

Supporting Information

Multicomponent domino cyclization of ethyl trifluoropyruvate with methyl ketones and amino alcohols – a new way to γ -lactam annulated oxazacycles

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Table S1. Crystallographic parameters and X-ray experiment details for **4d^c**, **5^{tc}**.

Compound	4d^c	5^{tc}
Empirical formula	C ₁₃ H ₁₂ F ₃ NO ₃	C ₁₃ H ₁₅ F ₆ NO ₆
Formula weight	287.24	395.26
Temperature/K	295(2)	295(2)
Crystal system	triclinic	monoclinic
Space group	P-1	P2 ₁ /n
a/Å	6.0290(7)	10.8844(8)
b/Å	8.1542(10)	13.1919(9)
c/Å	13.4778(17)	11.3734(9)
α/°	79.481(10)	90
β/°	86.443(10)	92.641(7)
γ/°	75.029(10)	90
Volume/Å ³	629.27(14)	1631.3(2)
Z	2	4
ρ _{calc} /cm ³	1.516	1.609
μ/mm ⁻¹	0.136	0.167
F(000)	296.0	808.0
Crystal size/mm ³	0.41 × 0.29 × 0.17	0.45 × 0.29 × 0.13
Radiation	MoKα (λ = 0.71073)	MoKα (λ = 0.71073)
2Θ range for data collection/°	7.366 to 61.83	7.144 to 62.028
Index ranges	-8 ≤ h ≤ 8, -11 ≤ k ≤ 10, -15 ≤ l ≤ 18	-15 ≤ h ≤ 13, -17 ≤ k ≤ 18, -16 ≤ l ≤ 15
Reflections collected	5481	11283
Independent reflections	3339 [R _{int} = 0.0434, R _{sigma} = 0.0881]	4404 [R _{int} = 0.0496, R _{sigma} = 0.0604]
Data/restraints/parameters	3339/0/186	4404/0/245
Goodness-of-fit on F ²	1.015	1.003
Final R indexes [I ≥ 2σ (I)]	R ₁ = 0.0702, wR ₂ = 0.1634	R ₁ = 0.0544, wR ₂ = 0.1455
Final R indexes [all data]	R ₁ = 0.1428, wR ₂ = 0.2335	R ₁ = 0.1148, wR ₂ = 0.2039
Largest diff. peak/hole / e Å ⁻³	0.26/-0.24	0.22/-0.21

Table S2. Crystallographic parameters and X-ray experiment details for **8c^c**, **11b**.

Compound	8c^c	11b
Empirical formula	C ₁₂ H ₁₈ F ₃ NO ₃	C ₁₅ H ₁₉ NO ₆ F ₆
Formula weight	281.27	423.31
Temperature/K	295(2)	295(2)
Crystal system	monoclinic	monoclinic
Space group	P2 ₁ /c	P2 ₁ /c
a/Å	10.6332(12)	19.1431(10)
b/Å	13.8923(13)	6.4882(4)
c/Å	9.5664(15)	16.4430(9)
α/°	90	90
β/°	102.235(13)	107.551(6)
γ/°	90	90
Volume/Å ³	1381.0(3)	1947.2(2)
Z	4	4
ρ _{calc} /cm ³	1.353	1.444
μ/mm ⁻¹	0.122	0.145
F(000)	592.0	872.0
Crystal size/mm ³	0.44 × 0.36 × 0.28	0.22 × 0.08 × 0.01
Radiation	Mo Kα (λ = 0.71073)	MoKα (λ = 0.71073)
2Θ range for data collection/°	7.056 to 61.722	5 to 52.742
Index ranges	-14 ≤ h ≤ 14, -18 ≤ k ≤ 19, -12 ≤ l ≤ 11	-23 ≤ h ≤ 23, -8 ≤ k ≤ 8, -20 ≤ l ≤ 20
Reflections collected	10666	15118
Independent reflections	3779 [R _{int} = 0.0582, R _{sigma} = 0.0803]	3972 [R _{int} = 0.1526, R _{sigma} = 0.0998]
Data/restraints/parameters	3779/0/210	3972/12/270
Goodness-of-fit on F ²	0.980	1.015
Final R indexes [I ≥ 2σ (I)]	R ₁ = 0.0611, wR ₂ = 0.1539	R ₁ = 0.0601, wR ₂ = 0.1547
Final R indexes [all data]	R ₁ = 0.1610, wR ₂ = 0.2201	R ₁ = 0.2070, wR ₂ = 0.2534
Largest diff. peak/hole / e Å ⁻³	0.30/-0.22	0.27/-0.28

Copies of ^1H , ^{13}C and ^{19}F NMR spectra for all compounds

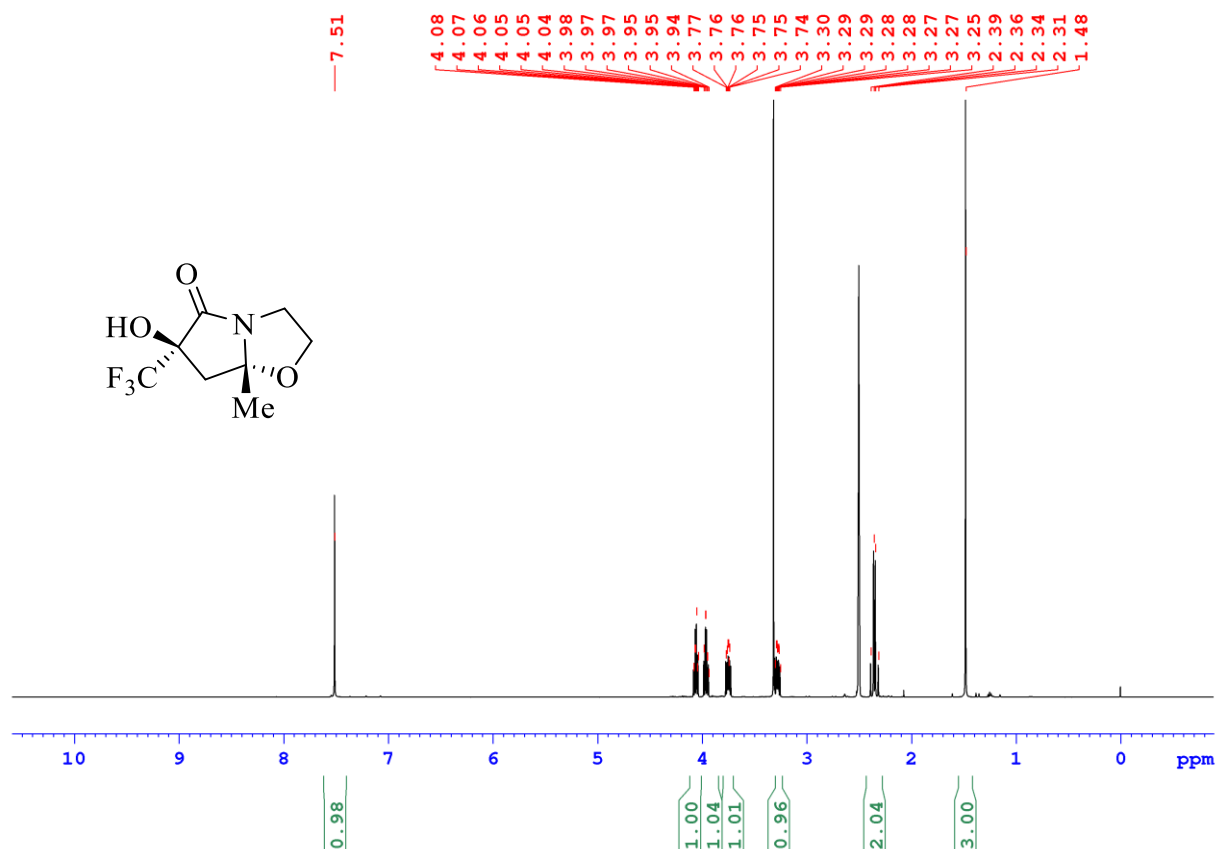


Figure S1. ^1H NMR spectrum of $4a^t$.

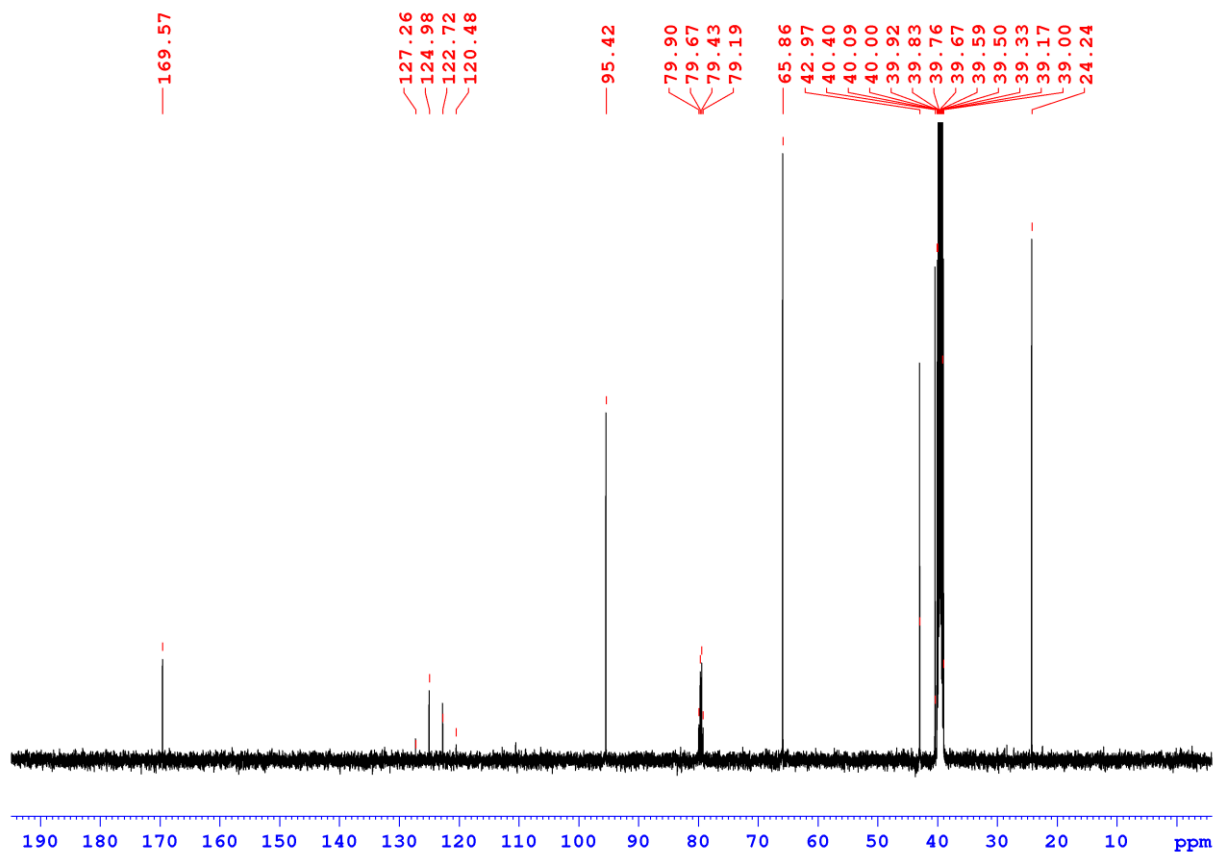


Figure S2. ^{13}C NMR spectrum of $4a^t$.



Figure S3. ¹⁹F NMR spectrum of **4a^t**.

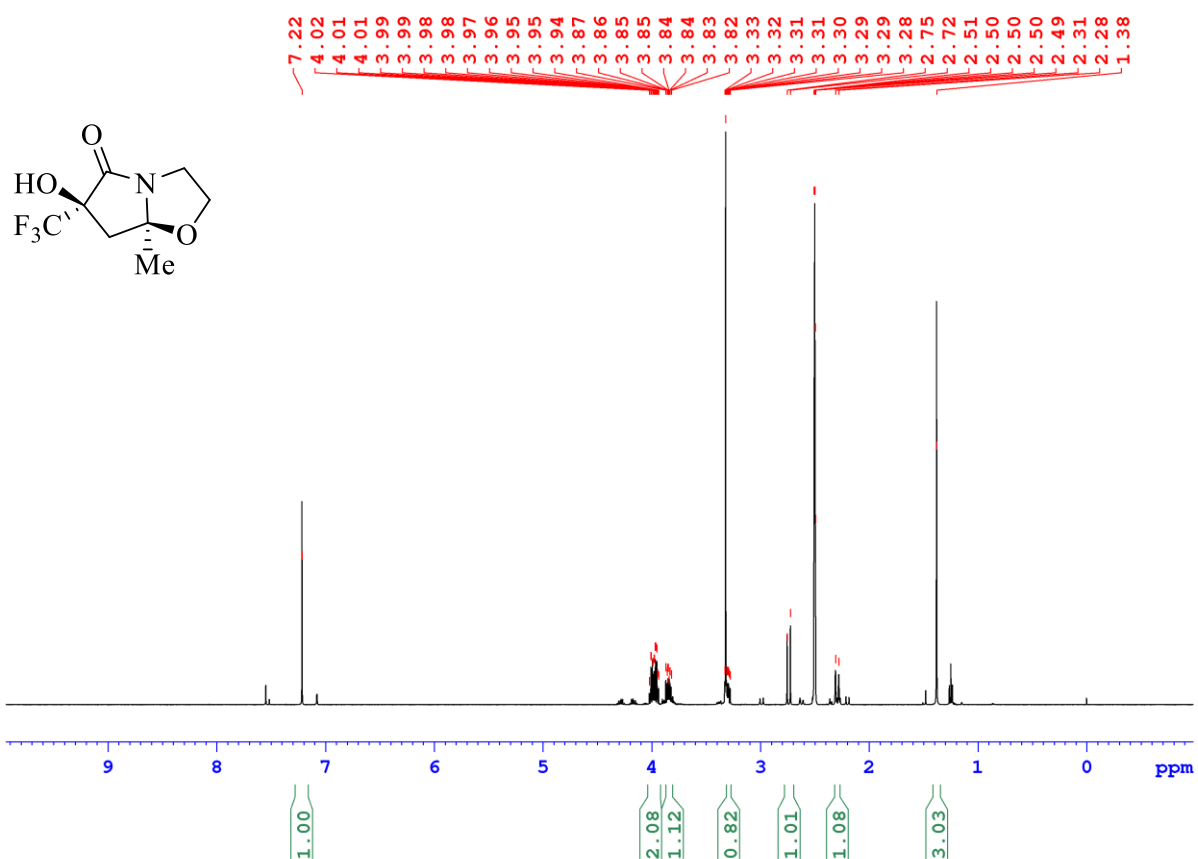


Figure S4. ¹H NMR spectrum of **4a^c** (mixed with **5^{ct}** (20%) and **4a^t** (2%)).

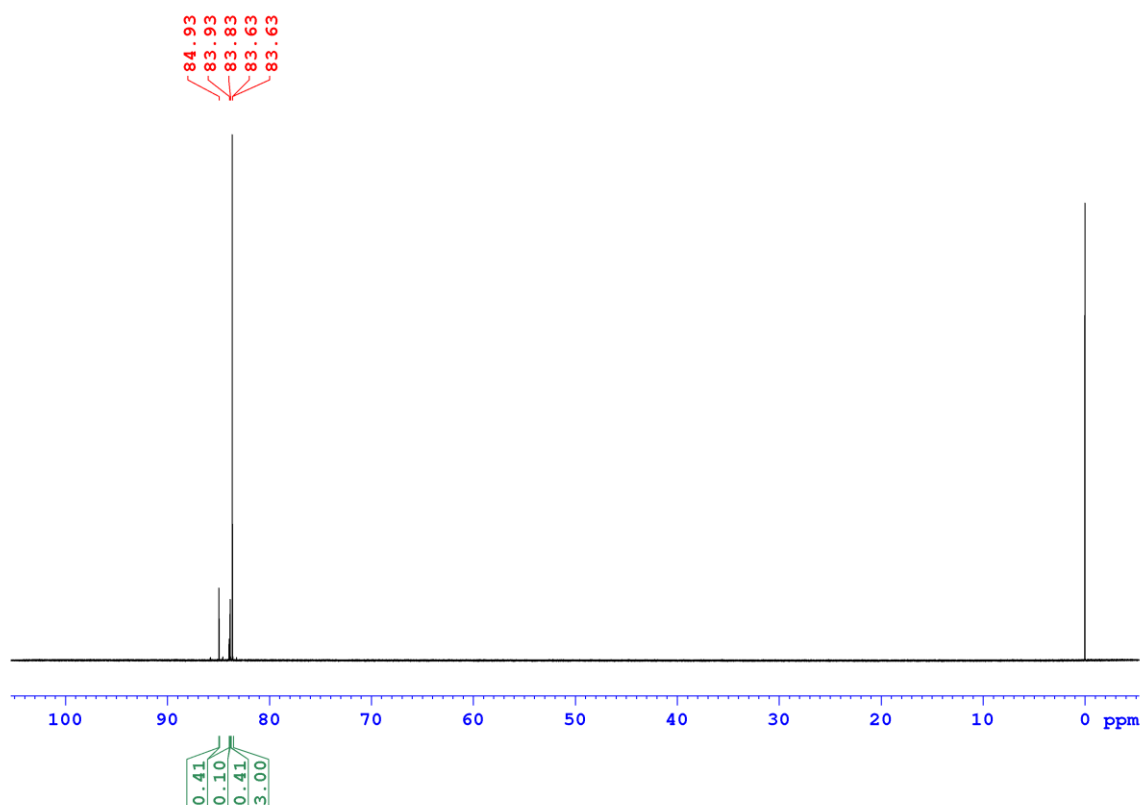


Figure S5. ¹⁹F NMR spectrum of **4a^c** (mixed with **5^{ct}** (20%) and **4a^t** (2%)).

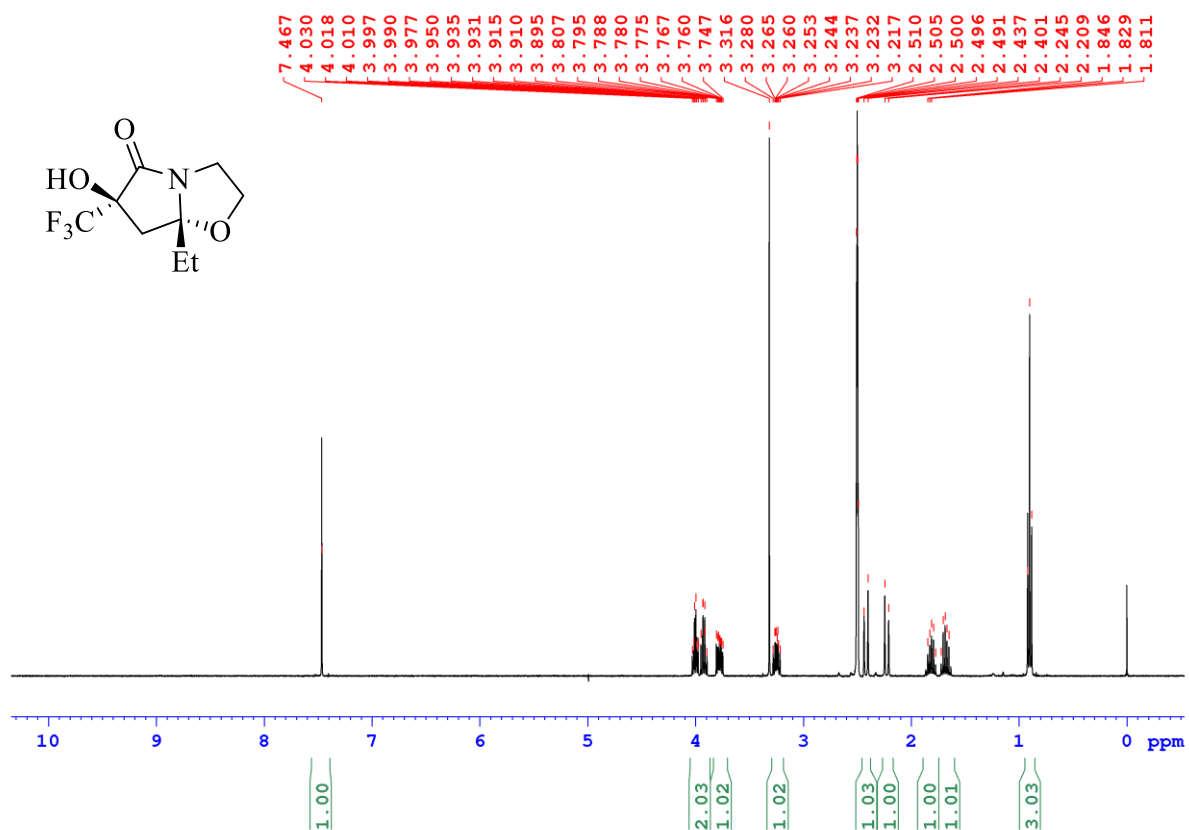


Figure S6. ¹H NMR spectrum of **4b^t**.

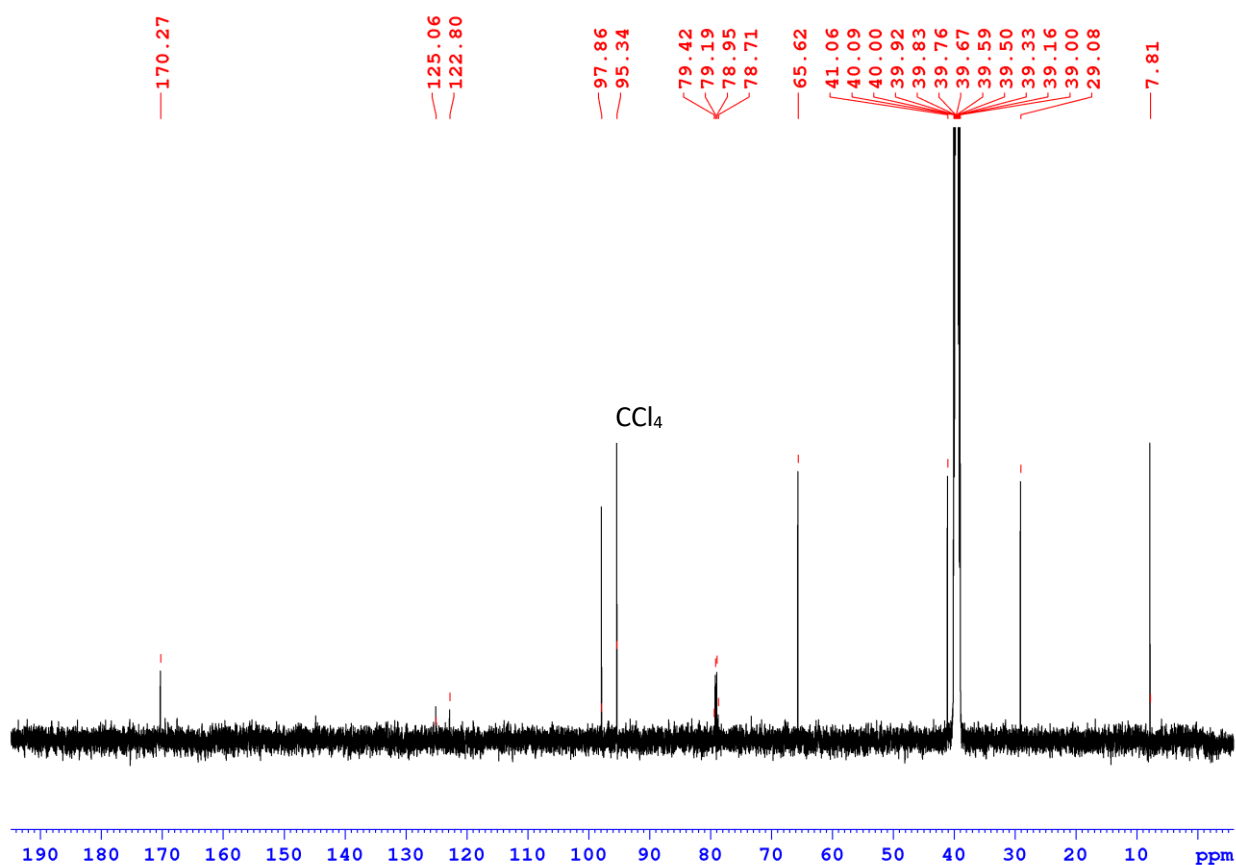


Figure S7. ¹H NMR spectrum of **4b^t**.

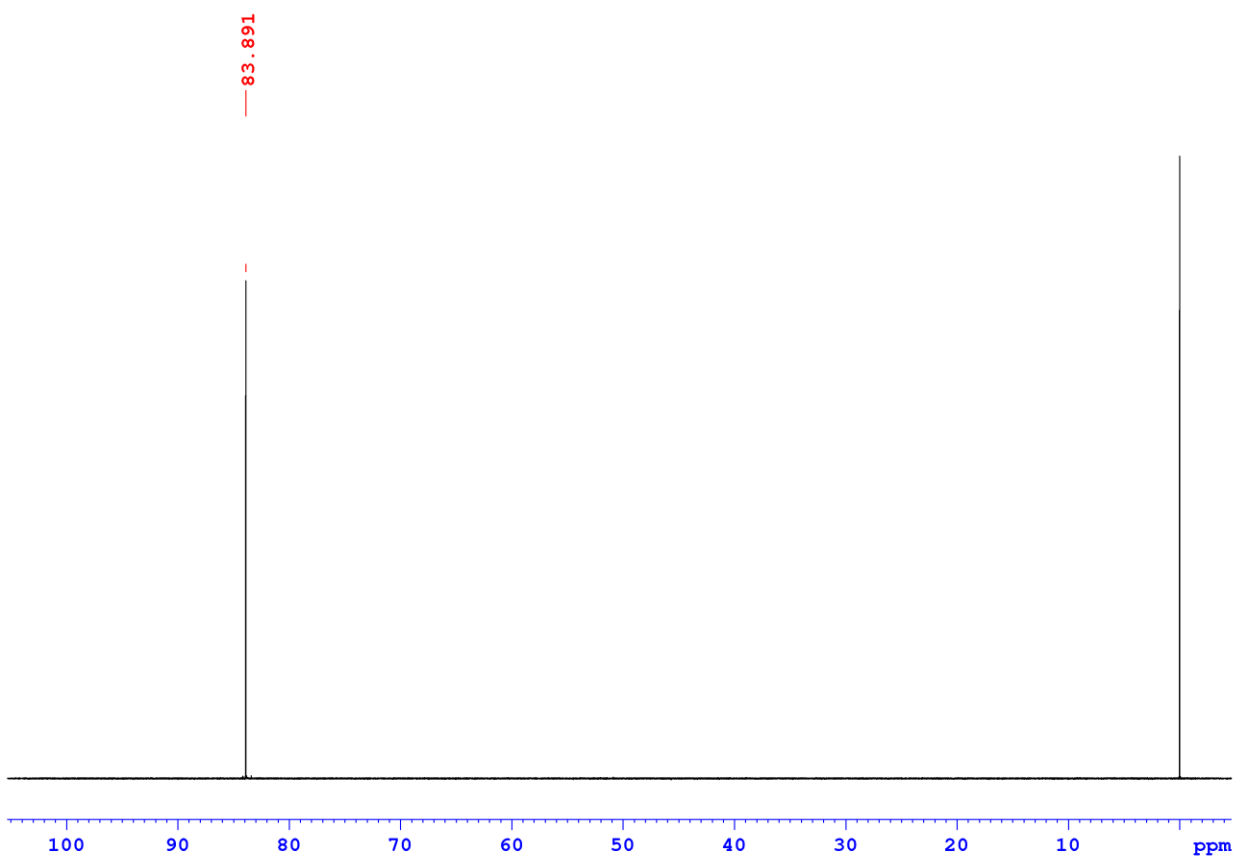


Figure S8. ¹⁹F NMR spectrum of **4b^t**.

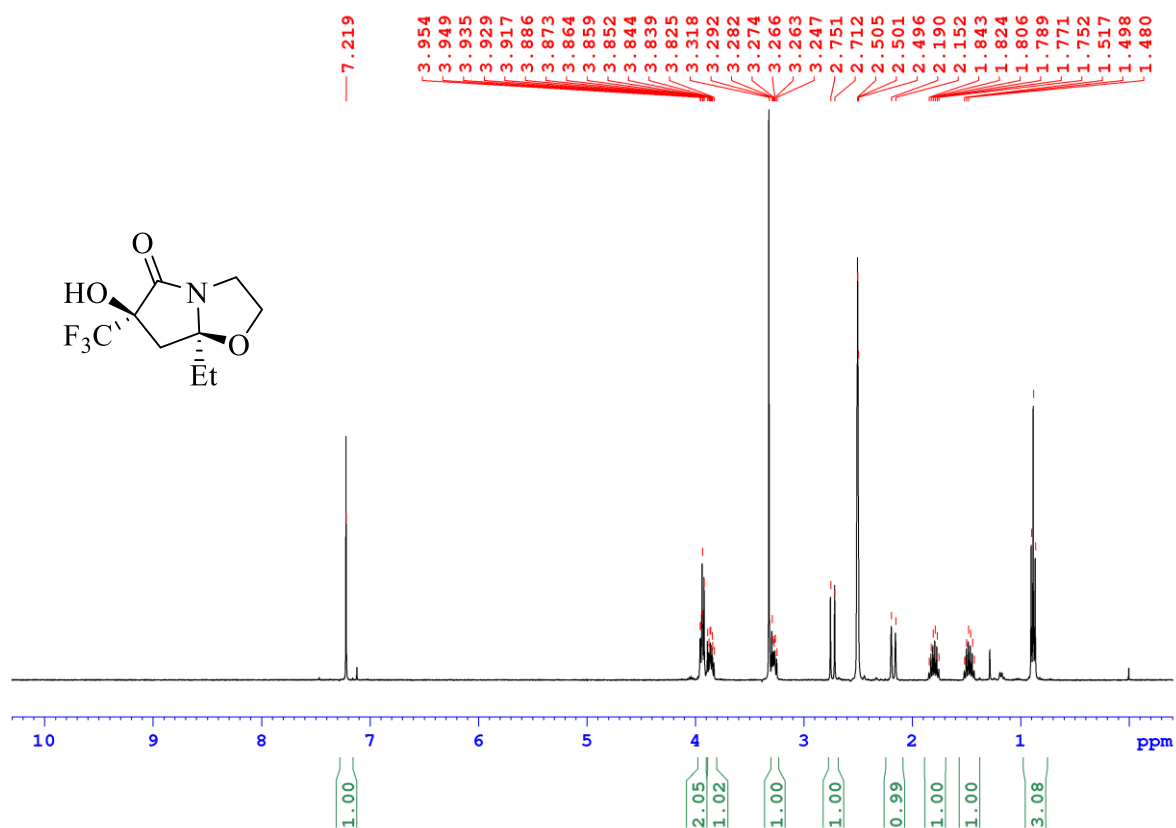


Figure S9. ¹H NMR spectrum of **4b^c** (mixed with **4b^t** in the ratio 90:10).

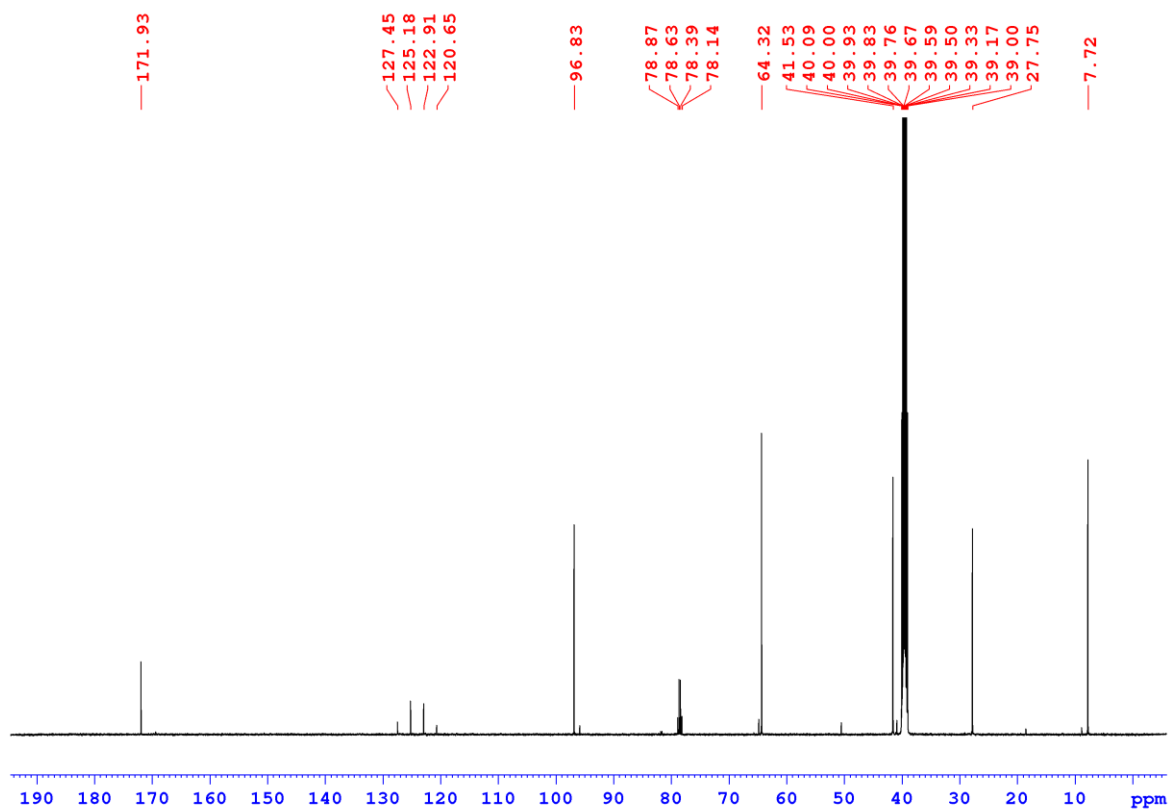


Figure S10. ¹³C NMR spectrum of **4b^c** (mixed with **4b^t** in the ratio 90:10).



Figure S11. ¹⁹F NMR spectrum of **4b^c** (mixed with **4b^t** in the ratio 90:10).

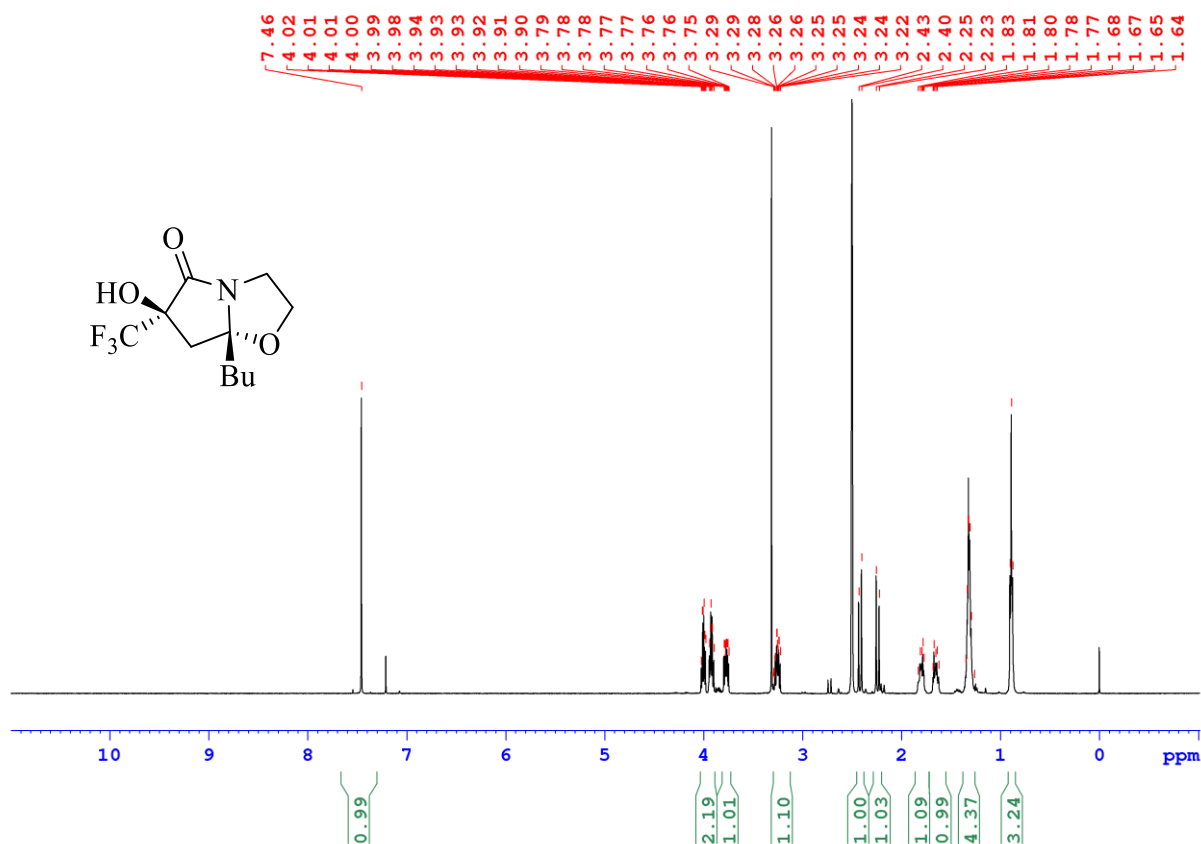


Figure S12. ¹H NMR spectrum of **4c^t** (mixed with **4c^c** in the ratio 90:10).

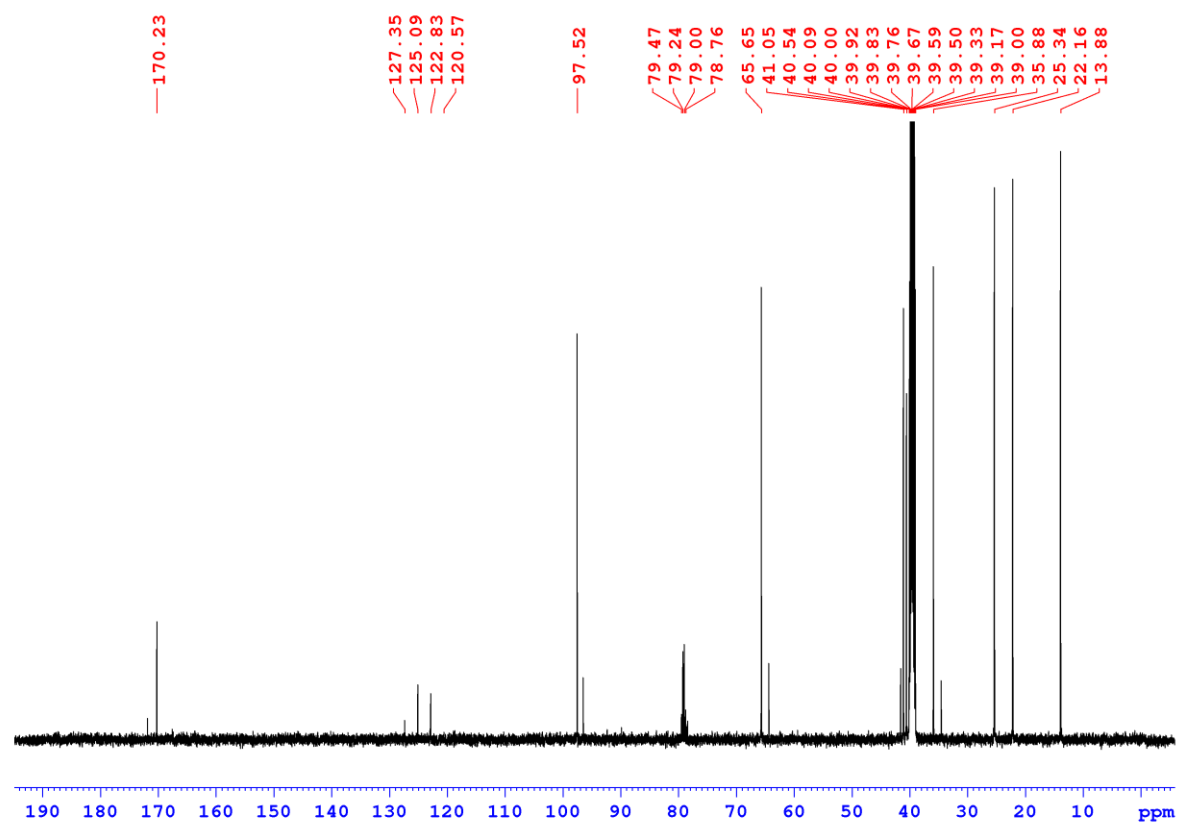


Figure S13. ¹³C NMR spectrum of **4c^t** (mixed with **4c^c** in the ratio 90:10).



Figure S14. ¹⁹F NMR spectrum of **4c^t** (mixed with **4c^c** in the ratio 90:10).

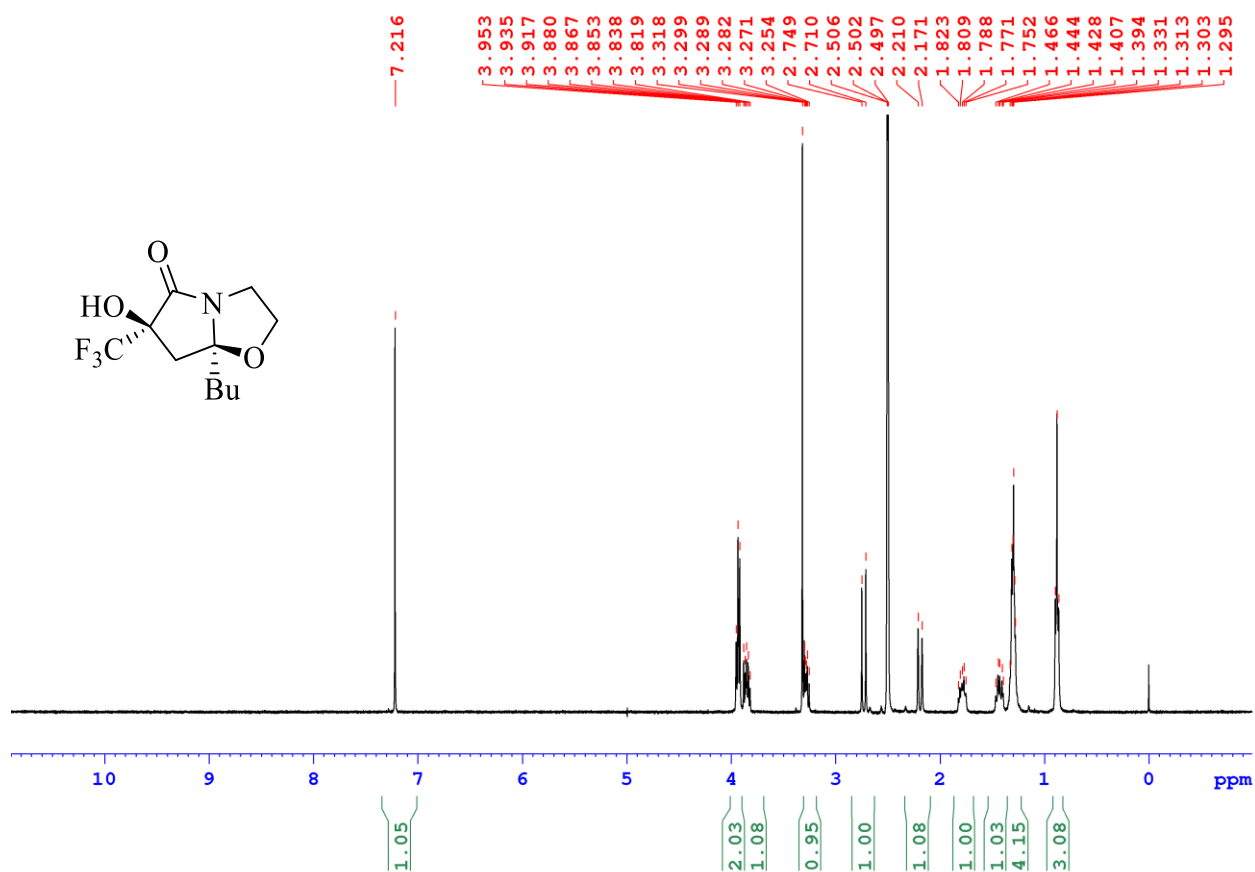


Figure S15. ¹H NMR spectrum of **4c**.

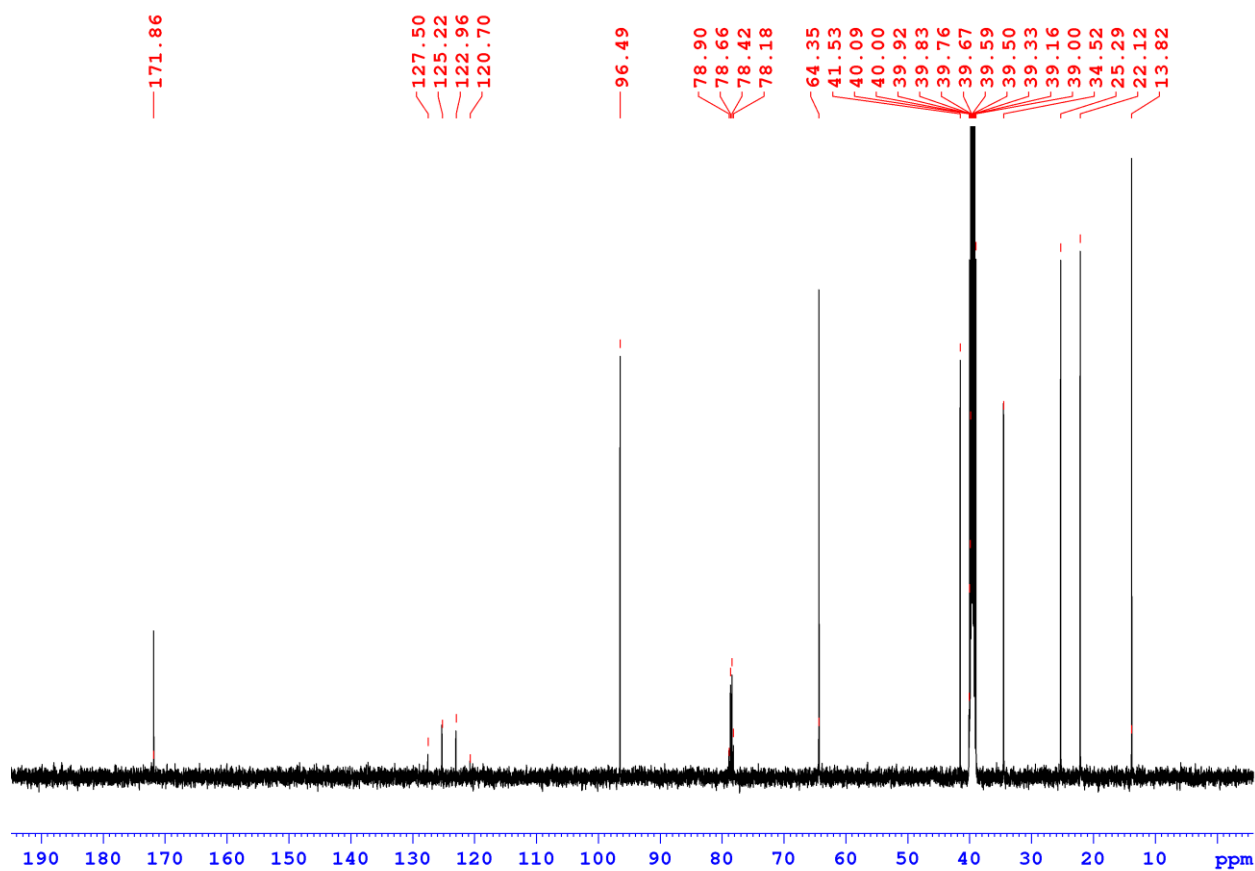


Figure S16. ¹³C NMR spectrum of **4c**.



Figure S17. ¹⁹F NMR spectrum of **4c**.

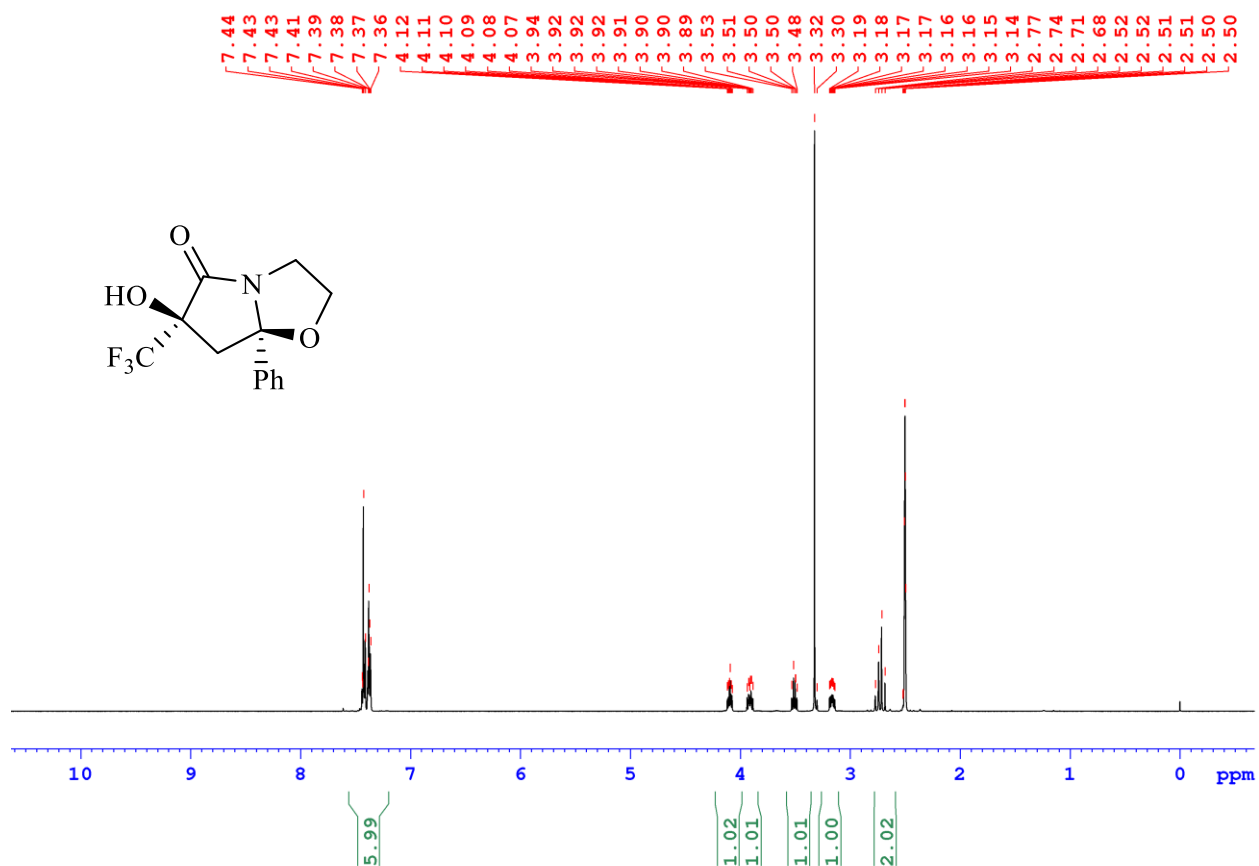


Figure S18. ¹H NMR spectrum of **4d**.

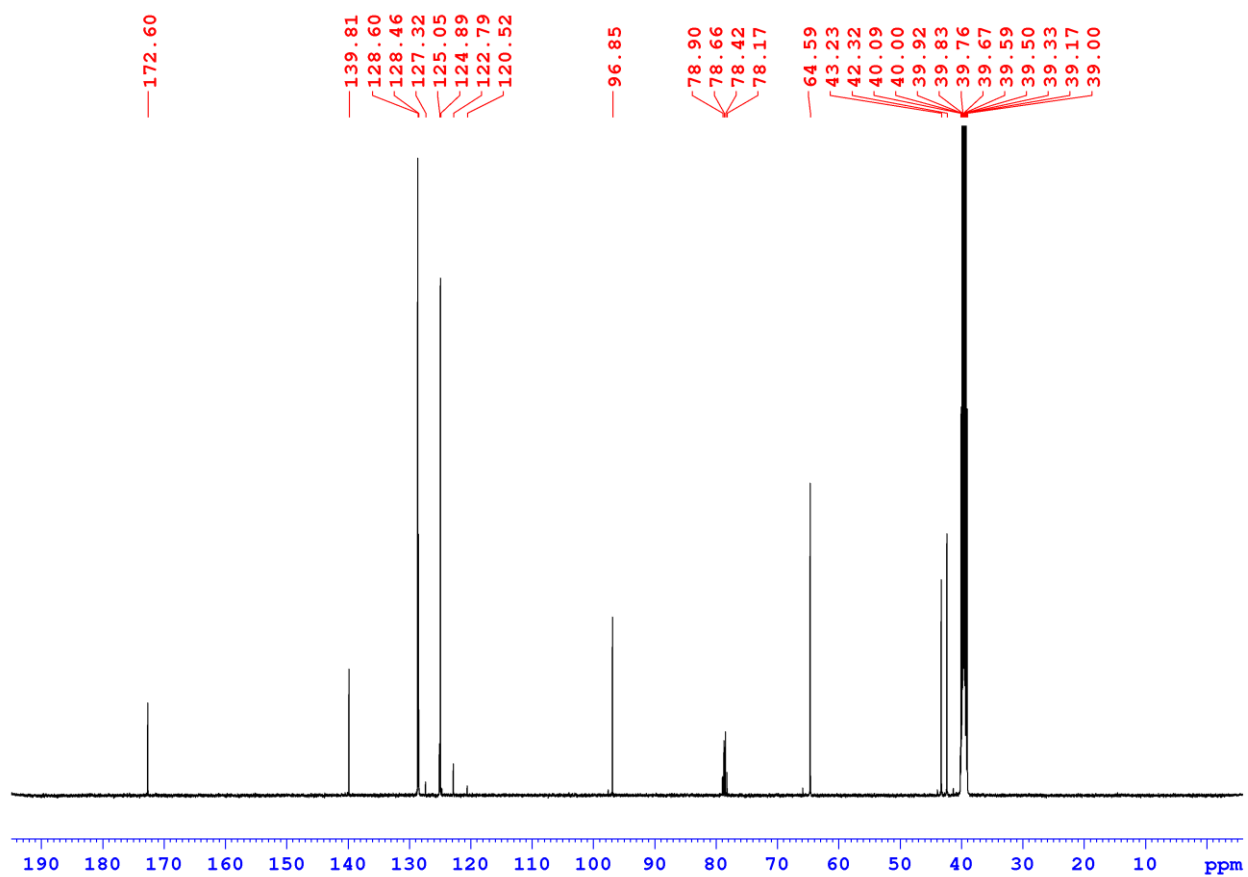


Figure S19. ¹³C NMR spectrum of **4d^c**.

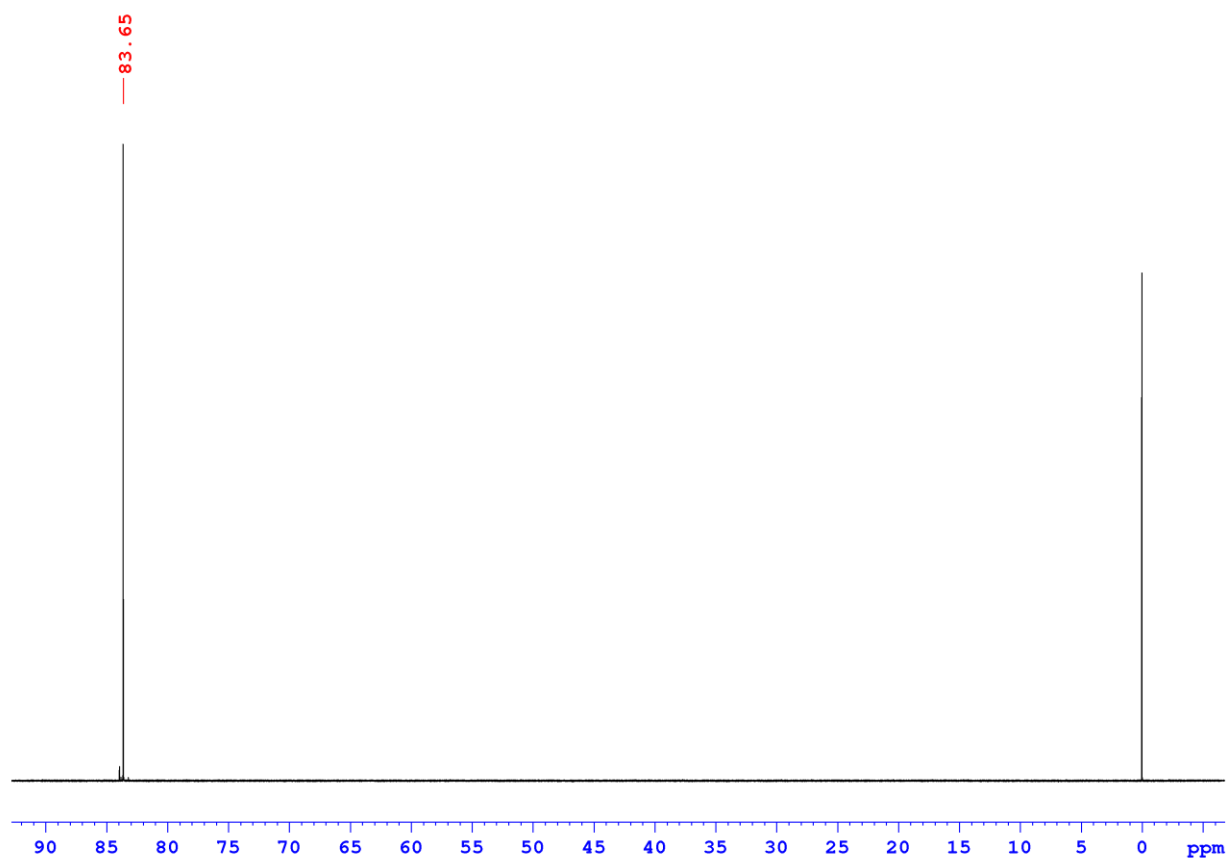


Figure S20. ¹⁹F NMR spectrum of **4d^c**.

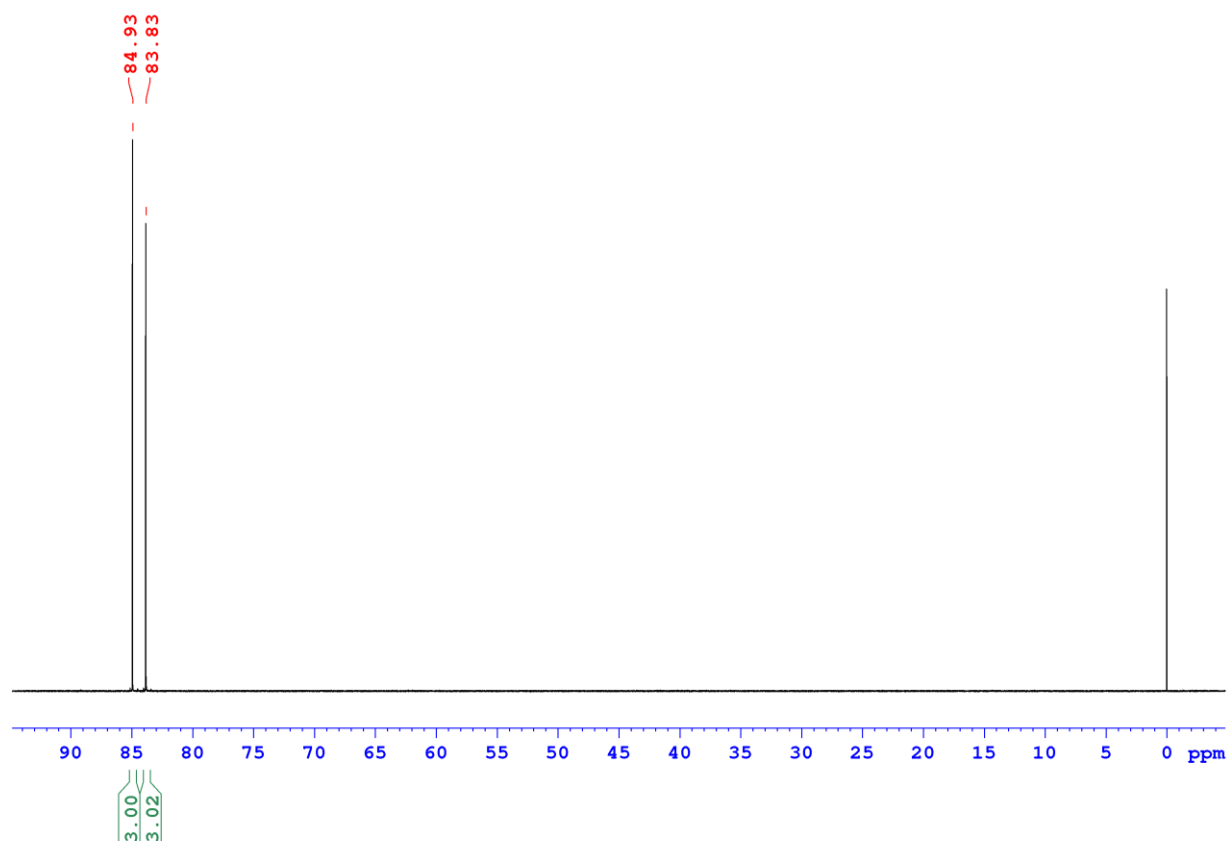


Figure S23. ¹⁹F NMR spectrum of **5^{tc}**.

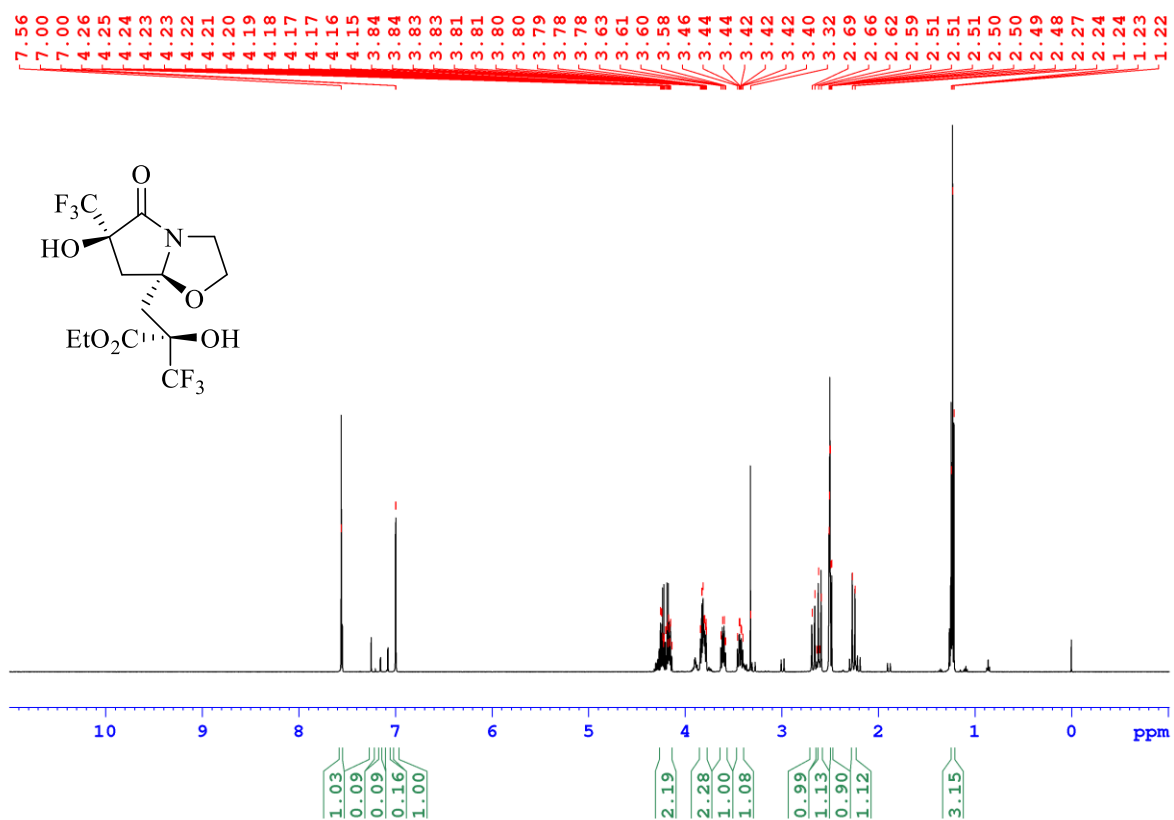


Figure S24. ¹H NMR spectrum of **5^{cc}** (mixed with **5^{tc}** in the ratio 80:20)

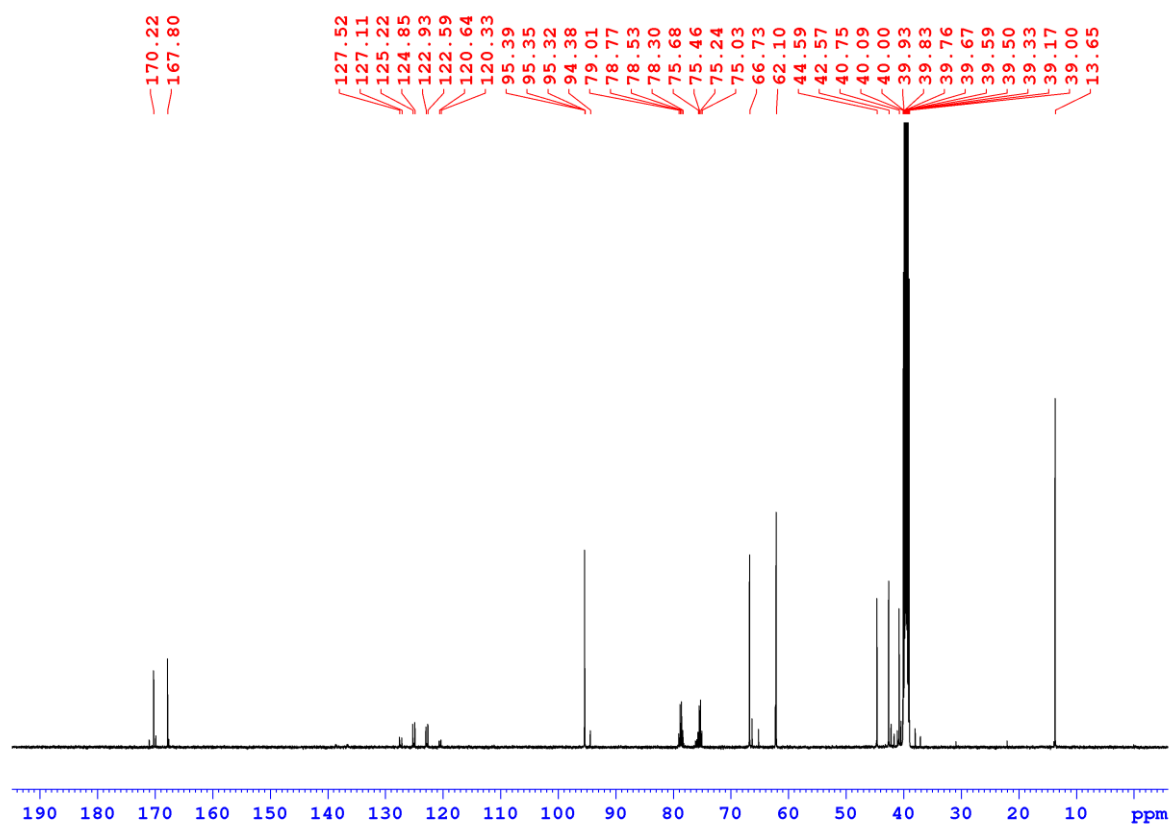


Figure S25. ¹³C NMR spectrum of **5^{cc}** (mixed with **5^{tc}** in the ratio 80:20).

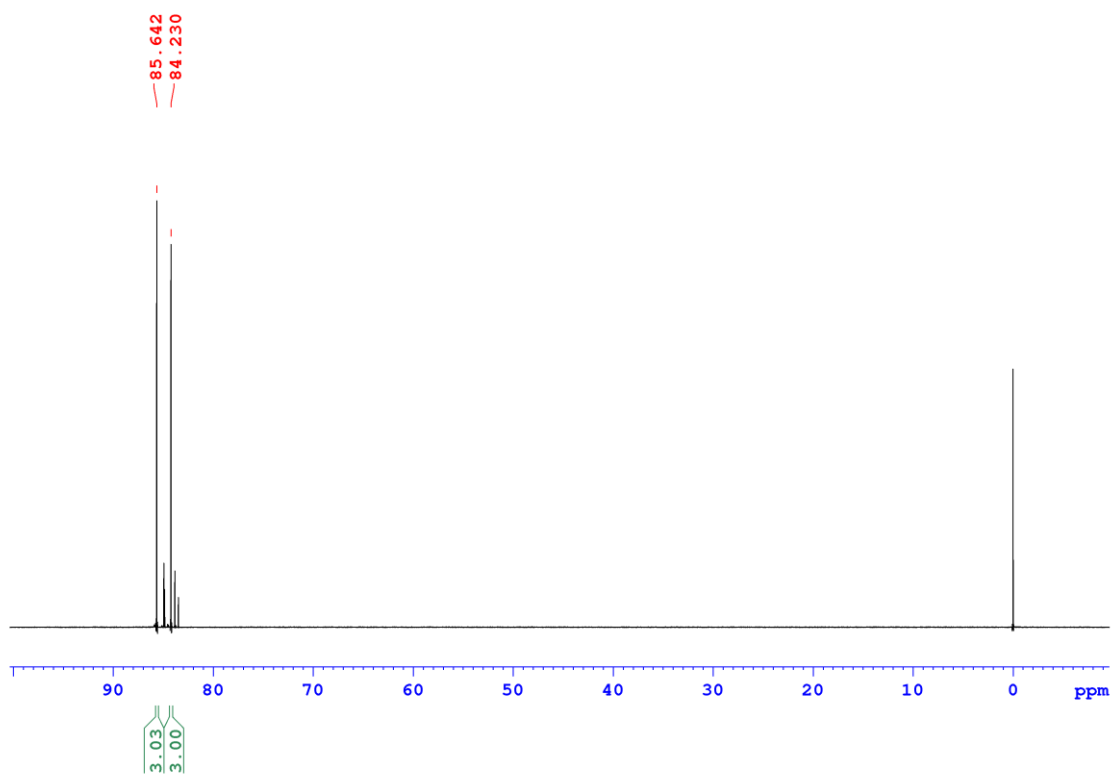


Figure S26. ¹⁹F NMR spectrum of **5^{cc}** (mixed with **5^{tc}** in the ratio 80:20).

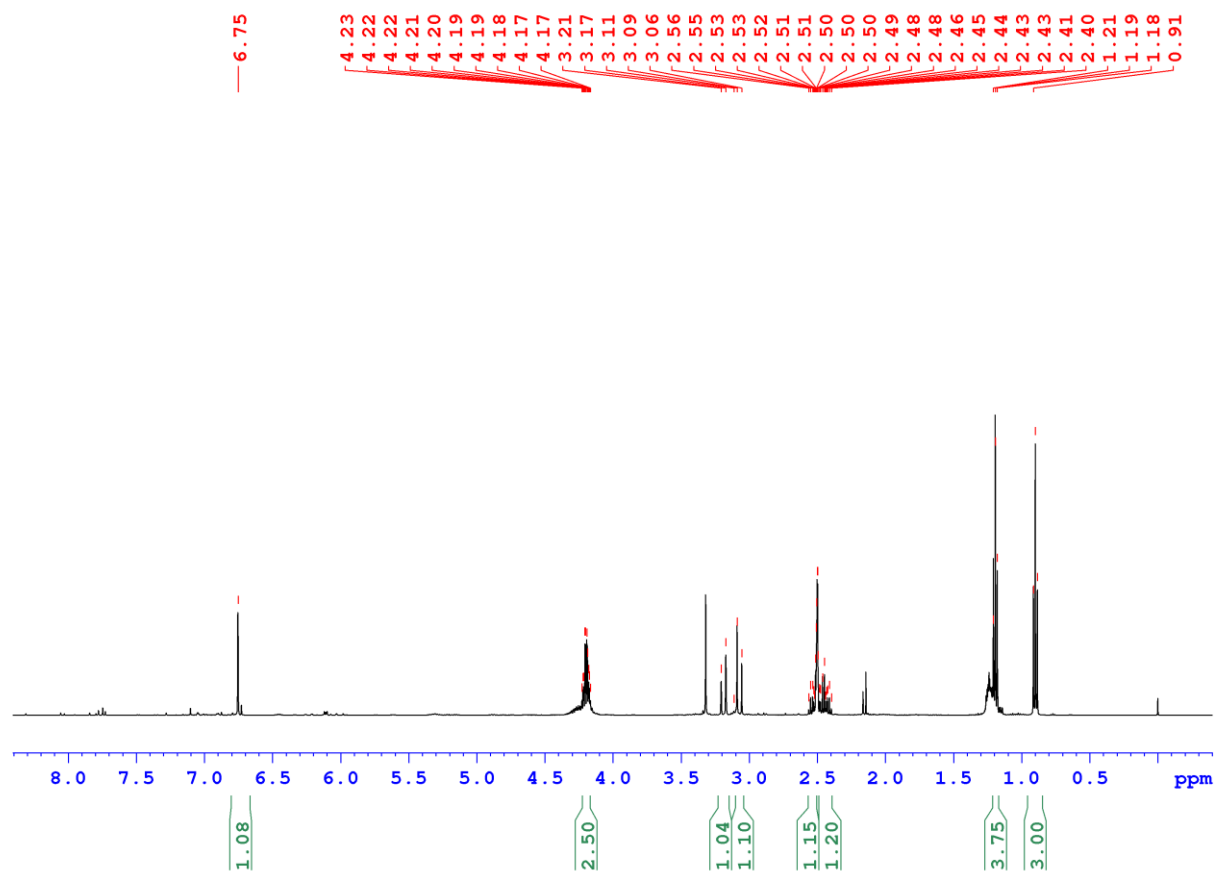


Figure S27. ¹H NMR spectrum of **6b**.

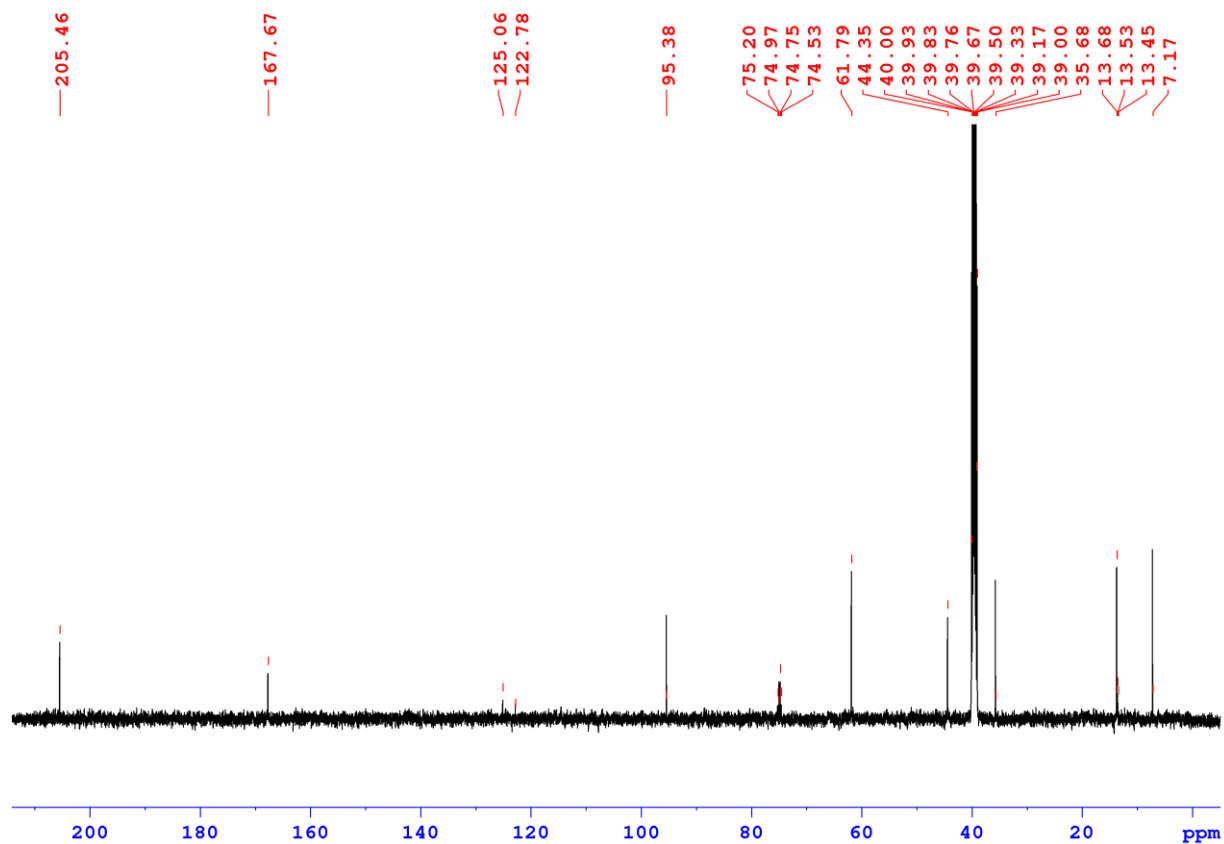


Figure S28. ¹³C NMR spectrum of **6b**.

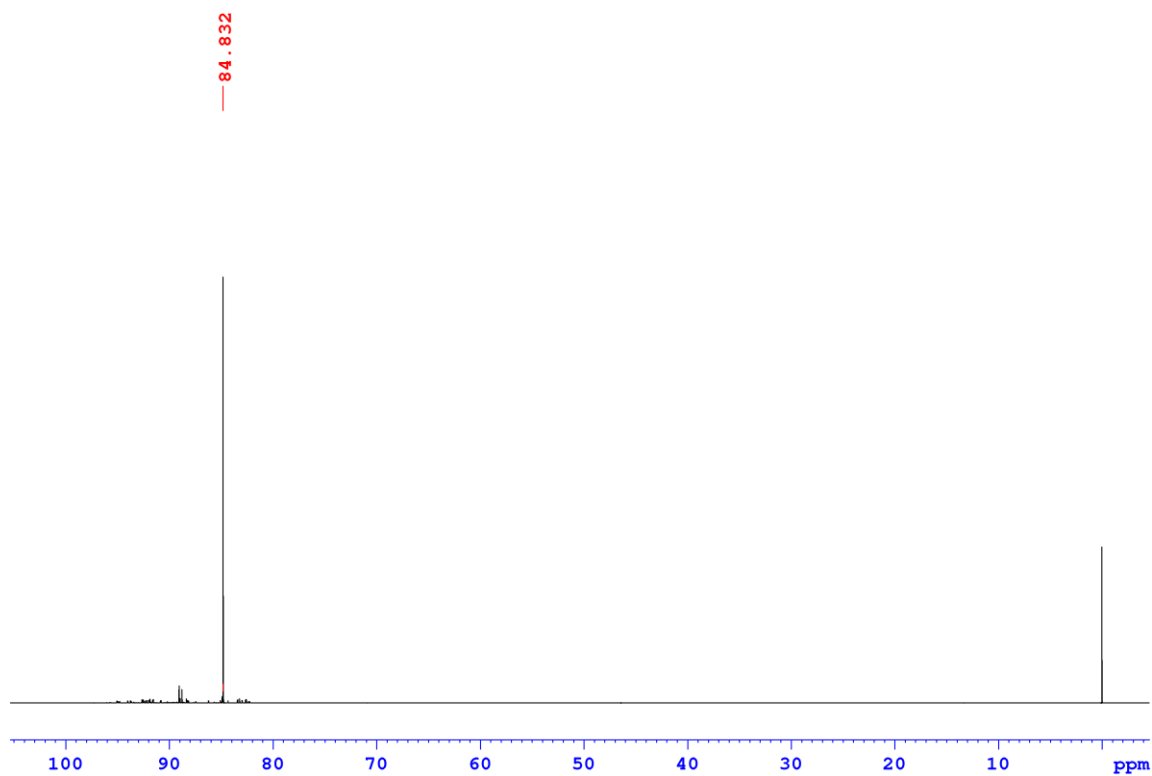


Figure S29. ^{19}F NMR spectrum of **6b**.

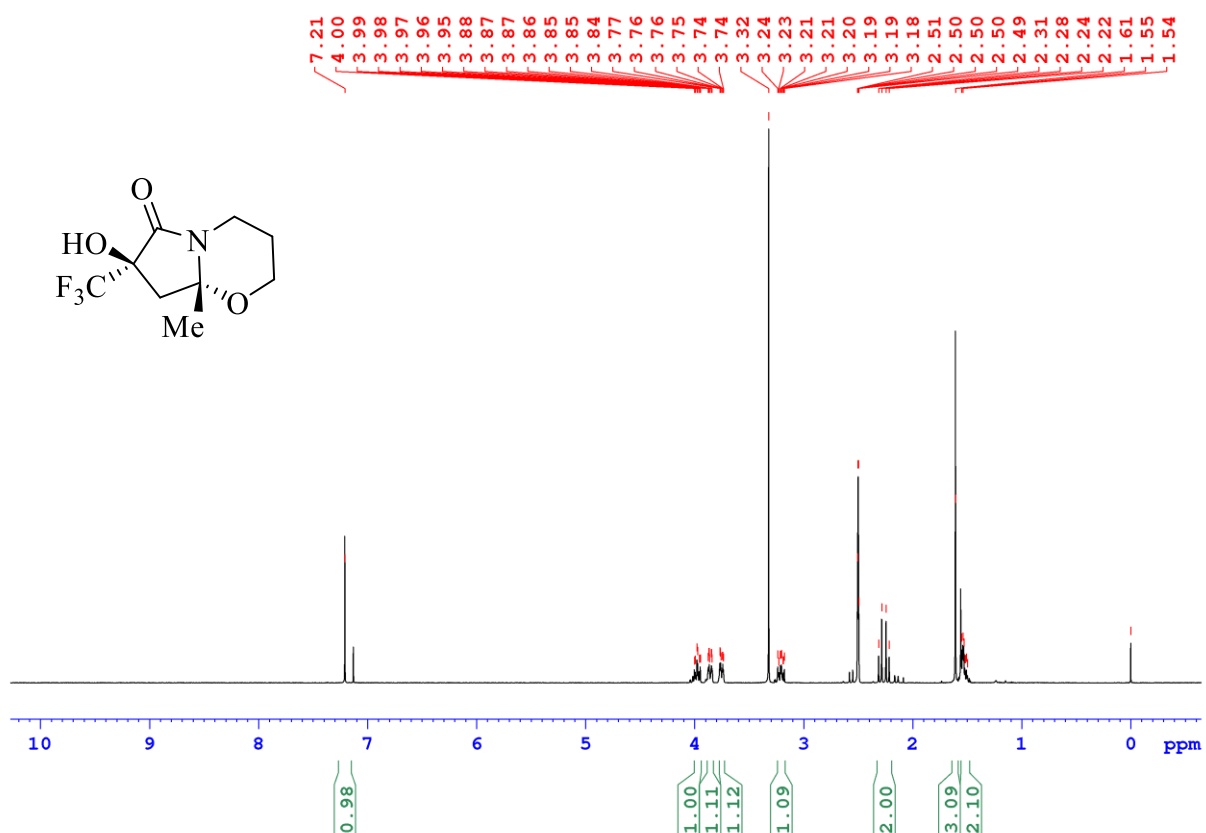


Figure S30. ^1H NMR spectrum of **8a^t** (mixed with **8a^c** in the ratio 84:16).

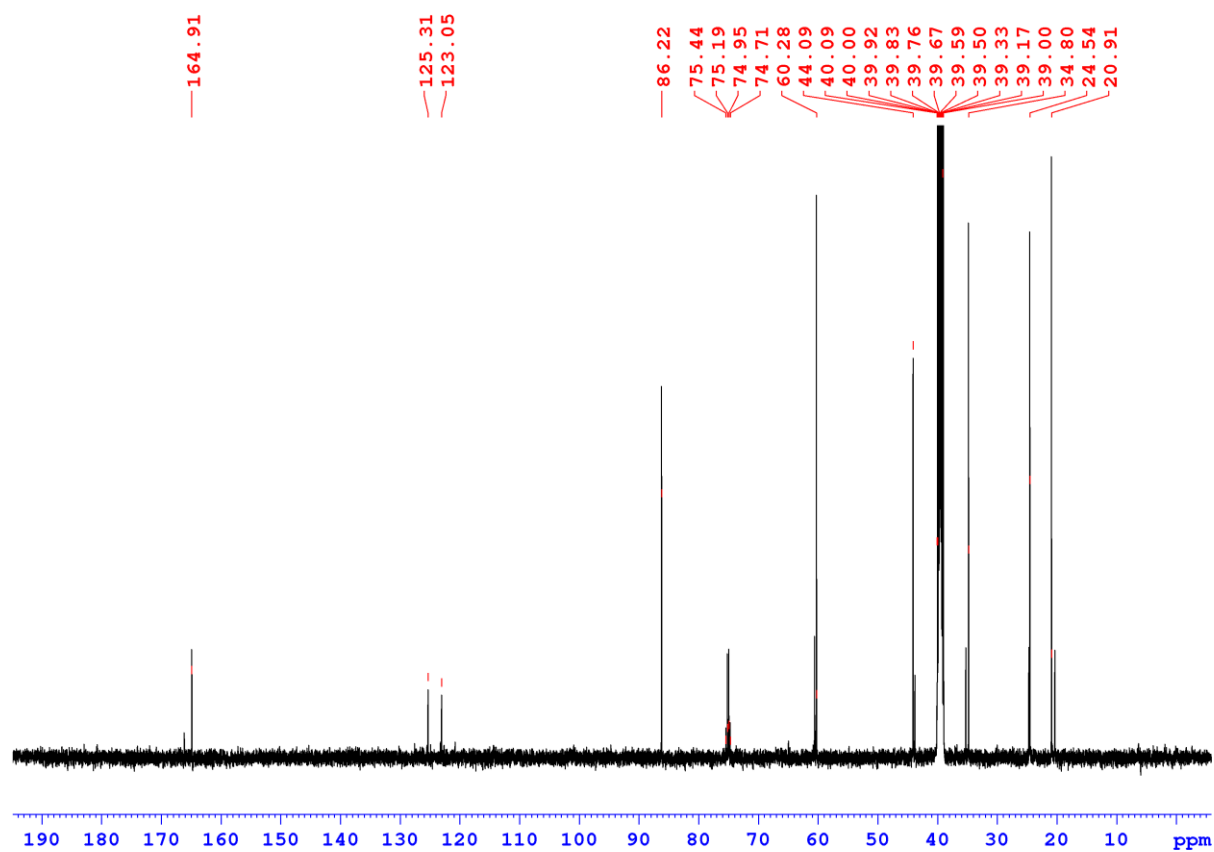


Figure S31. ¹³C NMR spectrum of **8a^t** (mixed with **8a^c** in the ratio 84:16).

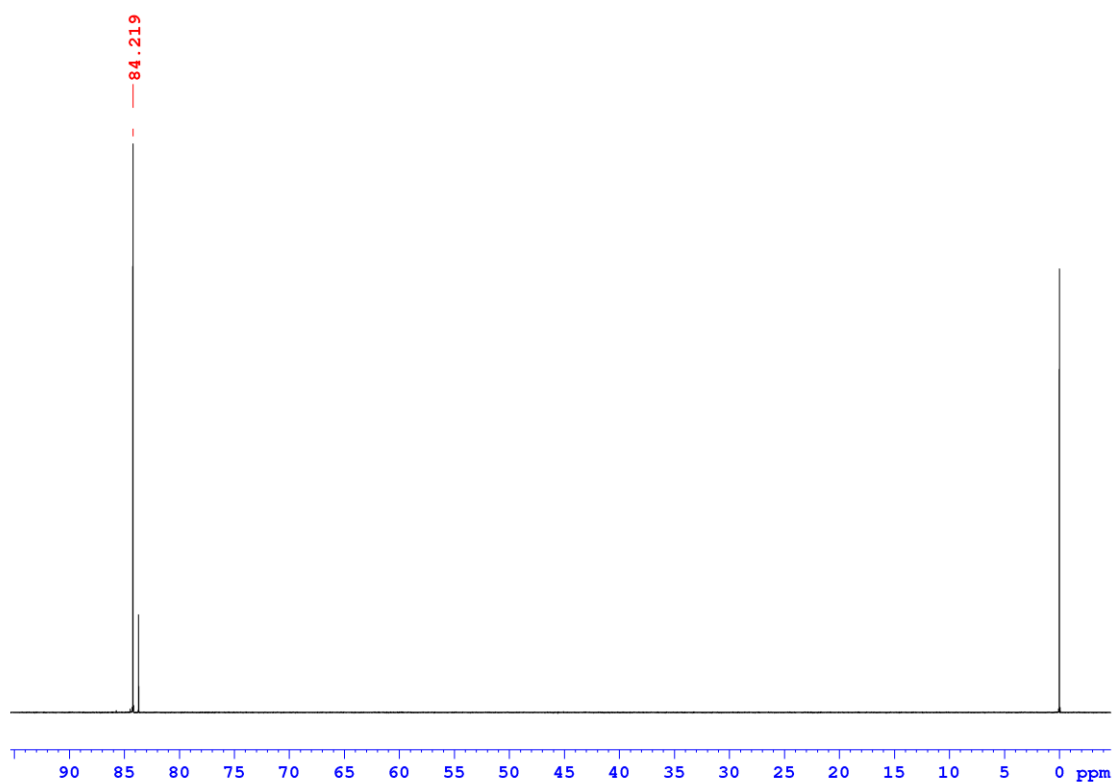


Figure S32. ¹⁹F NMR spectrum of **8a^t** (mixed with **8a^c** in the ratio 84:16).

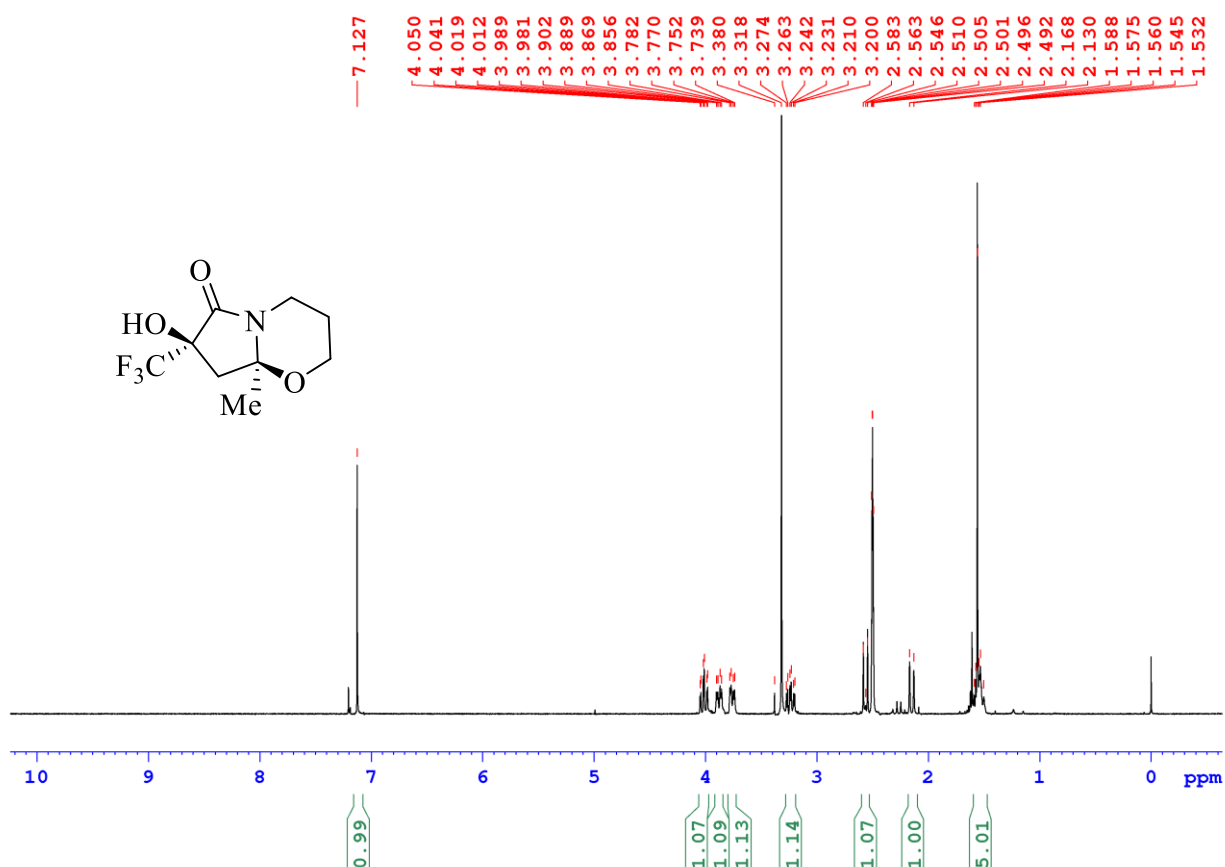


Figure S33. ¹H NMR spectrum of **8a^c** (mixed with **8a^t** in the ratio 89:11).

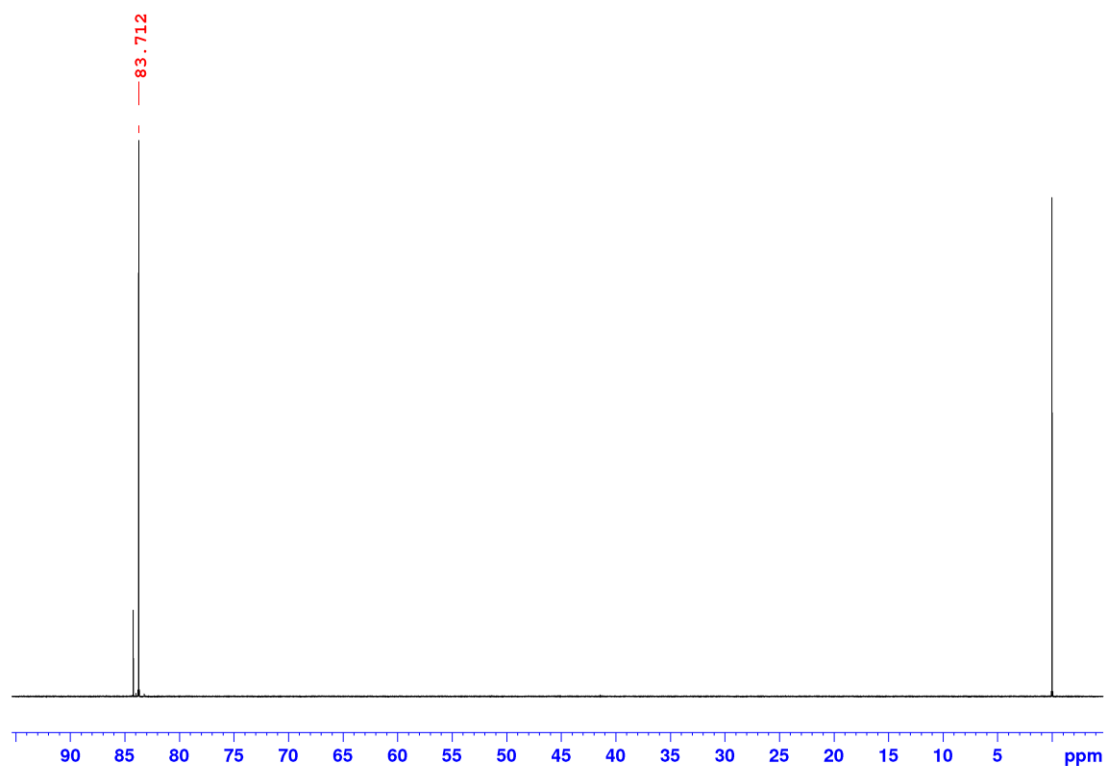


Figure S34. ¹⁹F NMR spectrum of **8a^c** (mixed with **8a^t** in the ratio 89:11).

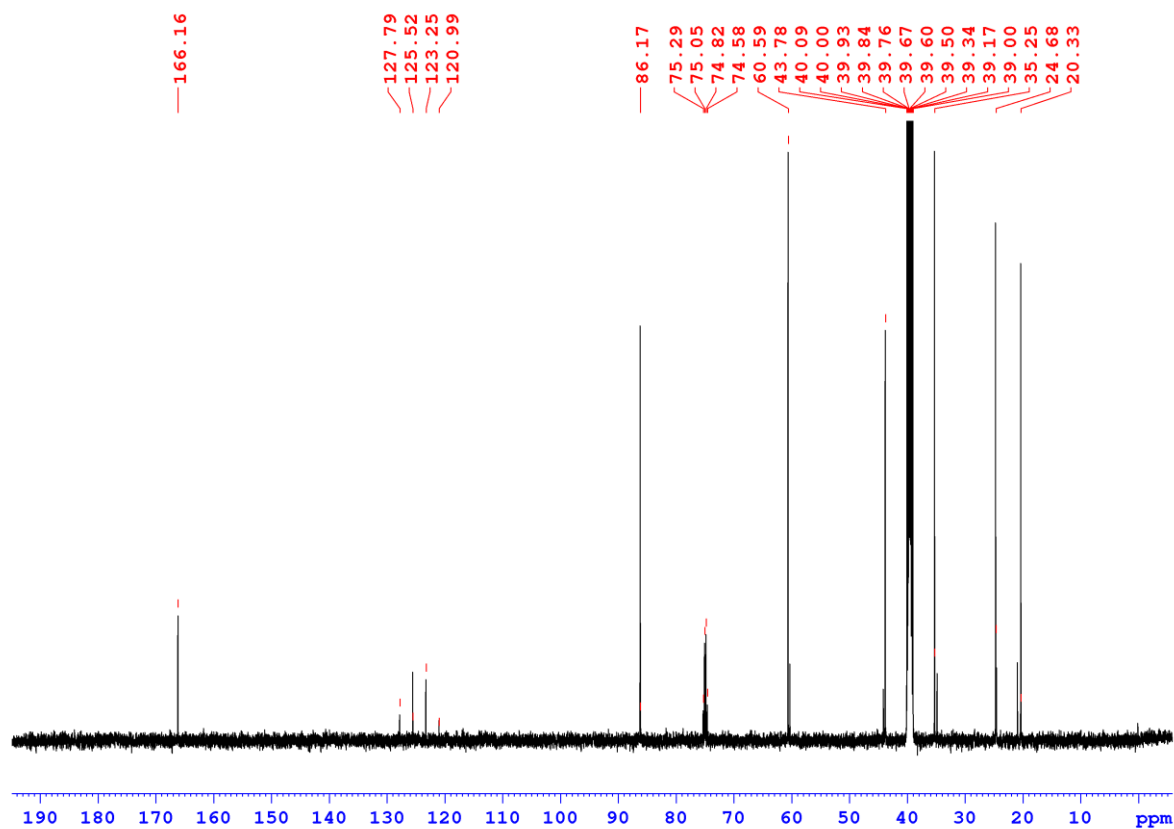


Figure S35. ¹³C NMR spectrum of **8a^c** (mixed with **8a^t** in the ratio 89:11).

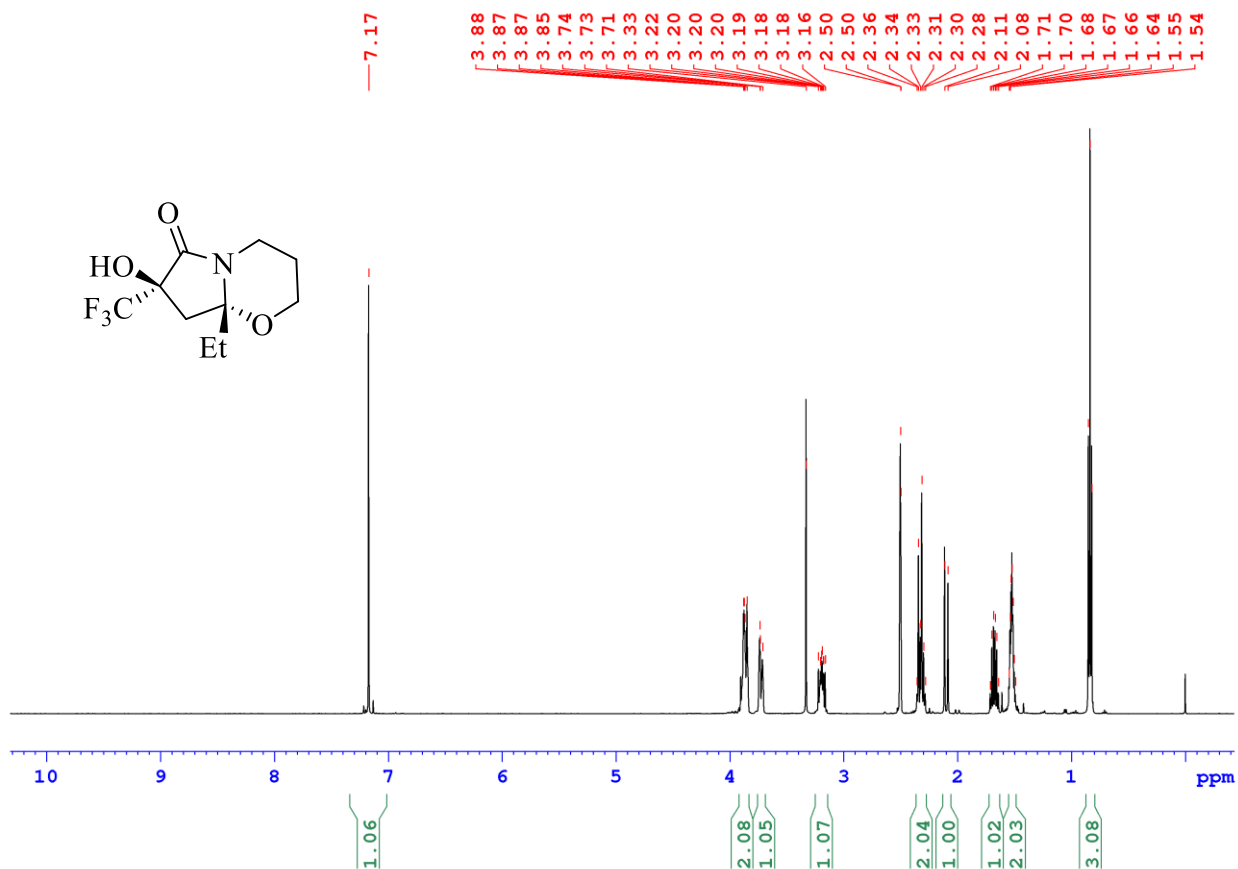


Figure S36. ¹H NMR spectrum of **8b^t**.

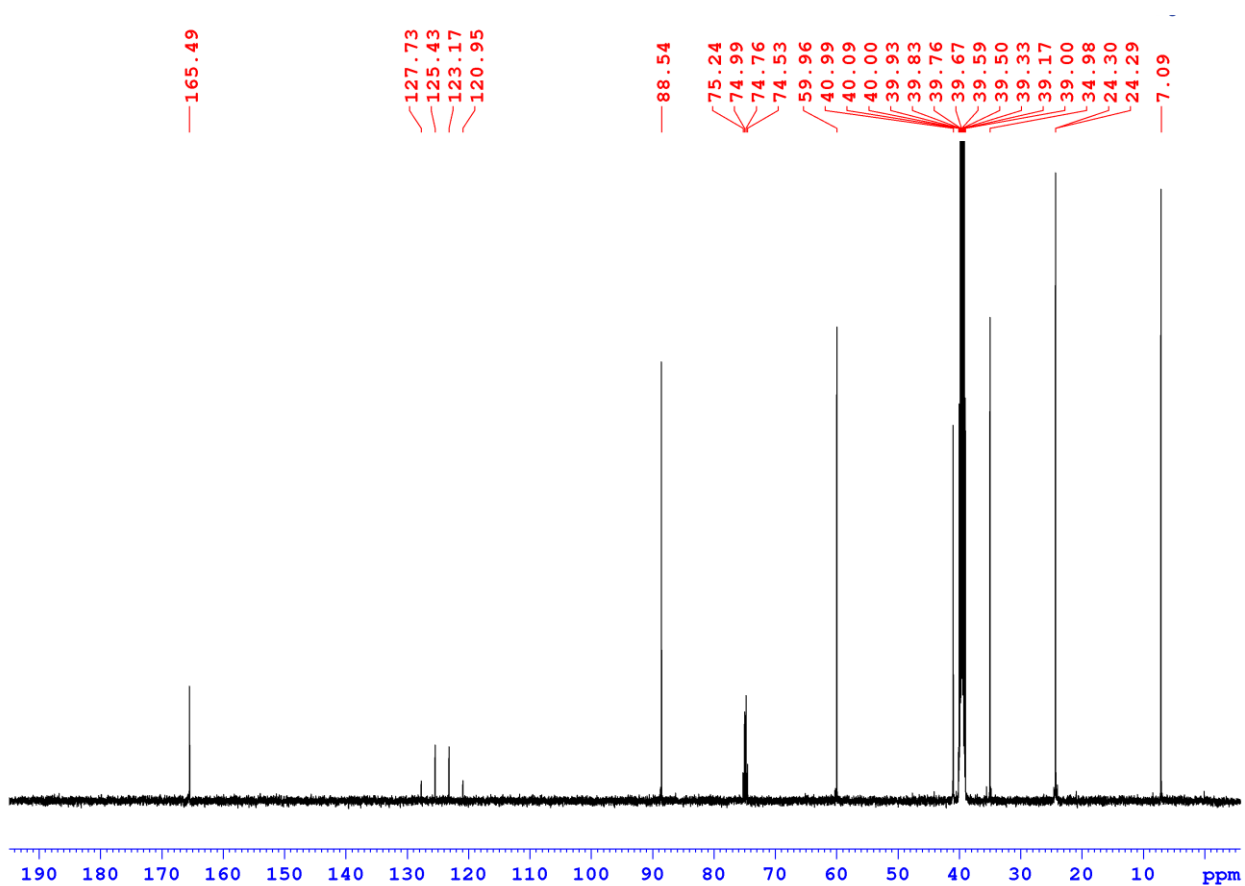


Figure S37. ^{13}C NMR spectrum of **8b^t**.



Figure S38. ^{19}F NMR spectrum of **8b^t**.

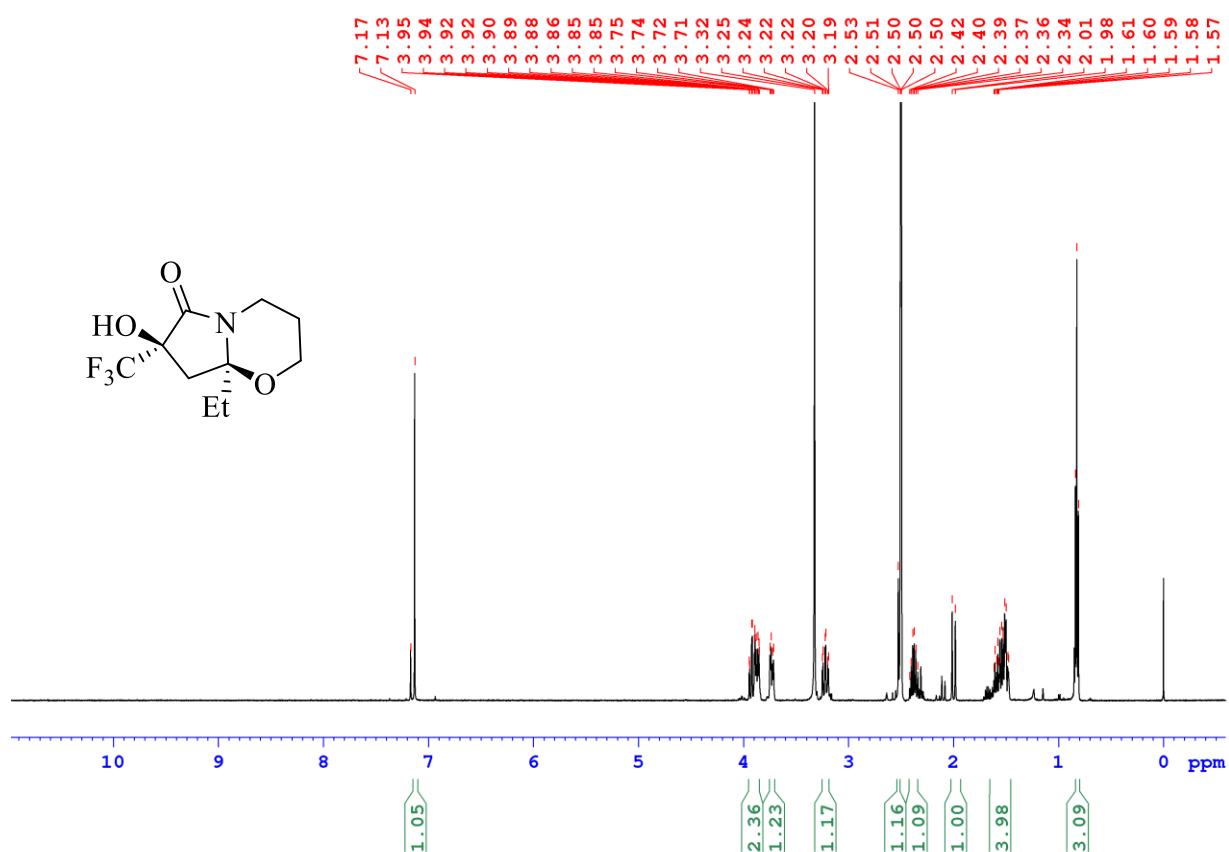


Figure S39. ¹H NMR spectrum of **8b^c** (mixed with **8b^t** in the ratio 81:19).

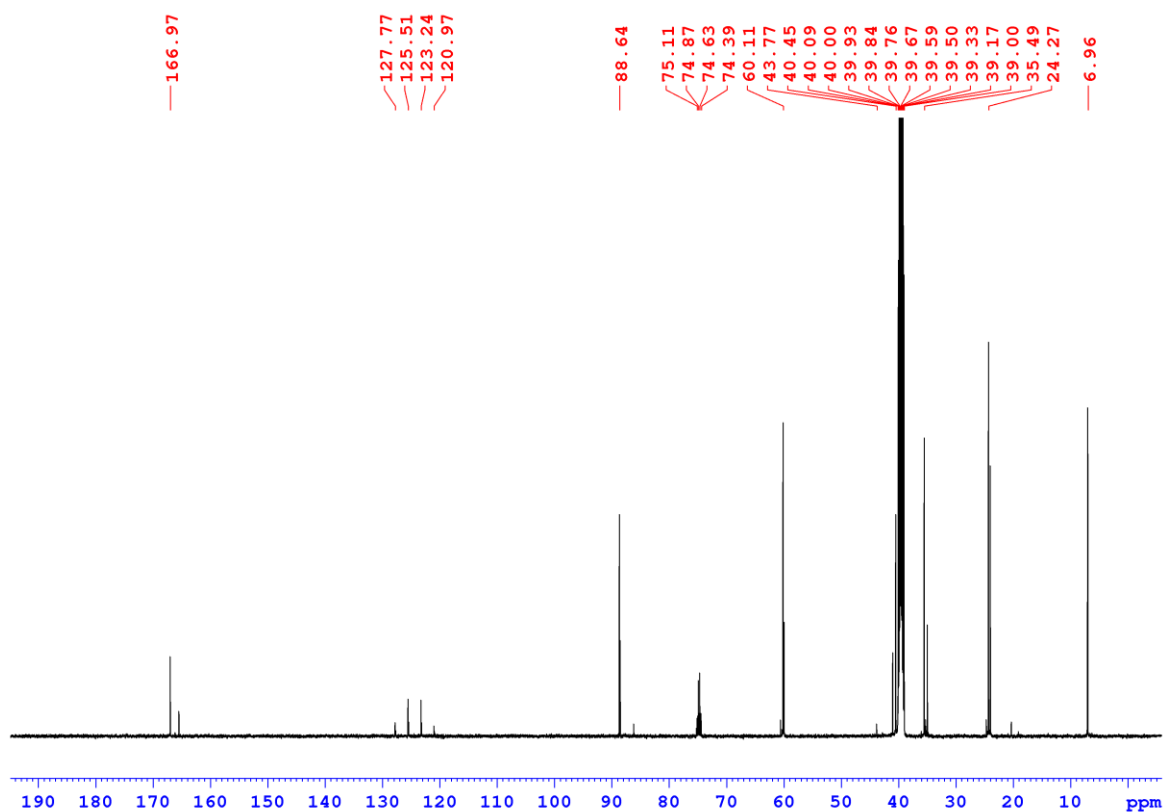


Figure S40. ¹³C NMR spectrum of **8b^c** (mixed with **8b^t** in the ratio 81:19).

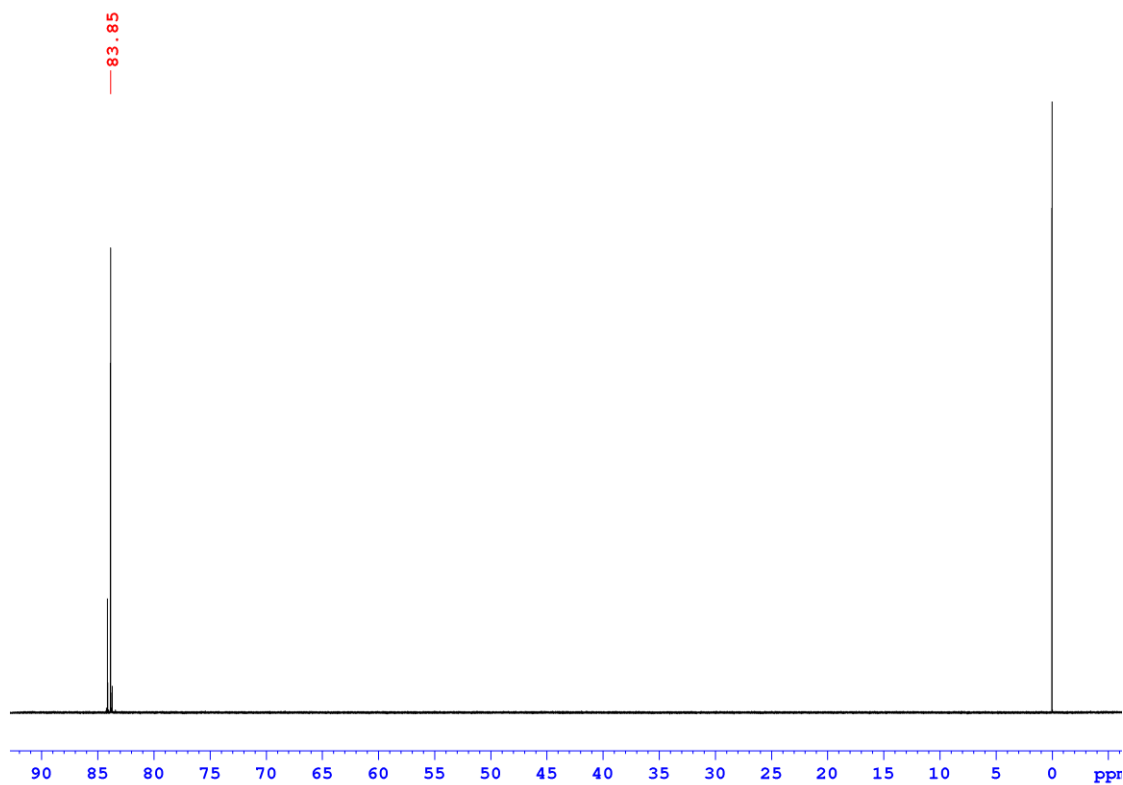


Figure S41. ^{19}F NMR spectrum of **8b^c** (mixed with **8b^t** in the ratio 81:19).

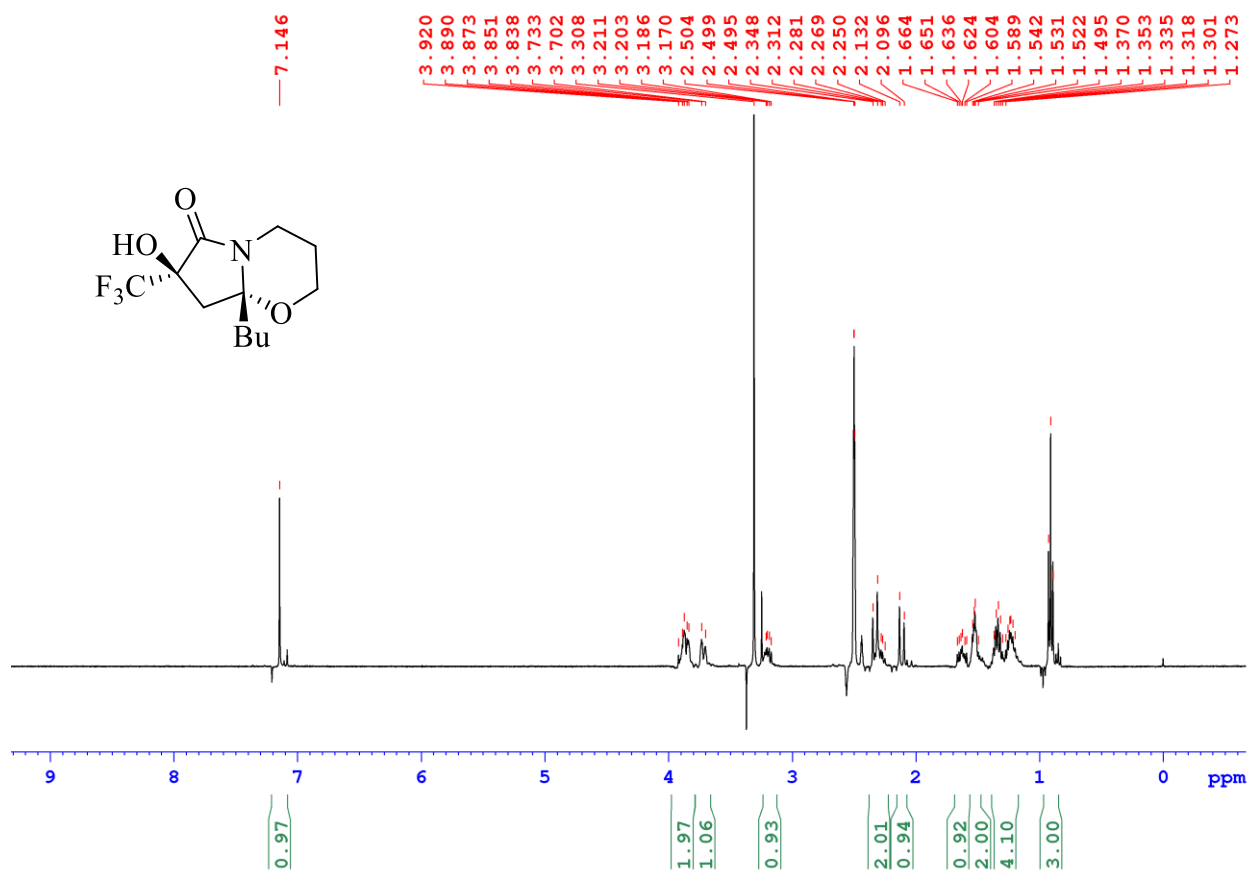


Figure S42. ^1H NMR spectrum of **8c^t**.



Figure S43. ¹⁹F NMR spectrum of **8c^t**.

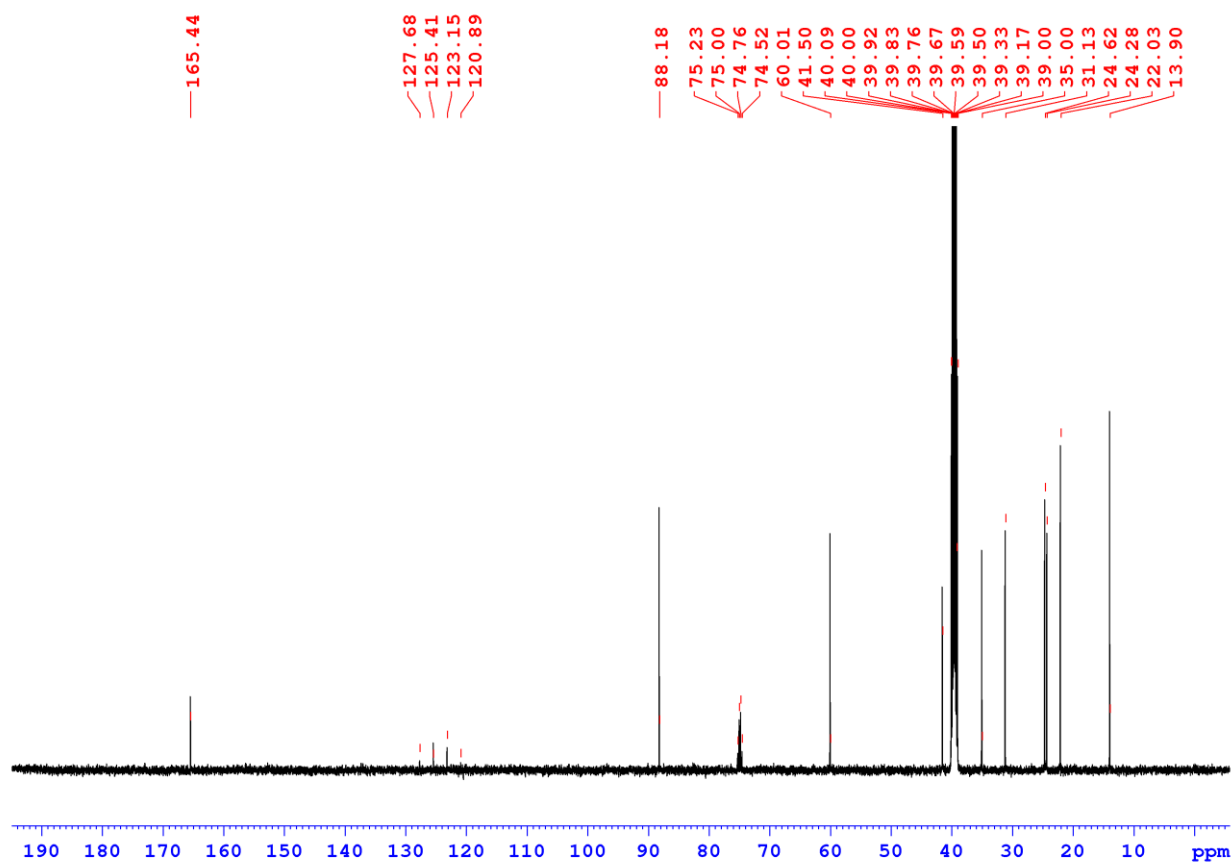


Figure S44. ¹³C NMR spectrum of **8c^t**.

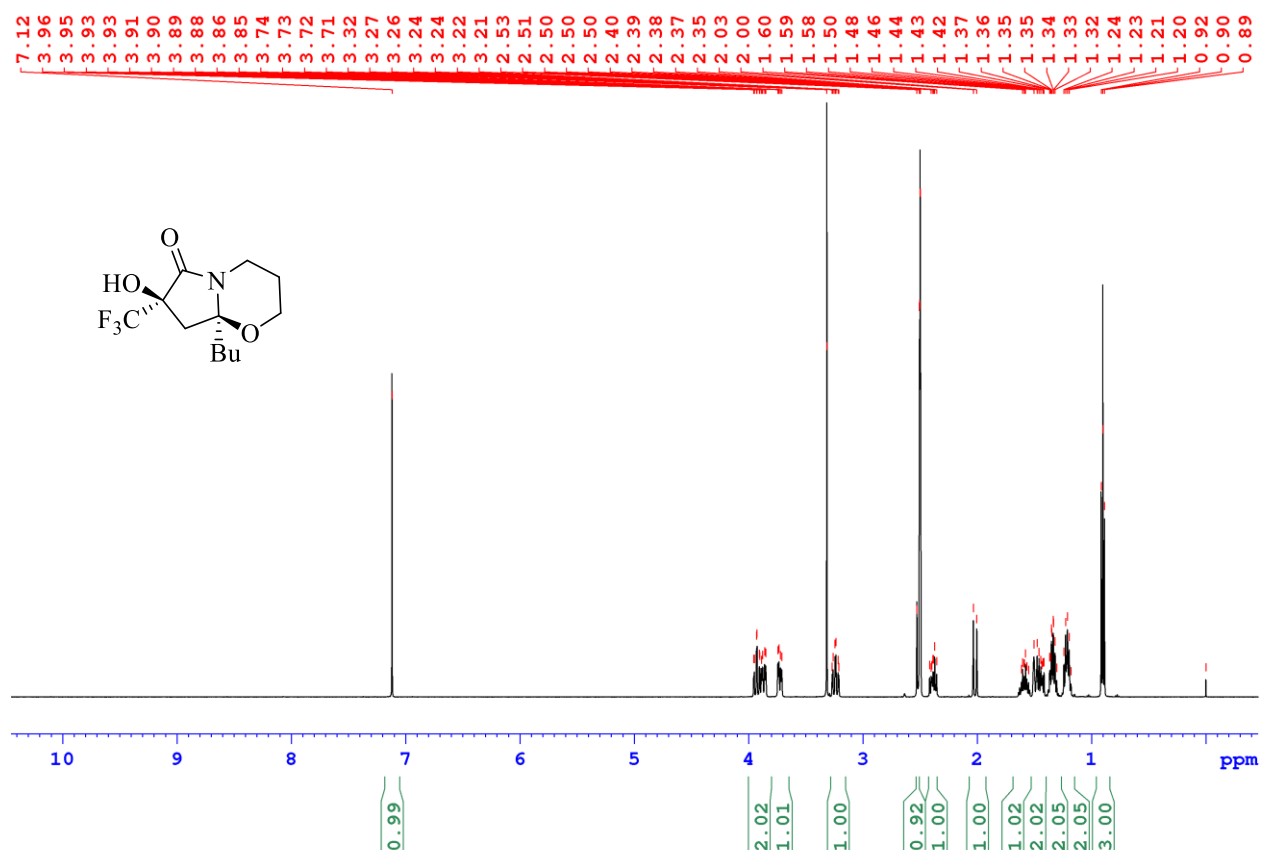


Figure S45. ¹H NMR spectrum of **8c**.

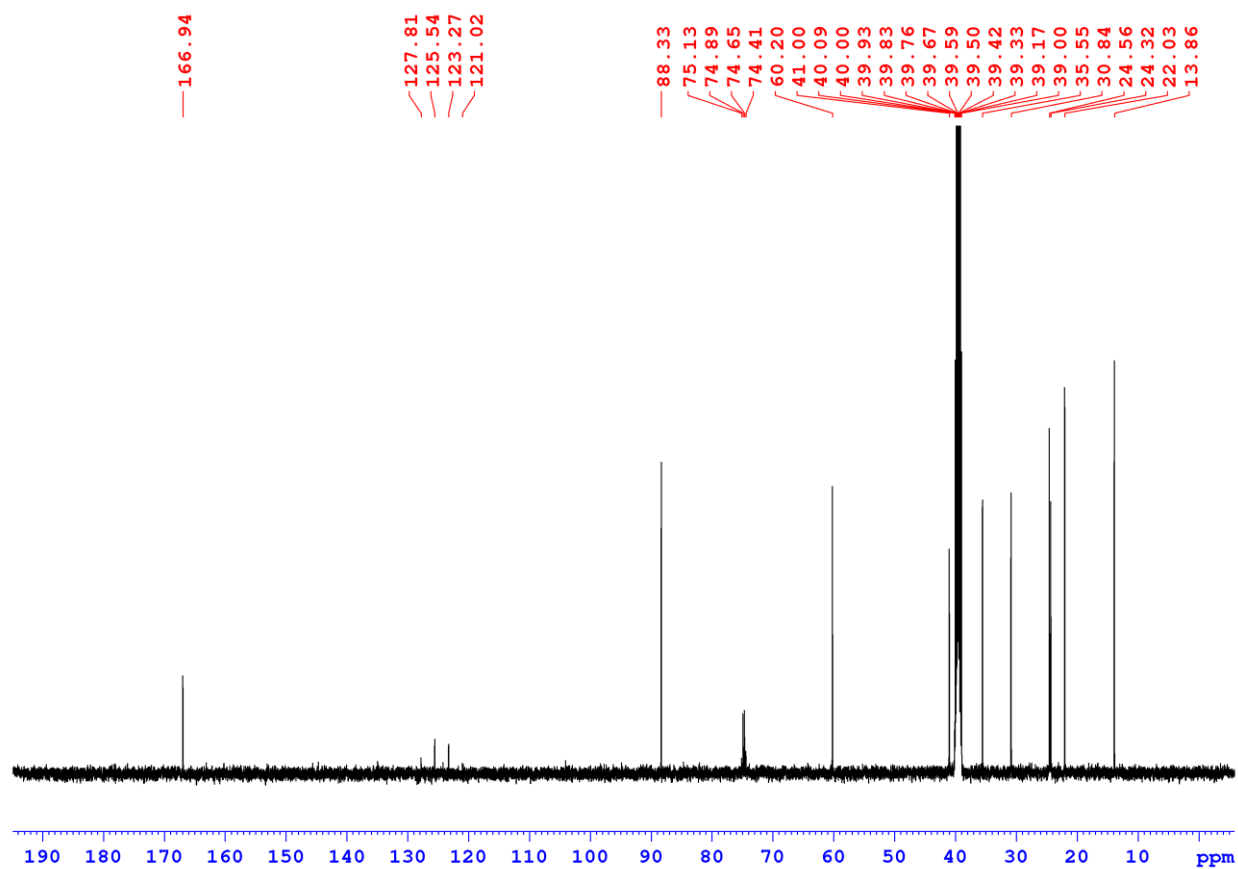


Figure S46. ¹³C NMR spectrum of **8c**.

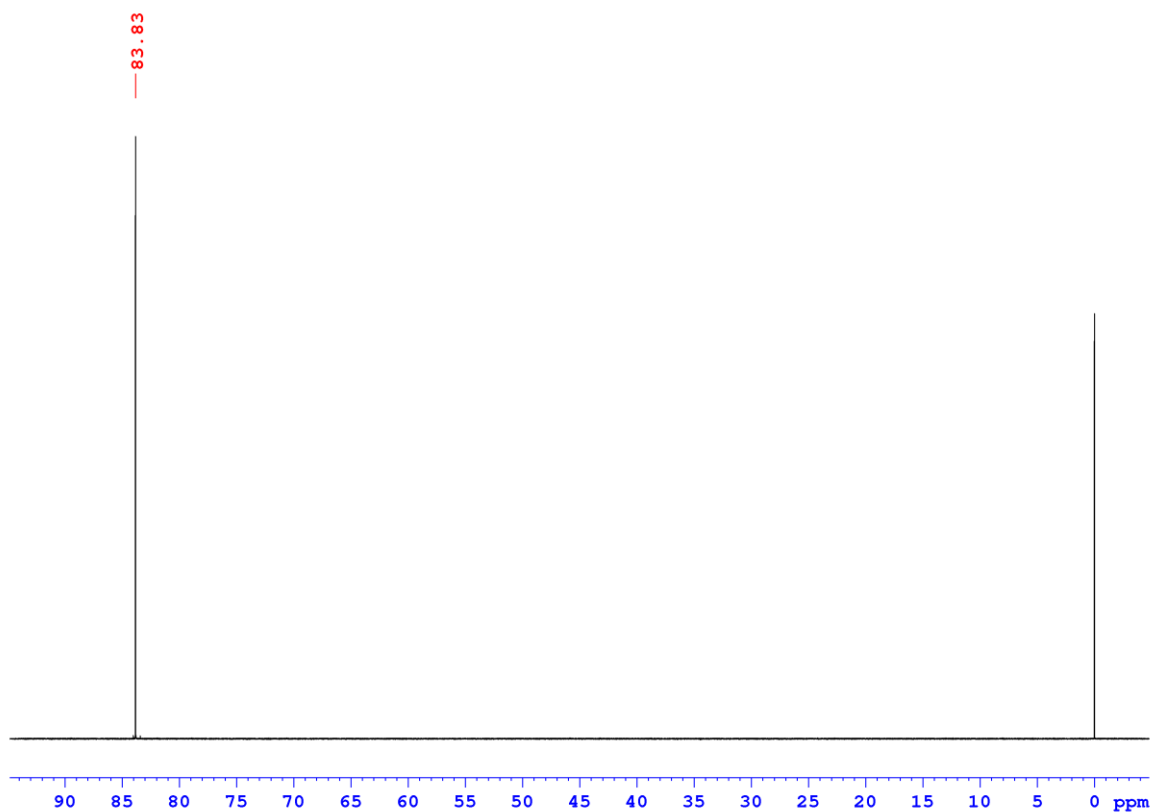


Figure S47. ¹⁹F NMR spectrum of **8c**.

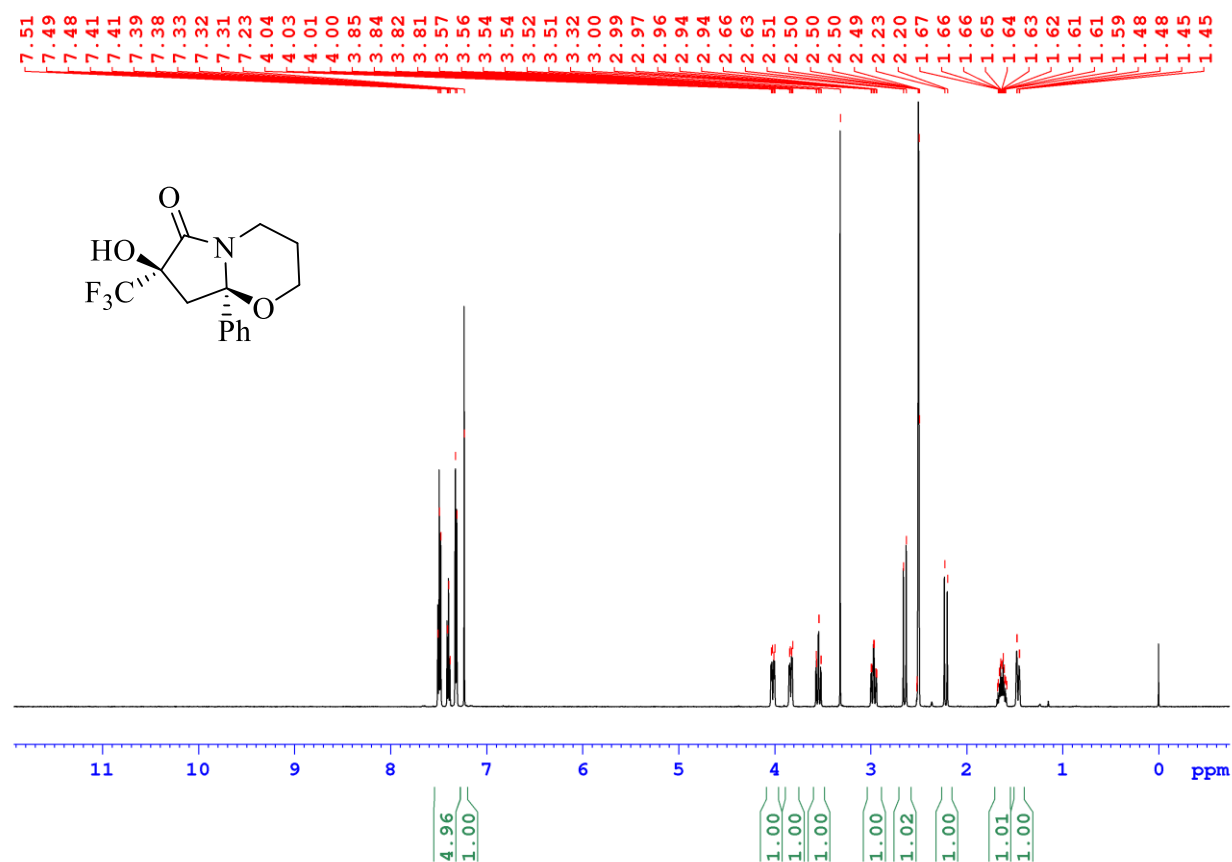


Figure S48. ¹H NMR spectrum of **8d**.

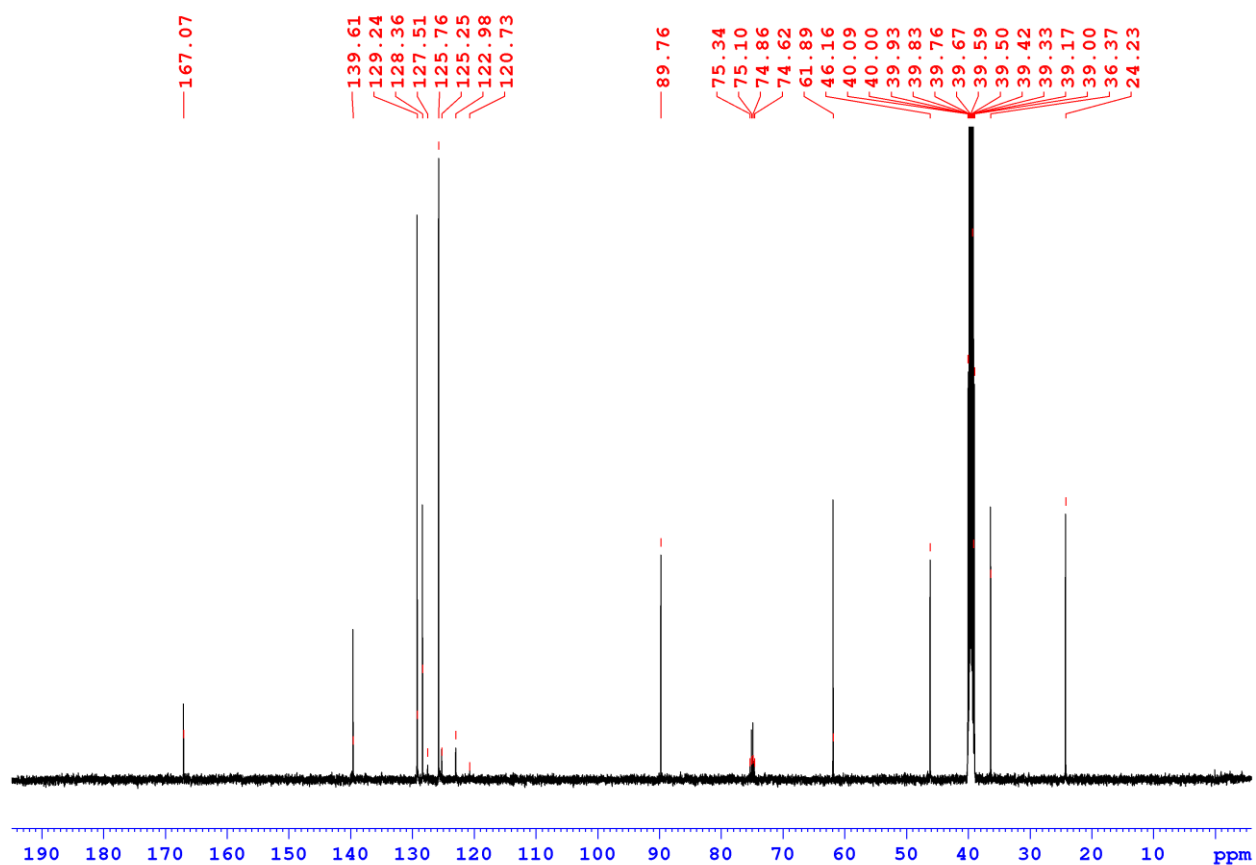


Figure S49. ¹³C NMR spectrum of **8d^t**.

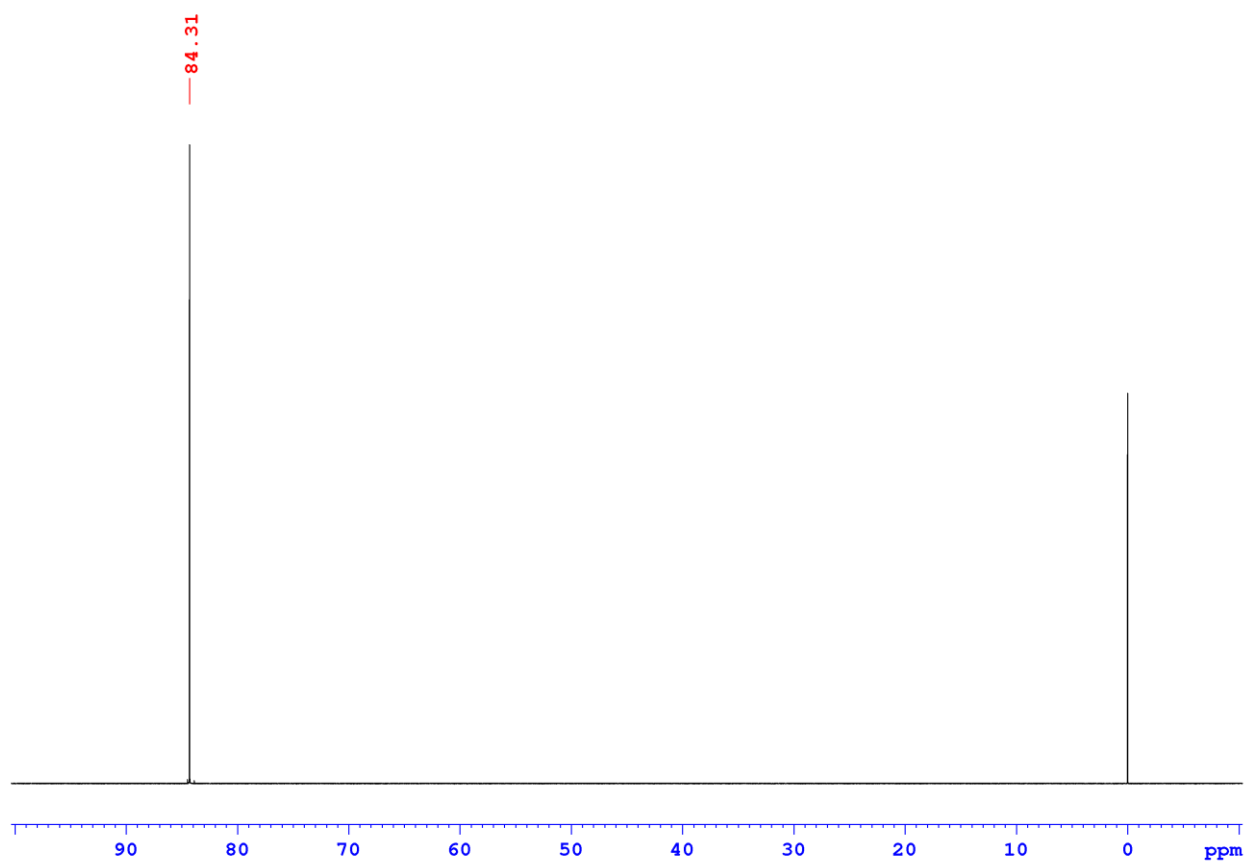


Figure S50. ¹⁹F NMR spectrum of **8d^t**.

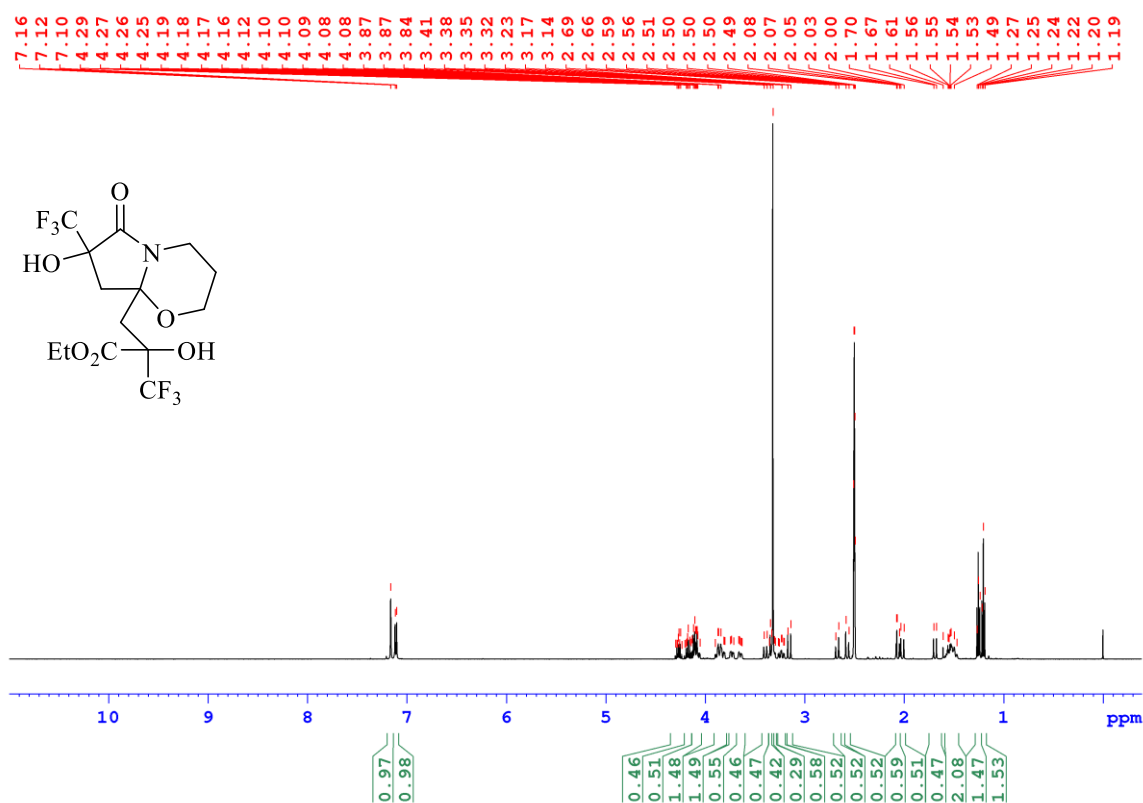


Figure S51. ¹H NMR spectrum of a mixture of **9** : **9'** in the $\approx 1 : 1$.

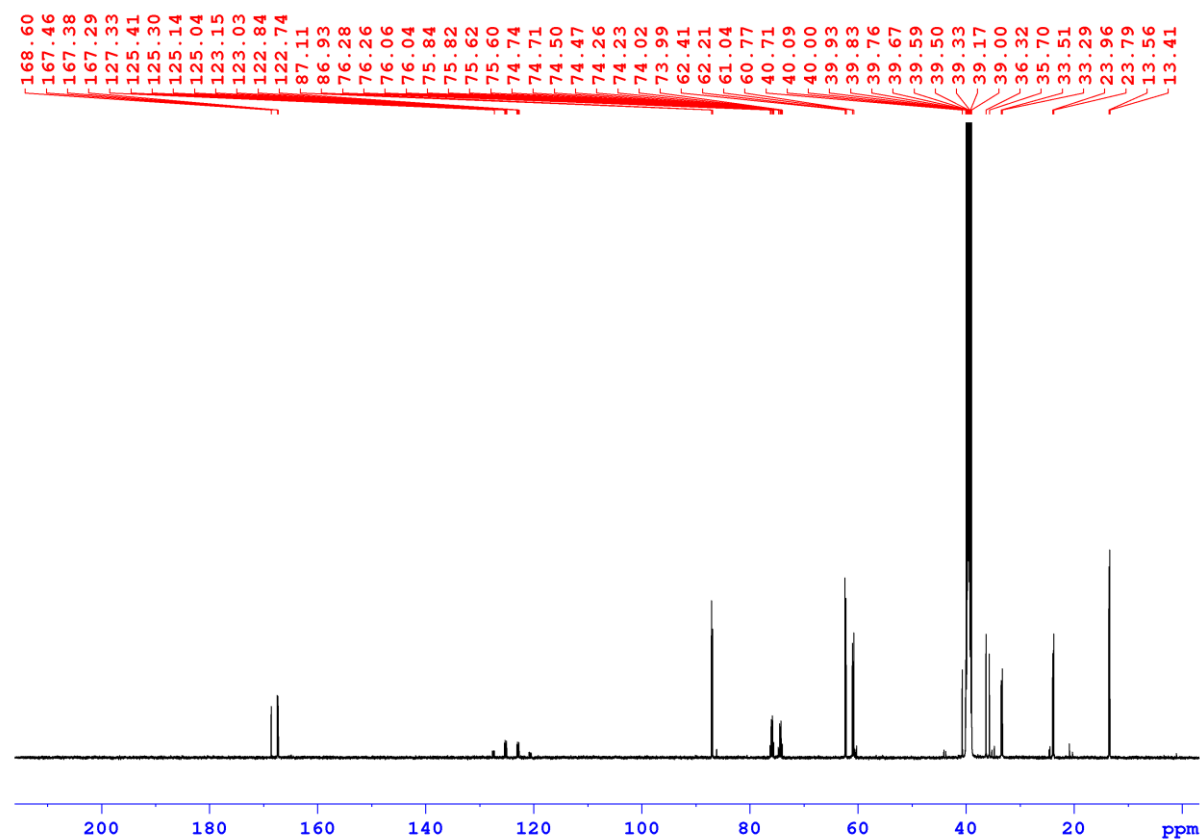


Figure S52. ¹³C NMR spectrum of a mixture of **9** : **9'** in the ratio $\approx 1 : 1$.

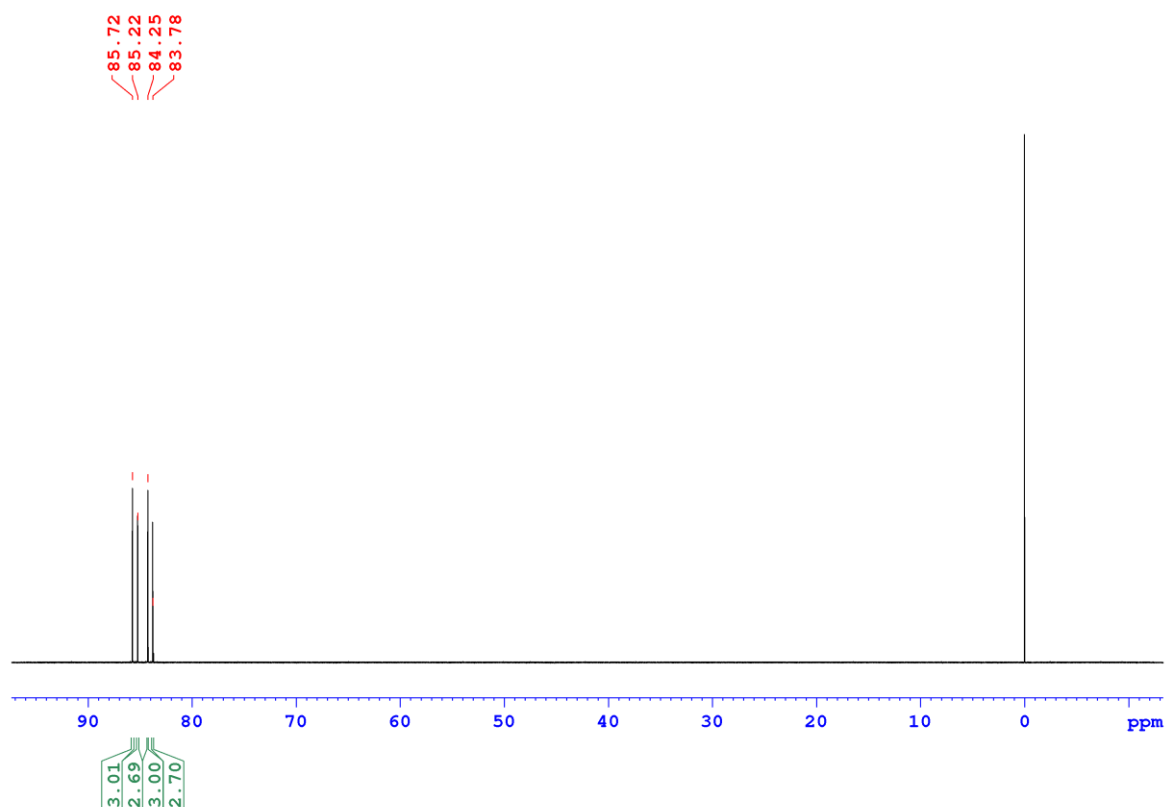


Figure S53. ^{19}F NMR spectrum of mixture of **9** : **9'** in the ratio $\approx 1 : 1$.

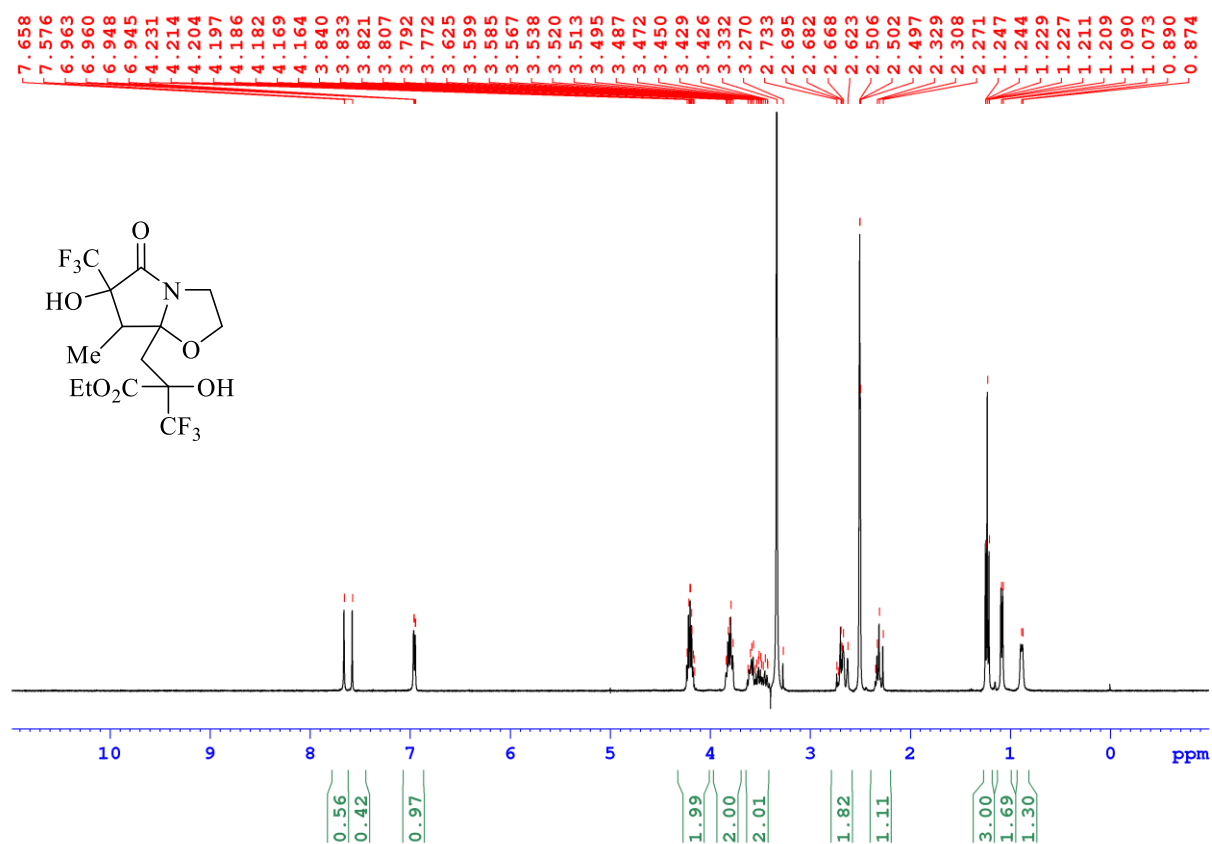


Figure S54. ^1H NMR spectrum of mixture of **10** : **10'** in the ratio 56 : 44.

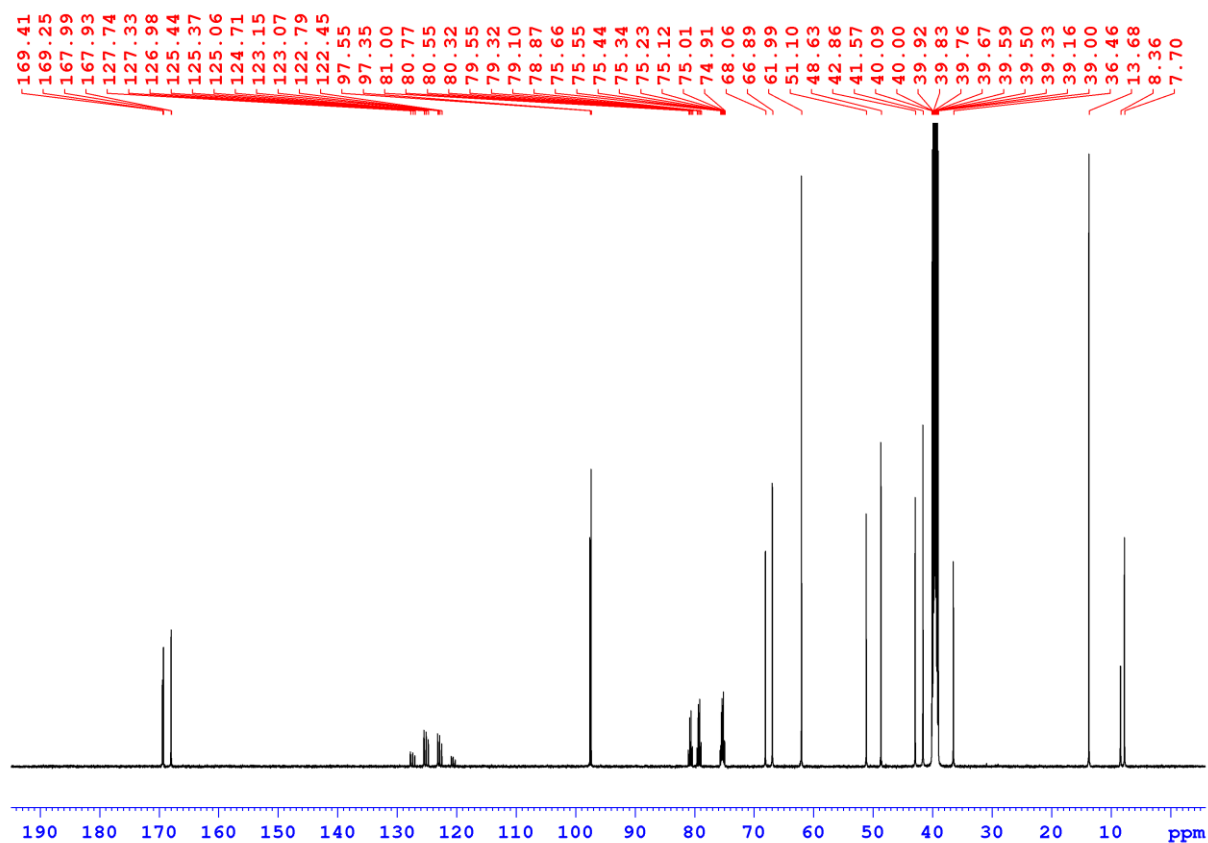


Figure S55. ¹³C NMR spectrum of mixture of **10** : **10'** in the ratio 56 : 44.

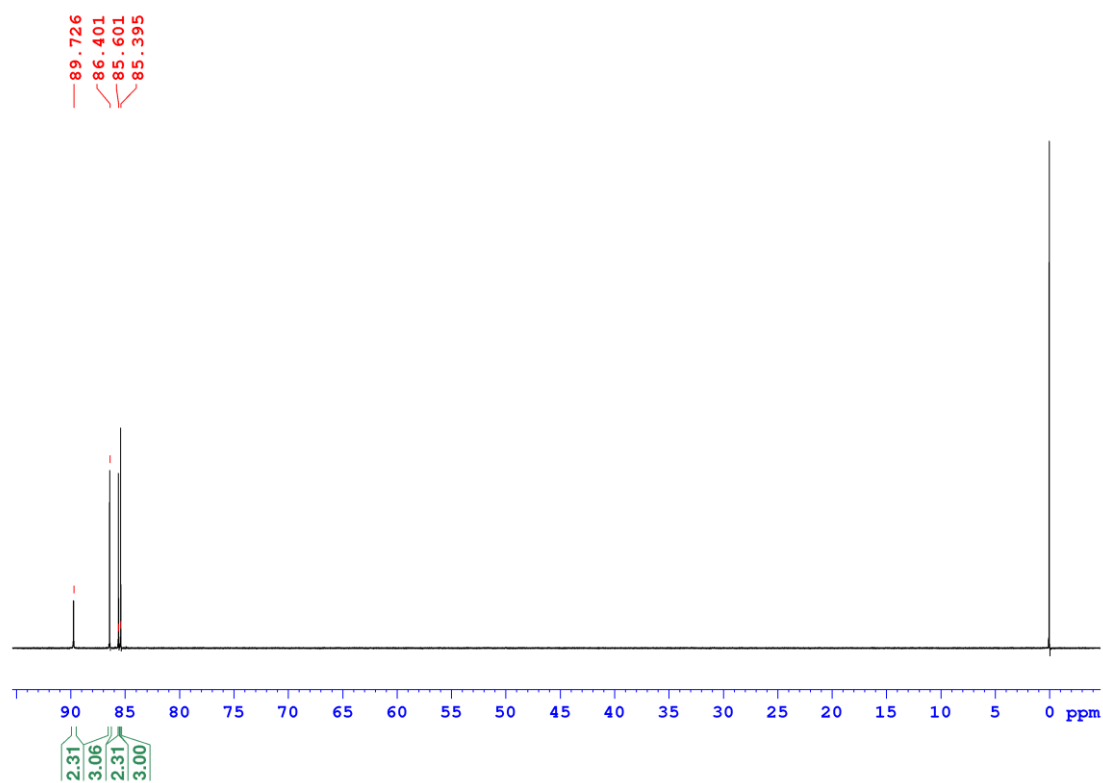


Figure S56. ¹⁹F NMR spectrum of mixture of **10** : **10'** in the ratio 56 : 44.

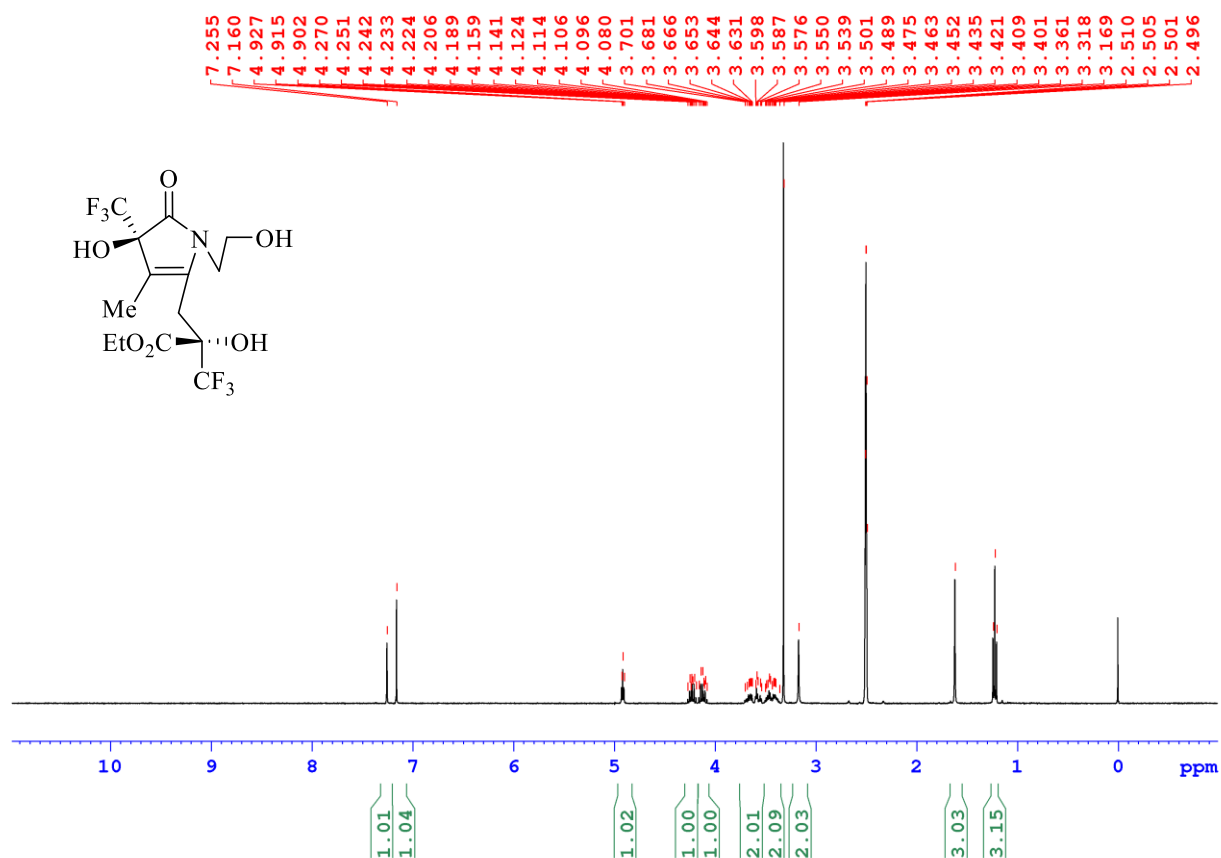


Figure S57. ¹H NMR spectrum of mixture of **11a**.

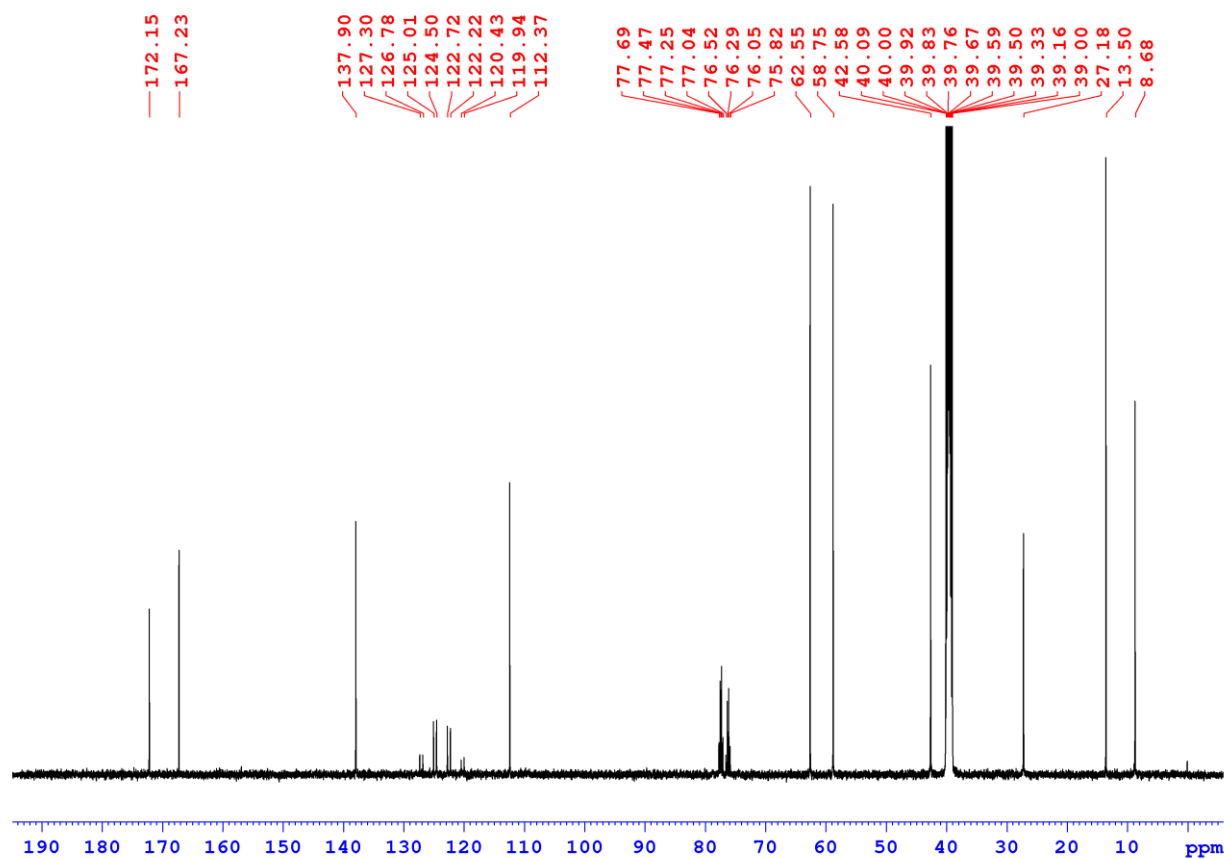


Figure S58. ¹³C NMR spectrum of mixture of **11a**.

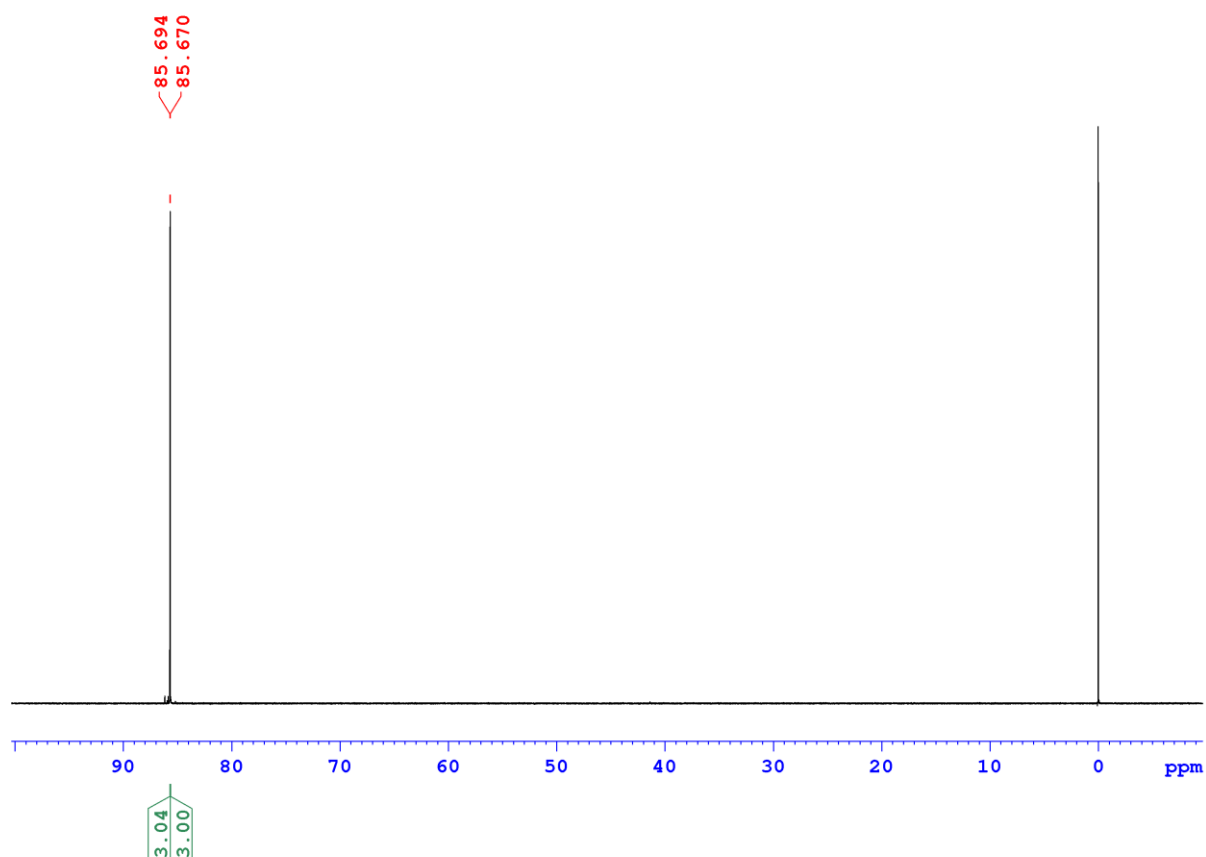


Figure S59. ¹⁹F NMR spectrum of mixture of **11a**.

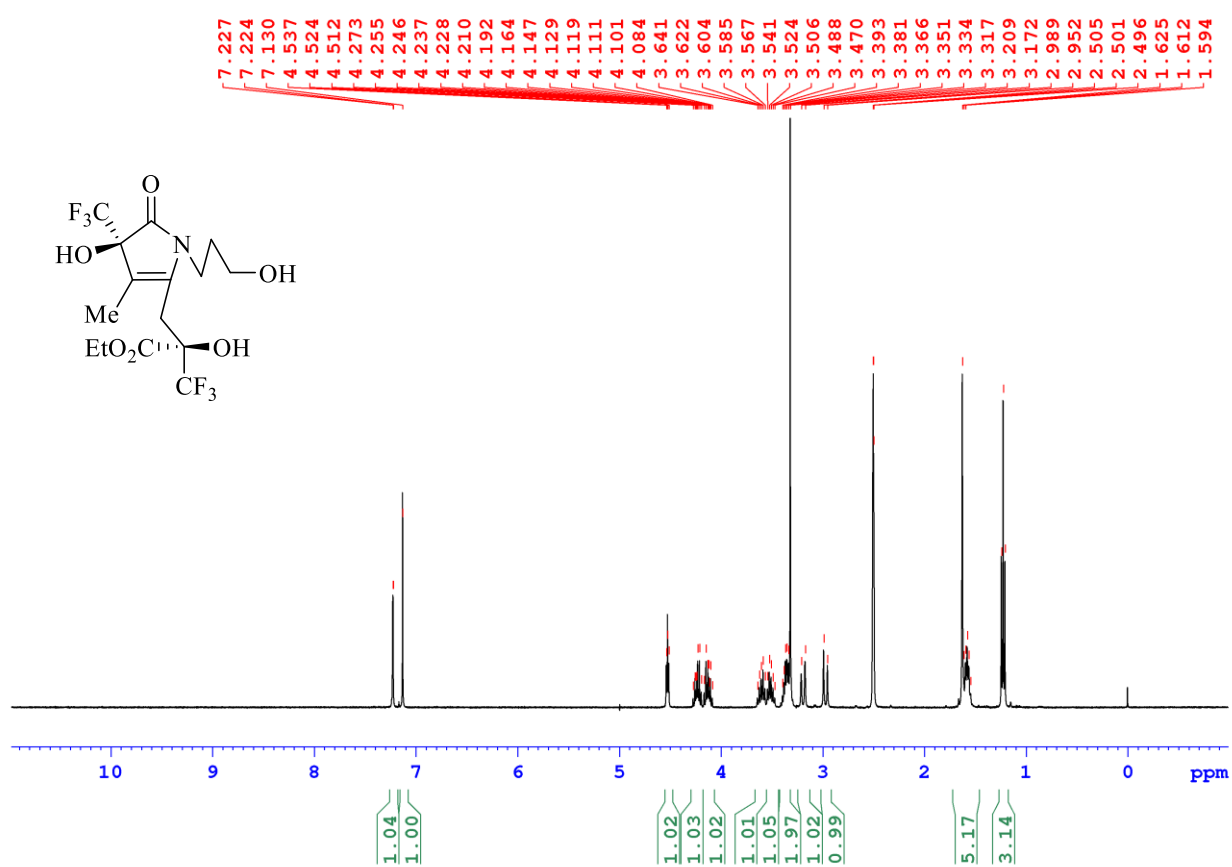


Figure S60. ¹H NMR spectrum of mixture of **11b**.

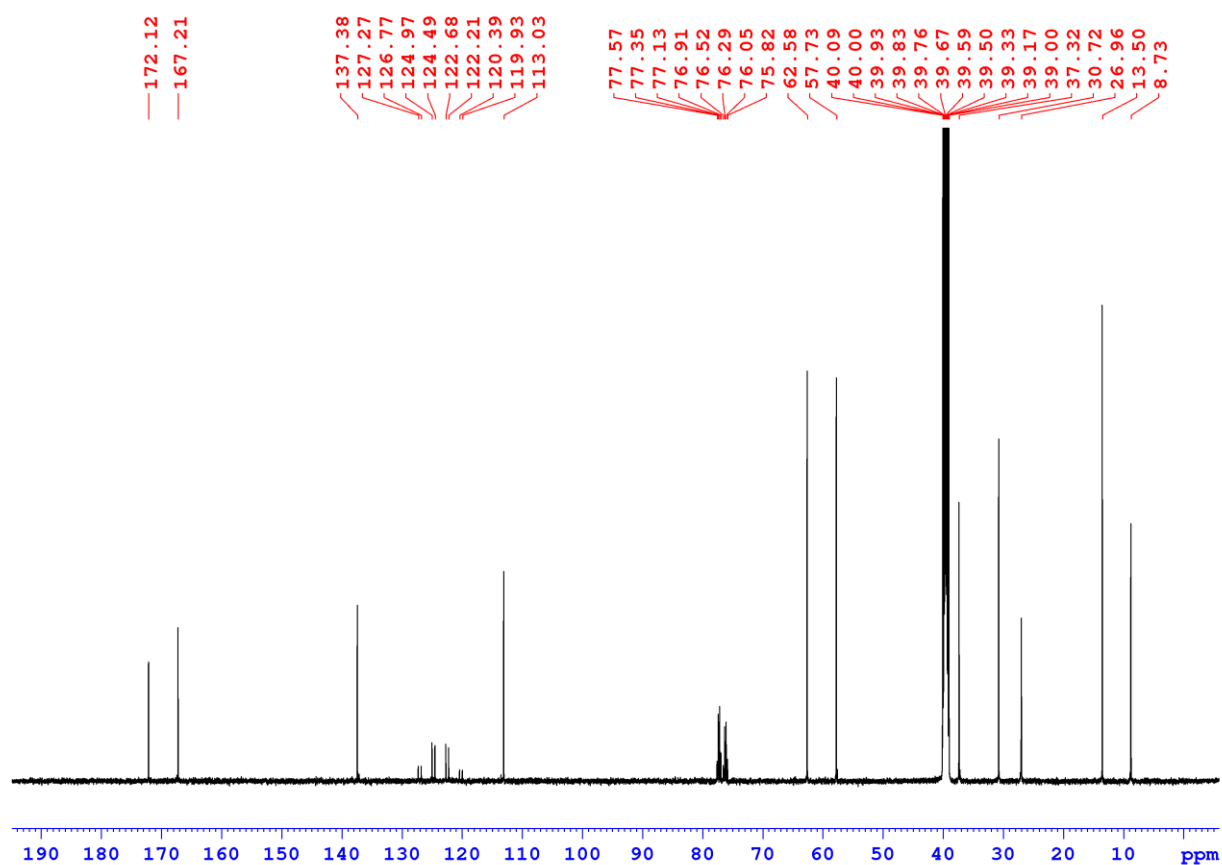


Figure S61. ^{13}C NMR (126 MHz, DMSO-d_6) spectrum of mixture of **11b**.

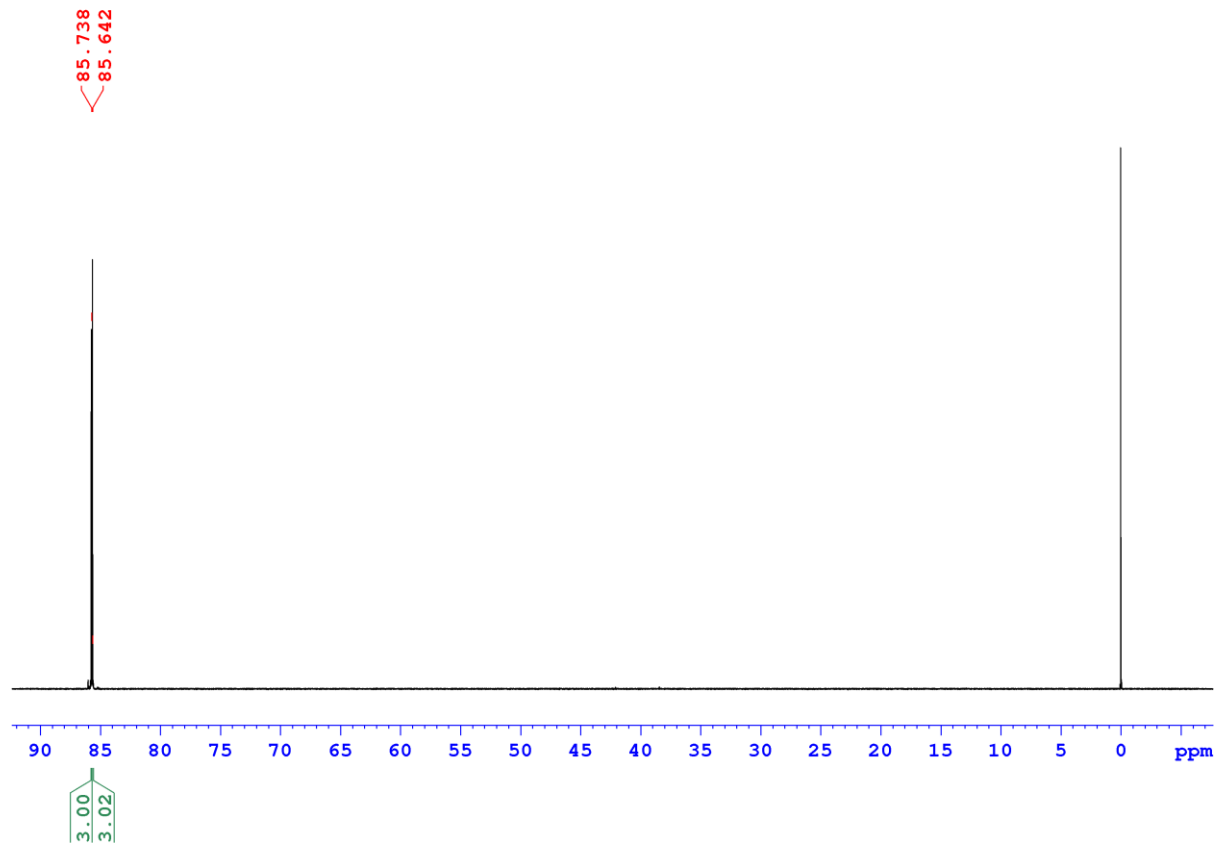


Figure S62. ^{19}F NMR spectrum of mixture of **11b**.

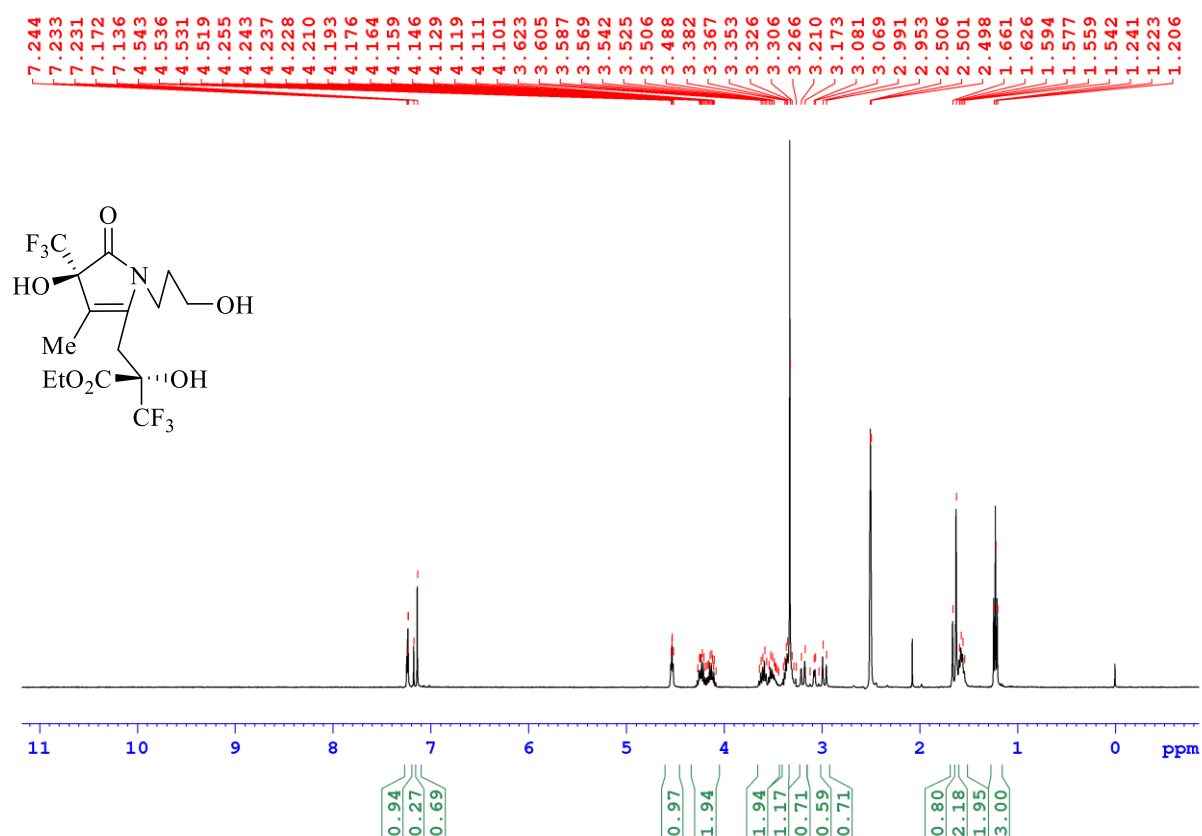


Figure S63. ¹H NMR spectrum of mixture of **11b** : **11b'** in the ratio 72 : 28.

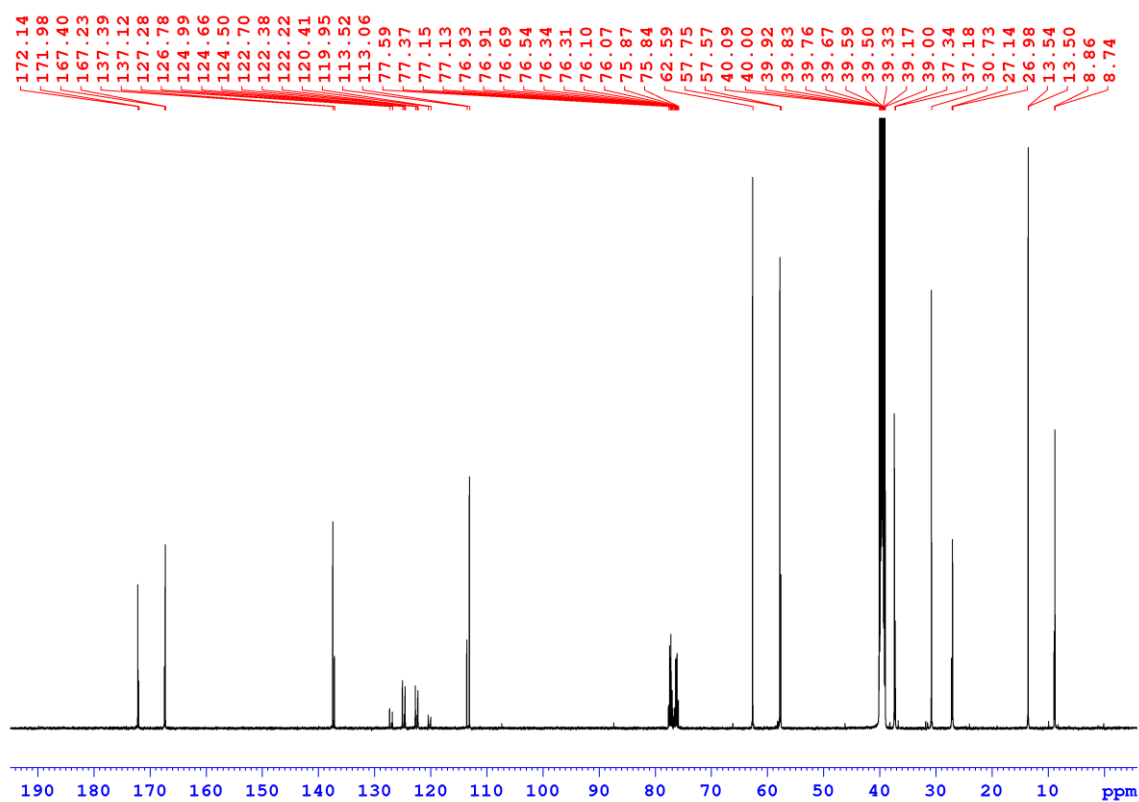


Figure S64. ¹³C NMR spectrum of mixture of **11** : **11'** in the ratio 72 : 28.

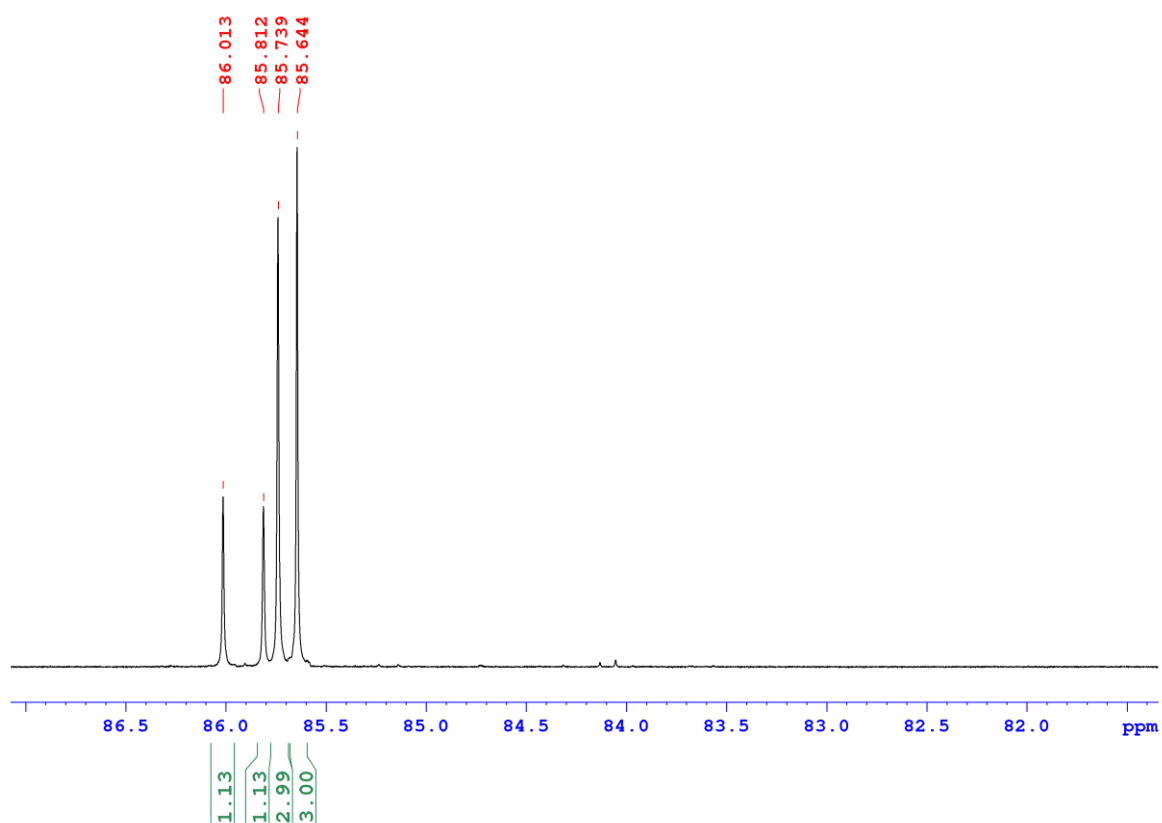


Figure S65. ^{19}F NMR spectrum of mixture of **11** : **11'** in the ratio 72 : 28.