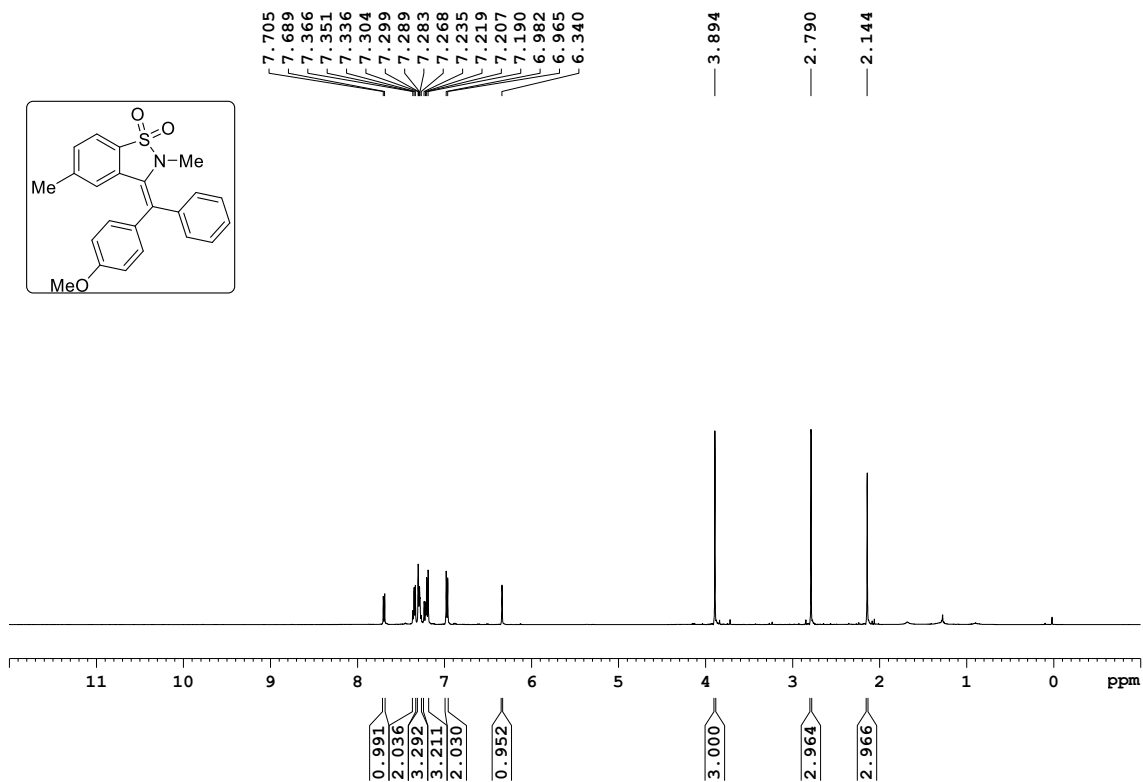


Figure S35. ¹H NMR spectrum of compound 3ac

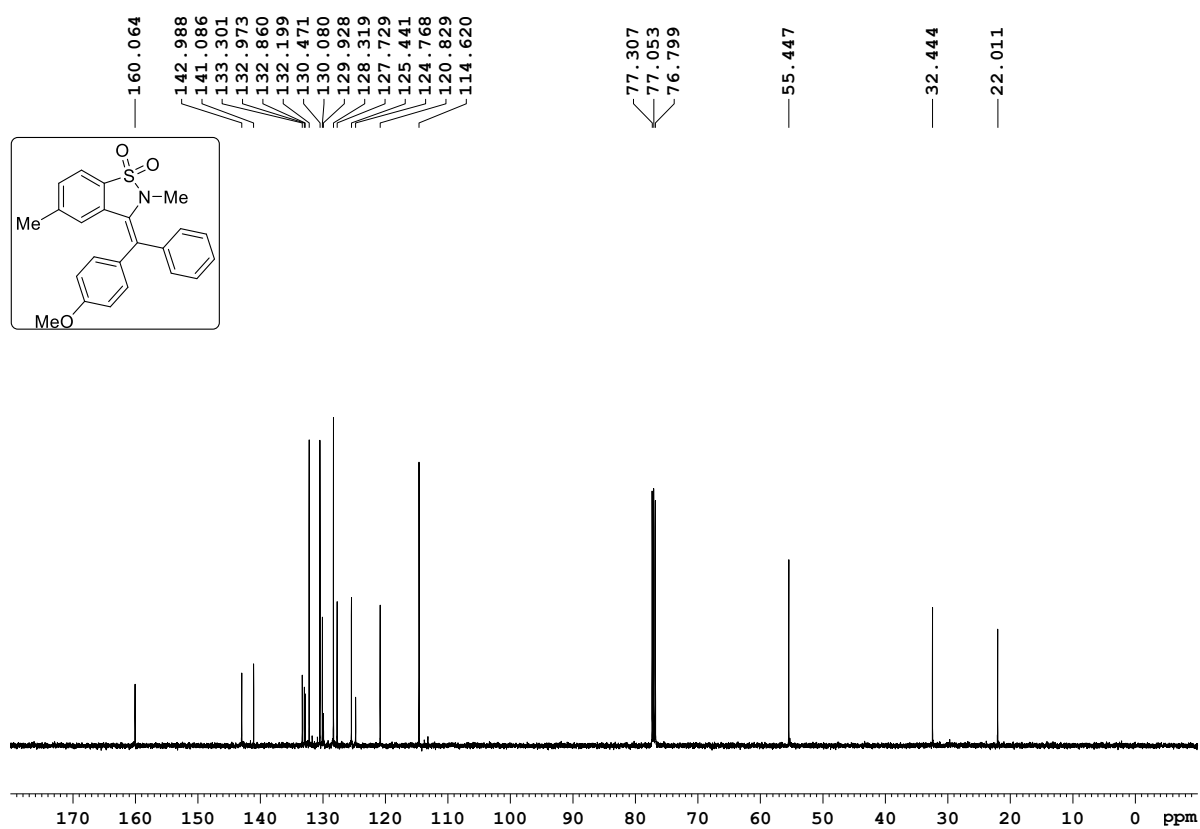


Figure S36. ¹³C NMR spectrum of compound 3ac

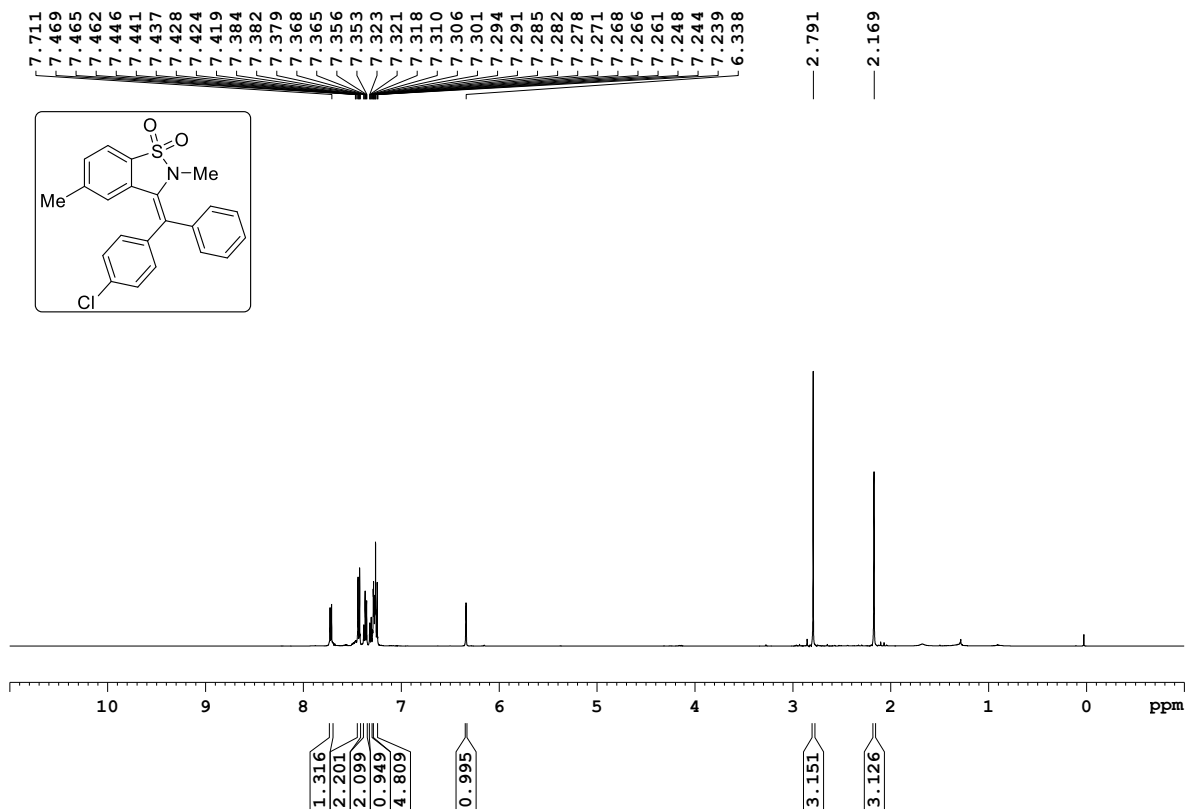


Figure S37. ¹H NMR spectrum of compound 3ad

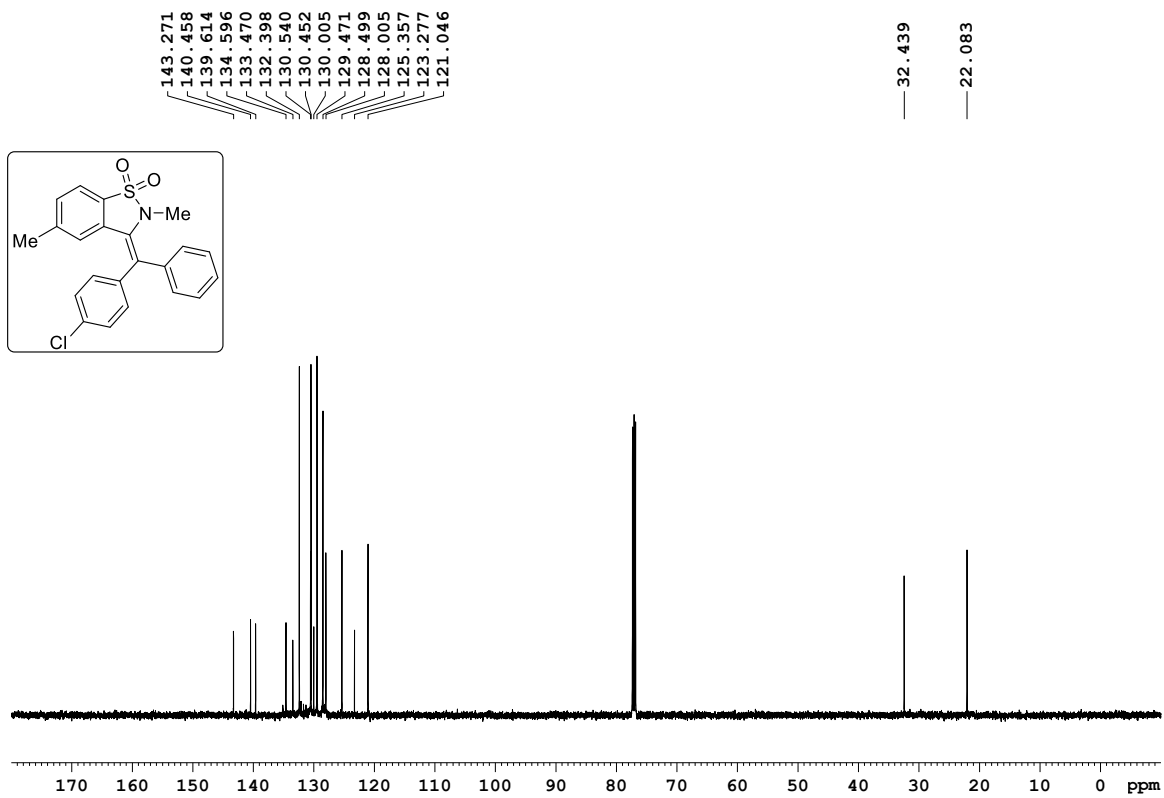


Figure S38. ¹³C NMR spectrum of compound 3ad

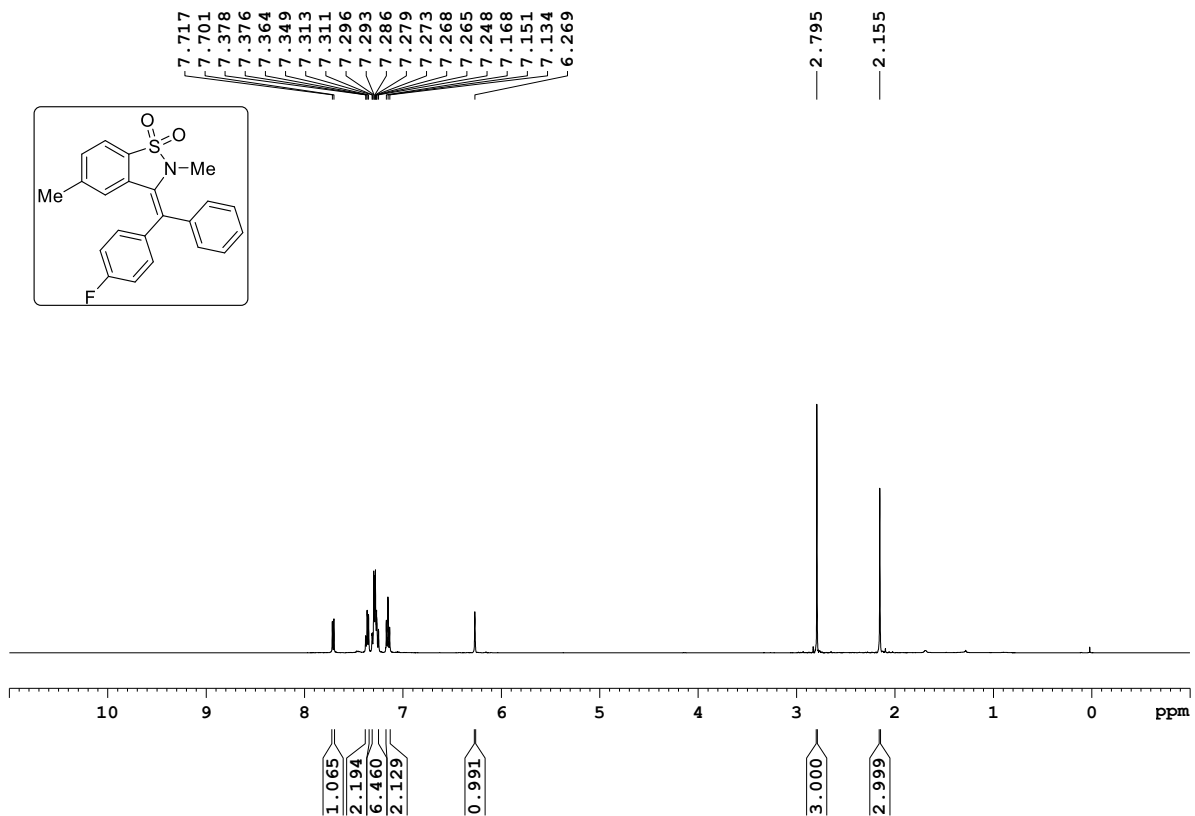


Figure S39. $^1\text{H NMR}$ spectrum of compound 3ae

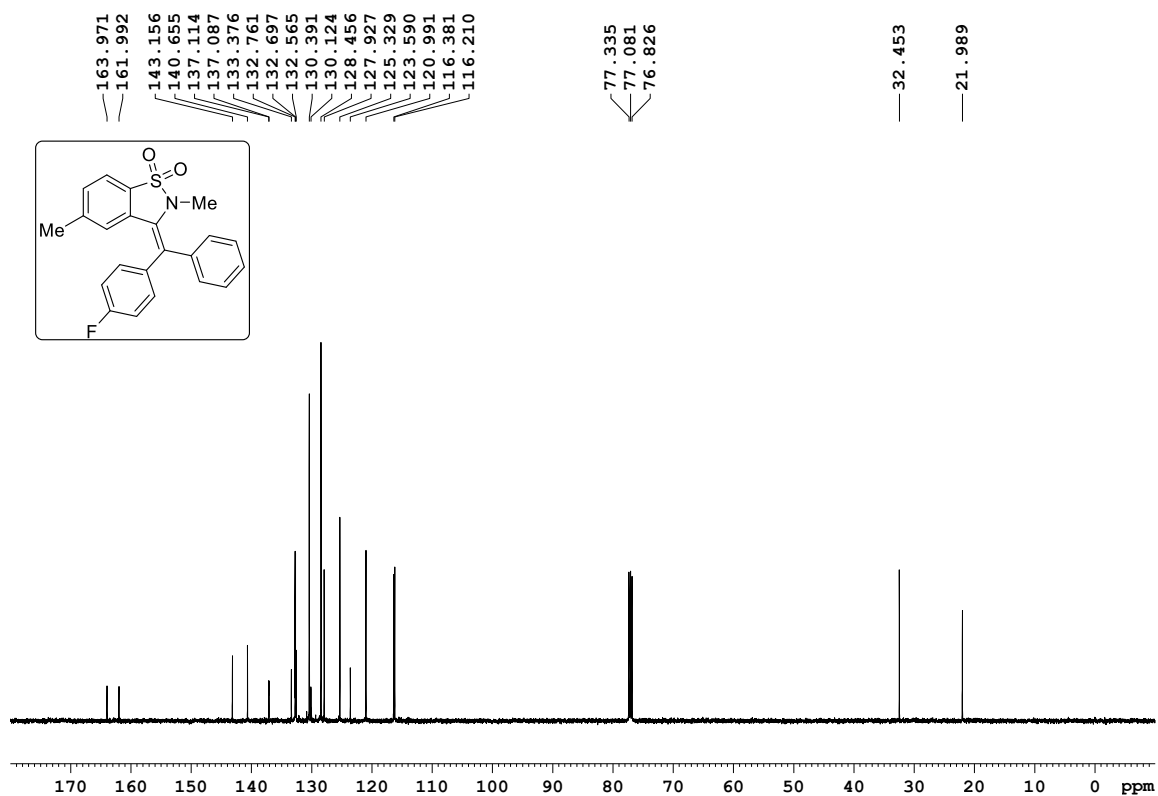


Figure S40. $^{13}\text{C NMR}$ spectrum of compound 3ae

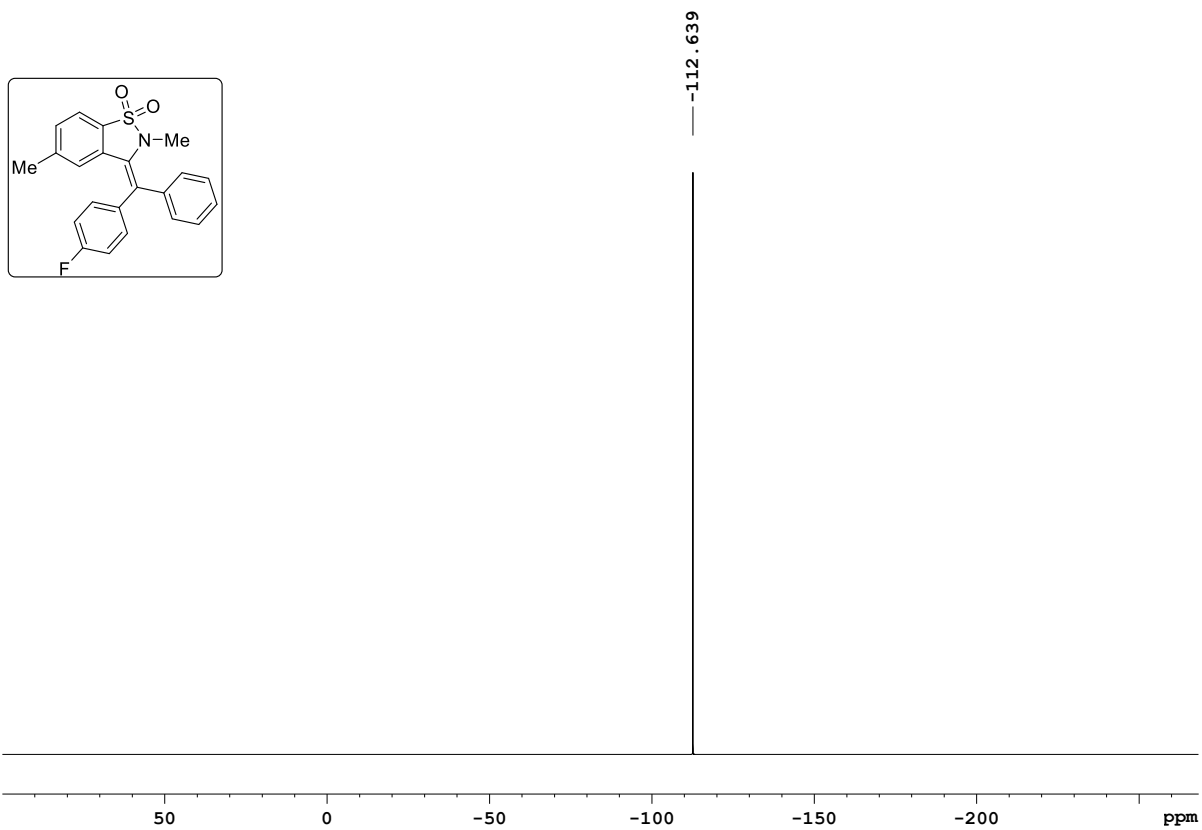


Figure S41. ^{19}F NMR spectrum of compound **3ae**

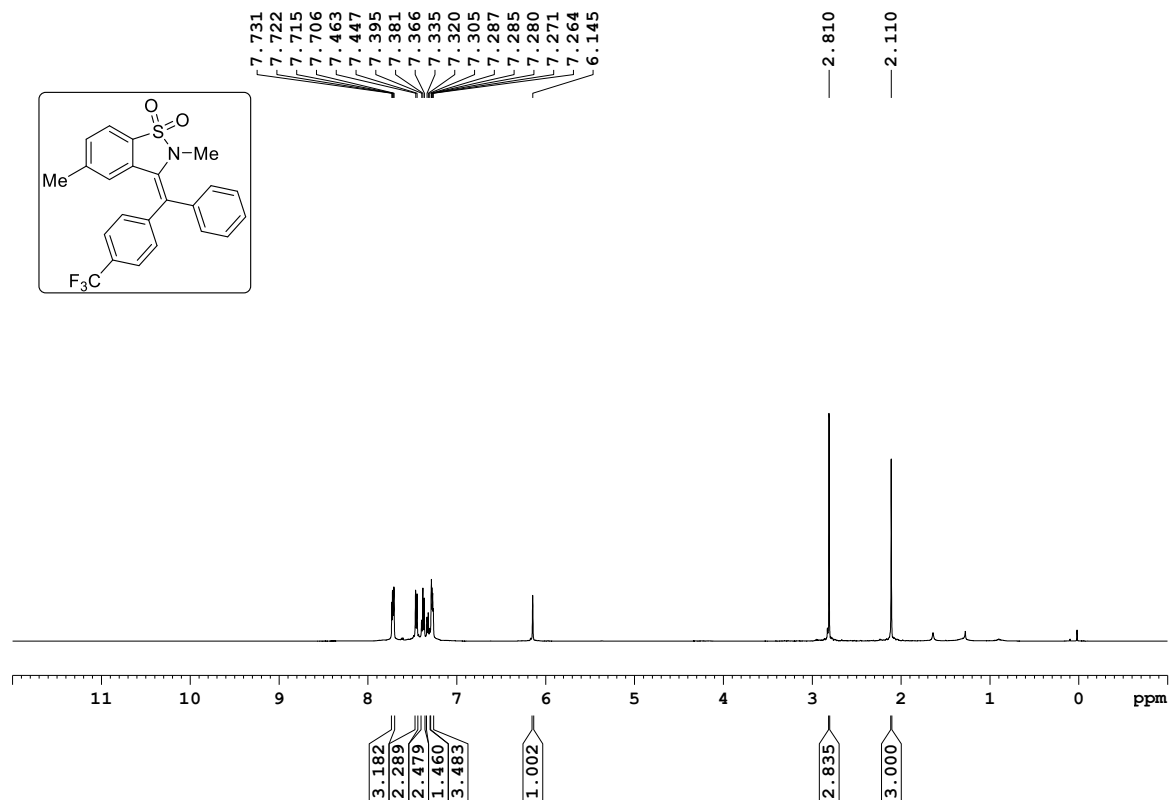


Figure S42. ¹H NMR spectrum of compound 3af

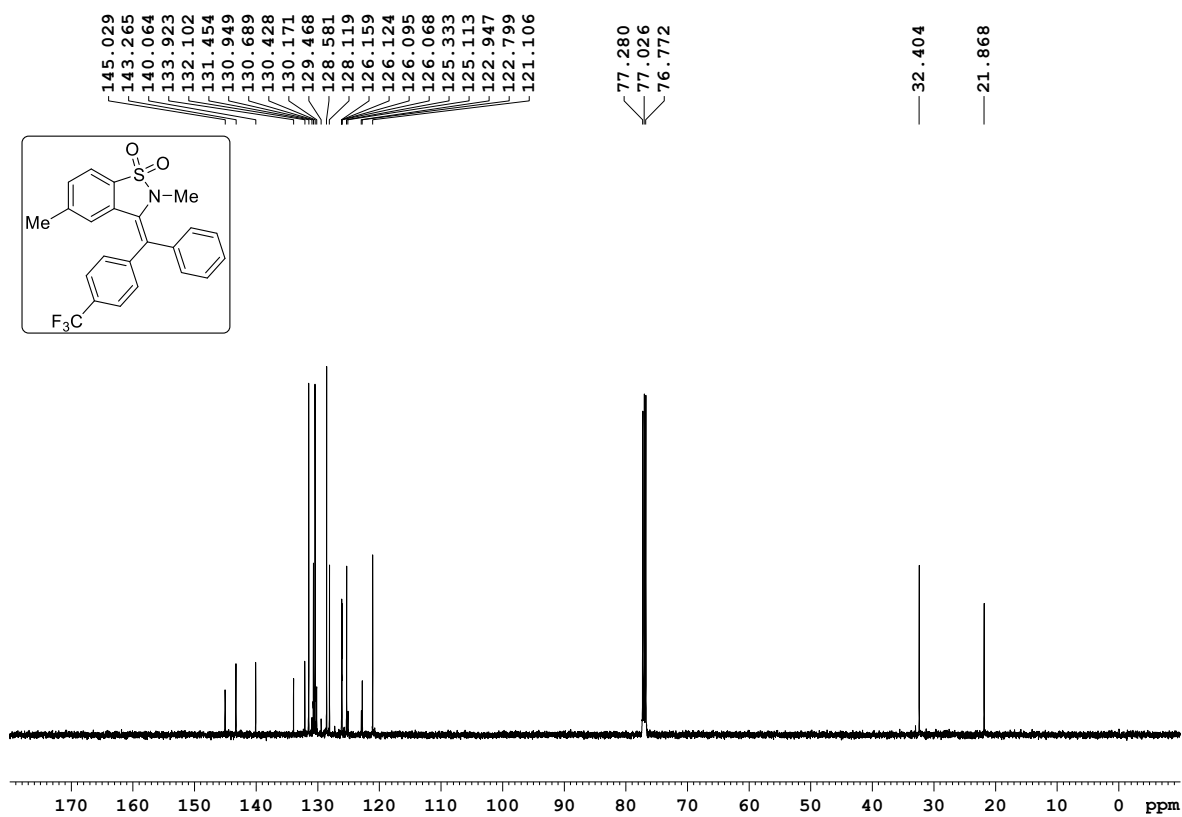


Figure S43. ¹³C NMR spectrum of compound 3af

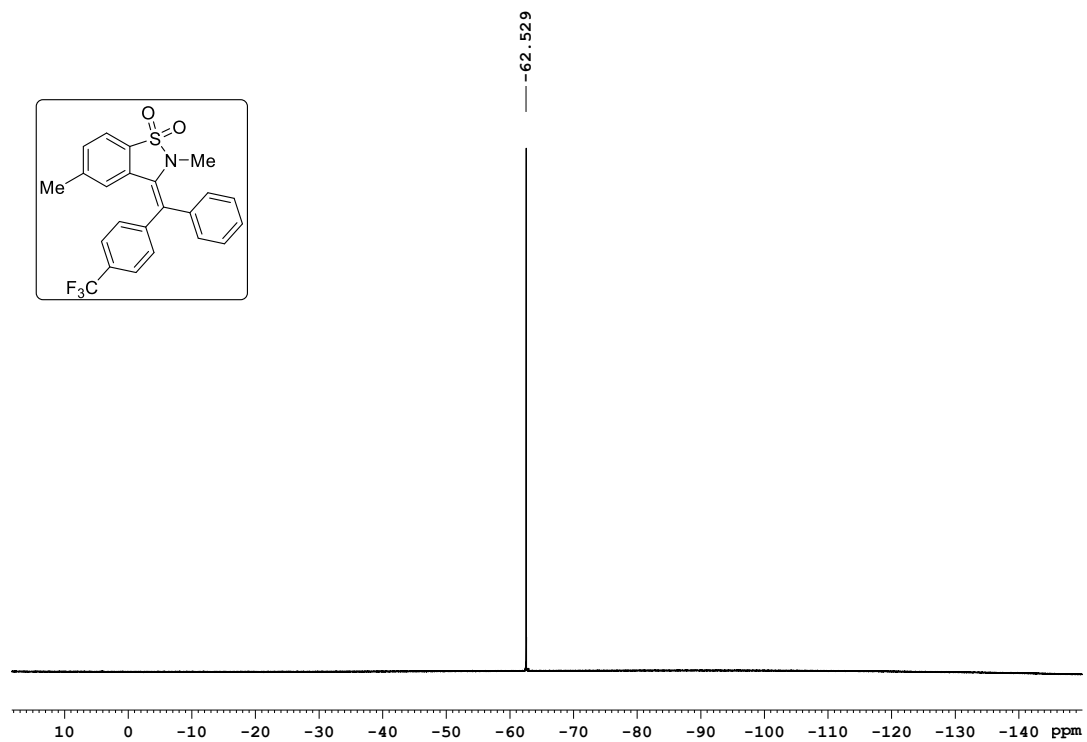


Figure S44. ^{19}F NMR spectrum of compound 3af

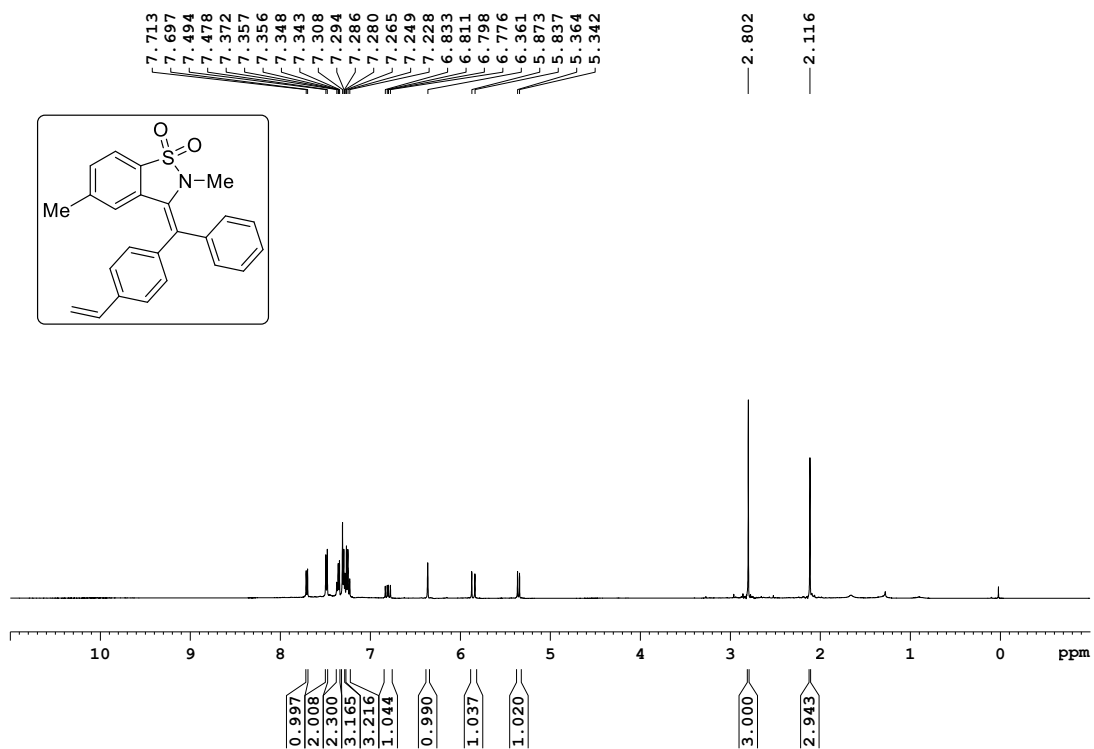


Figure S45. ¹H NMR spectrum of compound **3ag** (purity >96%)

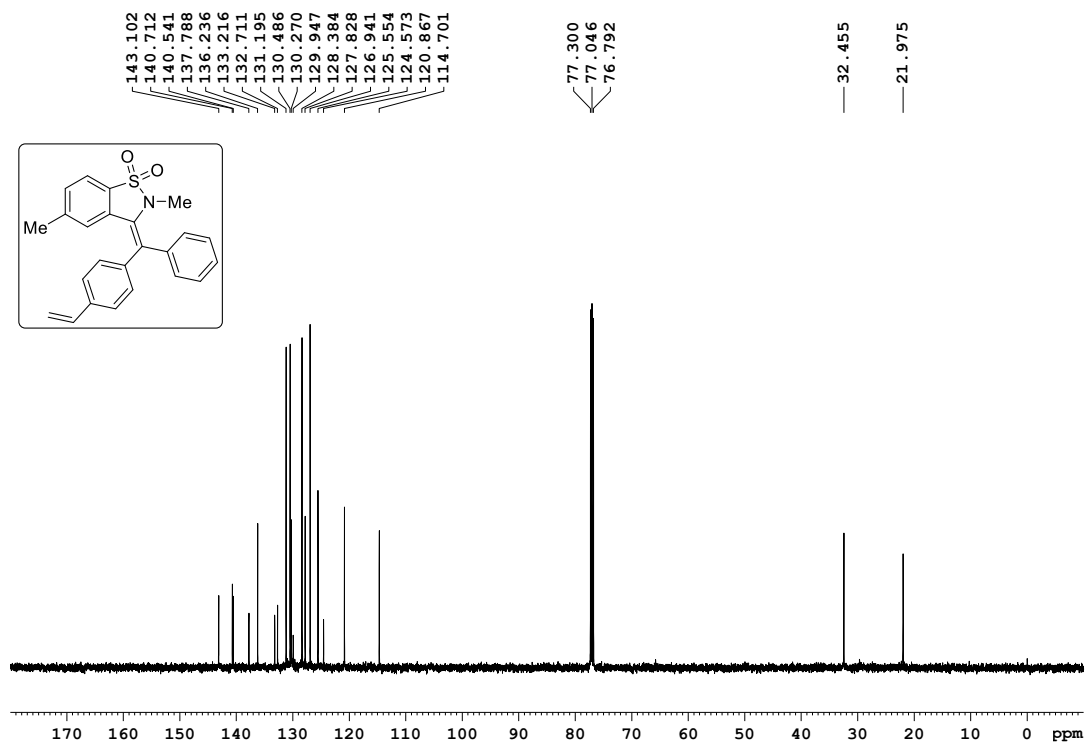


Figure S46. ¹³C NMR spectrum of compound **3ag** (purity >96%)

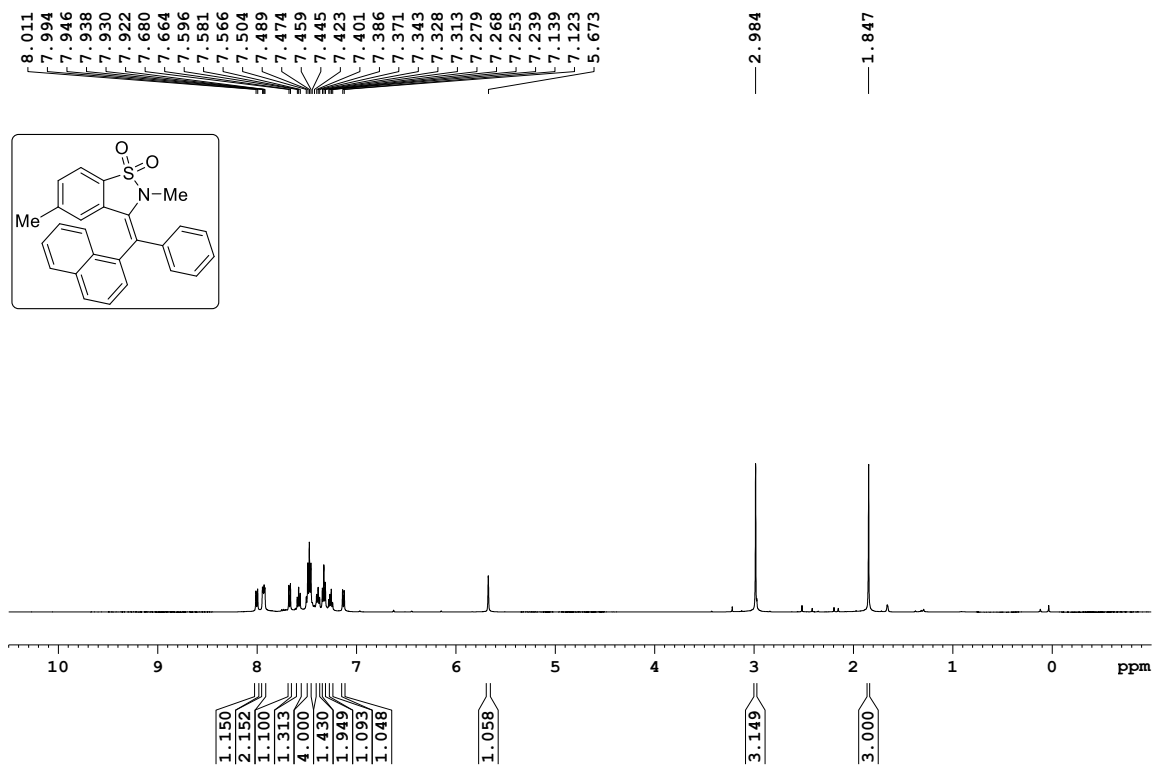


Figure S47. ¹H NMR spectrum of compound **3ah**

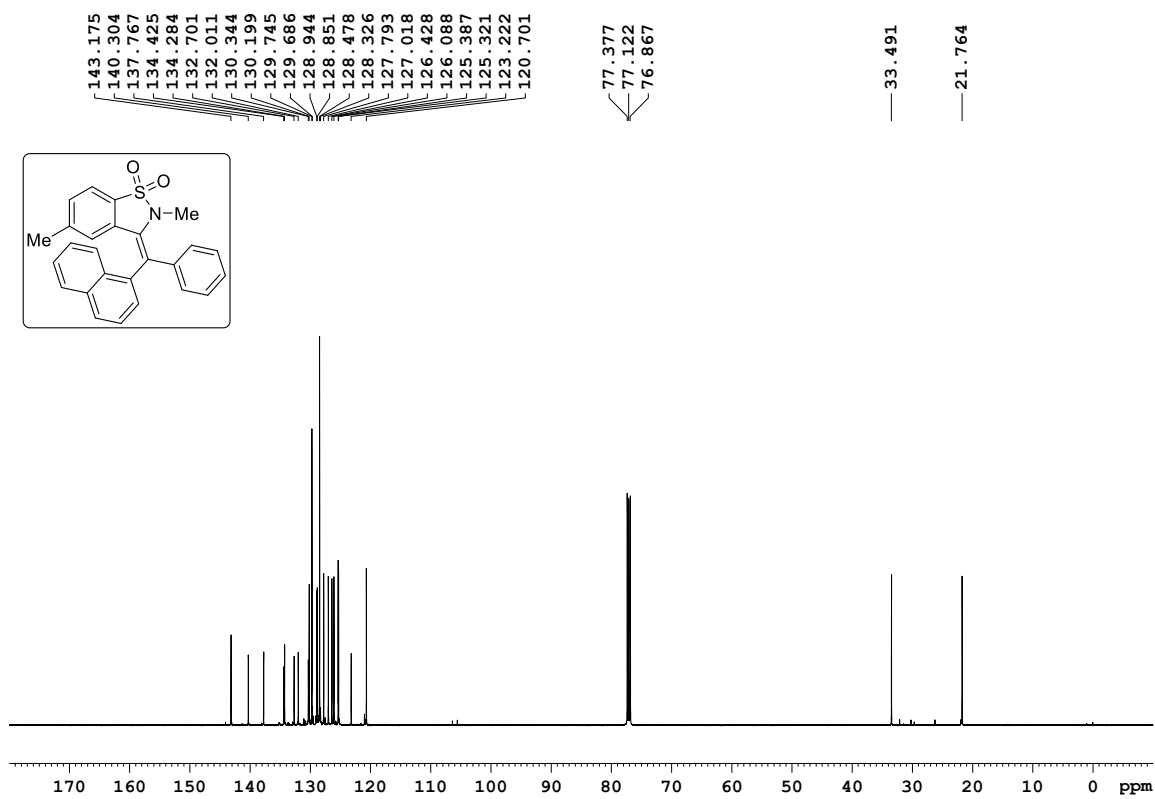


Figure S48. ¹³C NMR spectrum of compound **3ah** (purity ca 96%)

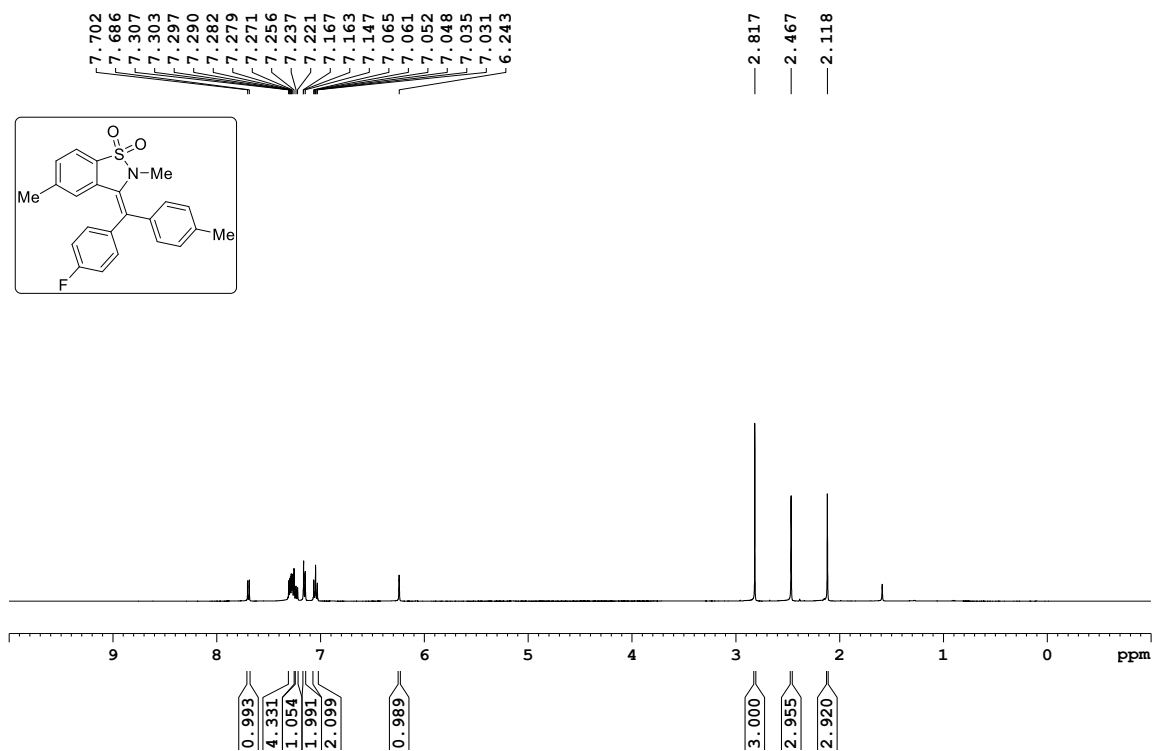


Figure S49. ¹H NMR spectrum of compound **3be**

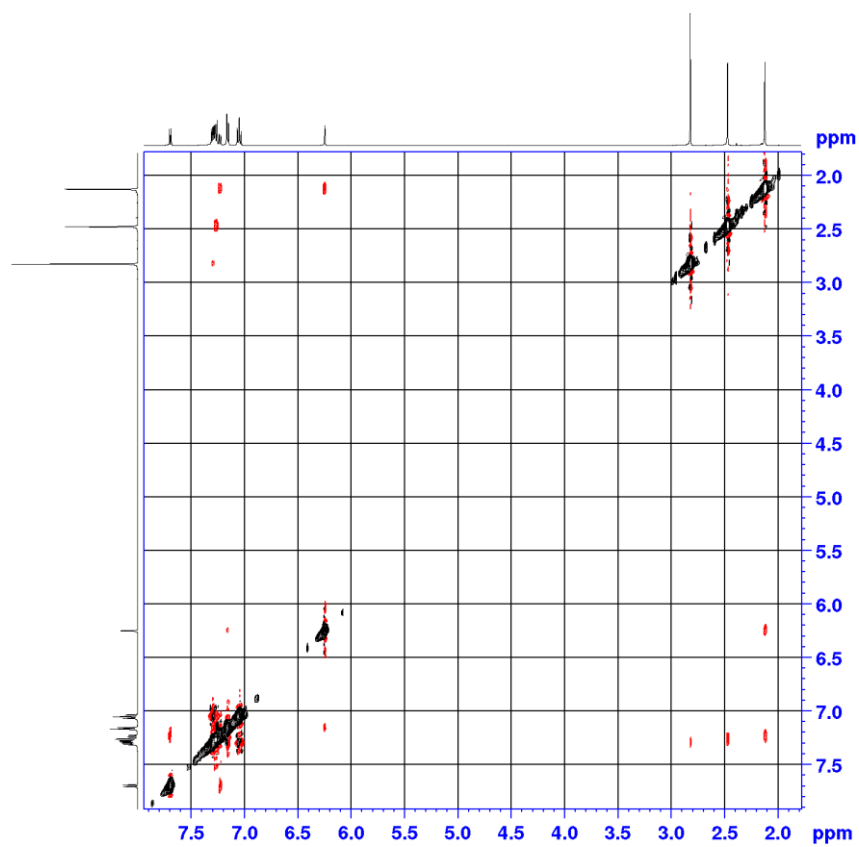


Figure S50. NOESY spectrum of compound **3be**

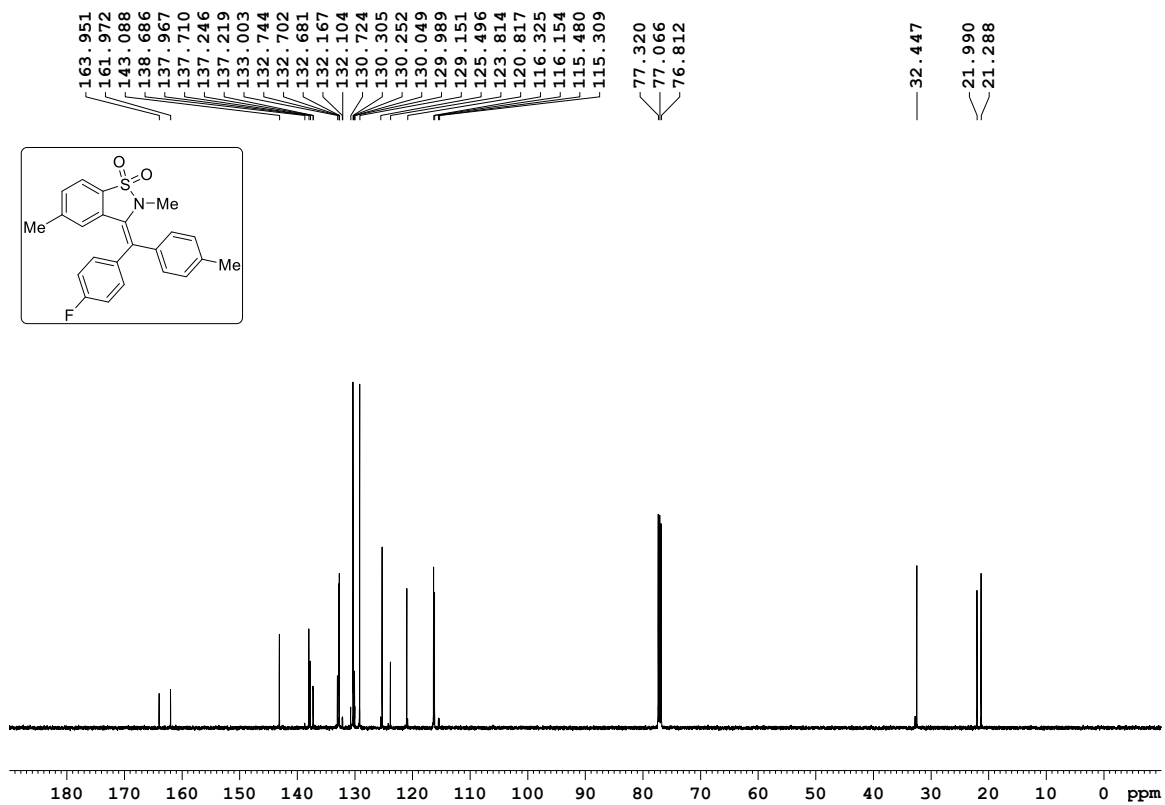


Figure S51. ¹³C NMR spectrum of compound 3be

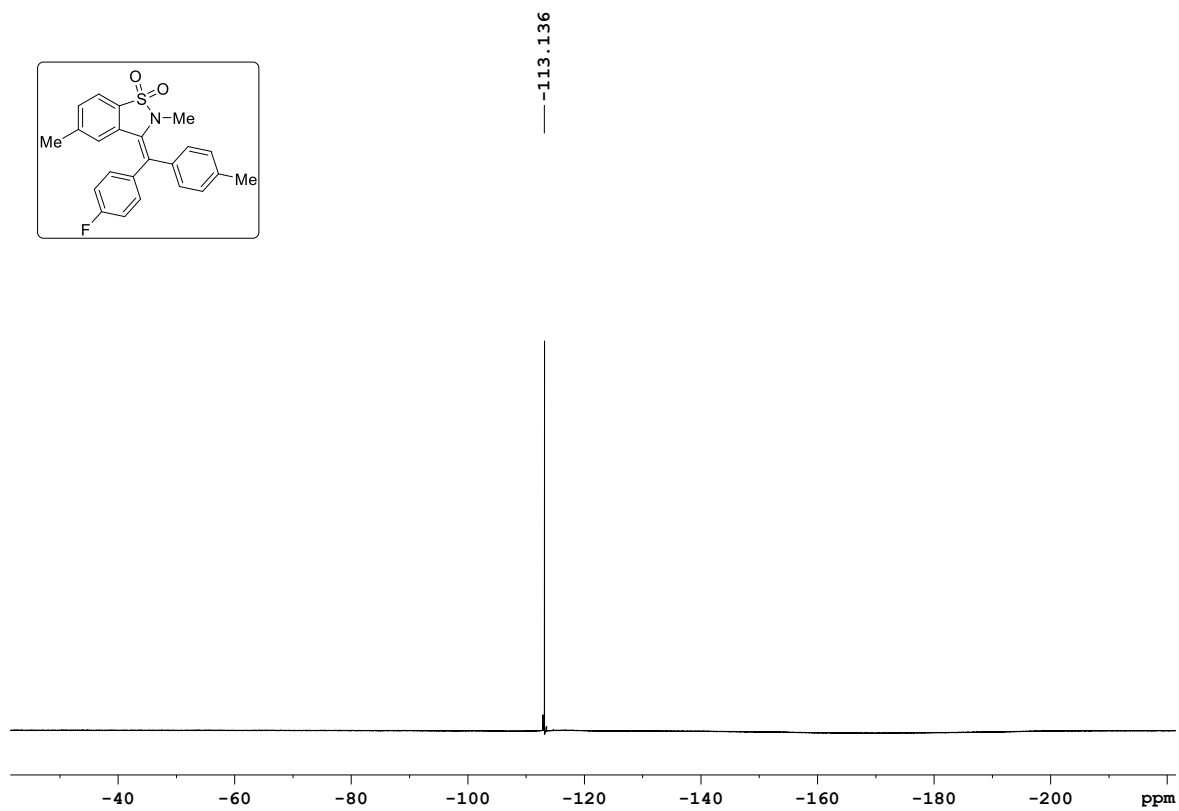


Figure S52. ¹⁹F NMR spectrum of compound 3be

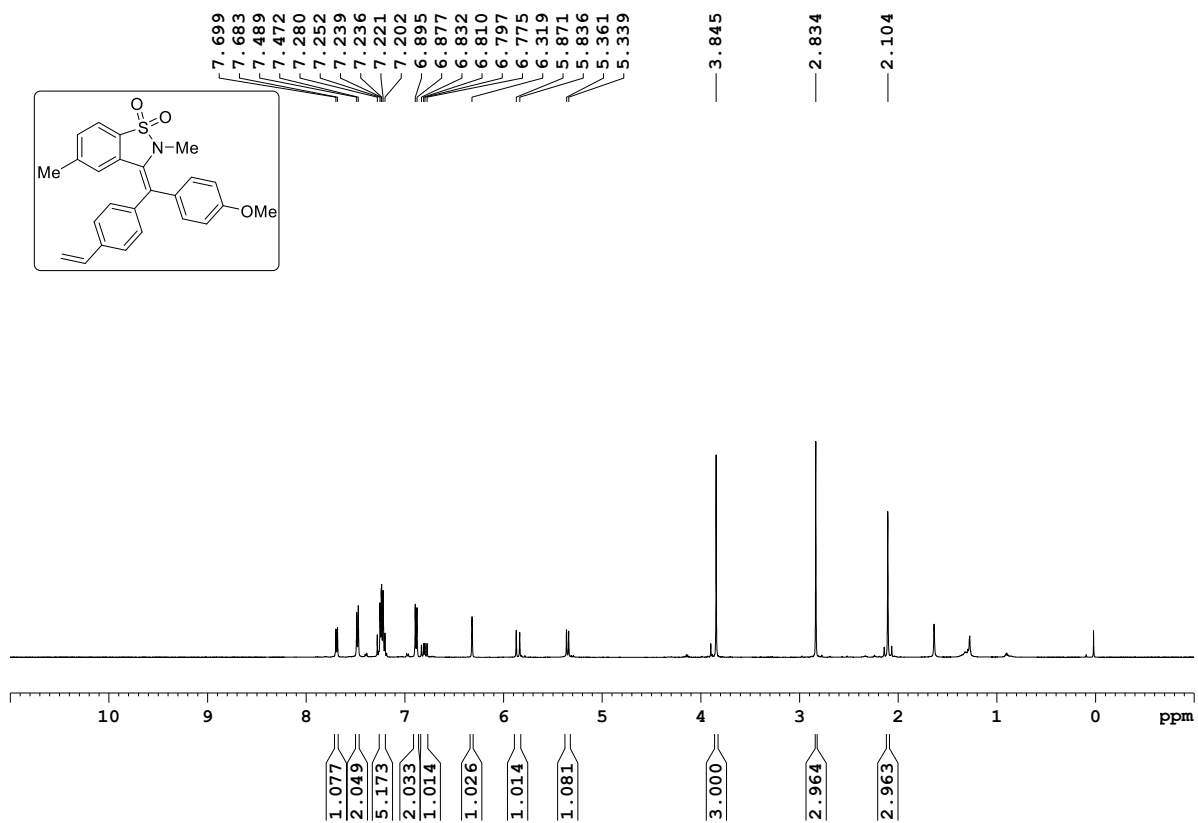


Figure S53. ¹H NMR spectrum of compound 3cg

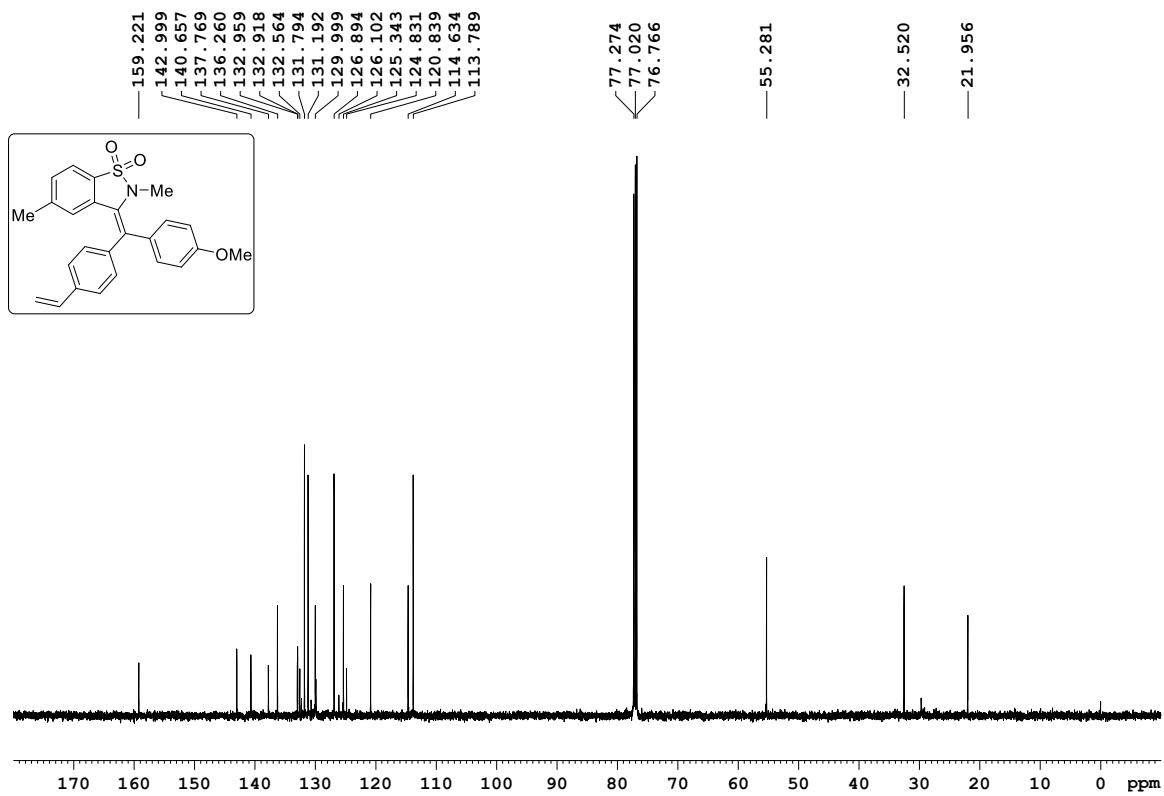


Figure S54. ¹³C NMR spectrum of compound 3cg

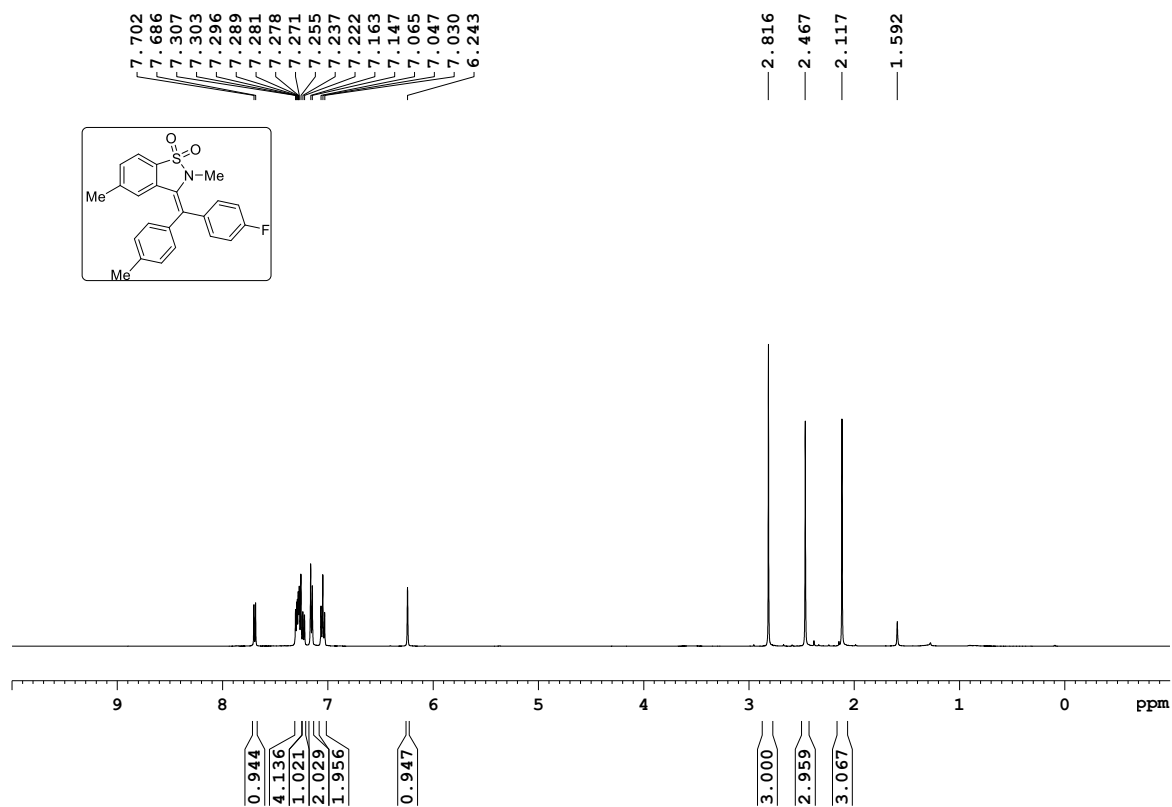


Figure S55. ¹H NMR spectrum of compound 3db

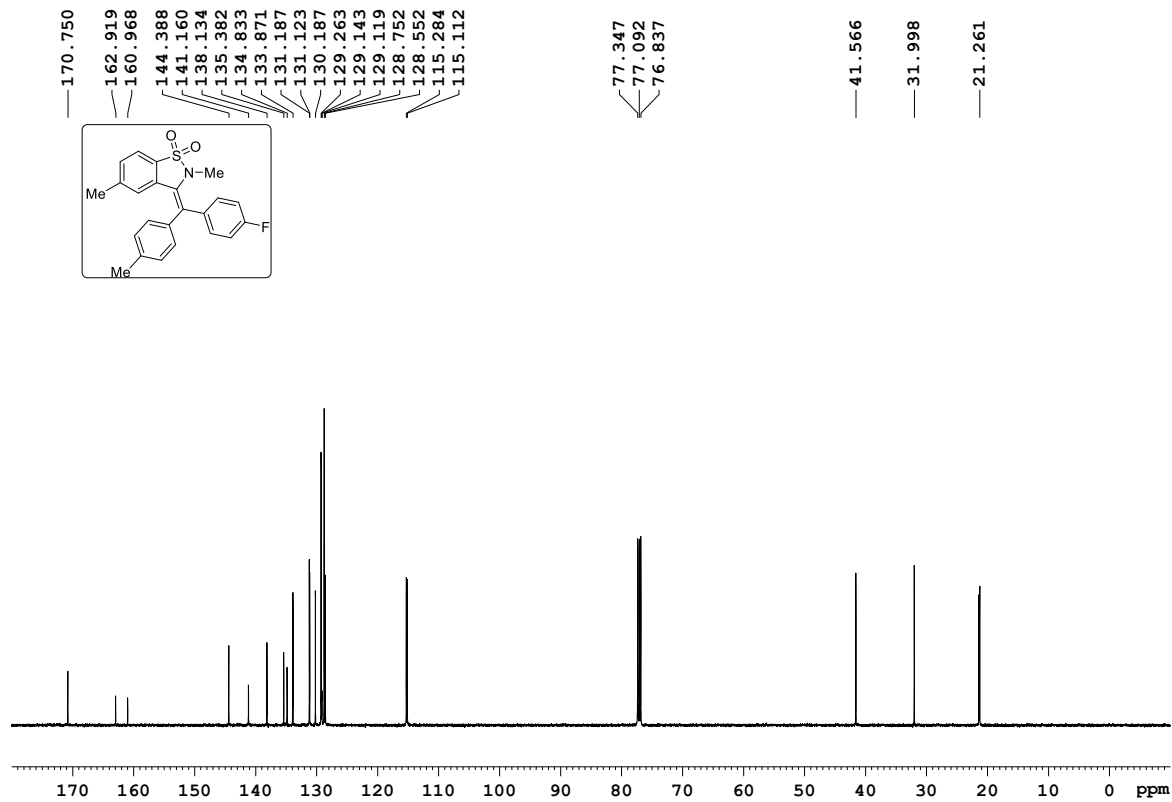


Figure S56. ¹³C NMR spectrum of compound 3db

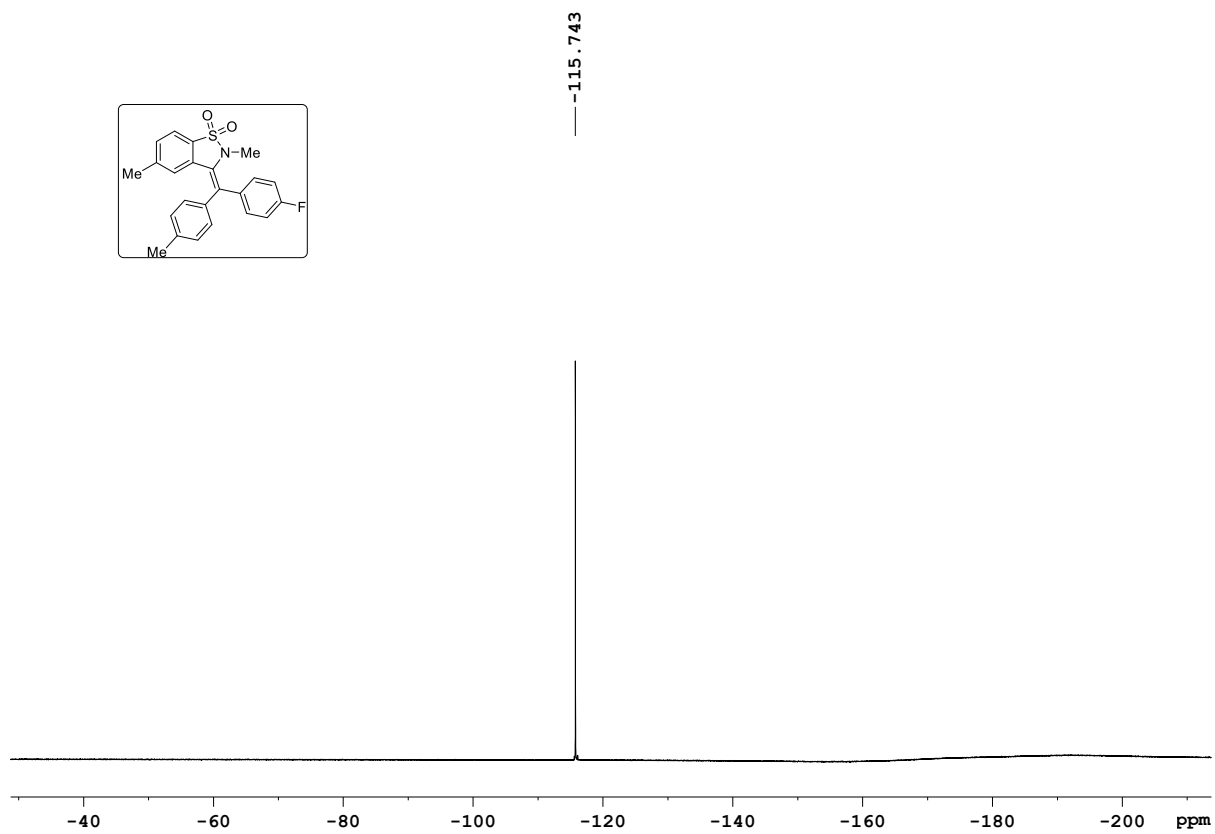


Figure S57. ^{19}F NMR spectrum of compound **3db**

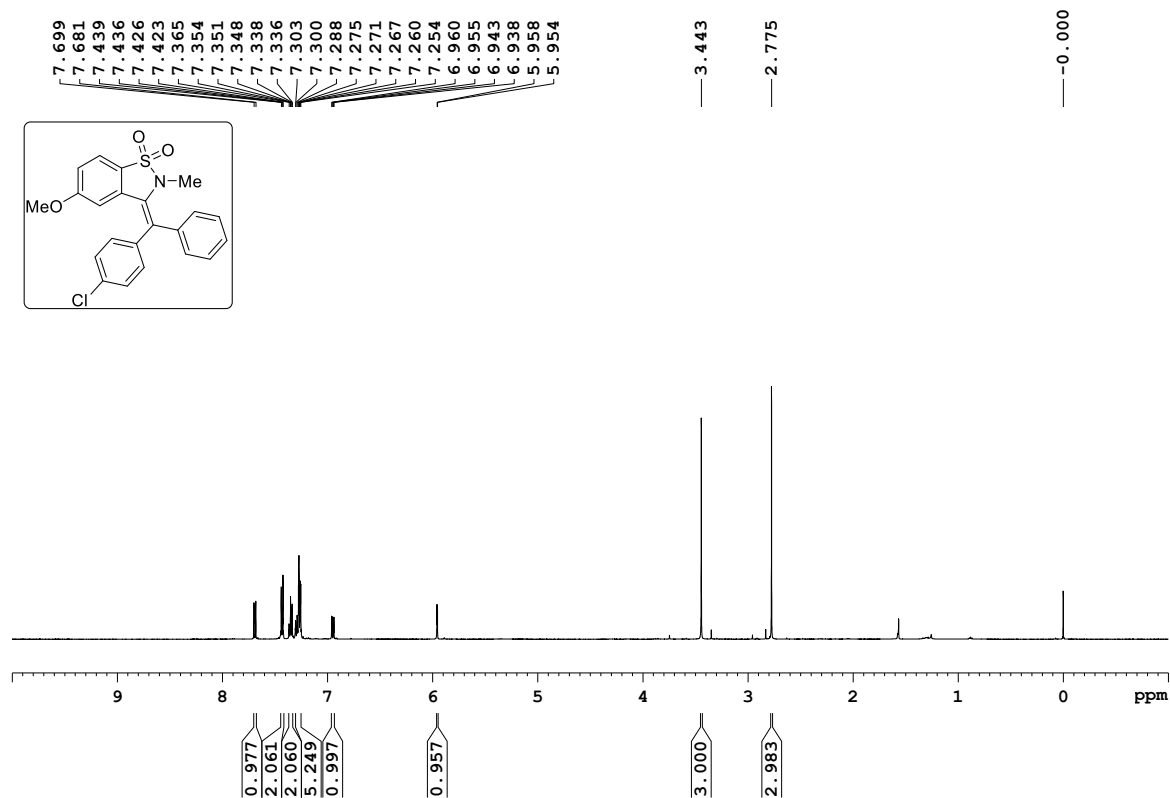


Figure S58. ¹H NMR spectrum of compound **3fd** (purity >96%)

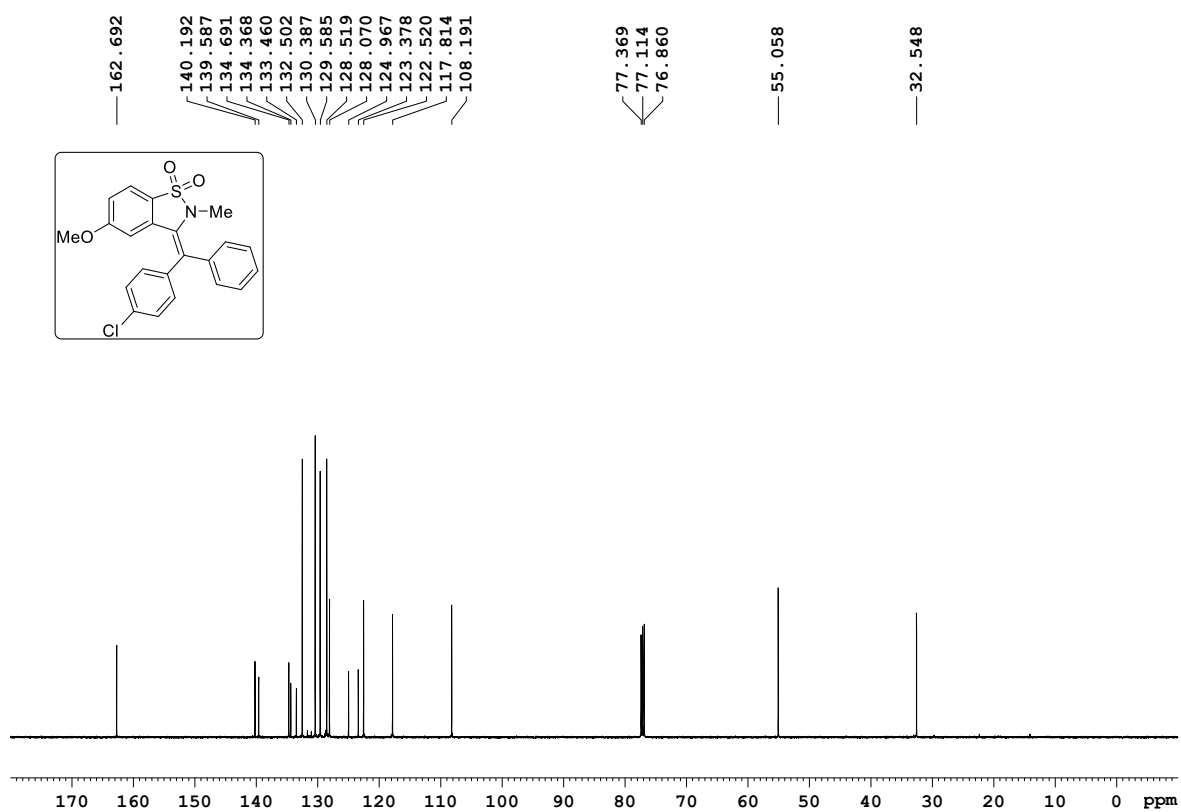


Figure S59. ¹³C NMR spectrum of compound **3fd** (purity >96%)

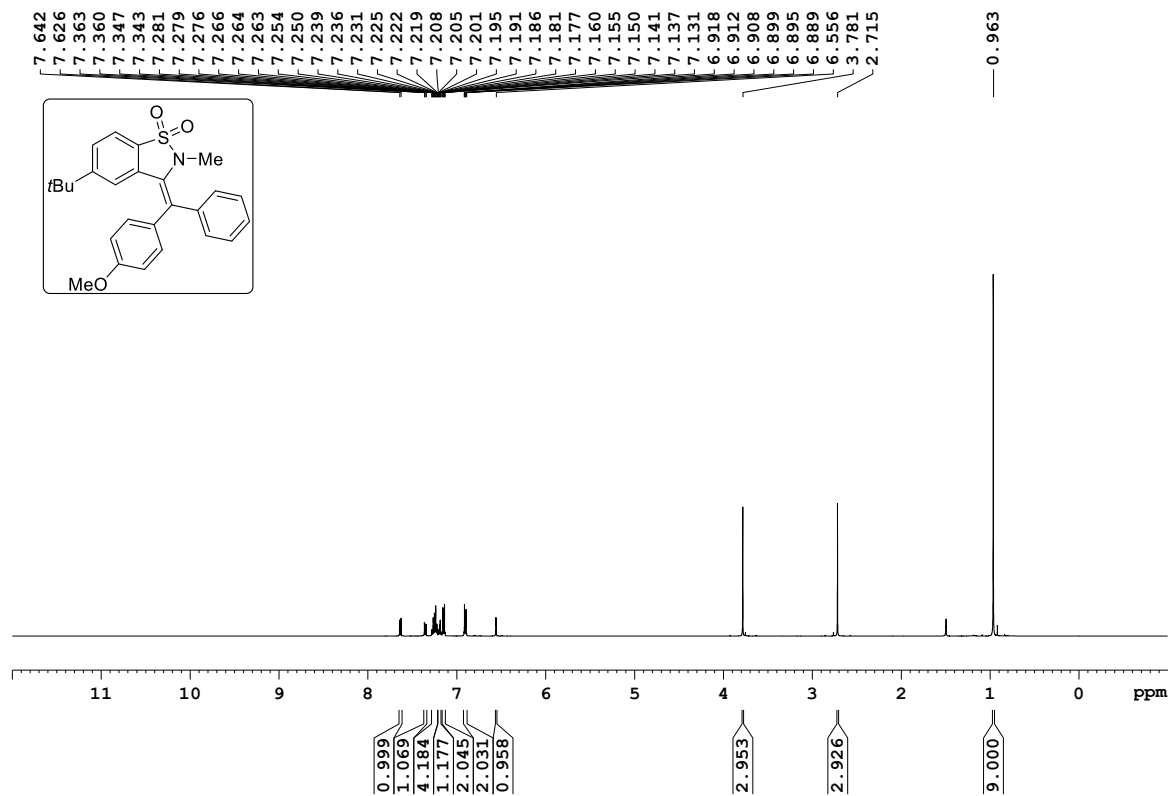


Figure S60. ¹H NMR spectrum of compound **3gc**

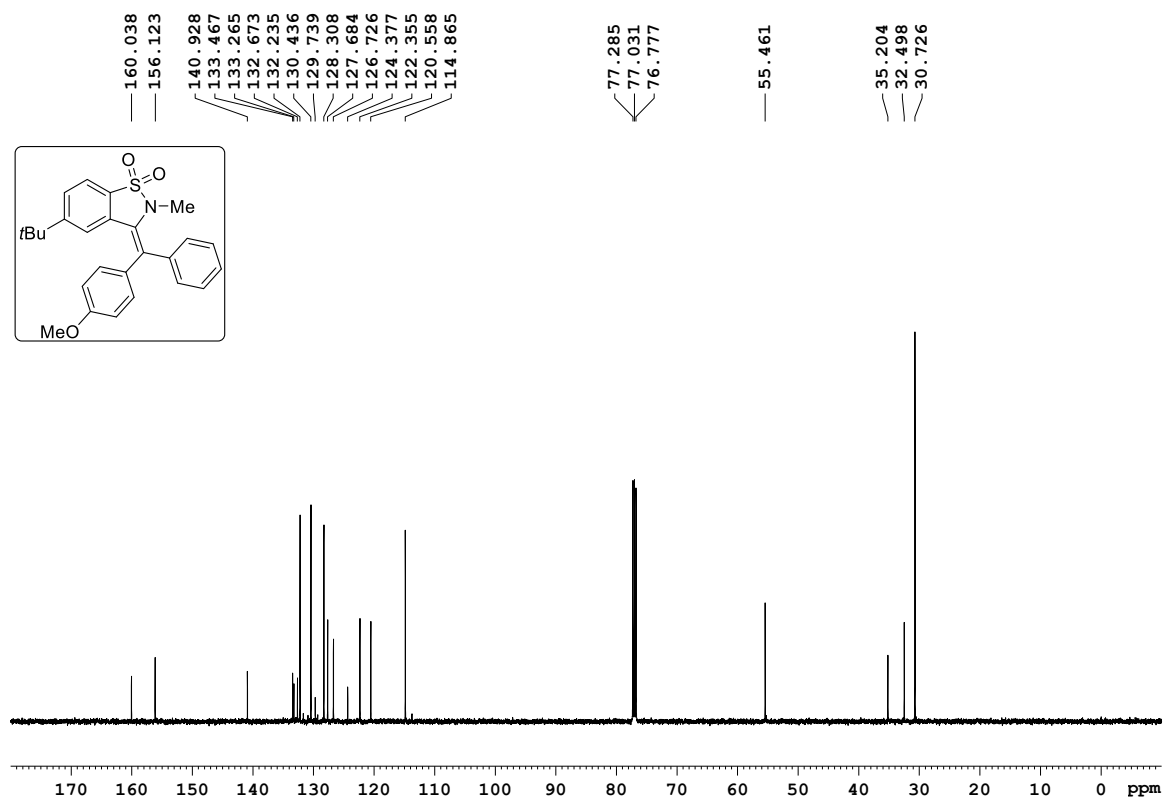


Figure S61. ¹³C NMR spectrum of compound **3gc**

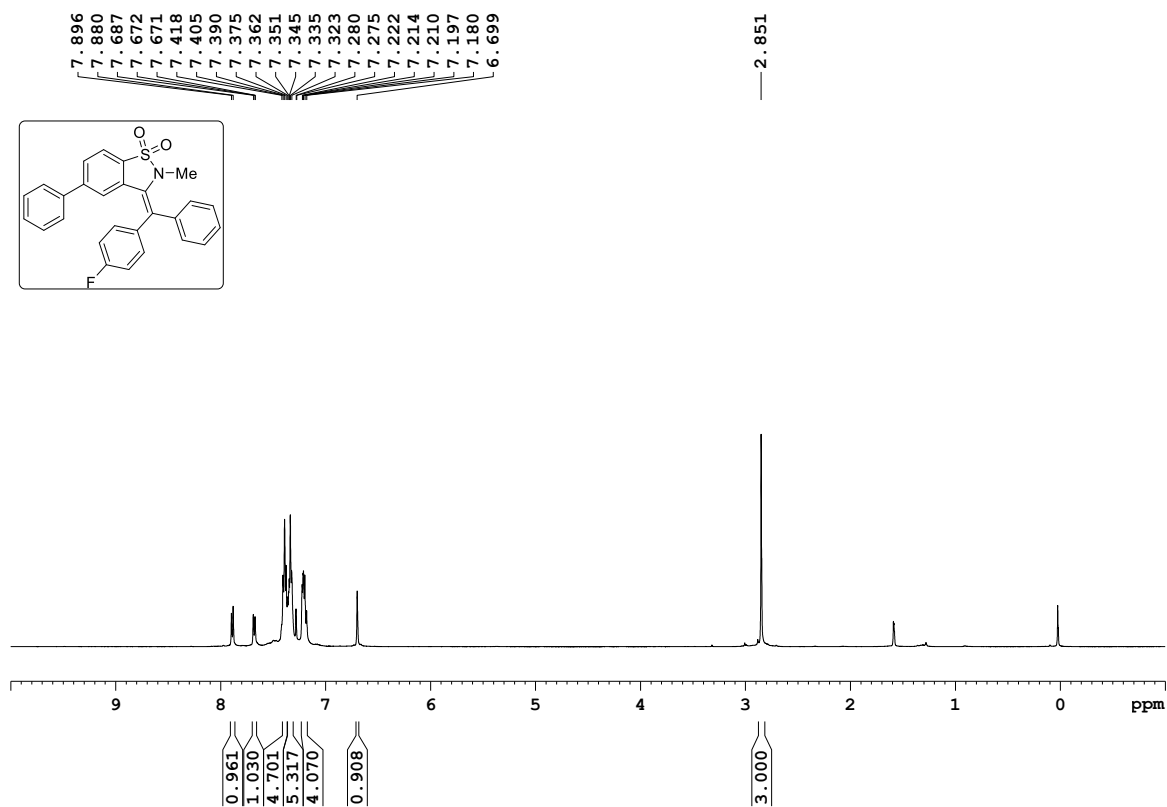


Figure S62. ¹H NMR spectrum of compound **3he**

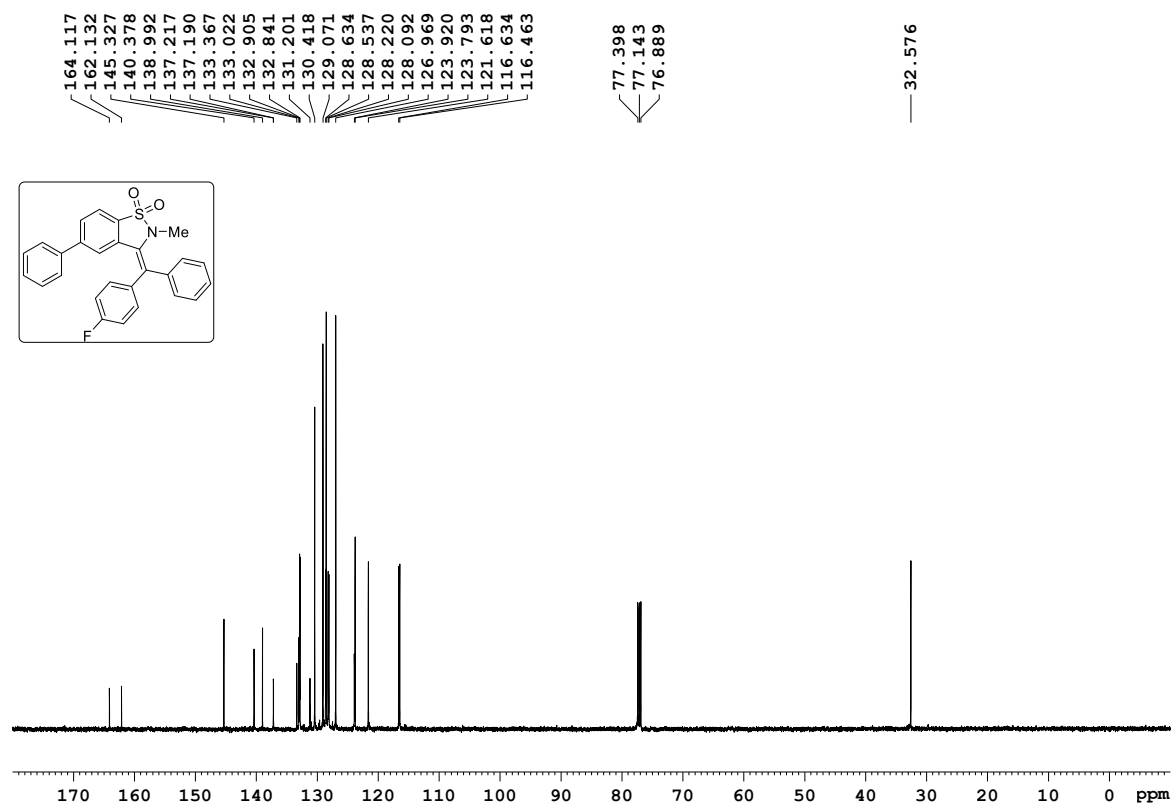


Figure S63. ¹³C NMR spectrum of compound **3he**

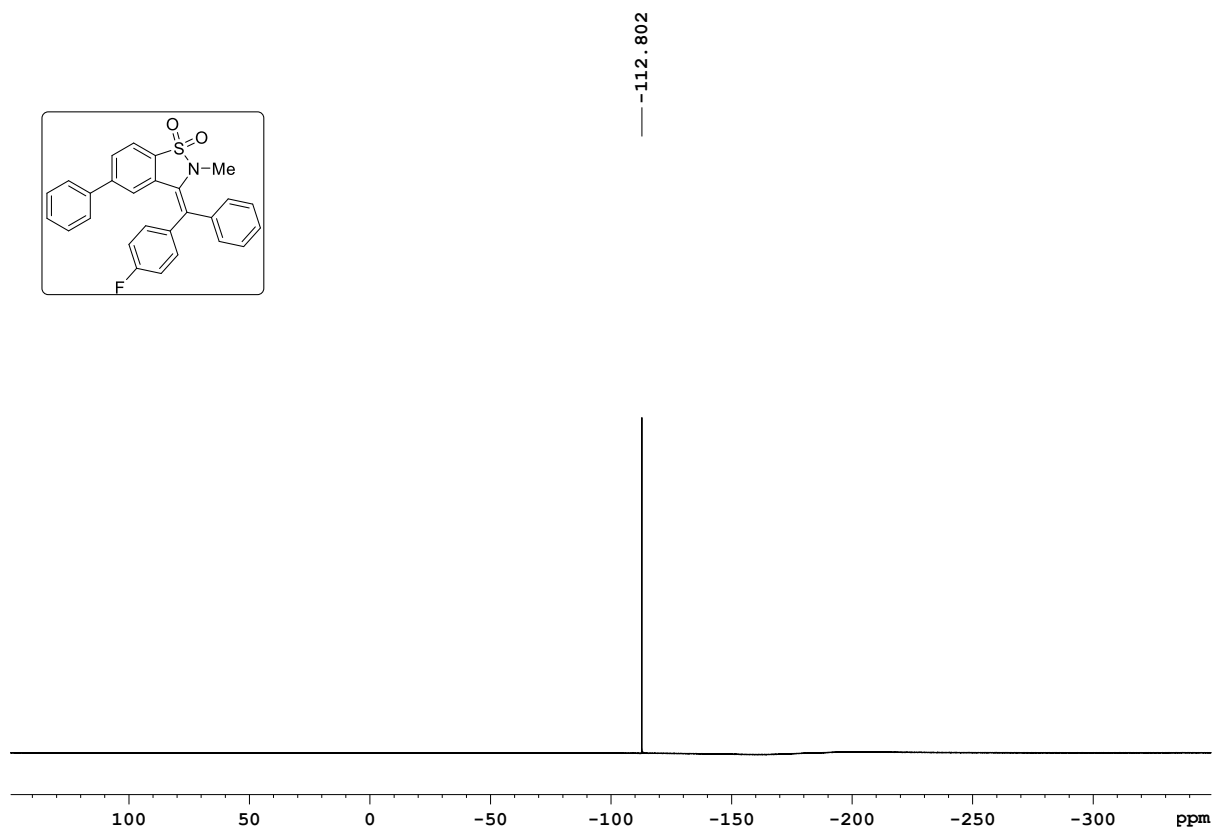


Figure S64. ^{19}F NMR spectrum of compound **3he**

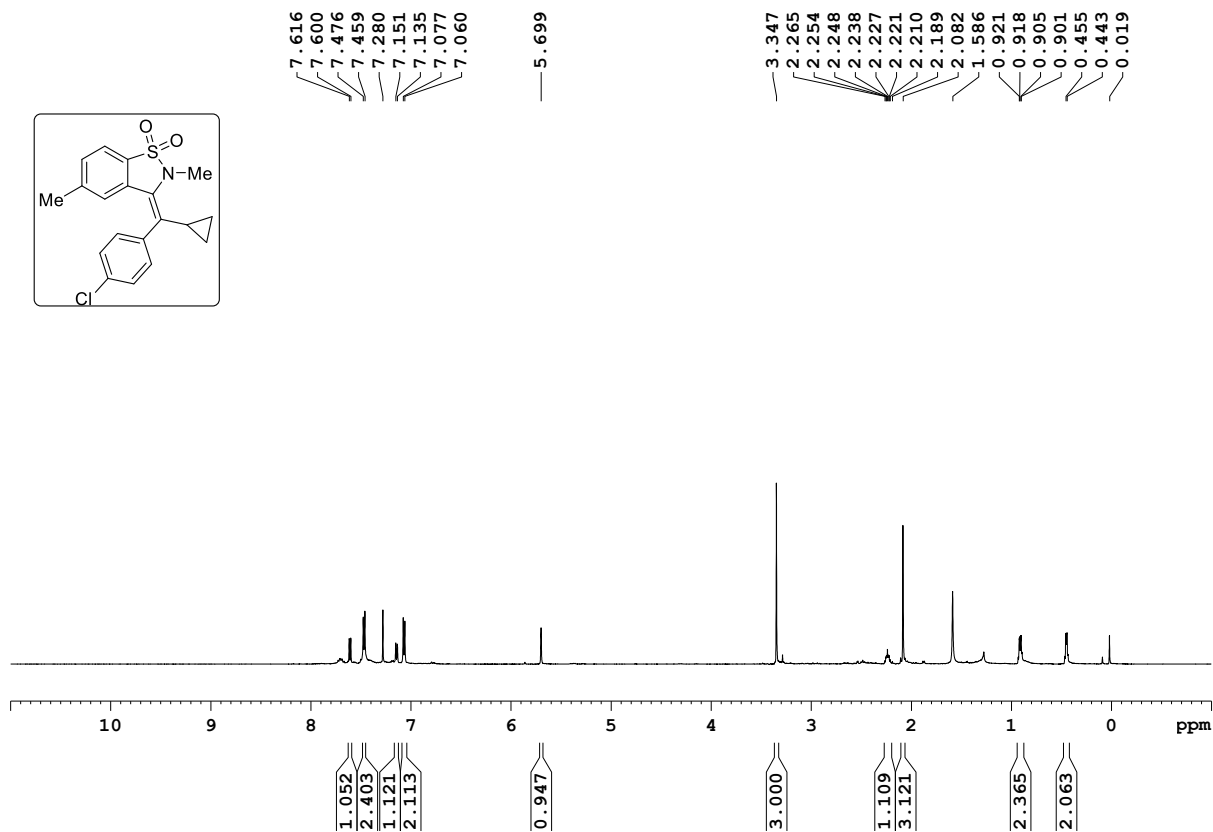


Figure S65. ¹H NMR spectrum of compound **3id** (purity >96%)

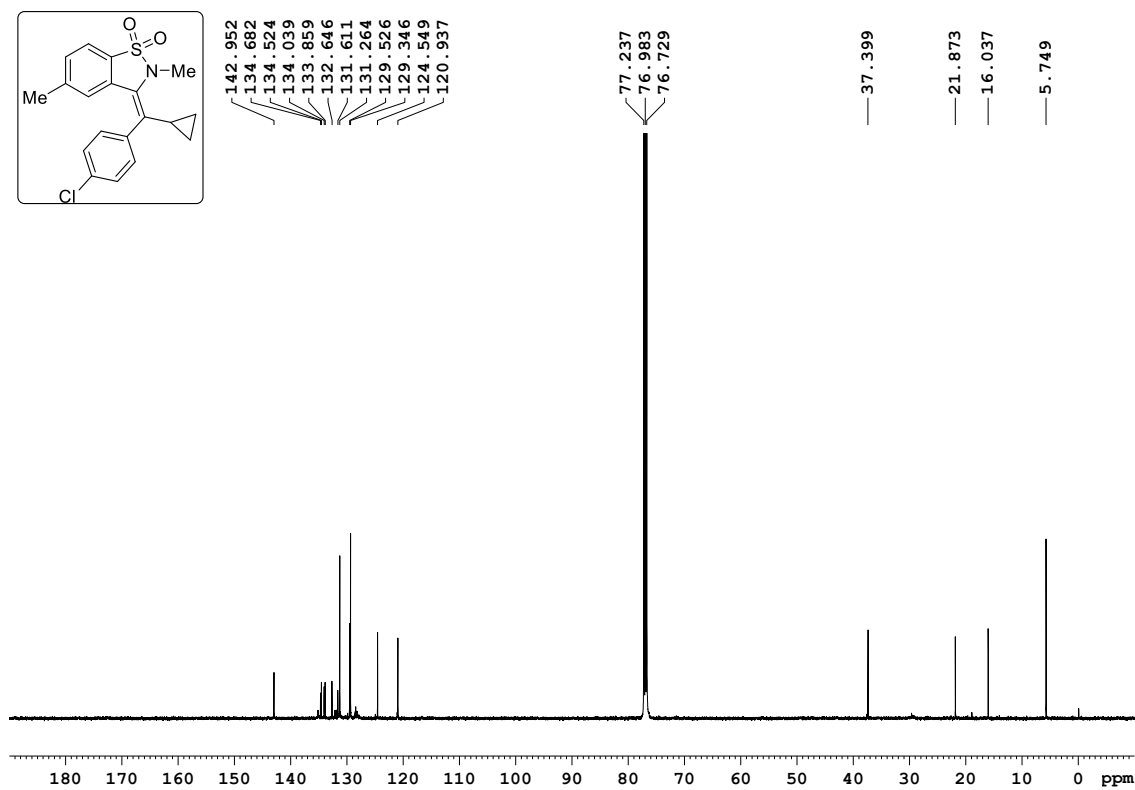


Figure S66. ¹³C NMR spectrum of compound **3id** (purity >96%)

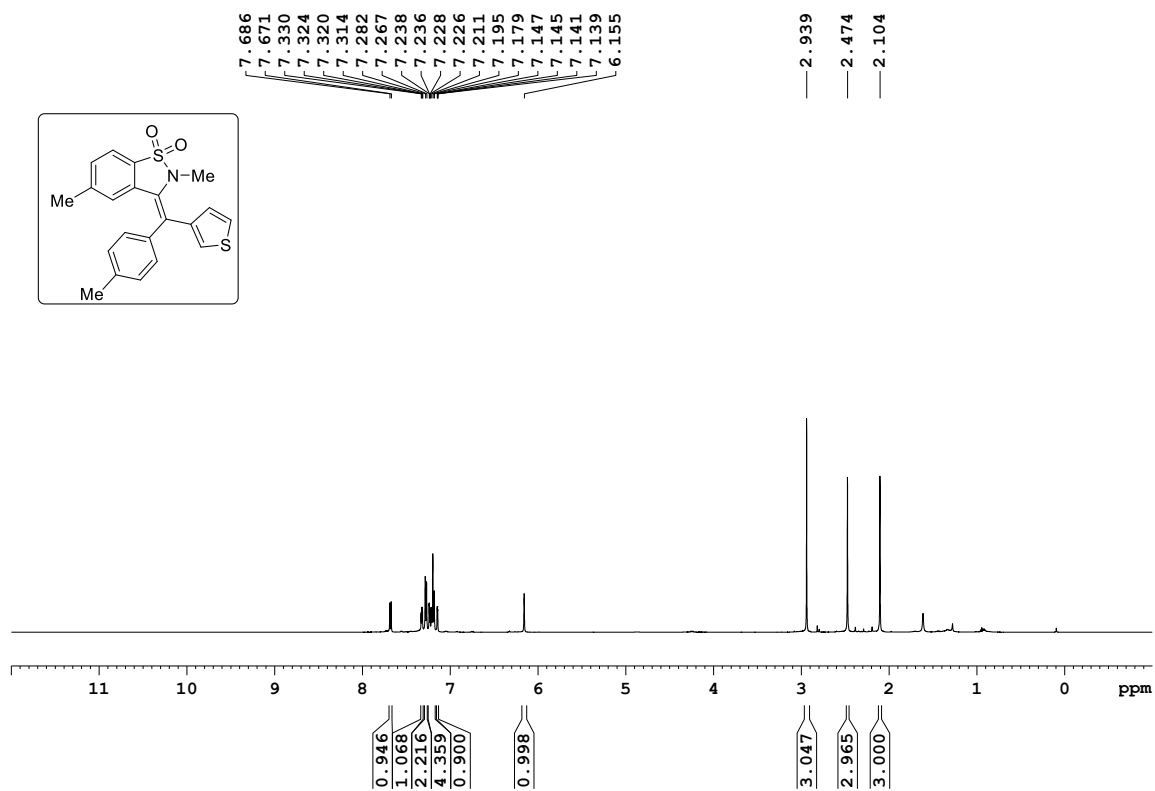


Figure S67. ¹H NMR spectrum of compound **3jb**

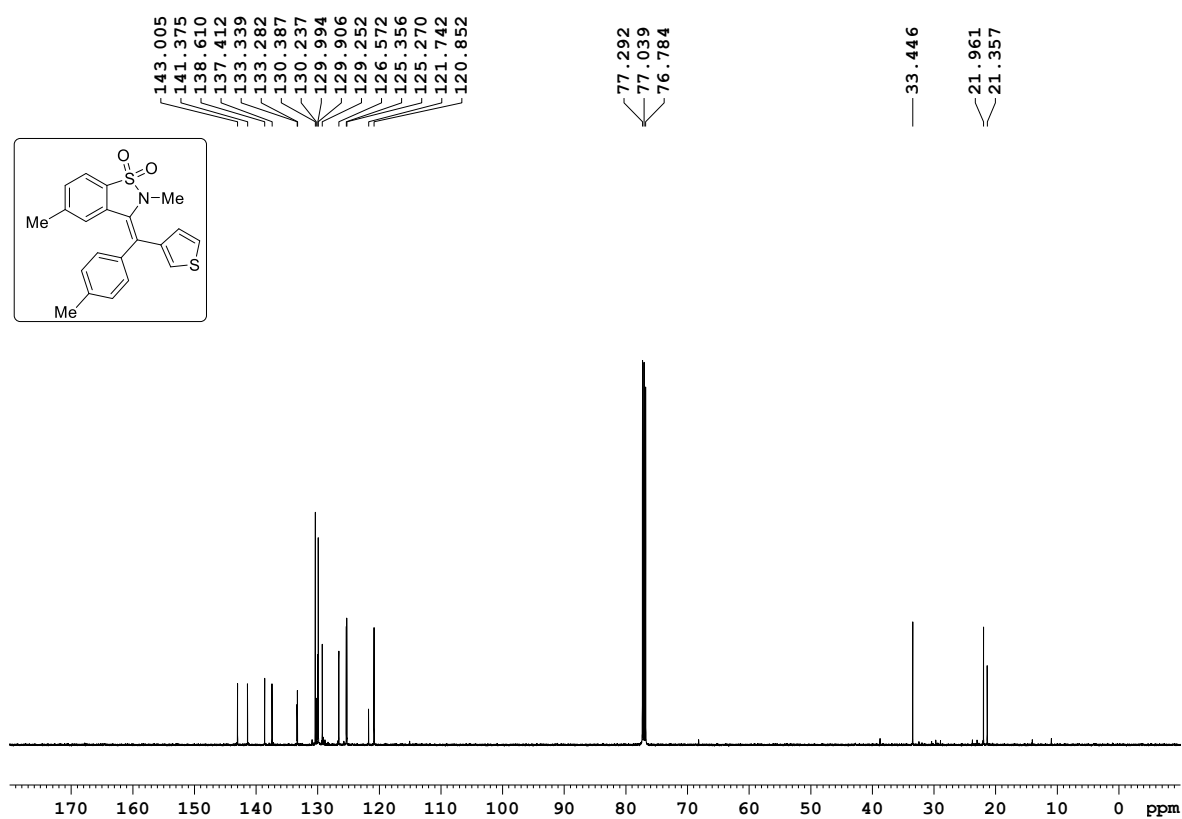


Figure S68. ¹³C NMR spectrum of compound **3jb**

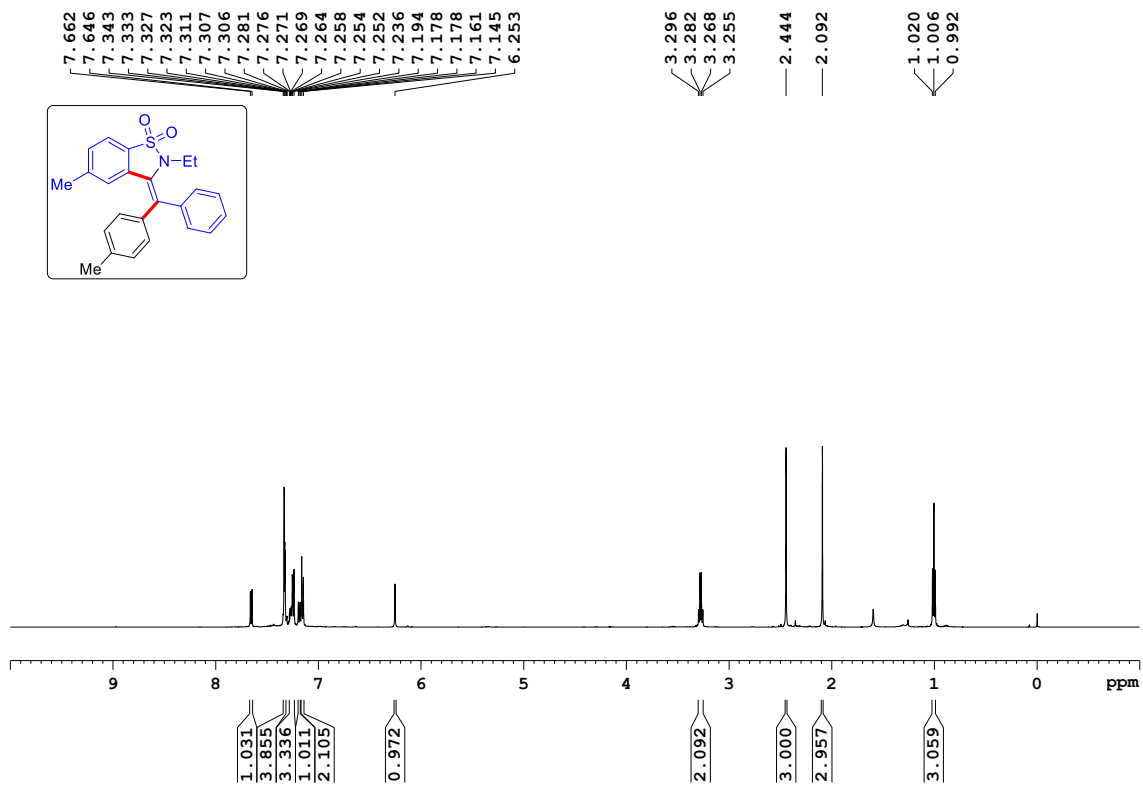


Figure S69. ¹H NMR spectrum of compound 31b

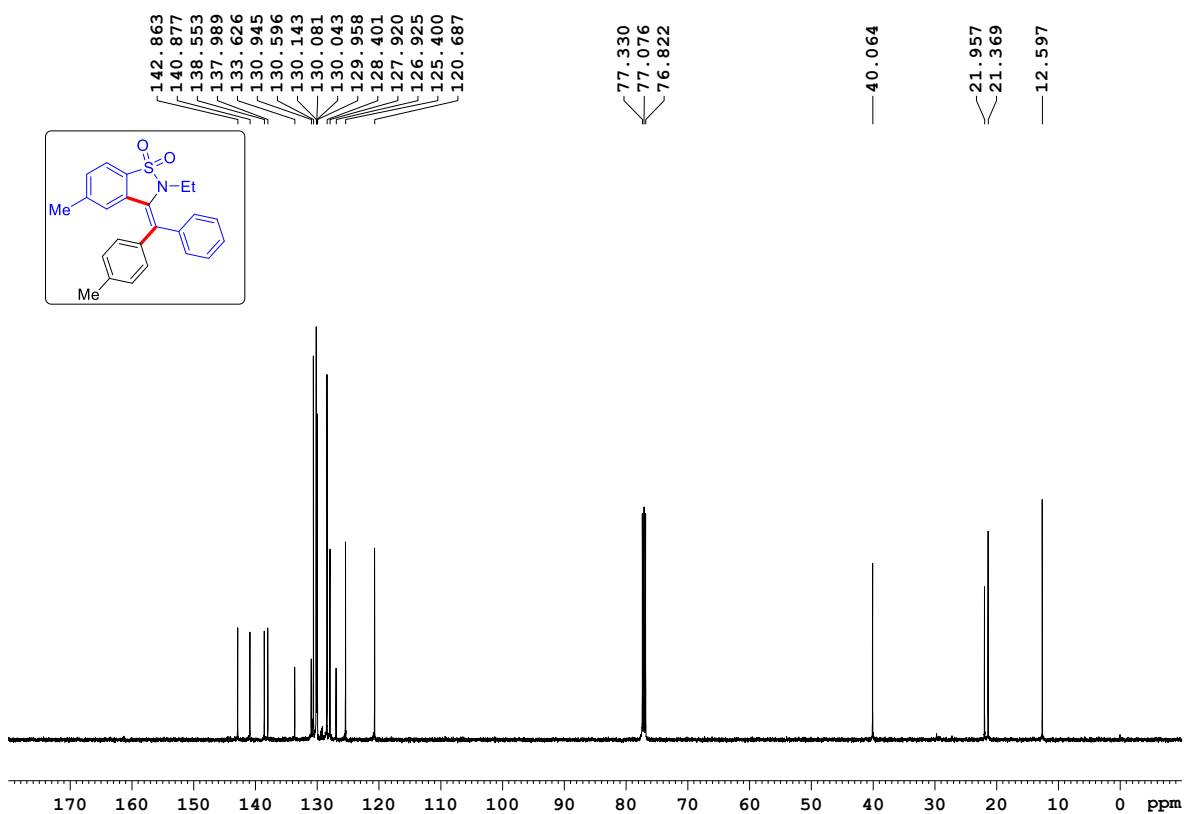


Figure S70. ¹³C NMR spectrum of compound 31b

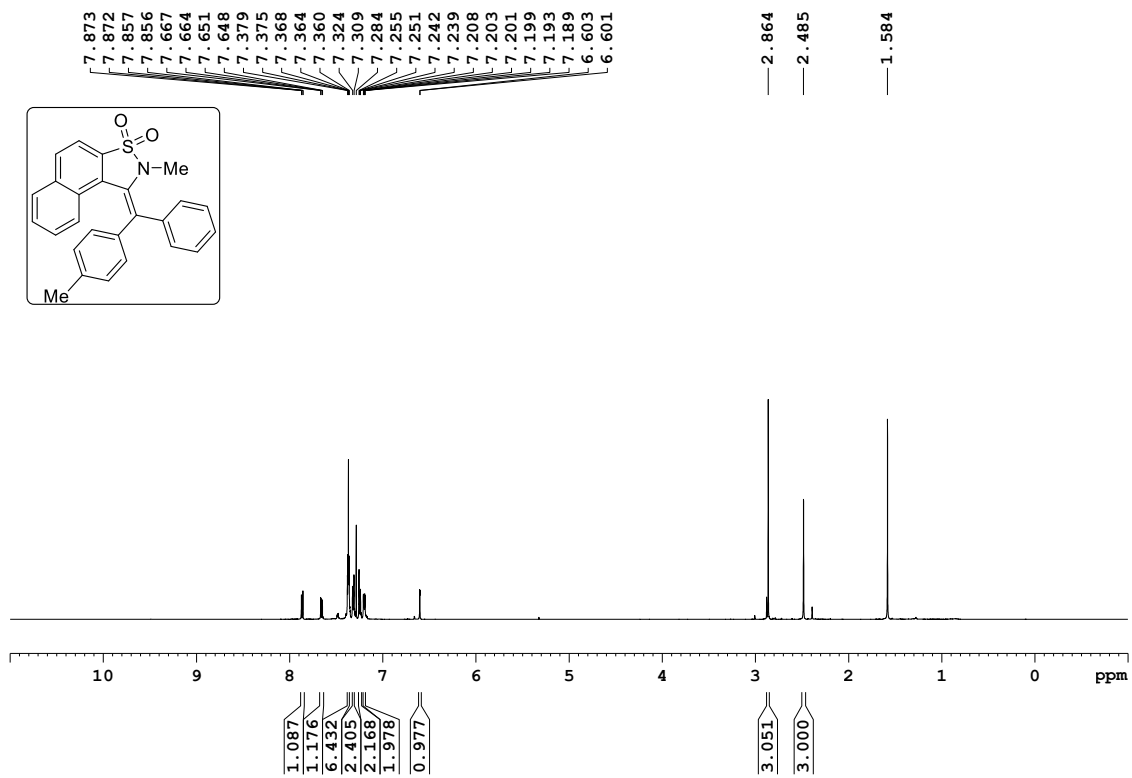


Figure S71. ¹H NMR spectrum of compound **3ob**

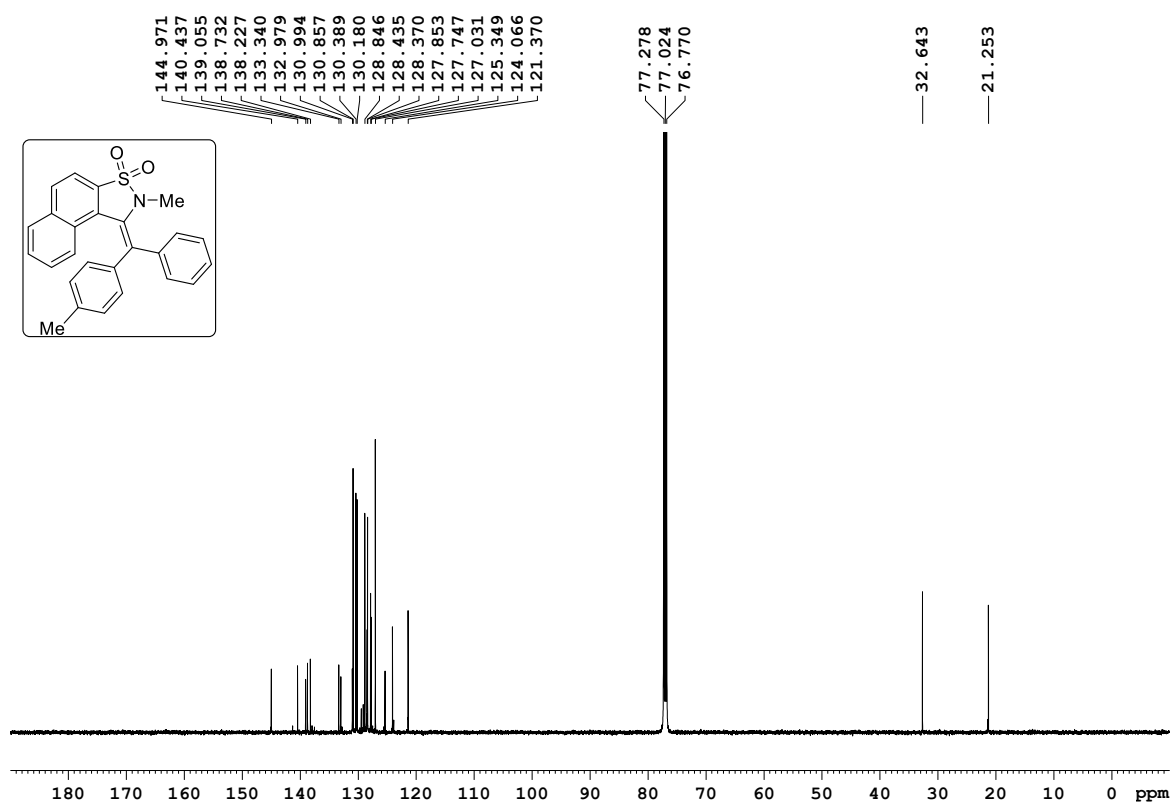


Figure S72. ¹³C NMR spectrum of compound **3ob**

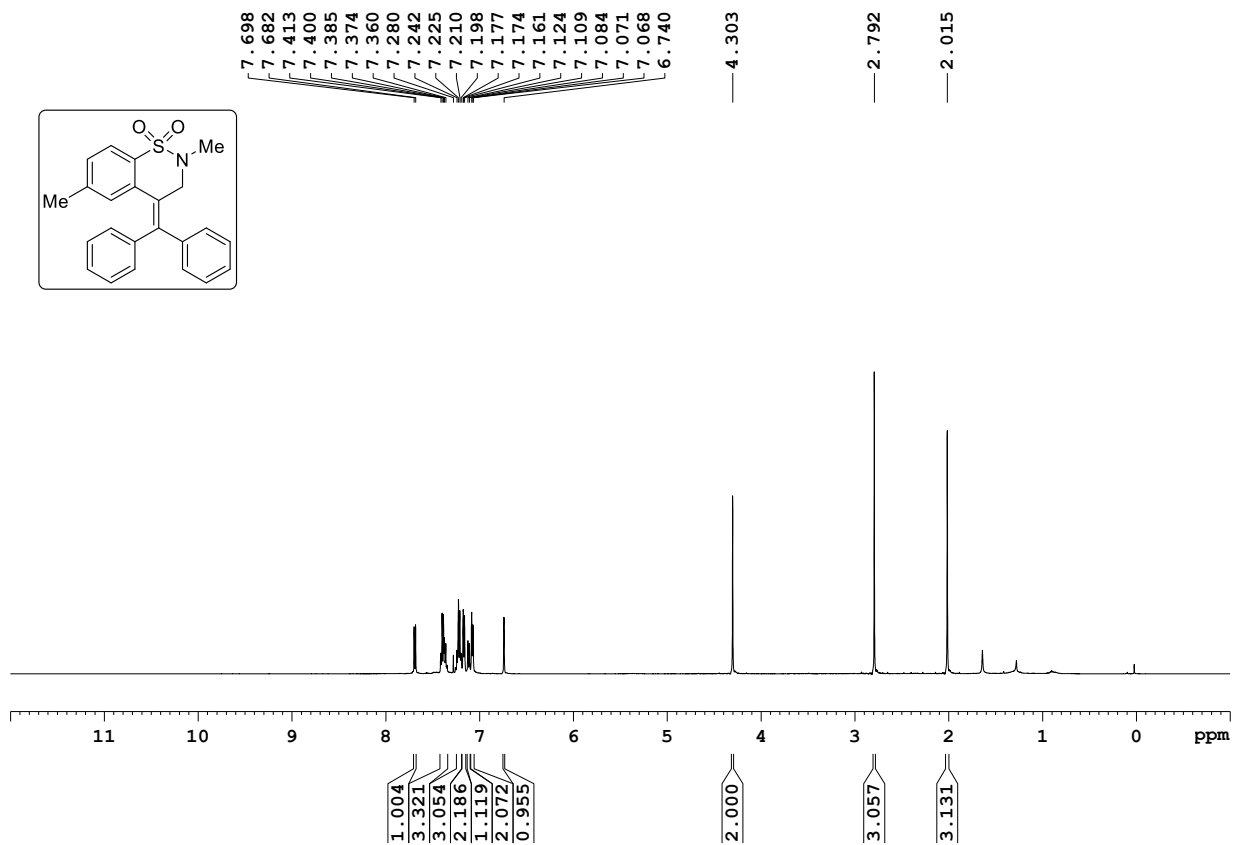


Figure S73. ¹H NMR spectrum of compound 5aa

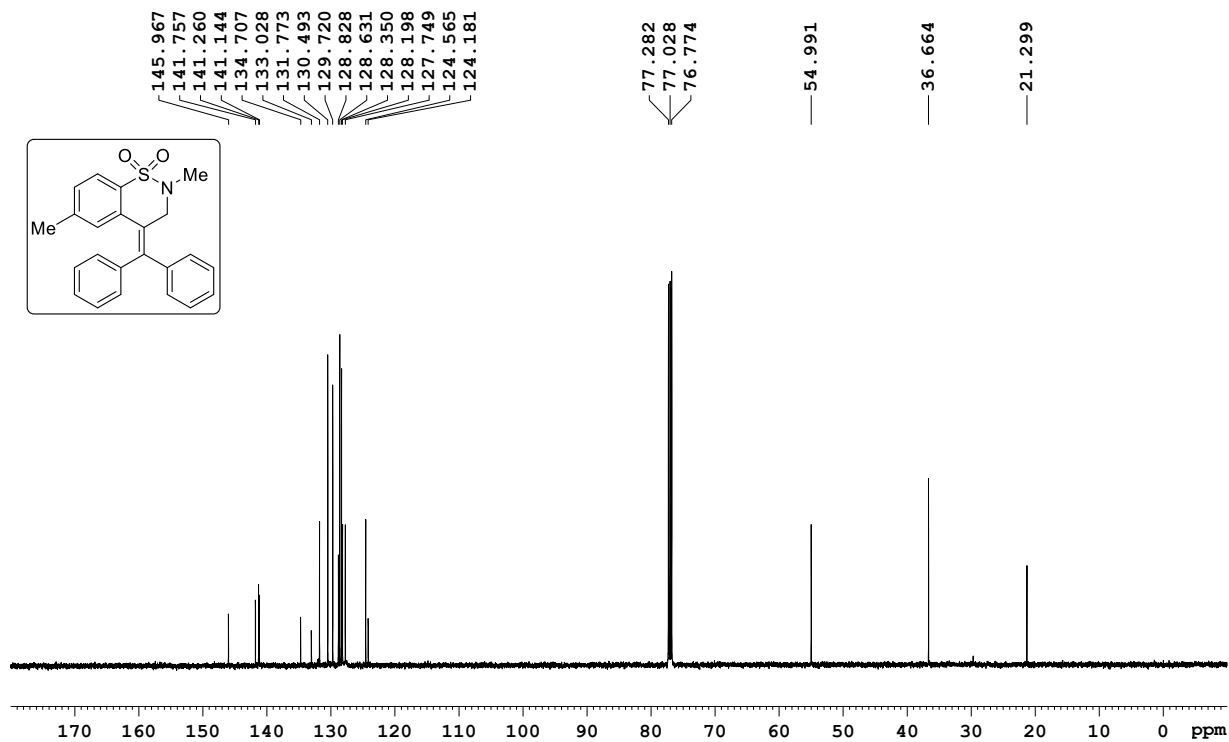


Figure S74. ¹³C NMR spectrum of compound 5aa

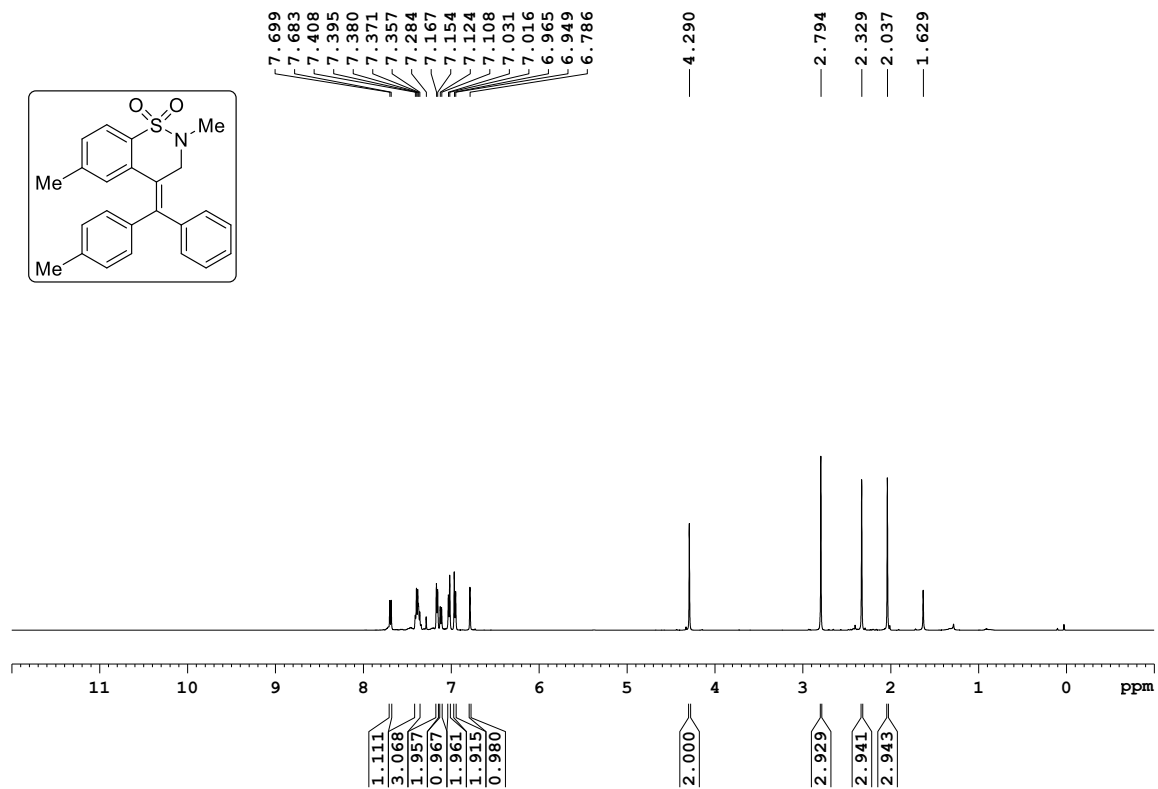


Figure S75. ¹H NMR spectrum of compound **5ab**

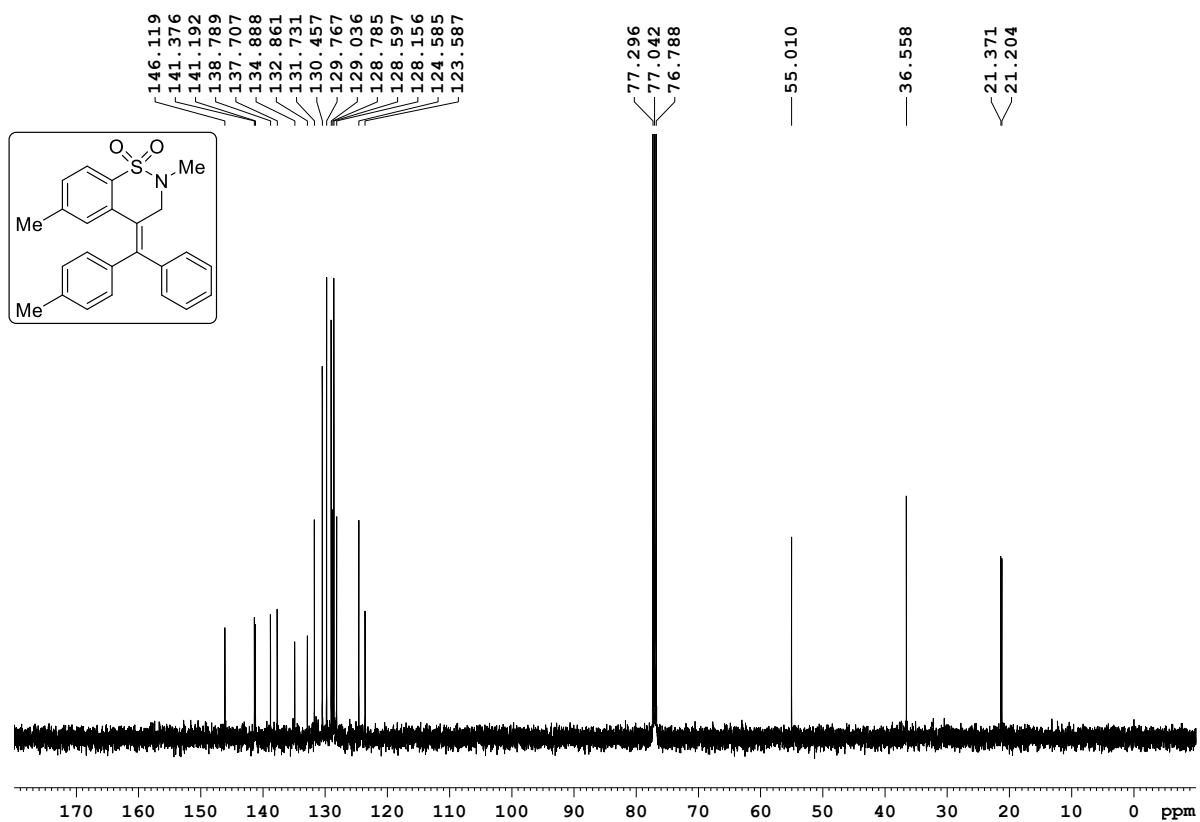


Figure S76. ¹³C NMR spectrum of compound **5ab**

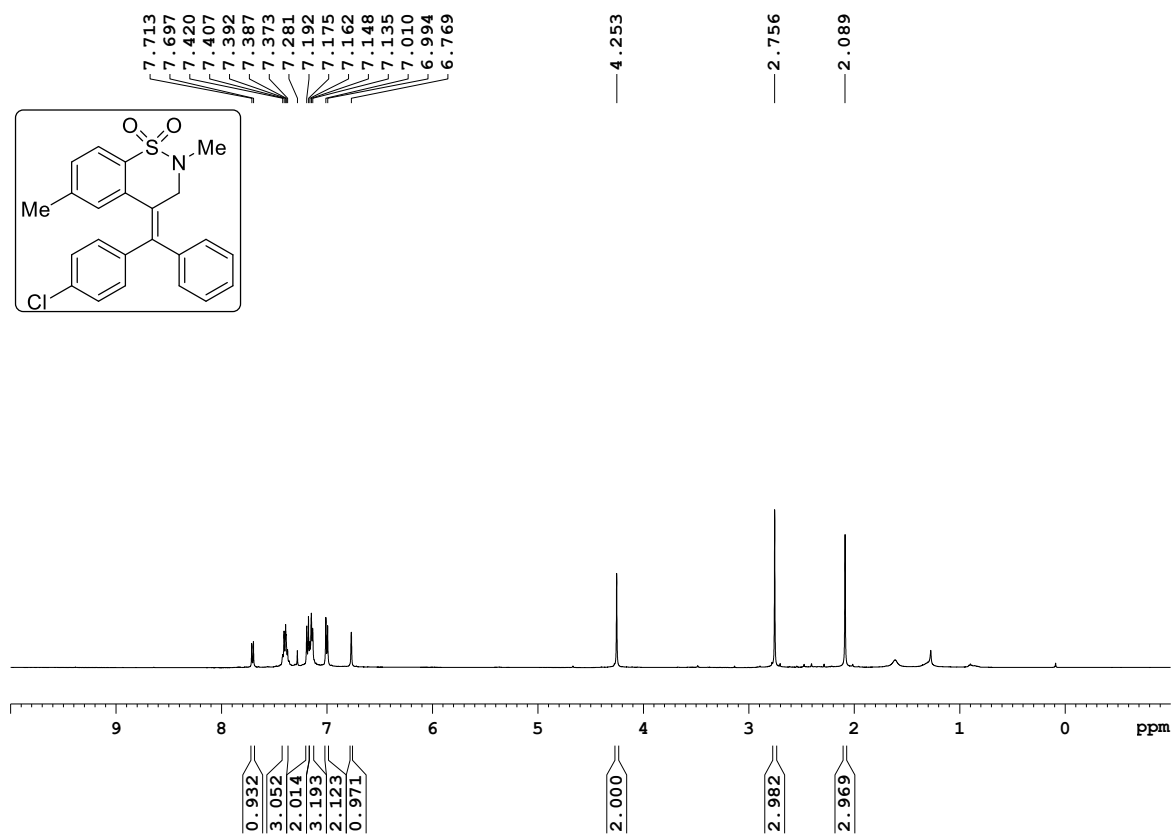


Figure S77. ¹H NMR spectrum of compound 5ad

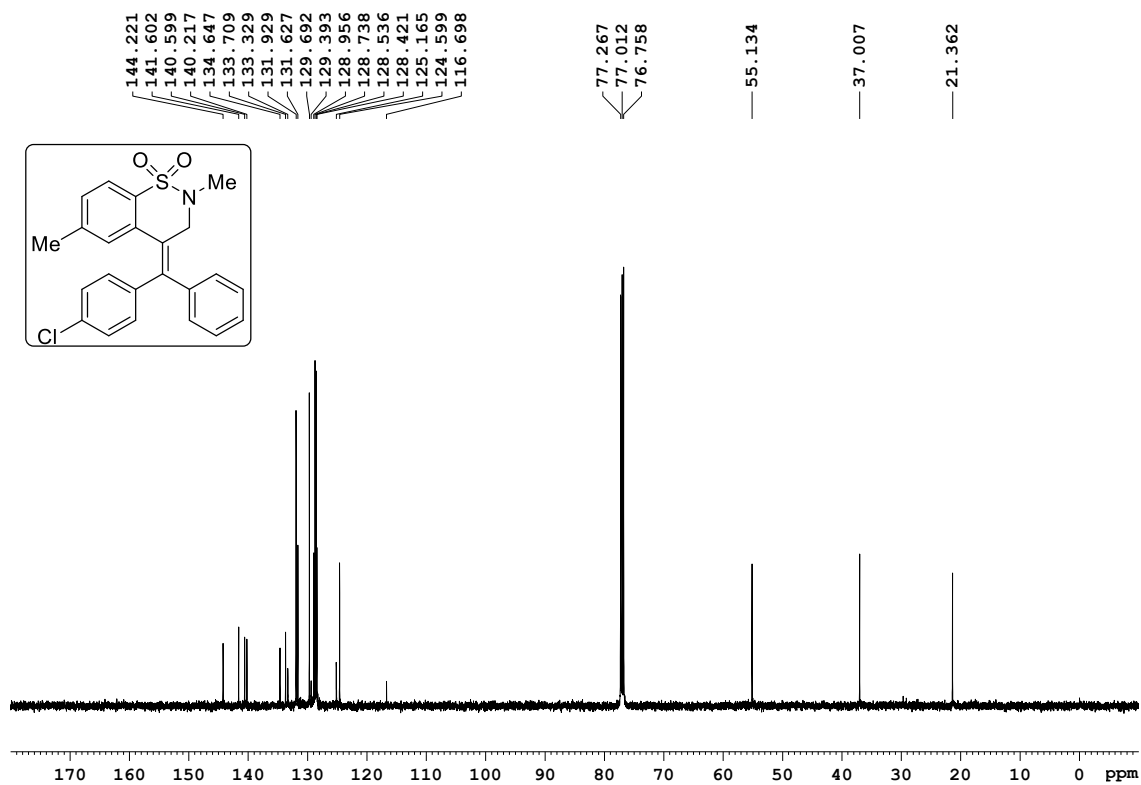


Figure S78. ¹³C NMR spectrum of compound 5ad

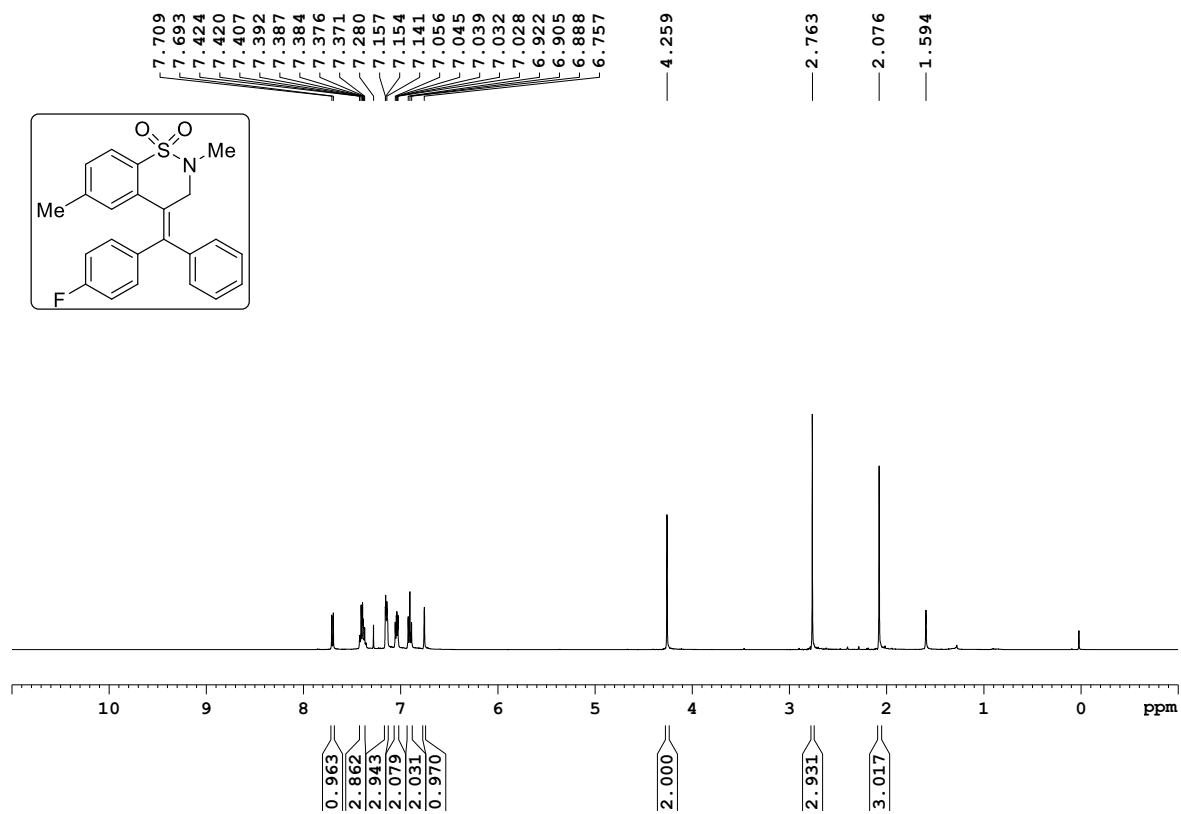


Figure S79. ¹H NMR spectrum of compound 5ae

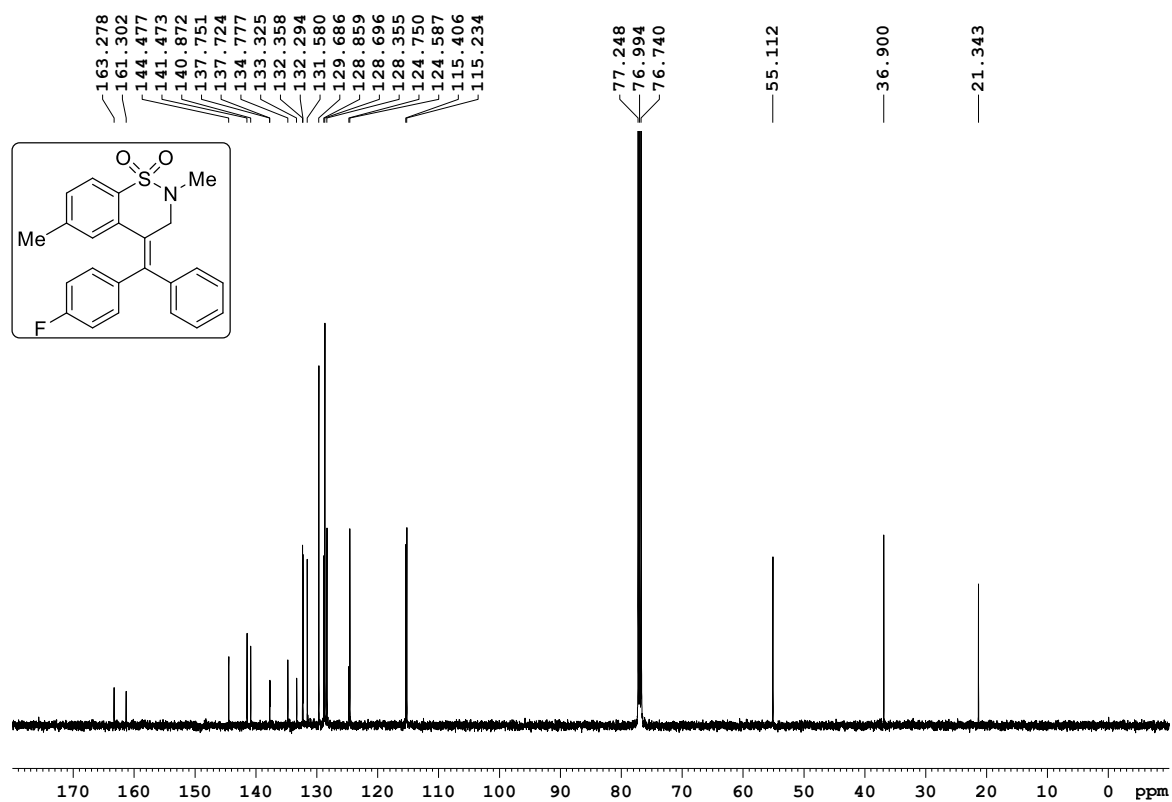


Figure S80. ¹³C NMR spectrum of compound 5ae

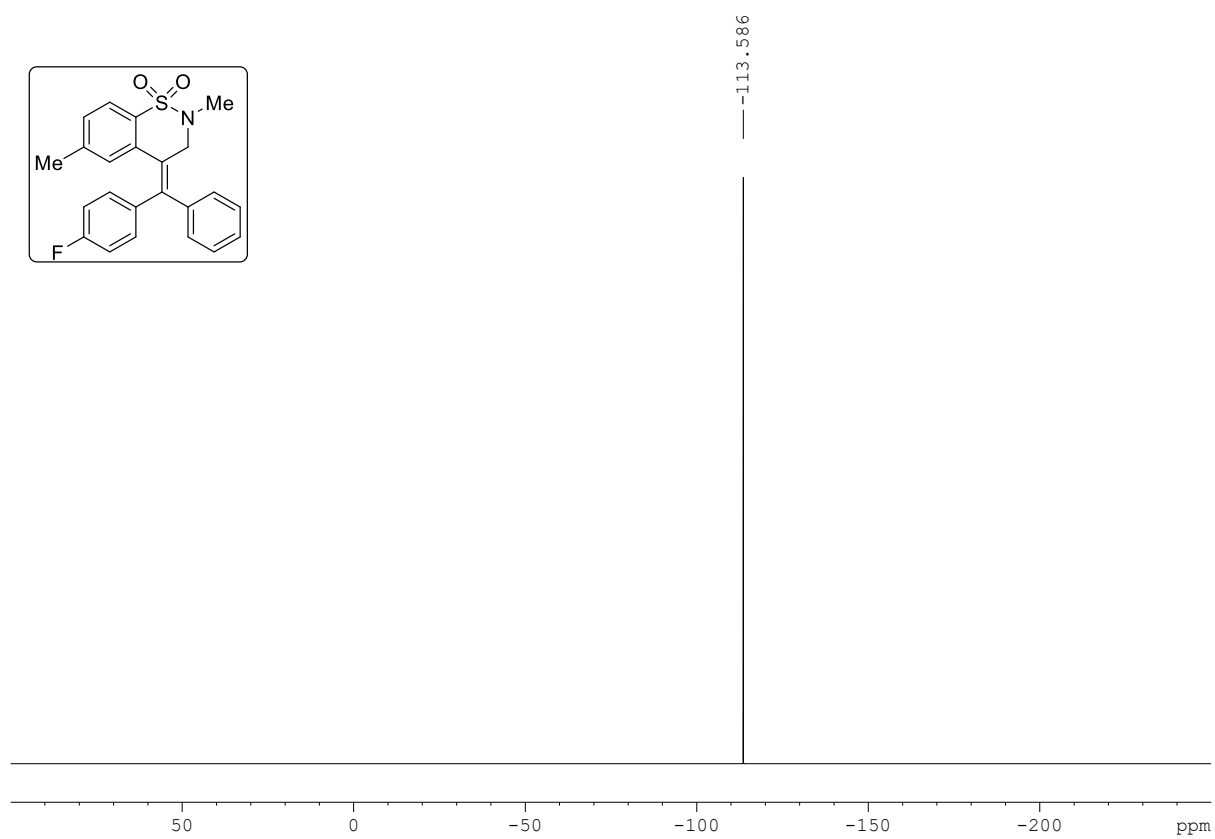


Figure S81. ^{19}F NMR spectrum of compound **5ae**

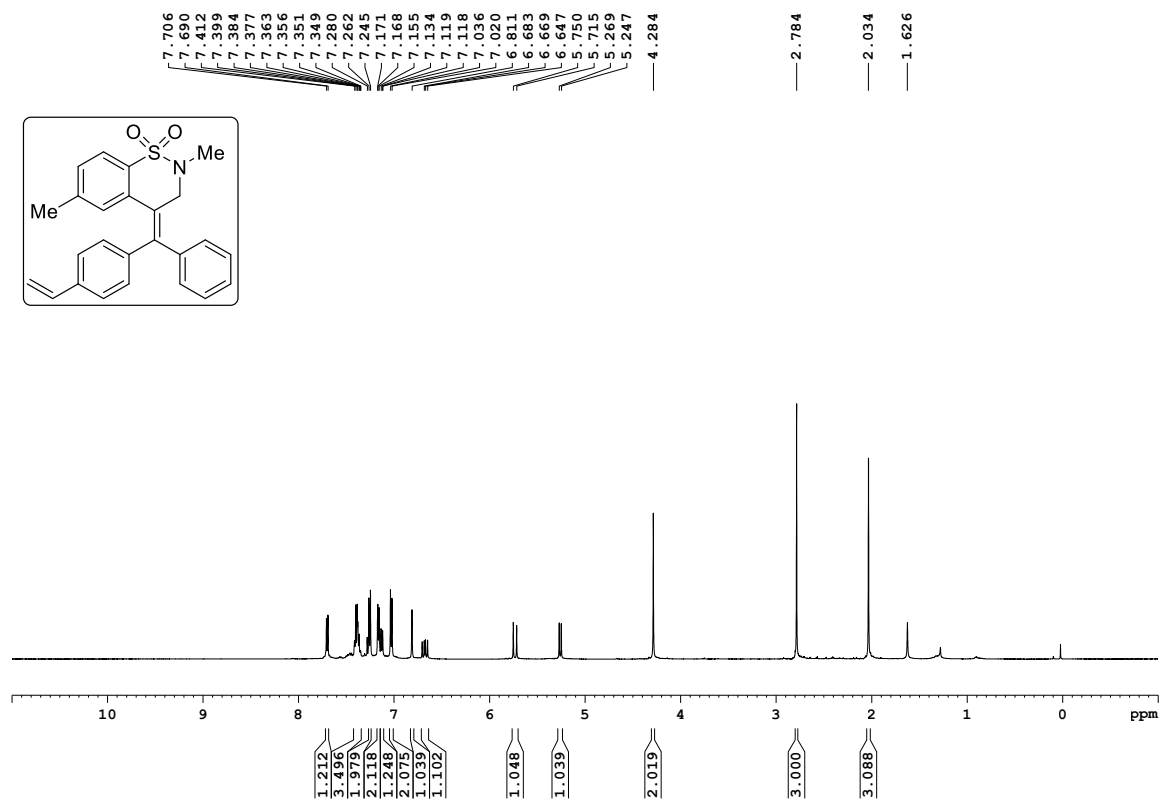


Figure S82. ¹H NMR spectrum of compound **5ag**

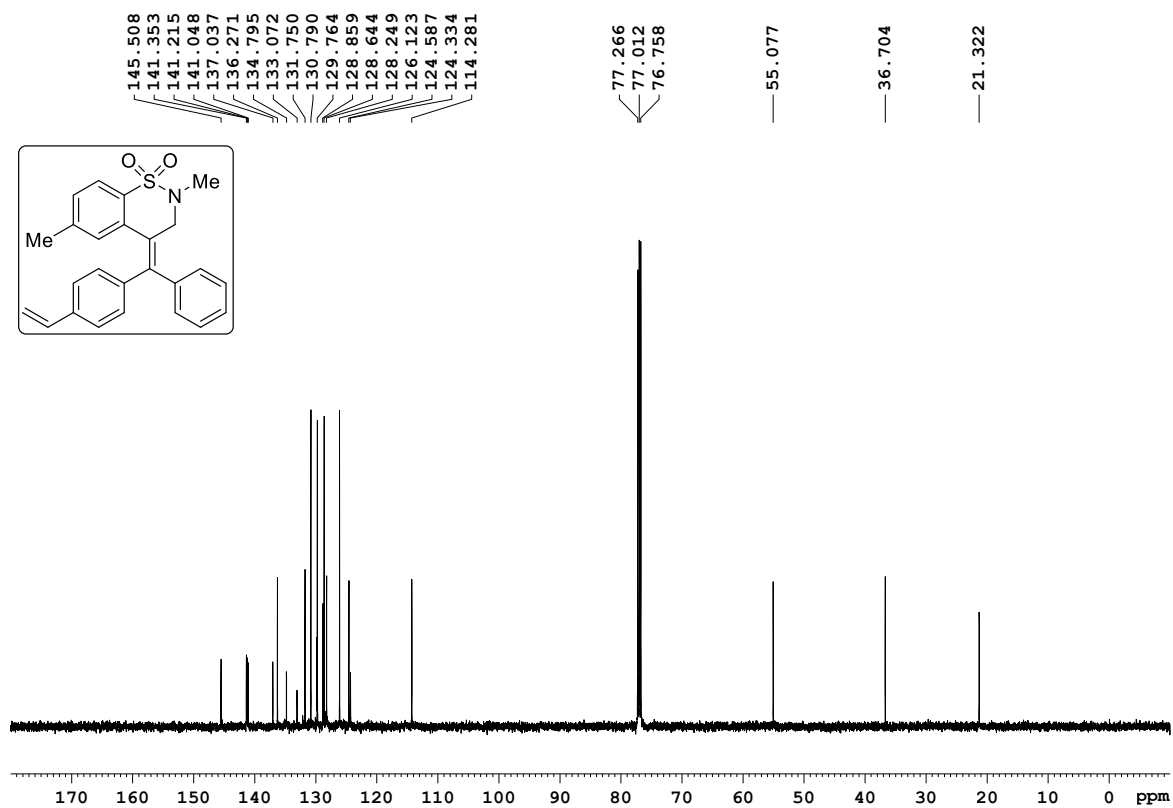


Figure S83. ¹³C NMR spectrum of compound **5ag**

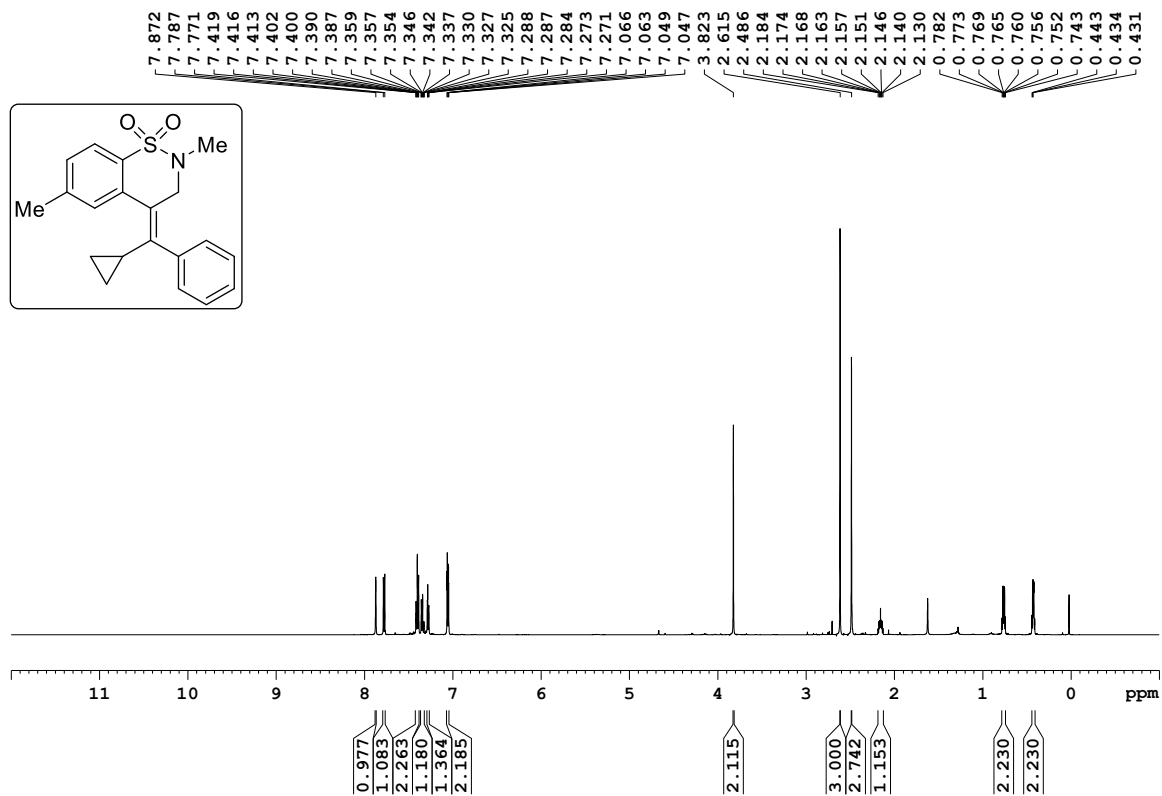


Figure S84. ¹H NMR spectrum of compound 5ah

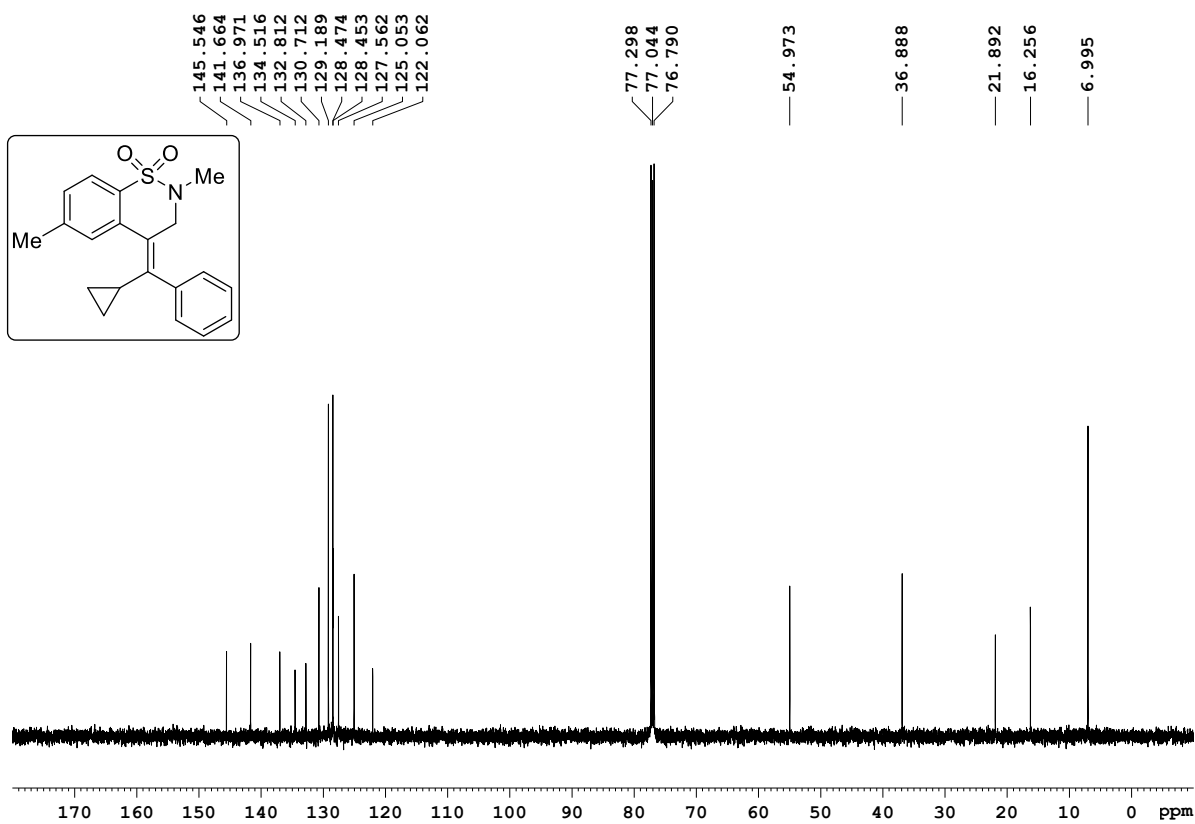


Figure S85. ¹³C NMR spectrum of compound 5ah

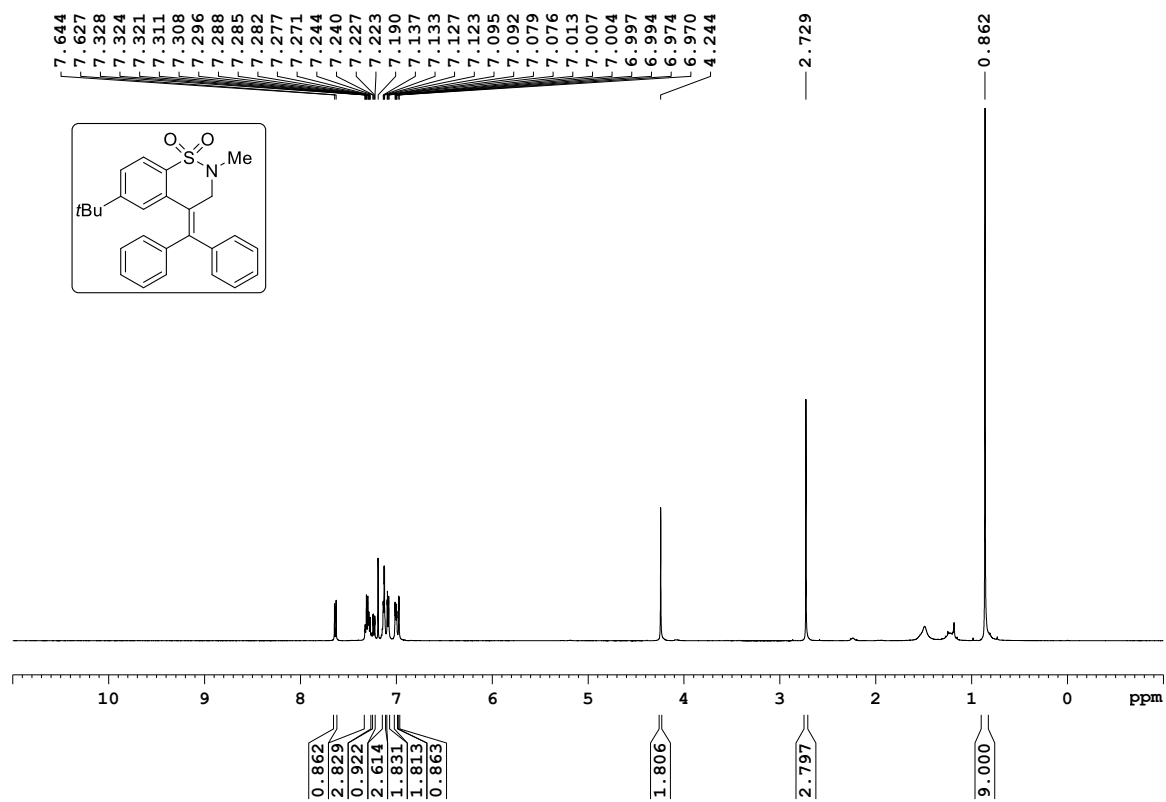


Figure S86. ¹H NMR spectrum of compound 5ba

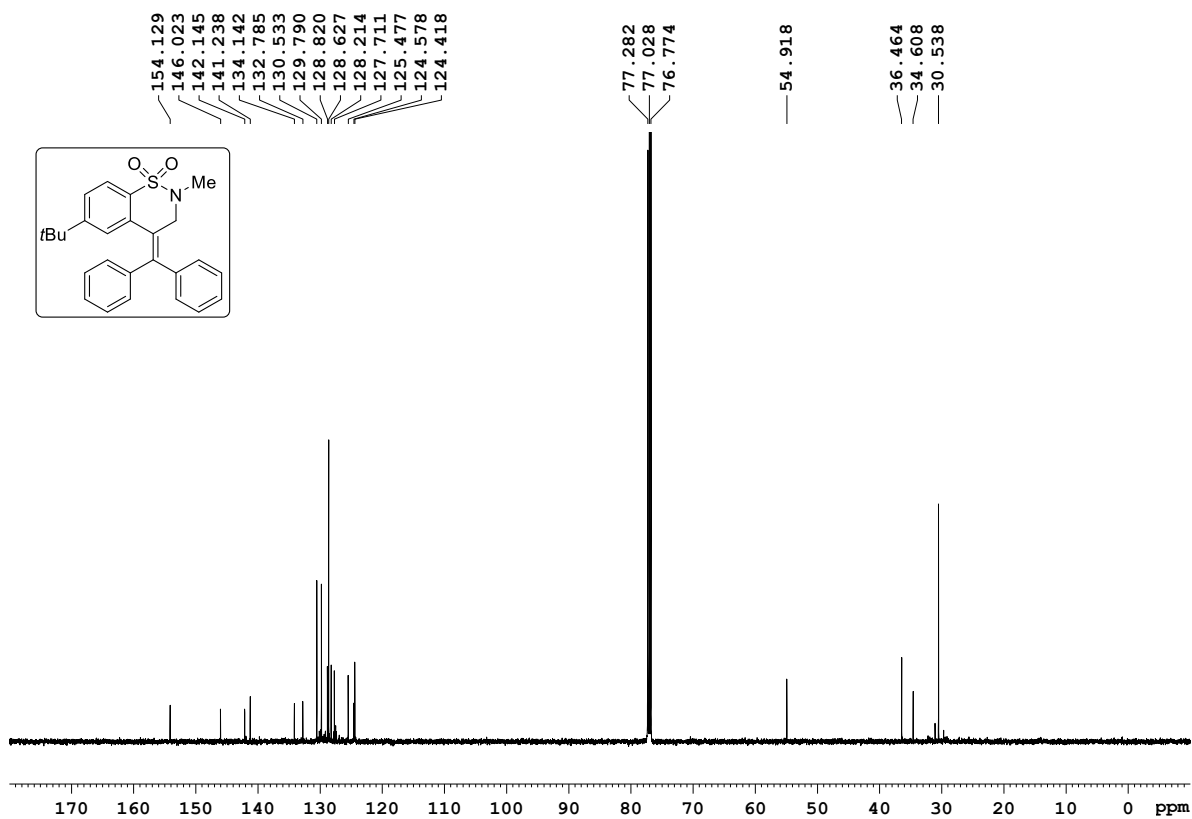


Figure S87. ¹³C NMR spectrum of compound 5ba

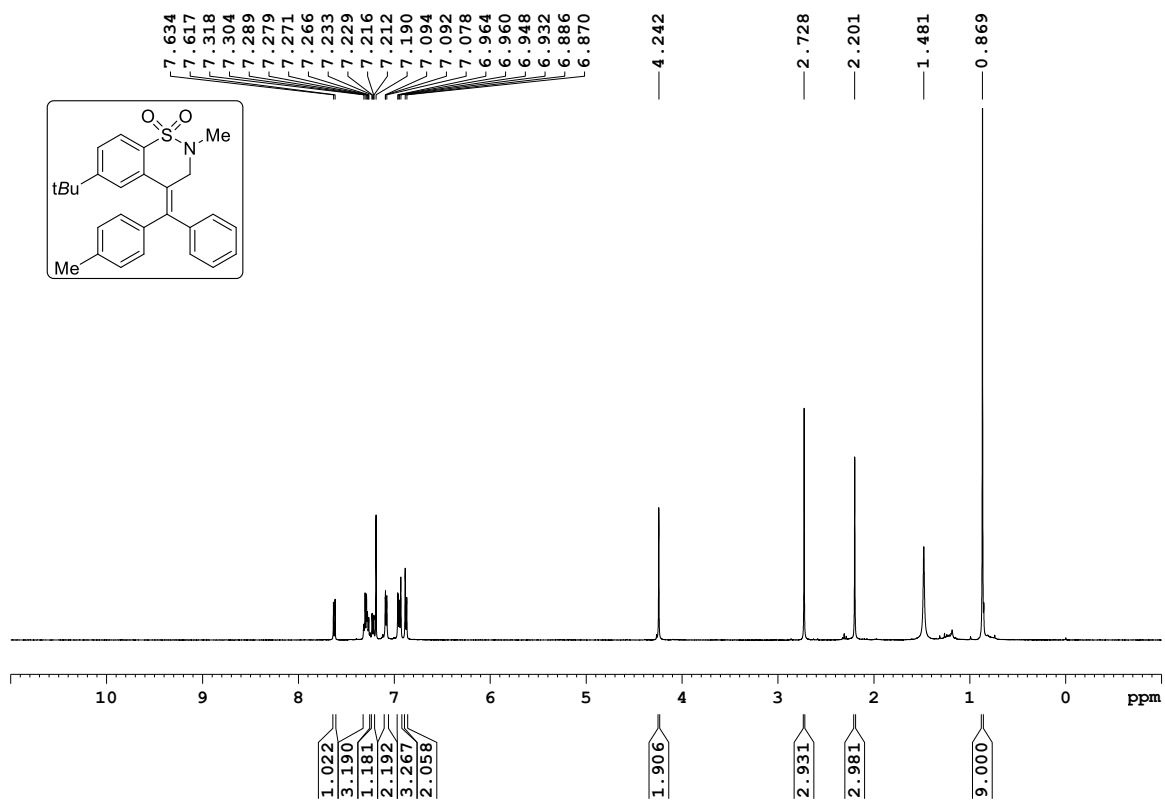


Figure S88. ¹H NMR spectrum of compound **5bb**

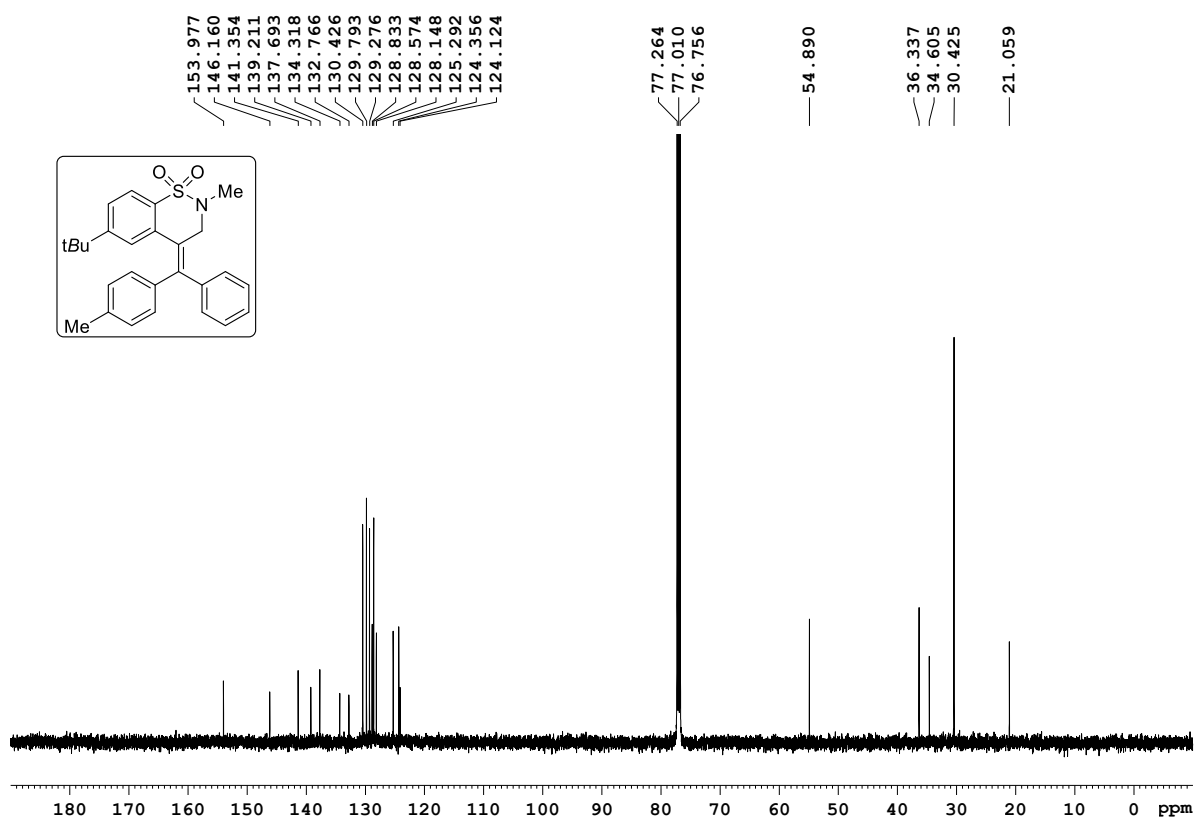


Figure S89. ¹³C NMR spectrum of compound **5bb**

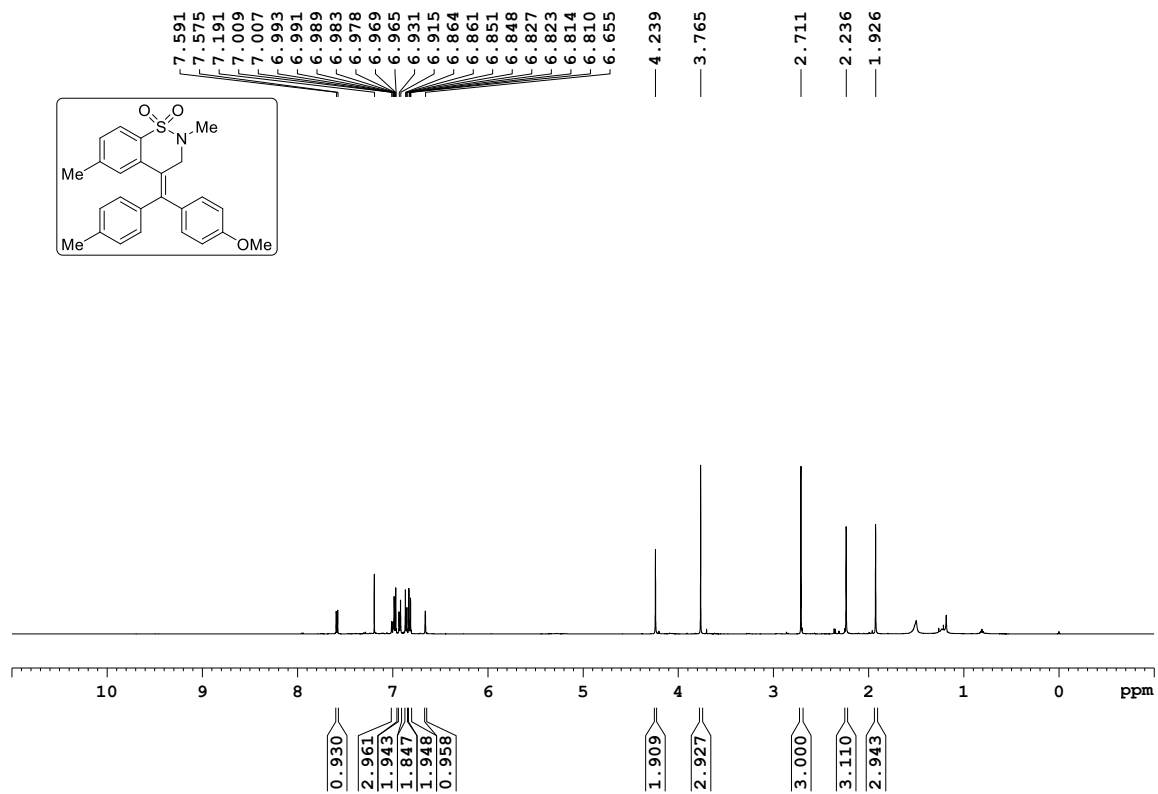


Figure S90. ¹H NMR spectrum of compound 5cb

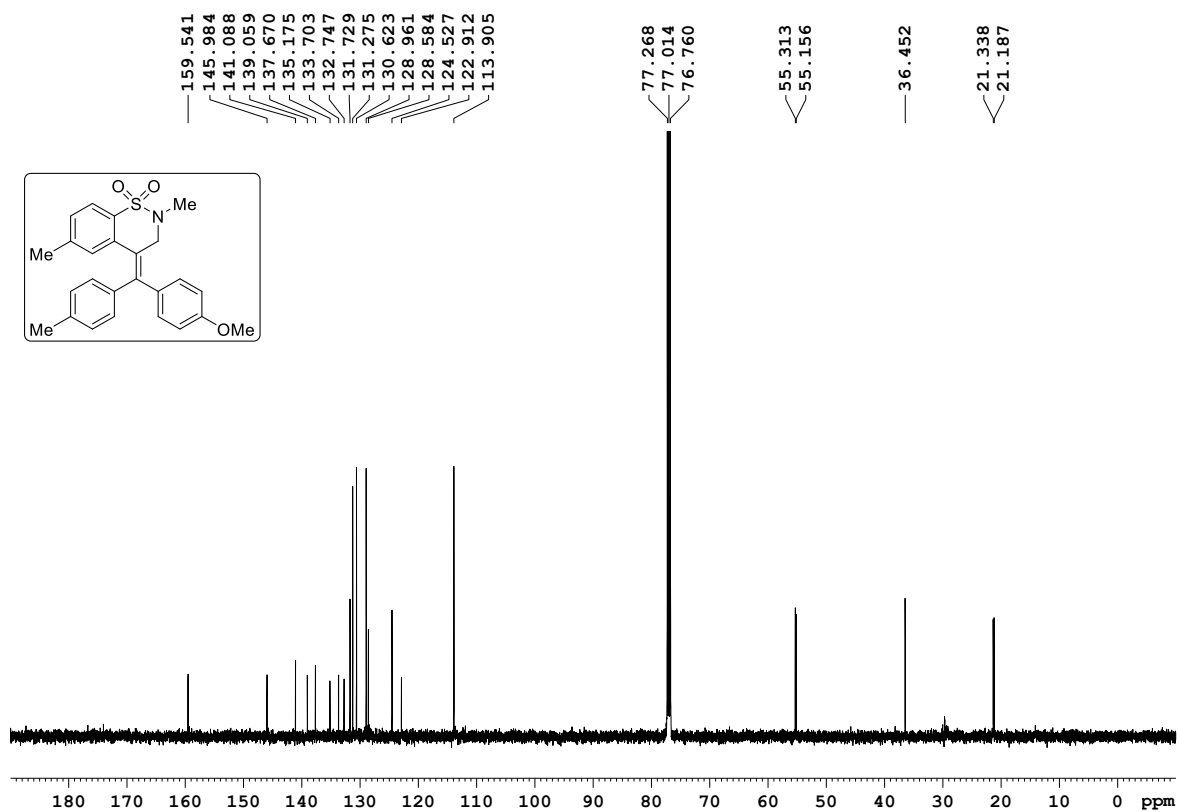


Figure S91. ¹³C NMR spectrum of compound 5cb