

Synthesis of 1,2,4-Trisubstituted-(1*H*)-Imidazoles Through Cu(OTf)₂/I₂ Catalyzed C-C Bond Cleavage of Chalcones and Benzylamines

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HRMS of reaction mixtures for different equivalents of benzylamine with one equivalent of chalcone at different intervals:

For the exploration of mechanism, we have conducted four different experiments using four different concentration of benzylamine in the reaction, ie, 1) both one equivalent of chalcone and benzylamine, 2) one equivalent of chalcone and two equivalent of benzylamine, 3) one equivalent of chalcone and four equivalent of benzylamine, 4) one equivalent of chalcone and six equivalent of benzylamine. All the reaction mixtures subjected for HRMS analysis at three different time intervals of 1 hour, 3 hour, and 6 hour respectively for finding the intermediates formed. After 1 hour of the reaction time, for both the reactions with one equivalent of benzylamine and two equivalent of benzylamine, HRMS analysis displayed the intermediate formation in the spectra (**Figure. S1, Figure S4**). However as the time progresses it clearly showed the imidazole formation $[(M+H)^+ = 311.1504]$. For higher equivalents of benzylamine, as the time progresses, it clearly displayed the peak for imidazole formation by decreasing the intensity of intermediate peaks (**Figure S7- Figure S12**). However in all the cases, we were unable to isolate the intermediates formed.

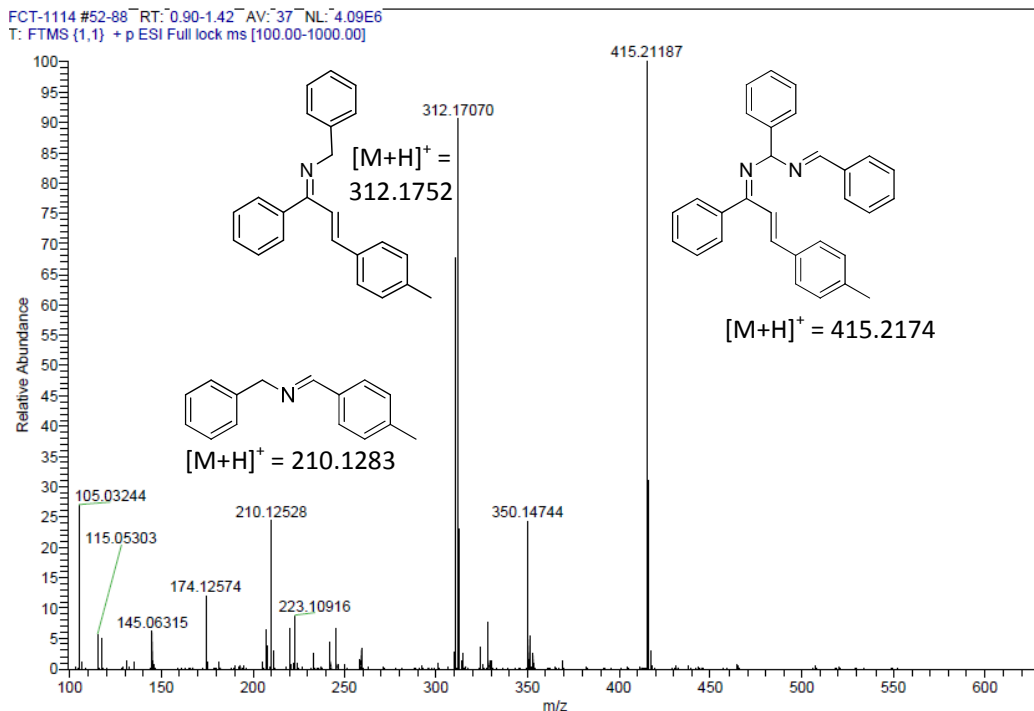


Figure S1. HRMS for the reaction mixture of 1 equivalent of benzylamine and chalcone after 1 hour of the reaction time.

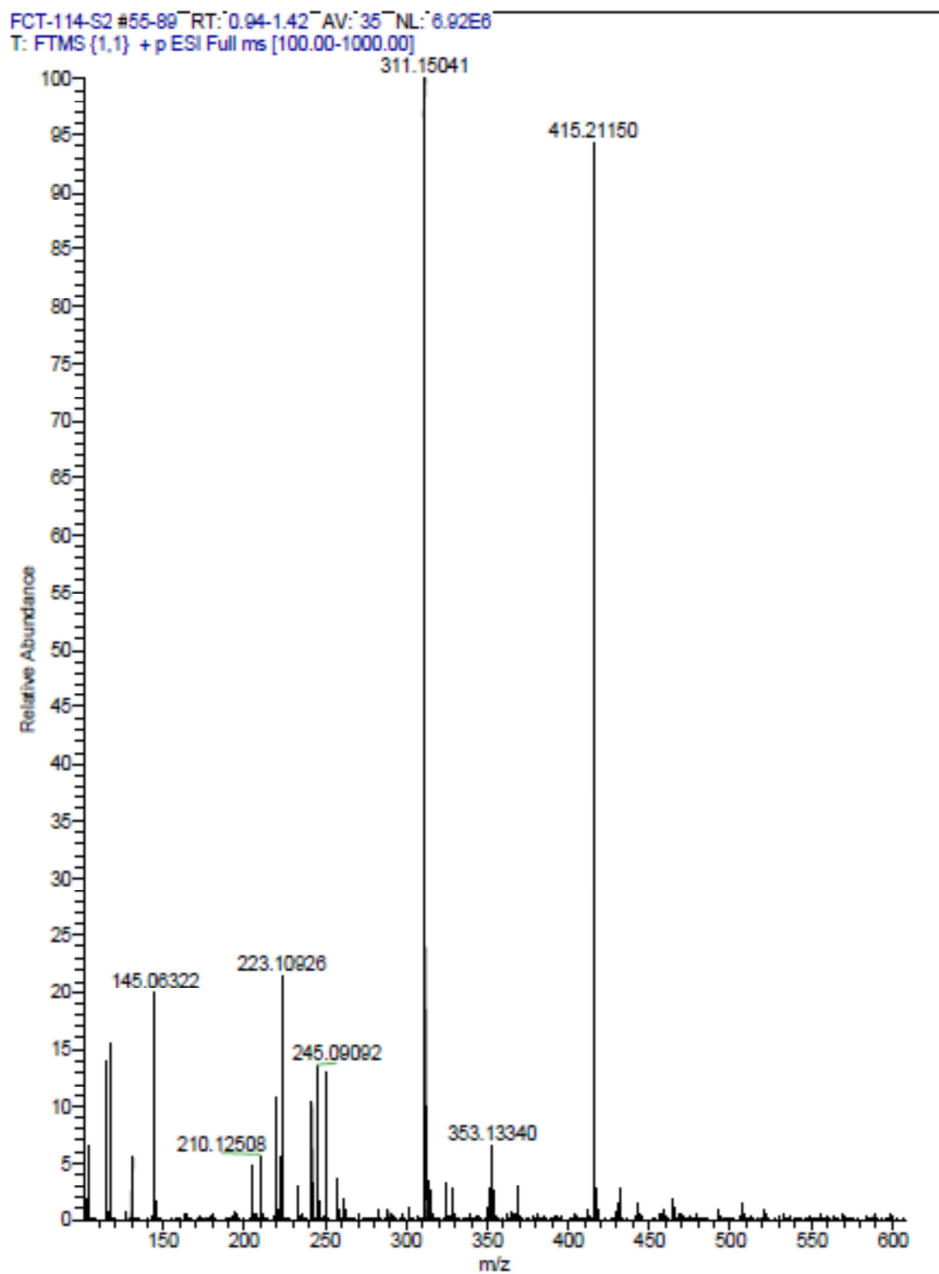


Figure S2. HRMS for the reaction mixture of 1 equivalent of benzylamine and chalcone after 3 hours of the reaction time.

FCT-114-S3 #51-78 RT: 0.88-1.25 AV: 28 NL: 1.38E7
T: FTMS (1,1) +p ESI Full lock ms [100.00-1000.00]

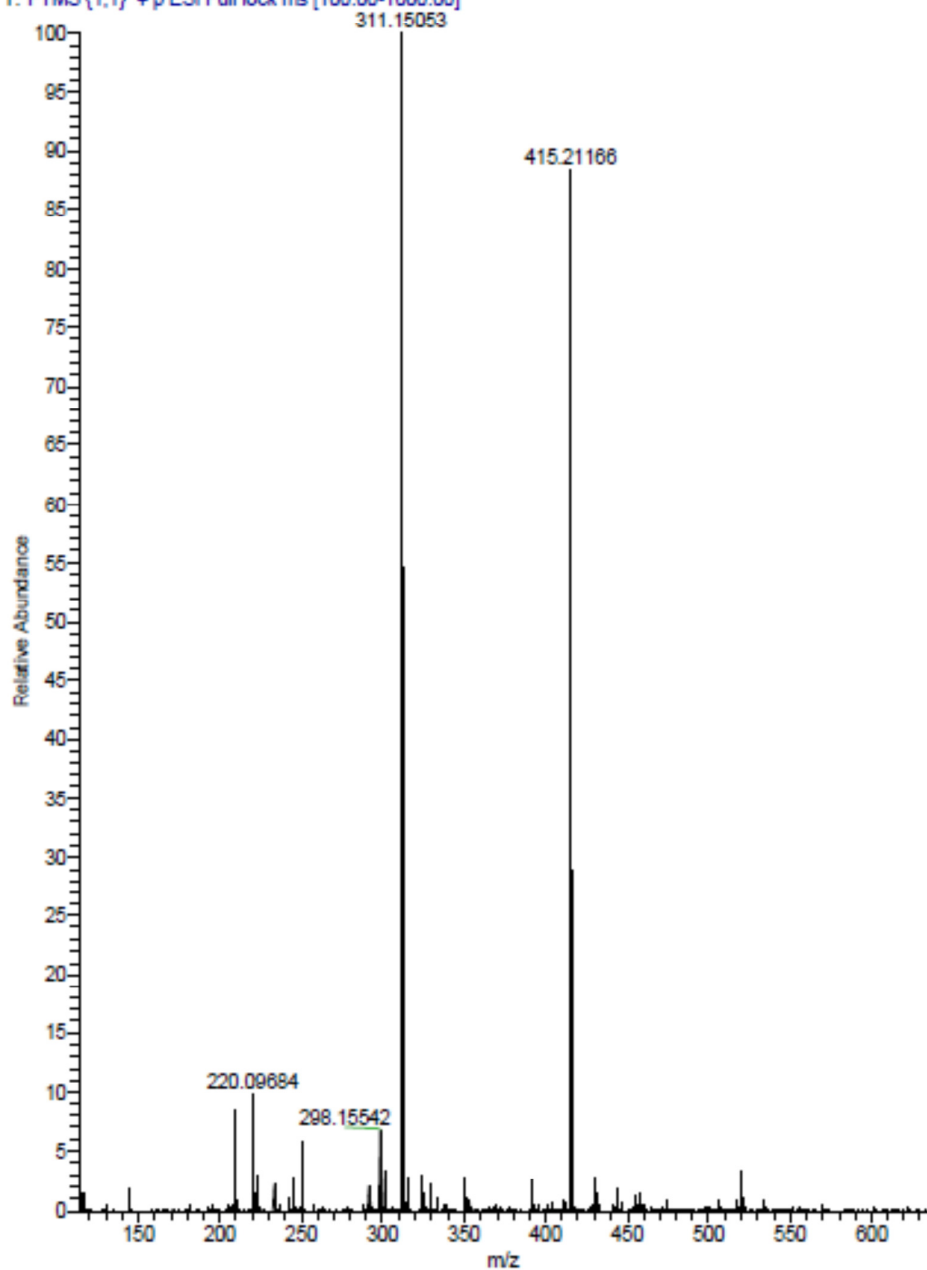


Figure S3. HRMS for the reaction mixture of 1 equivalent of benzylamine and chalcone after 6 hours of the reaction time.

FCT-1115 #53-81 RT: 0.92-1.31 AV: 29 NL: 6.39E6
T: FTMS (1,1) + p ESI Full ms [100.00-1000.00]

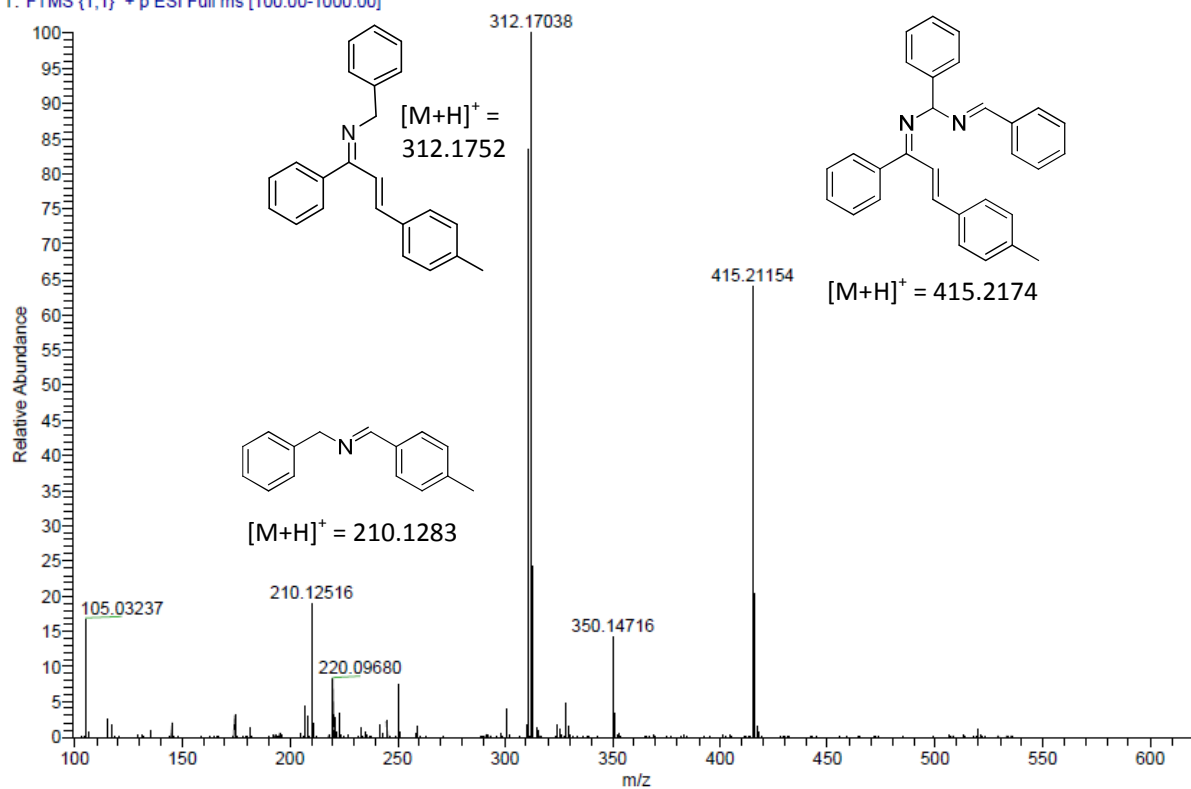


Figure S4. HRMS for the reaction mixture of 2 equivalents of benzylamine and 1 equivalent of chalcone after 1 hour of the reaction time.

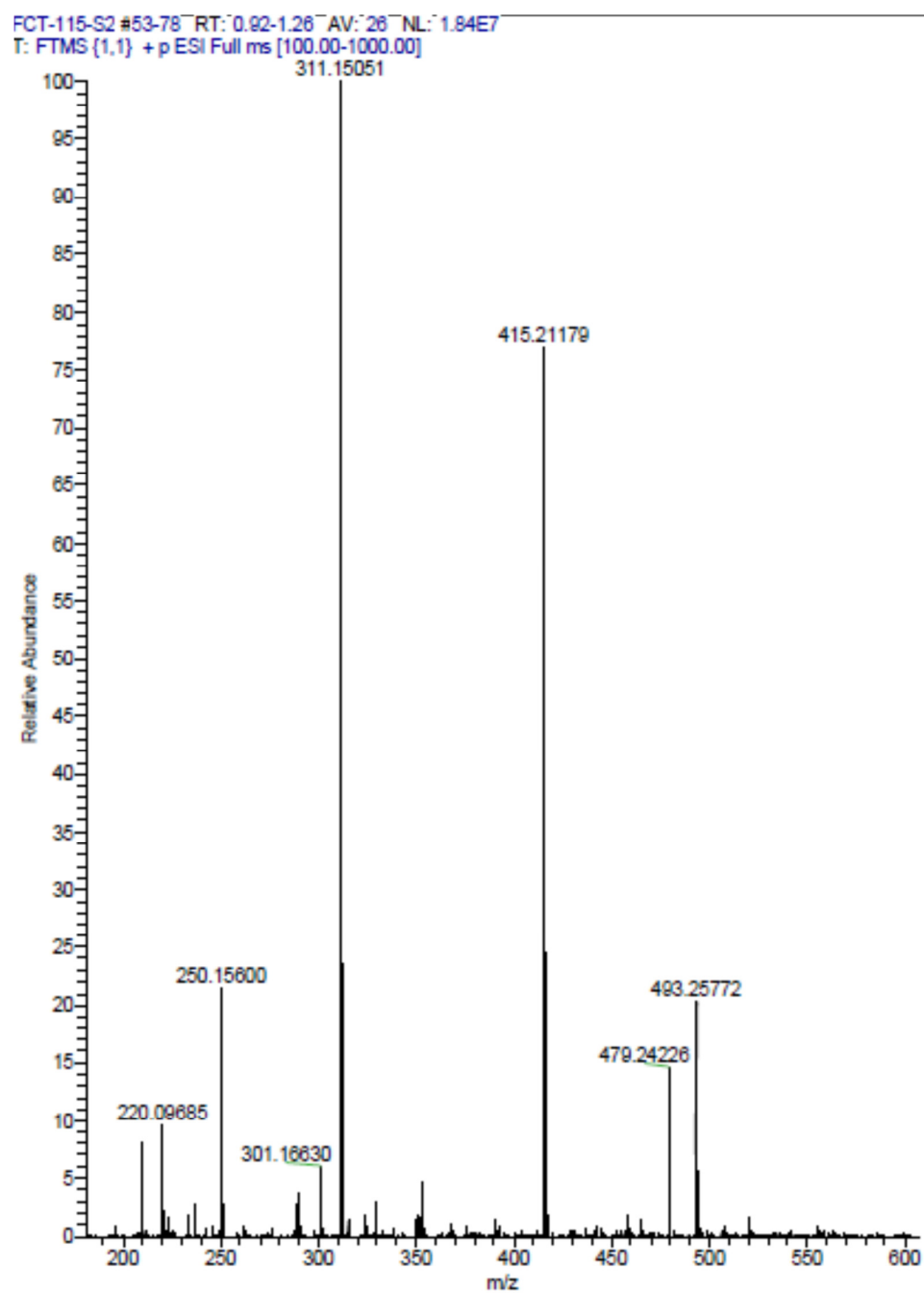


Figure S5. HRMS for the reaction mixture of 2 equivalents of benzylamine and 1 equivalent of chalcone after 3 hours of the reaction time.

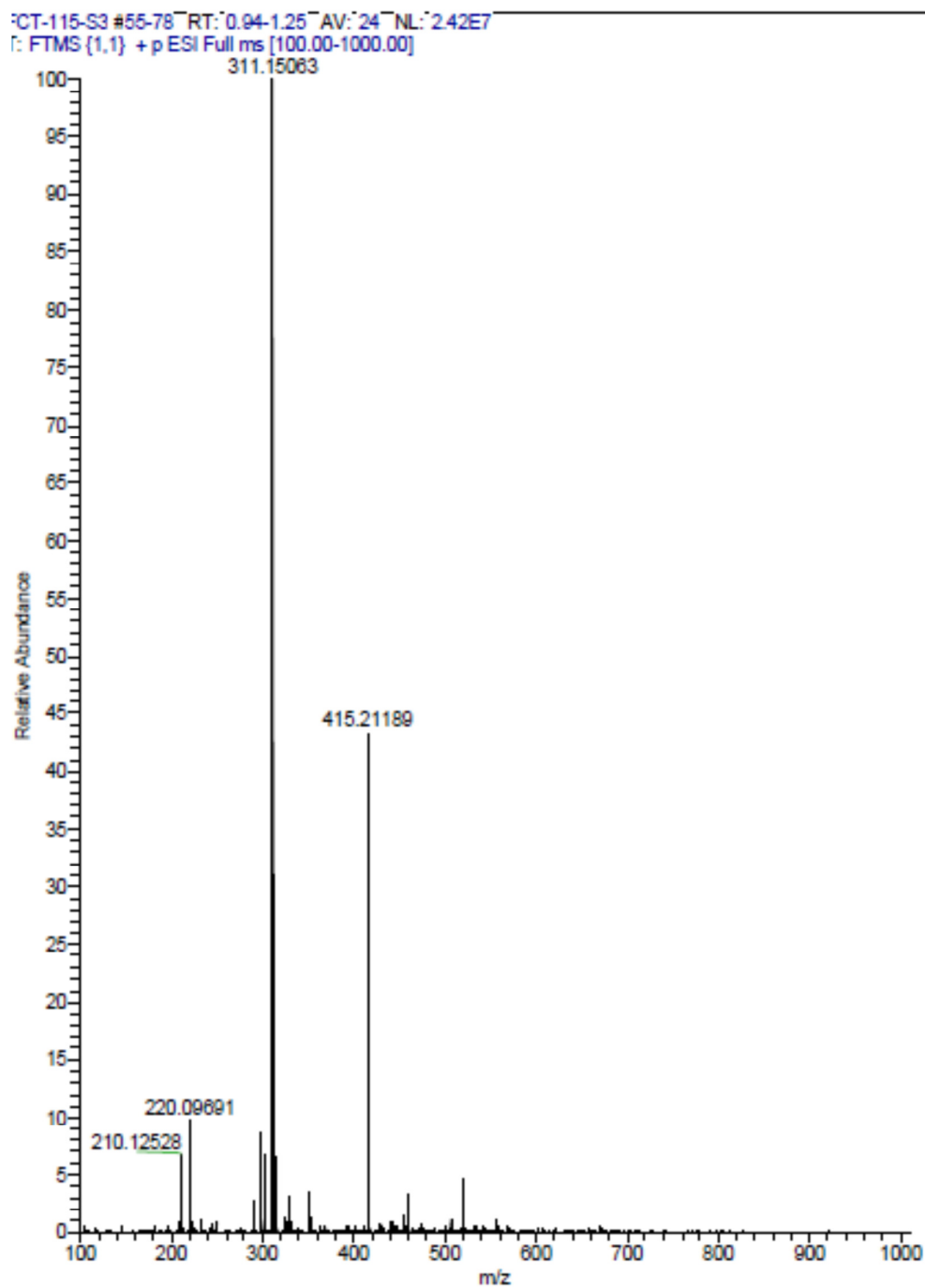


Figure S6. HRMS for the reaction mixture of 2 equivalents of benzylamine and 1 equivalent of chalcone after 6 hours of the reaction time.

FCT-1116 #55-80 RT: 0.94-1.28 AV: 26 NL: 6.42E7
T: FTMS (1,1) + p ESI Full ms [100.00-1000.00]

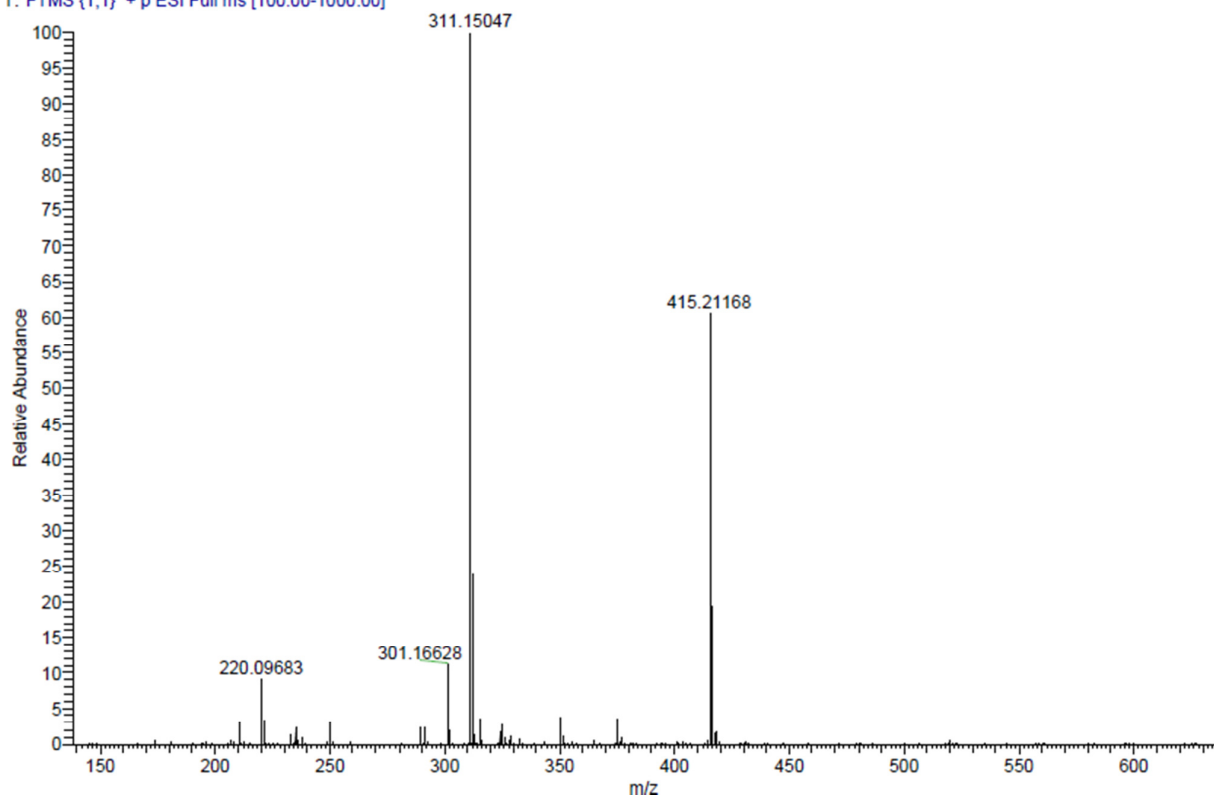


Figure S7. HRMS for the reaction mixture of 4 equivalents of benzylamine and 1 equivalent of chalcone after 1 hour of the reaction time.

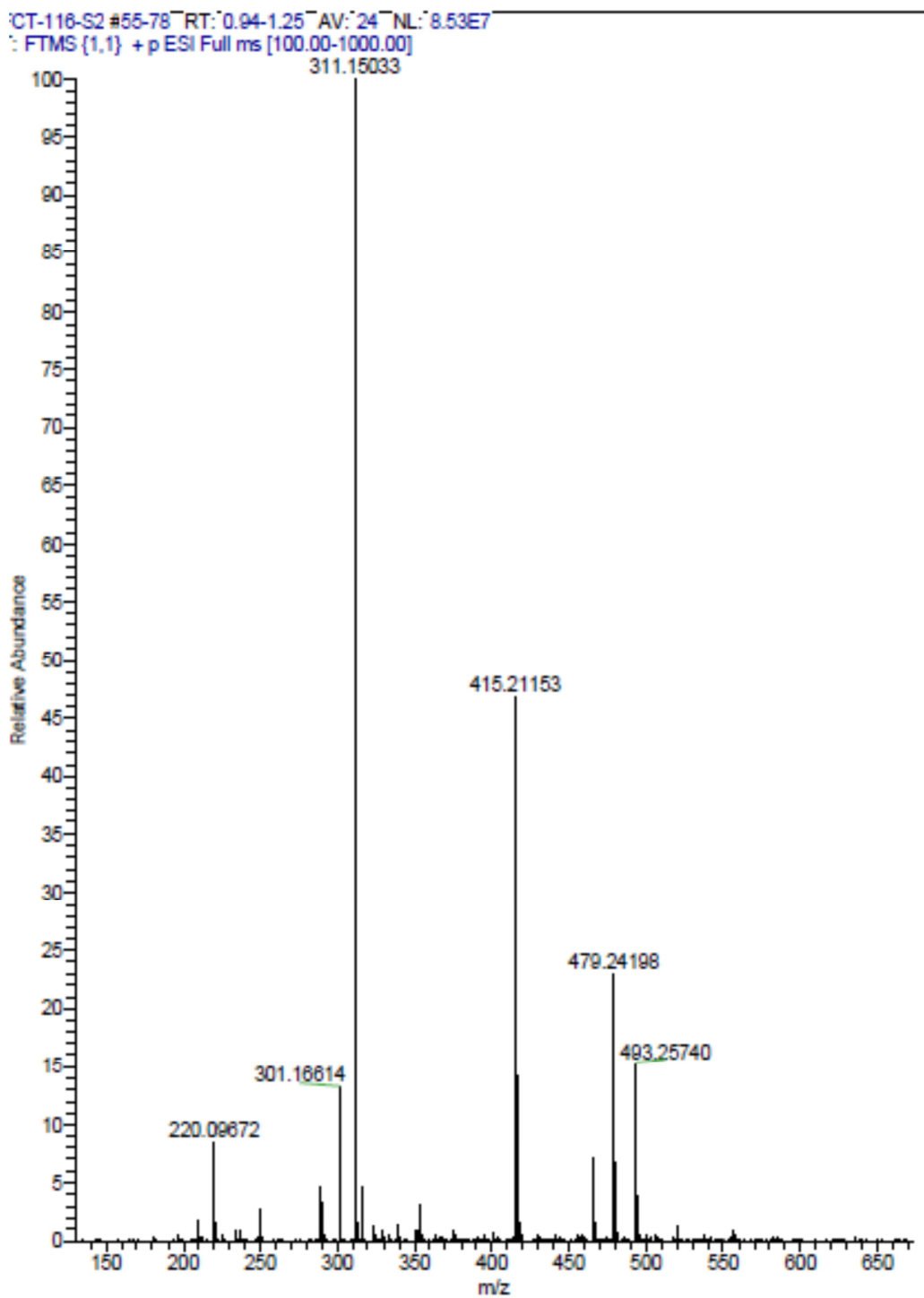


Figure S8. HRMS for the reaction mixture of 4 equivalents of benzylamine and 1 equivalent of chalcone after 3 hours of the reaction time.

FCT-1116-S3 #56-82 RT: 0.94-1.29 AV: 27 NL: 1.16E8
T: FTMS (1,1) + p ESI Full ms [100.00-1000.00]

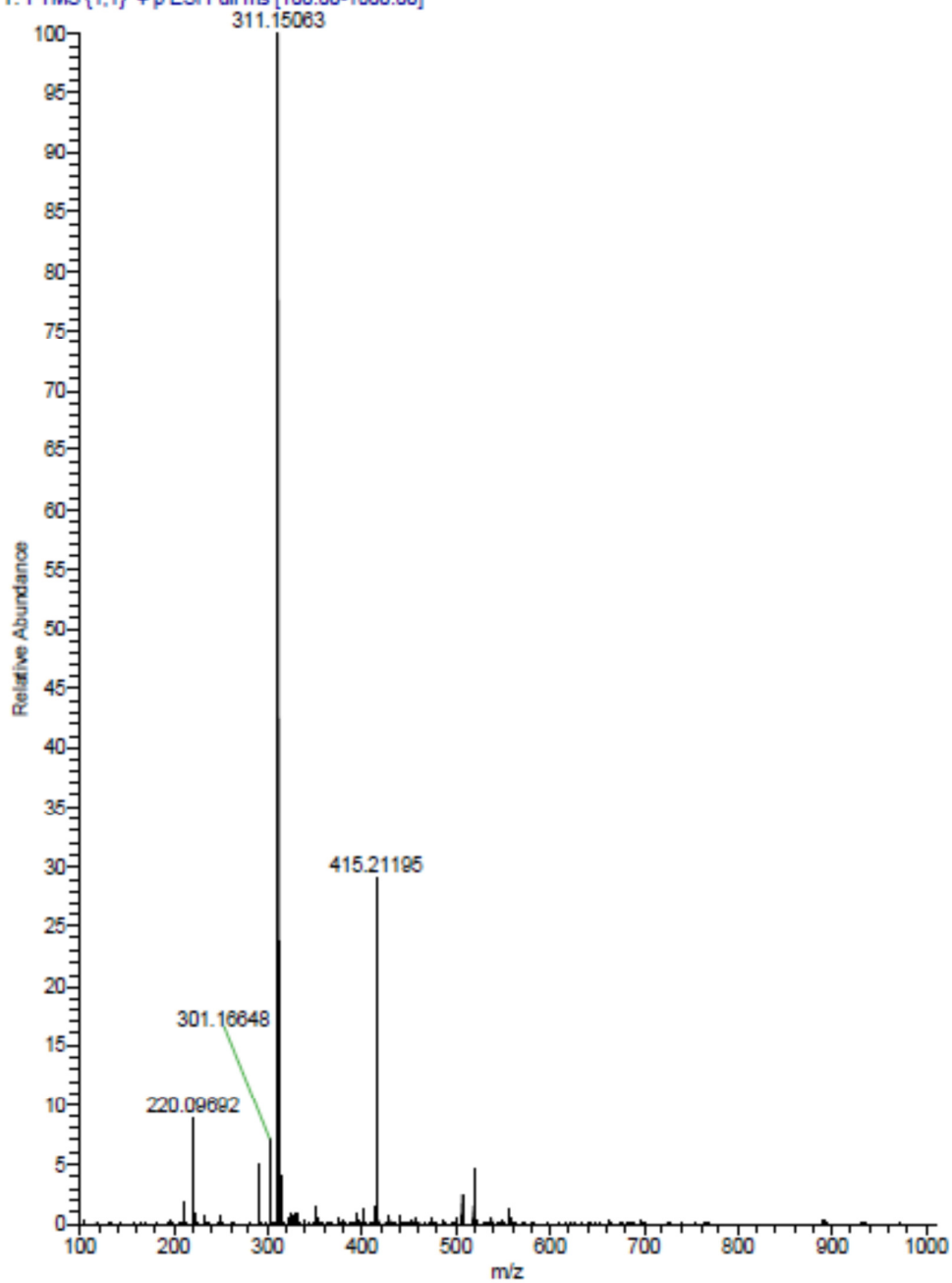


Figure S9. HRMS for the reaction mixture of 4 equivalents of benzylamine and 1 equivalent of chalcone after 6 hours of the reaction time.

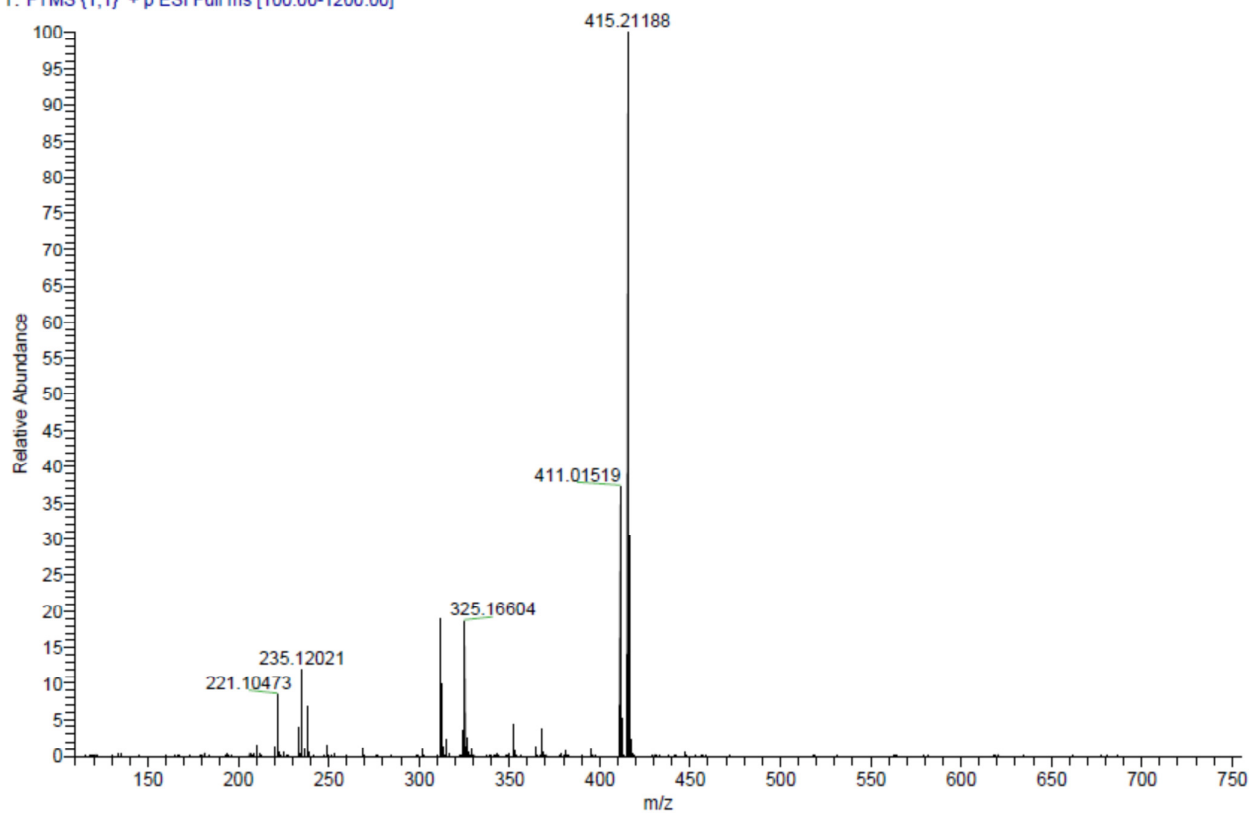


Figure S10. HRMS for the reaction mixture of 6 equivalents of benzylamine and 1 equivalent of chalcone after 1 hour of the reaction time.

FCT-1107-S2 #65-76 RT: 1.09-1.23 AV: 12 NL: 3.56E7
T: FTMS (1,1) + p ESI Full ms [100.00-1200.00]

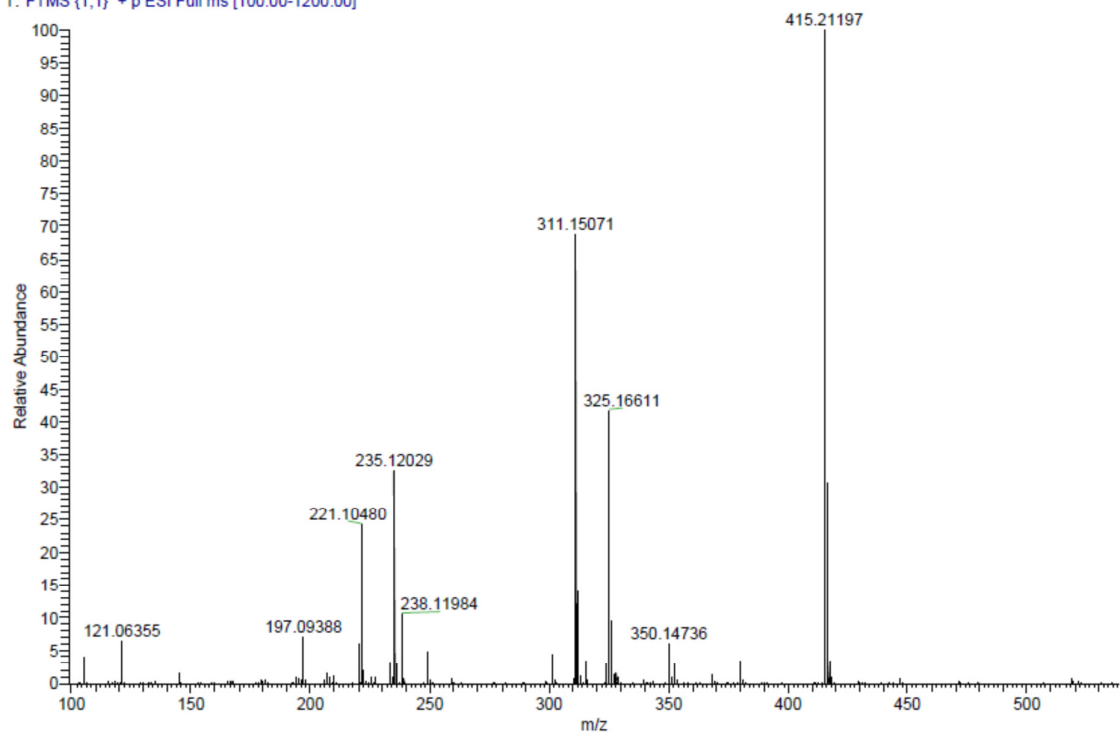


Figure S11. HRMS for the reaction mixture of 6 equivalents of benzylamine and 1 equivalent of chalcone after 3 hours of the reaction time.

FCT-1107-S3 #62-74 RT: 1.04-1.20 AV: 13 NL: 4.44E7
T: FTMS (1,1) + p ESI Full ms [100.00-1200.00]

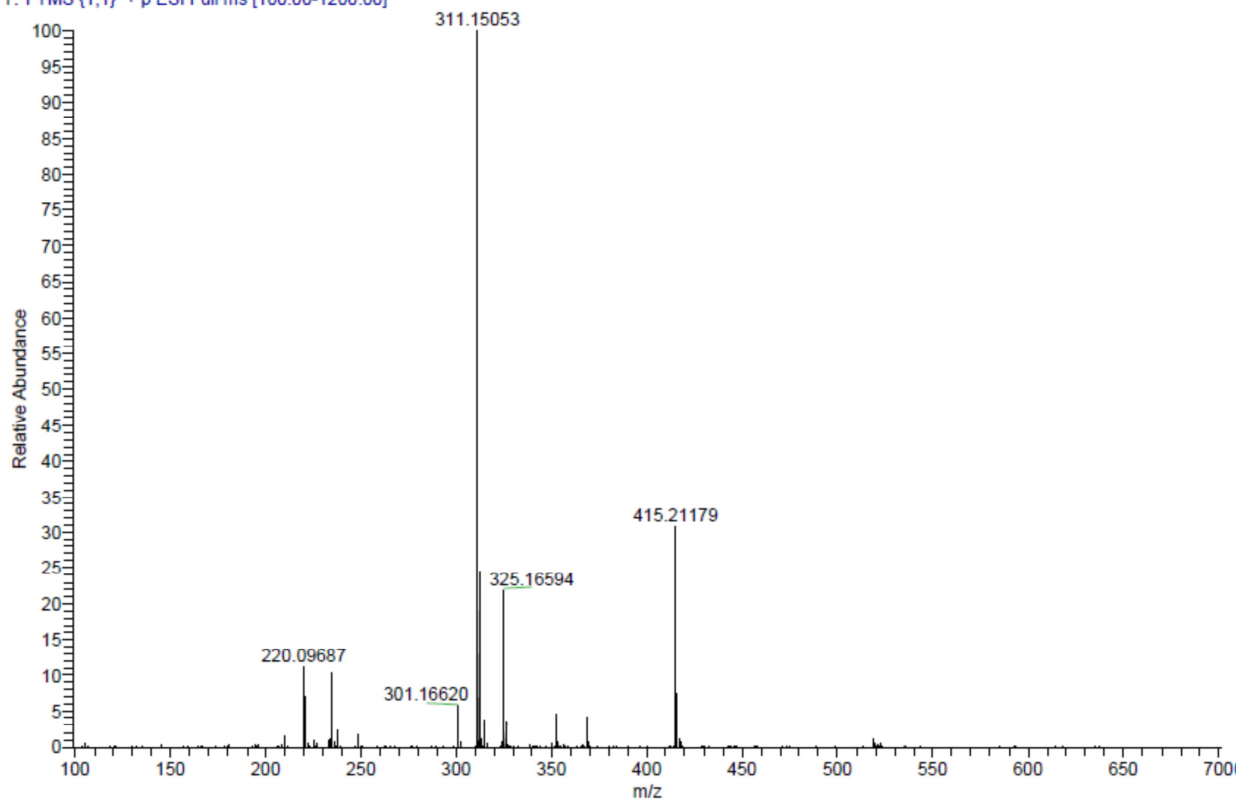


Figure S12. HRMS for the reaction mixture of 6 equivalents of benzylamine and 1 equivalent of chalcone after 6 hours of the reaction time.

Spectral Data:

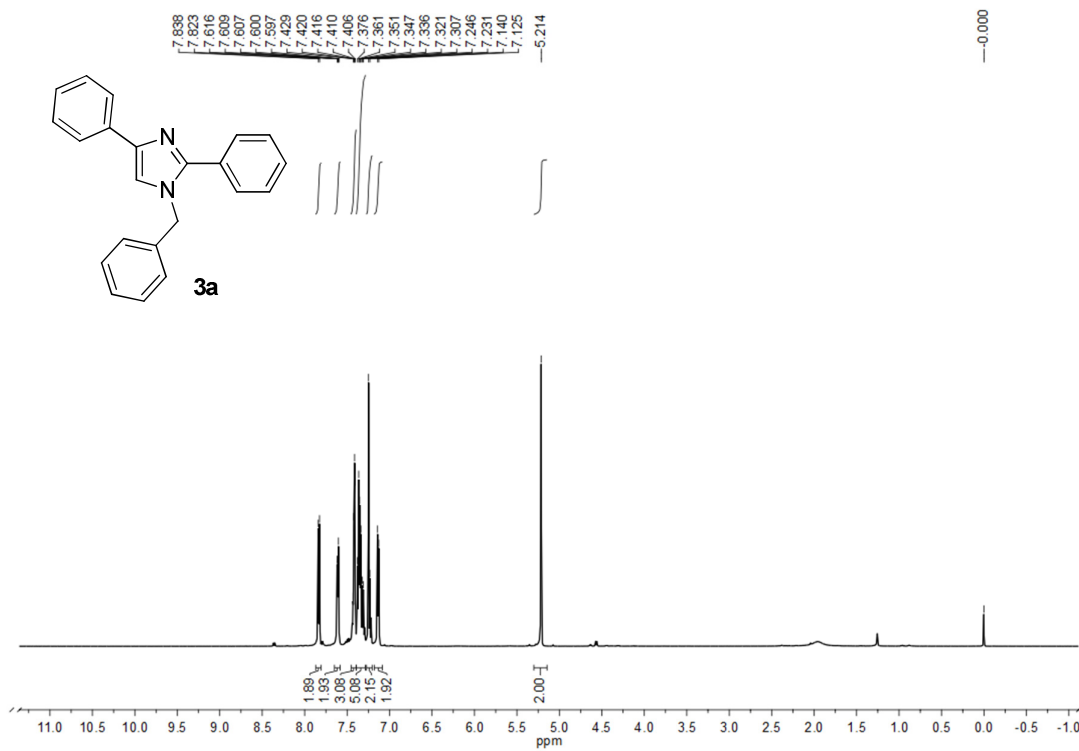


Figure S13. ¹H NMR of 3a

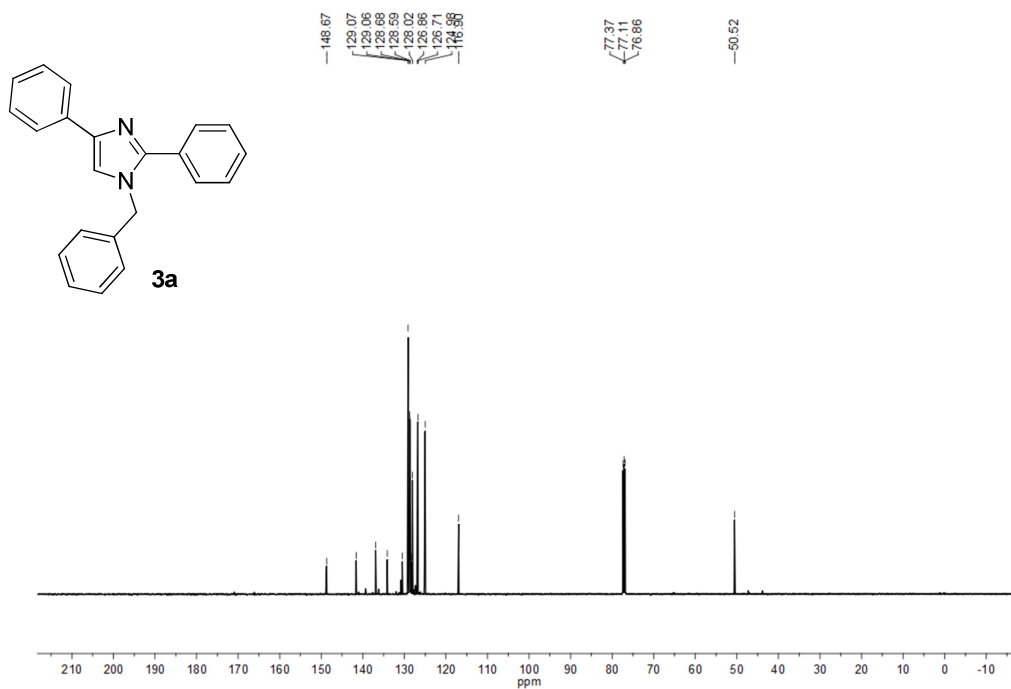


Figure S14. ¹³C NMR of 3a

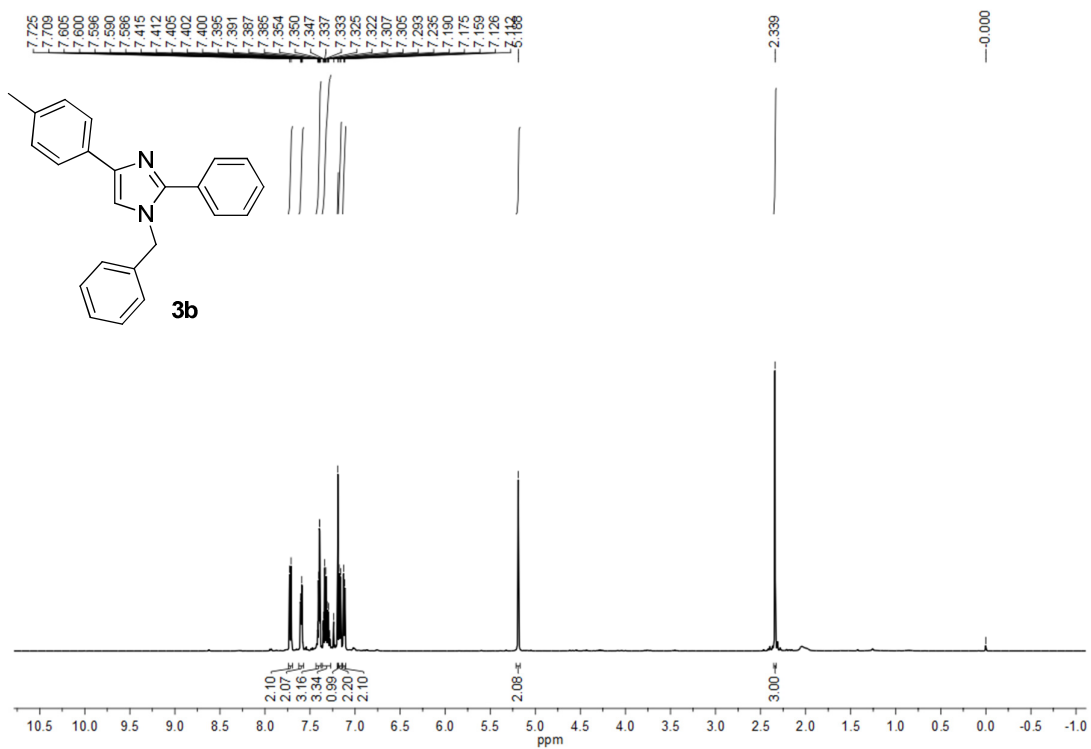


Figure S15. ^1H NMR of 3b

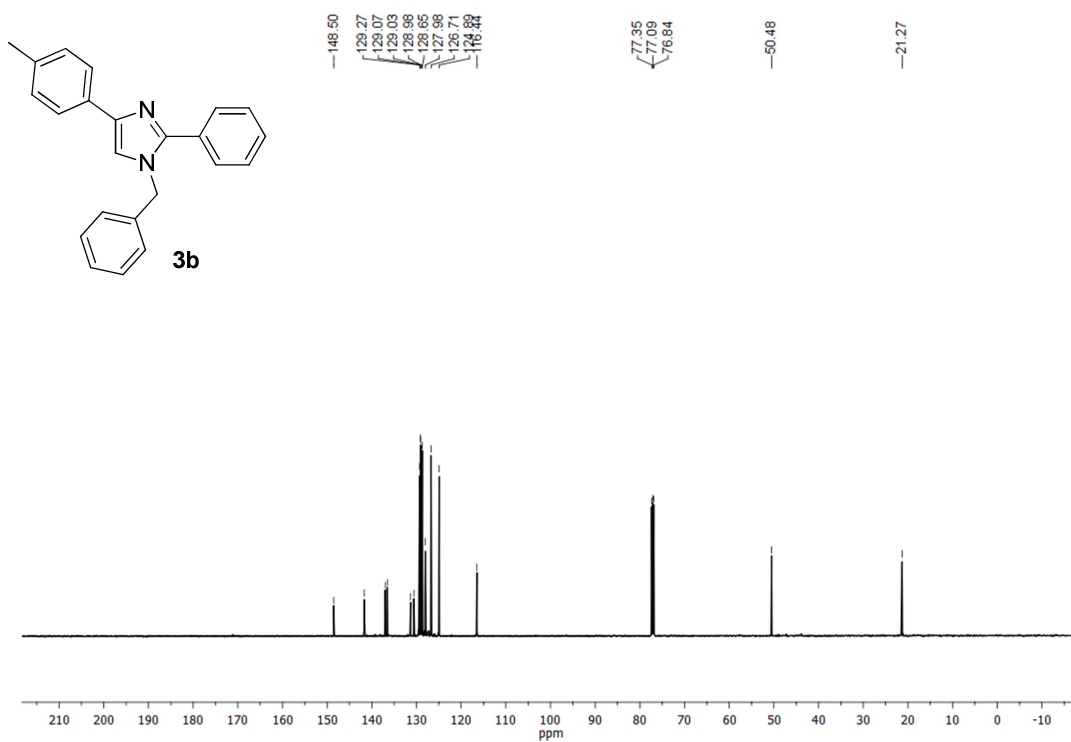


Figure S16. ^{13}C NMR of 3b

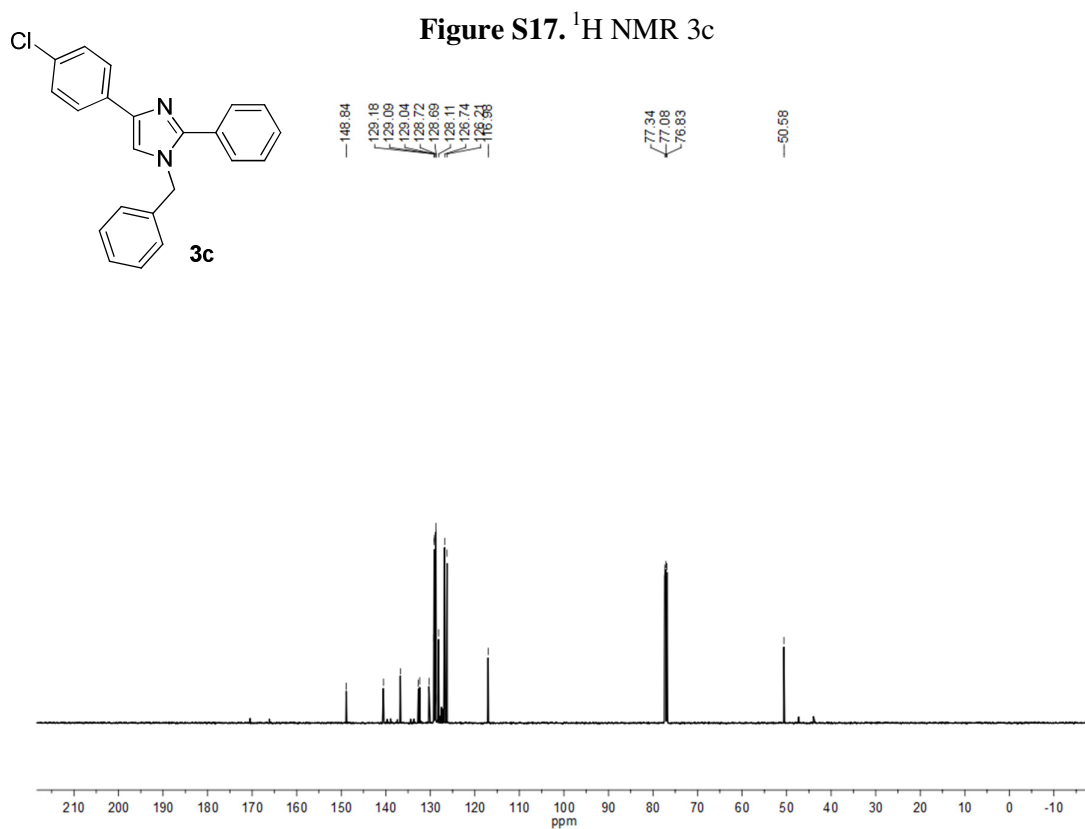
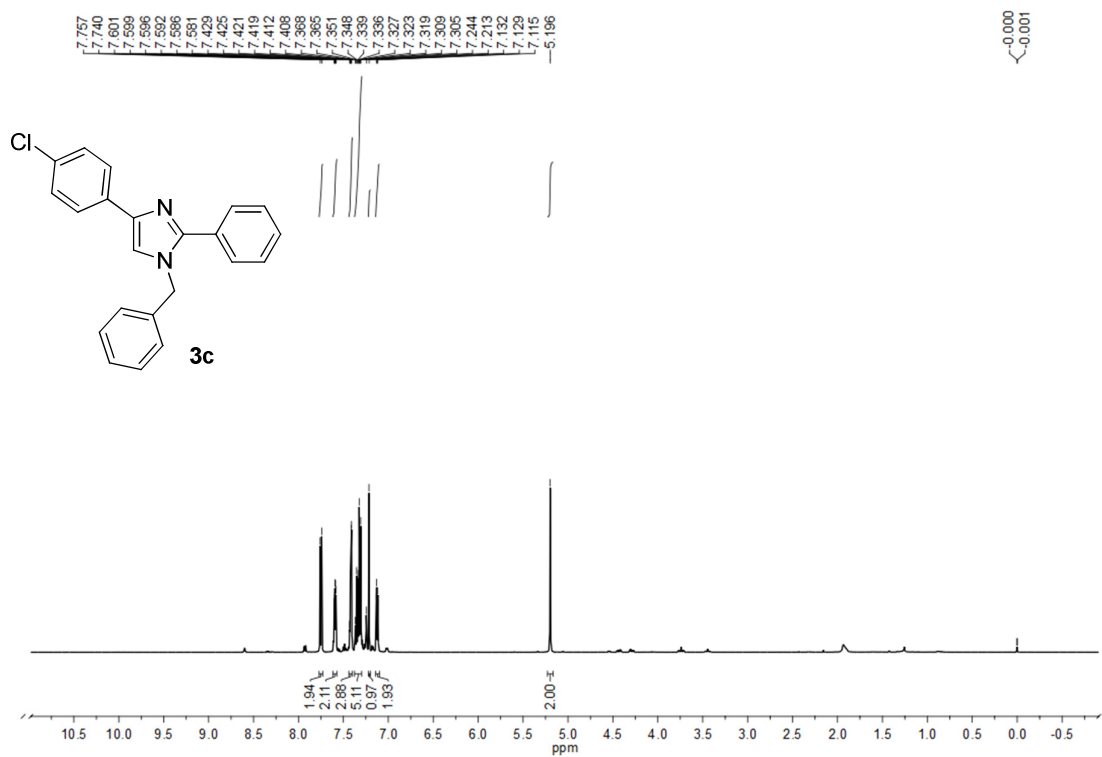


Figure S18. ¹³C NMR of 3c

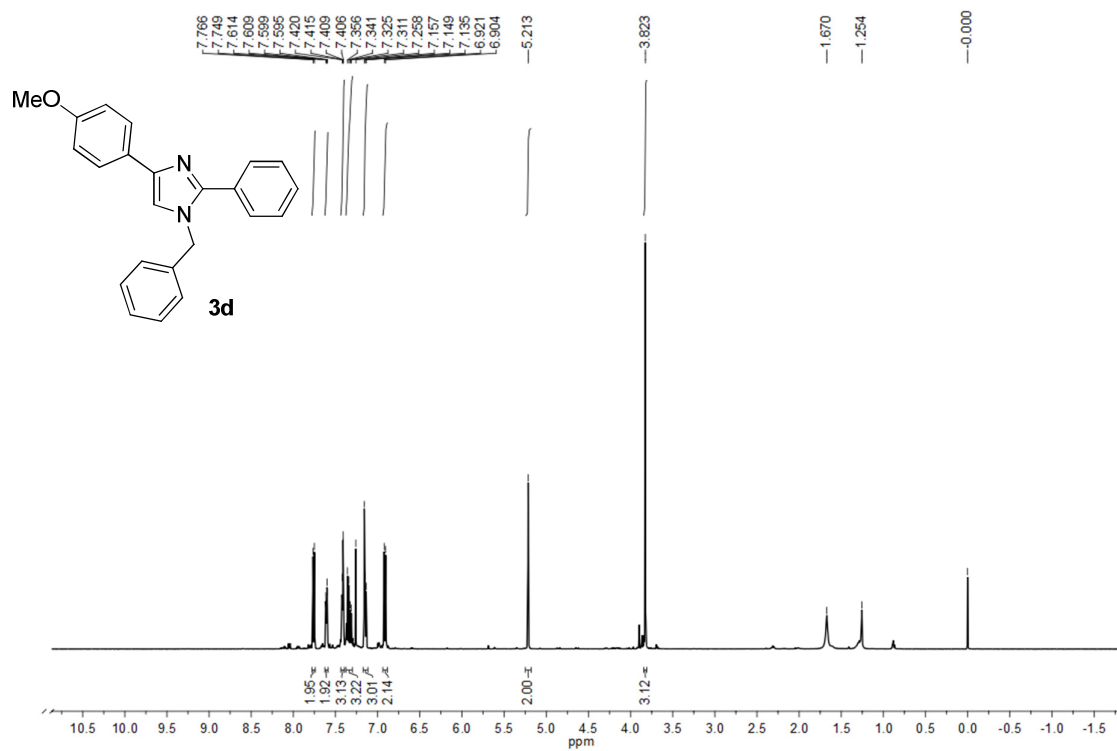


Figure S19. ^1H NMR of 3d

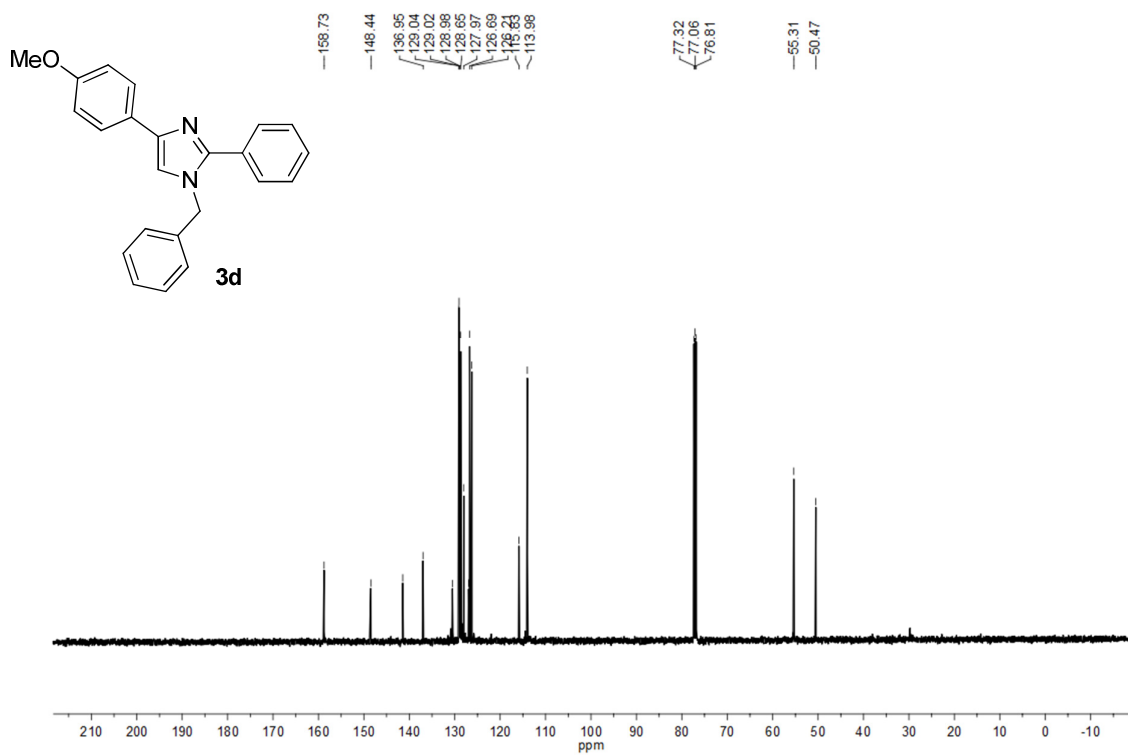


Figure S20. ^{13}C NMR of 3d

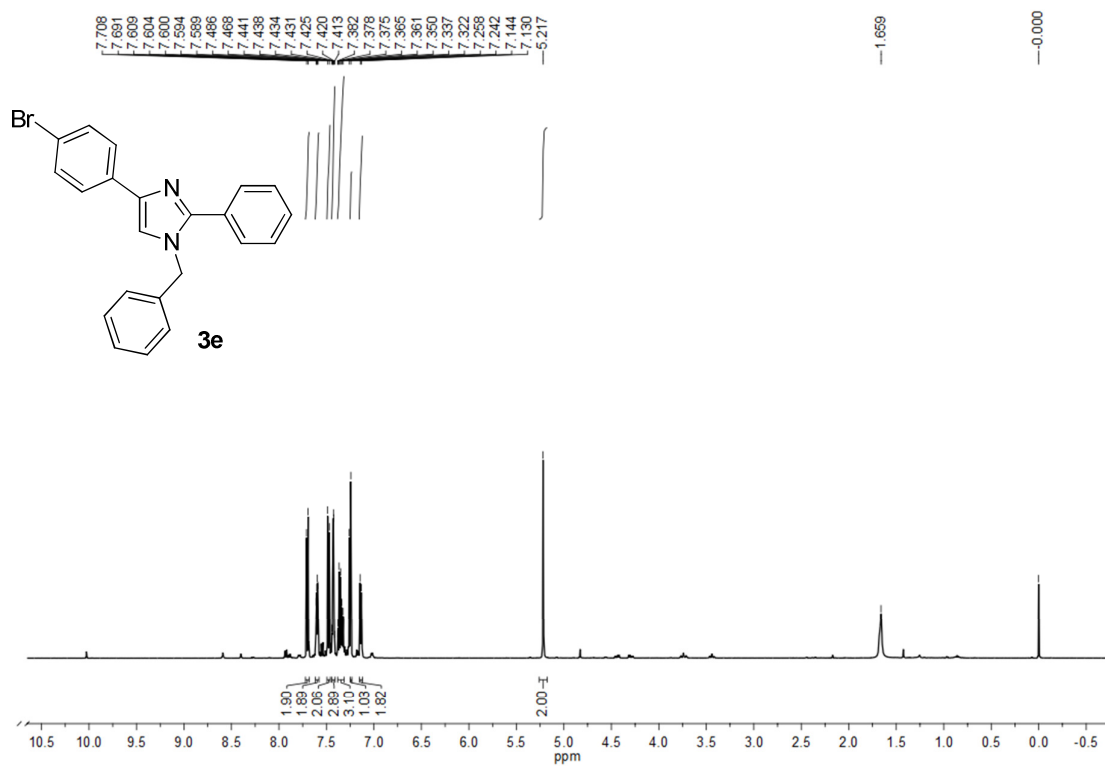


Figure S21. ¹H NMR of **3e**

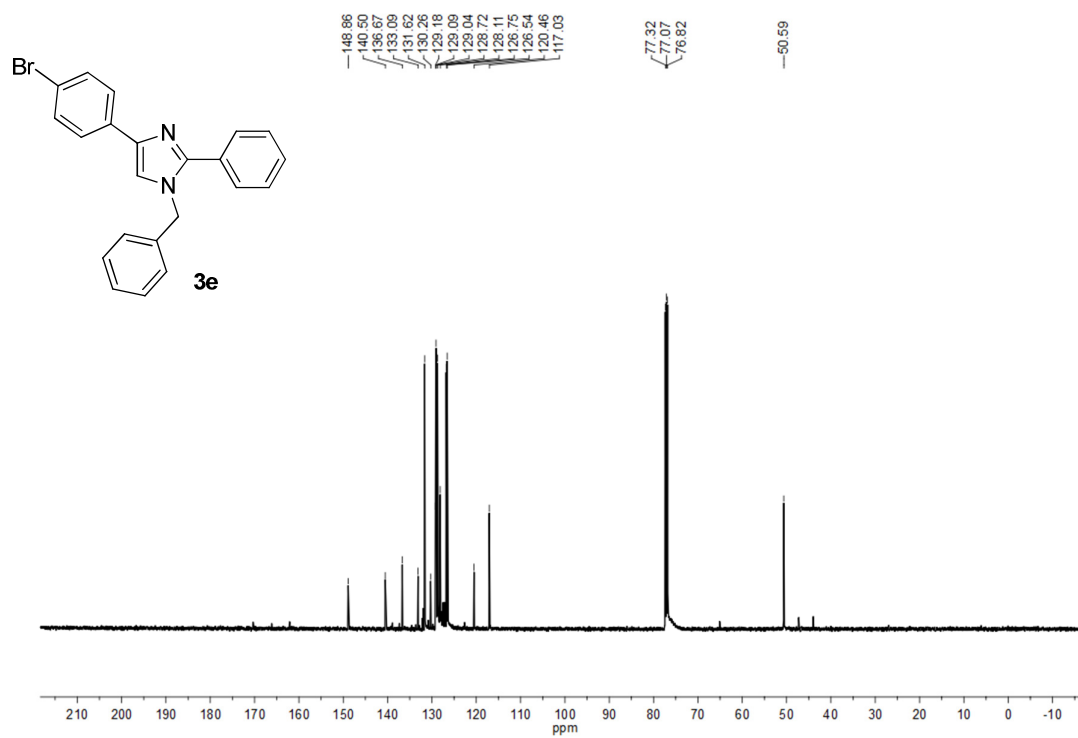


Figure S22. ¹³C NMR of **3e**

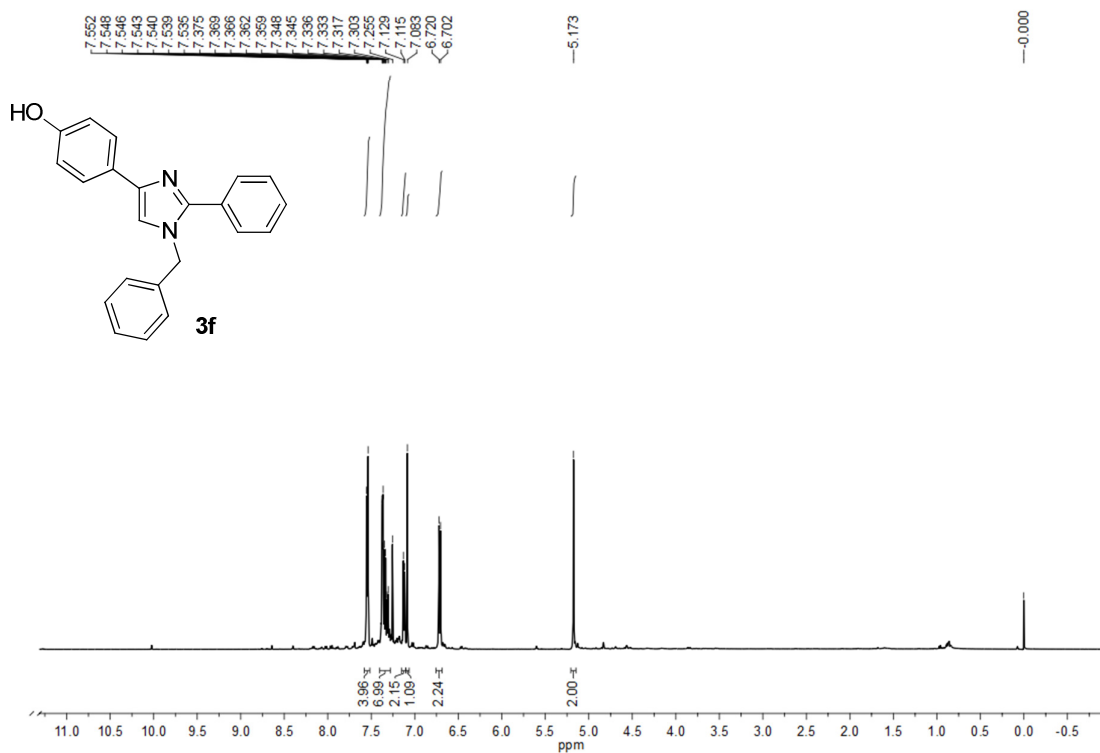


Figure S23. ¹H NMR of 3f

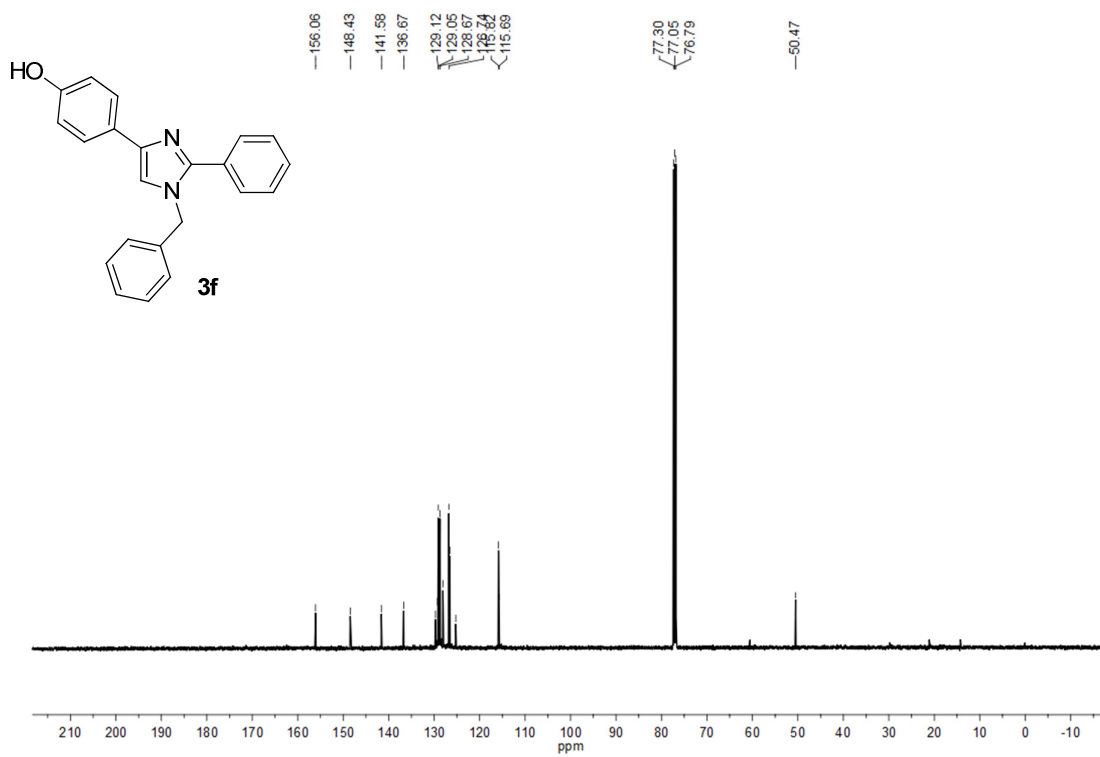


Figure S24. ¹³C NMR of 3f

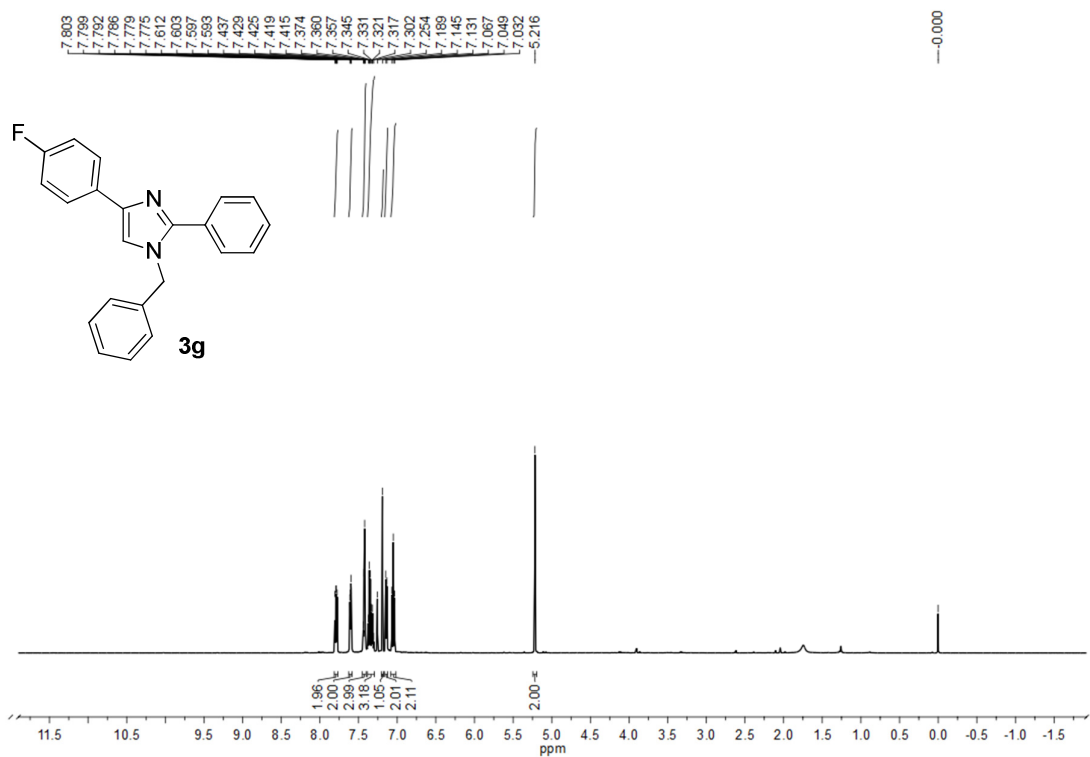


Figure S25. ¹H NMR of 3g

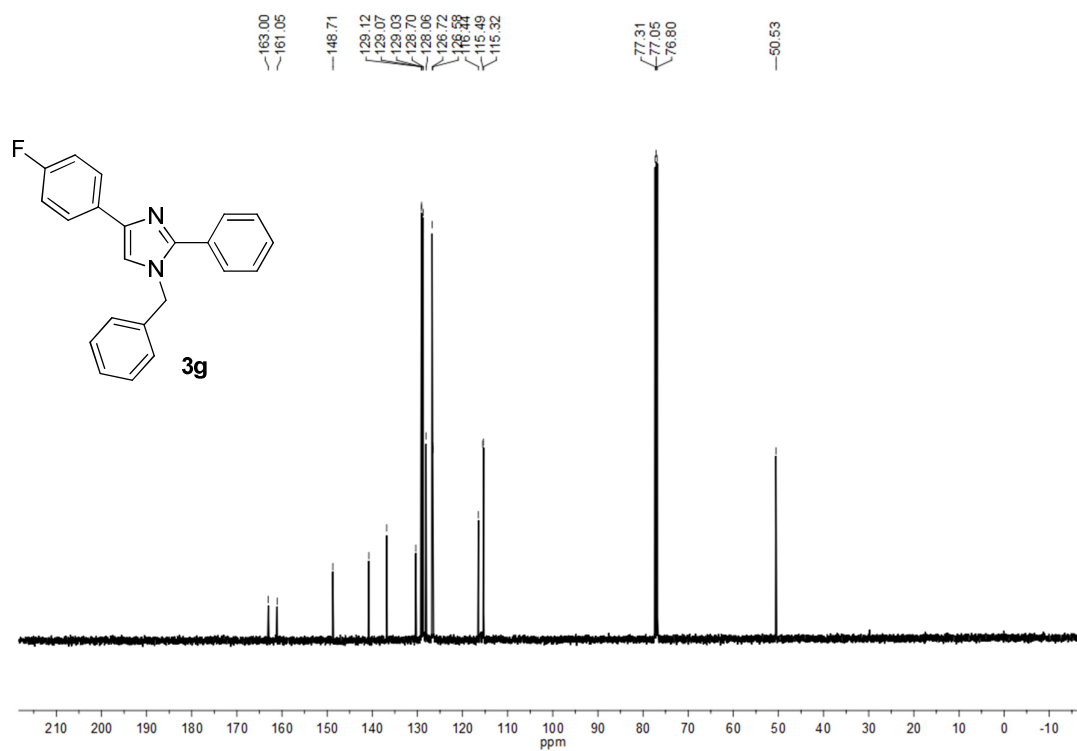


Figure S26. ¹³C NMR of 3g

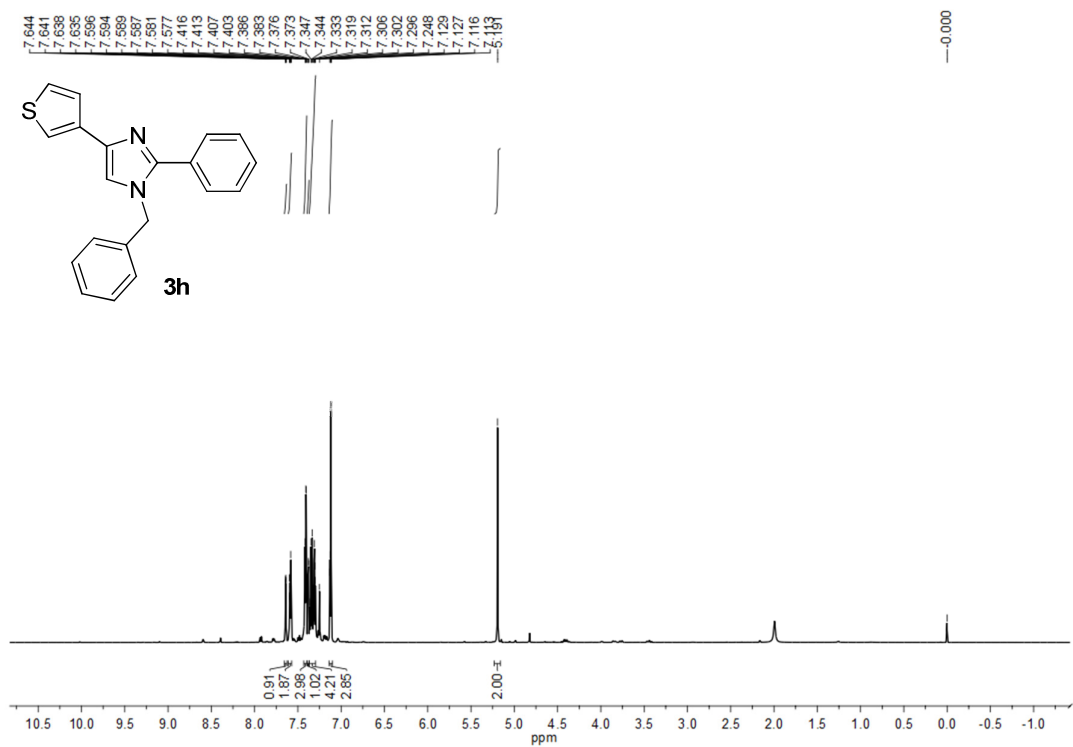


Figure S27. ¹H NMR of 3h

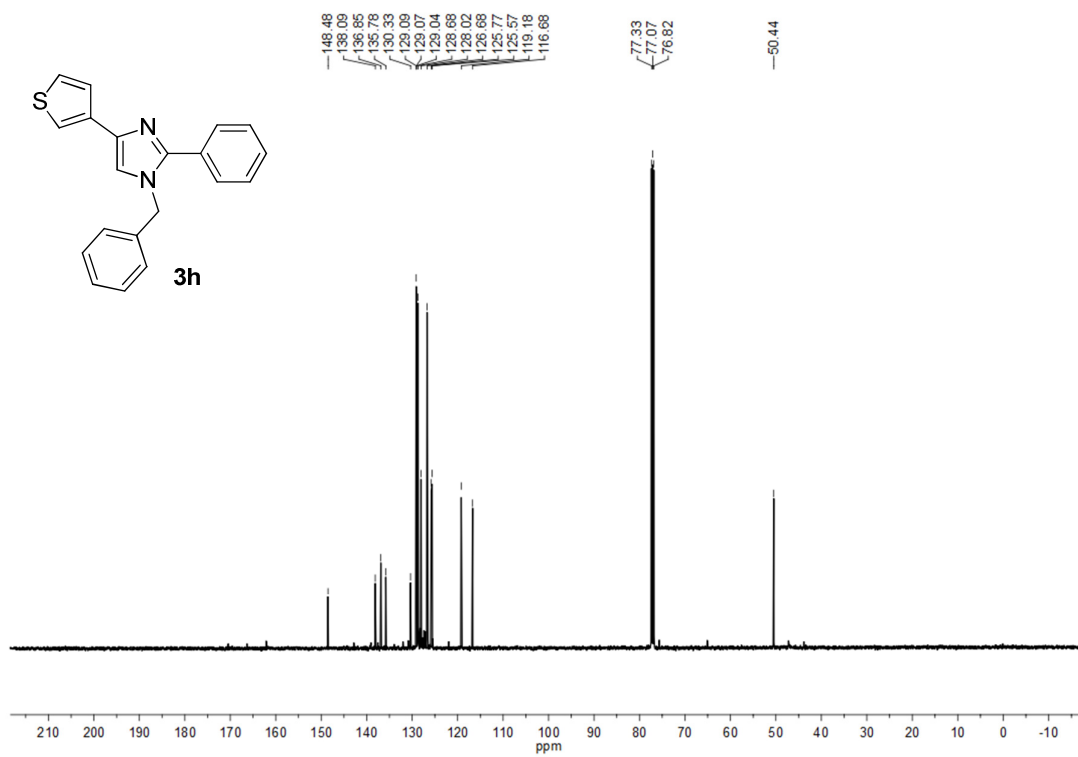


Figure S28. ¹³C NMR of 3h

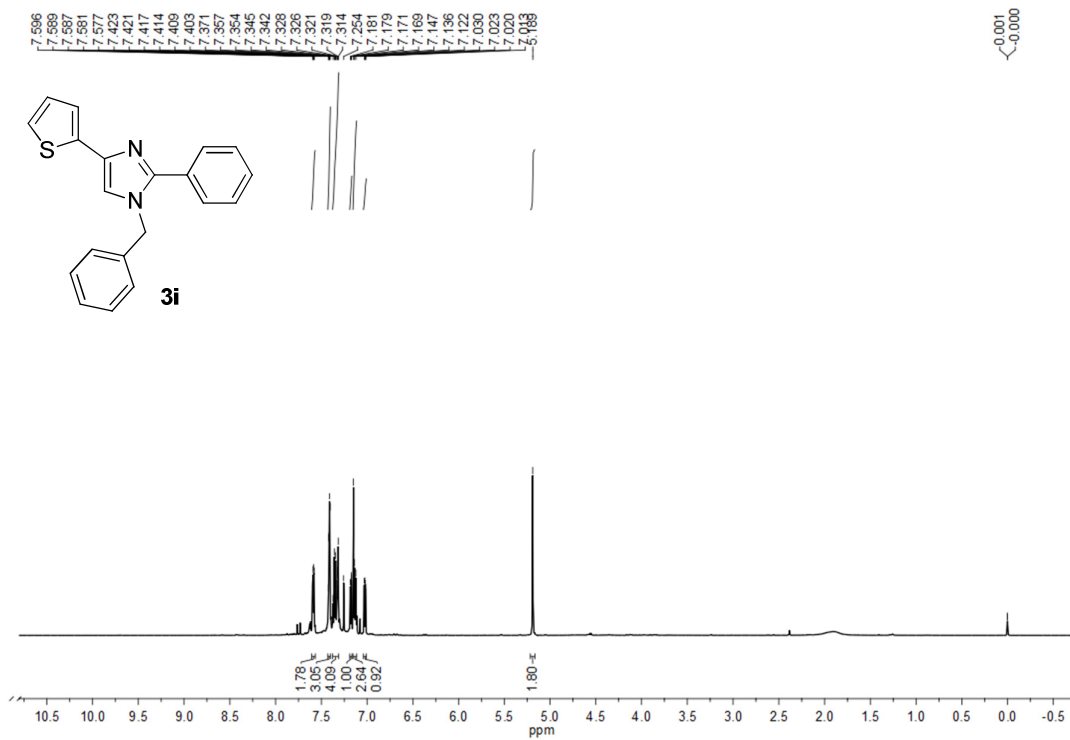


Figure S29. ¹H NMR of 3i

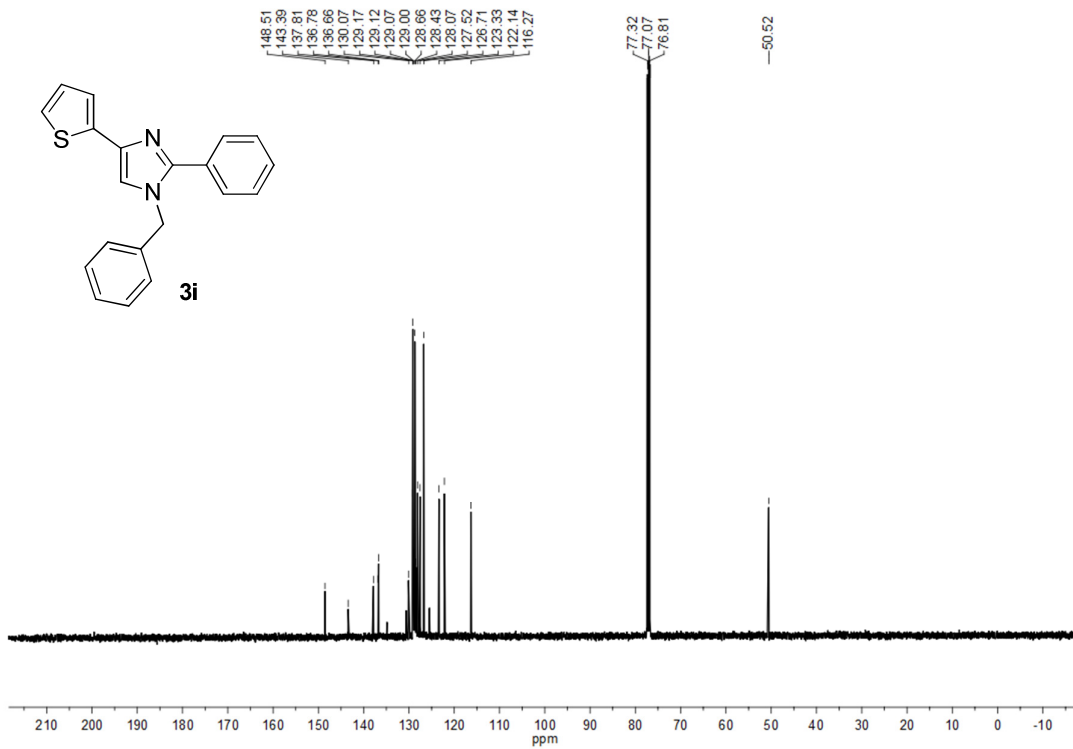


Figure S30. ¹³C NMR of 3i

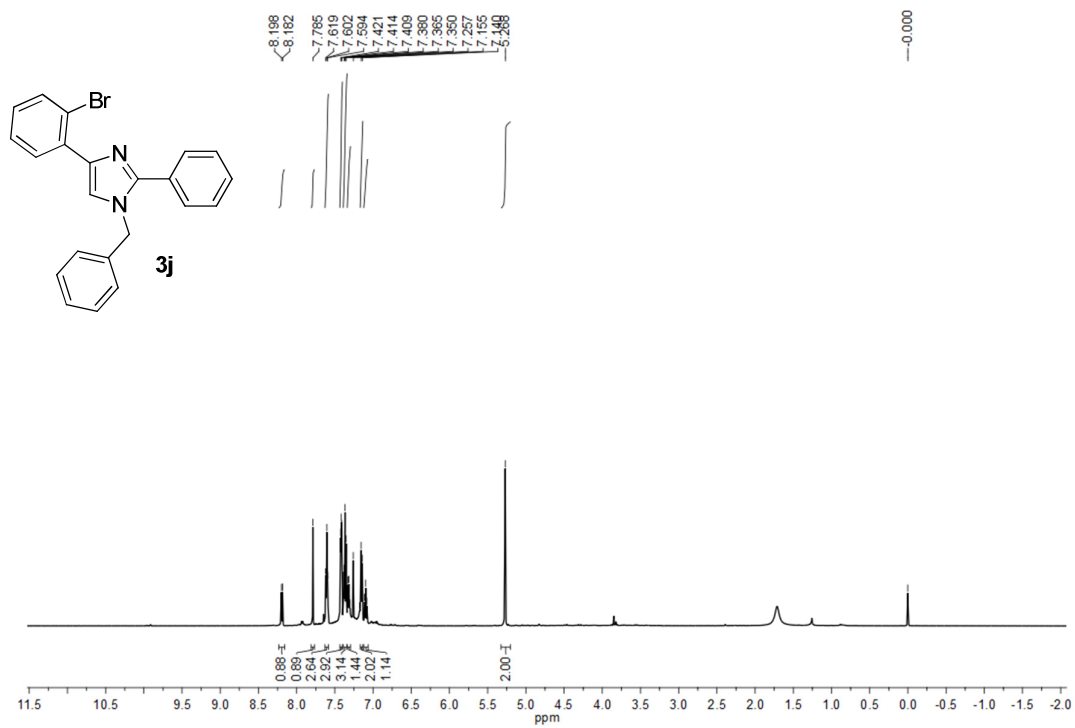


Figure S31. ¹H NMR of 3j

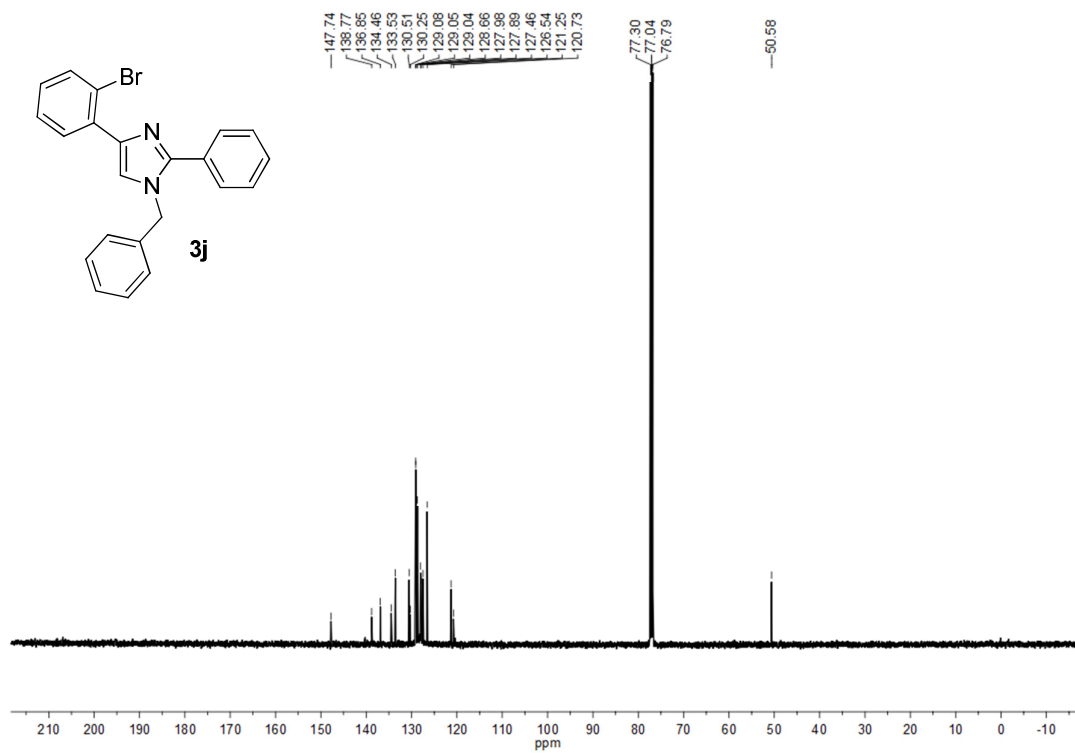


Figure S32. ¹³C NMR of 3j

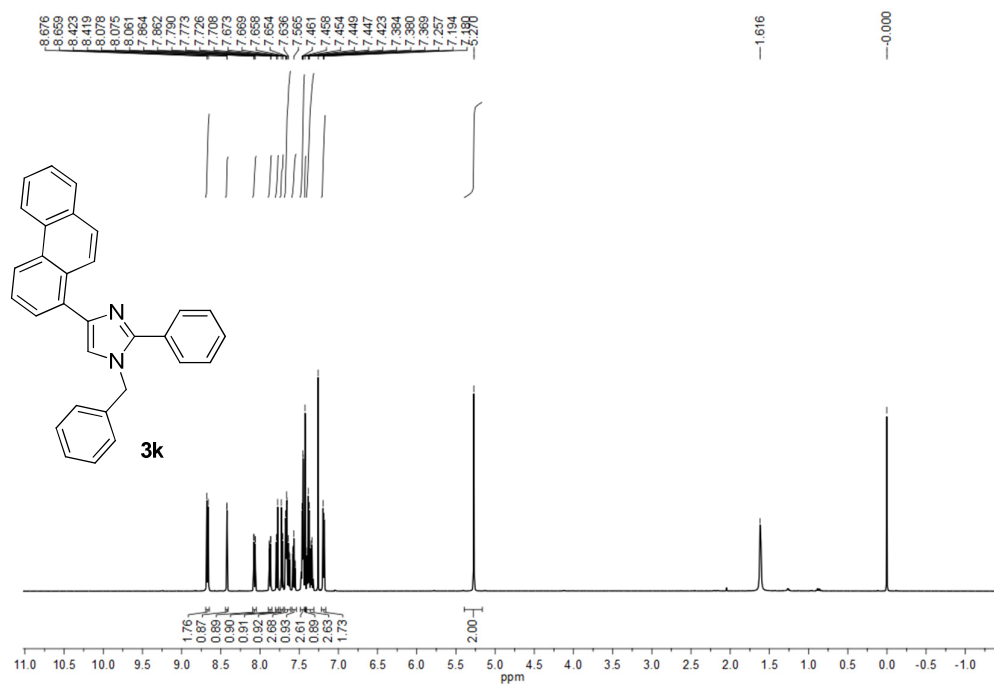


Figure S33. ¹H NMR of 3k

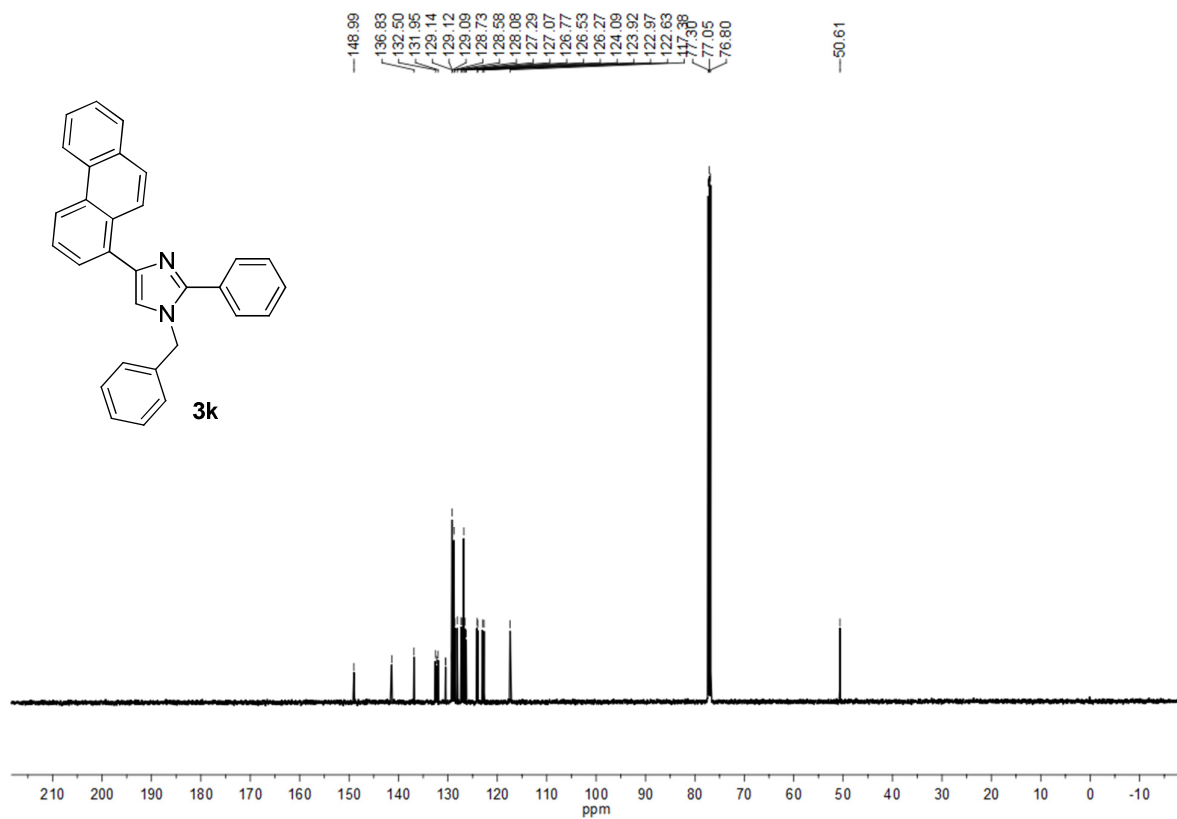


Figure S34. ¹³C NMR of 3k

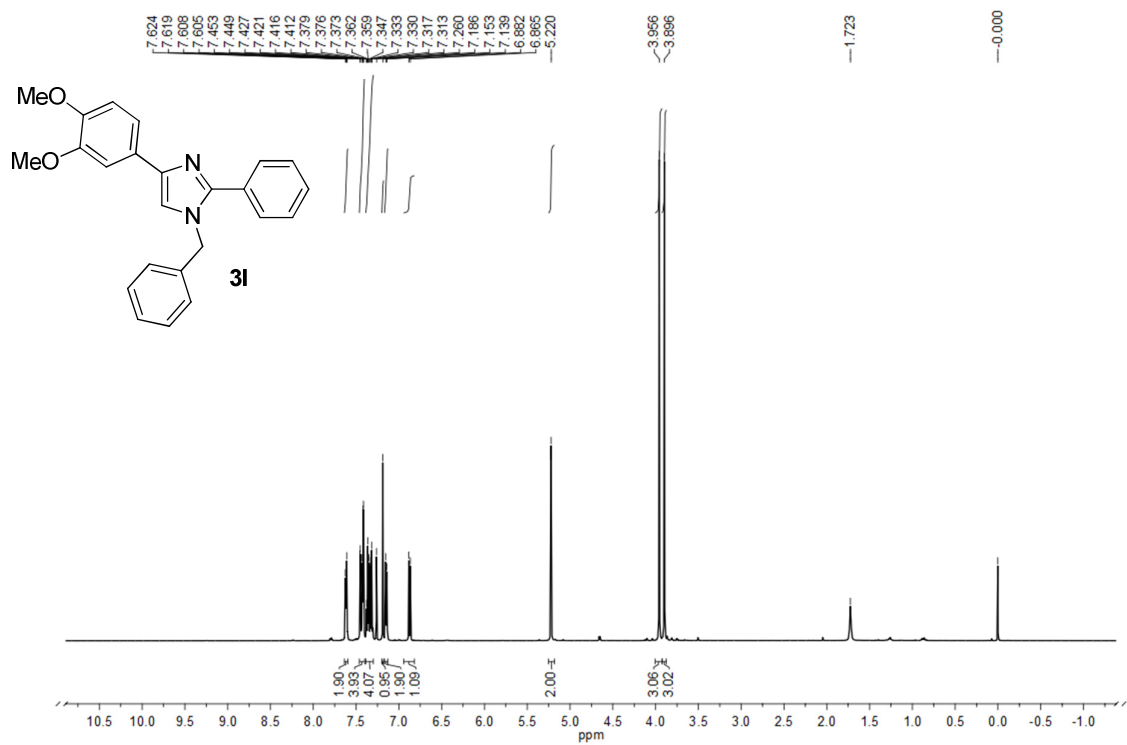


Figure S35. ¹H NMR of 3I

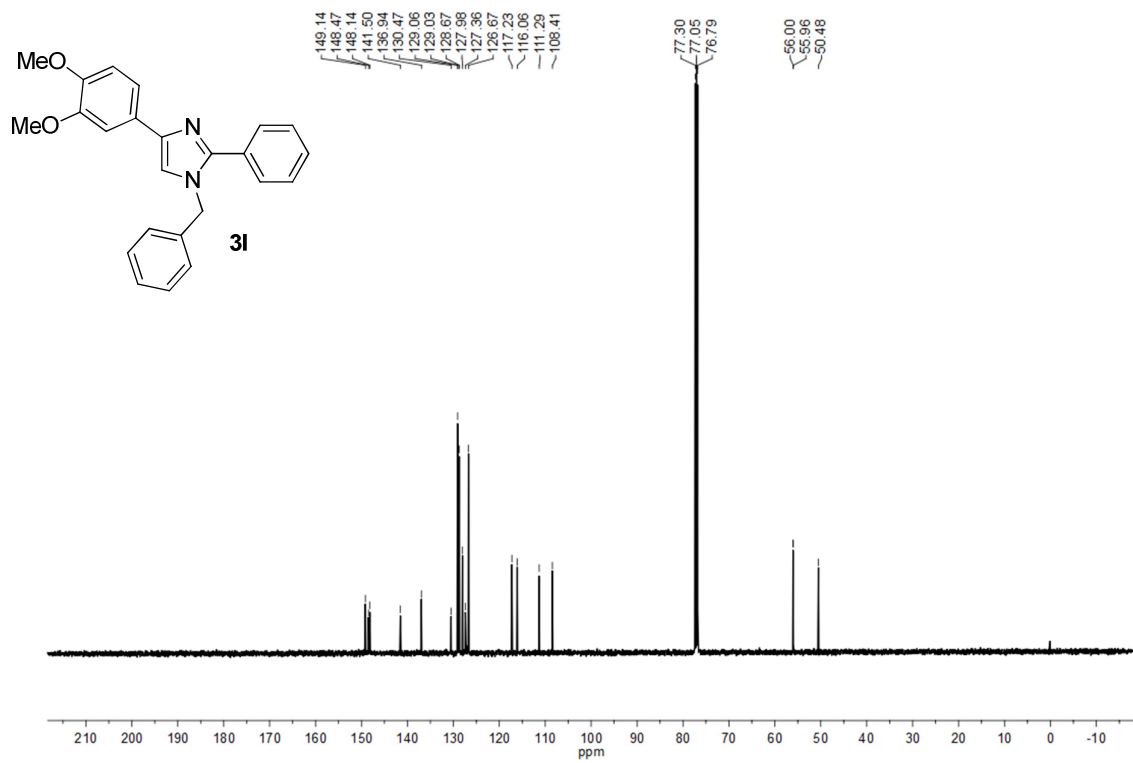


Figure S36. ¹³C NMR of 3I

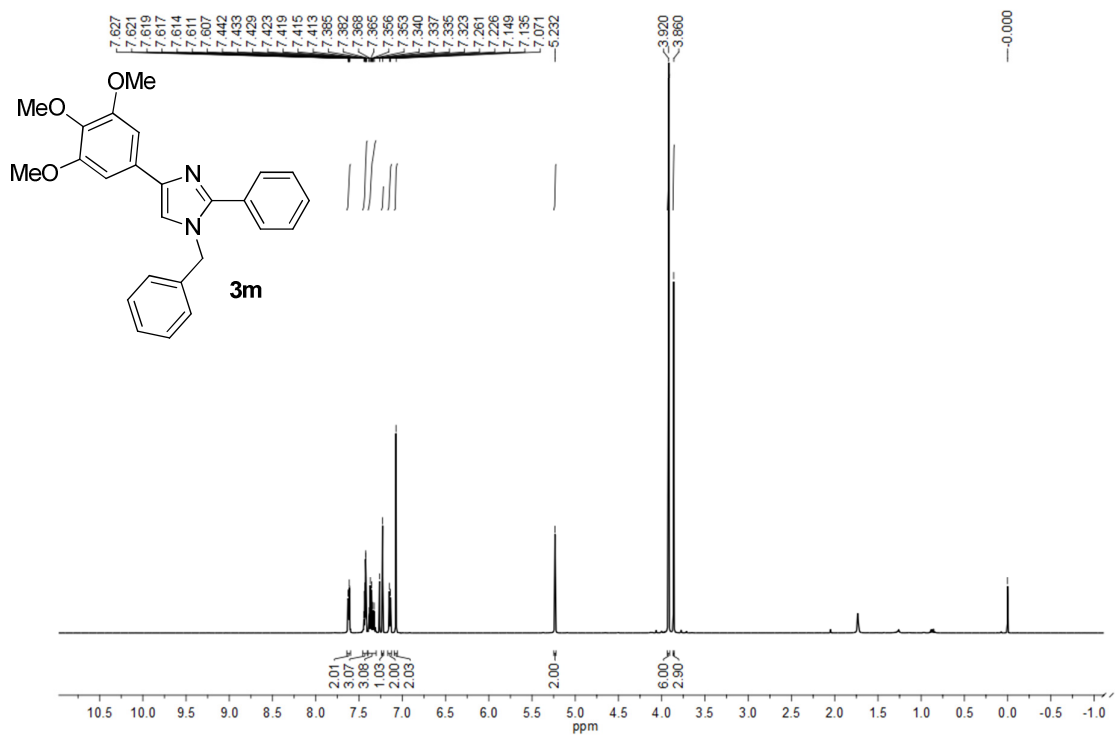


Figure S37. ¹H NMR of 3m

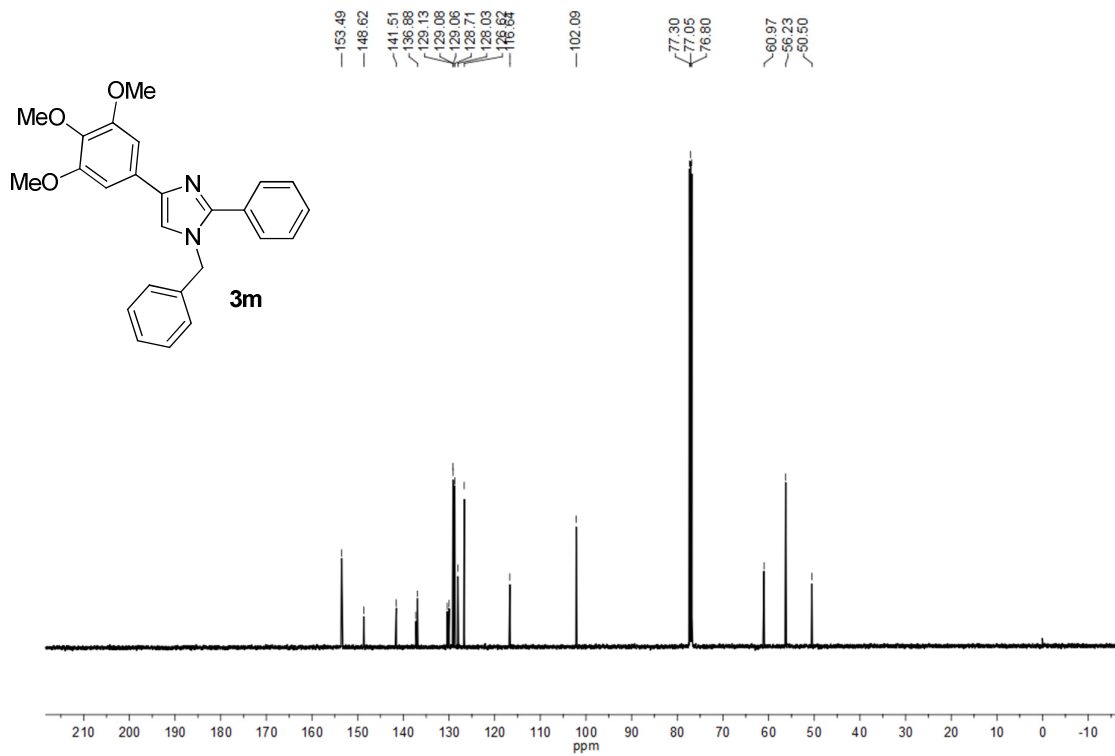


Figure S38. ¹³C NMR of 3m

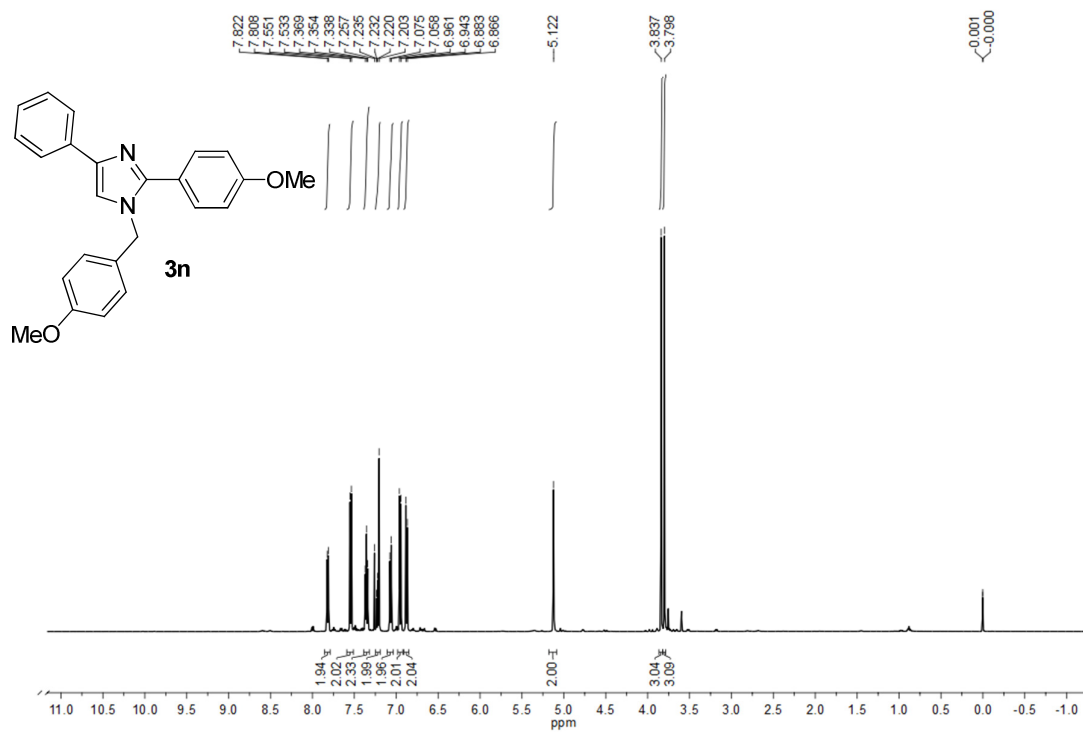


Figure S39. ^1H NMR of 3n

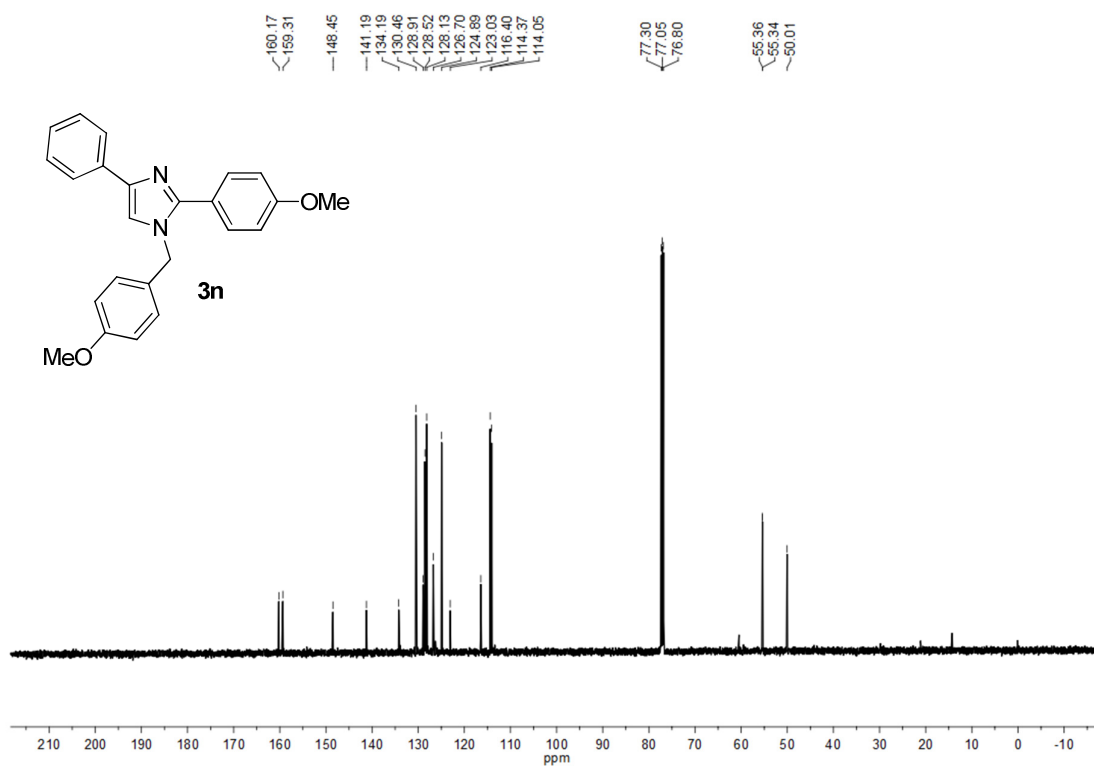


Figure S40. ^{13}C NMR of 3n

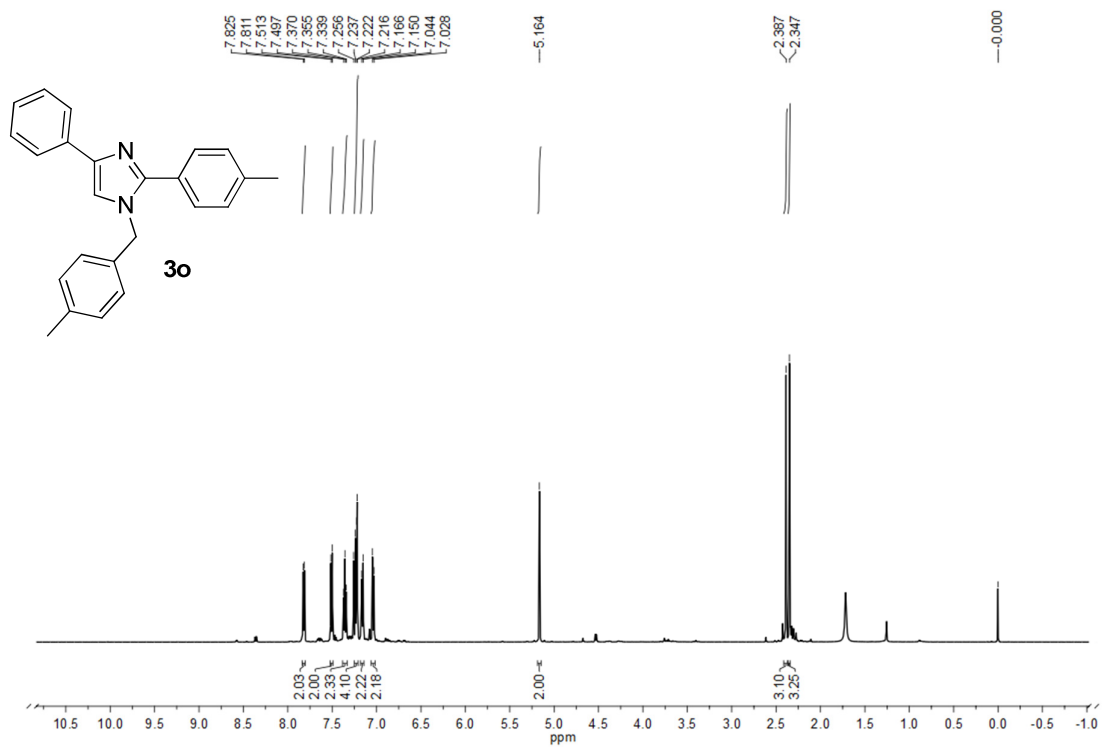


Figure S41. ¹H NMR of 3o

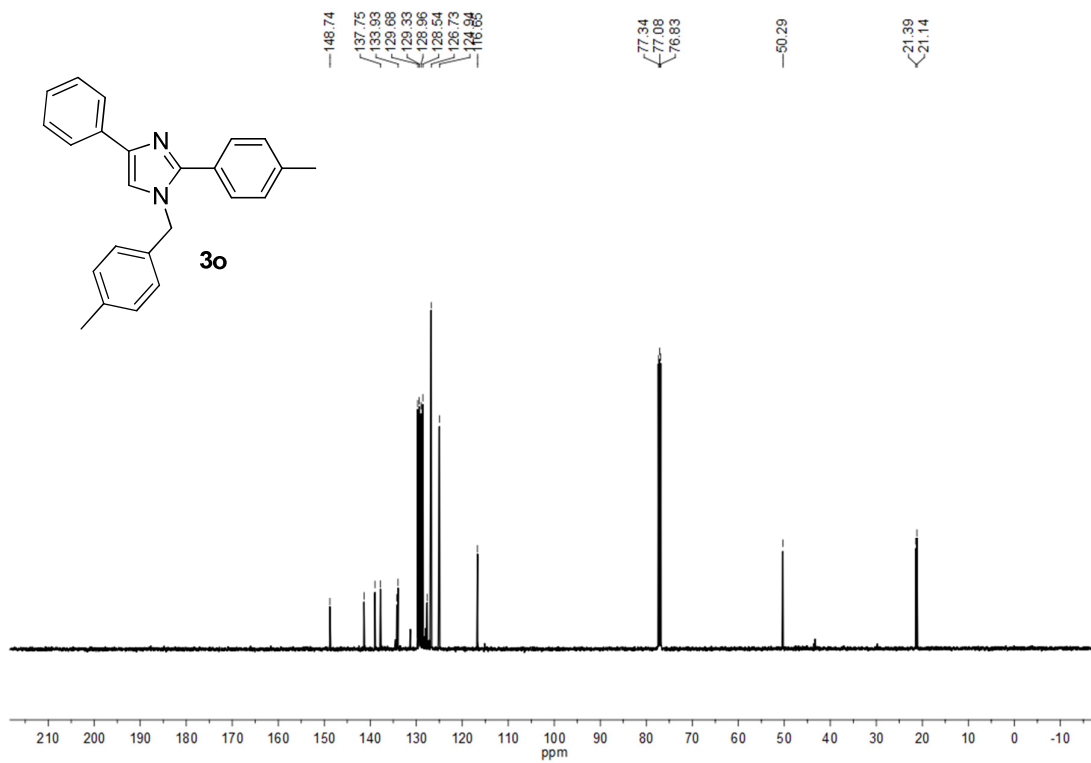


Figure S42. ¹³C NMR of 3o

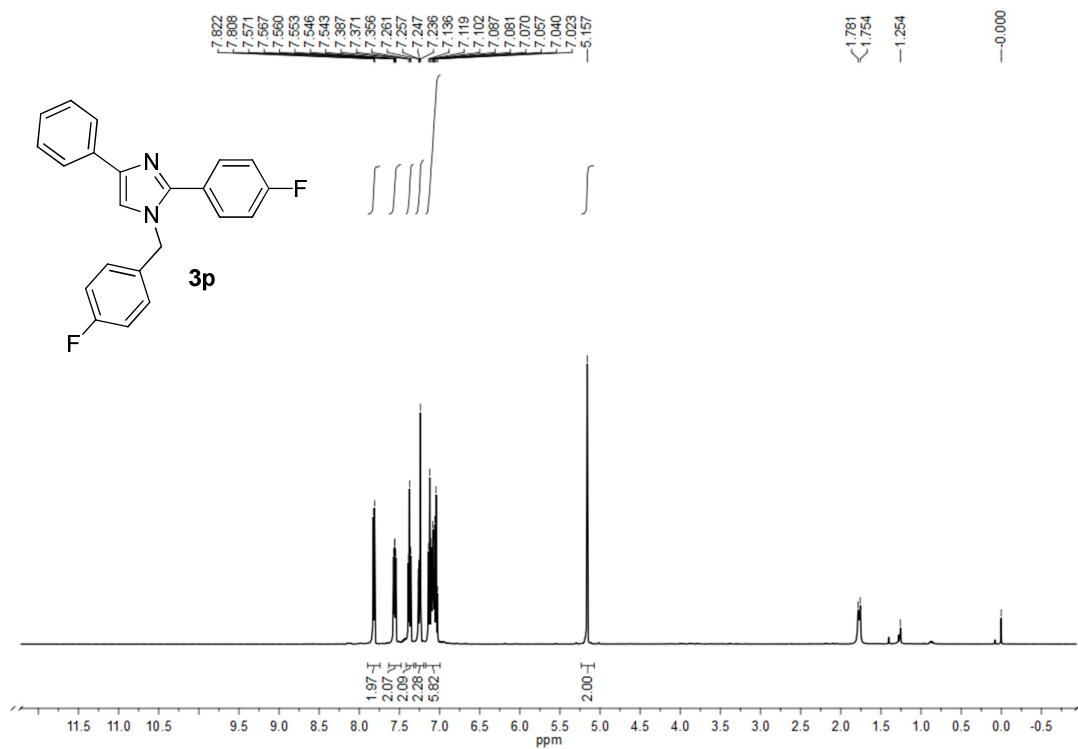


Figure S43. ¹H NMR of 3p

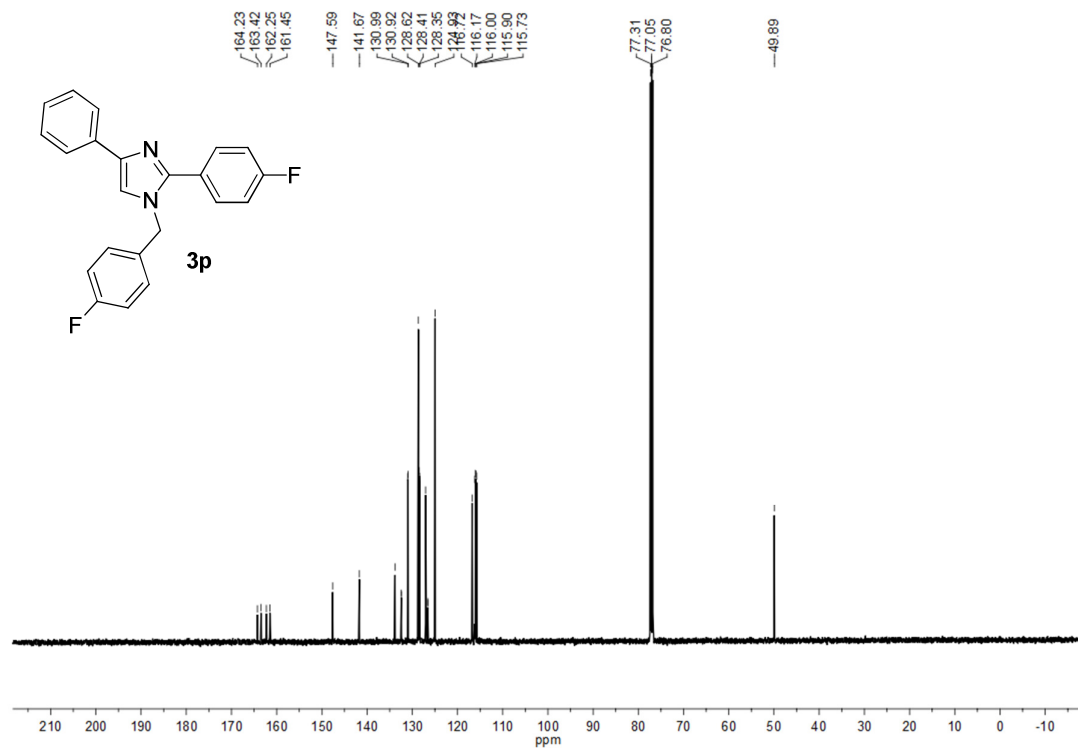


Figure S44. ¹³C NMR of 3p

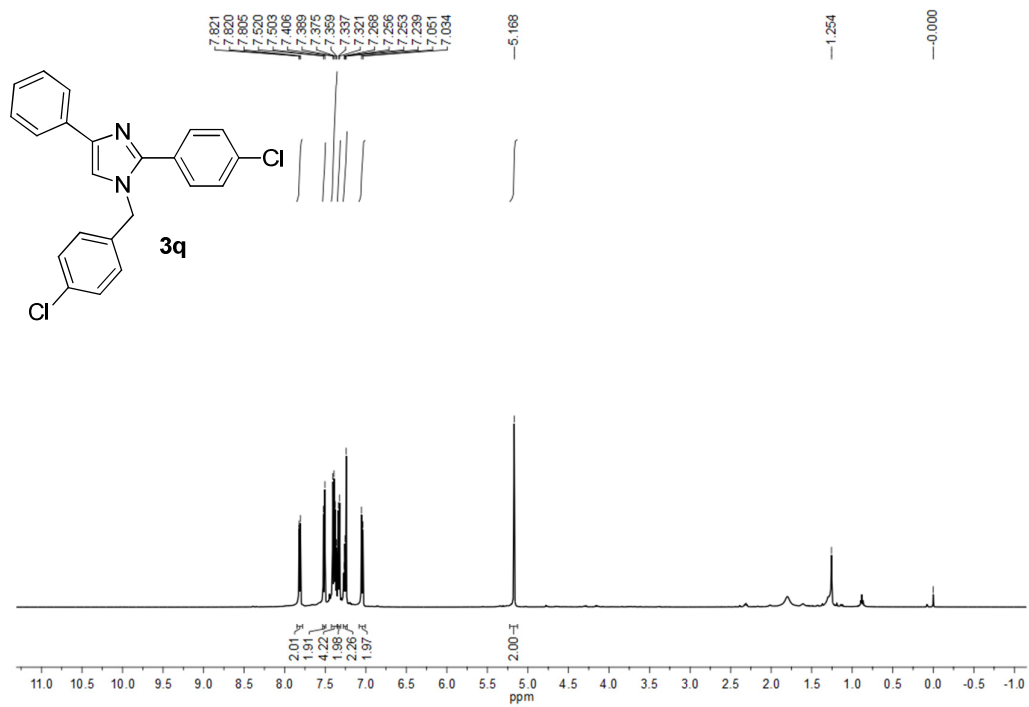


Figure S45. ¹H NMR of 3q

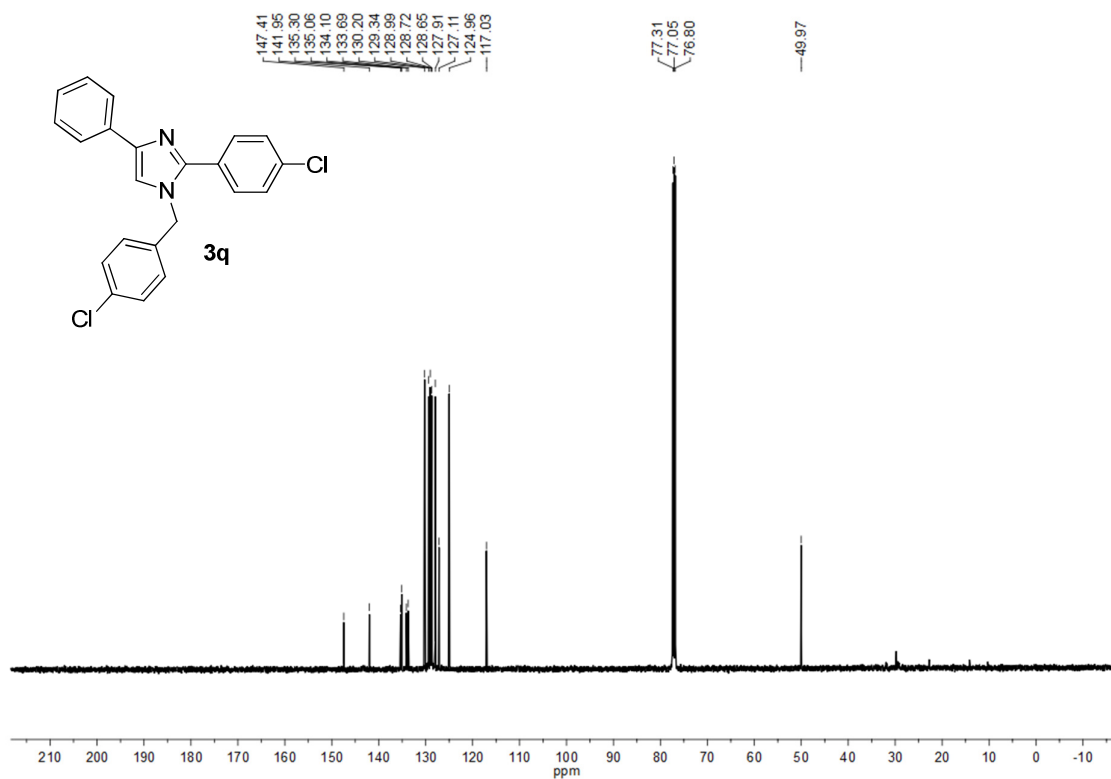


Figure S46. ¹³C NMR of 3q

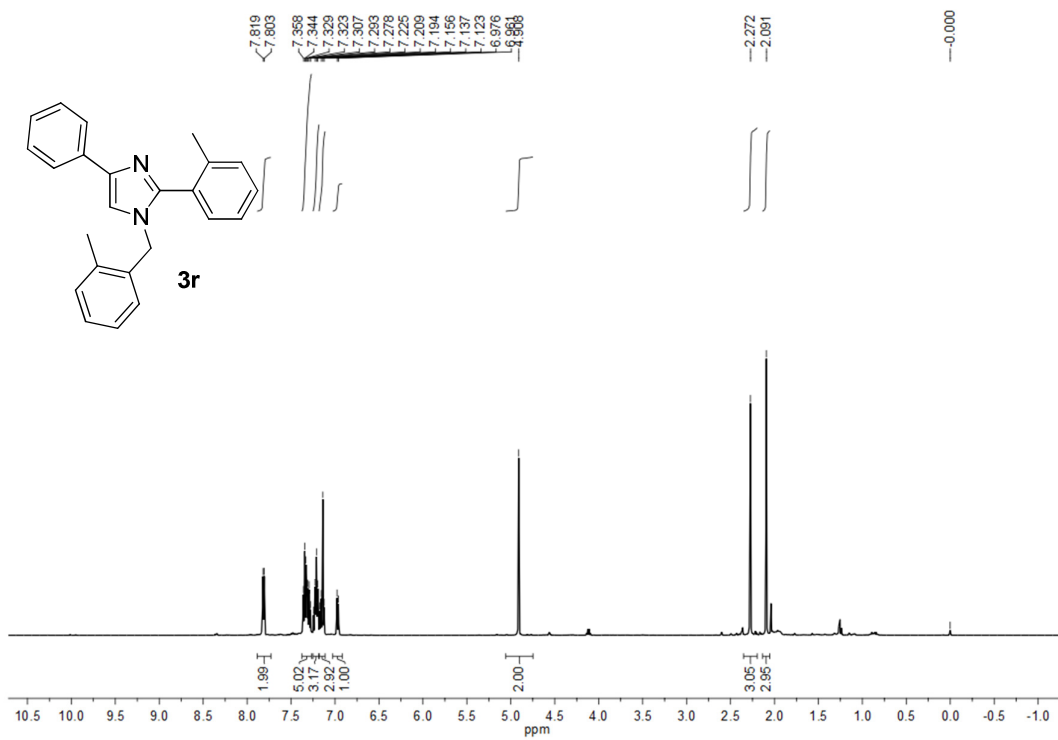


Figure S47. ^1H NMR of 3r

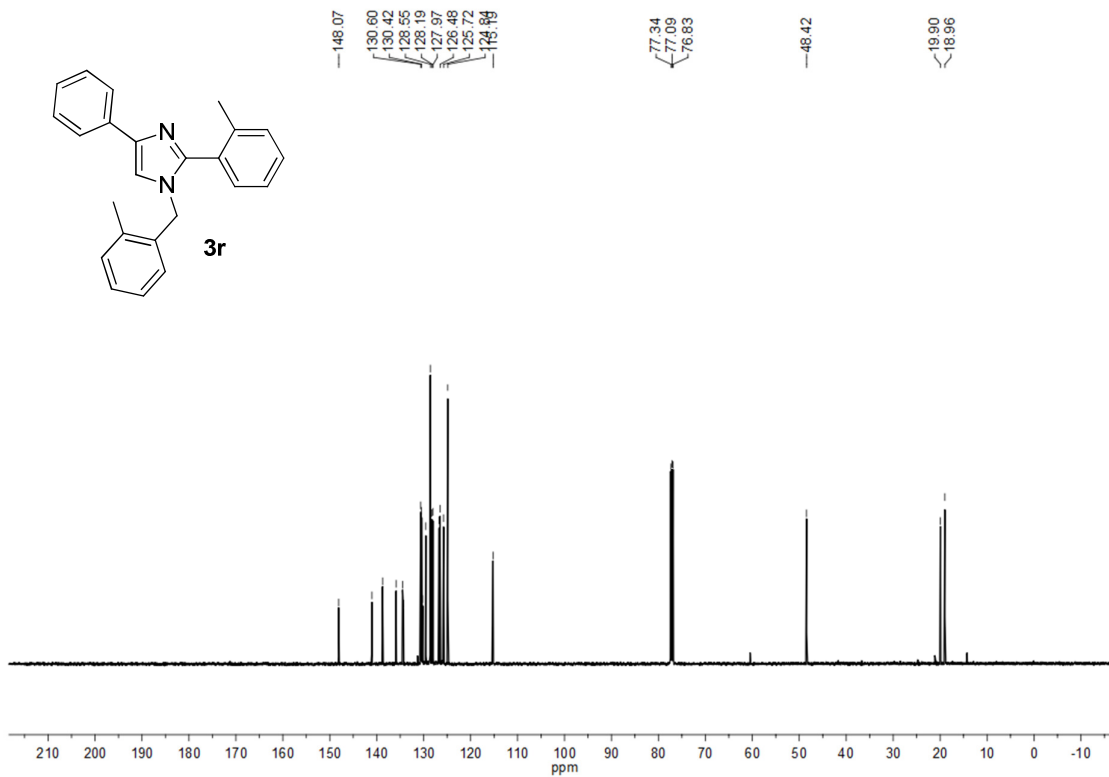


Figure S48. ^{13}C NMR of 3r

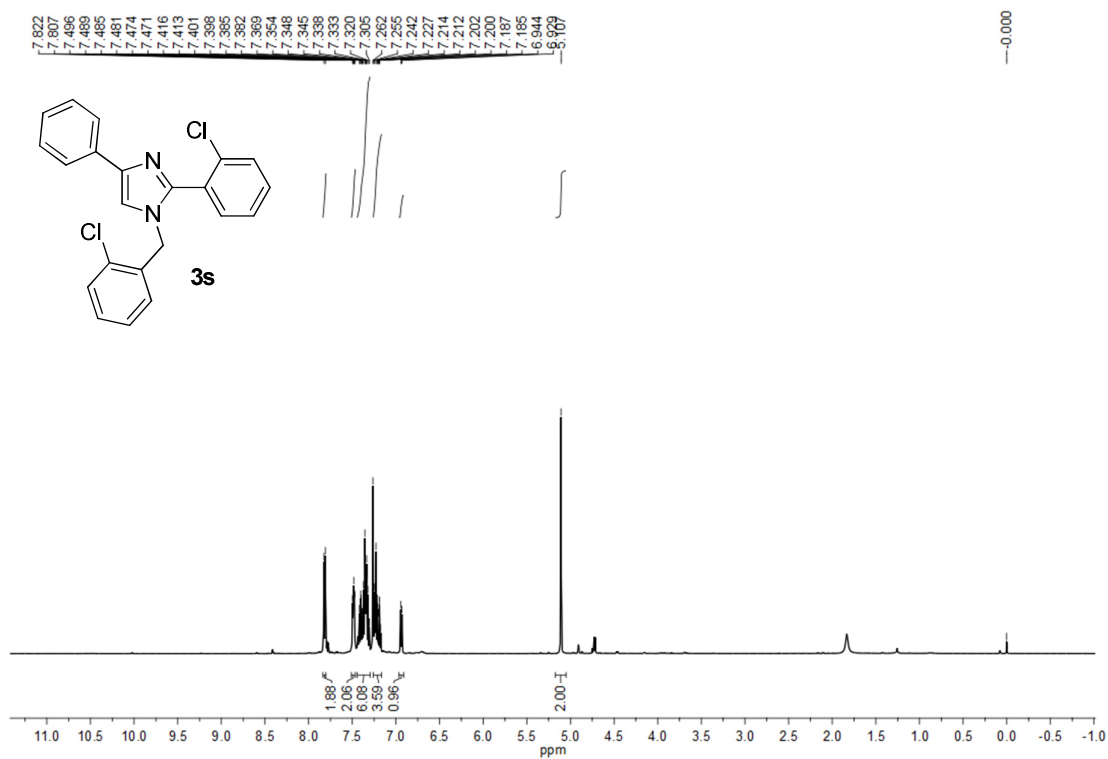


Figure S49. ¹H NMR of 3s

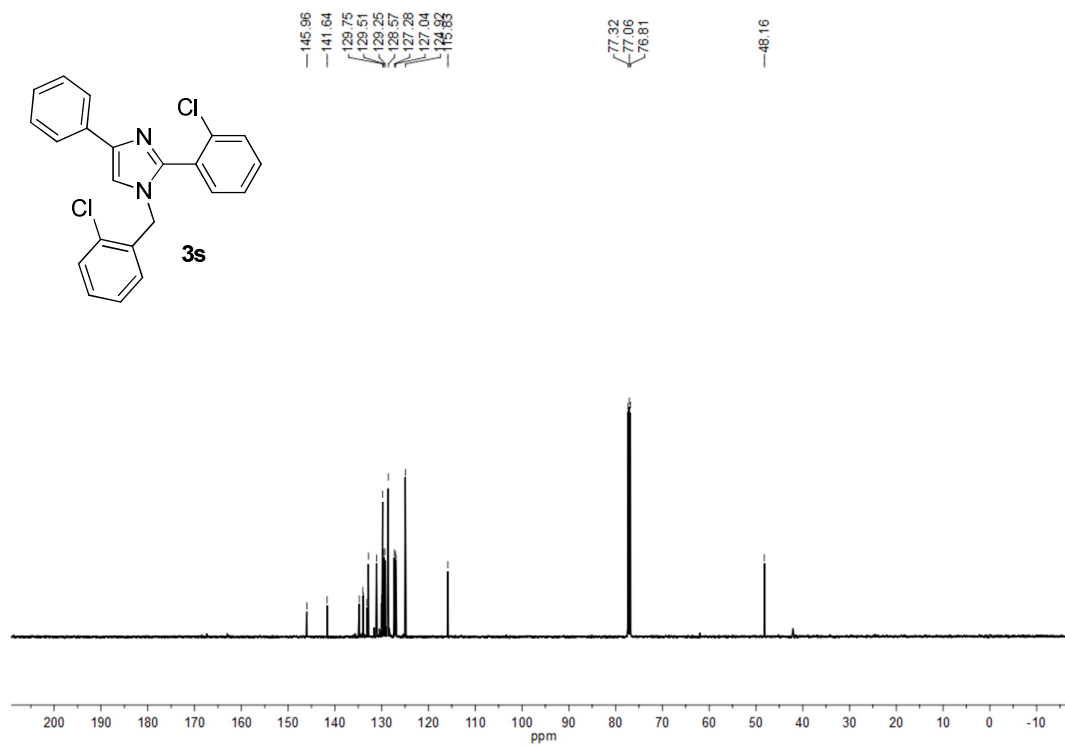


Figure S50. ¹³C NMR of 3s

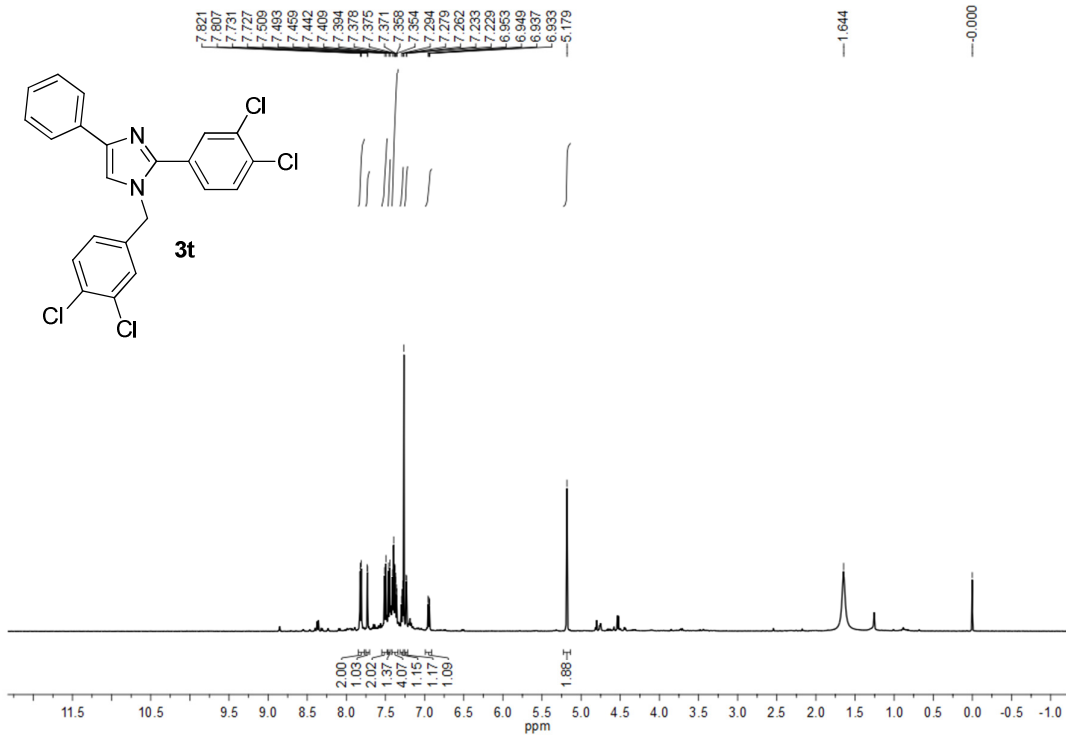


Figure S51. ¹H NMR of 3t

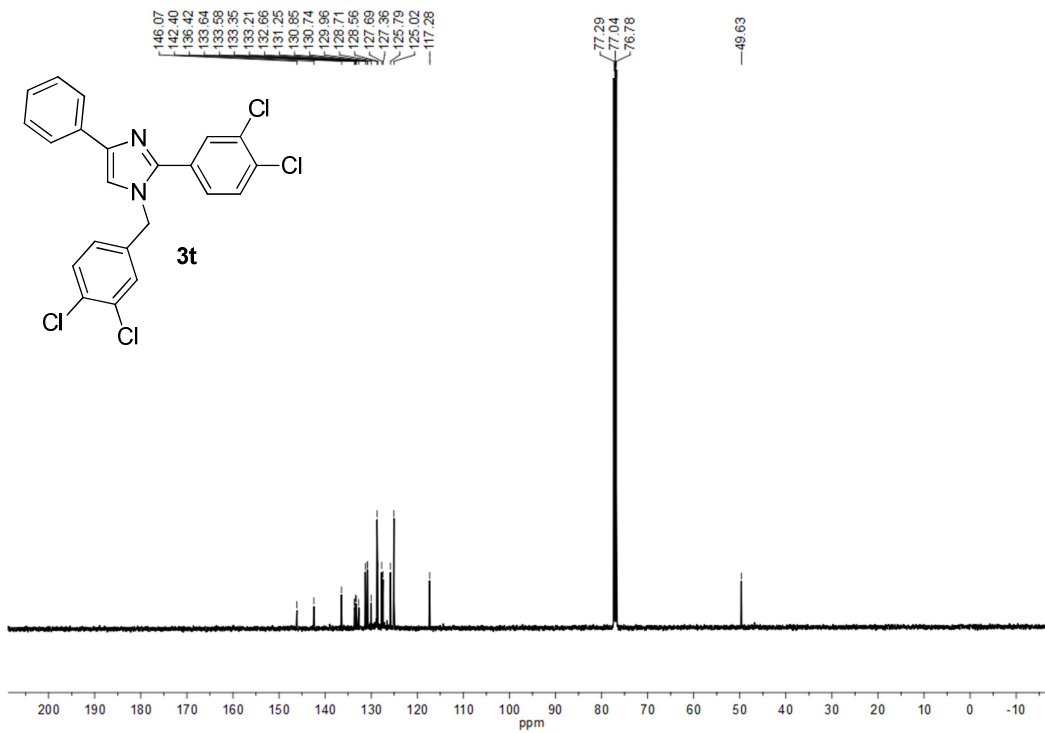


Figure S52. ¹³C NMR of 3t

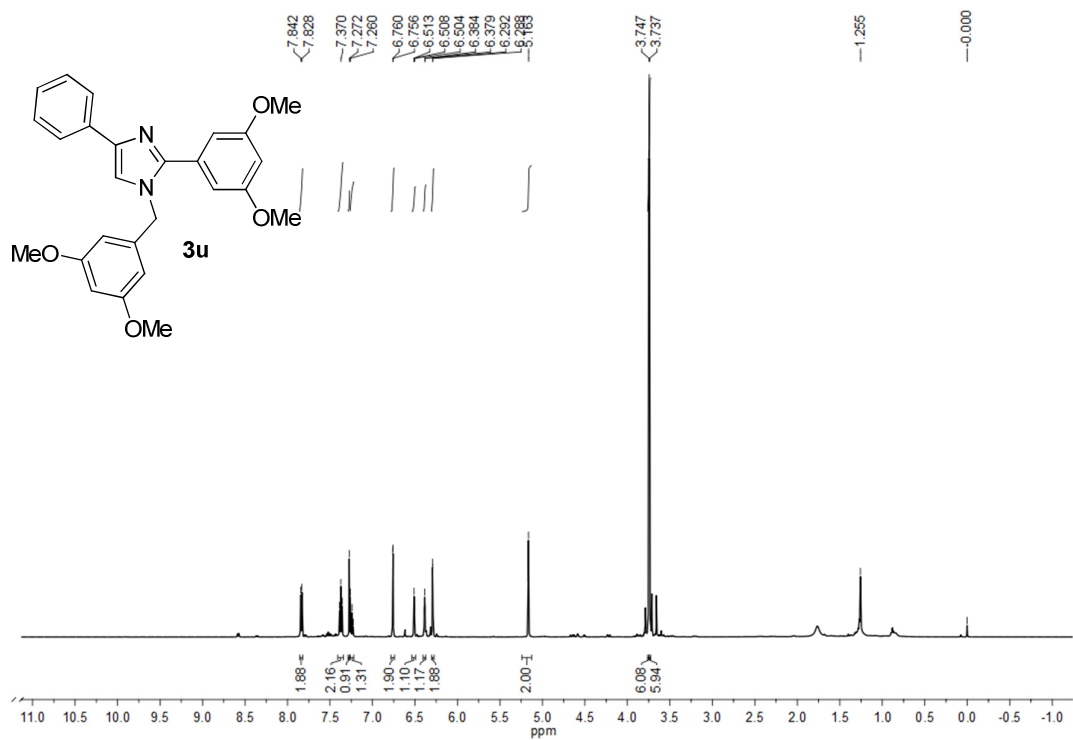


Figure S53. ^1H NMR 3u

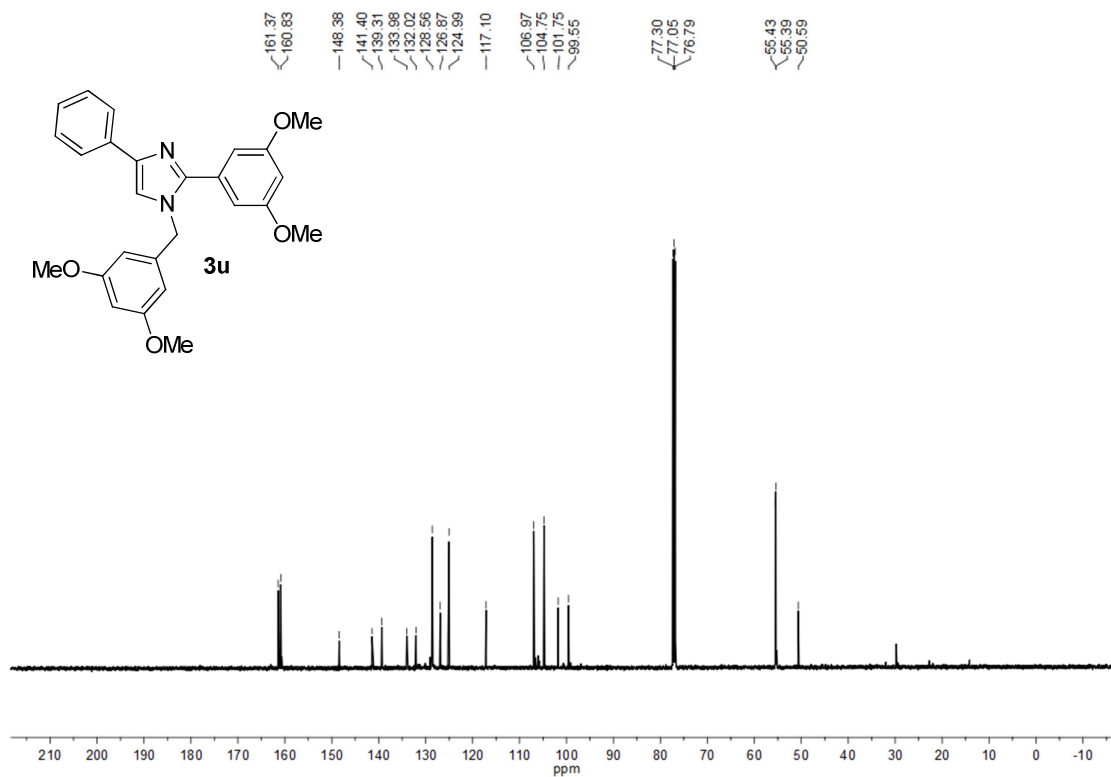


Figure S54. ^{13}C NMR of 3u

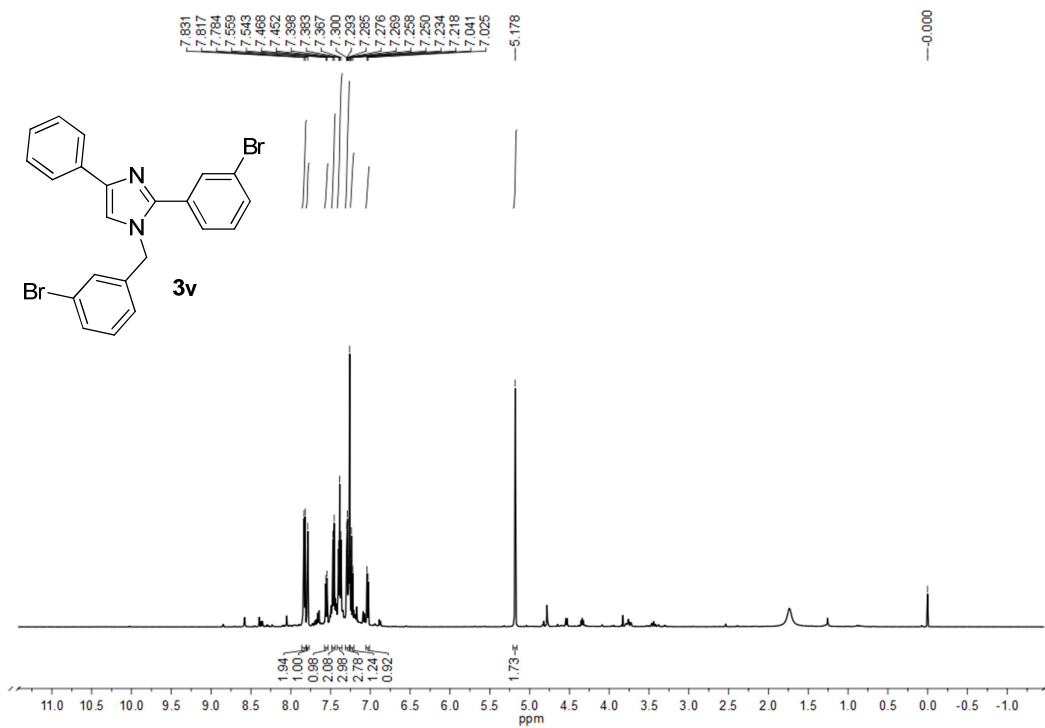


Figure S55. ¹H NMR of 3v

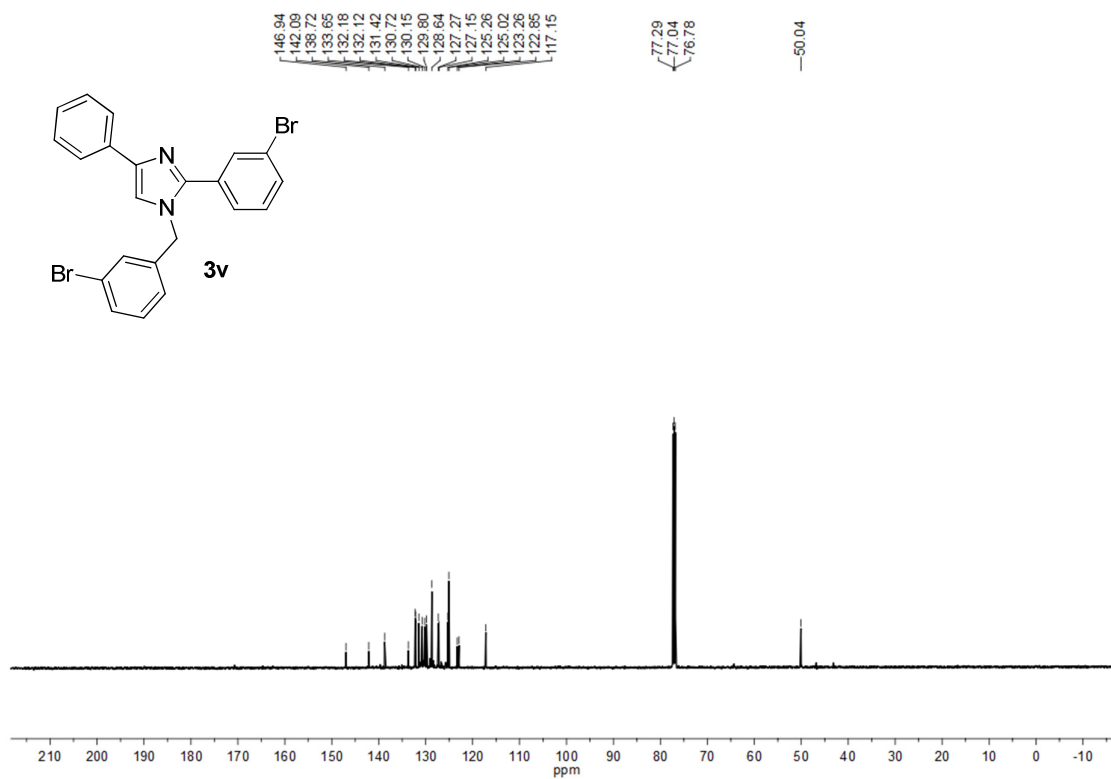


Figure S56. ¹³C NMR of 3v

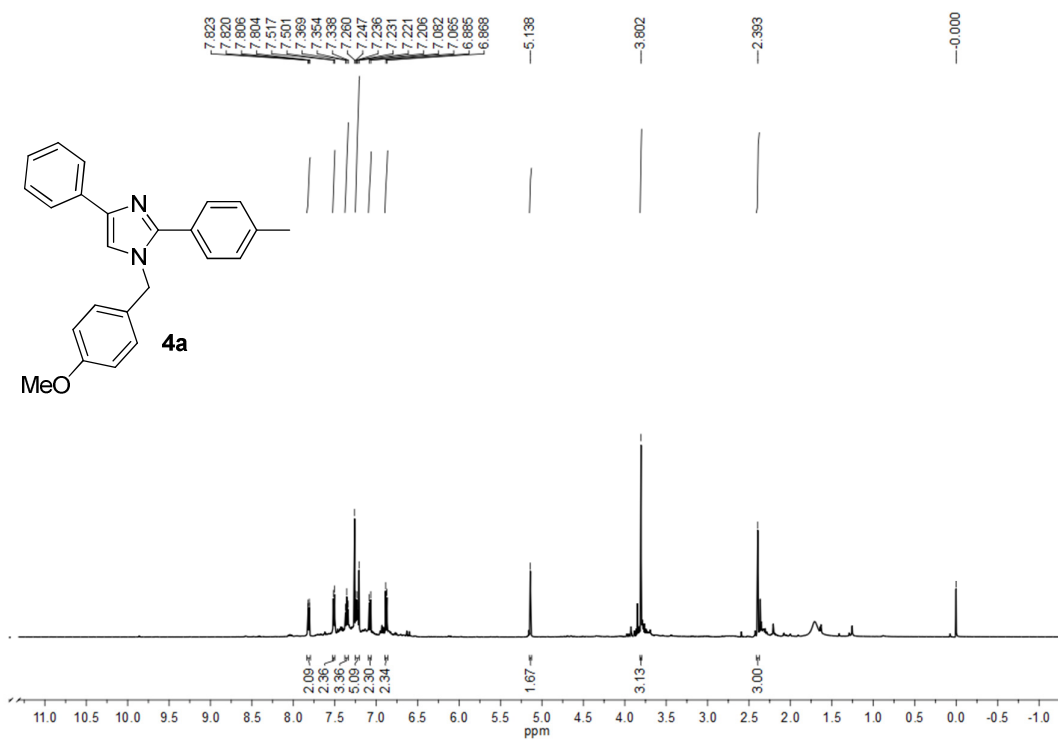


Figure S57. ¹H NMR of 4a

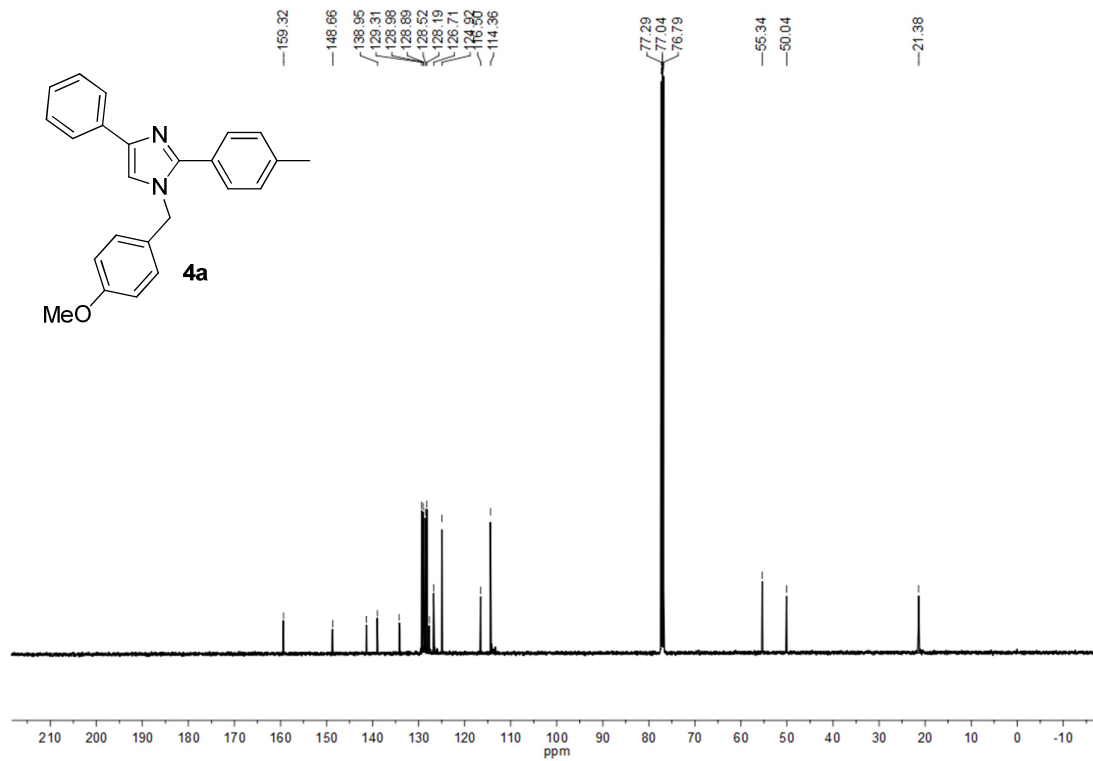


Figure S58. ¹³C NMR of 4a

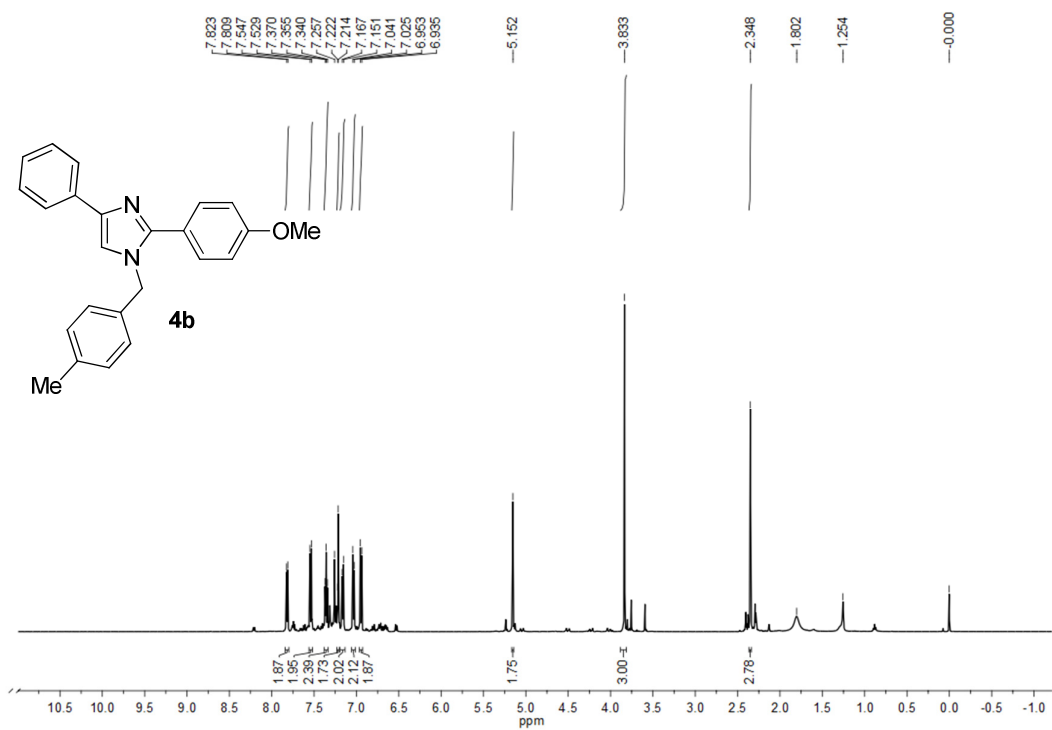


Figure S59. ¹H NMR of **4b**

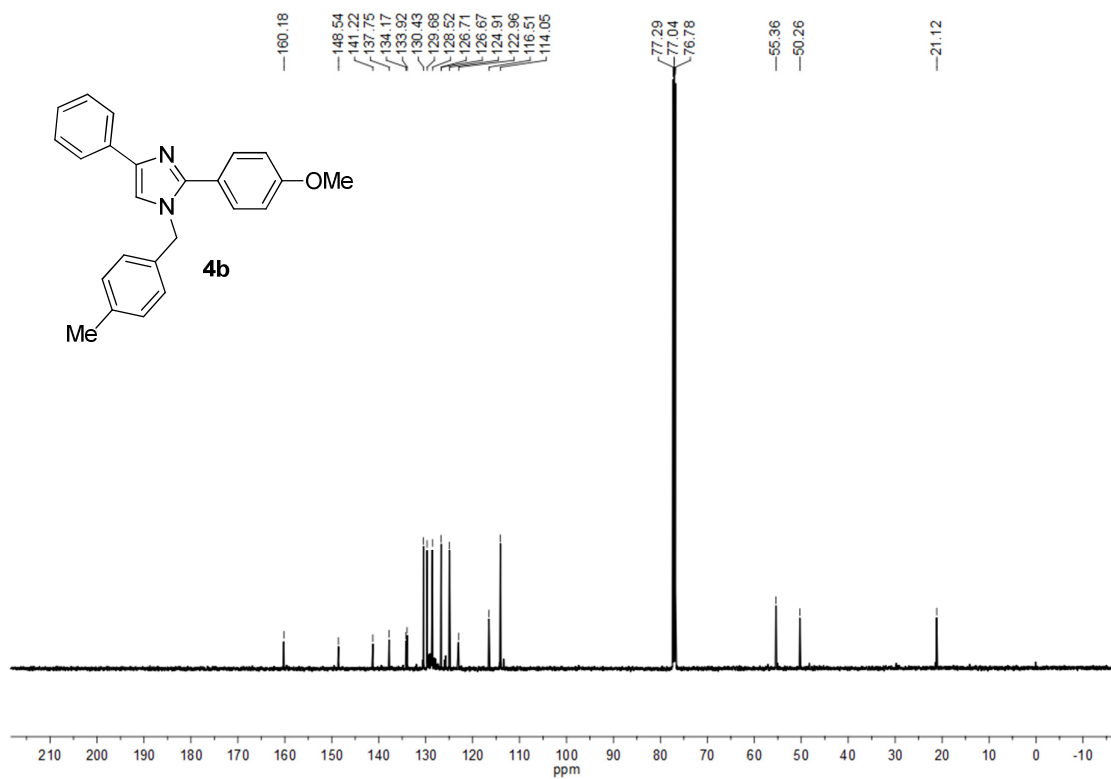


Figure S60. ¹³C NMR of **4b**

CCDC No: 1812406

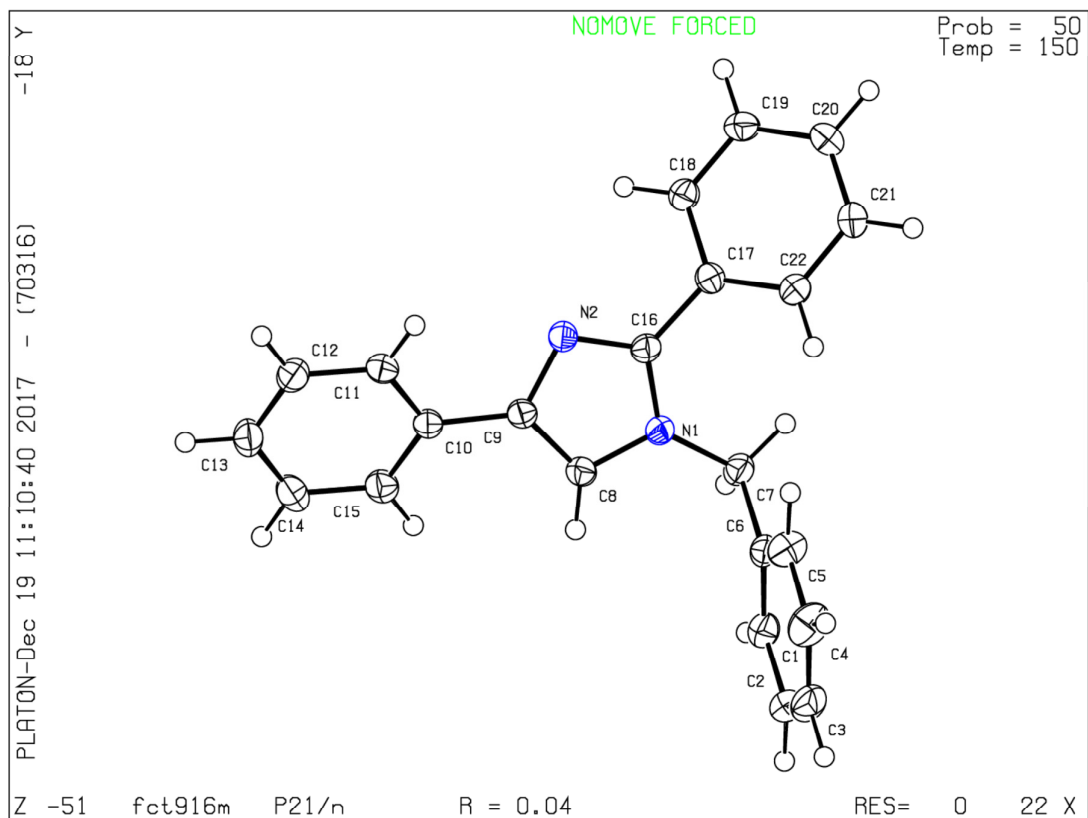


Figure S61. ORTEP drawing for compound 3a