

Electronic Supplementary Information

Chemoselective synthesis of 5,4'-imidazolinyI spirobarbiturates via NBS-promoted cyclization of unsaturated barbiturates and amidines

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1. NMR Spectra of products 3

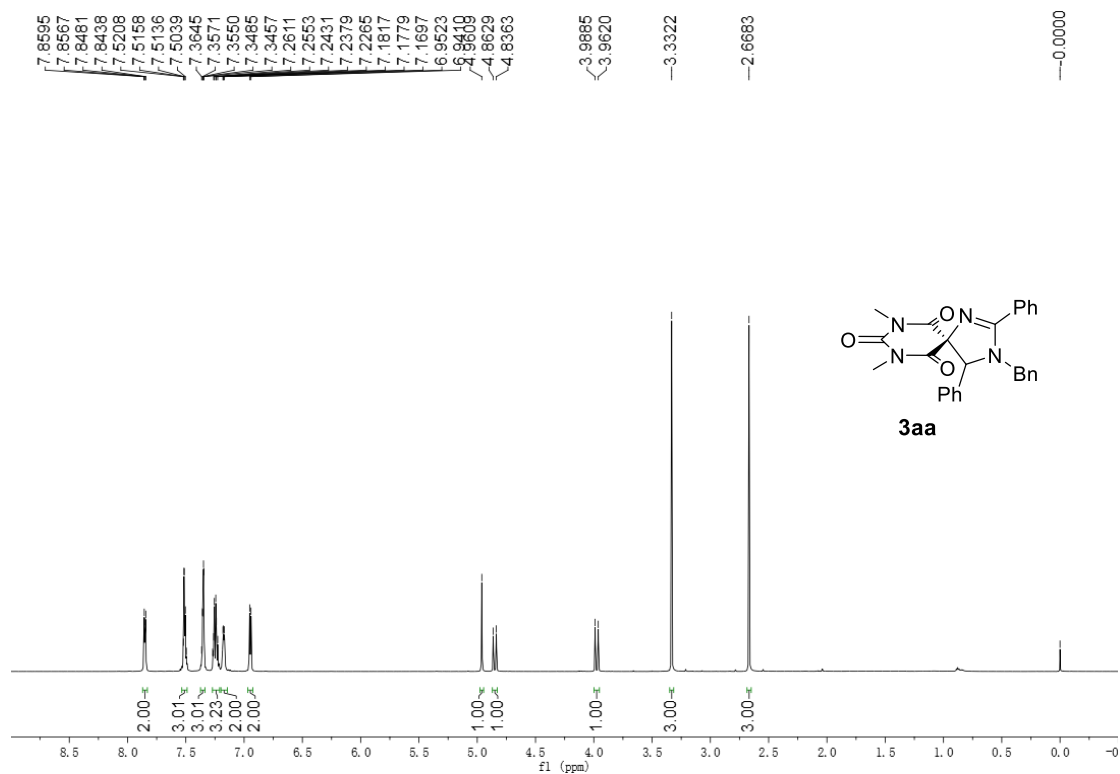


Figure S1. ¹H NMR (600 MHz, CDCl₃) of compound 3aa

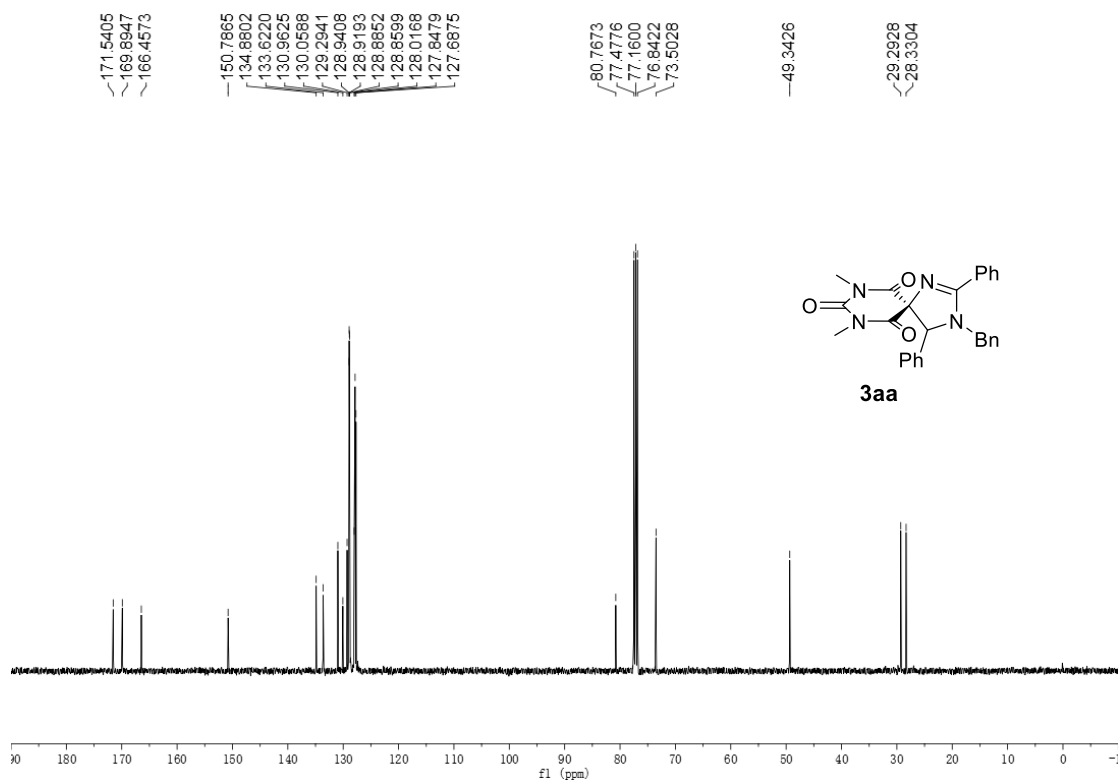


Figure S2. ¹³C NMR (100 MHz, CDCl₃) of compound 3aa

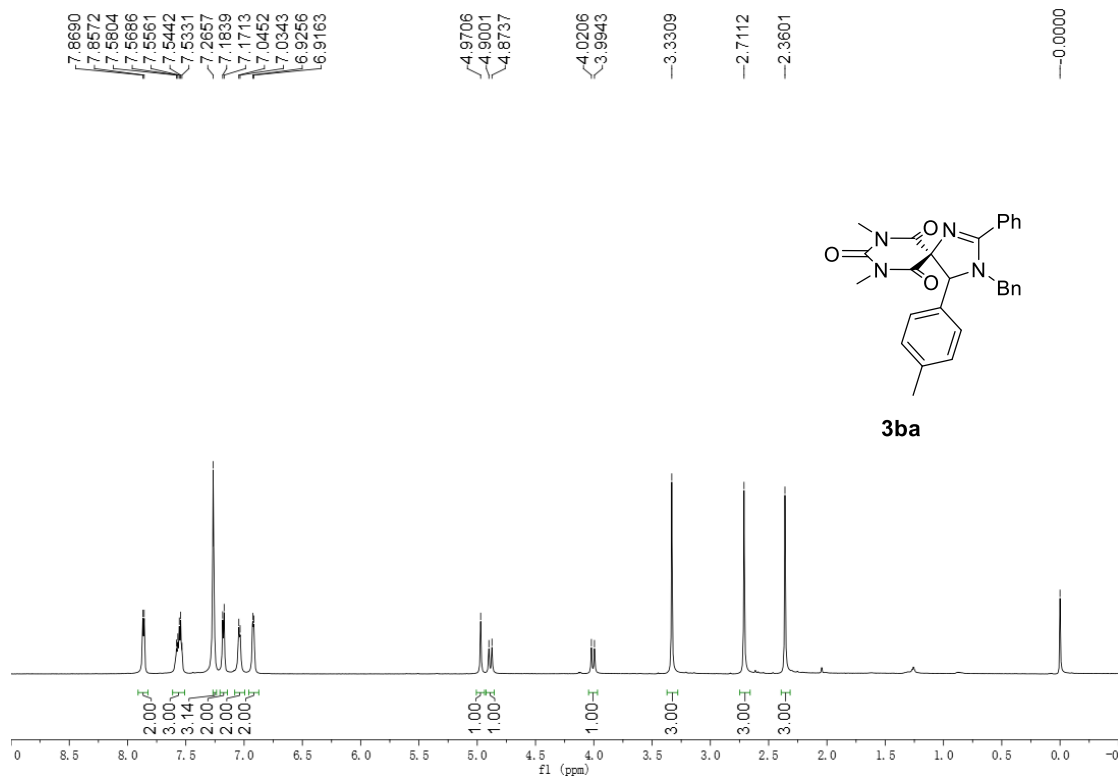


Figure S3. ¹H NMR (600 MHz, CDCl₃) of compound 3ba

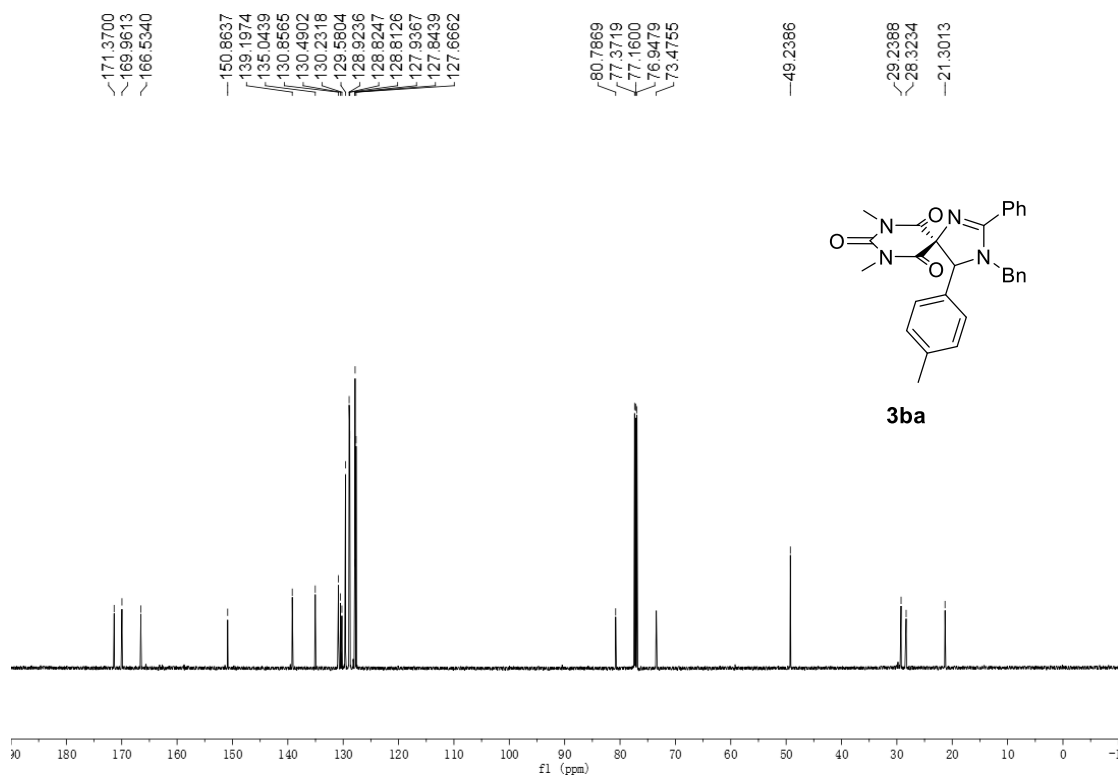


Figure S4. ¹³C NMR (150 MHz, CDCl₃) of compound 3ba

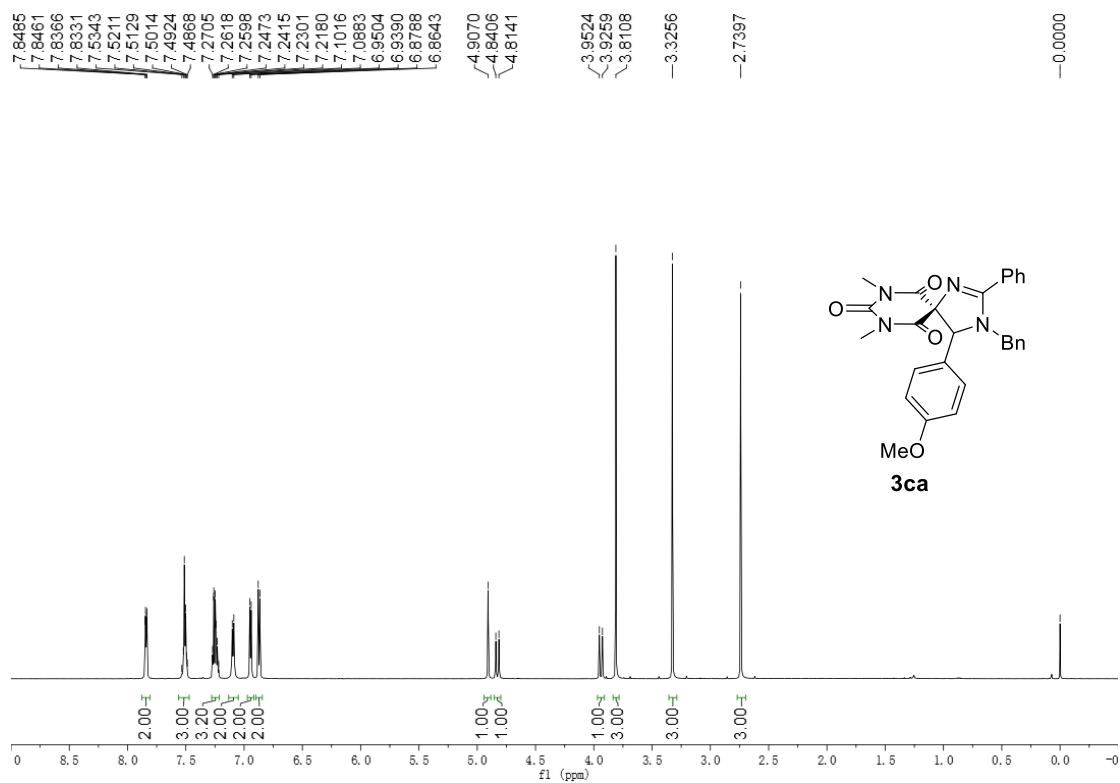


Figure S5. ¹H NMR (600 MHz, CDCl₃) of compound **3ca**

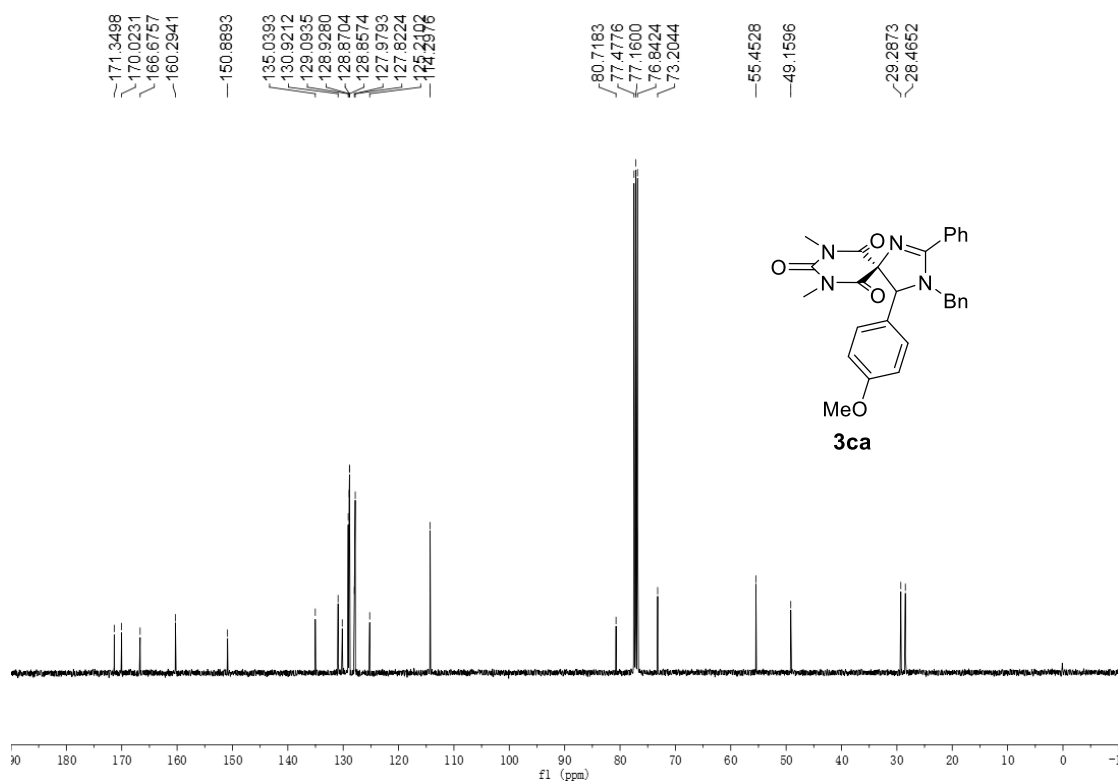


Figure S6. ¹³C NMR (100 MHz, CDCl₃) of compound **3ca**

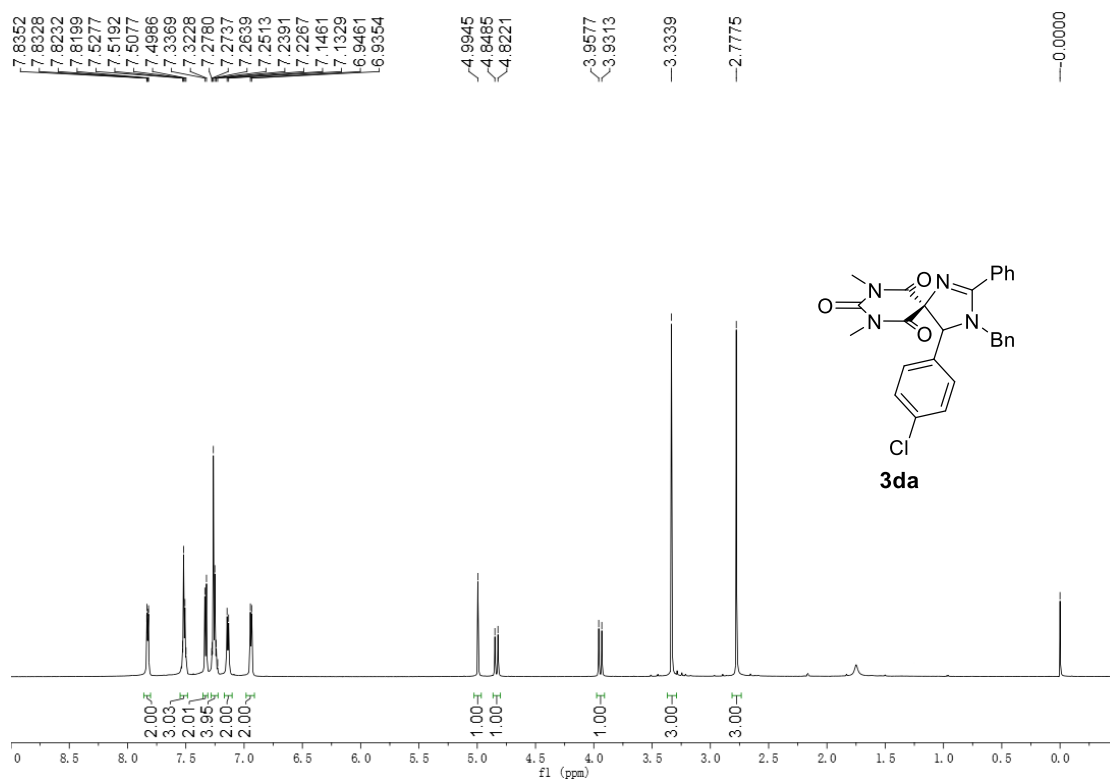


Figure S7. ¹H NMR (600 MHz, CDCl₃) of compound 3da

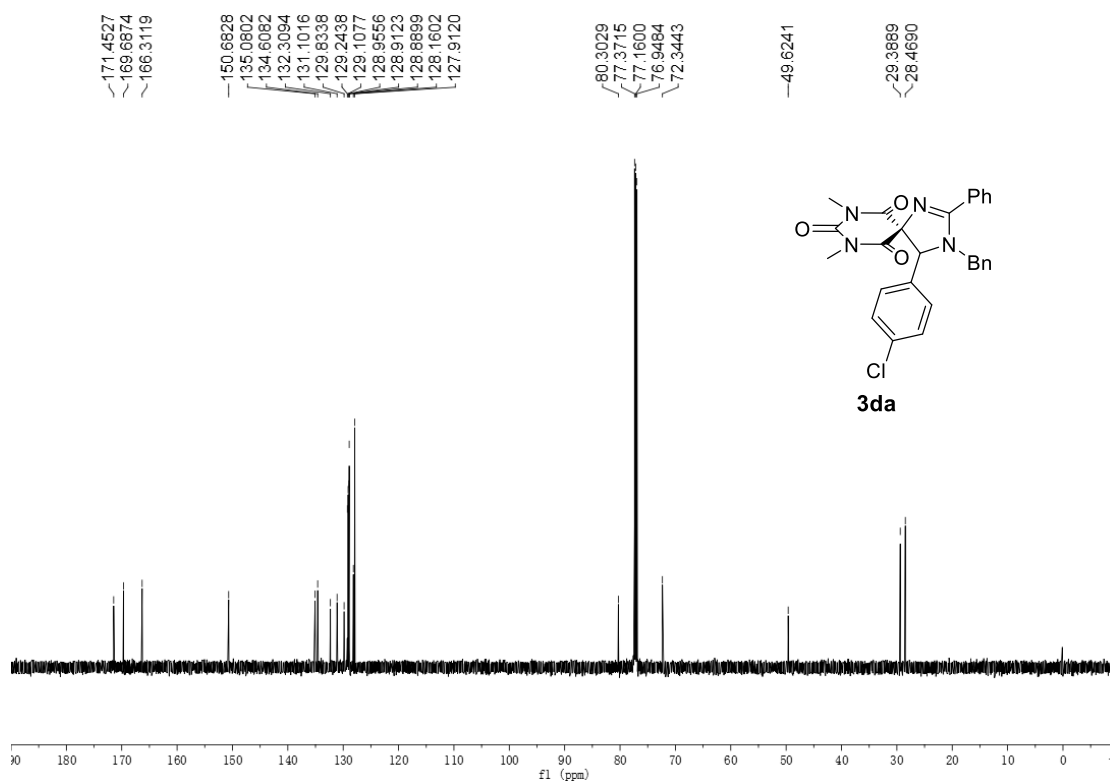


Figure S8. ¹³C NMR (150 MHz, CDCl₃) of compound 3da

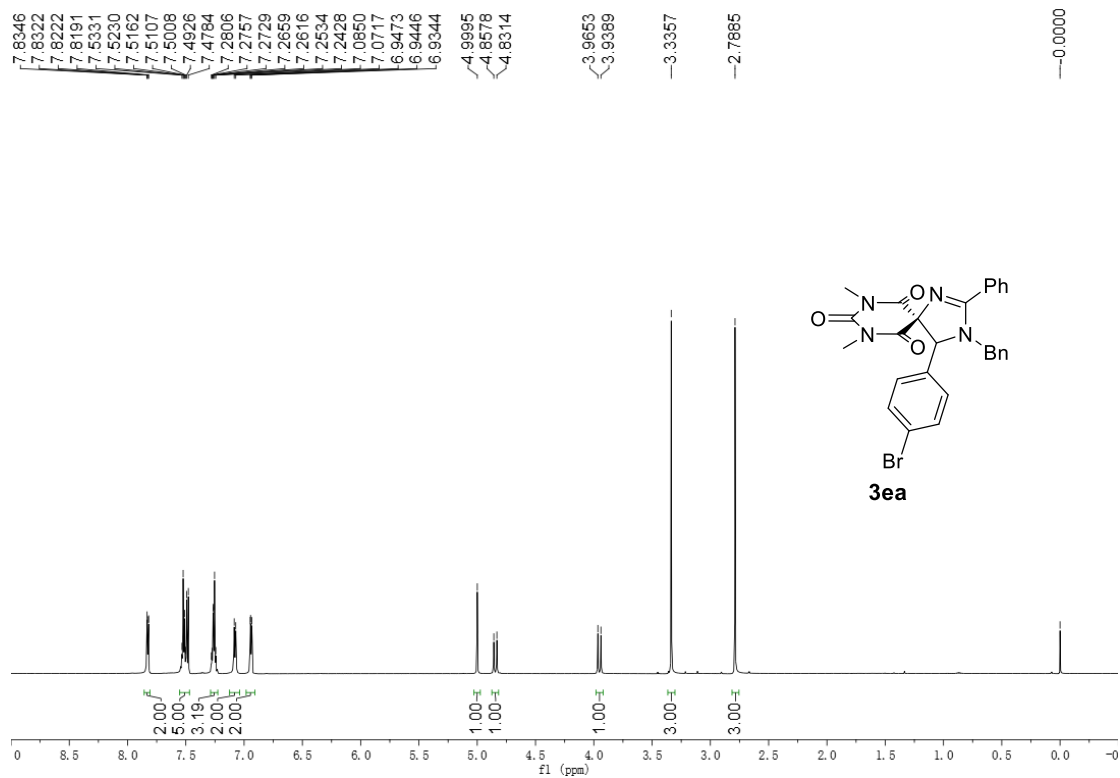


Figure S9. ¹H NMR (600 MHz, CDCl₃) of compound **3ea**

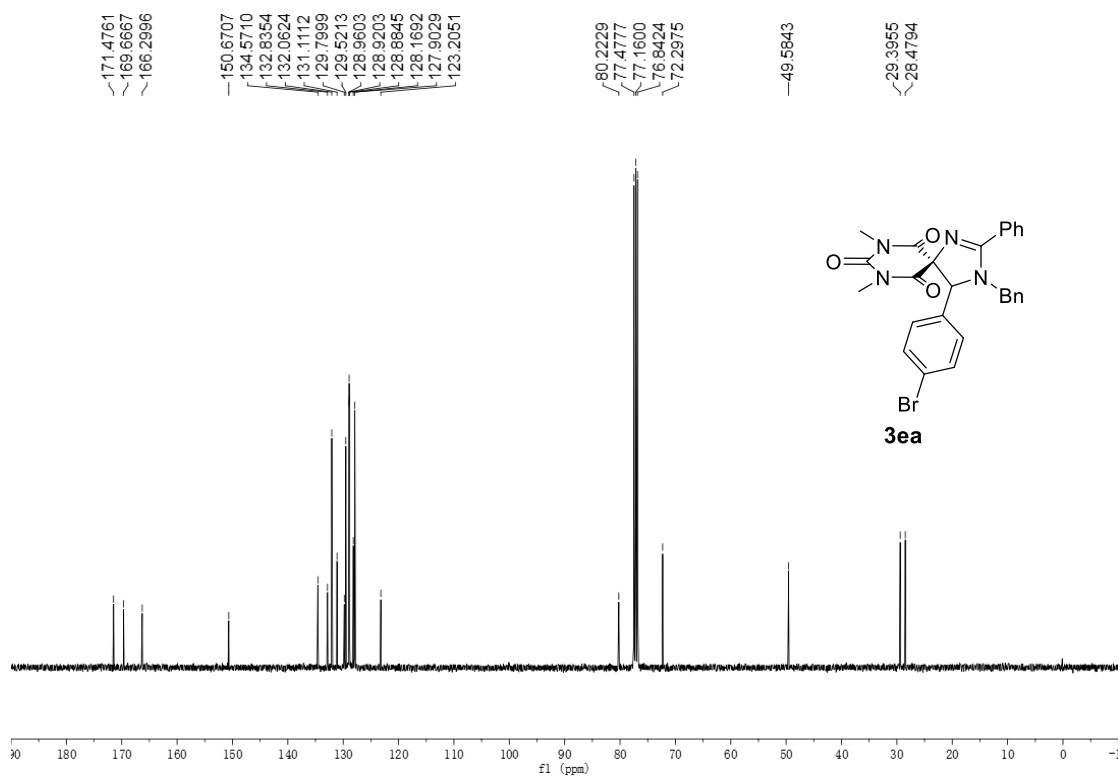


Figure S10. ¹³C NMR (100 MHz, CDCl₃) of compound **3ea**

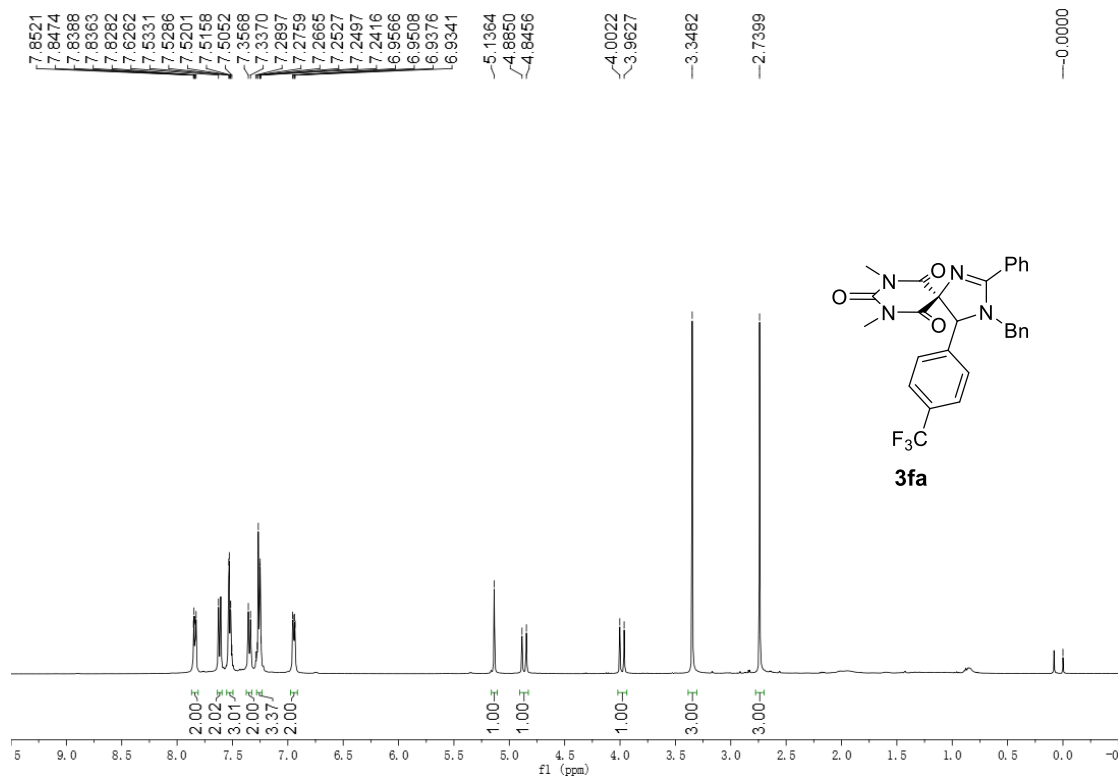


Figure S11. ¹H NMR (400 MHz, CDCl₃) of compound **3fa**

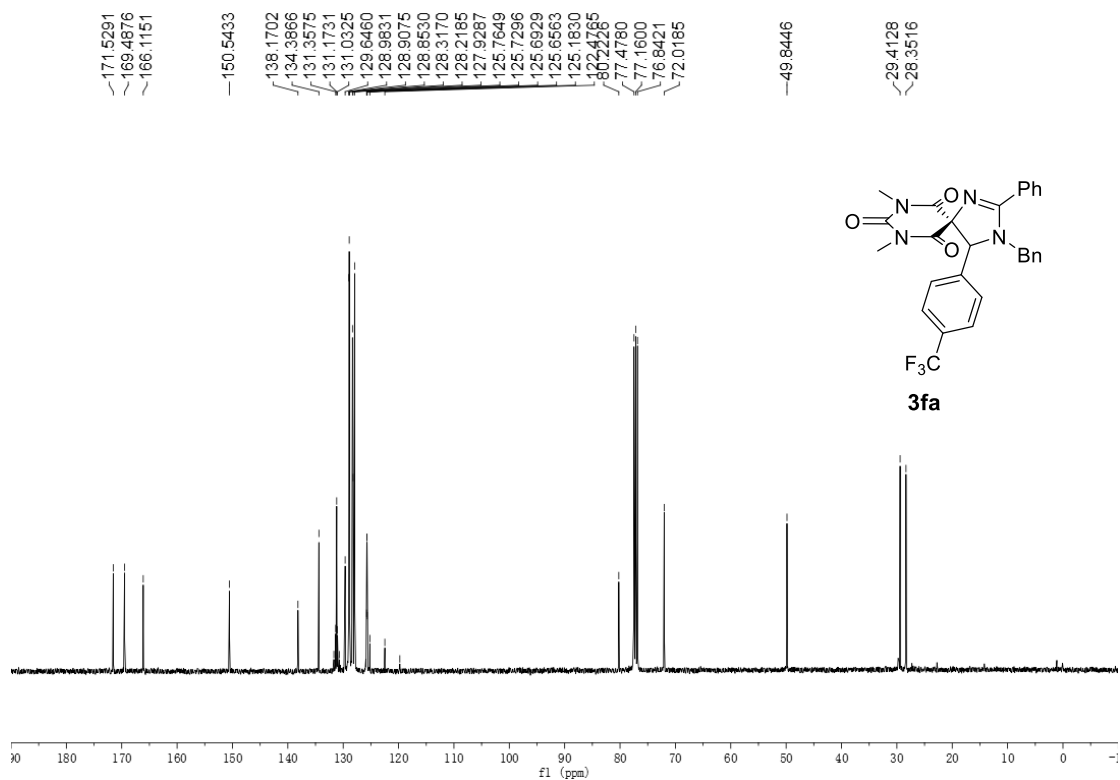


Figure S12. ¹³C NMR (100 MHz, CDCl₃) of compound **3fa**

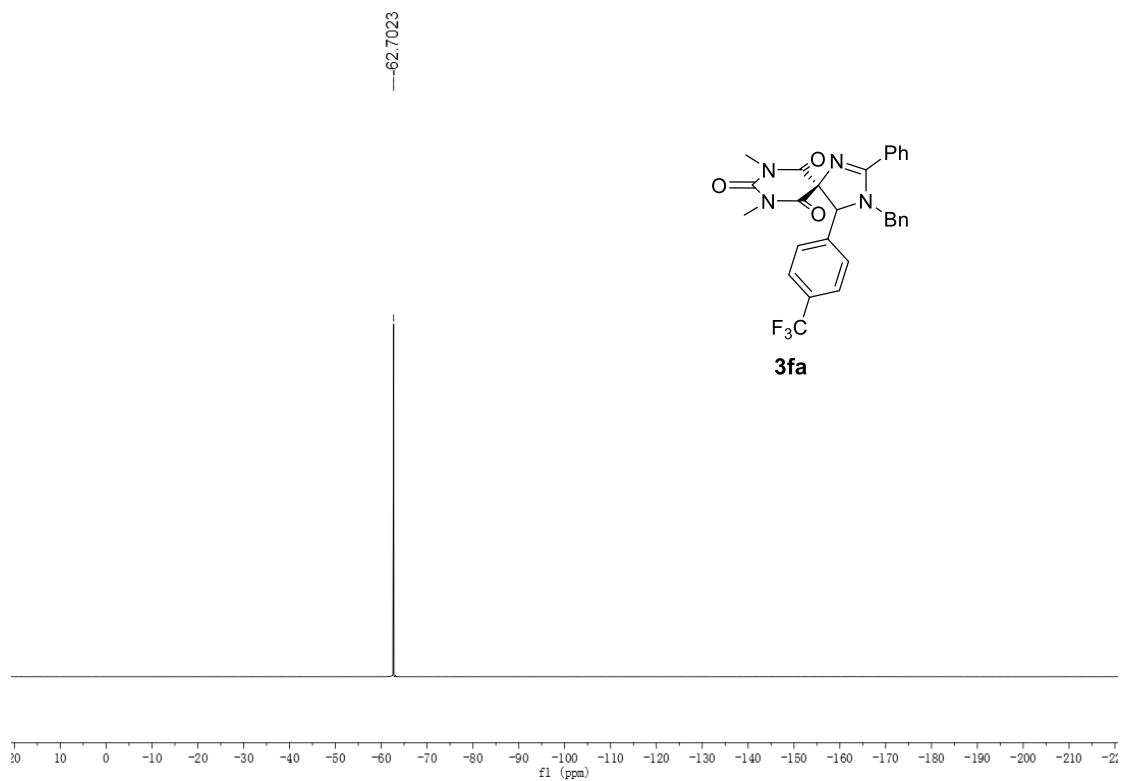


Figure S13. ^{19}F NMR (470 MHz, CDCl_3) of compound **3fa**

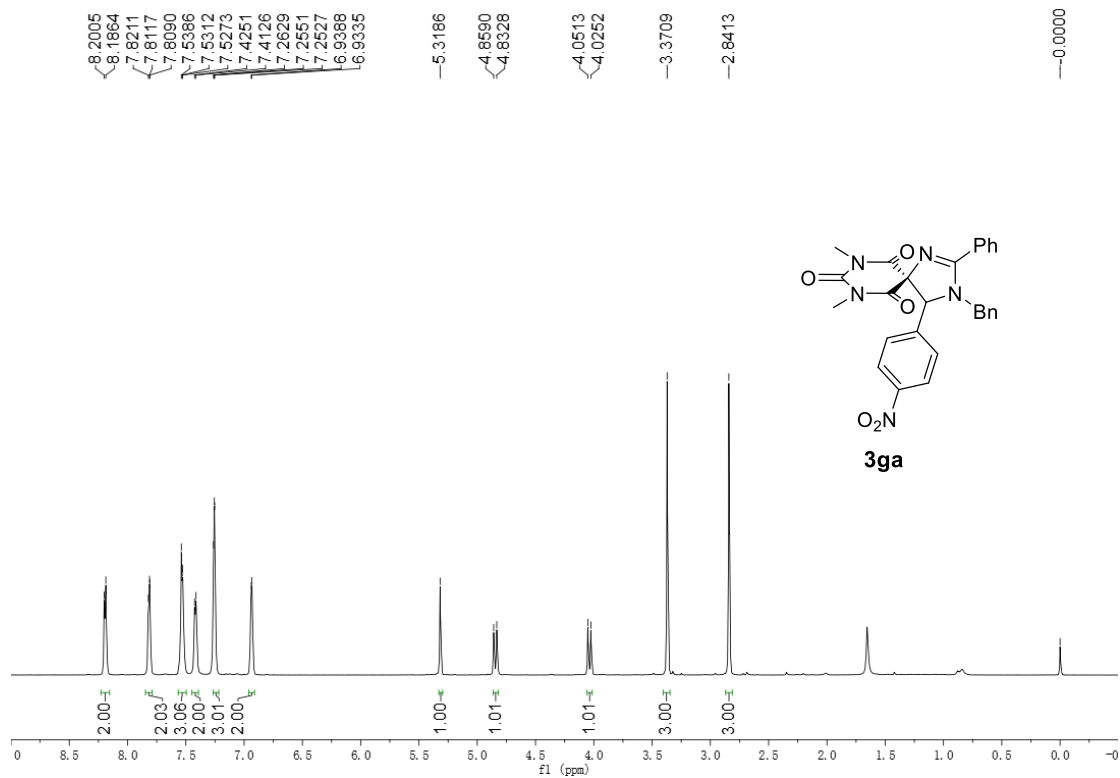


Figure S14. ¹H NMR (600 MHz, CDCl₃) of compound **3ga**

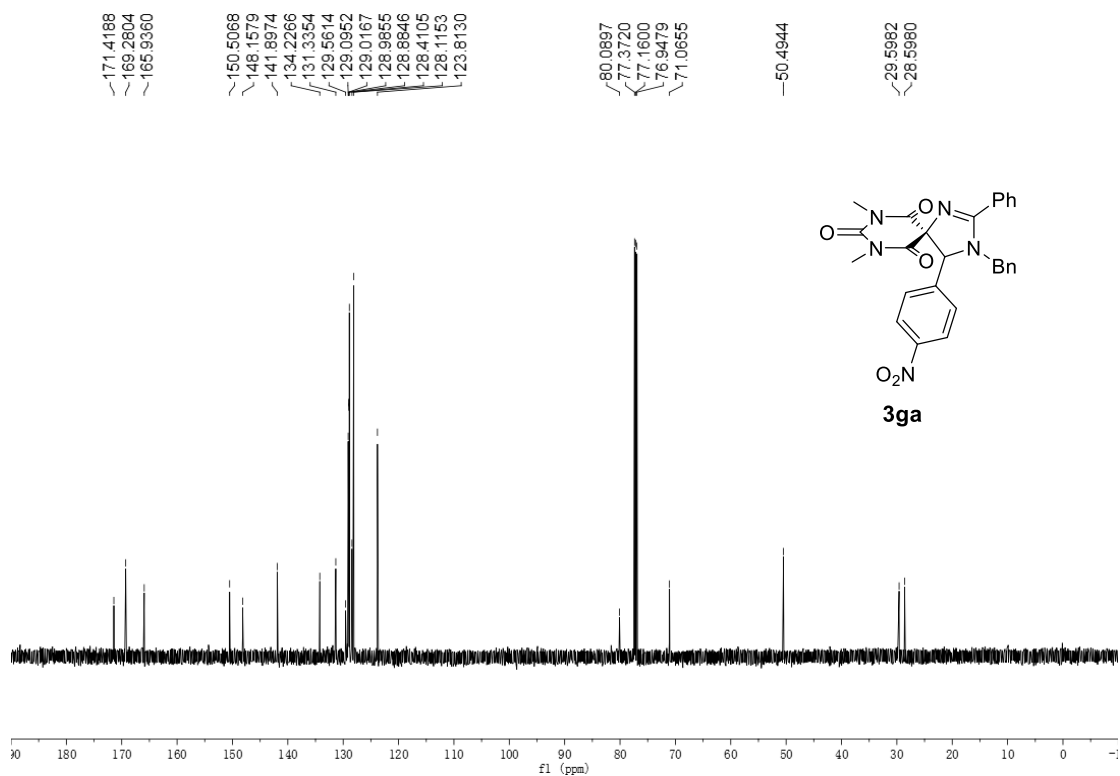


Figure S15. ¹³C NMR (150 MHz, CDCl₃) of compound **3ga**

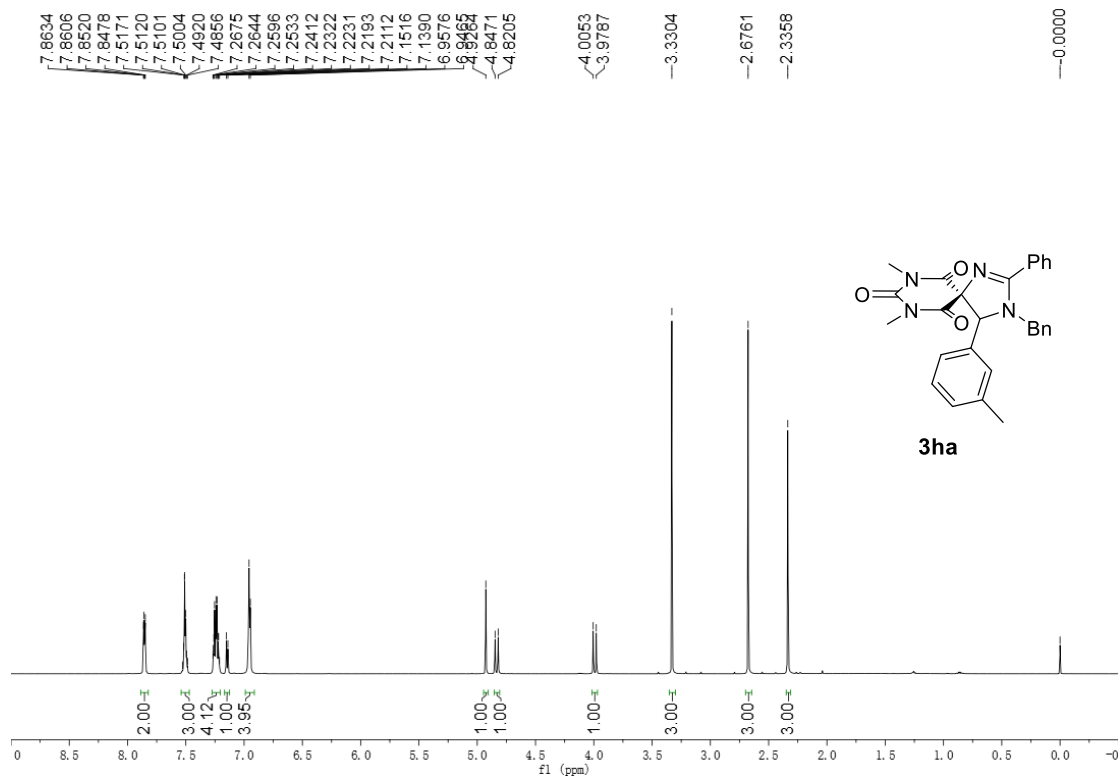


Figure S16. ¹H NMR (600 MHz, CDCl₃) of compound 3ha

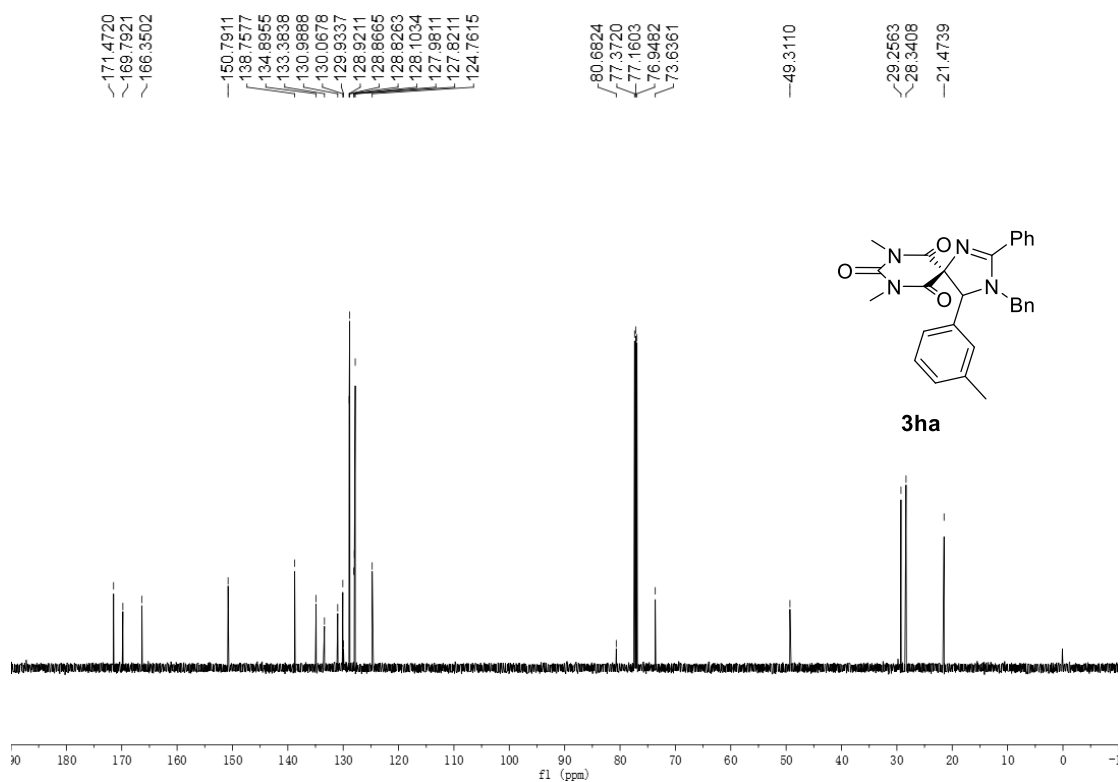


Figure S17. ¹³C NMR (150 MHz, CDCl₃) of compound 3ha

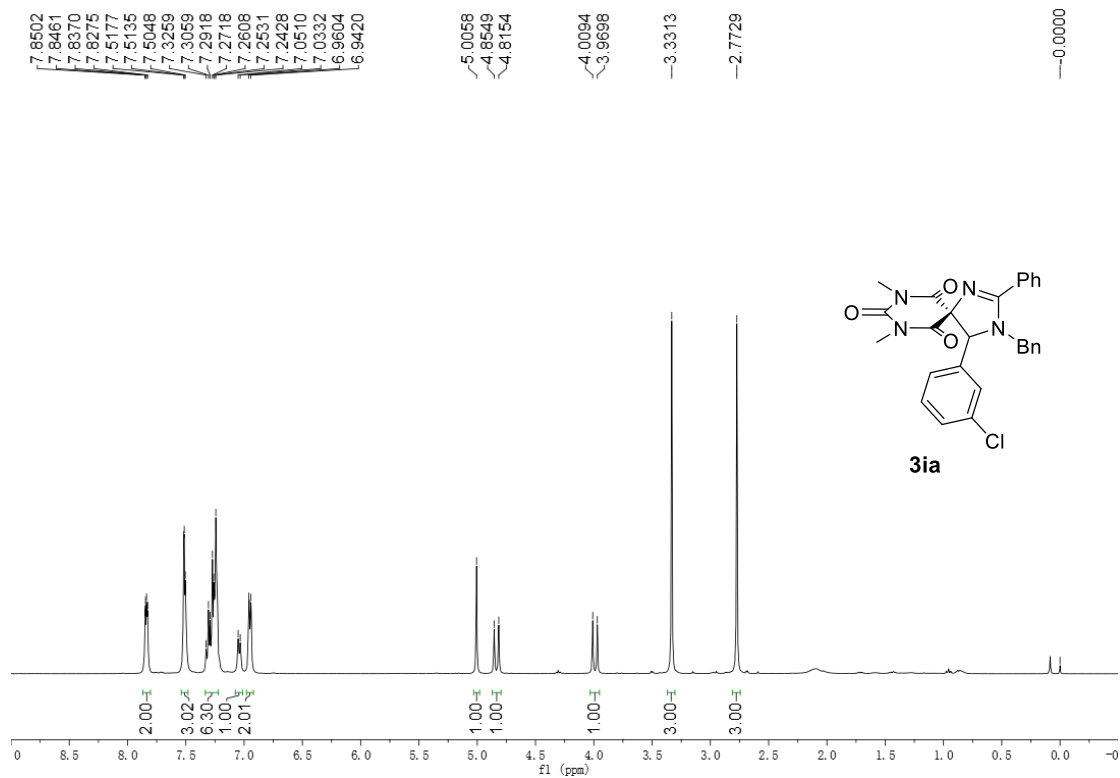


Figure S18. ¹H NMR (400 MHz, CDCl₃) of compound **3ia**

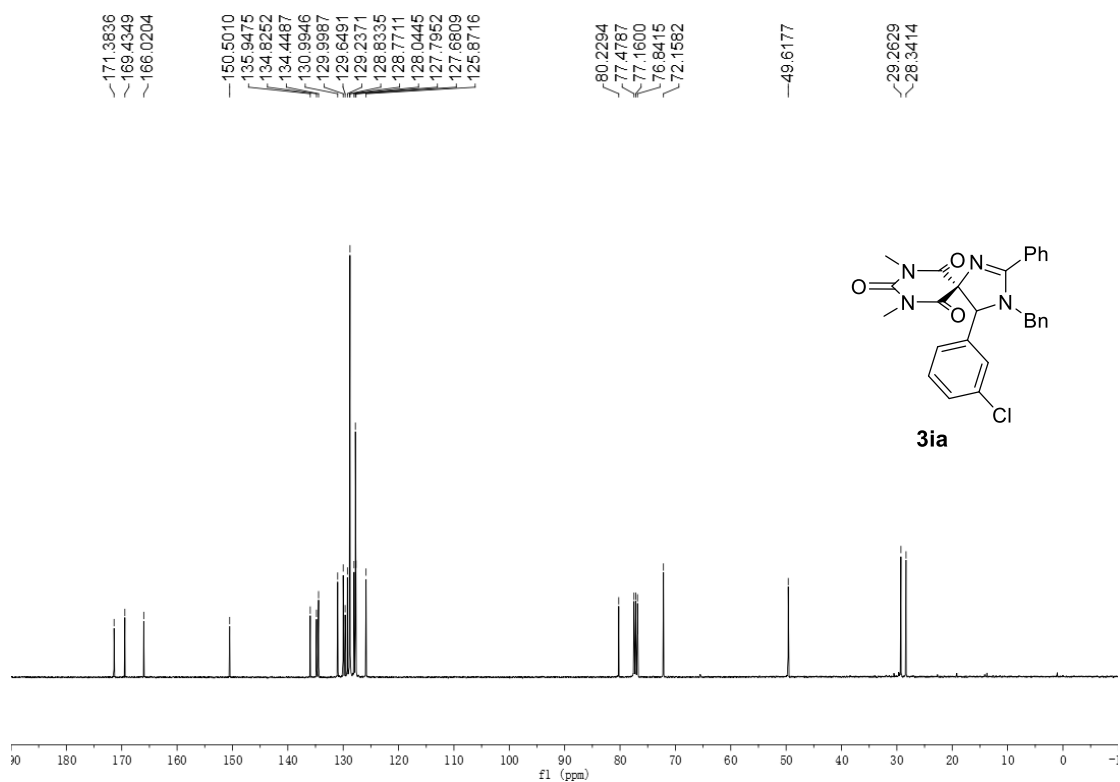


Figure S19. ¹³C NMR (100 MHz, CDCl₃) of compound **3ia**

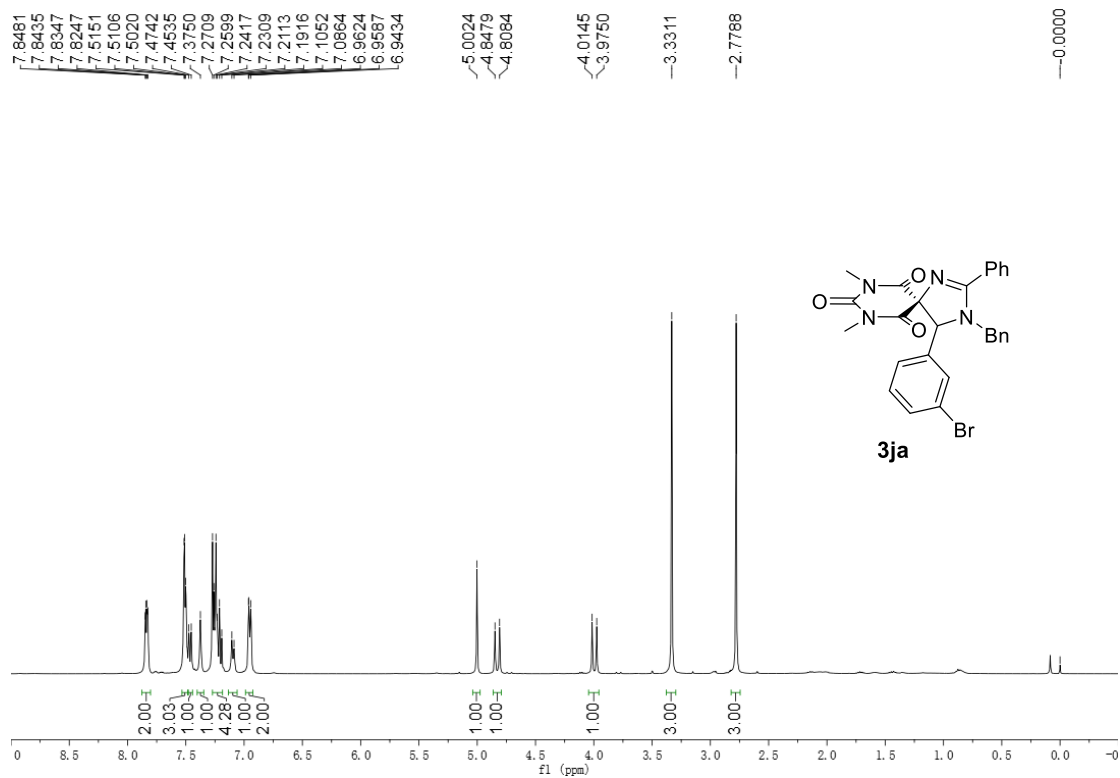


Figure S20. ¹H NMR (400 MHz, CDCl₃) of compound 3ja

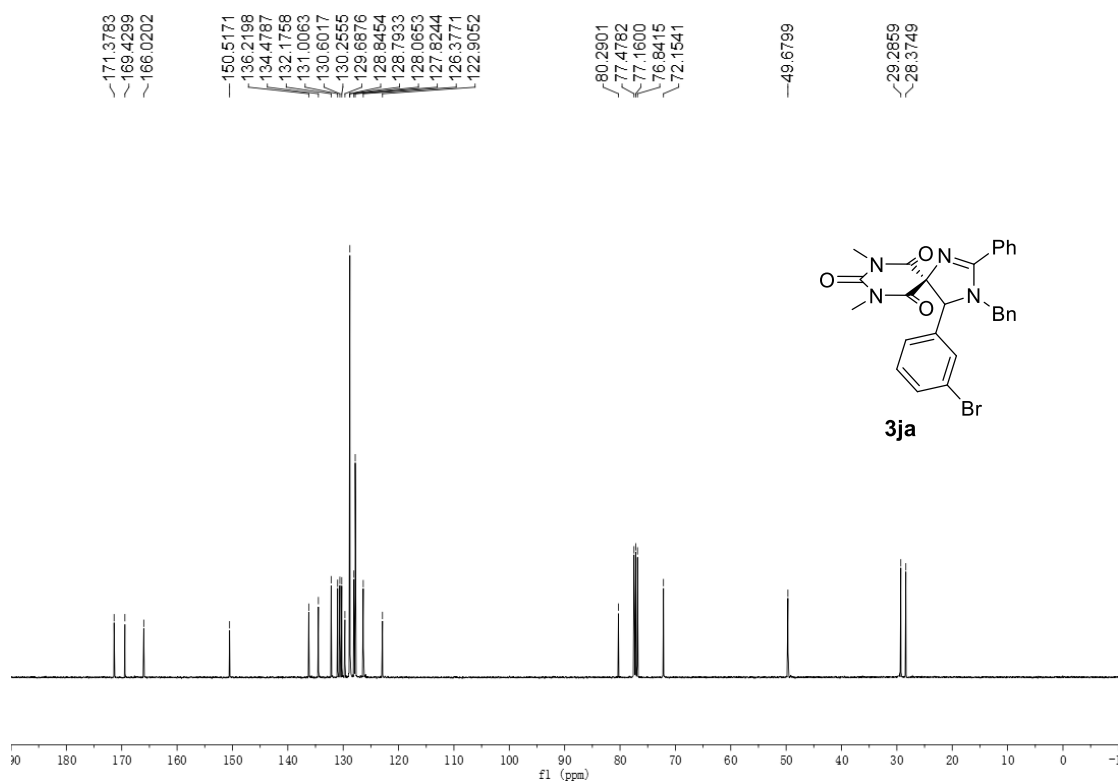
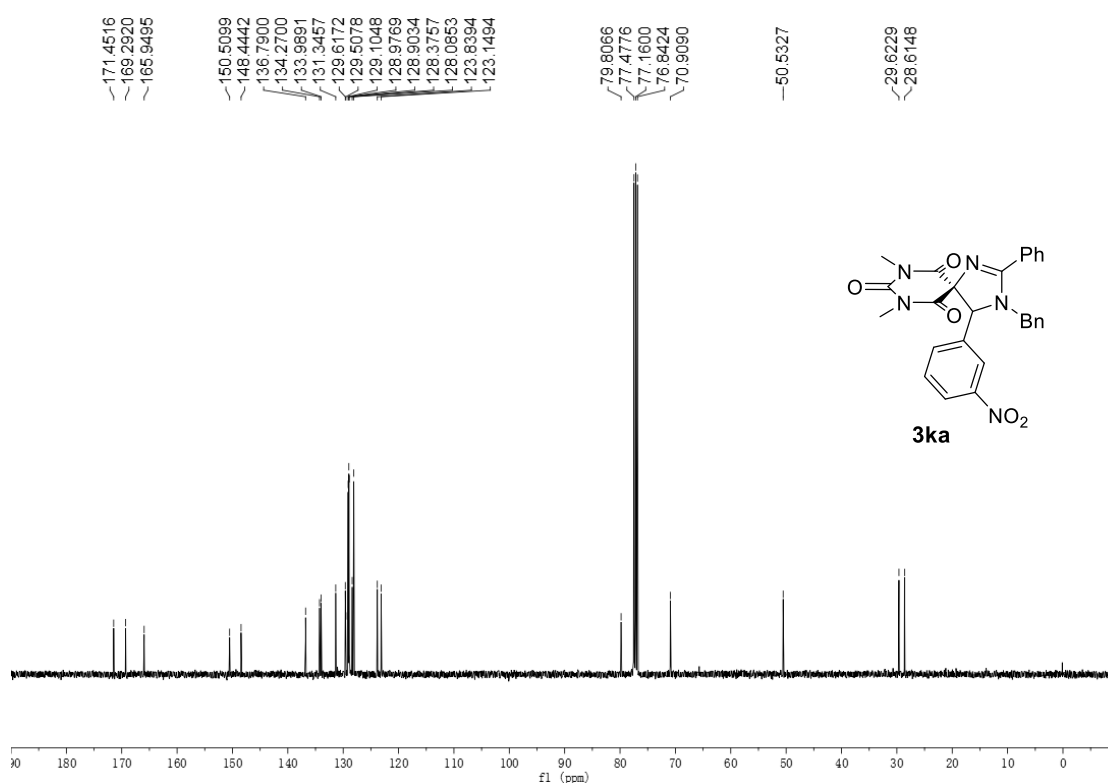
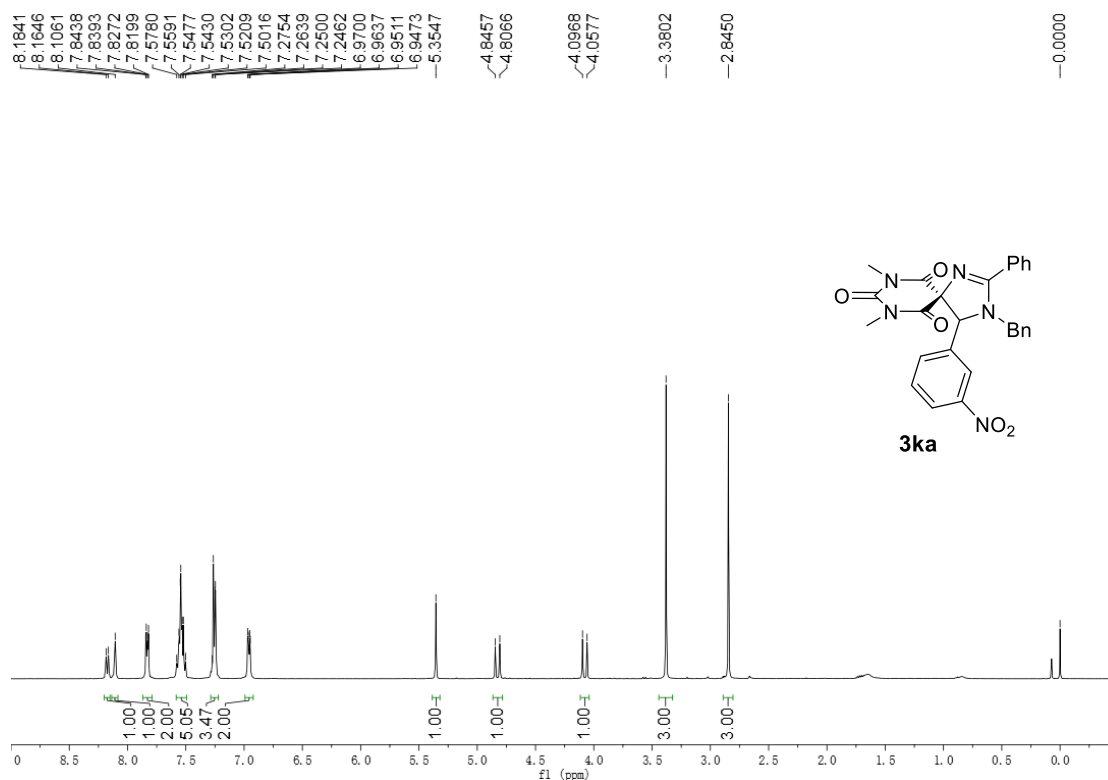


Figure S21. ¹³C NMR (100 MHz, CDCl₃) of compound 3ja



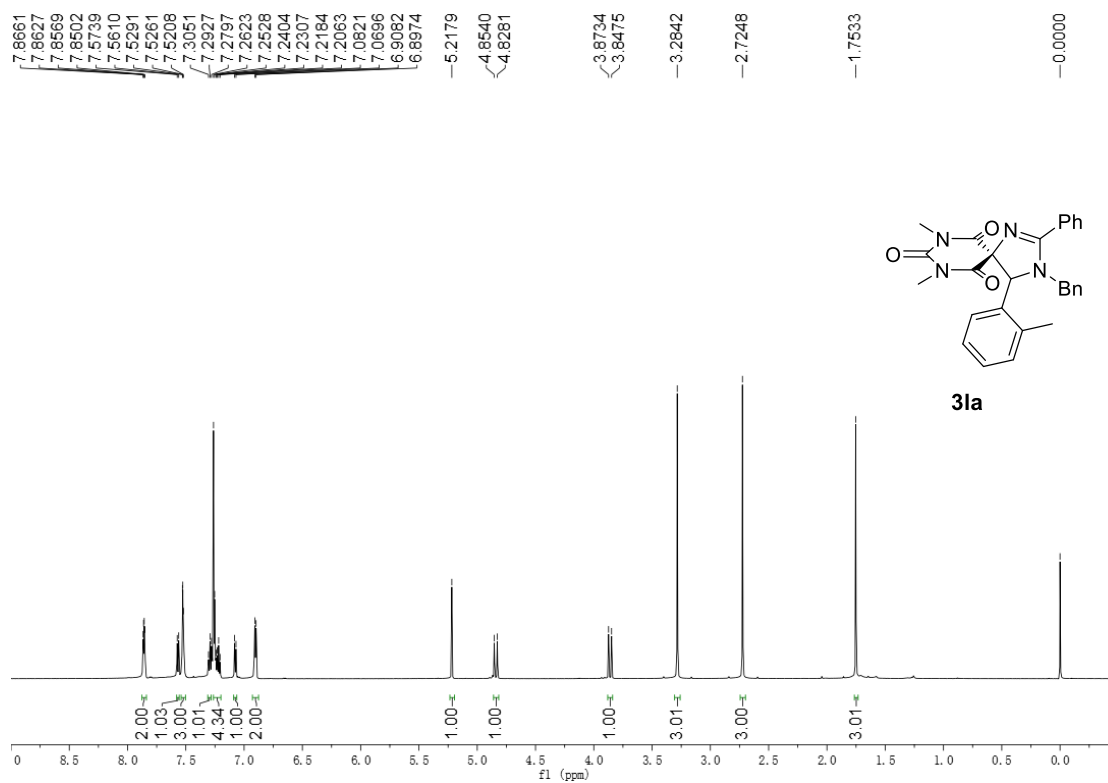


Figure S24. ^1H NMR (600 MHz, CDCl_3) of compound **3la**

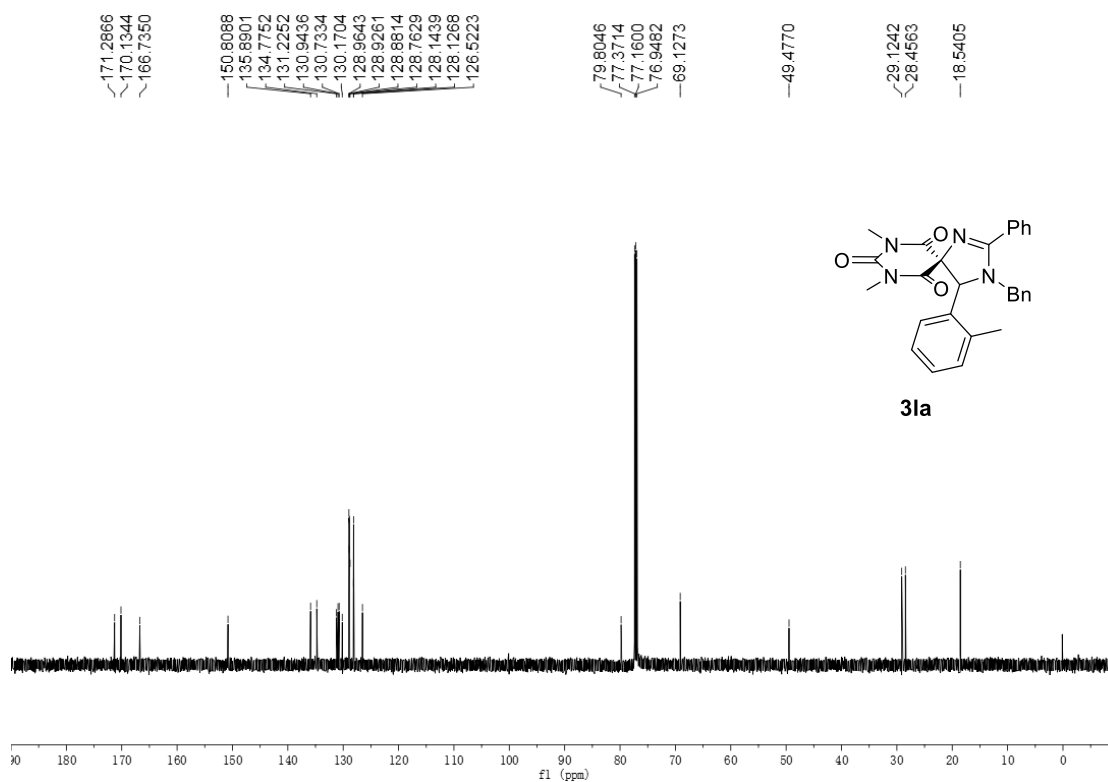
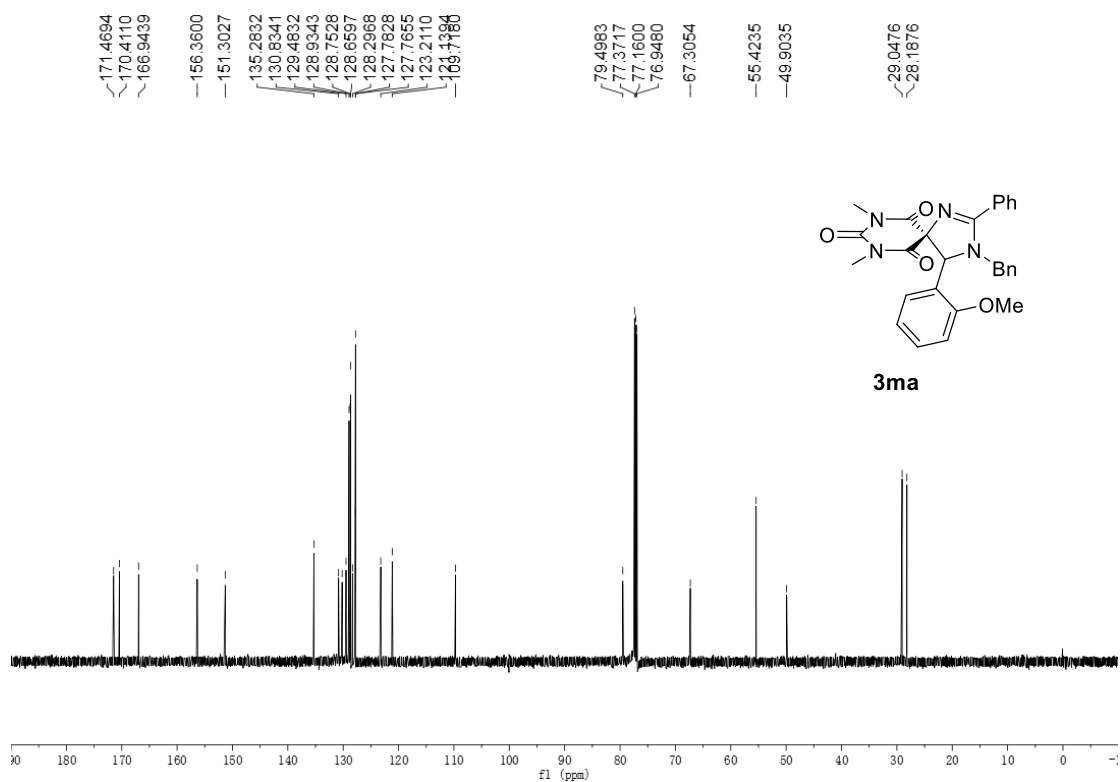
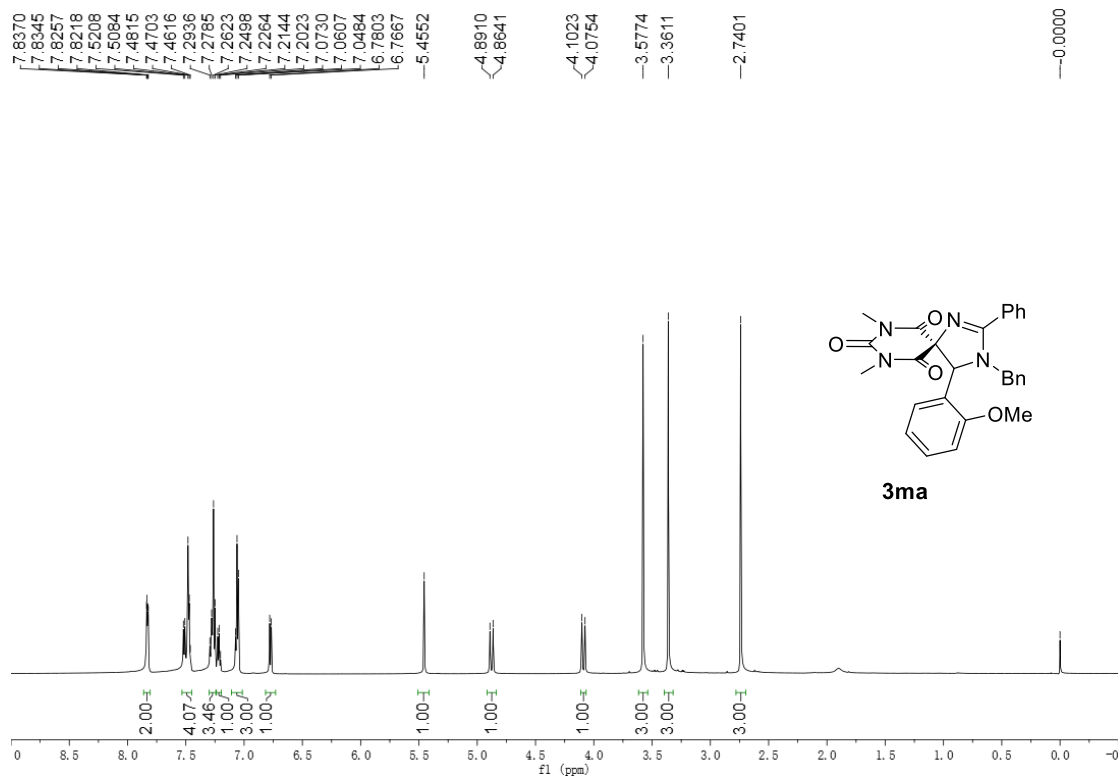


Figure S25. ^{13}C NMR (150 MHz, CDCl_3) of compound **3la**



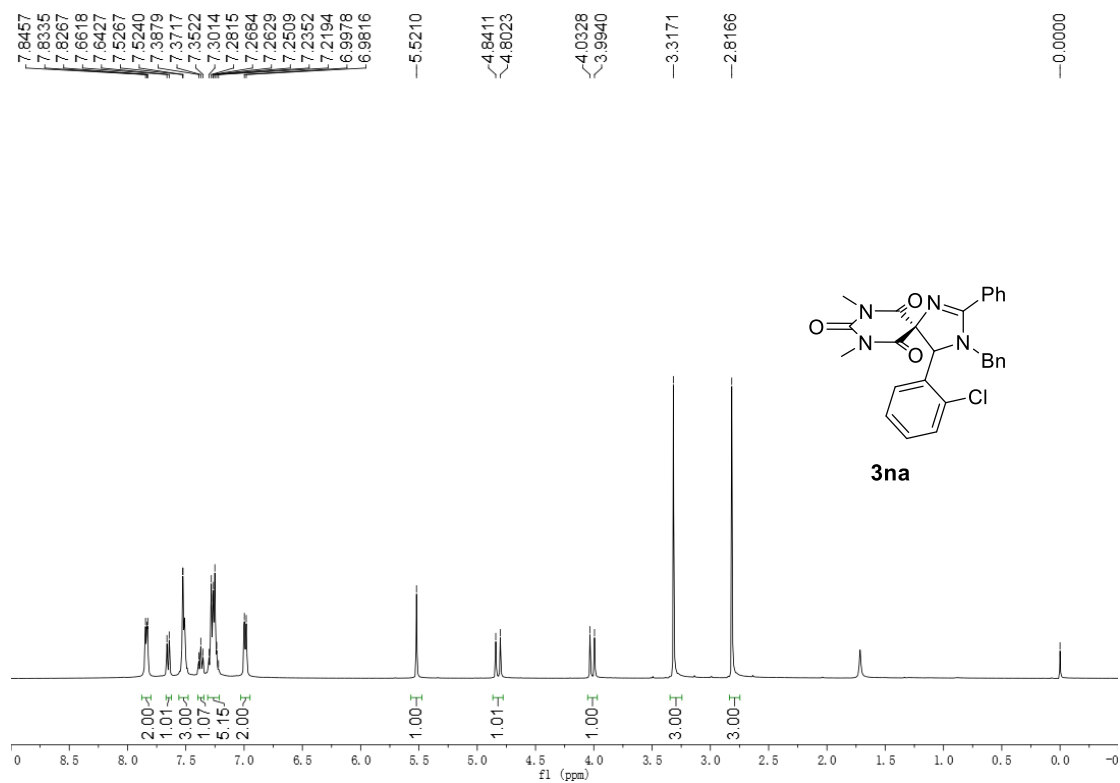


Figure S28. ^1H NMR (400 MHz, CDCl_3) of compound **3na**

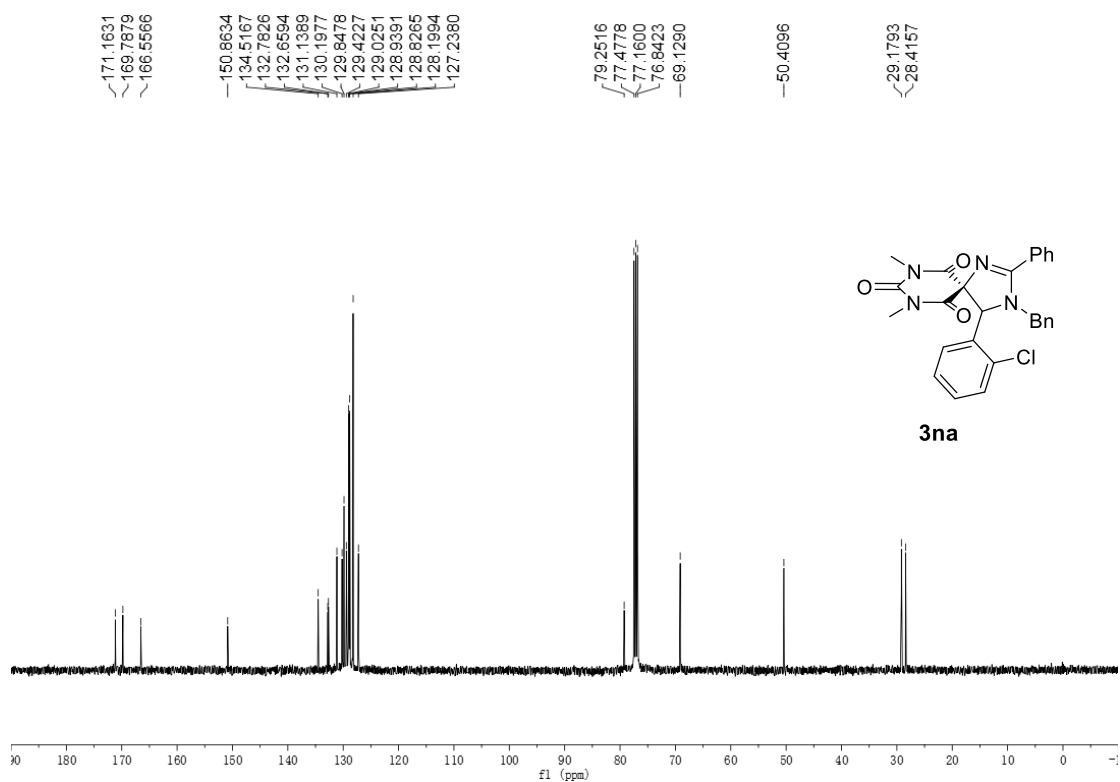


Figure S29. ^{13}C NMR (100 MHz, CDCl_3) of compound **3na**

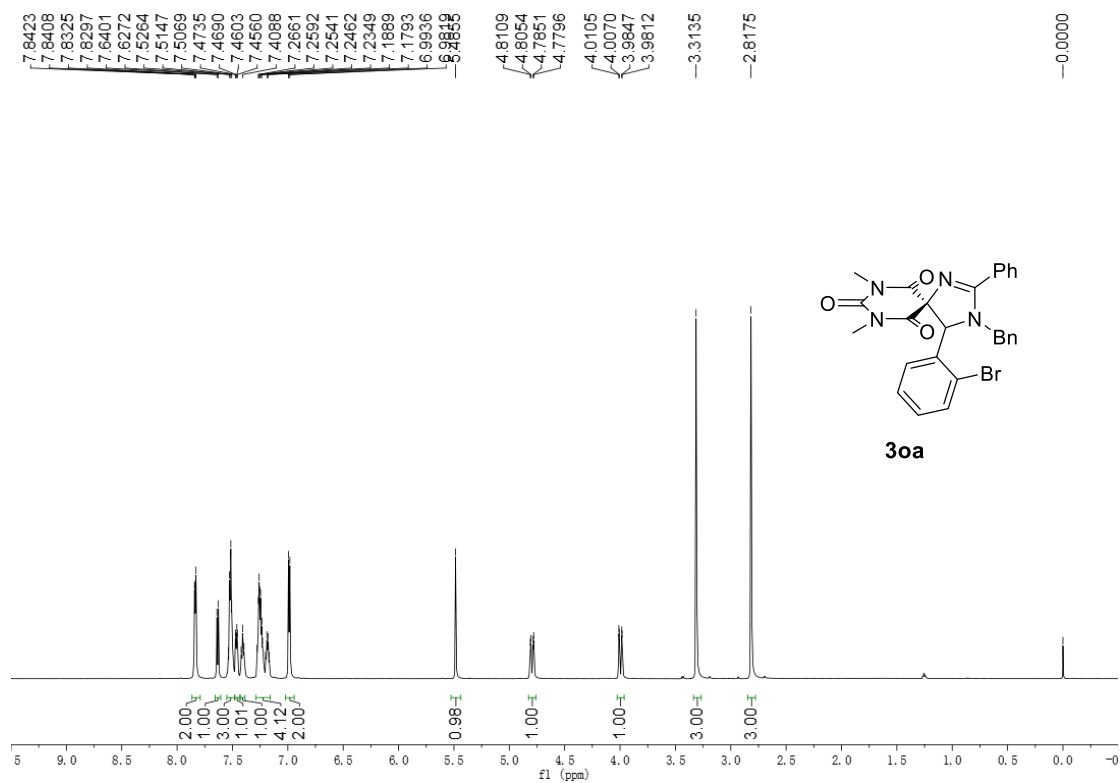


Figure S30. ¹H NMR (600 MHz, CDCl₃) of compound 3oa

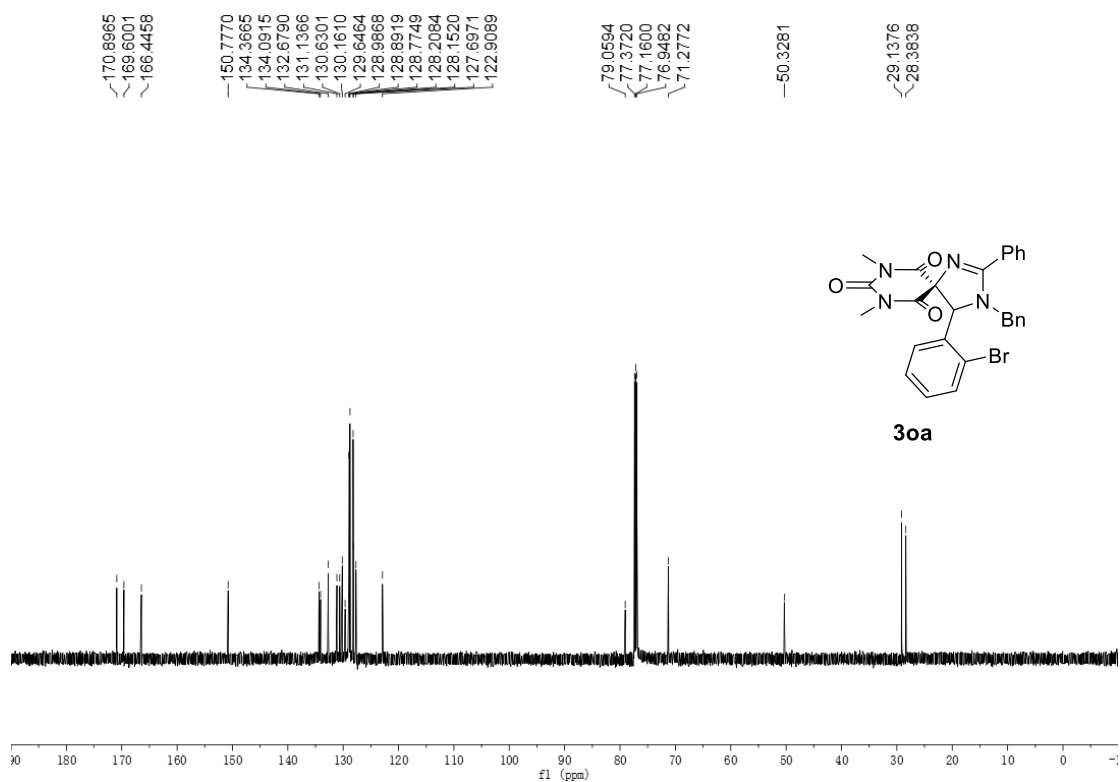


Figure S31. ¹³C NMR (150 MHz, CDCl₃) of compound 3oa

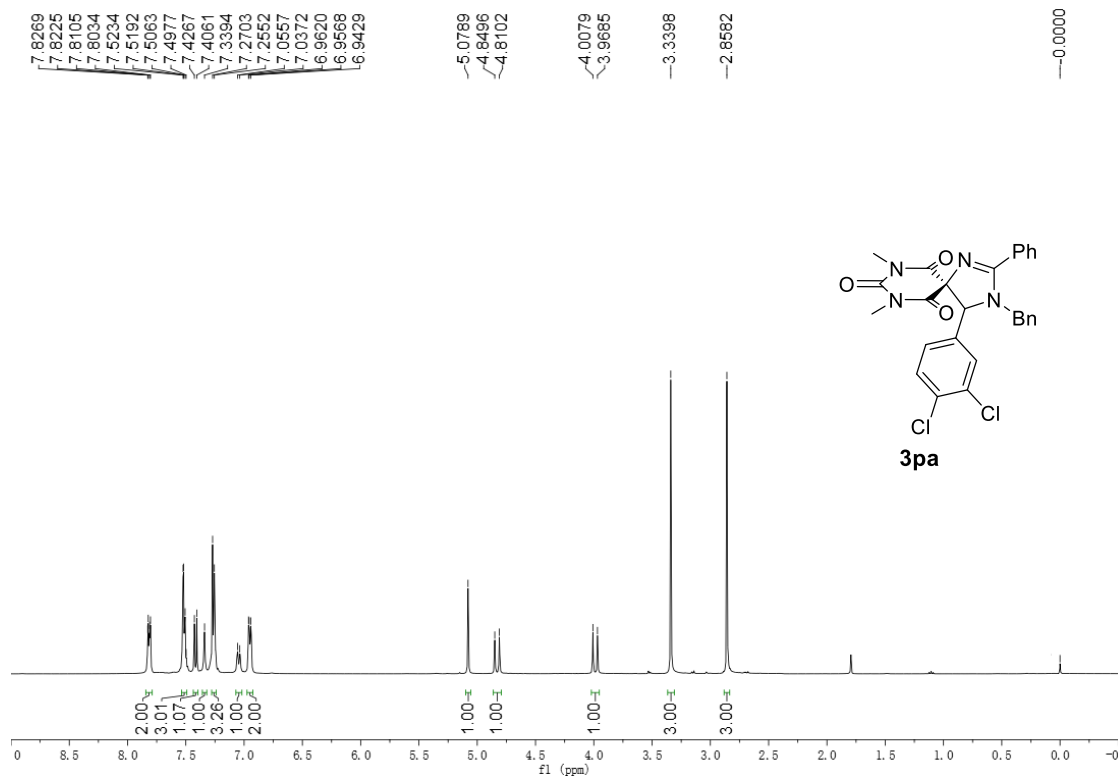


Figure S32. ¹H NMR (400 MHz, CDCl₃) of compound **3pa**

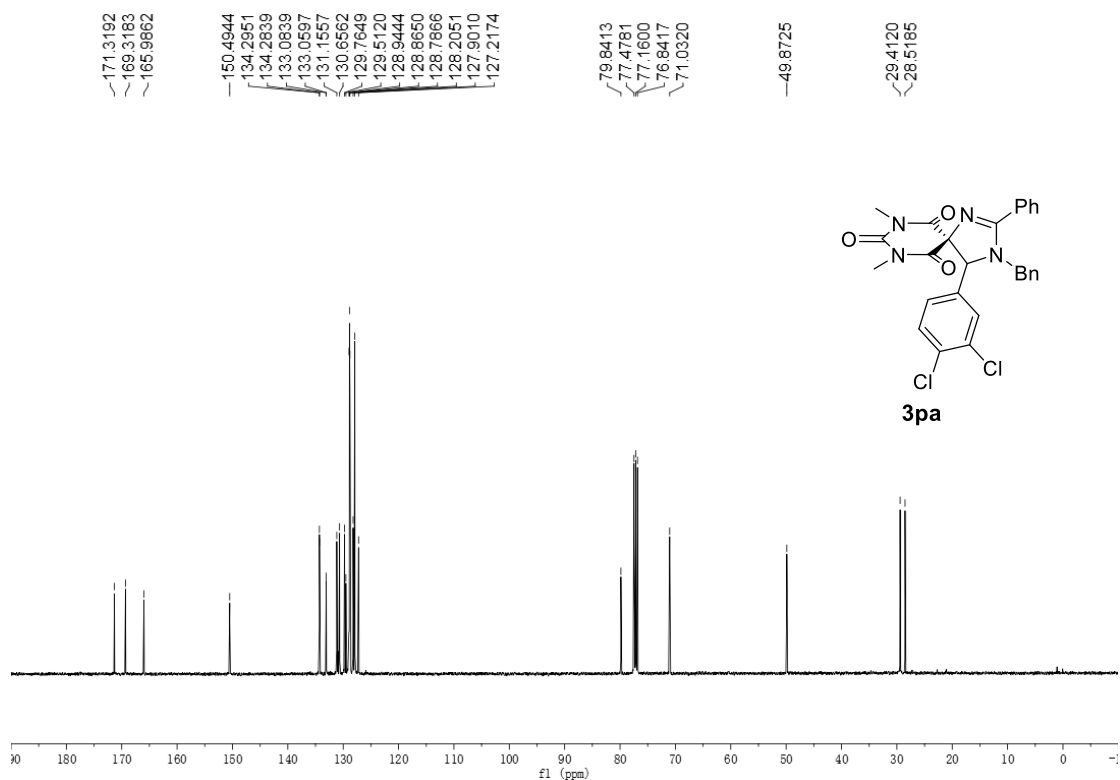
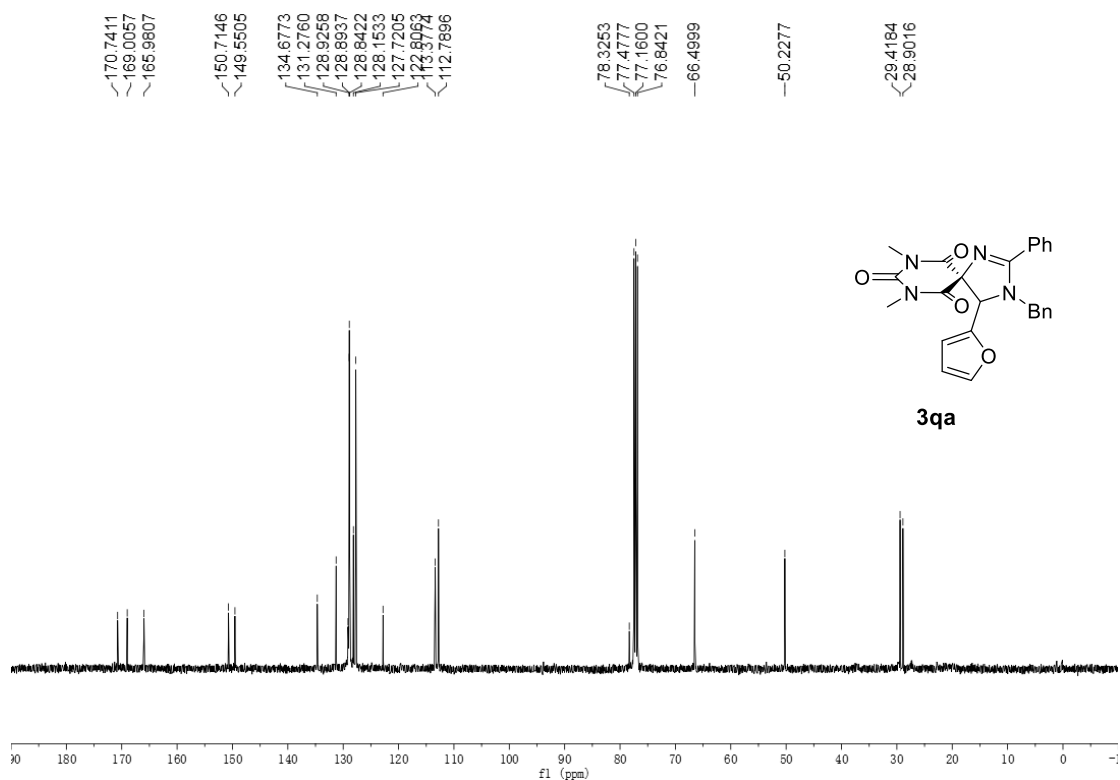
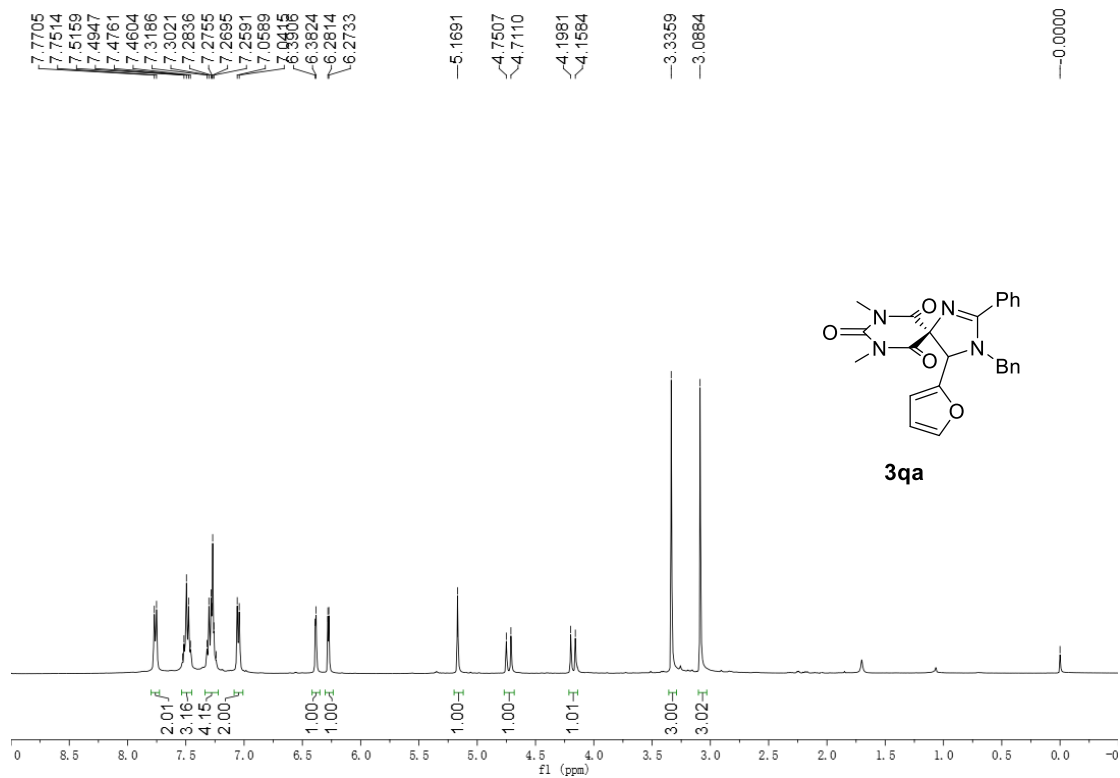


Figure S33. ¹³C NMR (100 MHz, CDCl₃) of compound **3pa**



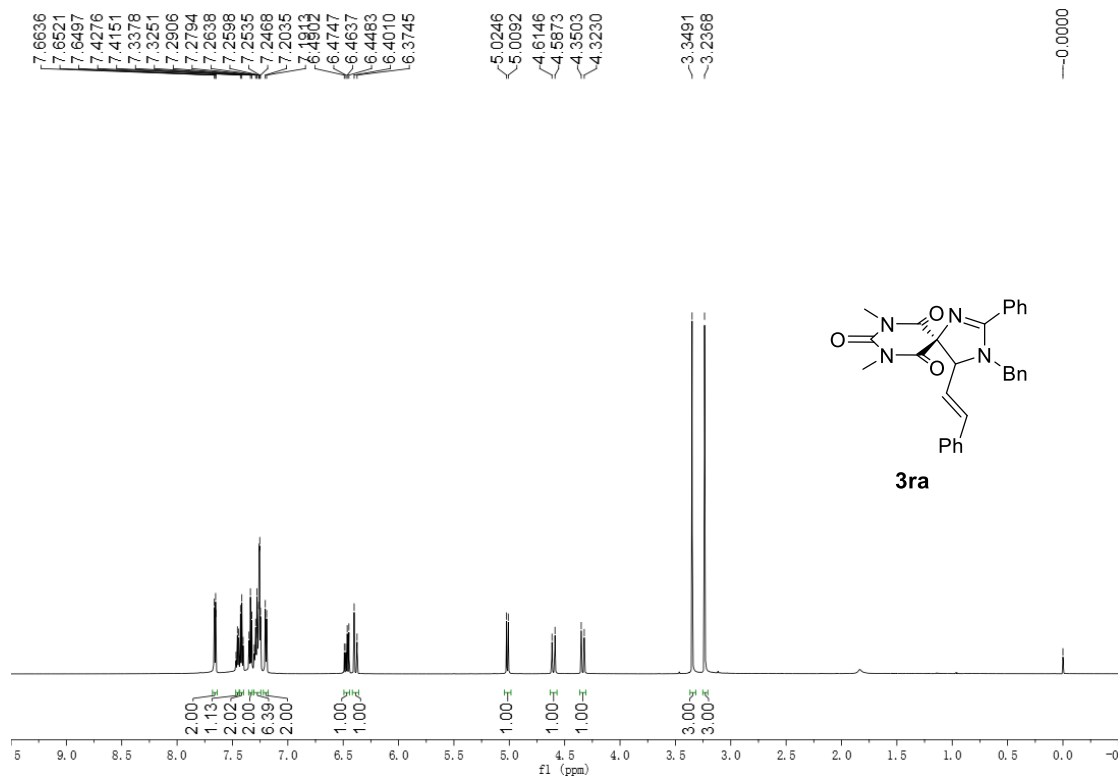


Figure S36. ¹H NMR (600 MHz, CDCl₃) of compound 3ra

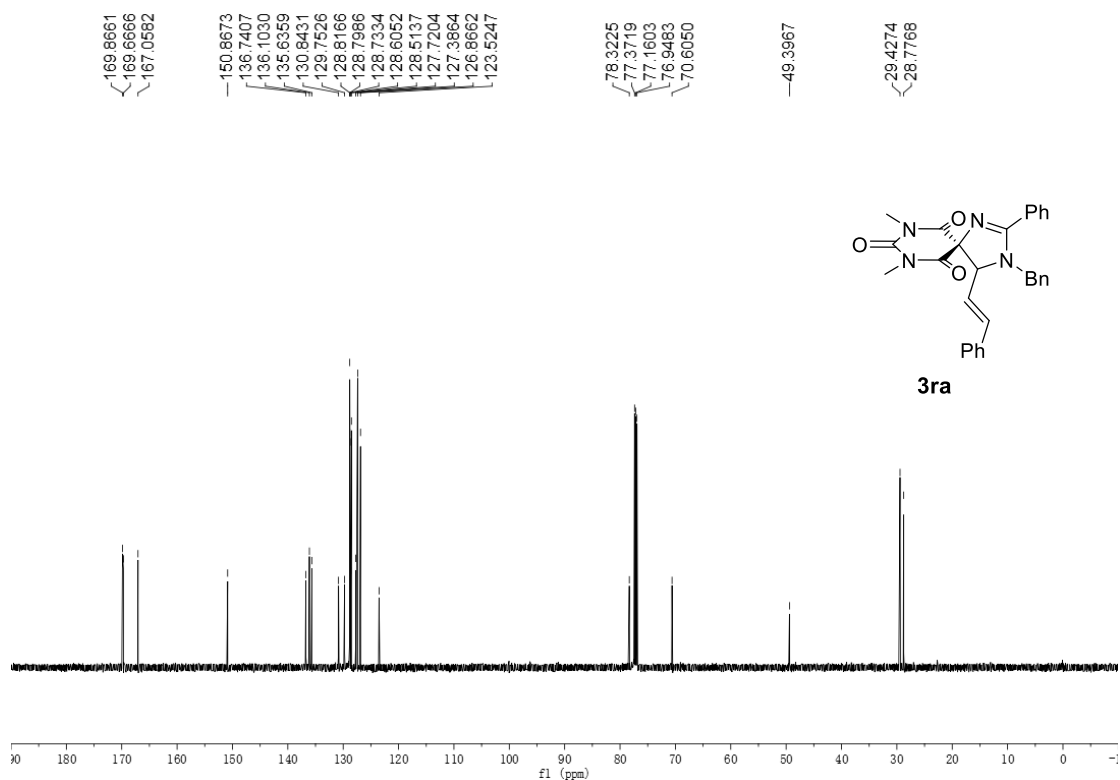


Figure S37. ¹³C NMR (150 MHz, CDCl₃) of compound 3ra

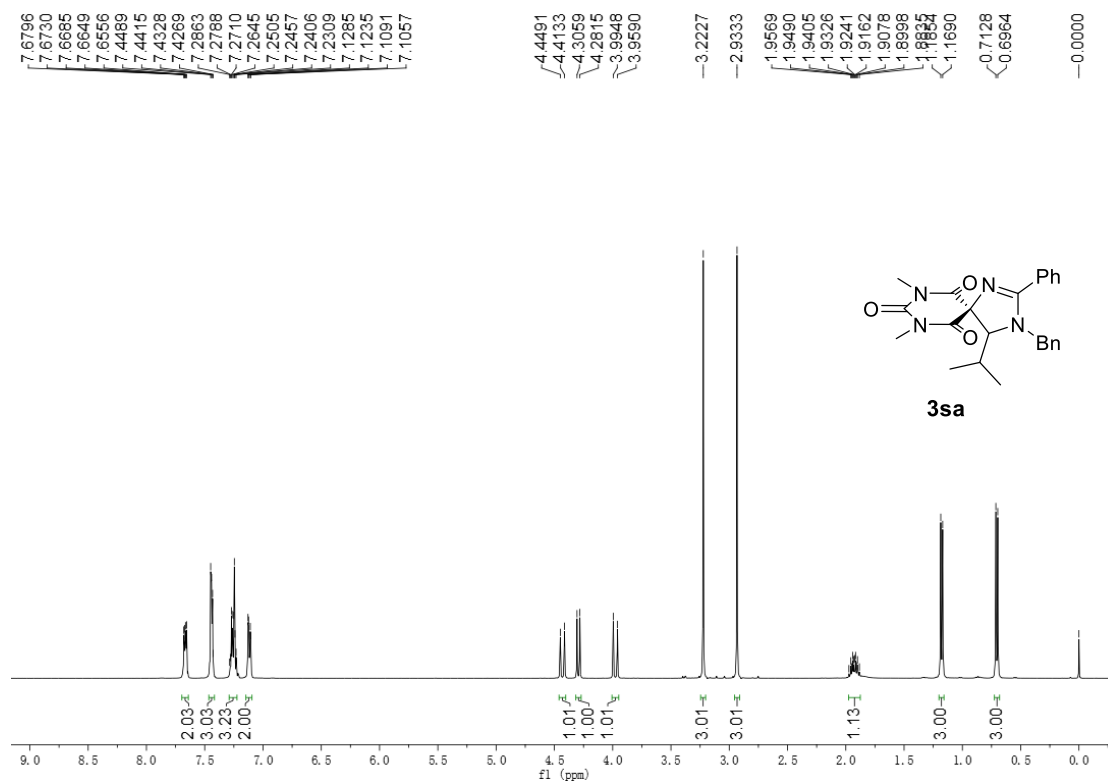


Figure S38. ¹H NMR (400 MHz, CDCl₃) of compound **3sa**

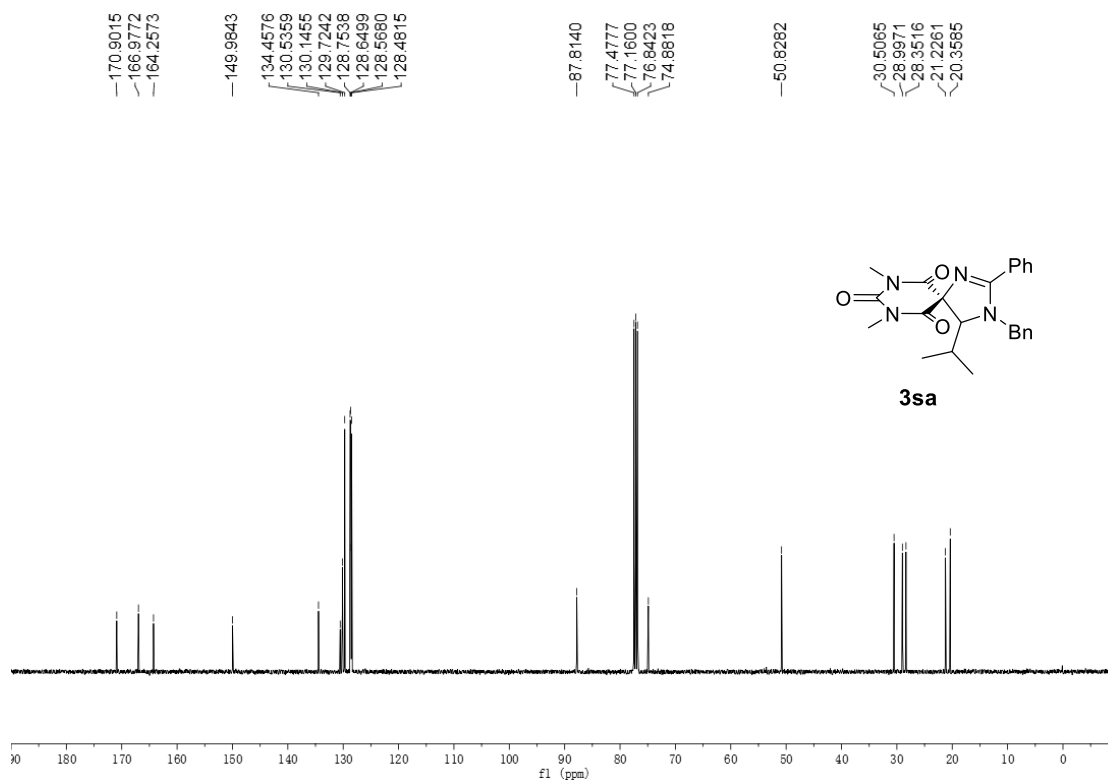


Figure S39. ¹³C NMR (100 MHz, CDCl₃) of compound **3sa**

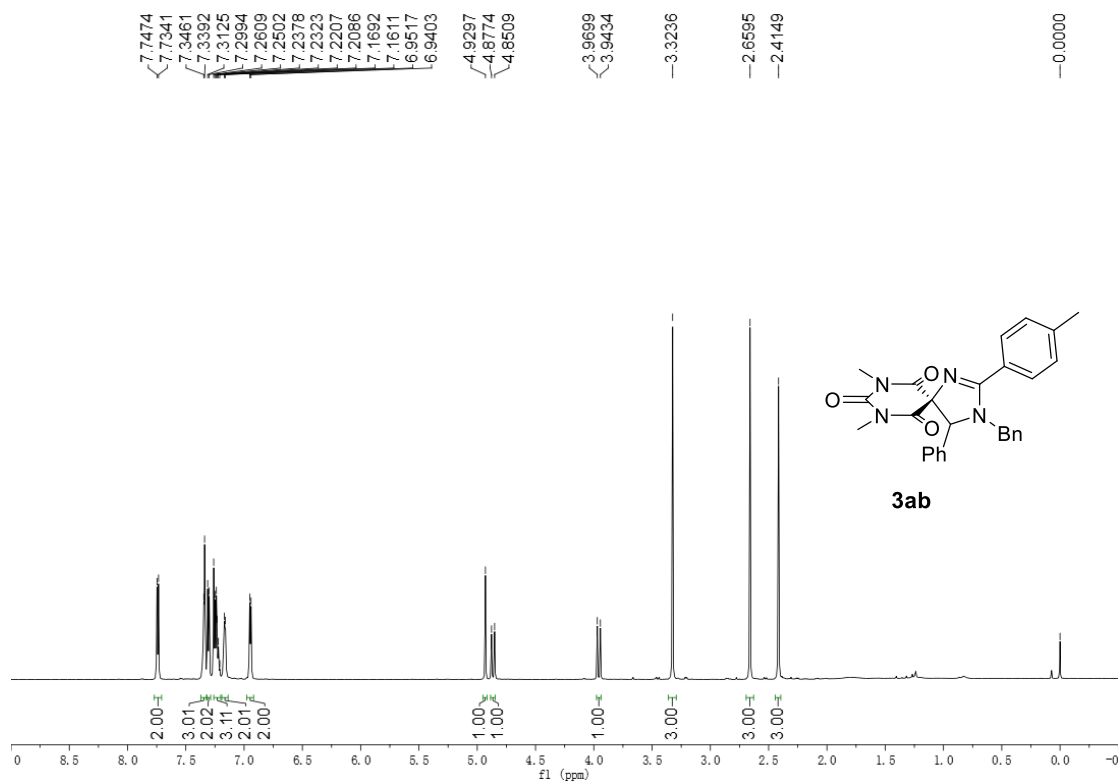


Figure S40. ¹H NMR (600 MHz, CDCl₃) of compound **3ab**

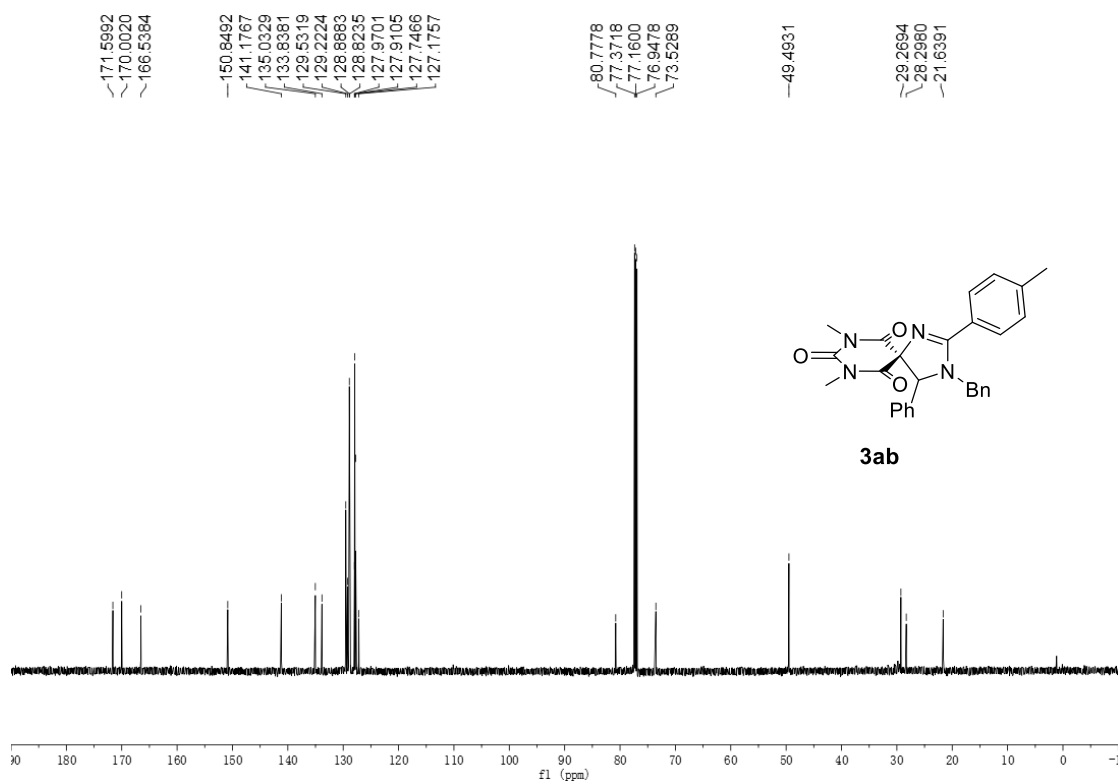


Figure S41. ¹³C NMR (150 MHz, CDCl₃) of compound **3ab**

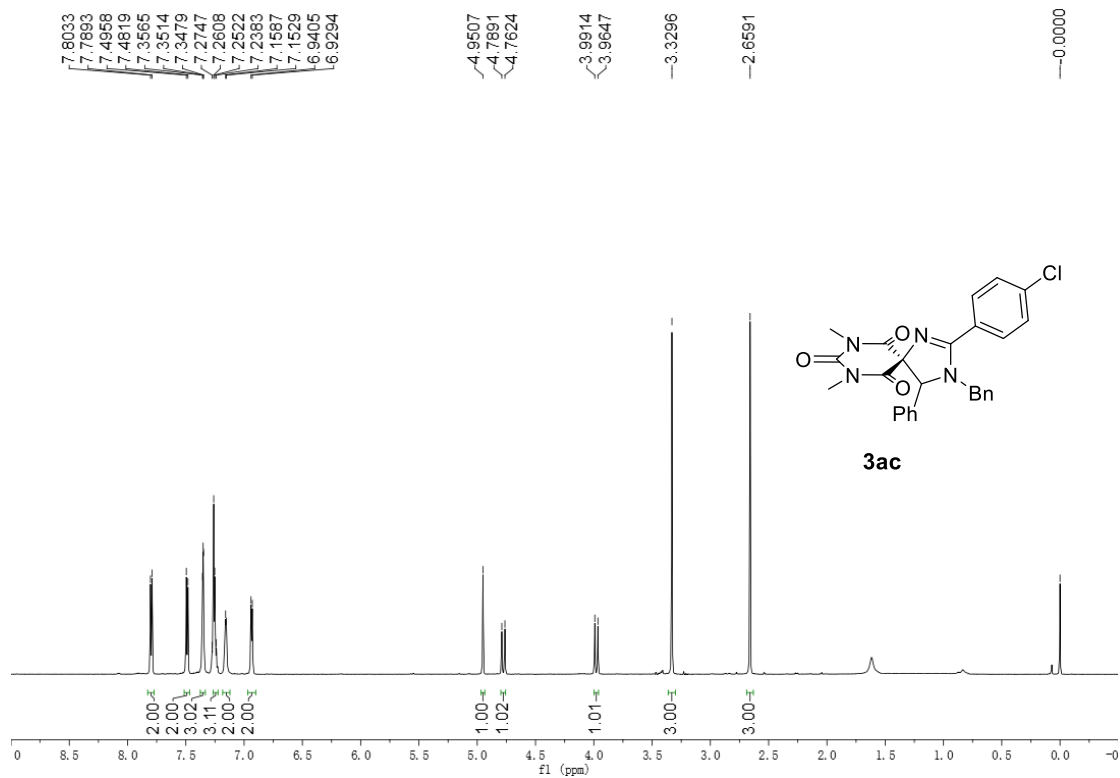


Figure S42. ¹H NMR (600 MHz, CDCl₃) of compound **3ac**

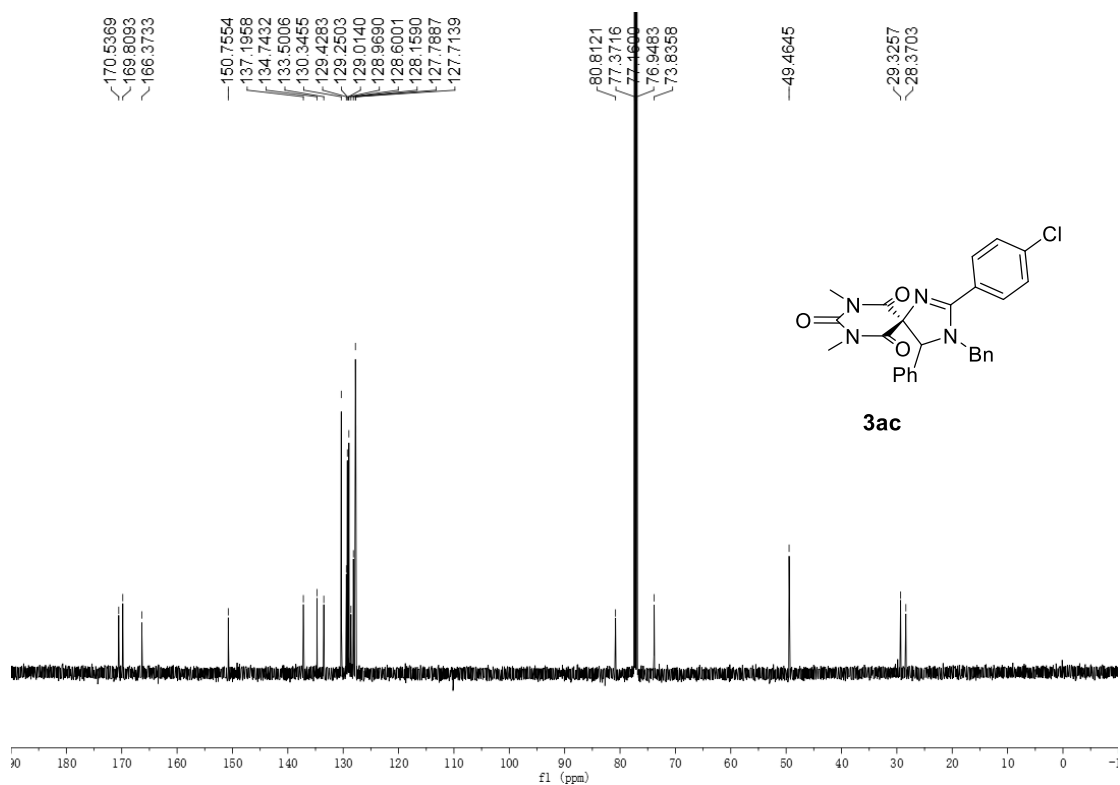


Figure S43. ¹³C NMR (150 MHz, CDCl₃) of compound **3ac**

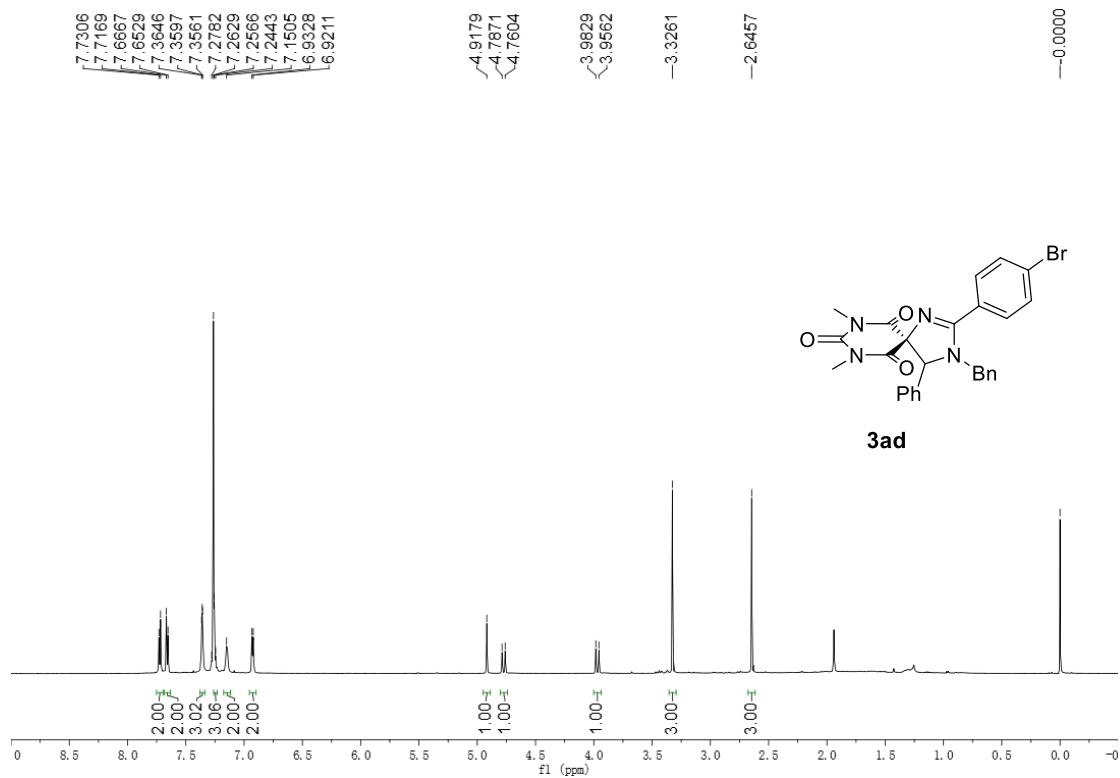


Figure S44. ¹H NMR (600 MHz, CDCl₃) of compound **3ad**

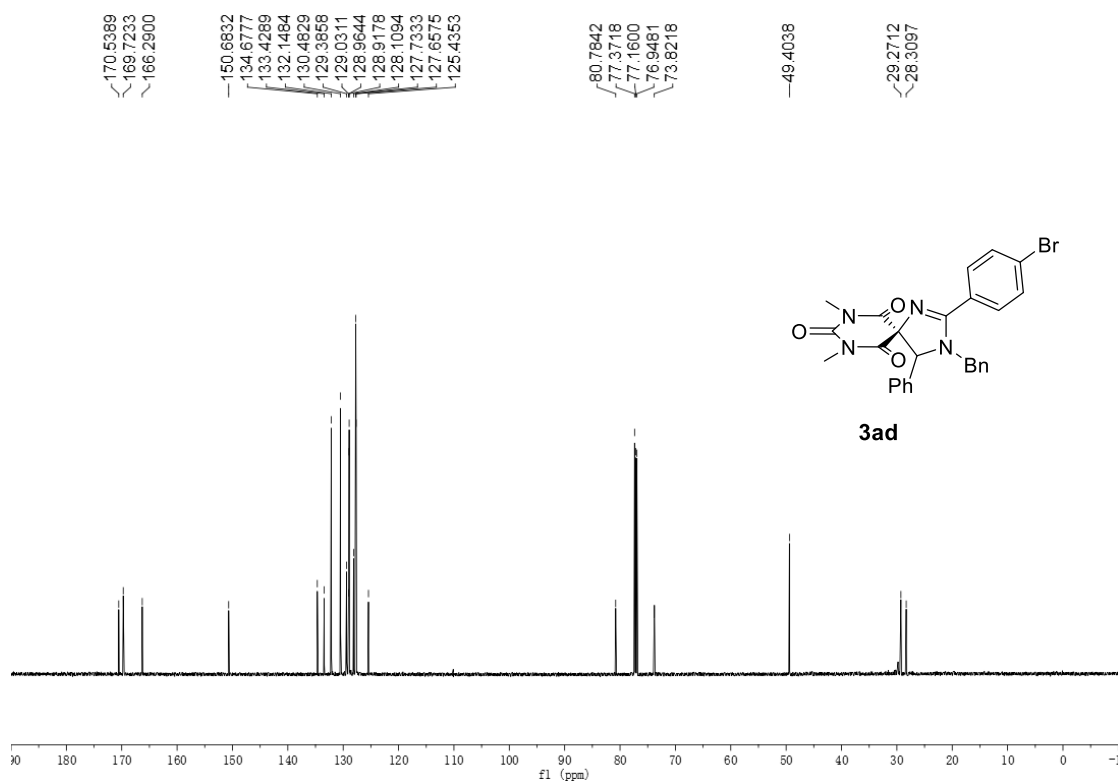


Figure S45. ¹³C NMR (150 MHz, CDCl₃) of compound **3ad**

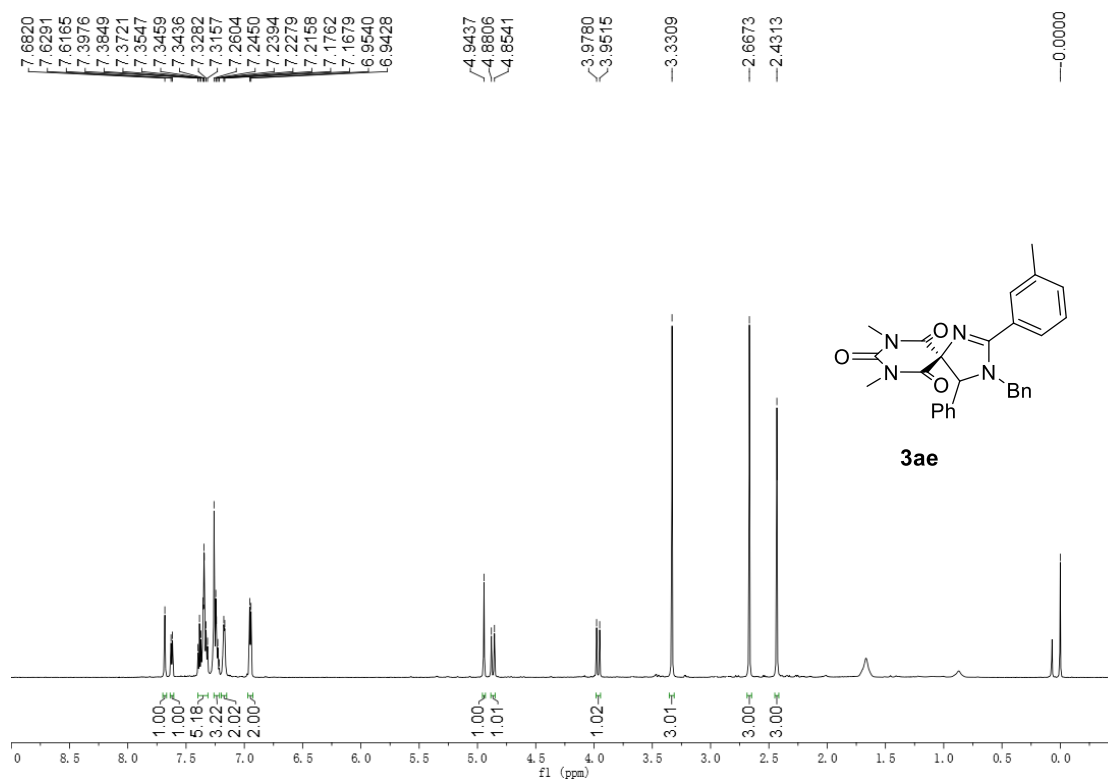


Figure S46. ¹H NMR (600 MHz, CDCl₃) of compound **3ae**

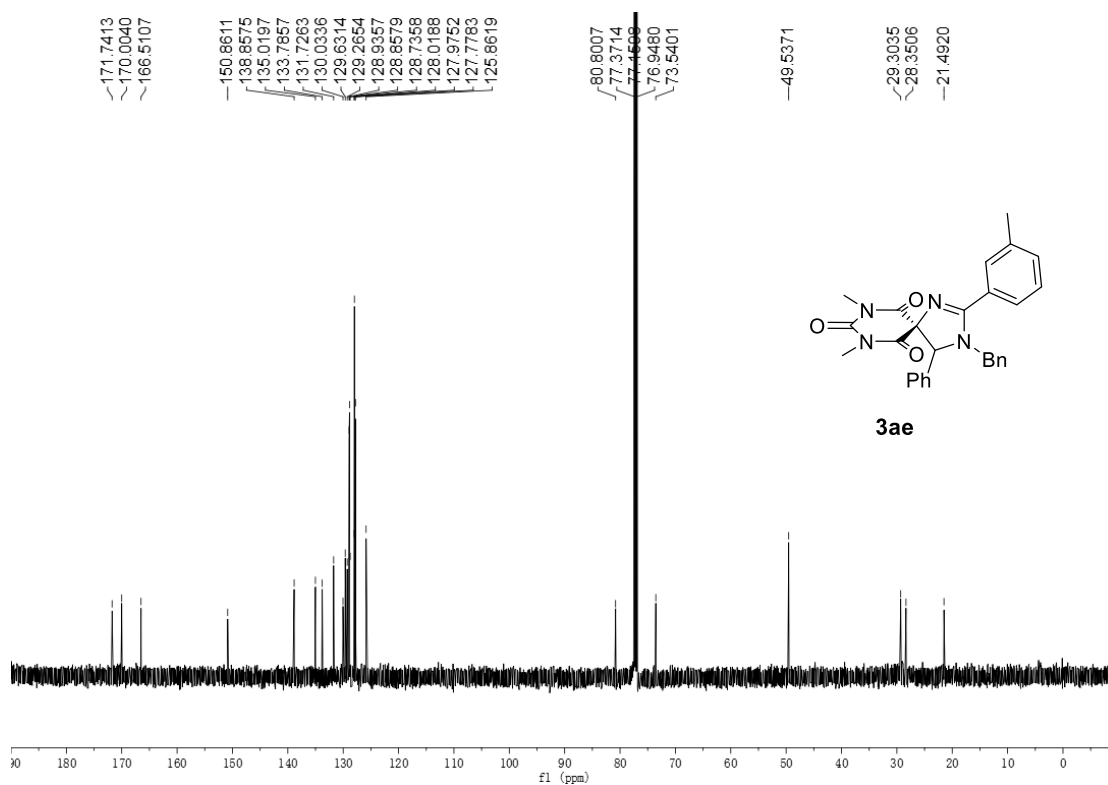


Figure S47. ¹³C NMR (150 MHz, CDCl₃) of compound **3ae**

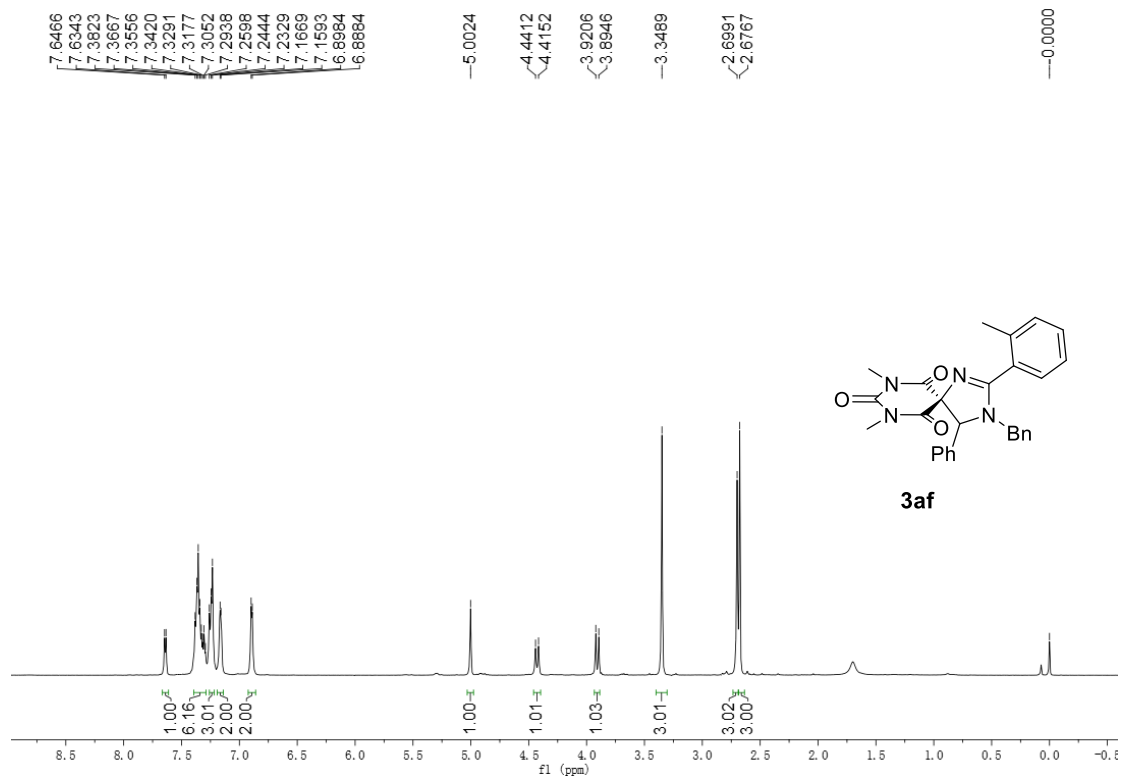


Figure S48. ¹H NMR (600 MHz, CDCl₃) of compound **3af**

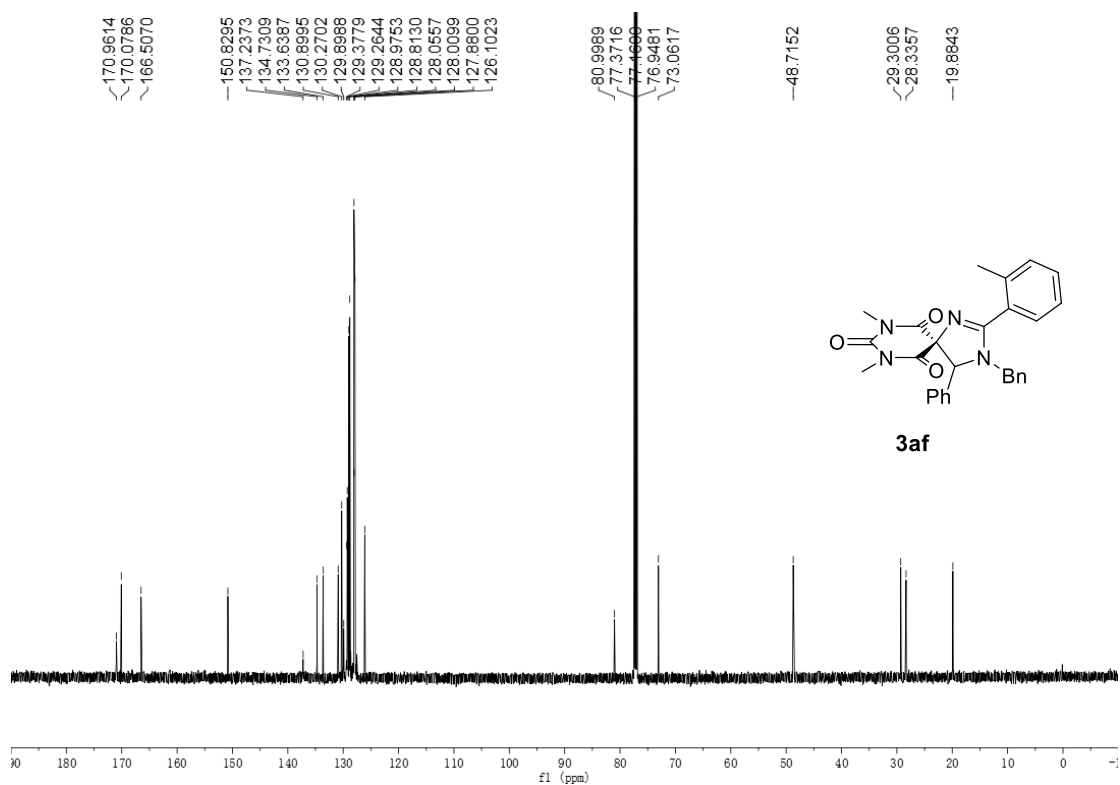


Figure S49. ¹³C NMR (150 MHz, CDCl₃) of compound **3af**

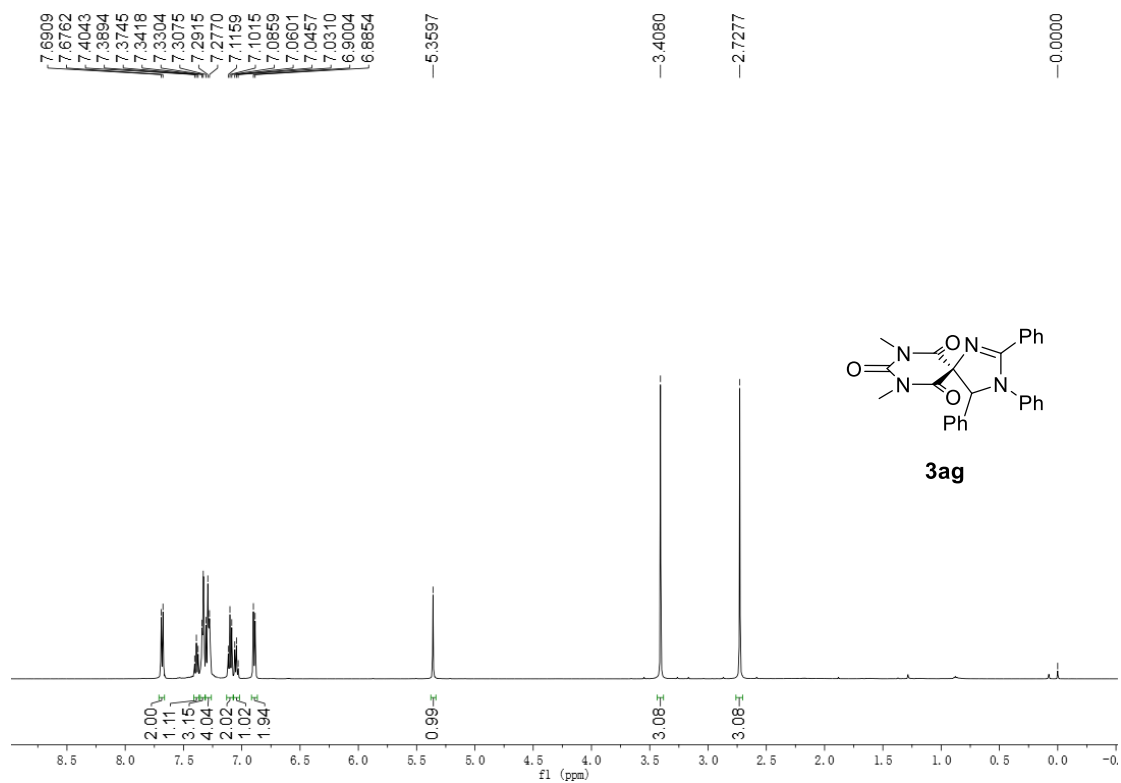


Figure S50. ^1H NMR (500 MHz, CDCl_3) of compound **3ag**

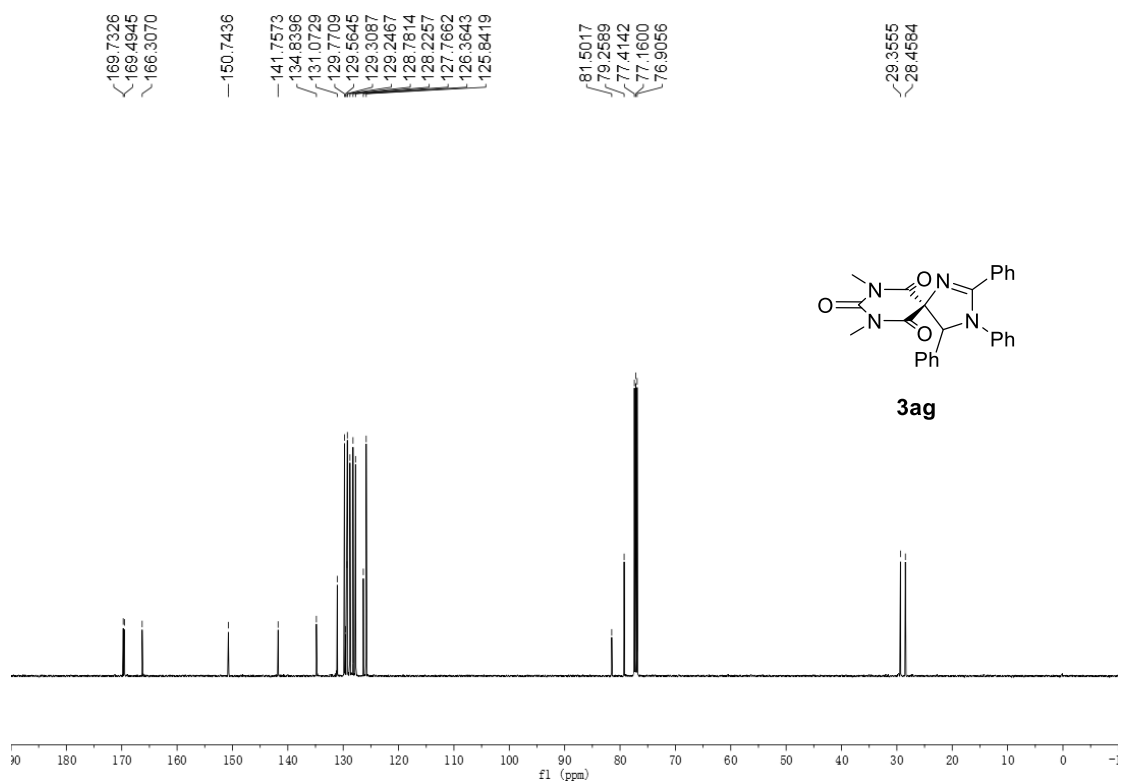


Figure S51. ^{13}C NMR (125 MHz, CDCl_3) of compound **3ag**

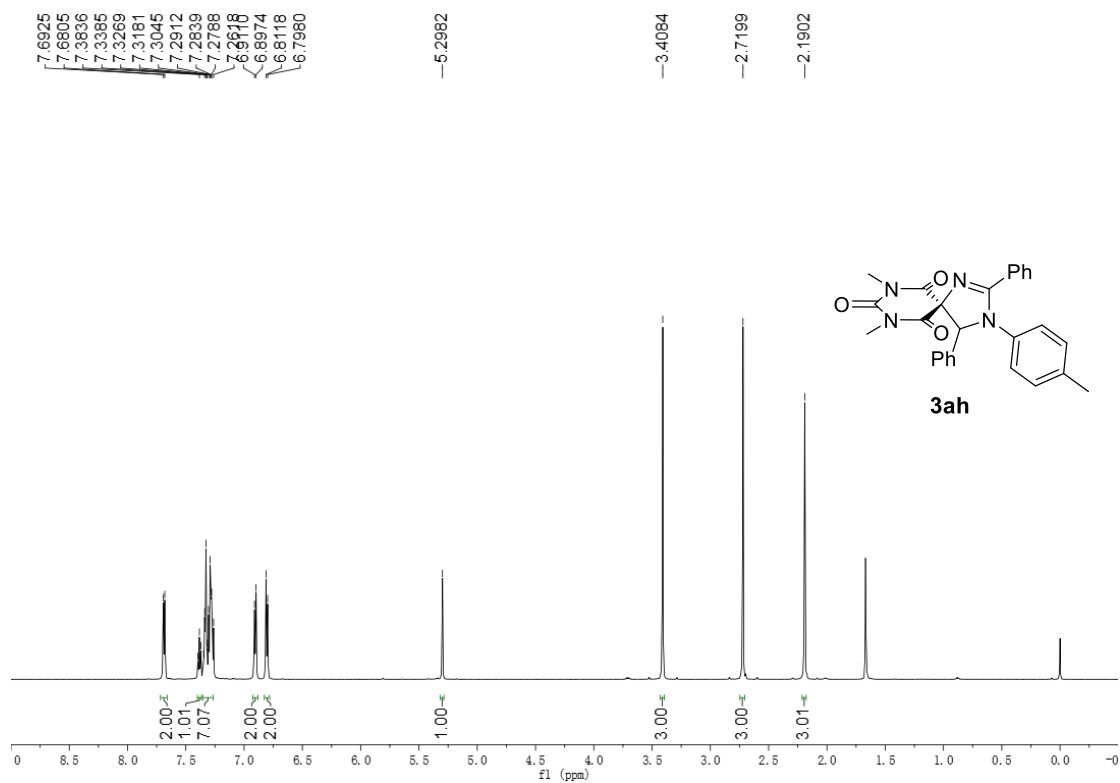


Figure S52. ¹H NMR (600 MHz, CDCl₃) of compound **3ah**

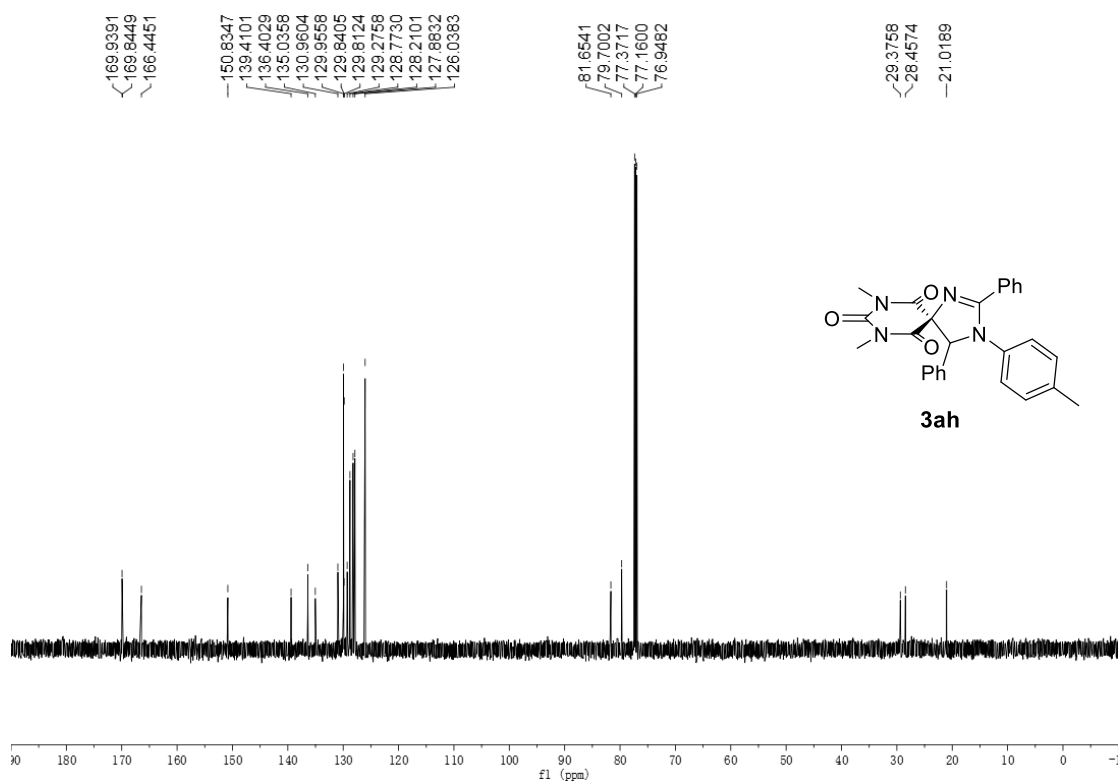
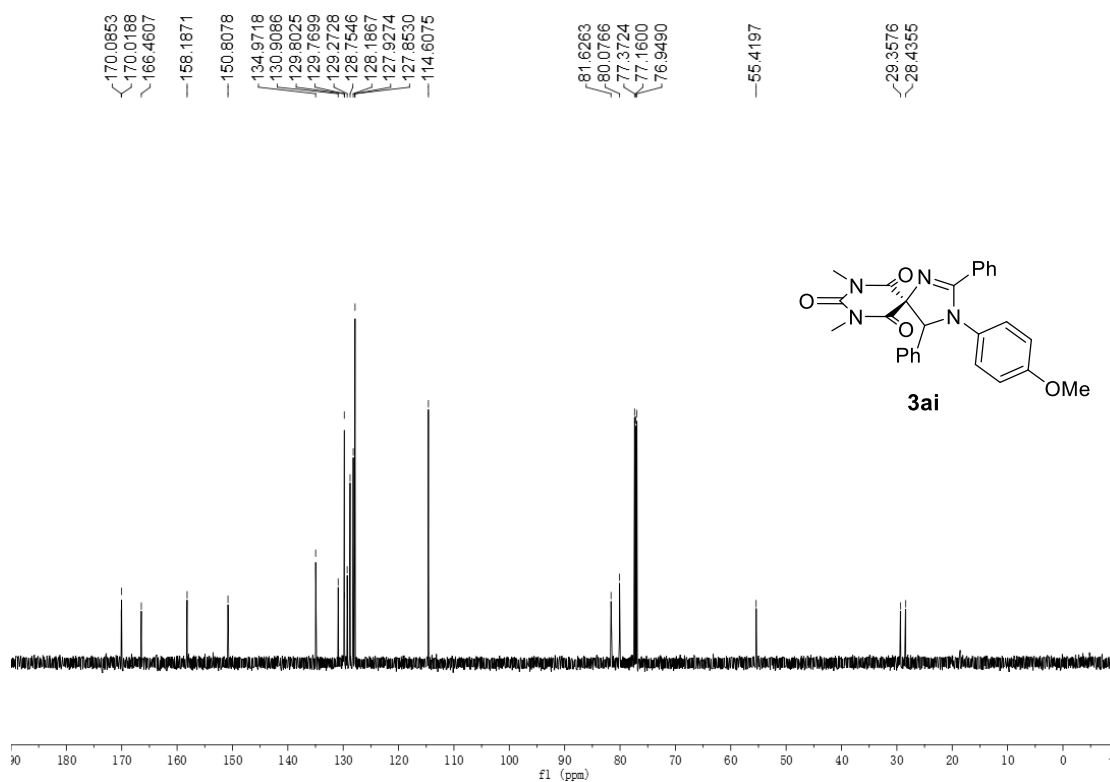
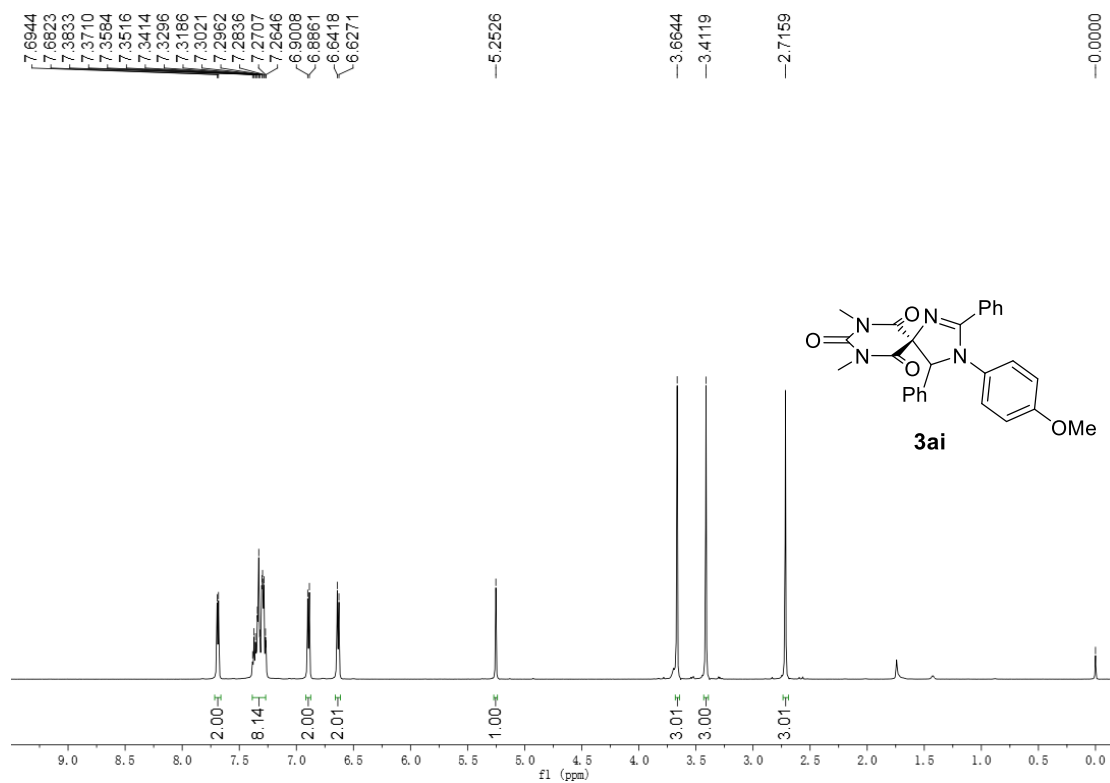


Figure S53. ¹³C NMR (150 MHz, CDCl₃) of compound **3ah**



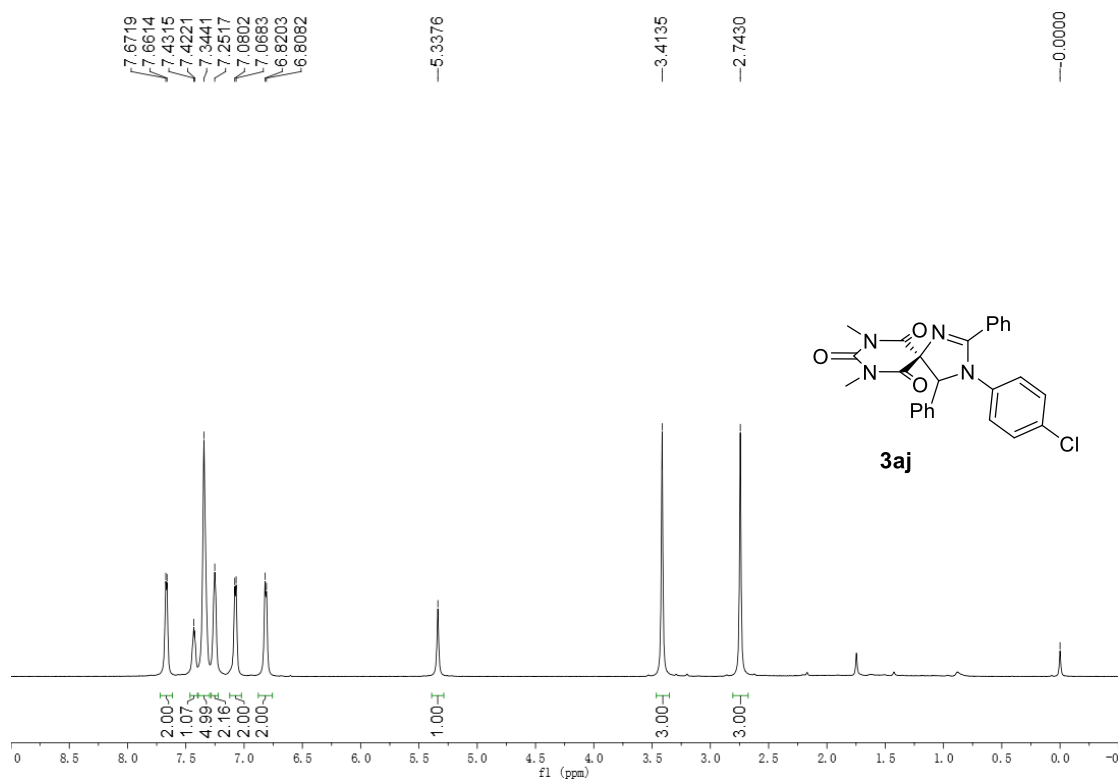


Figure S56. ¹H NMR (600 MHz, CDCl₃) of compound **3aj**

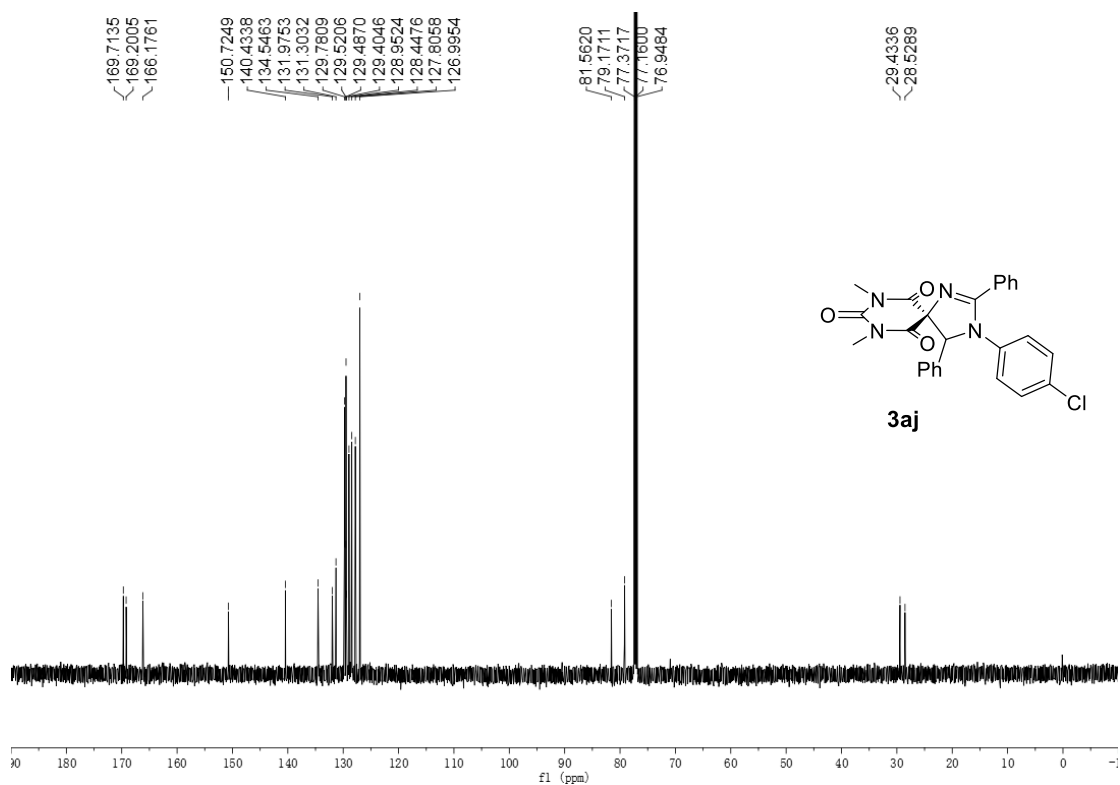


Figure S57. ¹³C NMR (150 MHz, CDCl₃) of compound **3aj**

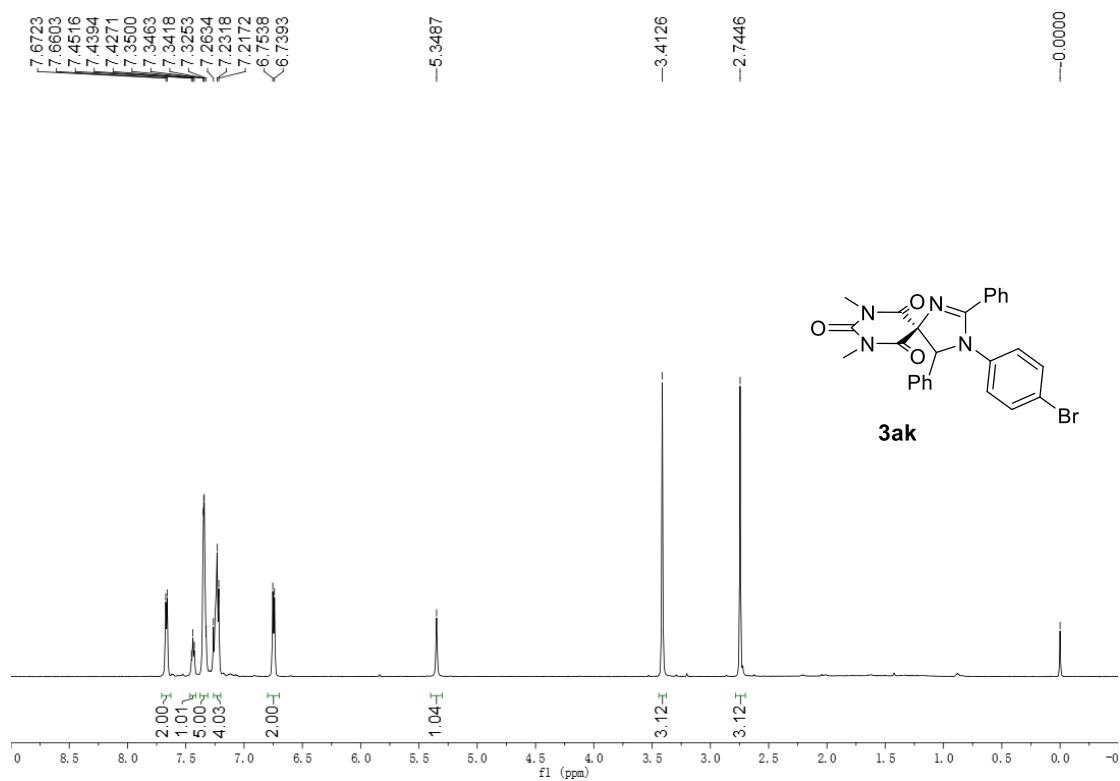


Figure S58. ¹H NMR (600 MHz, CDCl₃) of compound **3ak**

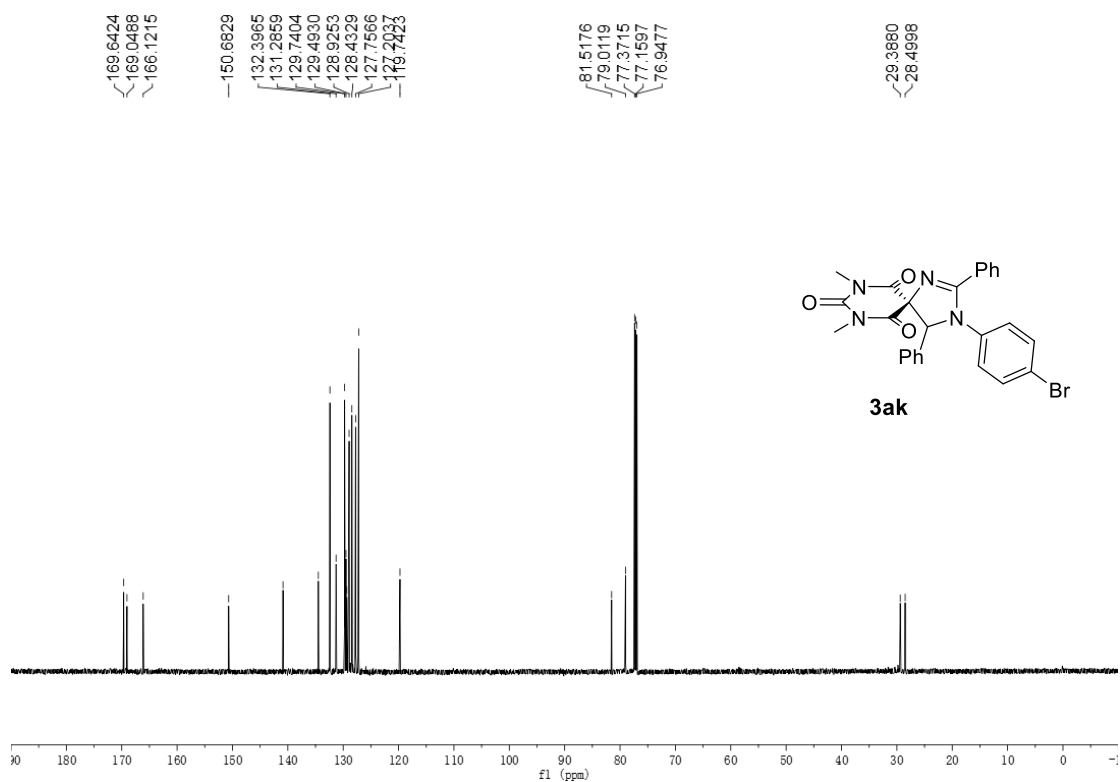


Figure S59. ¹³C NMR (150 MHz, CDCl₃) of compound **3ak**

2. Single-crystal X-ray crystallography of 30a

Single crystal of **30a** were obtained by slow evaporation from a mixture of acetone/*n*-hexane at 5 °C. Single-crystal X-ray diffraction data were collected on a diffractometer (Bruker APEX-II) equipped with a CCD area detector using graphite-monochromated MoK α radiation ($\lambda = 0.71073 \text{ \AA}$) in the scan range $4.538 < 2\theta < 54.968^\circ$. The structure was solved with the SHELXT structure solution program using Intrinsic Phasing and refined with the SHELXL refinement package using Least Squares minimisation. Crystallographic data have been deposited in the Cambridge Crystallographic Data Centre as deposition number CCDC 1982766.

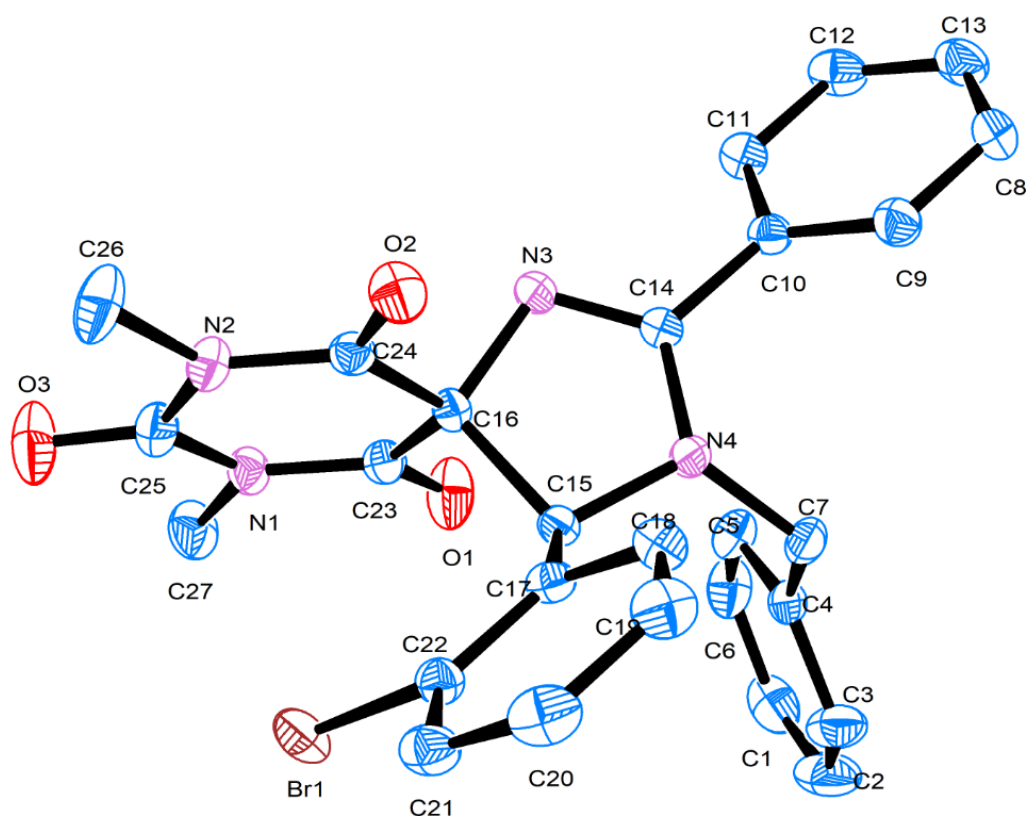


Figure S60. ORTEP Diagrams of 30a with 30% thermal ellipsoids

Table S1. Crystal data and structure refinement for 3oa

Identification code	CCDC 1982766
Empirical formula	C ₂₇ H ₂₃ BrN ₄ O ₃
Formula weight	531.40
Temperature/K	173
Crystal system	orthorhombic
Space group	Pbca
a/Å	8.3195(3)
b/Å	18.8155(8)
c/Å	30.0395(15)
α/°	90
β/°	90
γ/°	90
Volume/Å ³	4702.2(4)
Z	8
ρ _{calc} /cm ³	1.501
μ/mm ⁻¹	1.787
F(000)	2176.0
Crystal size/mm ³	0.15 × 0.12 × 0.1
Radiation	MoKα (λ = 0.71073)
2θ range for data collection/°	4.538 to 54.968
Index ranges	-9 ≤ h ≤ 10, -24 ≤ k ≤ 21, -38 ≤ l ≤ 26
Reflections collected	30027
Independent reflections	5385 [R _{int} = 0.0491, R _{sigma} = 0.0363]
Data/restraints/parameters	5385/0/318
Goodness-of-fit on F ²	1.036
Final R indexes [I ≥ 2σ (I)]	R ₁ = 0.0324, wR ₂ = 0.0745
Final R indexes [all data]	R ₁ = 0.0436, wR ₂ = 0.0796
Largest diff. peak/hole / e Å ⁻³	0.43/-0.52