

Supplementary Information

Hyphenating the Curtius Rearrangement with Morita-Baylis-Hillman Adducts: Synthesis of Biologically Active Acyloins and Vicinal Aminoalcohols

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General

The ^1H and ^{13}C spectra were recorded on Bruker at 250 MHz and 62.5 MHz respectively. The ^1H and ^{13}C spectra were also recorded on Inova instrument at 500 MHz and 125 MHz, respectively. The high resolution mass spectra were recorded using a Q-TOF Micromass equipment (Waters, UK).

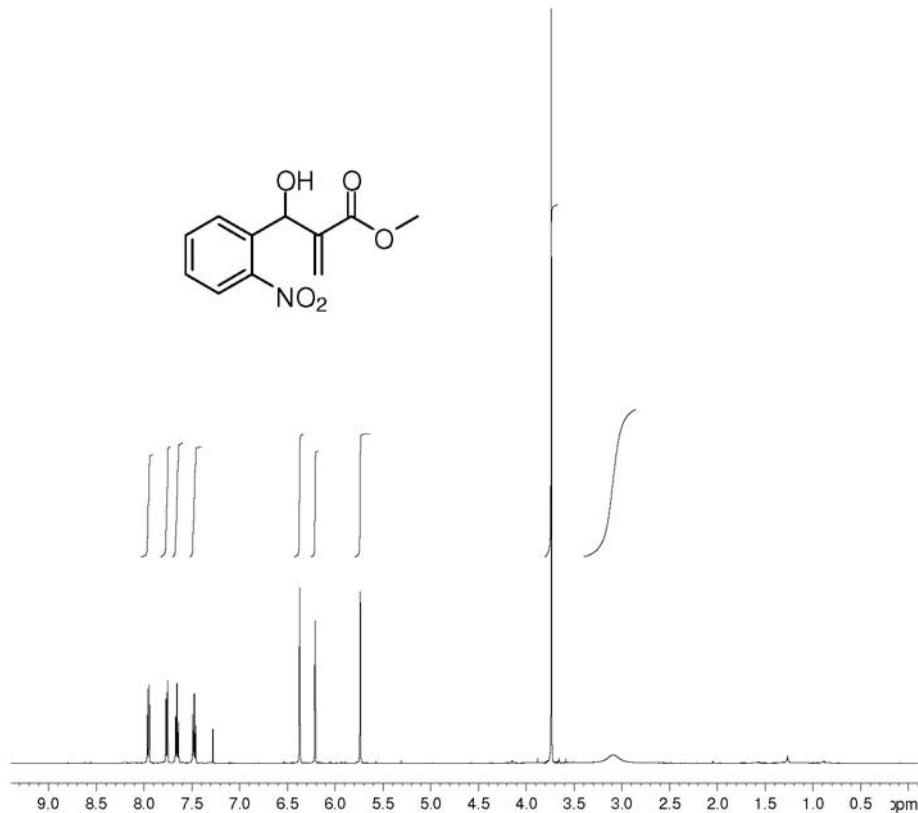


Figure S1. ^1H NMR (CDCl_3 , 500 MHz) of MBH adduct 3.

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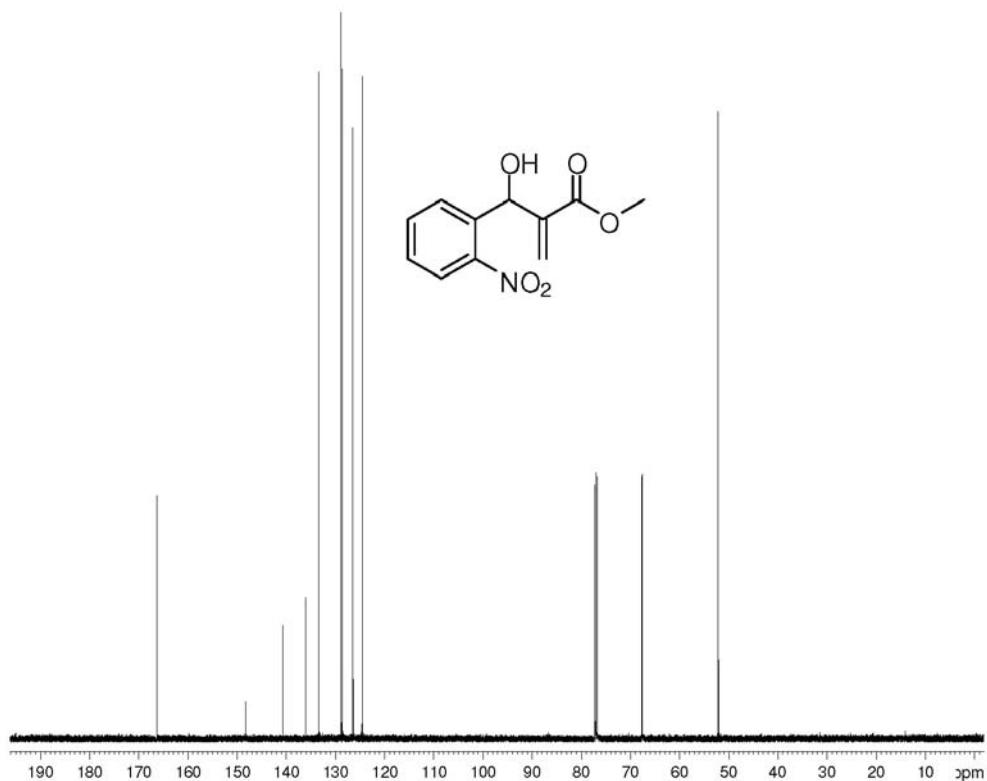


Figure S2. ¹³C NMR (CDCl_3 , 125 MHz) of MBH adduct 3.

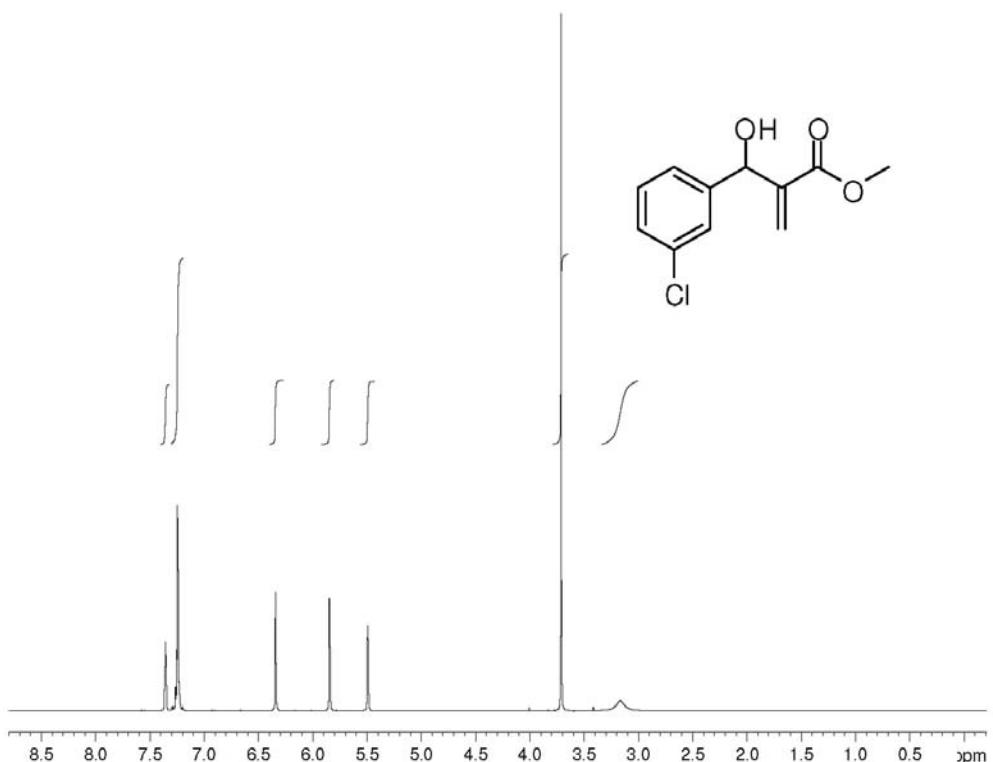


Figure S3. ¹H NMR (CDCl_3 , 250 MHz) of MBH adduct 4.

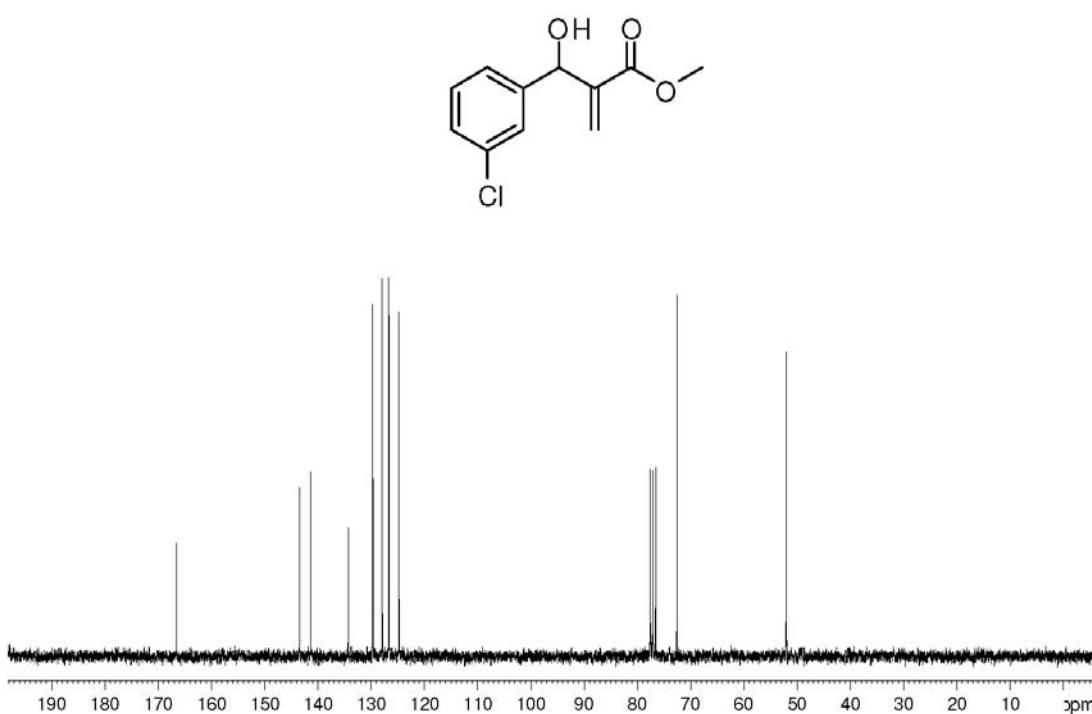


Figure S4. ^{13}C NMR (CDCl_3 , 62.5 MHz) of MBH adduct 4.

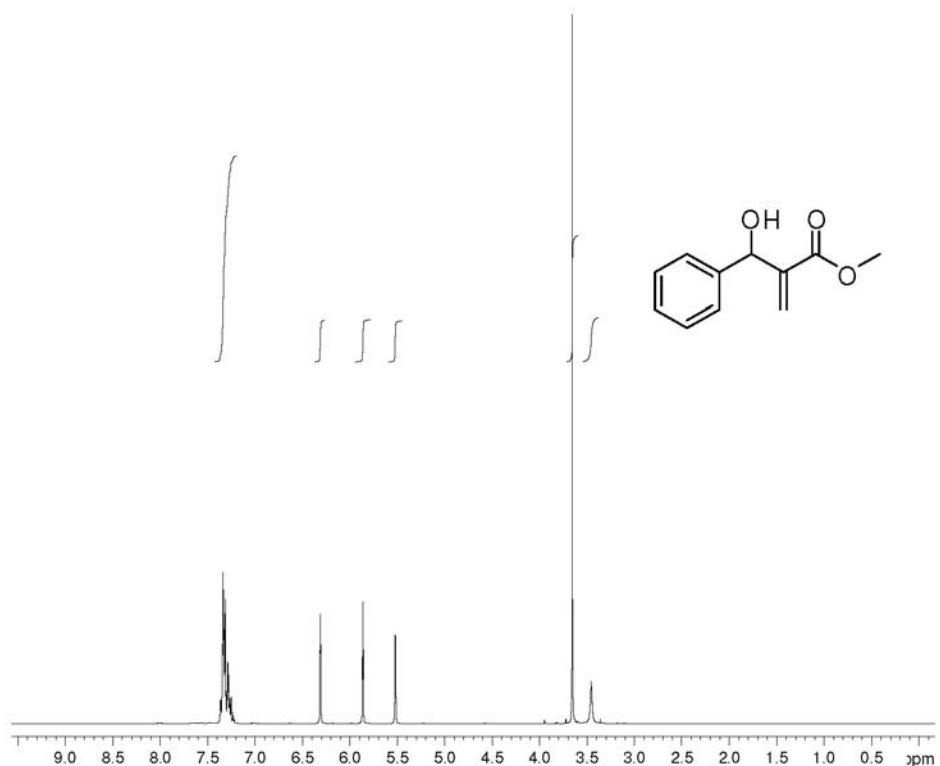


Figure S5. ^1H NMR (CDCl_3 , 250 MHz) of MBH adduct 5.

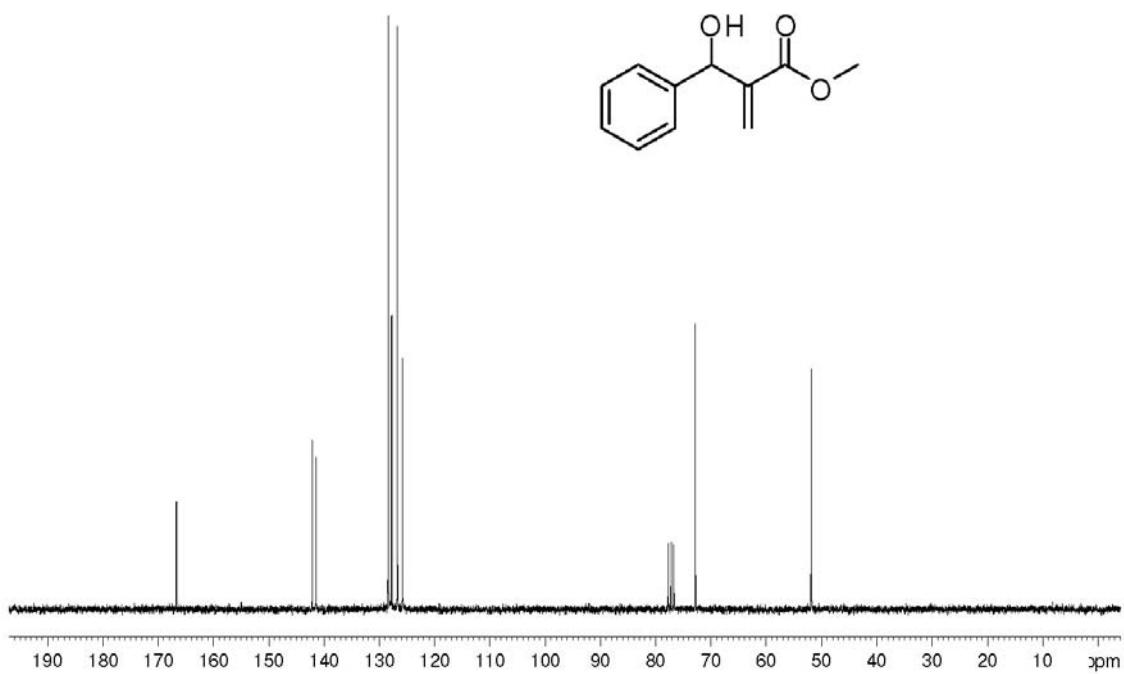


Figure S6. ^{13}C NMR (CDCl_3 , 62.5 MHz) of MBH adduct 5.

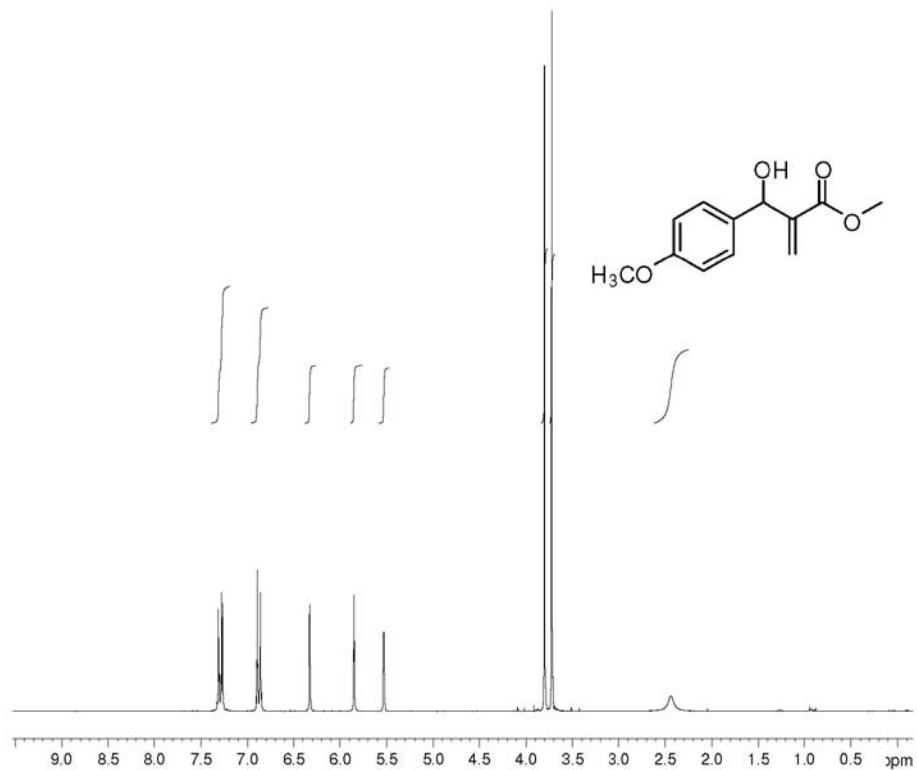


Figure S7. ^1H NMR (CDCl_3 , 250 MHz) of MBH adduct 6.

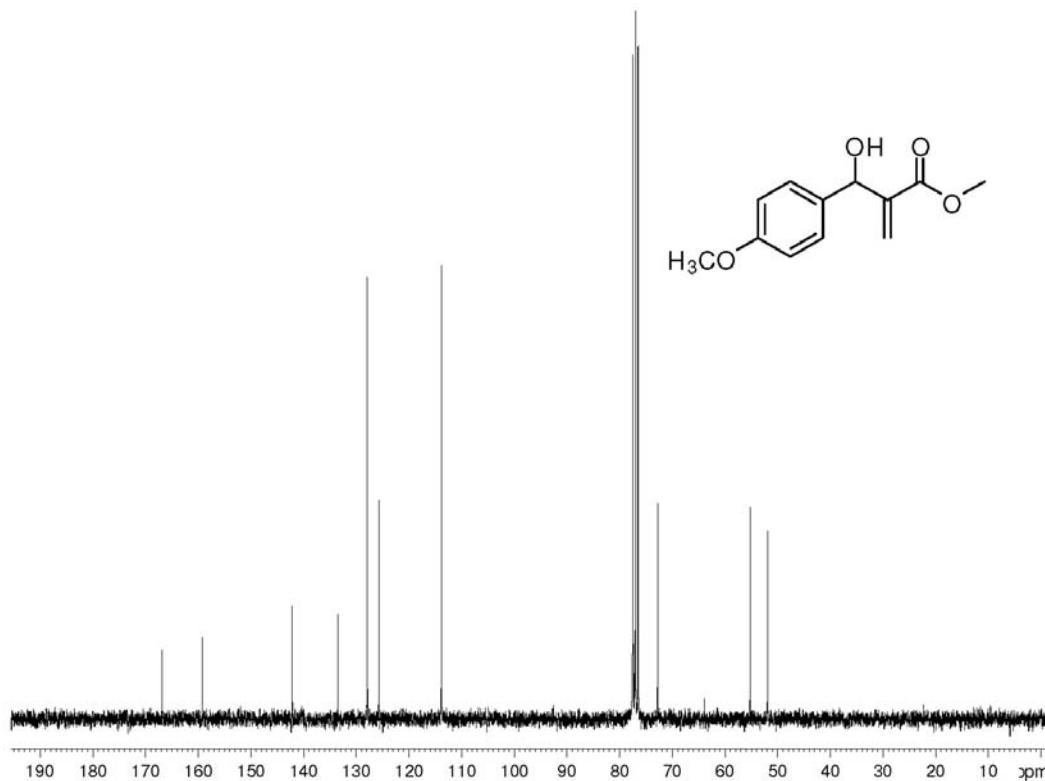


Figure S8. ^{13}C NMR (CDCl_3 , 62.5 MHz) of MBH adduct 6.

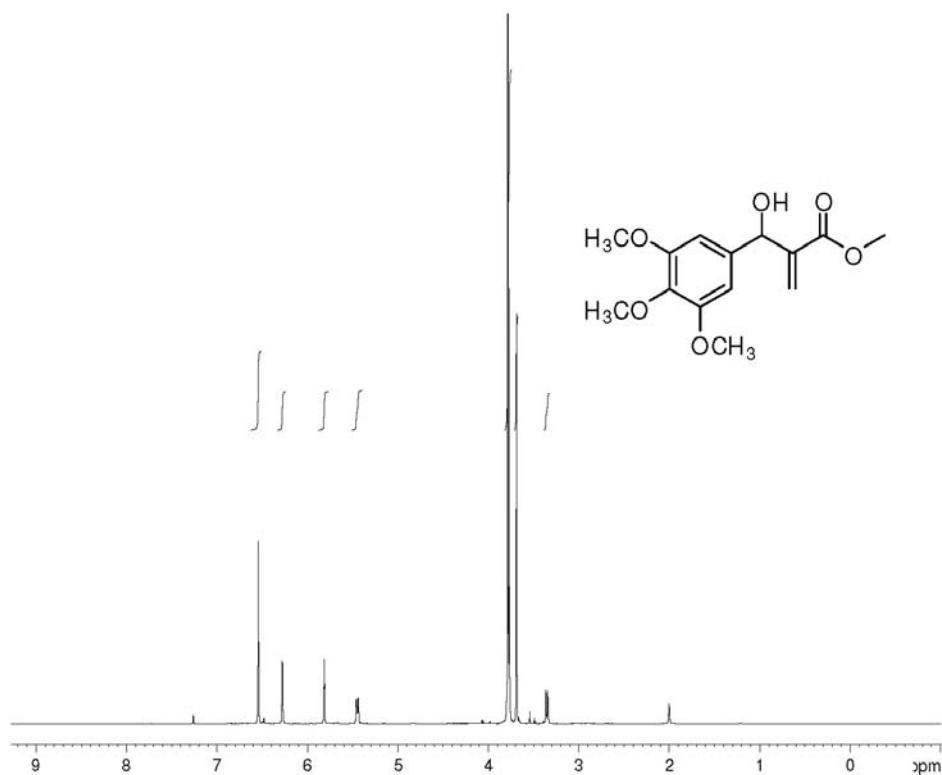


Figure S9. ^1H RMN (CDCl_3 , 250 MHz) of MBH adduct 7.

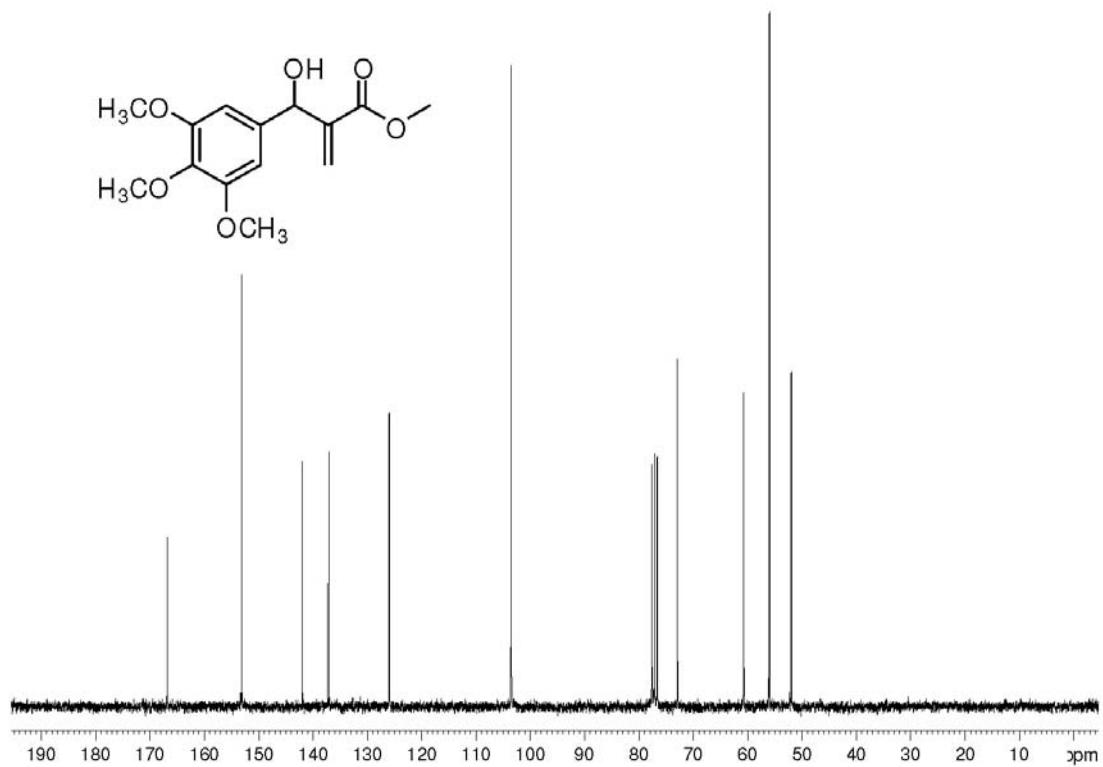


Figure S10. ^{13}C NMR (CDCl_3 , 62.5 MHz) of MBH adduct 7.

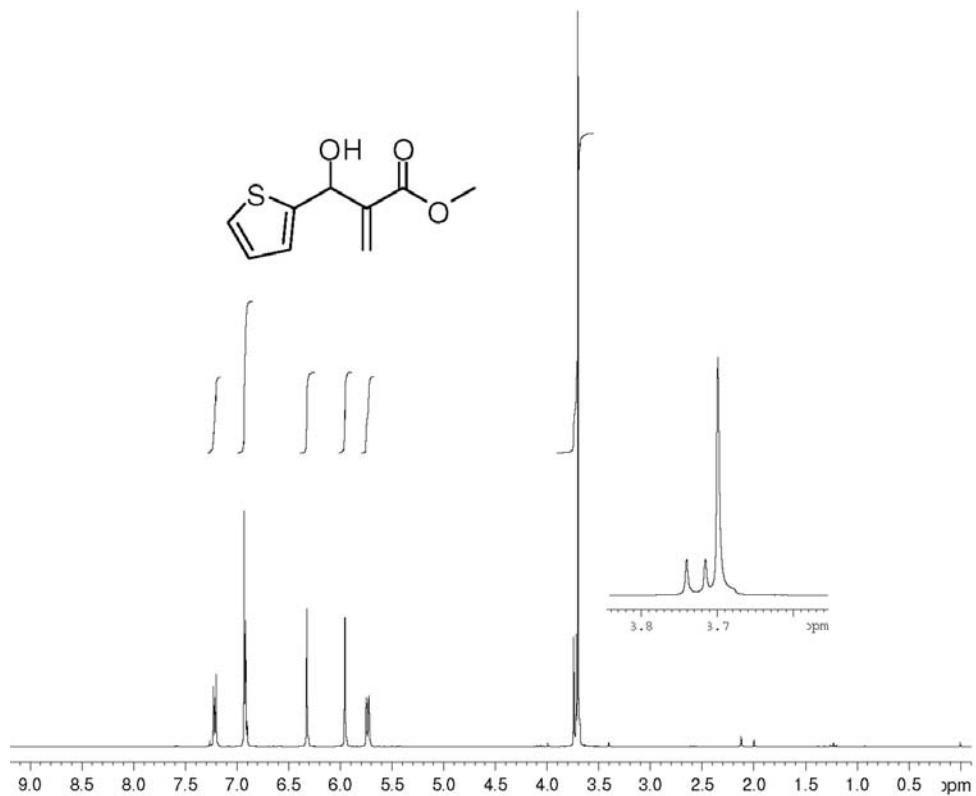


Figure S11. ^1H NMR (CDCl_3 , 250 MHz) of MBH adduct 8.

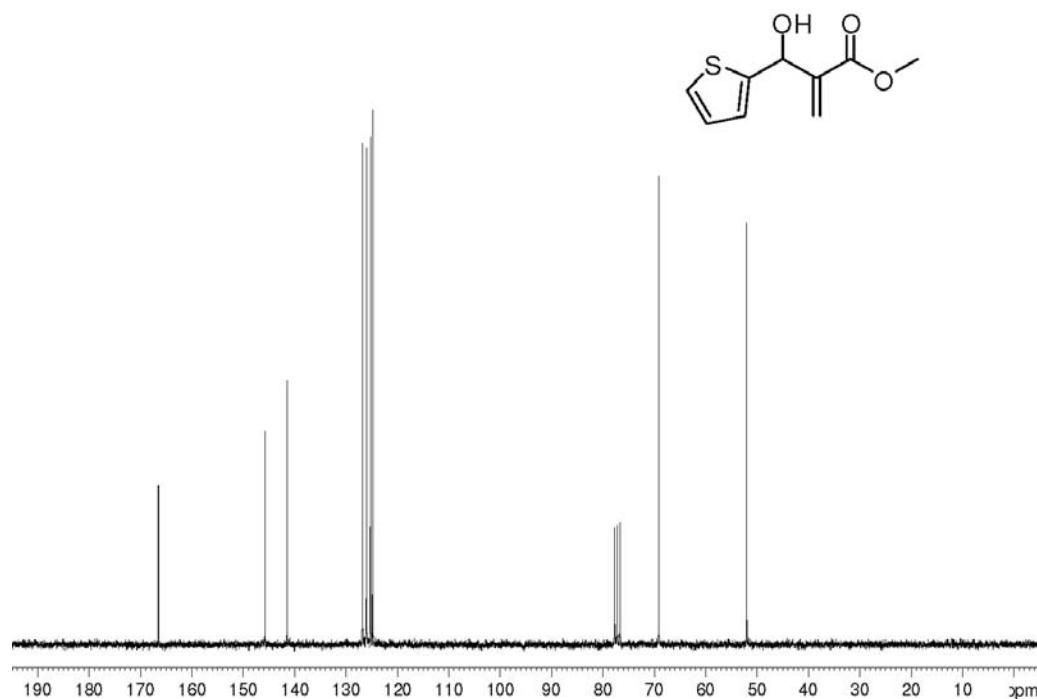


Figure S12. ¹³C NMR (CDCl₃, 62.5 MHz) of MBH adduct **8**.

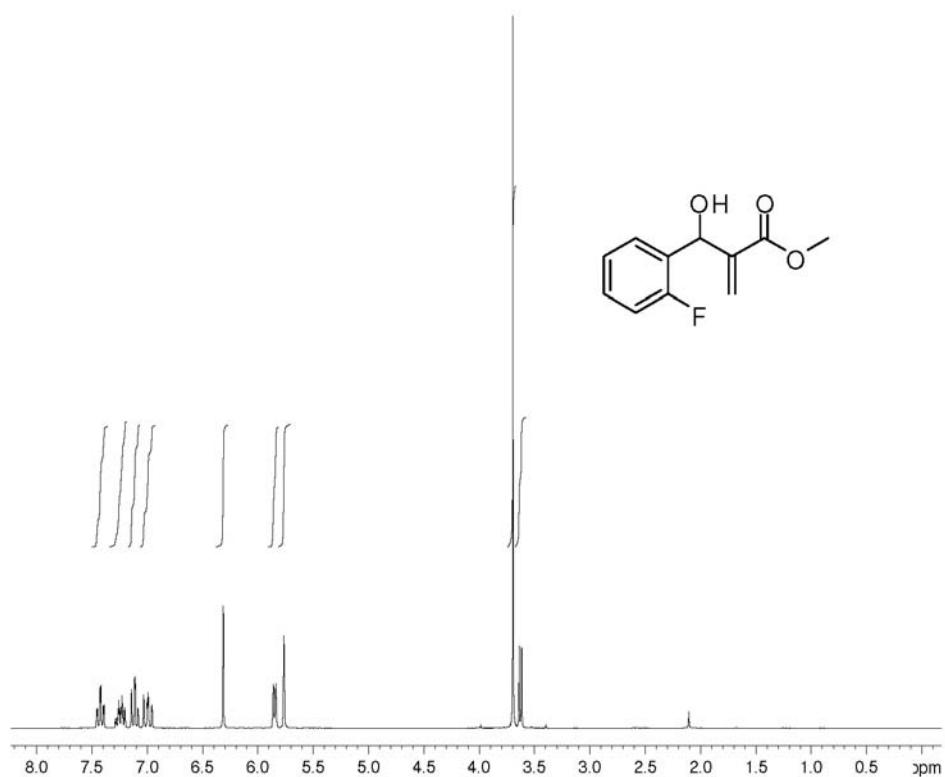


Figure S13. ¹H NMR (CDCl₃, 250 MHz) of MBH adduct **9**.

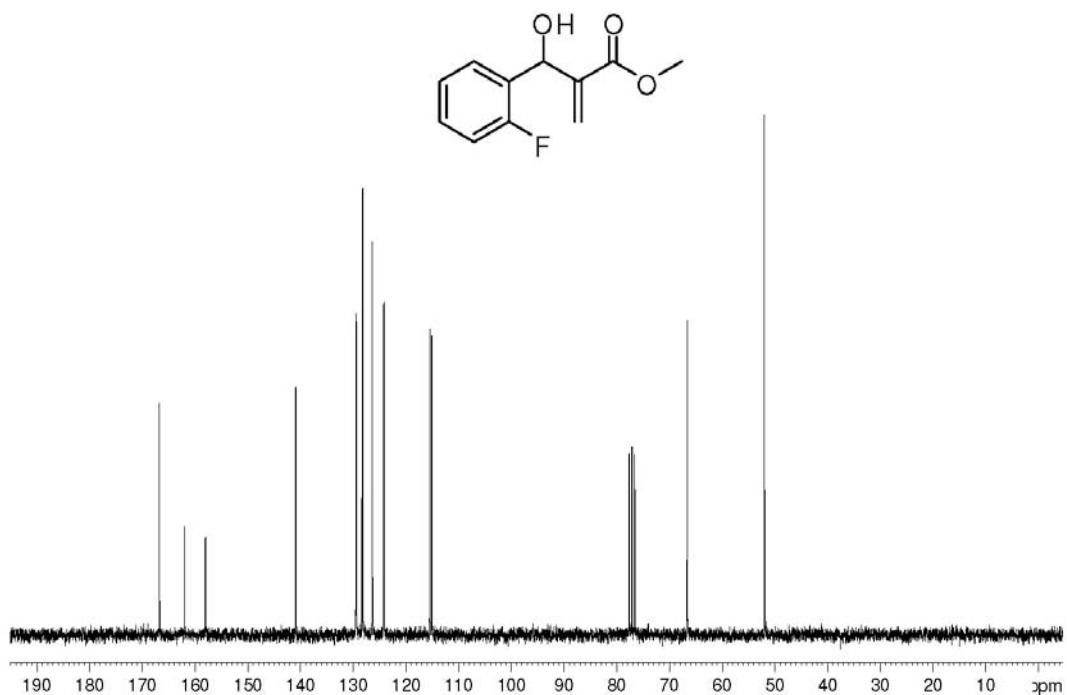


Figure S14. ¹³C NMR (CDCl₃, 62.5 MHz) of MBH adduct **9**.

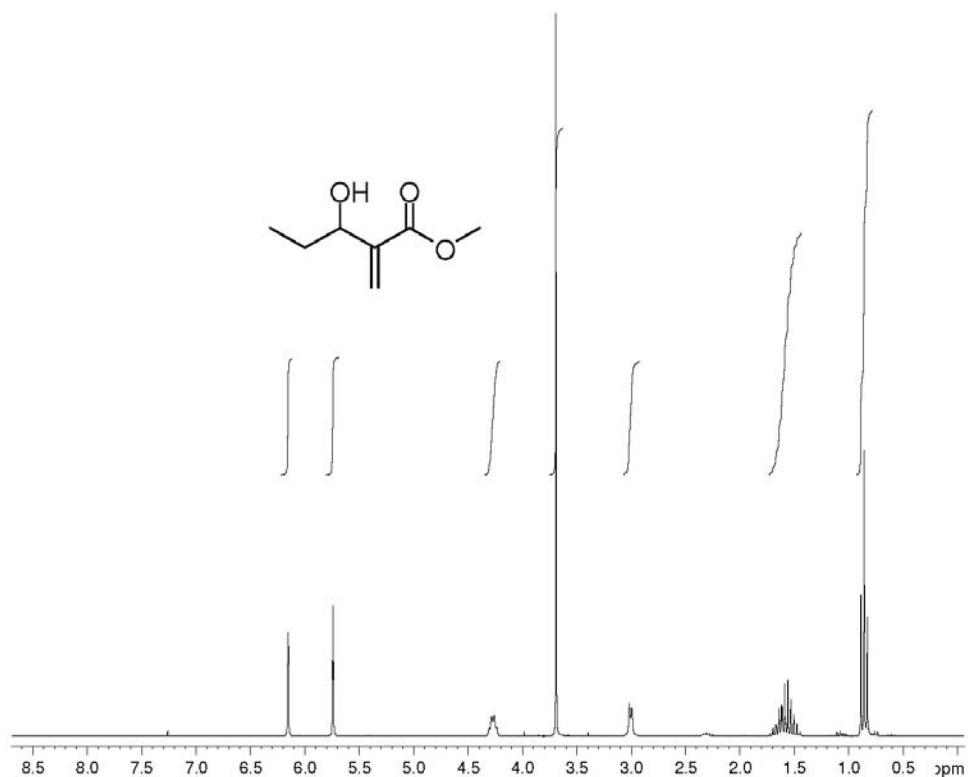


Figure S15. ¹H NMR (CDCl₃, 250 MHz) of MBH adduct **10**.

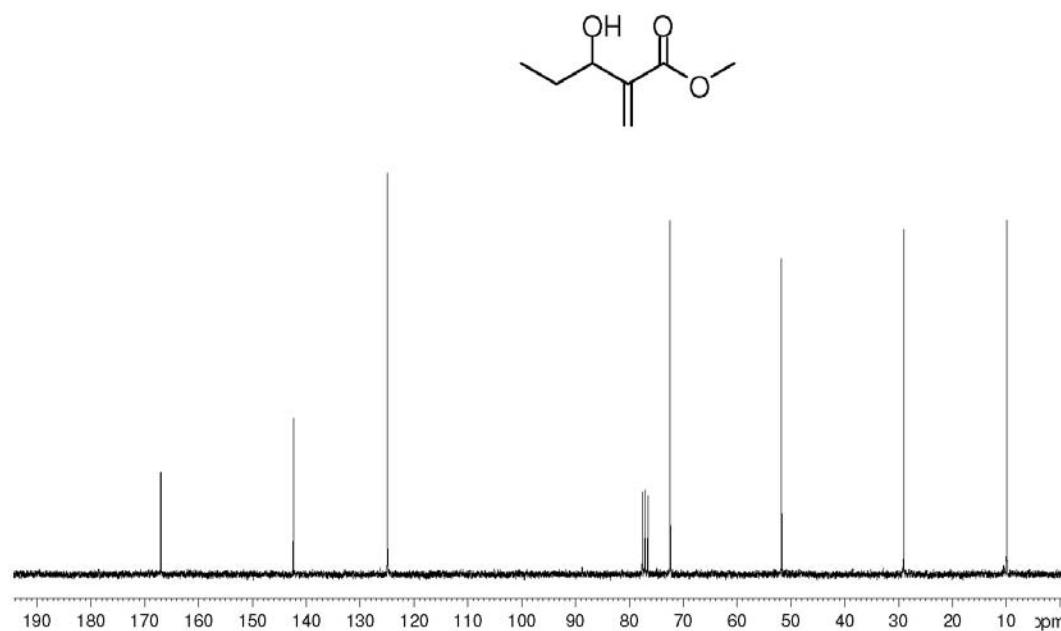


Figure S16. ^{13}C NMR (CDCl_3 , 62.5 MHz) of MBH adduct **10**.

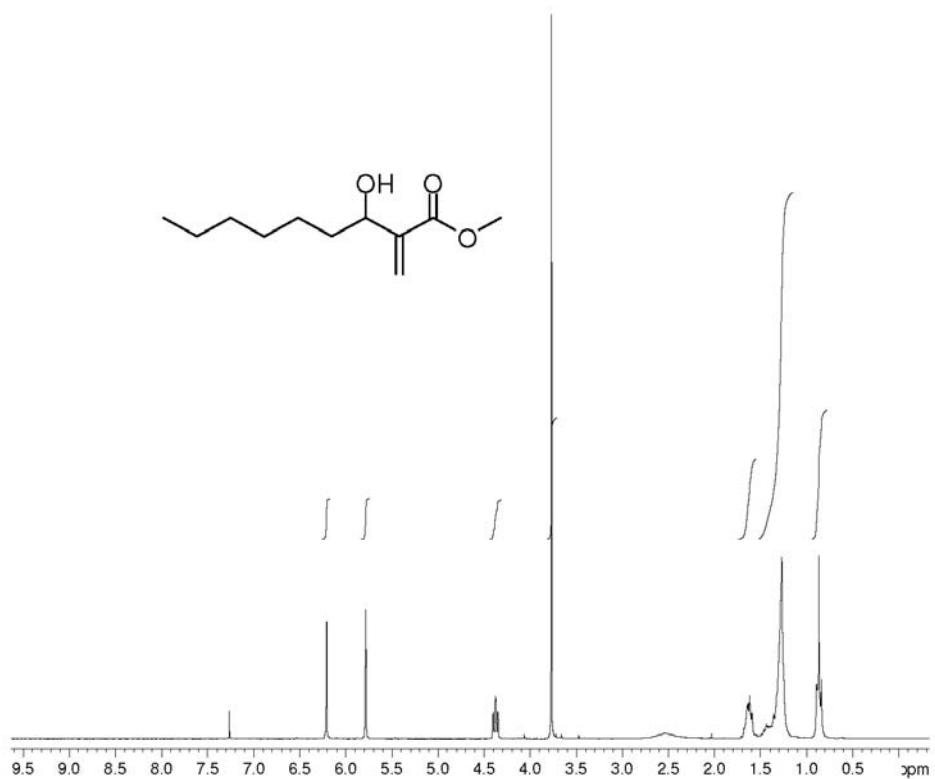


Figure S17. ^1H NMR (CDCl_3 , 250 MHz) of MBH adduct **11**.

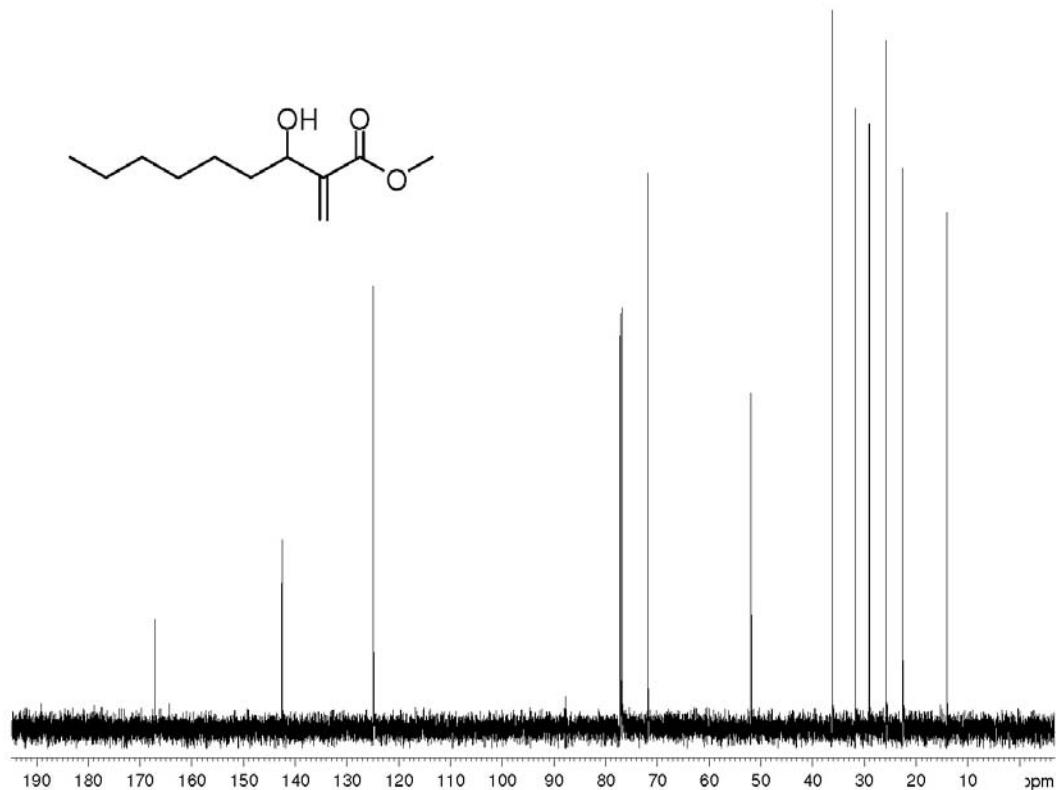


Figure S18. ¹³C NMR (CDCl_3 , 125 MHz) of MBH adduct **11**.

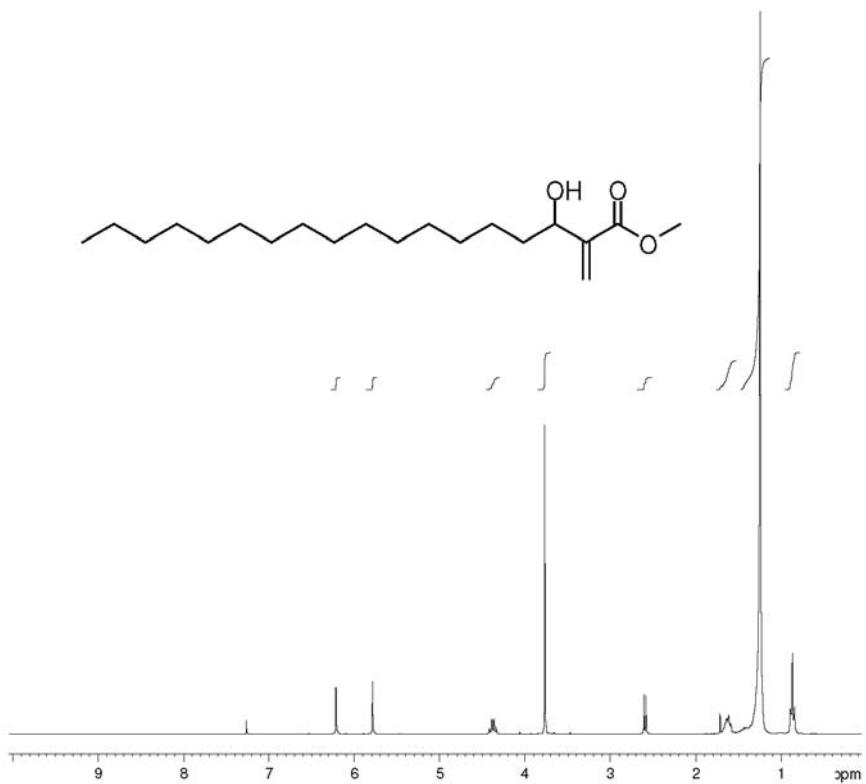


Figure S19. ¹H NMR (CDCl_3 , 250 MHz) of MBH adduct **12**.

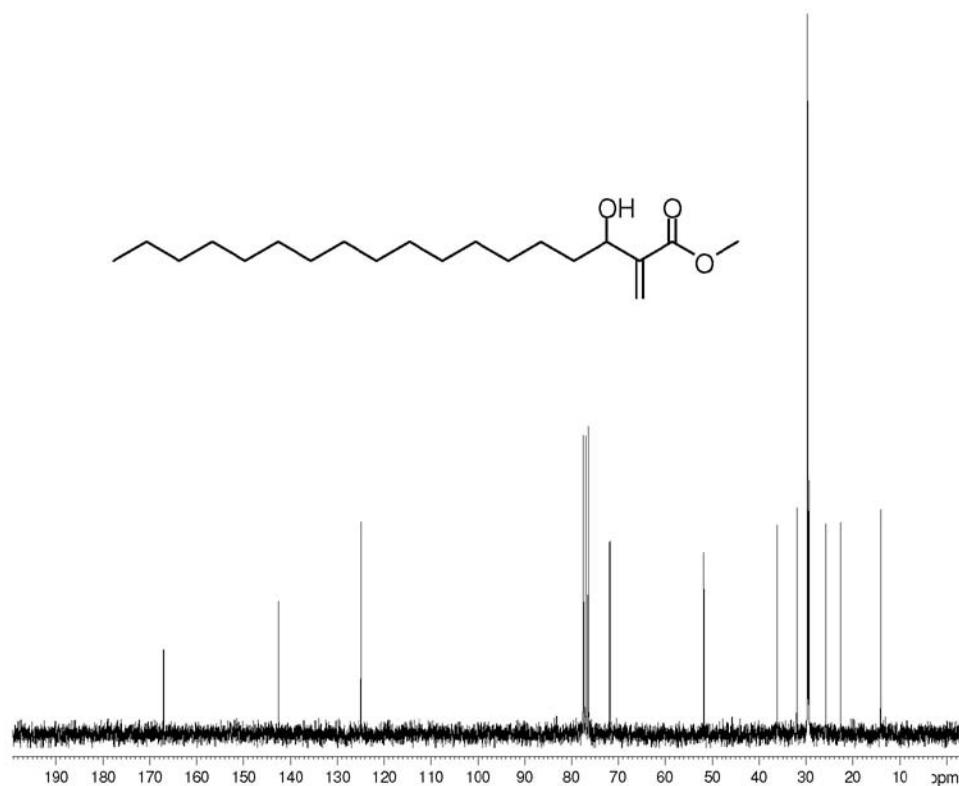


Figure S20. ^{13}C NMR (CDCl_3 , 125 MHz) of MBH adduct **12**.

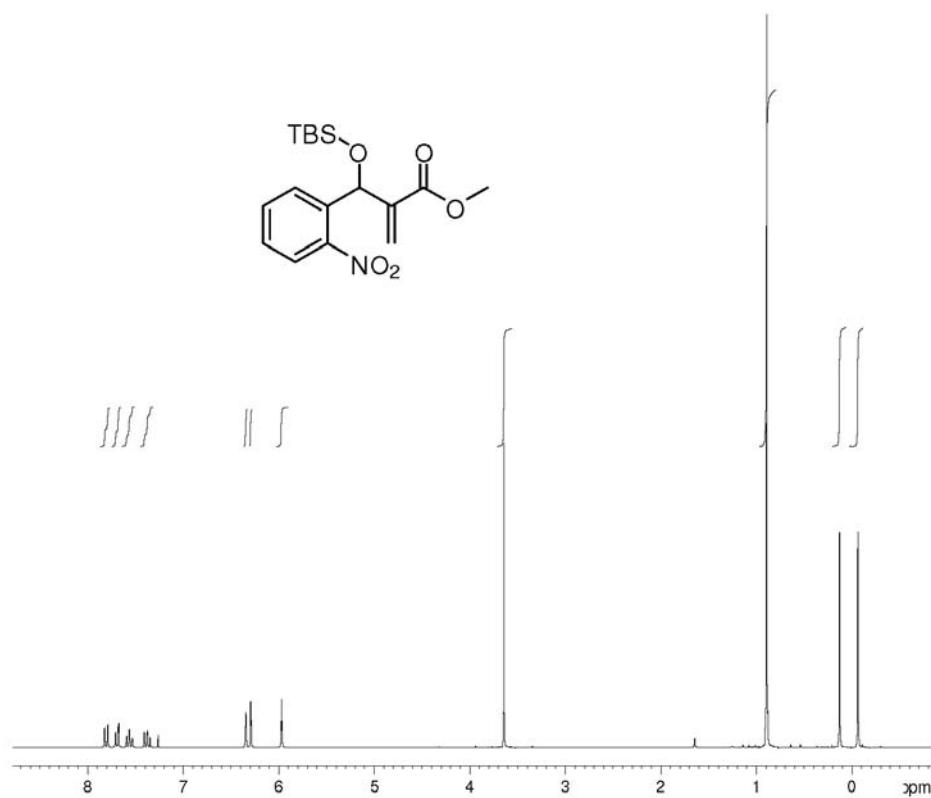


Figure S21. ^1H NMR (CDCl_3 , 250 MHz) of silylated MBH adduct **13**.

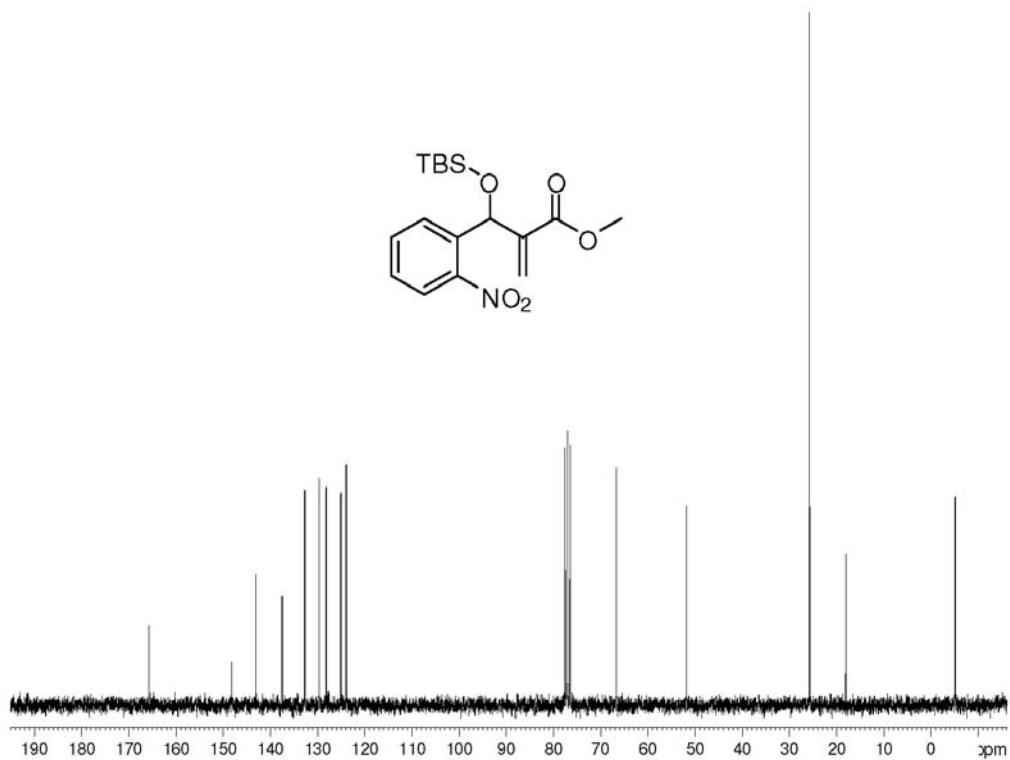


Figure S22. ^{13}C RMN (CDCl_3 , 62.5 MHz) of silylated MBH adduct **13**.

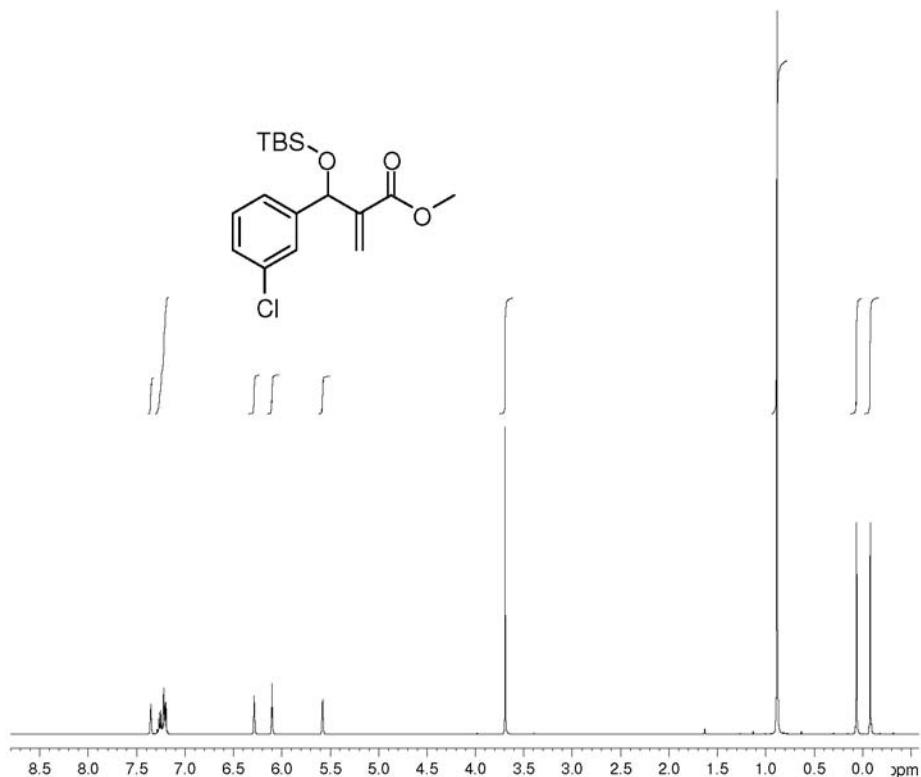


Figure S23. ^1H NMR (CDCl_3 , 250 MHz) of silylated MBH adduct **14**.

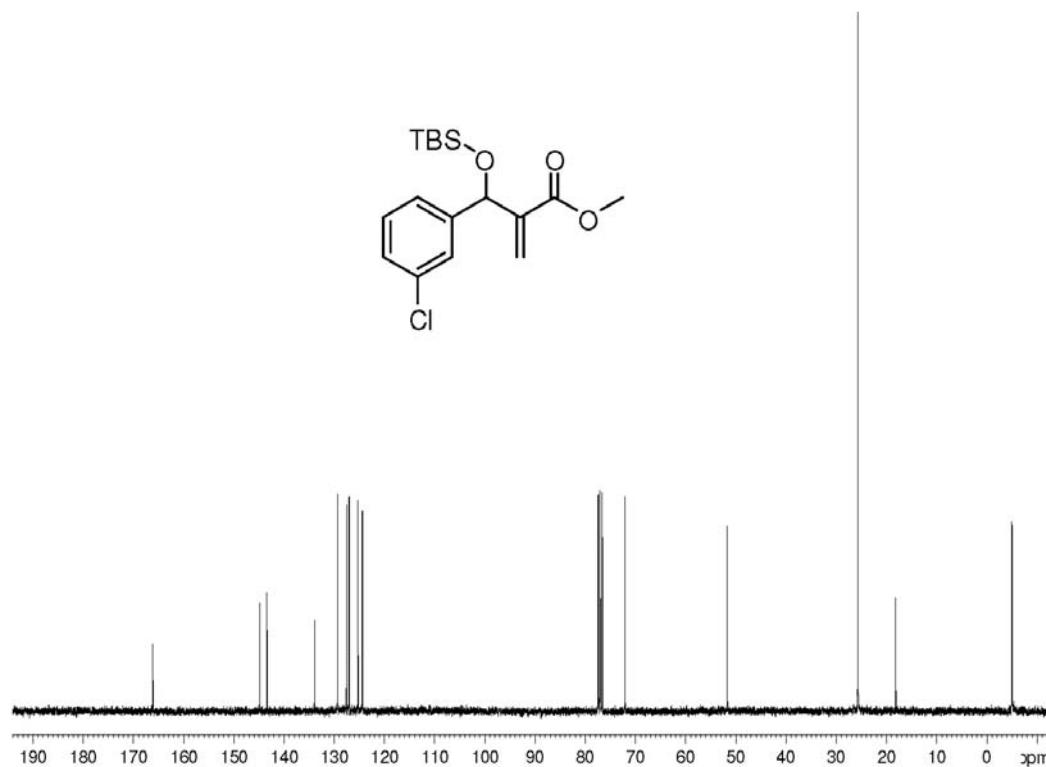


Figure S24. ^{13}C NMR (CDCl_3 , 62.5 MHz) of silylated MBH adduct **14**.

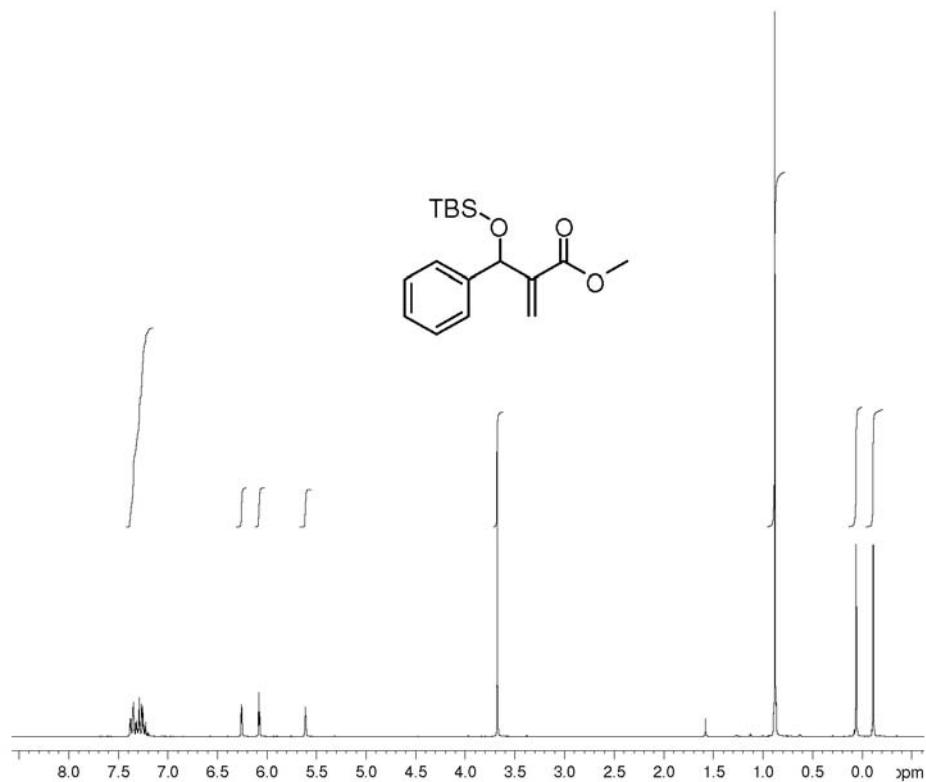


Figure S25. ^1H NMR (CDCl_3 , 250 MHz) of silylated MBH adduct **15**.

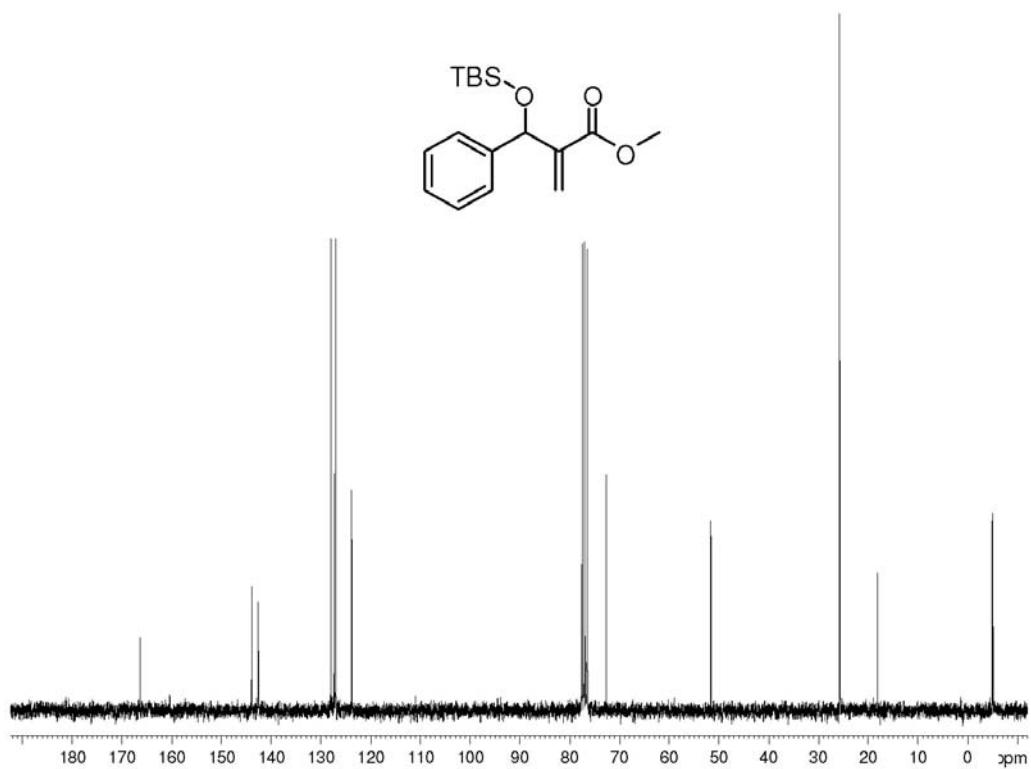


Figure S26. ^{13}C NMR (CDCl_3 , 62.5 MHz) of silylated MBH adduct **15**.

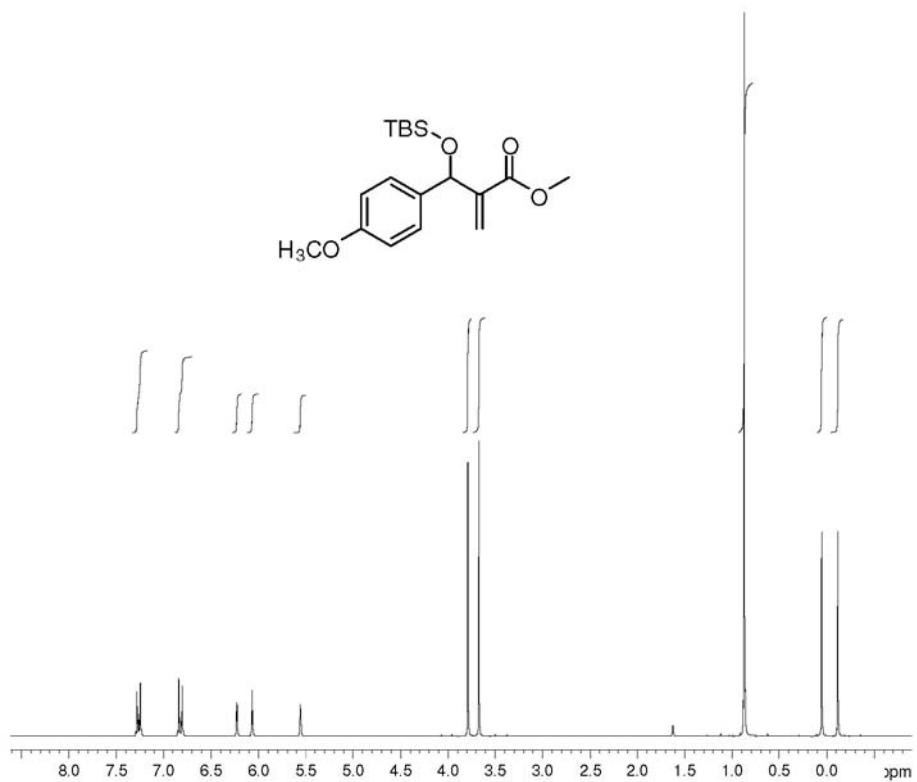


Figure S27. ^1H NMR (CDCl_3 , 250 MHz) of silylated MBH adduct **16**.

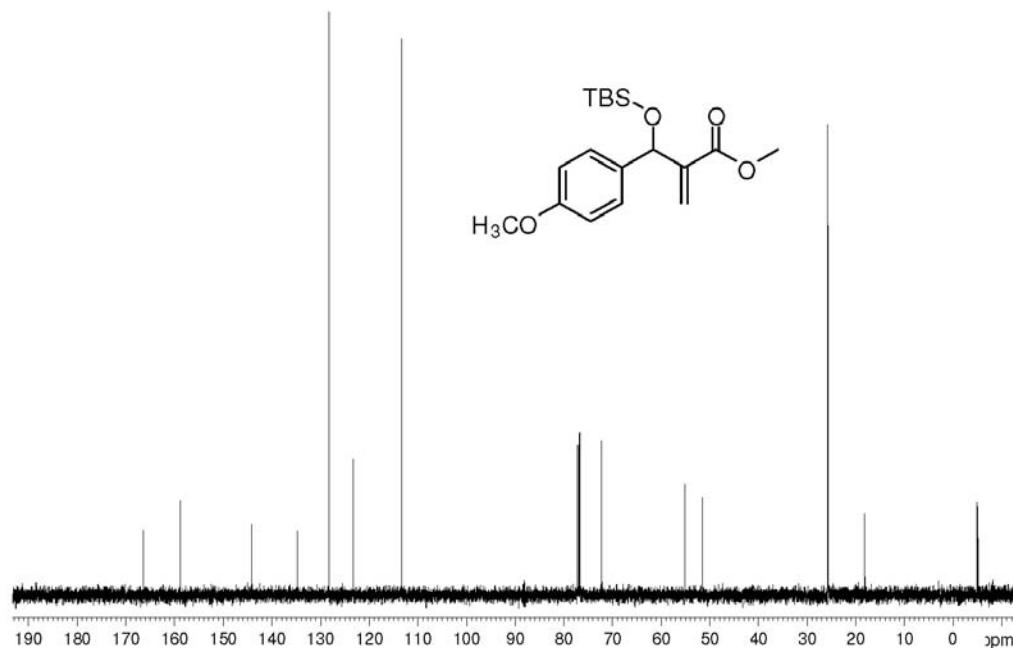


Figure S28. ¹³C NMR (CDCl₃, 125 MHz) of silylated MBH adduct **16**.

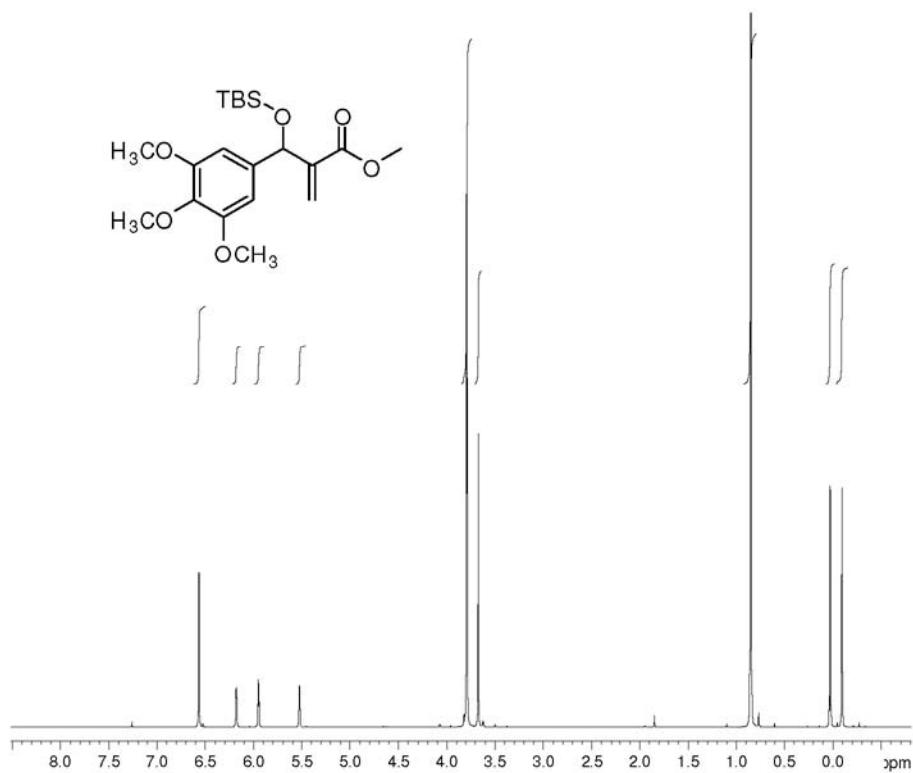


Figure S29. ¹H NMR (CDCl₃, 250 MHz) of silylated MBH adduct **17**.

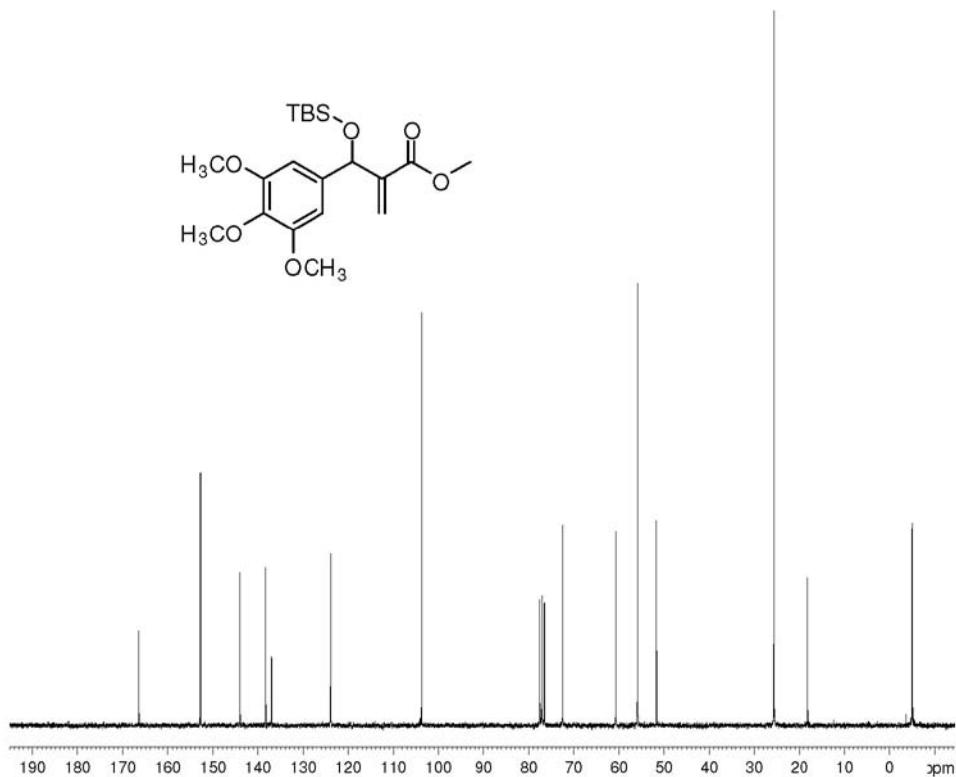


Figure S30. ^{13}C NMR (CDCl_3 , 62.5 MHz) of silylated MBH adduct **17**.

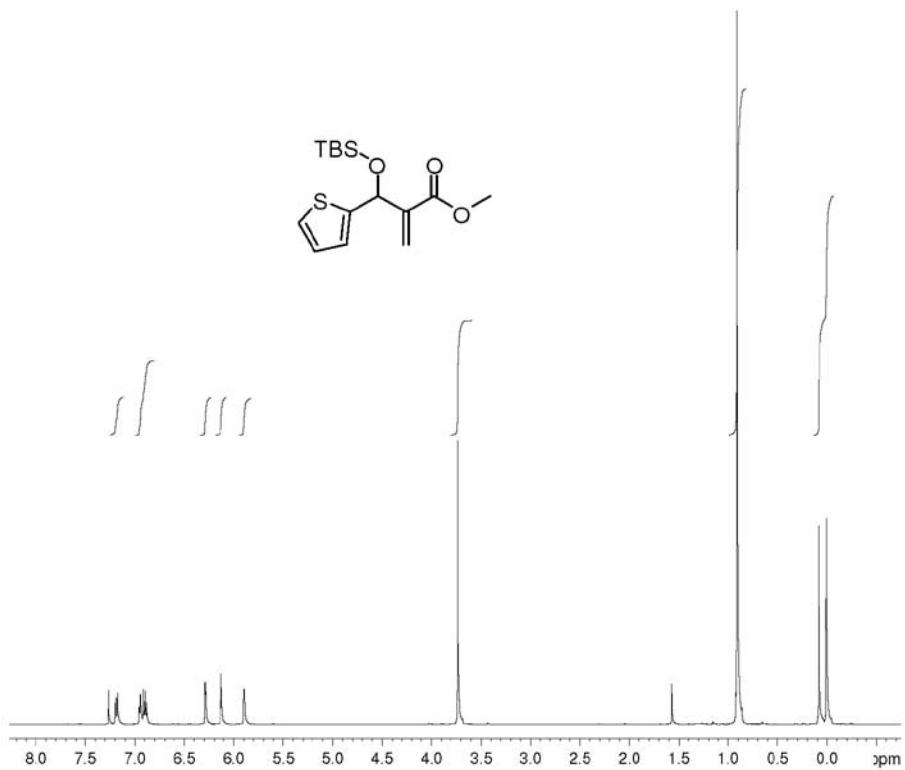


Figure S31. ^1H NMR (CDCl_3 , 250 MHz) of silylated MBH adduct **18**.

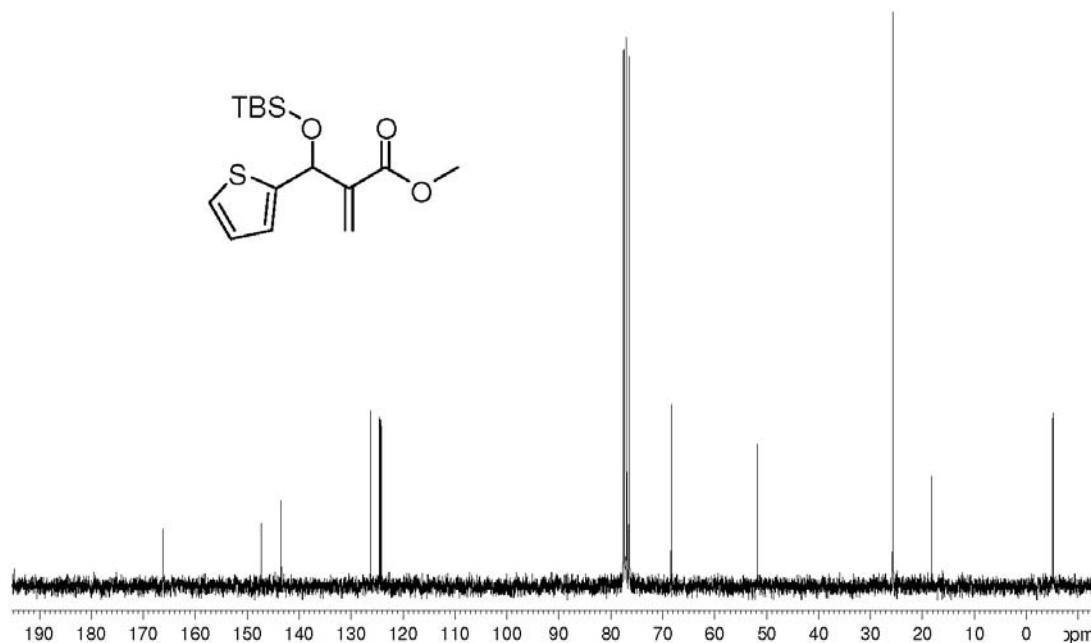


Figure S32. ^{13}C NMR (CDCl_3 , 62.5 MHz) of silylated MBH adduct **18**.

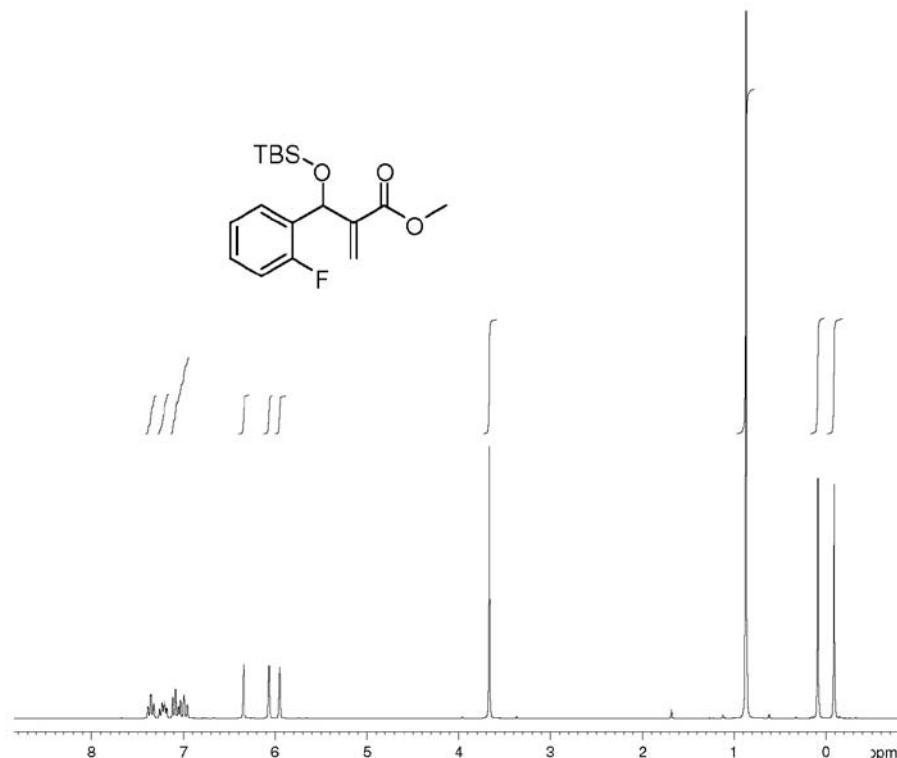


Figure S33. ^1H NMR (CDCl_3 , 250 MHz) of silylated MBH adduct **19**.

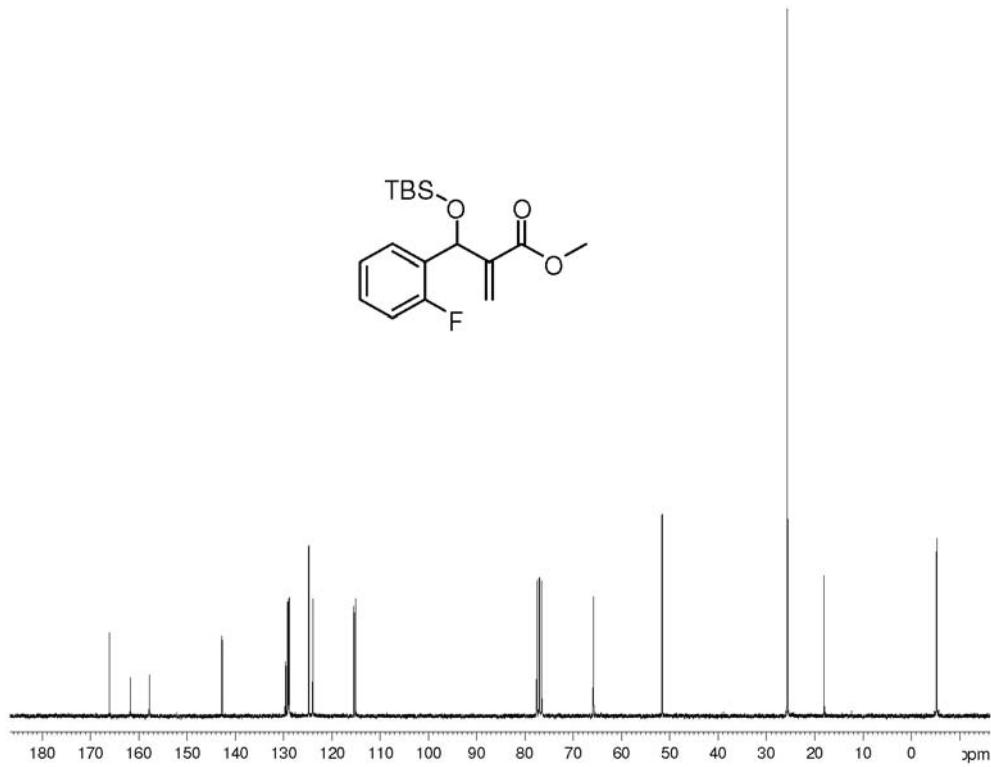


Figure S34. ^{13}C NMR (CDCl_3 , 62.5 MHz) of silylated MBH adduct **19**.

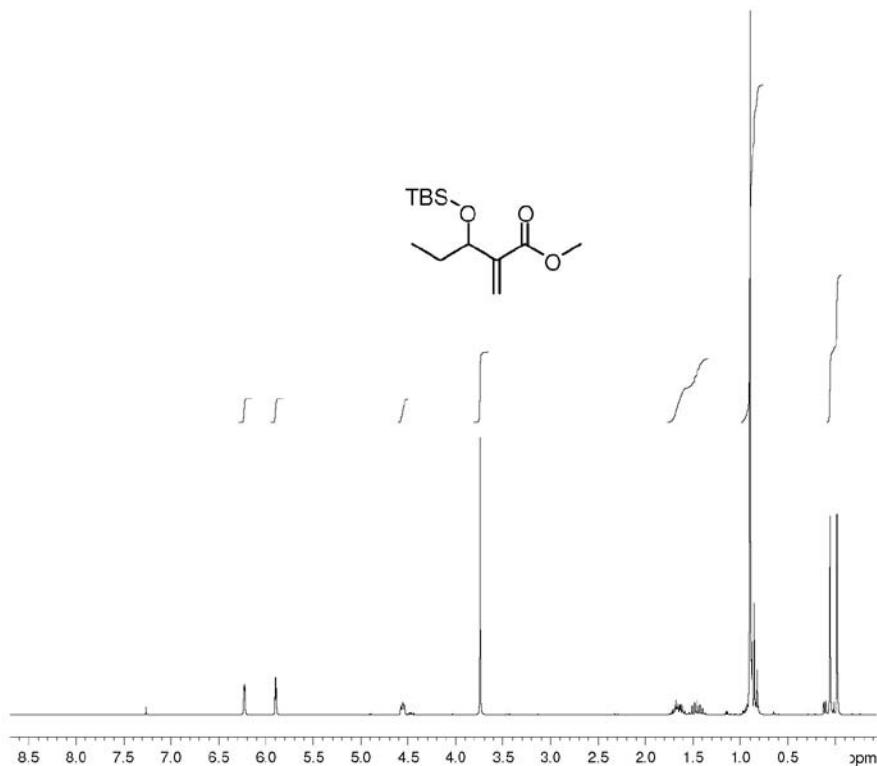


Figure S35. ^1H NMR (CDCl_3 , 250 MHz) of silylated MBH adduct **20**.

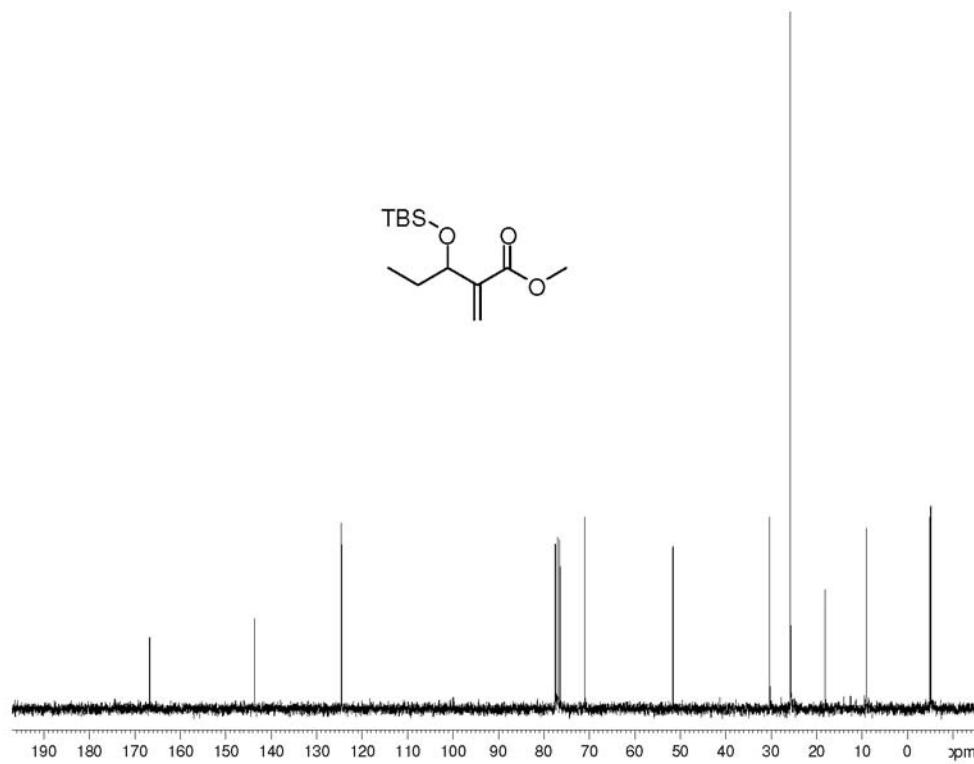


Figure S36. ^{13}C NMR (CDCl_3 , 62.5 MHz) of silylated MBH adduct **20**.

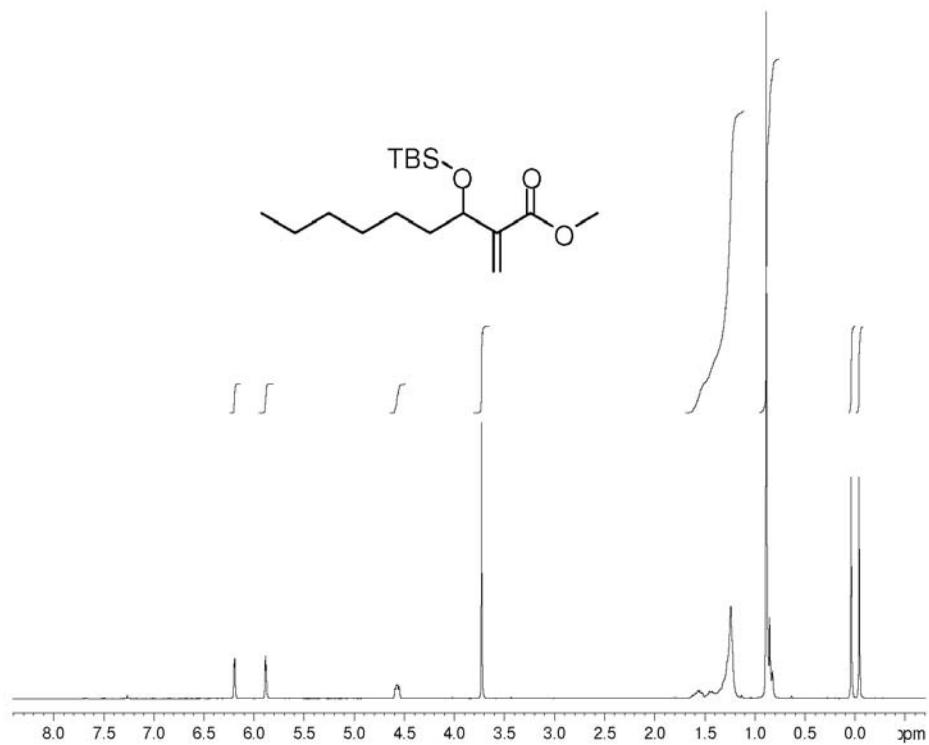


Figure S37. ^1H NMR (CDCl_3 , 250 MHz) of silylated MBH adduct **21**.

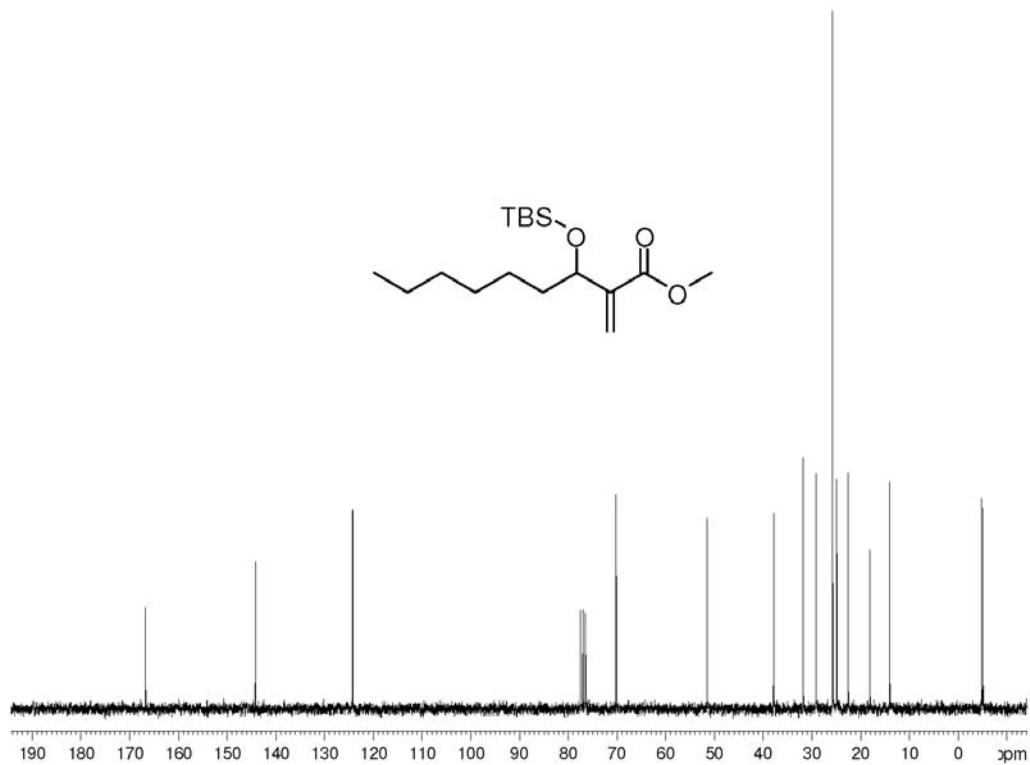


Figure S38. ^{13}C NMR (CDCl_3 , 62.5 MHz) of silylated MBH adduct **21**.

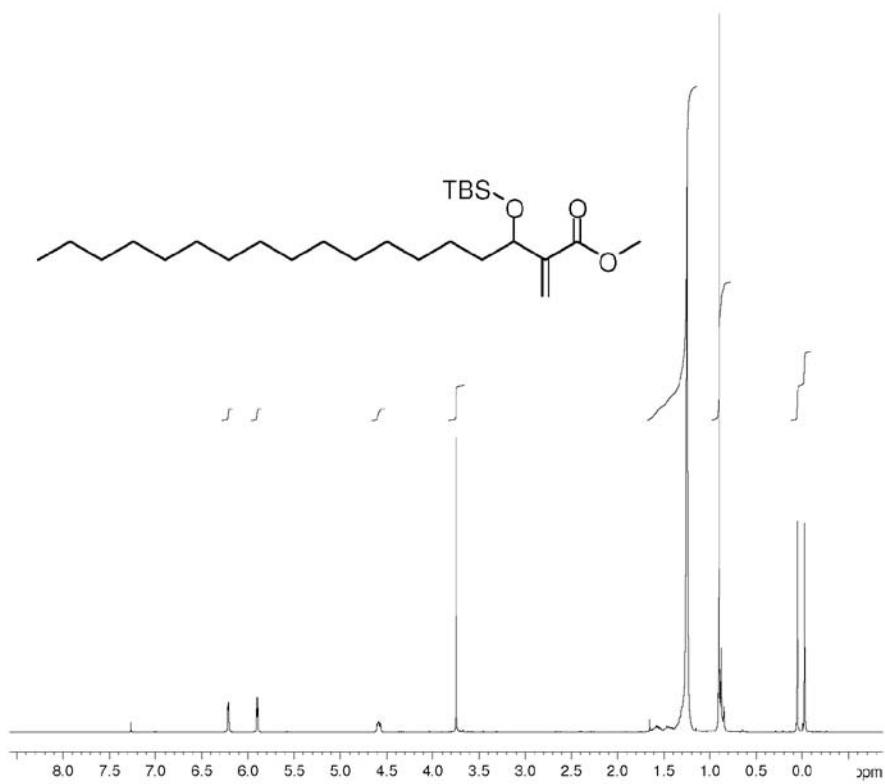


Figure S39. ^1H NMR (CDCl_3 , 250 MHz) of silylated MBH adduct **22**.

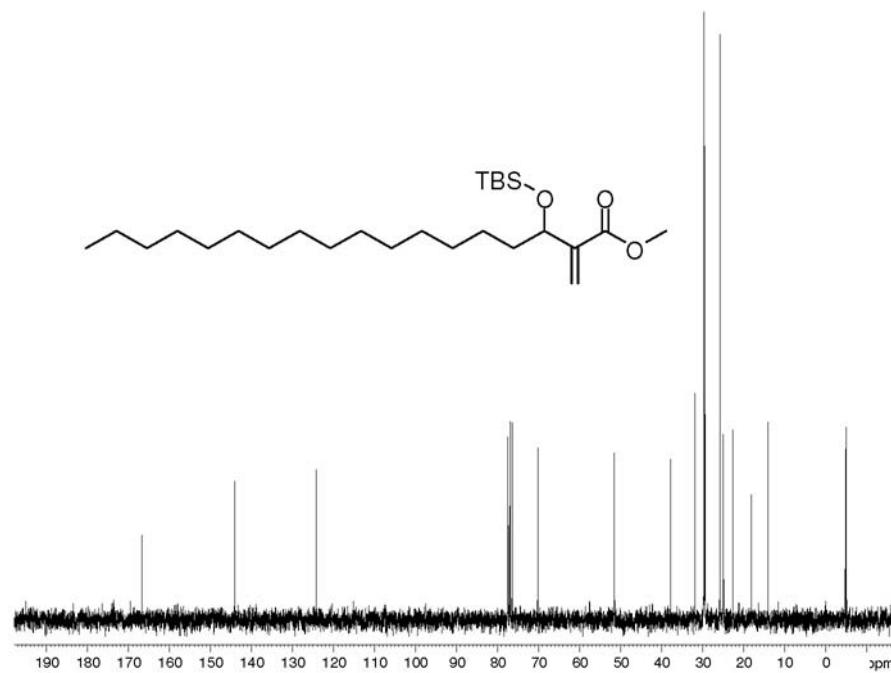


Figure S40. ^{13}C NMR (CDCl_3 , 62.5 MHz) of silylated MBH adduct **22**.

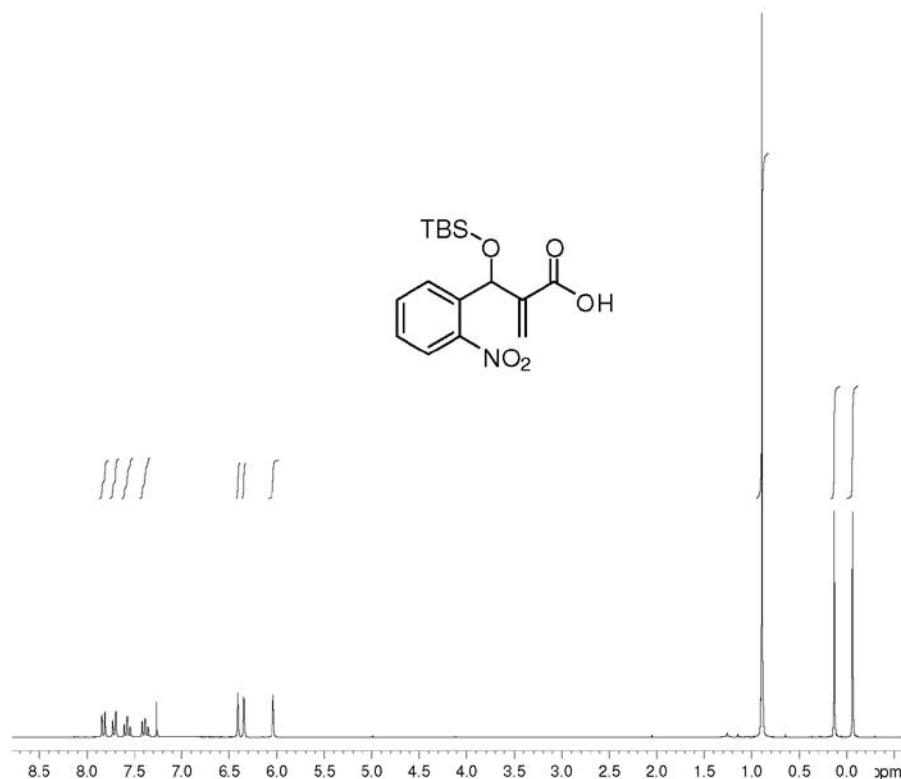


Figure S41. ^1H NMR (CDCl_3 , 250 MHz) of silylated acid **23**.

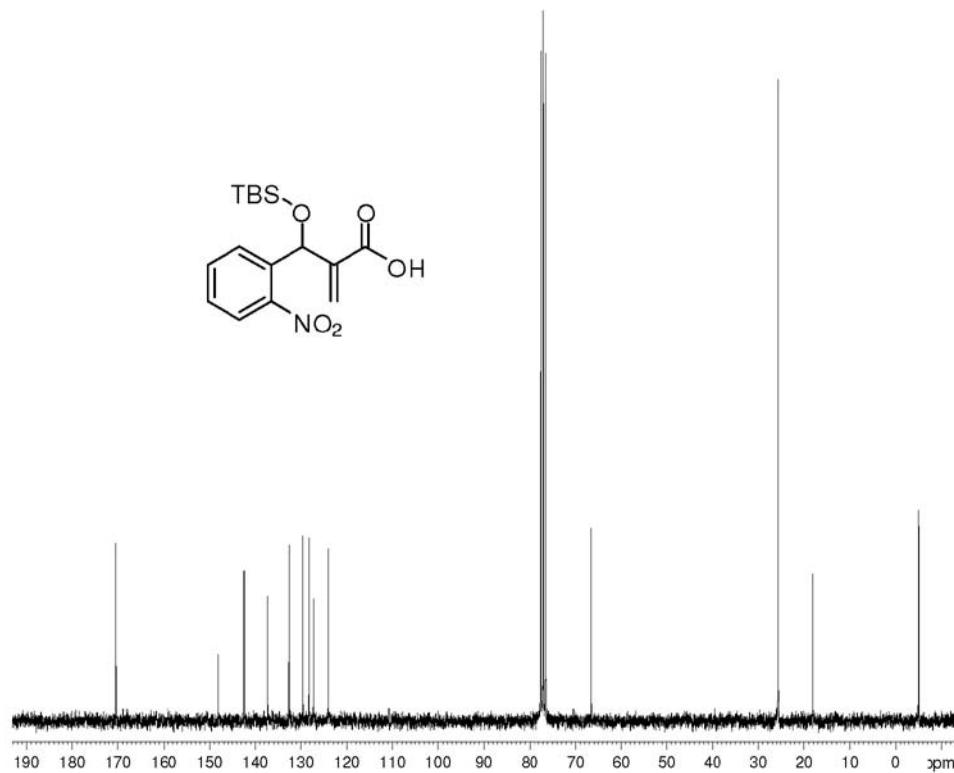


Figure S42. ^{13}C NMR (CDCl_3 , 62.5 MHz) of silylated acid 23.

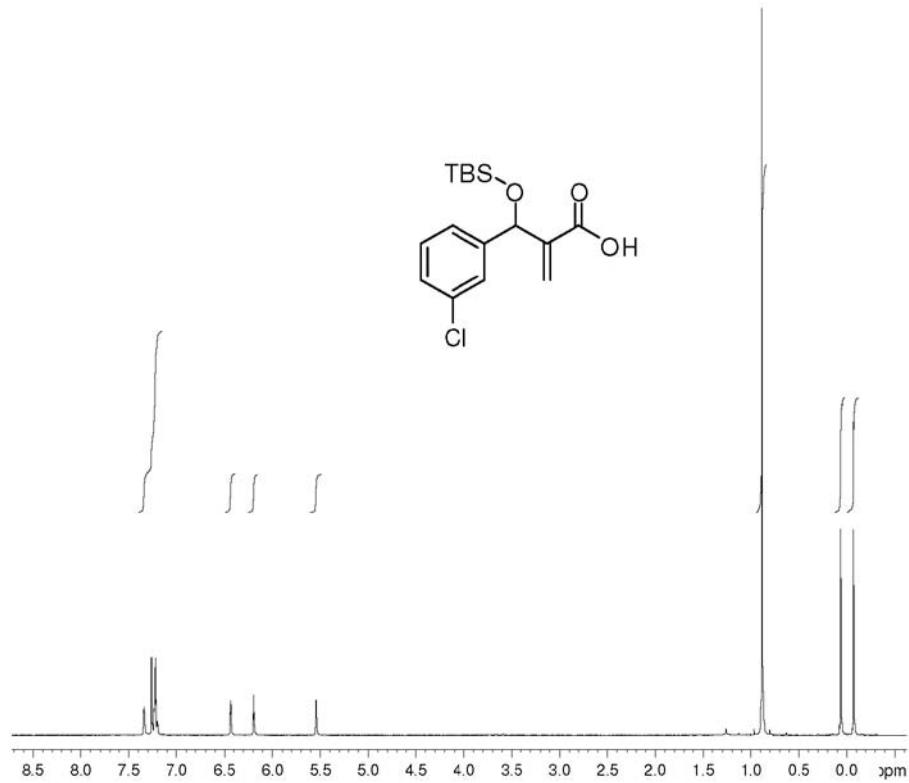


Figure S43. ^1H NMR (CDCl_3 , 250 MHz) of silylated acid 24.

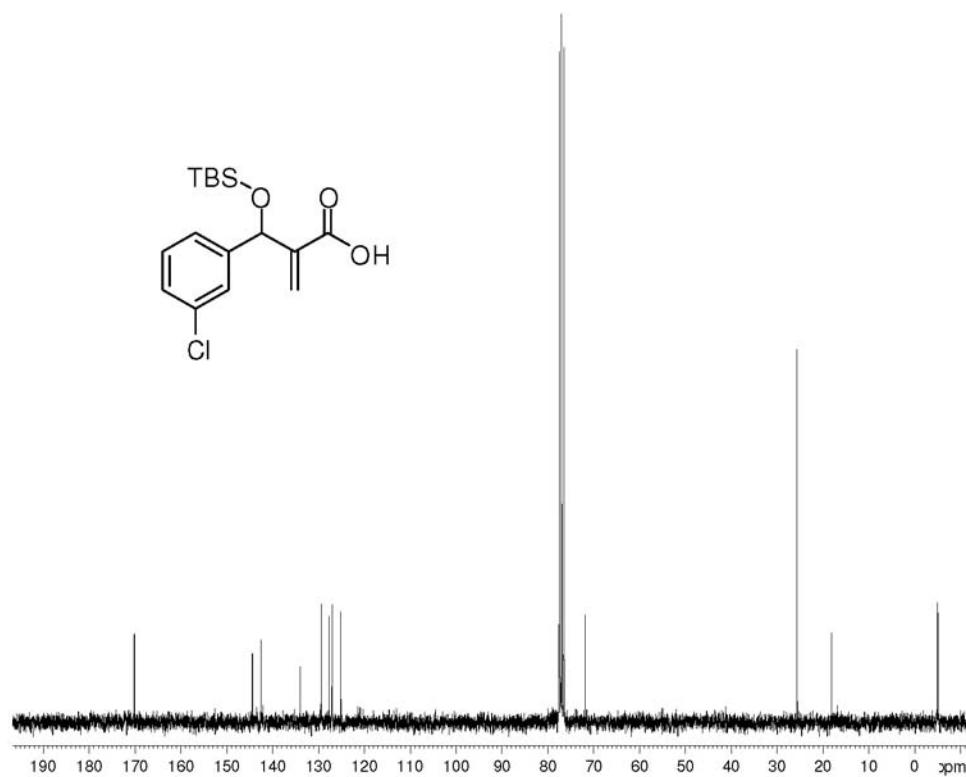


Figure S44. ^{13}C NMR (CDCl_3 , 62.5 MHz) of silylated acid 24.

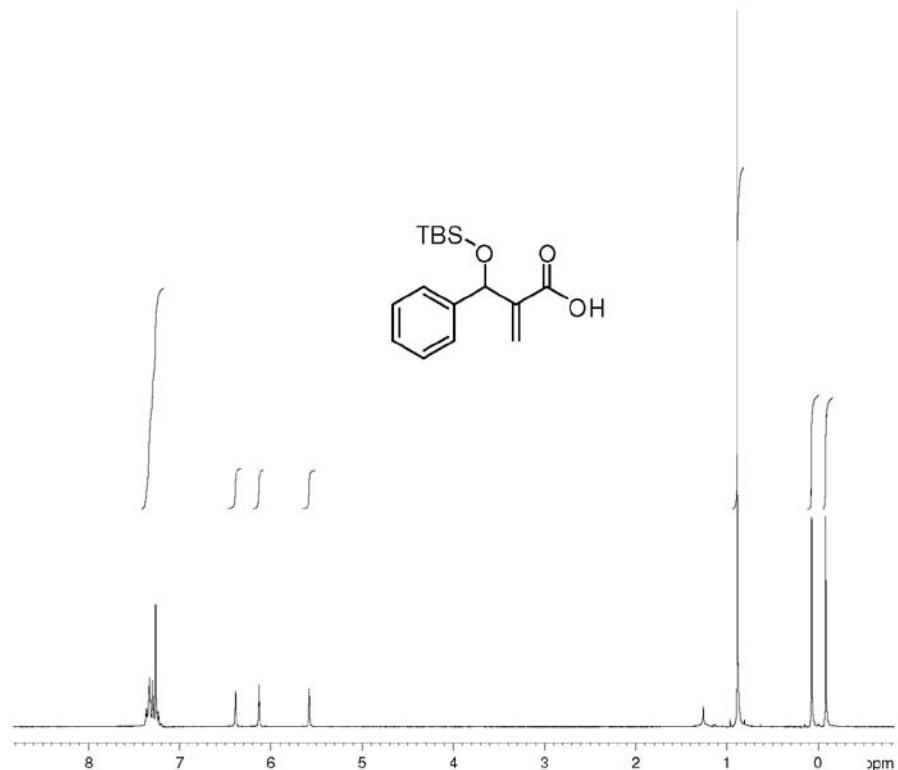


Figure S45. ^1H NMR (CDCl_3 , 250 MHz) of silylated acid 25.

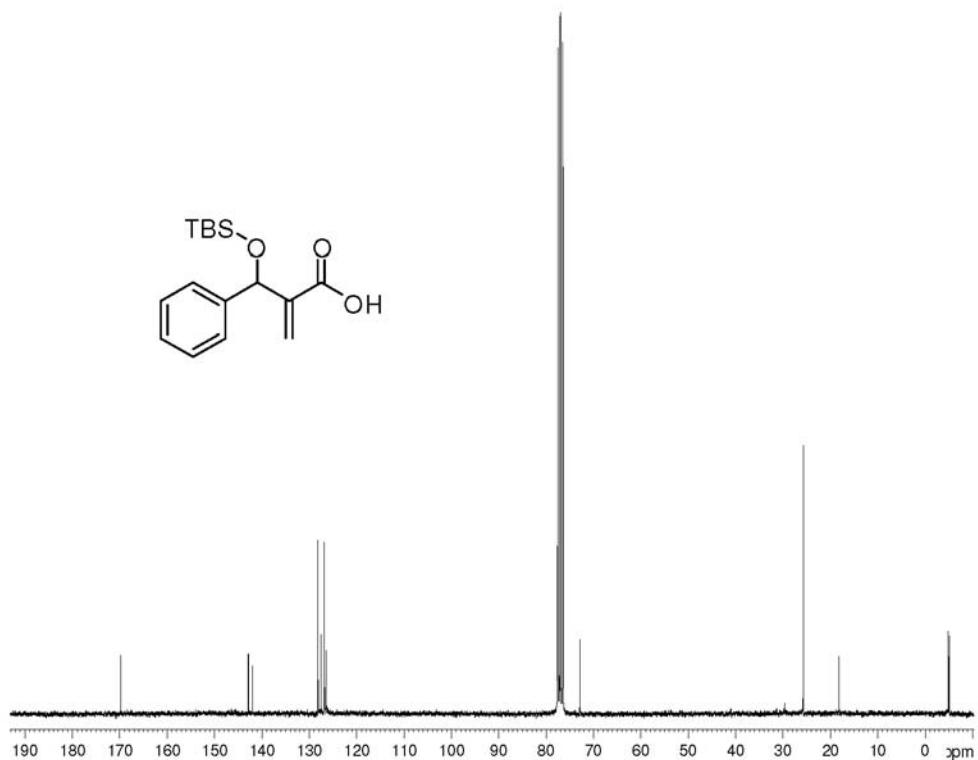


Figure S46. ^{13}C NMR (CDCl_3 , 62.5 MHz) of silylated acid **25**.

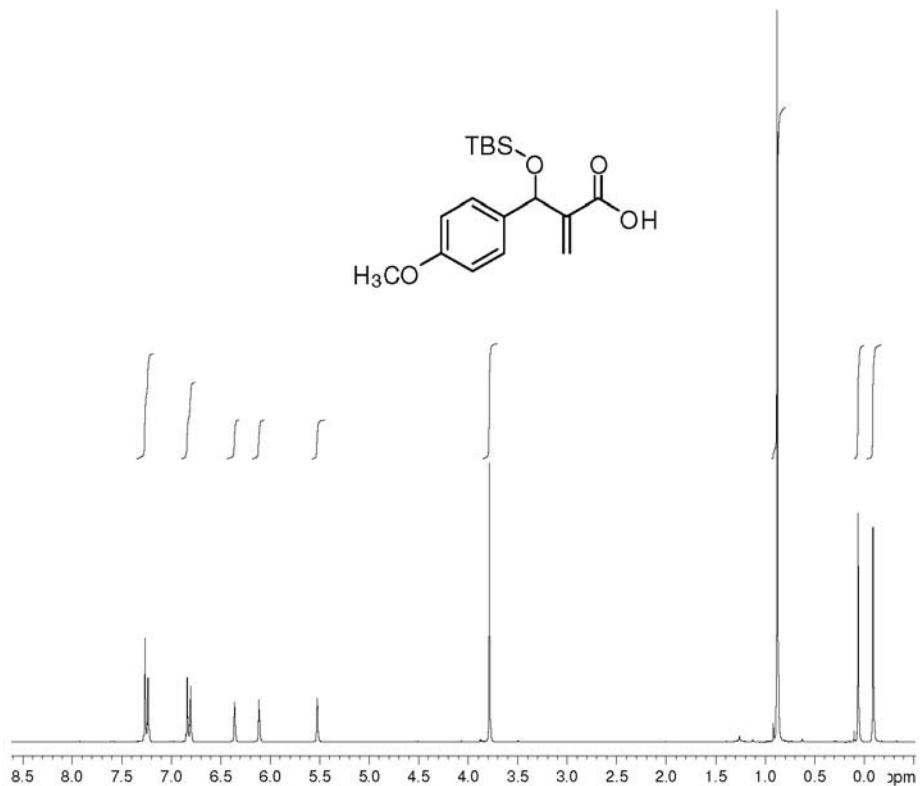


Figure S47. ^1H NMR (CDCl_3 , 250 MHz) of silylated acid **26**.

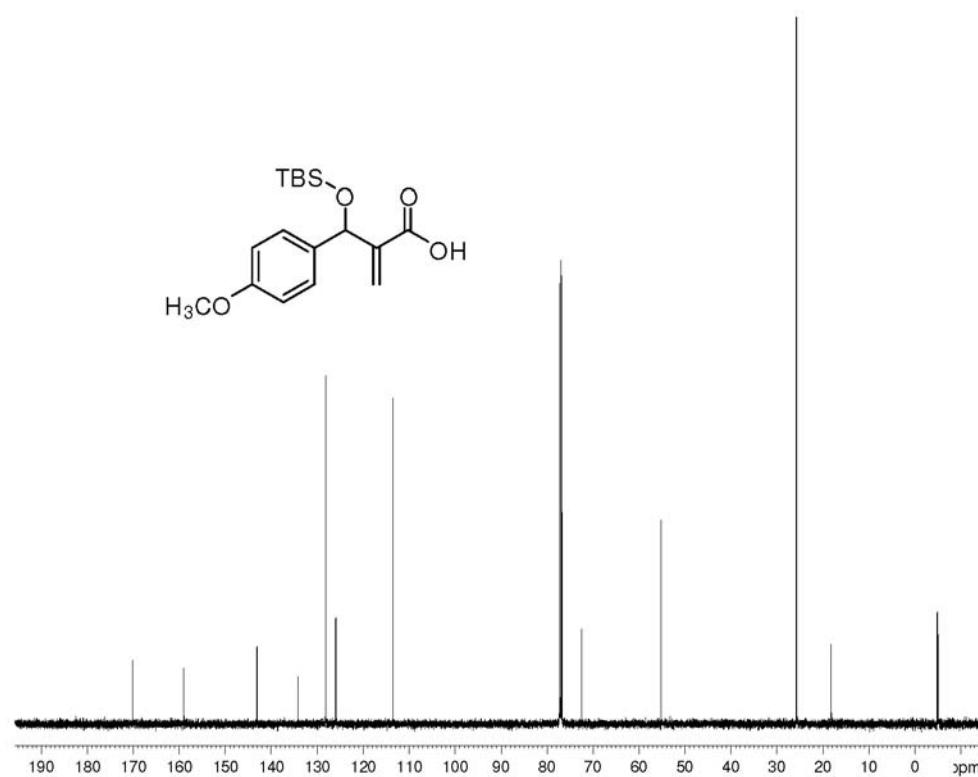


Figure S48. ^{13}C NMR (CDCl_3 , 125 MHz) of silylated acid 26.

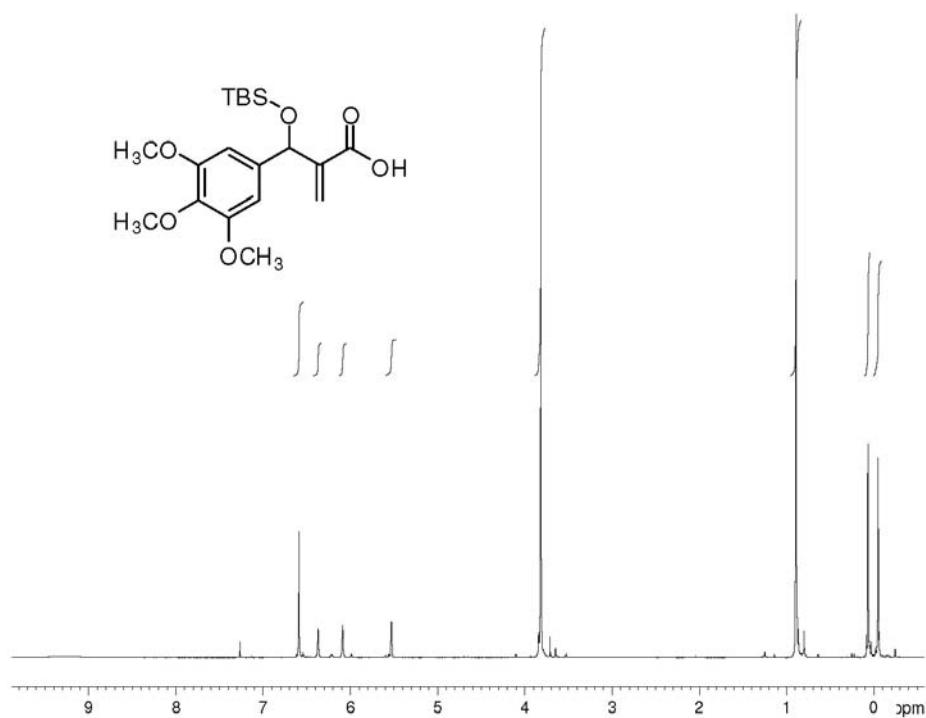


Figure S49. ^1H NMR (CDCl_3 , 250 MHz) of silylated acid 27.

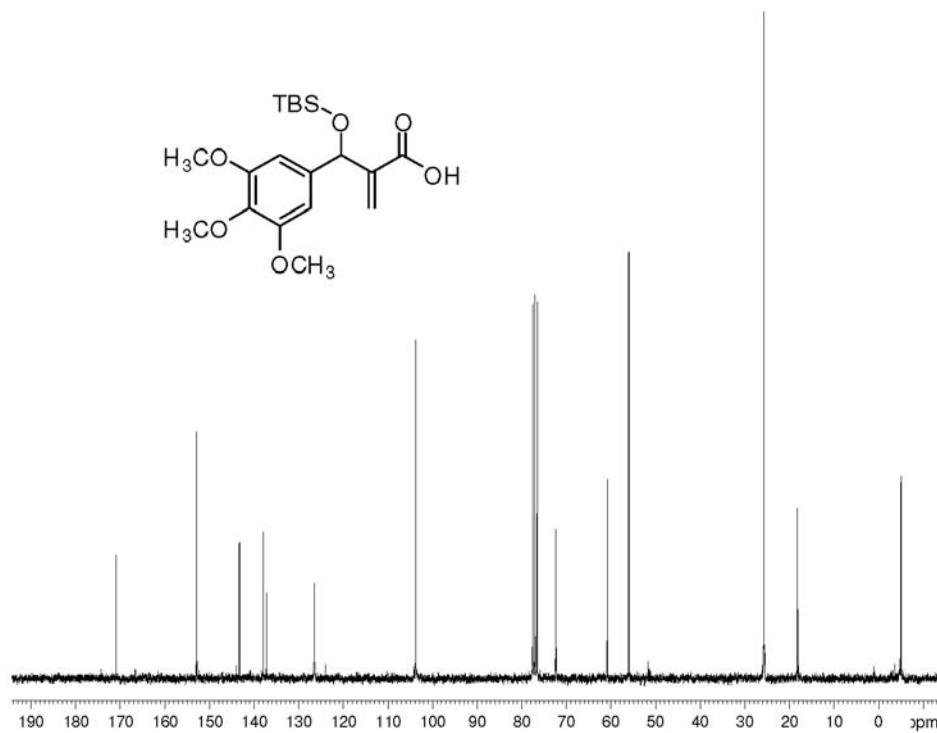


Figure S50. ^{13}C NMR (CDCl_3 , 62.5 MHz) of silylated acid 27.

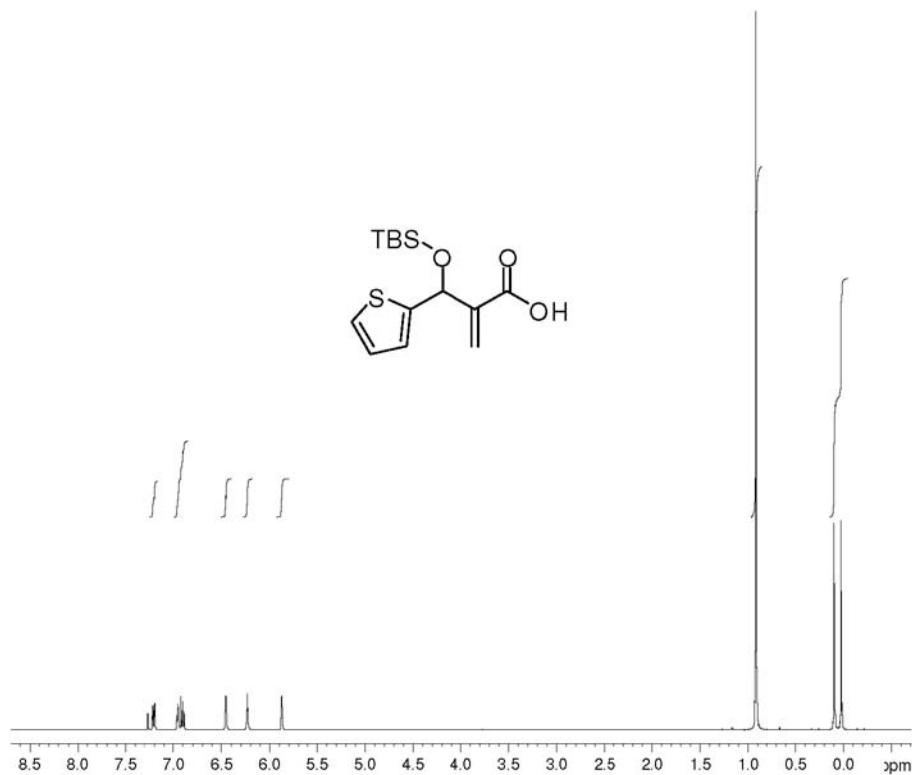


Figure S51. ^1H NMR (CDCl_3 , 250 MHz) of silylated acid 28.

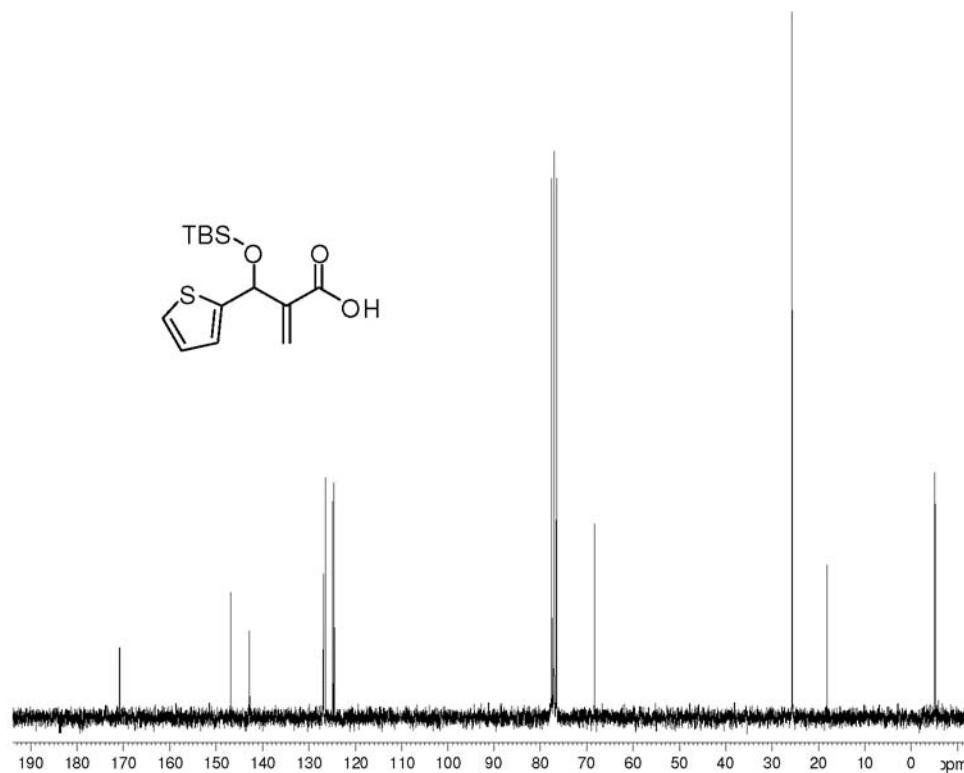


Figure S52. ^{13}C NMR (CDCl_3 , 62.5 MHz) of silylated acid 28.

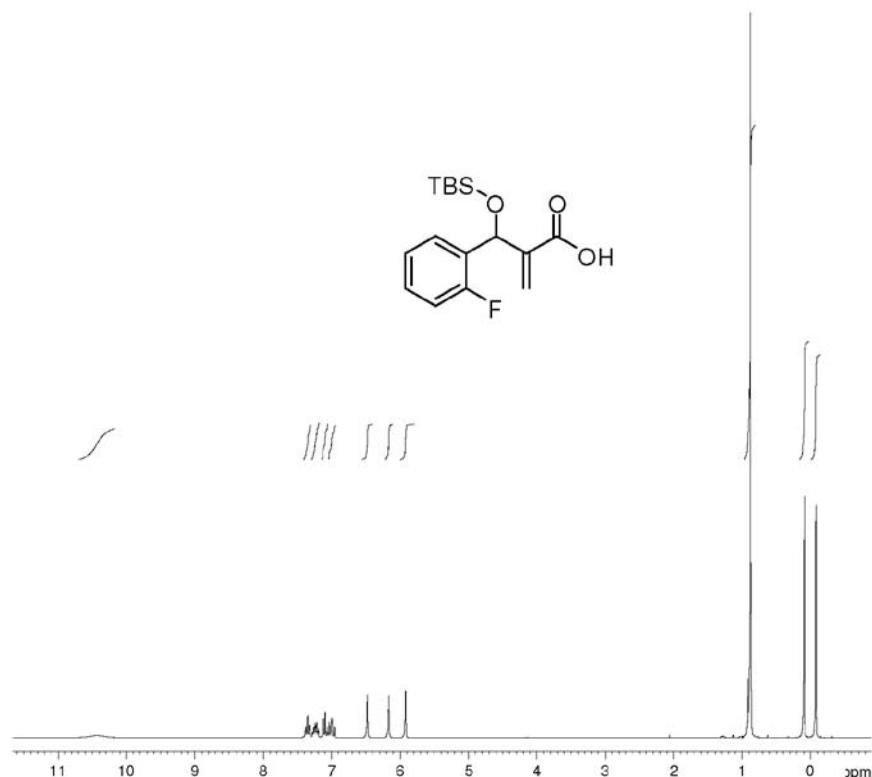


Figure S53. ^1H NMR (CDCl_3 , 250 MHz) of silylated acid 29.

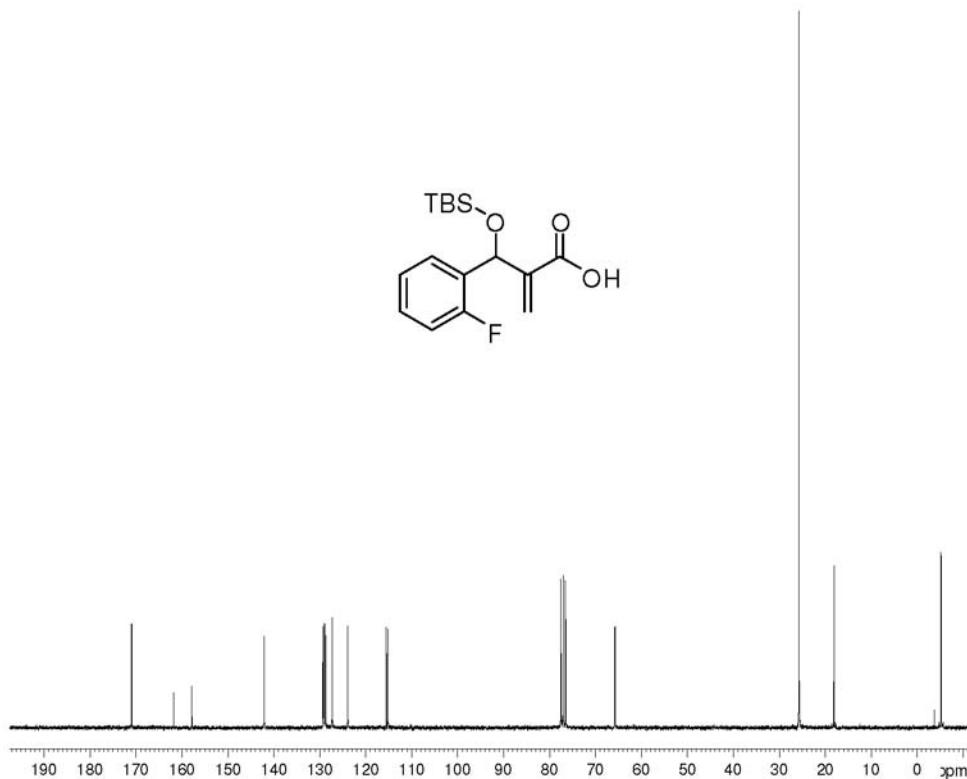


Figure S54. ¹³C NMR (CDCl_3 , 62.5 MHz) of silylated acid **29**.

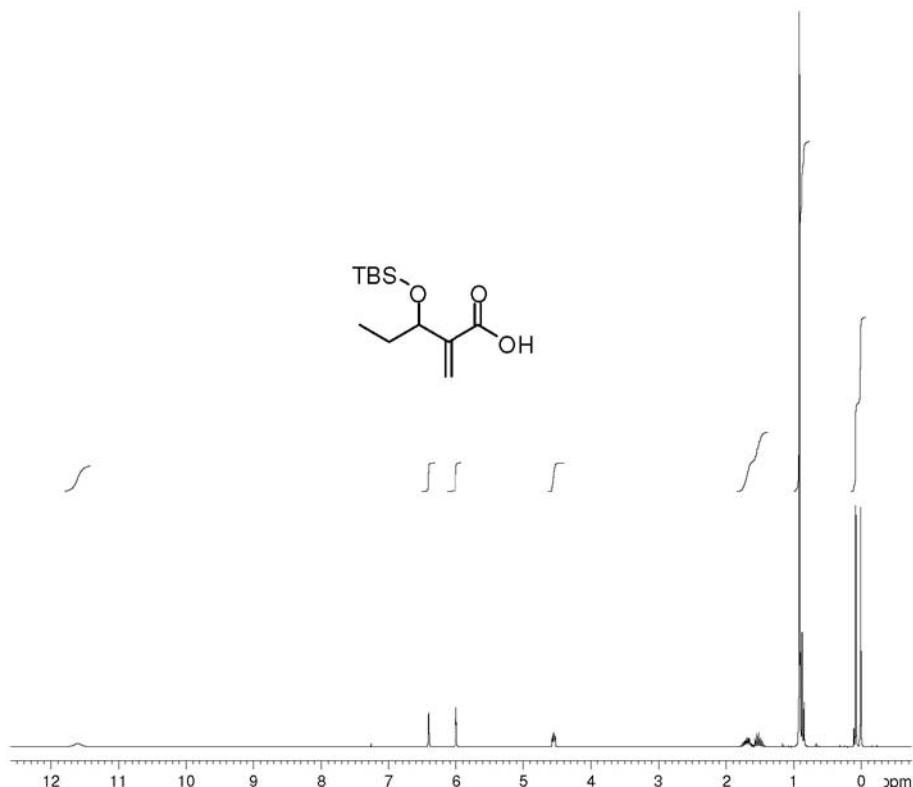


Figure S55. ¹H NMR (CDCl_3 , 250 MHz) of silylated acid **30**.

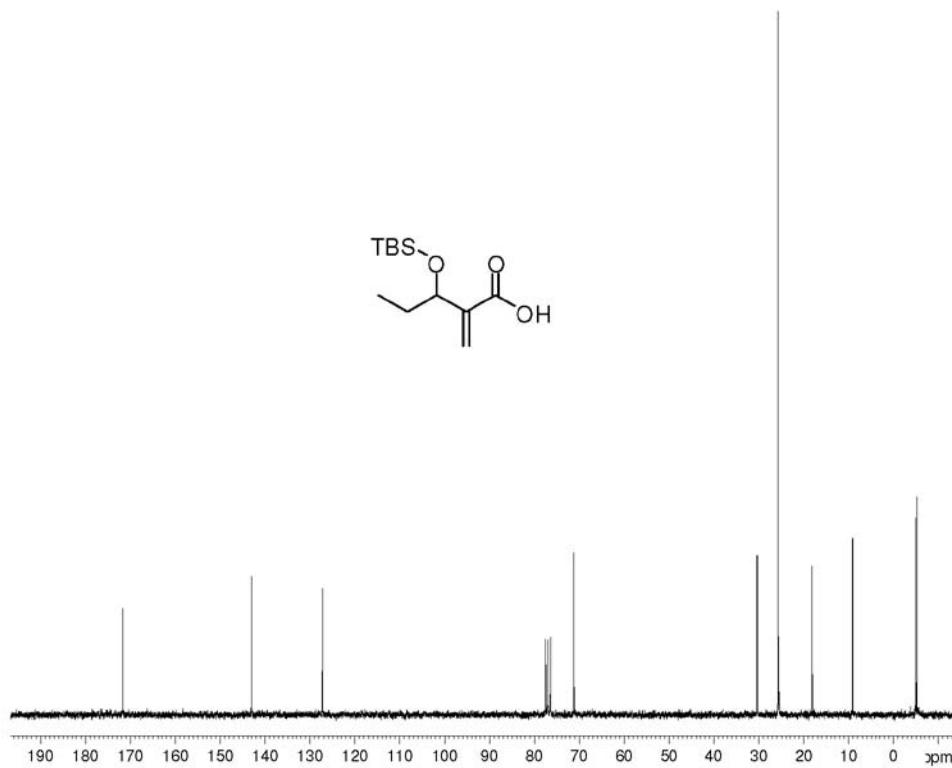


Figure S56. ^{13}C NMR (CDCl_3 , 62.5 MHz) of silylated acid 30.

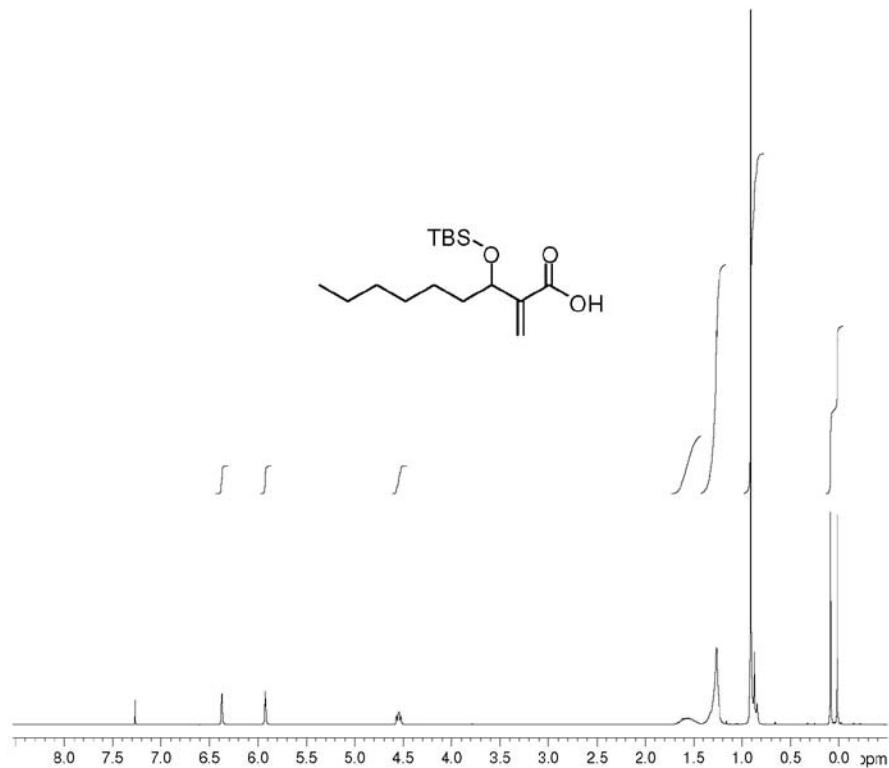


Figure S57. ^1H NMR (CDCl_3 , 250 MHz) of silylated acid 31.

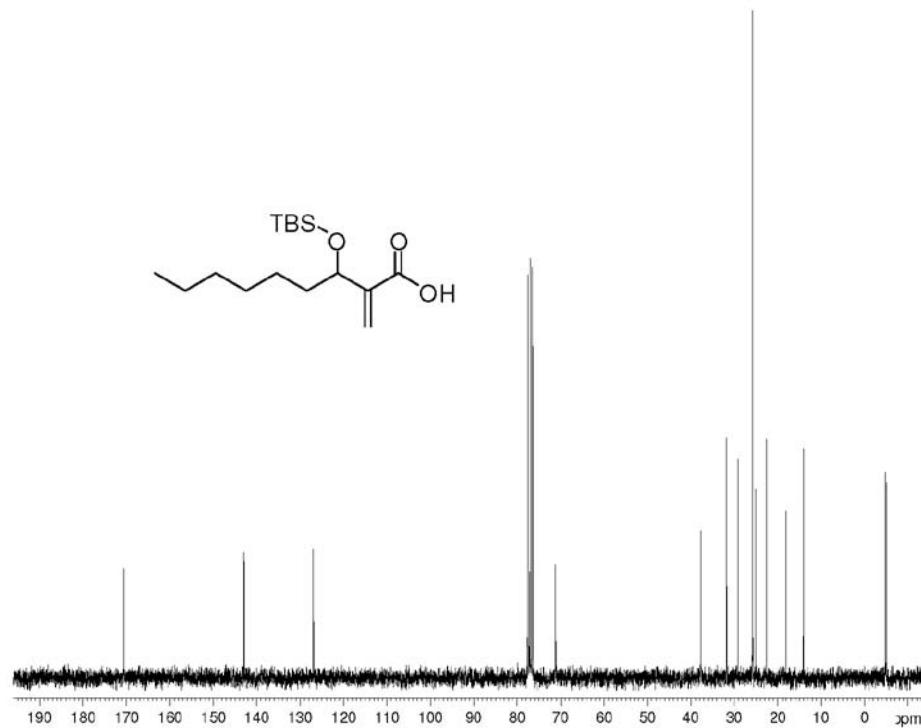


Figure S58. ^{13}C NMR (CDCl_3 , 62.5 MHz) of silylated acid 31.

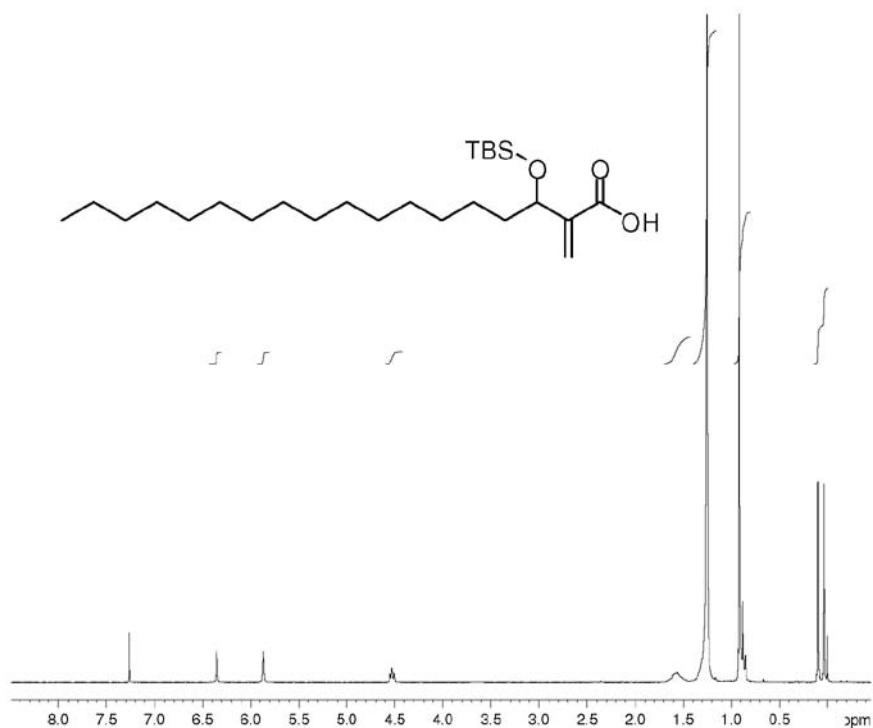


Figure S59. ^1H NMR (CDCl_3 , 250 MHz) of silylated acid 32.

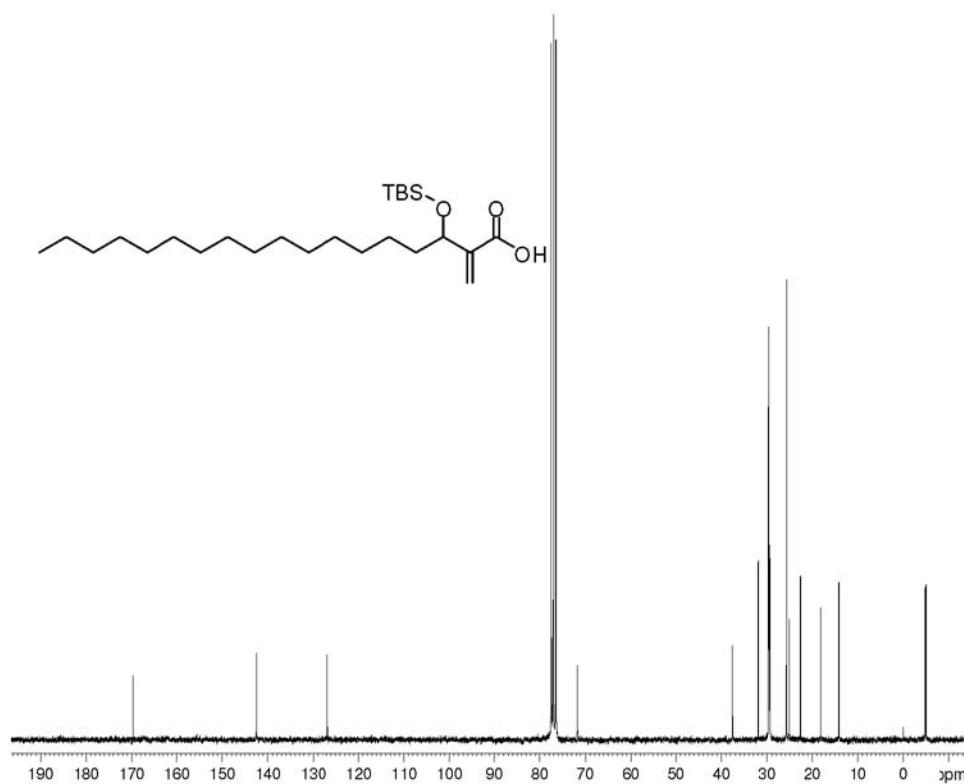


Figure S60. ^{13}C NMR (CDCl_3 , 62.5 MHz) of silylated acid 32.

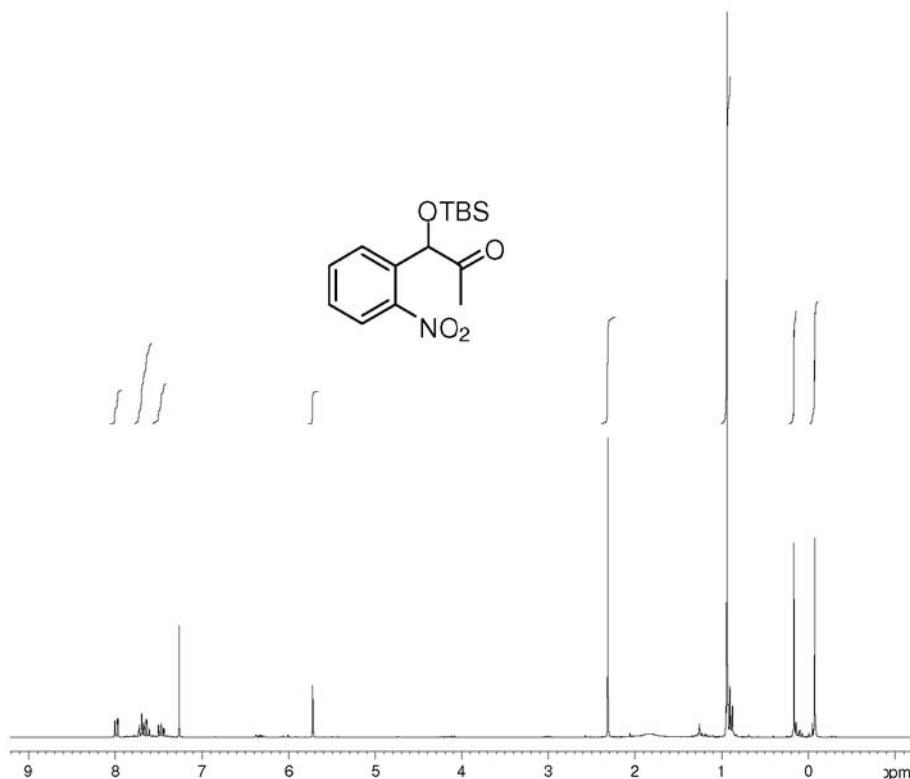
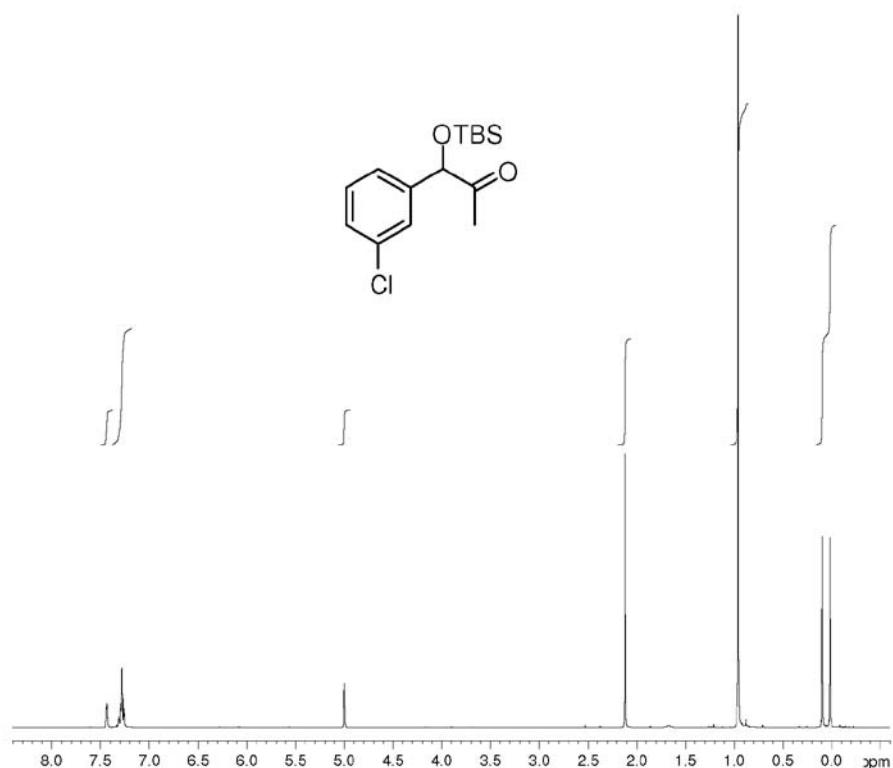
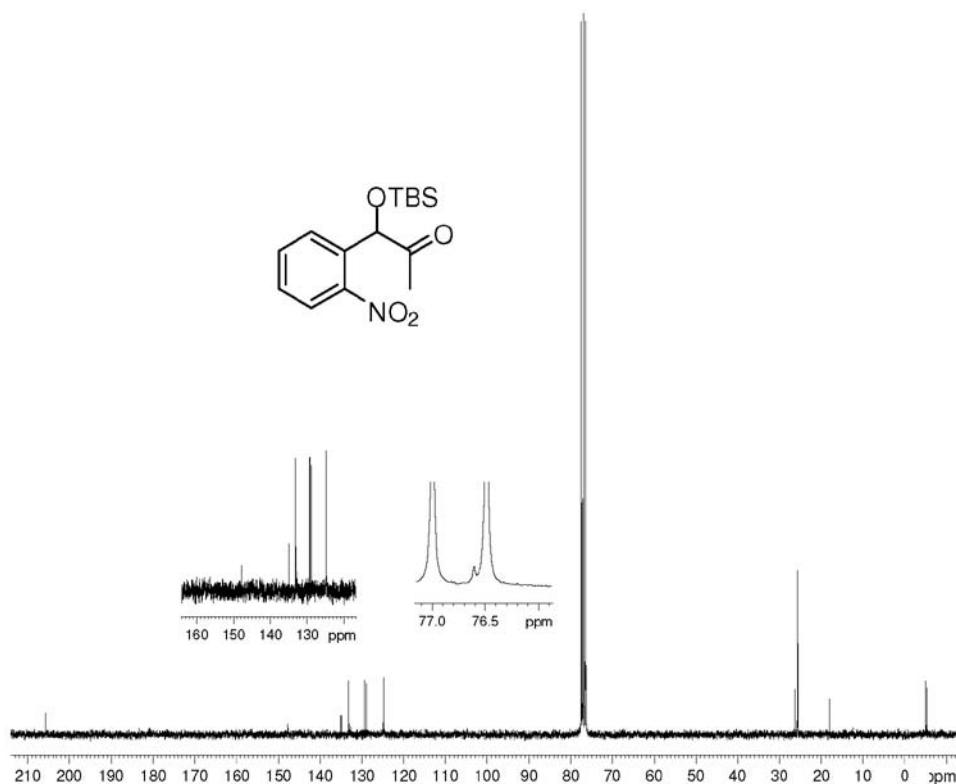


Figure S61. ^1H NMR (CDCl_3 , 250 MHz) of acyloin 33.



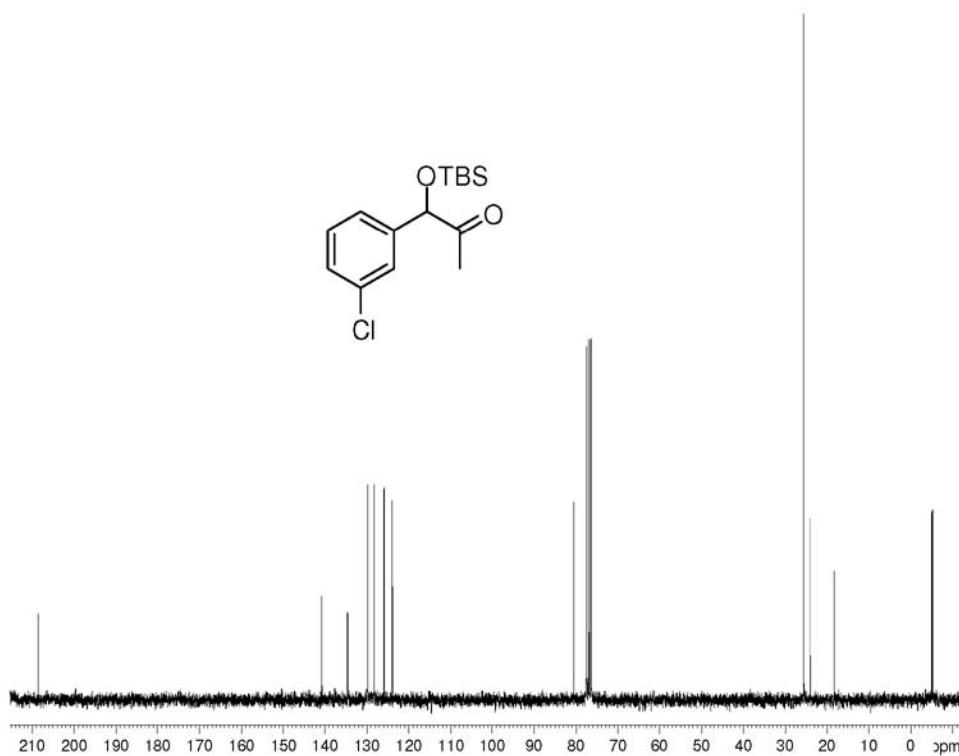


Figure S64. ^{13}C NMR (CDCl_3 , 62.5 MHz) of acyloin 34.

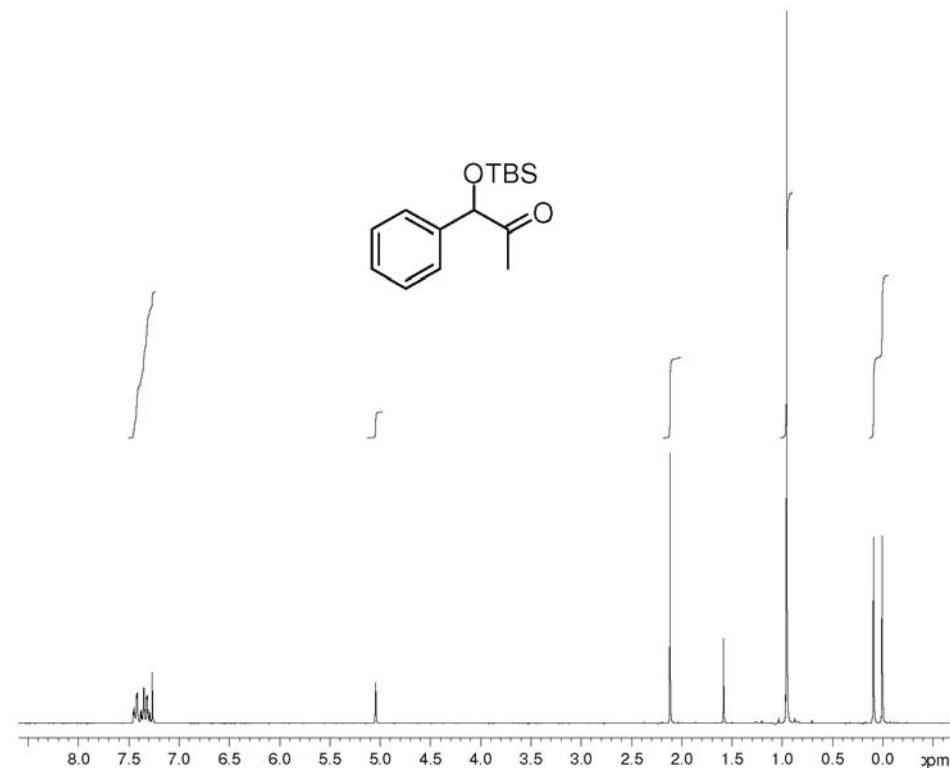


Figure S65. ^1H NMR (CDCl_3 , 250 MHz) of acyloin 35.

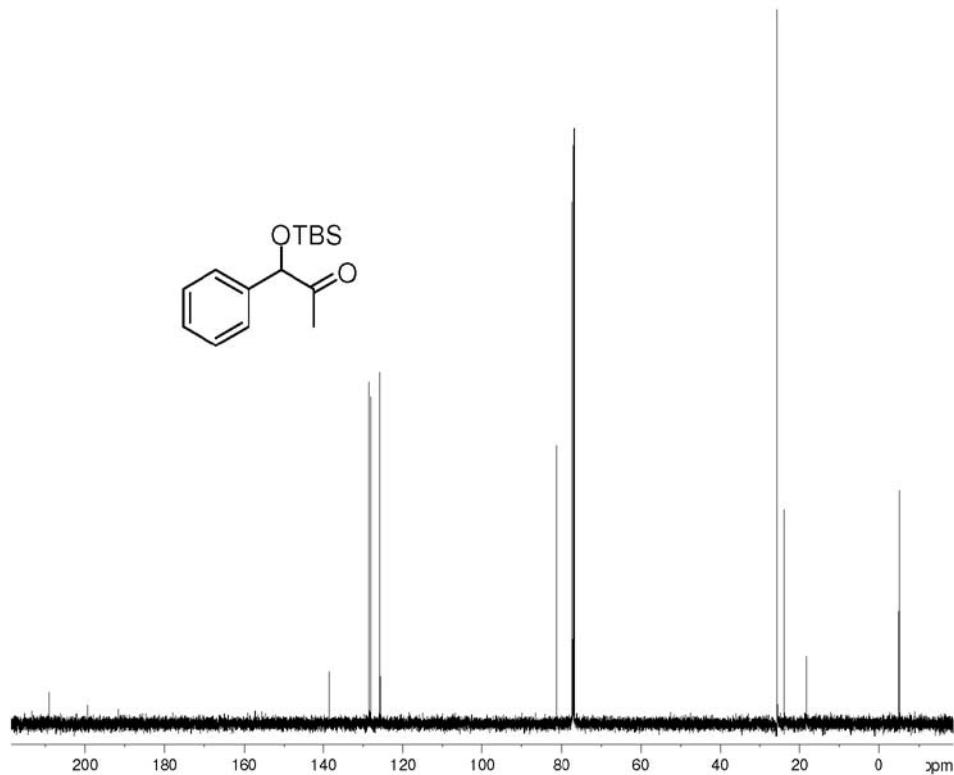


Figure S66. ^{13}C NMR (CDCl_3 , 125 MHz) of acyloin 35.

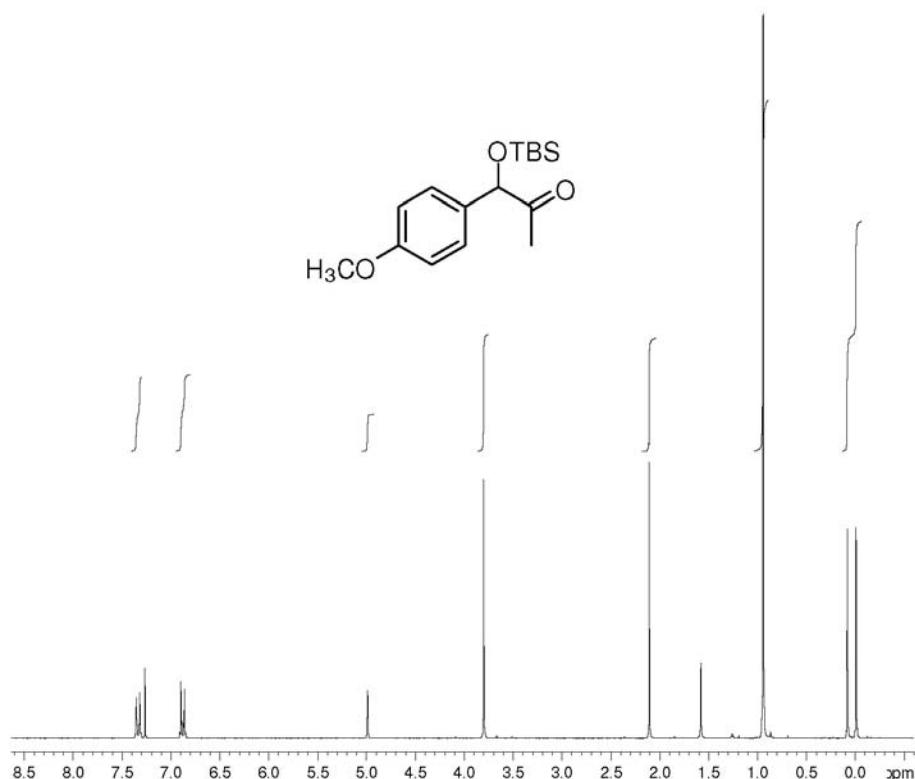


Figure S67. ^1H NMR (CDCl_3 , 250 MHz) of acyloin 36.

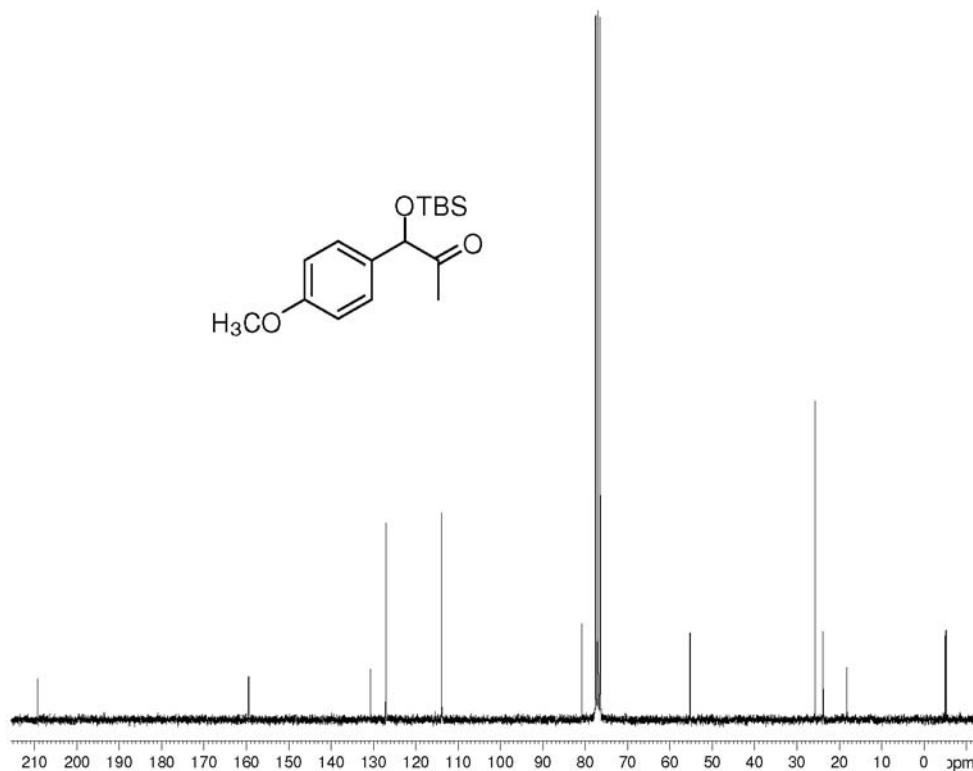


Figure S68. ^{13}C NMR (CDCl_3 , 62.5 MHz) of acyloin **36**.

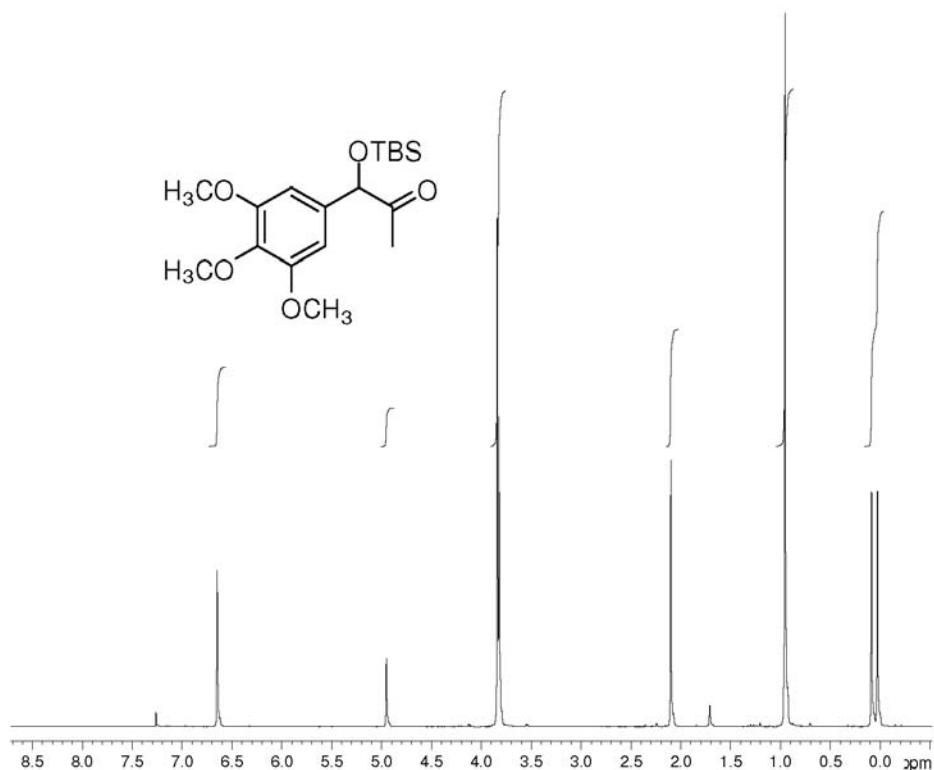


Figure S69. ^1H NMR (CDCl_3 , 250 MHz) of acyloin **37**.

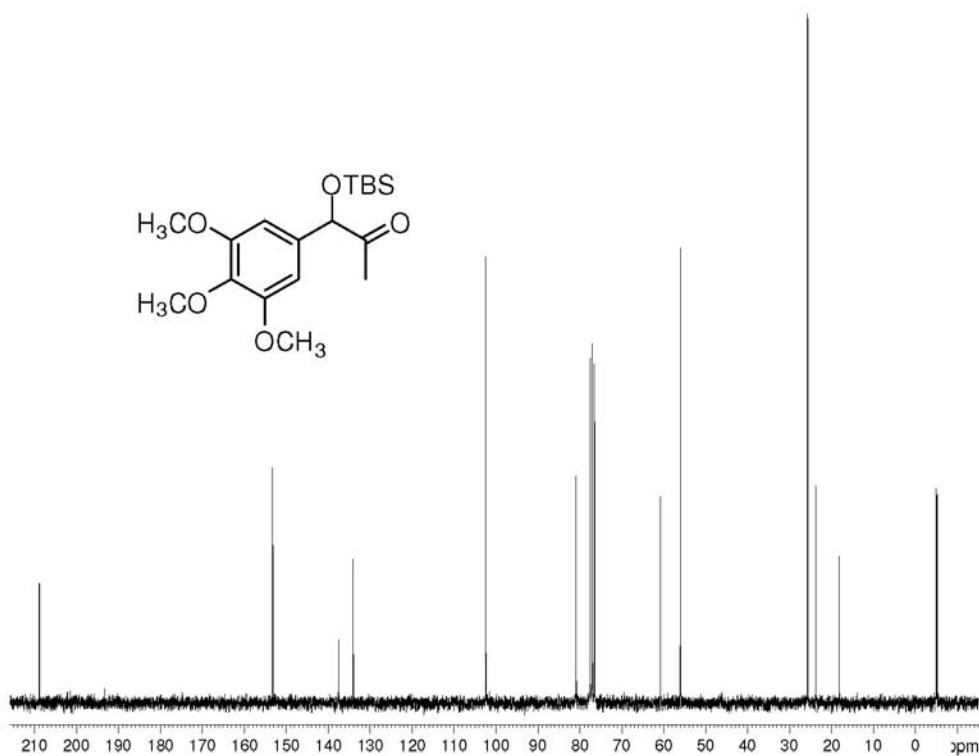


Figure S70. ^{13}C NMR (CDCl_3 , 62.5 MHz) of acyloin 37.

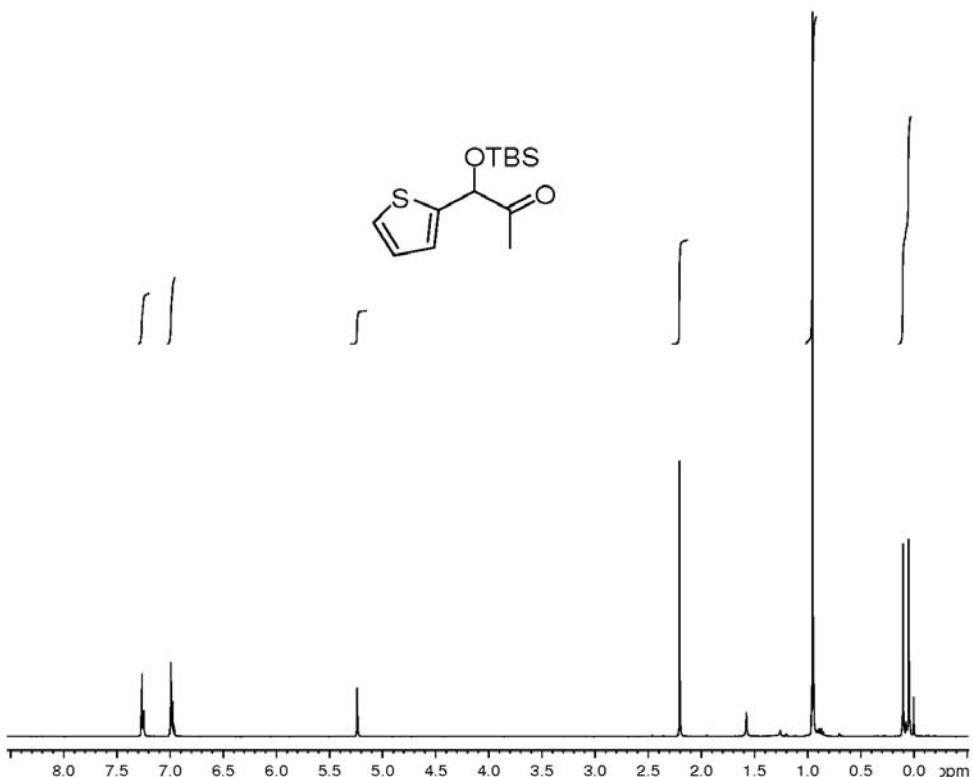


Figure S71. ^1H NMR (CDCl_3 , 250 MHz) of acyloin 38.

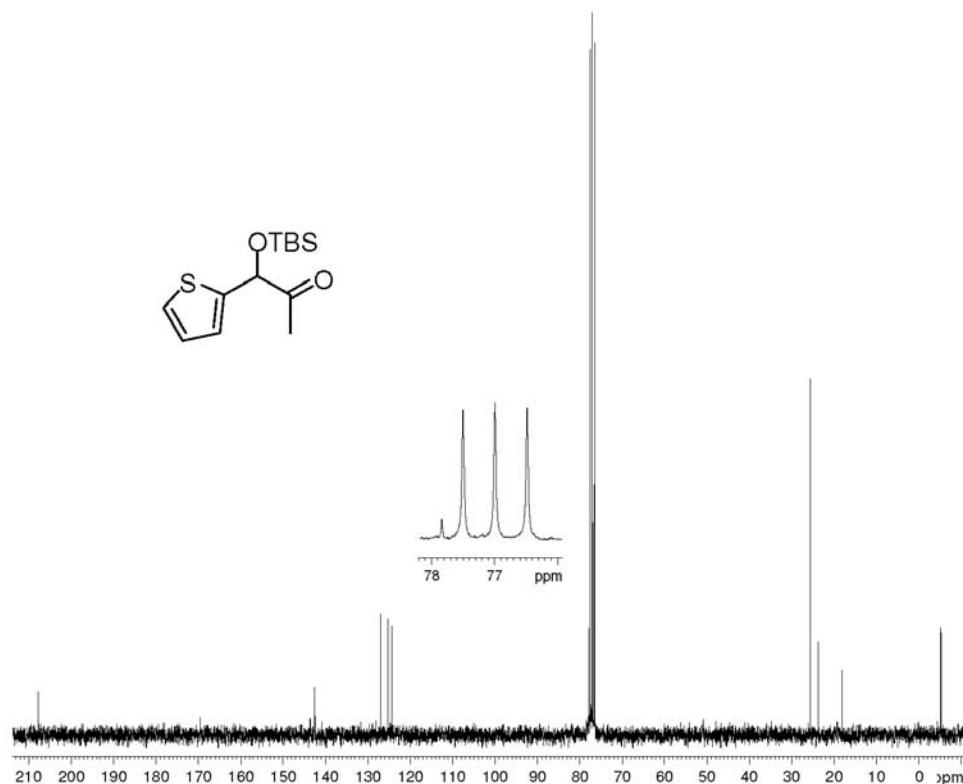


Figure S72. ^{13}C NMR (CDCl_3 , 62.5 MHz) of acyloin **38**.

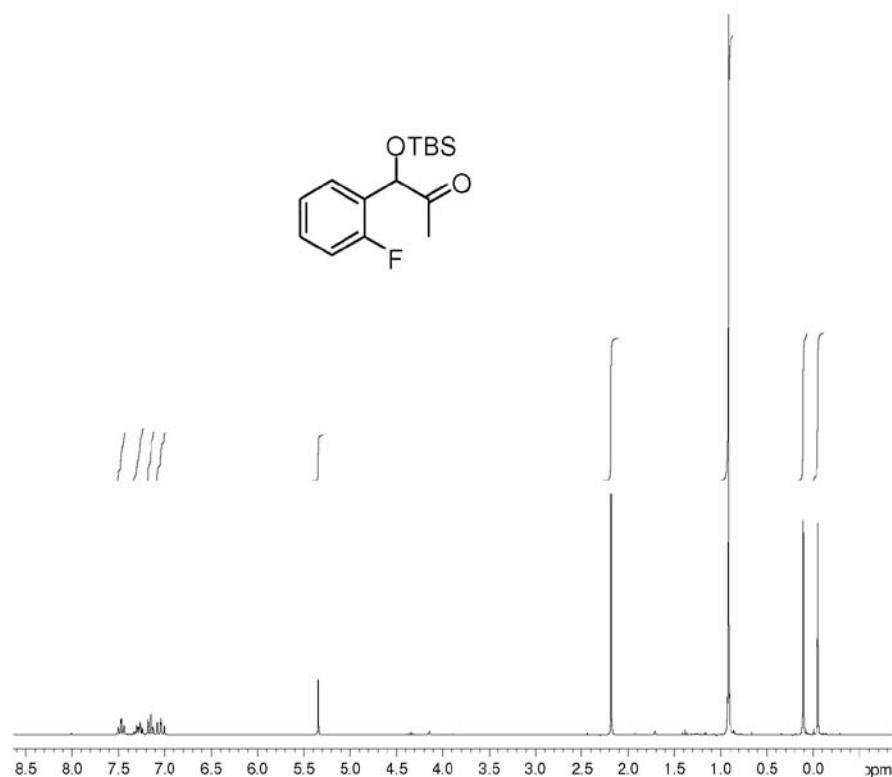


Figure S73. ^1H NMR (CDCl_3 , 250 MHz) of acyloin **39**.

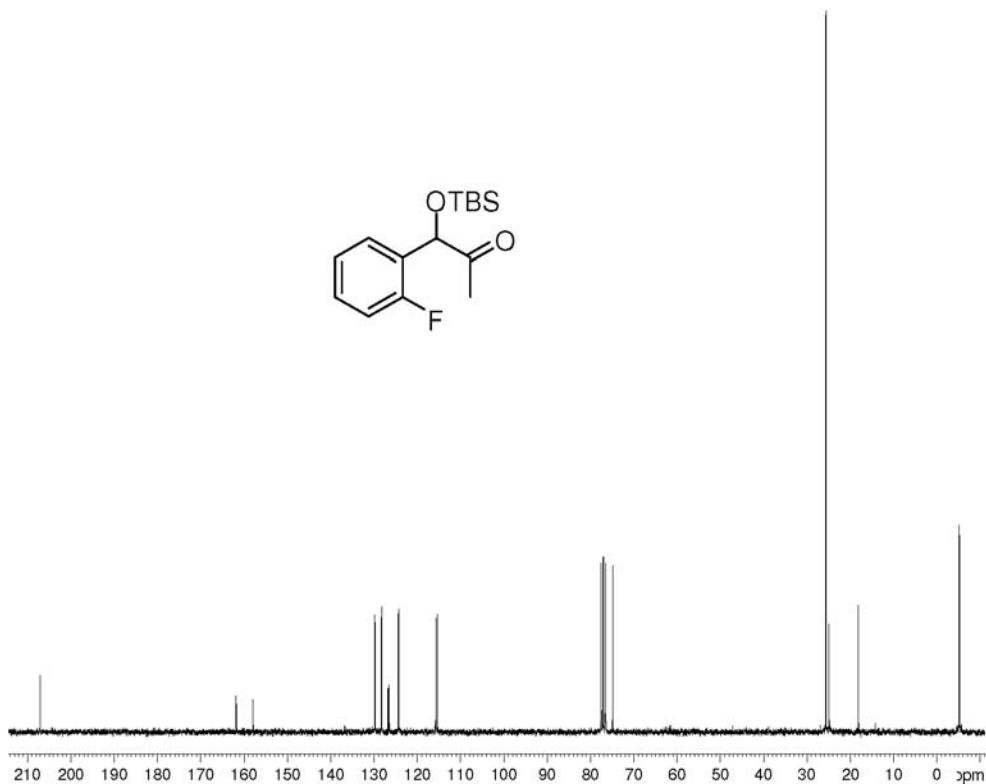


Figure S74. ^{13}C NMR (CDCl_3 , 62.5 MHz) of acyloin 39.

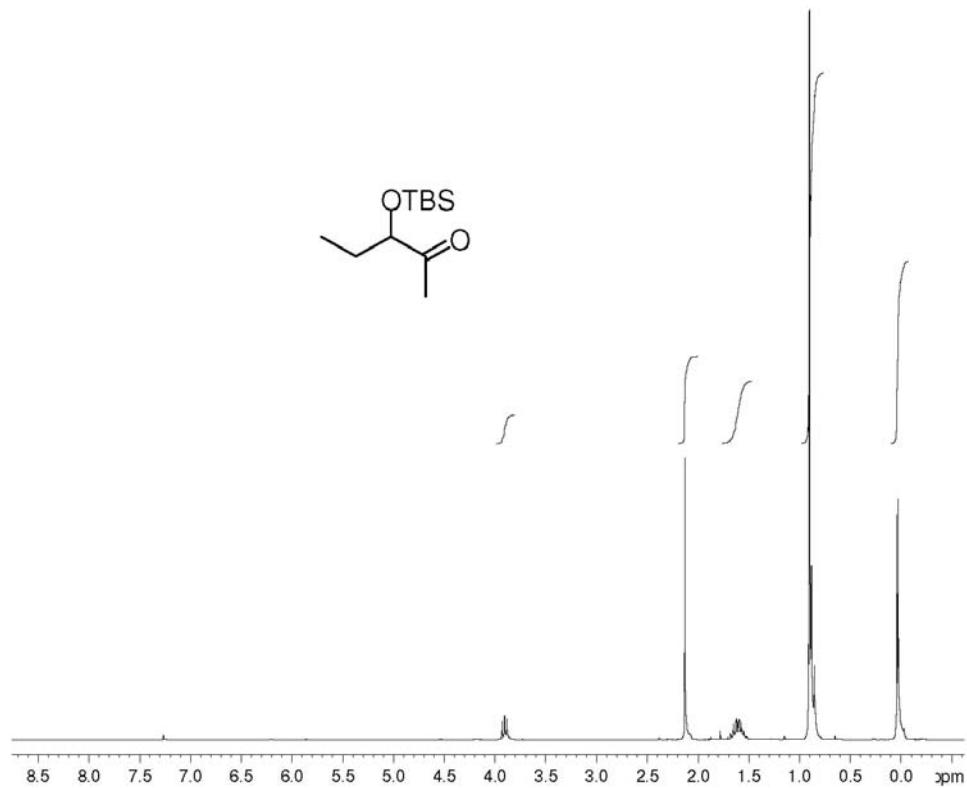


Figure S75. ^1H NMR (CDCl_3 , 250 MHz) of acyloin 40.

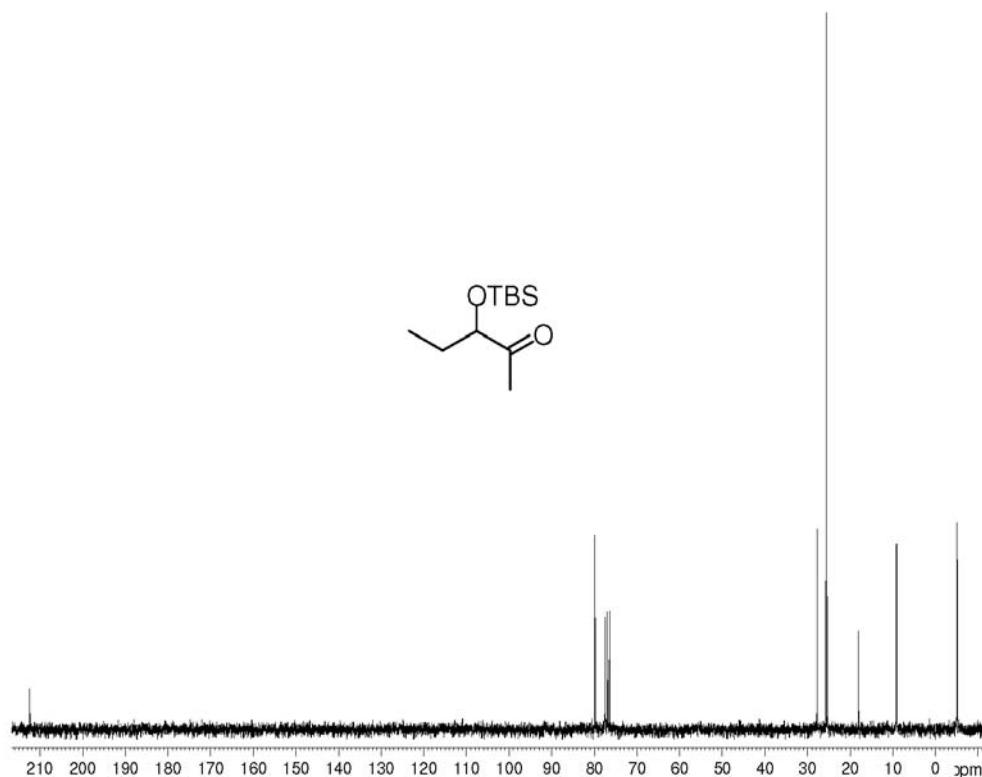


Figure S76. ^{13}C NMR (CDCl_3 , 62.5 MHz) of acyloin **40**.

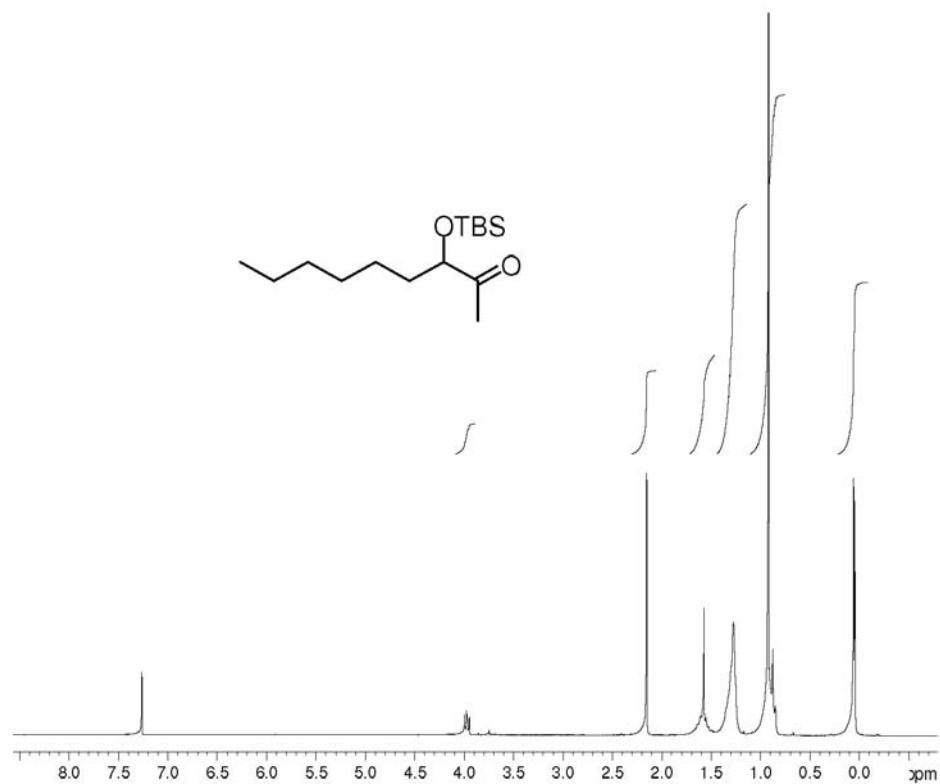


Figure S77. ^1H NMR (CDCl_3 , 250 MHz) of acyloin **41**.

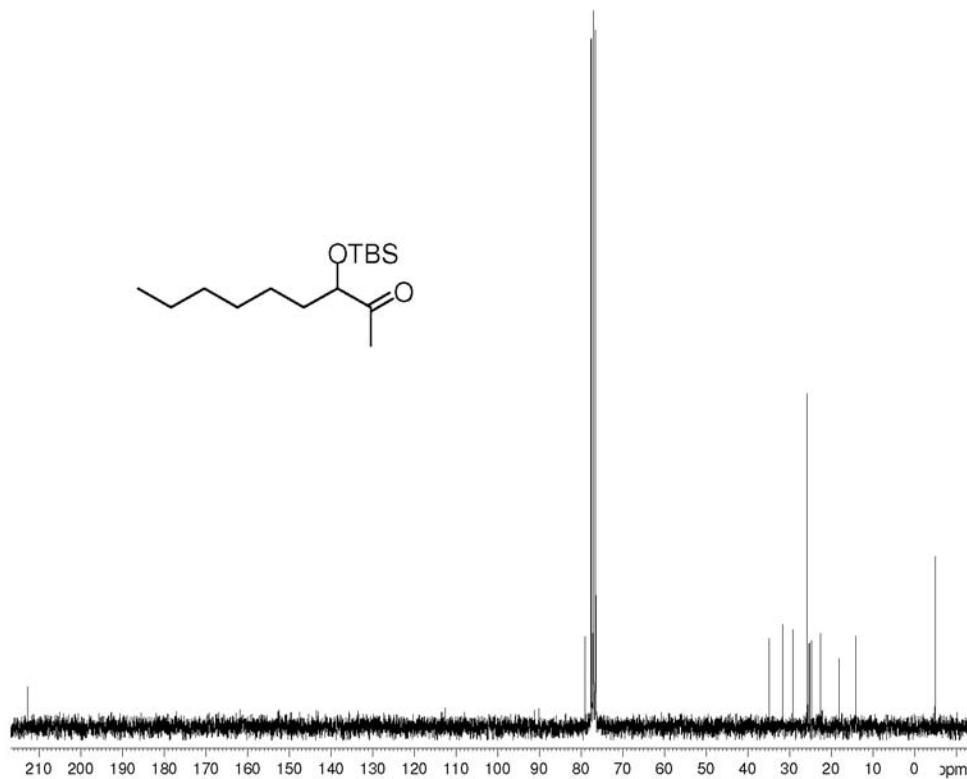


Figure S78. ^{13}C NMR (CDCl_3 , 62.5 MHz) of acyloin 41.

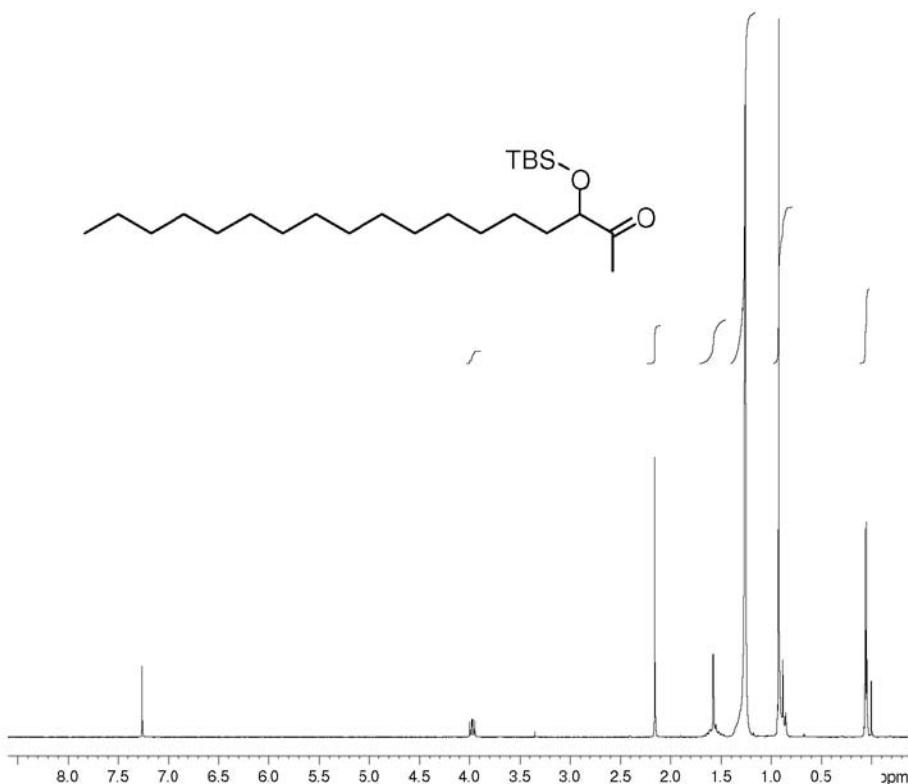


Figure S79. ^1H NMR (CDCl_3 , 250 MHz) of acyloin 42.

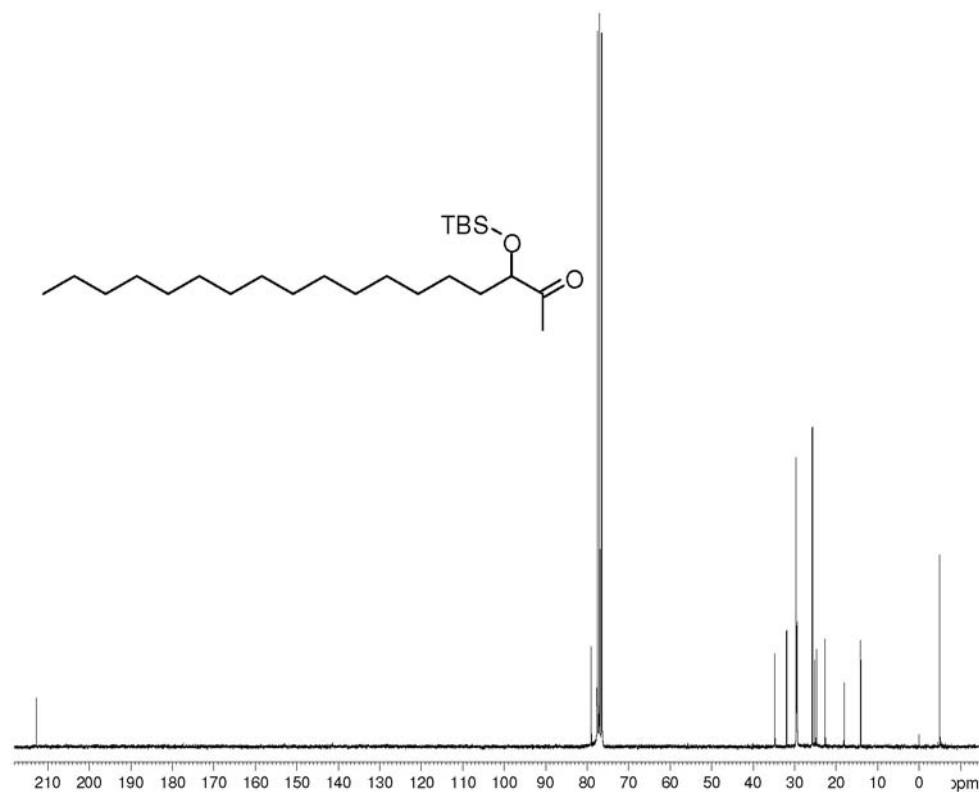


Figure S80. ^{13}C NMR (CDCl_3 , 62.5 MHz) of acyloin 41.

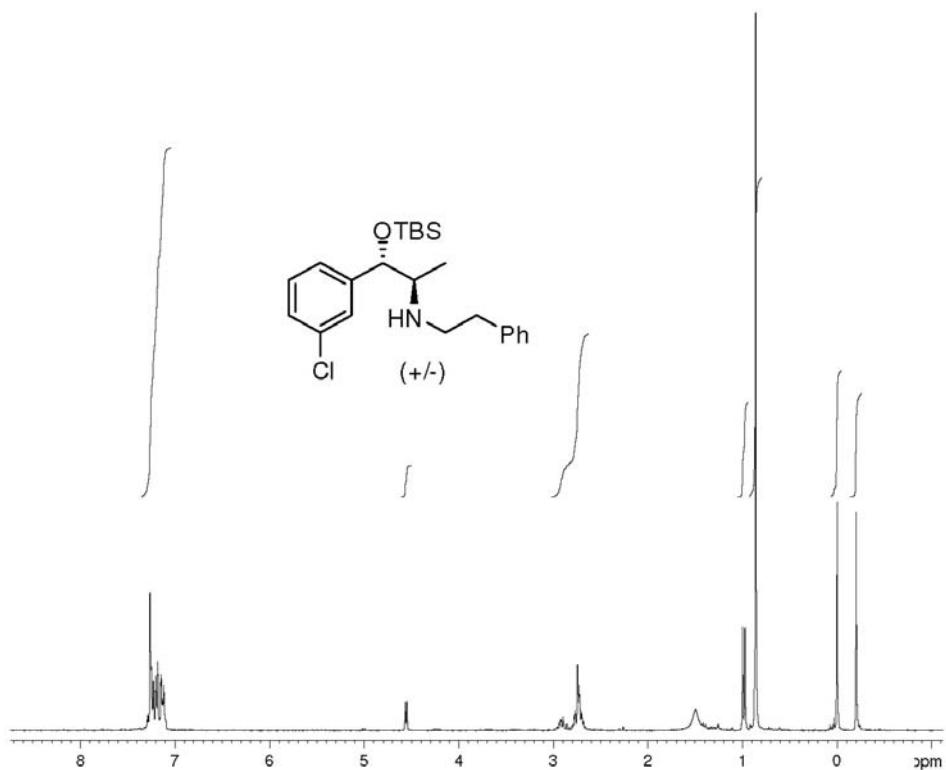


Figure S81. ^1H NMR (CDCl_3 , 250 MHz) of vicinal aminoalcohol 43.

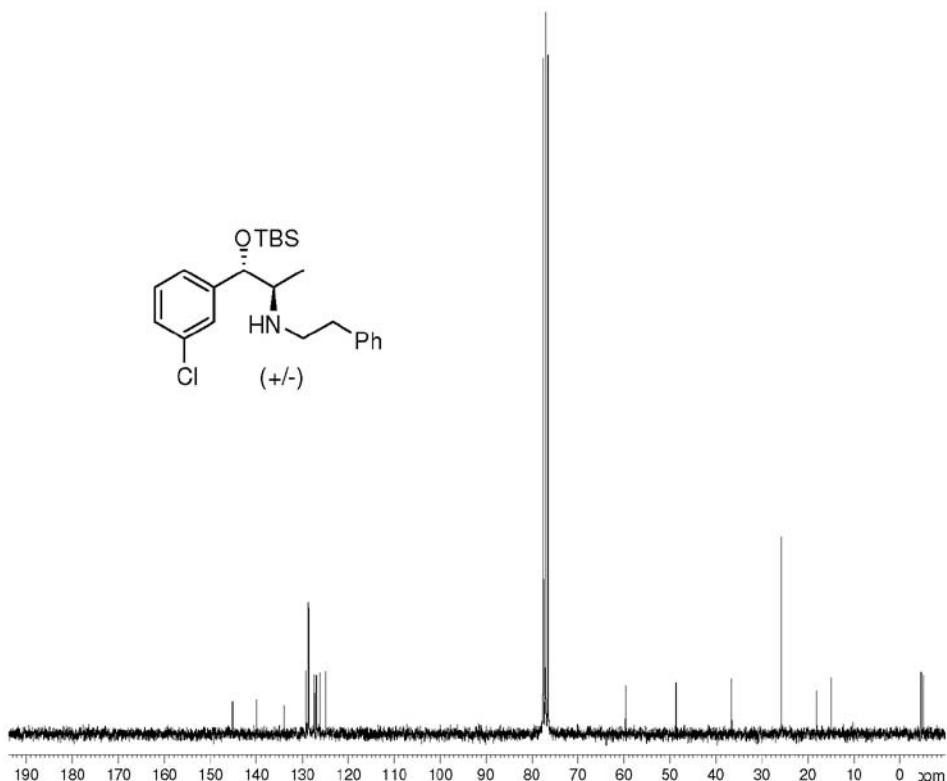


Figure S82. ^{13}C NMR (CDCl_3 , 62.5 MHz) of vicinal aminoalcohol **43**.

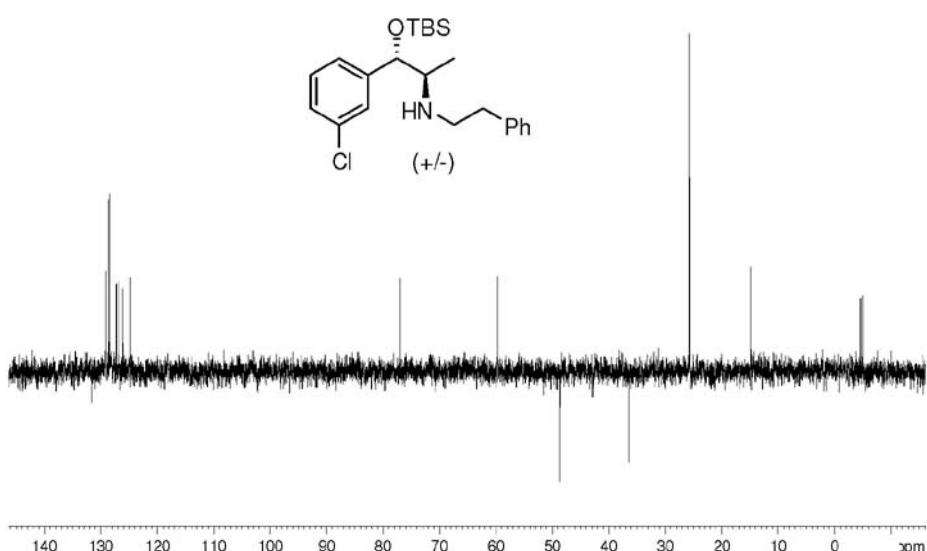


Figure S83. DEPT 135 (CDCl_3 , 62.5 MHz) of vicinal aminoalcohol **43**.

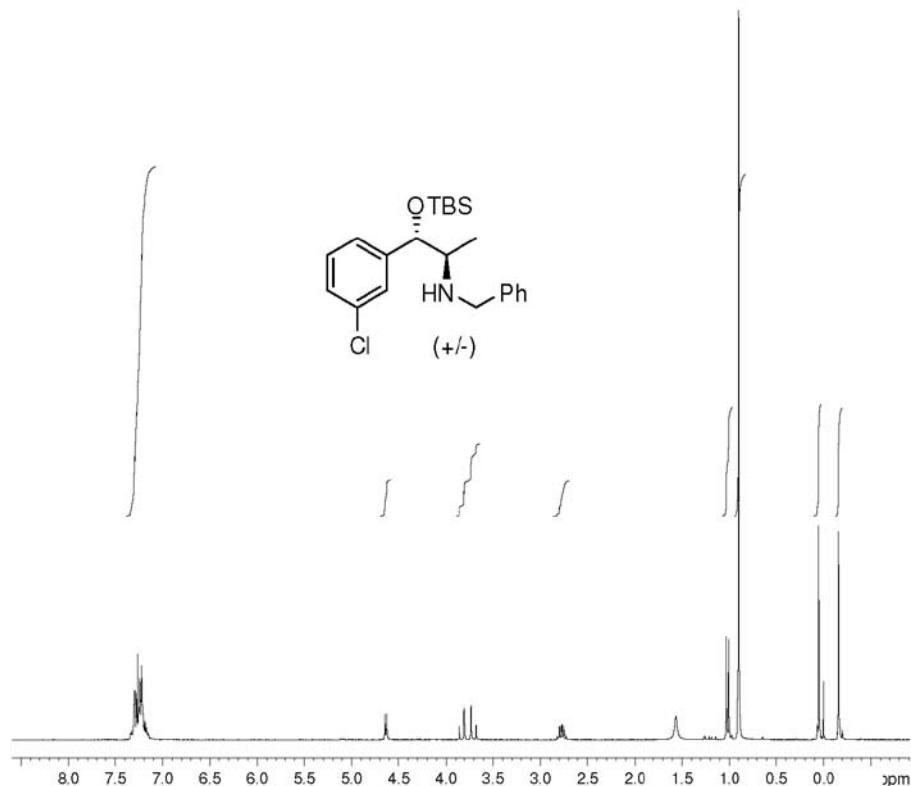


Figure S84. ¹H NMR (CDCl_3 , 250 MHz) of vicinal aminoalcohol **44**.

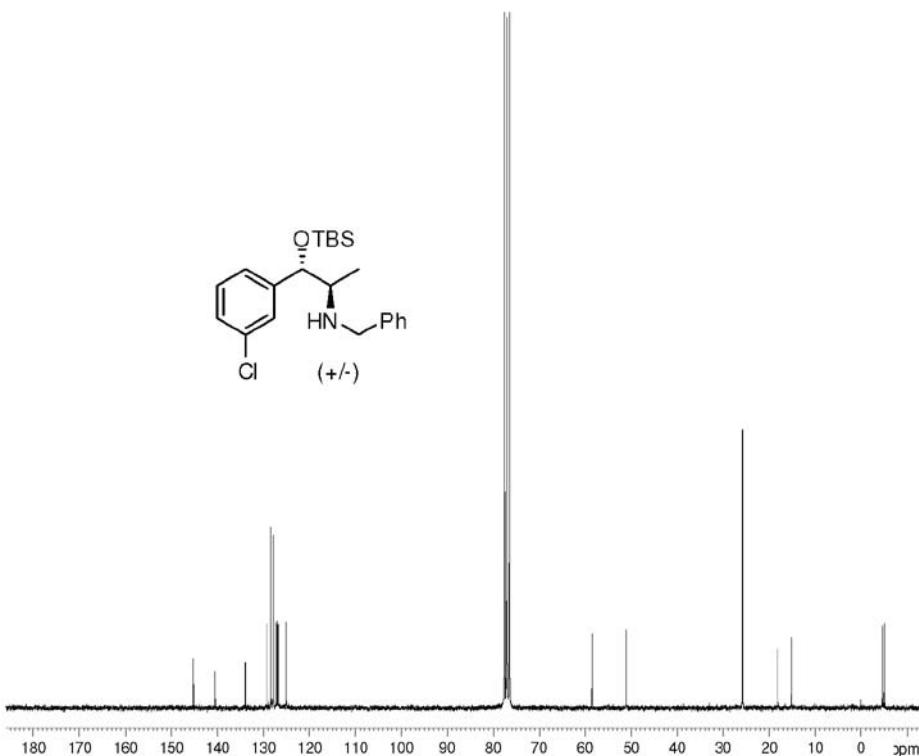


Figure S85. ¹³C NMR (CDCl_3 , 62.5 MHz) of vicinal aminoalcohol **44**.

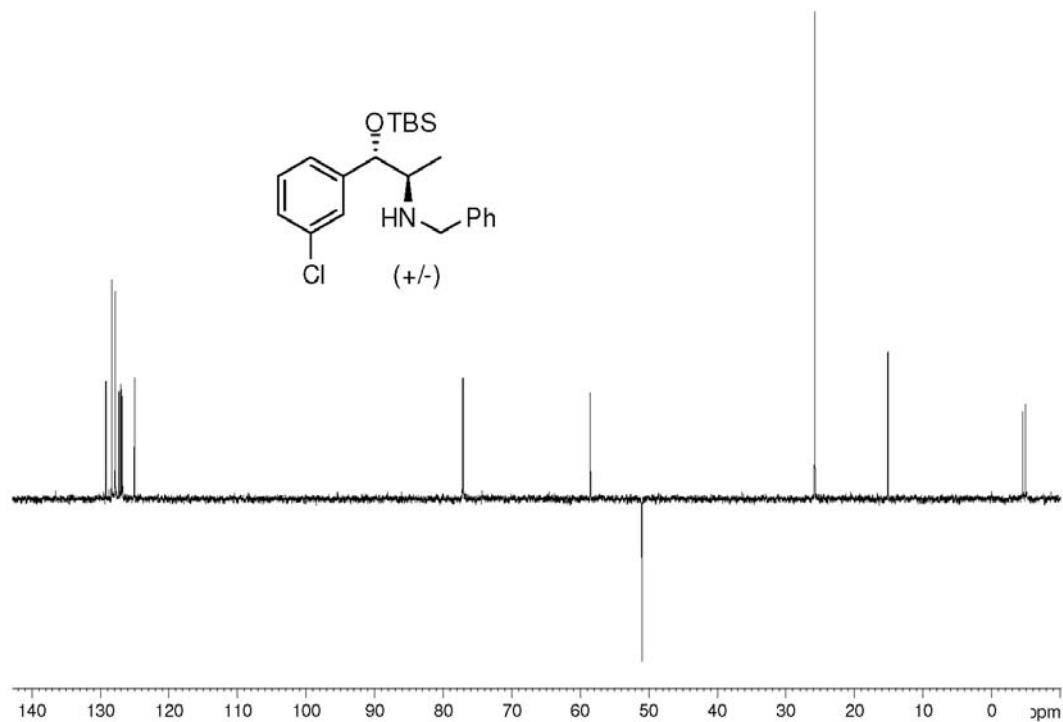


Figure S86. DEPT 135 (CDCl_3 , 62.5 MHz) of vicinal aminoalcohol **44**.

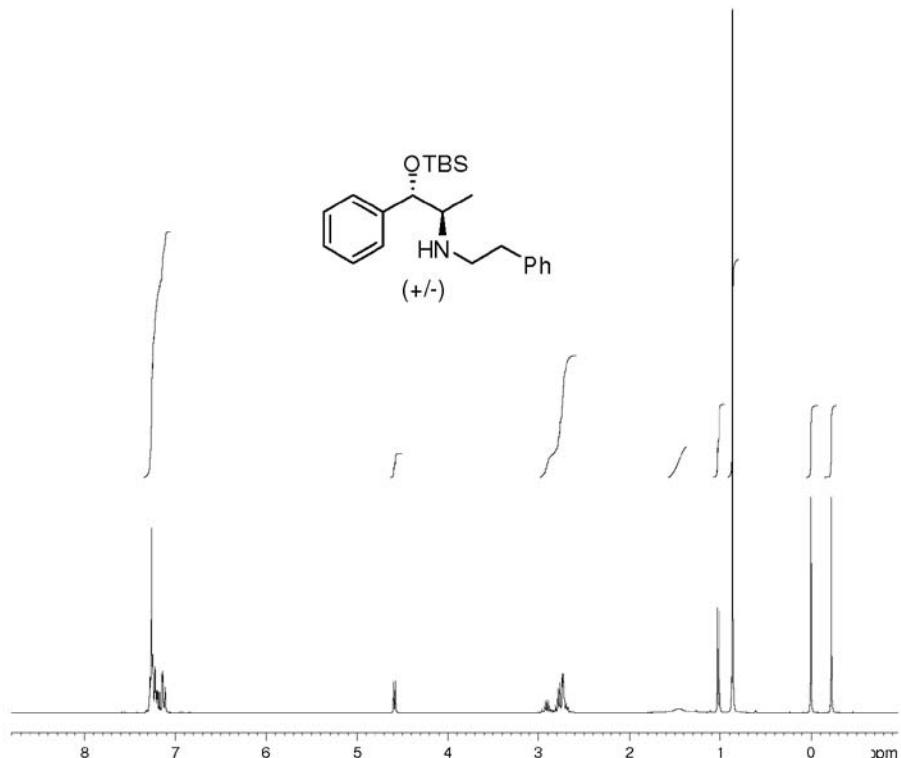


Figure S87. ¹H NMR (CDCl_3 , 250 MHz) of vicinal aminoalcohol **45**.

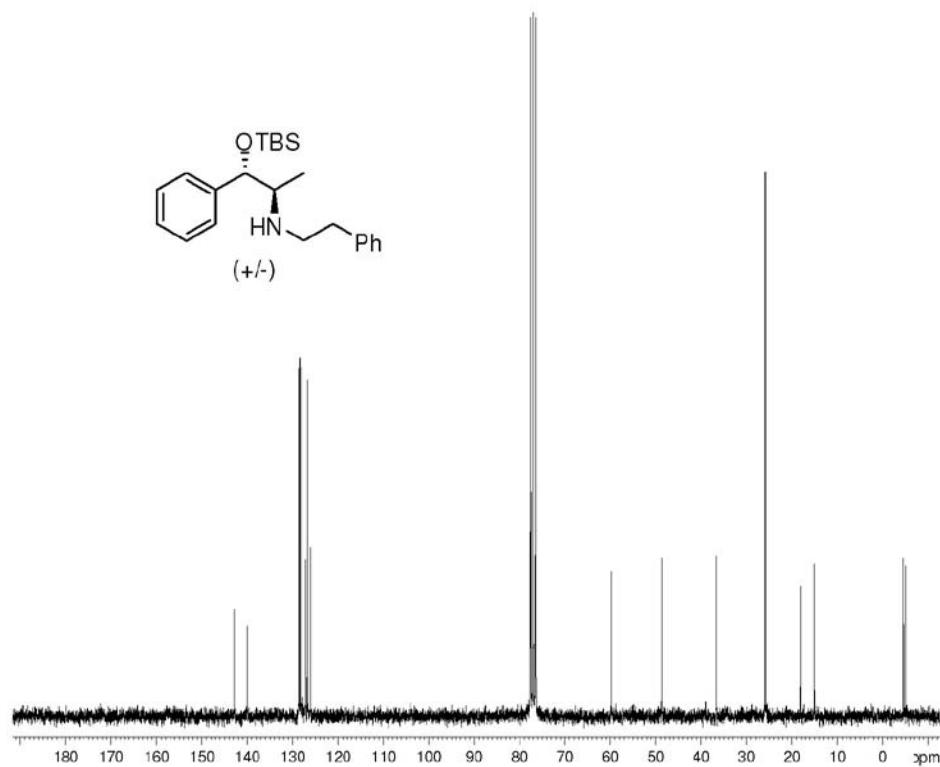


Figure S88. ^{13}C NMR (CDCl_3 , 62.5 MHz) of vicinal aminoalcohol **45**.

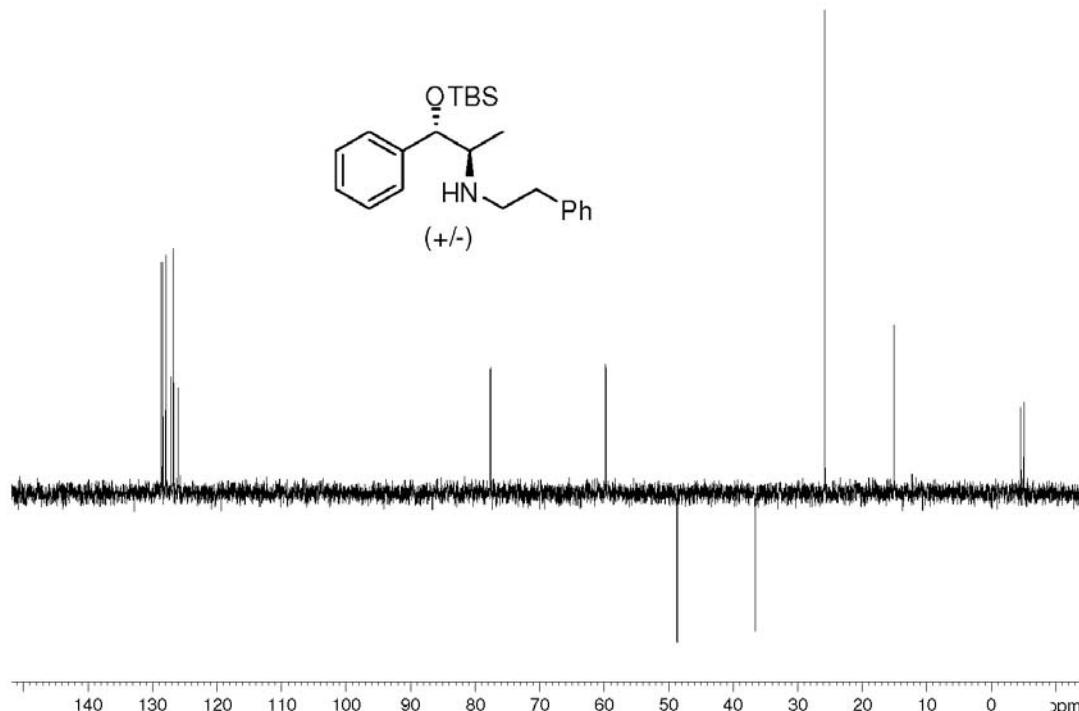


Figure S89. DEPT 135 (CDCl_3 , 62.5 MHz) of vicinal aminoalcohol **45**.

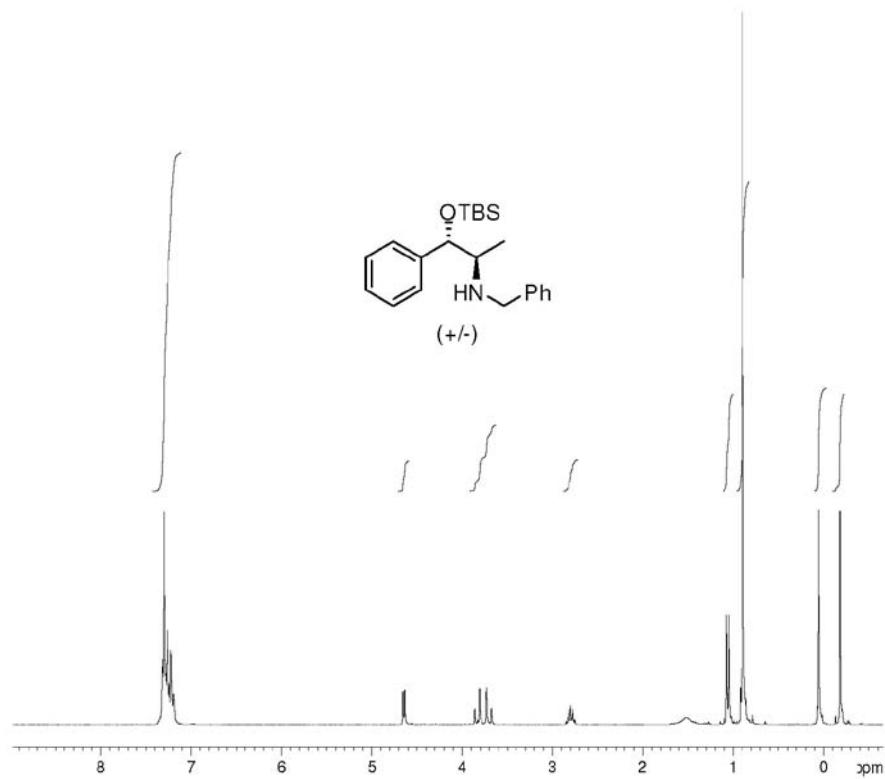


Figure S90. ¹H NMR (CDCl₃, 250 MHz) of vicinal aminoalcohol 46.

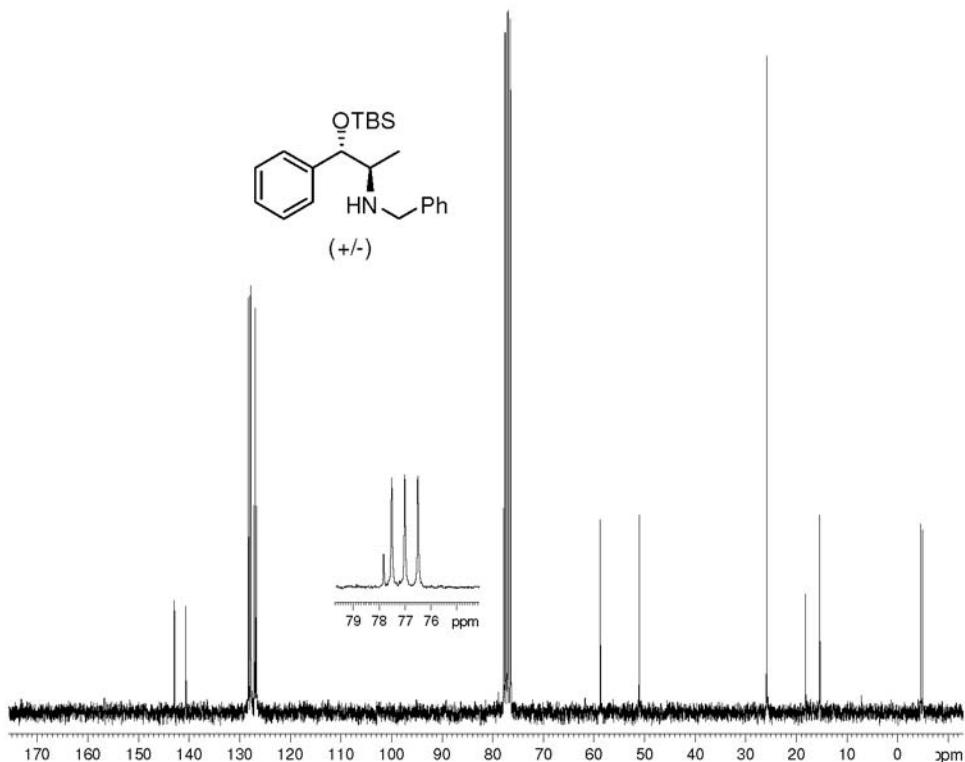


Figure S91. ¹³C NMR (CDCl₃, 62.5 MHz) of vicinal aminoalcohol 46.

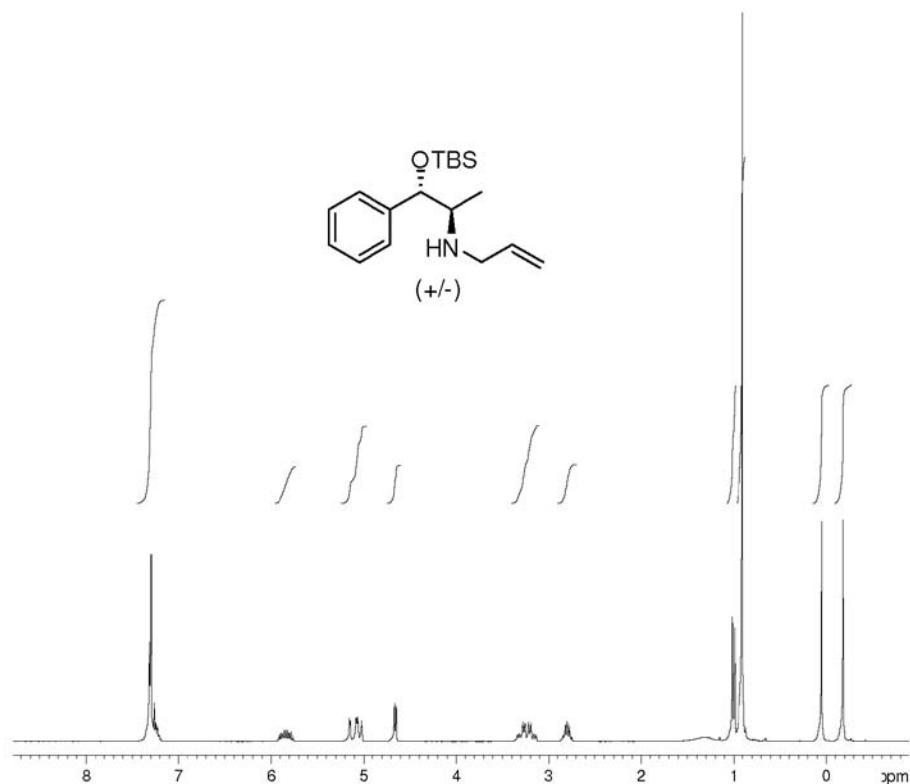


Figure S92. ¹H NMR (CDCl_3 , 250 MHz) of vicinal aminoalcohol **47**.

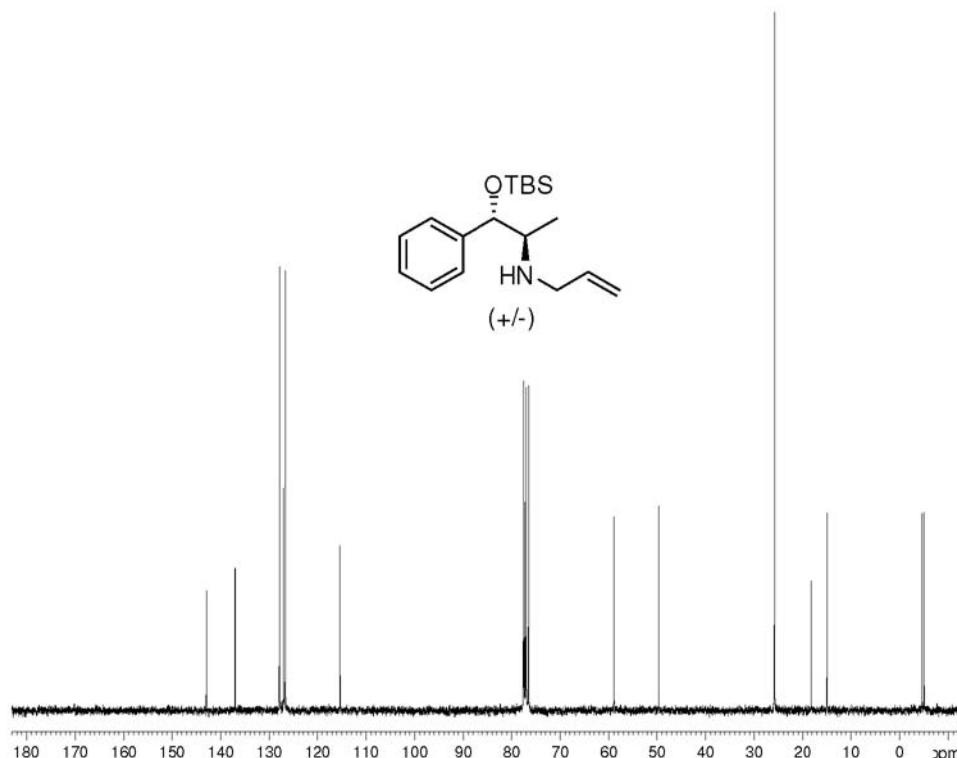


Figure S93. ¹³C NMR (CDCl_3 , 62.5 MHz) of vicinal aminoalcohol **47**.

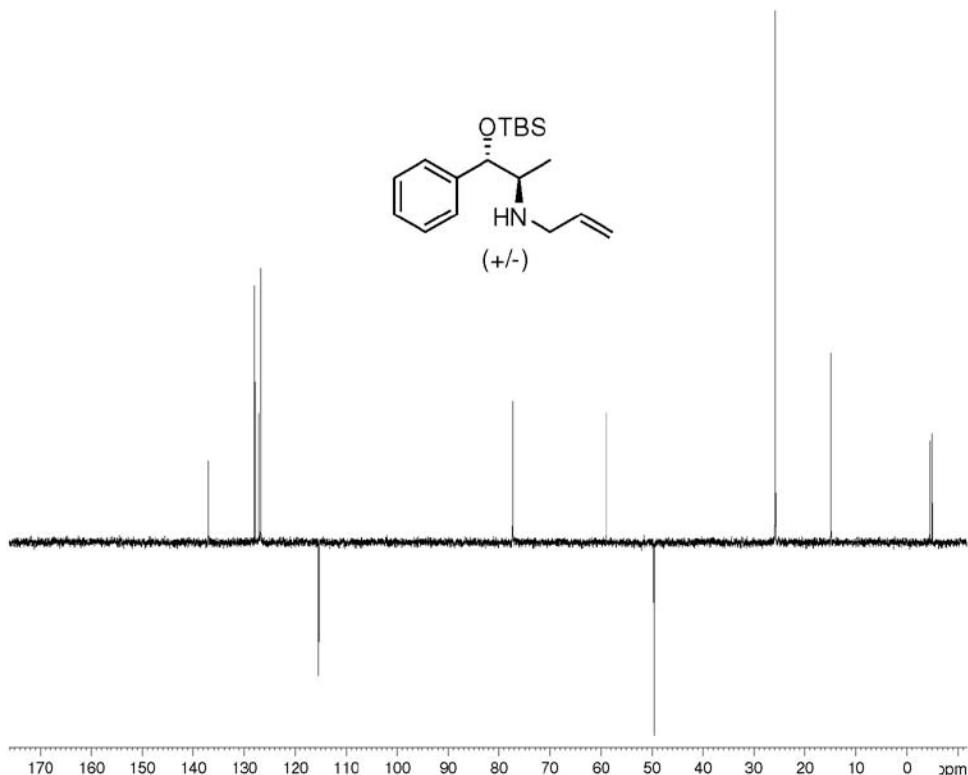


Figure S94. DEPT 135 (CDCl_3 , 62.5 MHz) of vicinal aminoalcohol **47**.

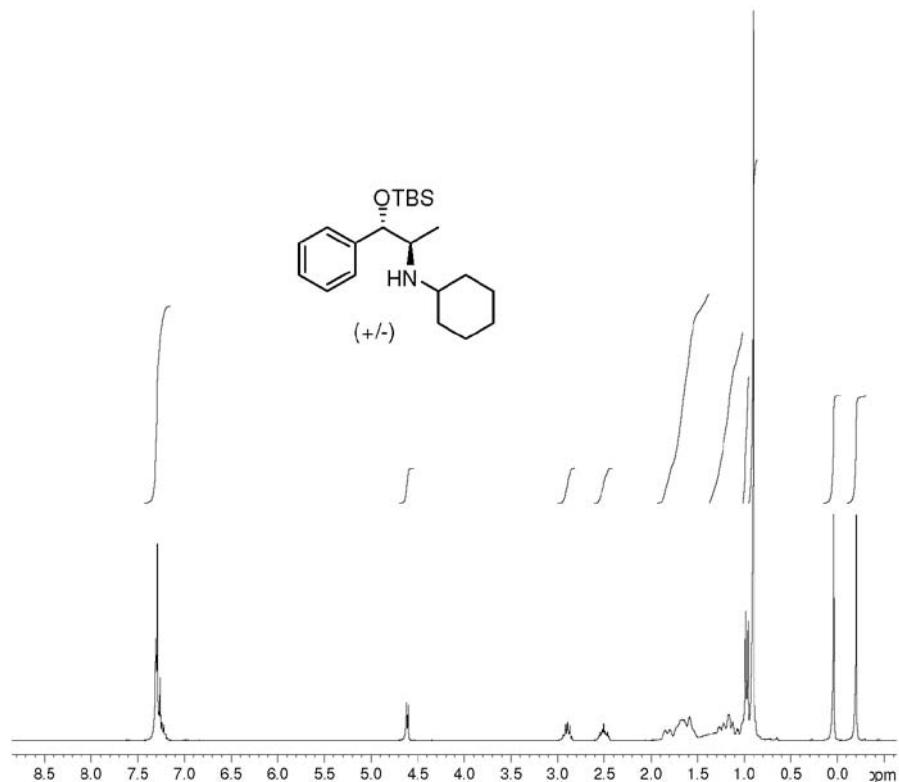


Figure S95. ¹H NMR (CDCl_3 , 250 MHz) of vicinal aminoalcohol **48**.

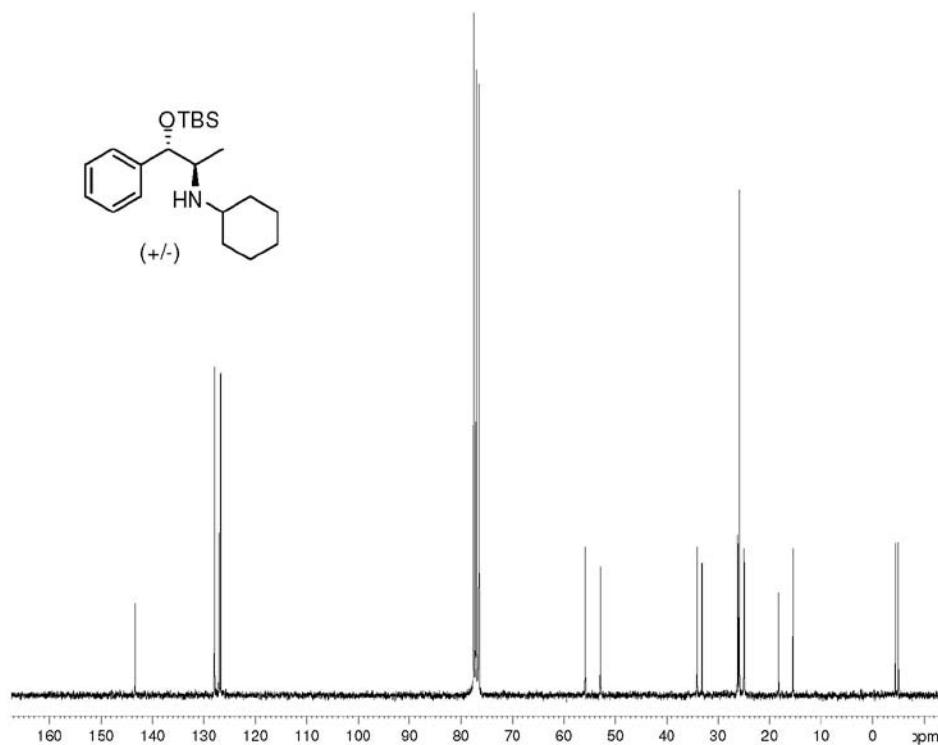


Figure S96. ^{13}C NMR (CDCl_3 , 62.5 MHz) of vicinal aminoalcohol **48**.

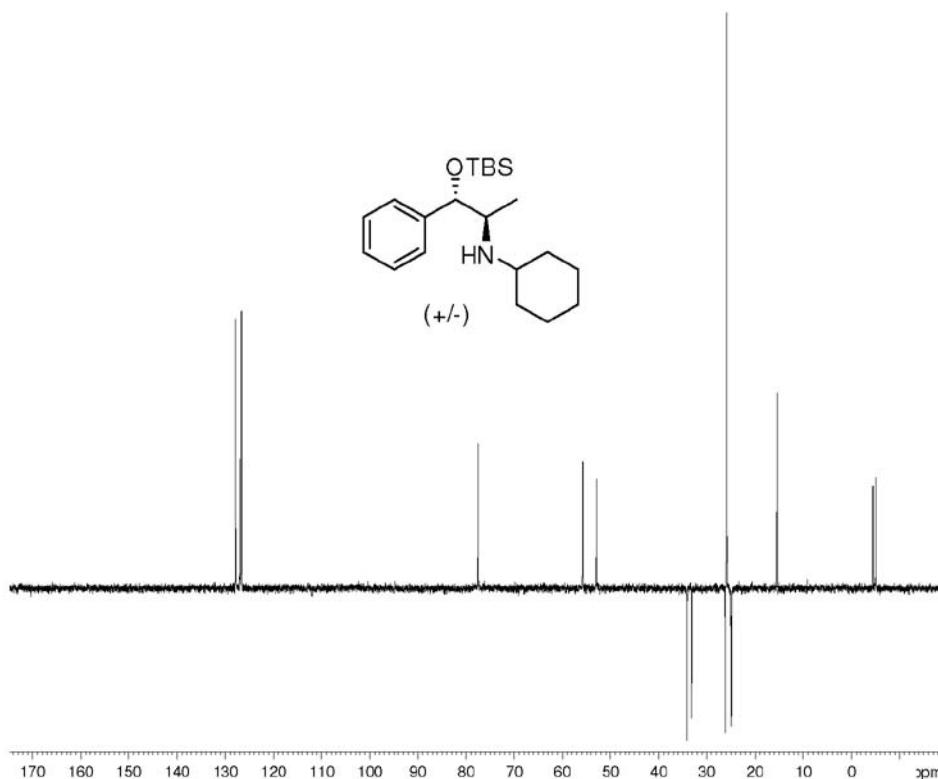


Figure S97. DEPT 135 (CDCl_3 , 62.5 MHz) of vicinal aminoalcohol **48**.

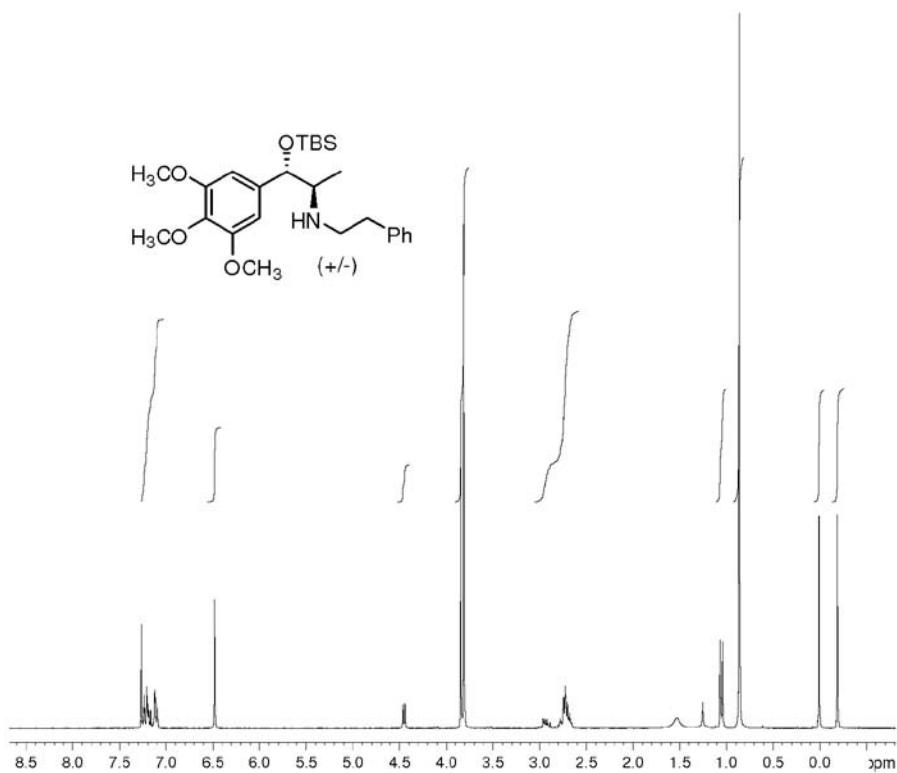


Figure S98. ^1H NMR (CDCl_3 , 250 MHz) of vicinal aminoalcohol **49**.

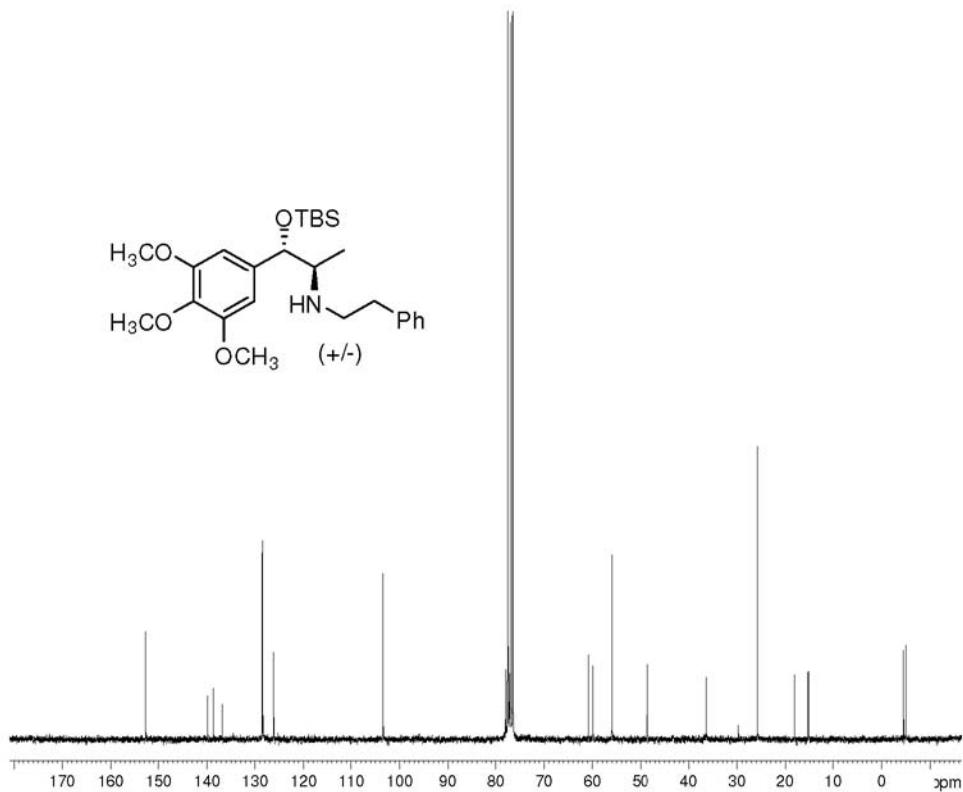


Figure S99. ^{13}C NMR (CDCl_3 , 62.5 MHz) of vicinal aminoalcohol **49**.

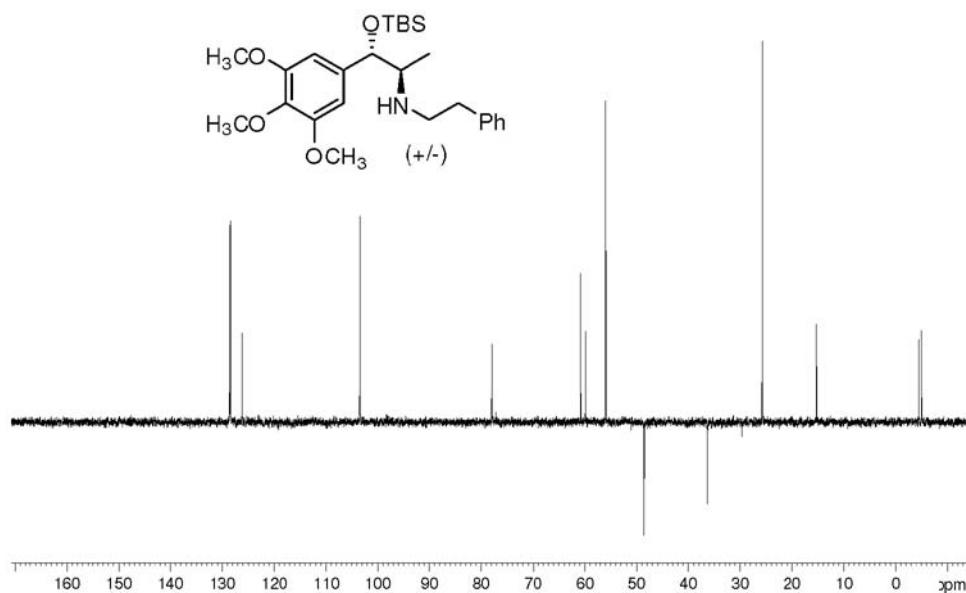


Figure S100. DEPT 135 (CDCl_3 , 62.5 MHz) of vicinal aminoalcohol **49**.

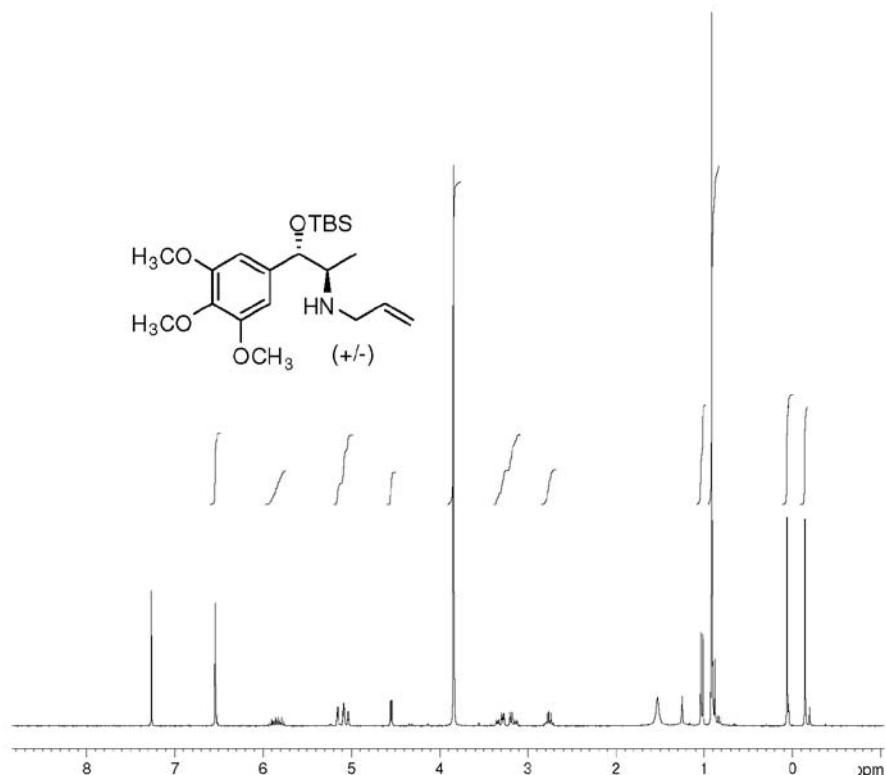


Figure S101. ^1H NMR (CDCl_3 , 250 MHz) of vicinal aminoalcohol **50**.

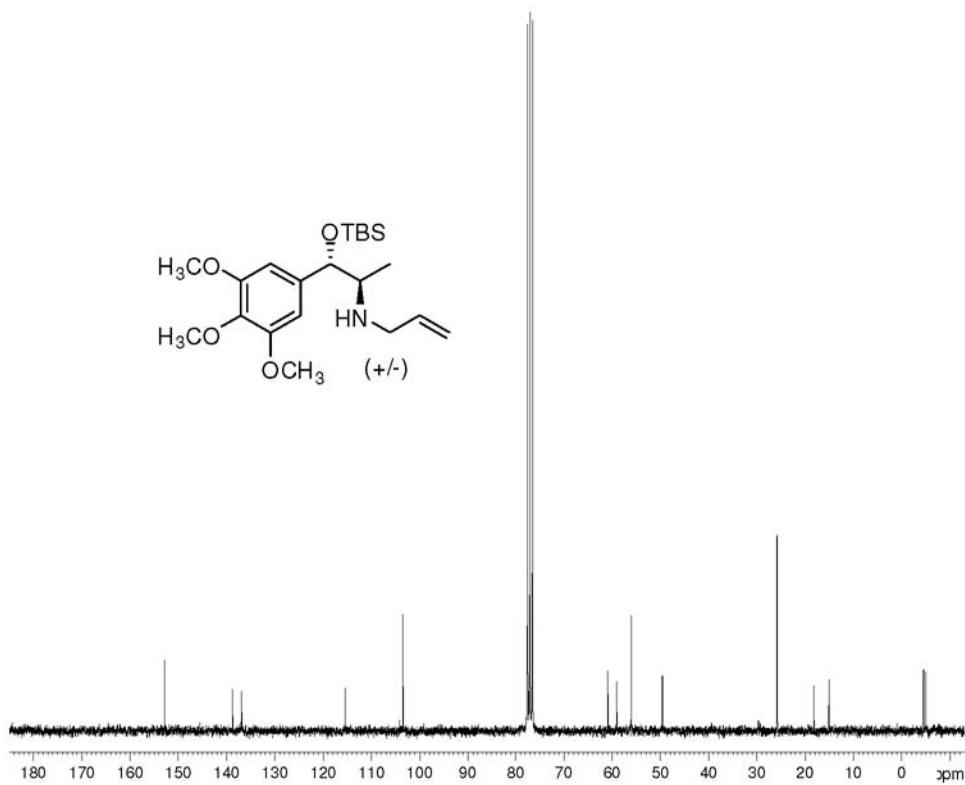


Figure S102. ^{13}C NMR (CDCl_3 , 62.5 MHz) of vicinal aminoalcohol **50**.

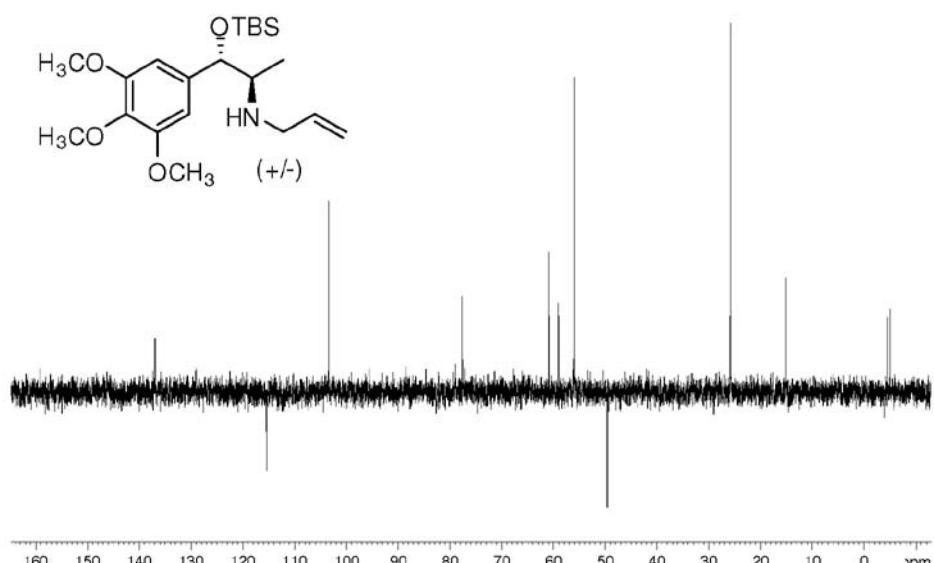


Figure S103. DEPT 135 (CDCl_3 , 62.5 MHz) of vicinal aminoalcohol **50**.

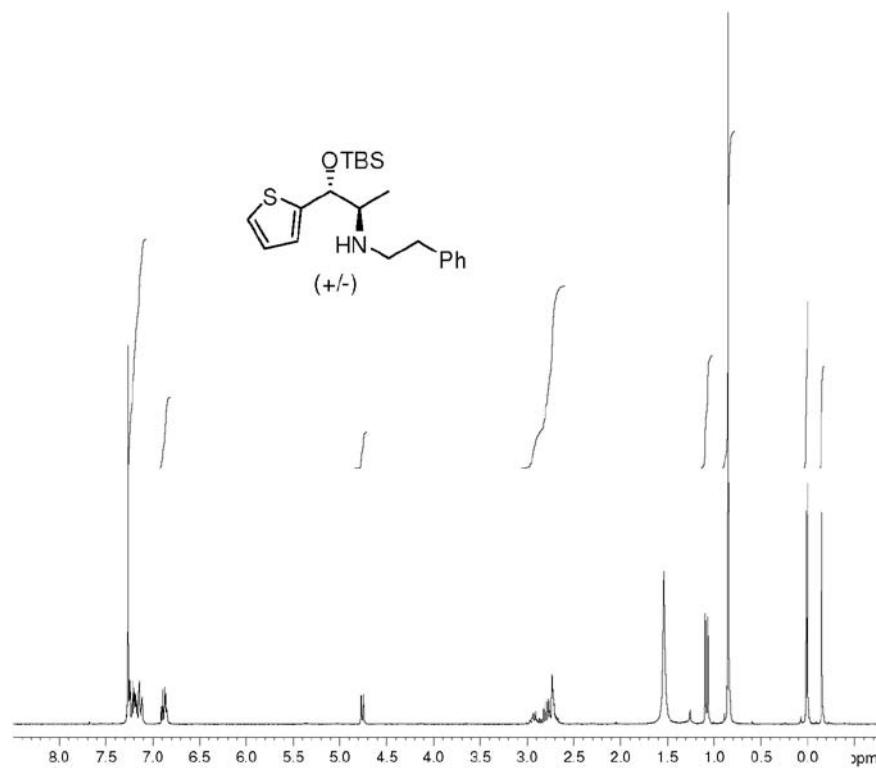


Figure S104. ¹H NMR (CDCl_3 , 250 MHz) of vicinal aminoalcohol **51**.

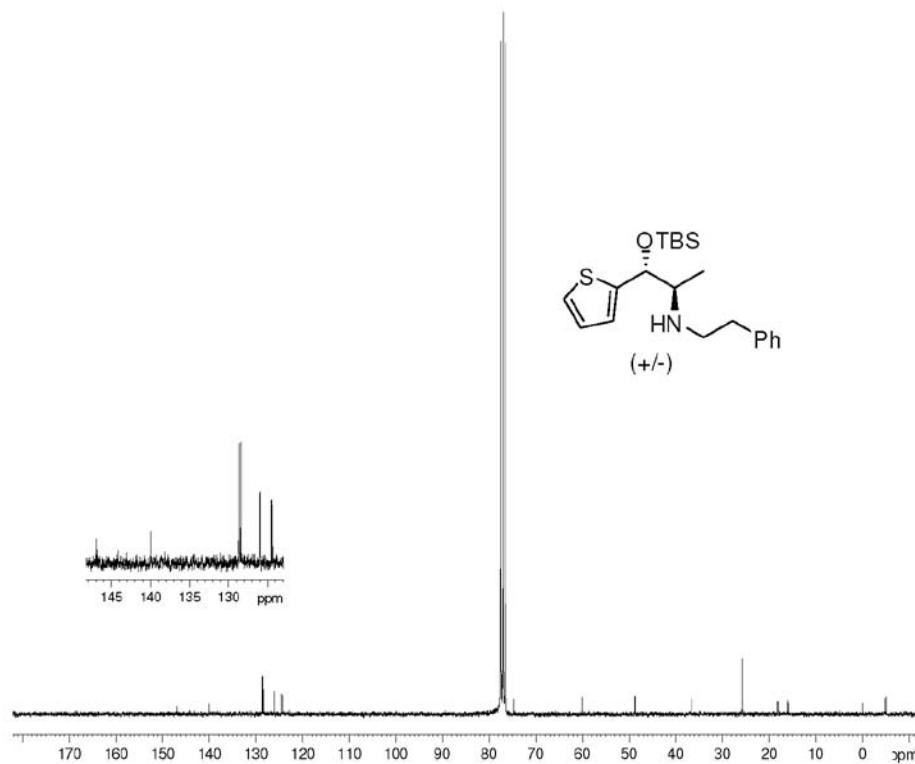


Figure S105. ¹³C NMR (CDCl_3 , 62.5 MHz) of vicinal aminoalcohol **51**.

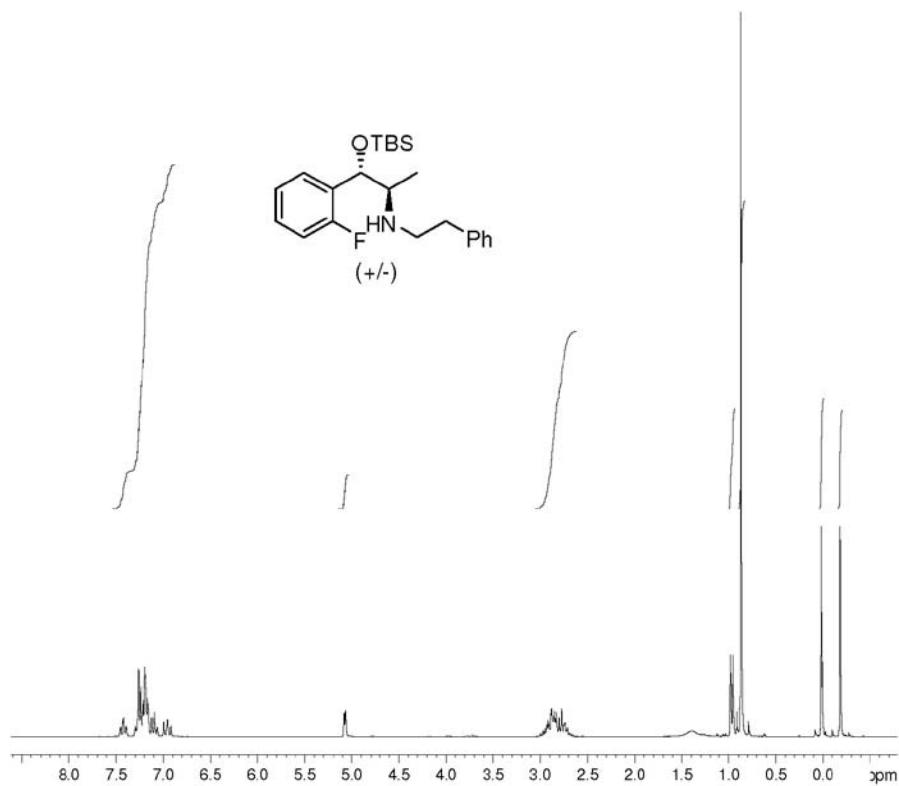


Figure S106. ^1H NMR (CDCl_3 , 250 MHz) of vicinal aminoalcohol **52**.

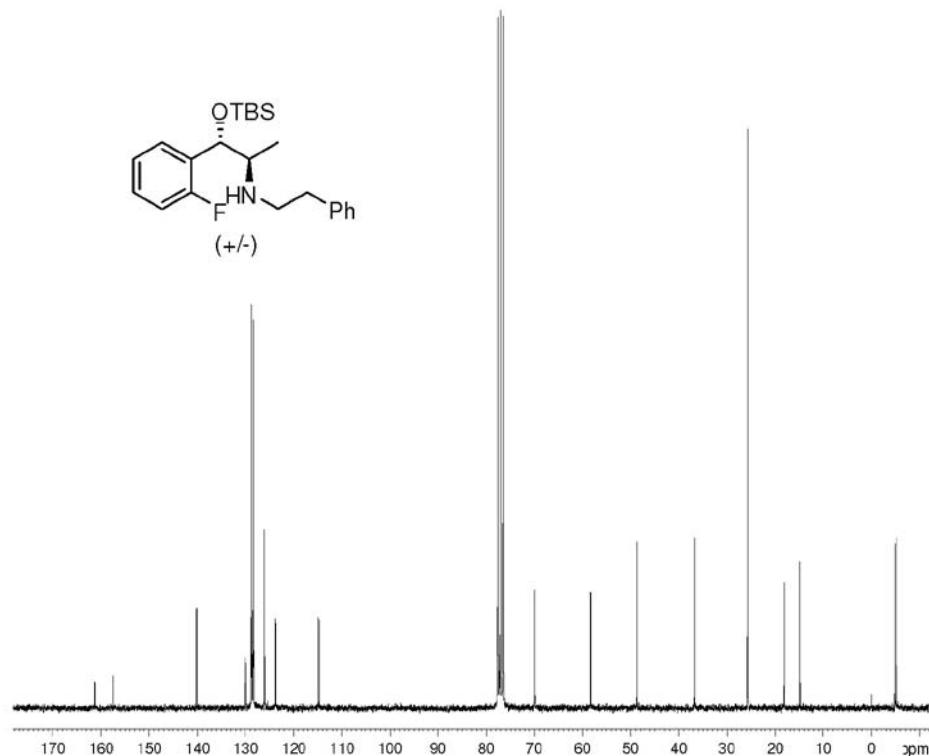


Figure S107. ^{13}C NMR (CDCl_3 , 62.5 MHz) of vicinal aminoalcohol **52**.

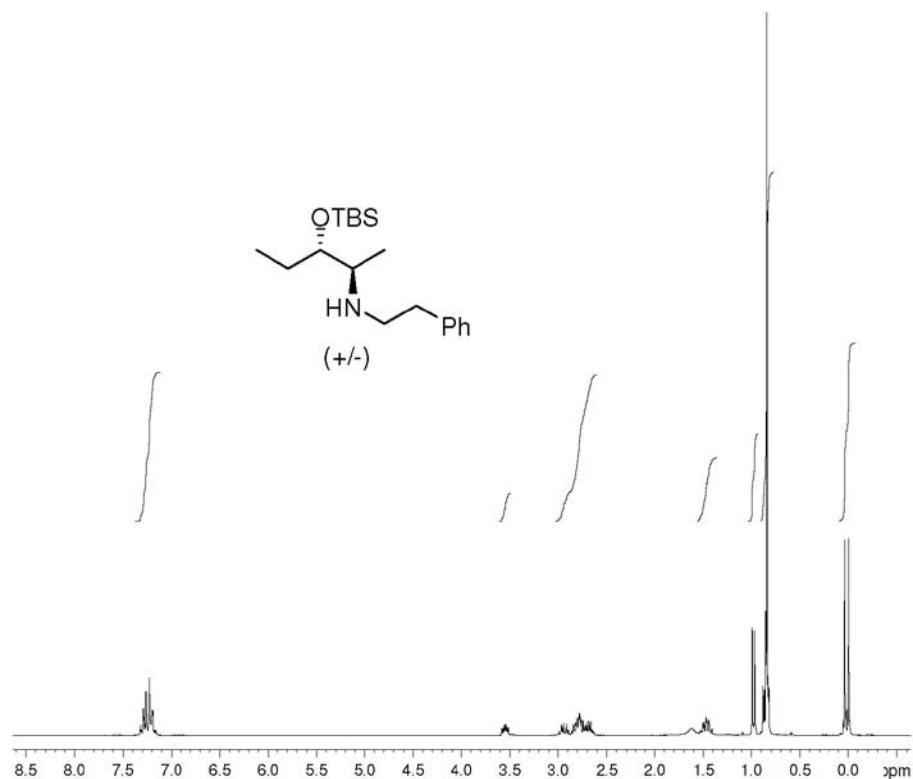


Figure S108. ¹H NMR (CDCl_3 , 250 MHz) of vicinal aminoalcohol **53**.

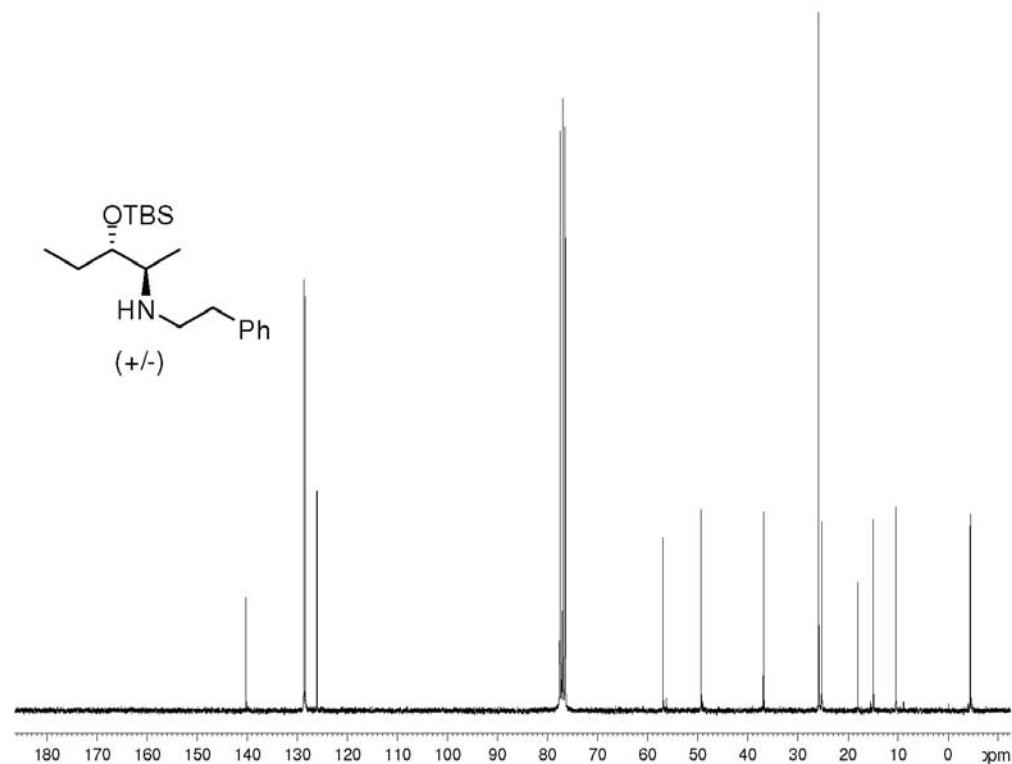


Figure S109. ¹³C NMR (CDCl_3 , 62.5 MHz) of vicinal aminoalcohol **53**.

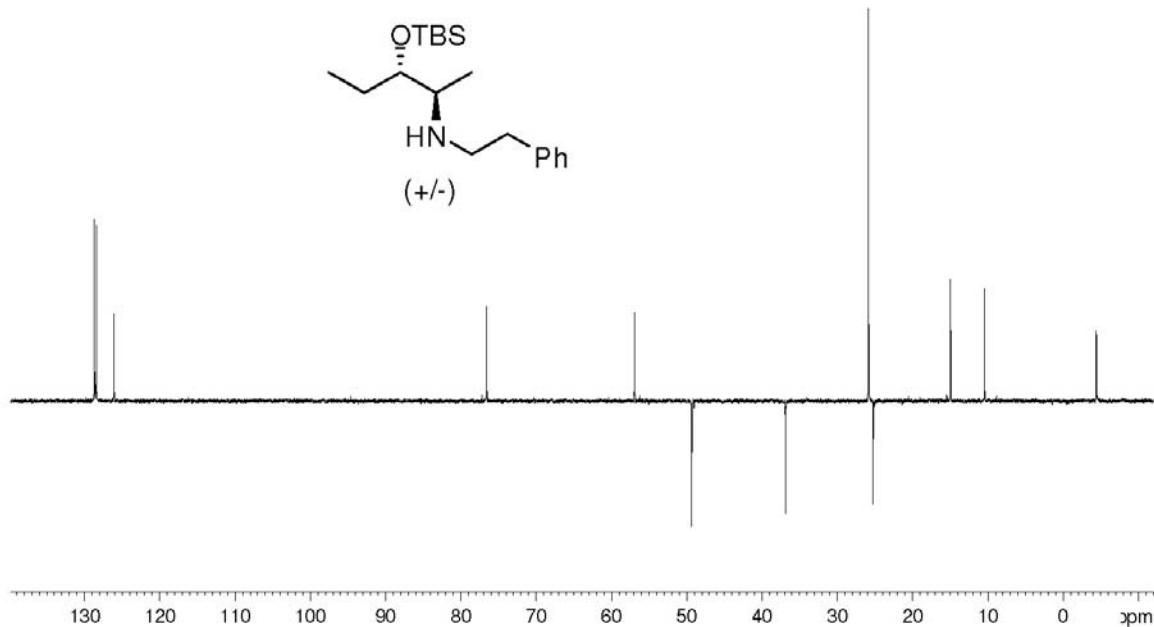


Figure S110. DEPT 135 (CDCl_3 , 62.5 MHz) of vicinal aminoalcohol **53**.

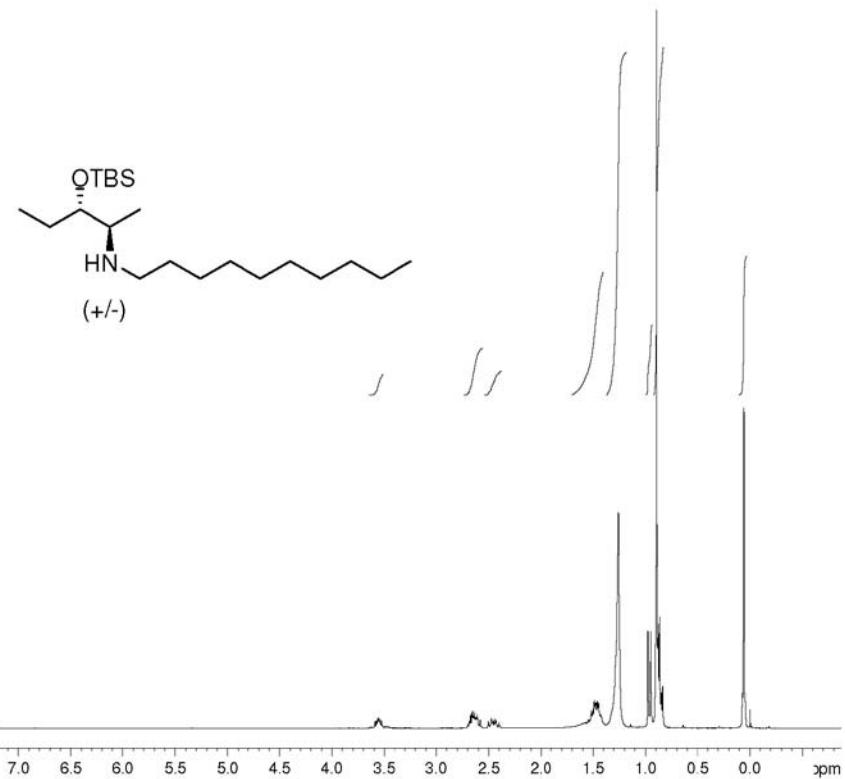


Figure S111. ¹H NMR (CDCl_3 , 250 MHz) of vicinal aminoalcohol **54**.

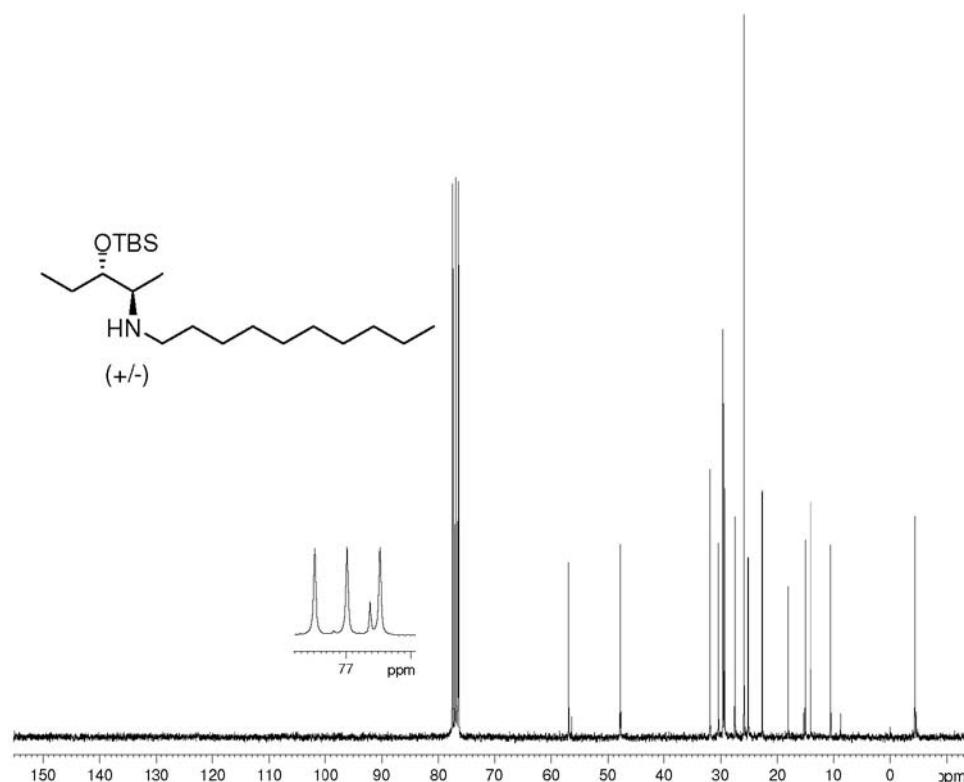


Figure S112. ^{13}C NMR (CDCl_3 , 62.5 MHz) of vicinal aminoalcohol **54**.

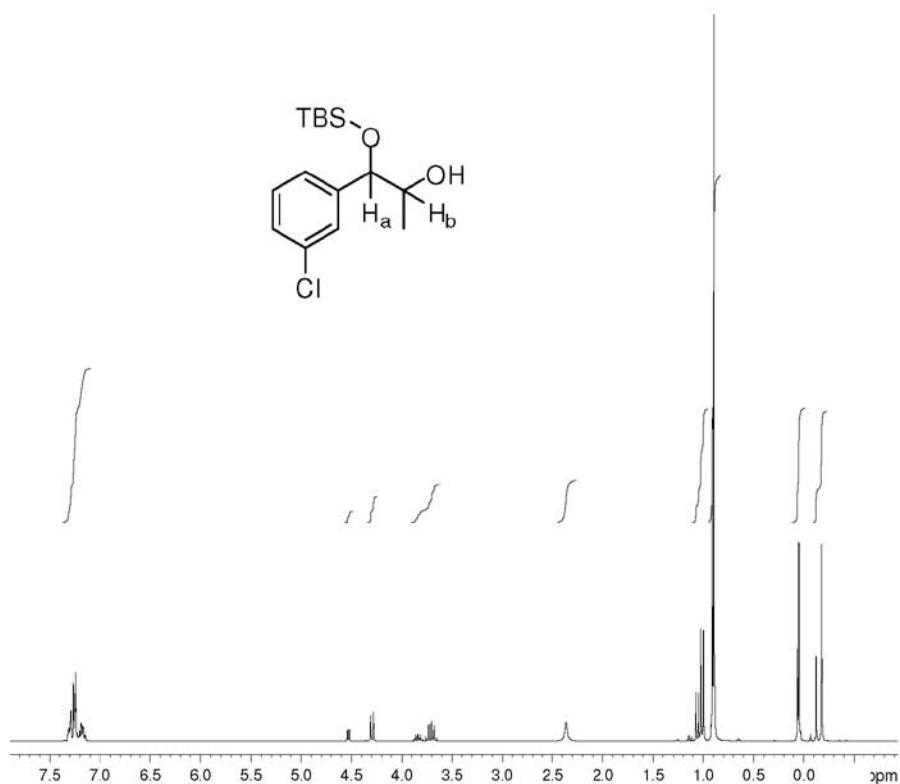


Figure S113. ^1H NMR (CDCl_3 , 250 MHz) of monosilylated diol **60**.

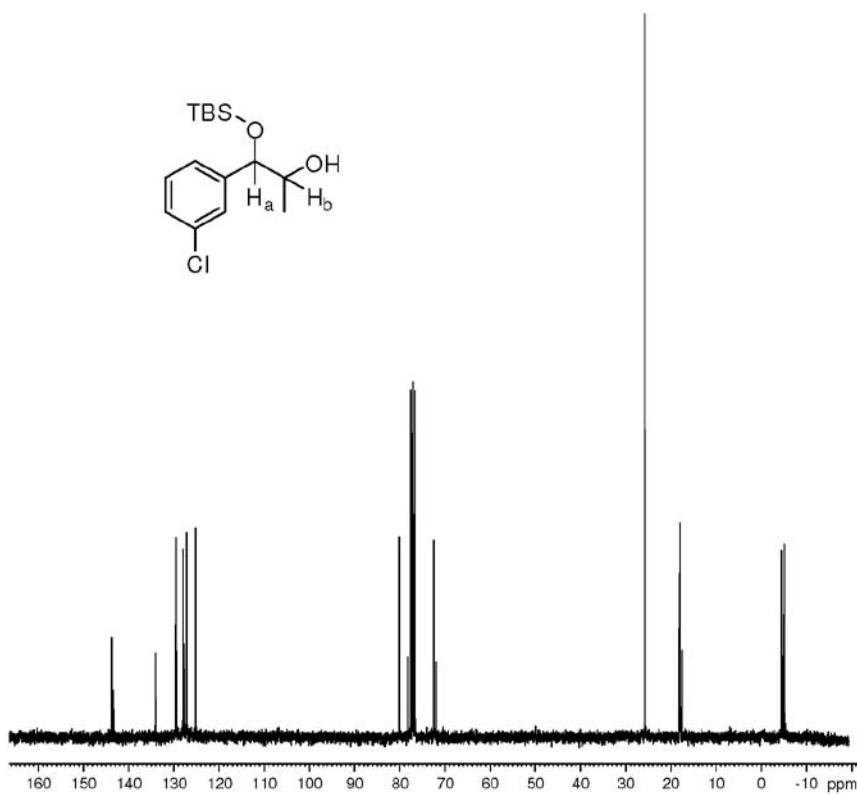


Figure S114. ¹³C NMR (CDCl₃, 75.4 MHz) of monosilylated diol **60**.

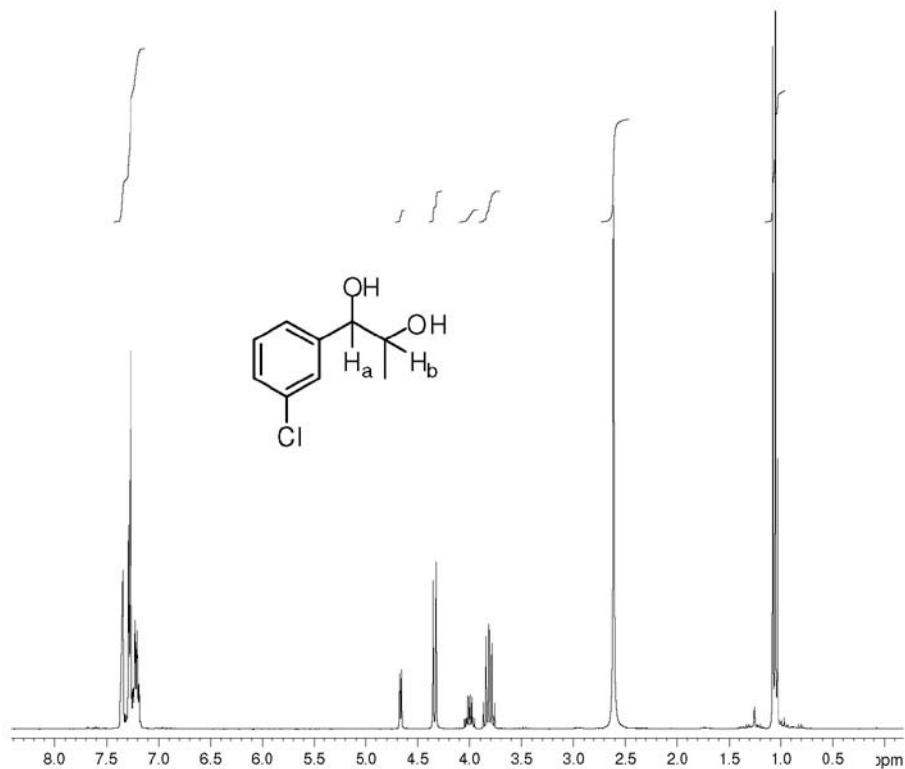


Figure S115. ¹H NMR (CDCl₃, 250 MHz) of diol **61**.

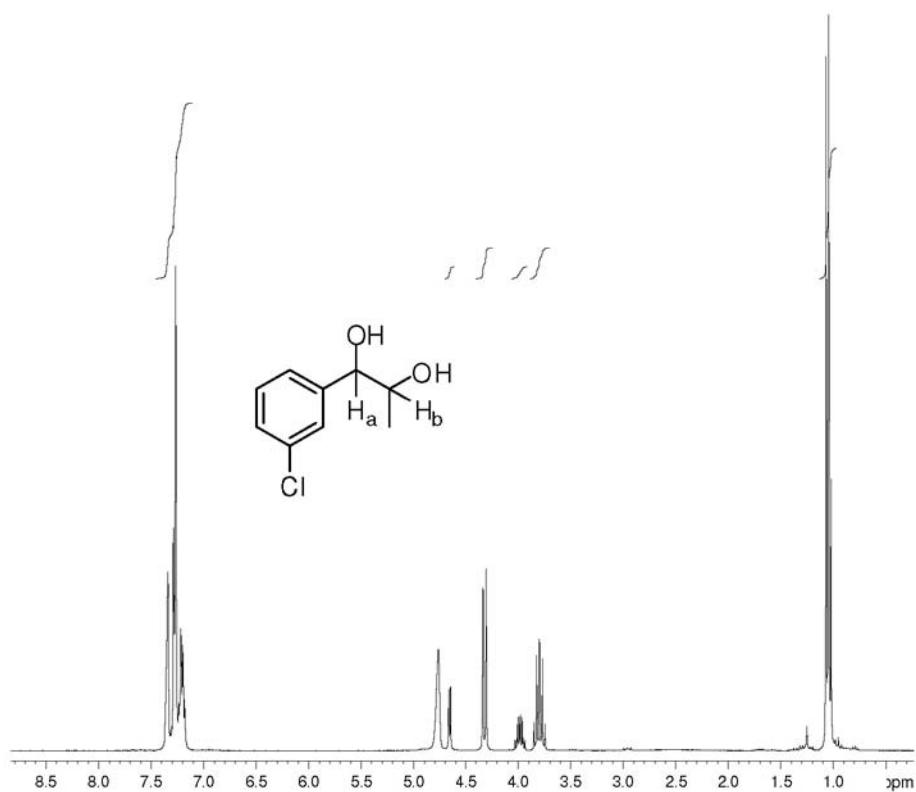


Figure S116. ¹H NMR ($\text{CDCl}_3 + 2$ drops of D_2O , 250 MHz) of diol **61**.

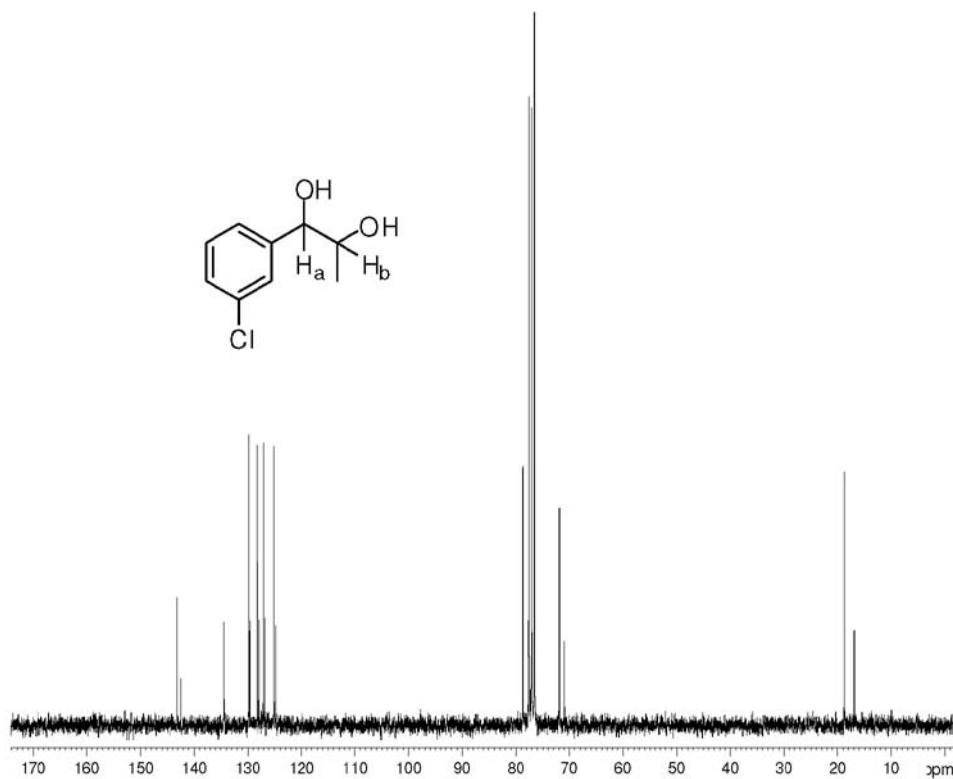


Figure S117. ¹³C NMR (CDCl_3 , 62.5 MHz) of diol **61**.

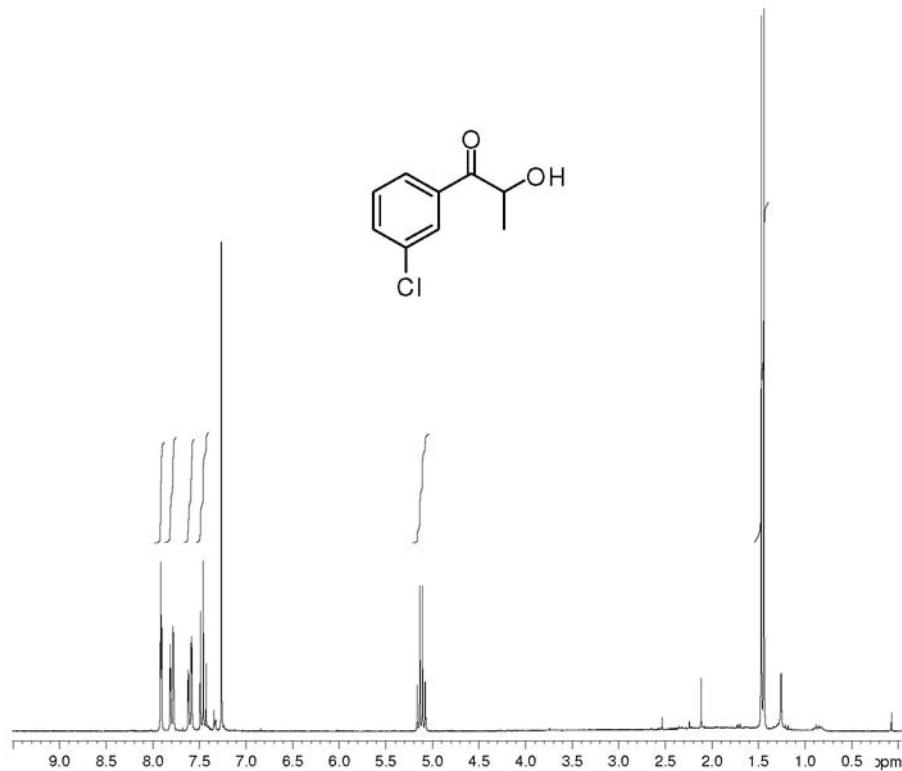


Figure S118. ¹H NMR (CDCl_3 , 250 MHz) of acyloin **59**.

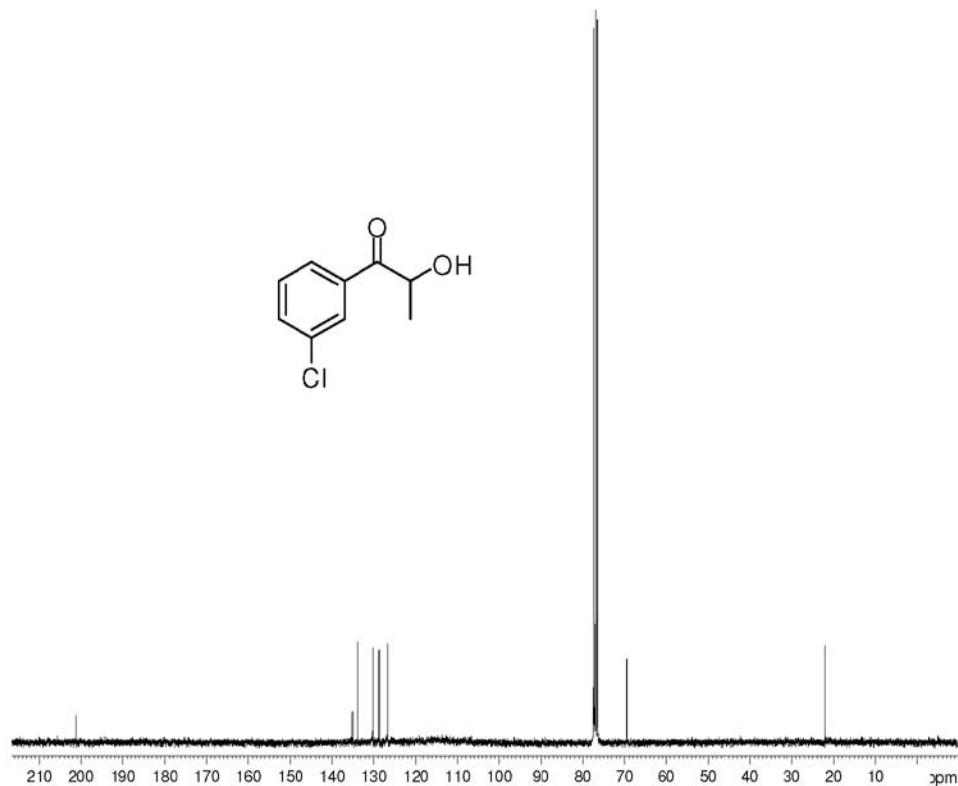


Figure S119. ¹³C NMR (CDCl_3 , 75.4 MHz) of acyloin **59**.

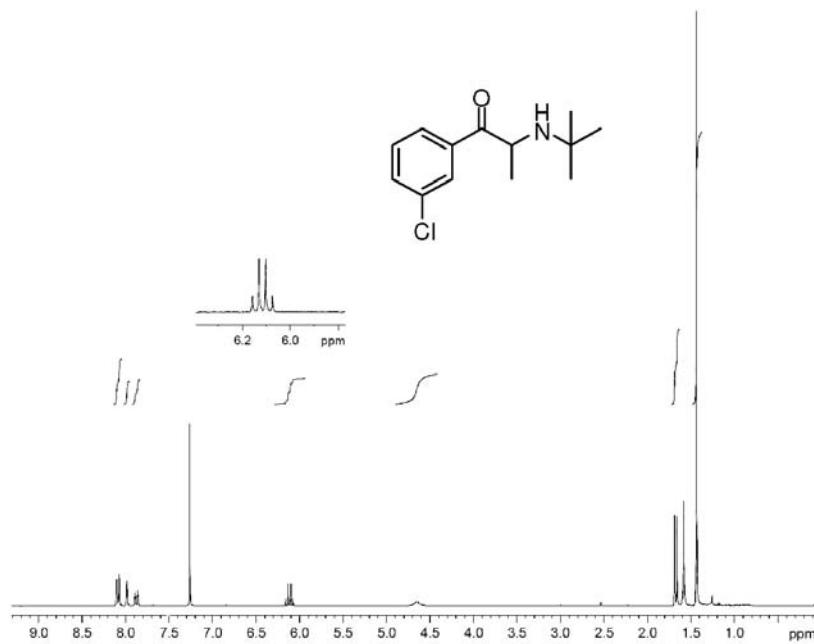


Figure S120. ¹H NMR (CDCl₃, 250 MHz) of bupropion (**1**).

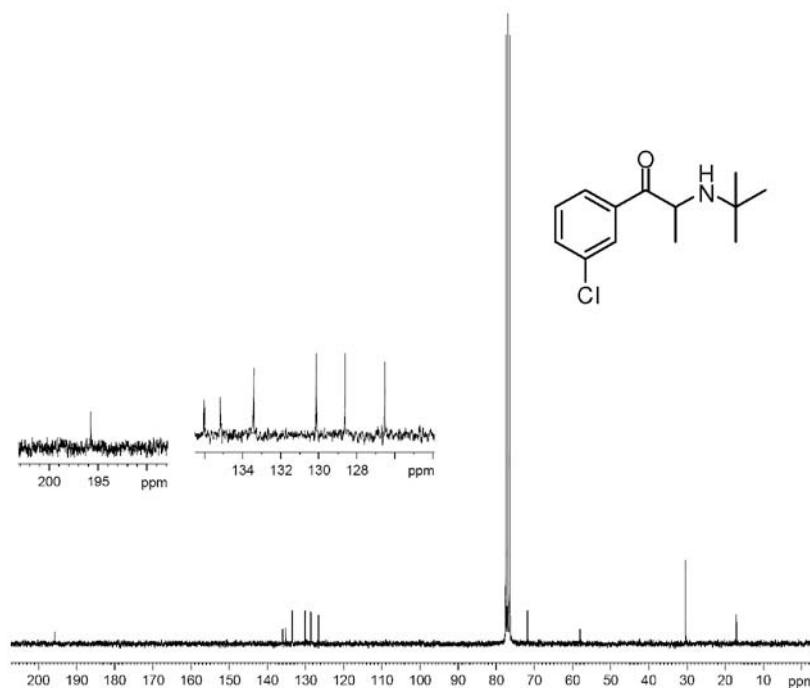


Figure S121. ¹³C NMR (CDCl₃, 62.5 MHz) of bupropion (**1**).

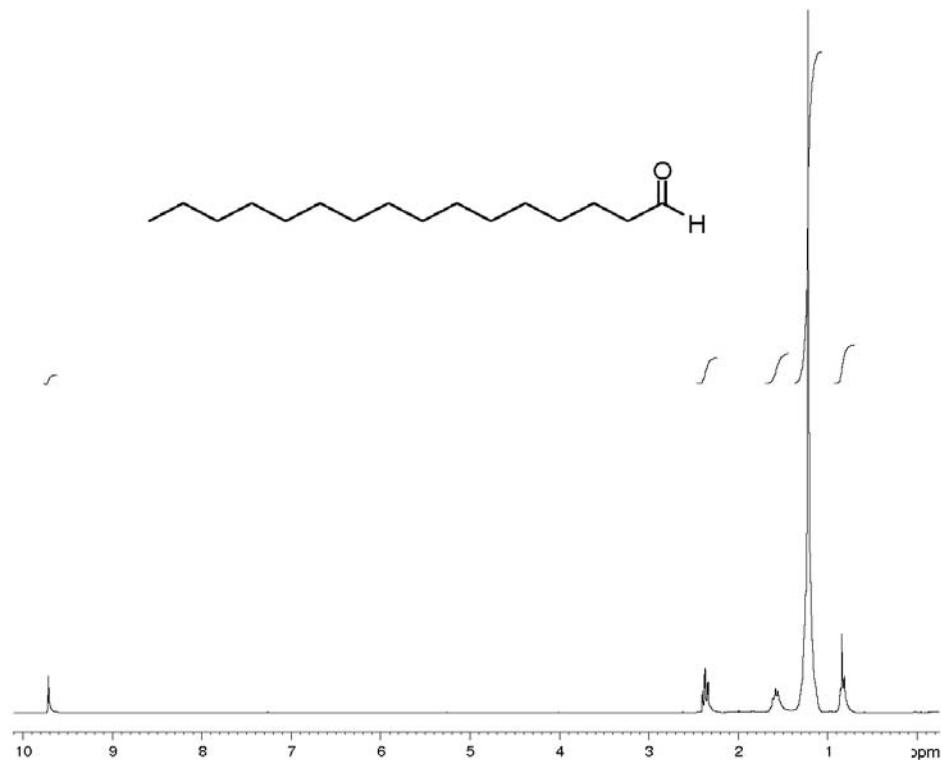


Figure S122. ¹H NMR (CDCl₃, 250 MHz) of hexadecanal.

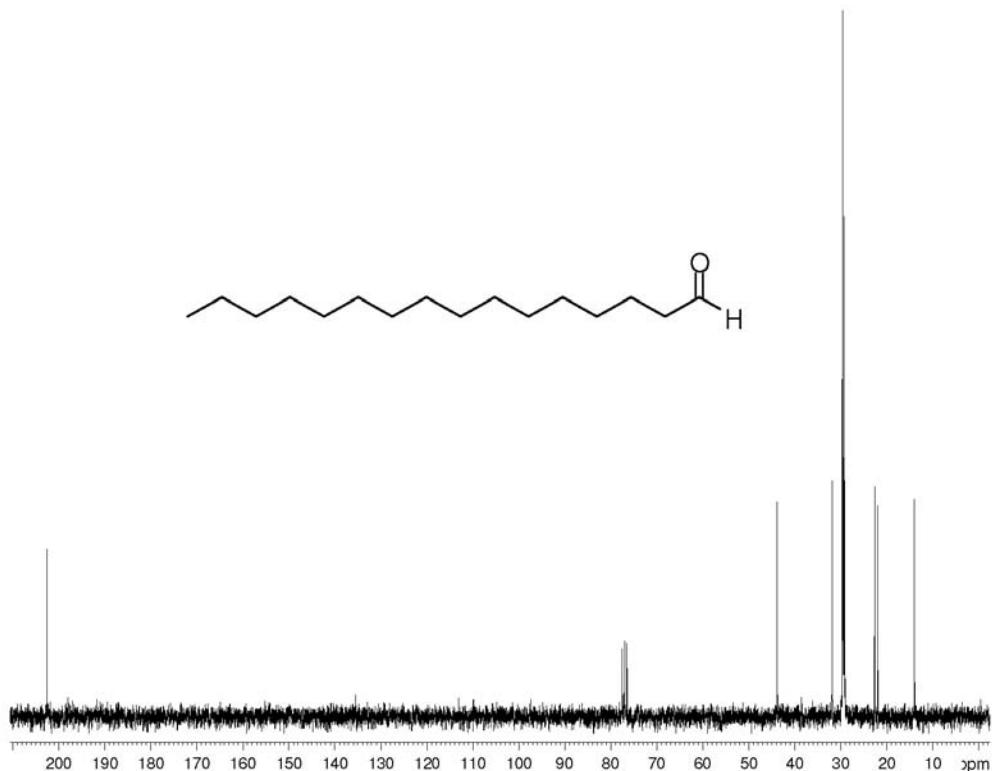


Figure S123. ¹³C NMR (CDCl₃, 62.5 MHz) of hexadecanal.

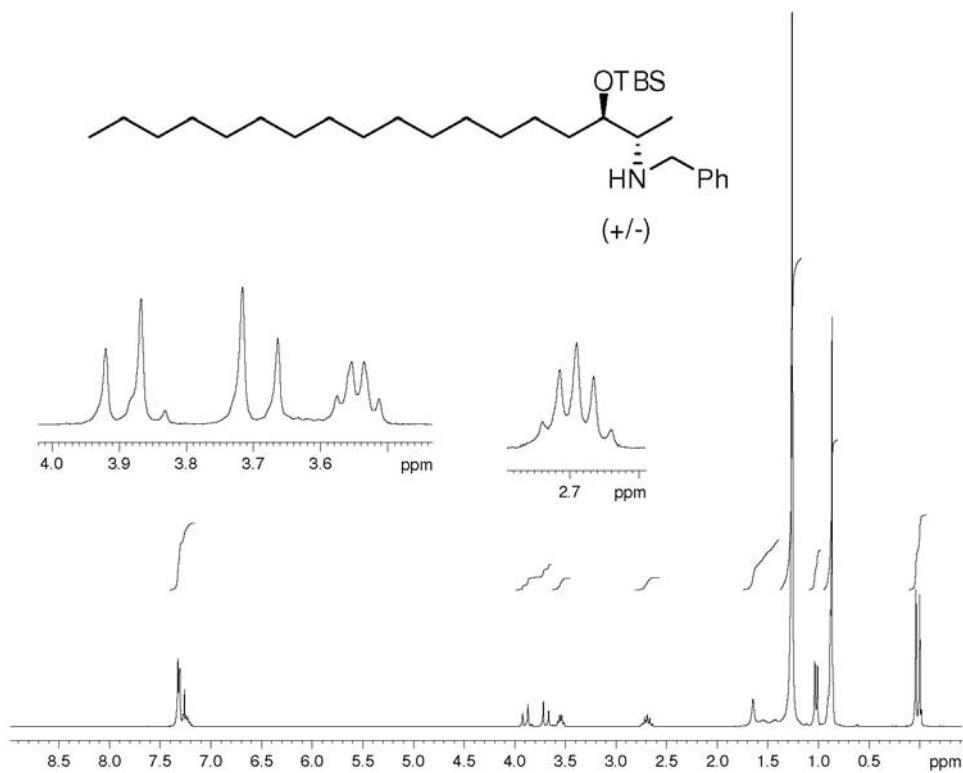


Figure S124. ¹H NMR (CDCl_3 , 250 MHz) of vicinal aminoalcohol **62**.

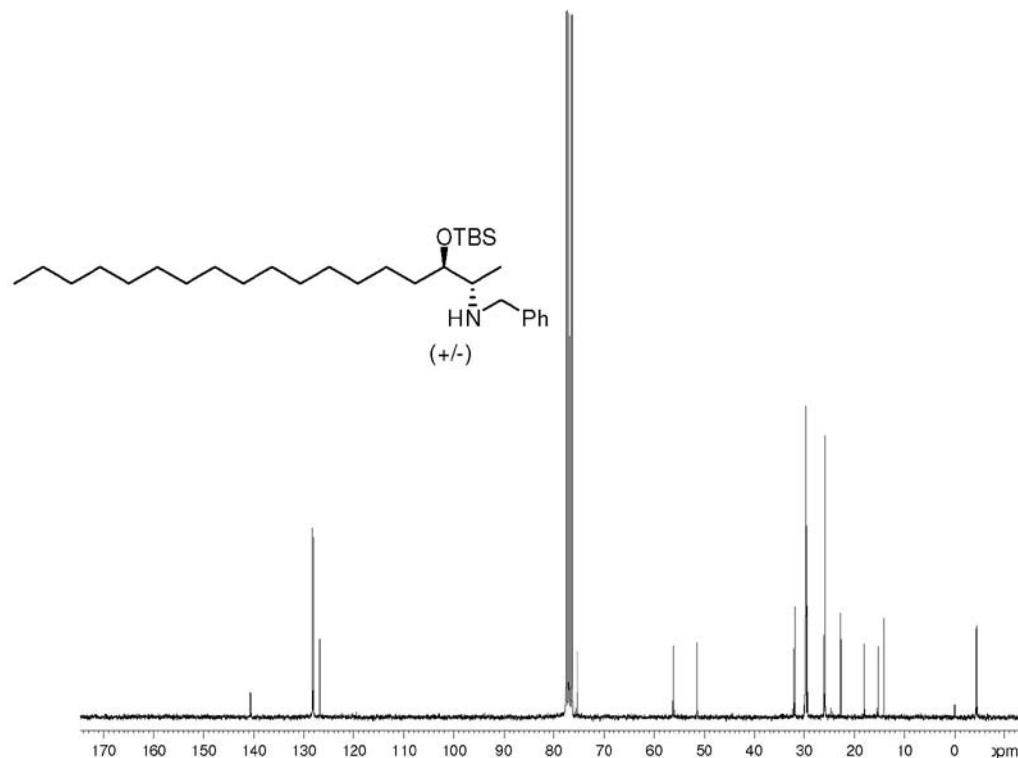


Figure S125. ¹³C NMR (CDCl_3 , 62.5 MHz) of vicinal aminoalcohol **62**.

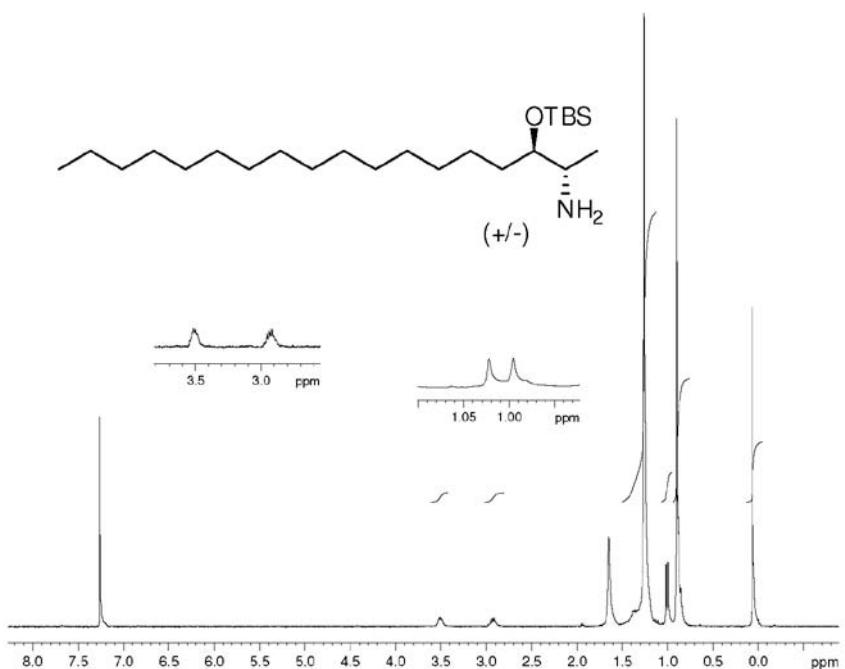


Figure S126. ^1H NMR (CDCl_3 , 250 MHz) of silylated aminoalcohol **63**.

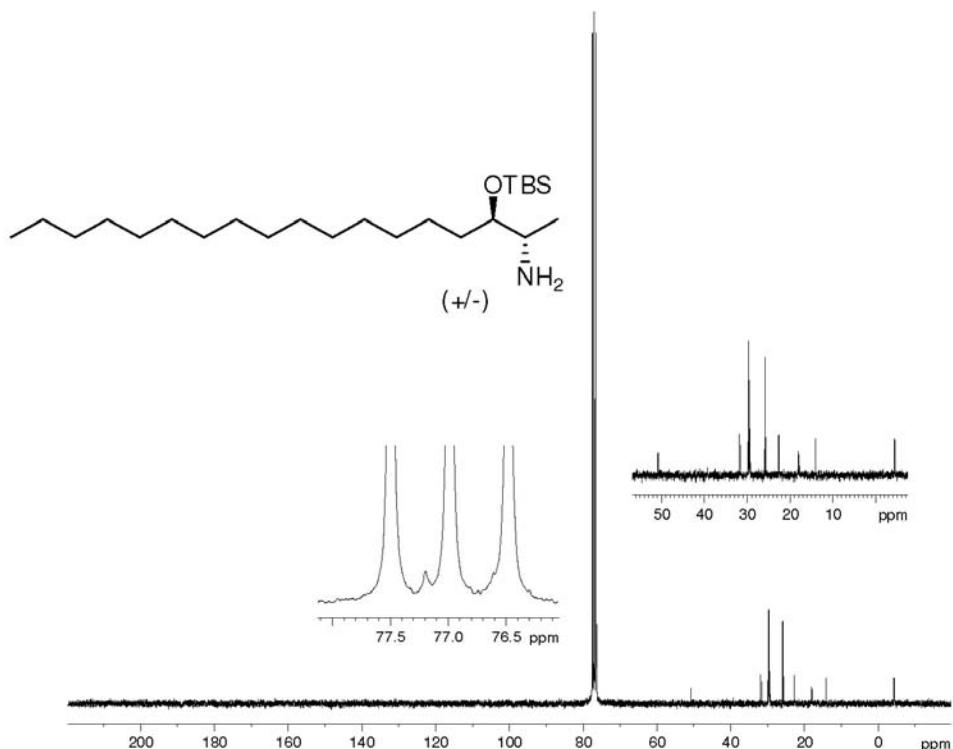


Figure S127. ^{13}C NMR (CDCl_3 , 62.5 MHz) of silylated aminoalcohol **63**.

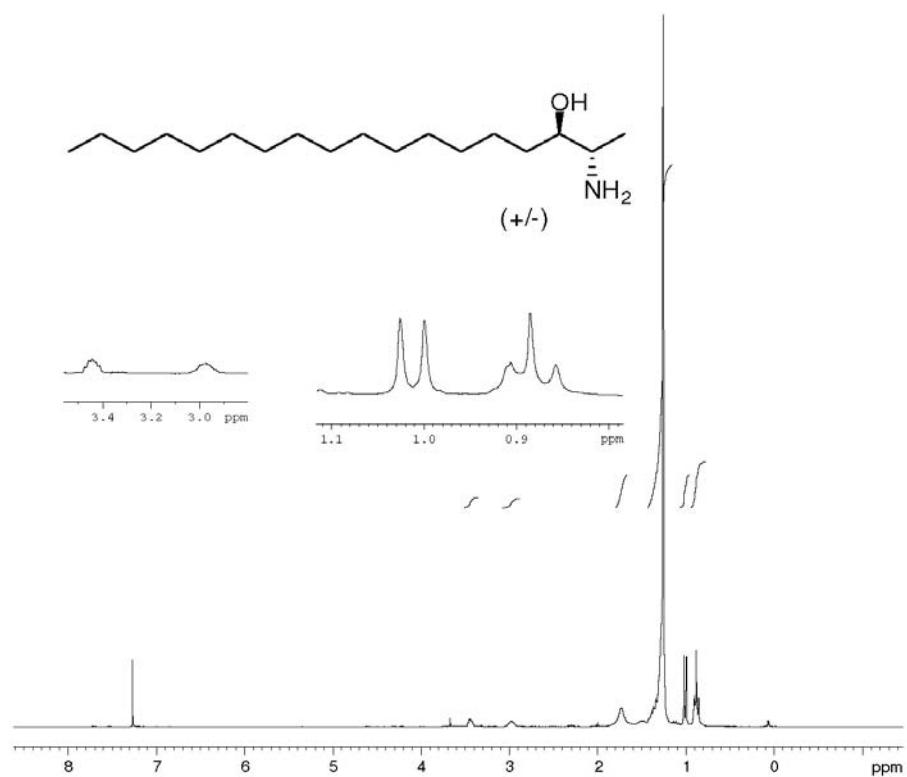


Figure S128. ¹H NMR (CDCl_3 , 250 MHz) of spisulosine (2).

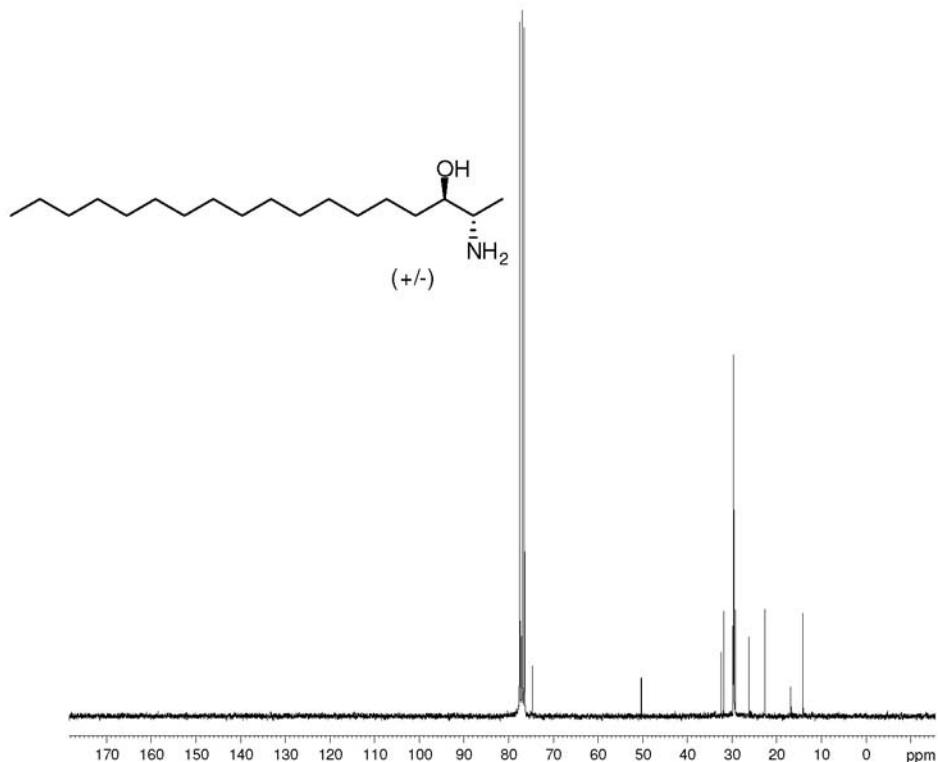


Figure S129. ¹³C NMR (CDCl_3 , 62.5 MHz) of spisulosine (2).