

Efficient Synthesis of 6,6'-Diamido-2,2'-dipicolylamine Ligands for Potential Phosphate Anion Sensing

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Table S1. Condition optimizations for the synthesis of compound **8** with anhydrides and acyl chlorides.

Entry	Product (R ₁)	Reactant	Solvent	Temp. (°C)	Time (h)	Yield ^a (%)
1	9a (-CH ₃)	(CH ₃ CO) ₂ O	CH ₂ Cl ₂	40	3h	N.D.
2			THF	65	3h	N.D.
3			Toluene	110	3h	35
4			Neat	140	3h	75
5			Neat	140	4h	96
6			Neat	140	5h	68
7	9a (-CH ₃)	CH ₃ COCl	Neat	51	4h	20
8	9a (-CH ₃)	CH ₃ COCl	Neat	140	4h	89
9	9b (-Ph)	PhCOCl	CH ₂ Cl ₂	40	3h	N.D.
10			THF	65	3h	N.D.
11			Toluene	110	3h	28
12			Neat	140	3h	56
13			Neat	140	6h	78
14			Neat	140	8h	95
15	9c (-CF ₃)	(CF ₃ CO) ₂ O	Neat	RT	3h	N.D.
16			Neat	40	3h	N.D.

^a Isolated yields of the corresponding products. (N.D. = not detected; RT = room temperature.)

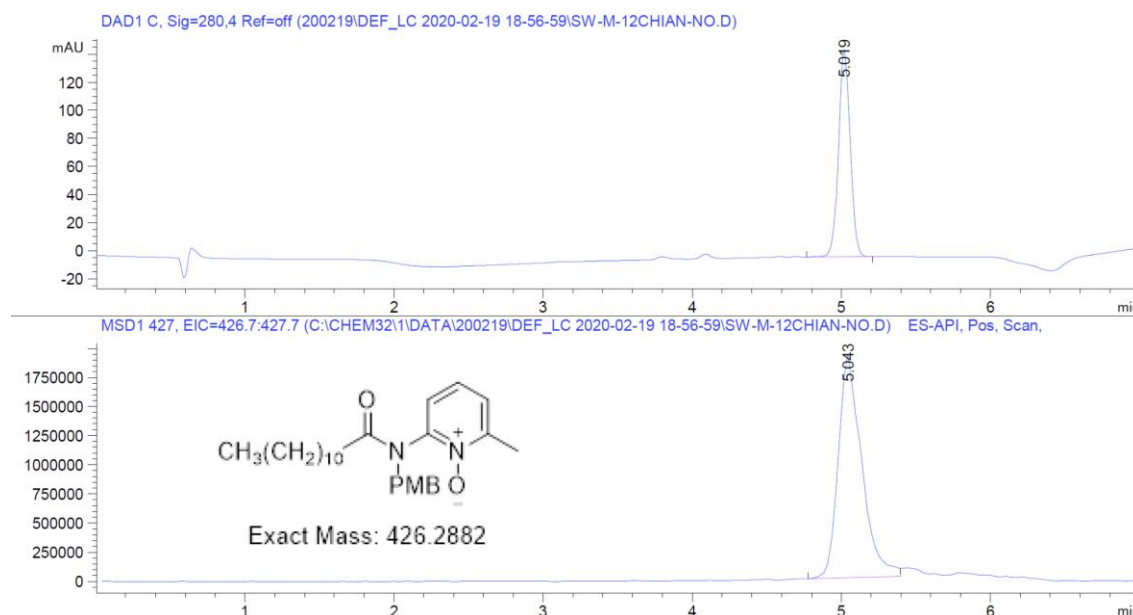


Figure S1. The LCMS spectra of intermediate.

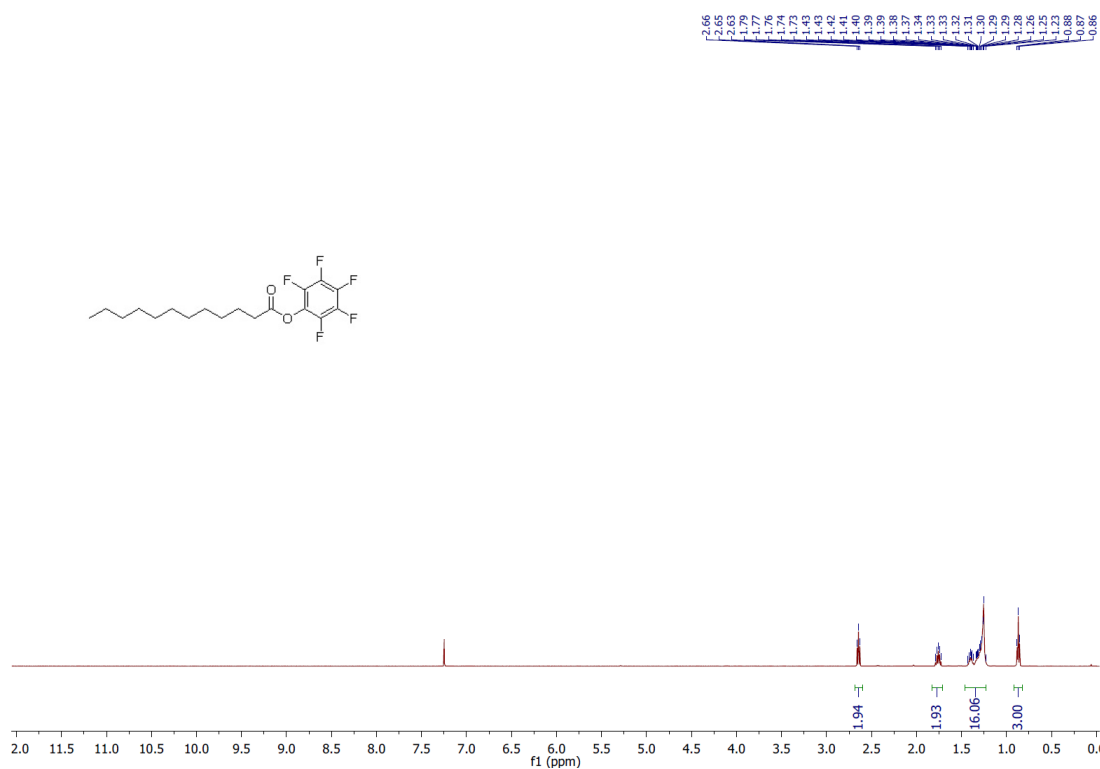


Figure S2. The ¹H spectra of compound **13**.

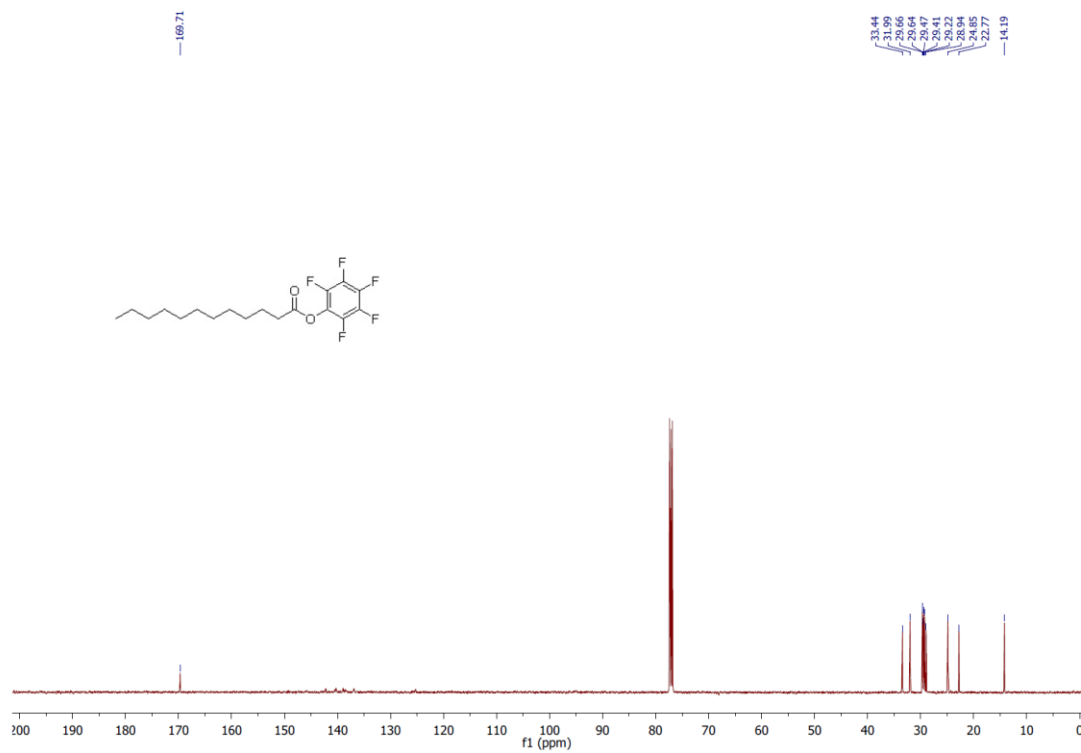


Figure S3. The ¹³C spectra of compound **13**.

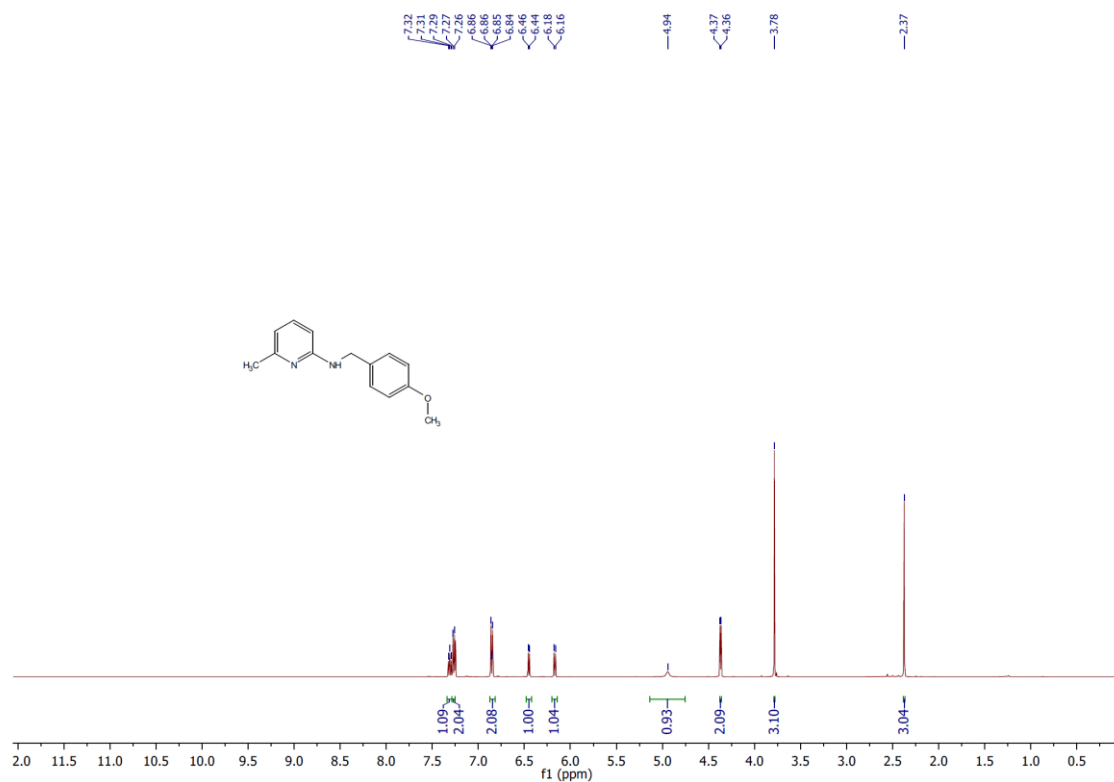


Figure S4. The ¹H spectra of compound 7.

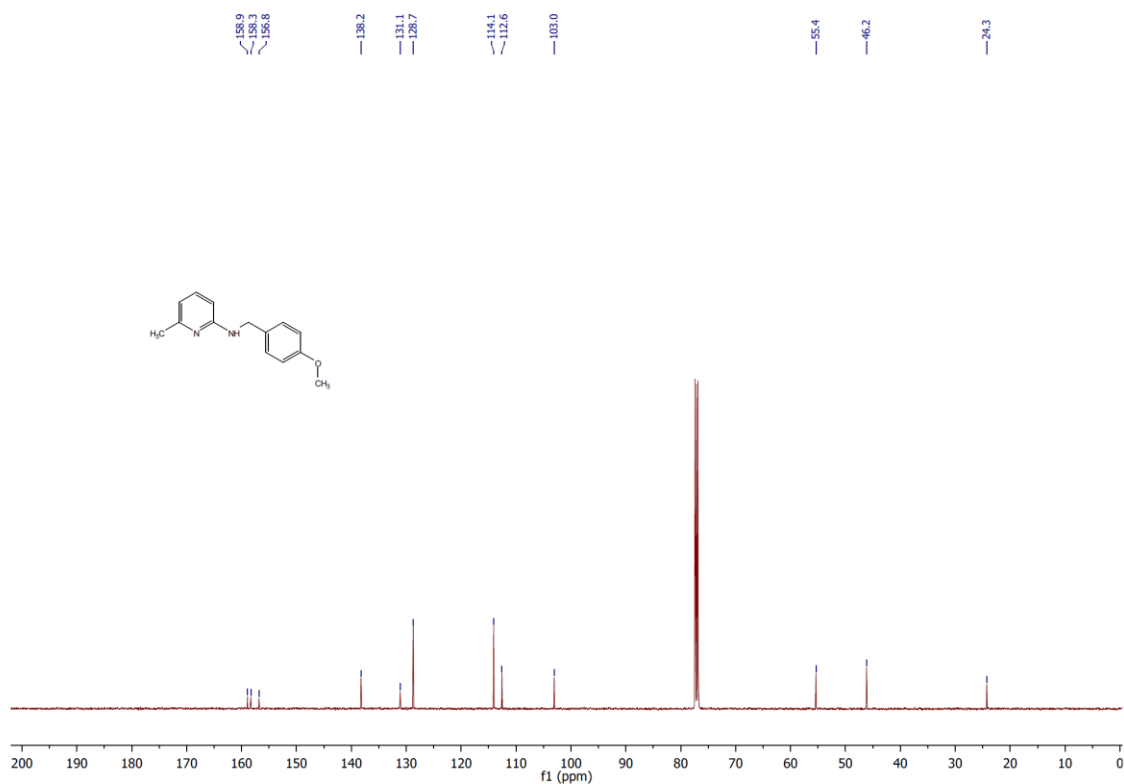


Figure S5. The ¹³C spectra of compound 7.

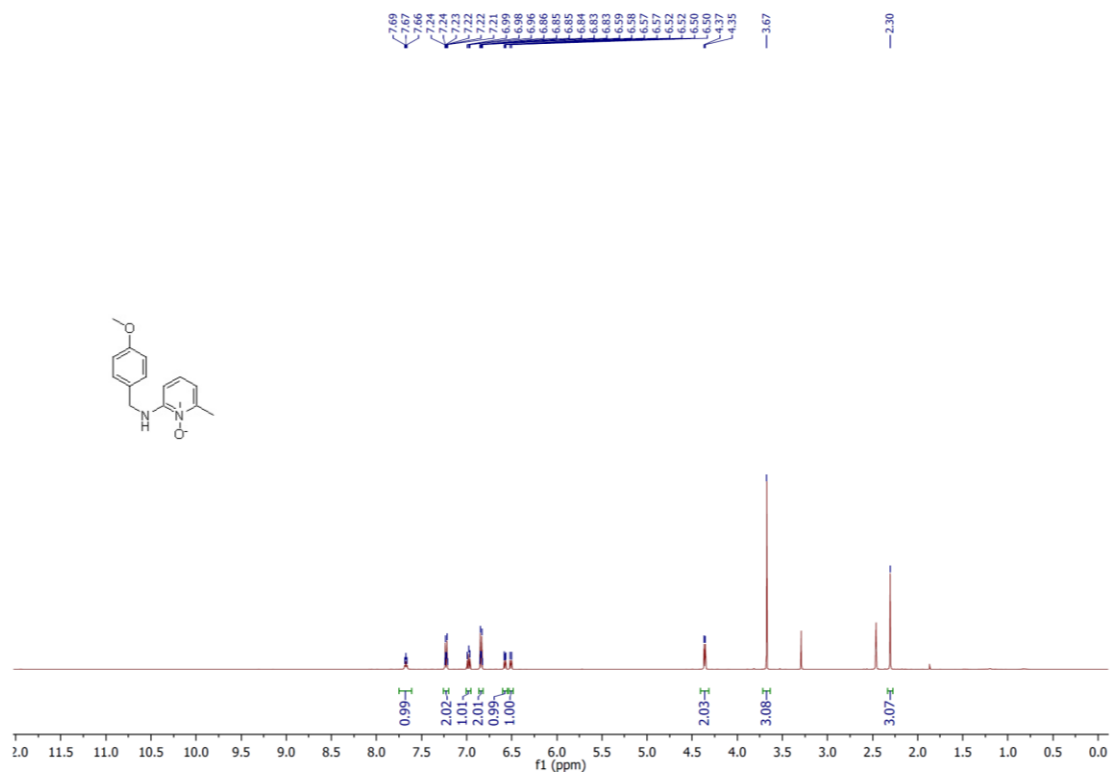


Figure S6. The ¹H spectra of compound **8**.

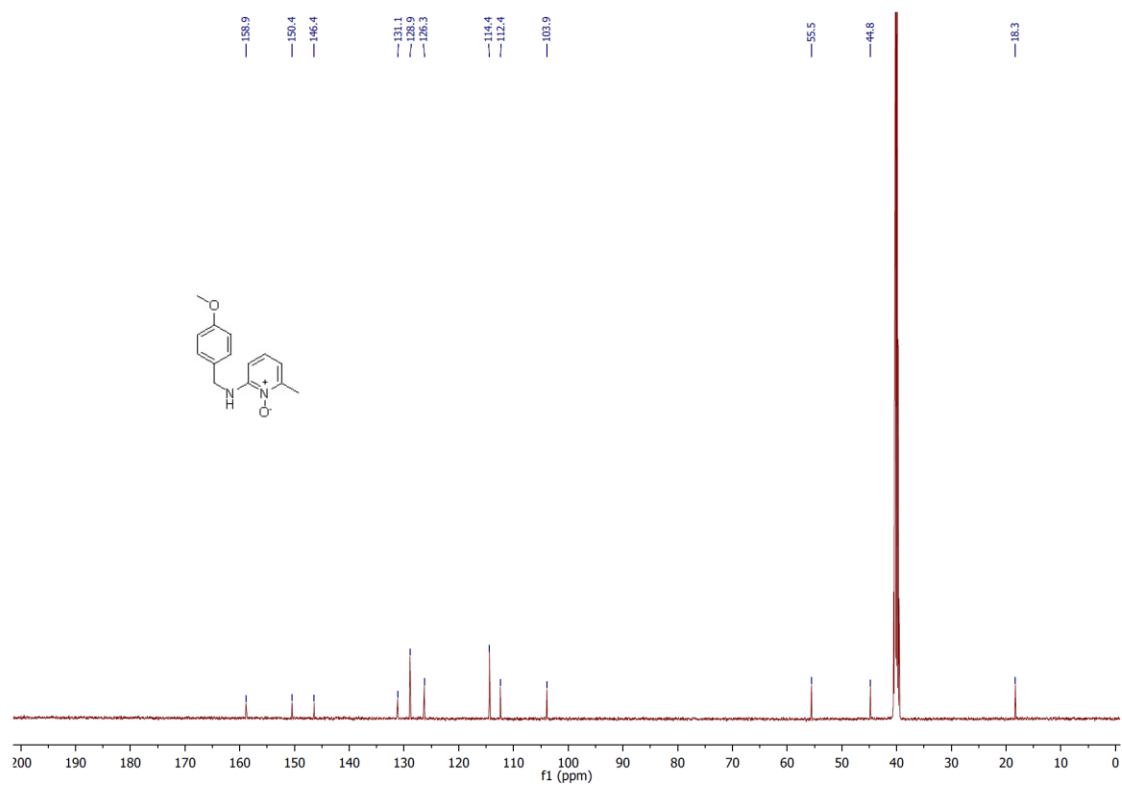


Figure S7. The ¹³C spectra of compound **8**.

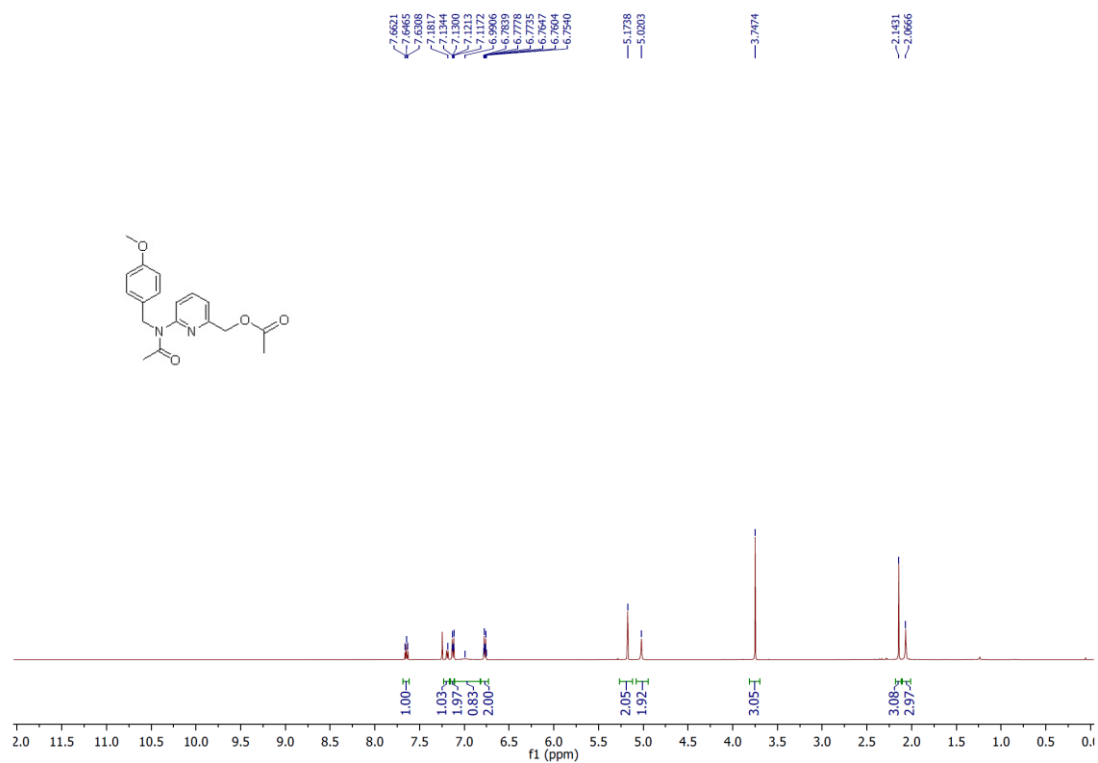


Figure S8. The ¹H spectra of compound **9a**.

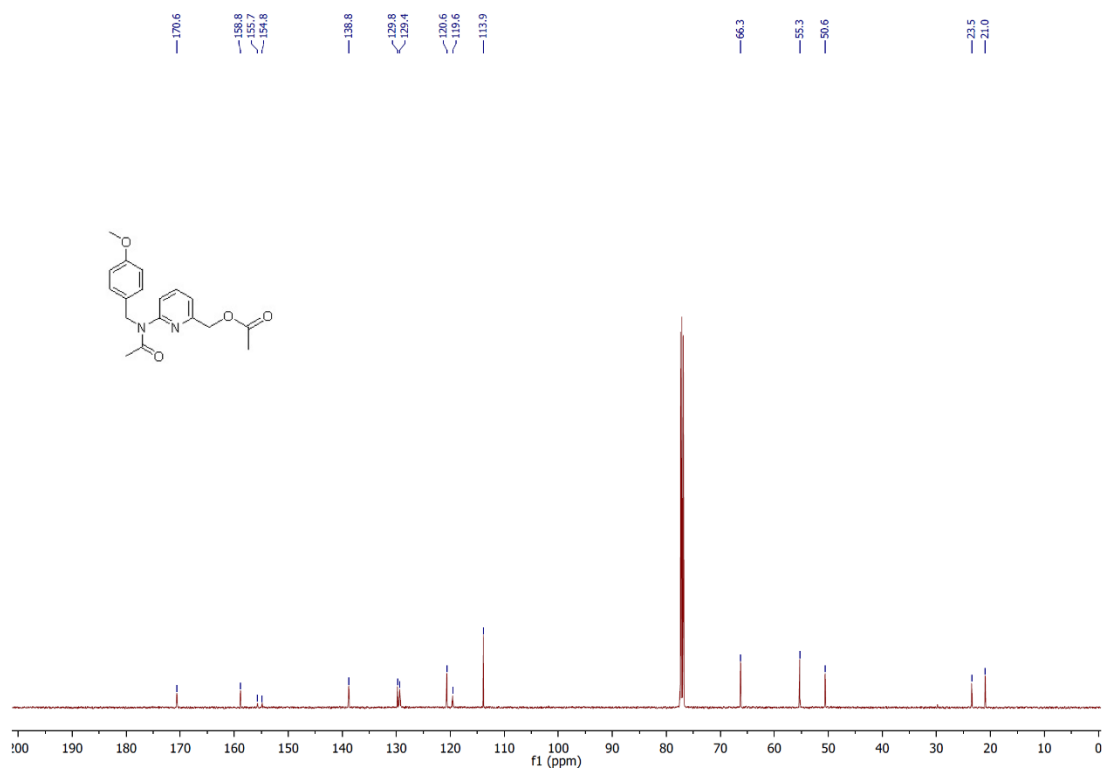


Figure S9. The ¹³C spectra of compound **9a**.

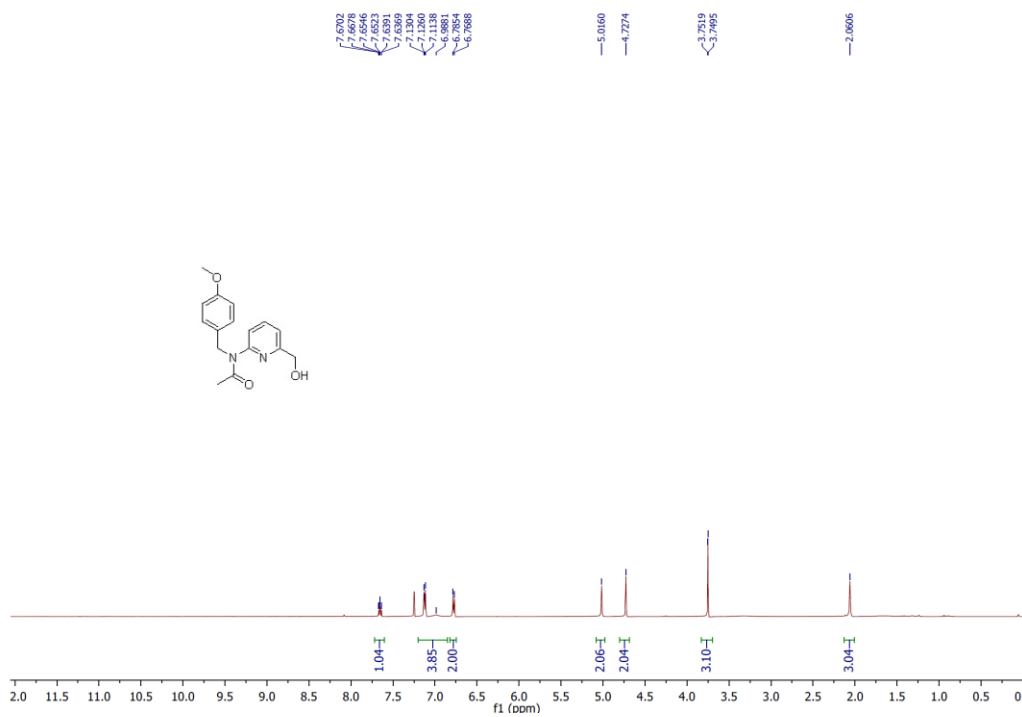


Figure S10. The ¹H spectra of compound **10a**.

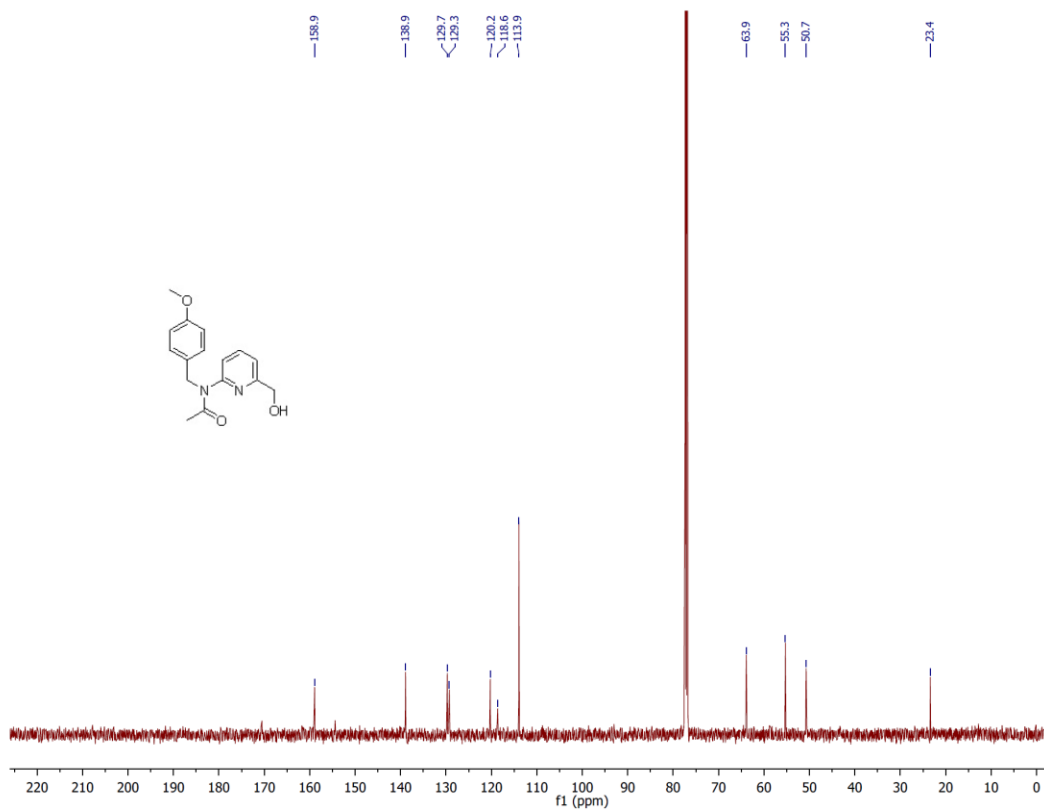


Figure S11. The ¹³C spectra of compound **10a**.

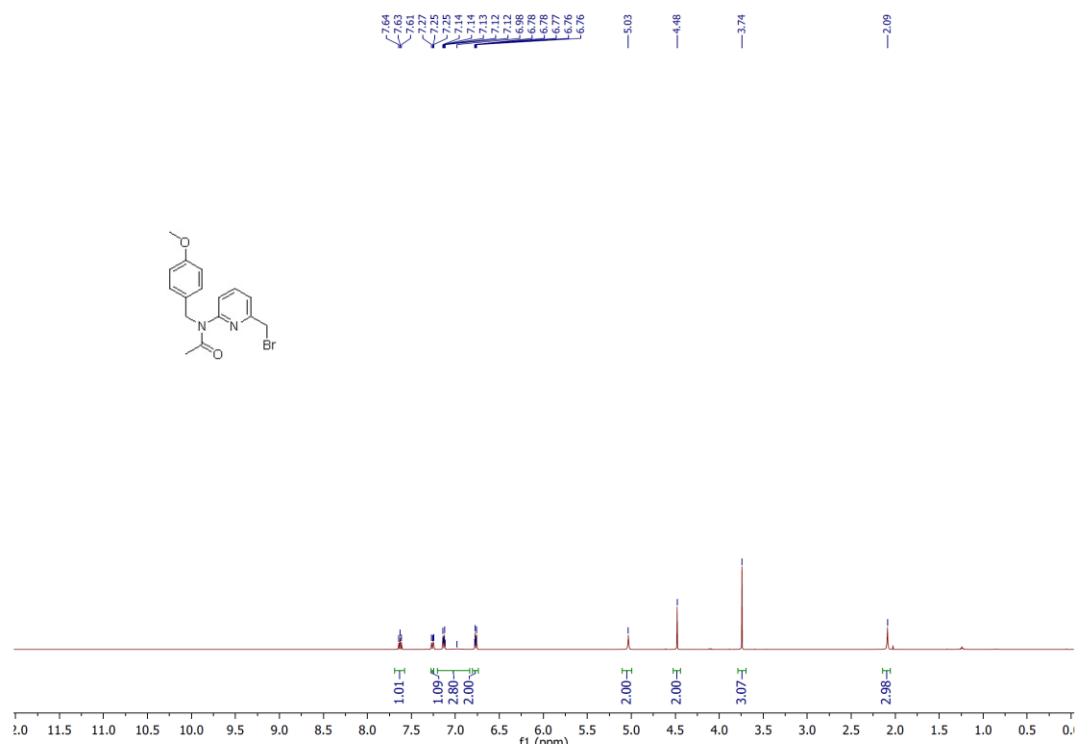


Figure S12. The ¹H spectra of compound **11a**.

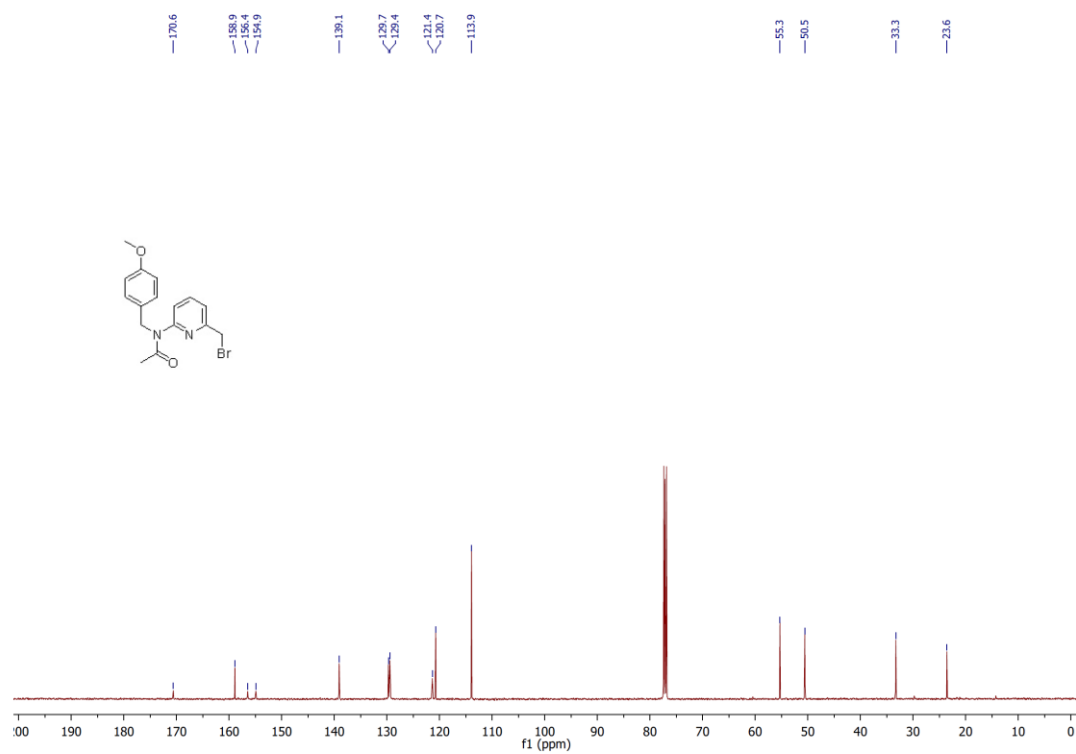


Figure S13. The ¹³C spectra of compound **11a**.

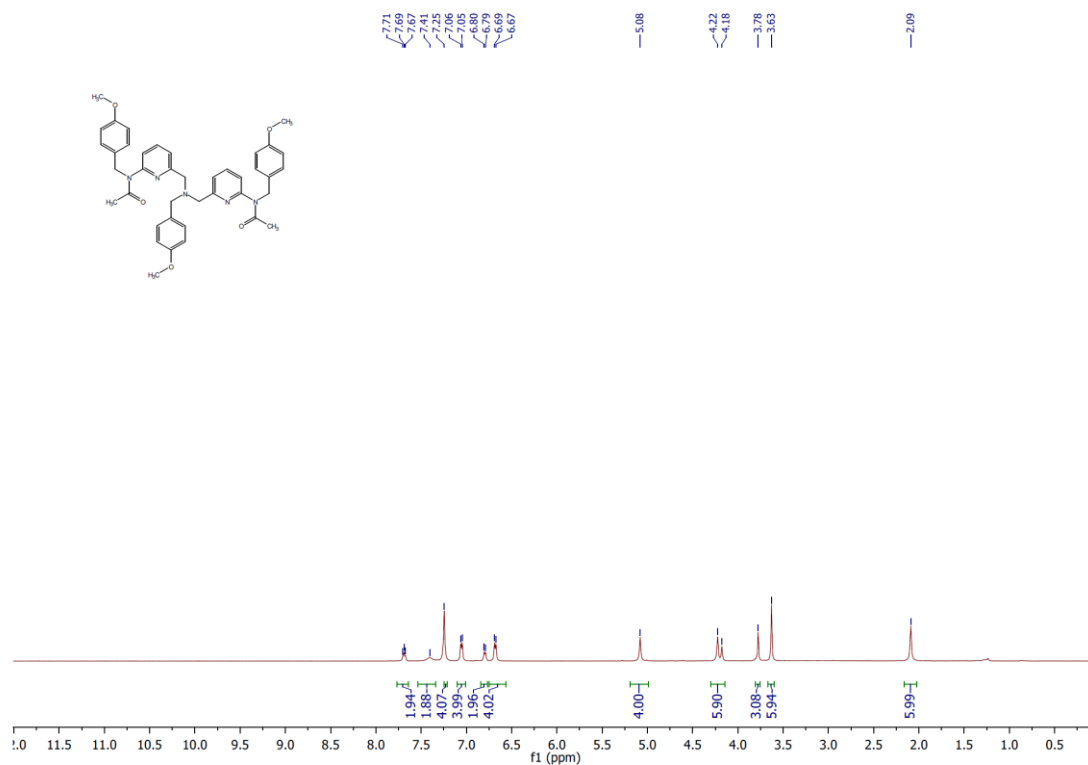


Figure S14. The ^1H spectra of compound **12a**.

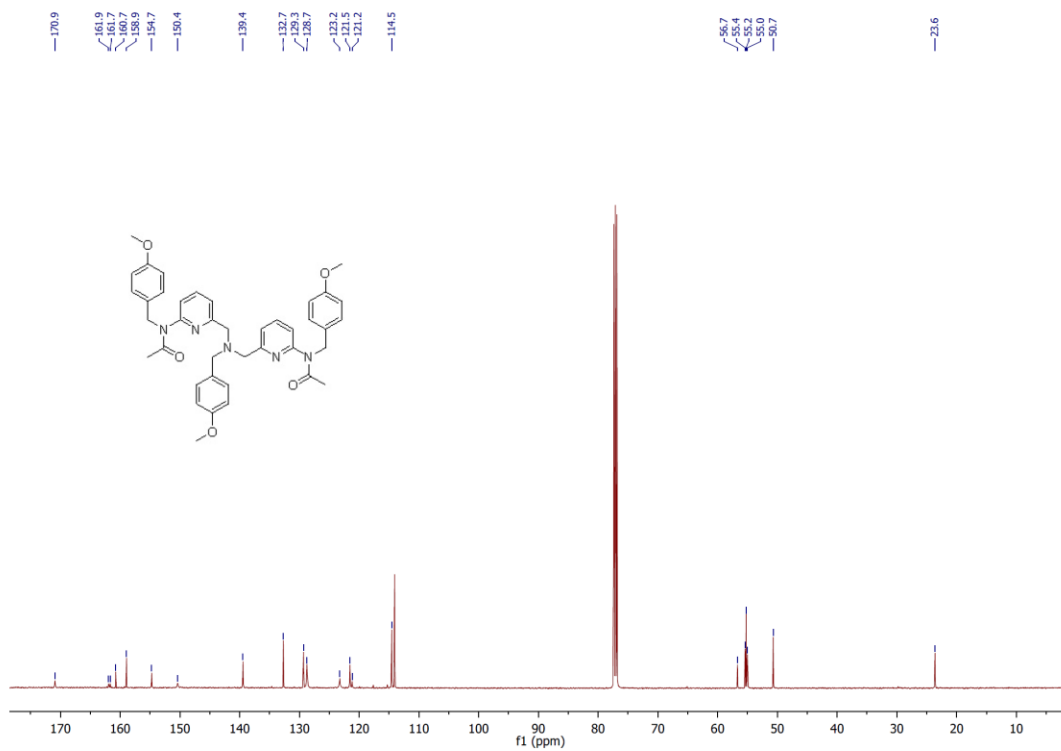


Figure S15. The ^{13}C spectra of compound **12a**.

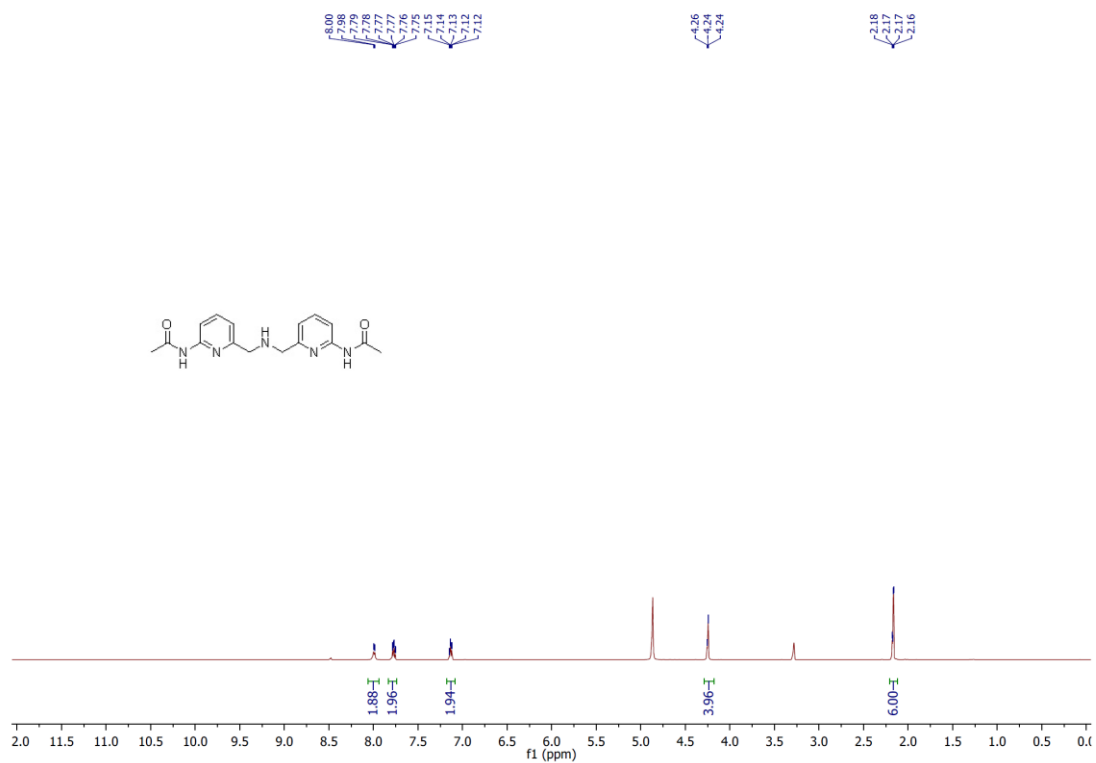


Figure S16. The ¹H spectra of compound **6a**.

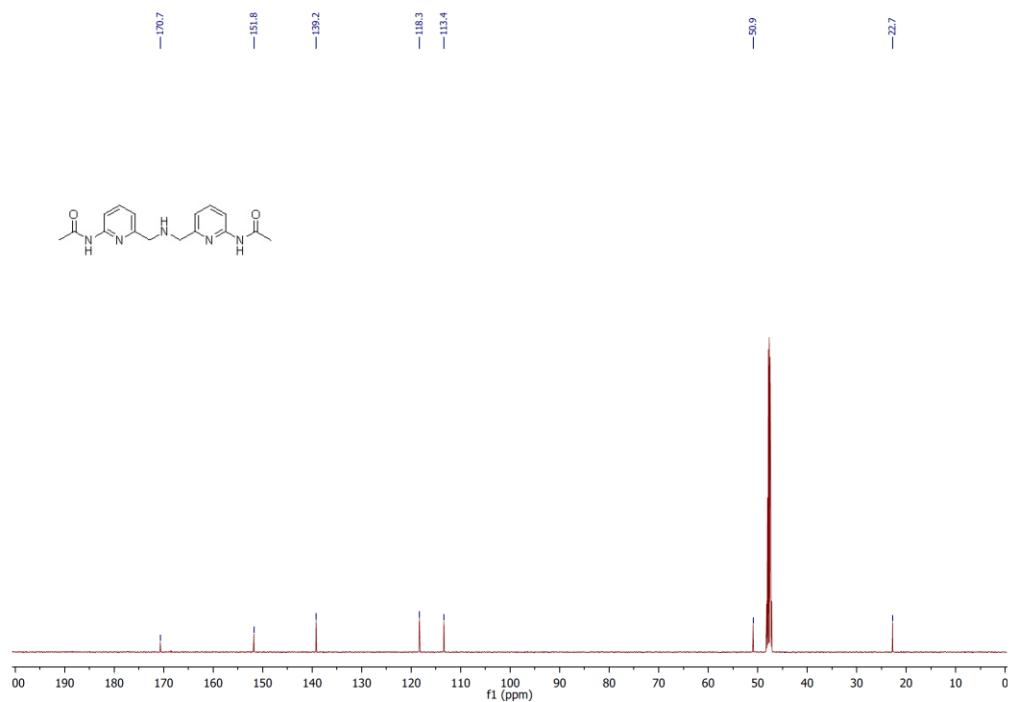


Figure S17. The ¹³C spectra of compound **6a**.

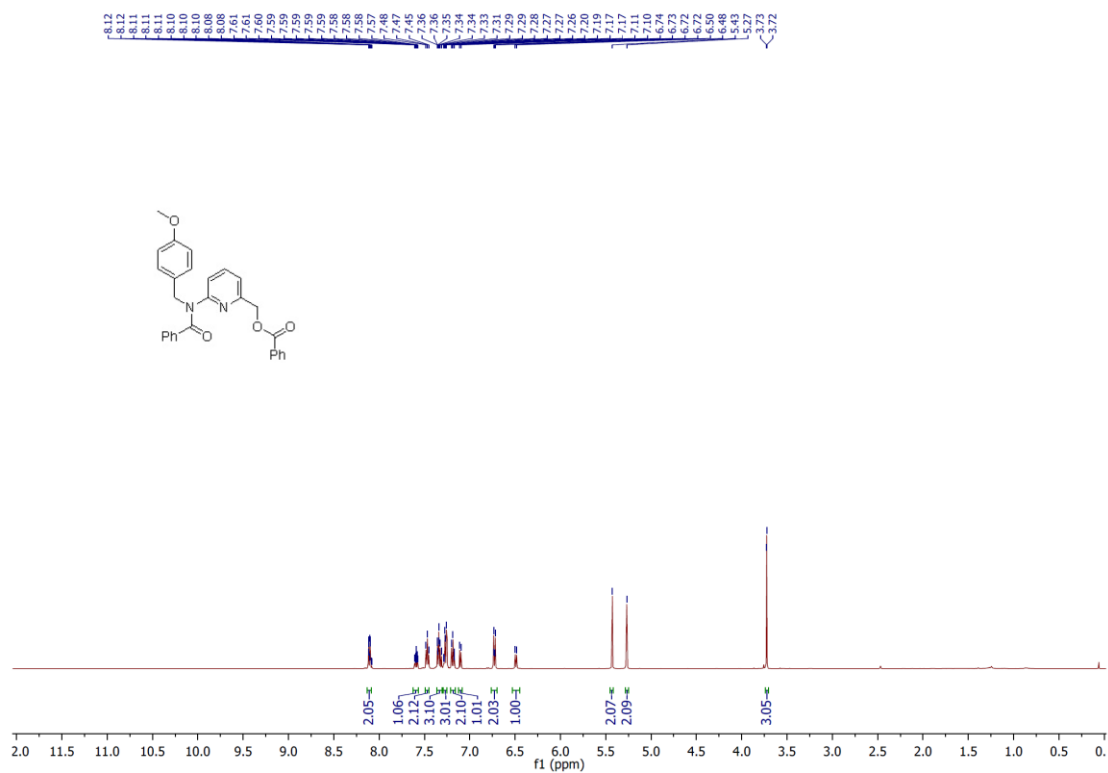


Figure S18. The ¹H spectra of compound **9b**.

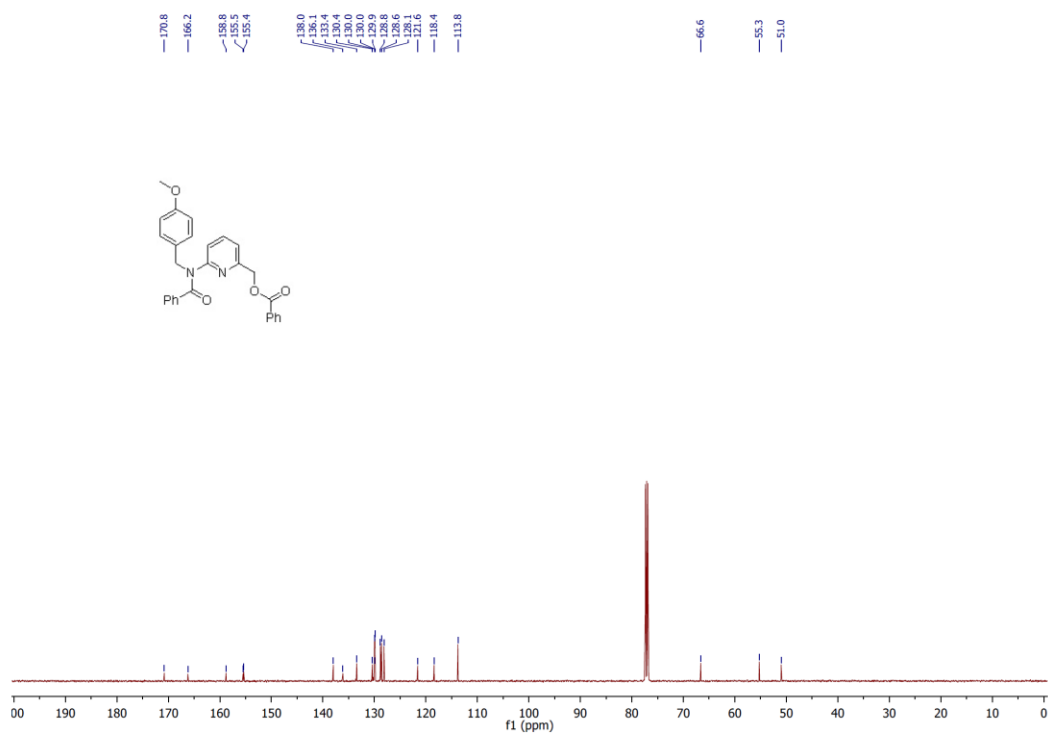


Figure S19. The ¹³C spectra of compound **9b**.

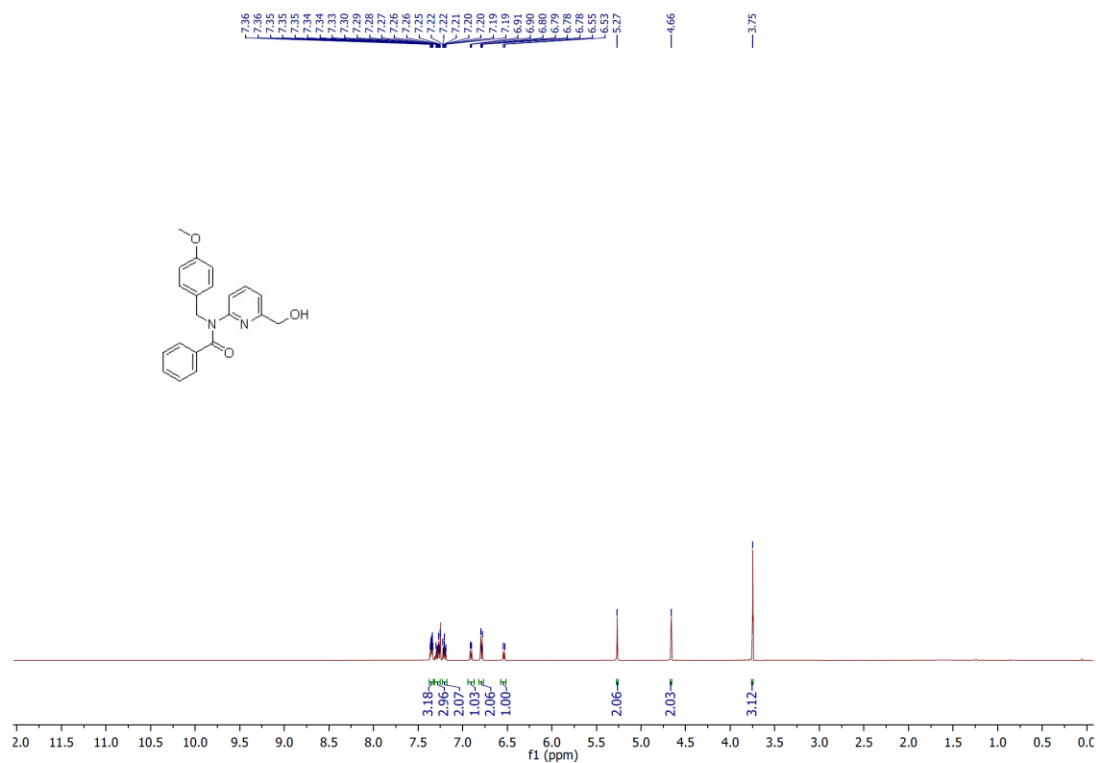


Figure S20. The ¹H spectra of compound **10b**.

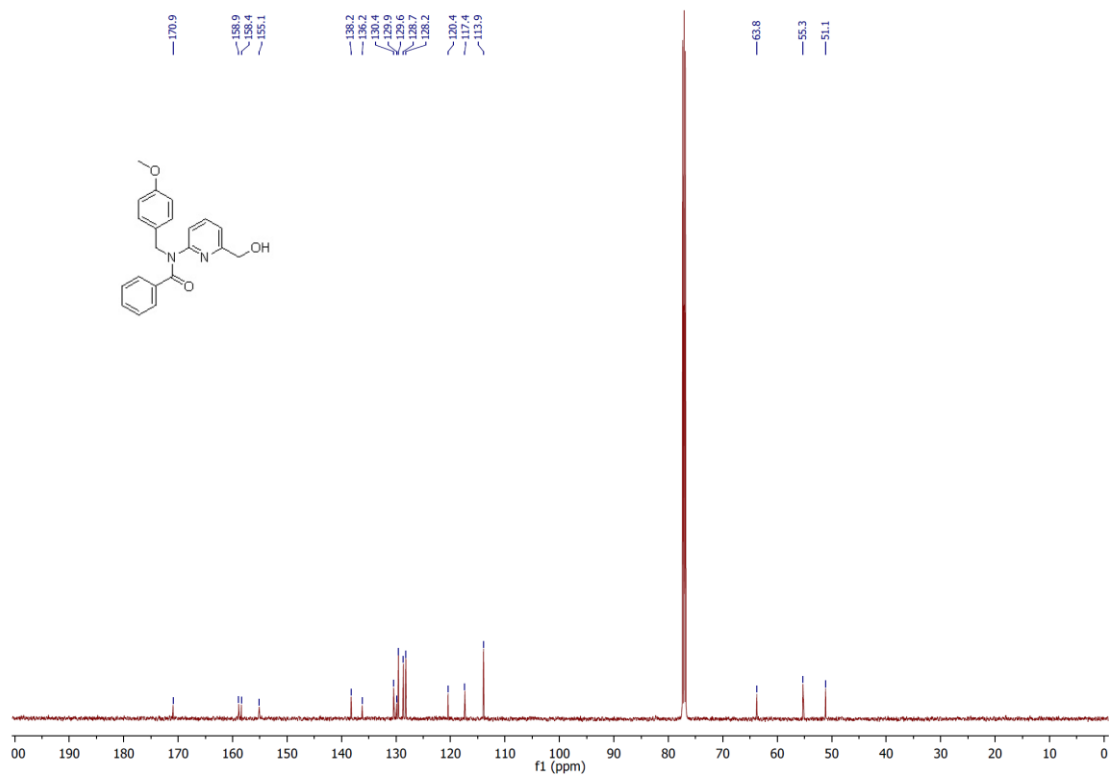


Figure S21. The ¹³C spectra of compound **10b**.

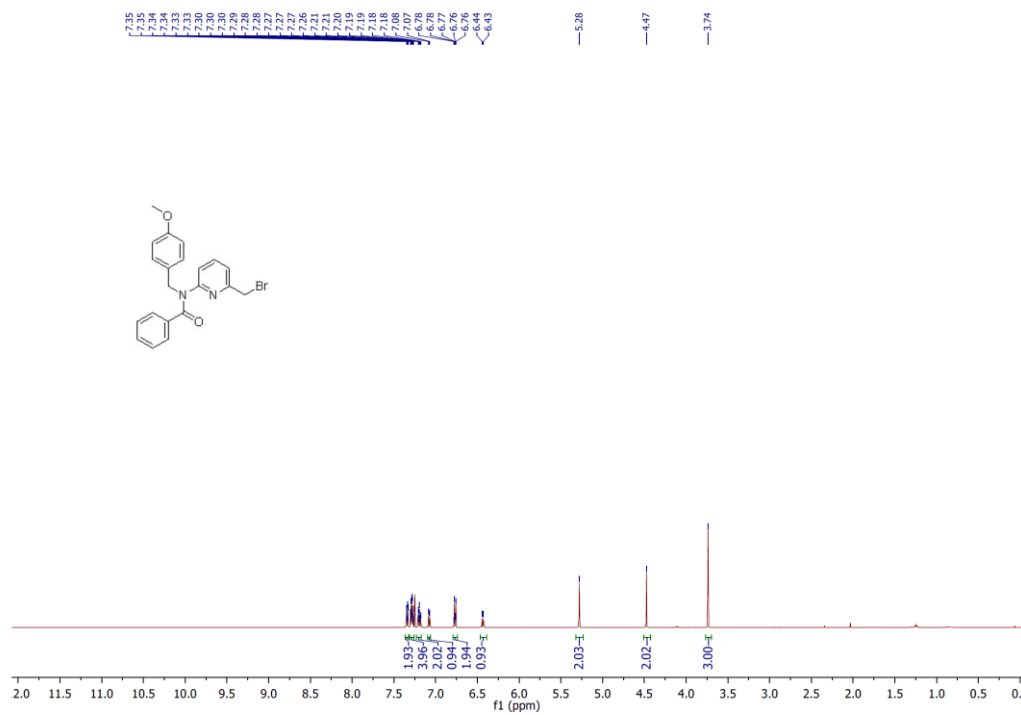


Figure S22. The ¹H spectra of compound **11b**.

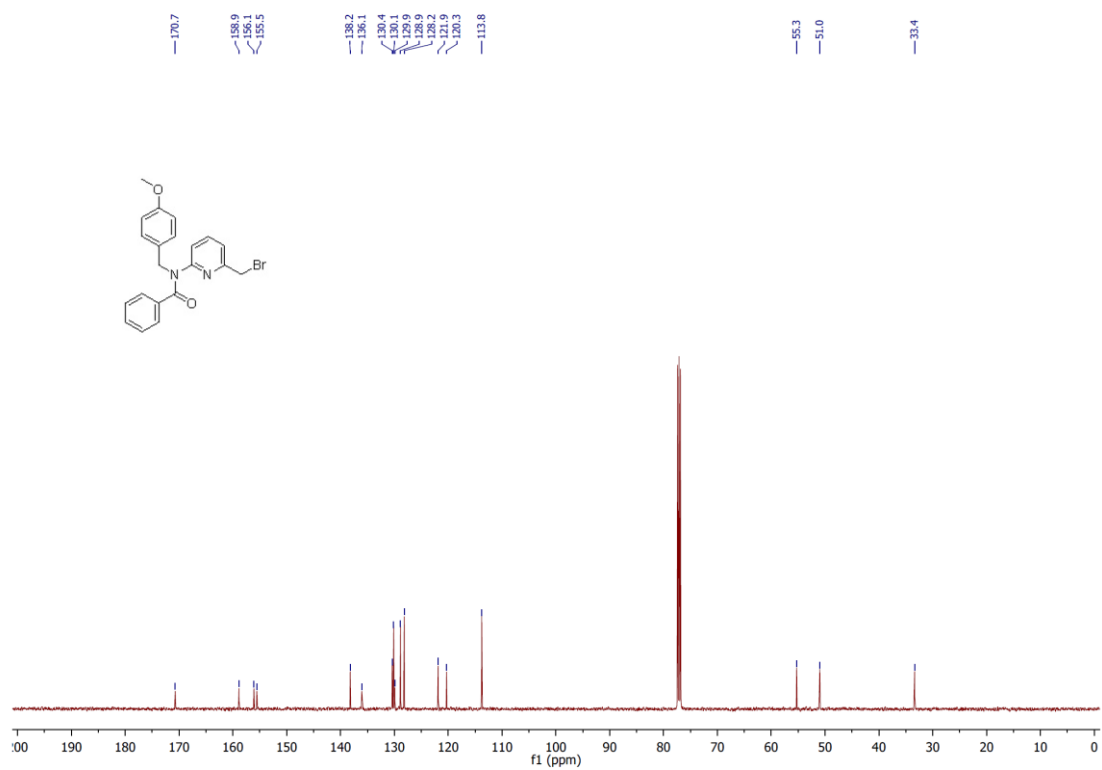


Figure S23. The ¹³C spectra of compound **11b**.

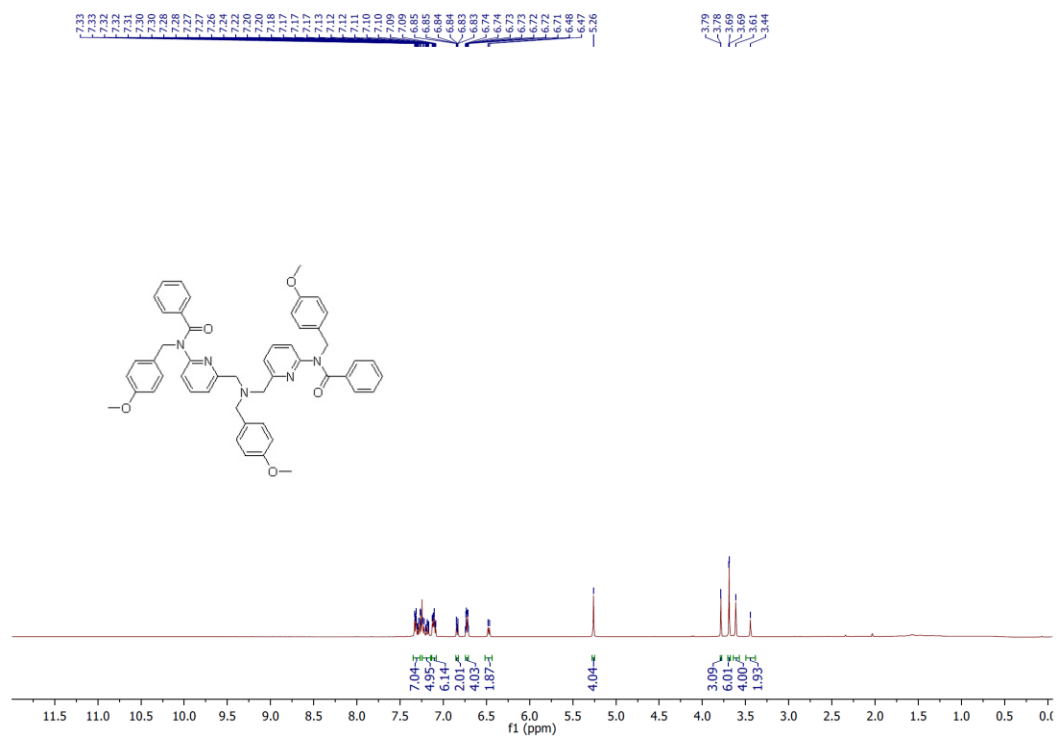


Figure S24. The ¹H spectra of compound **12b**.

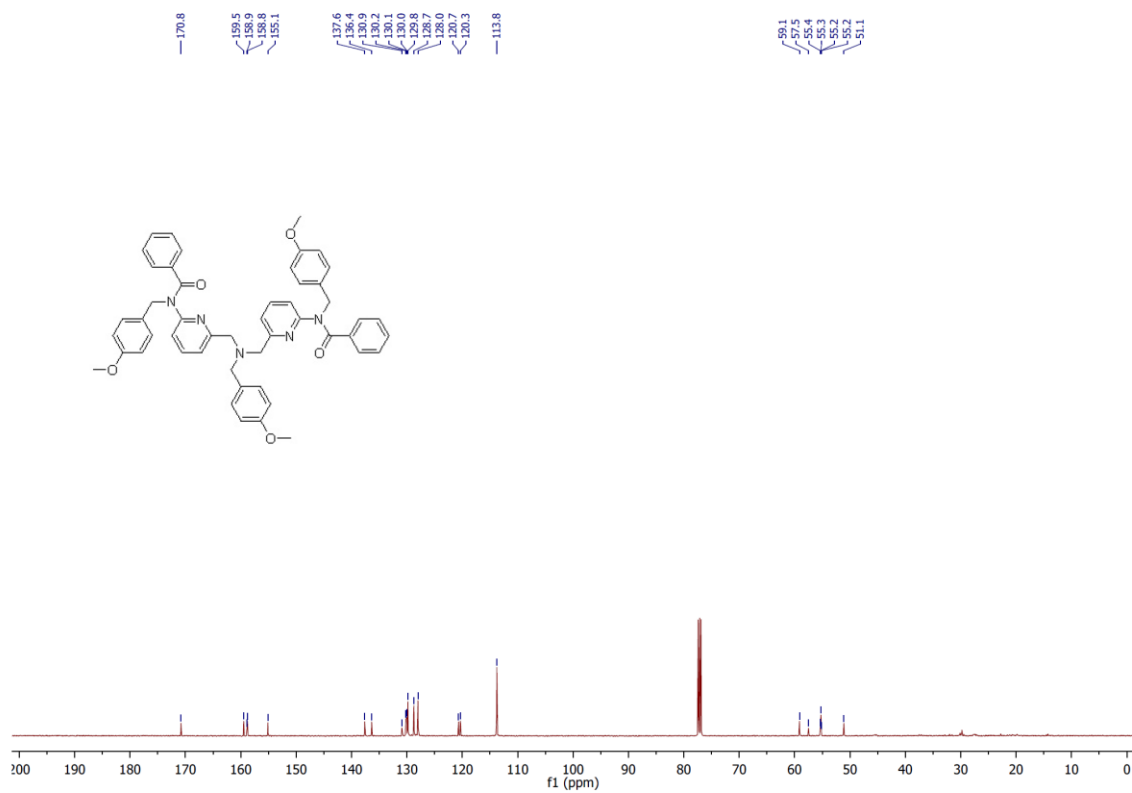


Figure S25. The ¹³C spectra of compound **12b**.

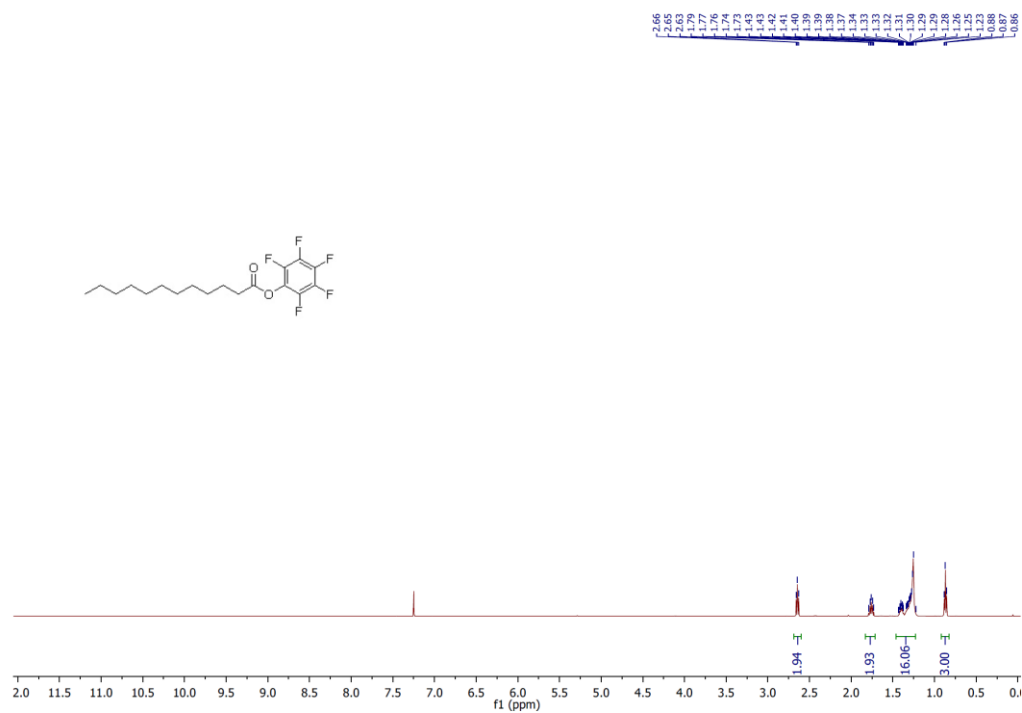


Figure S28. The ¹H spectra of compound **13**.

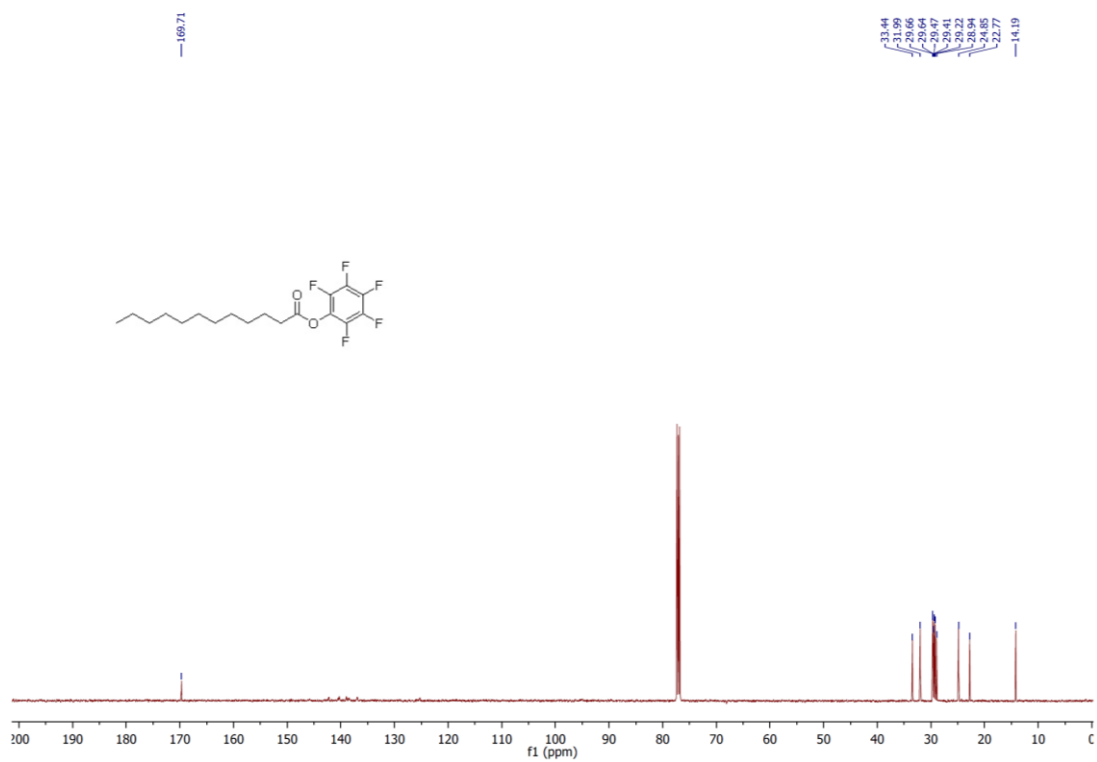


Figure S29. The ¹³C spectra of compound **13**.

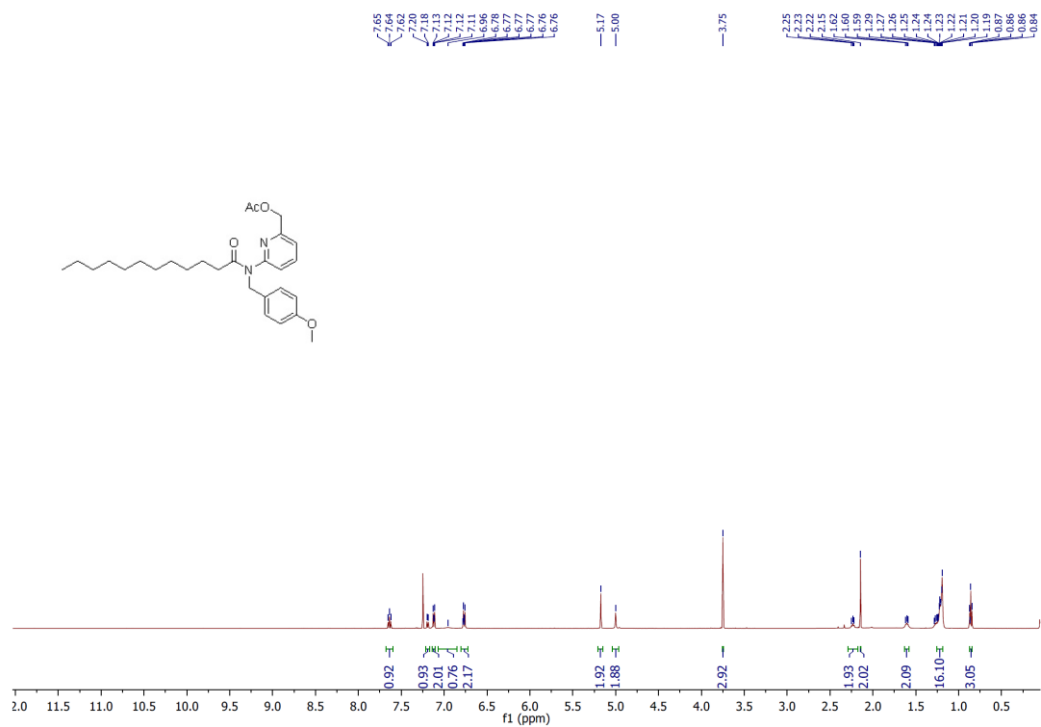


Figure S30. The ¹H spectra of compound **9d**.

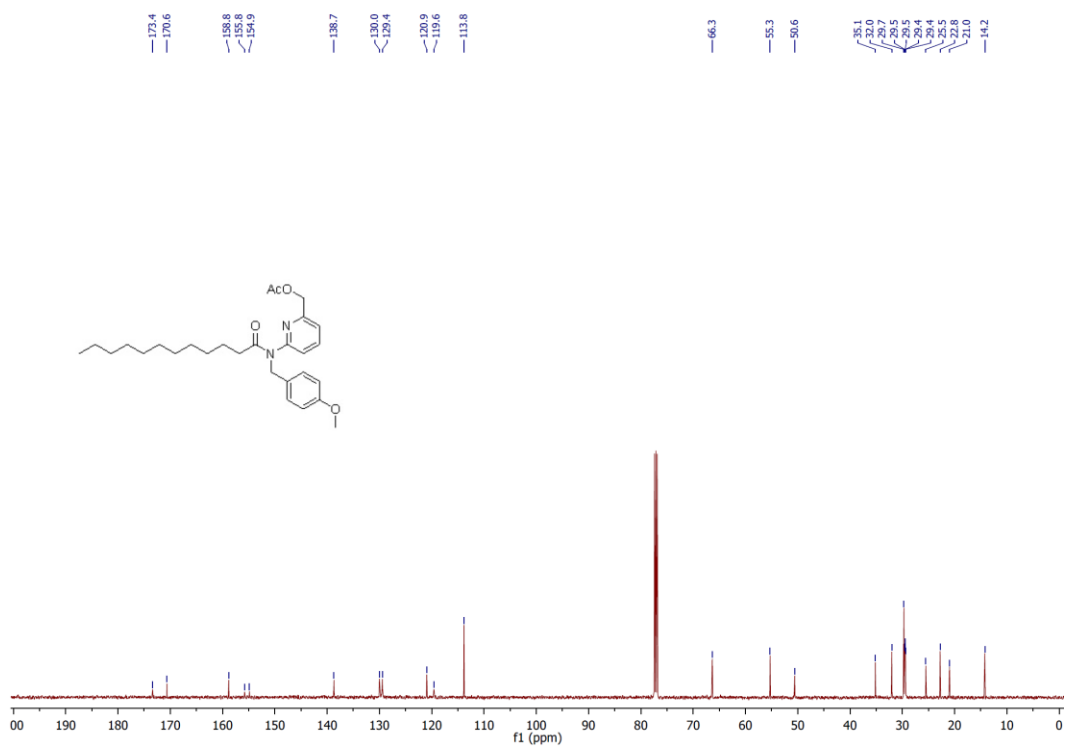


Figure S31. The ¹³C spectra of compound **9d**.

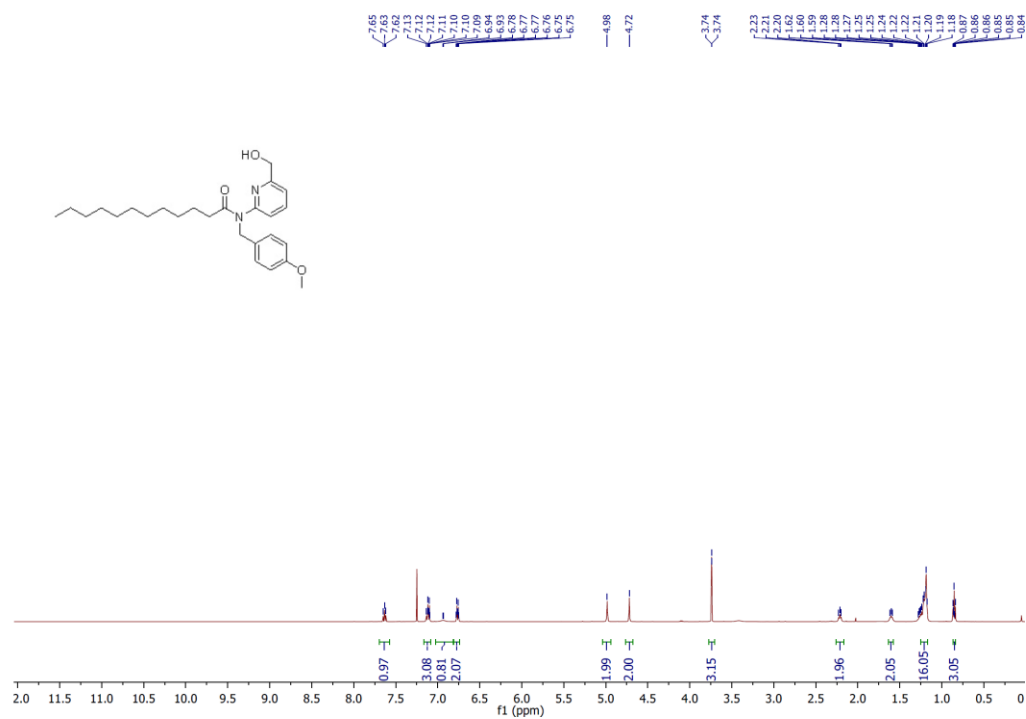


Figure S32. The ¹H spectra of compound **10d**.

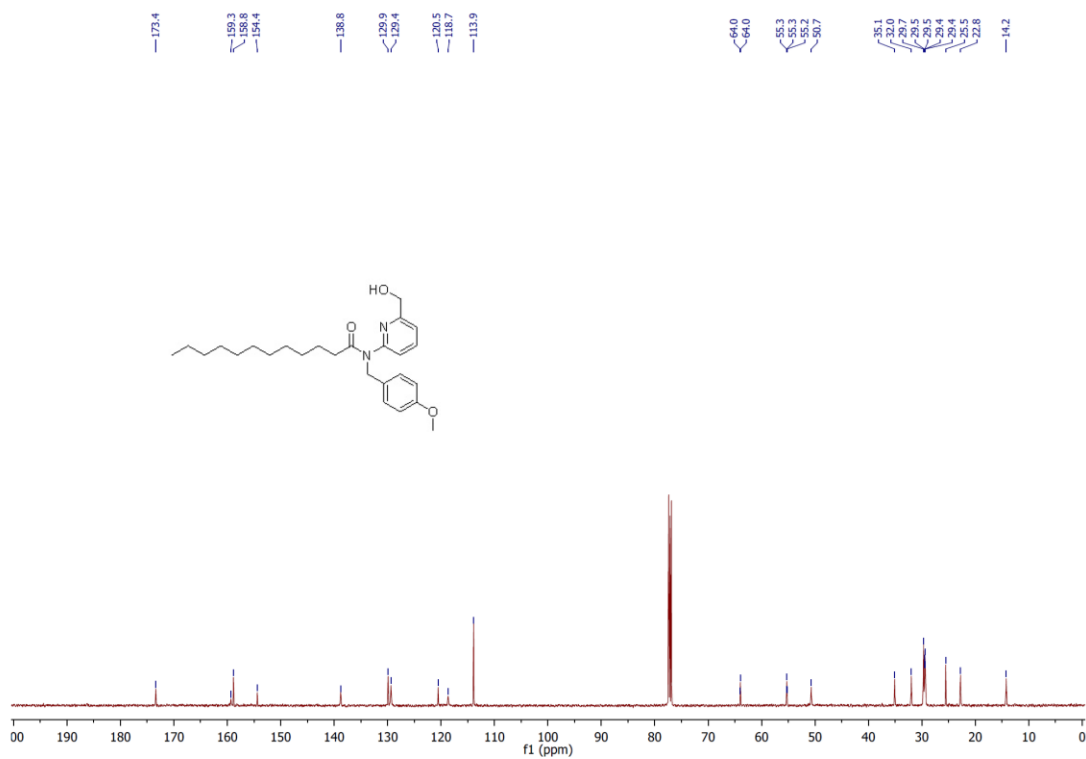


Figure S33. The ¹³C spectra of compound **10d**.

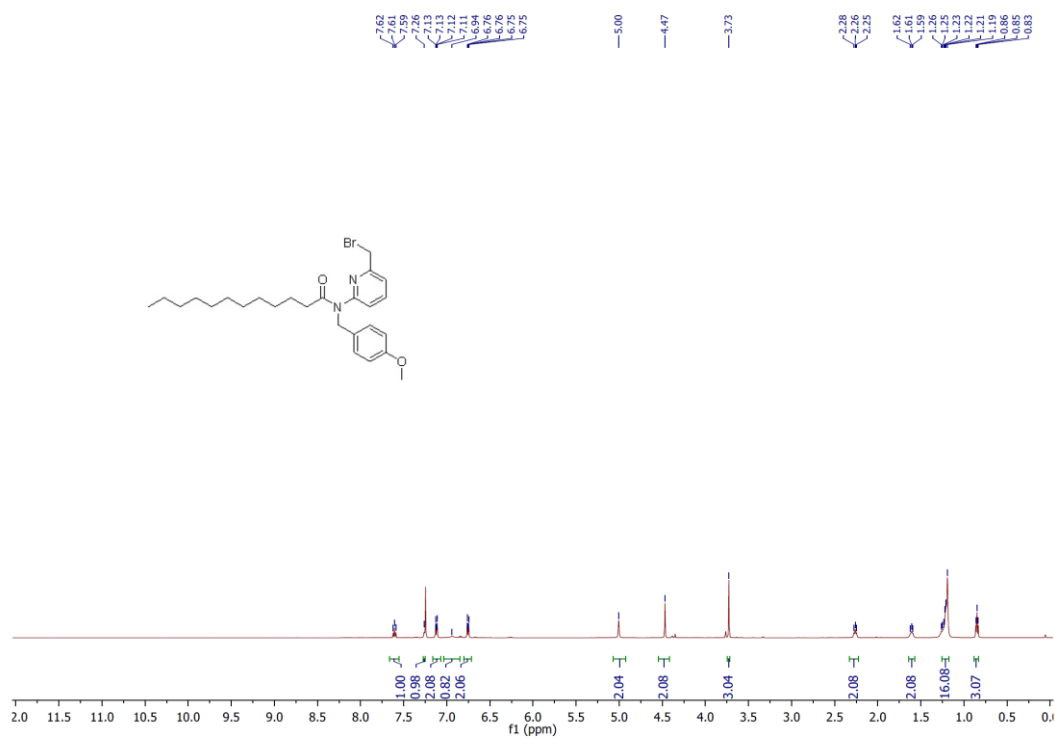


Figure S34. The ¹H spectra of compound **11d**.

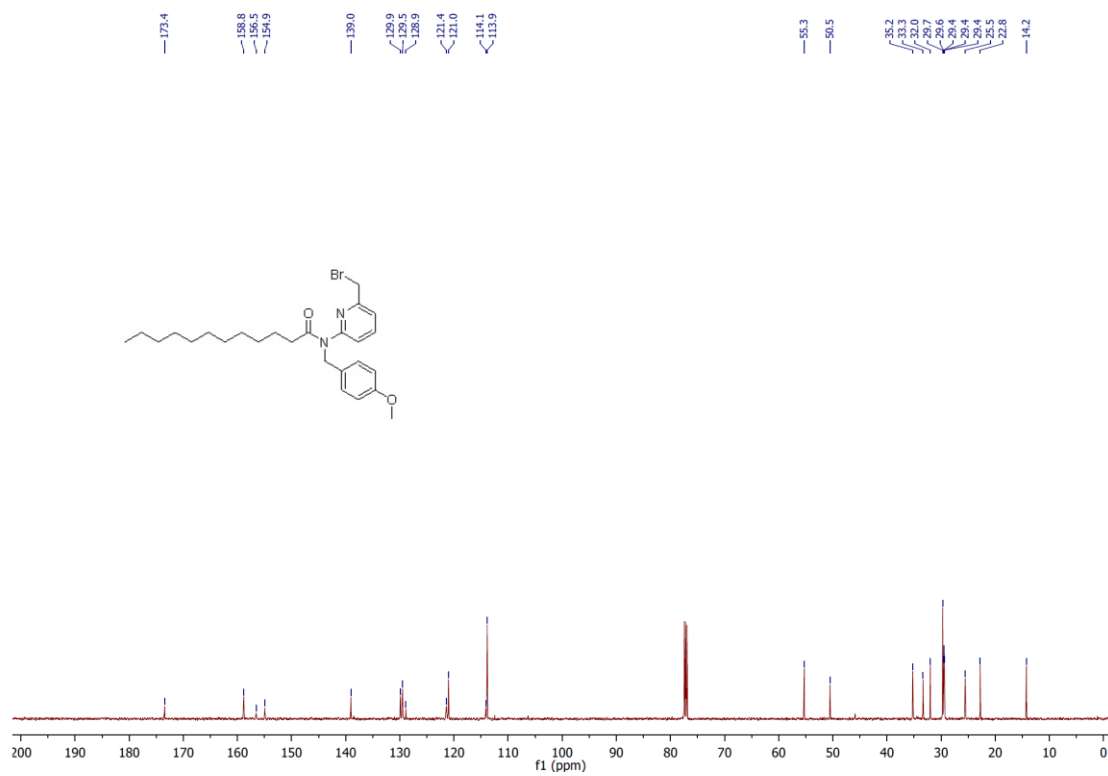


Figure S35. The ¹³C spectra of compound **11d**.

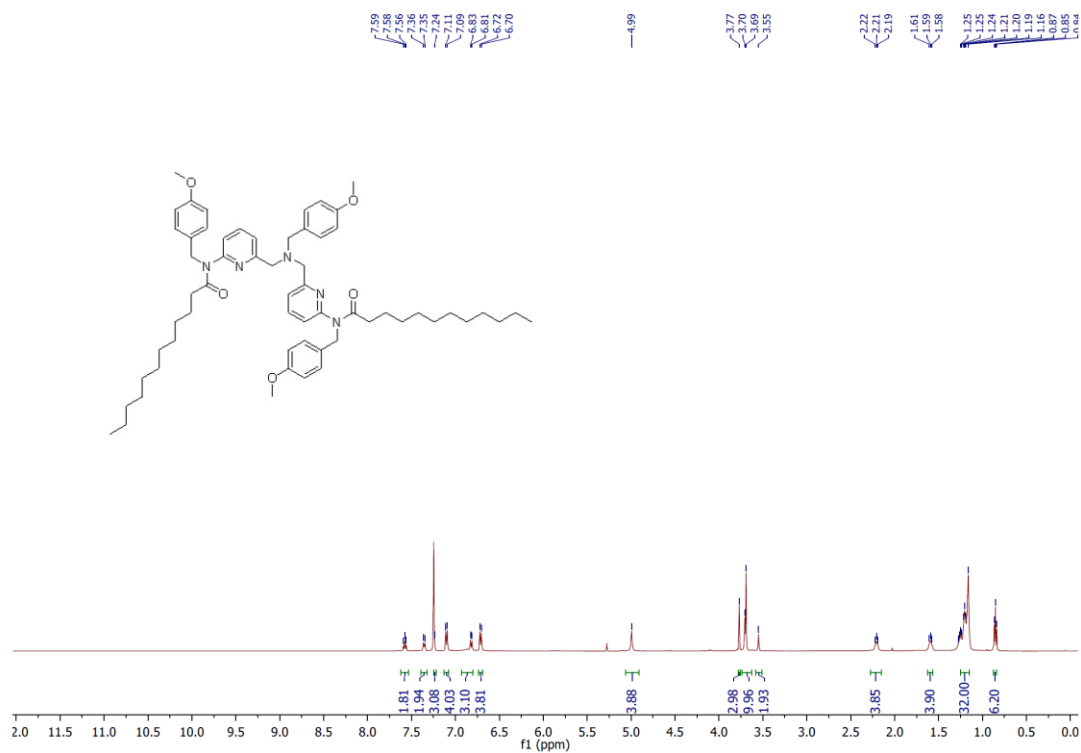


Figure S36. The ¹H spectra of compound **12d**.

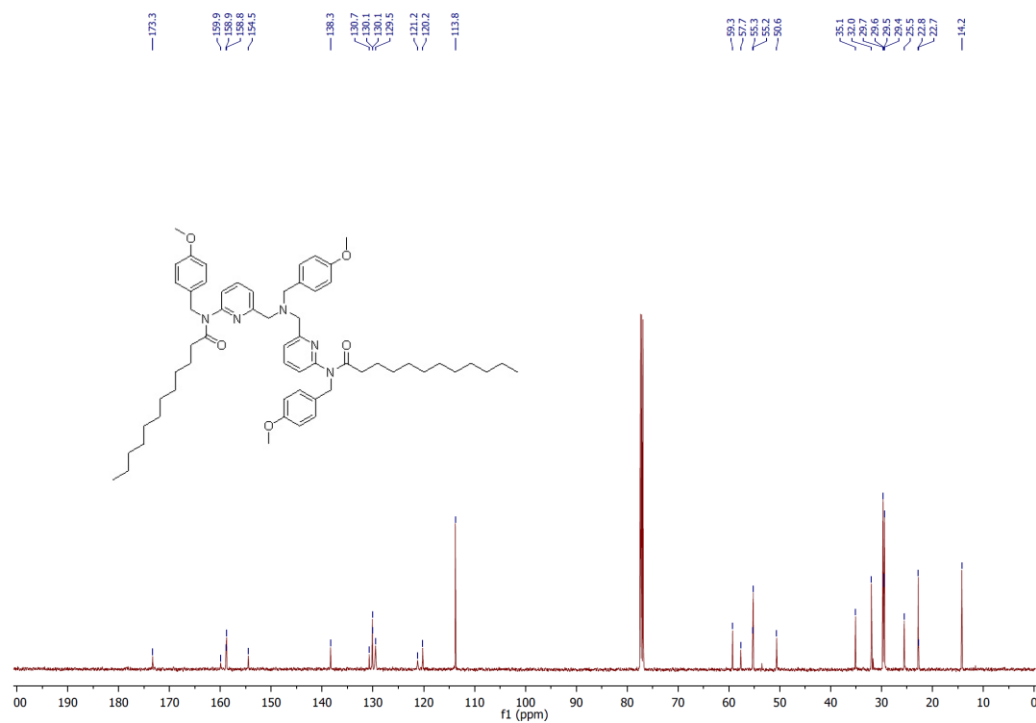


Figure S37. The ¹³C spectra of compound **12d**.

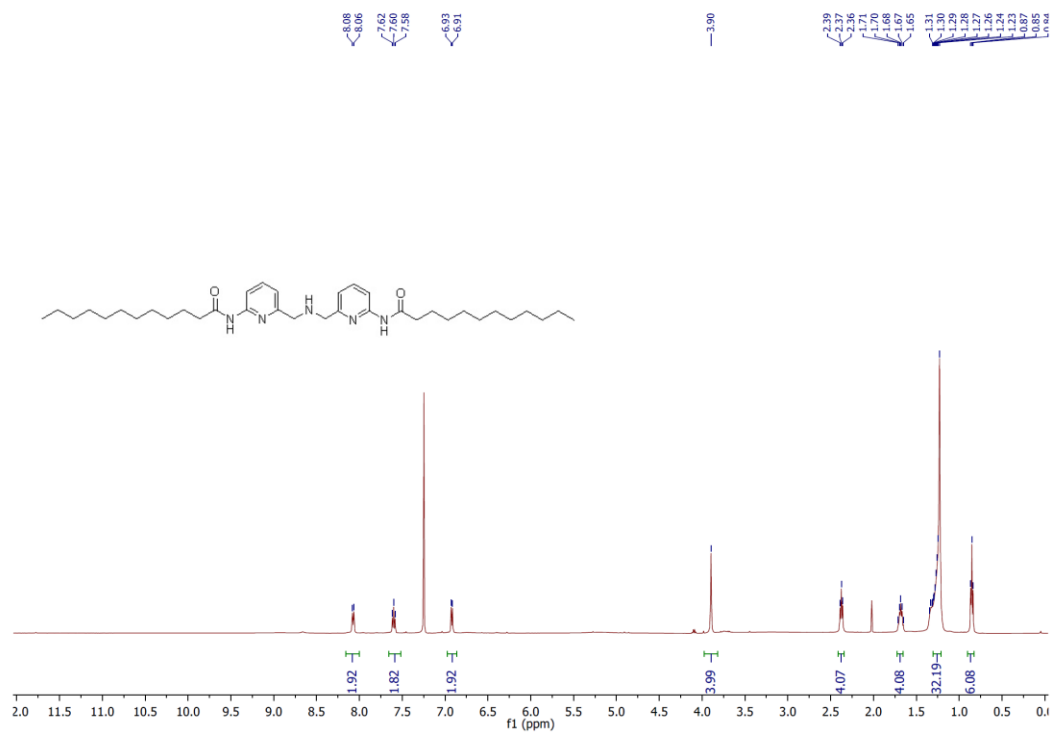


Figure S38. The ¹H spectra of compound **6d**.

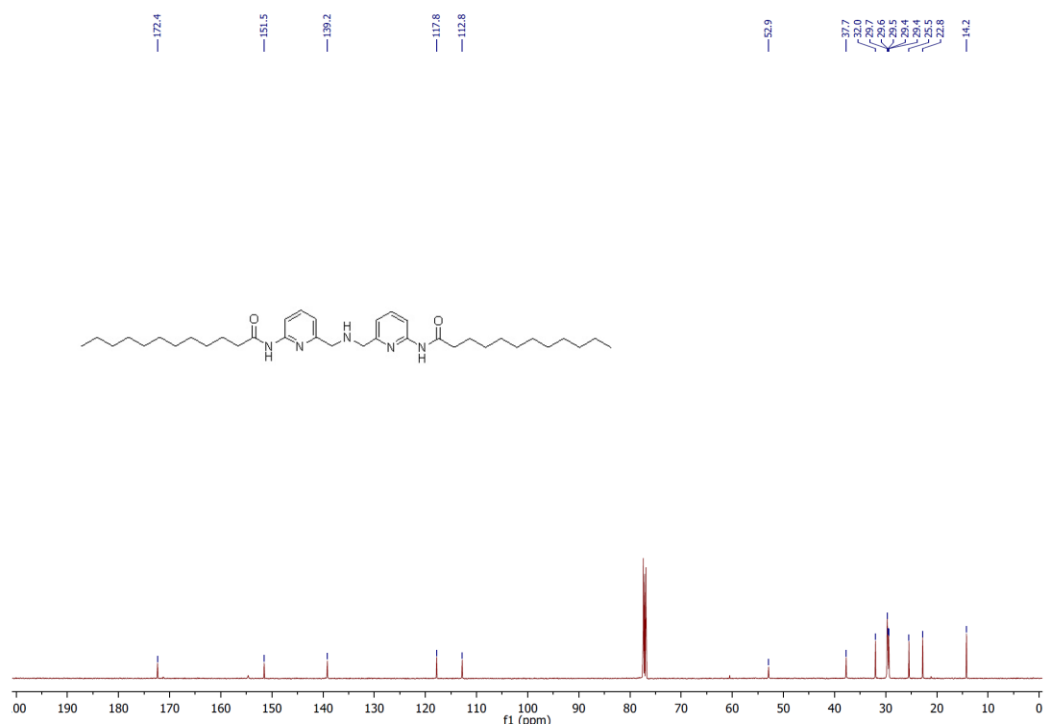


Figure S39. The ¹³C spectra of compound **6d**.

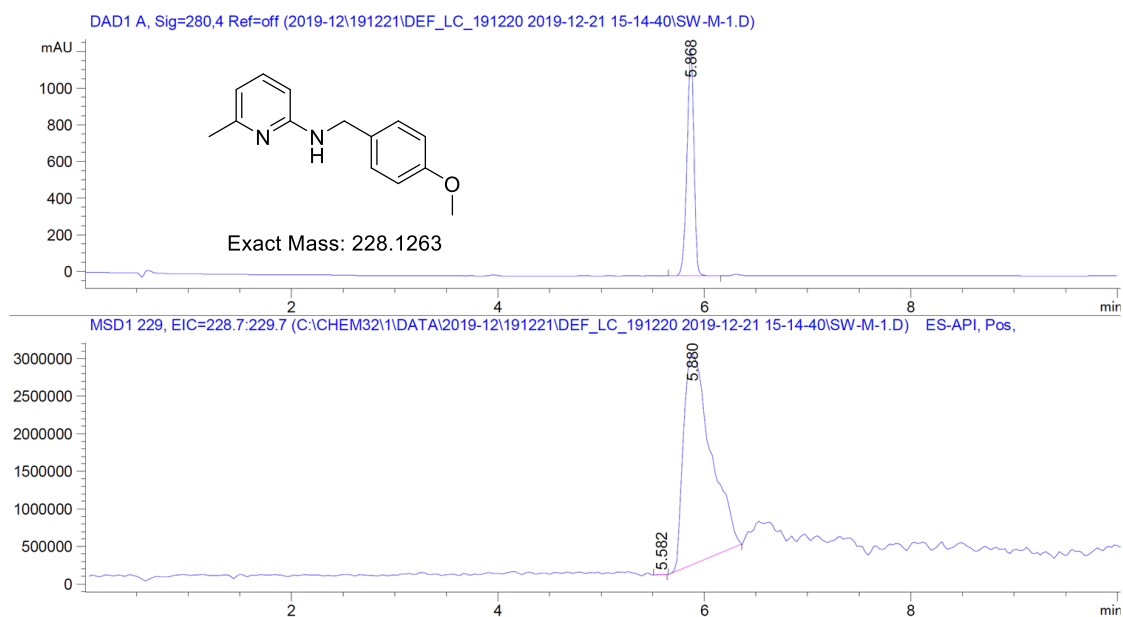


Figure S40. The LCMS spectra of compound **7**.

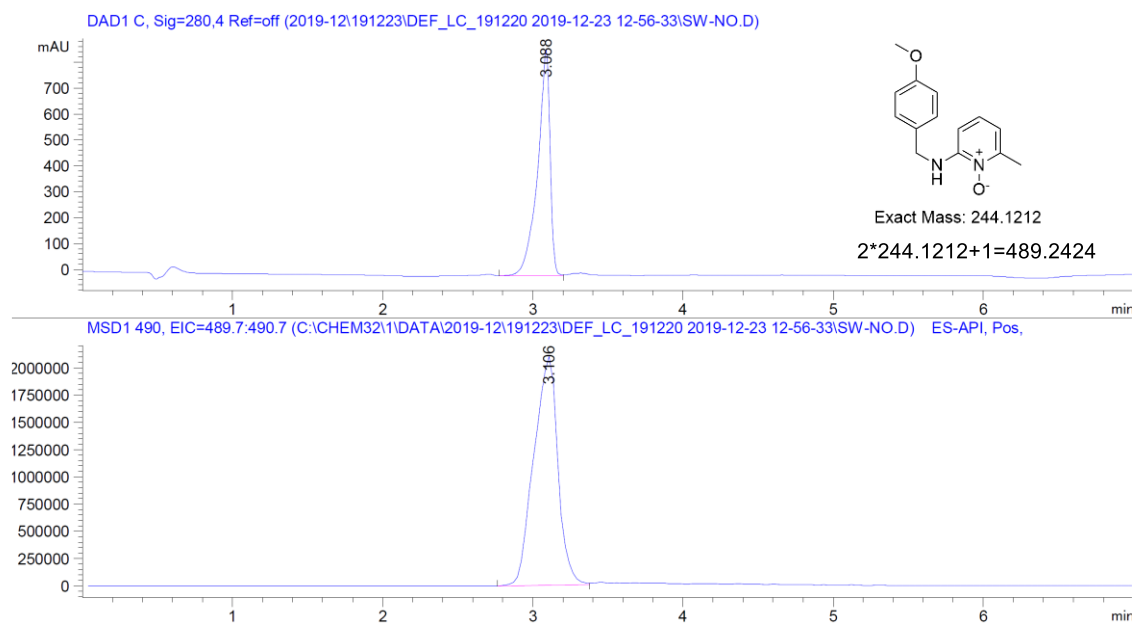


Figure S41. The LCMS spectra of compound **8**.

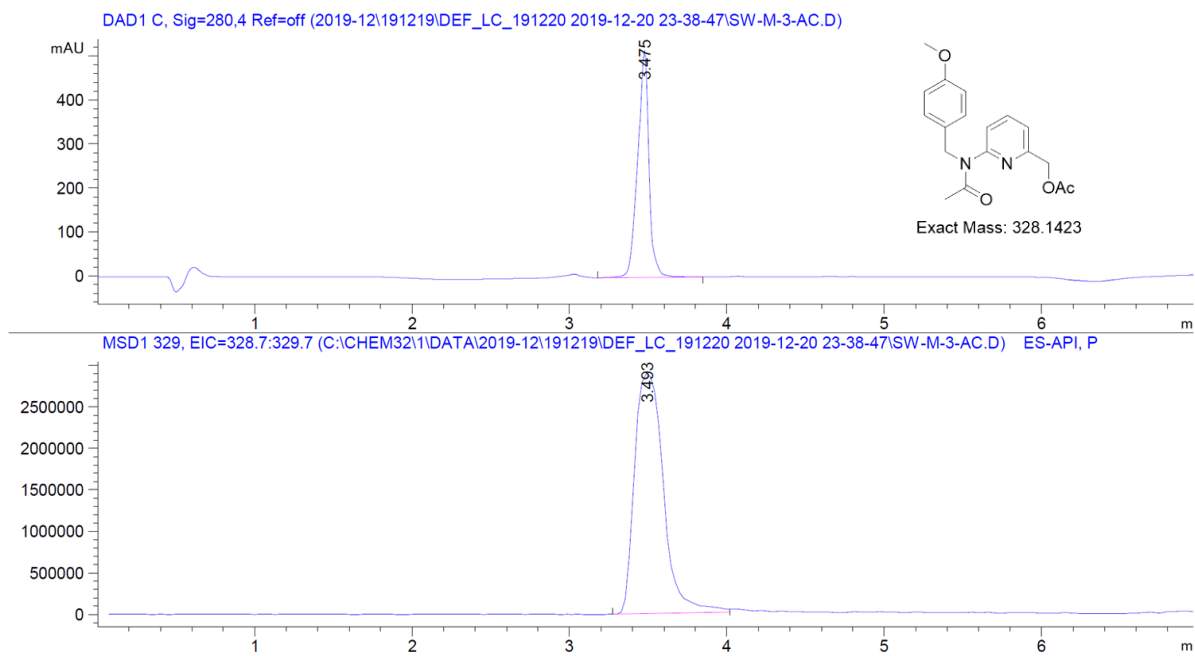


Figure S42. The LCMS spectra of compound **9a**.

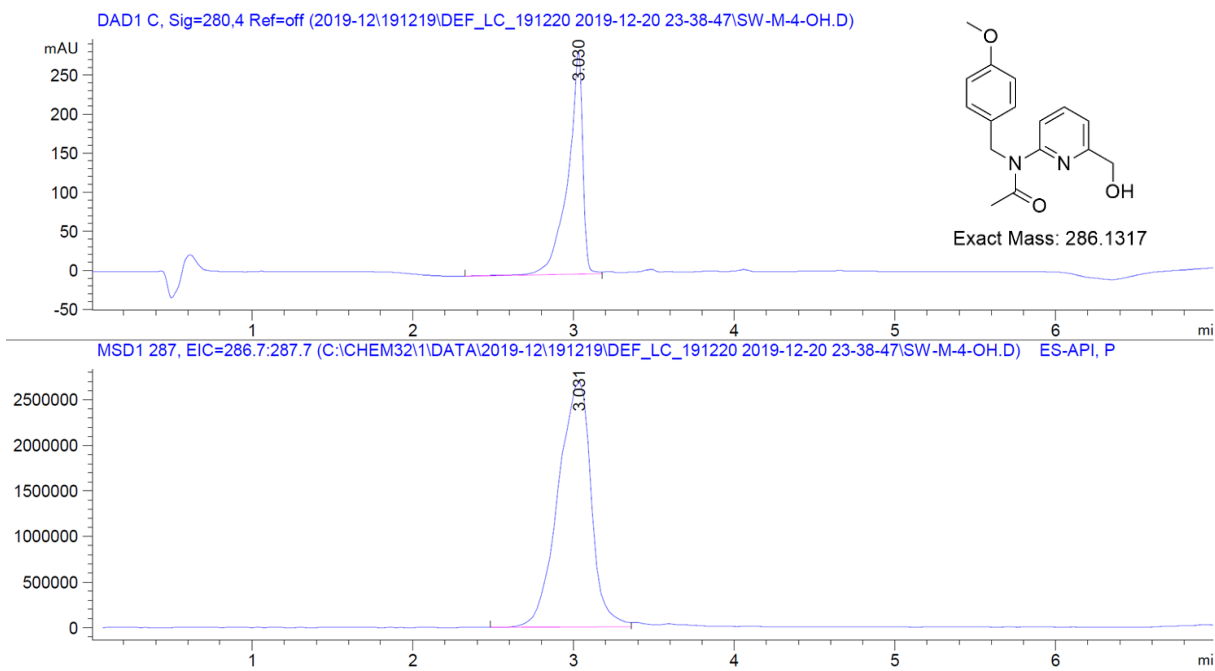


Figure S43. The LCMS spectra of compound **10a**.

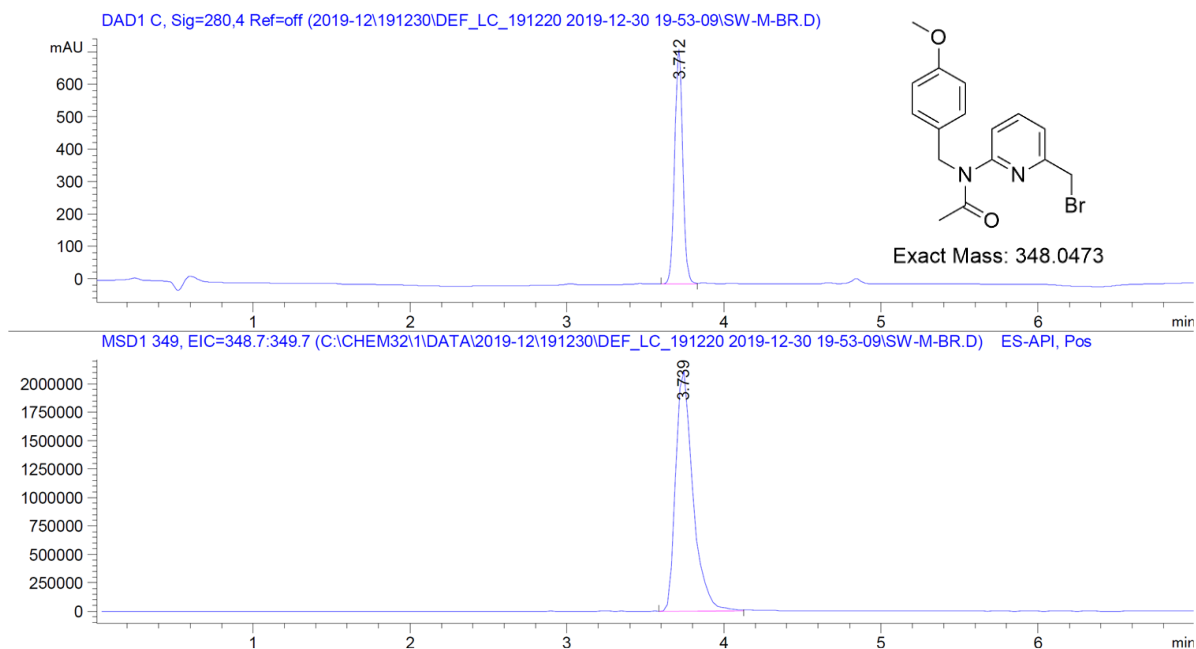


Figure S44. The LCMS spectra of compound **11a**.

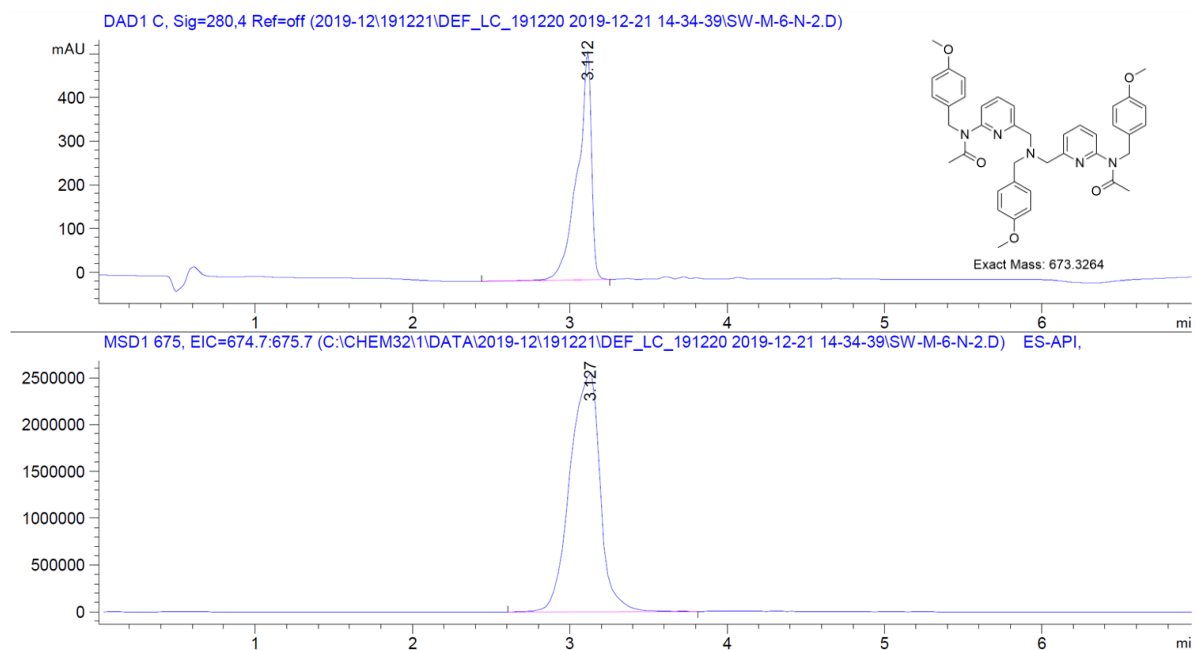


Figure S45. The LCMS spectra of compound **12a**.

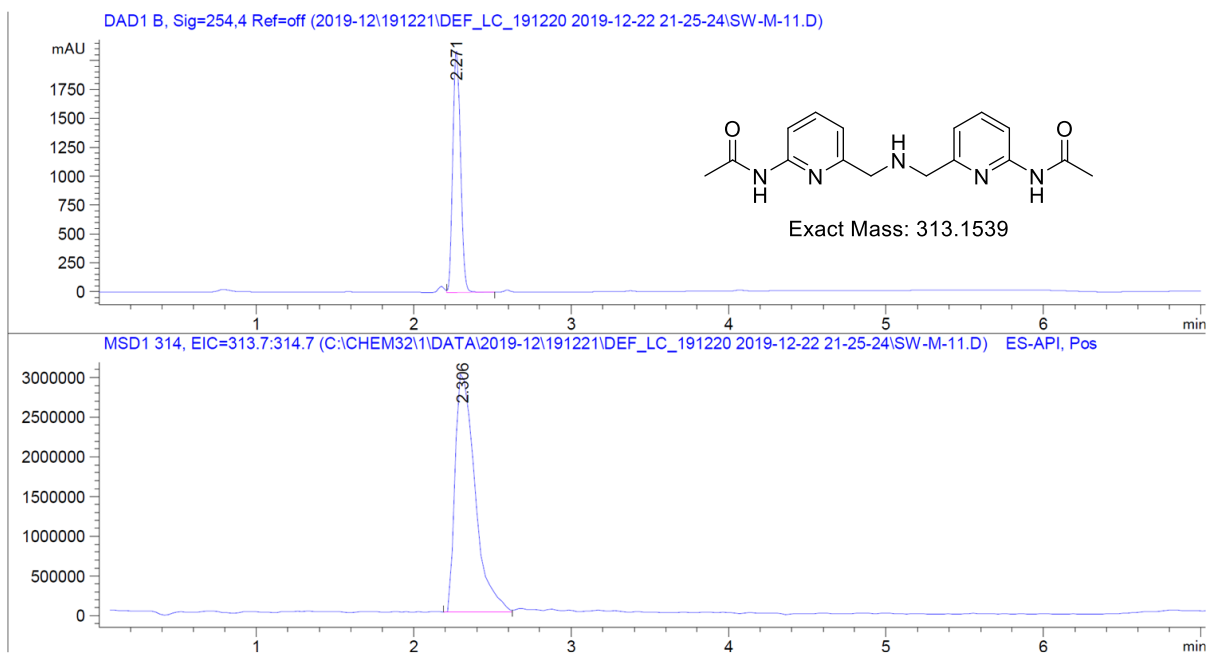


Figure S46. The LCMS spectra of compound **13a**.

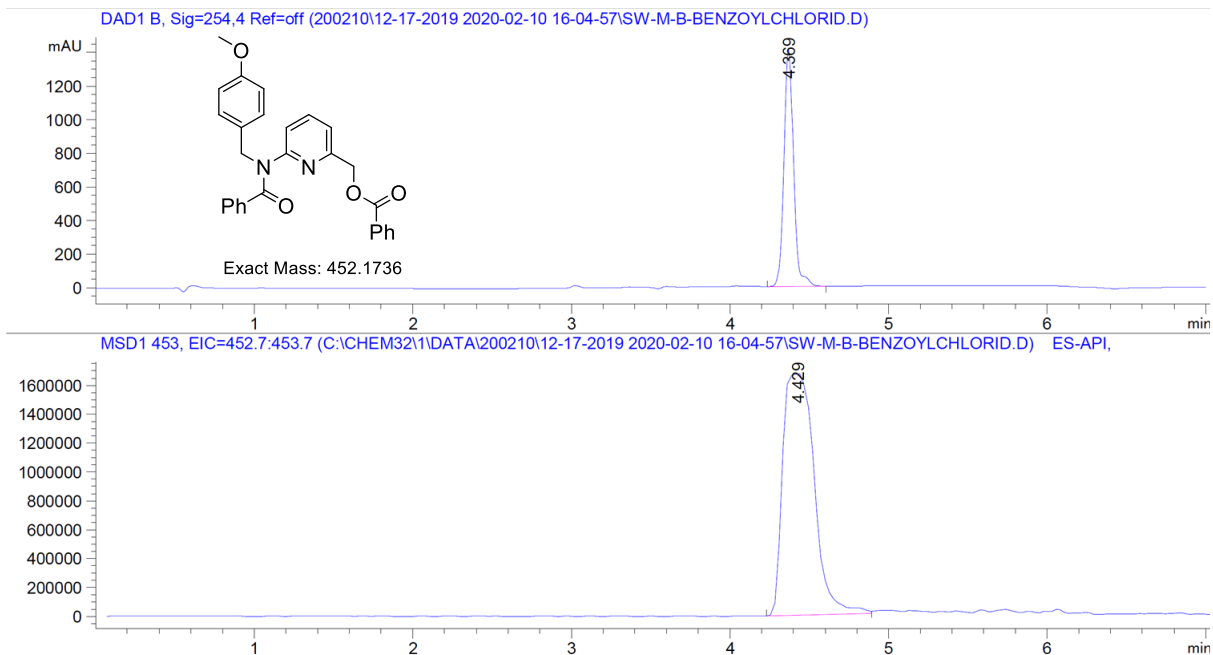


Figure S47. The LCMS spectra of compound **9b**.

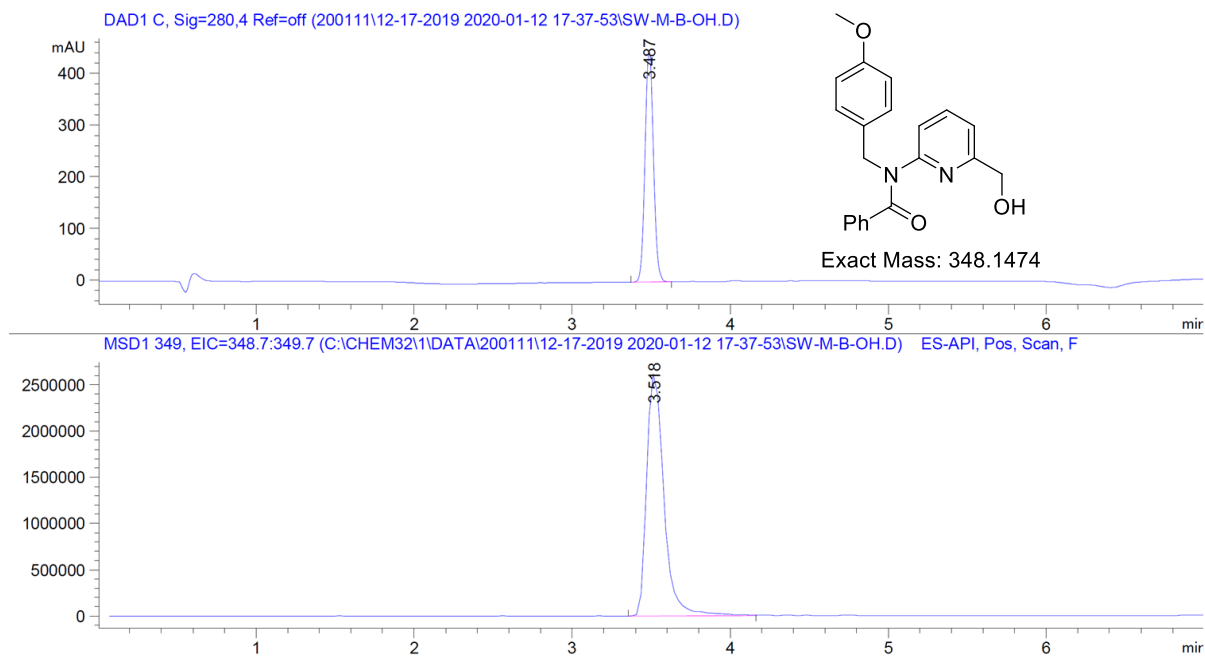


Figure S48. The LCMS spectra of compound **10b**.

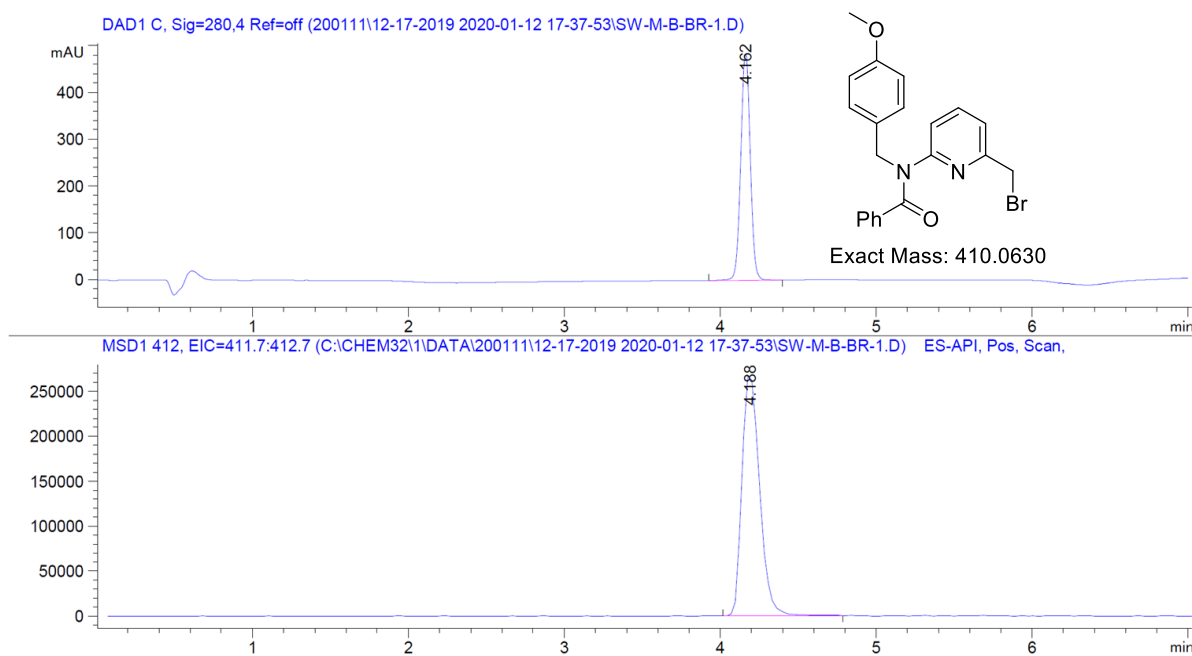


Figure S49. The LCMS spectra of compound **11b**.

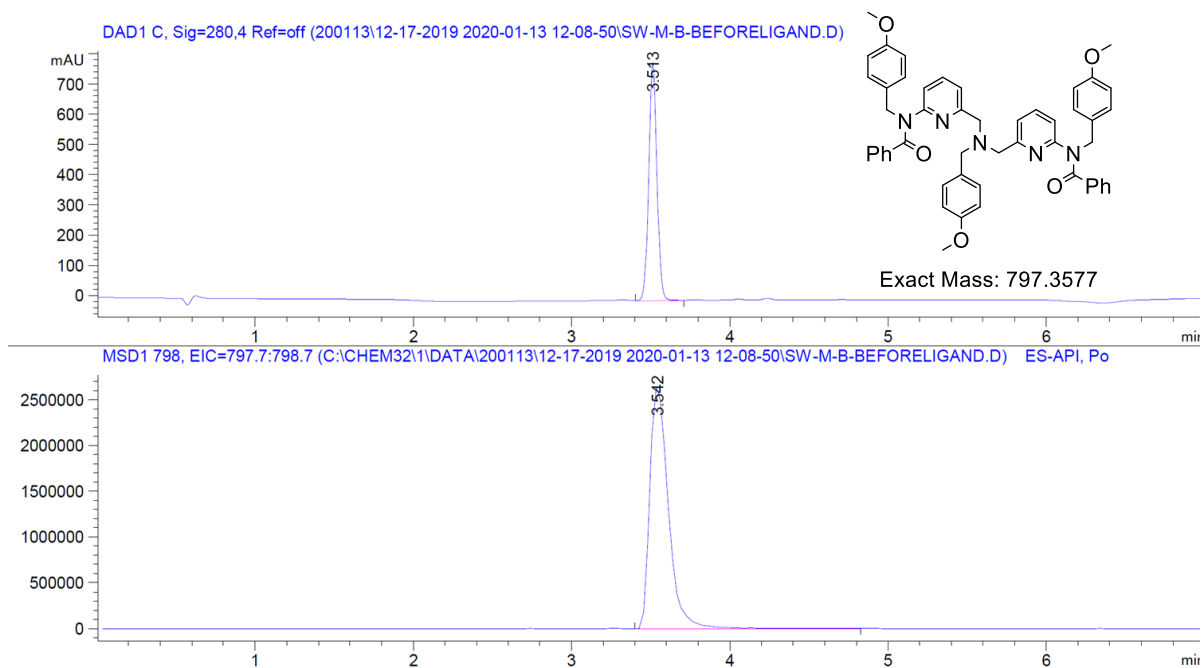


Figure S50. The LCMS spectra of compound **12b**.

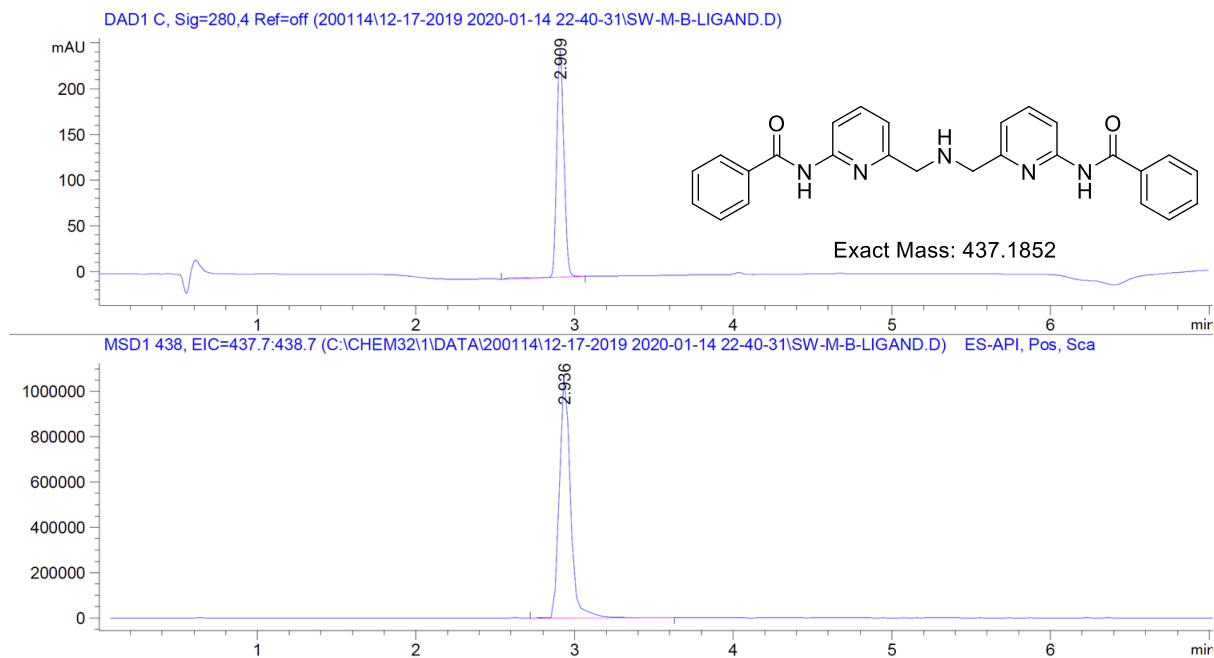


Figure S51. The LCMS spectra of compound **13b**.

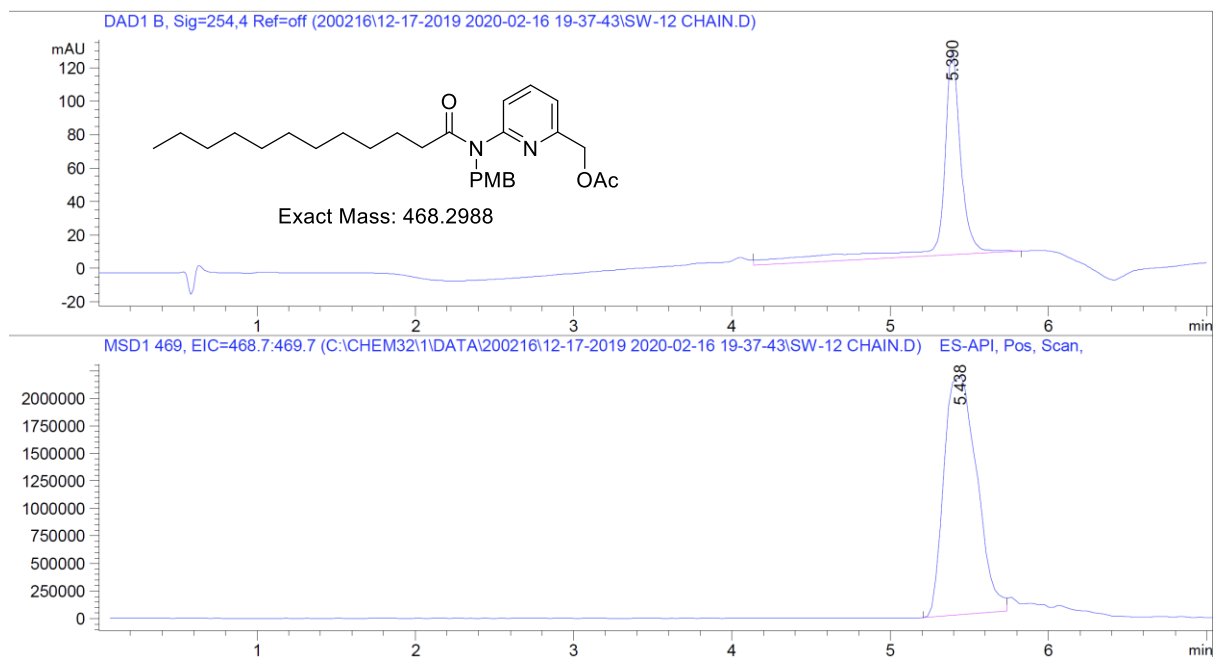


Figure S52. The LCMS spectra of compound **9d**.

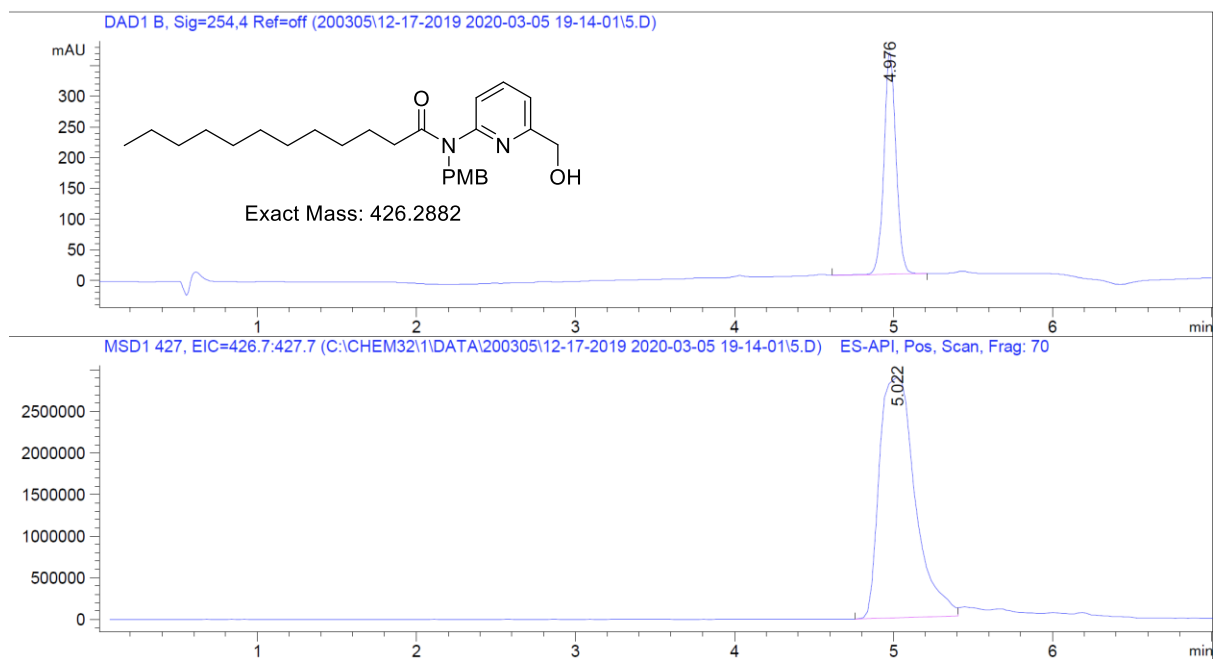


Figure S53. The LCMS spectra of compound **10d**.

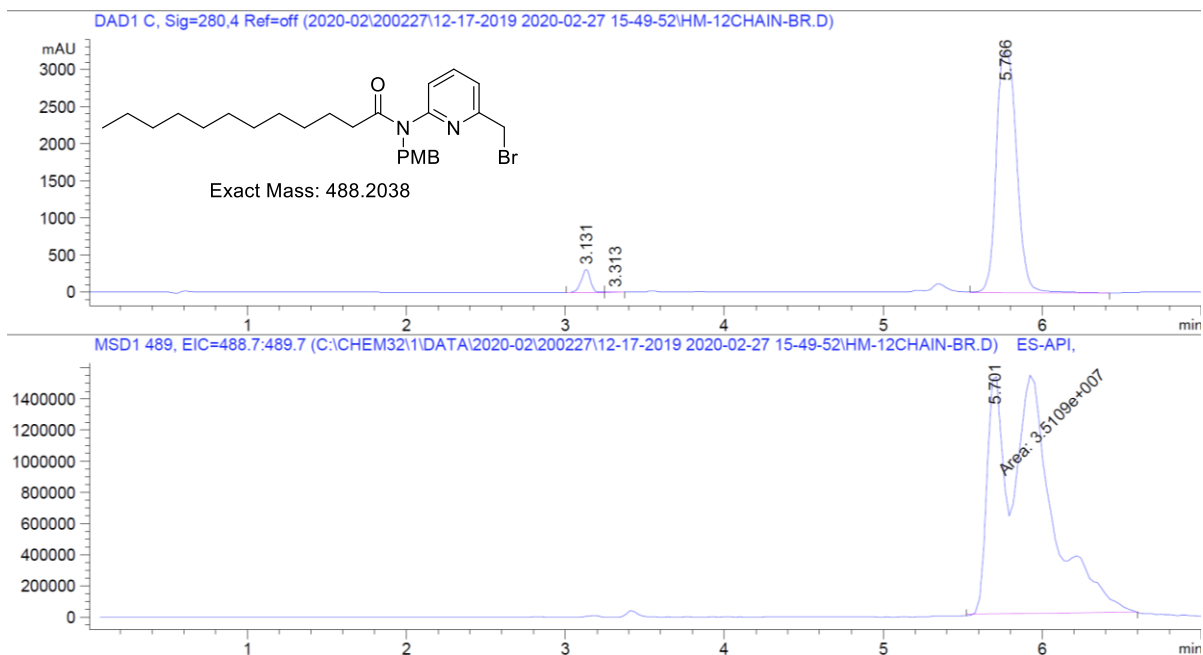


Figure S54. The LCMS spectra of compound **11d**.

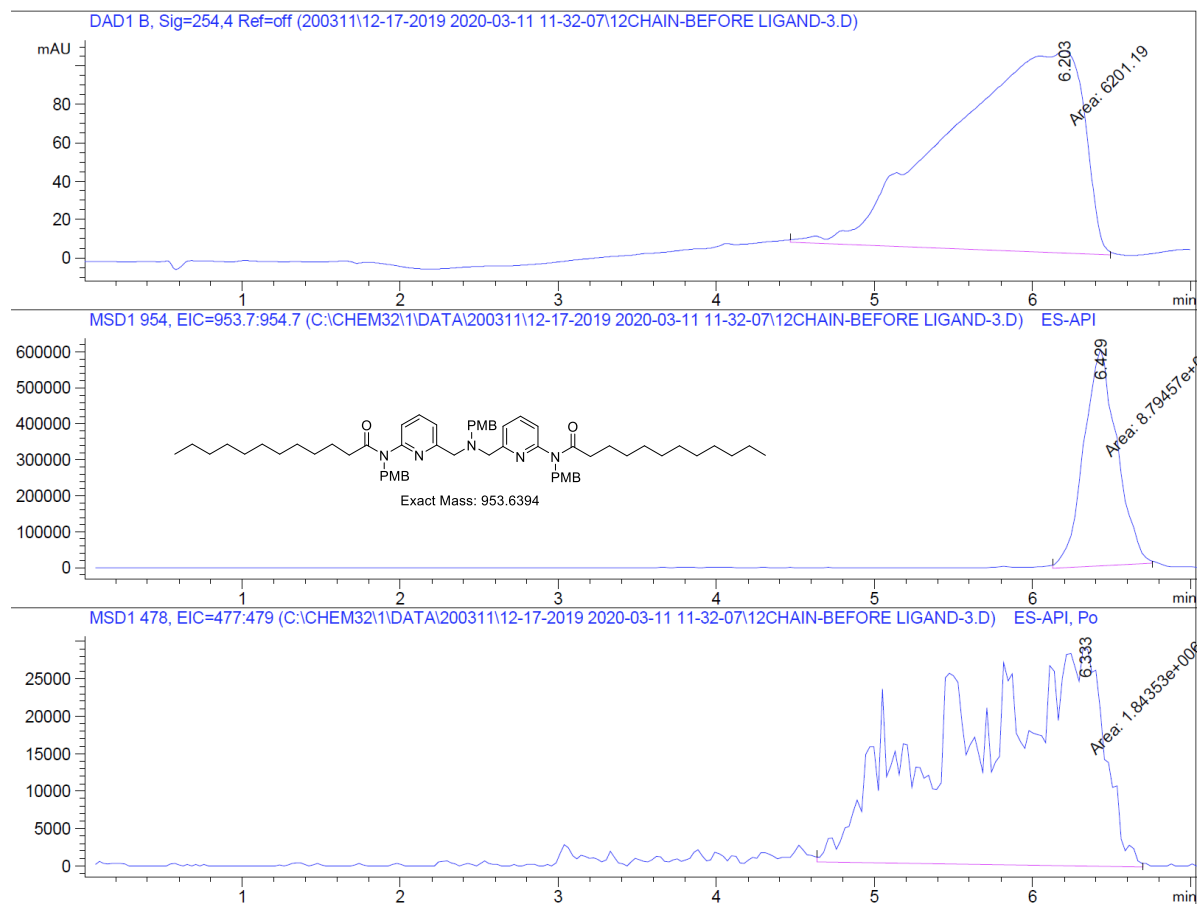


Figure S55. The LCMS spectra of compound **12d**.

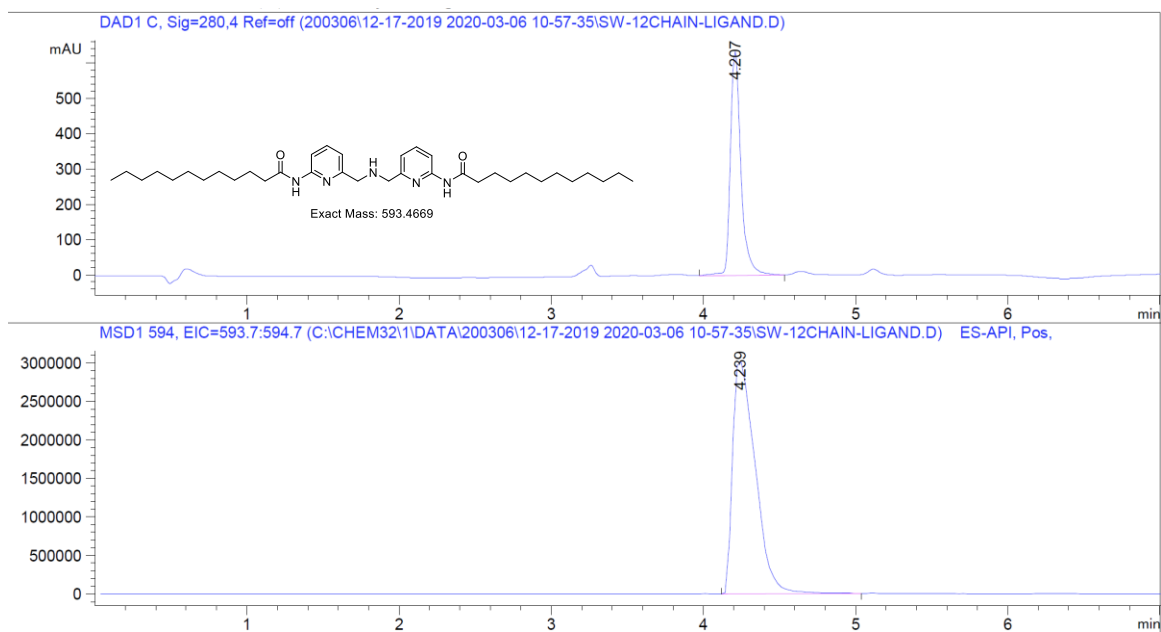


Figure S56. The LCMS spectra of compound **6d**.