

Synthesis and Biological Activity of Novel (*Z*)- and (*E*)-Verbenone Oxime Esters

Qiong Hu, Wen-Gui Duan *, Gui-Shan Lin *, Min Huang and Fu-Hou Lei

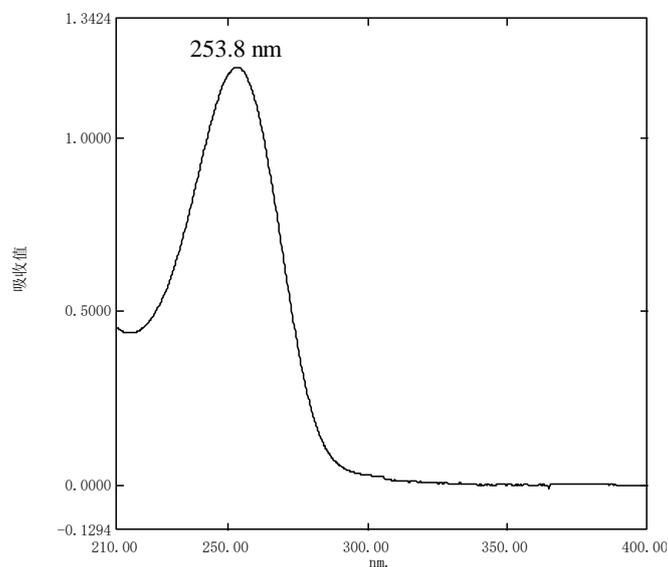


Figure S1. UV-vis spectrum of verbenone 2 in EtOH.

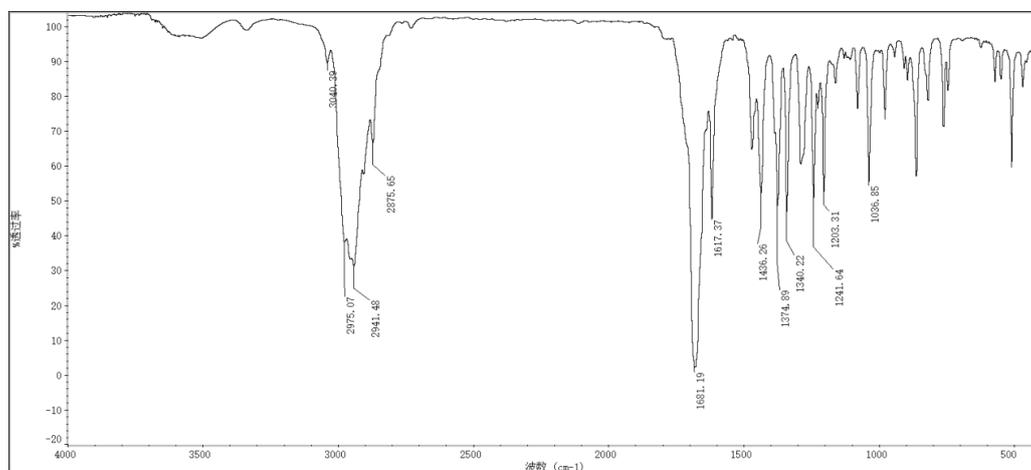


Figure S2. FTIR spectrum of verbenone 2.

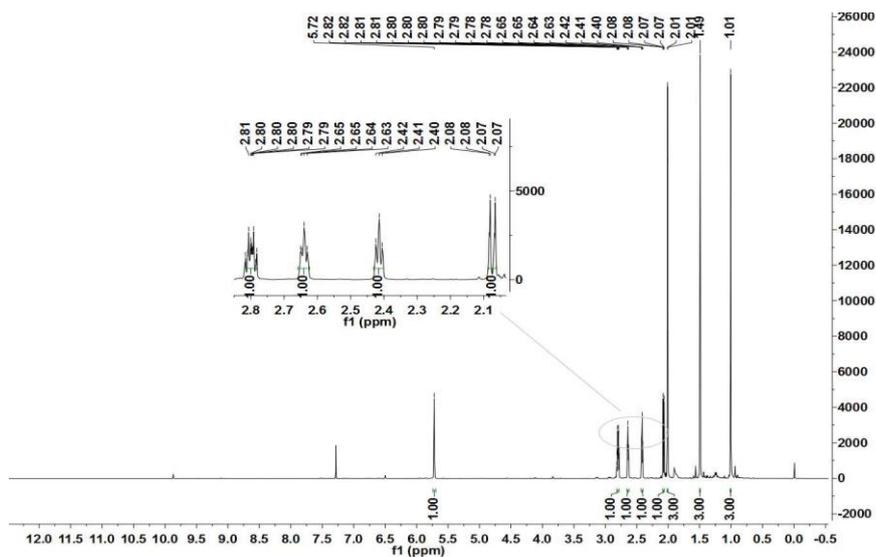


Figure S3. ¹H-NMR spectrum of verbenone 2 in CDCl₃.

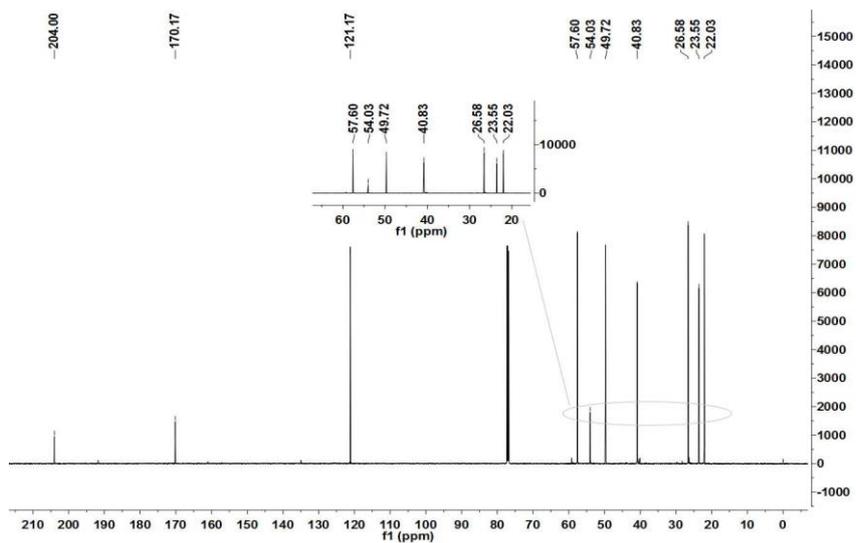


Figure S4. ¹³C-NMR spectrum of verbenone 2 in CDCl₃.

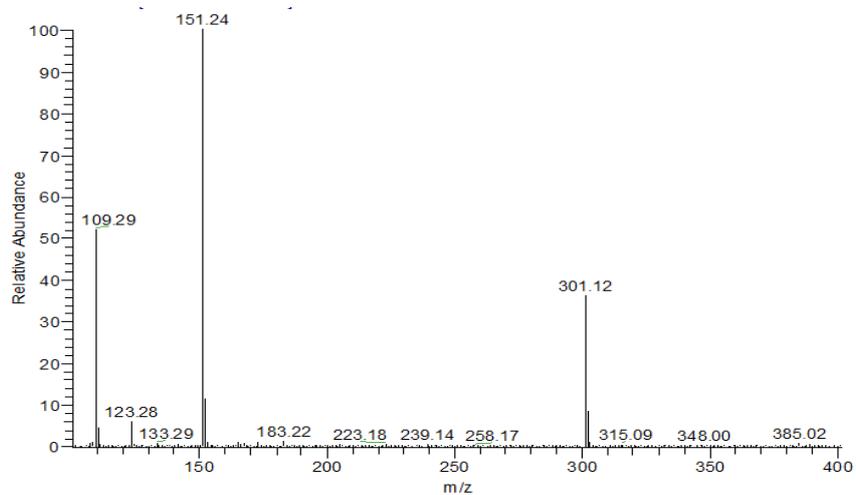


Figure S5. ESI-MS spectrum of verbenone 2.

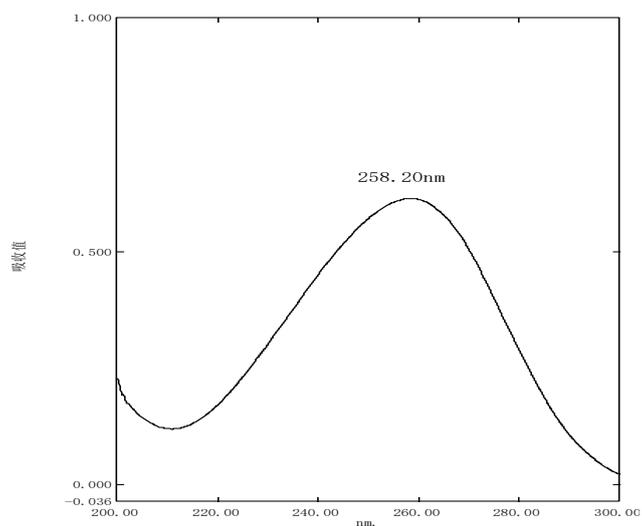


Figure S11. UV-vis spectrum of (Z)-verbenone oxime 3 in EtOH.

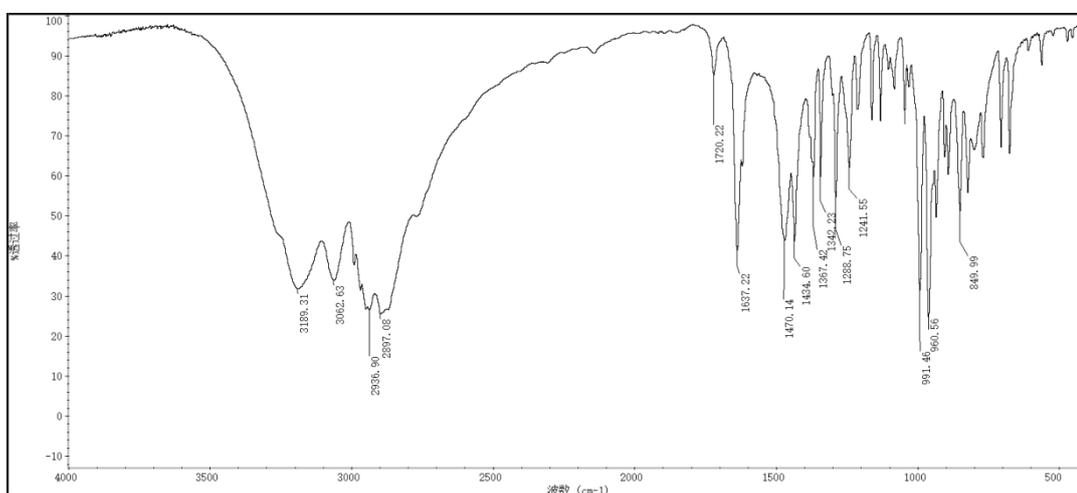


Figure S12. FTIR spectrum of (Z)-verbenone oxime 3.

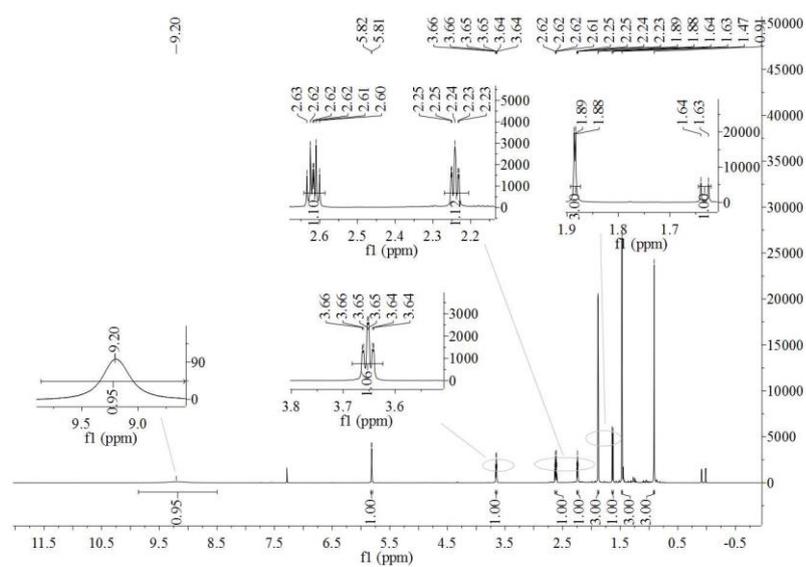


Figure S13. ¹H-NMR spectrum of (Z)-verbenone oxime 3 in CDCl₃.

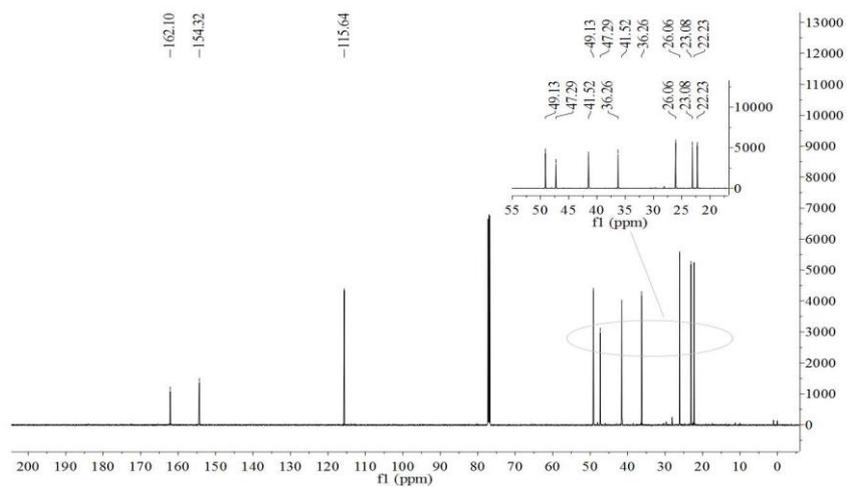


Figure S14. ^{13}C -NMR spectrum of (*Z*)-verbenone oxime **3** in CDCl_3 .

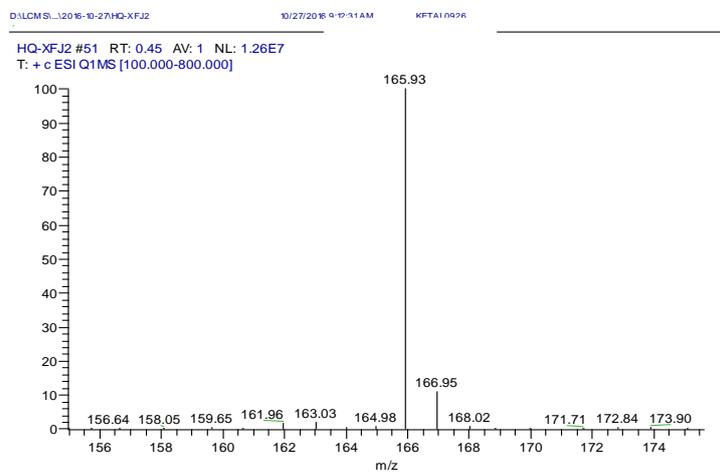


Figure S15. ESI-MS spectrum of (*Z*)-verbenone oxime **3**.

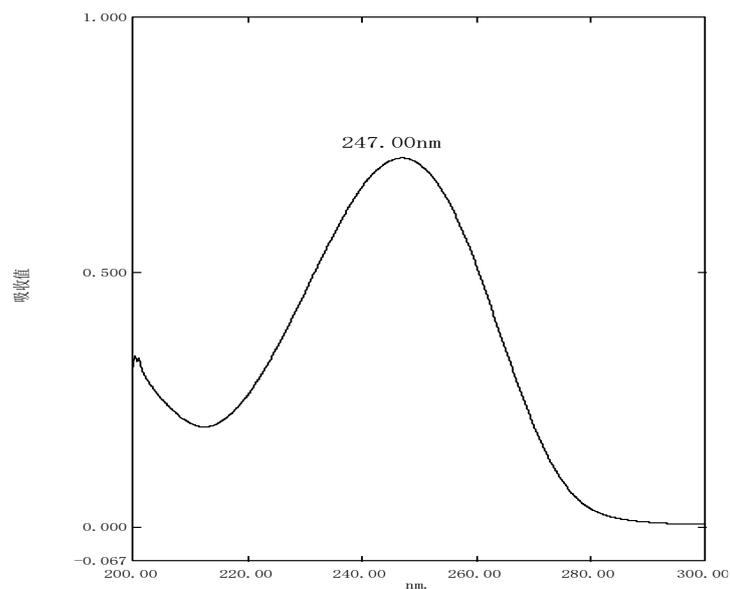


Figure S6. UV-vis spectrum of (*E*)-verbenone oxime **3** in EtOH.

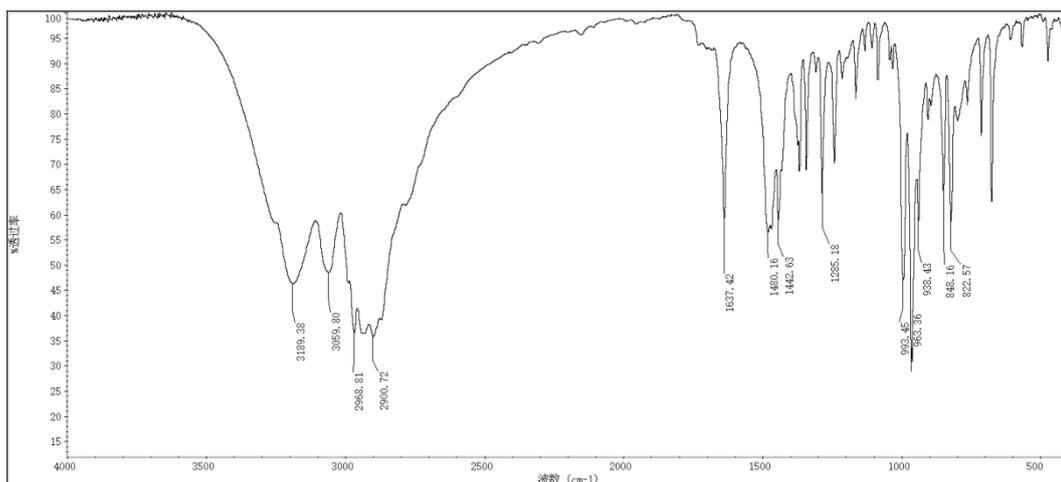


Figure S7. FTIR spectrum of (*E*)-verbenone oxime **3**.

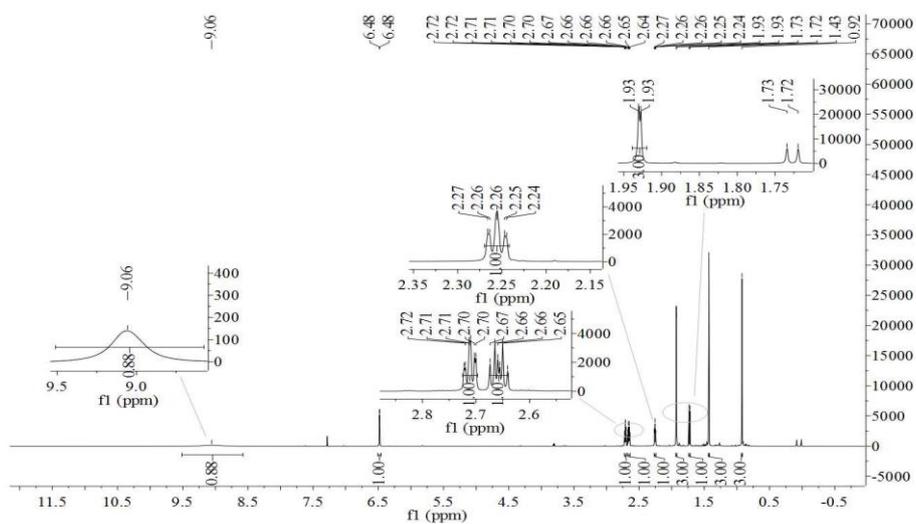


Figure S8. ¹H-NMR spectrum of (*E*)-verbenone oxime **3** in CDCl₃.

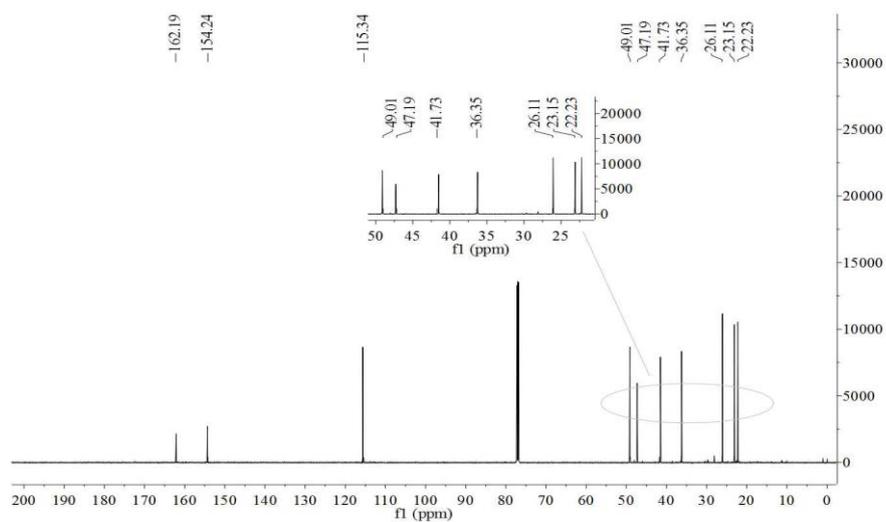


Figure S9. ¹³C-NMR spectrum of (*E*)-verbenone oxime **3** in CDCl₃.

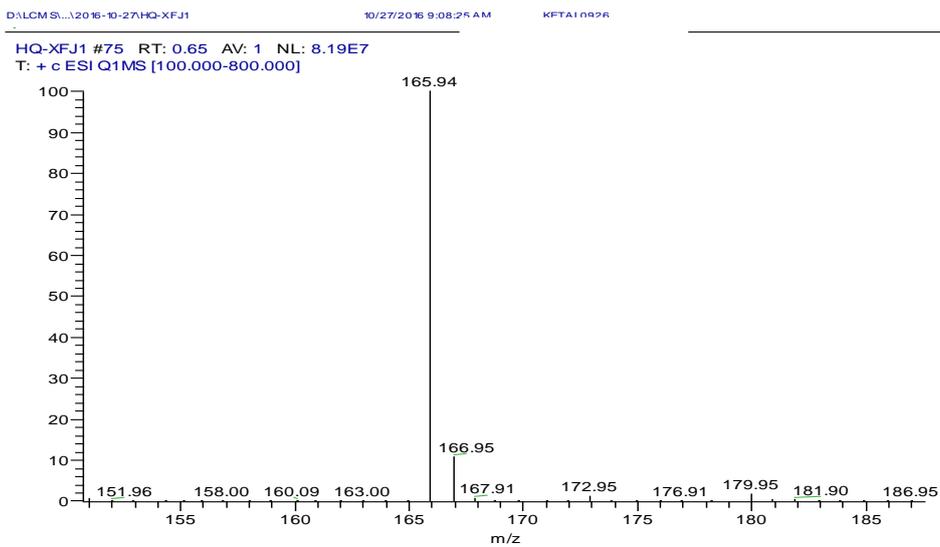


Figure S10. ESI-MS spectrum of (*E*)-verbenone oxime 3.

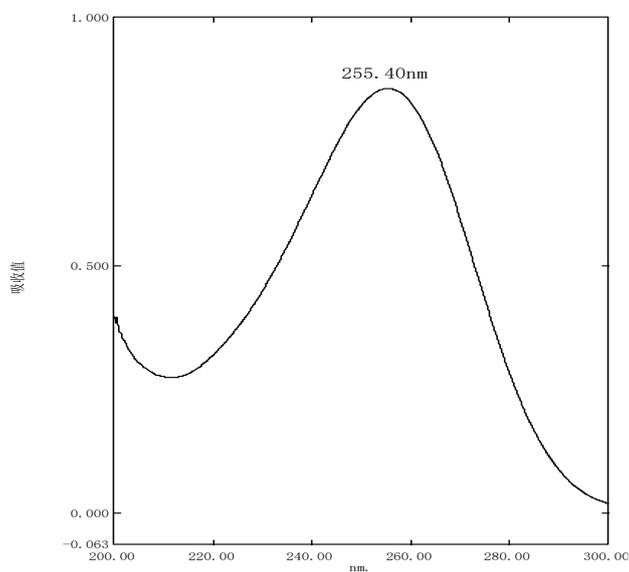


Figure S23. UV-vis spectrum of (*Z*)-verbenone *O*-*n*-pentanoyl oxime 4a in EtOH.

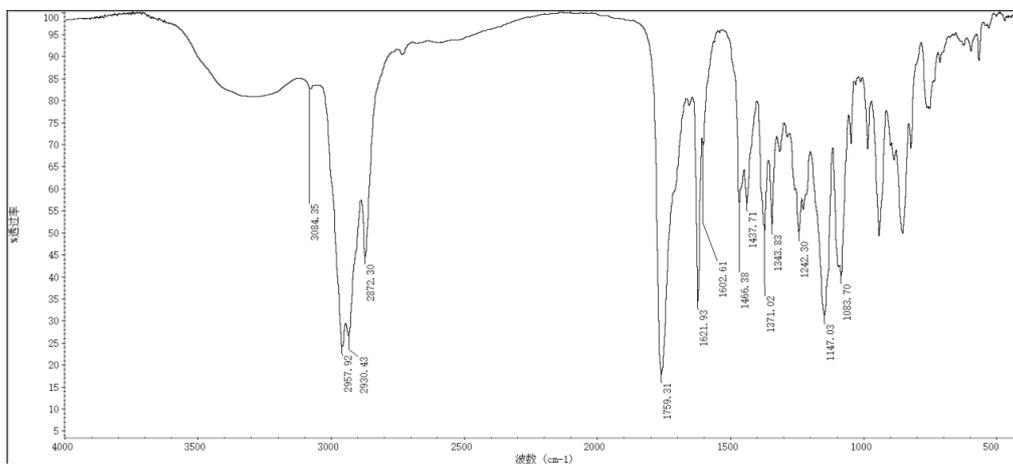


Figure S24. FTIR spectrum of (*Z*)-verbenone *O*-*n*-pentanoyl oxime 4a.

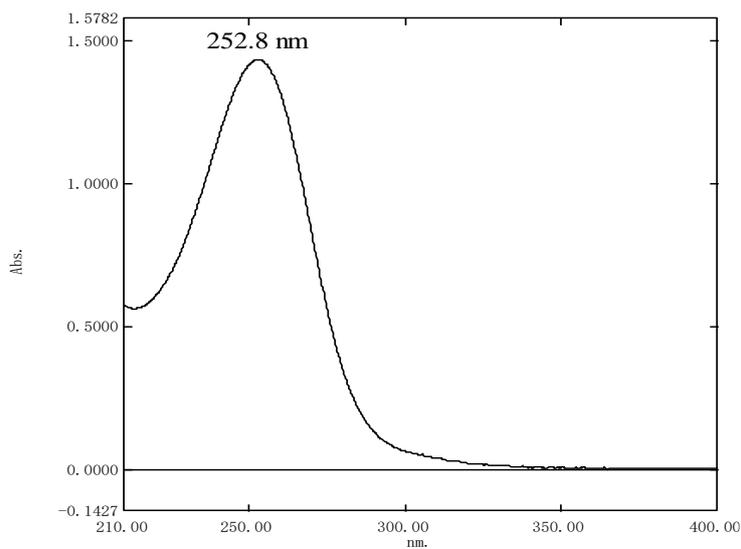


Figure S18. UV-vis spectrum of (*E*)-verbenone *O*-*n*-pentanoyl oxime **4a** in EtOH.

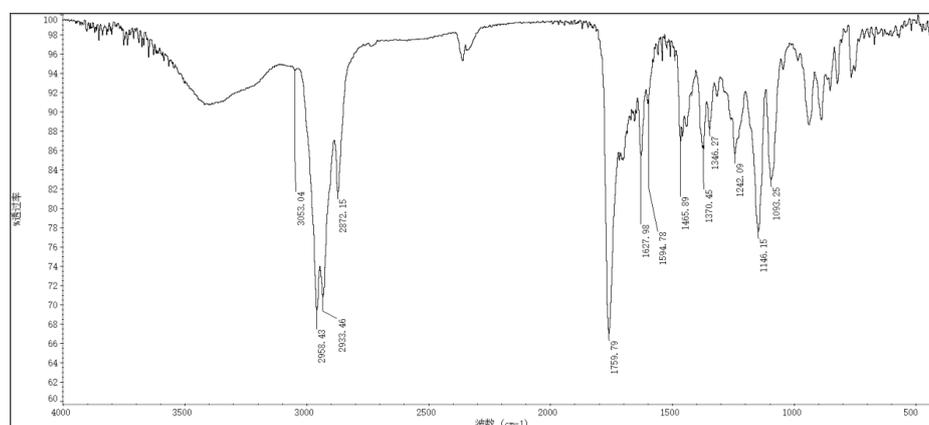


Figure S19. FTIR spectrum of (*E*)-verbenone *O*-*n*-pentanoyl oxime **4a**.

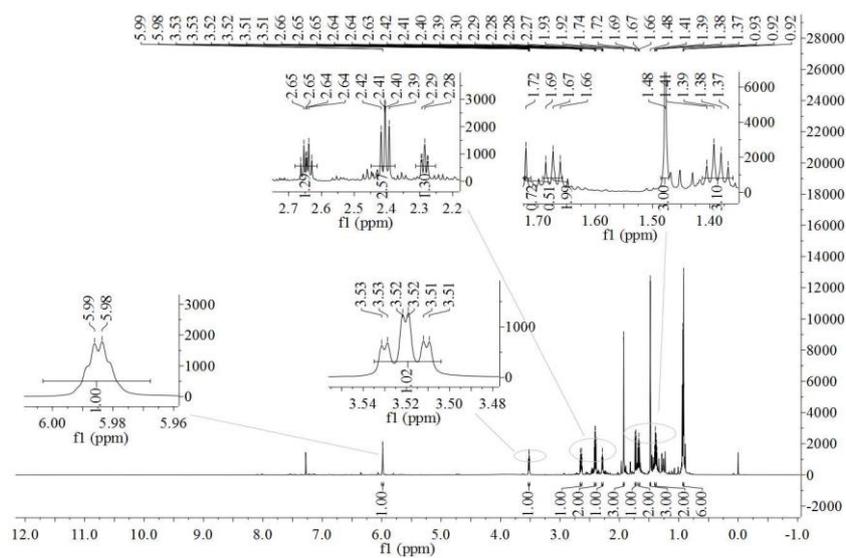


Figure S20. $^1\text{H-NMR}$ spectrum of (*E*)-verbenone *O*-*n*-pentanoyl oxime **4a** in CDCl_3 .

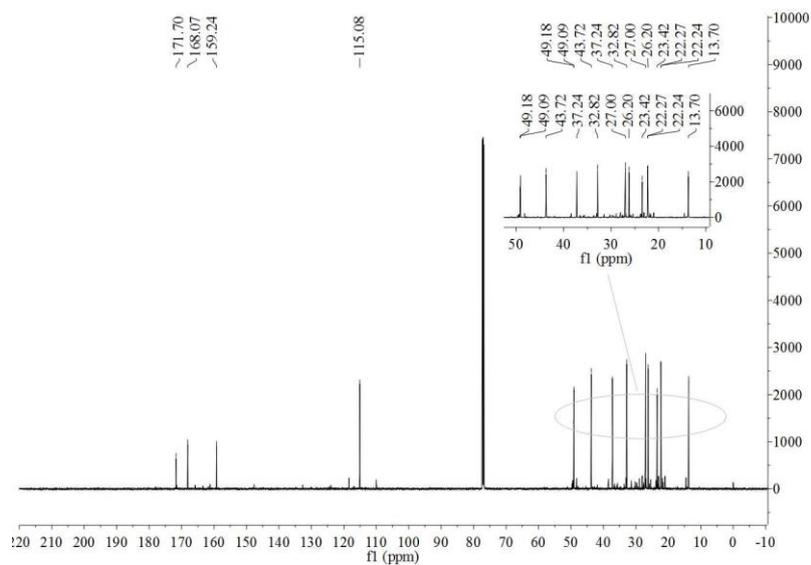


Figure S21. ^{13}C -NMR spectrum of (*E*)-verbenone *O*-*n*-pentanoyl oxime **4a** in CDCl_3 .

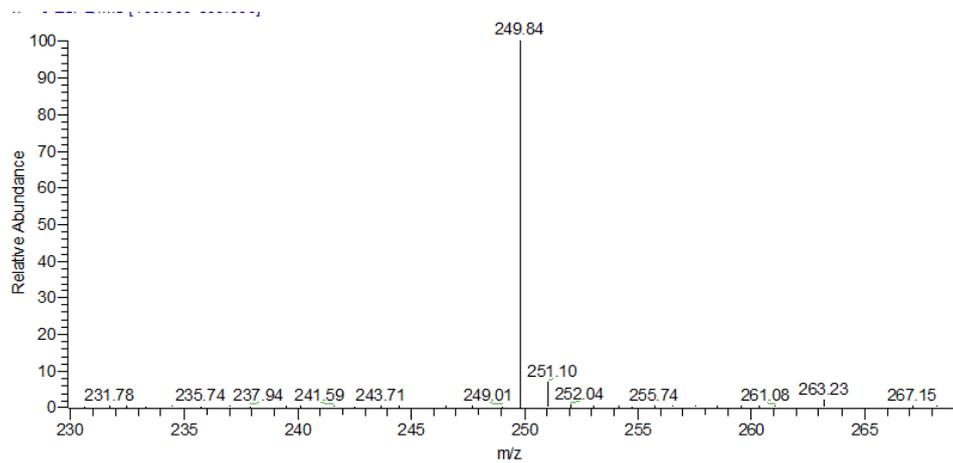


Figure S22. ESI-MS spectrum of (*E*)-verbenone *O*-*n*-pentanoyl oxime **4a**.

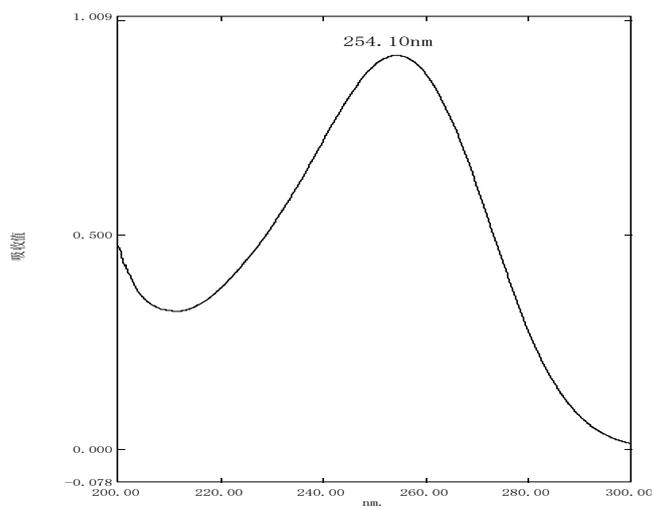


Figure S33. UV-vis spectrum of (*Z*)-verbenone *O*-*n*-hexanoyl oxime **4b** in EtOH .

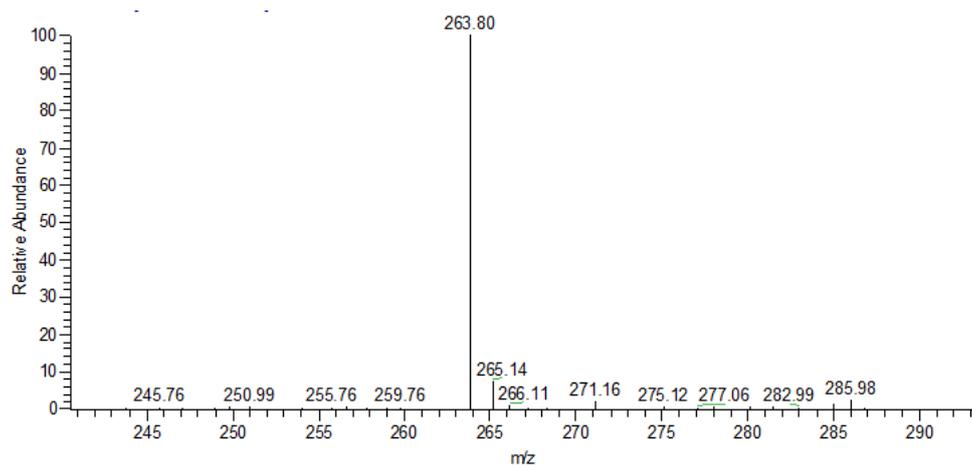


Figure S37. ESI-MS spectrum of (*Z*)-verbenone *O*-*n*-hexanoyl oxime **4b**.

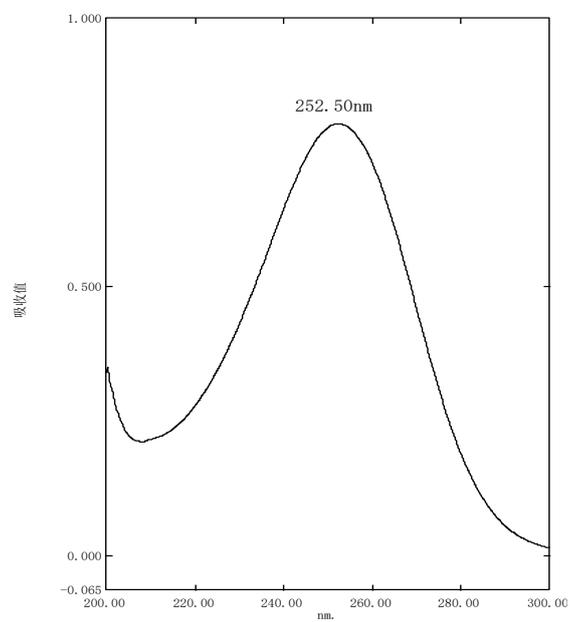


Figure S28. UV-vis spectrum of (*E*)-verbenone *O*-*n*-hexanoyl oxime **4b** in EtOH.

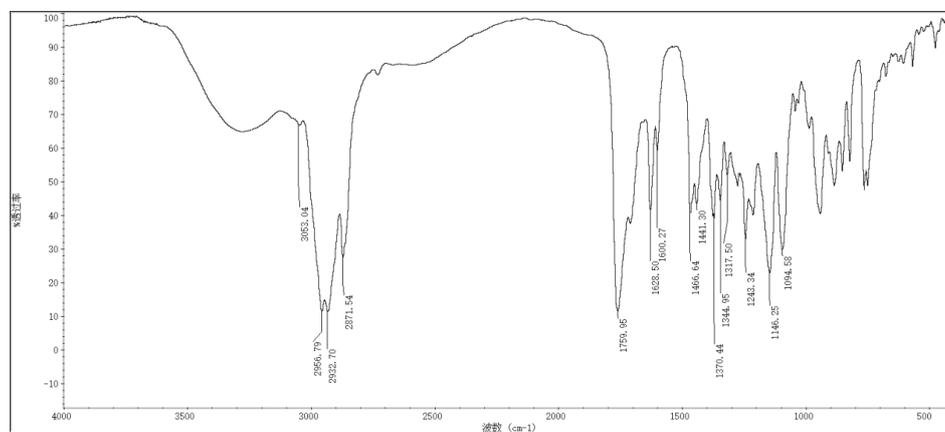


Figure S29. FTIR spectrum of (*E*)-verbenone *O*-*n*-hexanoyl oxime **4b**.

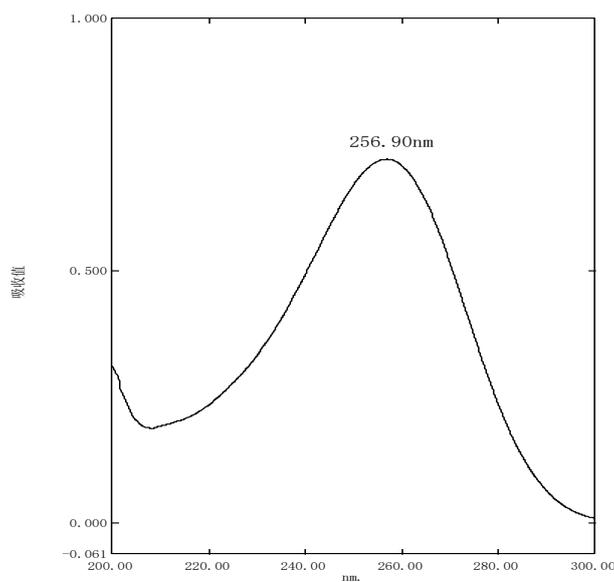


Figure S43. UV-vis spectrum of (Z)-verbenone O-cyclopentylcarbonyl oxime **4c** in EtOH.

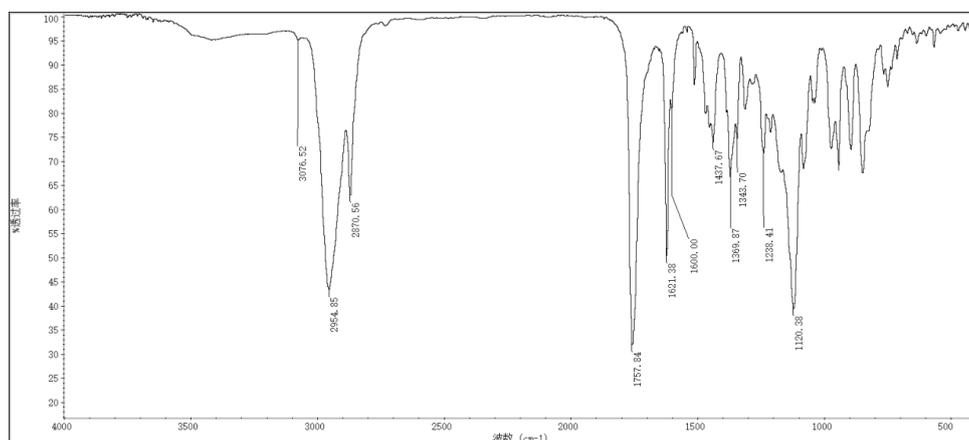


Figure S44. FTIR spectrum of (Z)-verbenone O-cyclopentylcarbonyl oxime **4c**.

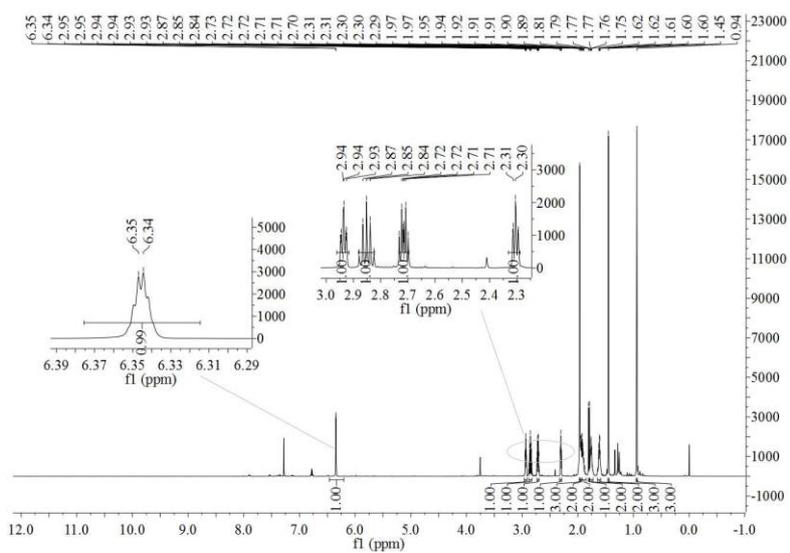


Figure S45. $^1\text{H-NMR}$ spectrum of (Z)-verbenone O-cyclopentylcarbonyl oxime **4c** in CDCl_3 .

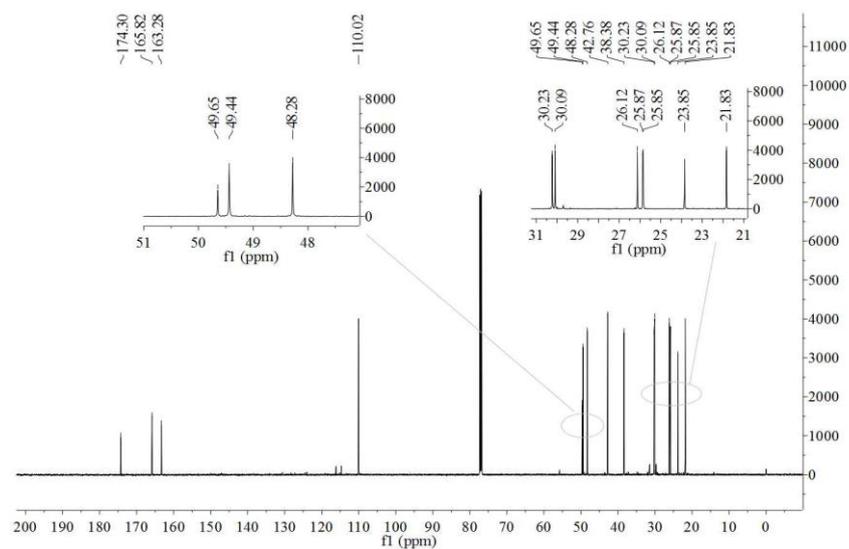


Figure S46. ^{13}C -NMR spectrum of (*Z*)-verbenone O-cyclopentylcarbonyl oxime **4c** in CDCl_3 .

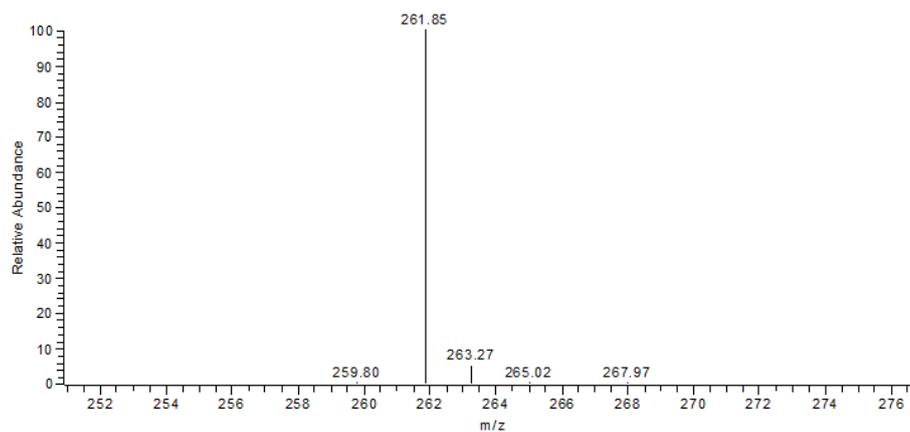


Figure S47. ESI-MS spectrum of (*Z*)-verbenone O-cyclopentylcarbonyl oxime **4c**.

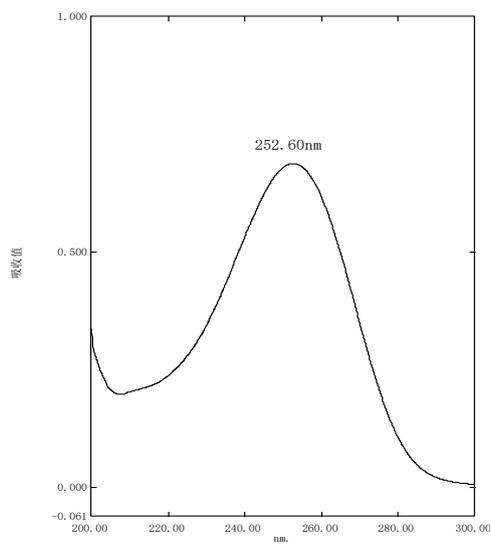


Figure S38. UV-vis spectrum of (*E*)-verbenone O-cyclopentylcarbonyl oxime **4c** in EtOH.

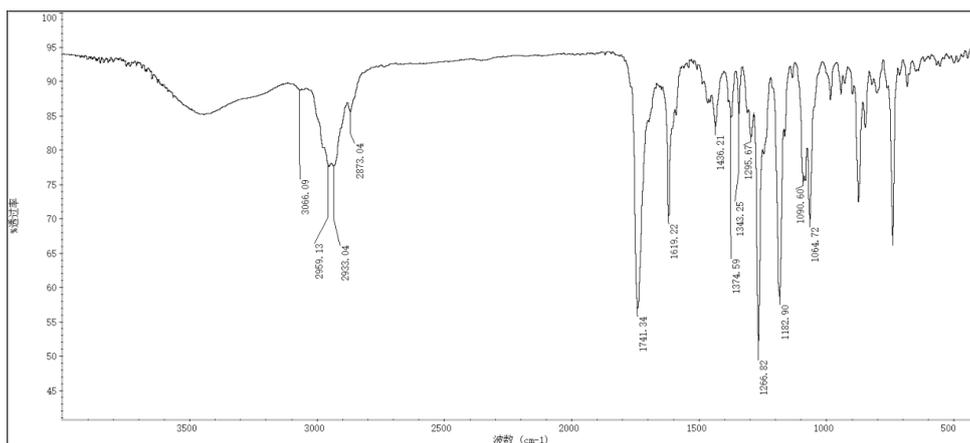


Figure S39. FTIR spectrum of (*E*)-verbenone O-cyclopentylcarbonyl oxime **4c**.

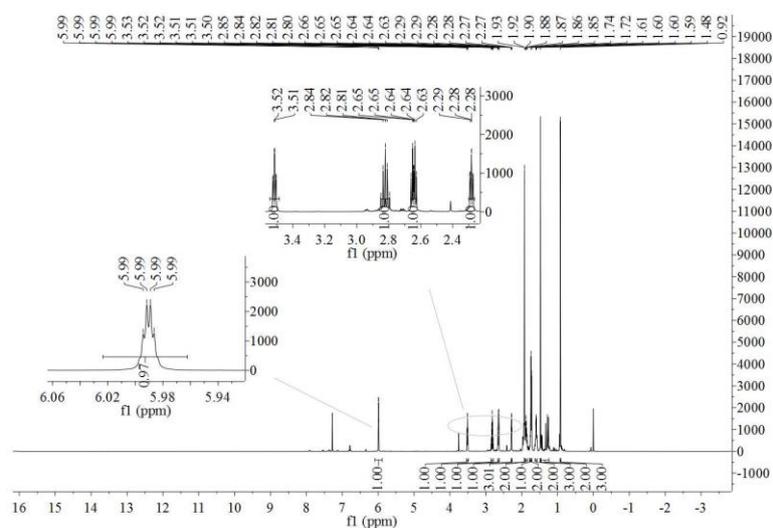


Figure S40. ¹H-NMR spectrum of (*E*)-verbenone O-cyclopentylcarbonyl oxime **4c** in CDCl₃.

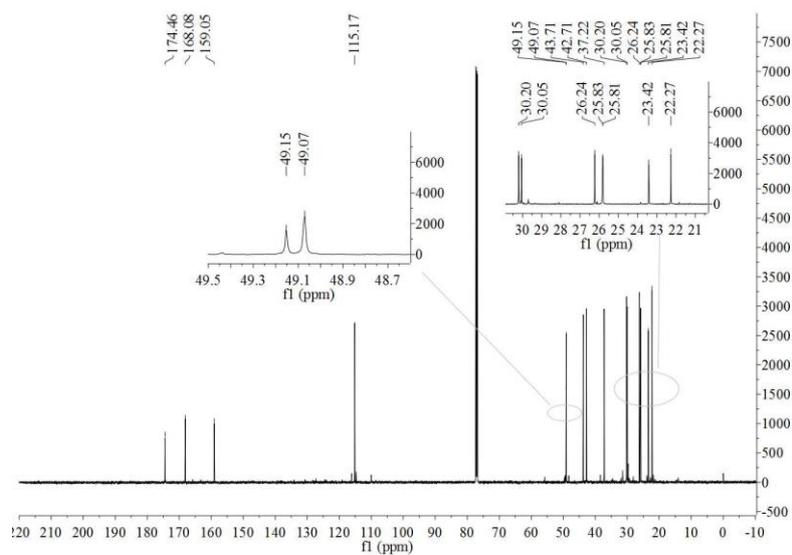


Figure S41. ¹³C-NMR spectrum of (*E*)-verbenone O-cyclopentylcarbonyl oxime **4c** in CDCl₃.

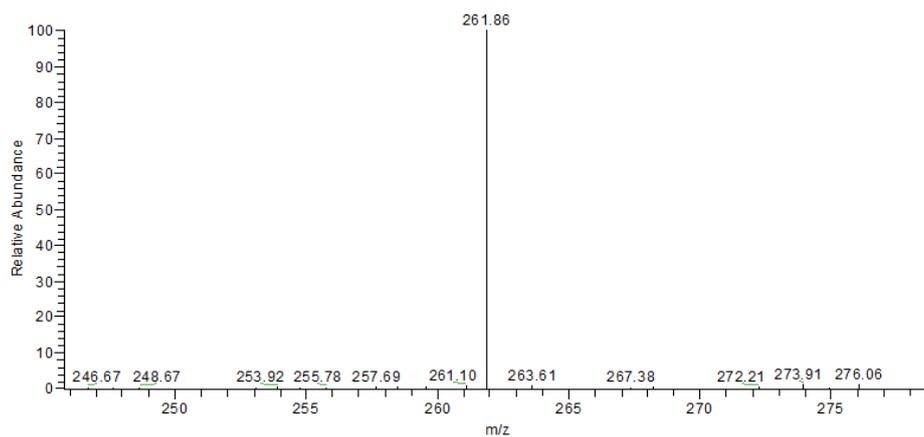


Figure S42. ESI-MS spectrum of (*E*)-verbenone O-cyclopentylcarbonyl oxime **4c**.

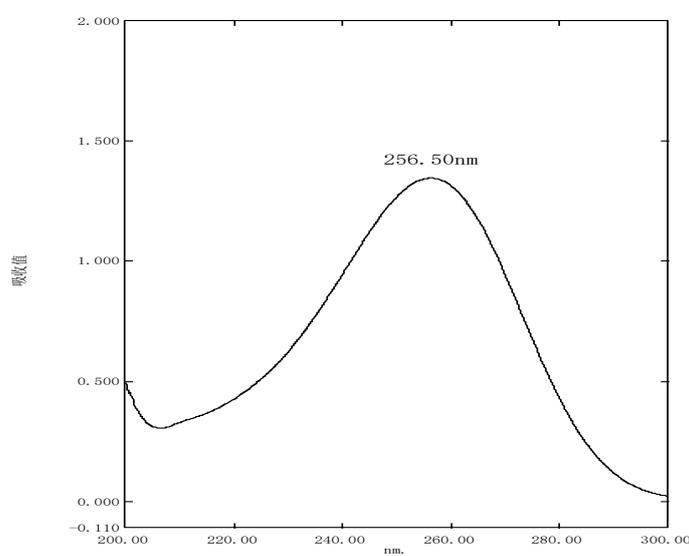


Figure S53. UV-vis spectrum of (*Z*)-verbenone O-cyclohexylcarbonyl oxime **4d** in EtOH.

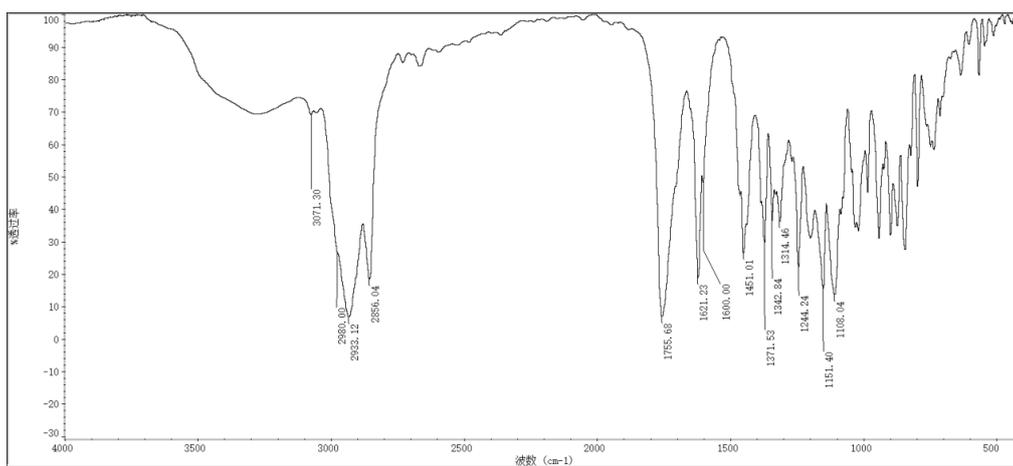


Figure S54. FTIR spectrum of (*Z*)-verbenone O-cyclohexylcarbonyl oxime **4d**.

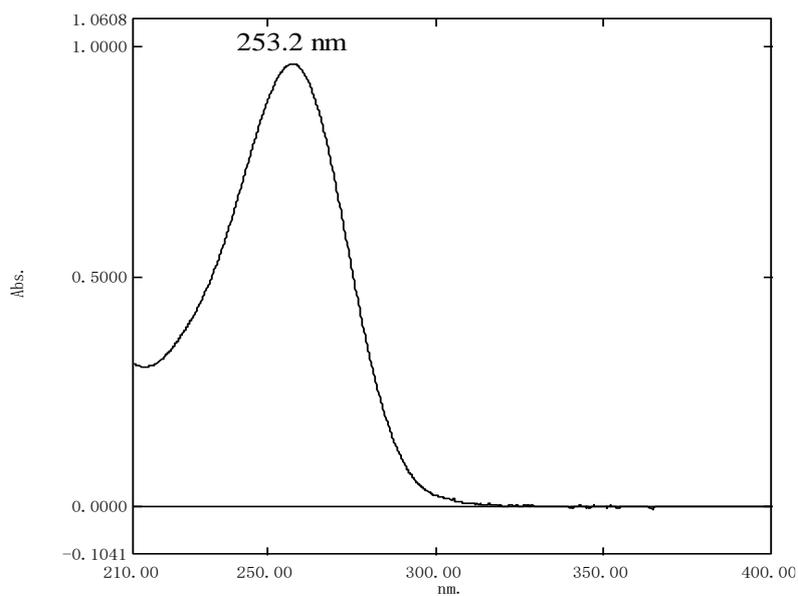


Figure S48. UV-vis spectrum of (*E*)-verbenone O-cyclohexylcarbonyl oxime **4d** in EtOH.

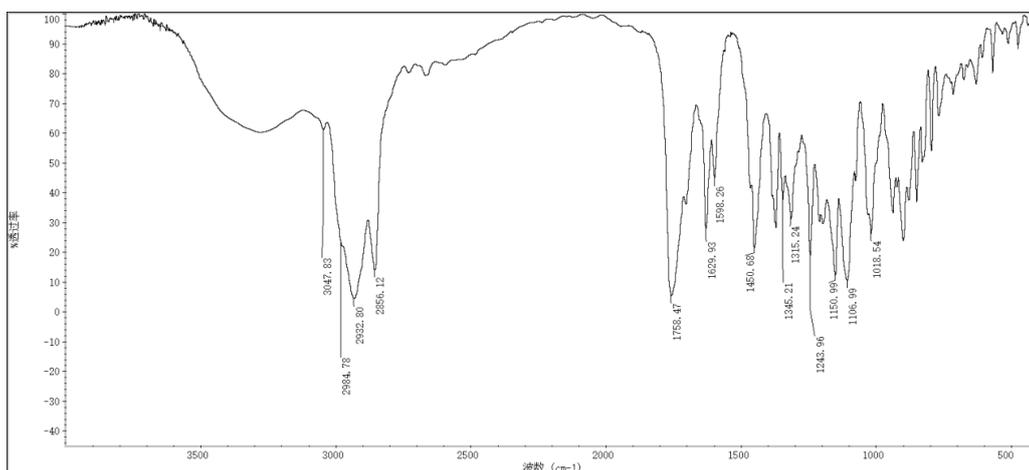


Figure S49. FTIR spectrum of (*E*)-verbenone O-cyclohexylcarbonyl oxime **4d**.

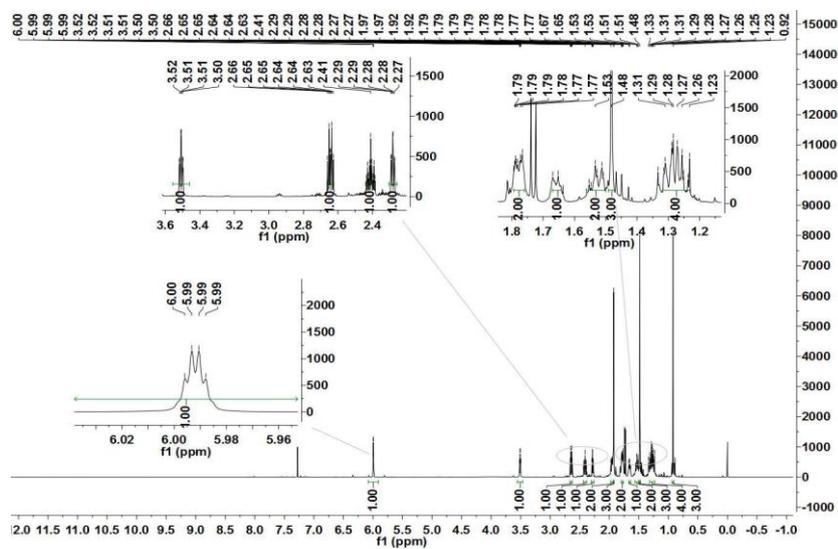


Figure S50. ¹H-NMR spectrum of (*E*)-verbenone O-cyclohexylcarbonyl oxime **4d** in CDCl₃.

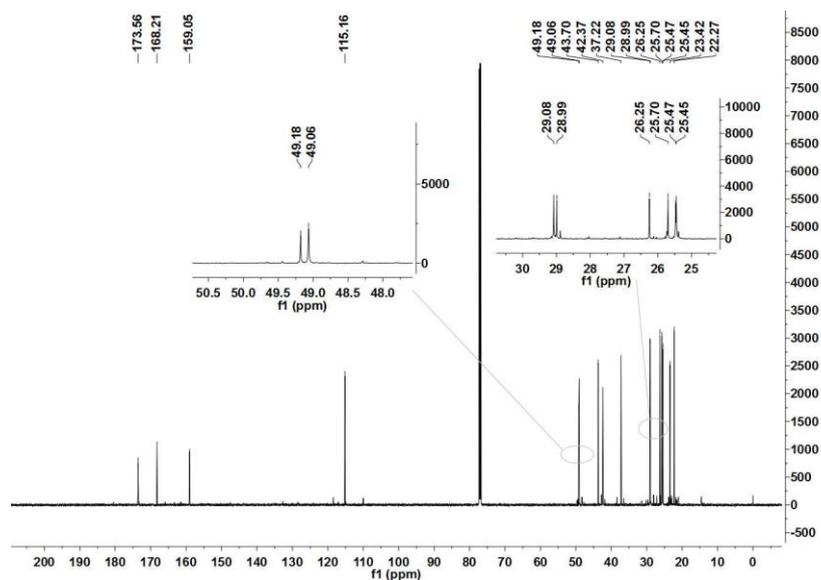


Figure S51. ^{13}C -NMR spectrum of (*E*)-verbenone O-cyclohexylcarbonyl oxime **4d** in CDCl_3 .

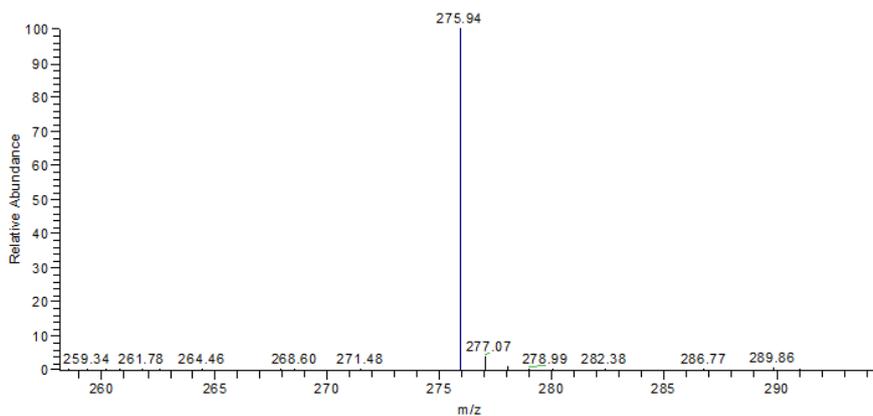


Figure S52. ESI-MS spectrum of (*E*)-verbenone O-cyclohexylcarbonyl oxime **4d**.

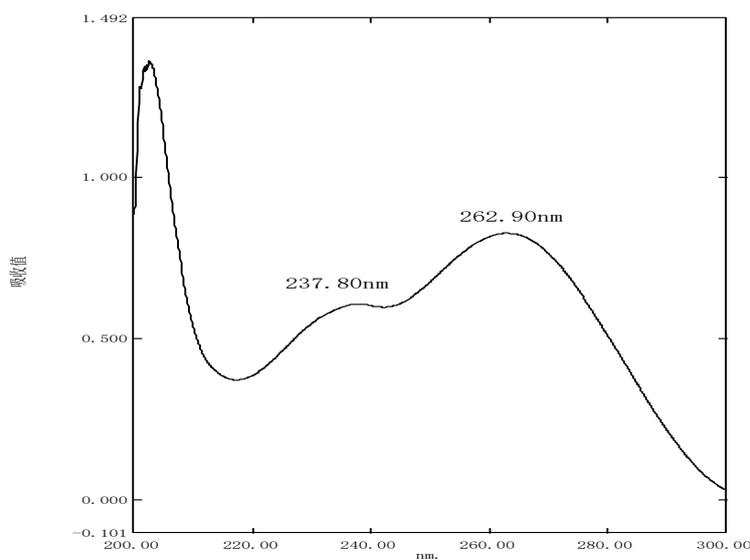


Figure S63. UV-vis spectrum of (*Z*)-verbenone O-(2'-methylbenzoyl) oxime **4e** in EtOH.

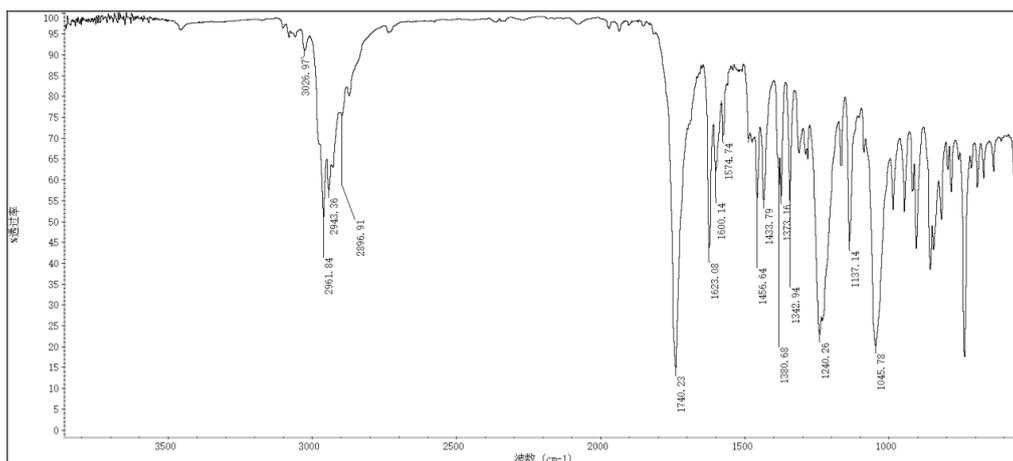


Figure S64. FTIR spectrum of (Z)-verbenone O-(2'-methylbenzoyl) oxime **4e**.

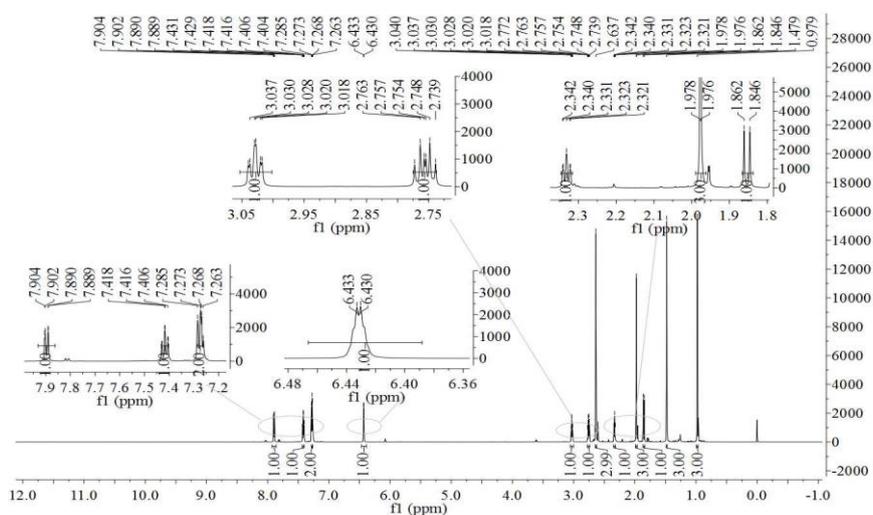


Figure S65. $^1\text{H-NMR}$ spectrum of (Z)-verbenone O-(2'-methylbenzoyl) oxime **4e** in CDCl_3 .

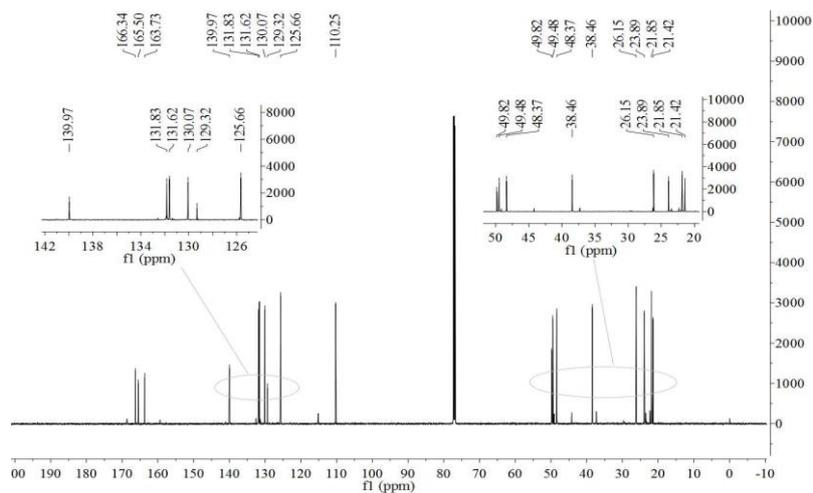


Figure S66. $^{13}\text{C-NMR}$ spectrum of (Z)-verbenone O-(2'-methylbenzoyl) oxime **4e** in CDCl_3 .

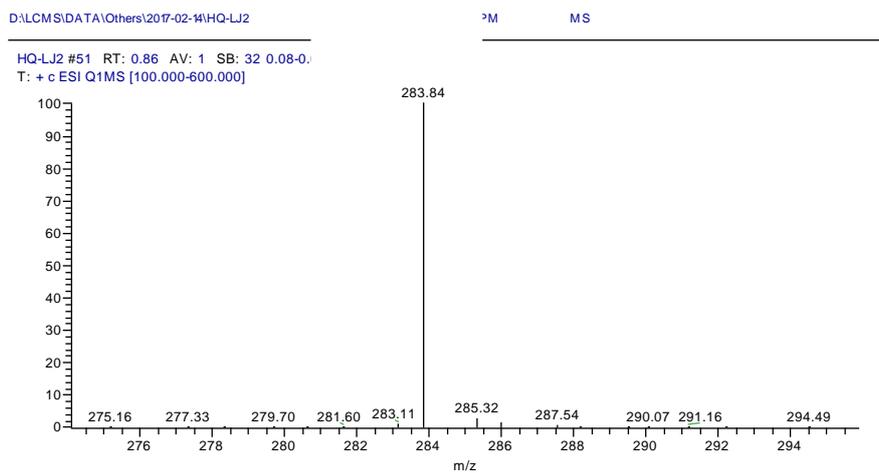


Figure S67. ESI-MS spectrum of (*Z*)-verbenone O-(2'-methylbenzoyl) oxime **4e**.

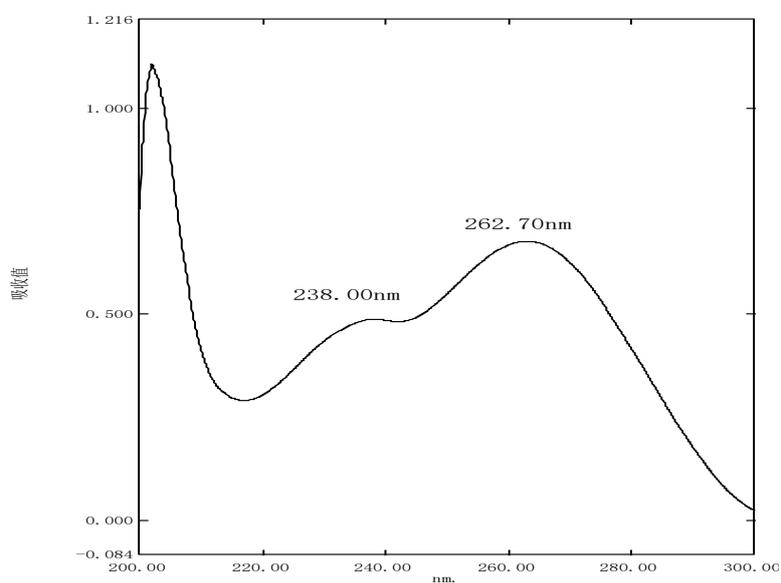


Figure S58. UV-vis spectrum of (*E*)-verbenone O-(2'-methylbenzoyl) oxime **4e** in EtOH.

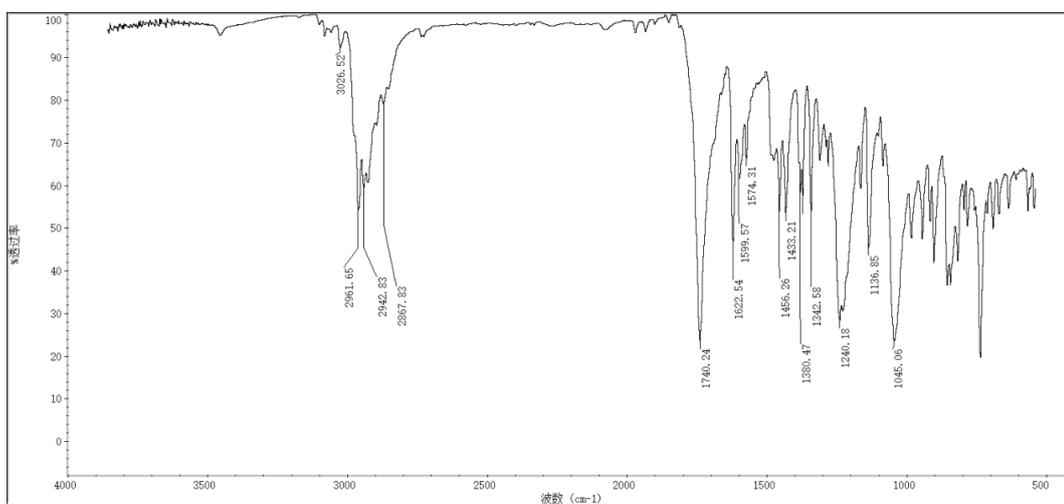


Figure S59. FTIR spectrum of (*E*)-verbenone O-(2'-methylbenzoyl) oxime **4e**.

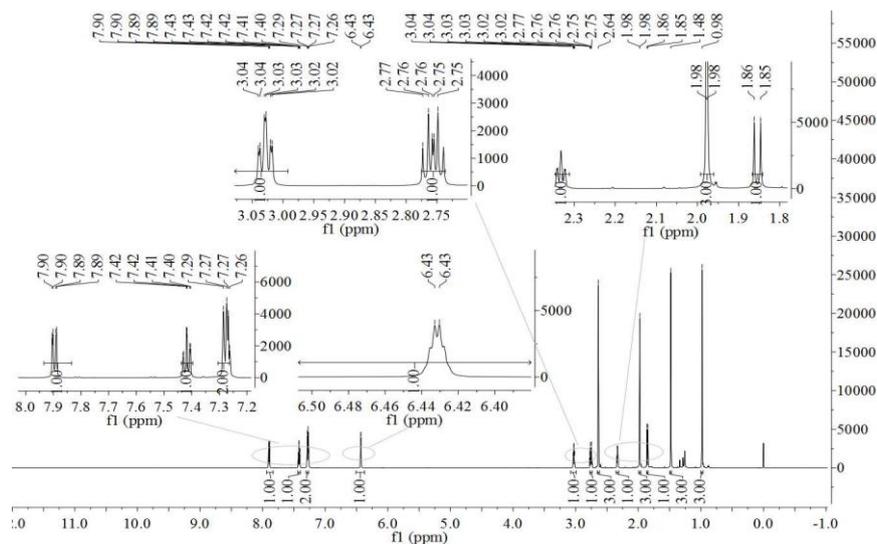


Figure S60. $^1\text{H-NMR}$ spectrum of *(E)*-verbenone O-(2'-methylbenzoyl) oxime **4e** in CDCl_3 .

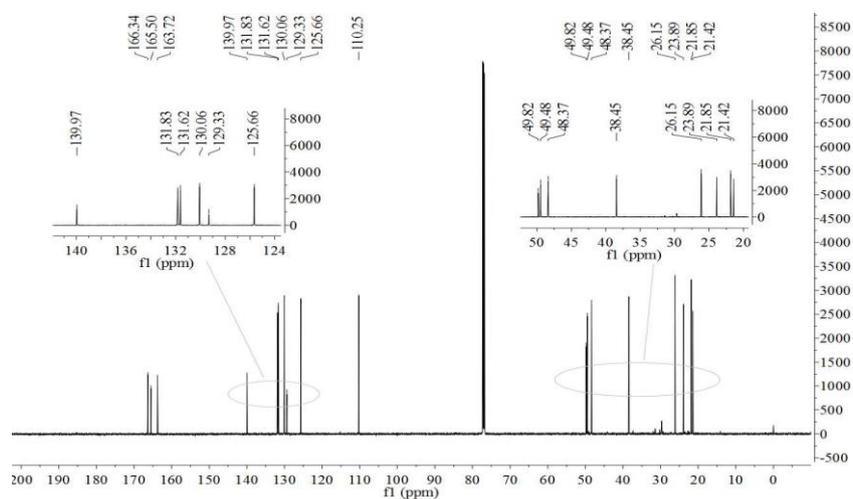


Figure S61. $^{13}\text{C-NMR}$ spectrum of *(E)*-verbenone O-(2'-methylbenzoyl) oxime **4e** in CDCl_3 .

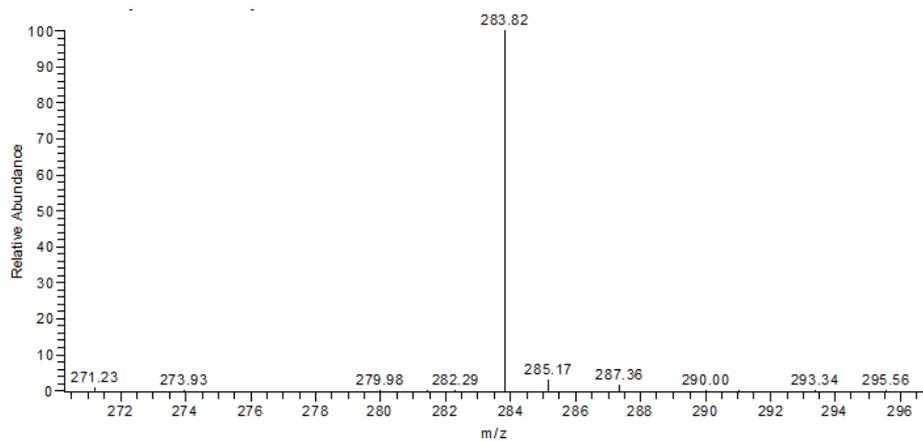


Figure S62. ESI-MS spectrum of *(E)*-verbenone O-(2'-methylbenzoyl) oxime **4e**.

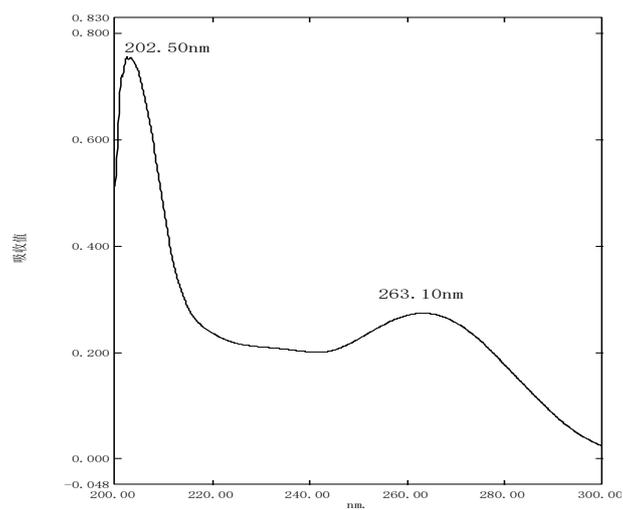


Figure S73. UV-vis spectrum of (Z)-verbenone O-(2'-chlorobenzoyl) oxime **4f** in EtOH.

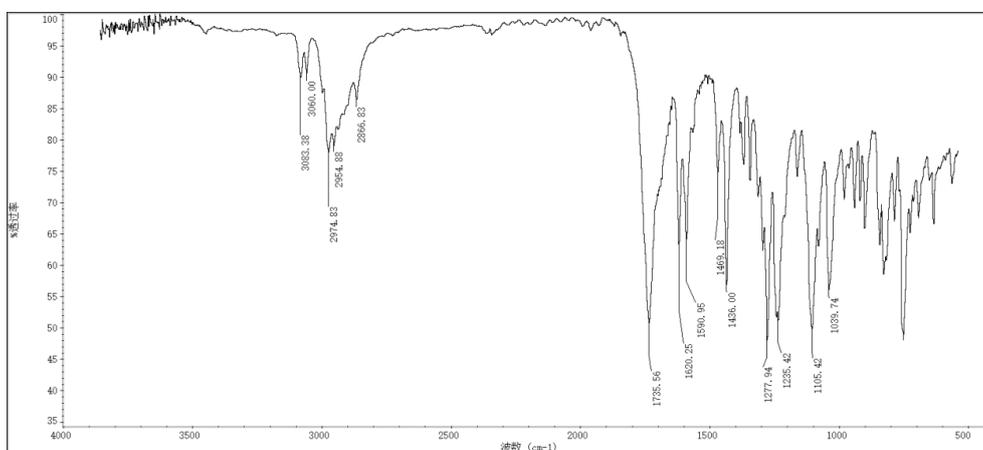


Figure S74. FTIR spectrum of (Z)-verbenone O-(2'-chlorobenzoyl) oxime **4f**.

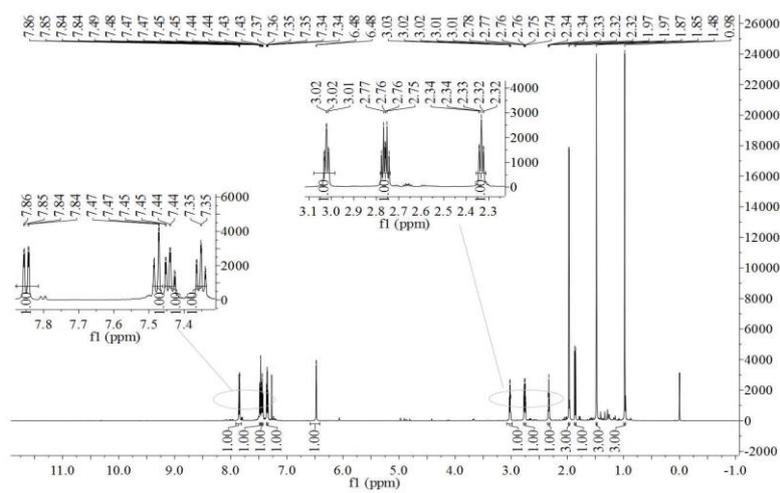


Figure S75. $^1\text{H-NMR}$ spectrum of (Z)-verbenone O-(2'-chlorobenzoyl) oxime **4f** in CDCl_3 .

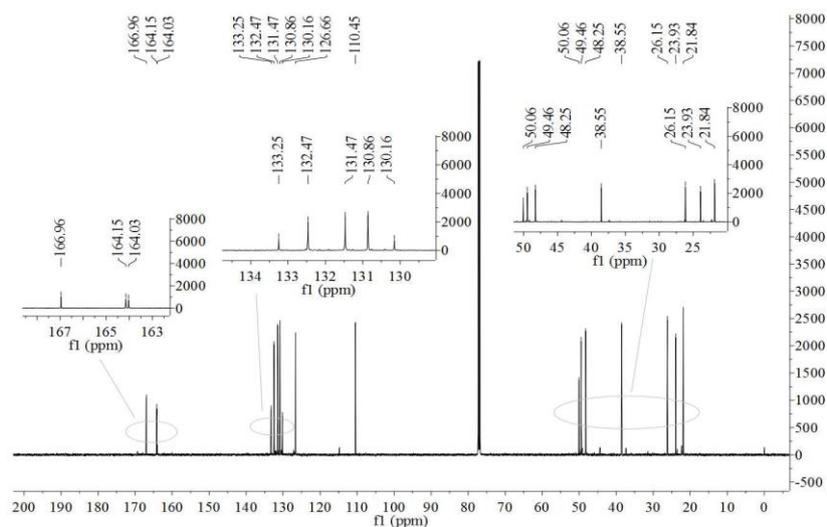


Figure S76. ¹³C-NMR spectrum of (Z)-verbenone O-(2'-chlorobenzoyl) oxime in CDCl₃.

D:\LCMS\DATA\Others\2017-02-14\HQ-LL2

3/22/2017 4:24:01PM

MS

HQ-LL2 #30-36 RT: 0.50-0.60 AV: 7 SB: 14

T: + c ESI Q1MS [100.000-600.000]

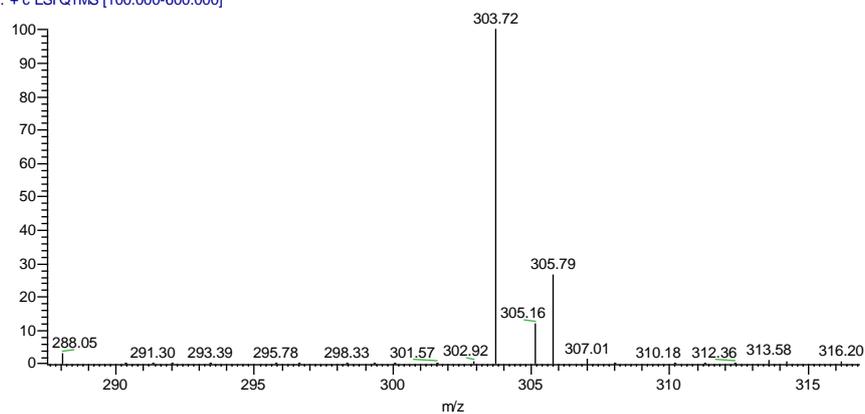


Figure S77. ESI-MS spectrum of (Z)-verbenone O-(2'-chlorobenzoyl) oxime 4f.

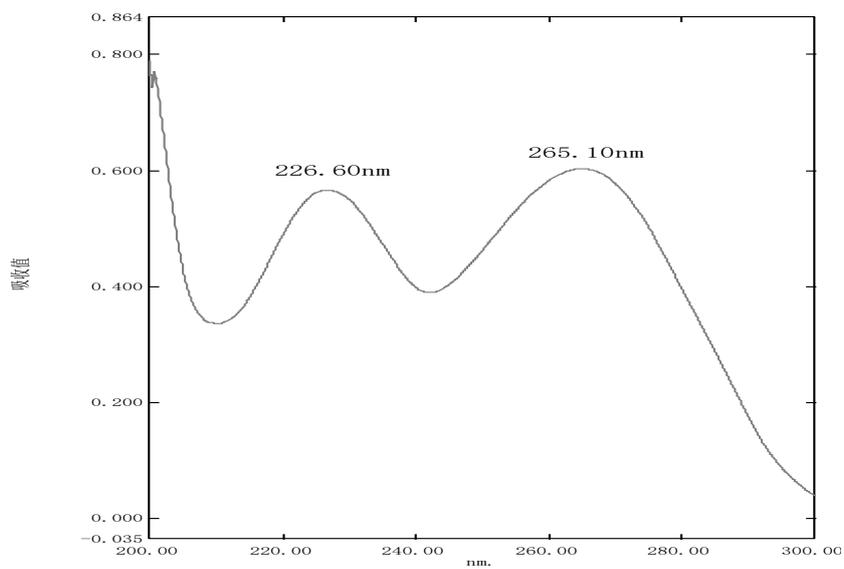


Figure S83. UV-vis spectrum of (Z)-verbenone O-(2'-fluorobenzoyl) oxime 4g in EtOH.

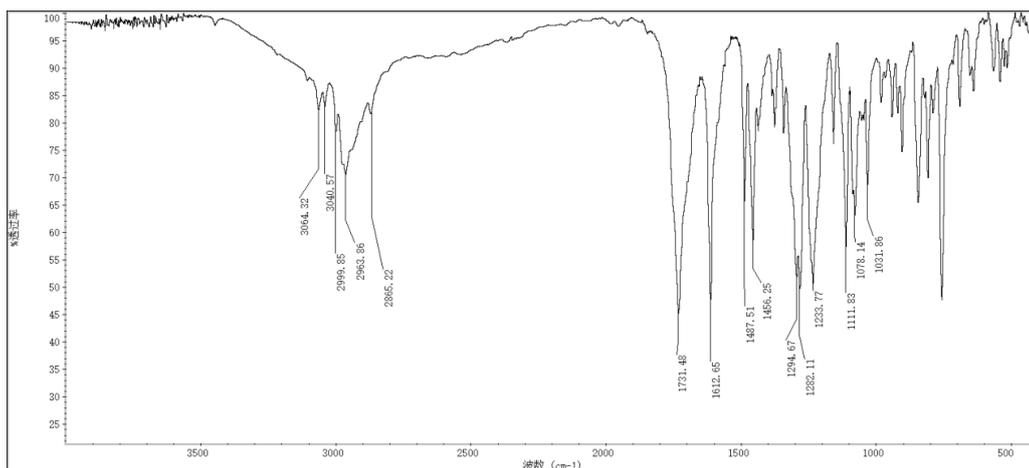


Figure S84. FTIR spectrum of (*Z*)-verbenone O-(2'-fluorobenzoyl) oxime **4g**.

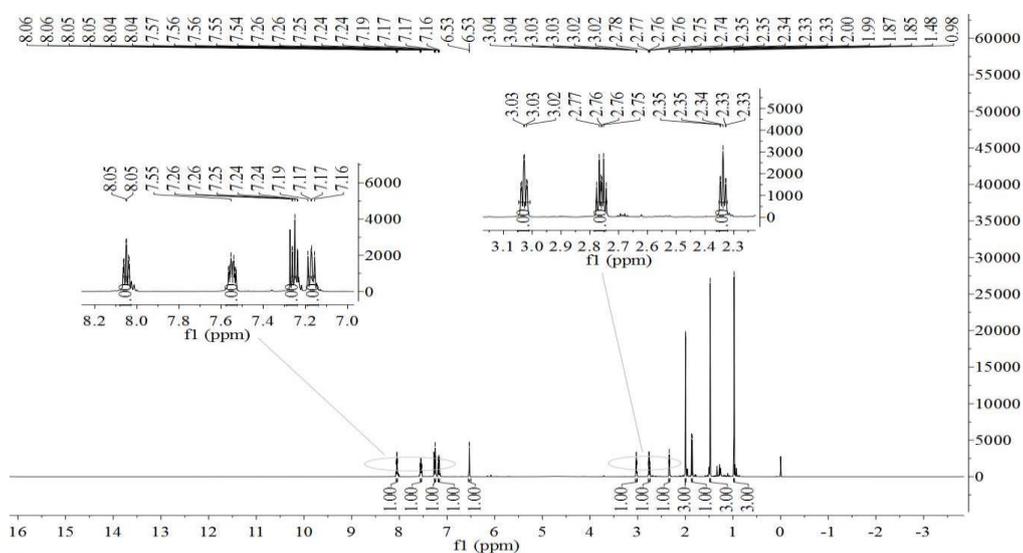


Figure S85. ¹H-NMR spectrum of (*Z*)-verbenone O-(2'-fluorobenzoyl) oxime **4g** in CDCl₃.

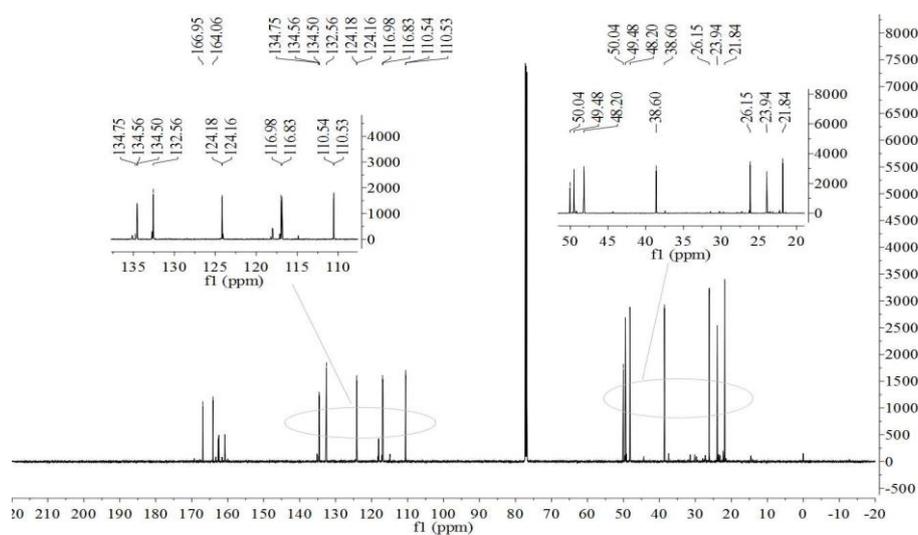


Figure S86. ¹³C-NMR spectrum of (*Z*)-verbenone O-(2'-fluorobenzoyl) oxime **4g** in CDCl₃.

HQ-LF1 #25 RT: 0.42 AV: 1 SB: 13 0.03-0.2
T: + c ESI Q1 MS [100.000-600.000]

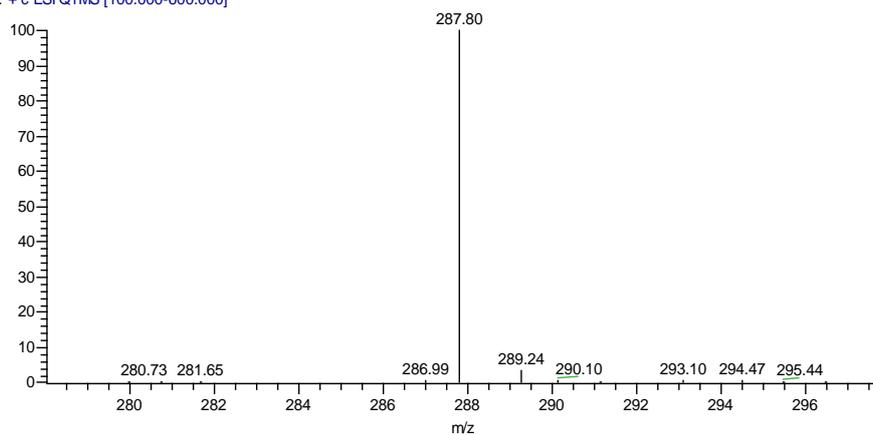


Figure S87. ESI-MS spectrum of (*Z*)-verbenone O-(2'-fluorobenzoyl) oxime **4g**.

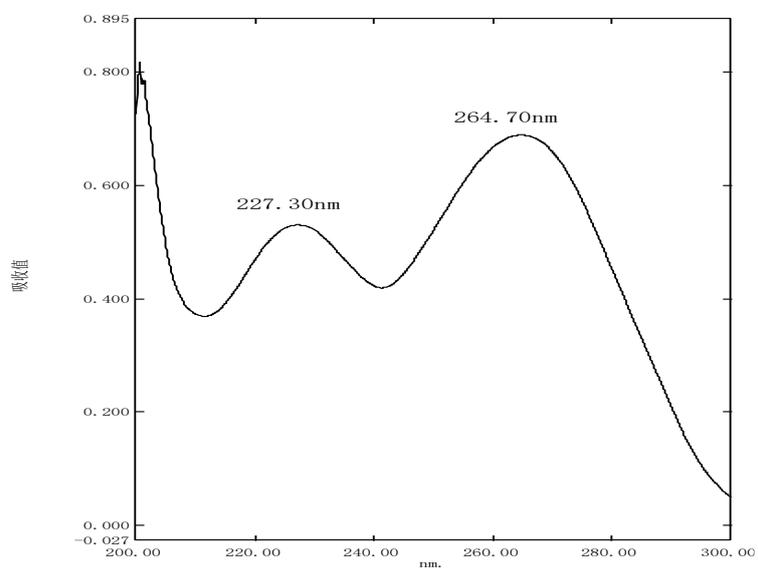


Figure S78. UV-vis spectrum of (*E*)-verbenone O-(2'-fluorobenzoyl) oxime **4g** in EtOH.

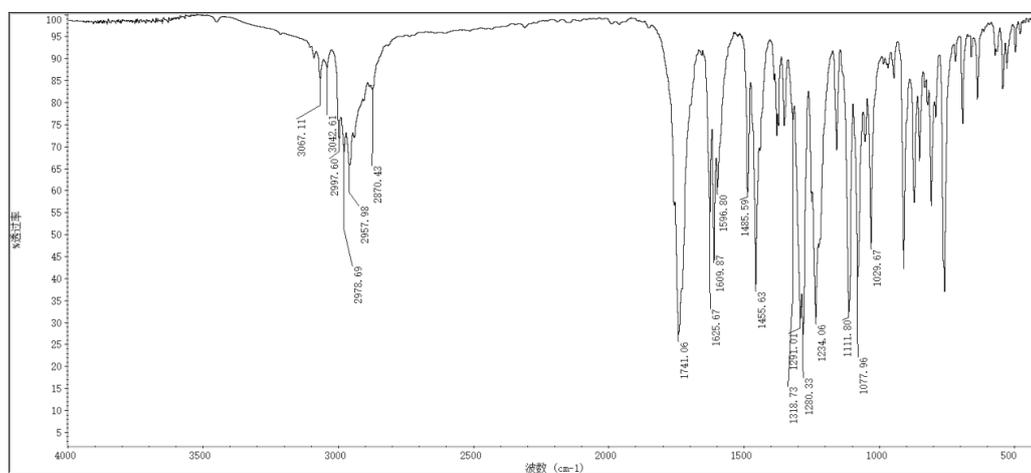


Figure S79. FTIR spectrum of (*E*)-verbenone O-(2'-fluorobenzoyl) oxime **4g**.

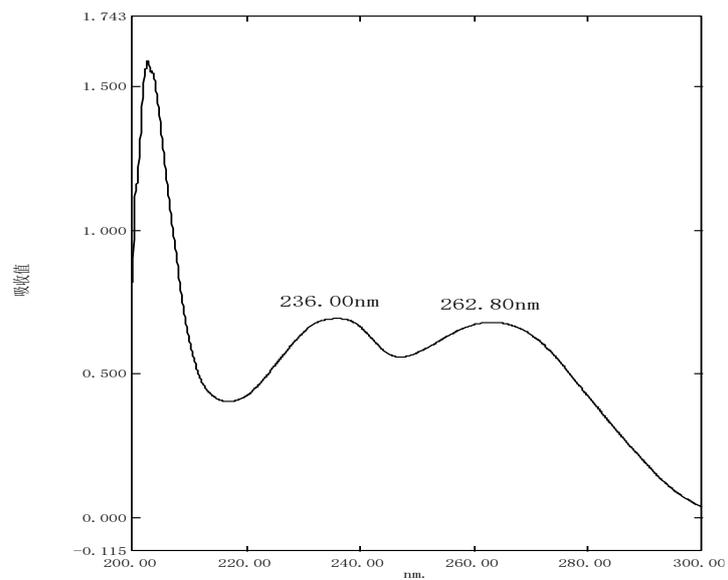


Figure S93. UV-vis spectrum of (Z)-verbenone O-(2'-methylbenzoyl) oxime **4h** in EtOH.

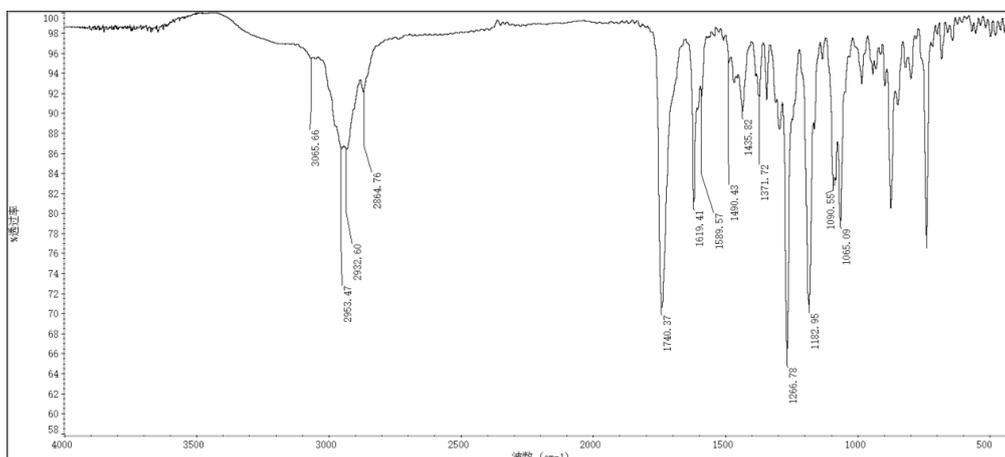


Figure S94. FTIR spectrum of (Z)-verbenone O-(3'-methylbenzoyl) oxime **4h**.

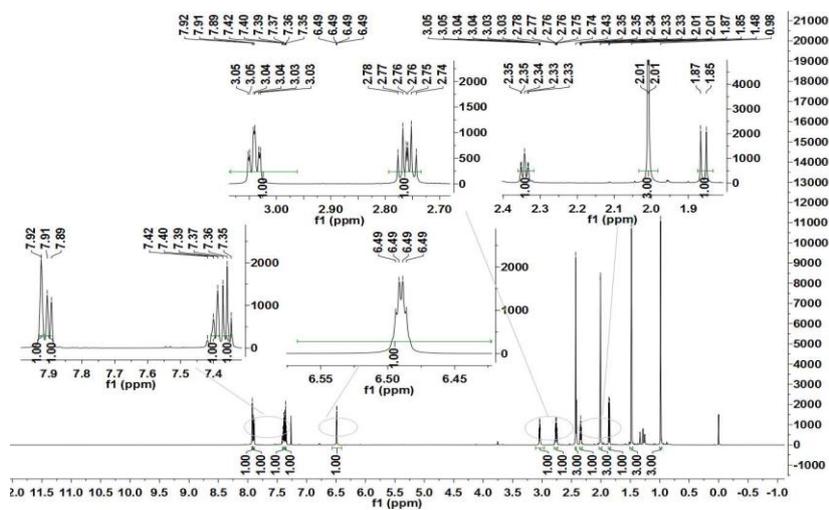


Figure S95. $^1\text{H-NMR}$ spectrum of (Z)-verbenone O-(3'-methylbenzoyl) oxime **4h** in CDCl_3 .

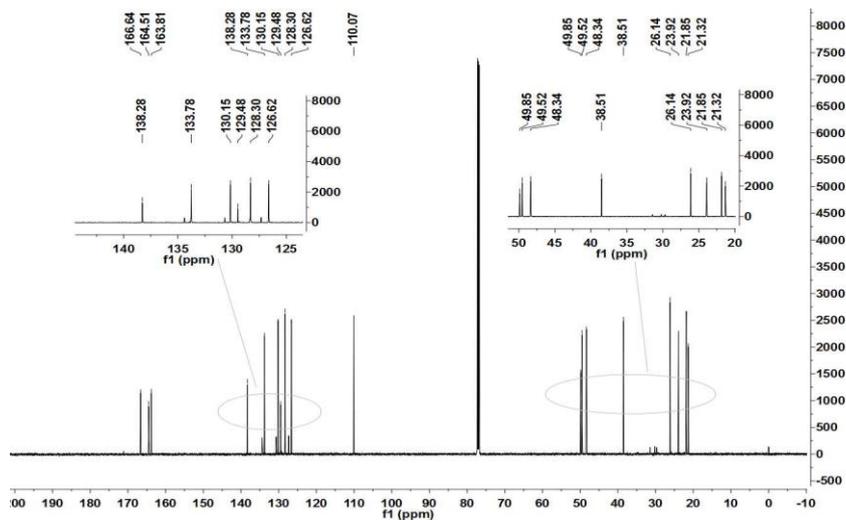


Figure S96. ¹³C-NMR spectrum of (Z)-verbenone O-(3'-methylbenzoyl) oxime **4h** in CDCl₃.

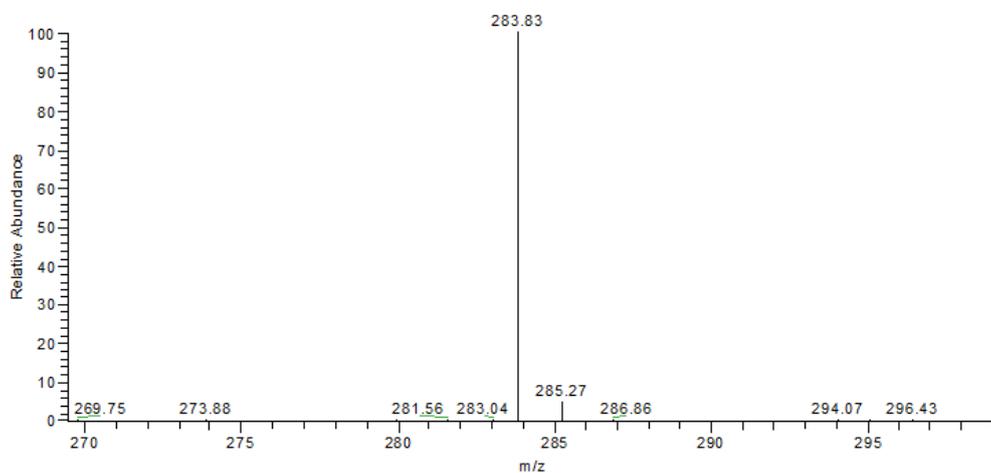


Figure S97. ESI-MS spectrum of (Z)-verbenone O-(3'-methylbenzoyl) oxime **4h**.

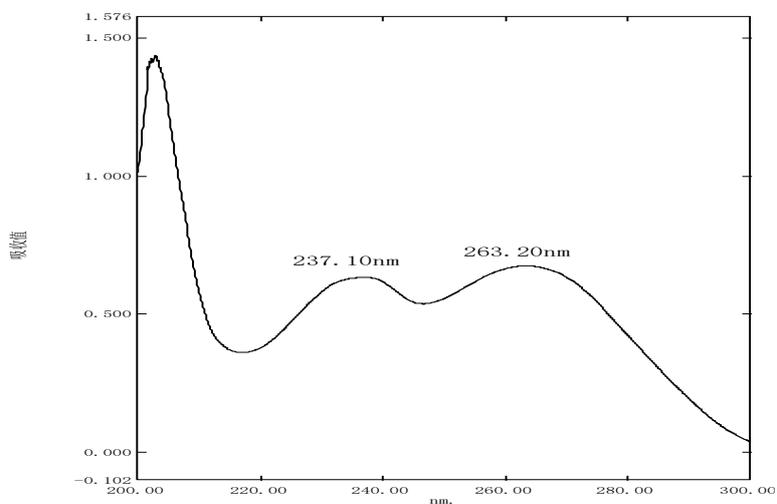


Figure S88. UV-vis spectrum of (E)-verbenone O-(3'-methylbenzoyl) oxime **4h** in EtOH.

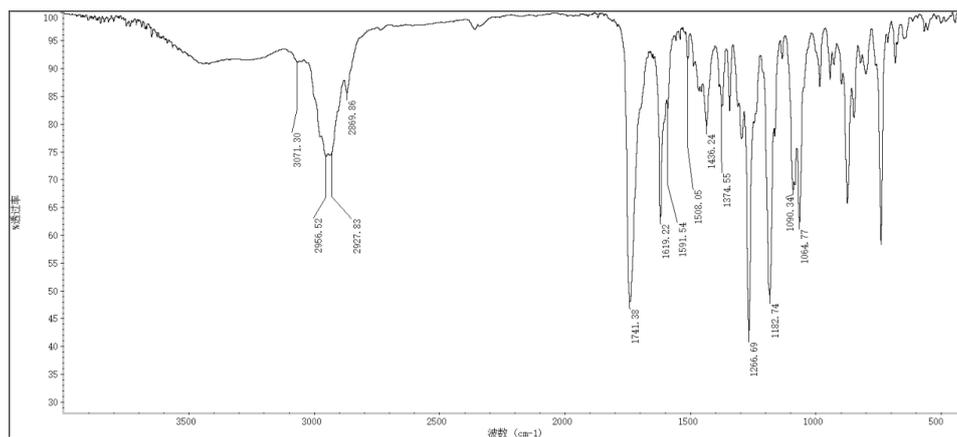


Figure S89. FTIR spectrum of (*E*)-verbenone O-(3'-methylbenzoyl) oxime **4h**.

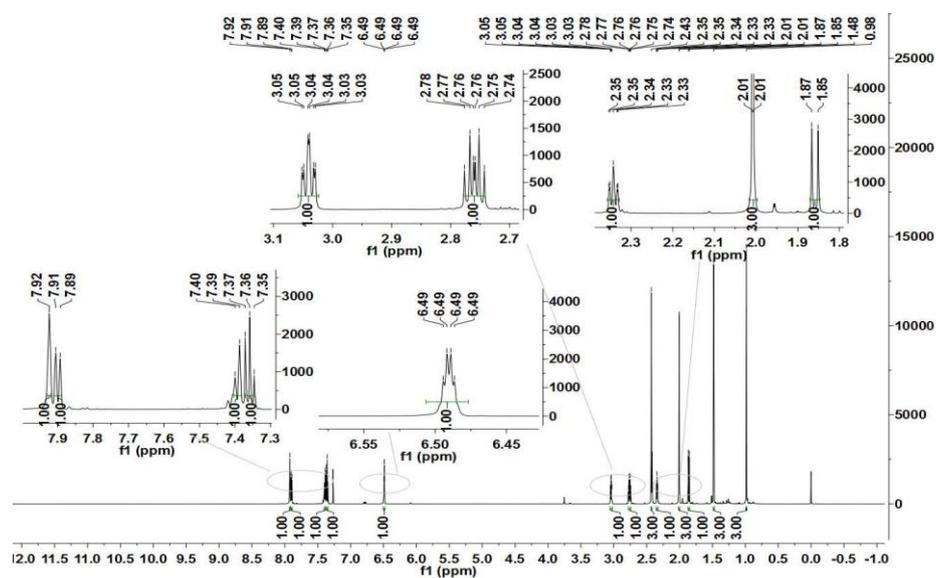


Figure S90. ¹H-NMR spectrum of (*E*)-verbenone O-(3'-methylbenzoyl) oxime **4h** in CDCl₃.

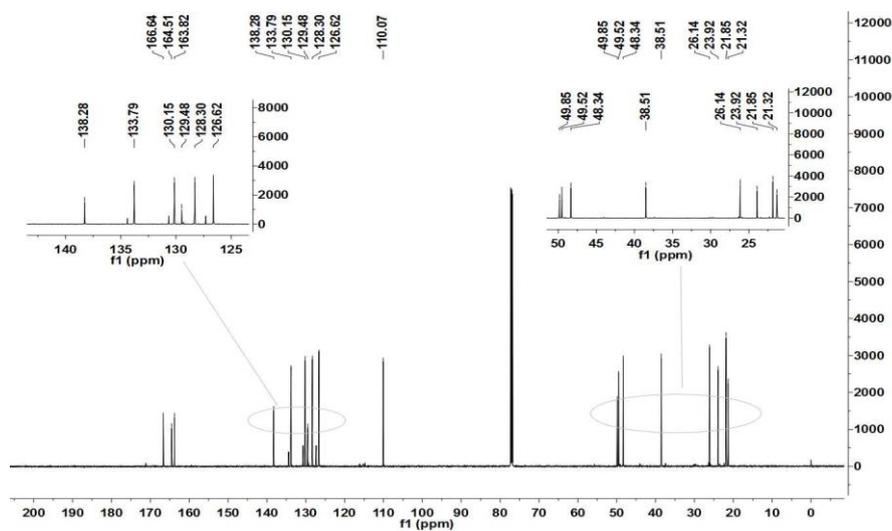


Figure S91. ¹³C-NMR spectrum of (*E*)-verbenone O-(3'-methylbenzoyl) oxime **4h** in CDCl₃.

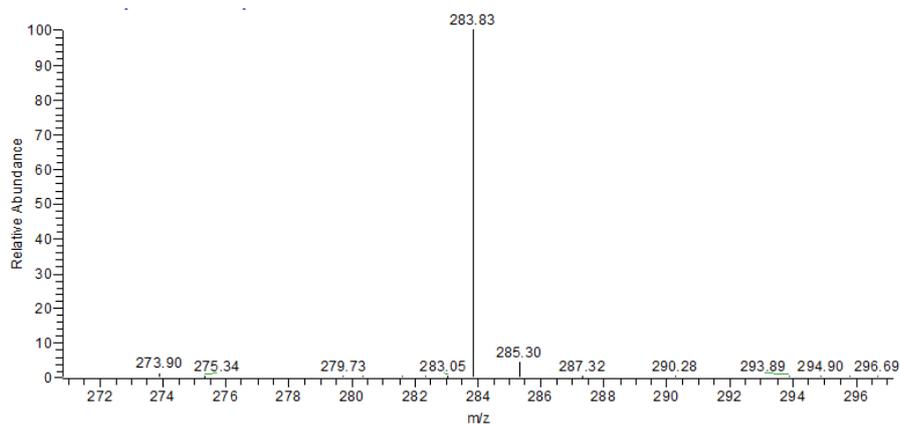


Figure S92. ESI-MS spectrum of (*E*)-verbenone O-(3'-methylbenzoyl) oxime **4h**.

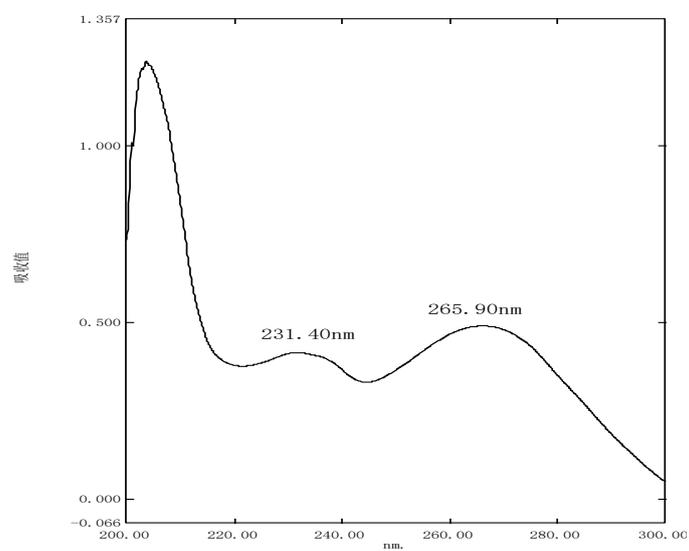


Figure S103. UV-vis spectrum of (*Z*)-verbenone O-(3'-chlorobenzoyl) oxime **4i** in EtOH.

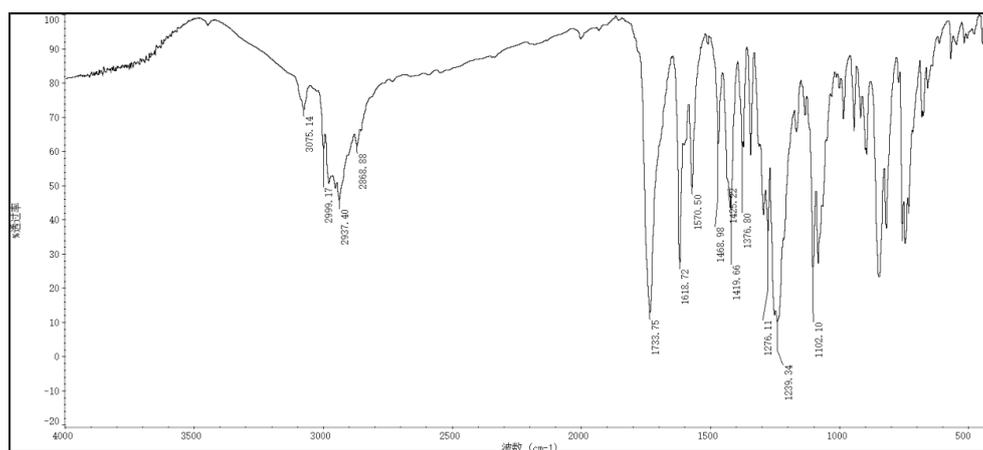


Figure S104. FTIR spectrum of (*Z*)-verbenone O-(3'-chlorobenzoyl) oxime **4i**.

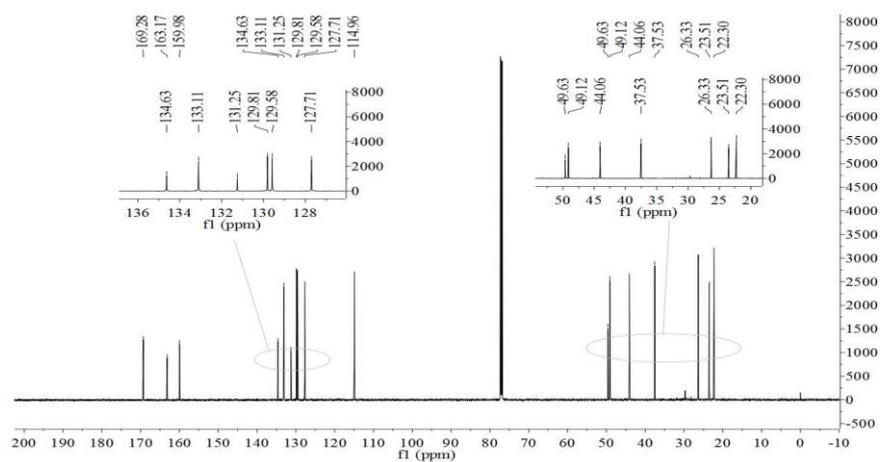


Figure S101. ^{13}C -NMR spectrum of *(E)*-verbenone O-(3'- chlorobenzoyl) oxime **4i** in CDCl_3 .

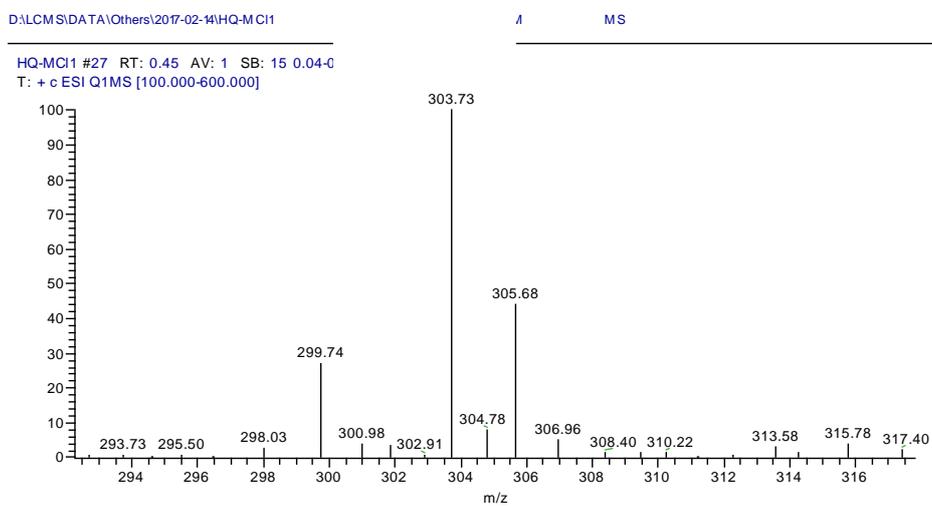


Figure S102. ESI-MS spectrum of *(E)*-verbenone O-(3'- chlorobenzoyl) oxime **4i**.

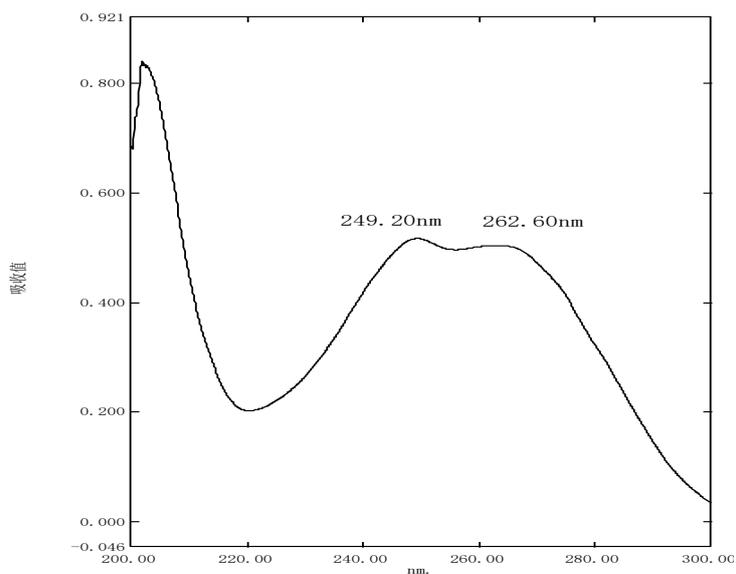


Figure S108. UV-vis spectrum of *(Z)*-verbenone O-(4'-bromobenzoyl) oxime **4j** in EtOH .

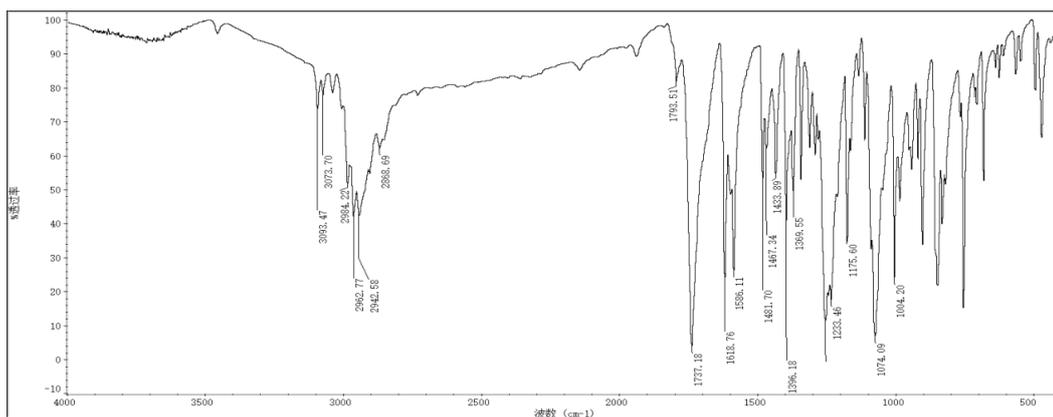


Figure S109. FTIR spectrum of (Z)-verbenone O-(4'-bromobenzoyl) oxime **4j**.

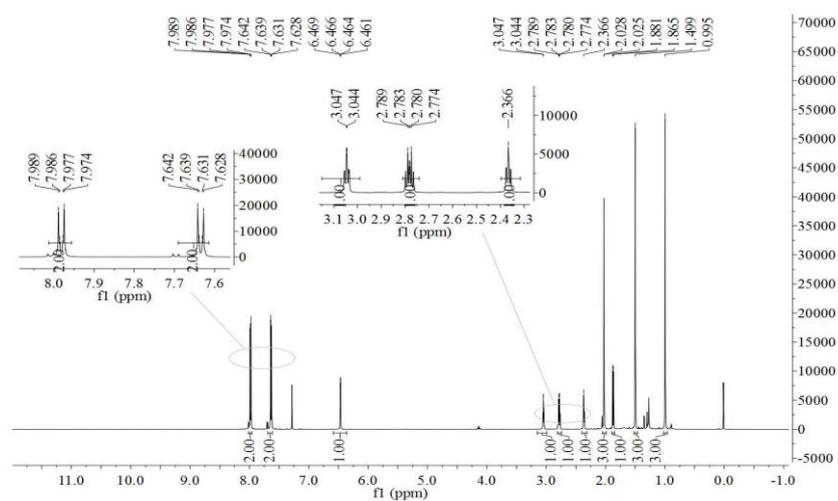


Figure S110. ¹H-NMR spectrum of (Z)-verbenone O-(4'-bromobenzoyl) oxime **4j** in CDCl₃.

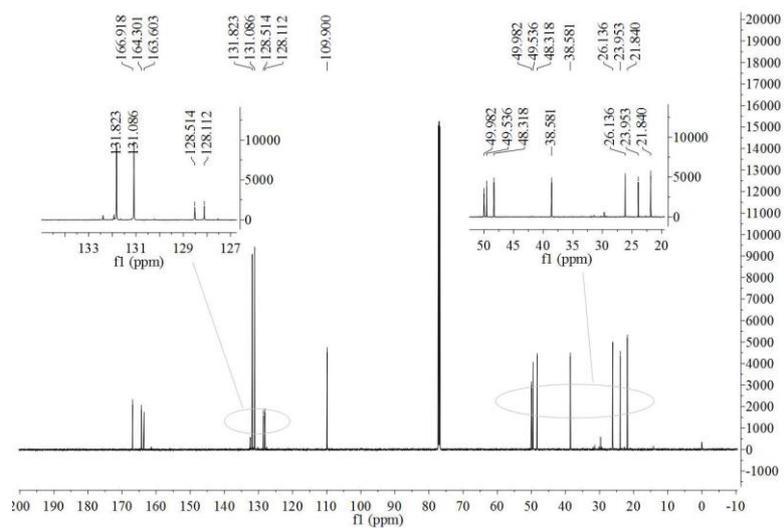


Figure S111. ¹³C-NMR spectrum of (Z)-verbenone O-(4'-bromobenzoyl) oxime **4j** in CDCl₃.

HQ-4Br2 #37 RT: 0.63 AV: 1 SB: 17 0.05-0.
T: + c ESI Q1MS [100.000-600.000]

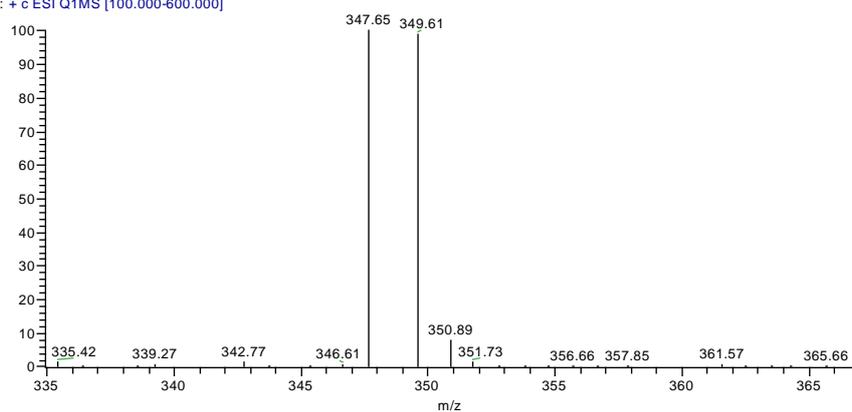


Figure S112. ESI-MS spectrum of (Z)-verbenone O-(4'-bromobenzoyl) oxime 4j.

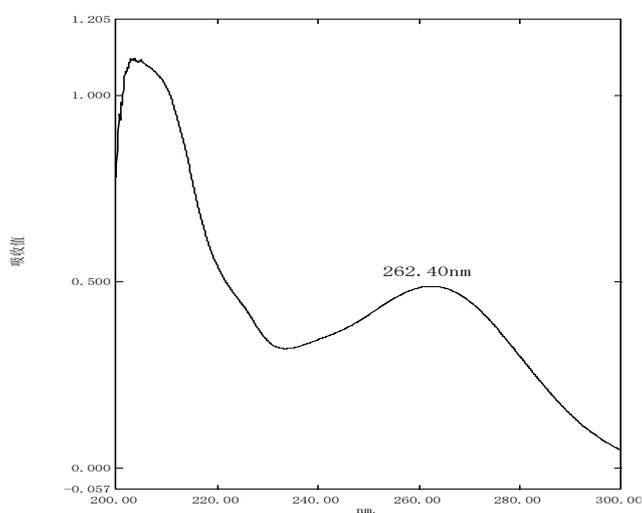


Figure S118. UV-vis spectrum of (Z)-verbenone O-(2', 3'-dichlorobenzoyl) oxime 4k in EtOH.

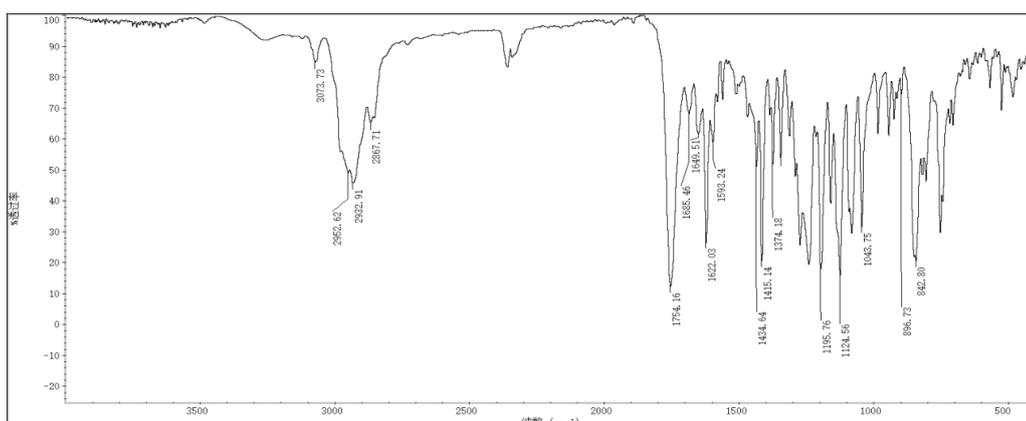


Figure S119. FTIR spectrum of (Z)-verbenone O-(2', 3'-dichlorobenzoyl) oxime 4k.

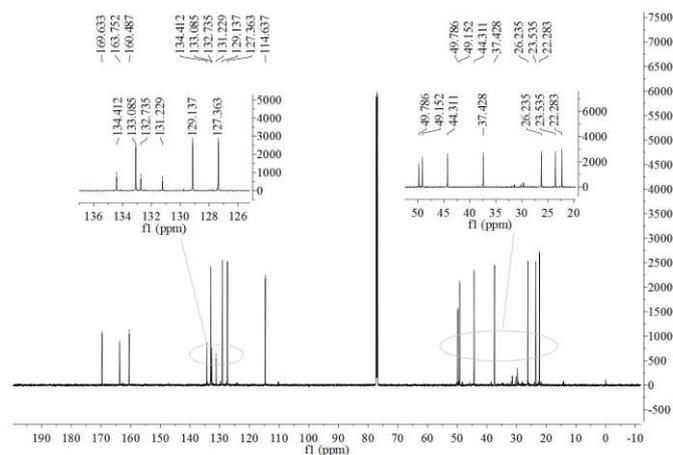


Figure S116. ^{13}C -NMR spectrum of (*E*)-verbenone O-(2', 3'-dichlorobenzoyl) oxime **4k** in CDCl_3 .

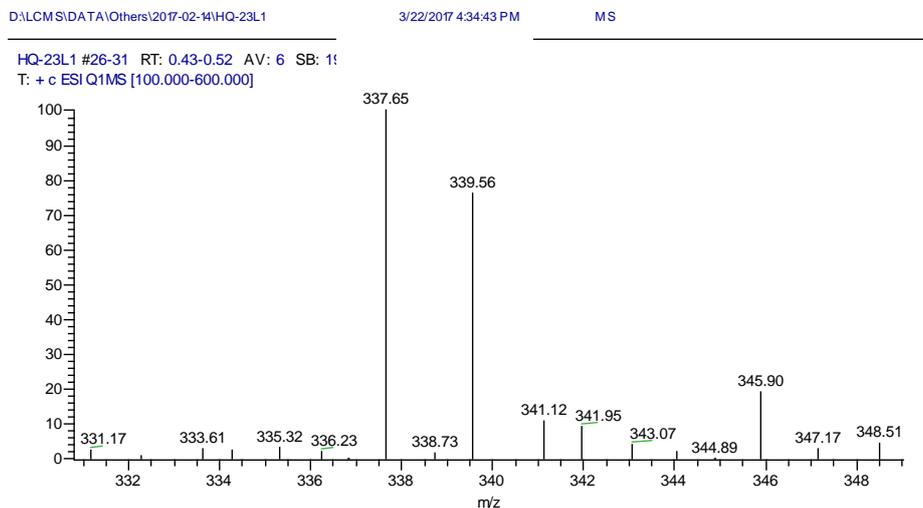


Figure S117. ESI-MS spectrum of (*E*)-verbenone O-(2', 3'-dichlorobenzoyl) oxime **4k**.

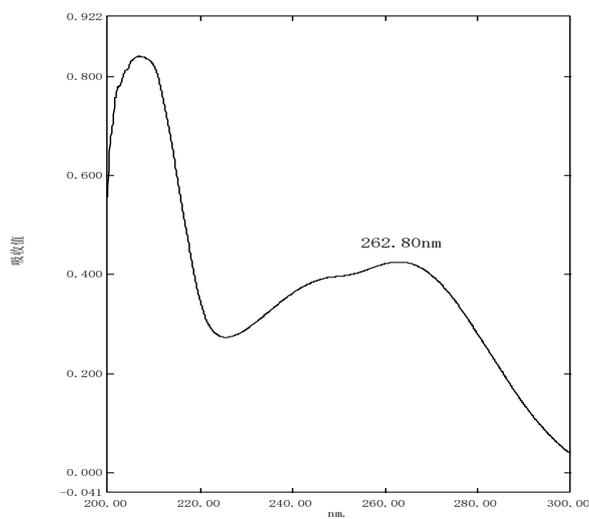


Figure S128. UV-vis spectrum of (*Z*)-verbenone O-(2', 4'-dichlorobenzoyl) oxime **4l** in EtOH.

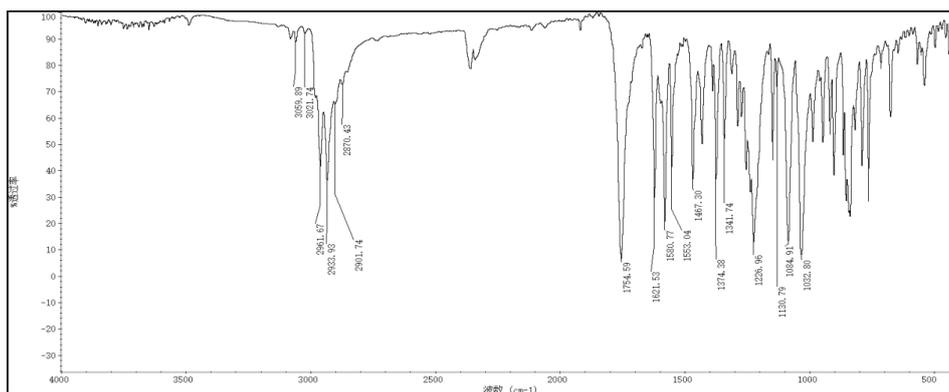


Figure S129. FTIR spectrum of (Z)-verbenone O-(2', 4'-dichlorobenzoyl) oxime **4l**.

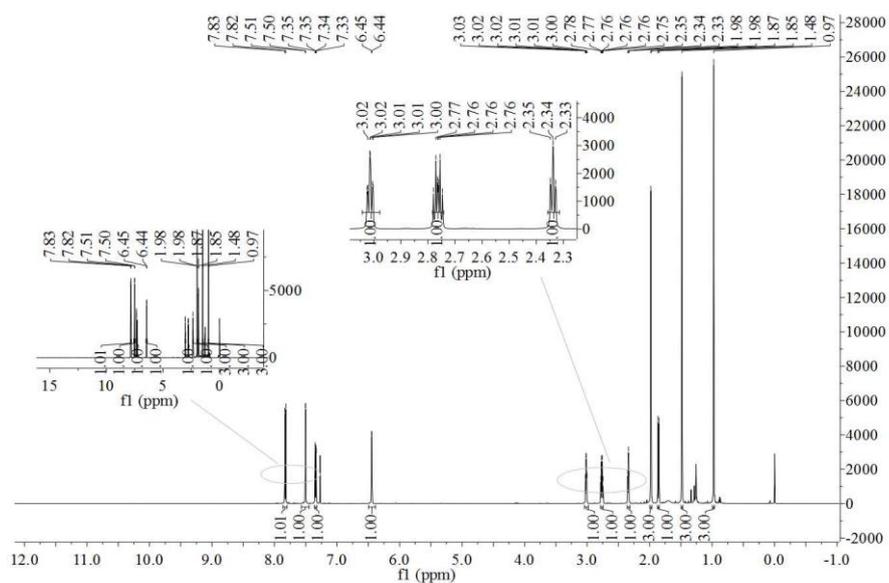


Figure S130. $^1\text{H-NMR}$ spectrum of (Z)-verbenone O-(2', 4'-dichlorobenzoyl) oxime **4l** in CDCl_3 .

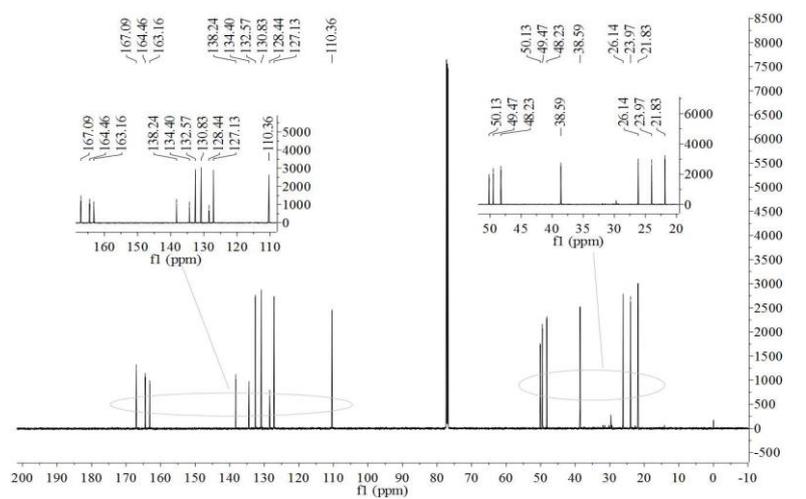


Figure S131. $^{13}\text{C-NMR}$ spectrum of (Z)-verbenone O-(2', 4'-dichlorobenzoyl) oxime **4l** in CDCl_3 .

HQ-24L2 #43 RT: 0.73 AV: 1 SB: 22 0.06-0
T: + c ESI Q1MS [100.000-600.000]

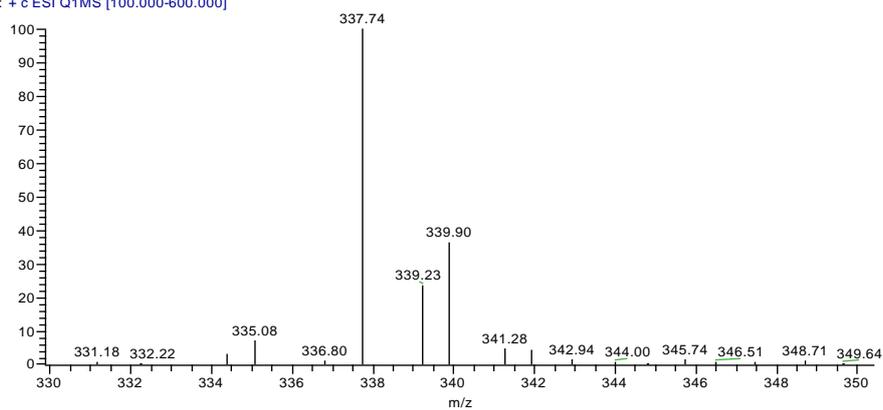


Figure S132. ESI-MS spectrum of (Z)-verbenone O-(2', 4'-dichlorobenzoyl) oxime 4l.

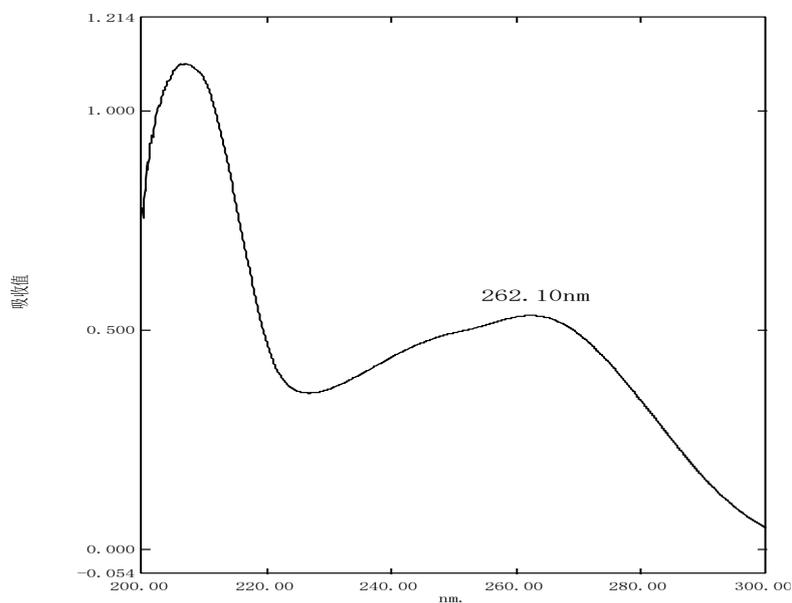


Figure S123. UV-vis spectrum of (E)-verbenone O-(2', 4'-dichlorobenzoyl) oxime 4l in EtOH.

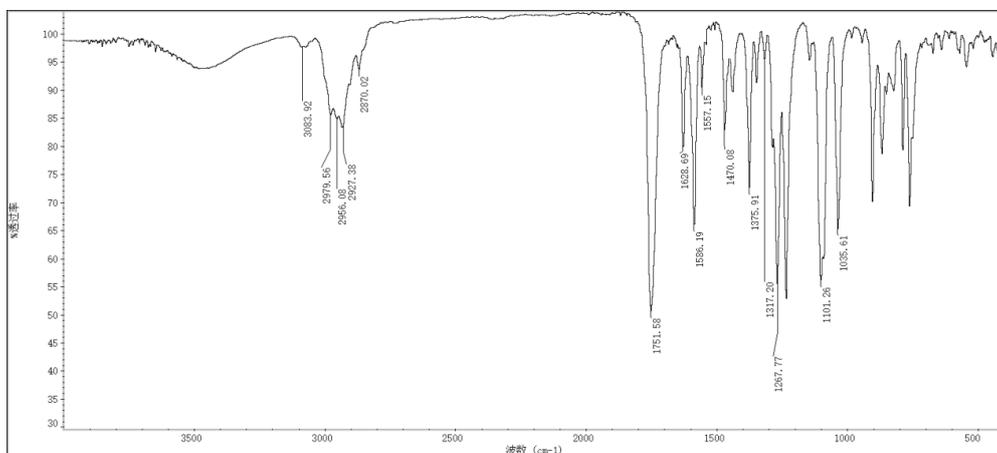


Figure S124. FTIR spectrum of (E)-verbenone O-(2', 4'-dichlorobenzoyl) oxime 4l.

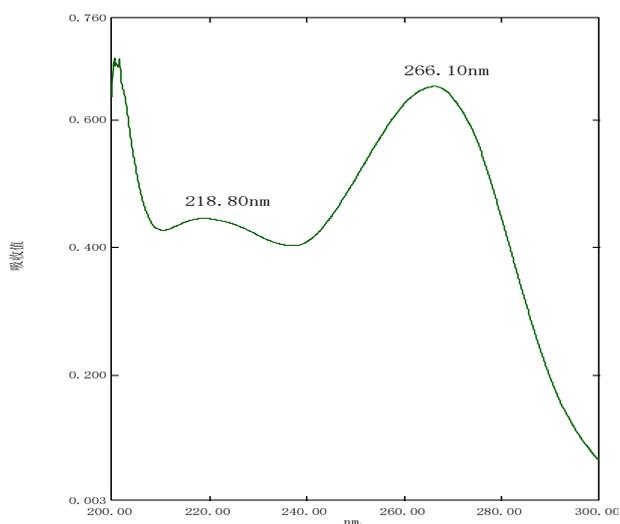


Figure S138. UV-vis spectrum of (Z)-verbenone O-2-chloropyridylcarbonyl oxime **4m** in EtOH.

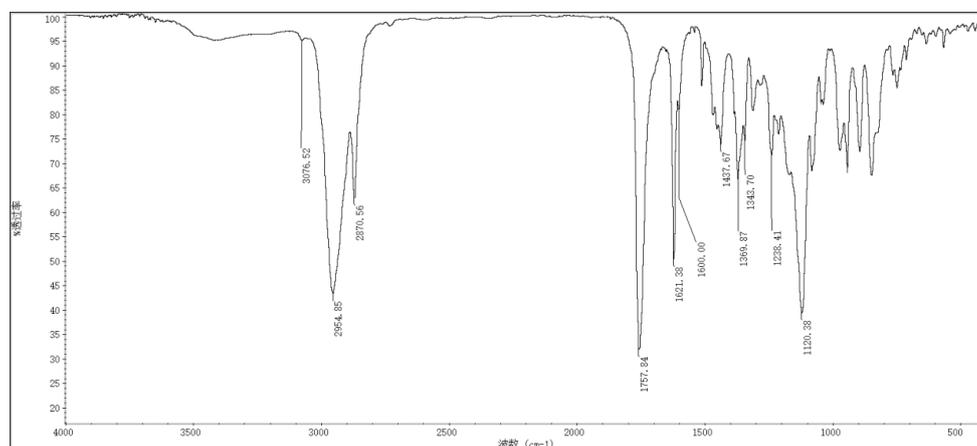


Figure S139. FTIR spectrum of (Z)-verbenone O-2-chloropyridylcarbonyl oxime **4m**.

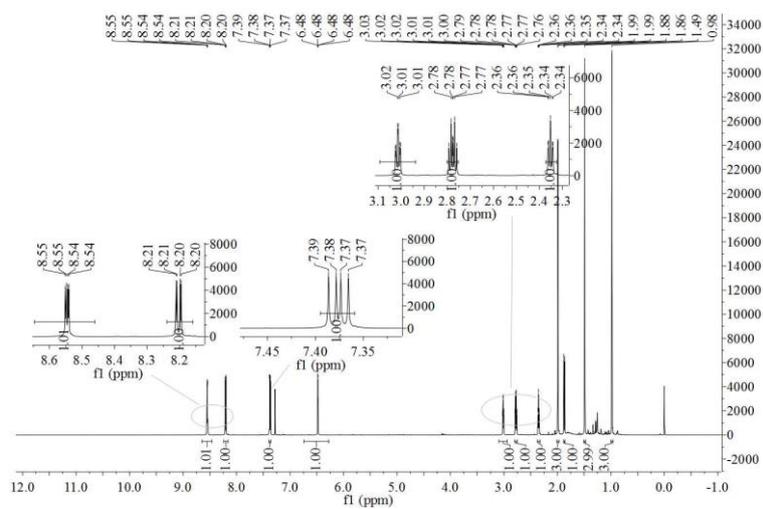


Figure S140. $^1\text{H-NMR}$ spectrum of (Z)-verbenone O-2-chloropyridylcarbonyl oxime **4m** in CDCl_3 .

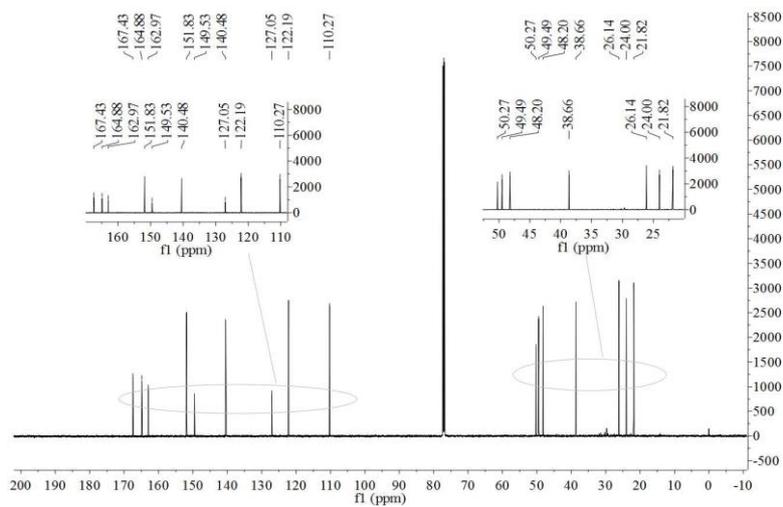


Figure S141. ^{13}C -NMR spectrum of (*Z*)-verbenone O-2-chloropyridylcarbonyl oxime **4m** in CDCl_3 .

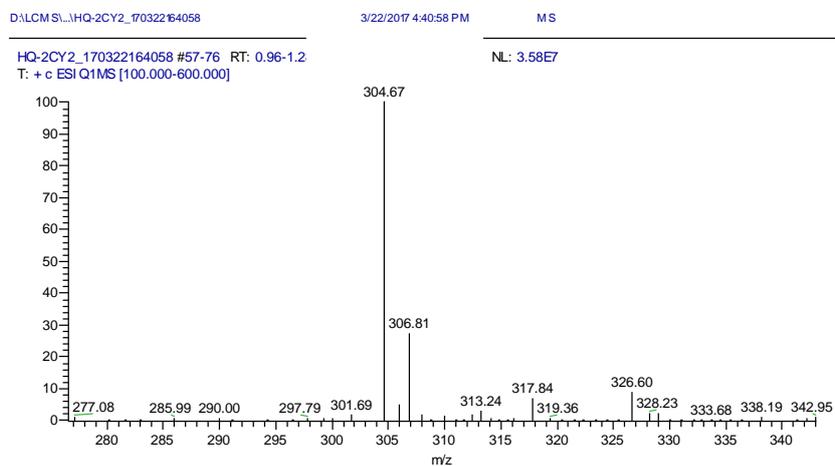


Figure S142. ESI-MS spectrum of (*Z*)-verbenone O-2-chloropyridylcarbonyl oxime **4m**

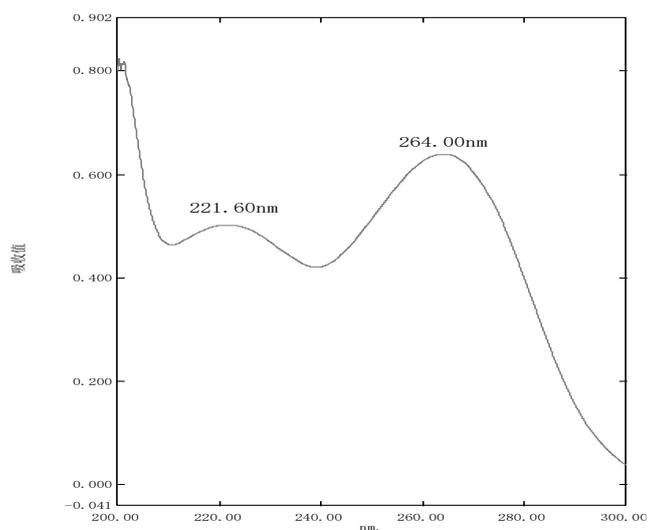


Figure S143. UV-vis spectrum of (*E*)-verbenone O-2-chloropyridylcarbonyl oxime **4m** in EtOH .

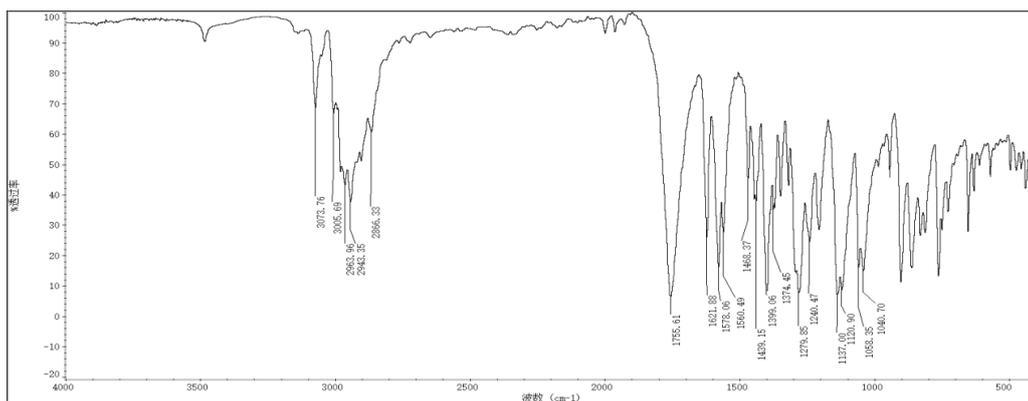


Figure S134. FTIR spectrum of (*E*)-verbenone O-2-chloropyridylcarbonyl oxime **4m**.

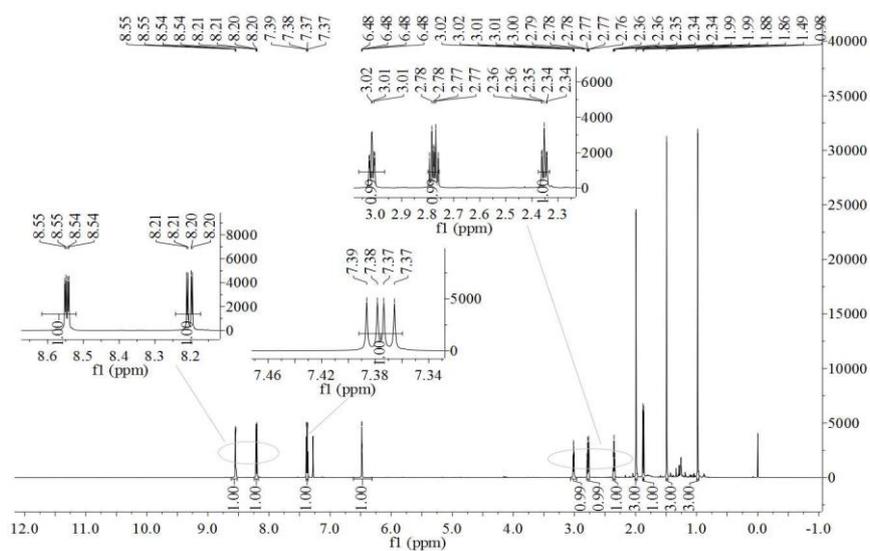


Figure S135. ¹H-NMR spectrum of (*E*)-verbenone O-2-chloropyridylcarbonyl oxime **4m** in CDCl₃.

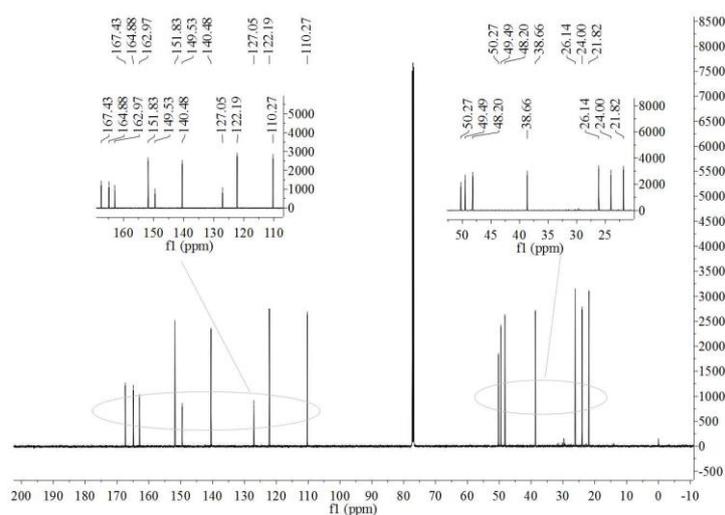


Figure S136. ¹³C-NMR spectrum of (*E*)-verbenone O-2-chloropyridylcarbonyl oxime **4m** in CDCl₃.

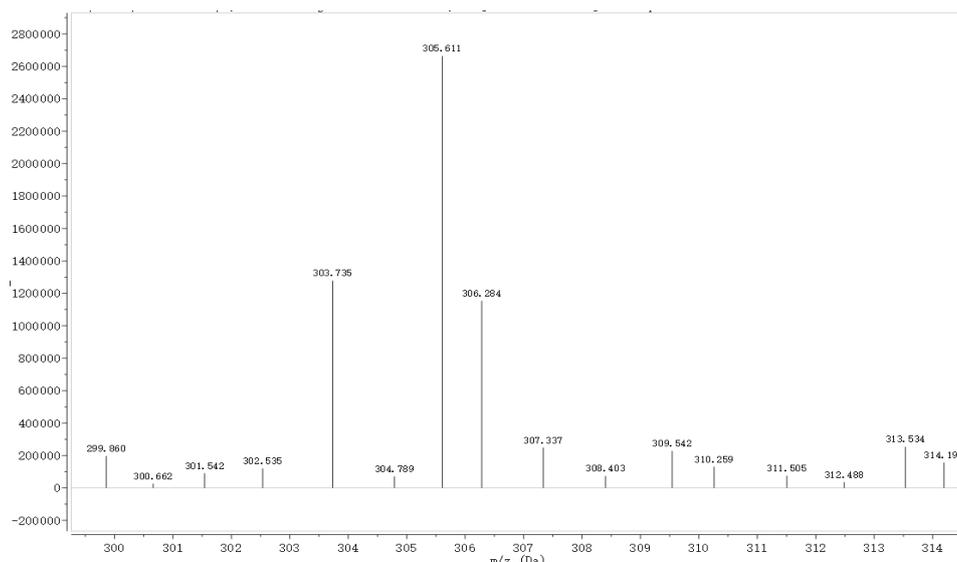


Figure S137. ESI-MS spectrum of (*E*)-verbenone O-2-chloropyridylcarbonyl oxime **4m**.

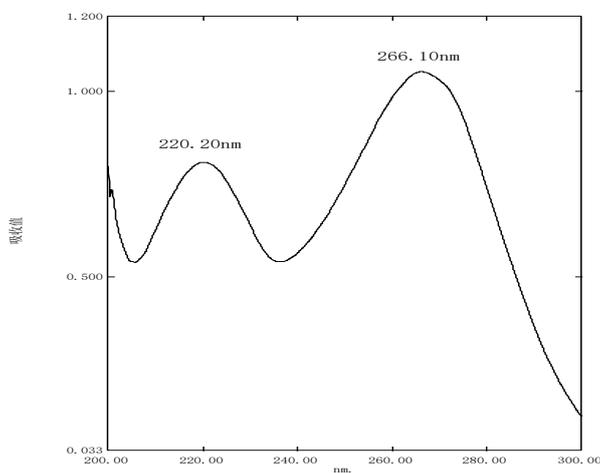


Figure S148. UV-vis spectrum of (*Z*)-verbenone O- β -pyridylcarbonyl oxime **4n** in EtOH.

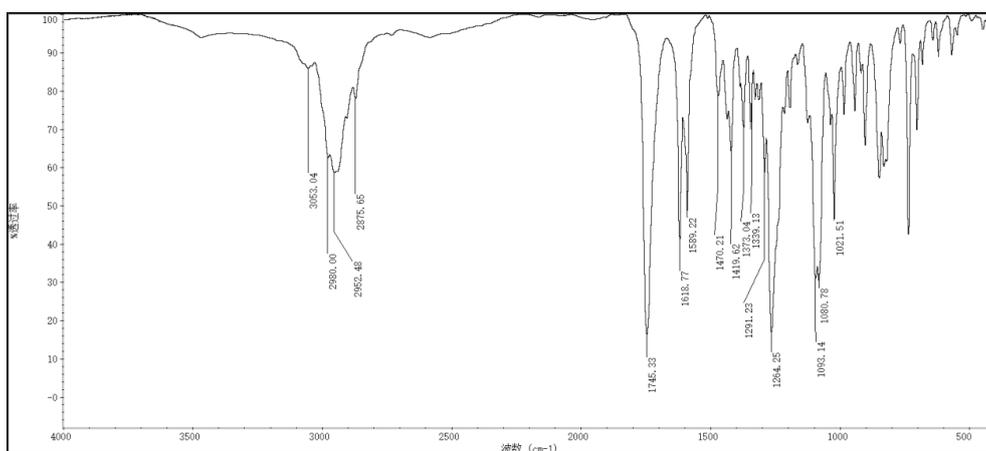


Figure S149. FTIR spectrum of (*Z*)-verbenone O- β -pyridylcarbonyl oxime **4n**.

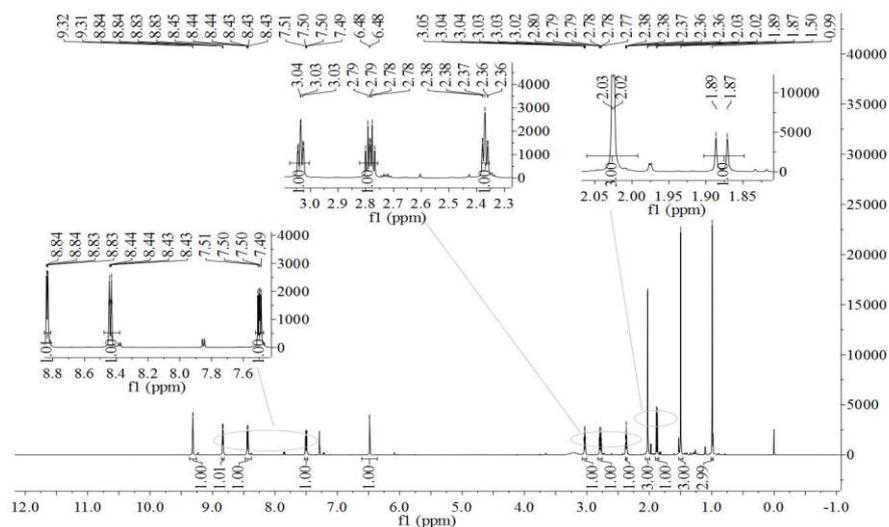


Figure S150. ¹H-NMR spectrum of (Z)-verbenone O-β-pyridylcarbonyl oxime 4n in CDCl₃.

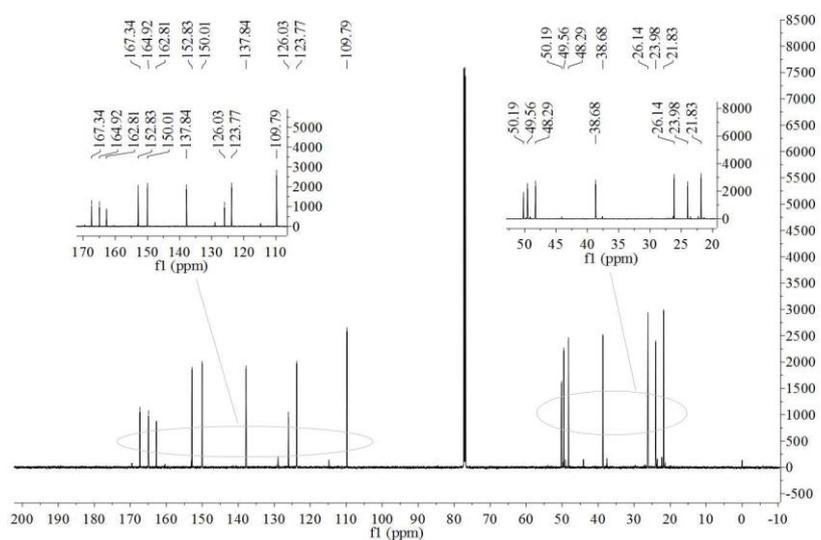


Figure S151. ¹³C-NMR spectrum of (Z)-verbenone O-β-pyridylcarbonyl oxime 4n in CDCl₃.

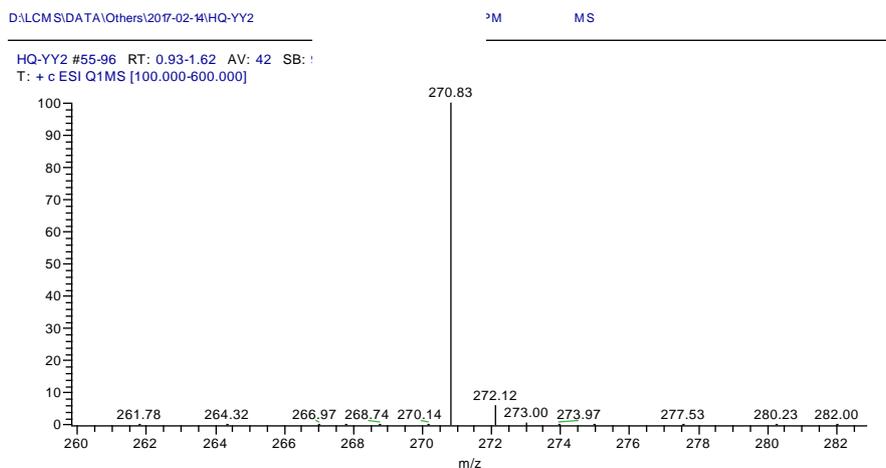


Figure S152. ESI-MS spectrum of (Z)-verbenone O-β-pyridylcarbonyl oxime 4n.

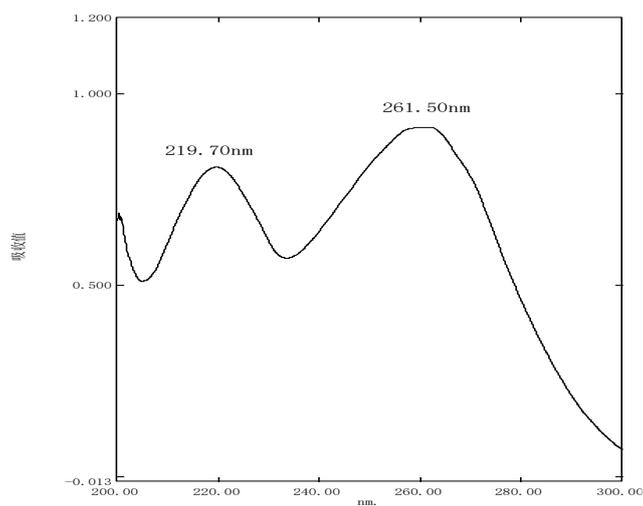


Figure S143. UV-vis spectrum of (*E*)-verbenone *O*- β -pyridylcarbonyl oxime **4n** in EtOH.

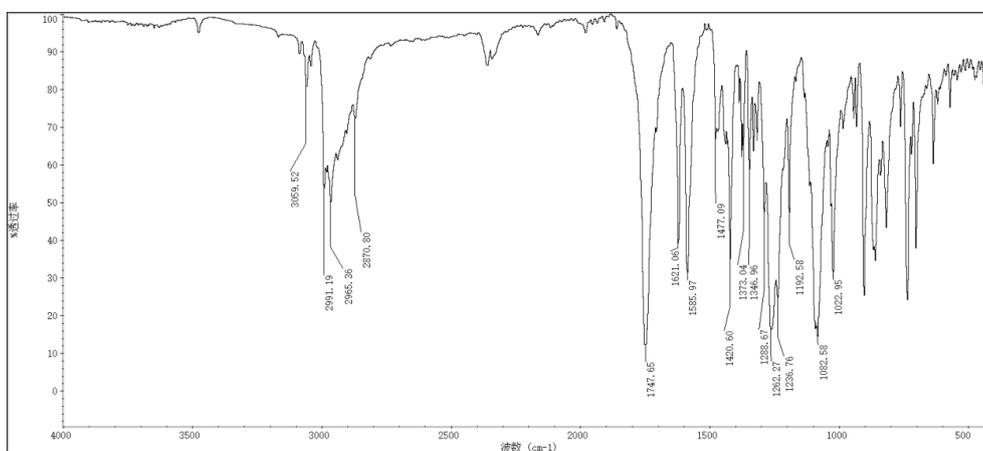


Figure S144. FTIR spectrum of (*E*)-verbenone *O*- β -pyridylcarbonyl oxime **4n**.

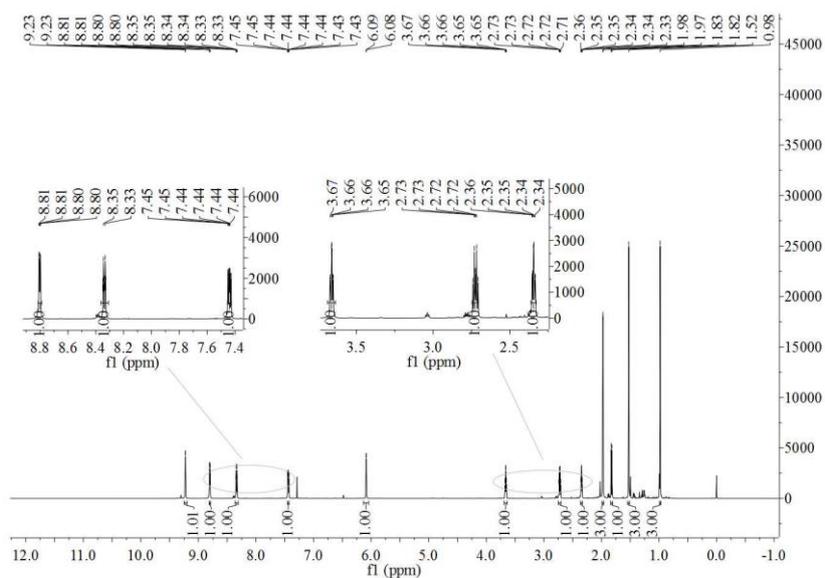


Figure S145. $^1\text{H-NMR}$ spectrum of (*E*)-verbenone *O*- β -pyridylcarbonyl oxime **4n** in CDCl_3 .

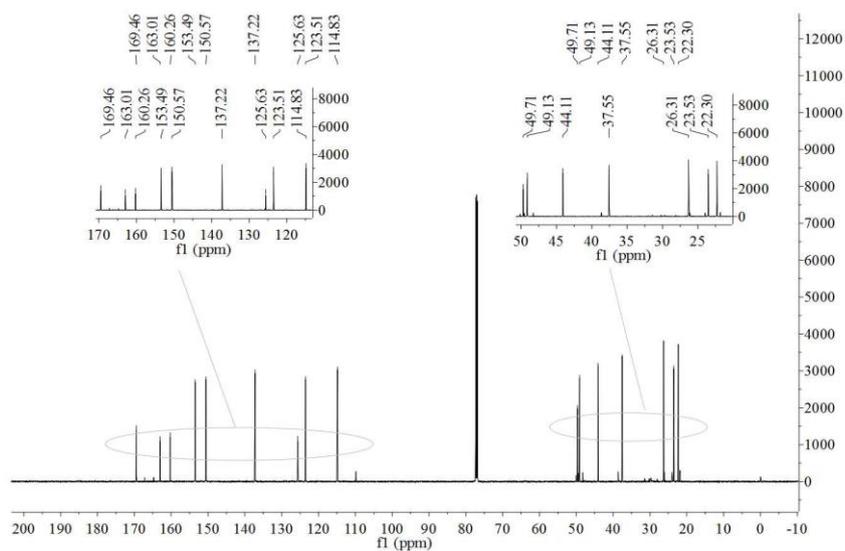


Figure S146. ^{13}C -NMR spectrum of (*E*)-verbenone *O*- β -pyridylcarbonyl oxime **4n** in CDCl_3 .

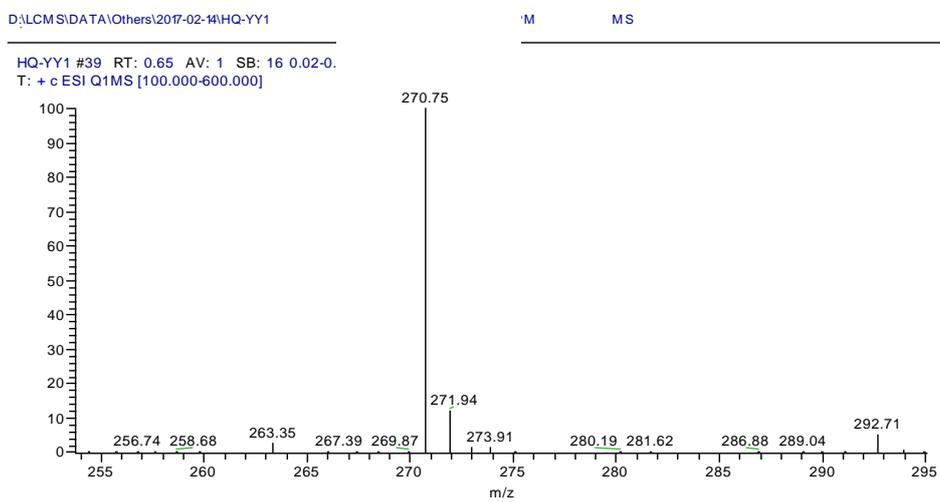


Figure S147. ESI-MS spectrum of (*E*)-verbenone *O*- β -pyridylcarbonyl oxime **4n**.