Supplementary Information

An Acetal Acylation Methodology for Producing Diversity of Trihalomethyl-1,3-dielectrophiles and 1,2-Azole Derivatives

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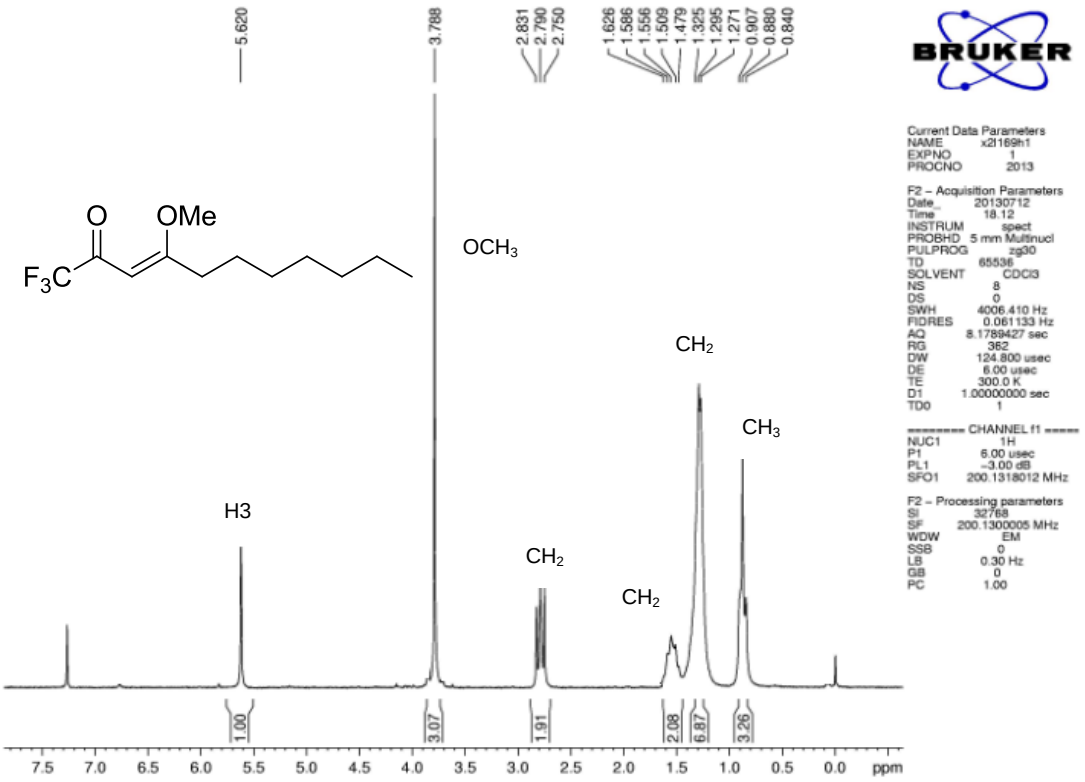

Figure S1. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of the 1,1,1-trifluoro-4-methoxyundec-3-en-2-one (**3a**).

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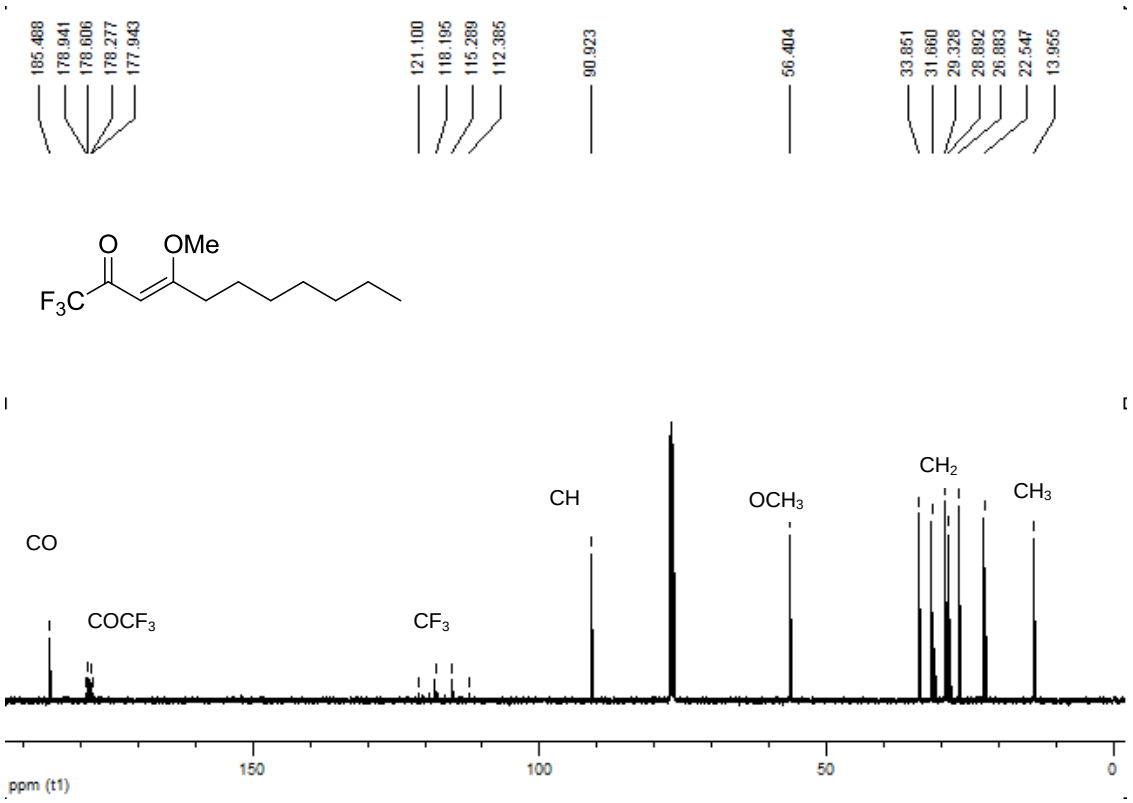


Figure S2. ^{13}C NMR spectrum (400 MHz, $CDCl_3$) of the 1,1,1-trifluoro-4-methoxyundec-3-en-2-one (**3a**).

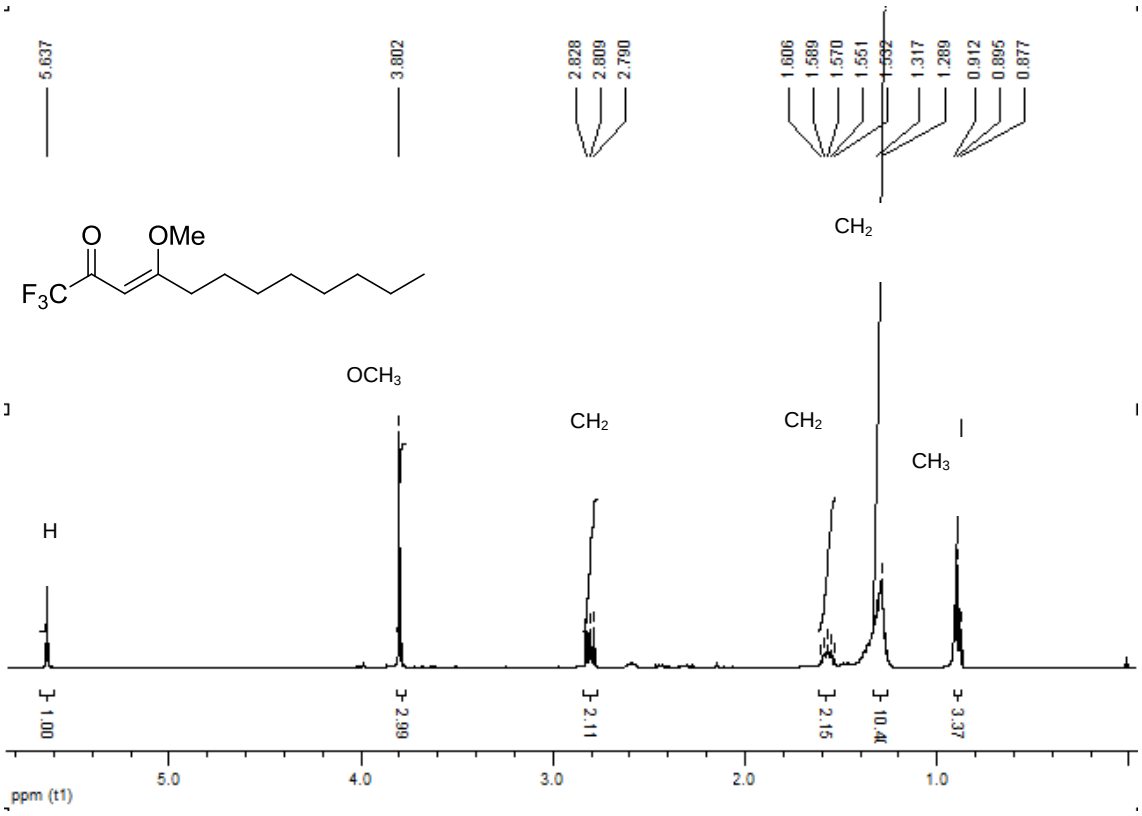


Figure S3. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of the 1,1,1-trifluoro-4-methoxydodec-3-en-2-one (**3b**).

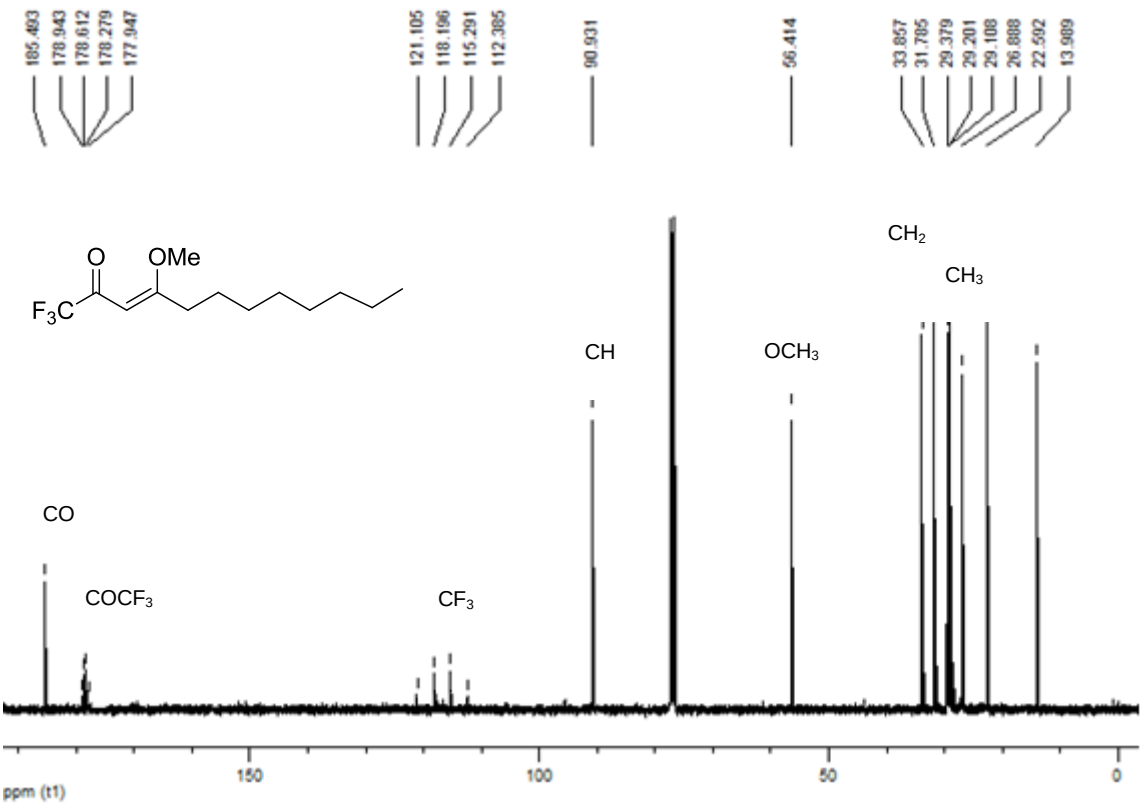


Figure S4. ^{13}C NMR spectrum (400 MHz, $CDCl_3$) of the 1,1,1-trifluoro-4-methoxydodec-3-en-2-one (**3b**).

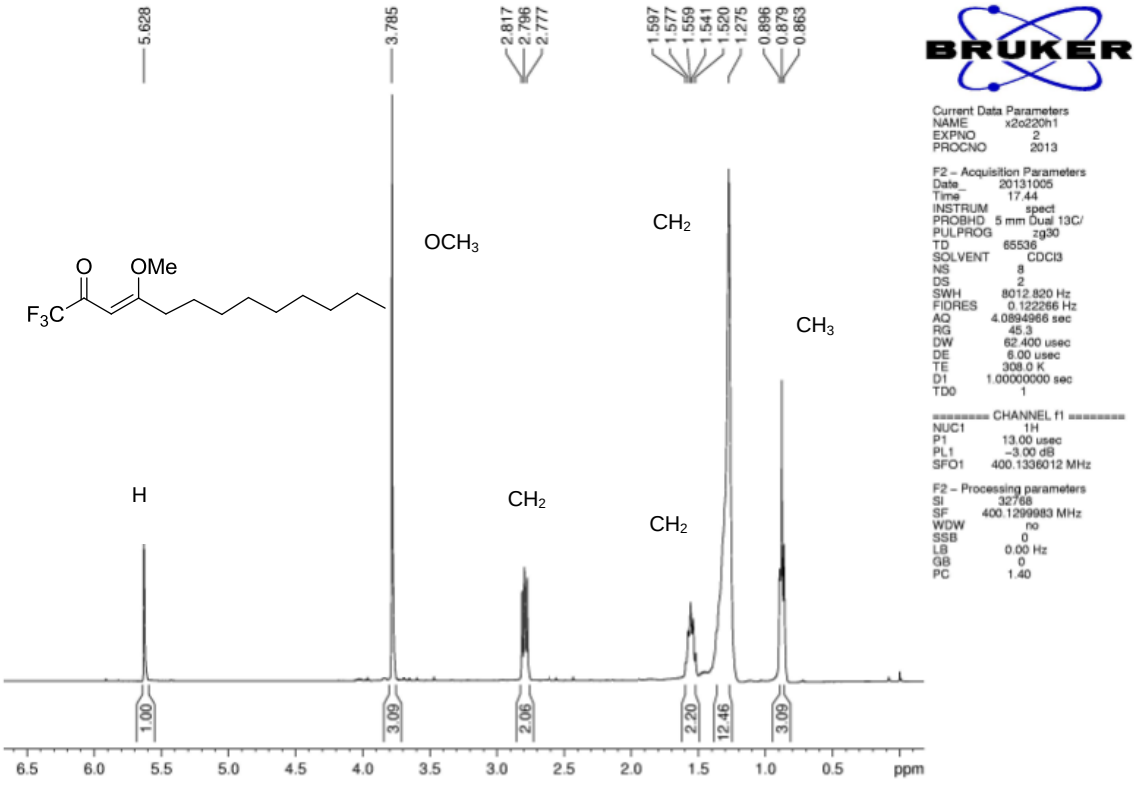


Figure S5. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of the 1,1,1-trifluoro-4-methoxytridec-3-en-2-one (**3c**).

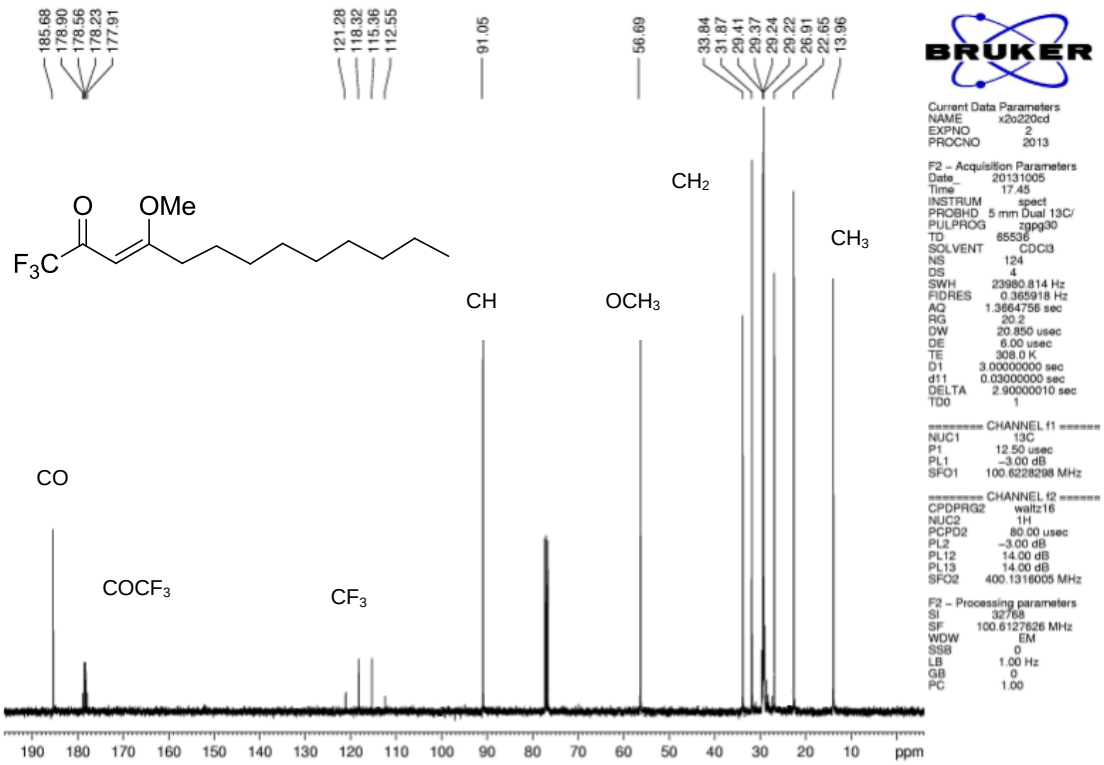


Figure S6. ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of the 1,1,1-trifluoro-4-methoxytridec-3-en-2-one (**3c**).

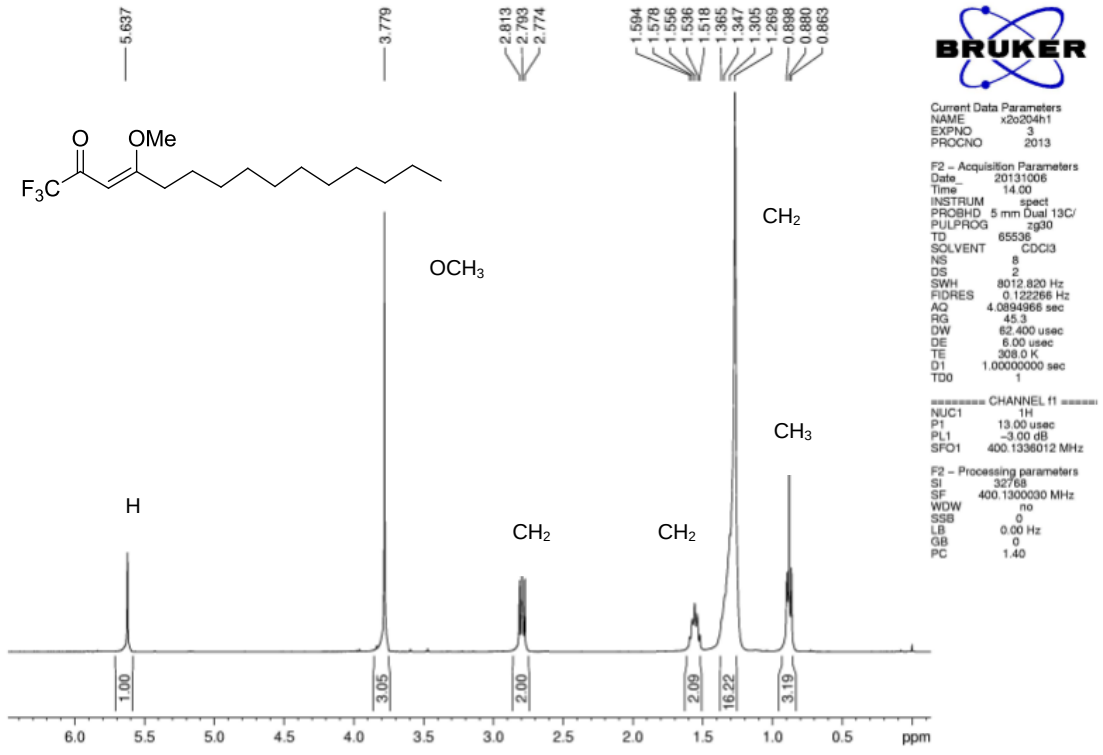


Figure S7. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of the 1,1,1-trifluoro-4-methoxypentadec-3-en-2-one (**3e**).

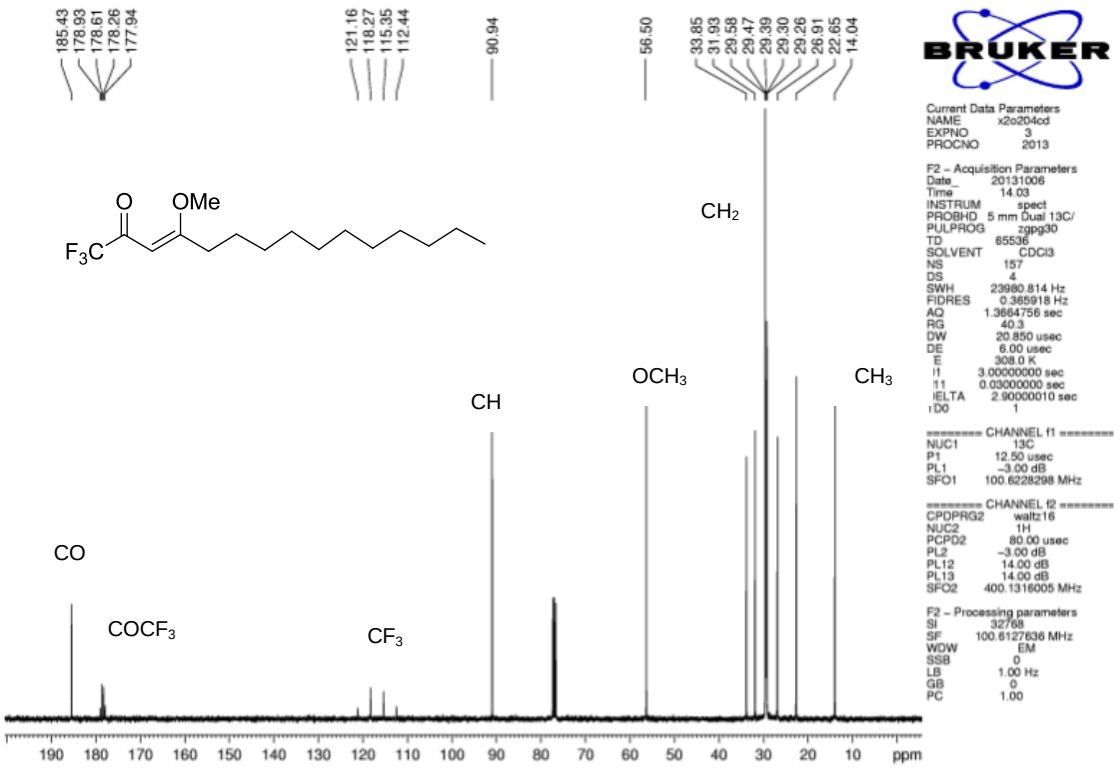


Figure S8. ^{1}H NMR spectrum (100 MHz, $CDCl_3$) of the 1,1,1-trifluoro-4-methoxypentadec-3-en-2-one (**3e**).

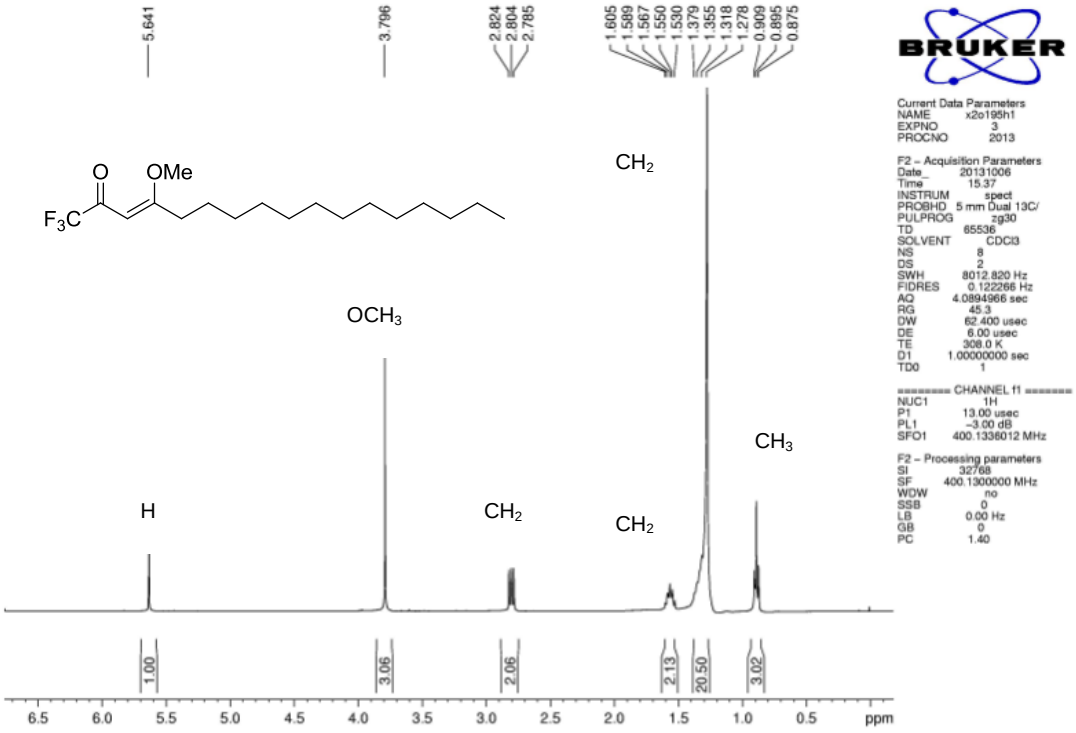


Figure S9. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of the 1,1,1-trifluoro-4-methoxyheptadec-3-en-2-one (**3f**).

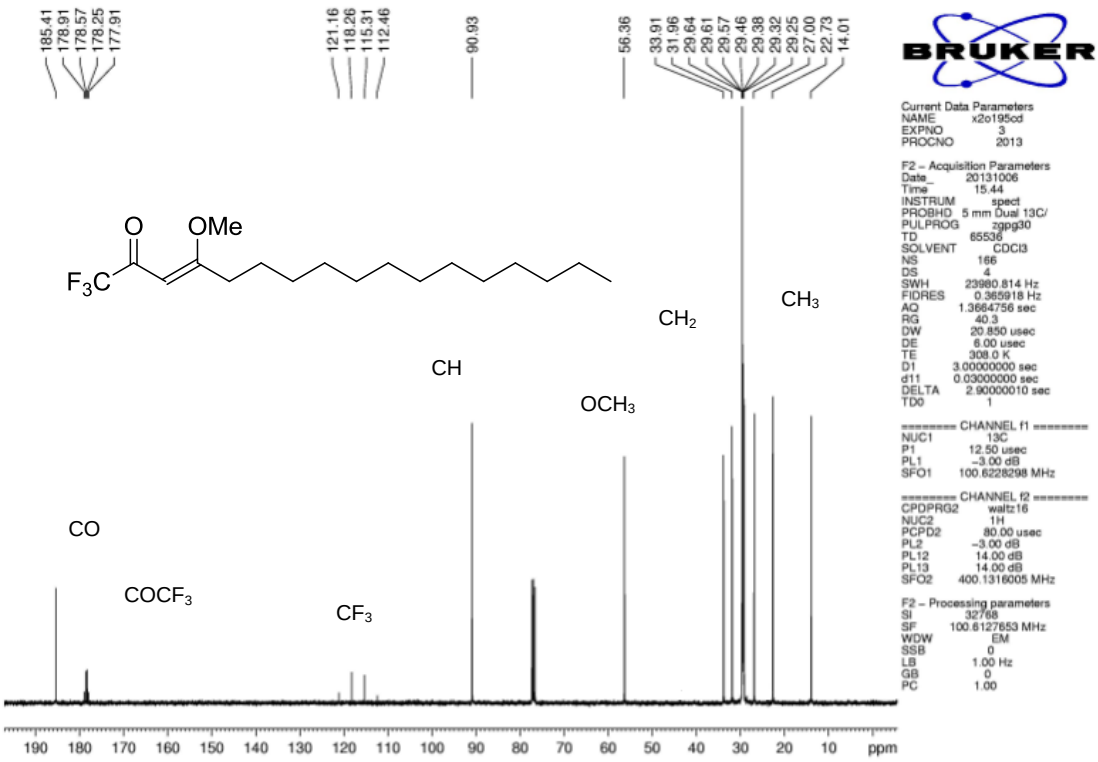


Figure S10. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of the 1,1,1-trifluoro-4-methoxyheptadec-3-en-2-one (**3f**).

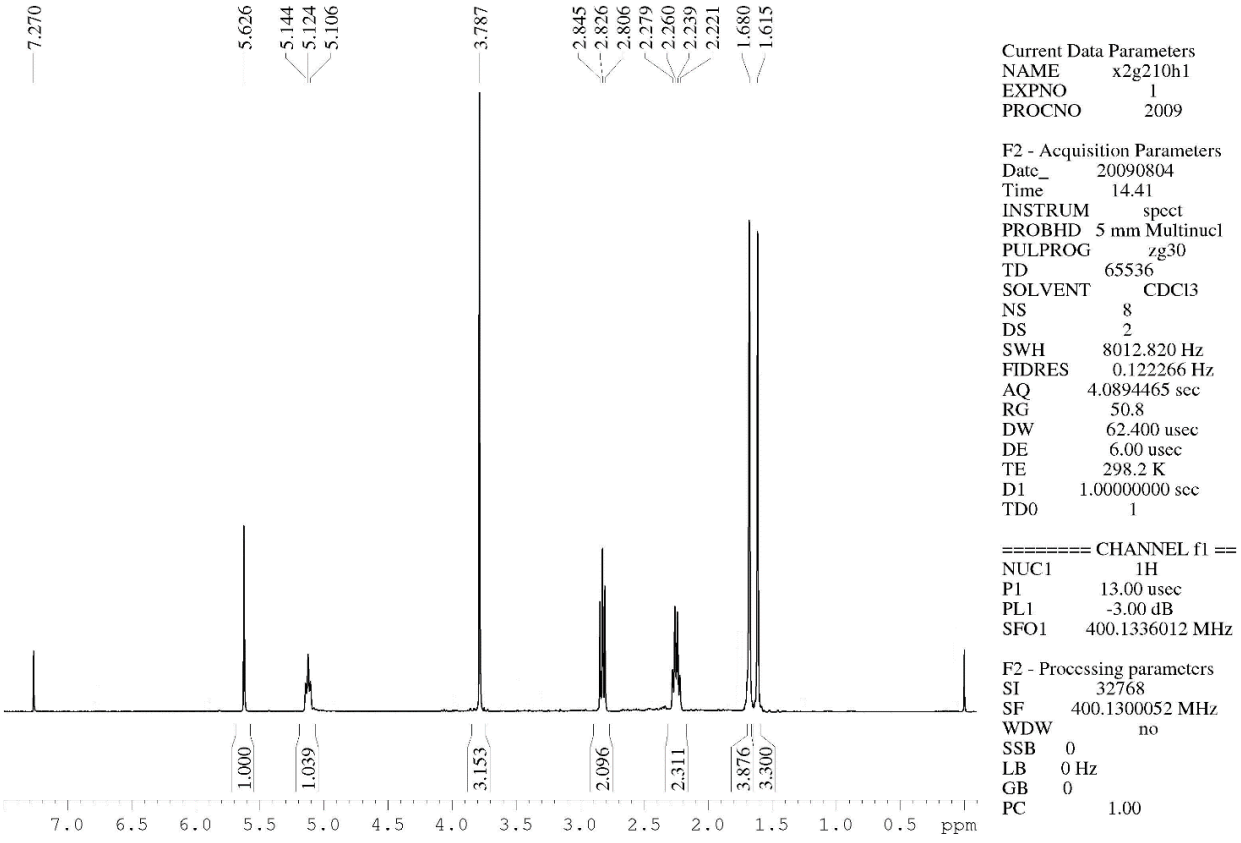


Figure S11. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of the 1,1,1-trifluoro-4-methoxy-8-methylnon-3,7-dien-2-one (**3g**).

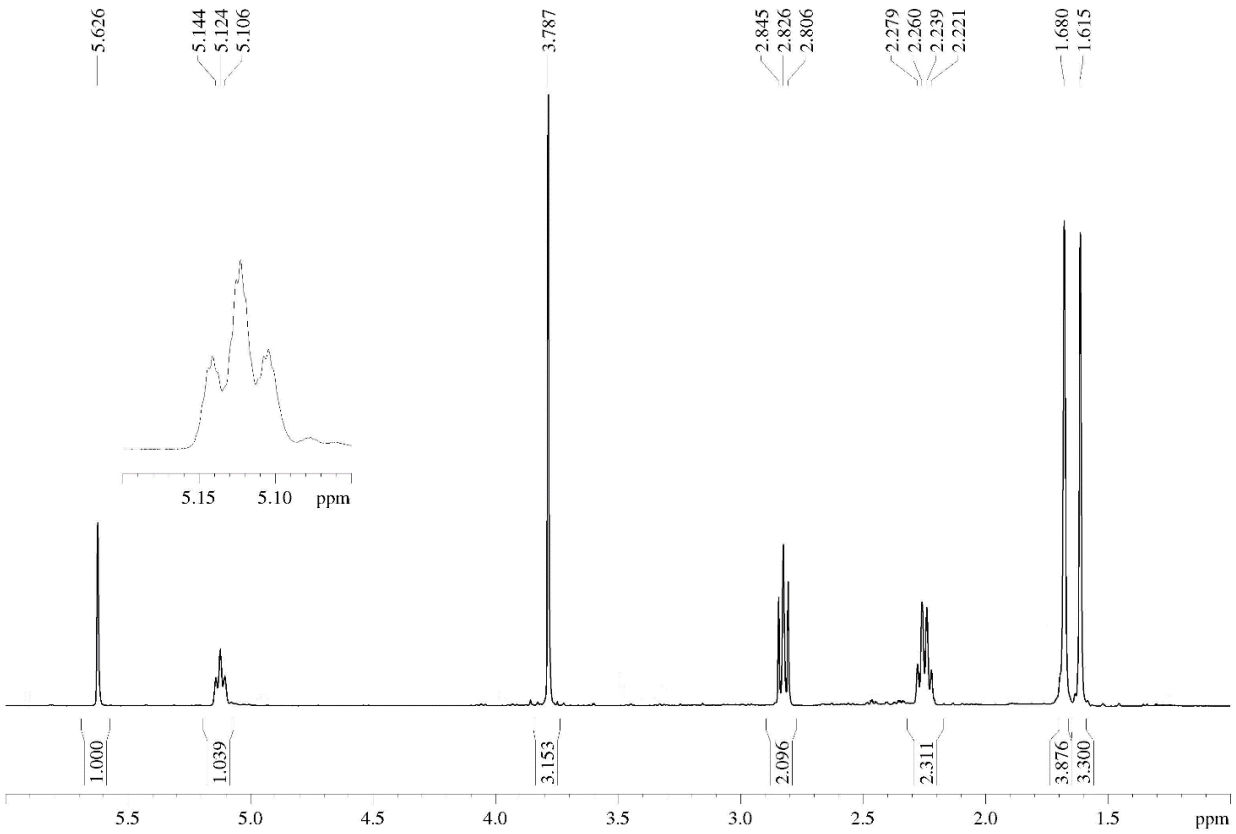


Figure S12. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of the 1,1,1-trifluoro-4-methoxy-8-methylnon-3,7-dien-2-one, expanded between 1.0-6.0 ppm (**3g**).

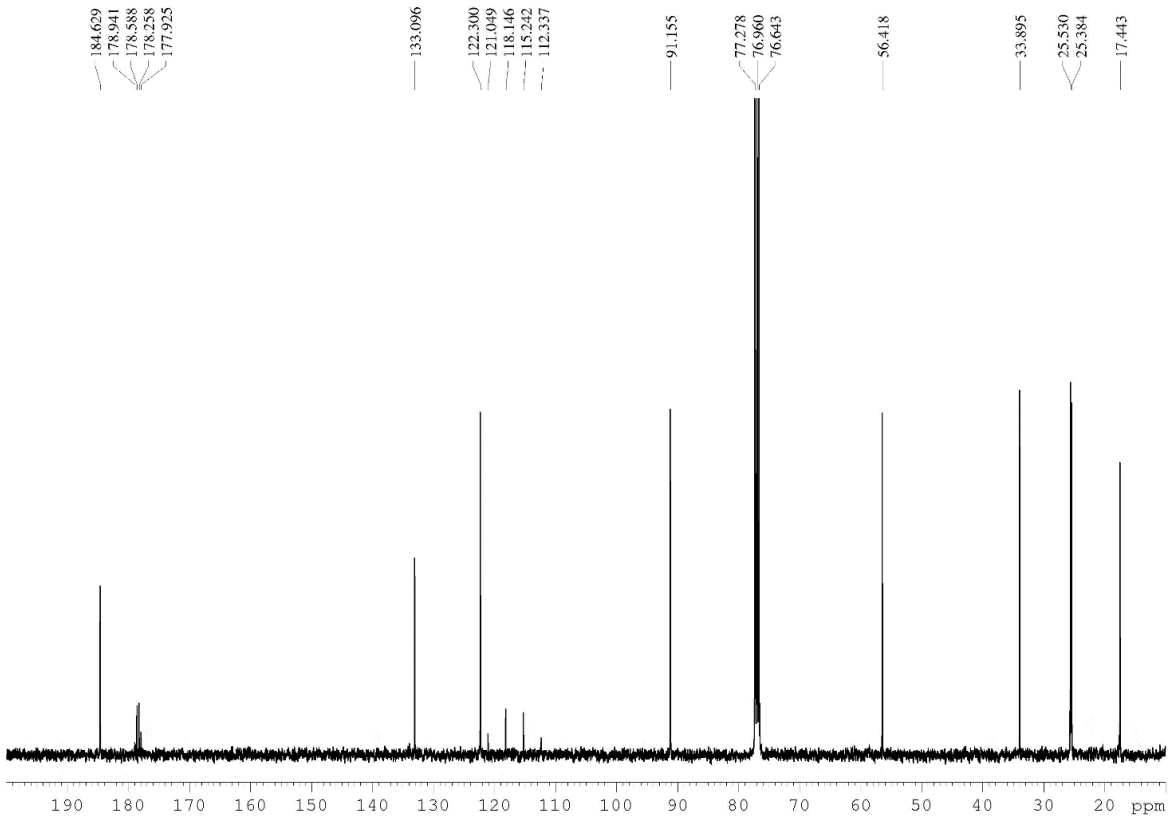


Figure S13. ^{13}C NMR spectrum (400 MHz, $CDCl_3$) of the 1,1,1-trifluoro-4-methoxy-8-methylnon-3,7-dien-2-one (**3g**).

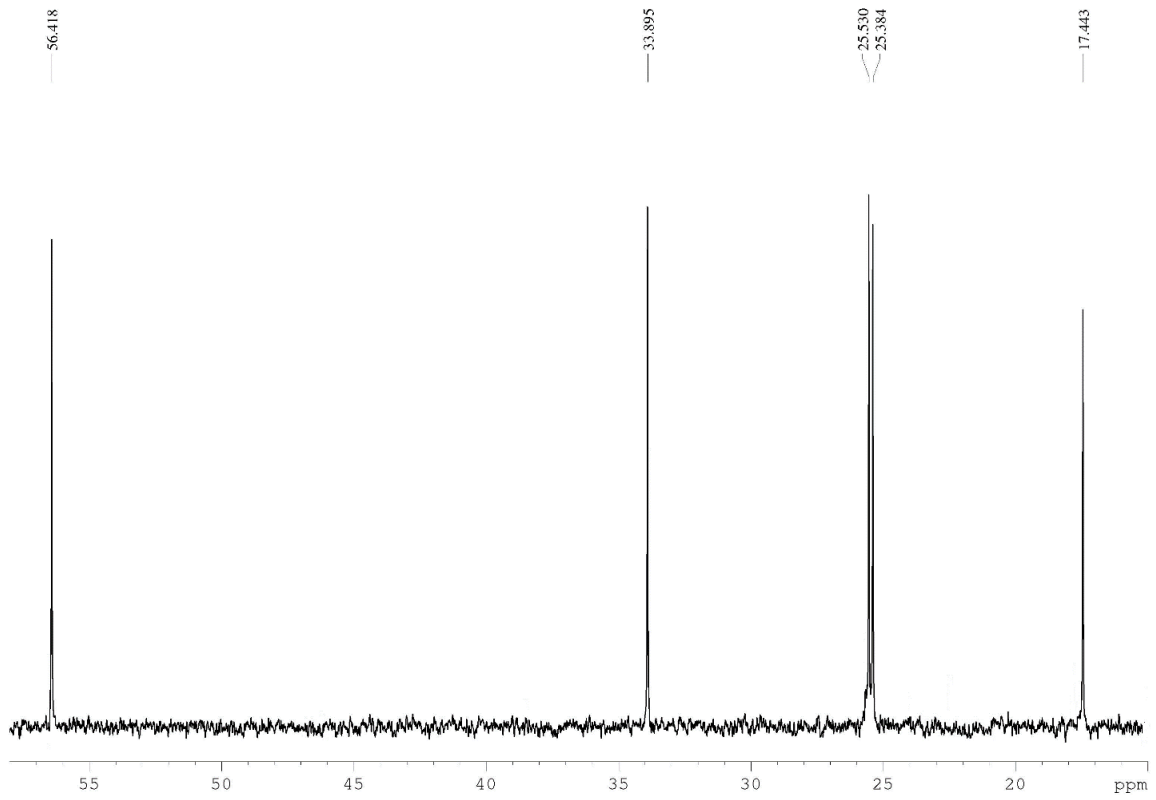


Figure S14. ^{13}C NMR spectrum (400 MHz, $CDCl_3$) of the 1,1,1-trifluoro-4-methoxy-8-methylnon-3,7-dien-2-one, expanded between 15-58 ppm (**3g**).

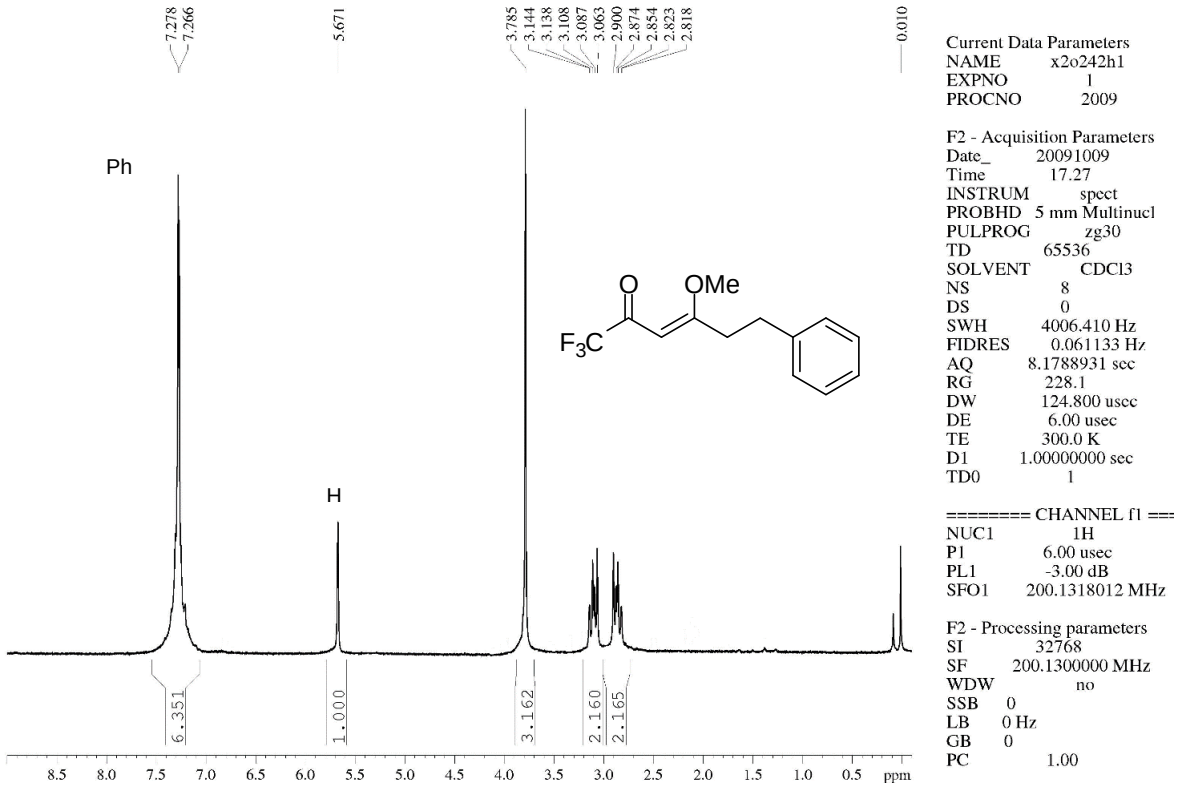


Figure S15. ^{1}H NMR spectrum (200 MHz, $CDCl_3$) of the 1,1,1-trifluoro-4-methoxy-6-phenylhex-3-en-2-one (**3h**).

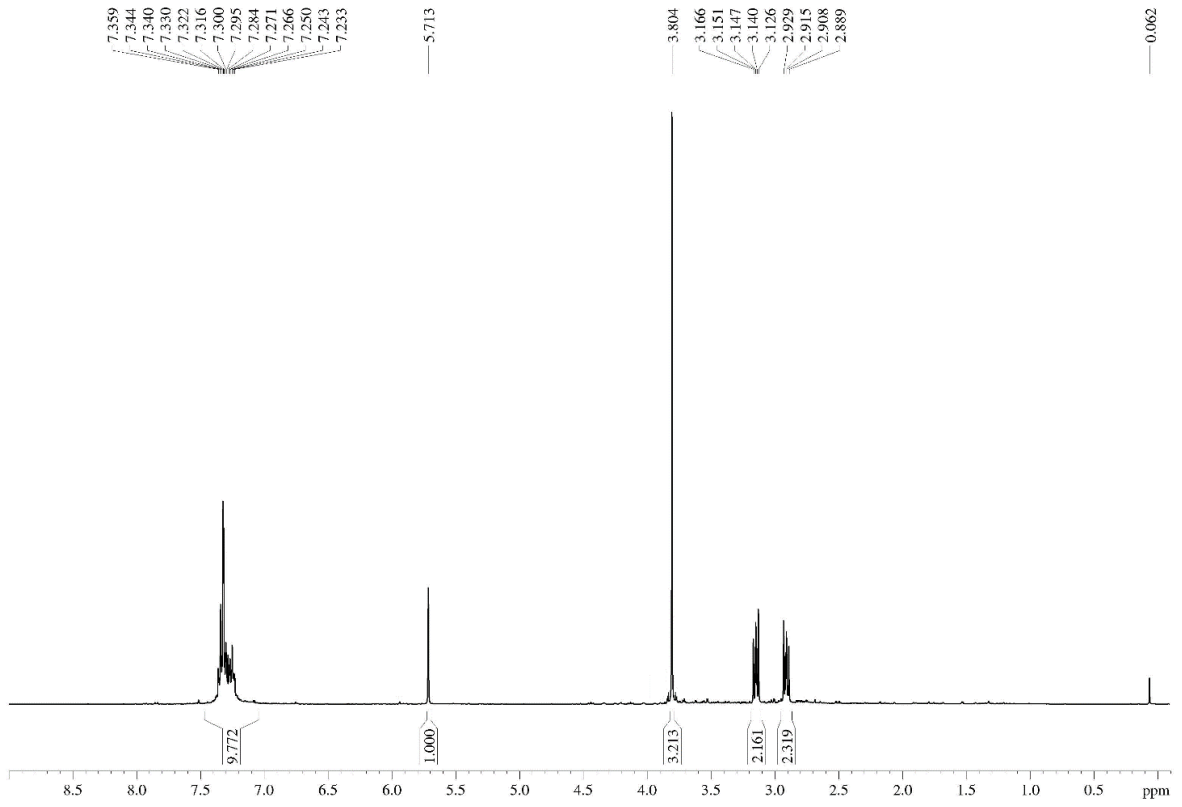


Figure S16. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of the 1,1,1-trifluoro-4-methoxy-6-phenylhex-3-en-2-one (**3h**).

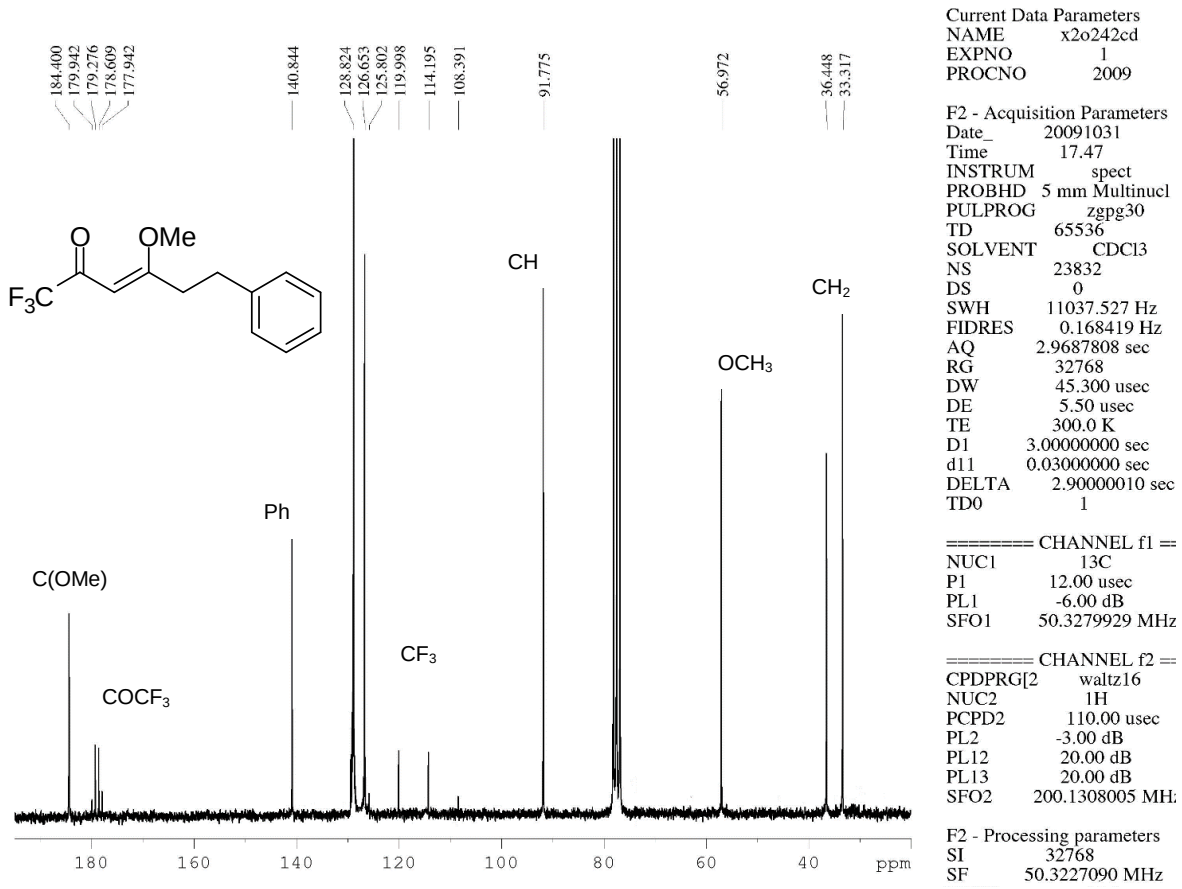


Figure S17. ^{13}C NMR spectrum (50 MHz, $CDCl_3$) of the 1,1,1-trifluoro-4-methoxy-6-phenylhex-3-en-2-one (**3h**).

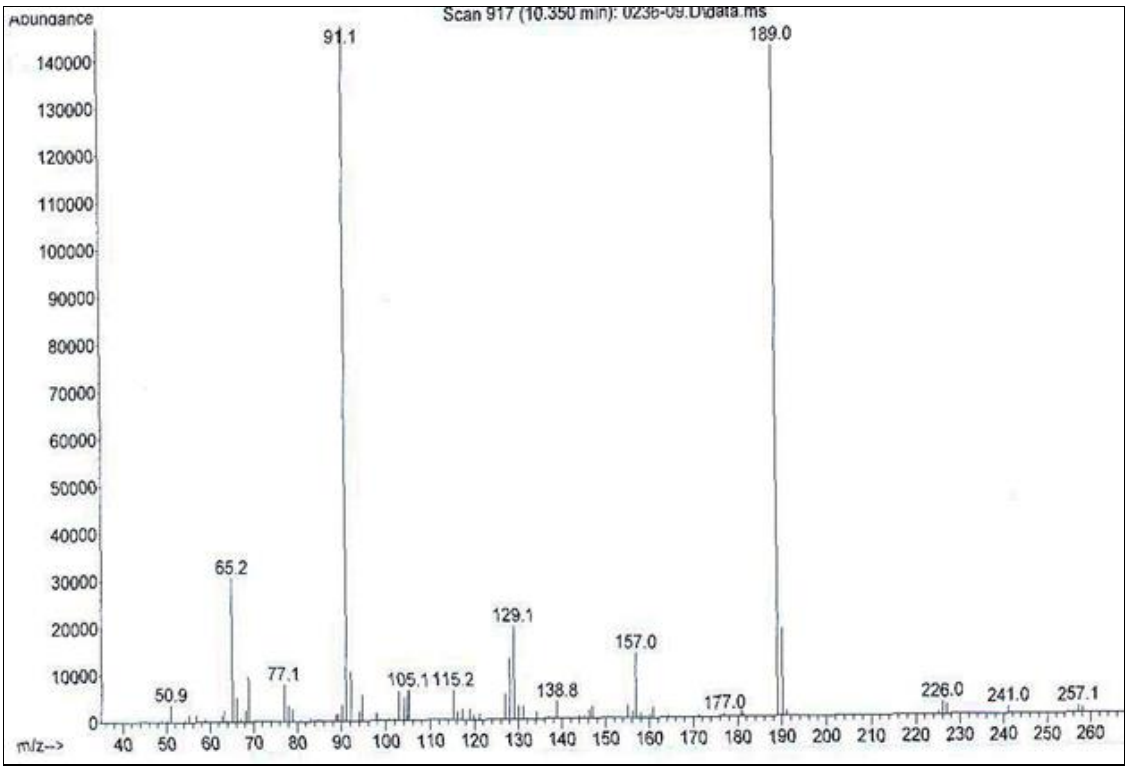


Figure S18. EI mass spectrum (70 eV) of the 1,1,1-trifluoro-4-methoxy-6-phenylhex-3-en-2-one (**3h**).

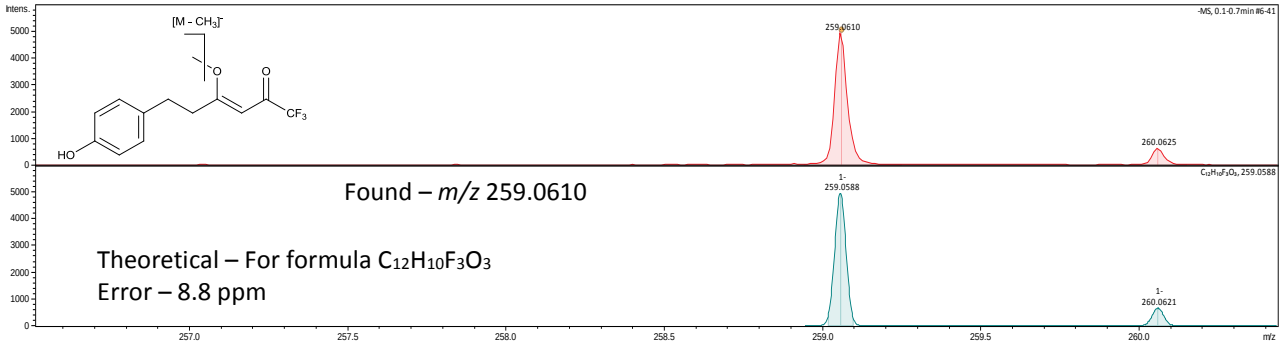


Figure S19. ESI-MS spectrum of the 1,1,1-trifluoro-4-methoxy-6-(4-hydroxyphenyl)hex-3-en-2-one (**3i**).

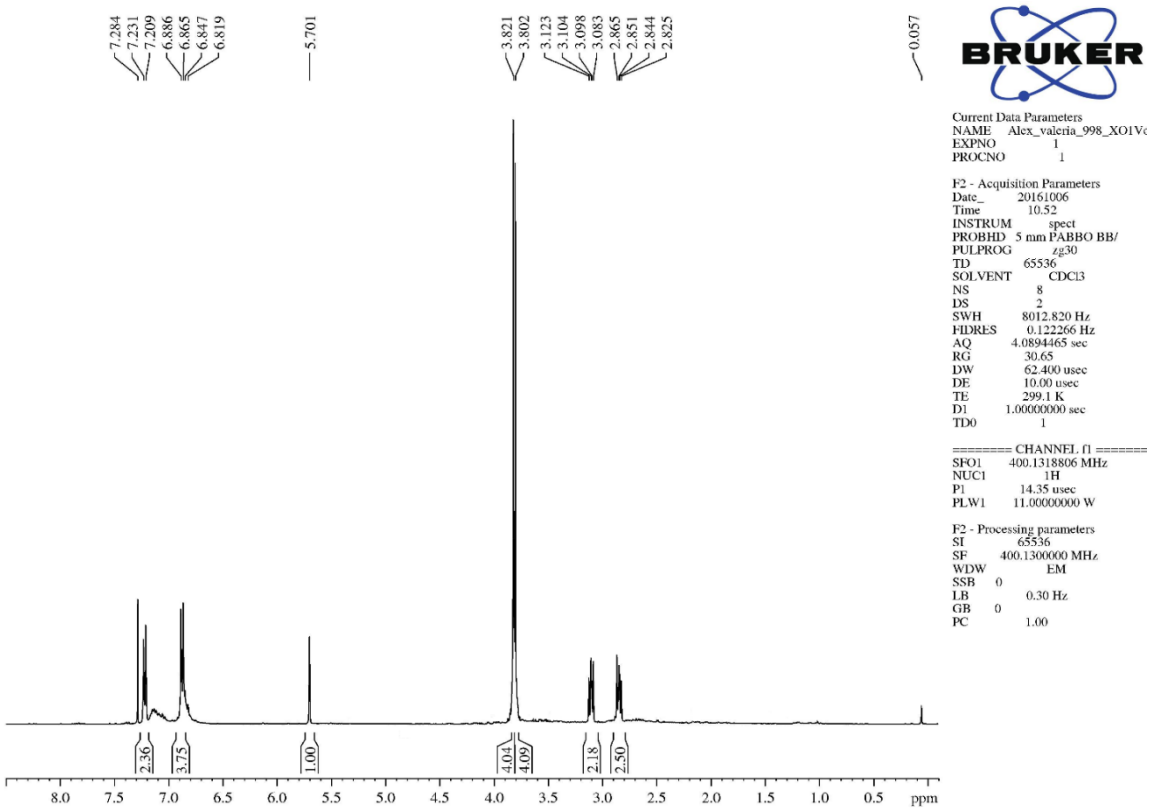


Figure S20. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of the 1,1,1-trifluoro-4-methoxy-6-(4-methoxy phenyl)hex-3-en-2-one (**3j**).

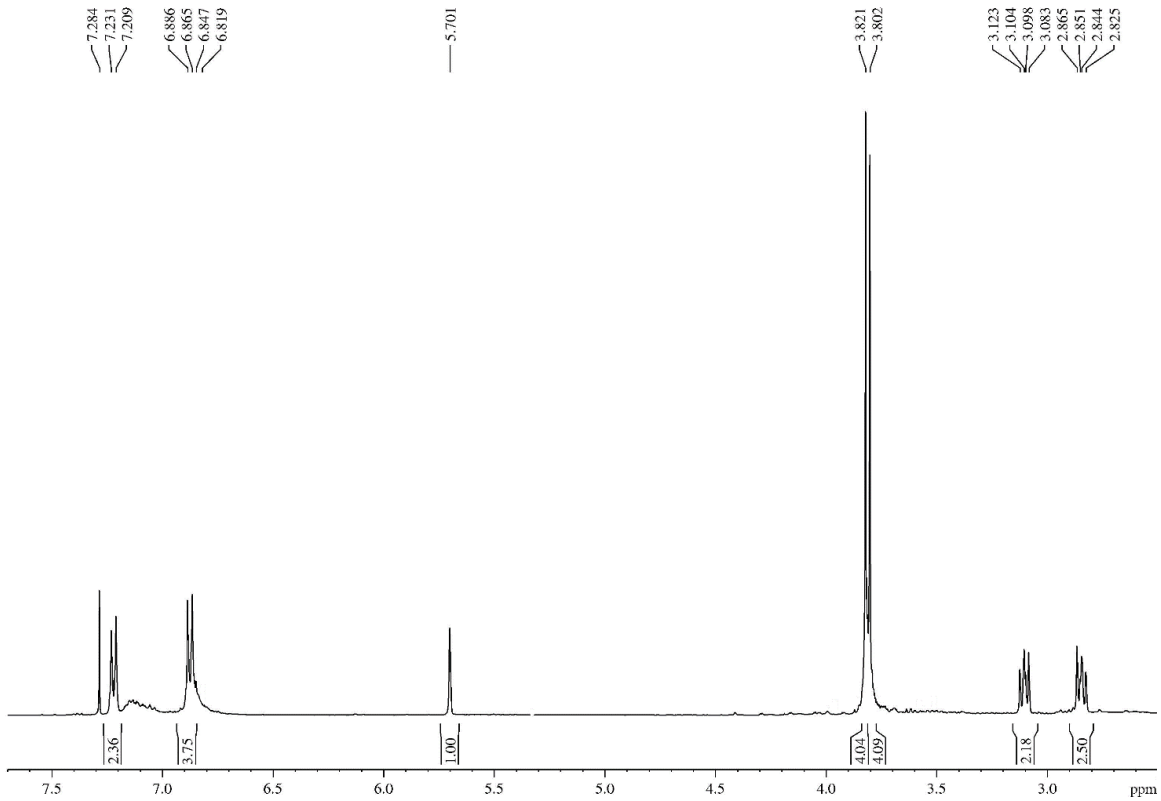


Figure S21. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of the 1,1,1-trifluoro-4-methoxy-6-(4-methoxy phenyl)hex-3-en-2-one (**3j**).

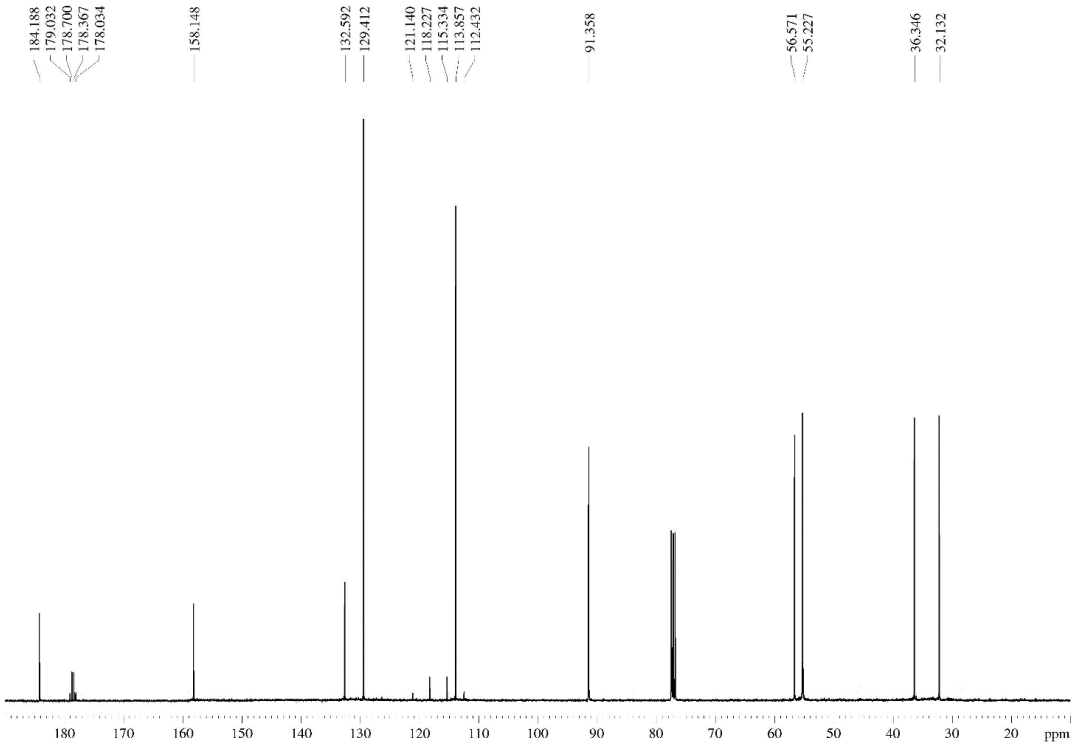


Figure S22. ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of the 1,1,1-trifluoro-4-methoxy-6-(4-methoxy phenyl)hex-3-en-2-one (**3j**).

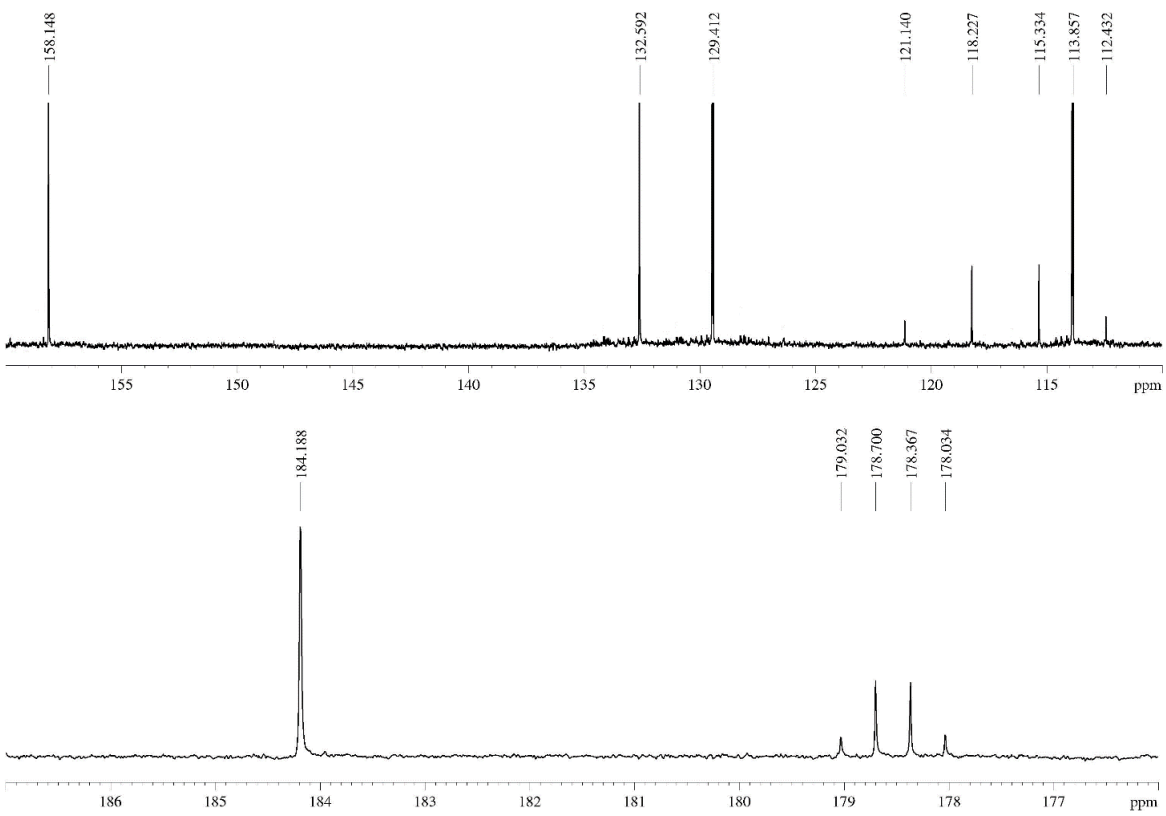


Figure S23. ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of the 1,1,1-trifluoro-4-methoxy-6-(4-methoxy phenyl)hex-3-en-2-one, expanded between 110-160 ppm (top), and 176-187 ppm (bottom) (**3j**).

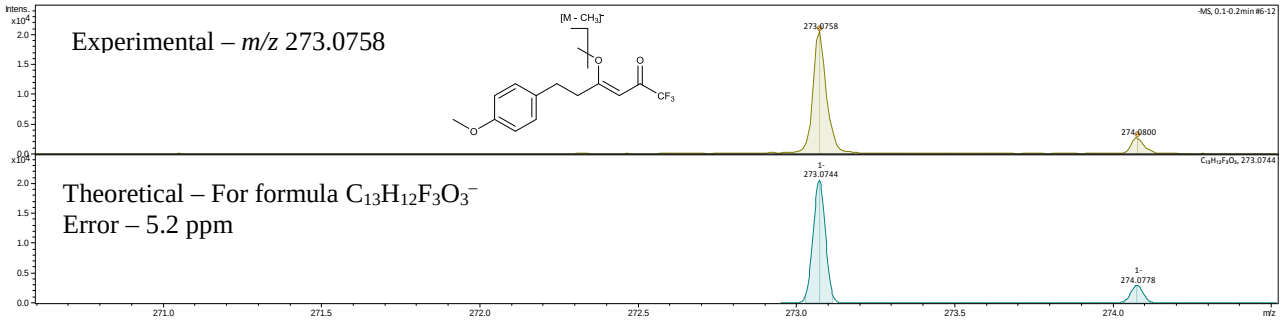


Figure S24. ESI-MS spectrum of the 1,1,1-trifluoro-4-methoxy-6-(4-methoxy phenyl)hex-3-en-2-one (**3j**).

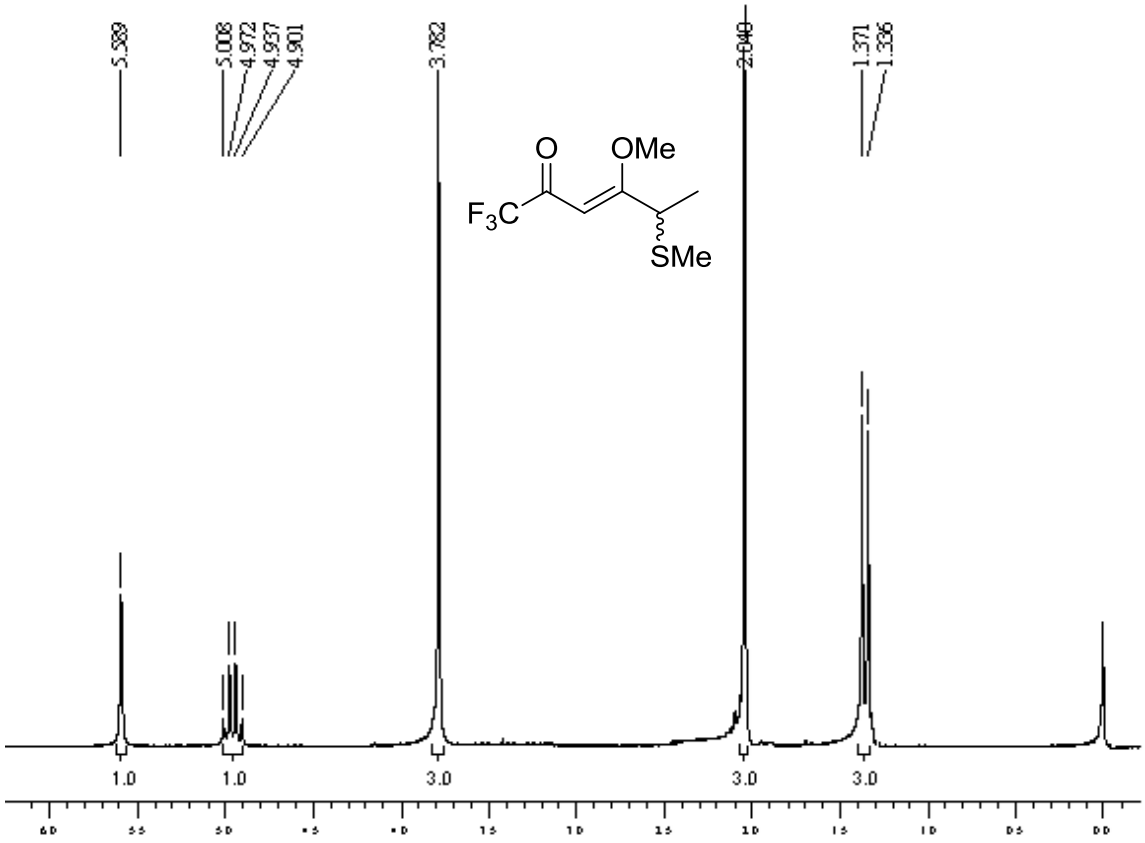


Figure S25. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of the 1,1,1-trifluoro-4-methoxy-5-thiomethylhex-3-en-2-one (**3k**).

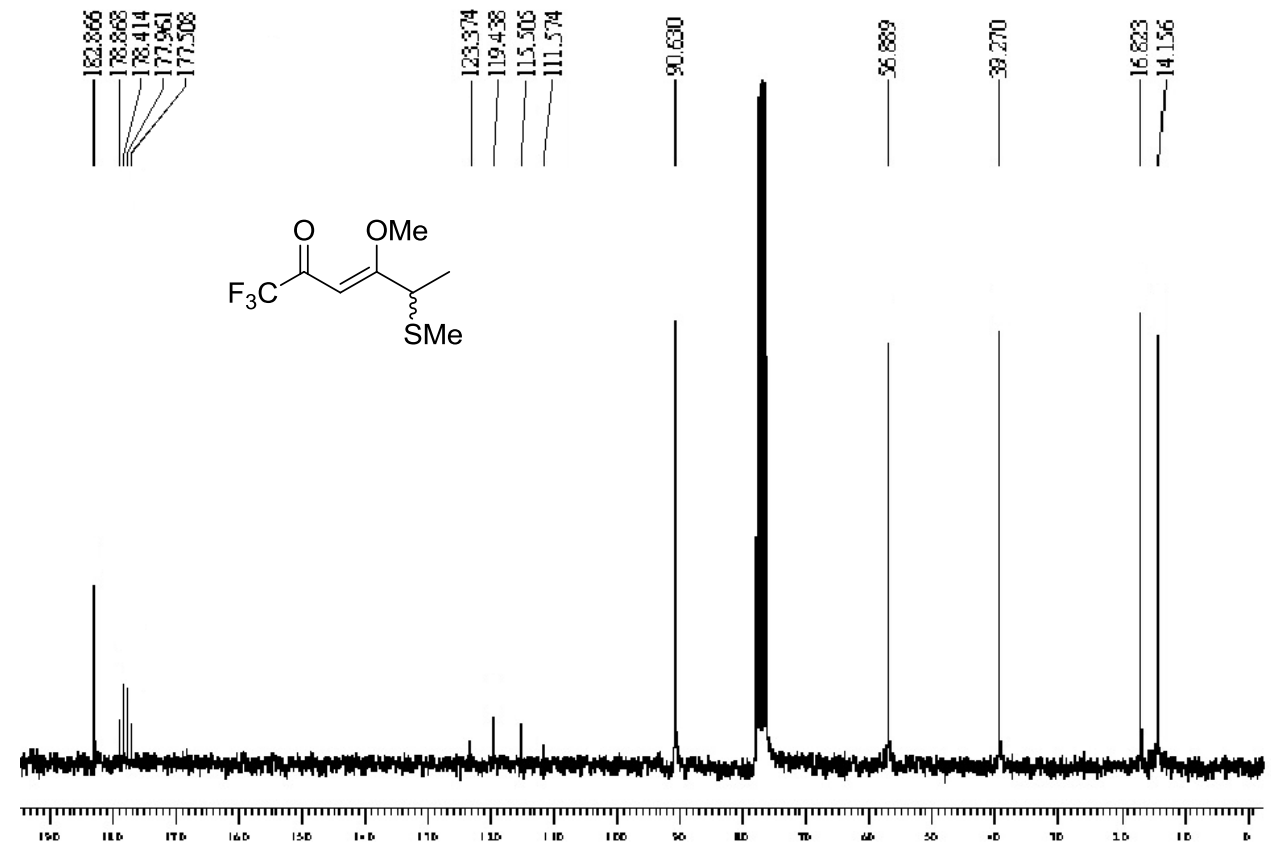


Figure S26. ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of the 1,1,1-trifluoro-4-methoxy-5-thiomethylhex-3-en-2-one (**3k**).

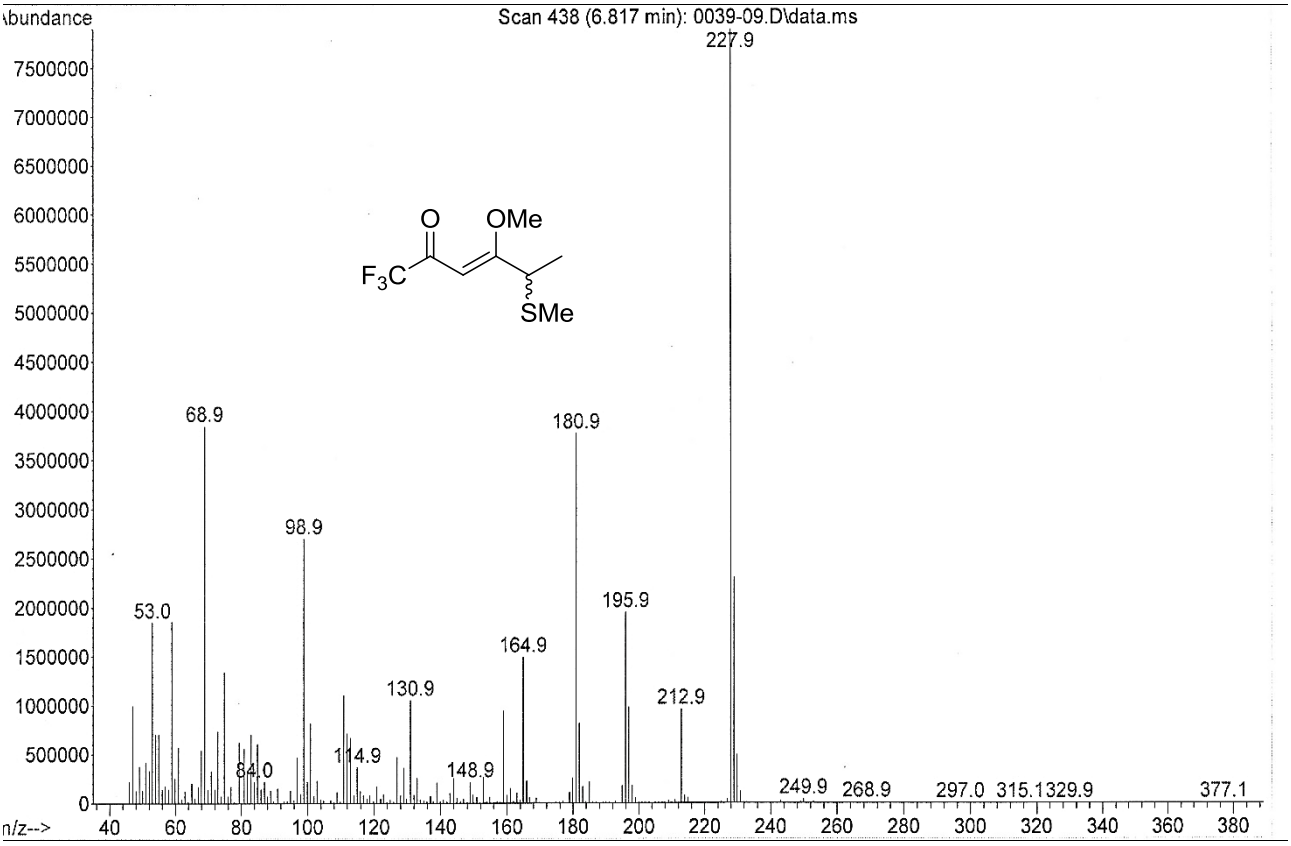


Figure S27. EI mass spectrum (70 eV) of the 1,1,1-trifluoro-4-methoxy-5-thiomethylhex-3-en-2-one (**3k**).

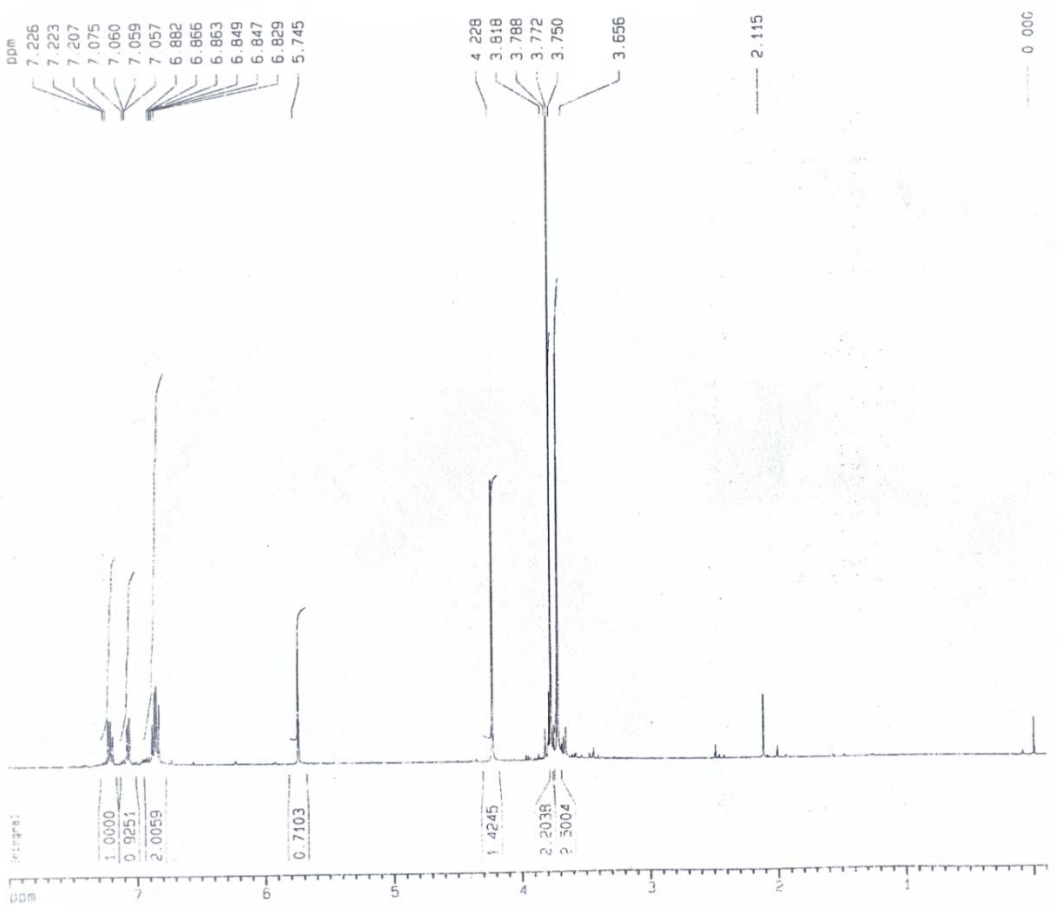


Figure S28. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of the 1,1,1-trifluoro-4-methoxy-5-(2-methoxy phenyl)pent-3-en-2-one (**3l**).

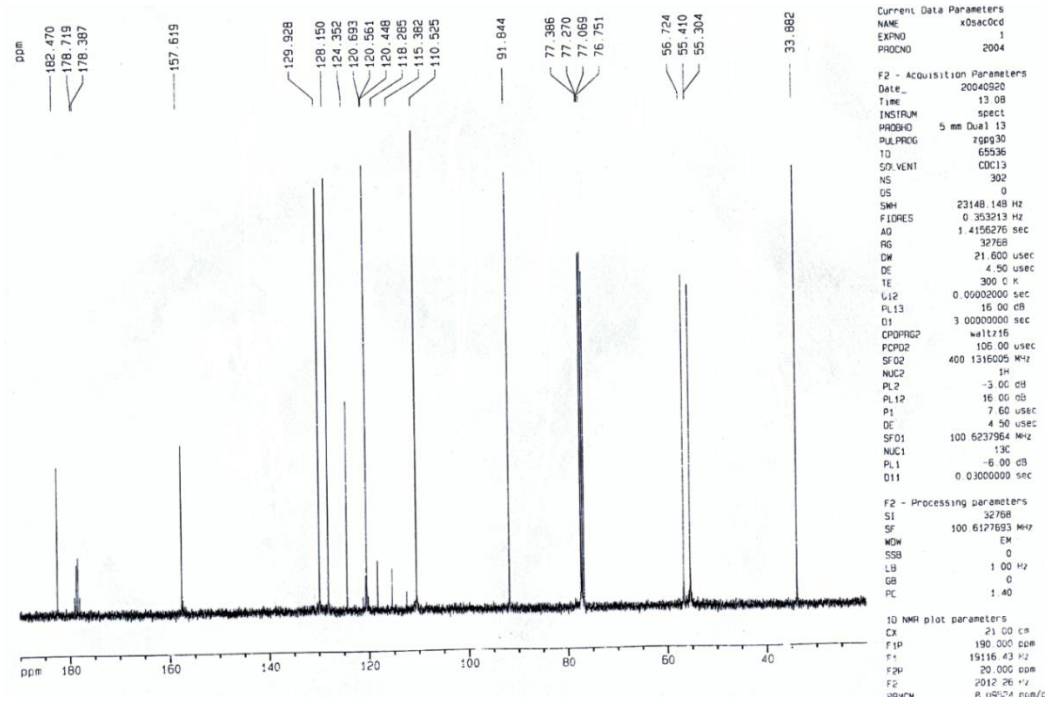


Figure S29. ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of the 1,1,1-trifluoro-4-methoxy-5-(2-methoxy phenyl)pent-3-en-2-one (**3l**).

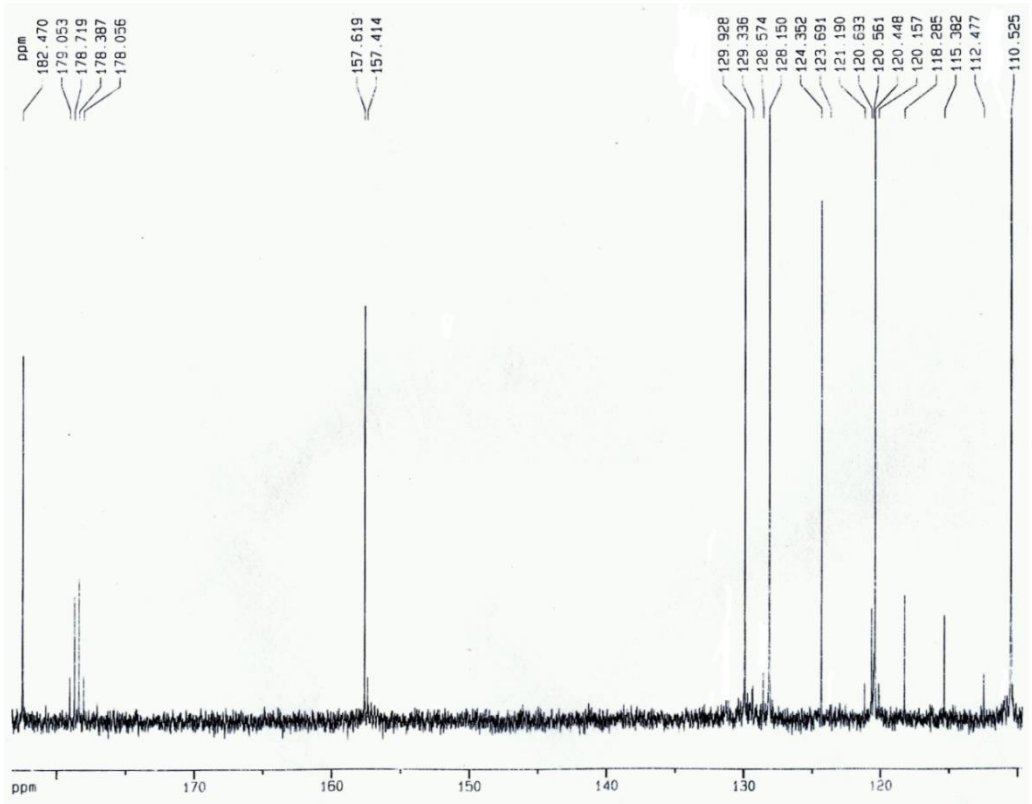


Figure S30. ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of the 1,1,1-trifluoro-4-methoxy-5-(2-methoxy phenyl)pent-3-en-2-one, expanded between 110-185 ppm (**3l**).

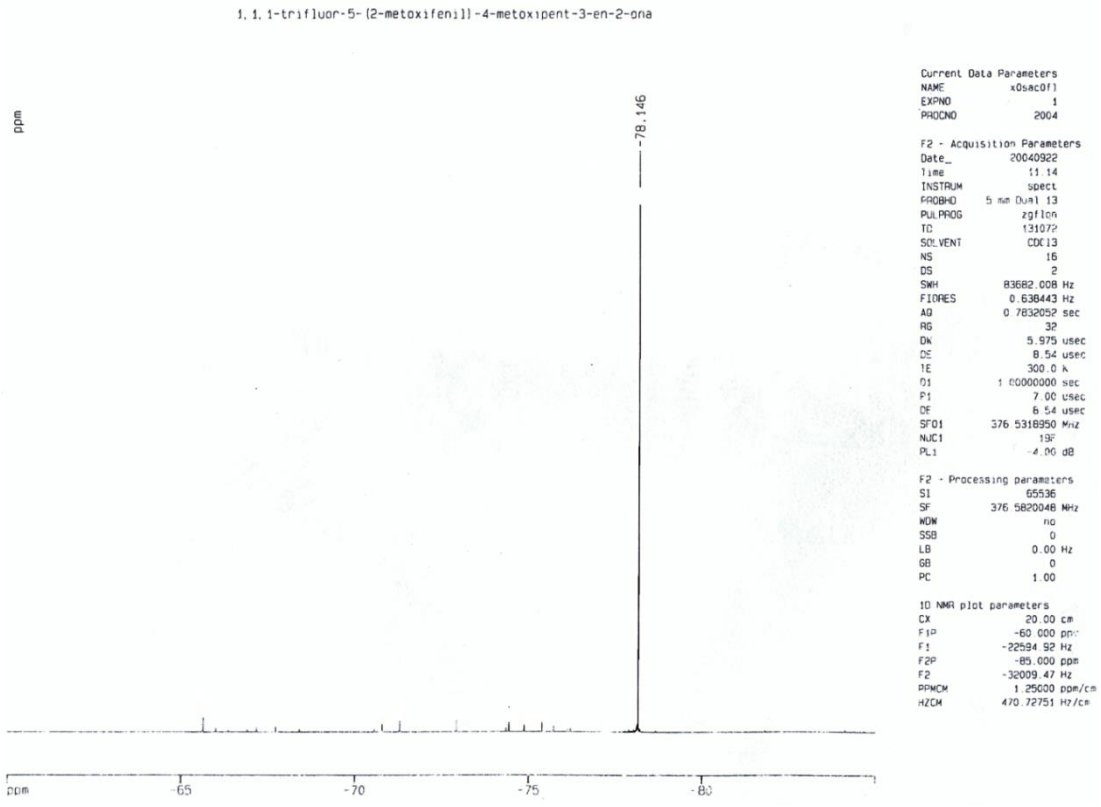


Figure S31. ^{19}F NMR spectrum (100 MHz, $CDCl_3$) of the 1,1,1-trifluoro-4-methoxy-5-(2-methoxy phenyl)pent-3-en-2-one (**3l**).

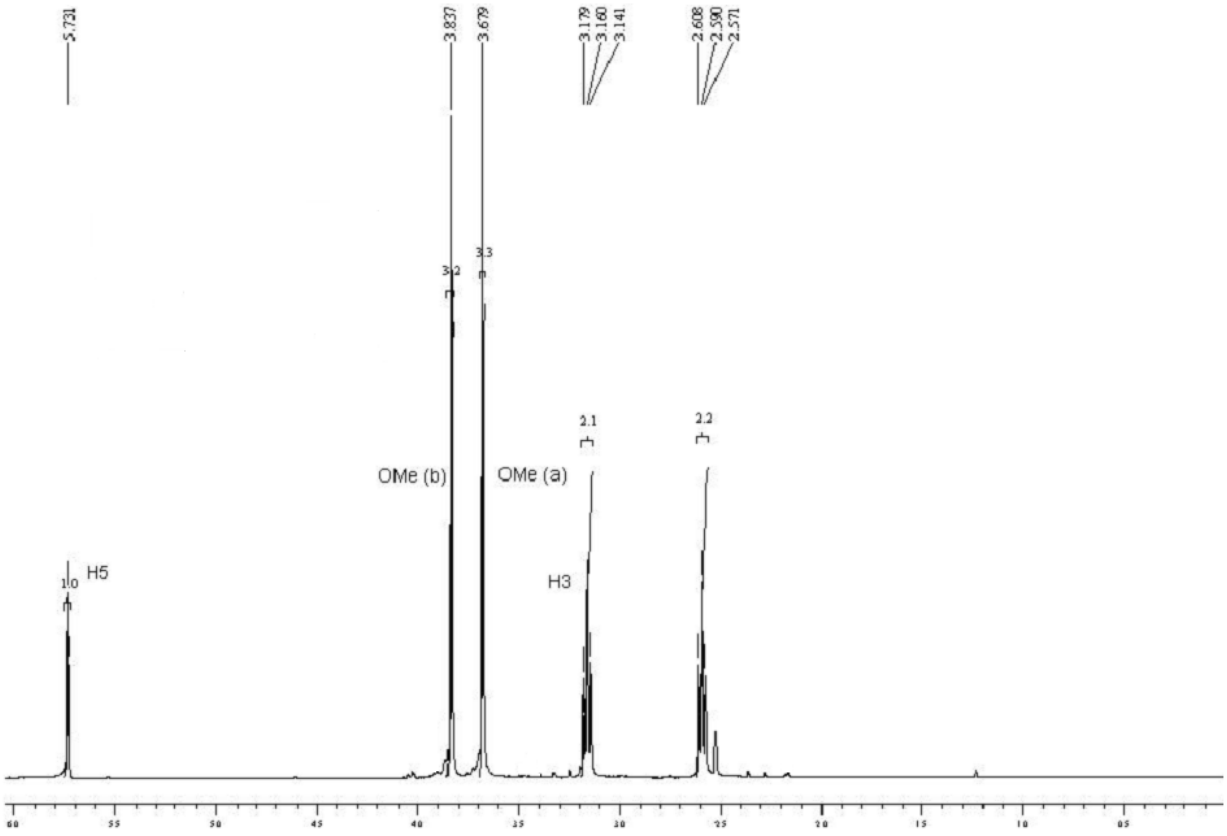


Figure S32. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of the methyl 7,7,7-trifluoro-4-methoxy-6-oxohep-4-enoate (**3m**).

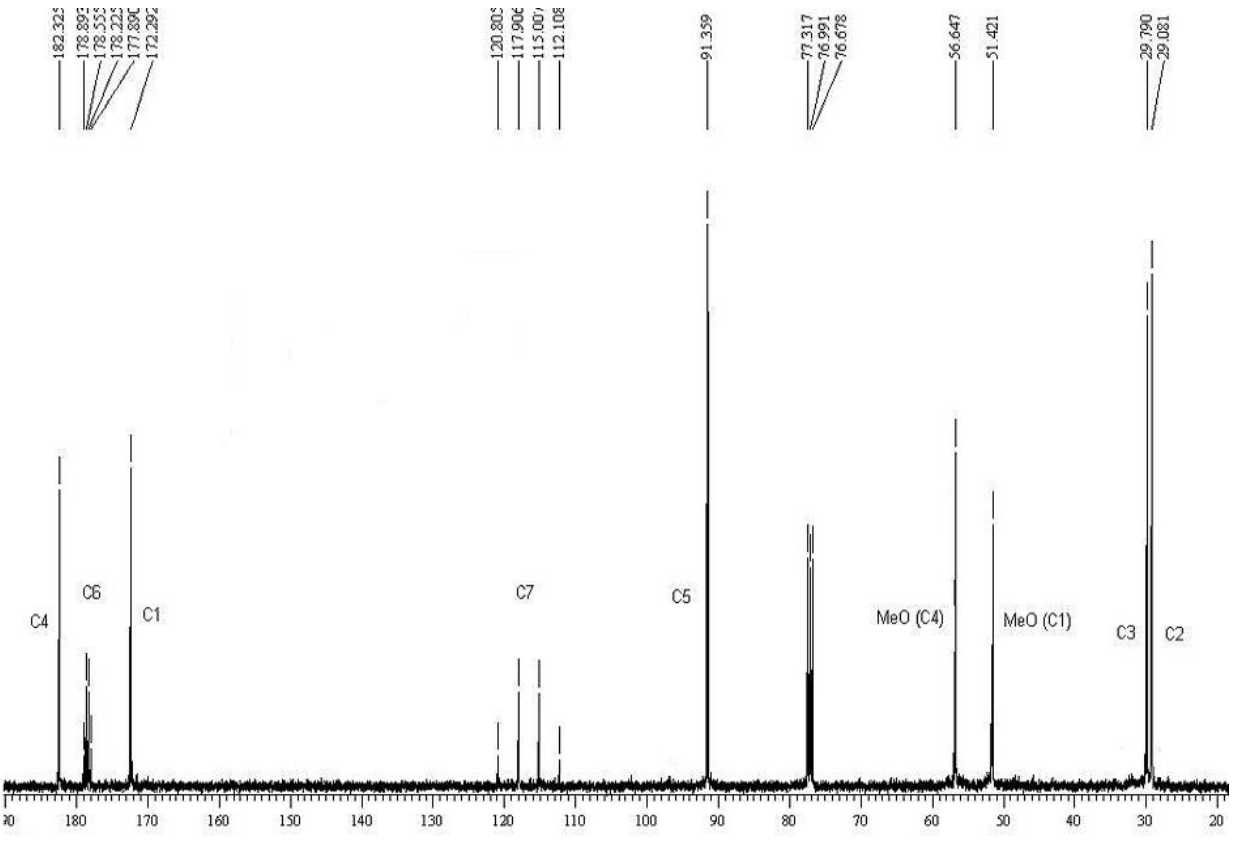


Figure S33. ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of the methyl 7,7,7-trifluoro-4-methoxy-6-oxohept-4-enoate (**3m**).

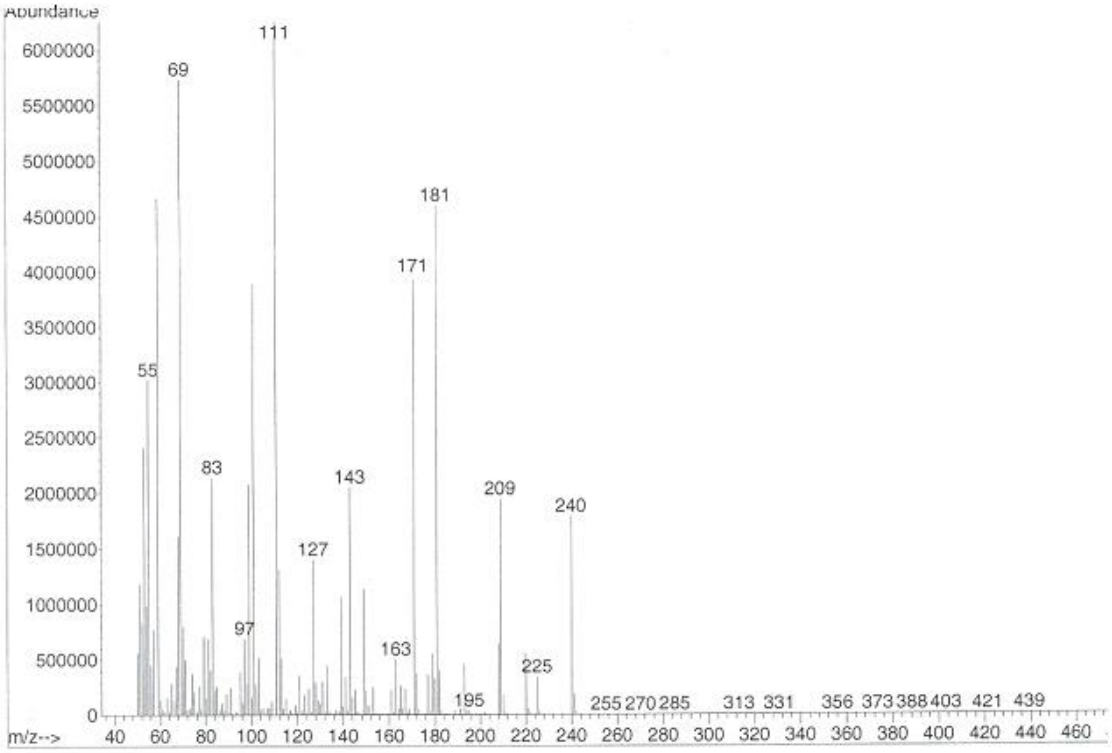


Figure S34. EI Mass spectrum (70 eV) of the methyl 7,7,7-trifluoro-4-methoxy-6-oxohept-4-enoate (**3m**).

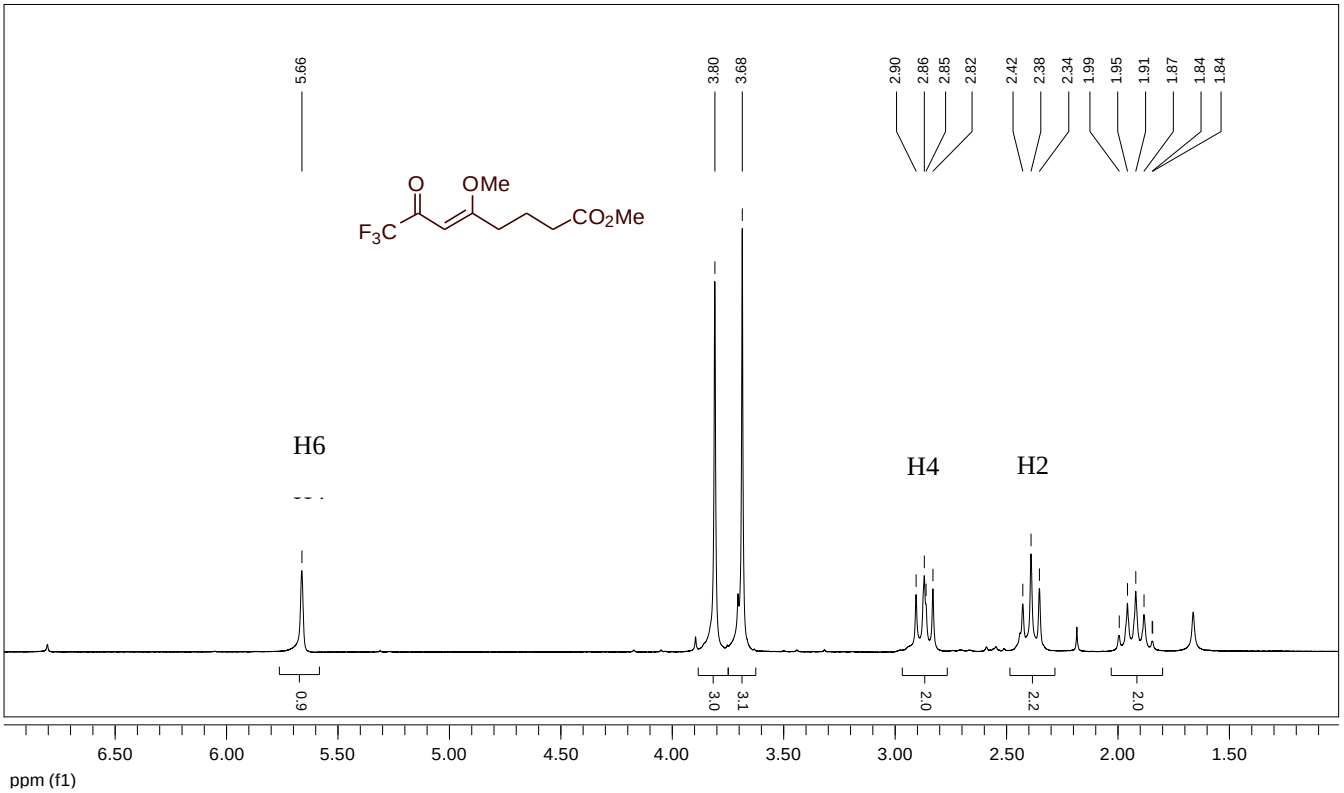


Figure S35. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of the methyl 8,8,8-trifluoro-5-methoxy-7-oxooct-4-enoate (**3m**).

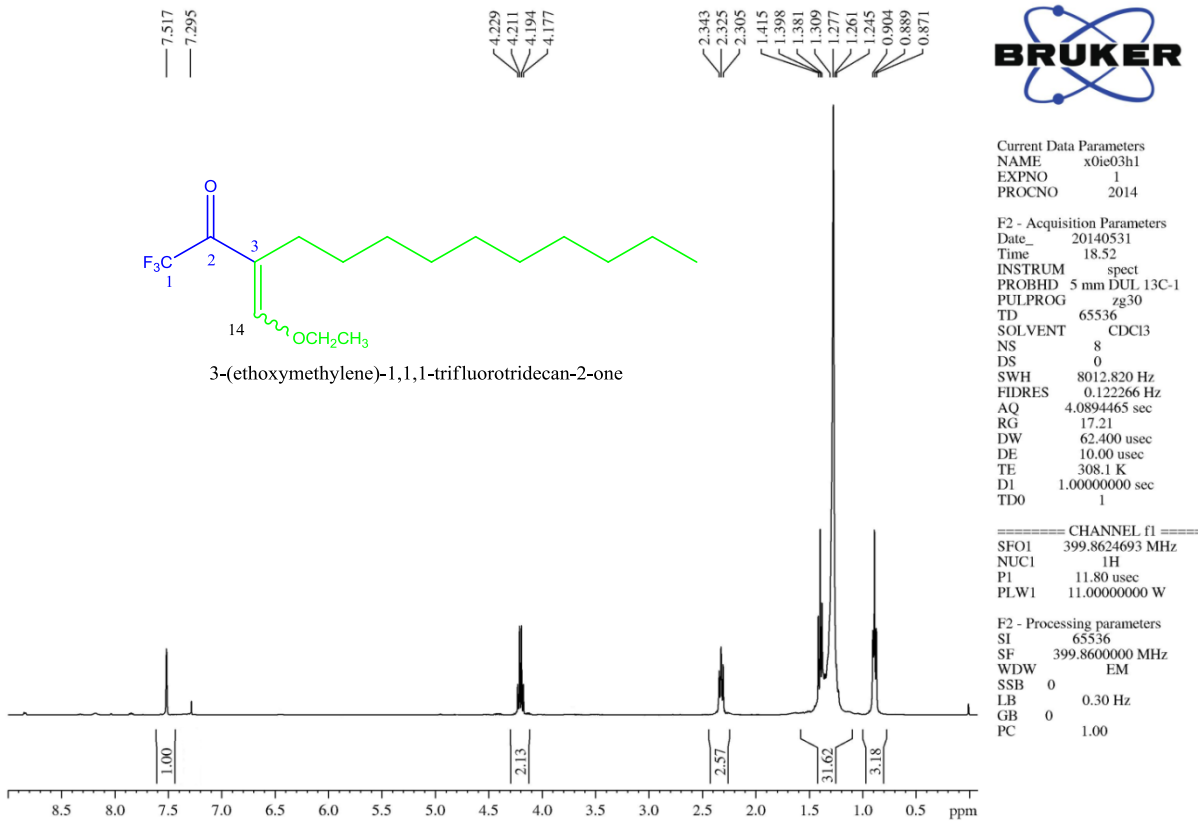


Figure S36. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of the 3-(ethoxymethylene)-1,1,1-trifluorotridecan-2-one (**3v**).

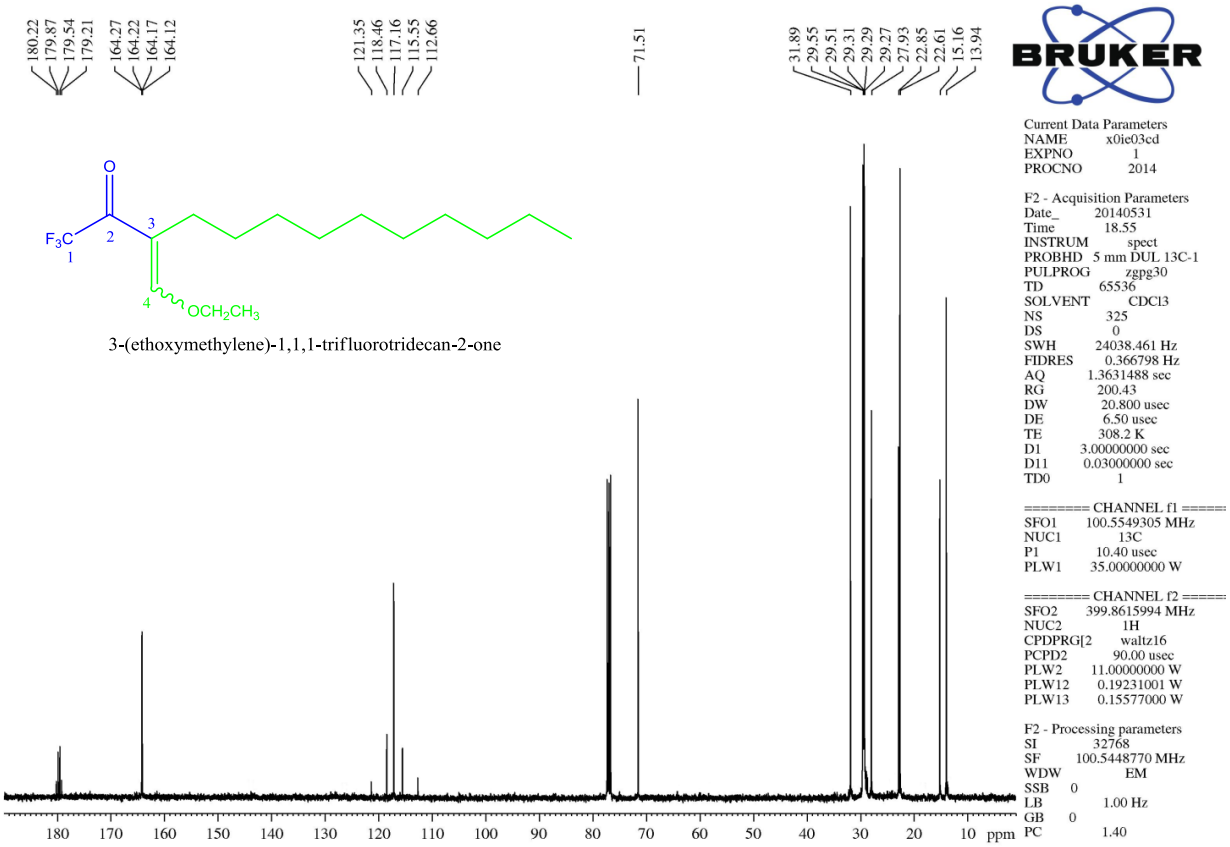


Figure S37. ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of the 3-(ethoxymethylene)-1,1,1-trifluorotridecan-2-one (**3v**).

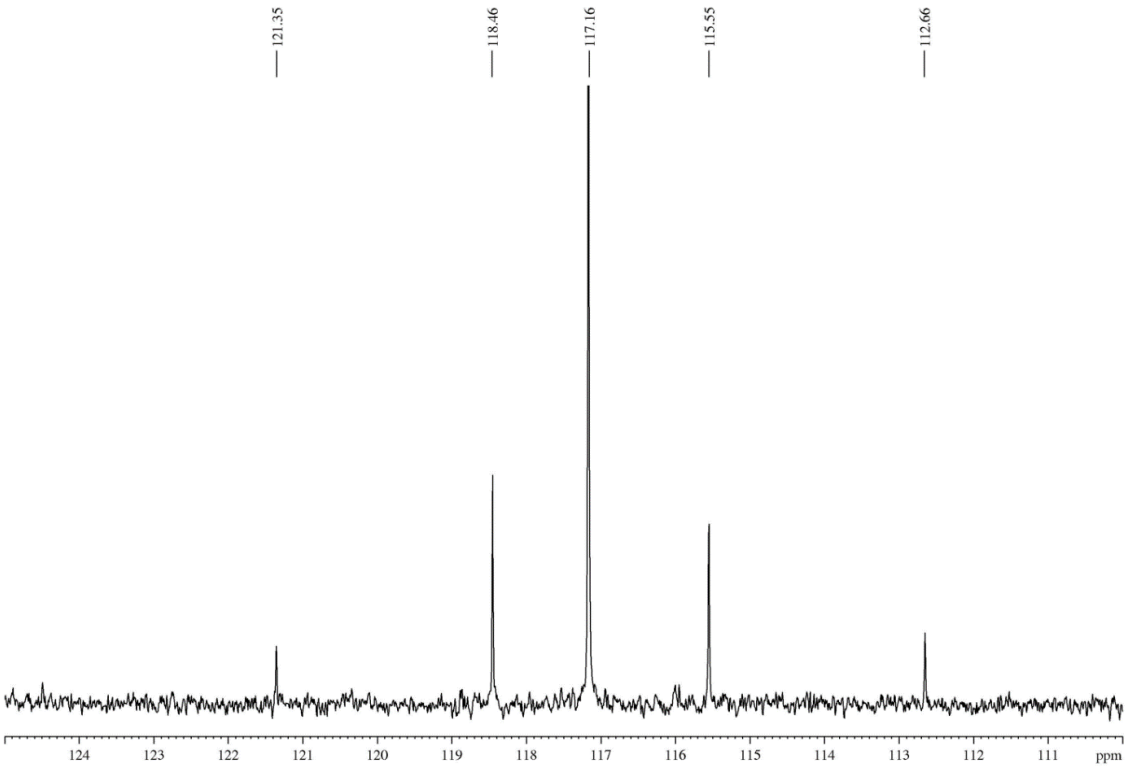


Figure S38. ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of the 3-(ethoxymethylene)-1,1,1-trifluorotridecan-2-one, expanded between 110-125 ppm (**3v**).

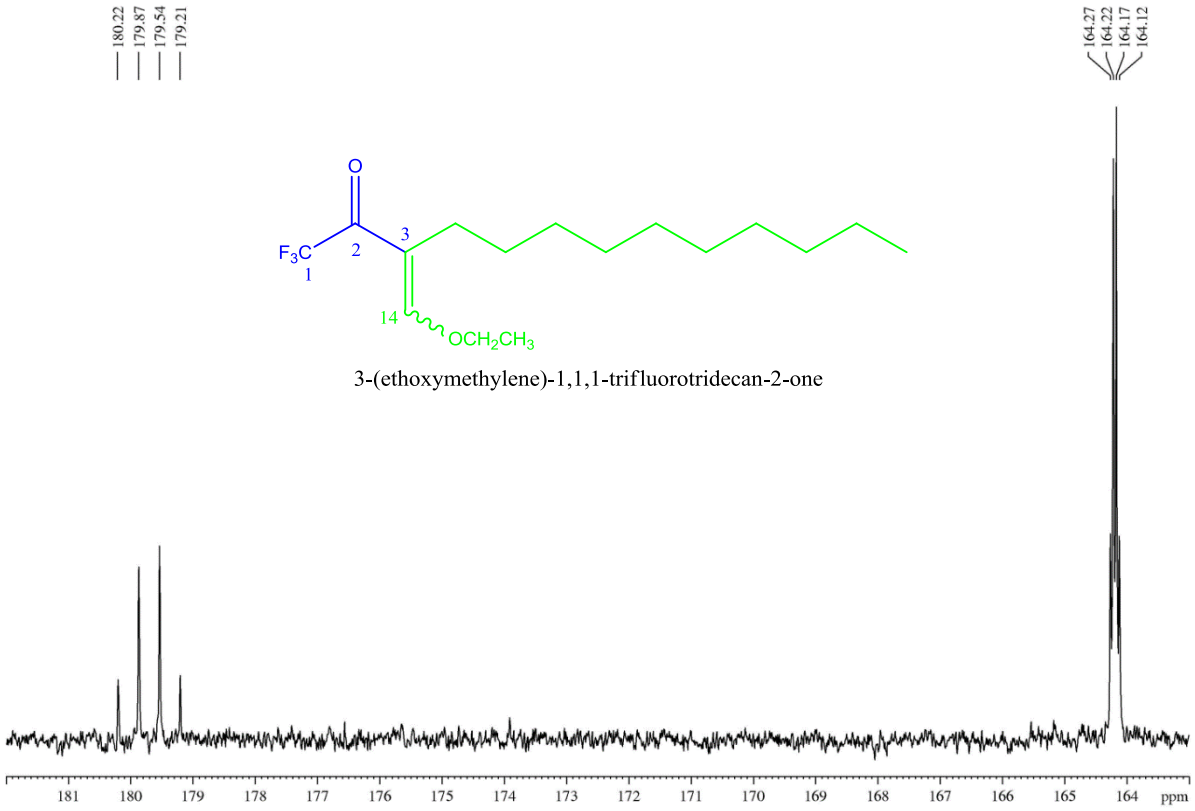


Figure S39. ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of the 3-(ethoxymethylene)-1,1,1-trifluorotridecan-2-one, expanded between 163-182 ppm (**3v**).

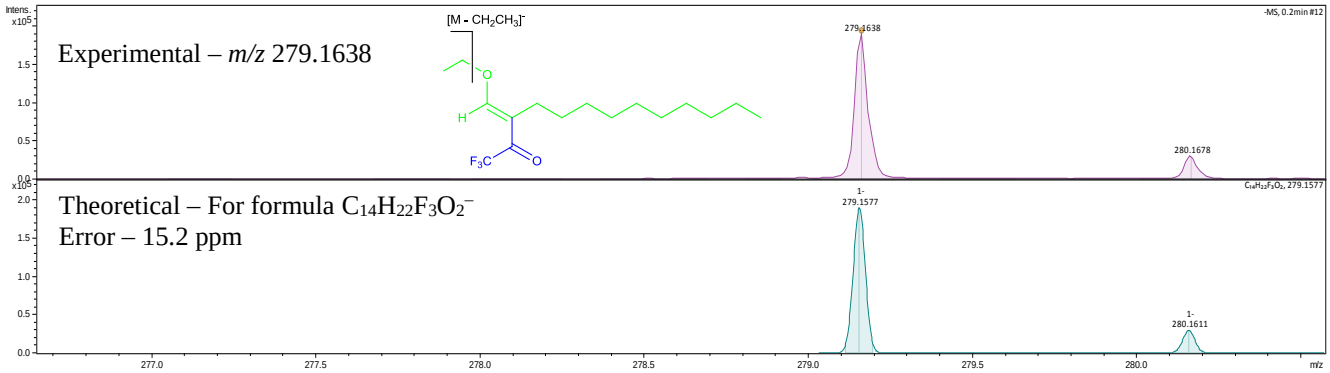


Figure S40. ESI MS spectrum of the 3-(ethoxymethylene)-1,1,1-trifluorotridecan-2-one, experimental and simulated spectra.

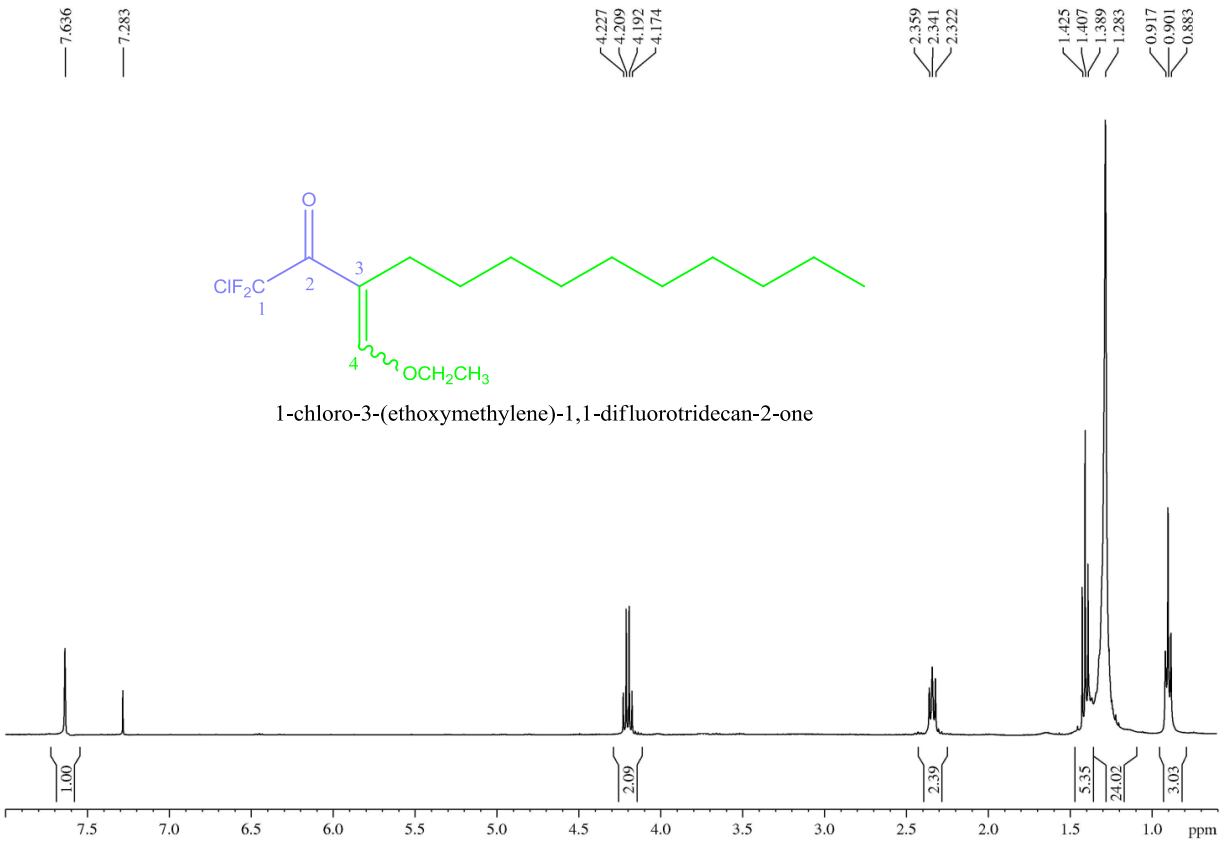


Figure S41. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of the 1-chloro-3-(ethoxymethylene)-1,1-difluorotridecan-2-one (**7v**).

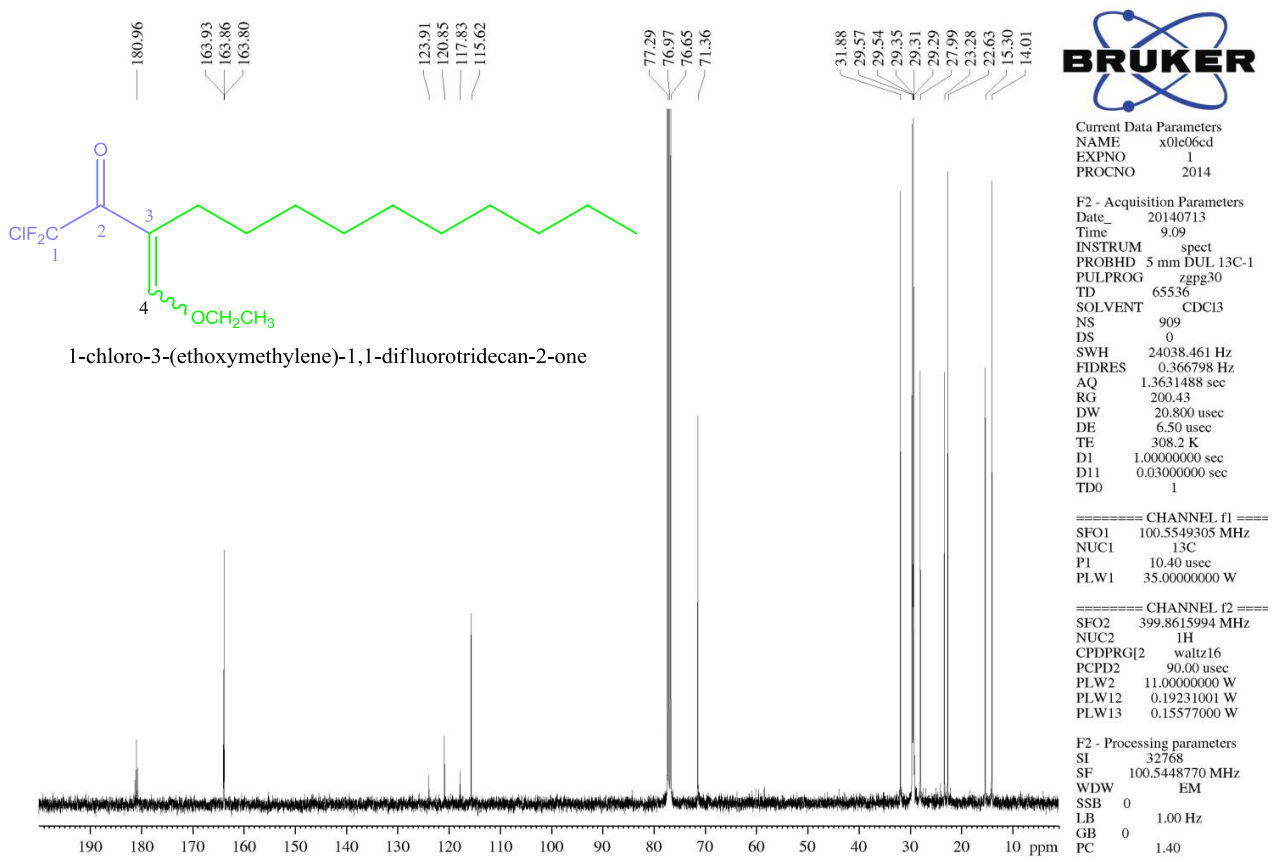


Figure S42. ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of the 1-chloro-3-(ethoxymethylene)-1,1-difluorotridecan-2-one (**7v**).

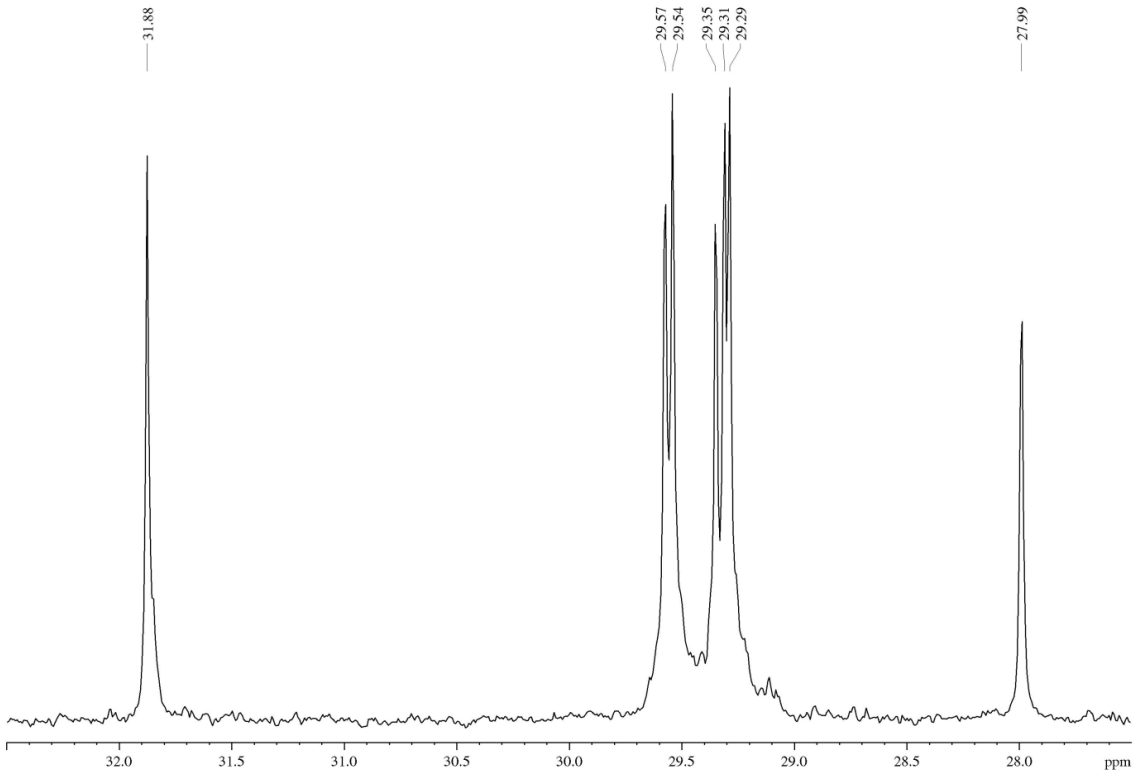


Figure S43. ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of the 1-chloro-3-(ethoxymethylene)-1,1-difluorotridecan-2-one, expanded between 27-33 ppm (**7v**).

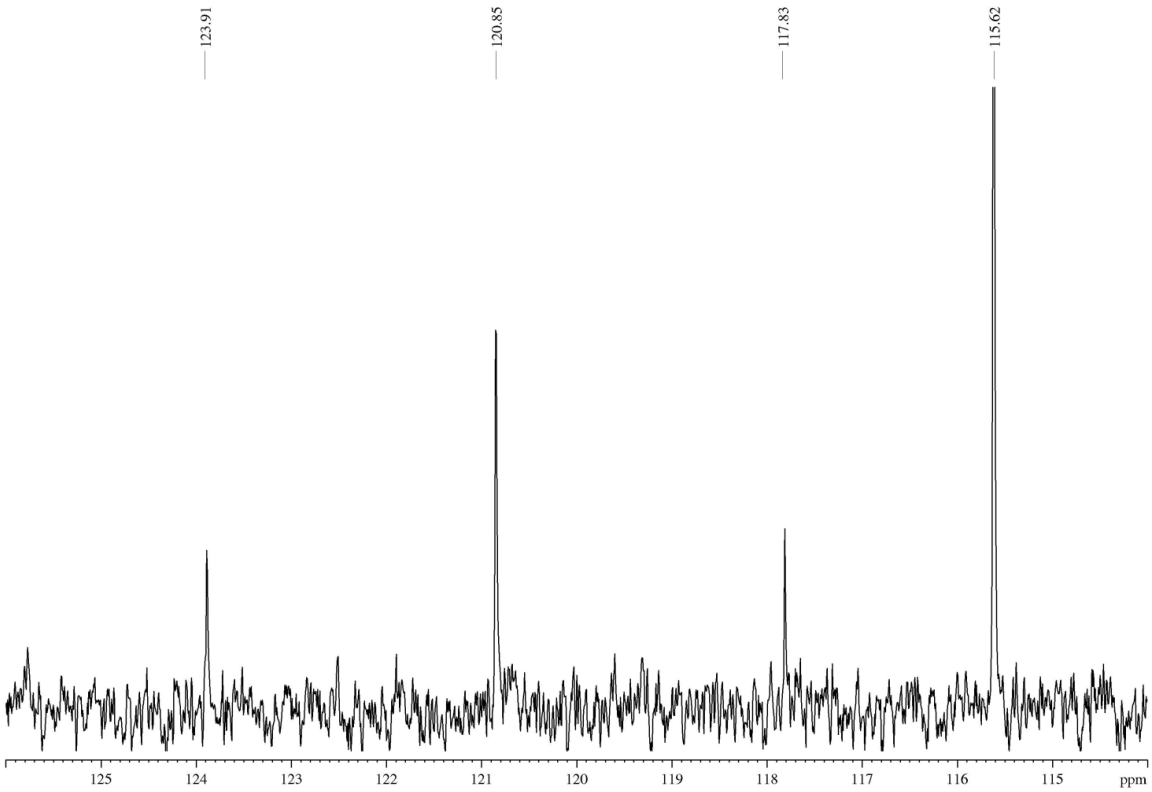


Figure S44. ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of the 1-chloro-3-(ethoxymethylene)-1,1-difluorotridecan-2-one, expanded between 114-126 ppm (**7v**).

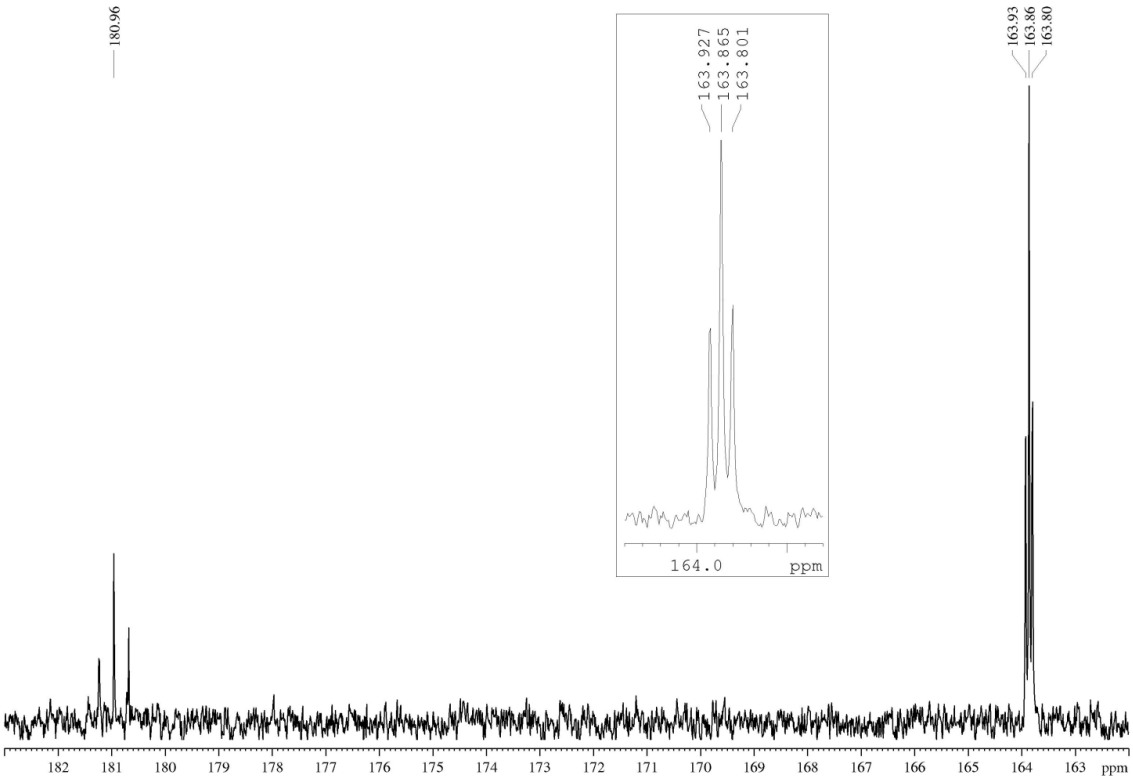


Figure S45. ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of the 1-chloro-3-(ethoxymethylene)-1,1-difluorotridecan-2-one, expanded between 162-183 ppm (**7v**).

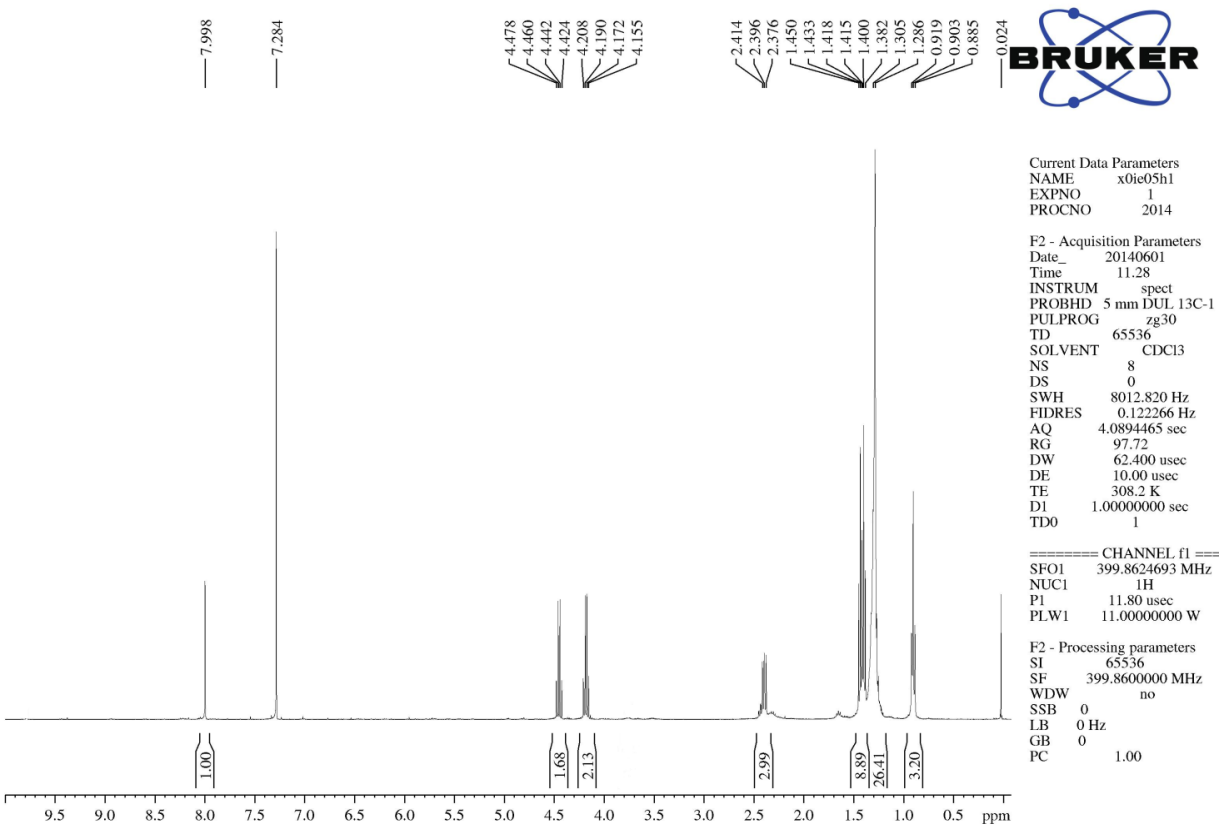


Figure S46. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of the 1,1,1-trichloro-3-(ethoxymethylene)tridecan-2-one (**4v**) and ethyl trichloroacetate.

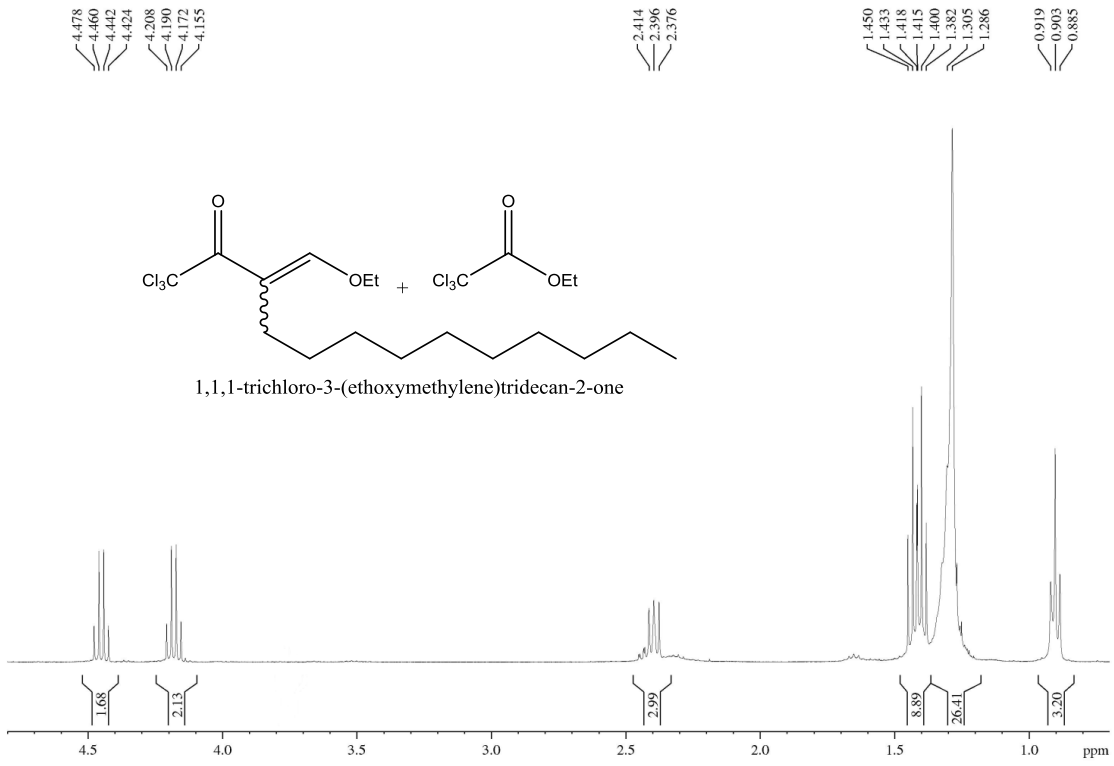


Figure S47. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of the 1,1,1-trichloro-3-(ethoxymethylene)tridecan-2-one (**4v**) and ethyl trichloroacetate.

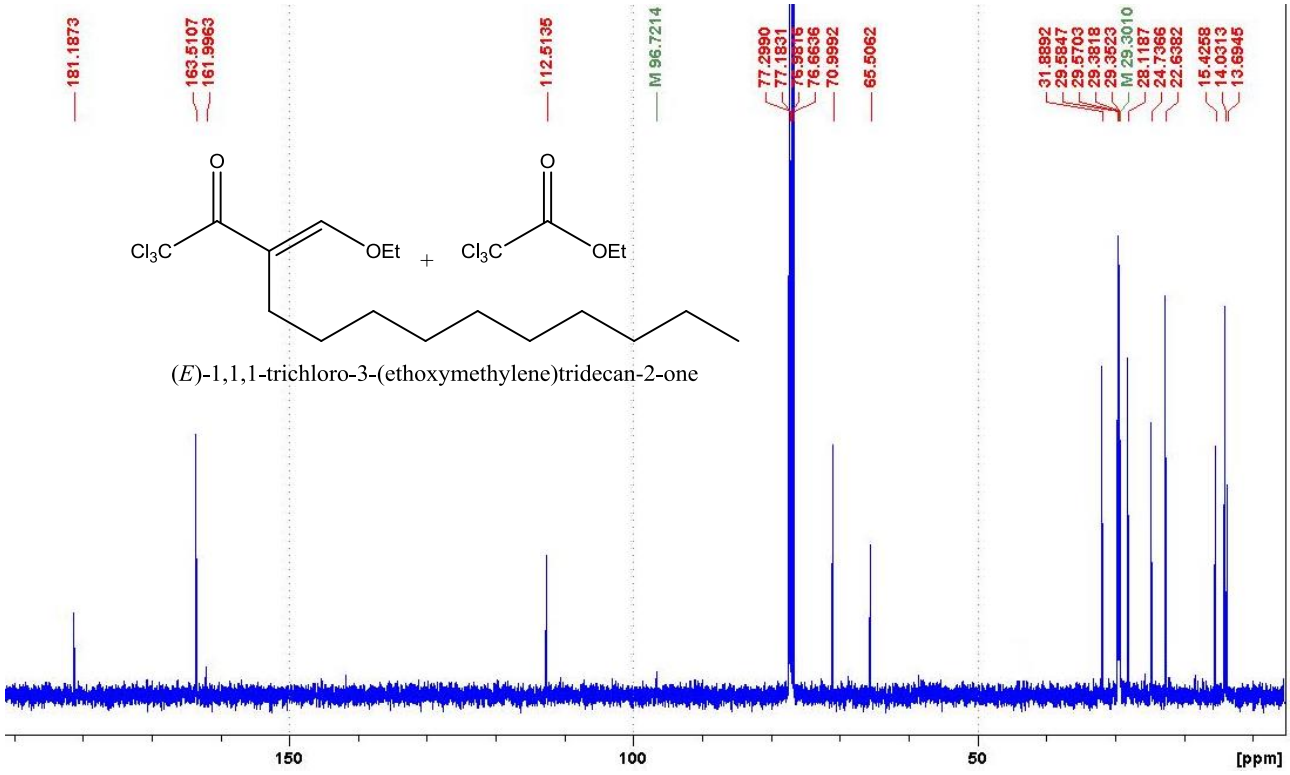


Figure S48. ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of the 1,1,1-trichloro-3-(ethoxymethylene)-tridecan-2-one (**4v**) and ethyl trichloroacetate.

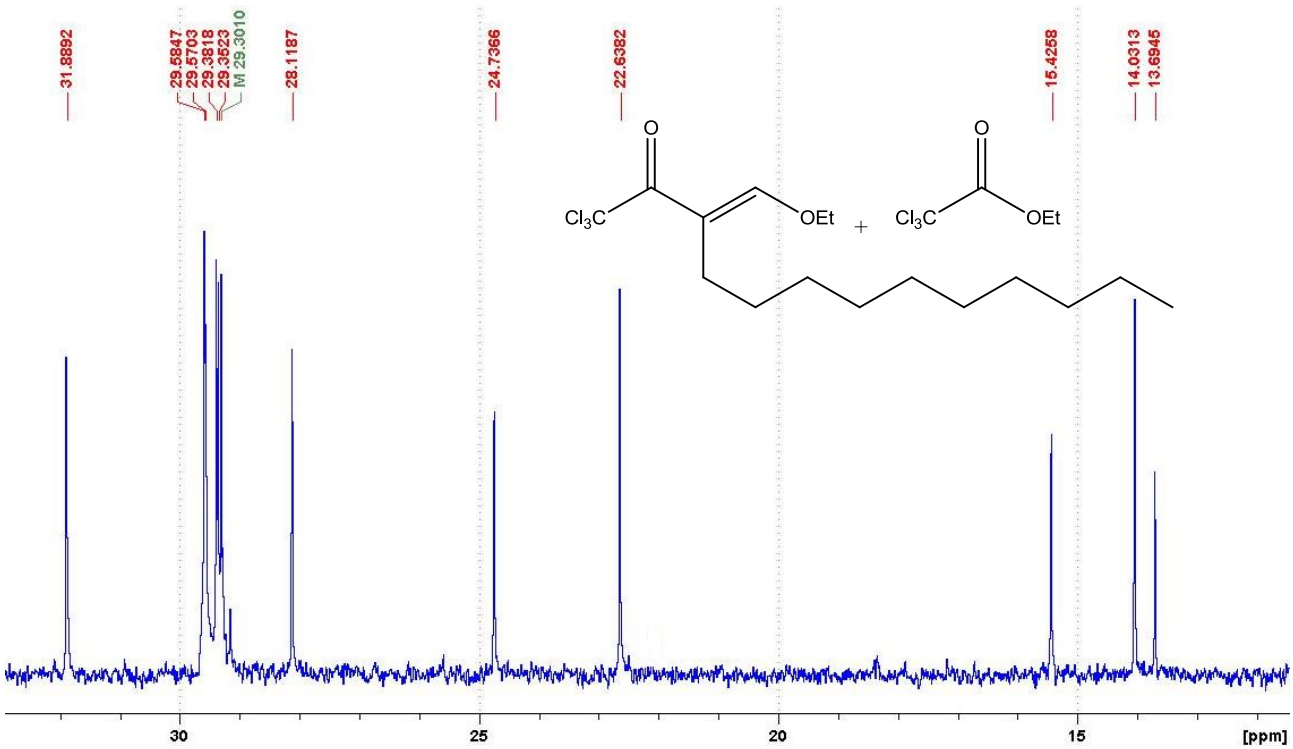


Figure S49. ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of the 1,1,1-trichloro-3-(ethoxymethylene)-tridecan-2-one (**4v**) and ethyl trichloroacetate, expanded between 12-33 ppm.

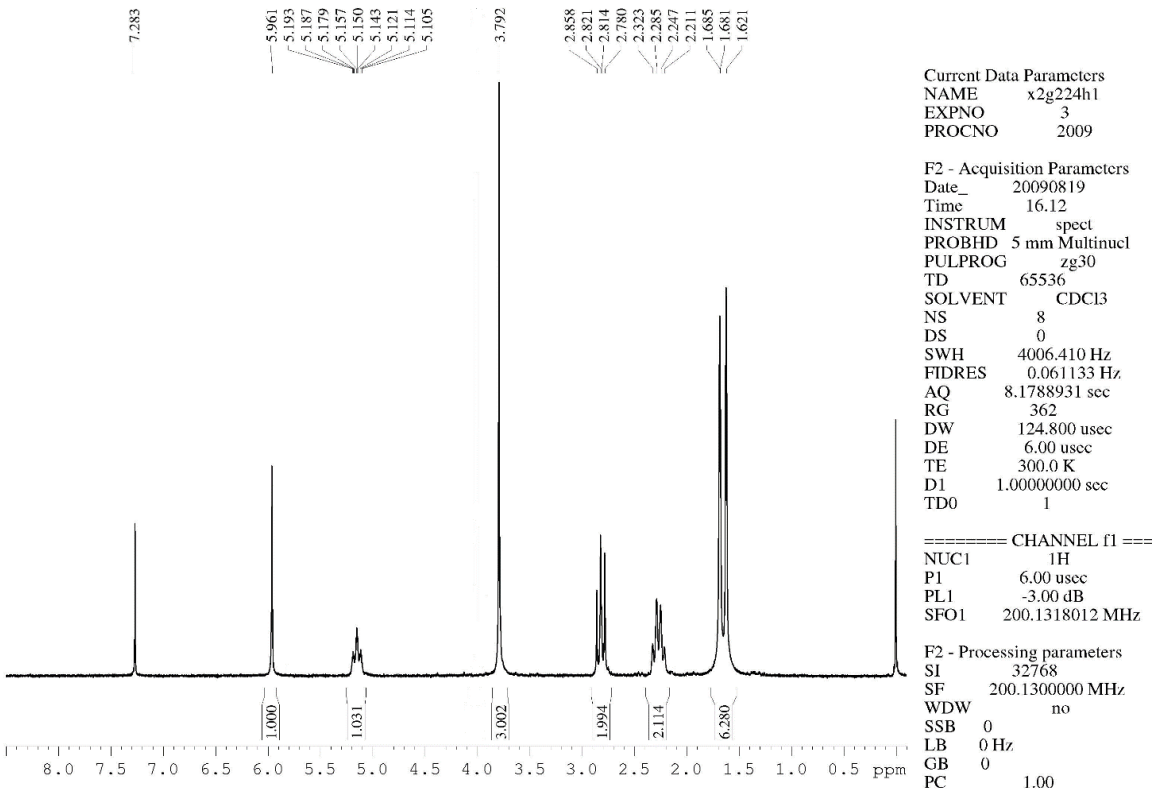


Figure S50. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of the 1,1,1-trichloro-4-methoxy-8-methylnon-3,7-dien-2-one (**4g**).

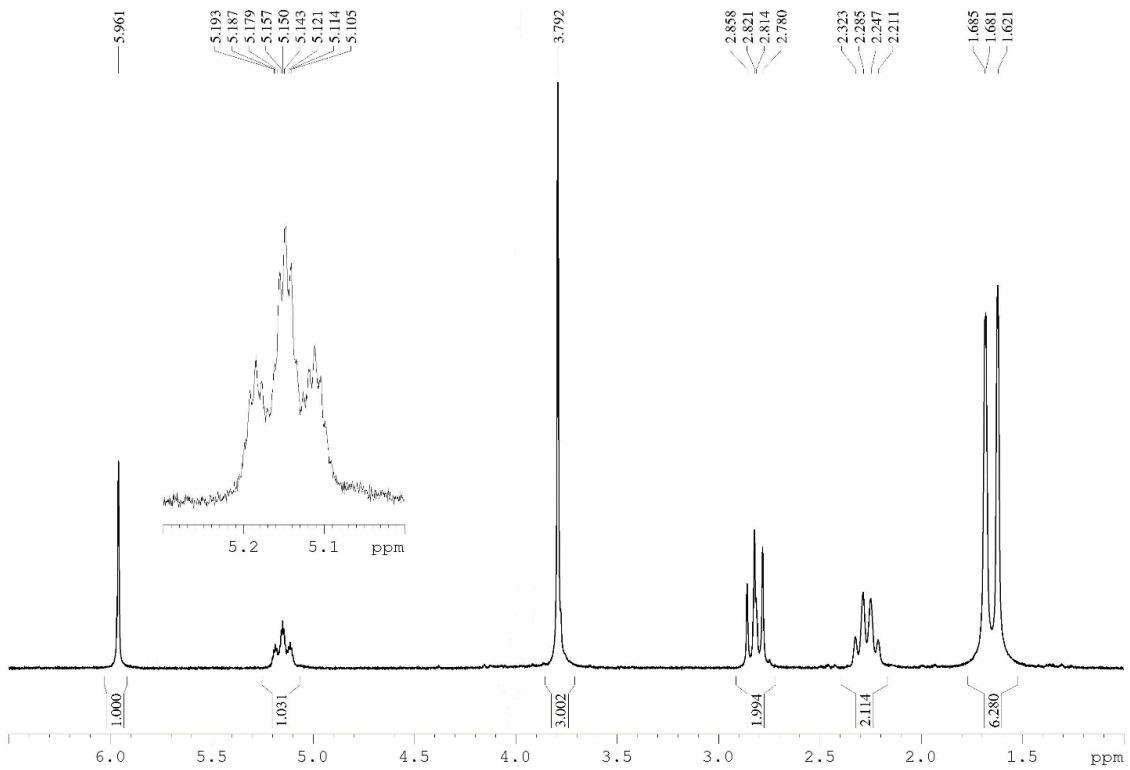


Figure S51. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of the 1,1,1-trichloro-4-methoxy-8-methylnon-3,7-dien-2-one (**4g**), expanded between 5.0-5.3 ppm.

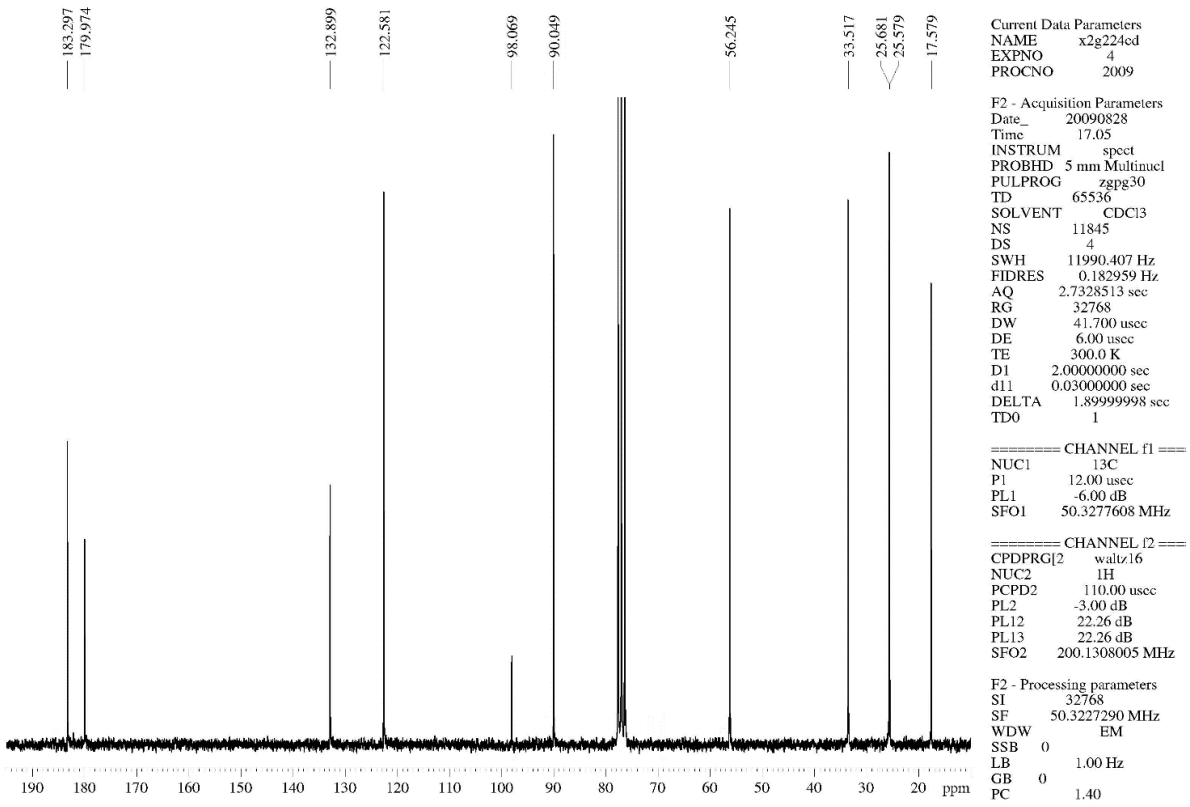


Figure S52. ^{13}C NMR spectrum (400 MHz, $CDCl_3$) of the 1,1,1-trichloro-4-methoxy-8-methylnon-3,7-dien-2-one (**4g**), expanded between 1.0-6.0 ppm.

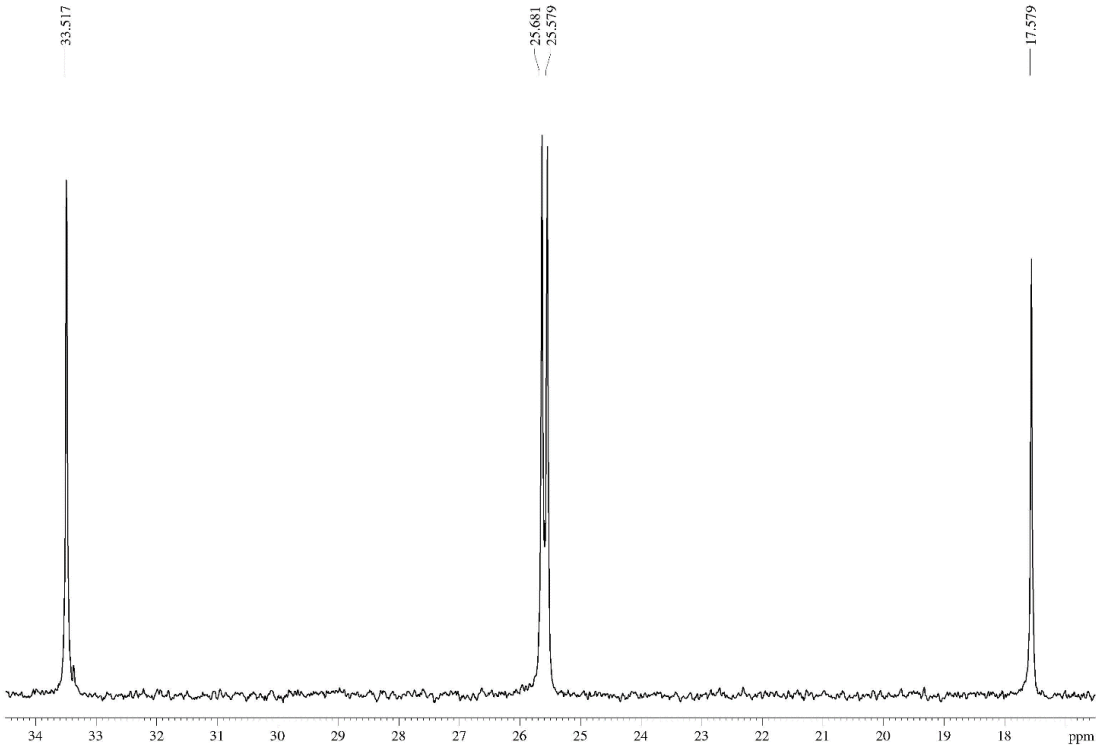


Figure S53. ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of the 1,1,1-trichloro-4-methoxy-8-methylnon-3,7-dien-2-one (**4g**), expanded between 1.0-6.0 ppm.

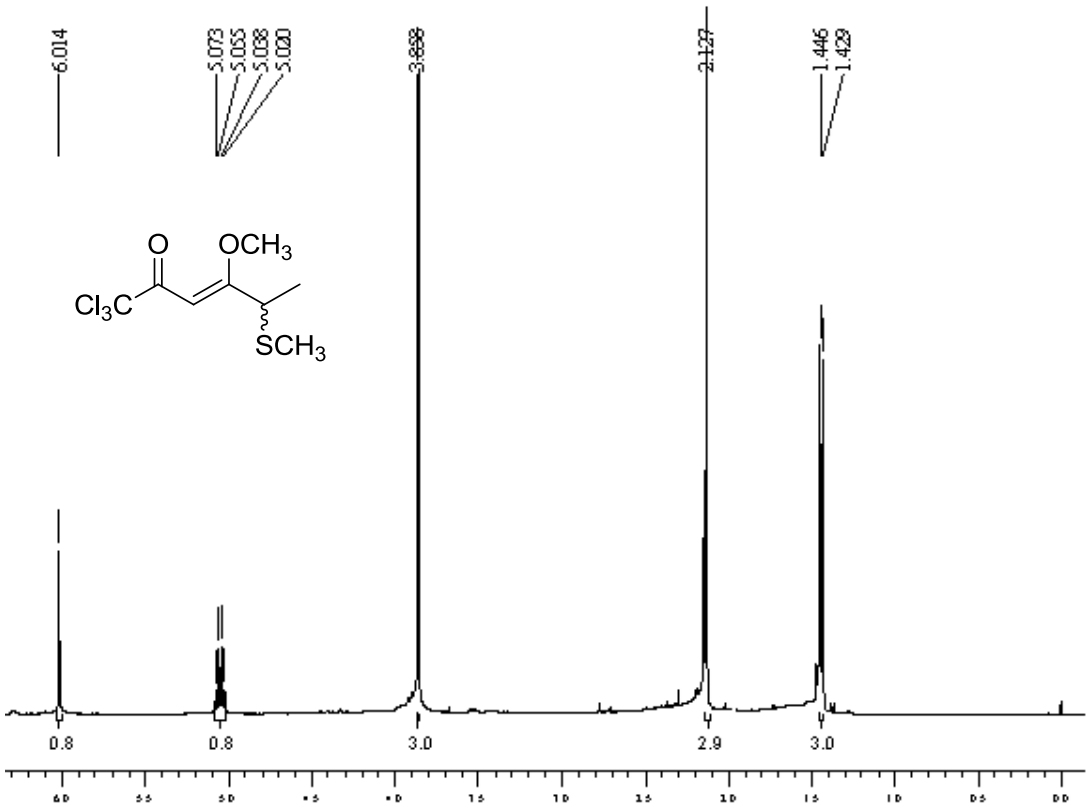


Figure S54. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of the 1,1,1-trichloro-4-methoxy-5-thiomethylhex-3-en-2-one (**4k**).

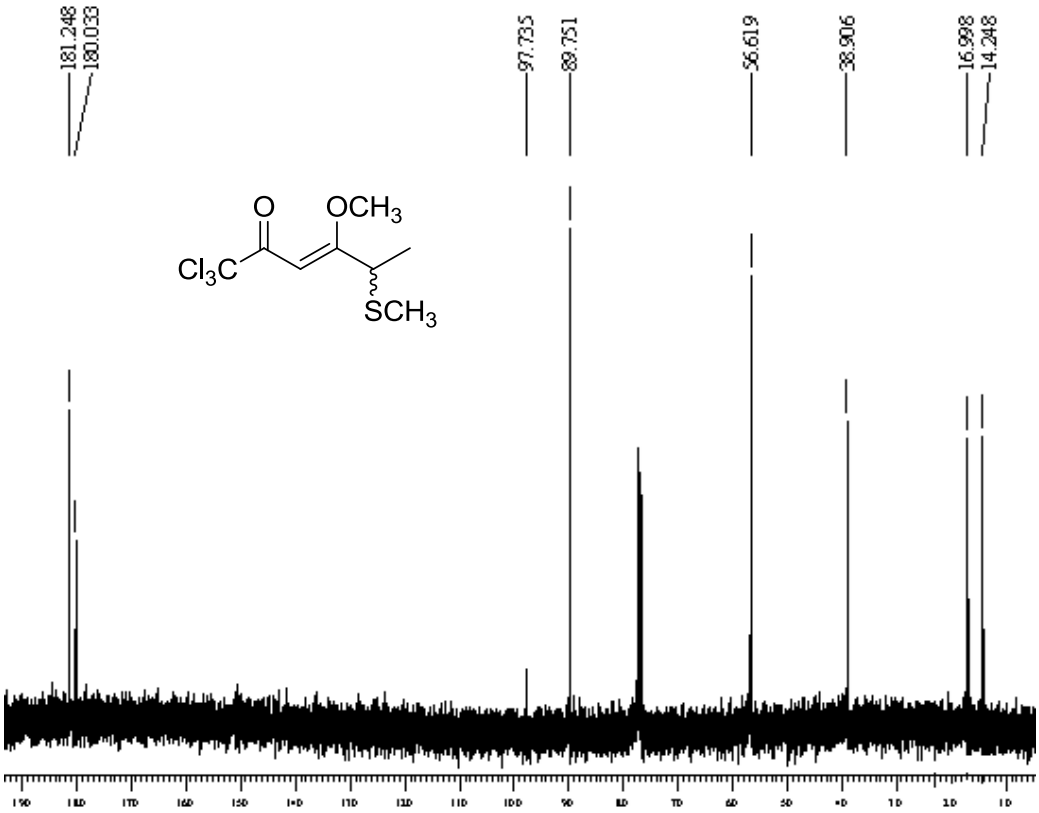


Figure S55. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of the 1,1,1-trichloro-4-methoxy-5-thiomethylhex-3-en-2-one (**4k**).

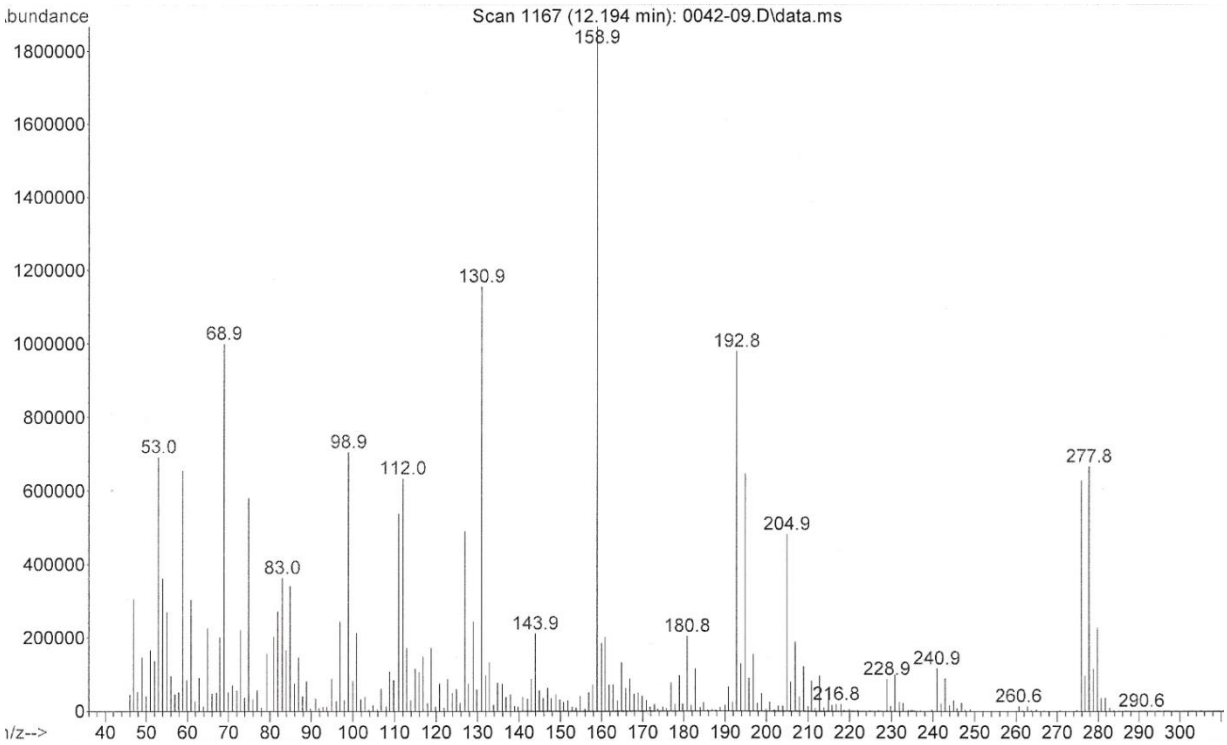


Figure S56. EI mass spectrum (70 eV) of the 1,1,1-trichloro-4-methoxy-5-thiomethylhex-3-en-2-one (**4k**).

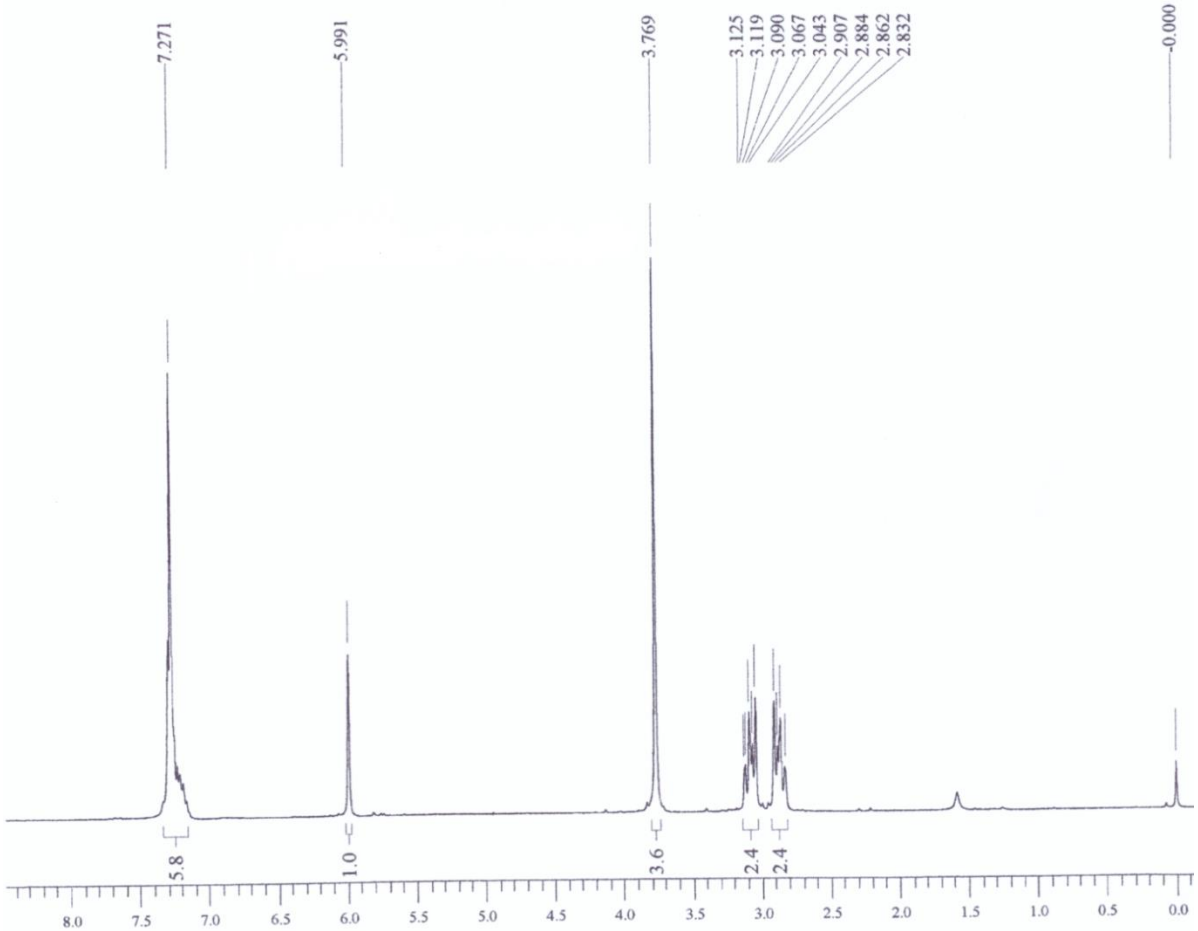


Figure S57. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of the 1,1,1-trichloro-4-methoxy-6-phenylhex-3-en-2-one (**4h**).

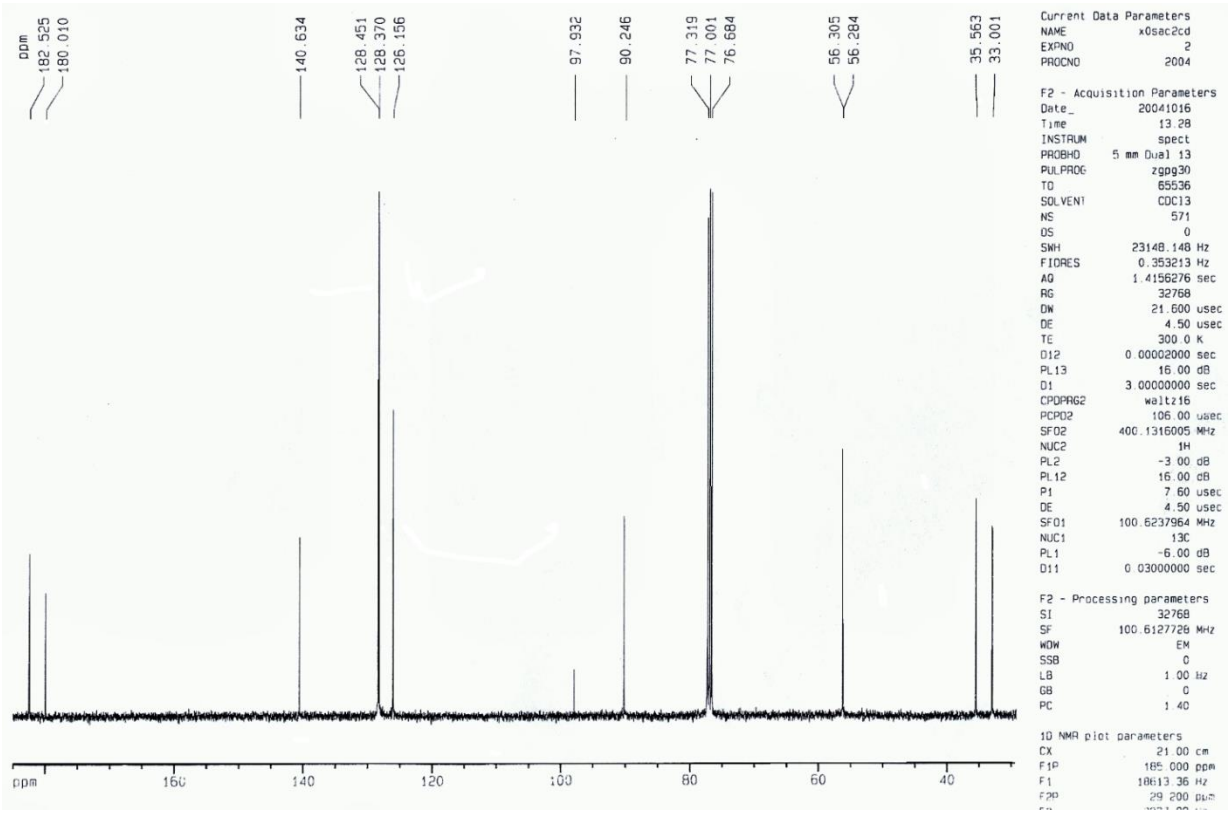


Figure S58. ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of the 1,1,1-trichloro-4-methoxy-6-phenylhex-3-en-2-one (**4h**).

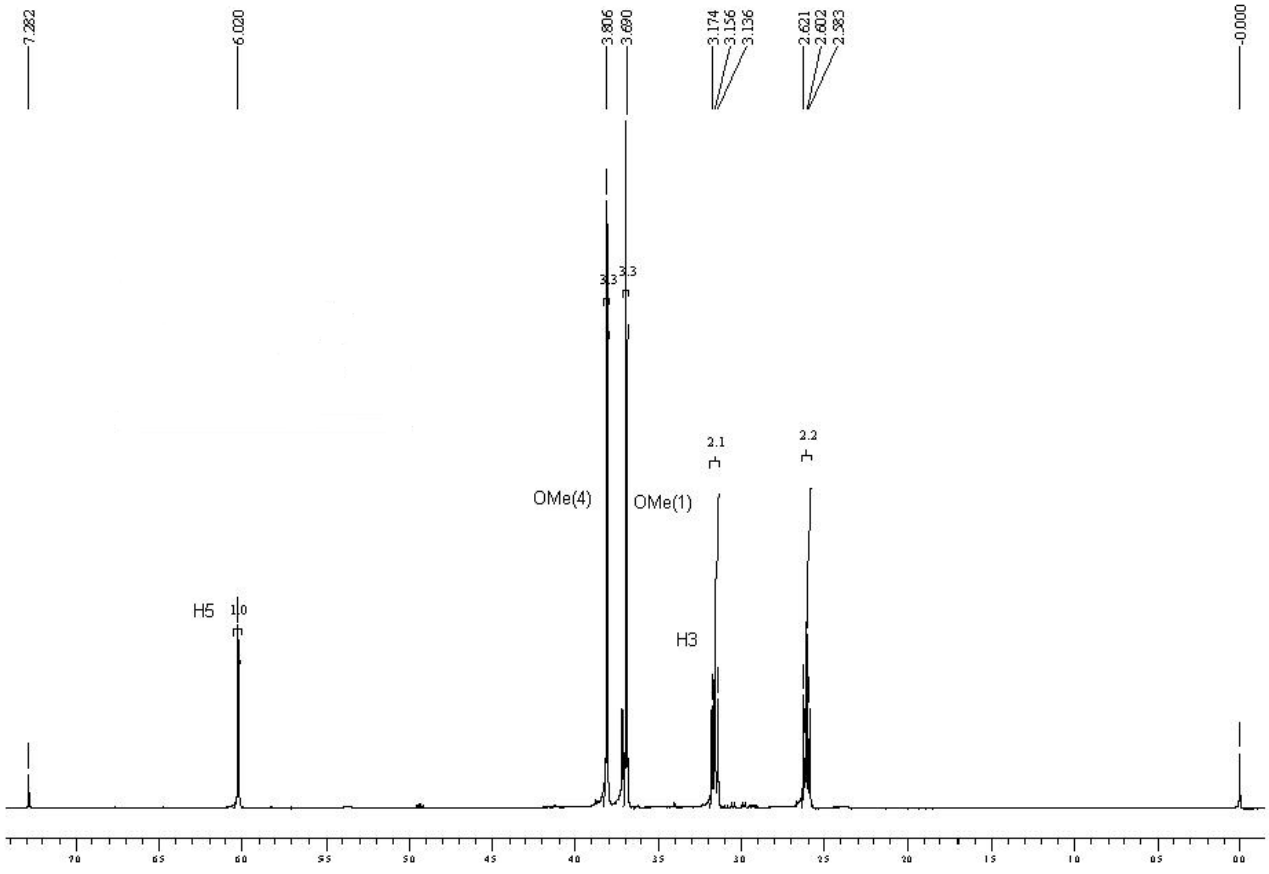


Figure S59. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of the methyl 7,7,7-trichloro-4-methoxy-6-oxohept-4-enoate (**4m**).

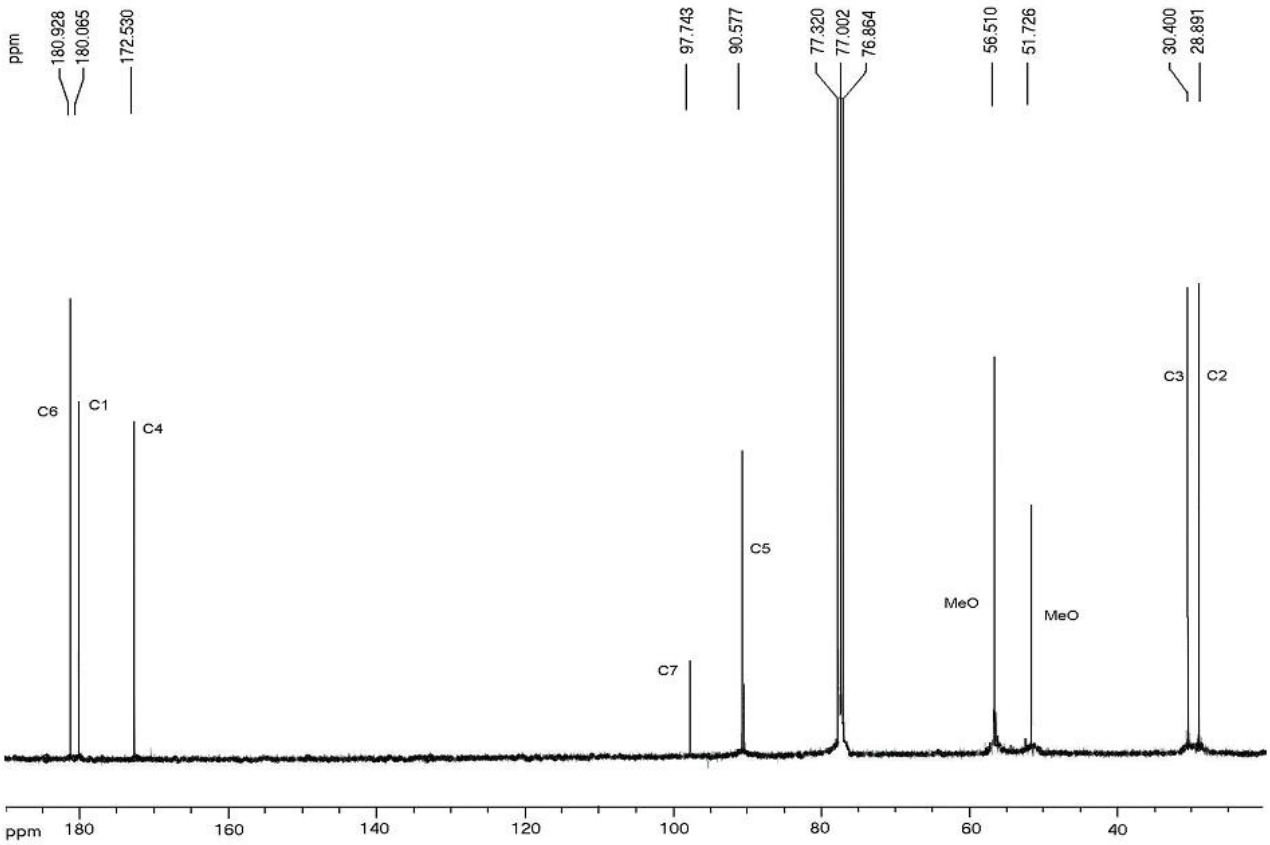


Figure S60. ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of the methyl 7,7,7-trichloro-4-methoxy-6-oxohept-4-enoate (**4m**).

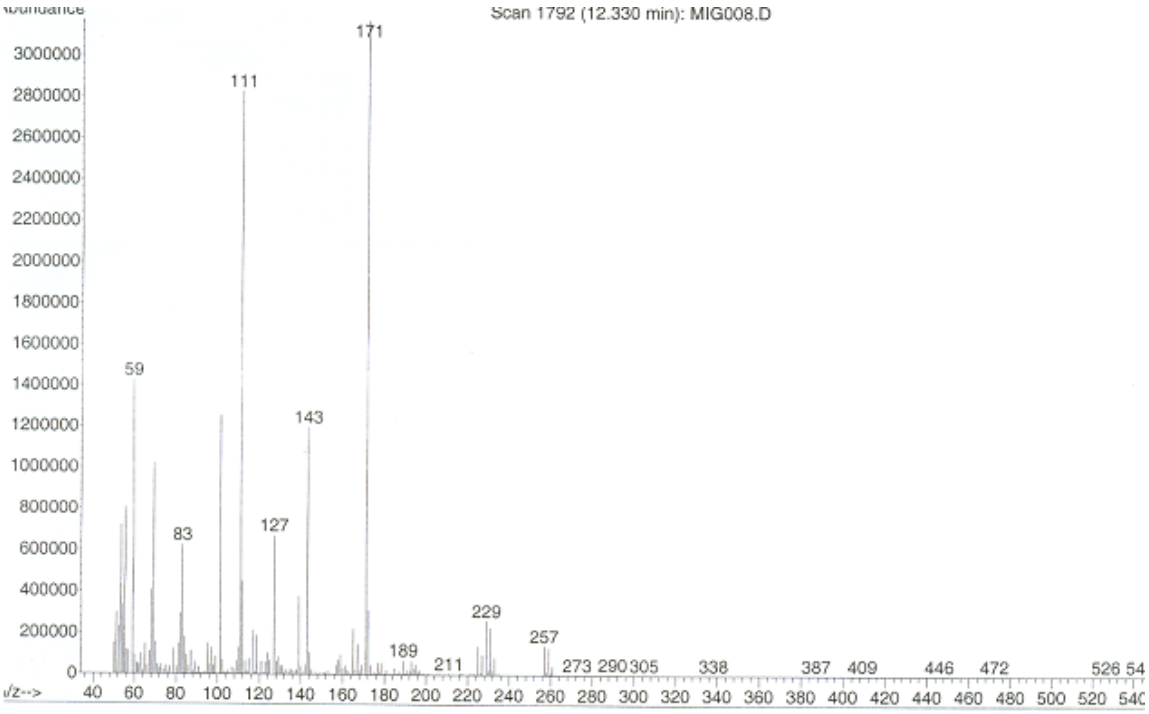


Figure S61. EI mass spectrum (70 eV) of the methyl 7,7,7-trichloro-4-methoxy-6-oxohept-4-enoate (**4n**).

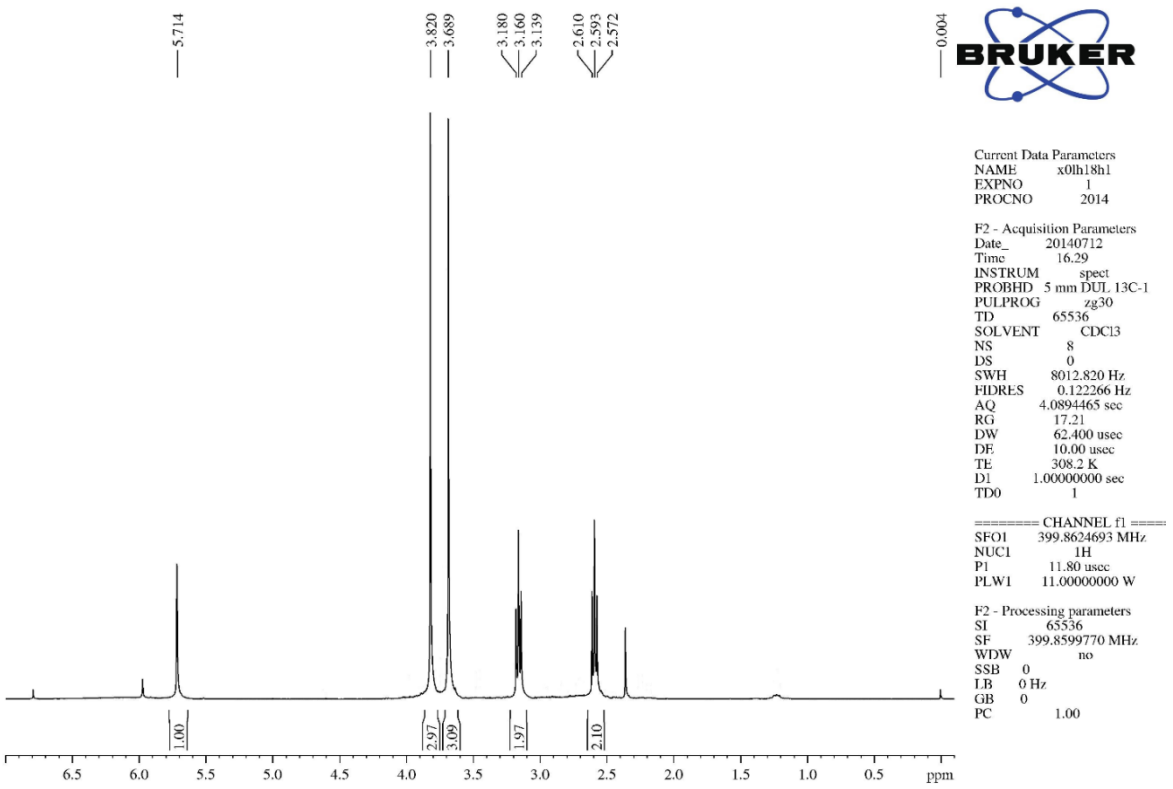


Figure S62. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of the methyl 7-chloro-7,7-difluoro-4-methoxy-6-oxohept-4-enoate (**7m**).

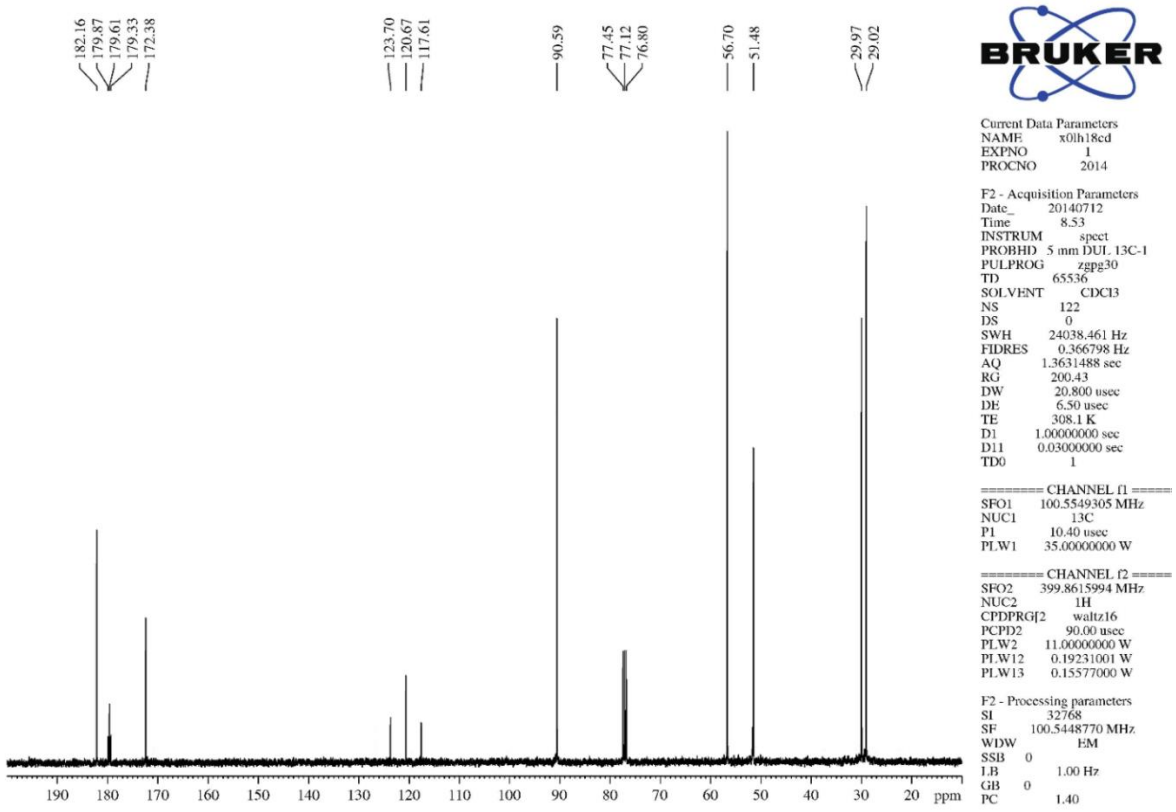


Figure S63. ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of the methyl 7-chloro-7,7-difluoro-4-methoxy-6-oxohept-4-enoate (**7m**).

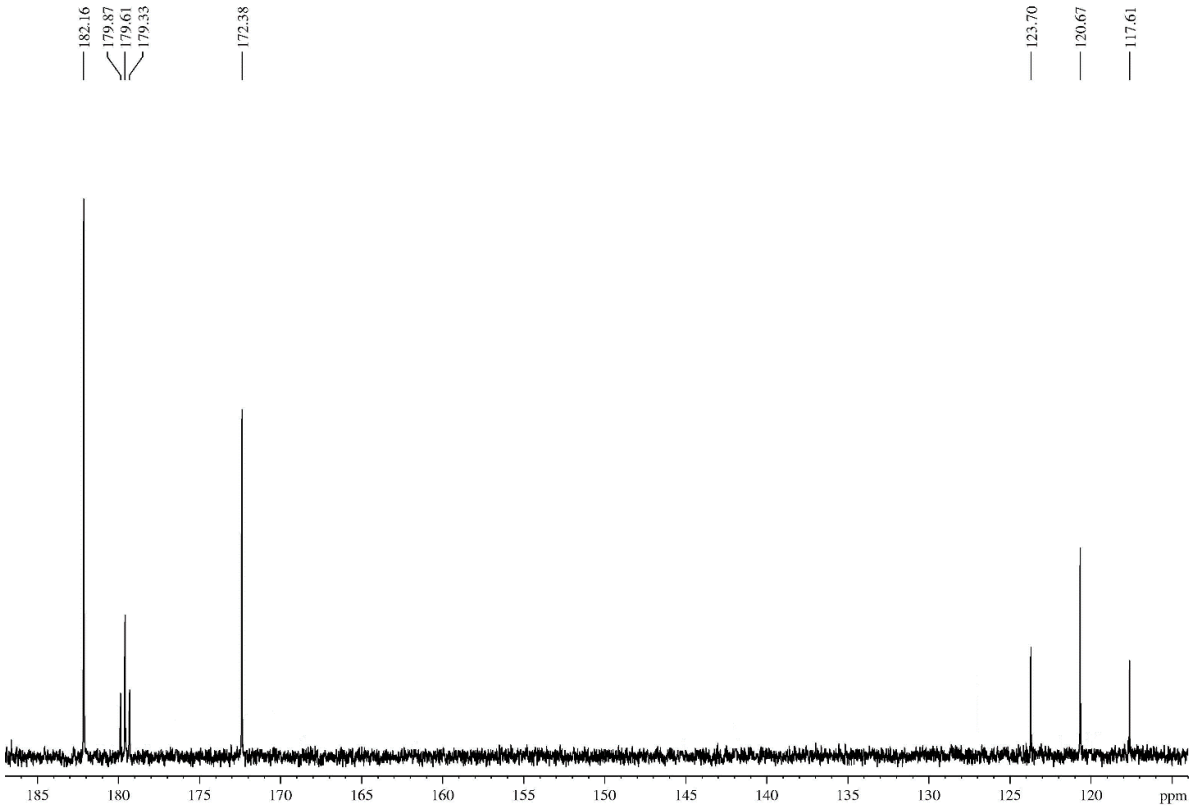


Figure S64. ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of the methyl 7-chloro-7,7-difluoro-4-methoxy-6-oxohept-4-enoate (**7m**), expanded between 114-187 ppm.

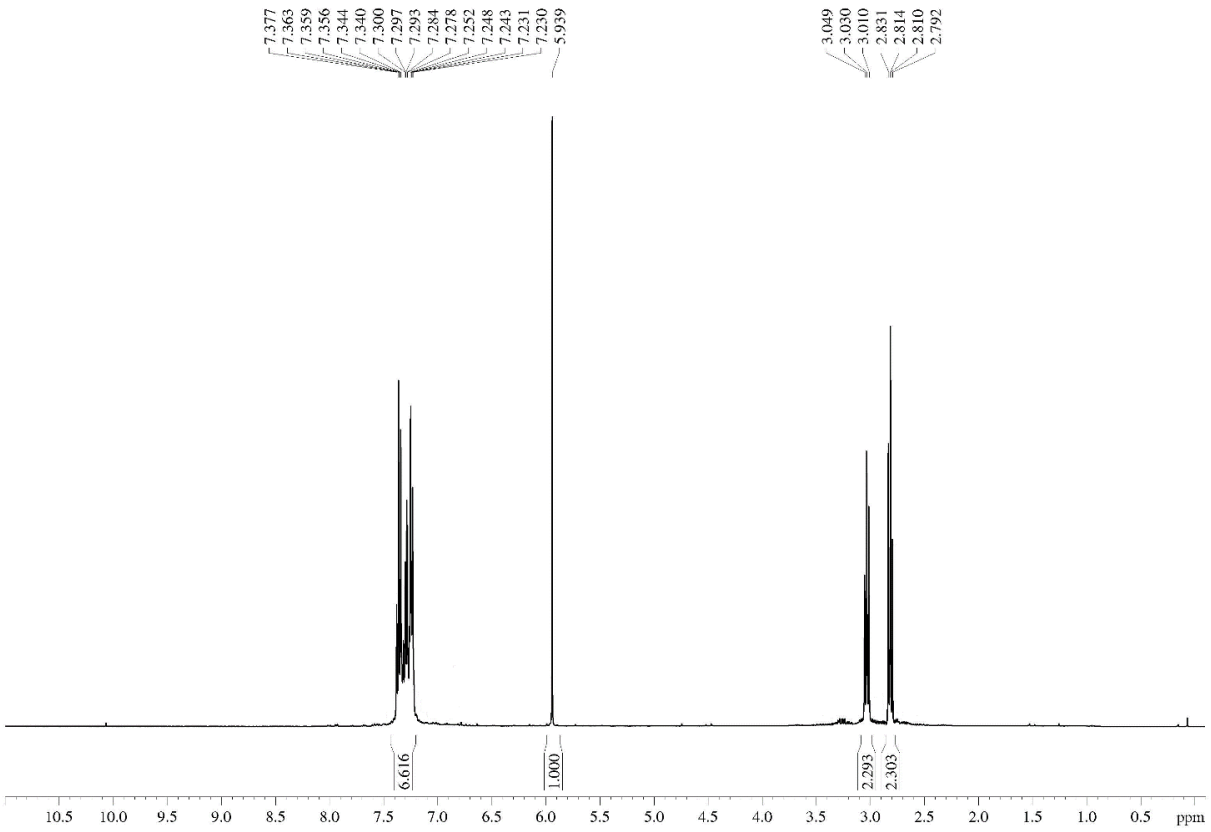


Figure S65. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of the 1,1,1-trifluoro-6-phenylhexan-2,4-dione (**5h**).

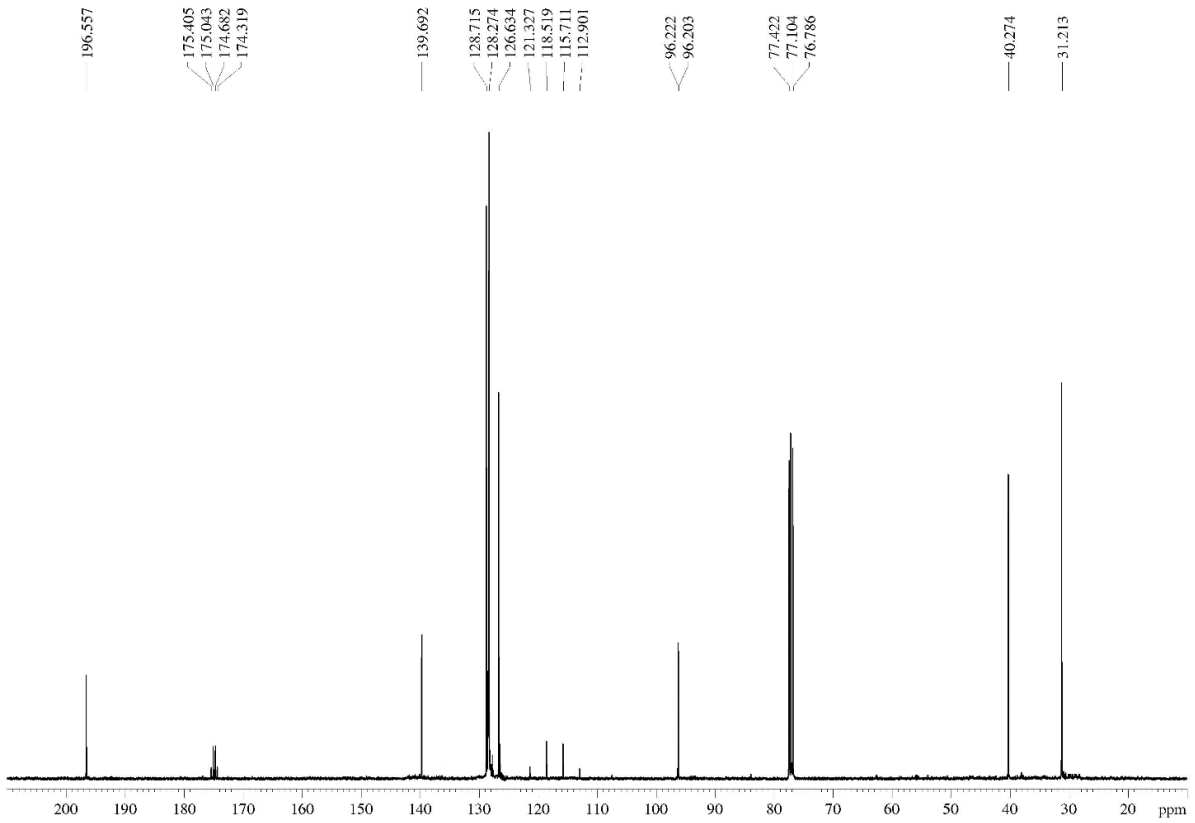


Figure S66. ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of the 1,1,1-trifluoro-4-methoxy-6-phenylhexan-2,4-dione (**5h**).

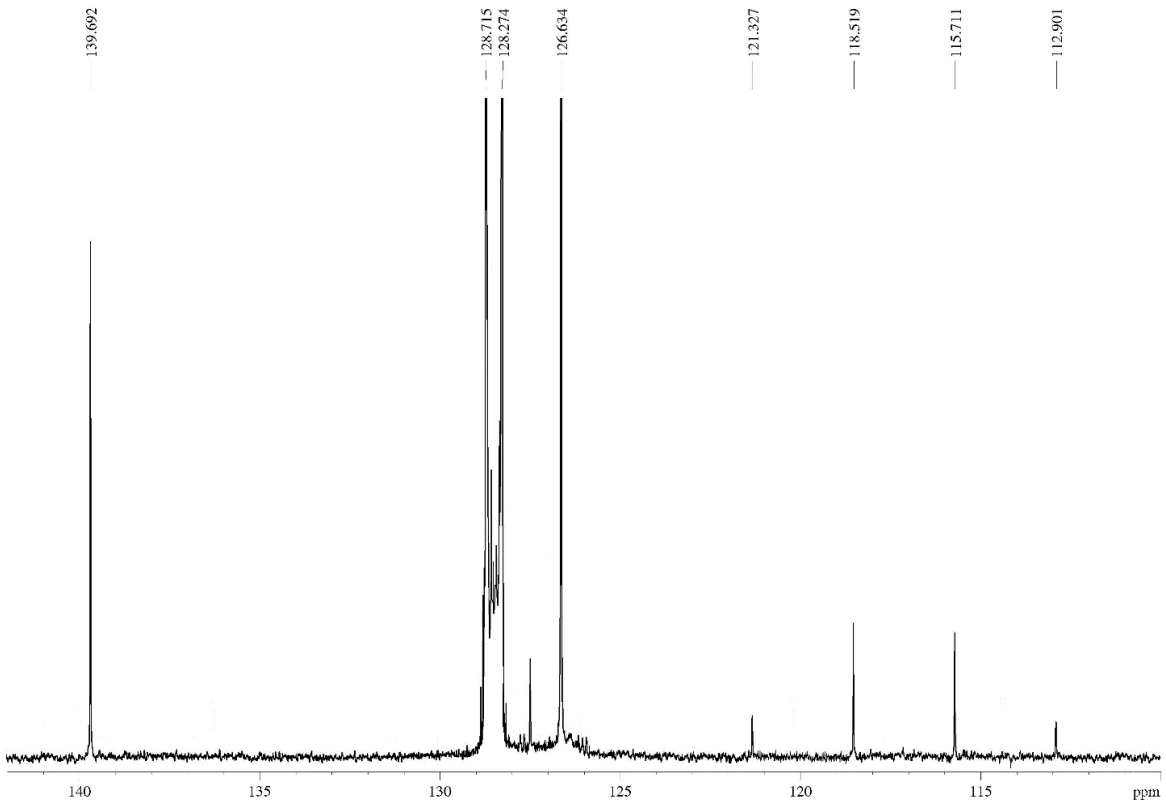


Figure S67. ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of the 1,1,1-trifluoro-4-methoxy-6-phenylhexan-2,4-dione (**5h**), expanded between 110-150 ppm.

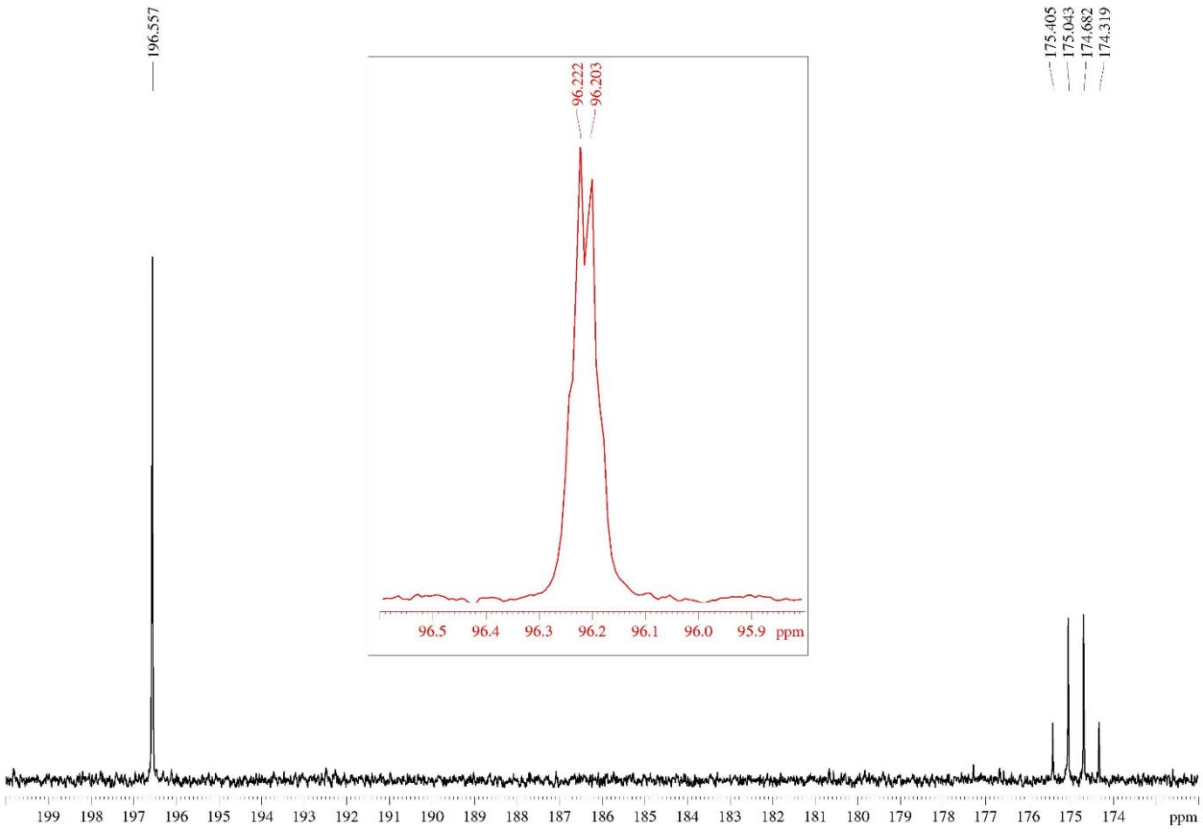


Figure S68. ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of the 1,1,1-trifluoro-4-methoxy-6-phenylhexan-2,4-dione (**5h**), expanded between 172-200 ppm.

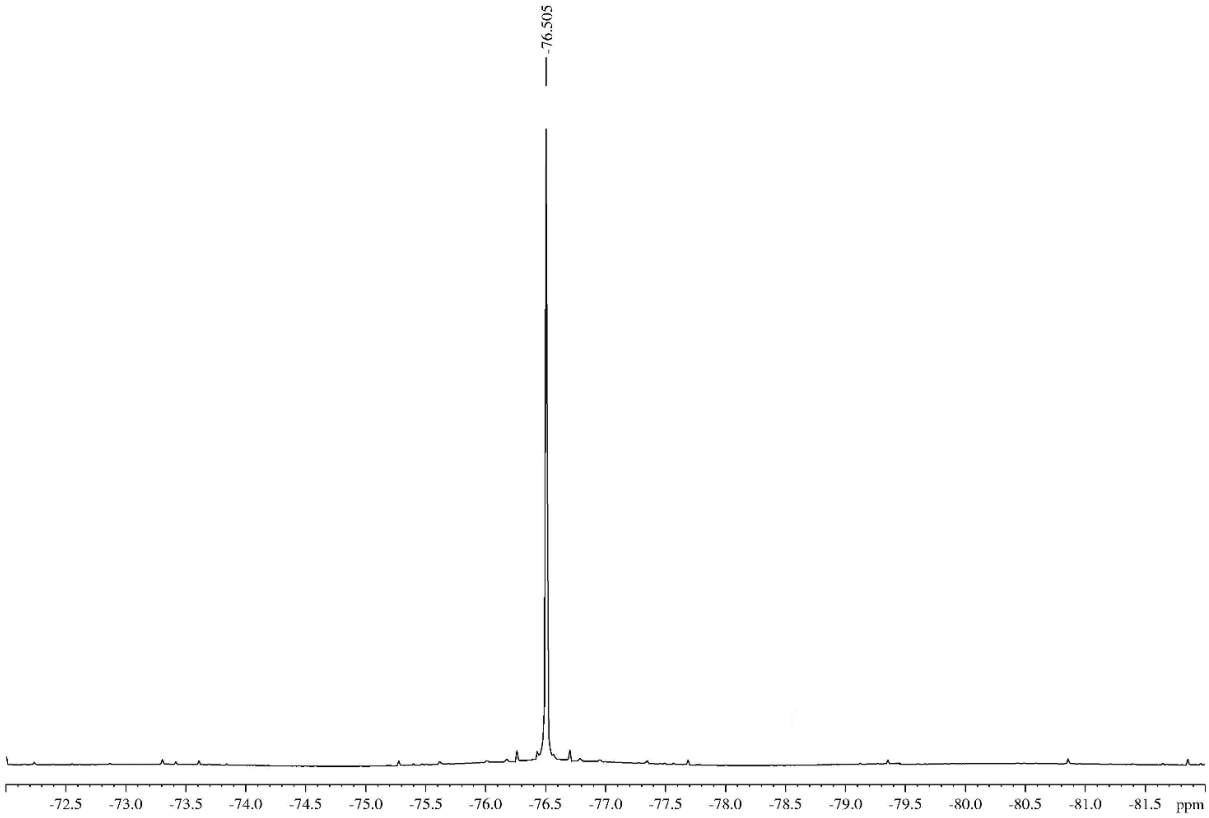


Figure S69. ^{19}F NMR spectrum (376 MHz, $CDCl_3$) of the 1,1,1-trifluoro-6-phenylhexan-2,4-dione (**5h**).

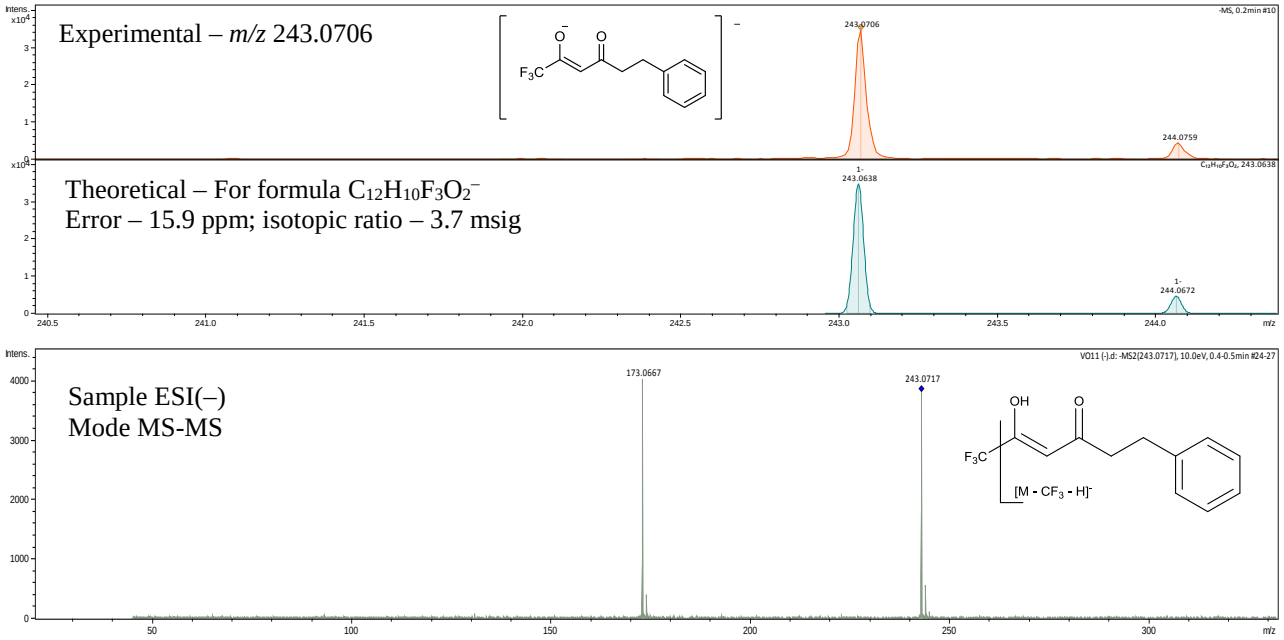


Figure S70. ESI MS-MS spectrum of the 1,1,1-trifluoro-4-methoxy-6-phenylhexan-2,4-dione (**5h**).

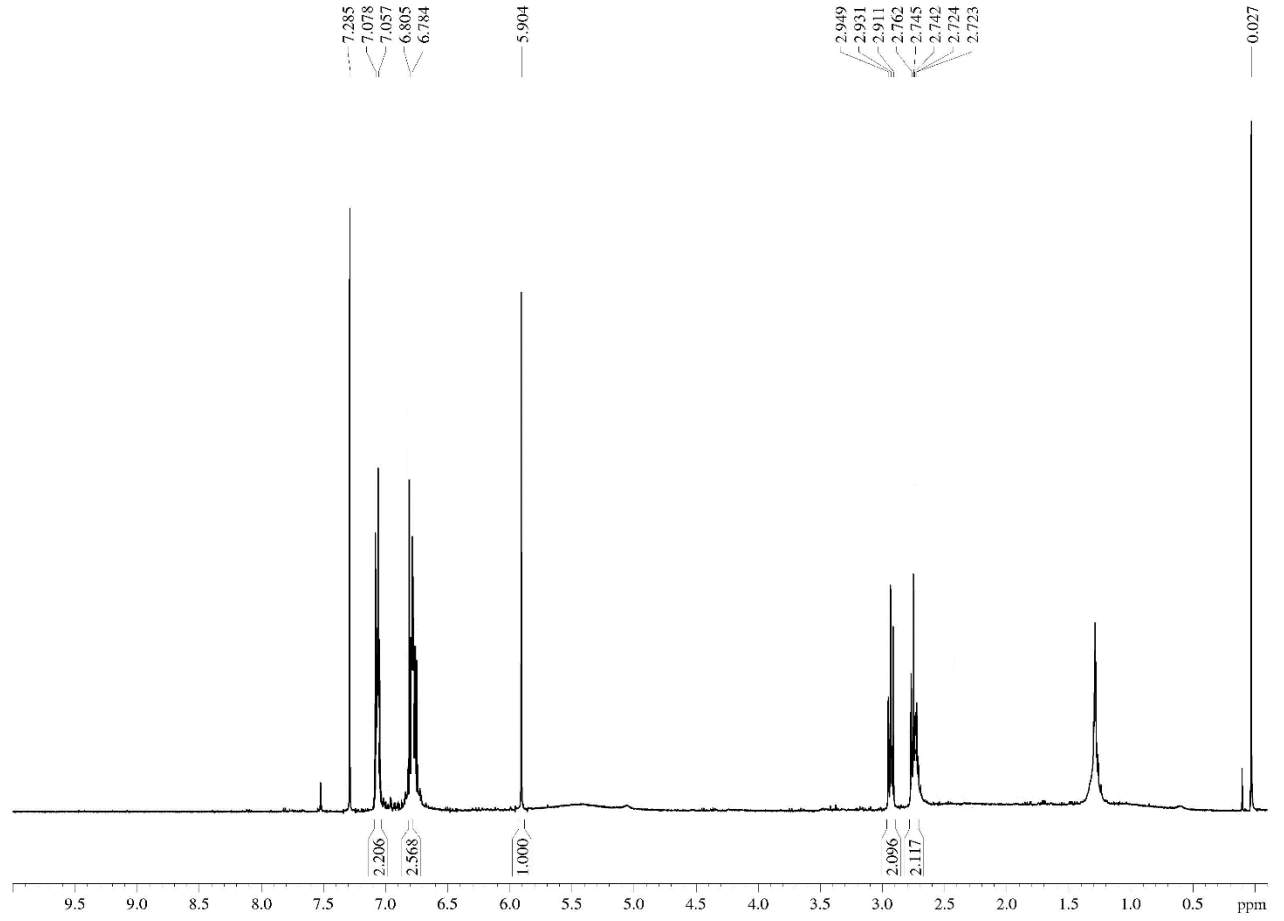


Figure S71. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of the 1,1,1-trifluoro-6-(4-hydroxyphenyl)-hexan-2,4-dione (**5i**).

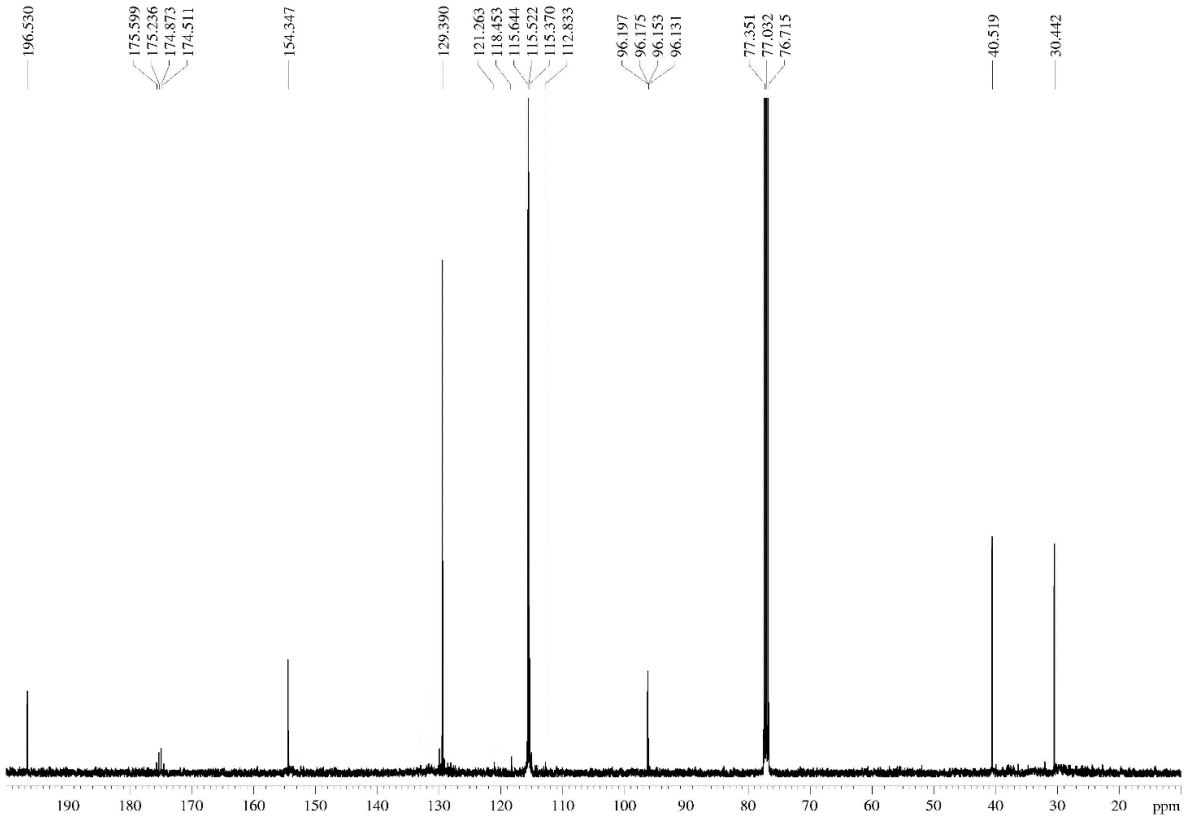


Figure S72. ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of the 1,1,1-trifluoro-6-(4-hydroxyphenyl)-hexan-2,4-dione (**5i**).

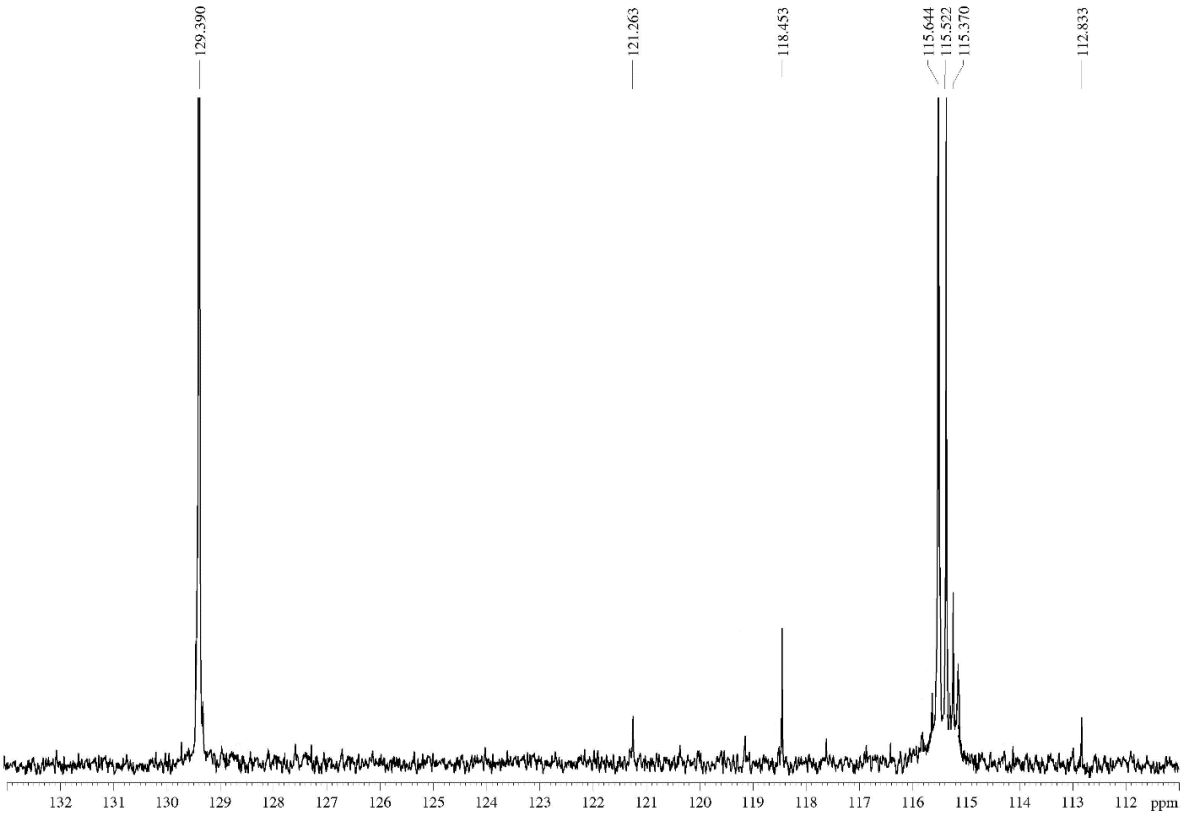


Figure S73. ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of the 1,1,1-trifluoro-4-methoxy-6-(4-hydroxyphenyl)-hex-3-en-2-one (**5i**), expanded between 111-131 ppm.

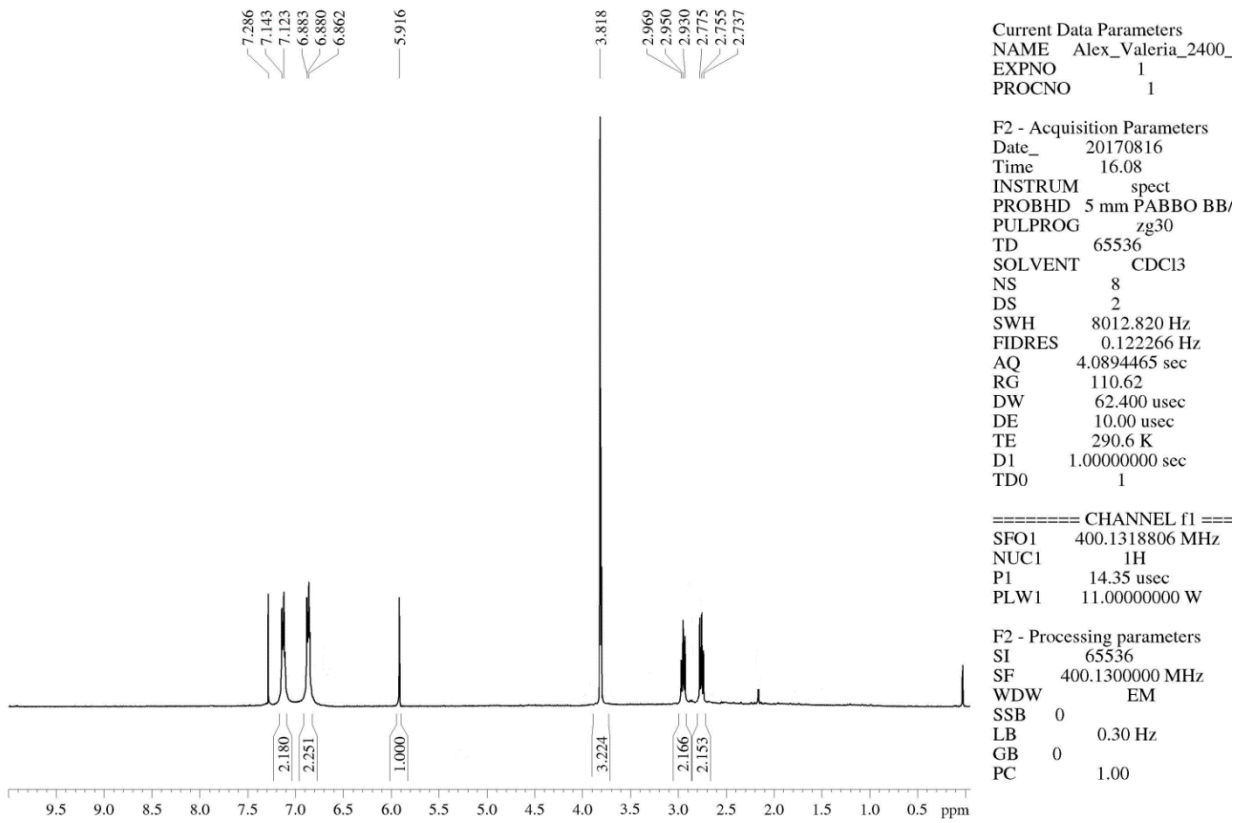


Figure S74. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of the 1,1,1-trifluoro-6-(4-methoxyphenyl)hexan-2,4-dione (**5j**).

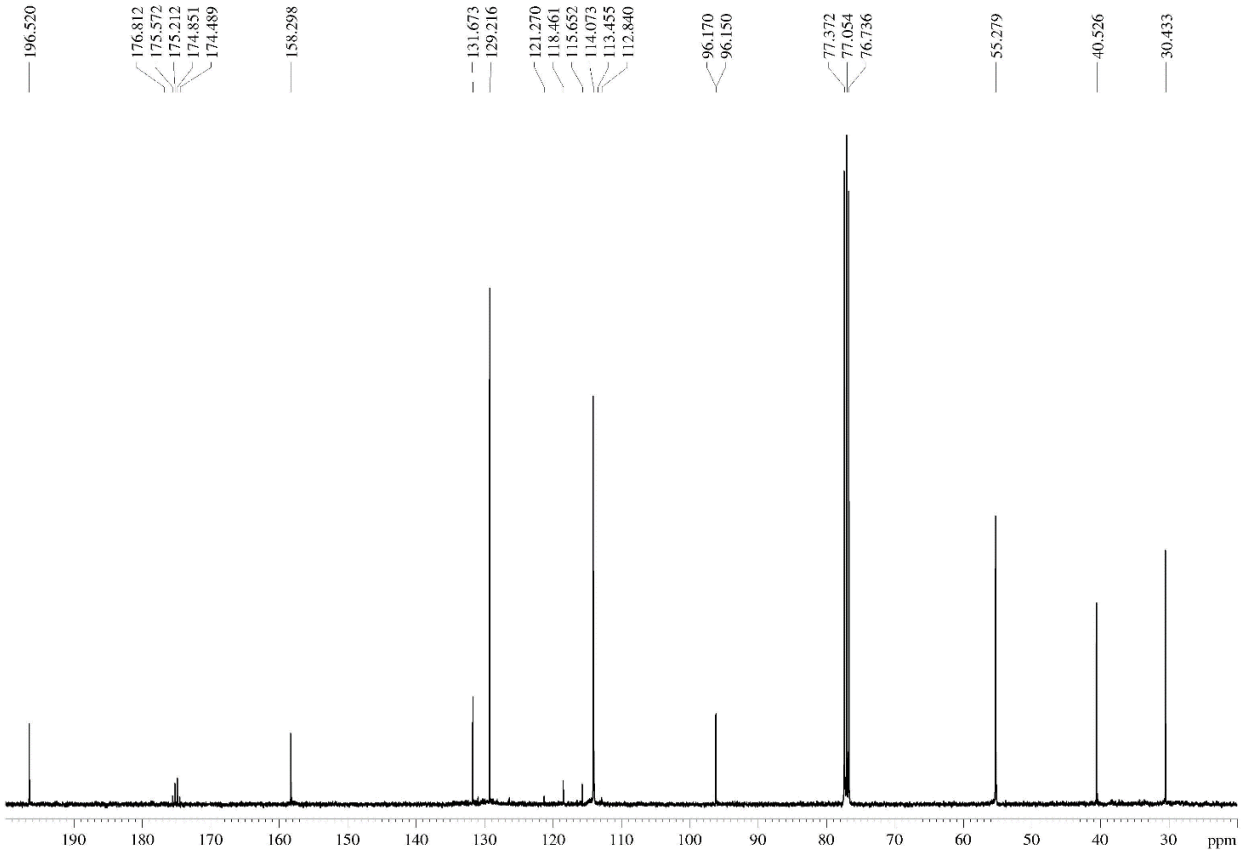


Figure S75. ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of the 1,1,1-trifluoro-6-(4-methoxyphenyl)hexan-2,4-dione (**5j**).

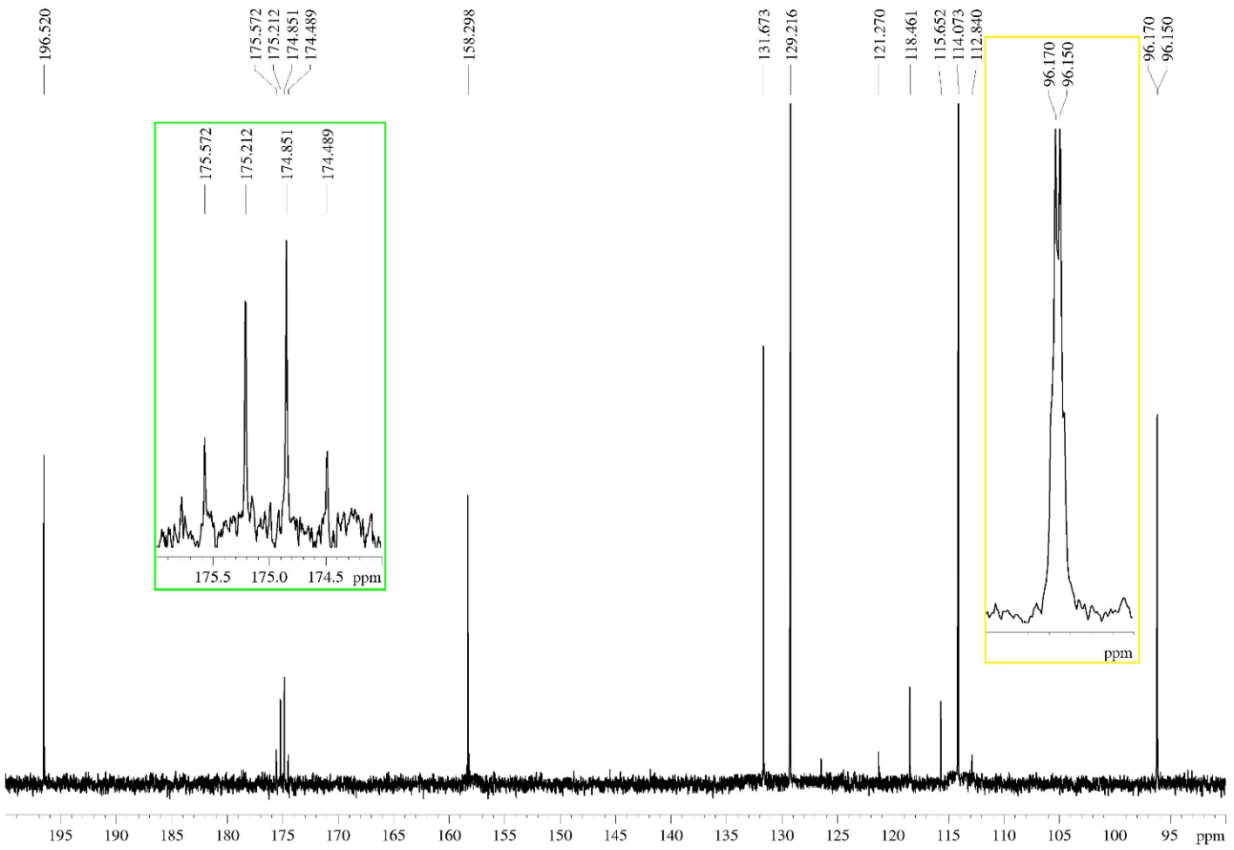


Figure S76. ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of the 1,1,1-trifluoro-6-(4-methoxyphenyl)hexan-2,4-dione (**5j**), expanded between 90-200 ppm.

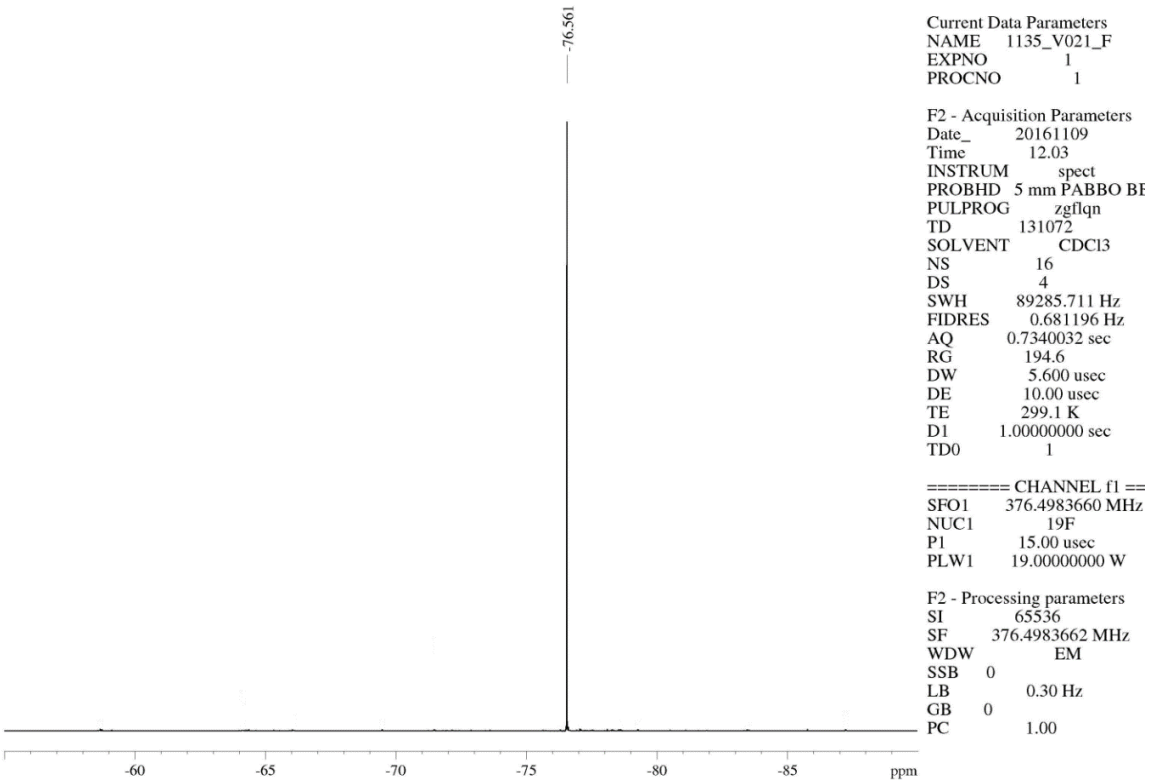


Figure S77. ^{19}F NMR spectrum (376 MHz, $CDCl_3$) of the 1,1,1-trifluoro-6-(4-methoxyphenyl)hexan-2,4-dione (**5j**).

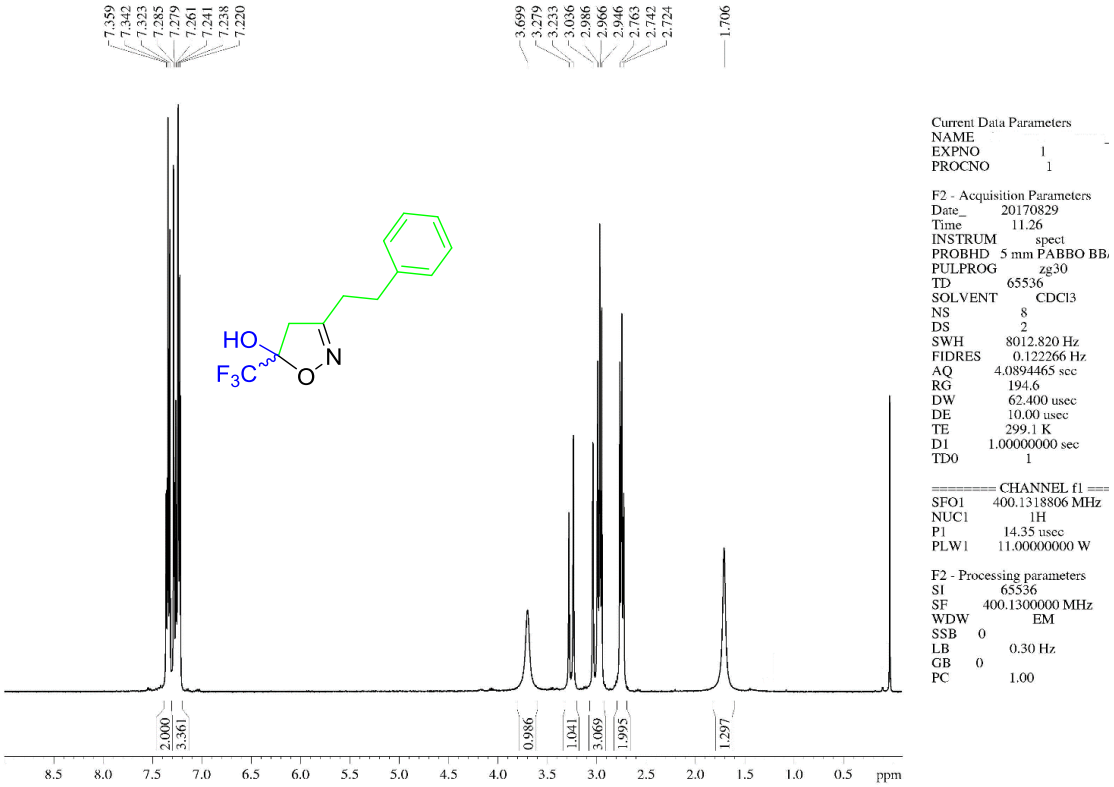


Figure S78. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of the 5-hydroxy-3-phenethyl-5-(trifluoromethyl)-4,5-dihydroisoxazol (**8h**).

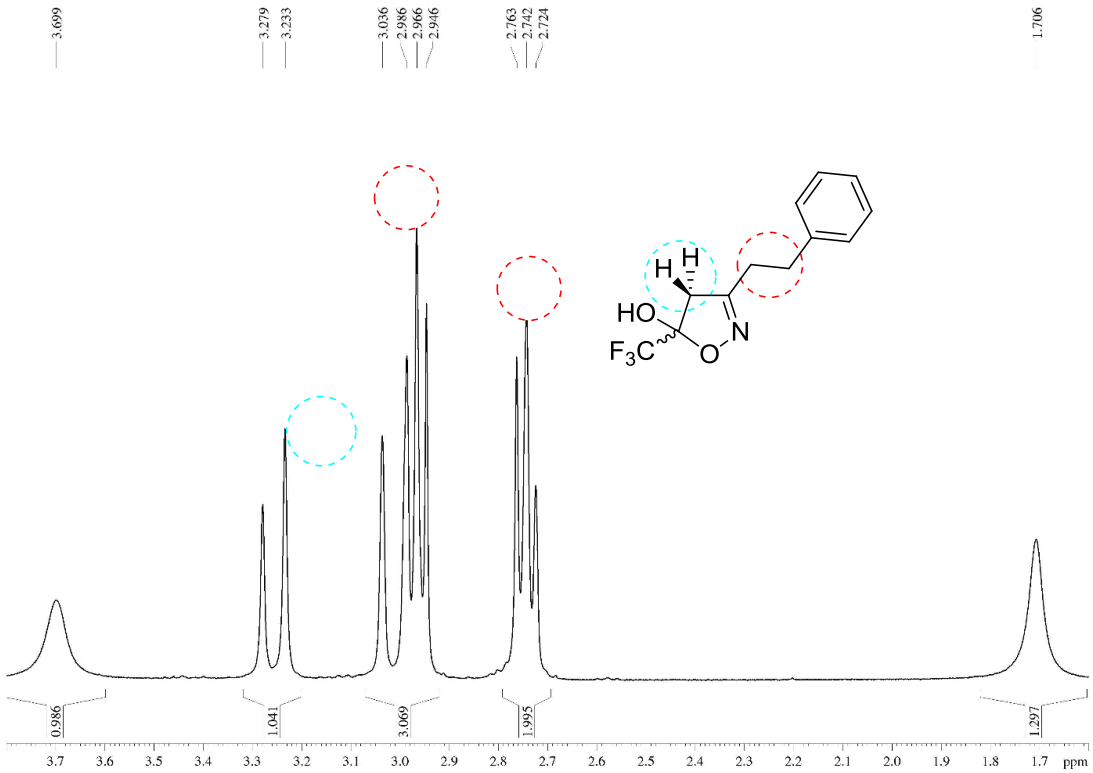


Figure S79. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of the 5-hydroxy-3-phenethyl-5-(trifluoromethyl)-4,5-dihydroisoxazol (**8h**), expanded between 1.6-3.8 ppm.

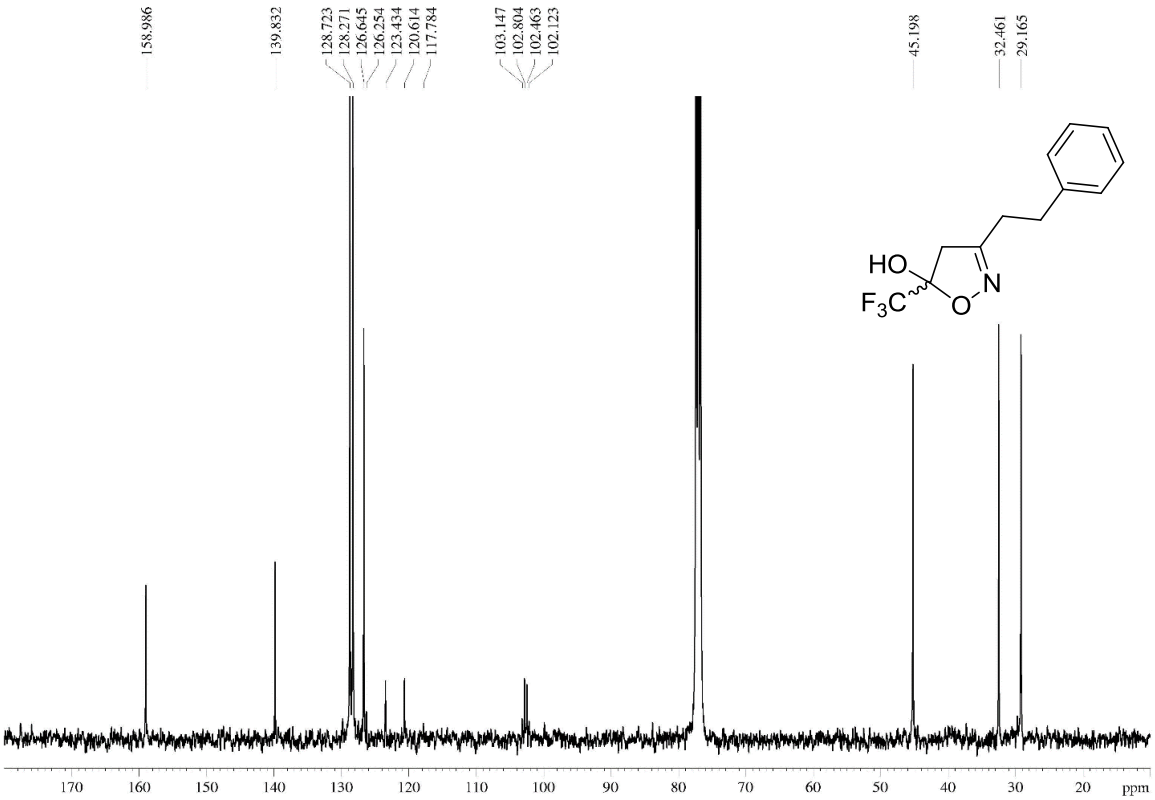


Figure S80. ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of the 5-hydroxy-3-phenethyl-5-(trifluoromethyl)-4,5-dihydroisoxazol (**8h**).

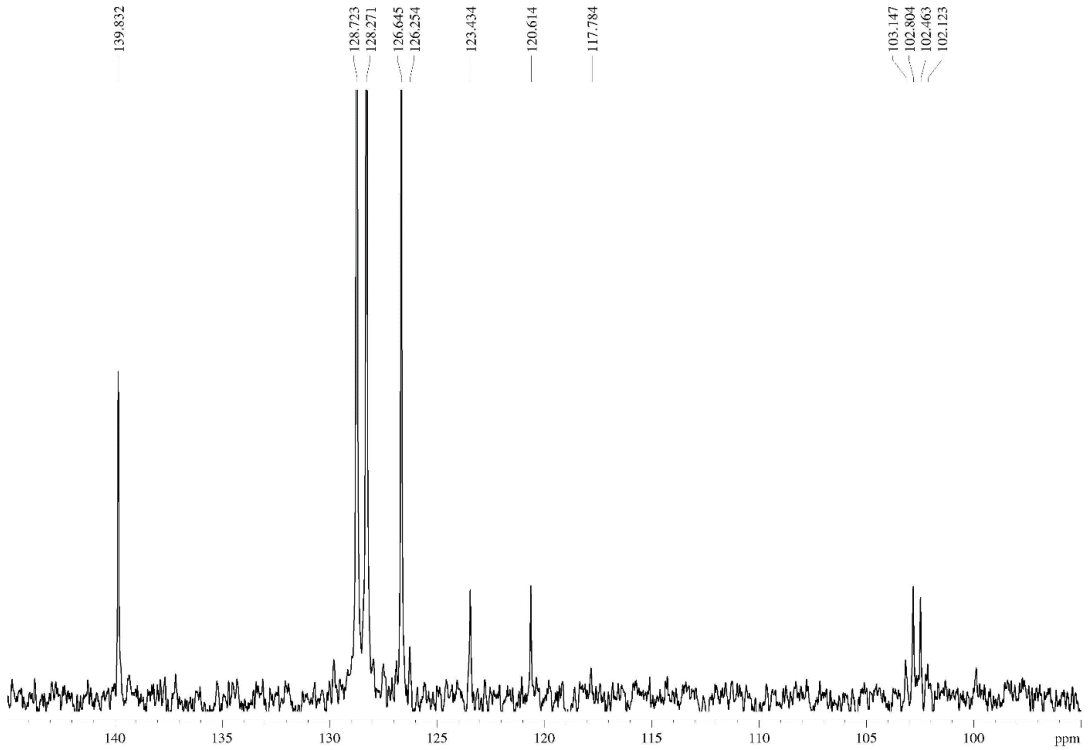


Figure S81. ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of the 5-hydroxy-3-phenethyl-5-(trifluoromethyl)-4,5-dihydroisoxazol (**8h**), expanded between 95-145 ppm.

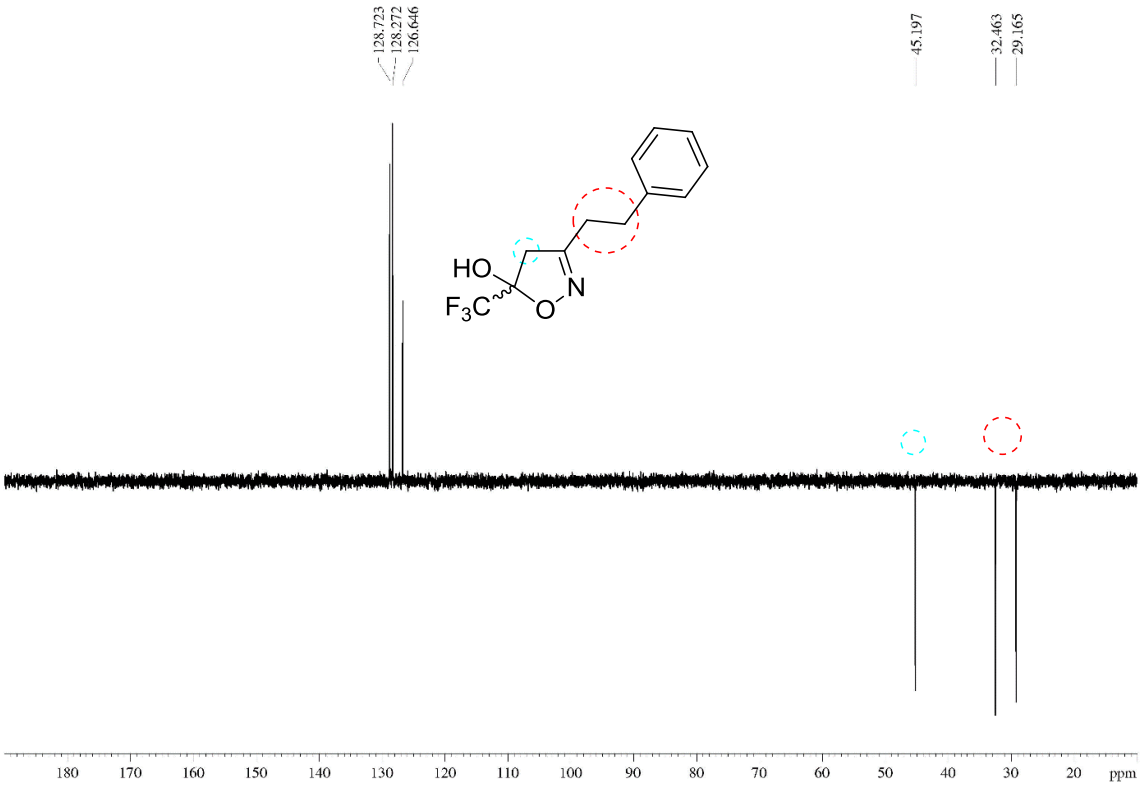


Figure S82. ^{13}C DEPT NMR spectrum (100 MHz, $CDCl_3$) of the 5-hydroxy-3-phenethyl-5-(trifluoromethyl)-4,5-dihydroisoxazole (**8h**), expanded between 1.6-3.8 ppm.

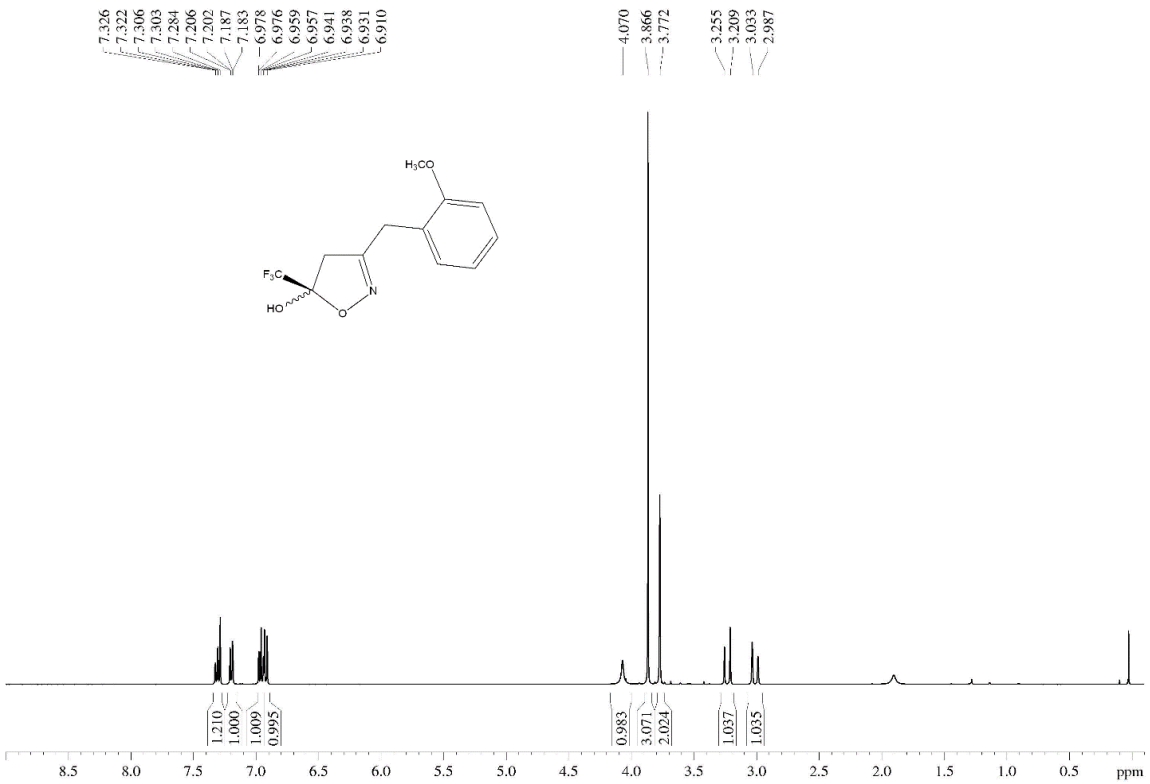


Figure S83. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of the 5-hydroxy-3-(2-methoxyphenyl)methyl-5-(trifluoromethyl)-4,5-dihydroisoxazole (**8l**).

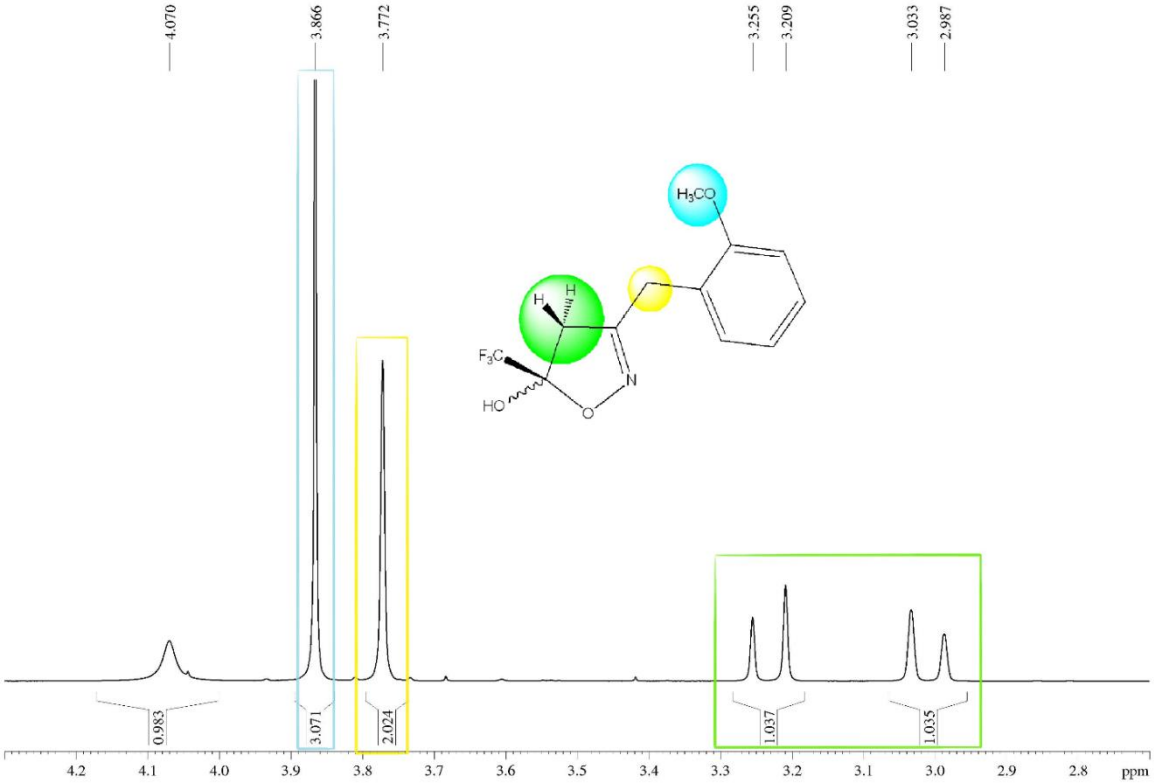


Figure S84. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of the 5-hydroxy-3-(2-methoxyphenyl)methyl-5-(trifluoromethyl)-4,5-dihydroisoxazole (**8l**), expanded between 2.7-4.3 ppm.

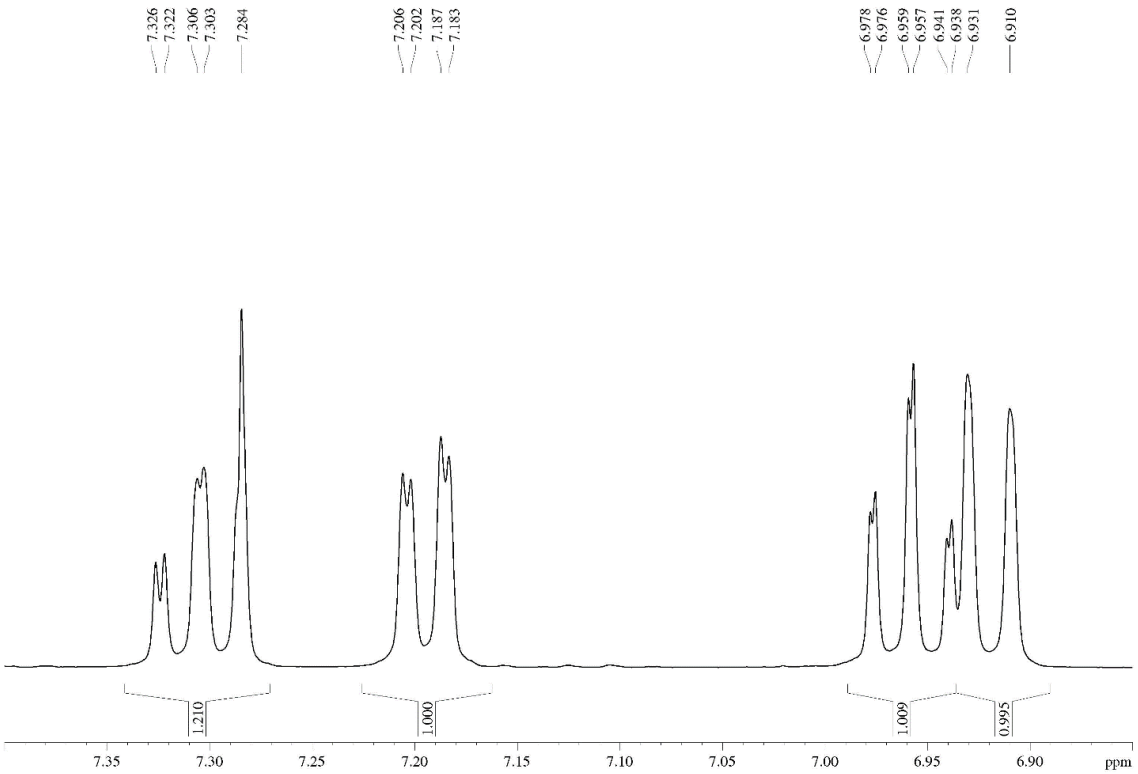


Figure S85. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of the 5-hydroxy-3-(2-methoxyphenyl)methyl-5-(trifluoromethyl)-4,5-dihydroisoxazole (**8l**), expanded between 6.8-7.4 ppm.

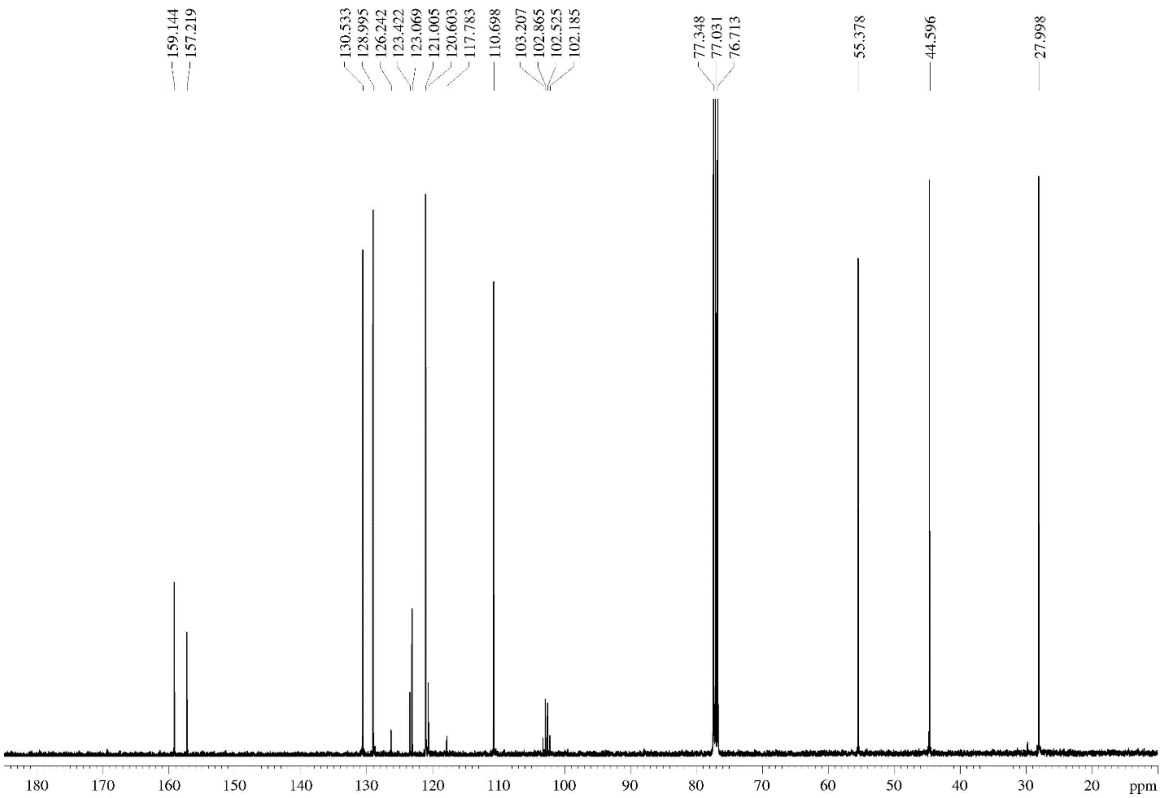


Figure S86. ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of the 5-hydroxy-3-(2-methoxyphenyl)methyl-5-(trifluoromethyl)-4,5-dihydroisoxazole (**8l**).

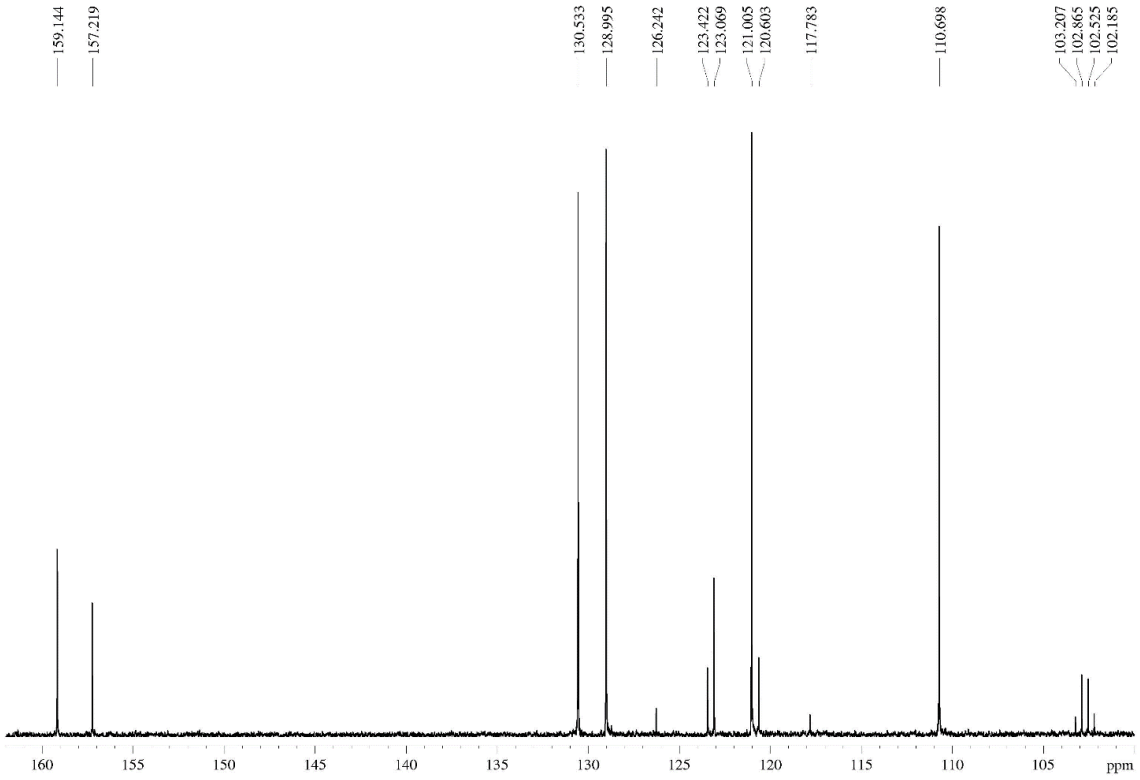


Figure S87. ^{13}C NMR spectrum (100 MHz, $CDCl_3$) of the 5-hydroxy-3-(2-methoxyphenyl)methyl-5-(trifluoromethyl)-4,5-dihydroisoxazole (**8l**), expanded between 100-162 ppm.

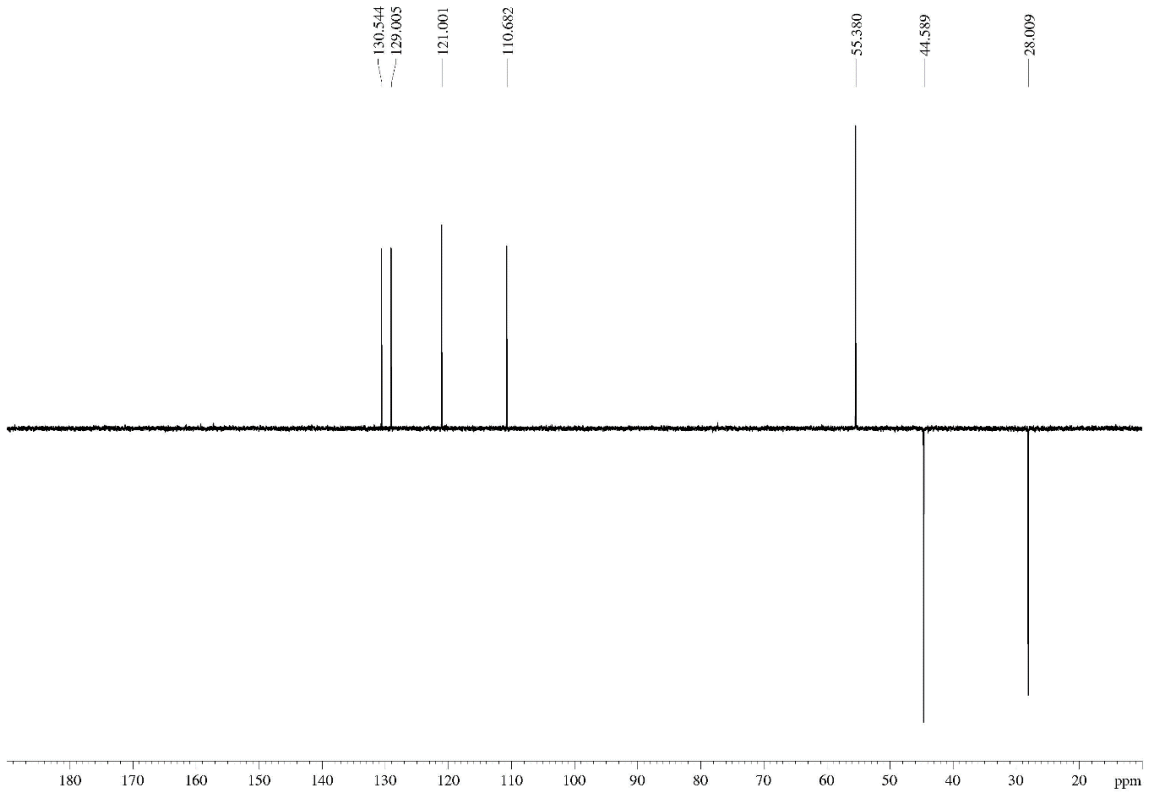


Figure S88. ^{13}C DEPT NMR spectrum (100 MHz, $CDCl_3$) of the 5-hydroxy-3-(2-methoxyphenyl)methyl-5-(trifluoromethyl)-4,5-dihydroisoxazole (**8l**).

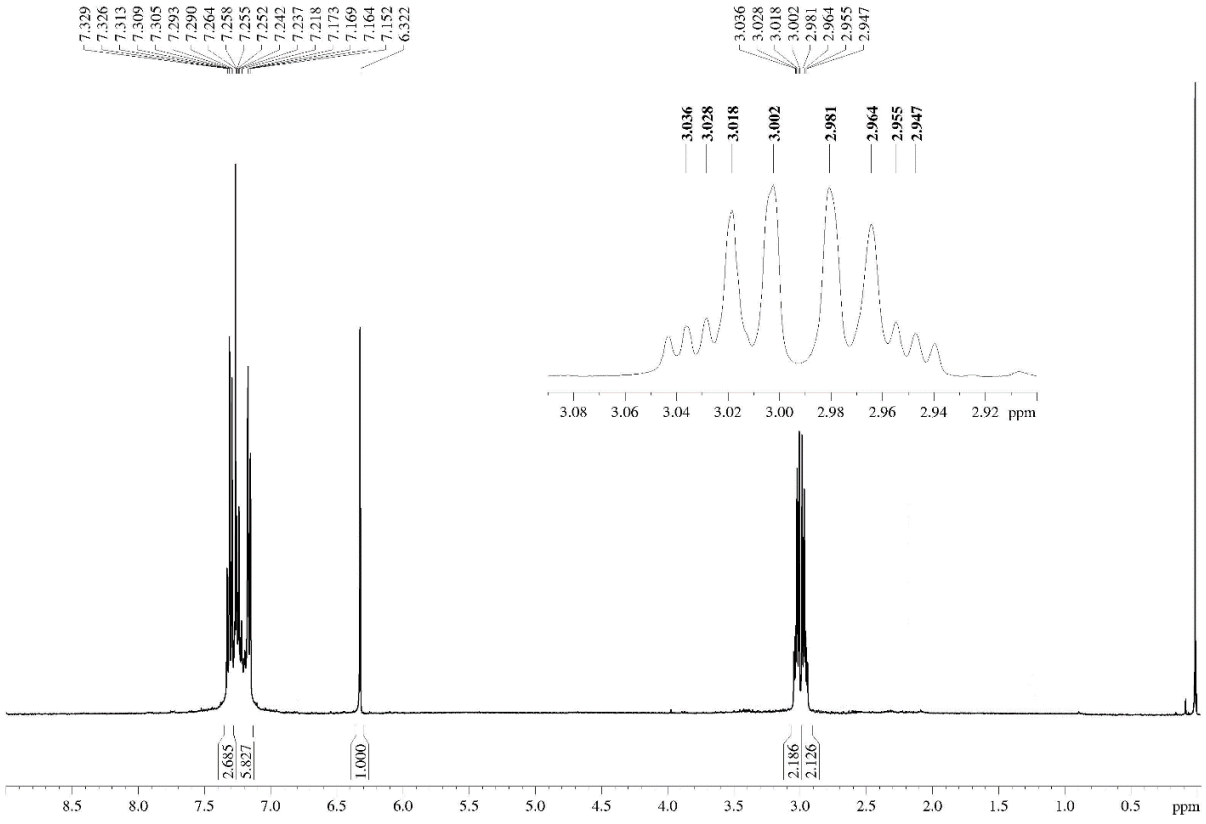


Figure S89. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of 3-(2-phenylethyl)-5-trifluoromethyl-1*H*-pyrazole (**12h**).

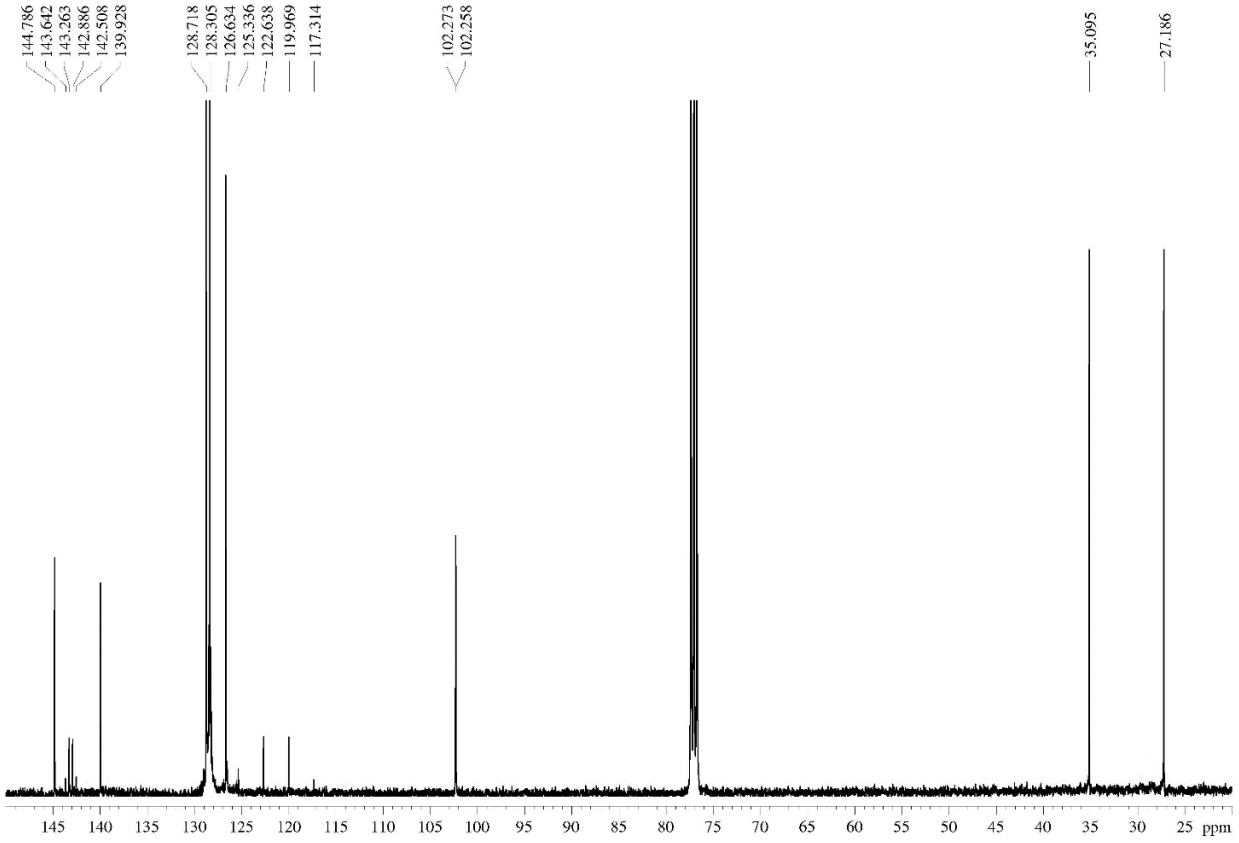


Figure S90. ^{13}C NMR spectrum (400 MHz, $CDCl_3$) of 3-(2-phenylethyl)-5-trifluoromethyl-1*H*-pyrazole (**12h**).

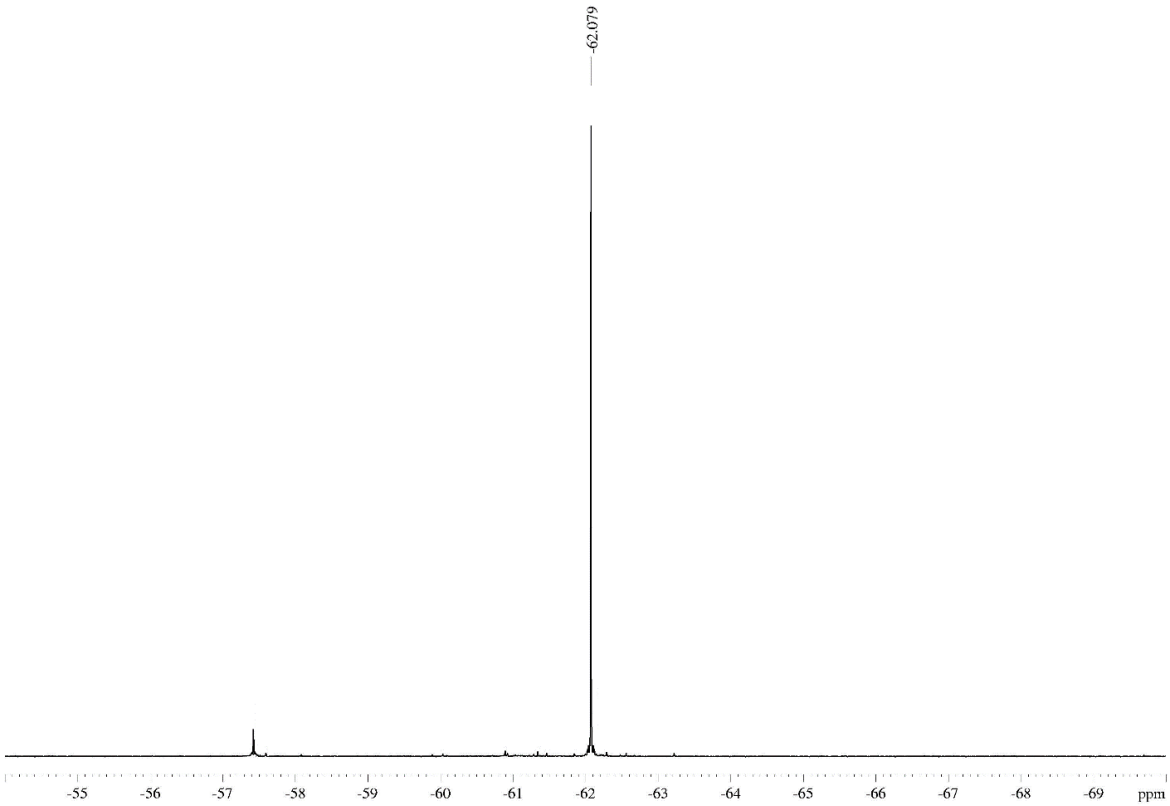


Figure S91. ^{19}F NMR spectrum (376 MHz, $CDCl_3$) of 3-(2-phenylethyl)-5-trifluoromethyl-1*H*-pyrazole (**12h**).

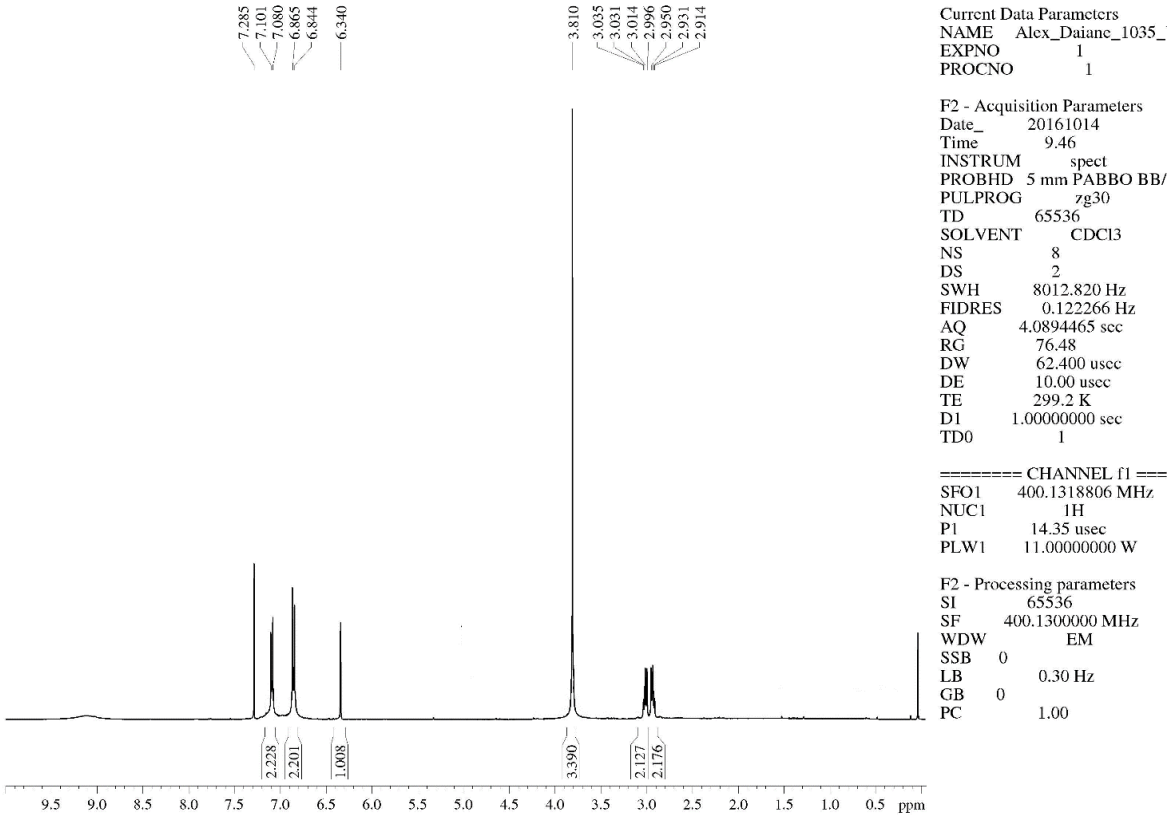


Figure S92. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of 3-[2-(4-methoxyphenyl)ethyl]-5-trifluoromethyl-1*H*-pyrazole (**12j**).

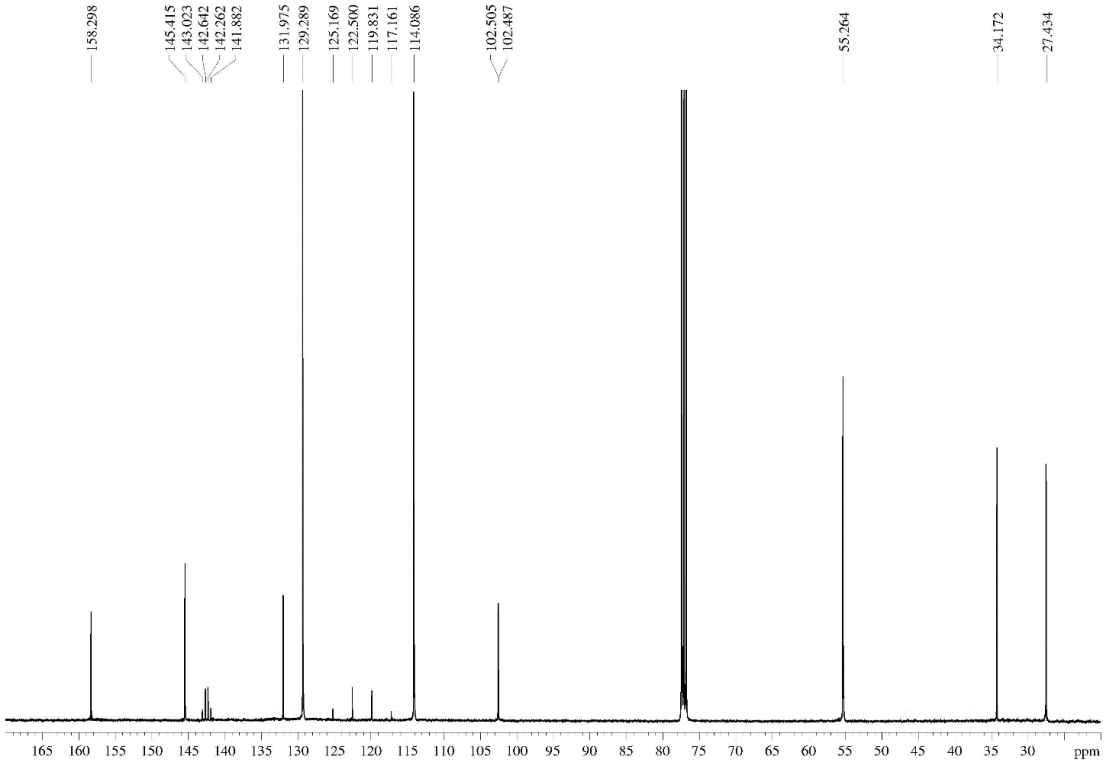


Figure S93. ^{13}C NMR spectrum (400 MHz, $CDCl_3$) of 3-[2-(4-methoxyphenyl)ethyl]-5-trifluoromethyl-1*H*-pyrazole (**12j**).

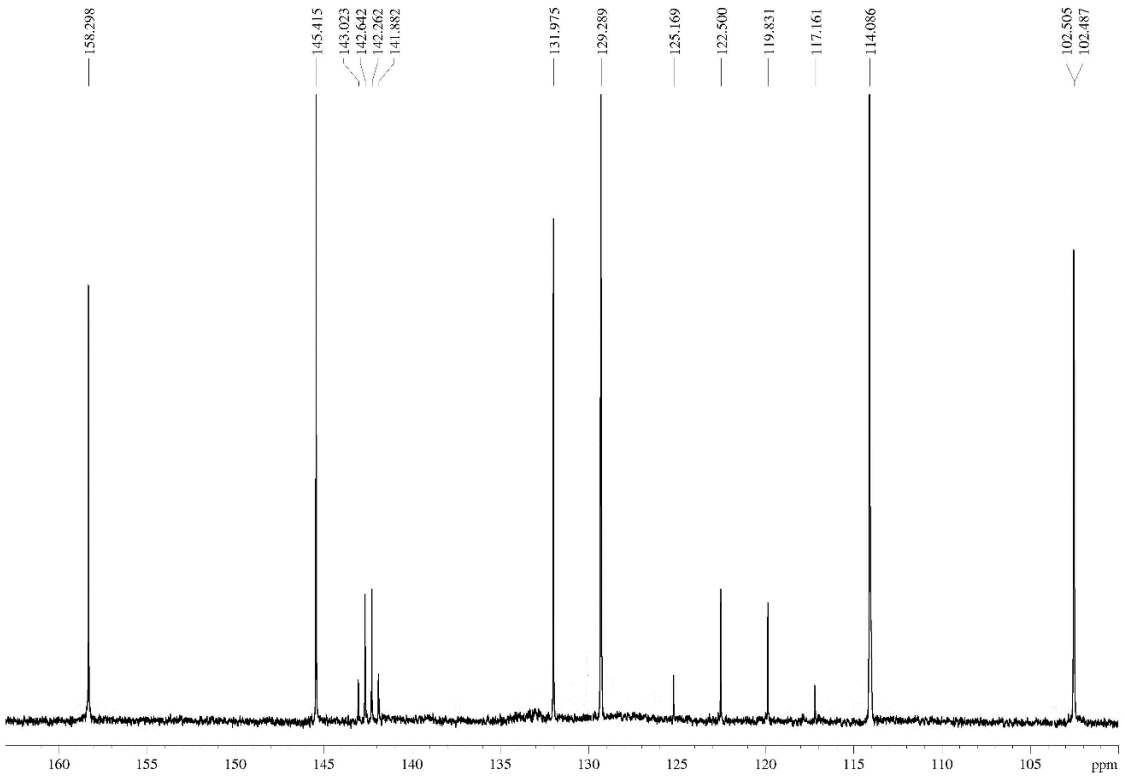


Figure S94. ^{13}C NMR spectrum (400 MHz, $CDCl_3$) of 3-[2-(4-methoxyphenyl)ethyl]-5-trifluoromethyl-1*H*-pyrazole (**12j**), expanded between 100-163 ppm.

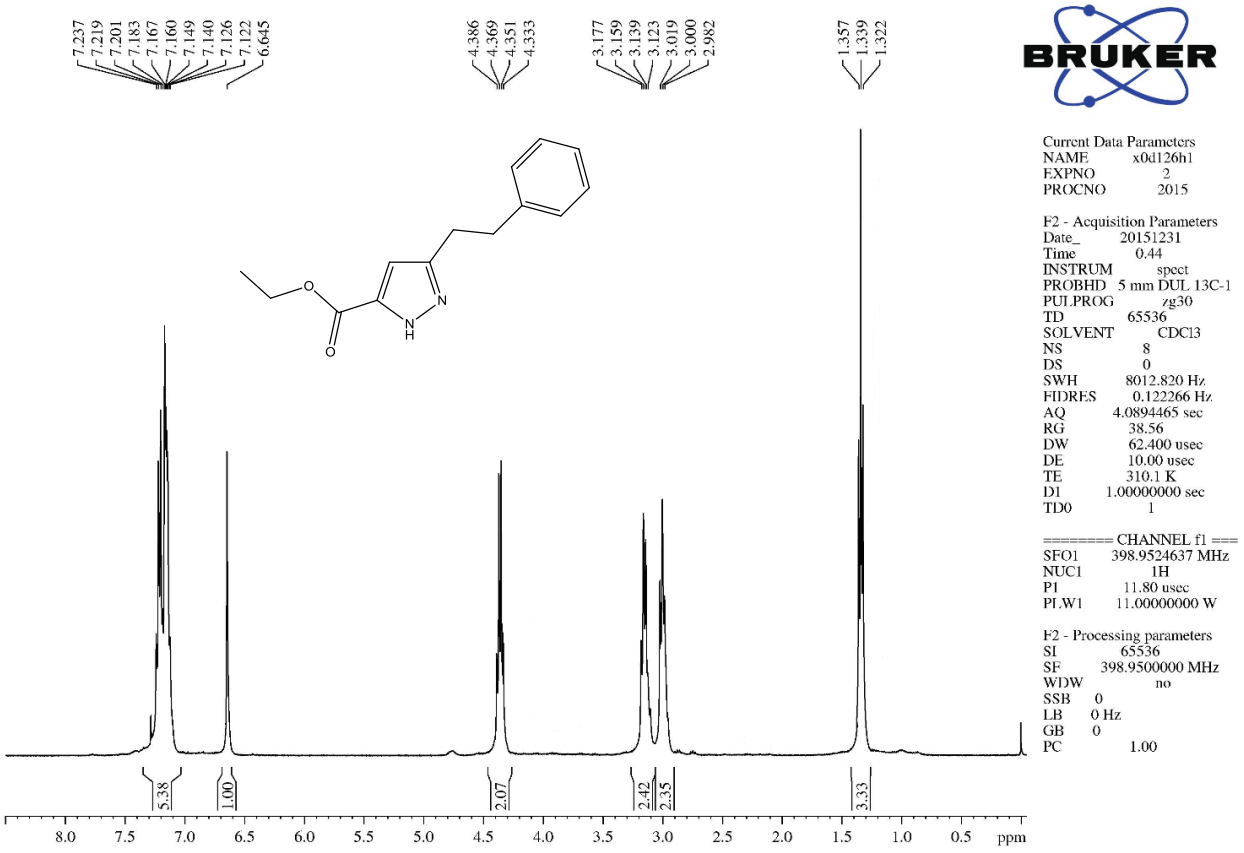


Figure S95. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of ethyl 3-(2-phenylethyl)-1*H*-pyrazole-5-carboxylate(**13h**).

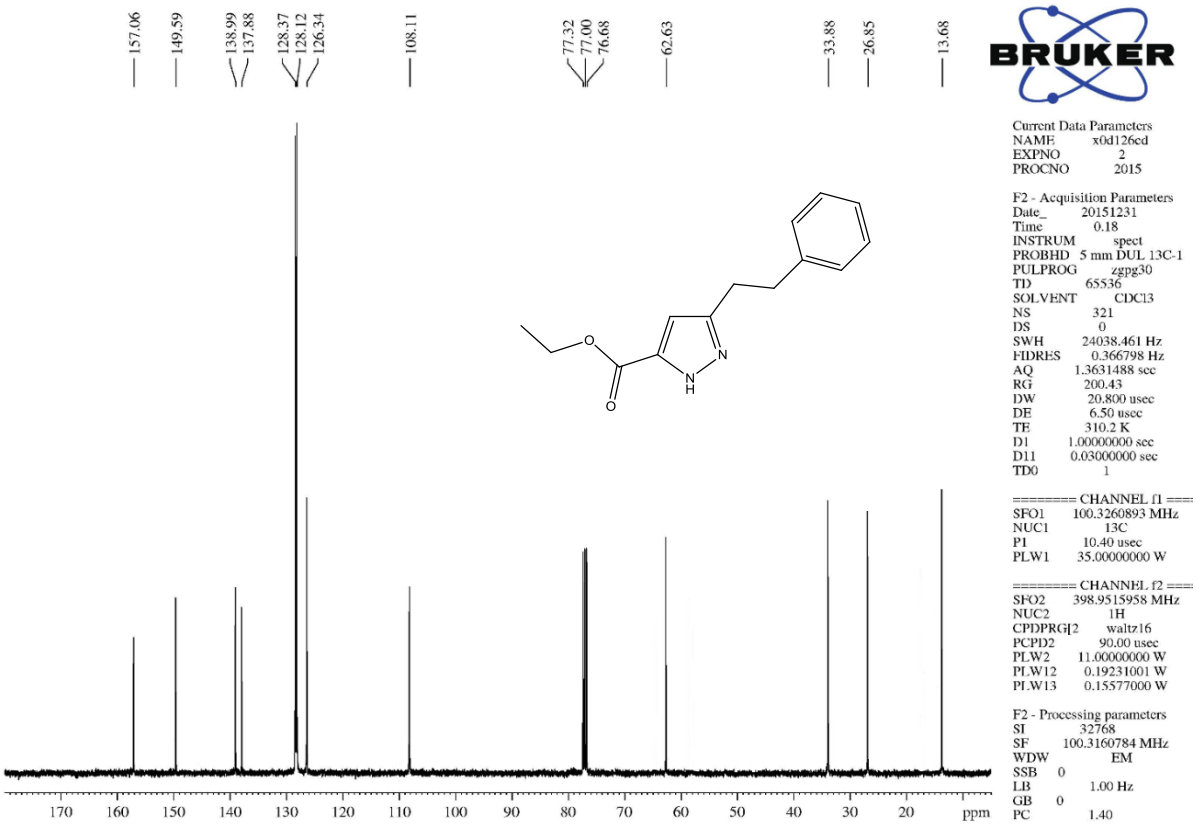


Figure S96. ^{13}C NMR spectrum (400 MHz, $CDCl_3$) of ethyl 3-(2-phenylethyl)-1*H*-pyrazole-5-carboxylate (**13h**).

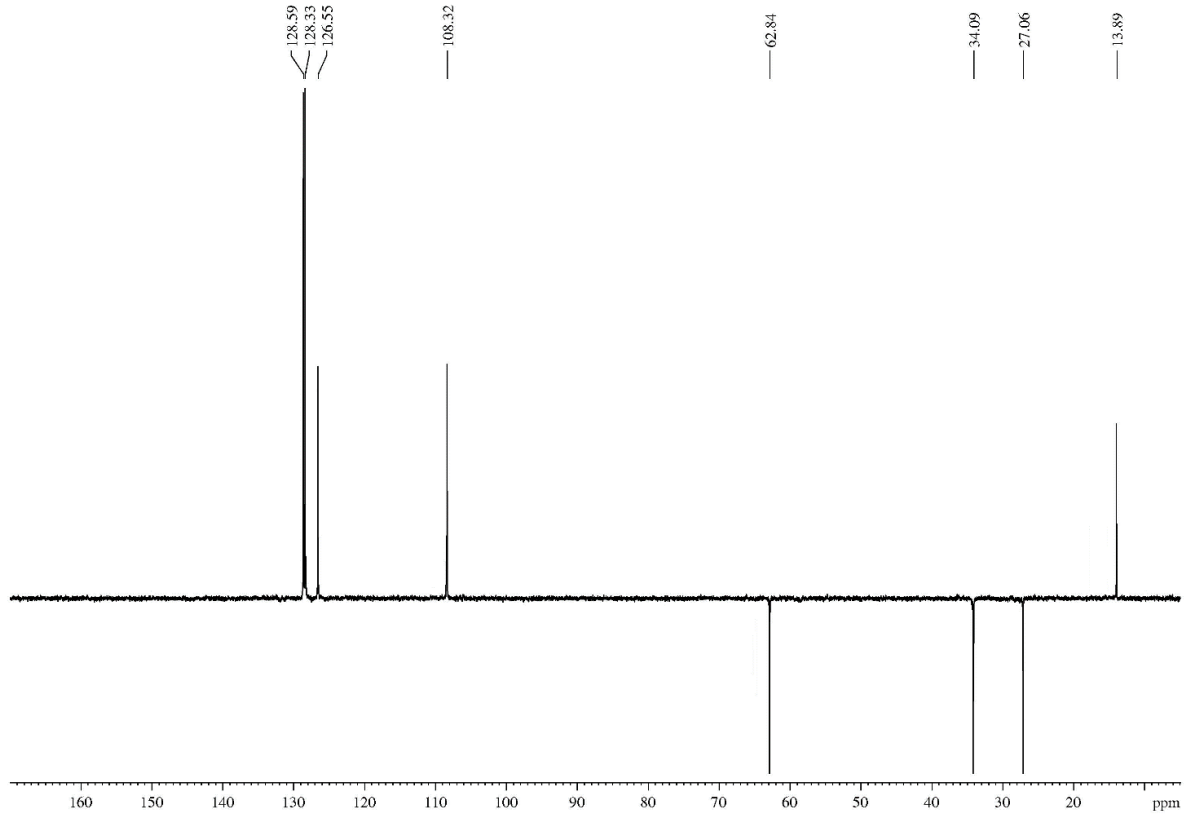


Figure S97. DEPT135 ^{13}C NMR spectrum (400 MHz, $CDCl_3$) of ethyl 3-(2-phenylethyl)-1*H*-pyrazole-5-carboxylate (**13h**).

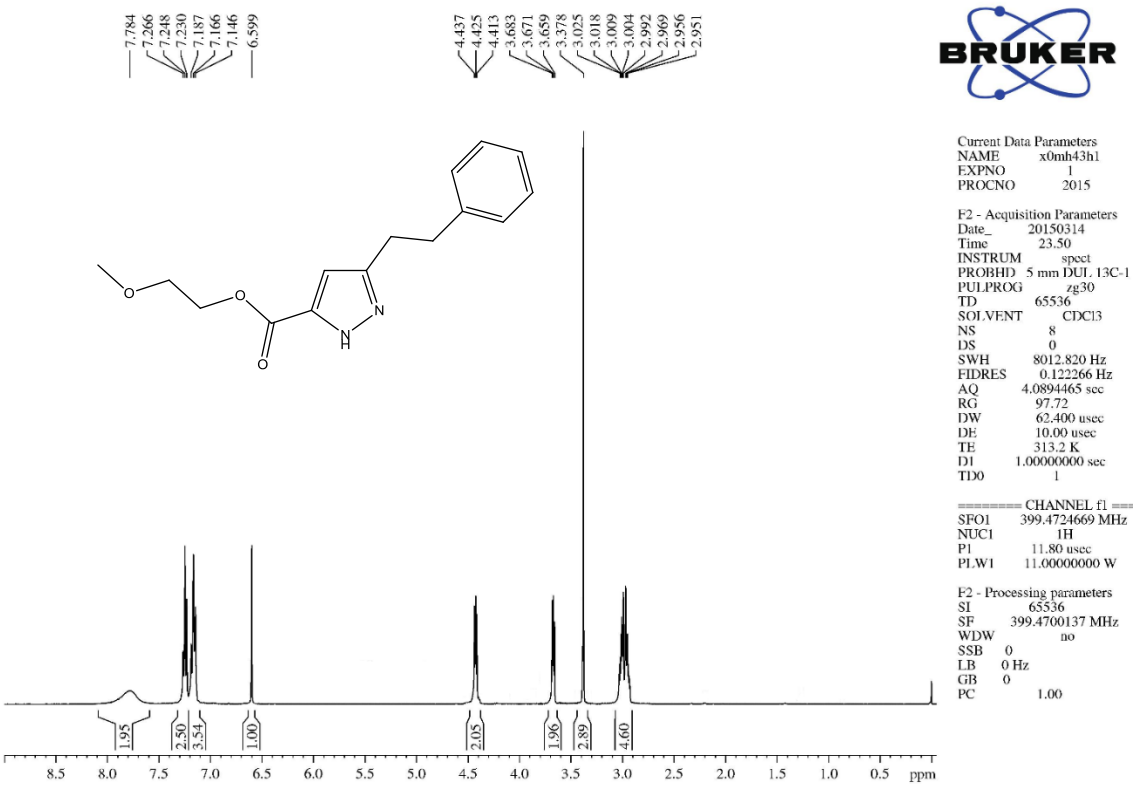


Figure S98. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of 2-methoxyethyl 3-(2-phenylethyl)-1*H*-pyrazole-5-carboxylate (**14h**).

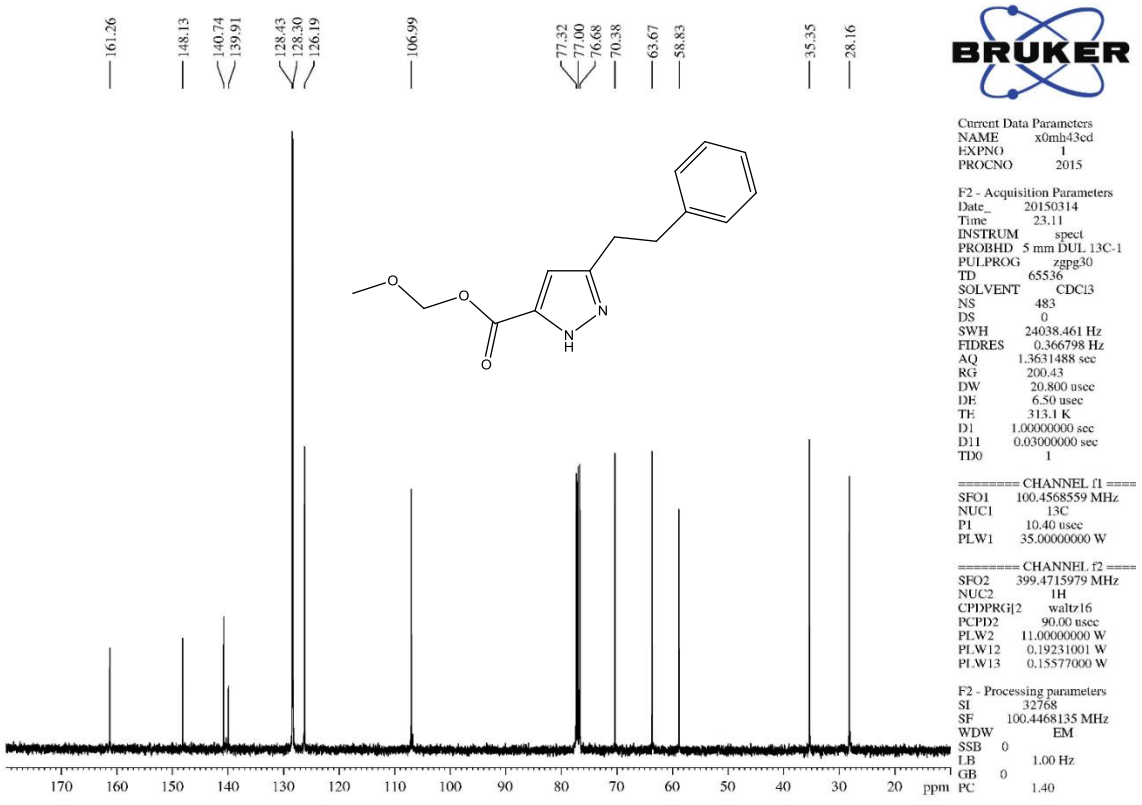


Figure S99. ^{13}C NMR spectrum (400 MHz, $CDCl_3$) of 2-methoxyethyl 3-(2-phenylethyl)-1*H*-pyrazole-5-carboxylate (**14h**).

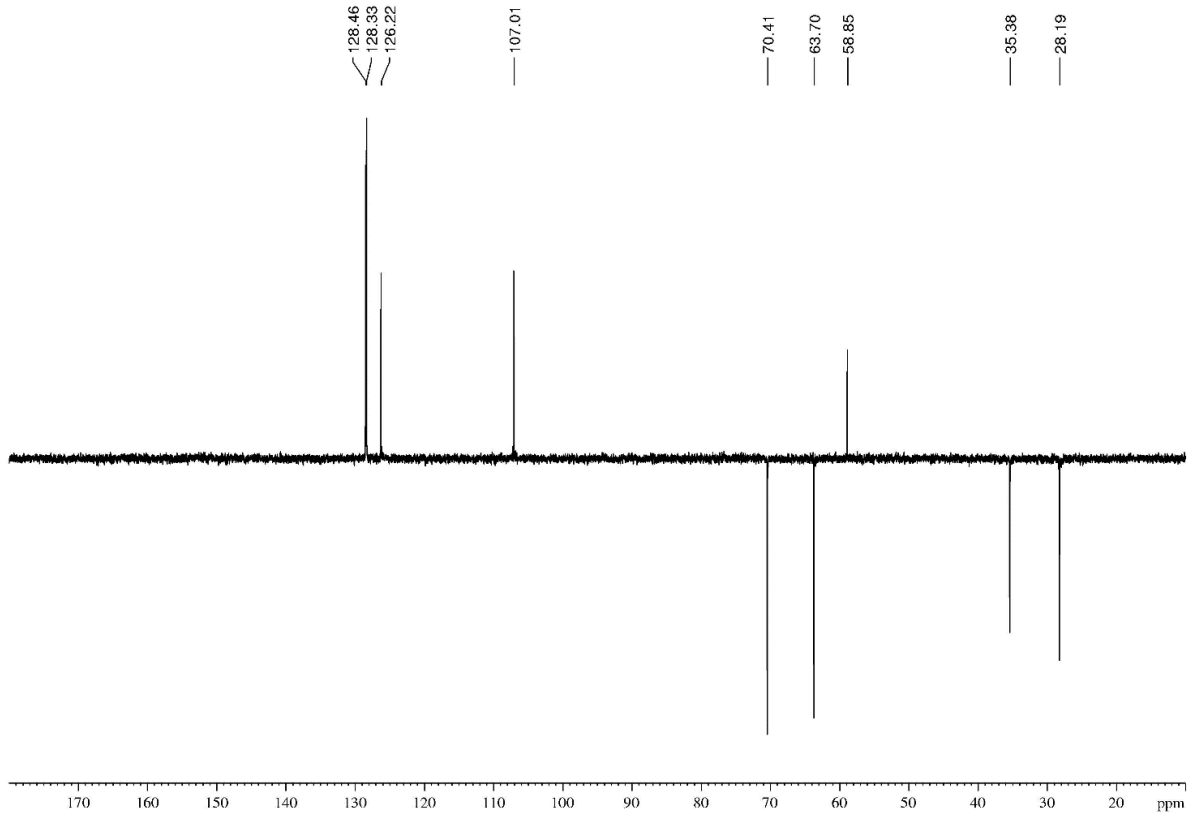


Figure S100. DEPT135 ^{13}C NMR spectrum (400 MHz, $CDCl_3$) of 2-methoxyethyl 3-(2-phenylethyl)-1*H*-pyrazole-5-carboxylate (**14h**).

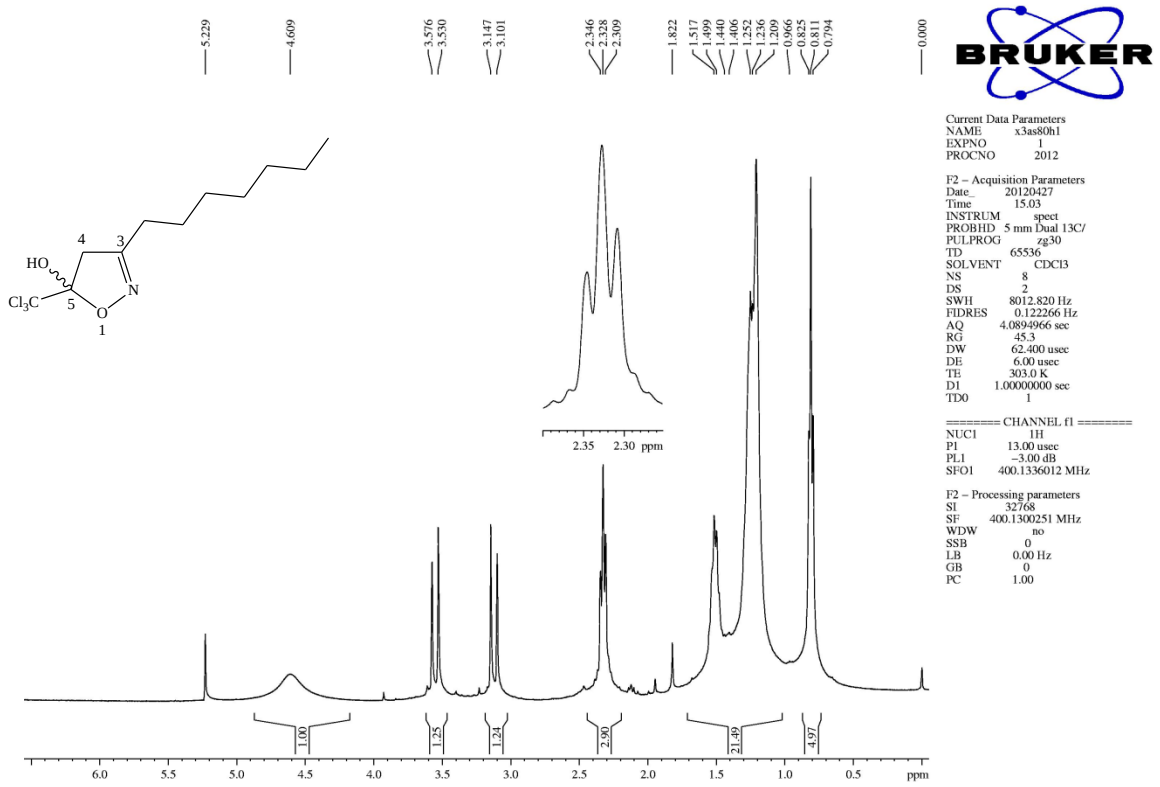


Figure S101. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of 5-trichloromethyl-3-heptyl-5-hydroxy-4,5-dihydroisoxazole (**9a**).

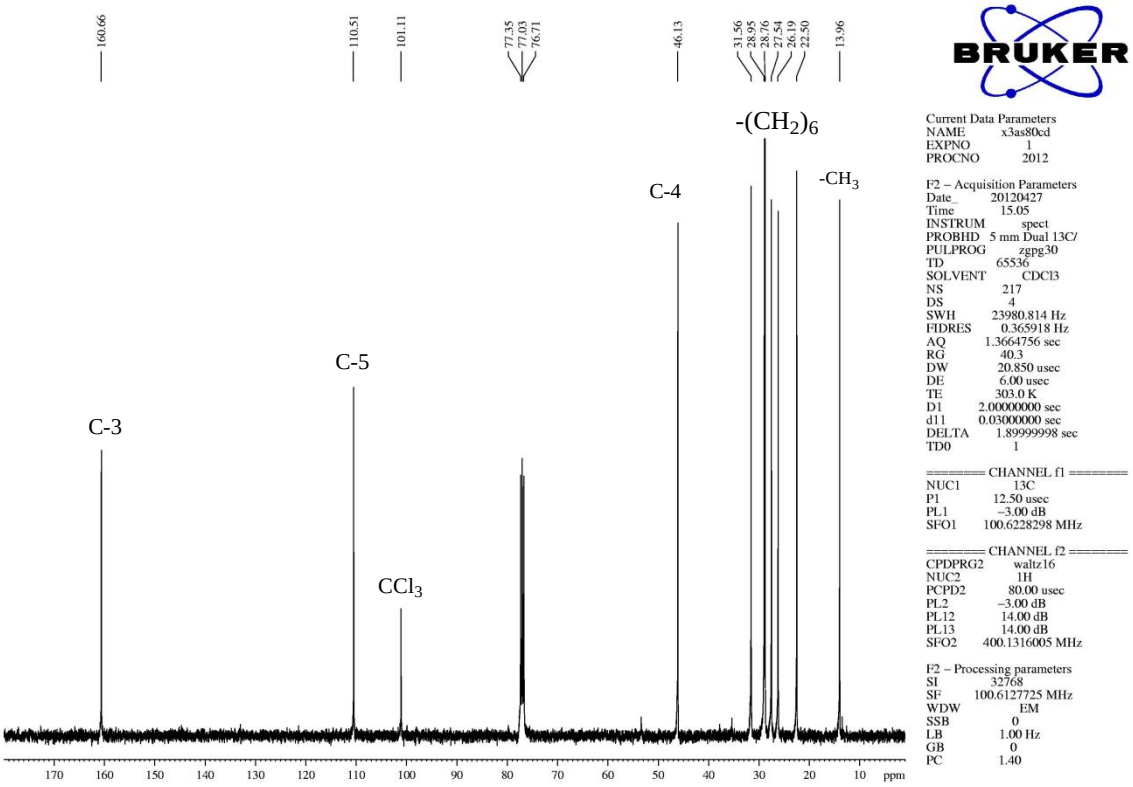


Figure S102. ^{13}C NMR spectrum (400 MHz, $CDCl_3$) of 5-trichloromethyl-3-heptyl-5-hydroxy-4,5-dihydroisoxazole (**9a**).

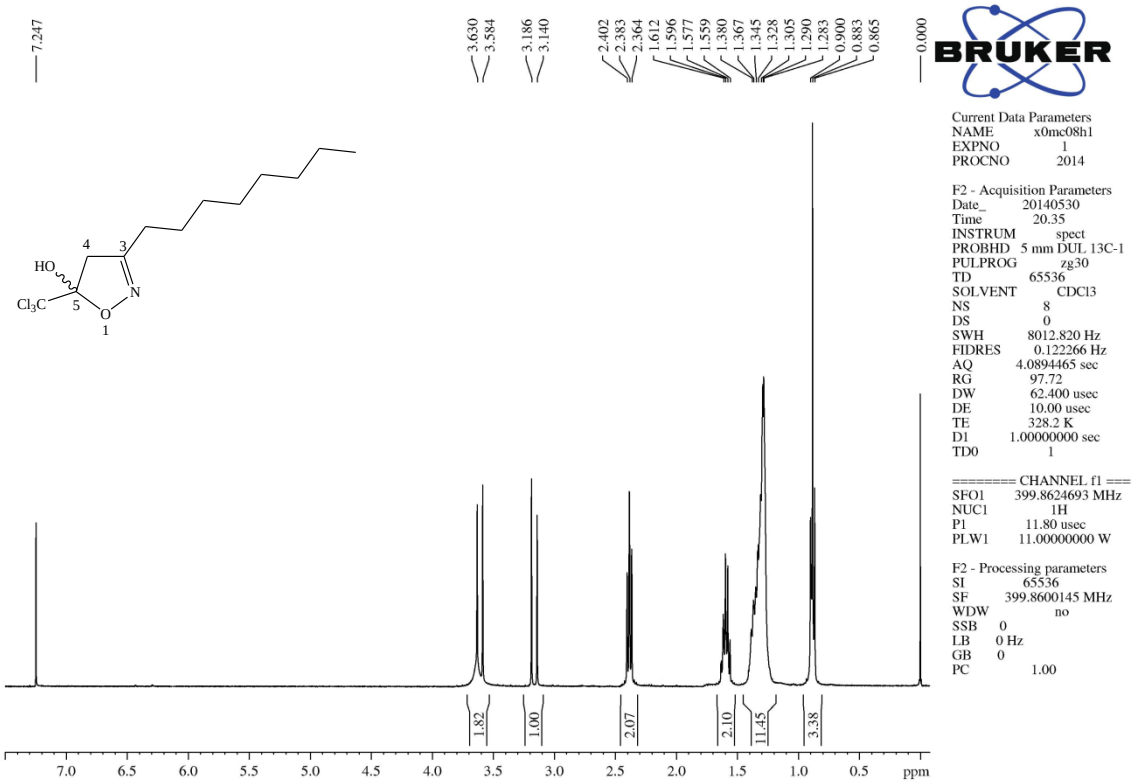


Figure S103. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of 5-trichloromethyl-3-octyl-5-hydroxy-4,5-dihydroisoxazole (**9b**).

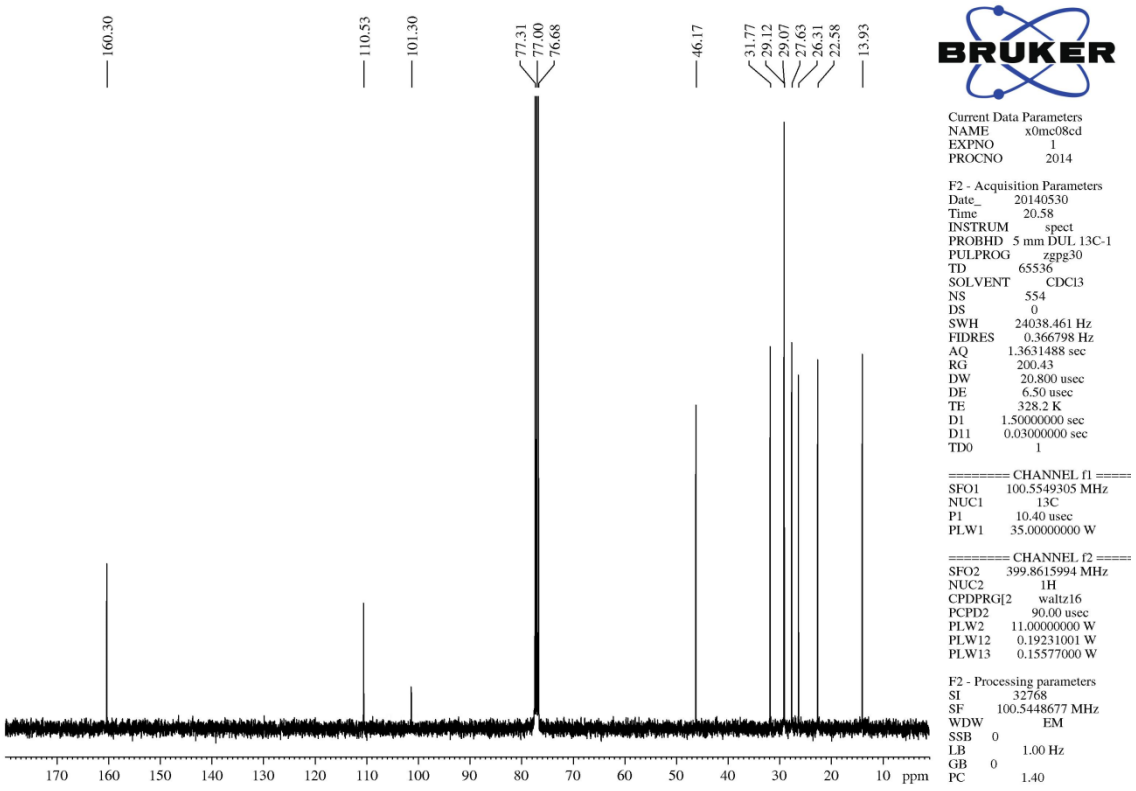


Figure S104. ^{13}C NMR spectrum (400 MHz, $CDCl_3$) of 5-trichloromethyl-3-octyl-5-hydroxy-4,5-dihydroisoxazole (**9b**).

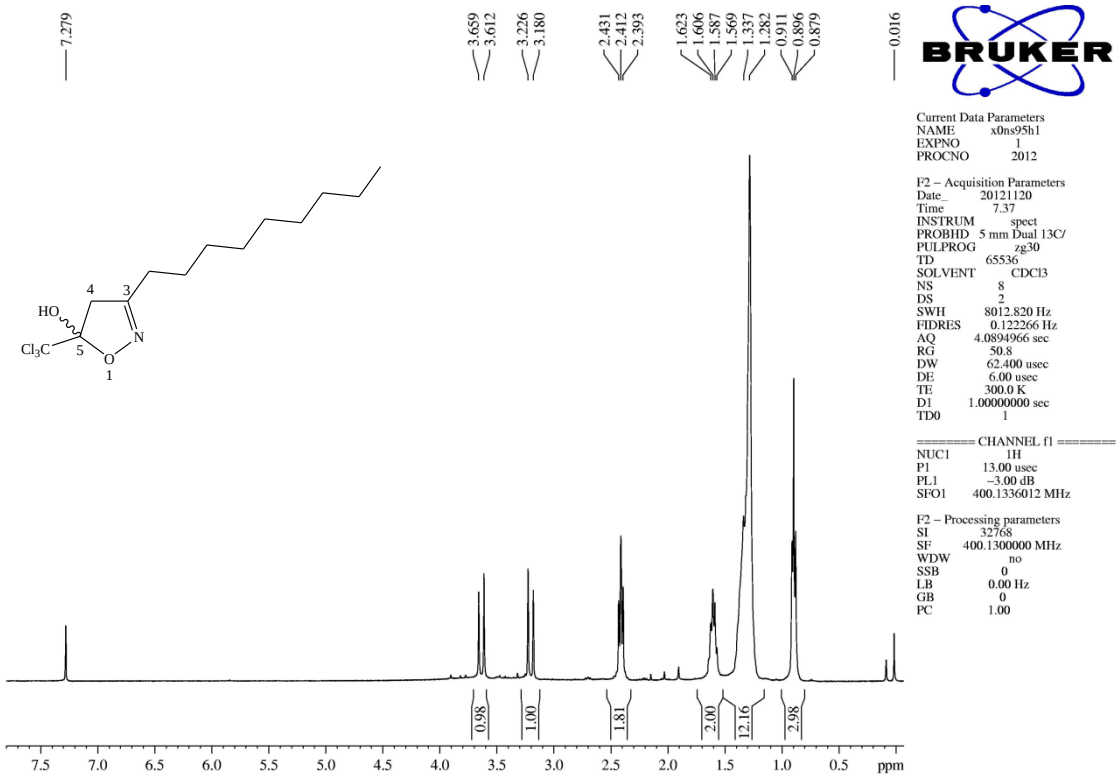


Figure S105. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of 5-trichloromethyl-3-nonyl-5-hydroxy-4,5-dihydroisoxazole (**9c**).

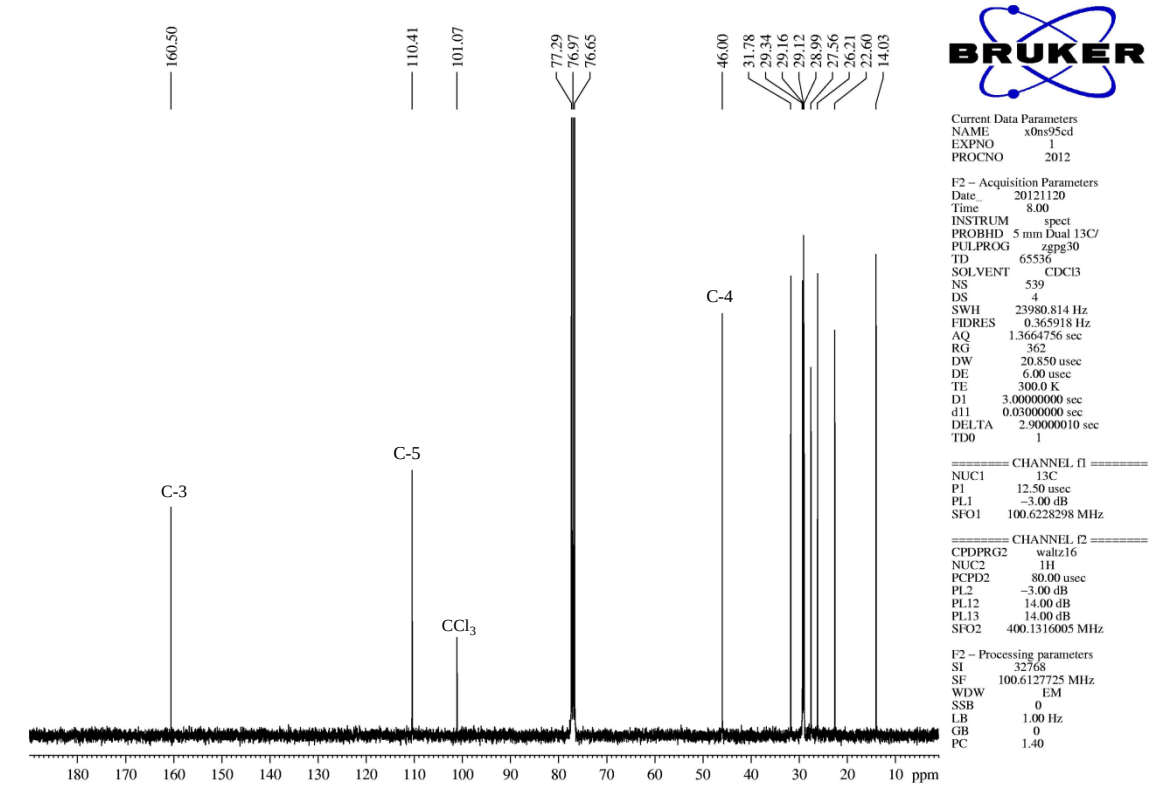


Figure S106. ^{13}C NMR spectrum (400 MHz, $CDCl_3$) of 5-trichloromethyl-3-nonyl-5-hydroxy-4,5-dihydroisoxazole (**9c**).

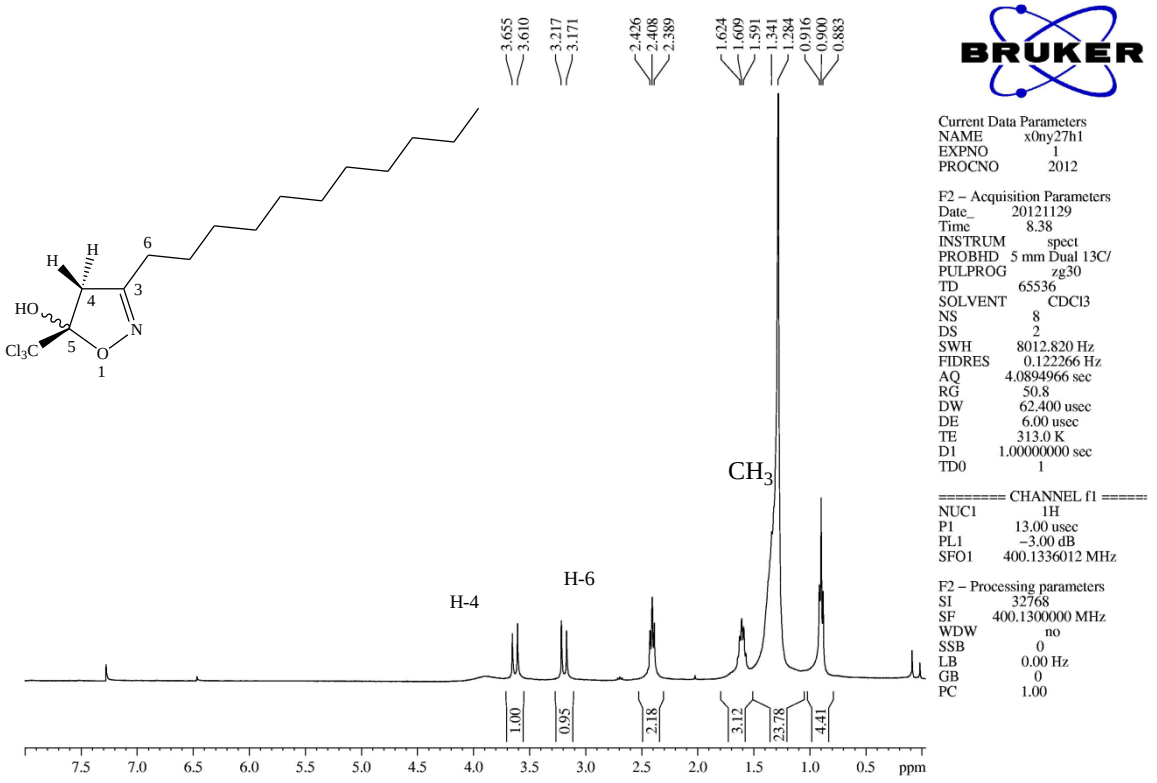


Figure S107. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of 5-trichloromethyl-3-undecyl-5-hydroxy-4,5-dihydroisoxazole (**9e**).

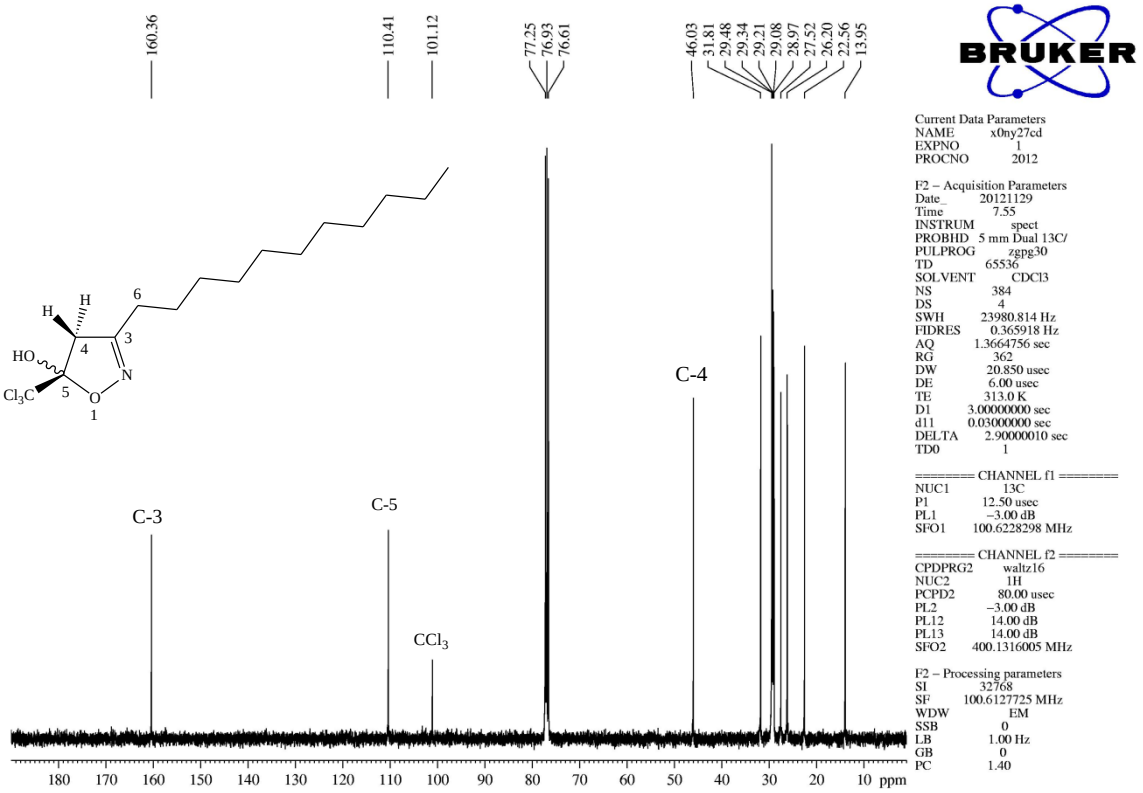


Figure S108. ^{13}C NMR spectrum (400 MHz, $CDCl_3$) of 5-trichloromethyl-3-undecyl-5-hydroxy-4,5-dihydroisoxazole (**9e**).

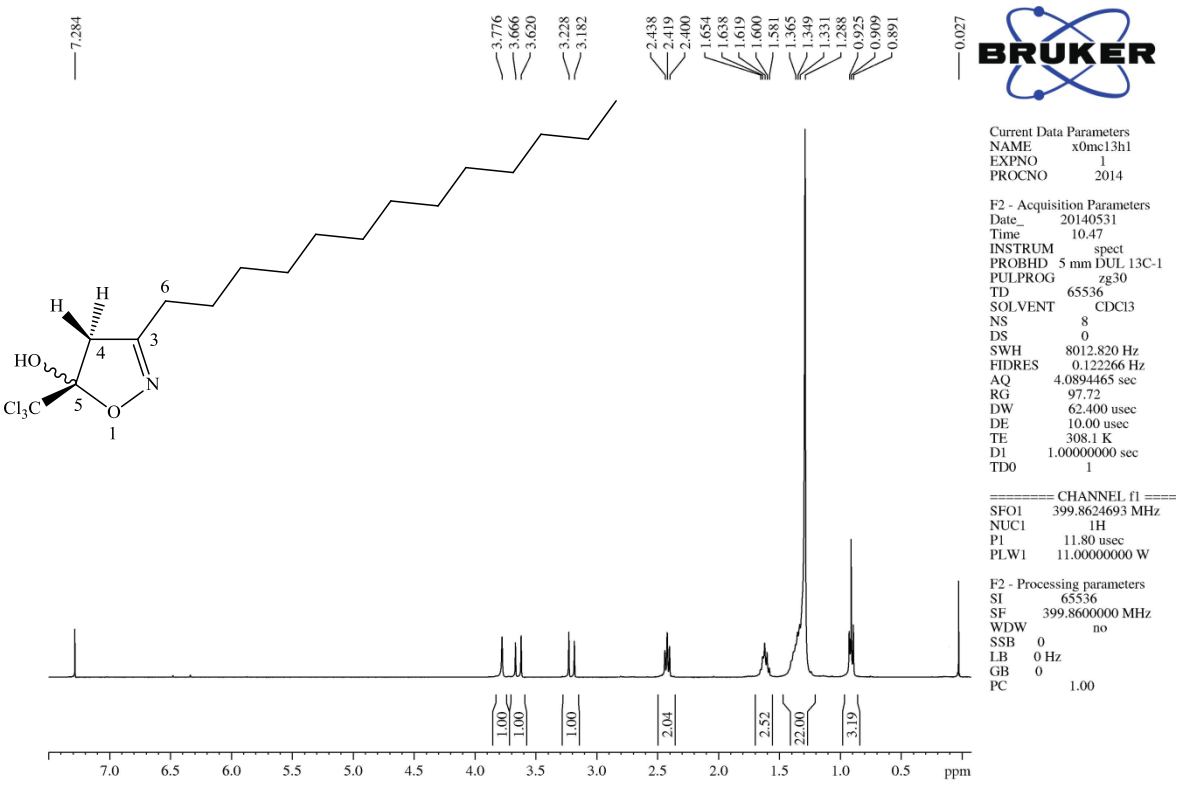


Figure S109. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of 5-trichloromethyl-3-tridecyl-5-hydroxy-4,5-dihydroisoxazole (**9f**).

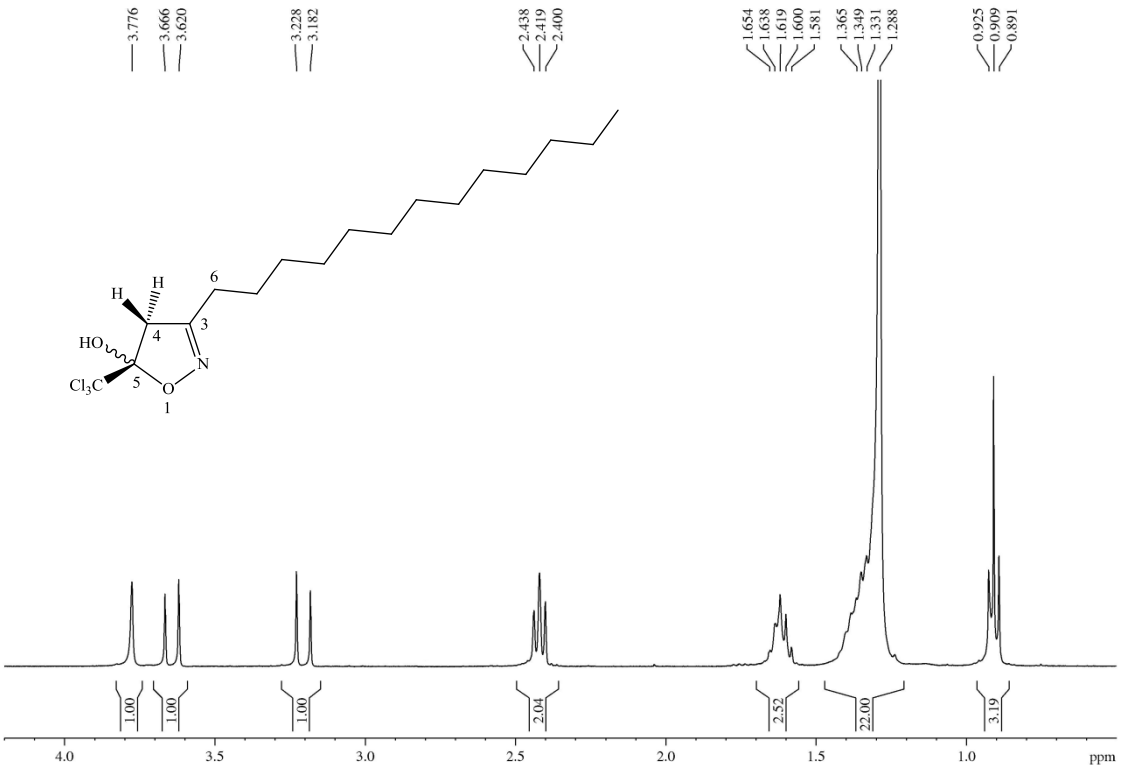


Figure S110. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of 5-trichloromethyl-3-tridecyl-5-hydroxy-4,5-dihydroisoxazole (**9f**), expanded between 0.6-4.2 ppm.

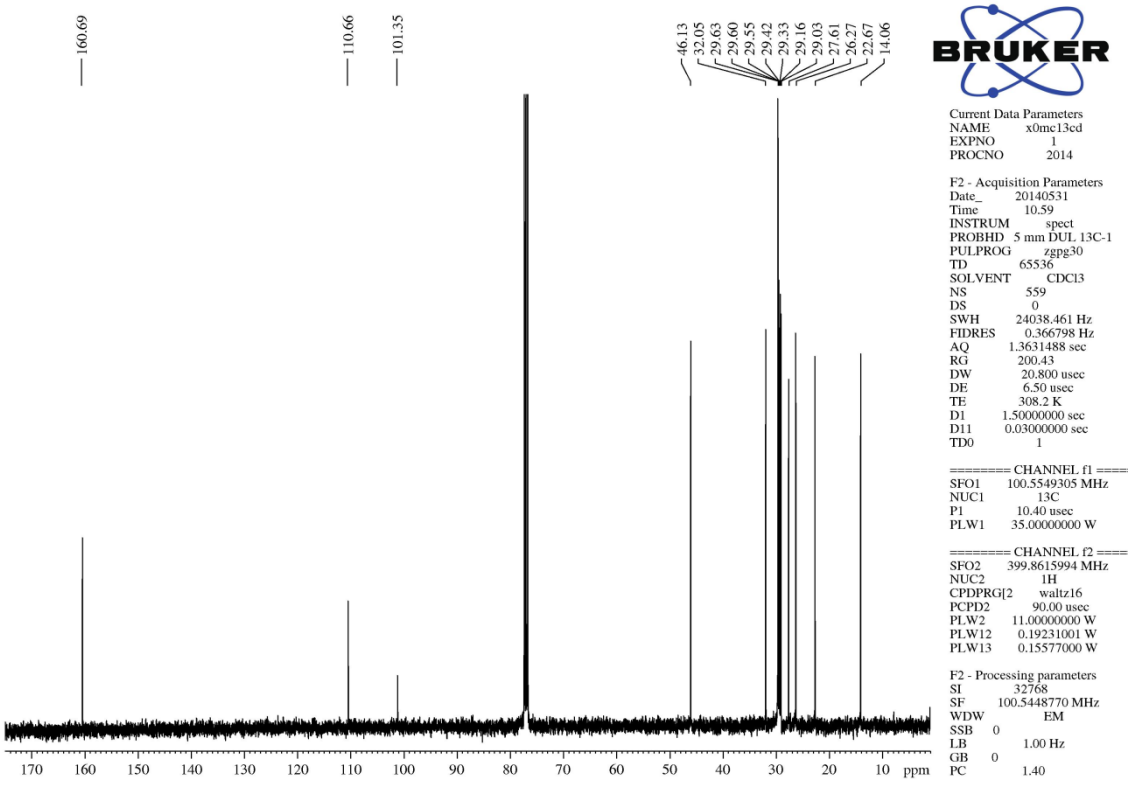


Figure S111. ^{13}C NMR spectrum (400 MHz, $CDCl_3$) of 5-trichloromethyl-3-tridecyl-5-hydroxy-4,5-dihydroisoxazole (**9f**).

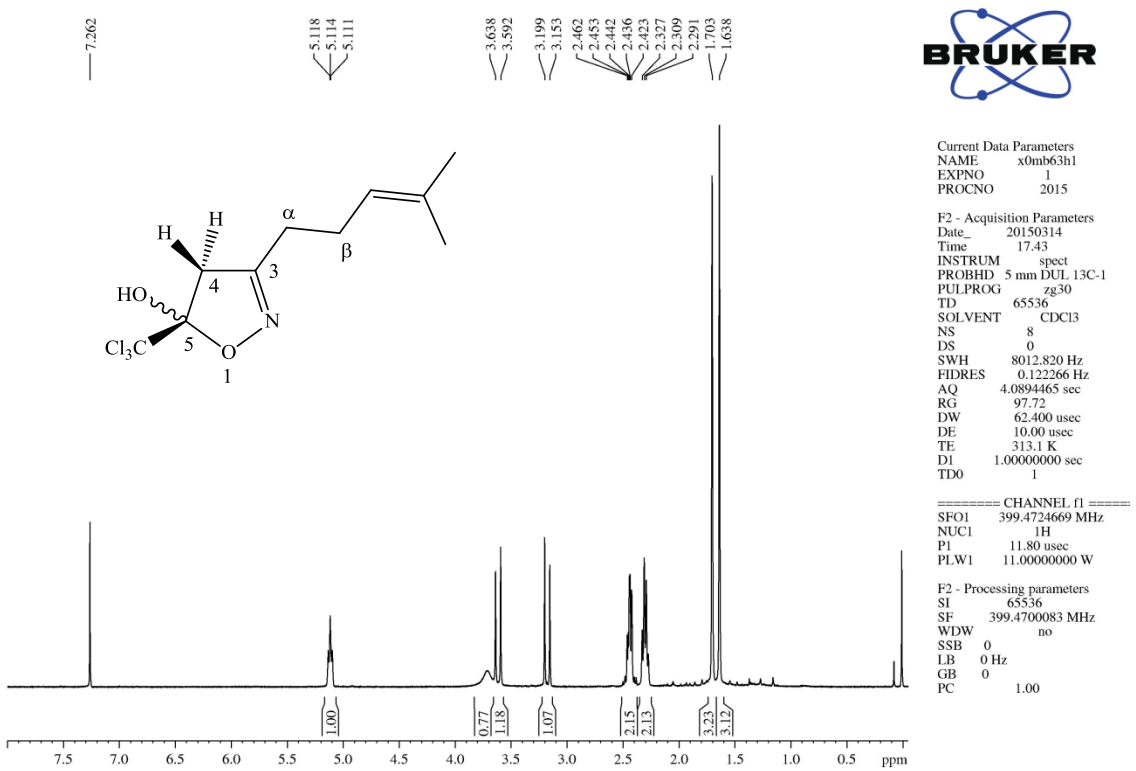


Figure S112. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of 5-trichloromethyl-3-(4-methyl-3-penten-1-yl)-5-hydroxy-4,5-dihydroisoxazole (**9g**).

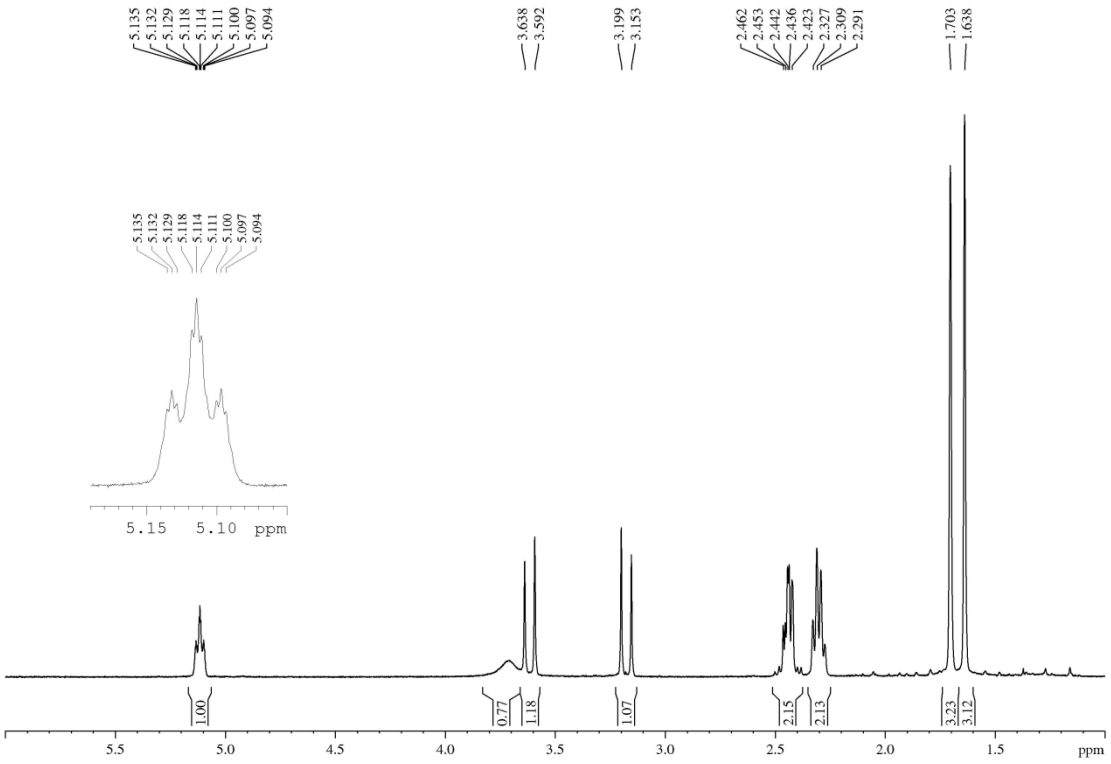


Figure S113. ^{1}H NMR spectrum (400 MHz, $CDCl_3$) of 5-trichloromethyl-3-(4-methyl-3-penten-1-yl)-5-hydroxy-4,5-dihydroisoxazole (**9g**), expanded between 1.0-6.0 ppm.

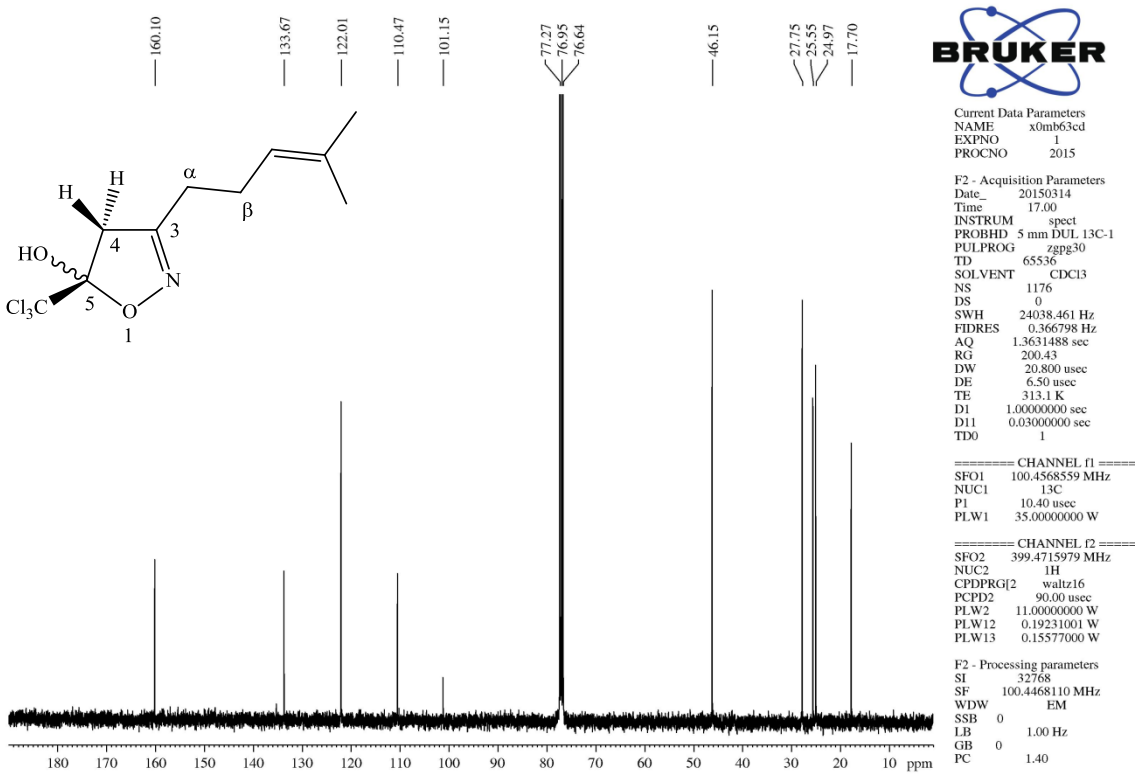


Figure S114. ^{13}C NMR spectrum (400MHz, $CDCl_3$) of 5-trichloromethyl-3-(4-methyl-3-penten-1-yl)-5-hydroxy-4,5-dihydroisoxazole (**9g**).

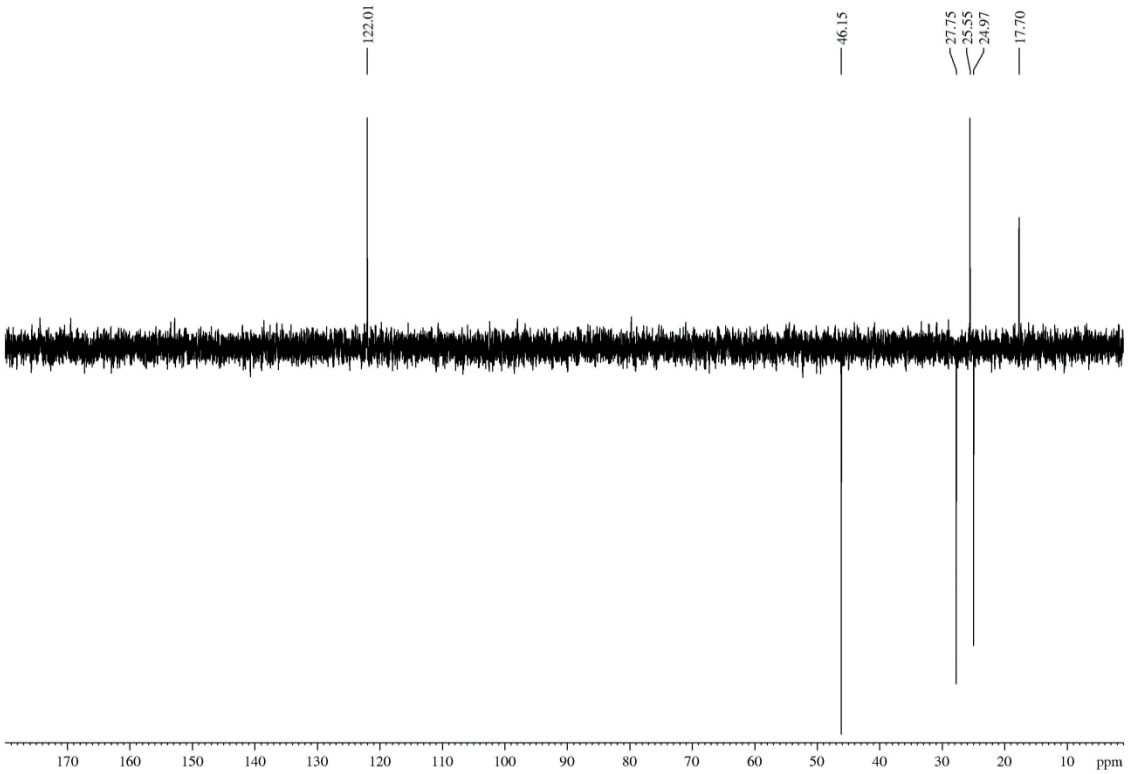


Figure S115. ^{13}C DEPT NMR spectrum (400MHz, $CDCl_3$) of of 5-trichloromethyl-3-(4-methyl-3-penten-1-yl)-5-hydroxy-4,5-dihydroisoxazole (**9g**).

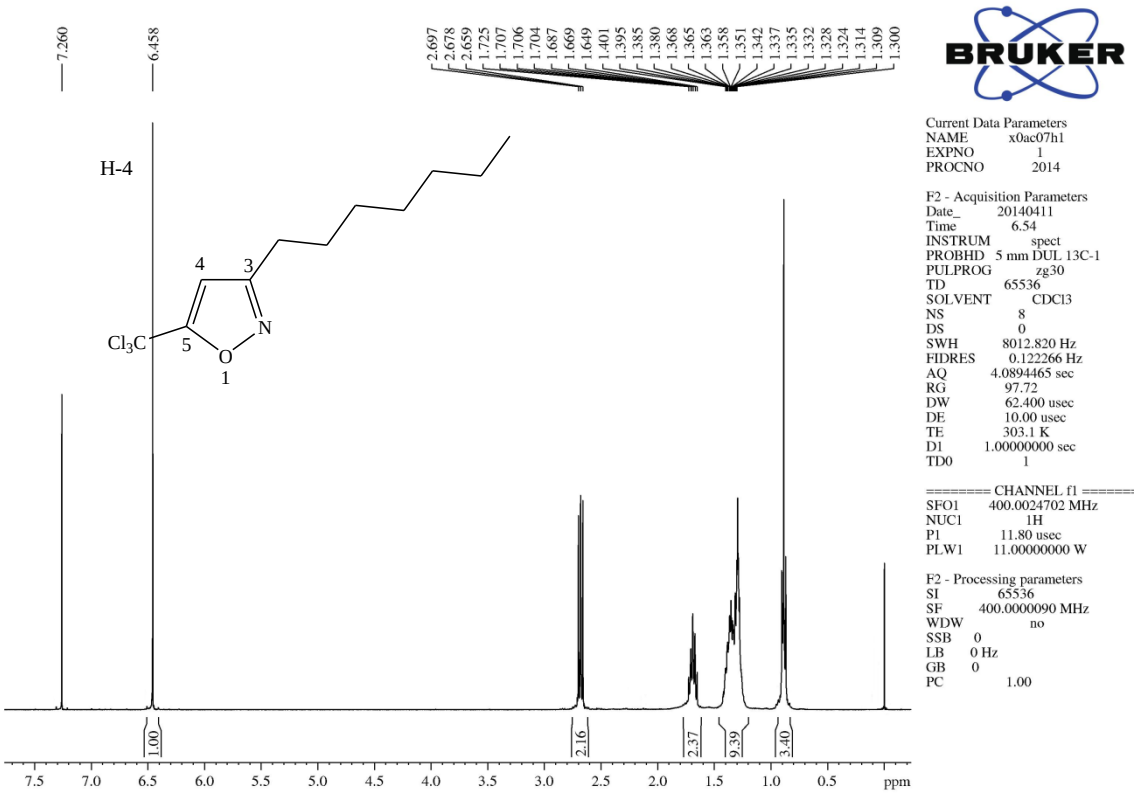


Figure S116. ^{1}H NMR spectrum (400MHz, $CDCl_3$) of 5-trichloromethyl-3-heptylisoxazole (**11a**).

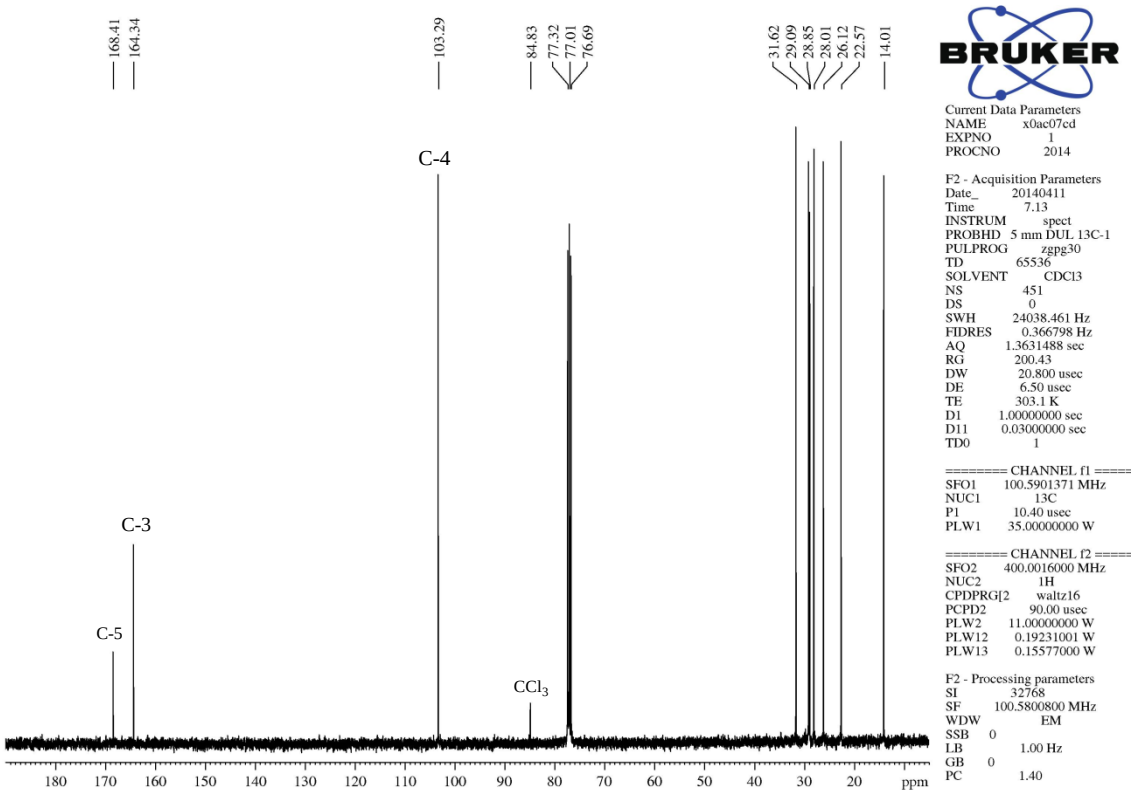


Figure S117. ^{13}C NMR spectrum (400MHz, $CDCl_3$) of 5-trichloromethyl-3-heptylisoxazole (**11a**).

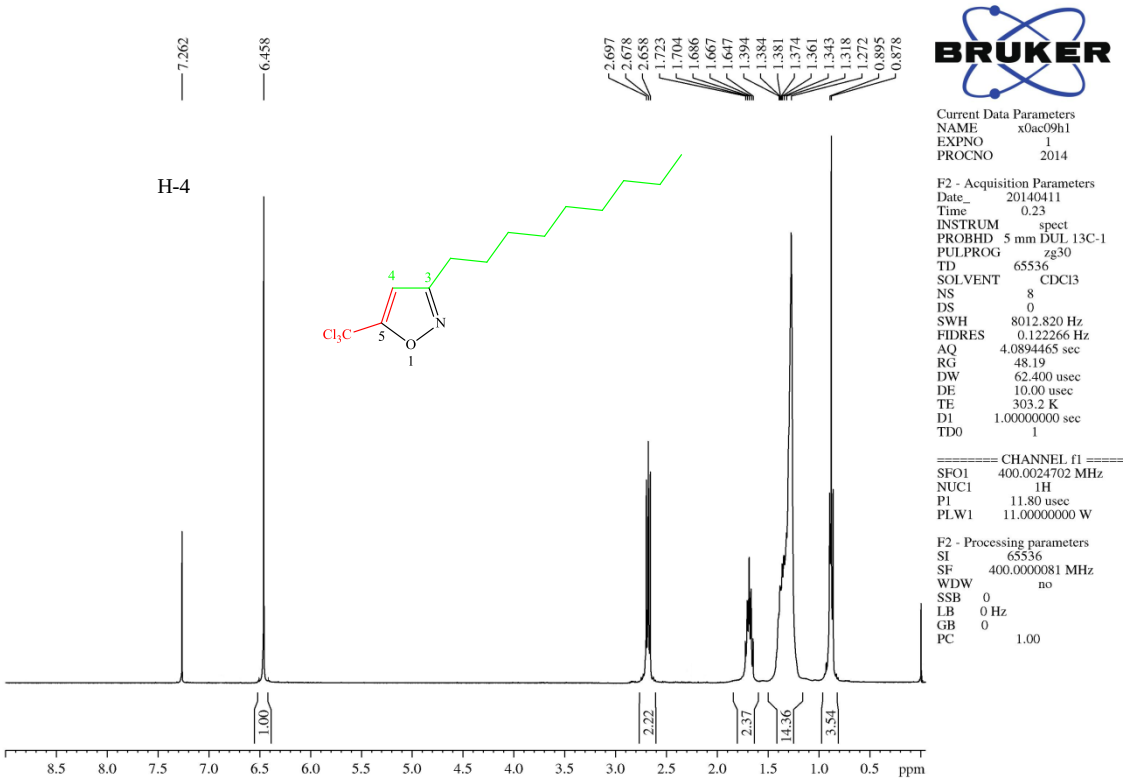


Figure S118. ^{1}H NMR spectrum (400MHz, $CDCl_3$) of 5-trichloromethyl-3-nonylisoxazole (**11d**).

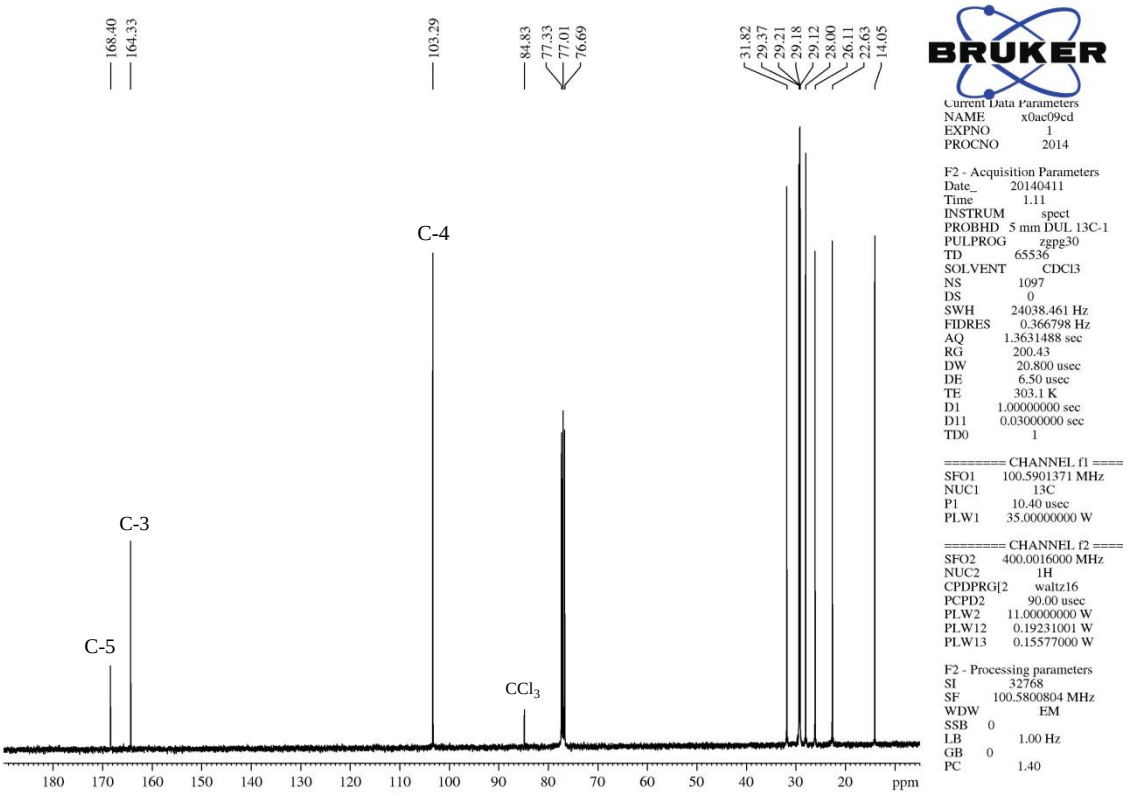


Figure S119. ^{13}C NMR spectrum (400MHz, $CDCl_3$) of 5-trichloromethyl-3-nonylisoxazole (**11d**).

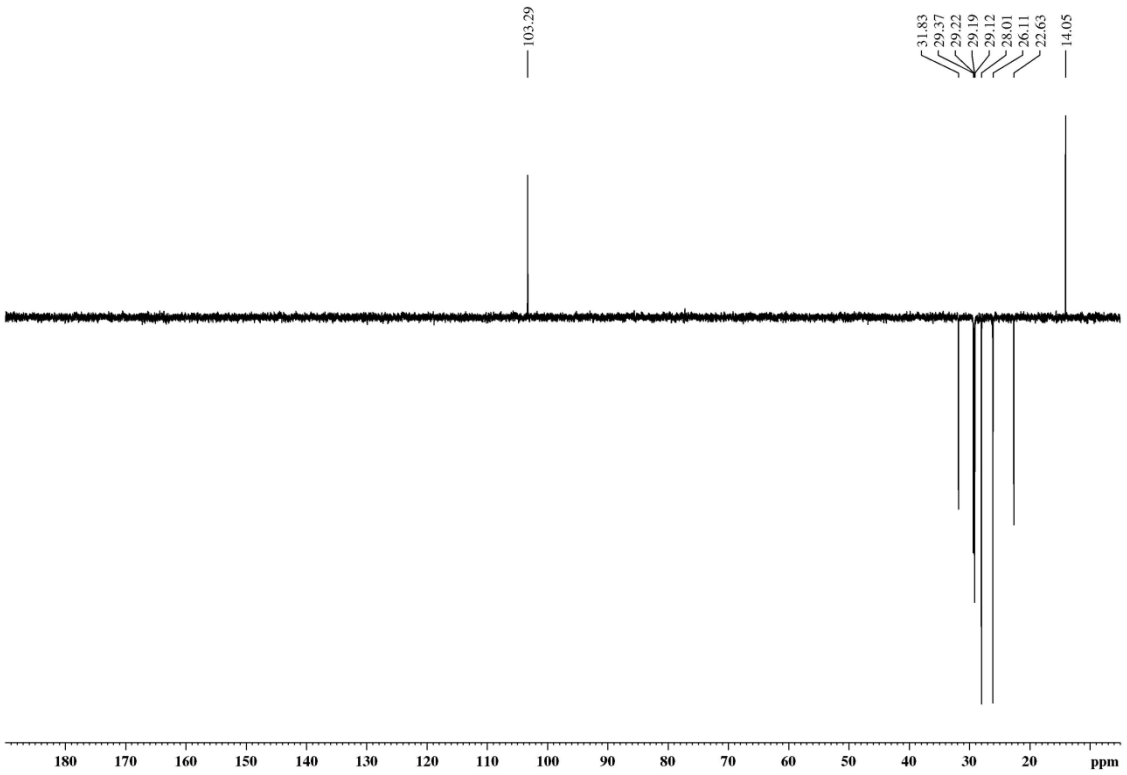


Figure S120. ^{13}C DEPT135 NMR (400 MHz, $CDCl_3$) spectrum of 5-trichloromethyl-3-nonylisoxazole (**11d**).

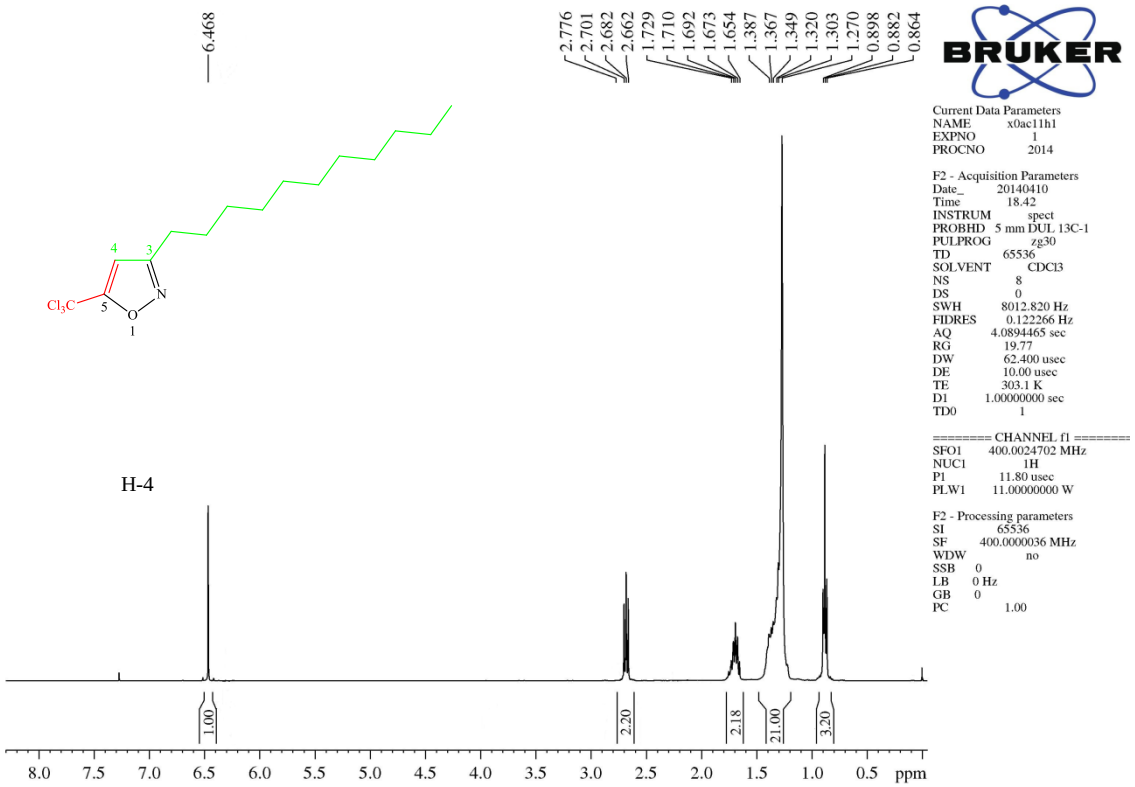


Figure S121. ^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 5-trichloromethyl-3-undecylisoxazole (**11e**).

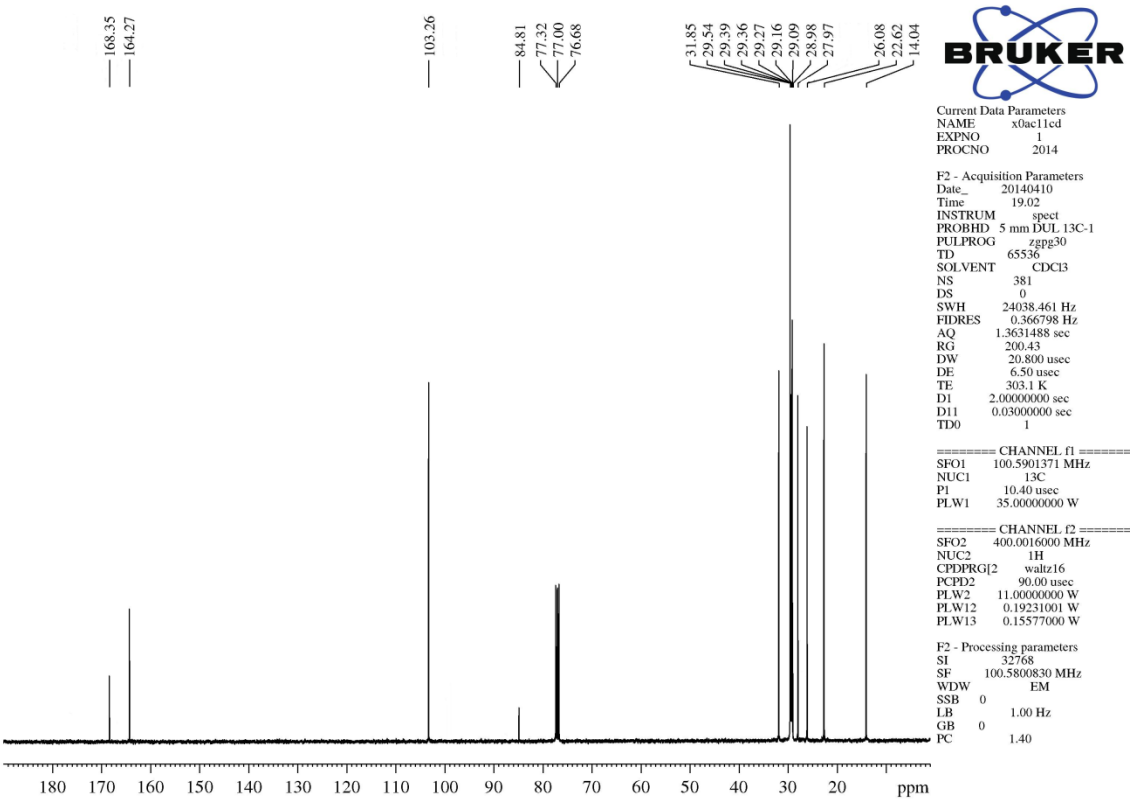


Figure S122. ^{13}C NMR (400 MHz, $CDCl_3$) spectrum of 5-trichloromethyl-3-undecylisoxazole (**11e**).

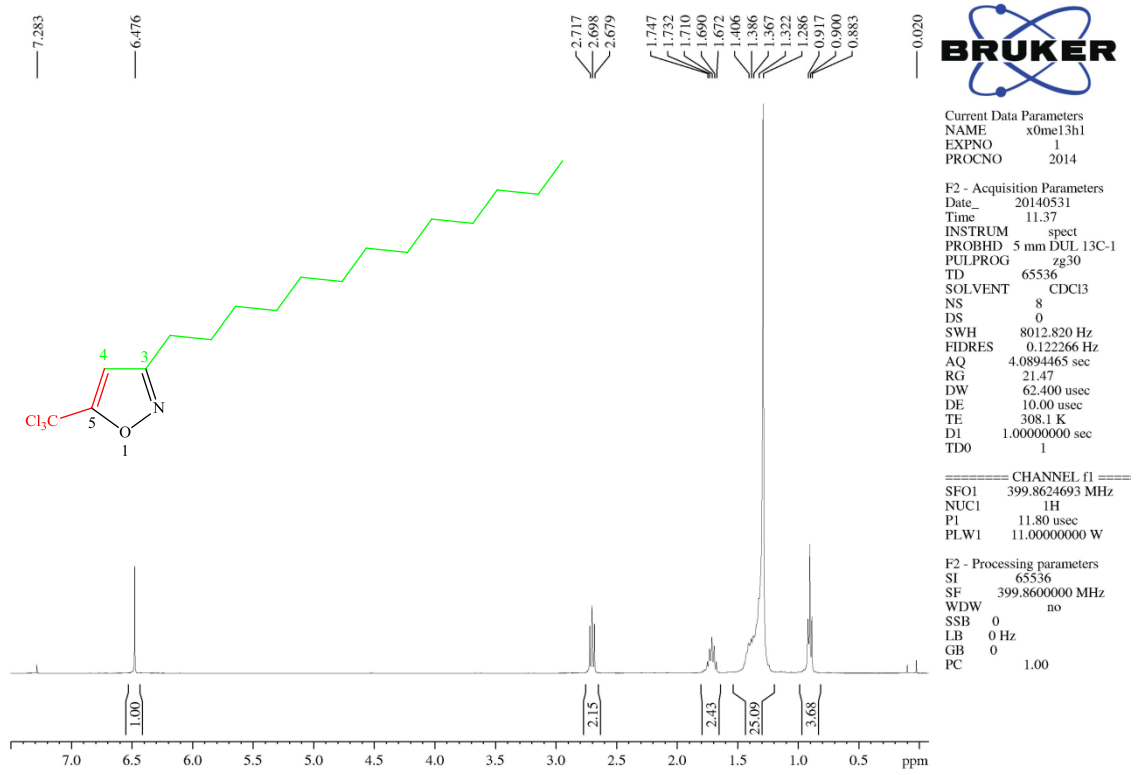


Figure S123. ^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 5-trichloromethyl-3-tridecylisoxazole (**11f**).

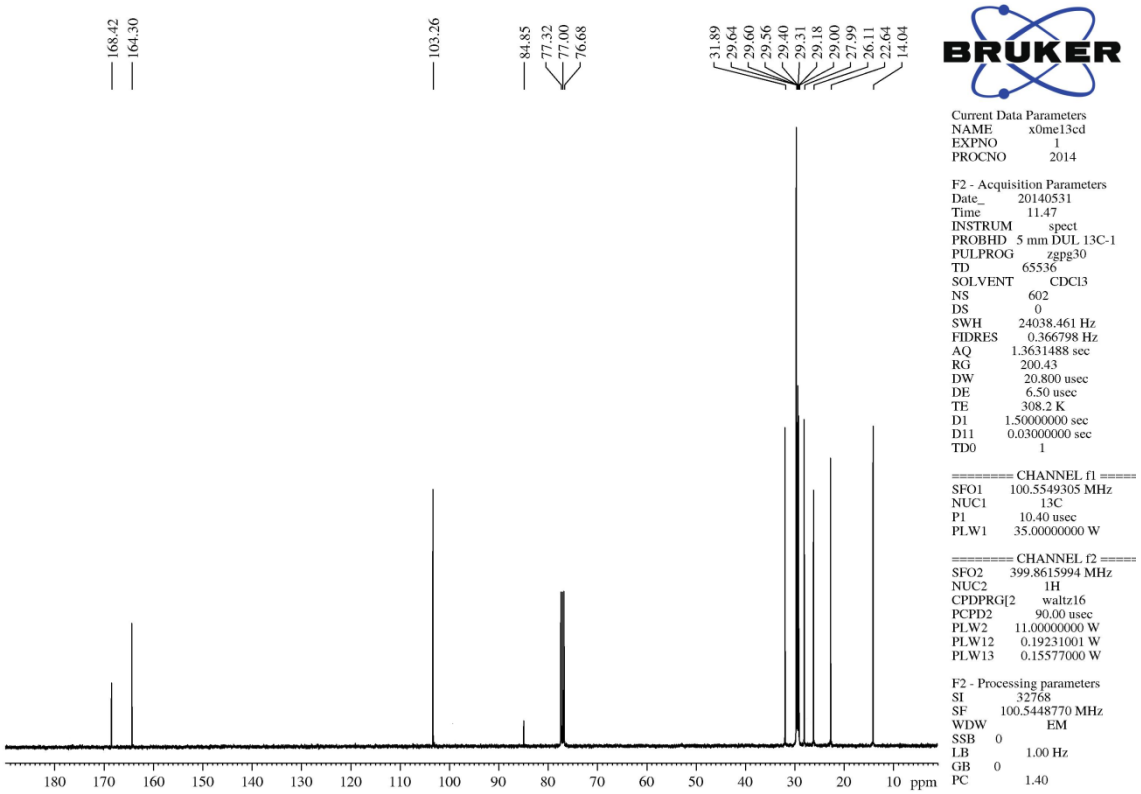
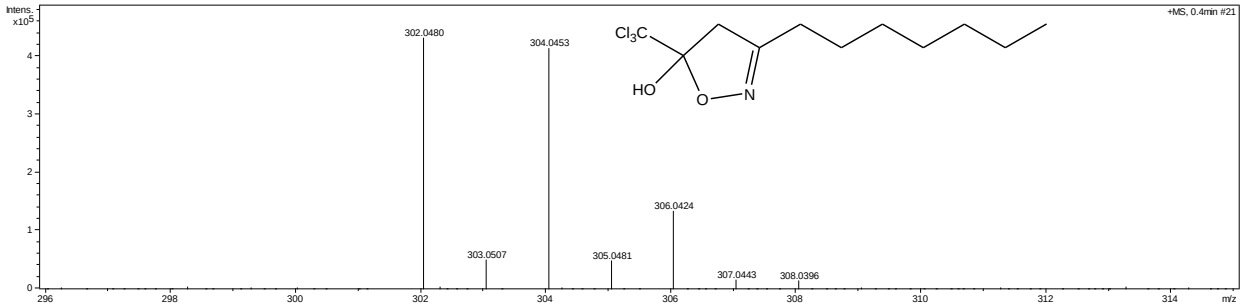


Figure S124. ^{13}C NMR (400 MHz, $CDCl_3$) spectrum of 5-trichloromethyl-3-tridecylisoxazole (**11f**).

MS



MS2

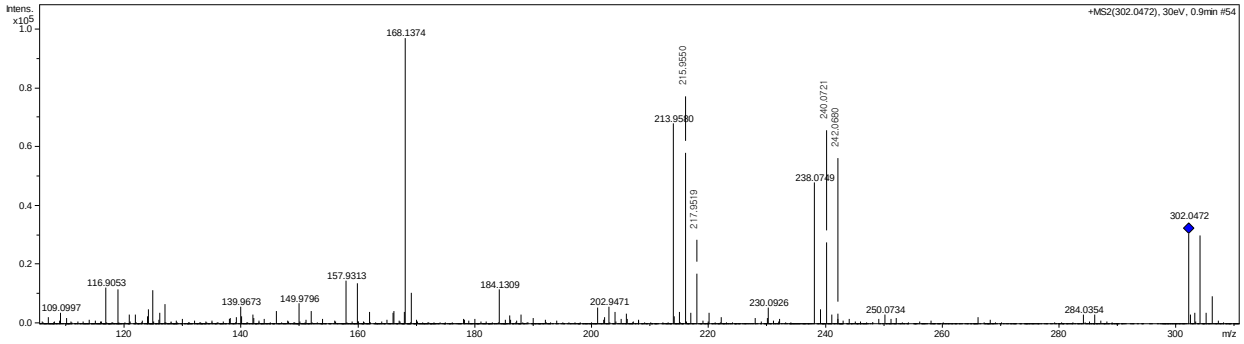
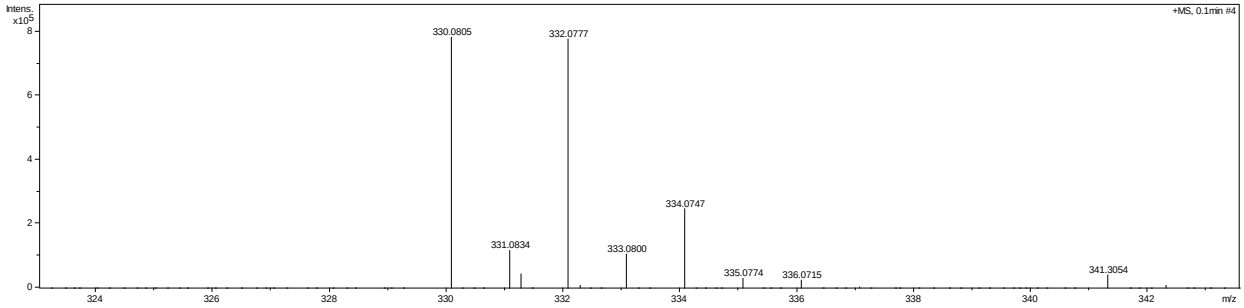


Figure S125. Mass spectra of 5-trichloromethyl-3-heptyl-5-hydroxy-4,5-dihydro isoxazole (**9a**).

MS



MS2

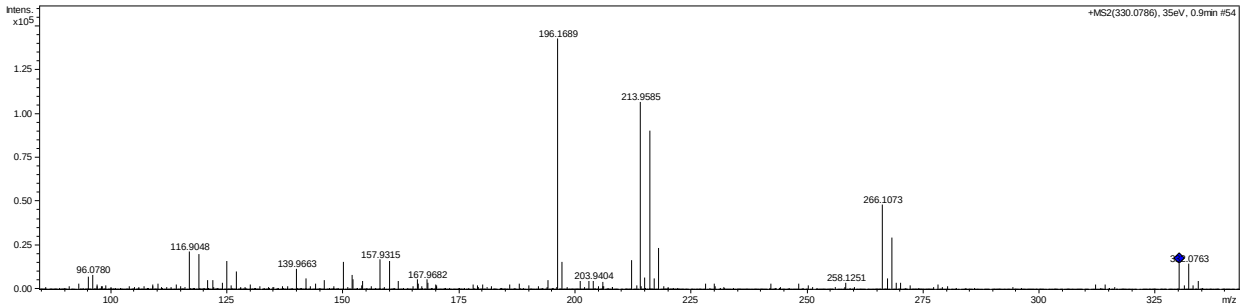
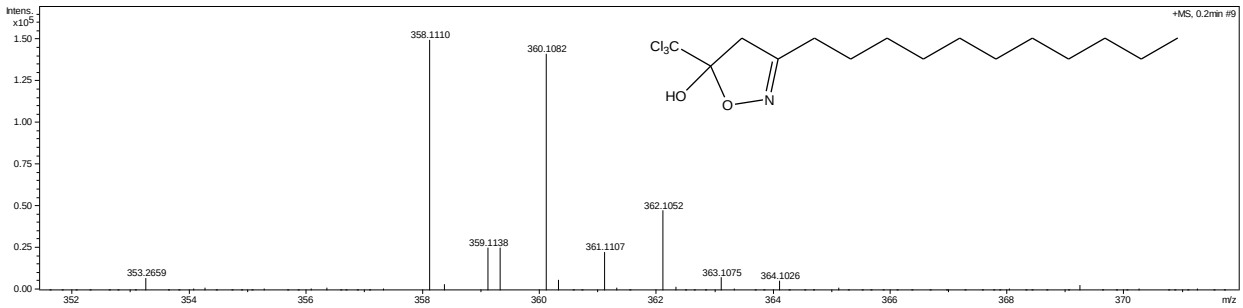


Figure S126. Mass spectra of 5-trichloromethyl-3-nonyl-5-hydroxy-4,5-dihydroisoxazole (**9d**).

MS



MS 2

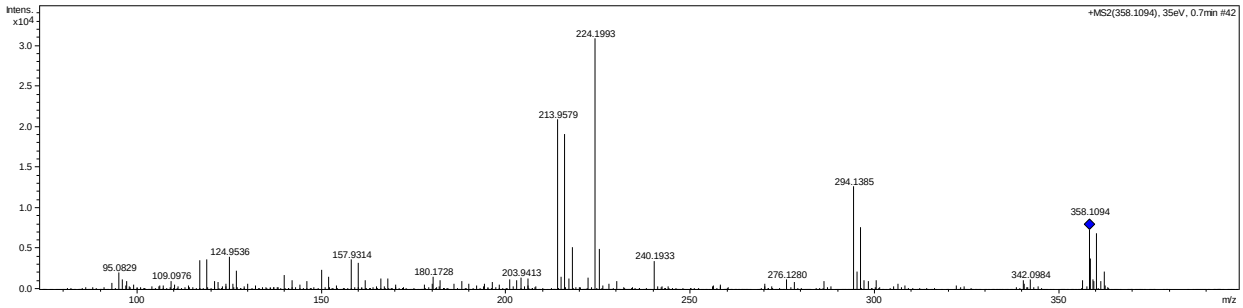
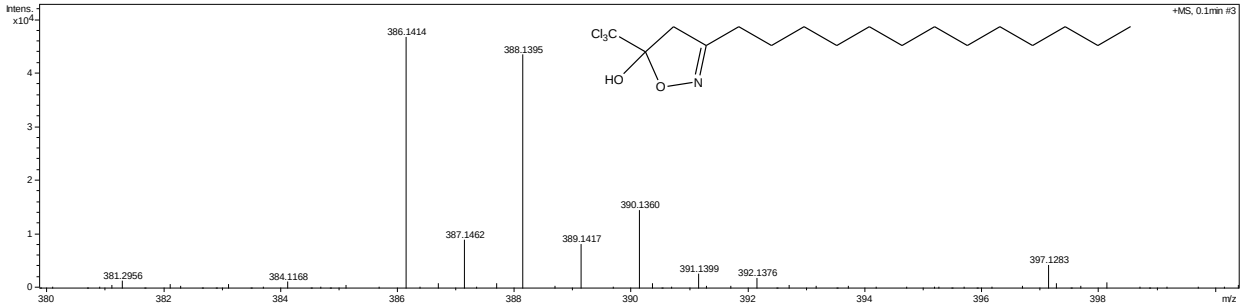


Figure S127. Mass spectra of 5-trichloromethyl-3-undecyl-5-hydroxy-4,5-dihydroisoxazole (**9e**)

MS



MS2

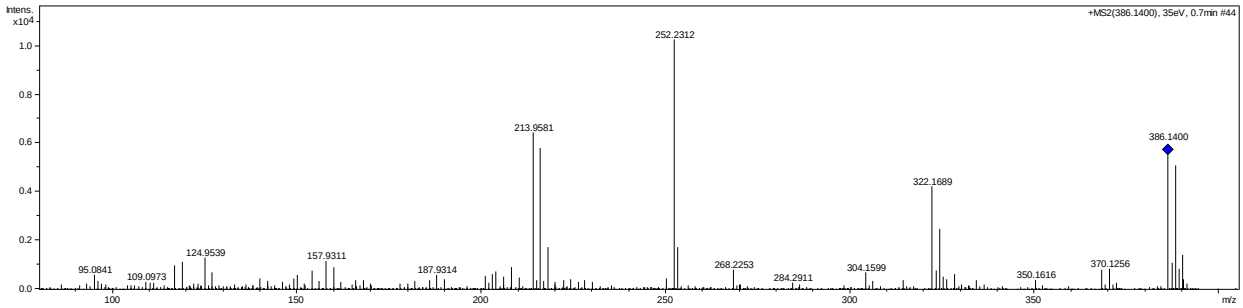
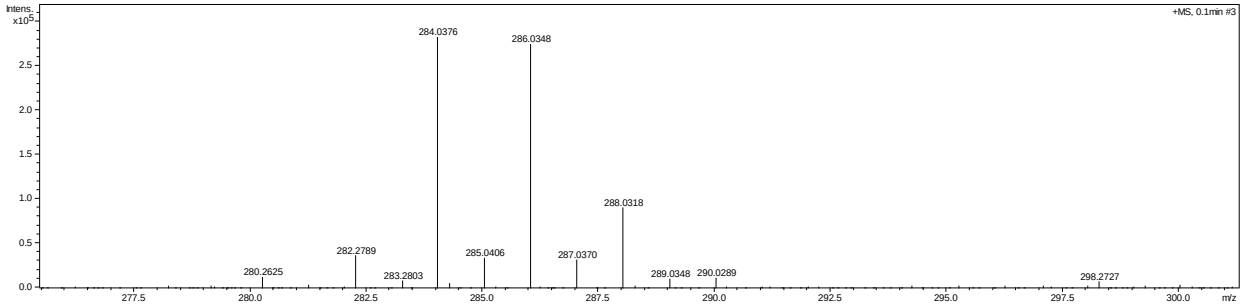


Figure S128. Mass spectra of 5-trichloromethyl-3-tridecyl-5-hydroxy-4,5-dihydroisoxazole (**9f**).

MS



MS2

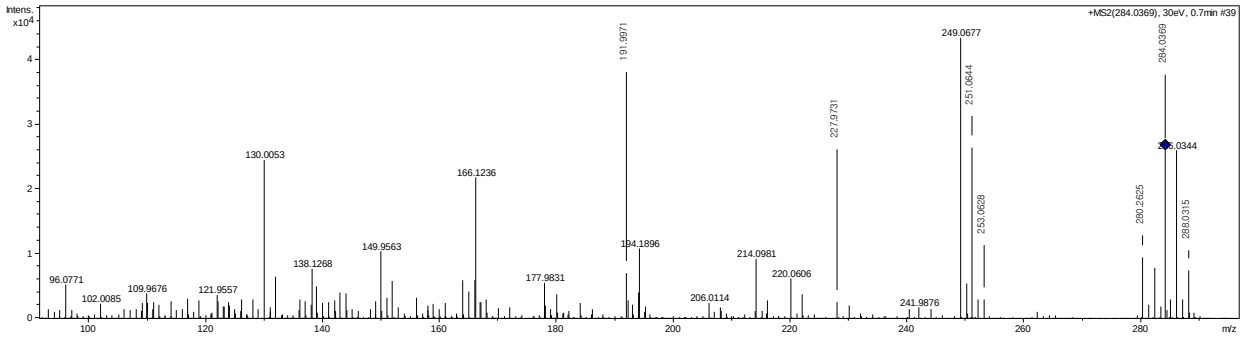
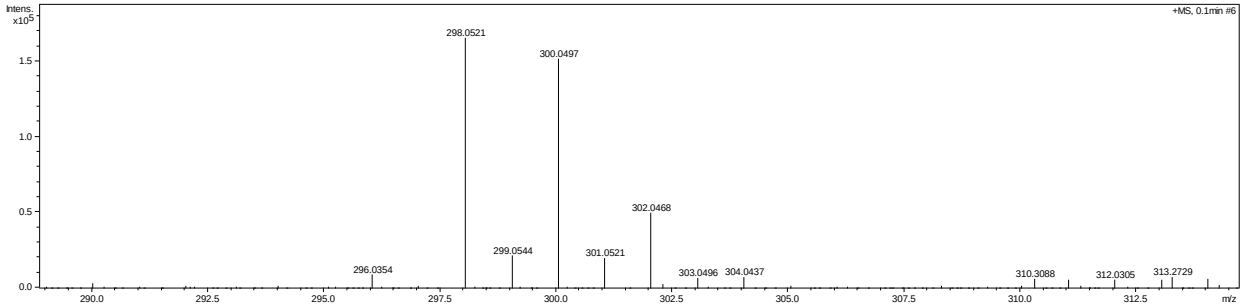


Figure S129. Mass spectra of of 5-trichloromethyl-3-heptylisoxazole (**11a**).

MS



MS2

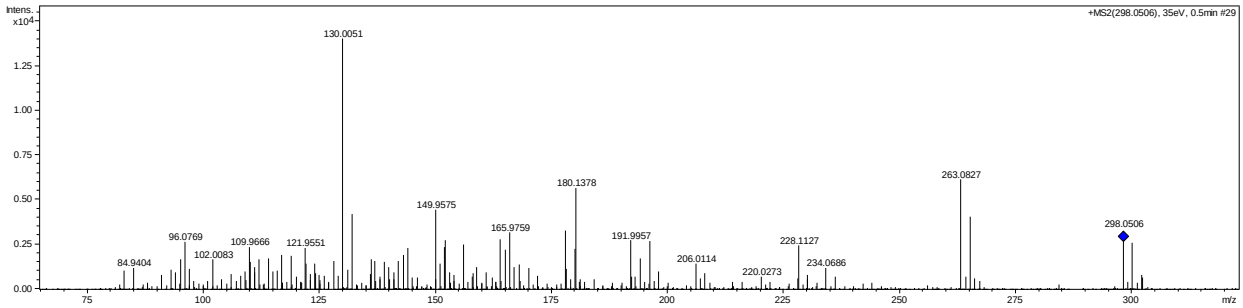
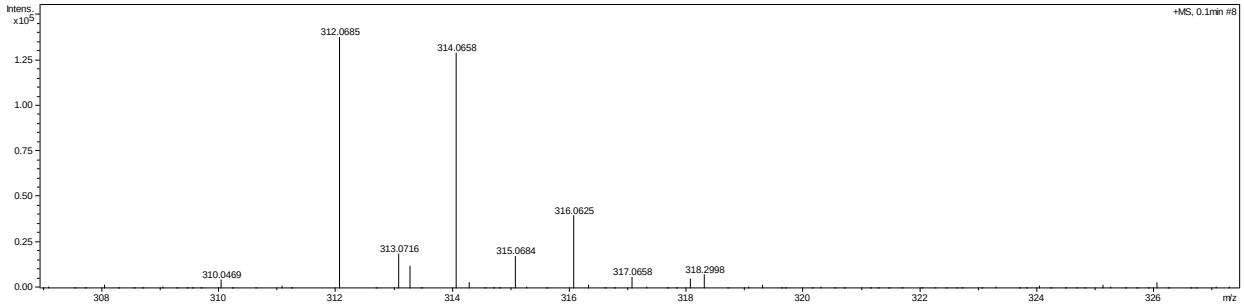


Figure S130. Mass spectra of of 5-trichloromethyl-3-octylisoxazole (**11c**).

MS



MS2

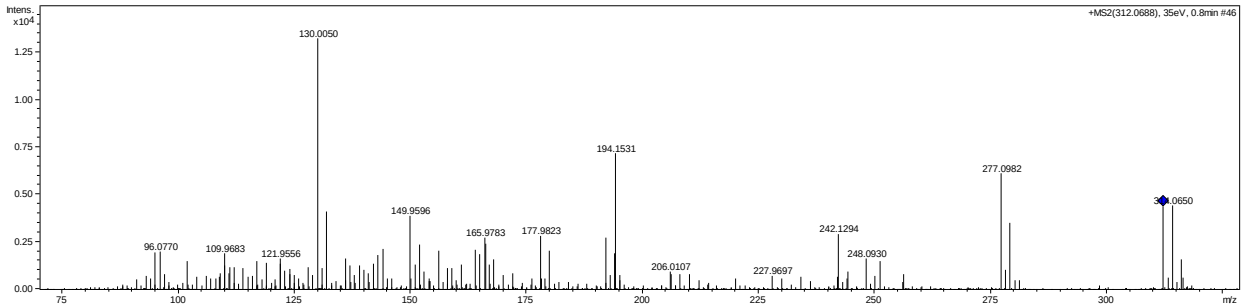
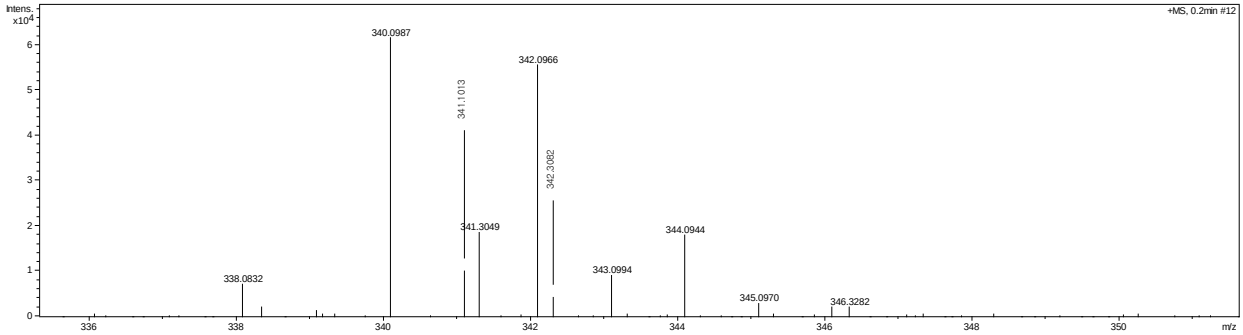


Figure S131. Mass spectra of of 5-trichloromethyl-3-nonylisoxazole (**11d**).

MS



MS2

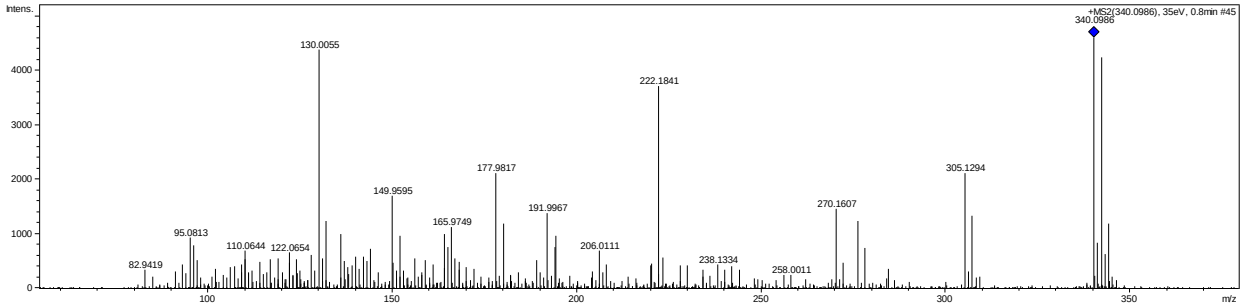


Figure S132. Mass spectra of of 5-trichloromethyl-3-undecylisoxazole (**11e**).

Table S1. ^{1}H NMR data for 5-hydroxy-5-trichloromethyl-4,5-dihydroisoxazoles (**9a**-**g**)

Compound	δ / ppm (multiplicity, J / Hz)					
(yield / %)	H-4a	H-4b	α-CH_2	β-CH_2	Other	Me
9a (85)	3.55 (d, 18.4)	3.12 (d, 18.4)	2.32 (t, 7.6)	1.51 (m)	1.25 (6H, m, 3CH_2)	0.81 (t, 6.8)
9b (92)	3.55 (d, 18.4)	3.12 (d, 18.4)	2.32 (t, 7.6)	1.51 (m)	1.49 (2H, m, CH_2); 1.23 (8H, m, -CH_2-)	0.81 (t, 6.8)
9c (95)	3.60 (d, 18.5)	3.16 (d, 18.5)	2.38 (t, 7.6)	1.58 (m)	1.58 (2H, m, CH_2); 1.33 (10H, m, -CH_2-)	0.88 (t, 6.8)
9d (92)	3.63 (d, 18.4)	3.20 (t, 18.4)	2.41 (t, 7.6)	1.58 (m)	1.59 (2H, m, CH_2); 1.33 (12H, m, -CH_2-)	0.89 (t, 6.8)
9e (90)	3.63 (d, 18.4)	3.19 (d, 18.4)	2.41 (t, 7.8)	1.60 (m)	1.26-1.34 (16H, m, –CH_2–)	0.90 (t, 6.8)
9f (69)	3.64 (d, 18.4)	3.20 (d, 18.4)	2.40 (t, 7.6)	1.58 (m)	1.27 (18H, m, –CH_2–)	0.90 (t, 6.8)
9g (89)	3.61 (d, 18.4)	3.17 (d, 18.4)	2.44 (m)	2.30 (m)	5.11 (tt, 7.0 and 1.2); 1.70 (s, 3H, Me); 1,63 (s, 3H, Me)	–

δ: chemical shift; J: coupling constant; d: doublet; t: triplet; m: multiplet; tt: triplet of triplet.

Table S2. ^{13}C NMR data for 5-hydroxy-5-trichloromethyl-4,5-dihydroisoxazoles (**9a**-**g**)

Compound	δ / ppm				
	C-3	C-4	C-5	CCl_3	Other
9a	160.6	46.1	110.5	101.1	31.3; 28.6; 27.5; 26.1; 22.4; 13.9
9b	160.7	46.1	110.5	101.1	31.5; 28.9; 28.8; 27.5; 26.2; 22.5; 14.0
9c	160.5	46.0	110.4	101.0	31.8; 29.3; 29.16; 29.12; 28.9; 27.5; 26.2; 22.6; 14.0
9d	160.3	46.0	110.4	101.1	31.8; 29.5; 29.3; 29,2; 29.1; 28.9; 27.5; 26.2; 22.5; 13.9
9e	160.5	46.0	110.4	101.1	31.8; 29.6; 29.57; 29.5; 29.4; 29.3; 29.1; 27.5; 26.2; 22.6; 14.0
9f	160.7	46.1	110.4	101.5	31.8; 29.6; 29.57; 29.5; 29.4; 29.3; 29.1; 29.0; 27.5; 26.2; 22.6; 14.0
9g	160.1	46.1	110.5	101.1	133.6; 122.0; 27.7; 25.5; 24.9; 17.7

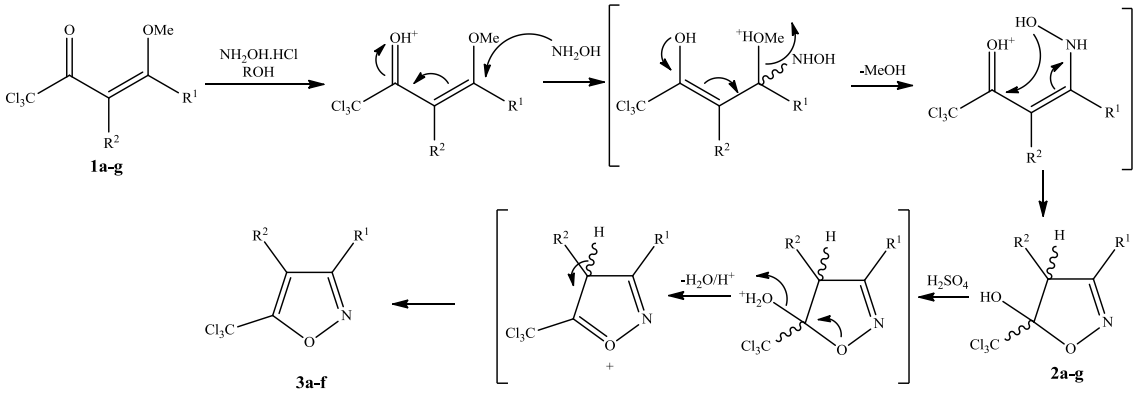
Table S3. ^{1}H NMR data for 5-trichloromethylisoxazoles (**11a**-**f**)

Compound	δ / ppm (multiplicity, J / Hz)				
(yield / %)	H-4	α-CH_2	β-CH_2	Other	Me
11a (95)	6.45 (s)	2.67 (t, 7.6)	1.70 (m)	1.25-1.40 (6H, m, –CH_2–)	0.88 (t, 7.2)
11b (92)	6.46 (s)	2.68 (t, 7.6)	1.69 (m)	1.25-1.40 (8H, m, –CH_2–)	0.88 (t, 7.0)
11c (95)	6.45 (s)	2.67 (t, 7.6)	1.70 (m)	1.28-1.38 (10H, m, –CH_2–)	0.88 (t, 7.2)
11d (92)	6.43 (s)	2.68 (t, 7.6)	1.58 (m)	1.26-1.40 (12H, m, –CH_2–)	0.89 (t, 7.0)
11e (95)	6.45 (s)	2.67 (t, 7.8)	1.60 (m)	1.26-1.34 (16H, m, –CH_2–)	0.88 (t, 7.2)
11f (96)	6.64 (s)	2.68 (t, 7.6)	1.58 (m)	1.27-1.35 (18H, m, –CH_2–)	0.89 (t, 7.2)

δ: chemical shift; J: coupling constant; s: singlet; t: triplet; m: multiplet.

Table S4. ^{13}C NMR data for 5-trichloromethylisoxazoles (**11a**-**f**)

Compound	δ / ppm				
	C-3	C-4	C-5	CCl_3	Other
11a	168.4	103.3	164.3	84.8	31.6; 29.0; 28.0; 26.1; 22.6; 14.0
11b	168.4	103.3	164.3	84.8	31.6; 29.1; 28.8; 28.1; 26.1; 22.5; 14.0
11c	168.5	103.3	164.3	84.8	31.8; 29.4; 29.2; 29.18; 29.1; 28.0; 26.1; 22.6; 14.0
11d	168.5	103.3	164.3	84.8	31.8; 29.5; 29.4; 29.3; 29.16; 29.1; 27.9; 26.1; 22.6; 14.0
11e	168.4	103.2	164.3	84.9	31.8; 29.6; 29.57; 29.5; 29.4; 29.3; 29.1; 27.5; 26.2; 22.6; 14.0
11f	168.4	103.2	164.3	84.8	31.9; 29.64; 29.6; 29.5; 29.4; 29.3; 29.2; 29.0; 28.0; 26.1; 22.6; 14.0



Scheme S1. Proposed reaction mechanism for cyclocondensation and dehydration process.