

SUPPORTING INFORMATION

Asymmetric Synthesis of Contact Sex Pheromone of *Tetropium fuscum* and Its Enantiomer

Xueyang Wang ¹, Jianan Wang ¹, Fengbo Ma ², Qinghua Bian ¹, Min Wang ¹
and Jiangchun Zhong ^{1,*}

¹ Department of Applied Chemistry, China Agricultural University,
2 West Yuanmingyuan Road, Beijing 100193, China

² College of Environmental Sciences, Sichuan Agricultural University,
211 Huimin Road, Chengdu 611130, China

* Correspondence: zhong@cau.edu.cn; Tel.: +86-010-6273-1356

Table of Contents

1. ¹ H, ¹³ C NMR Spectra of the Products.....	S2
2. HPLC Chromatography of the Compounds.....	S17

1. ^1H , ^{13}C NMR Spectra of the Products

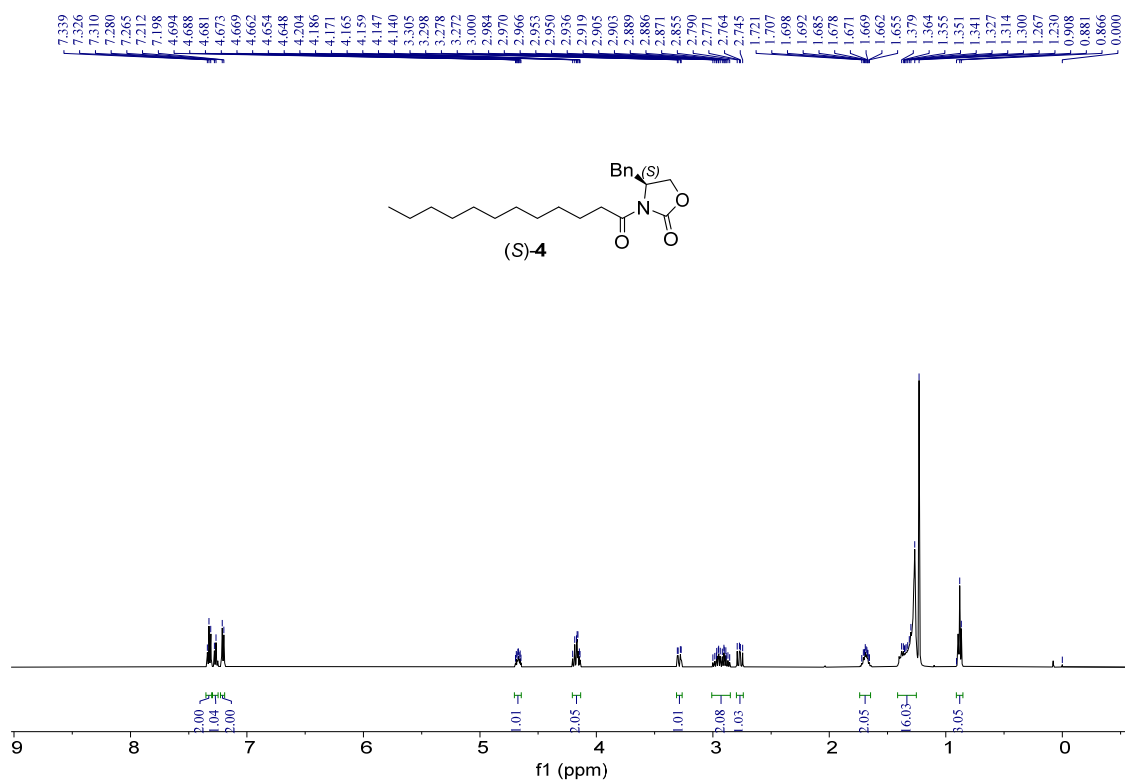


Figure S1. ^1H NMR Spectrum of (S)-4-benzyl-3-dodecanoyloxazolidin-2-one ((S)-4) (500 MHz, CDCl_3)

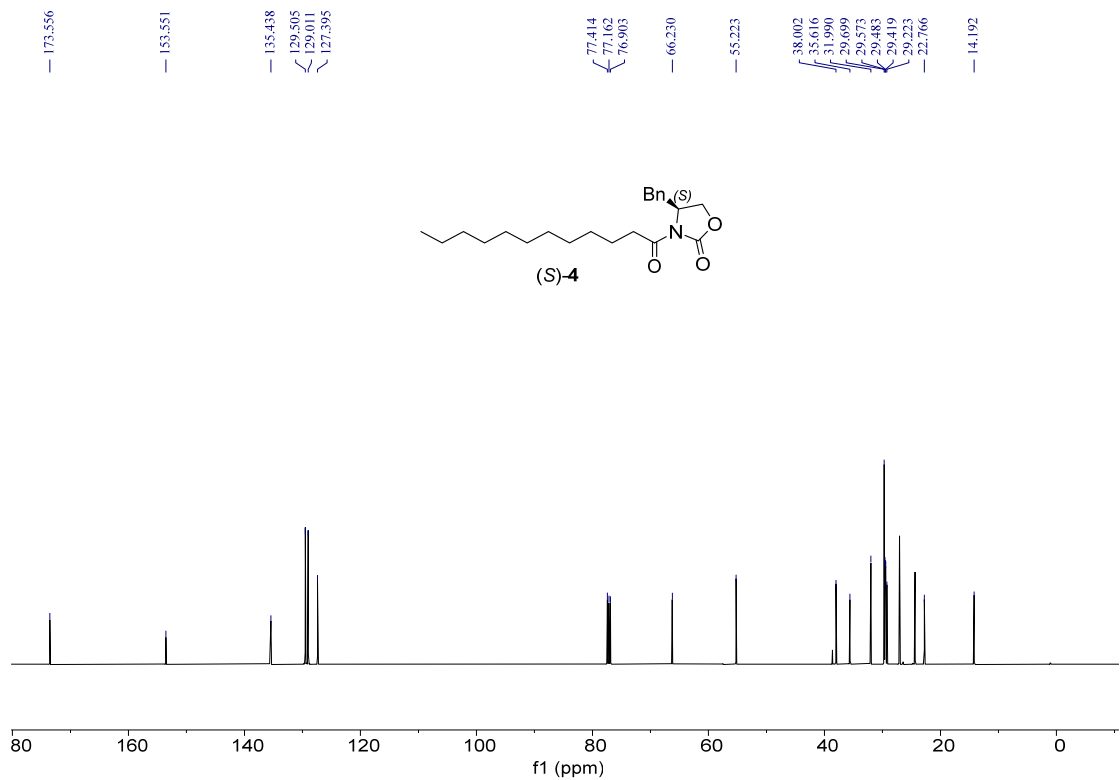
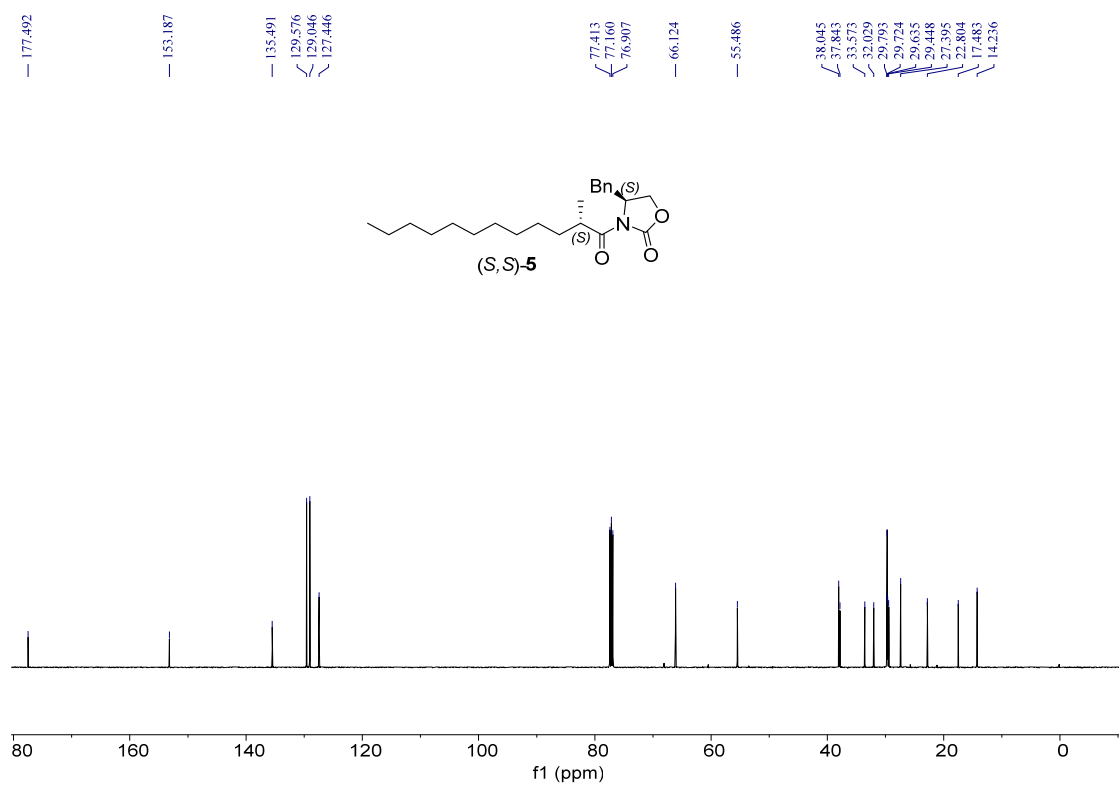
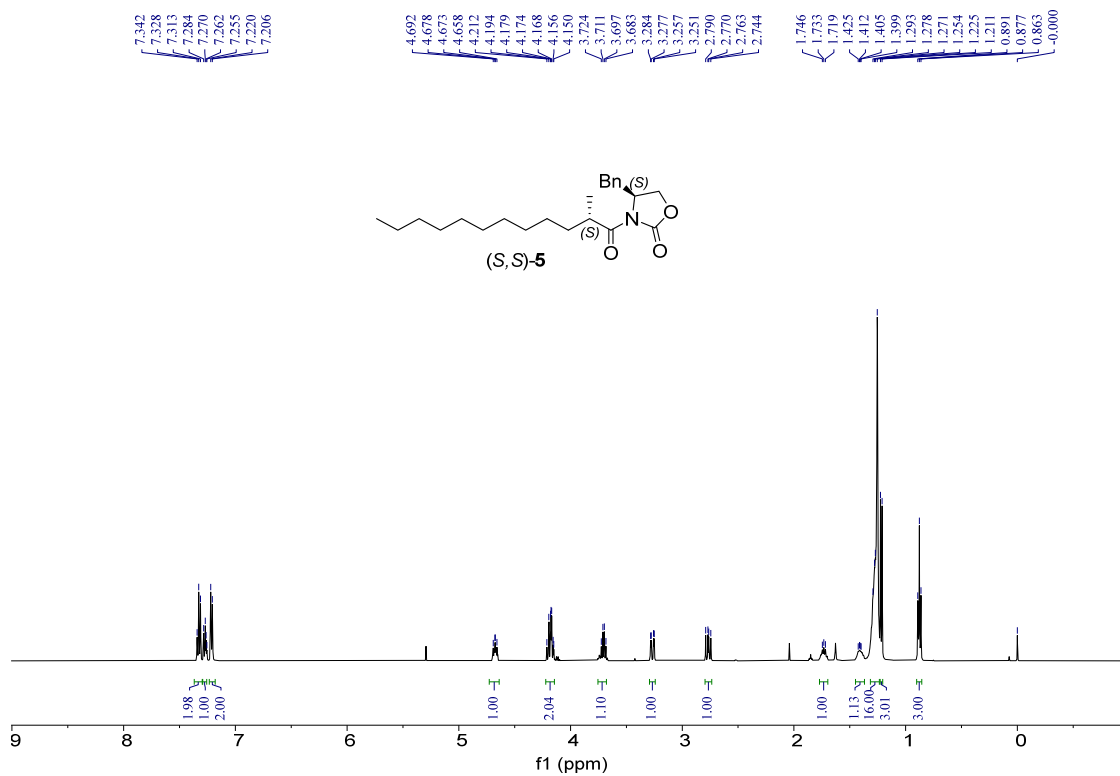


Figure S2. ^{13}C NMR Spectrum of (S)-4-benzyl-3-dodecanoyloxazolidin-2-one ((S)-4) (126 MHz, CDCl_3)



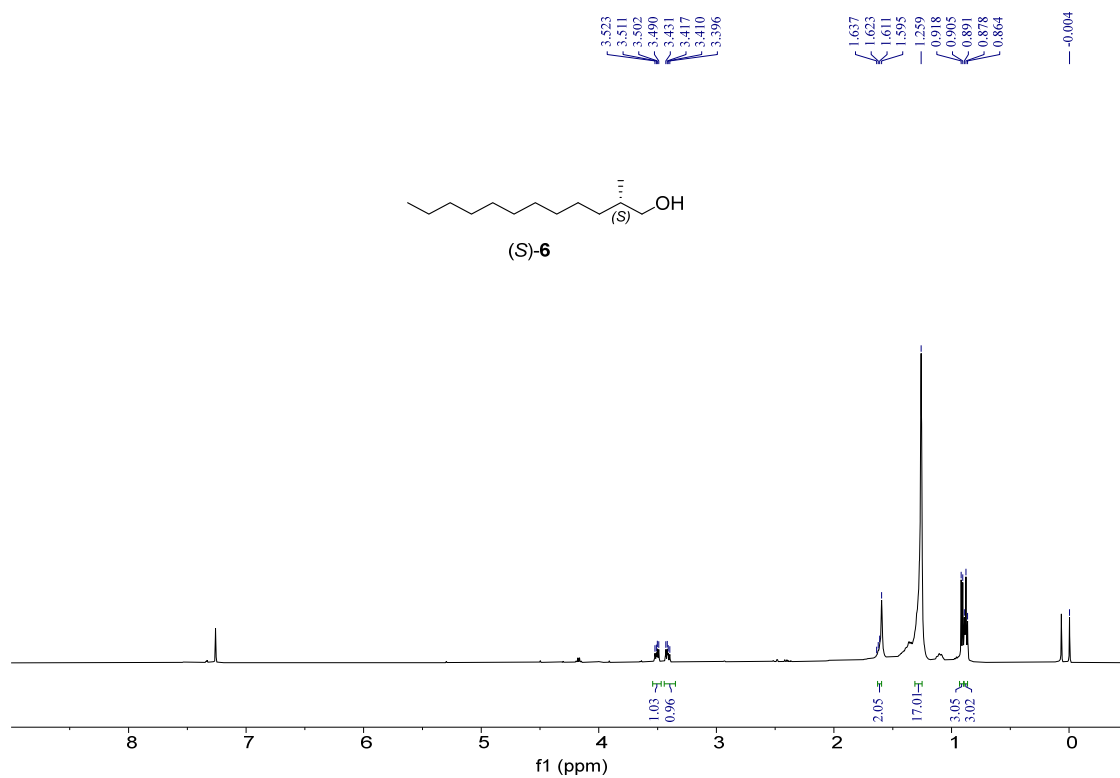


Figure S5. ¹H NMR Spectrum of (S)-2-methyldodecan-1-ol ((S)-6) (500 MHz, CDCl₃)

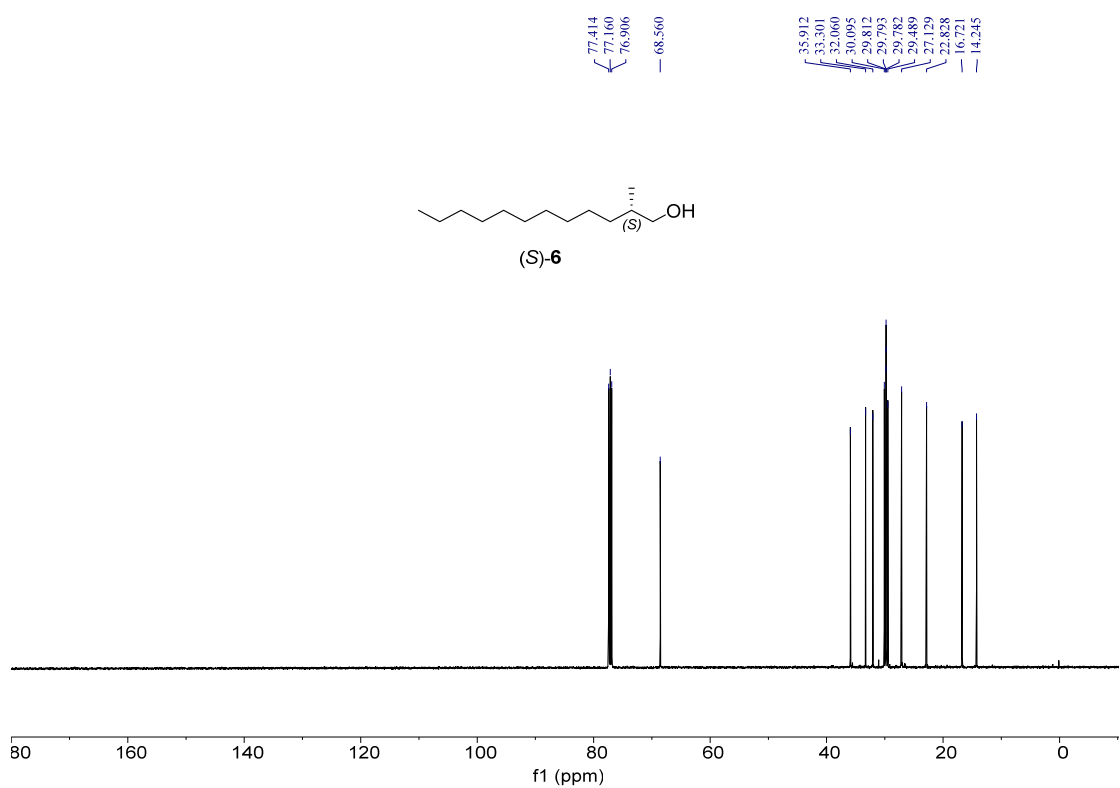


Figure S6. ¹³C NMR Spectrum of (S)-2-methyldodecan-1-ol ((S)-6) (126 MHz, CDCl₃)

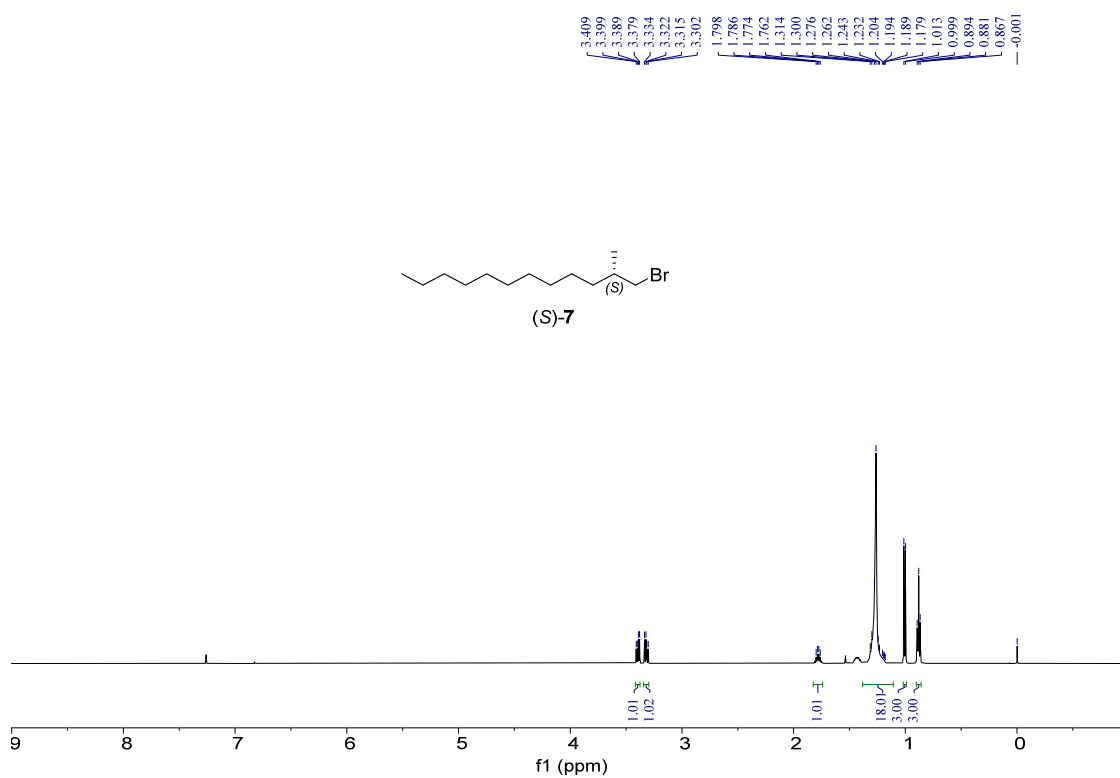


Figure S7. ¹H NMR Spectrum of (S)-1-bromo-2-methyldodecane ((S)-7) (500 MHz, CDCl₃)

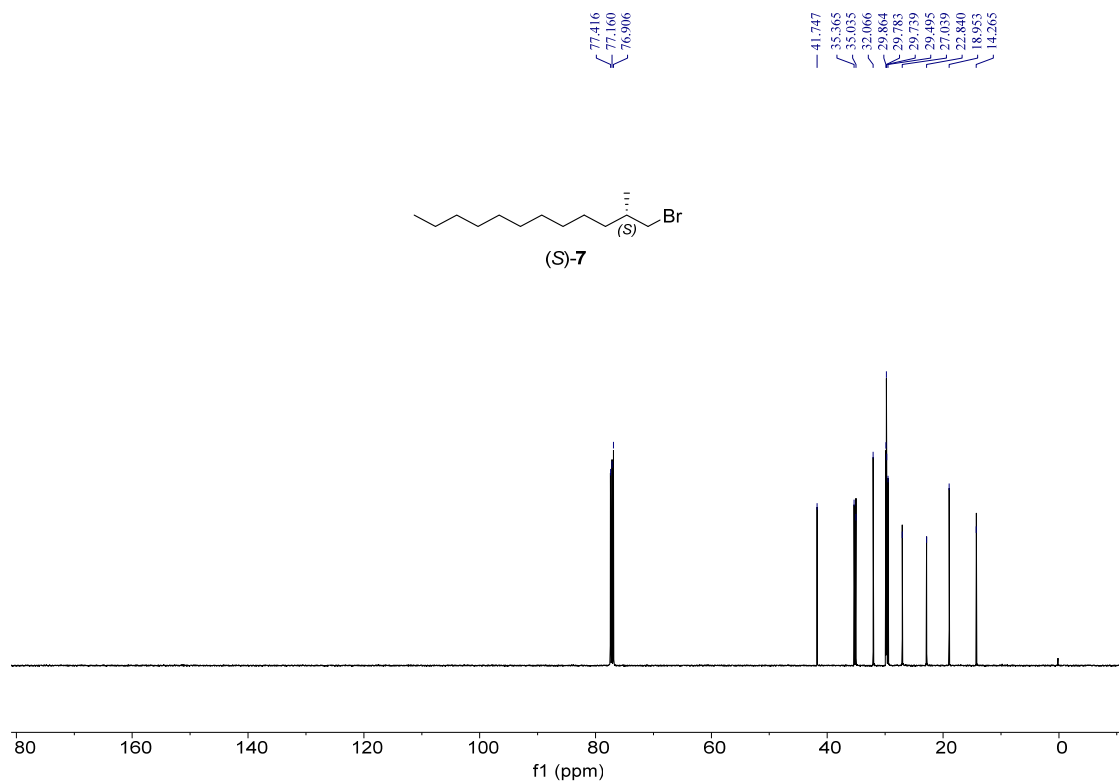


Figure S8. ¹³C NMR Spectrum of (S)-1-bromo-2-methyldodecane ((S)-7) (126 MHz, CDCl₃)

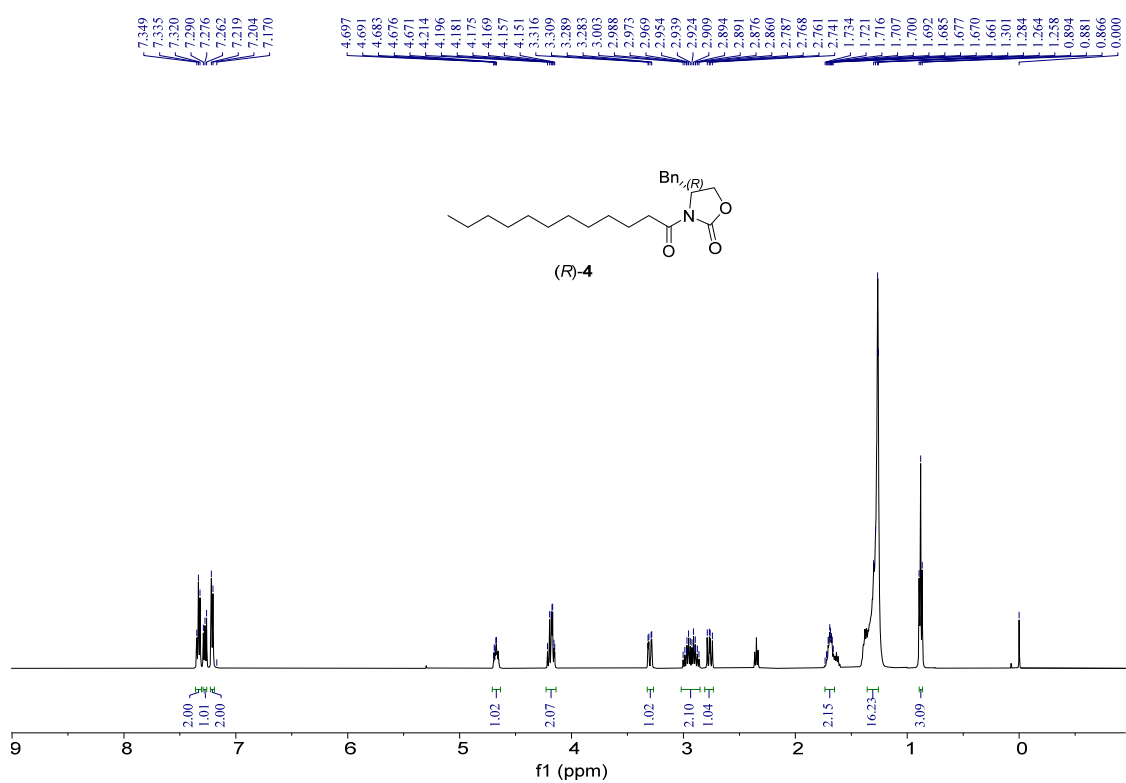


Figure S9. ¹H NMR Spectrum of (R)-4-benzyl-3-dodecanoyloxazolidin-2-one ((R)-4) (500 MHz, CDCl₃)

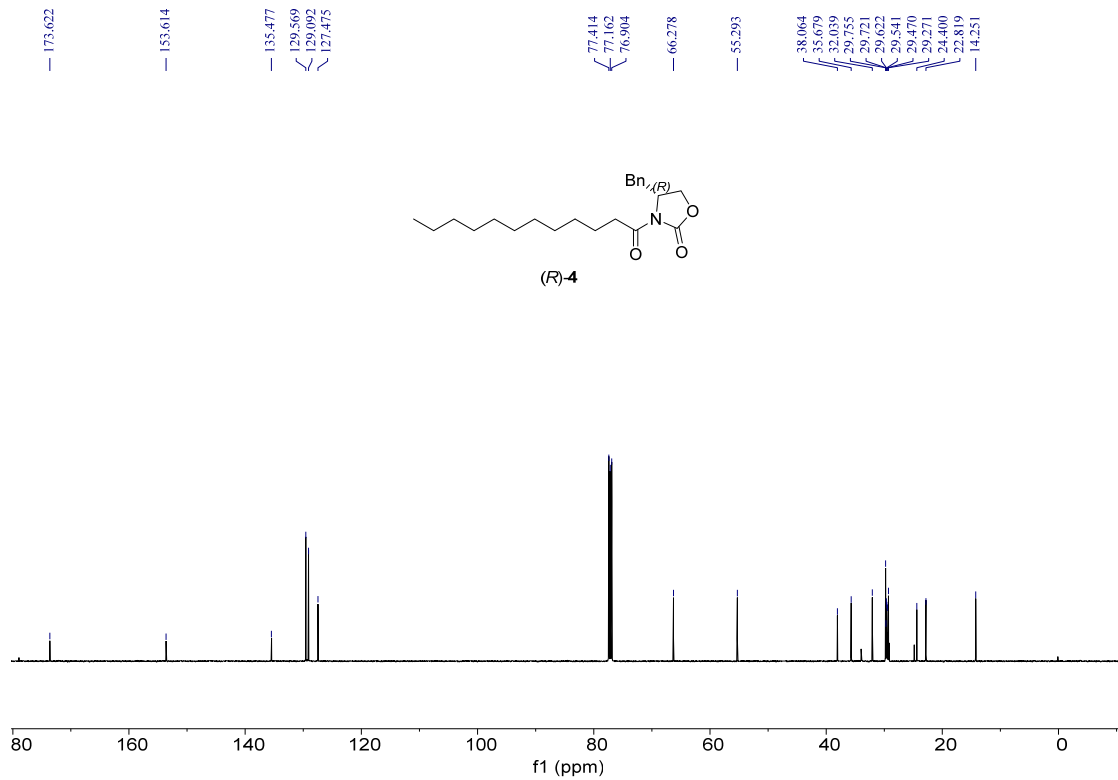


Figure S10. ¹³C NMR Spectrum of (R)-4-benzyl-3-dodecanoyloxazolidin-2-one ((R)-4) (126 MHz, CDCl₃)

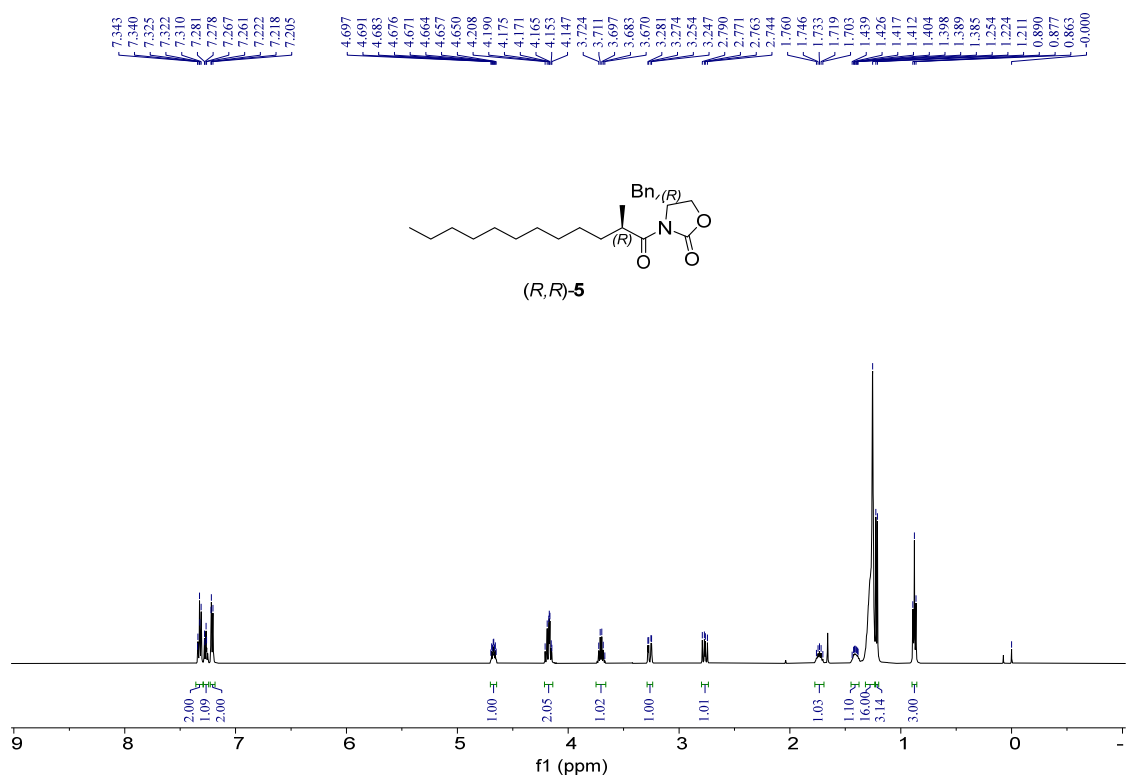


Figure S11. ¹H NMR Spectrum of (R)-4-benzyl-3-((R)-2-methyldodecanoyl)oxazolidin-2-one((R,R)-5) (500 MHz, CDCl₃)

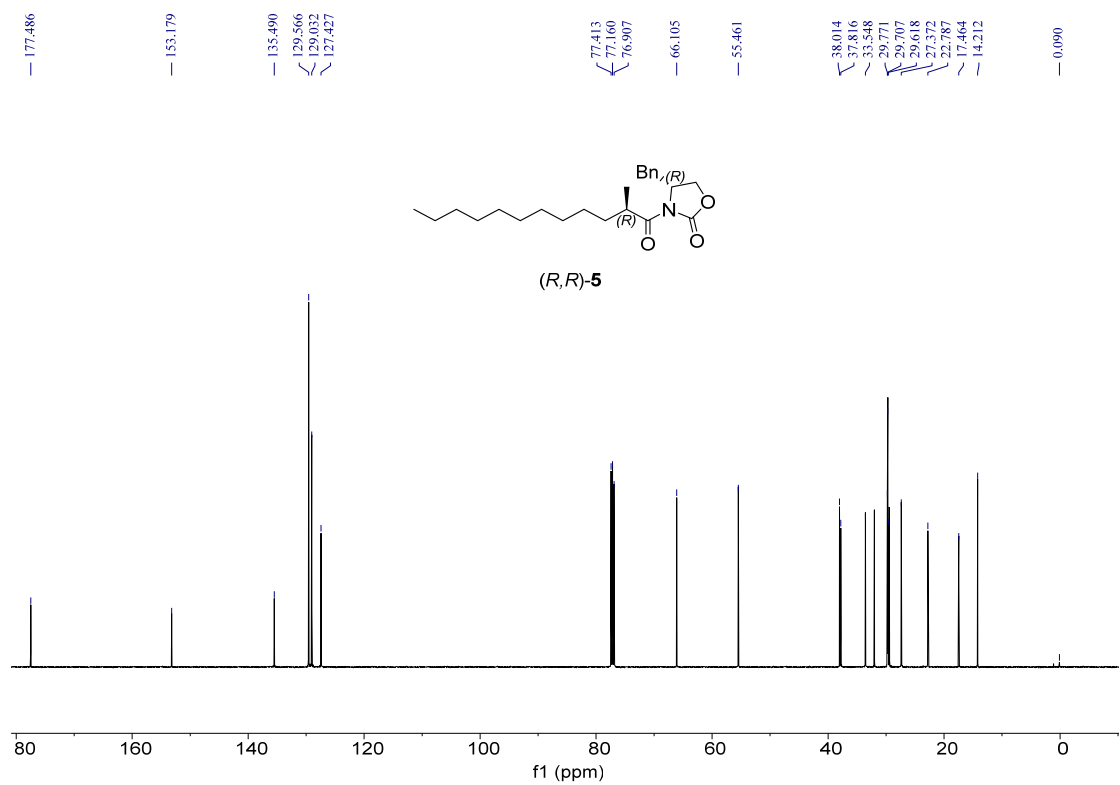


Figure S12. ¹³C NMR Spectrum of (R)-4-benzyl-3-((R)-2-methyldodecanoyl)oxazolidin-2-one((R,R)-5) (126 MHz, CDCl₃)

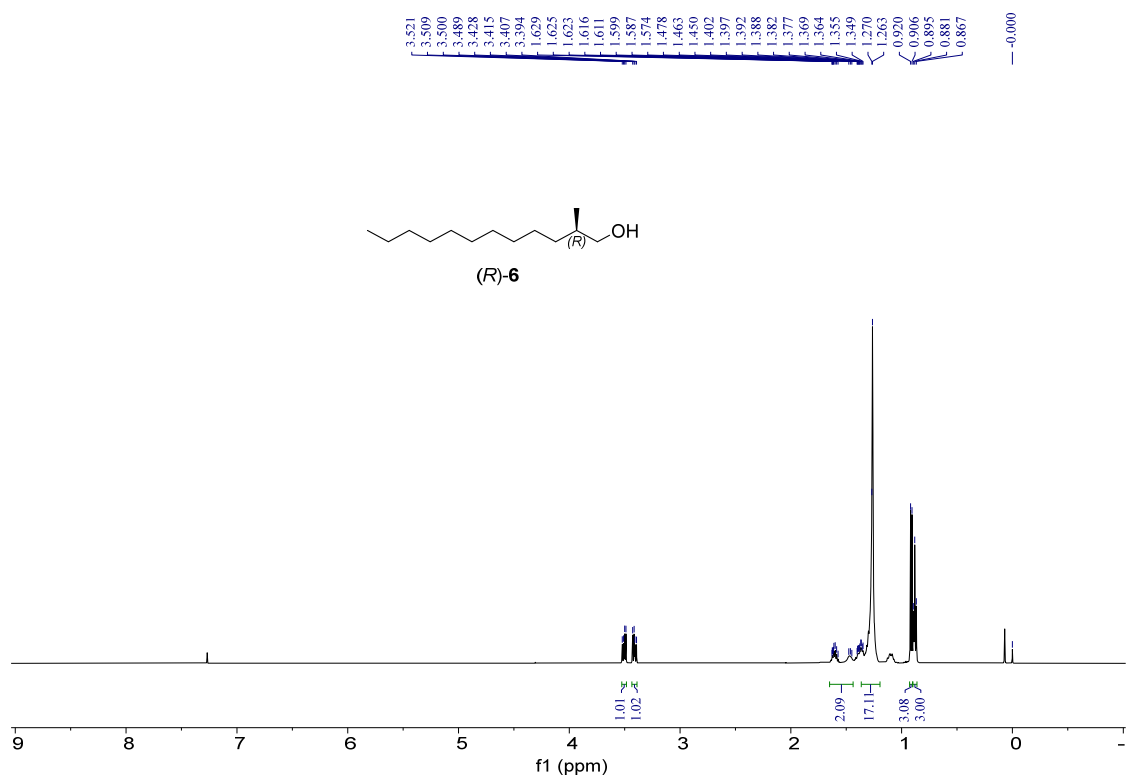


Figure S13. ¹H NMR Spectrum of (R)-2-methyldodecan-1-ol ((R)-6) (500 MHz, CDCl₃)

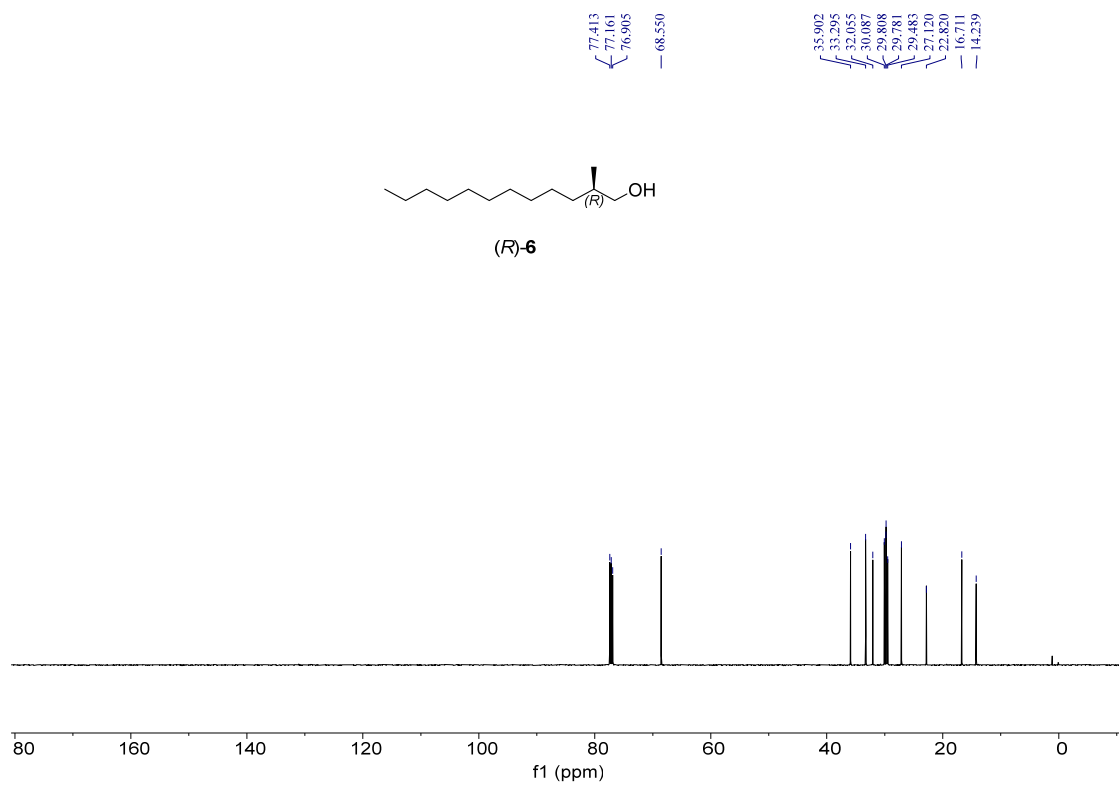


Figure S14. ¹³C NMR Spectrum of (R)-2-methyldodecan-1-ol ((R)-6) (126 MHz, CDCl₃)

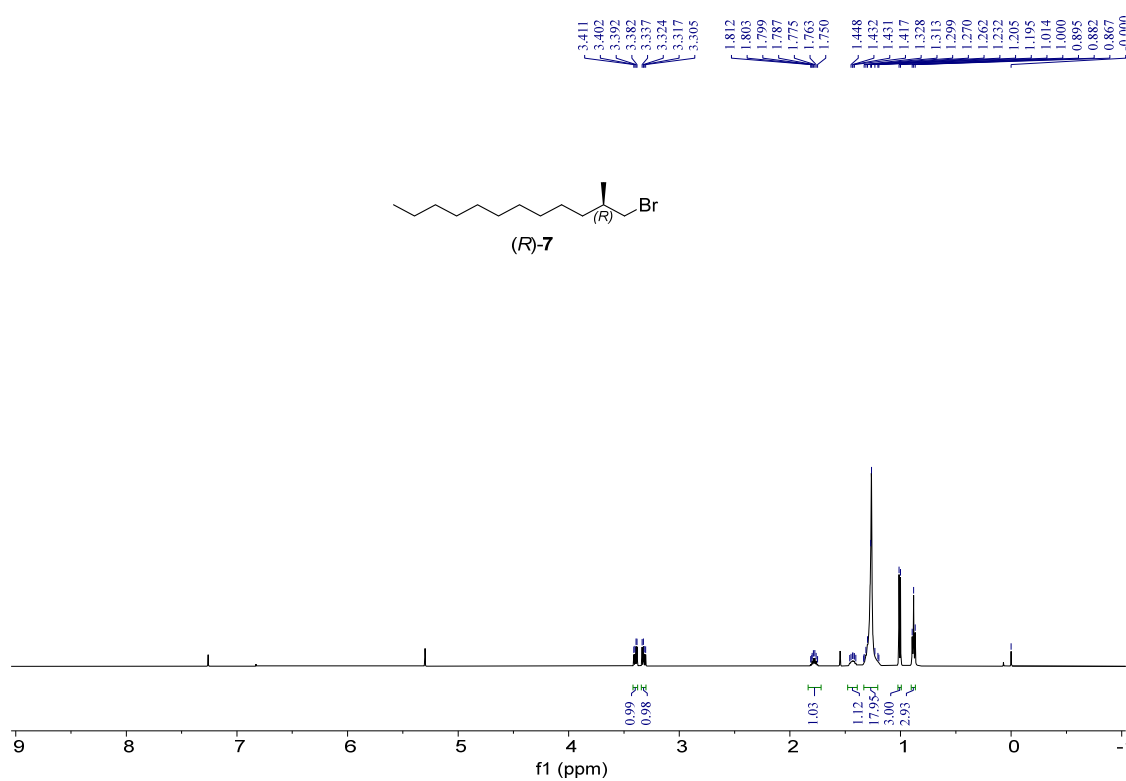


Figure S15. ¹H NMR Spectrum of (R)-1-bromo-2-methyldodecane ((R)-7) (500 MHz, CDCl₃)

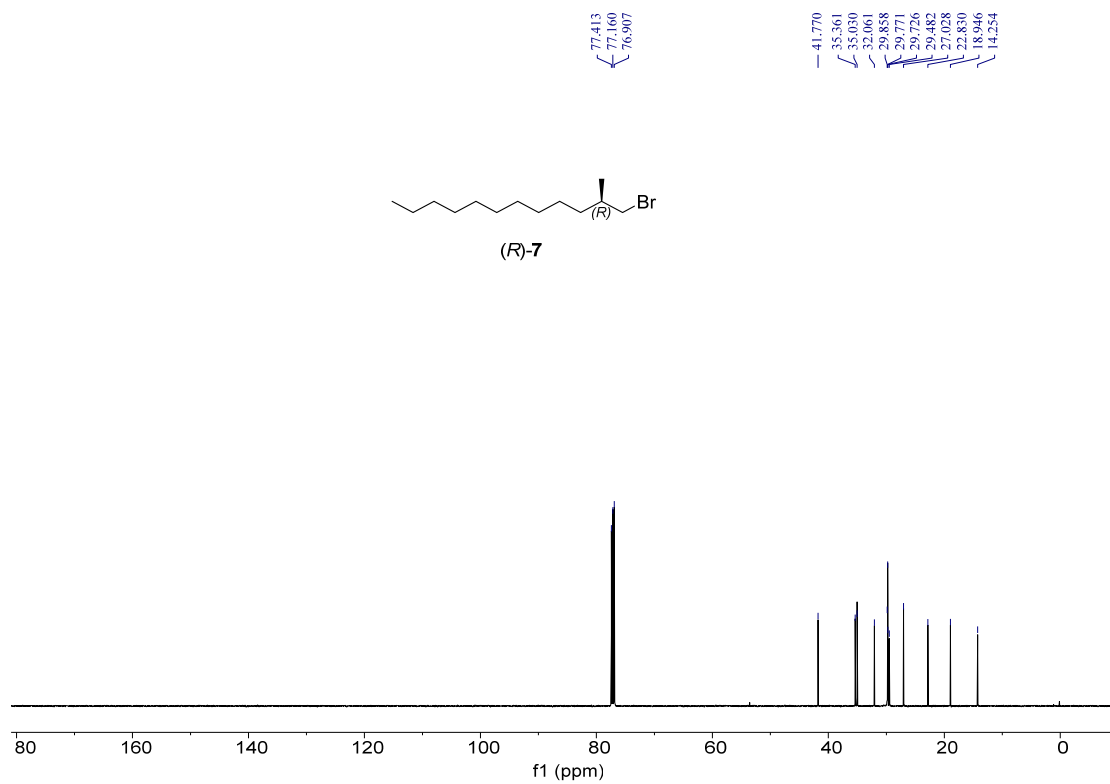


Figure S16. ¹³C NMR Spectrum of (R)-1-bromo-2-methyldodecane ((R)-7) (126 MHz, CDCl₃)

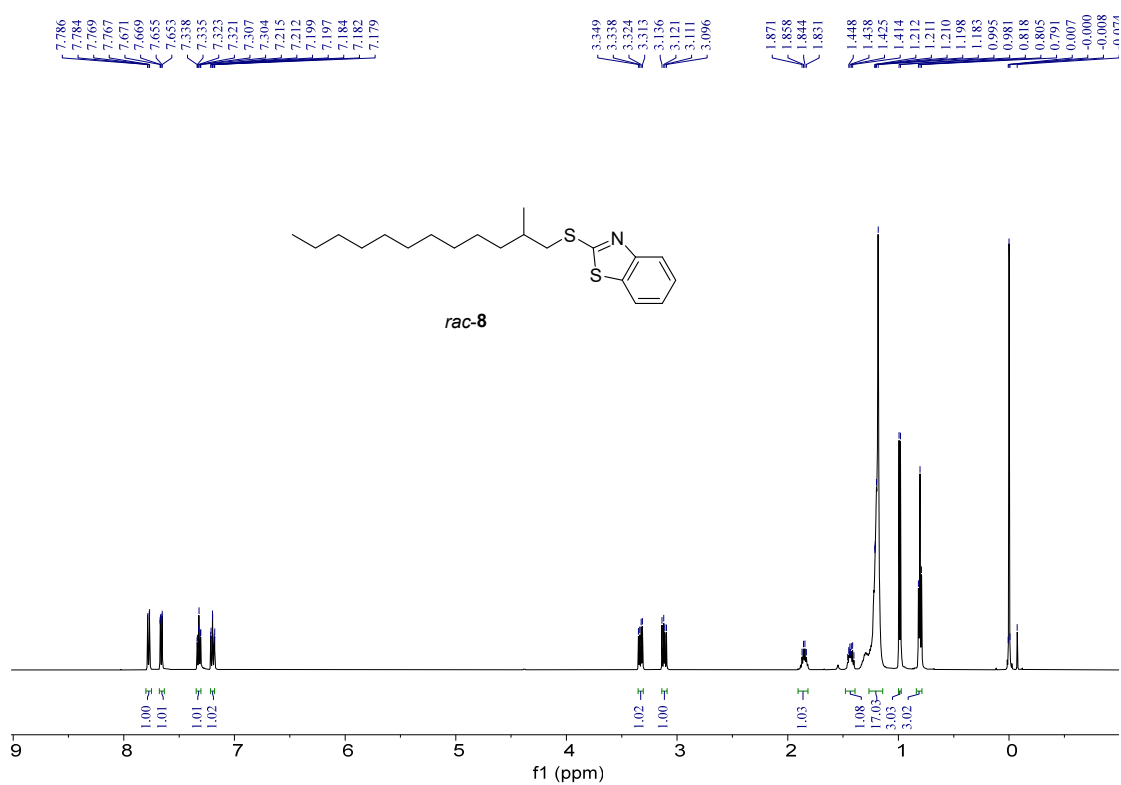


Figure S17. ¹H NMR Spectrum of 2-((2-methyldodecyl)thio)benzo[d]thiazole (*rac-8*) (500 MHz, CDCl₃)

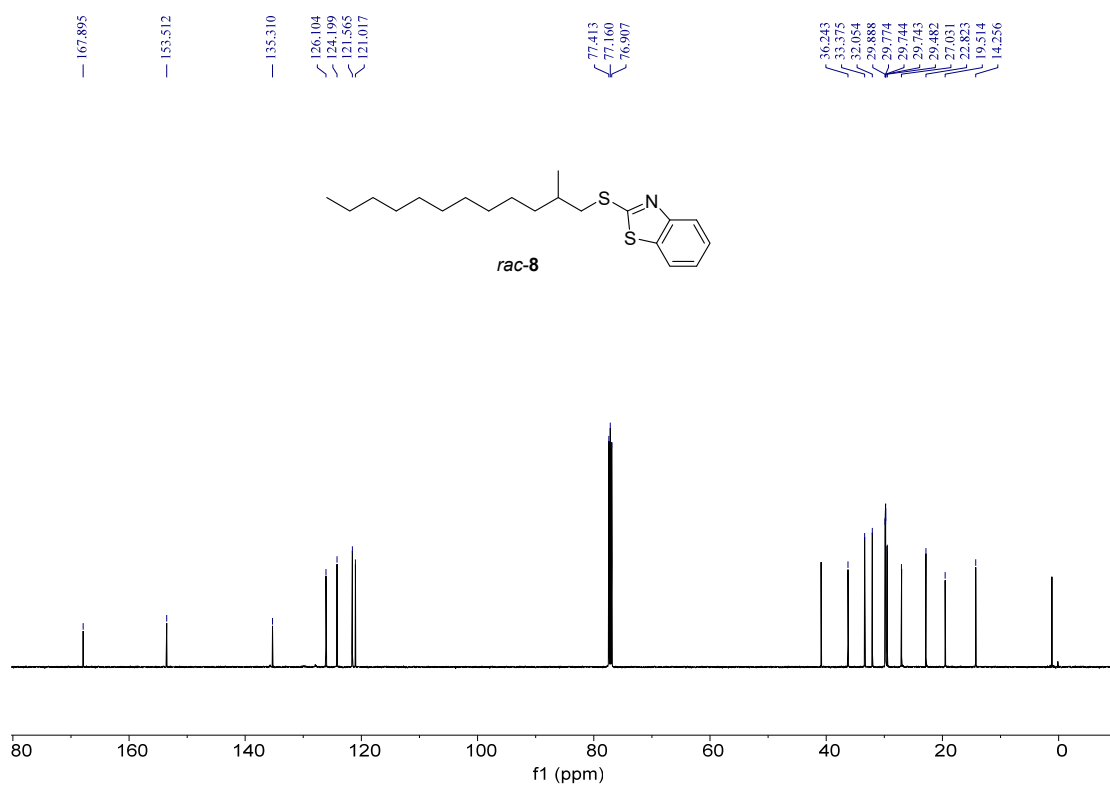


Figure S18. ¹³C NMR Spectrum of 2-((2-methyldodecyl)thio)benzo[d]thiazole (*rac-8*) (126 MHz, CDCl₃)

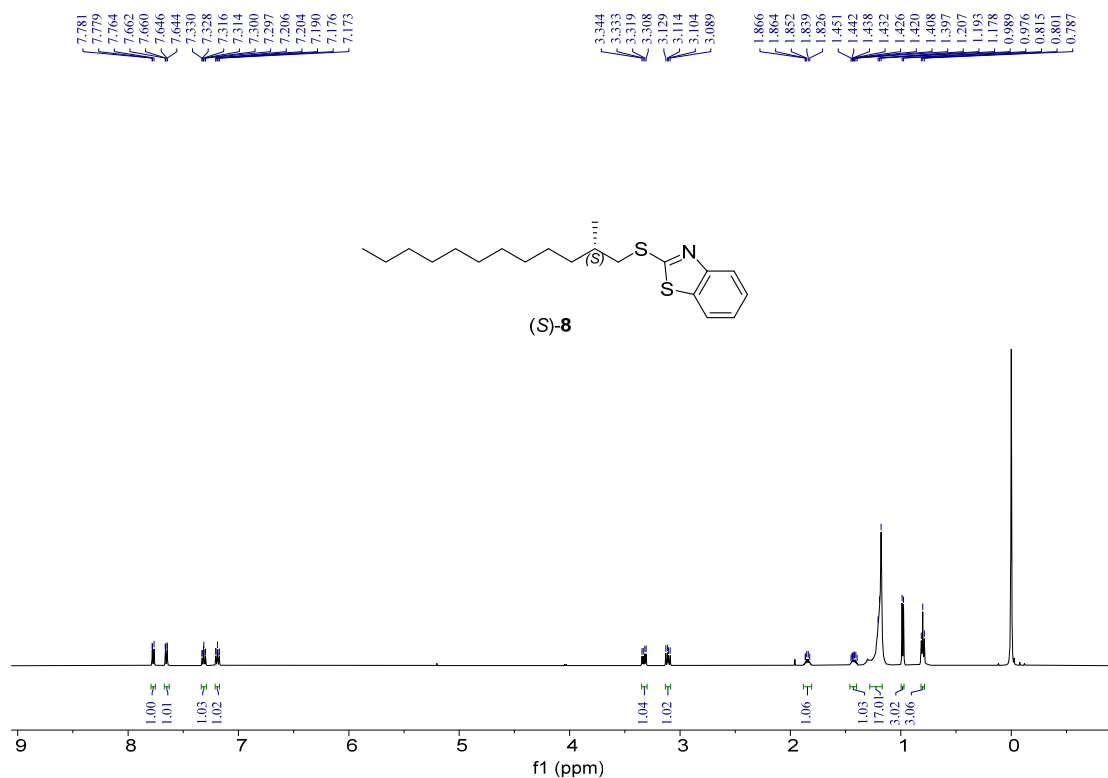


Figure S19. ¹H NMR Spectrum of (S)-2-((2-methyldodecyl)thio)benzo[d]thiazole ((S)-8) (500 MHz, CDCl₃)

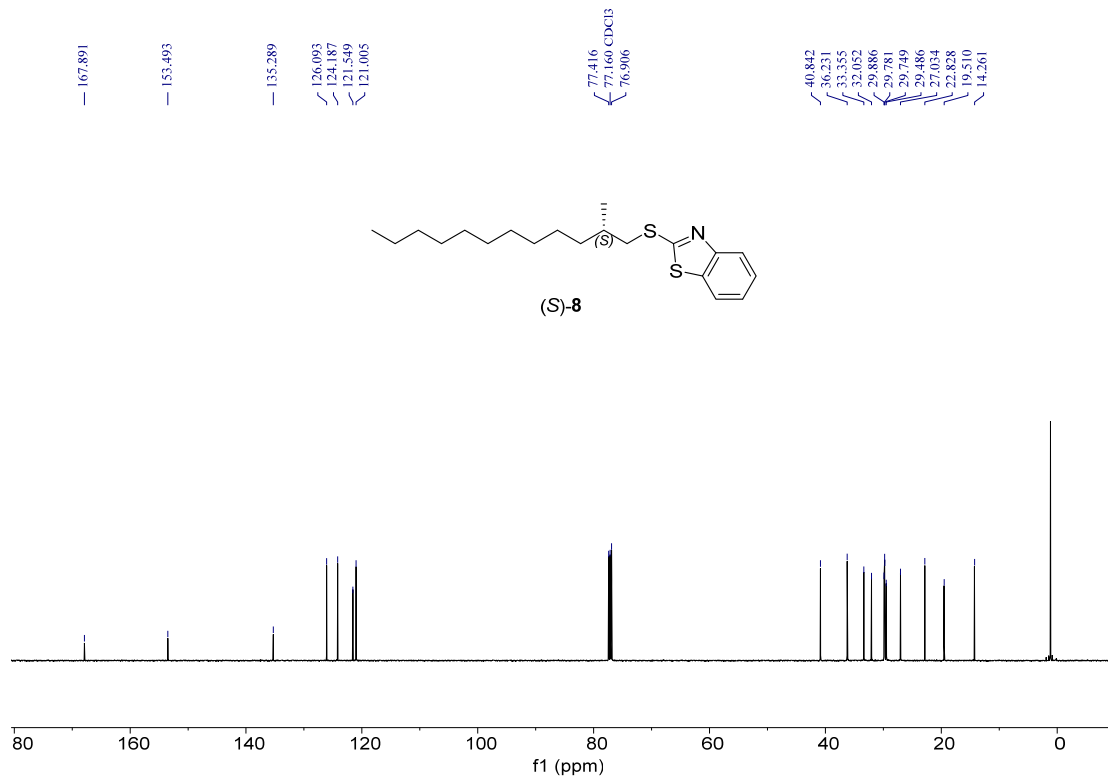


Figure S20. ¹³C NMR Spectrum of (S)-2-((2-methyldodecyl)thio)benzo[d]thiazole ((S)-8) (126 MHz, CDCl₃)

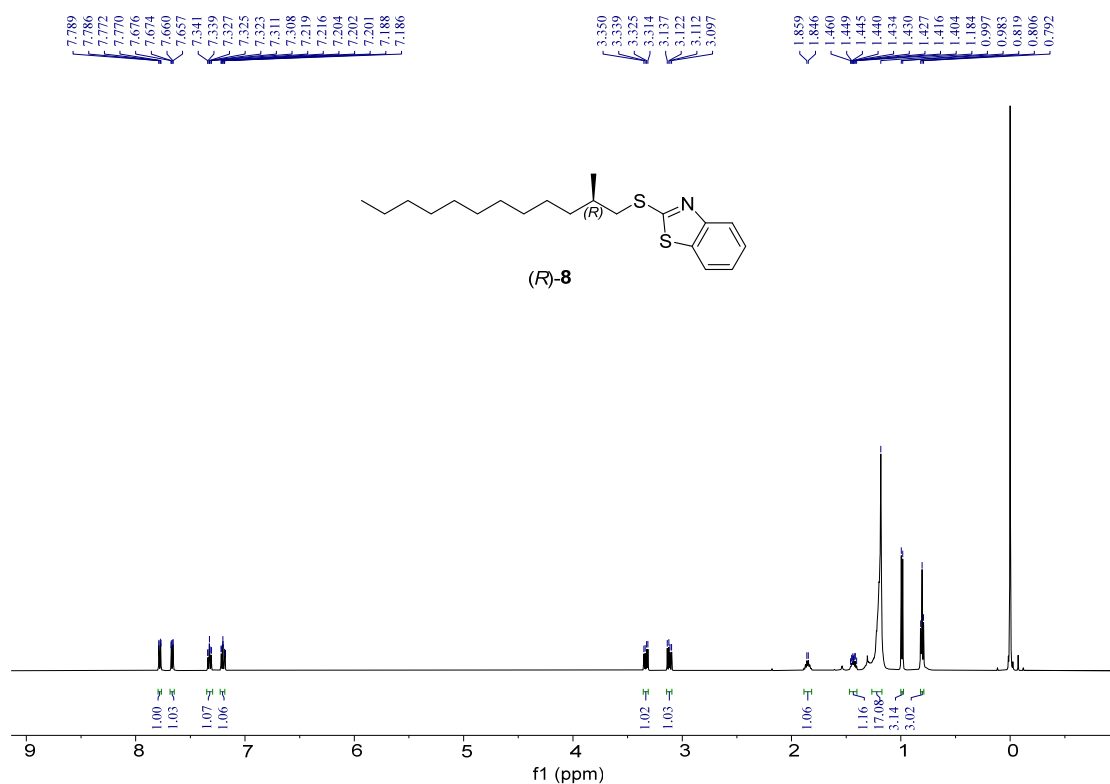


Figure S21. ¹H NMR Spectrum of (R)-2-((2-methyldodecyl)thio)benzo[d]thiazole ((R)-8) (500 MHz, CDCl₃)

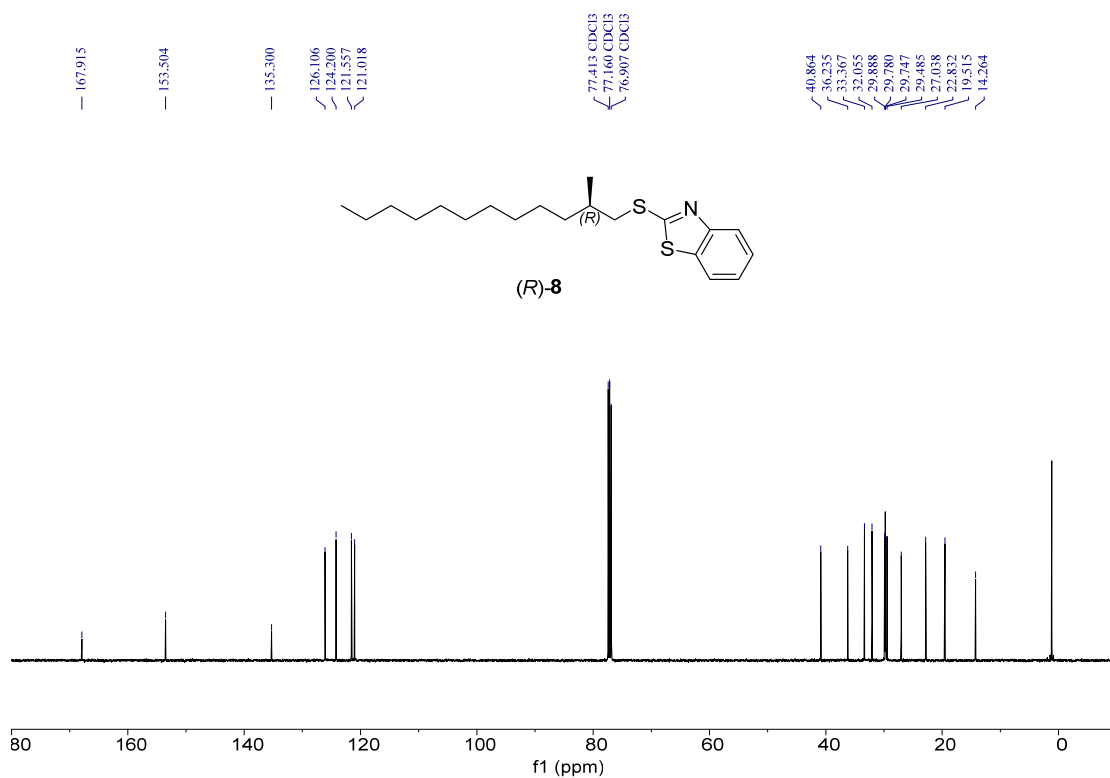


Figure S22. ¹³C NMR Spectrum of (R)-2-((2-methyldodecyl)thio)benzo[d]thiazole ((R)-8) (126 MHz, CDCl₃)

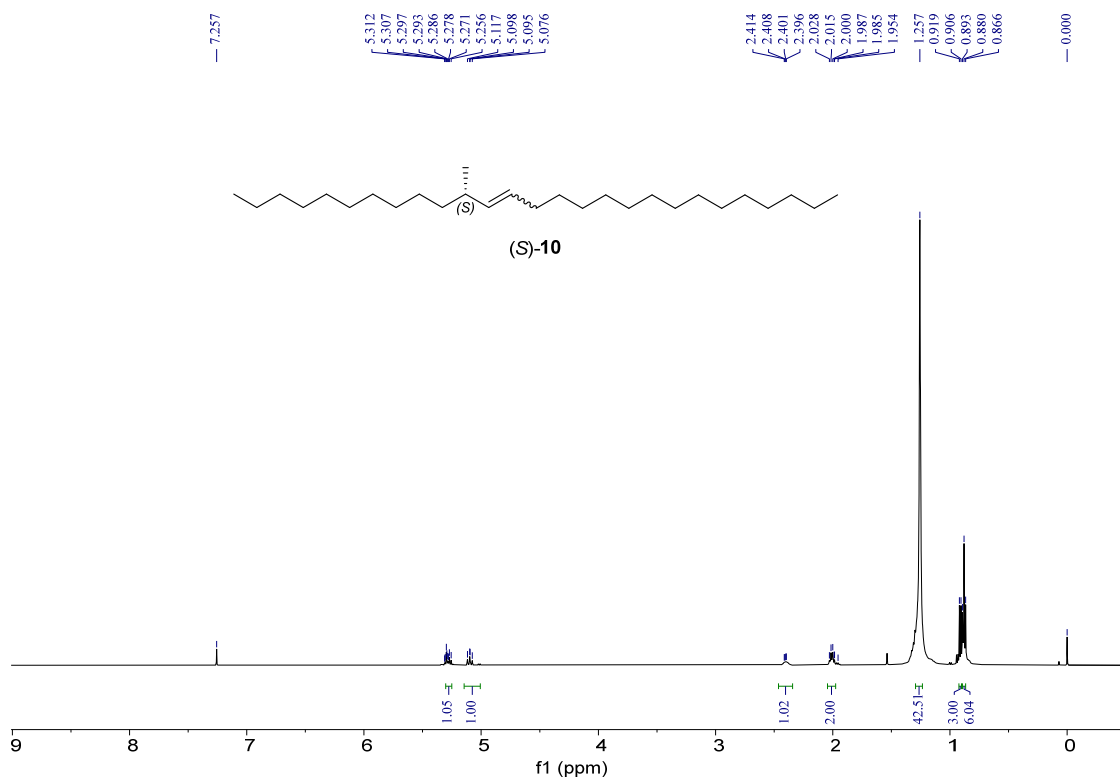


Figure S23. ^1H NMR Spectrum of (S)-11-methylheptacos-9-ene ((S)-10) (500 MHz, CDCl_3)

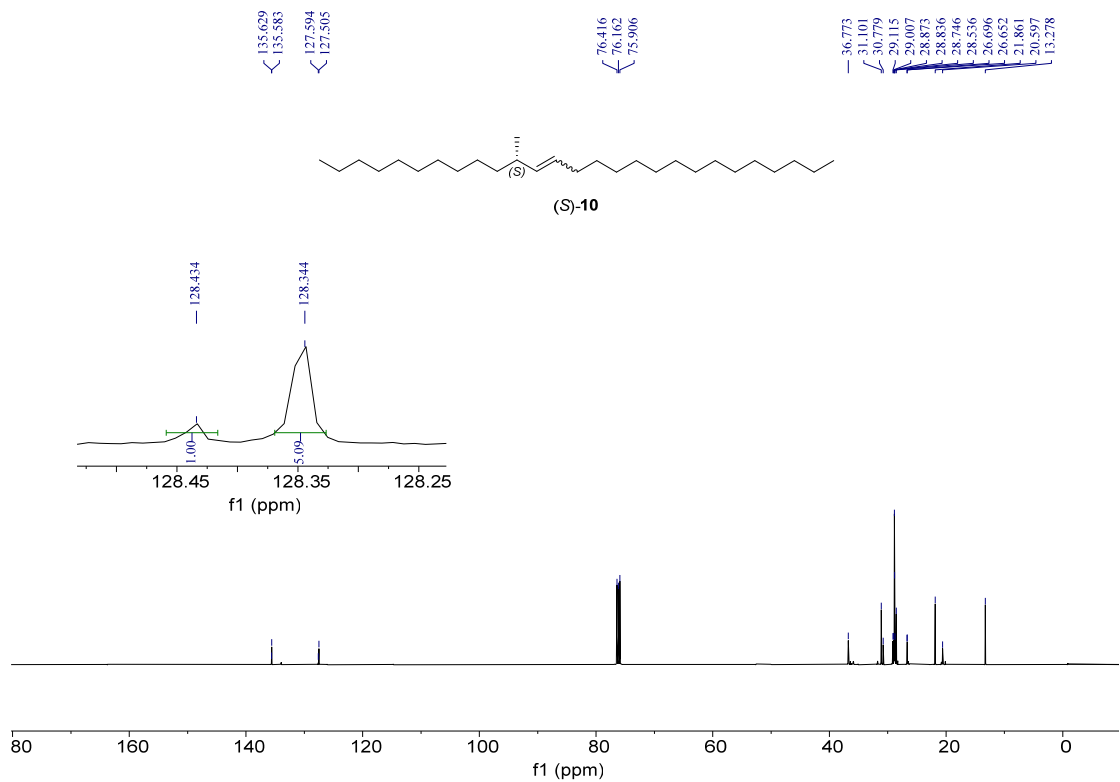


Figure S24. ^{13}C NMR Spectrum of (S)-11-methylheptacos-9-ene ((S)-10) (126 MHz, CDCl_3)

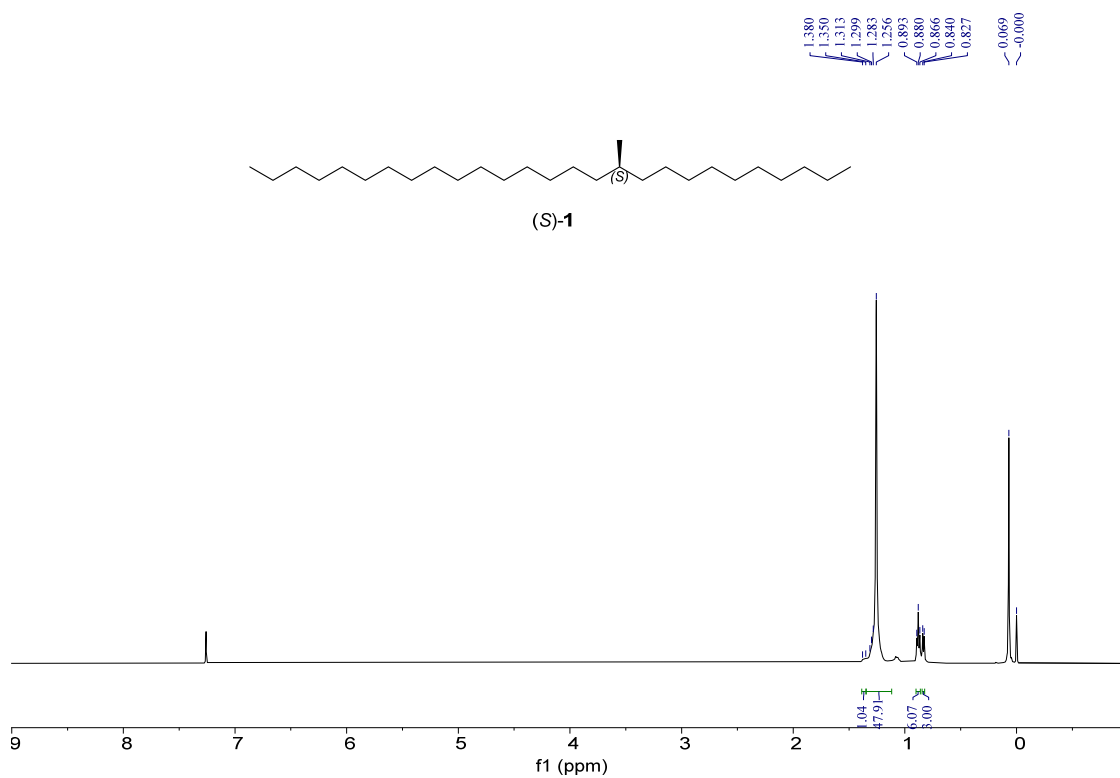


Figure S25. ¹H NMR Spectrum of (S)-11-methylheptacosane ((S)-1) (500 MHz, CDCl₃)

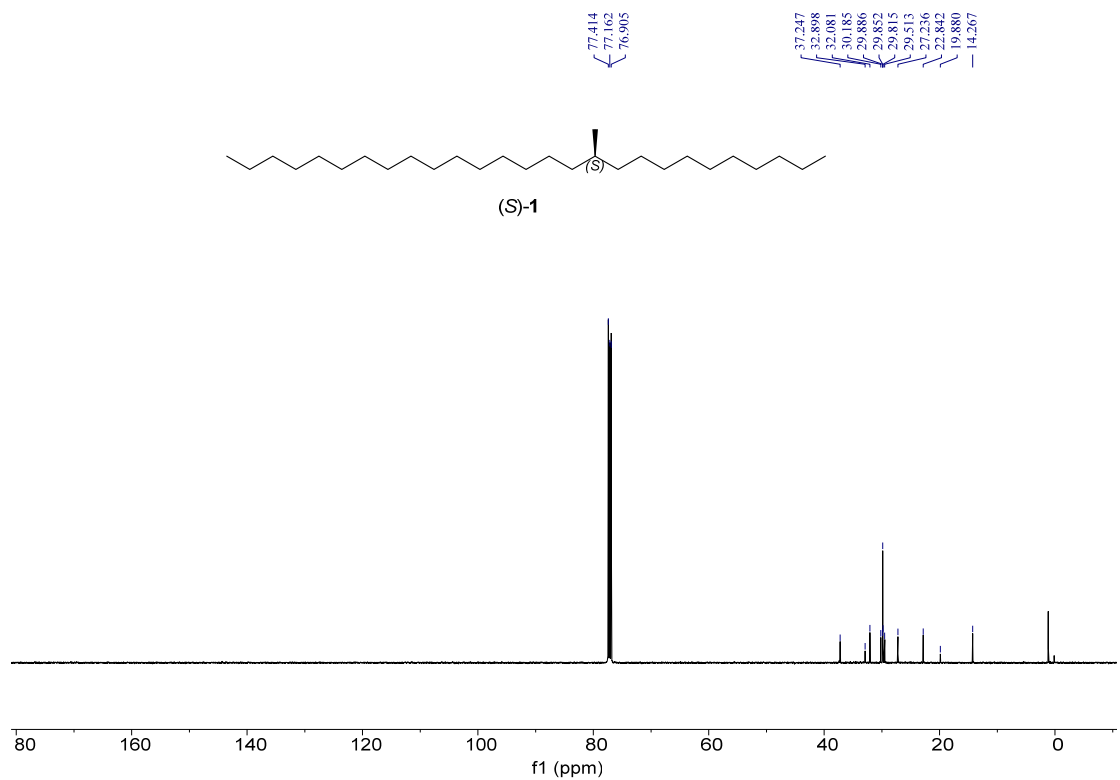


Figure S26. ¹³C NMR Spectrum of (S)-11-methylheptacosane ((S)-1) (126 MHz, CDCl₃)

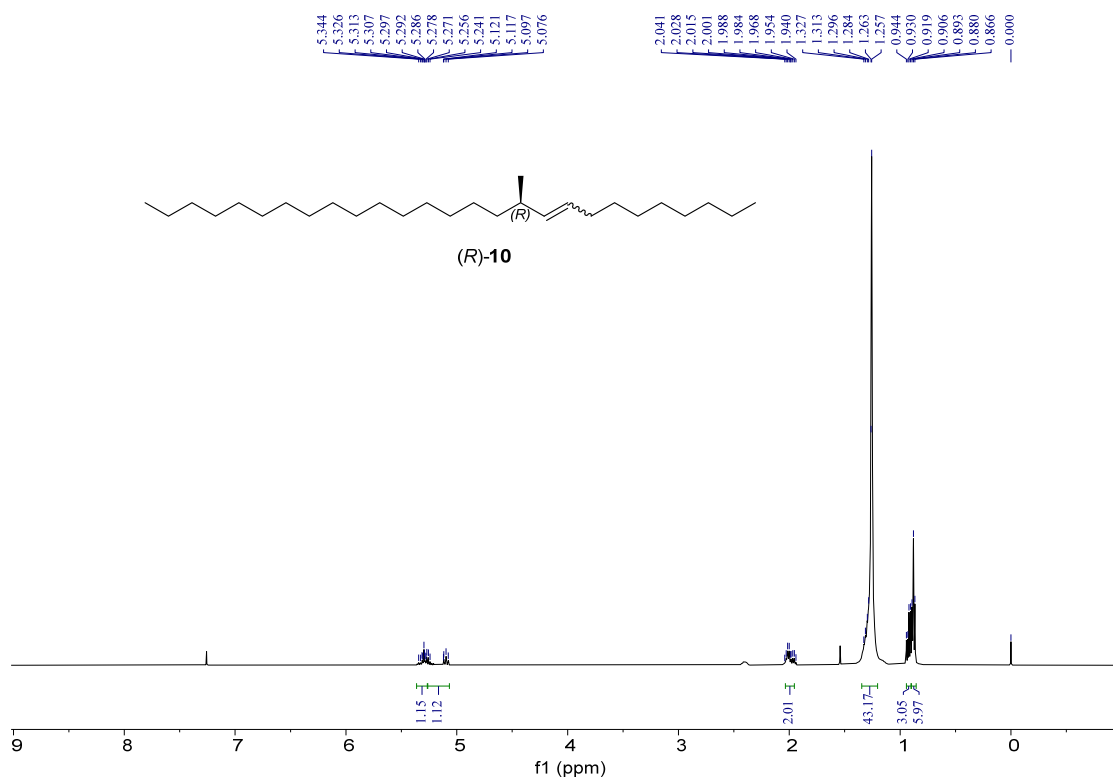


Figure S27. ¹H NMR Spectrum of (R)-11-methylheptacos-9-ene ((R)-10) (500 MHz, CDCl₃)

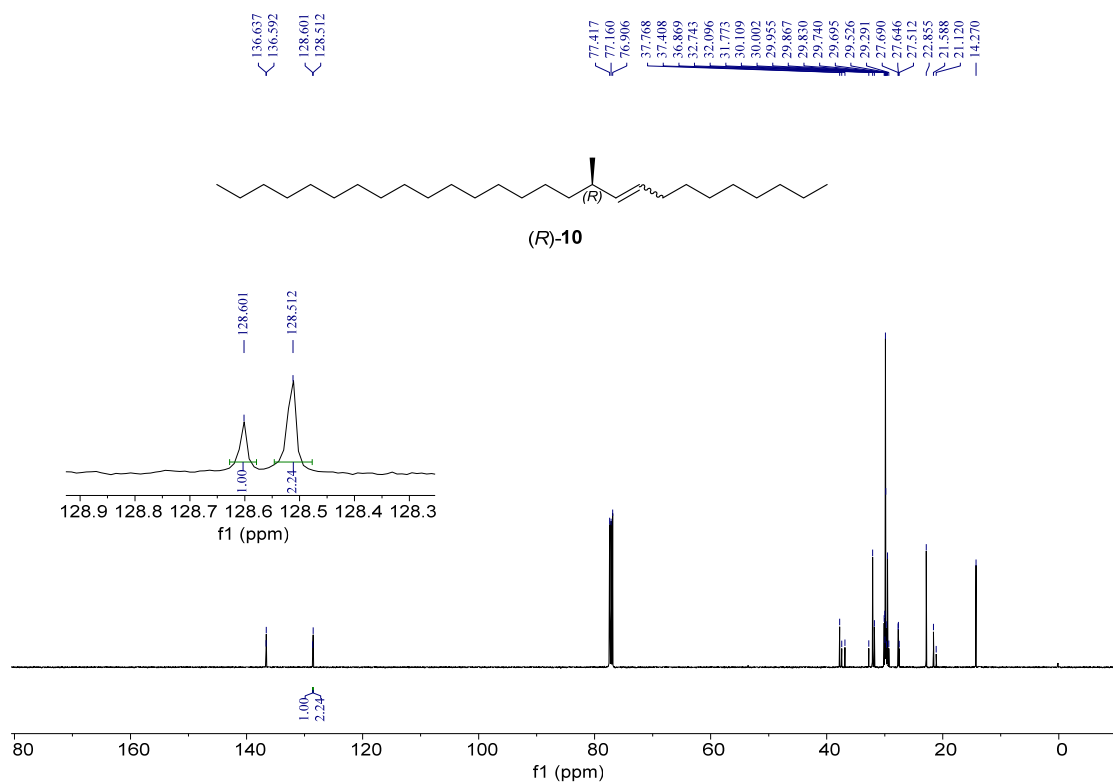


Figure S28. ¹³C NMR Spectrum of (R)-11-methylheptacos-9-ene ((R)-10) (126 MHz, CDCl₃)

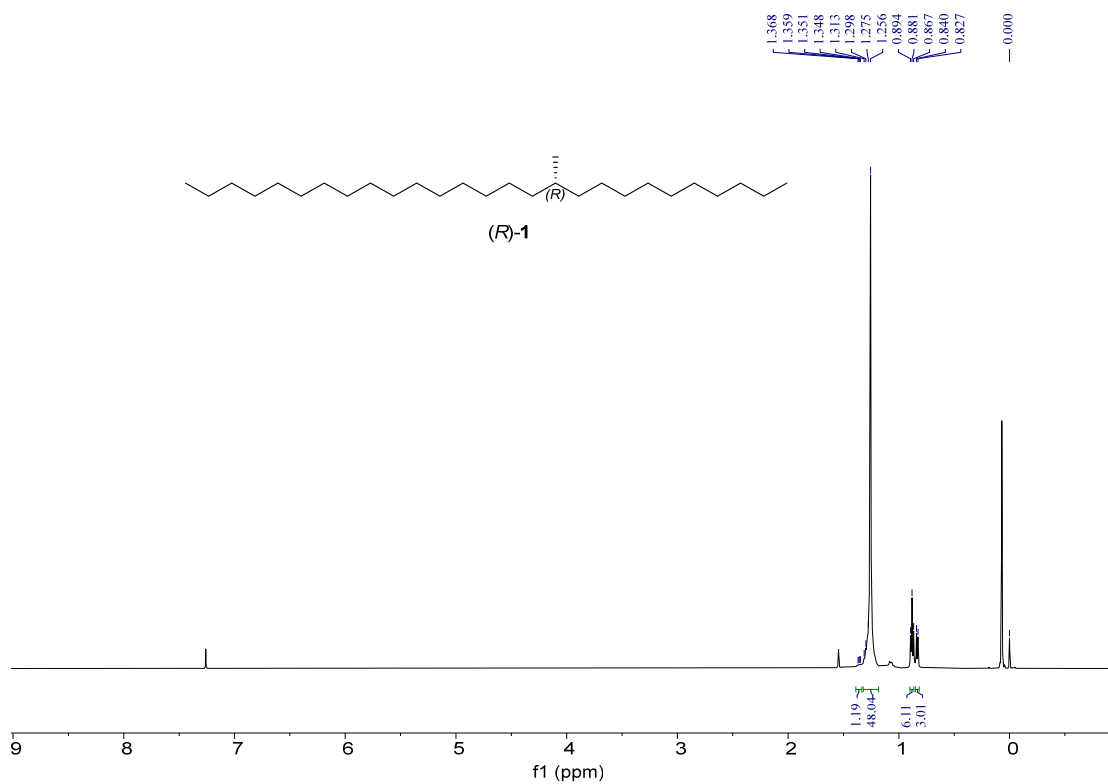


Figure S29. ¹H NMR Spectrum of (R)-11-methylheptacosane ((R)-1) (500 MHz, CDCl₃)

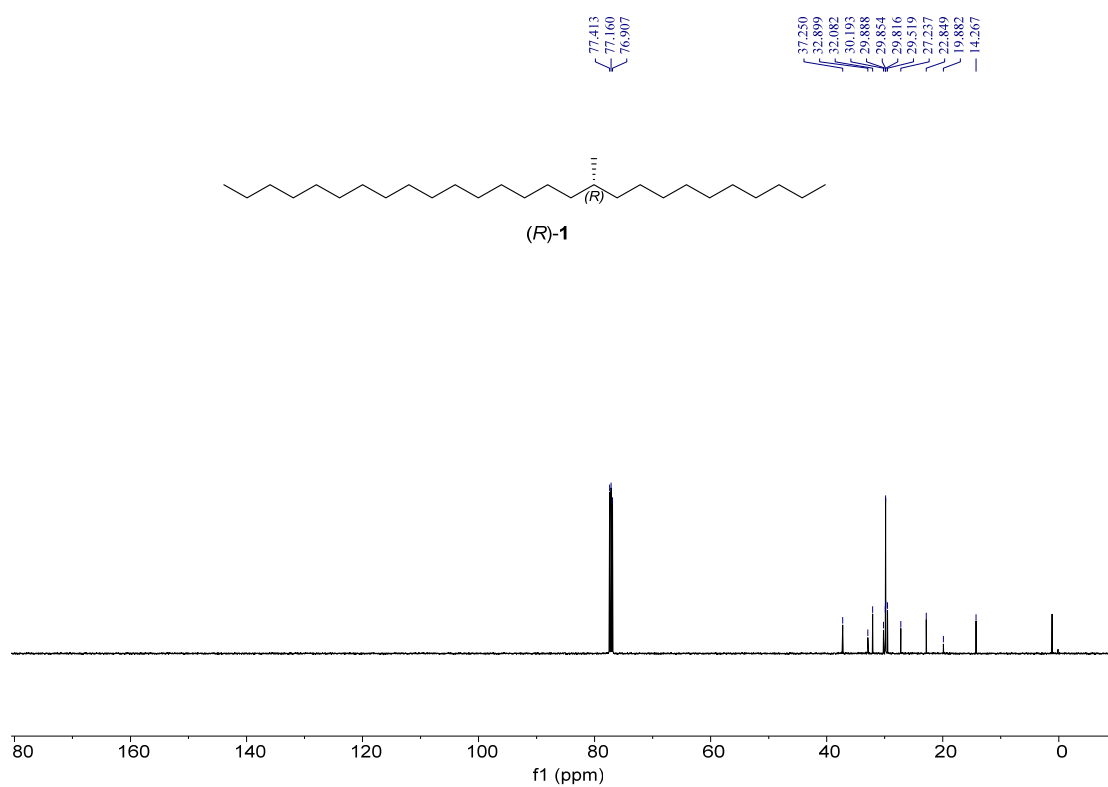
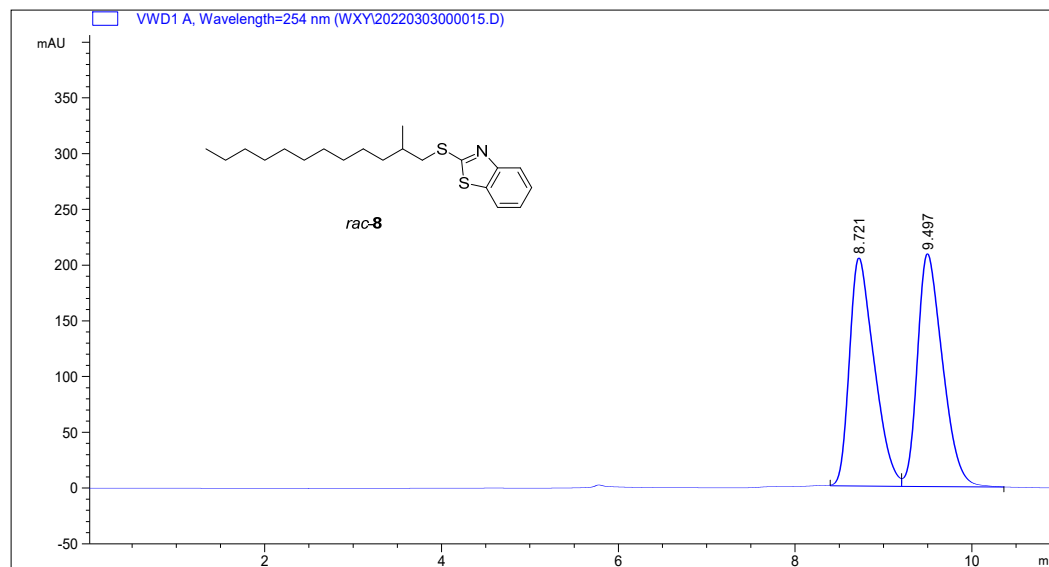


Figure S30. ¹³C NMR Spectrum of (R)-11-methylheptacosane ((R)-1) (126 MHz, CDCl₃)

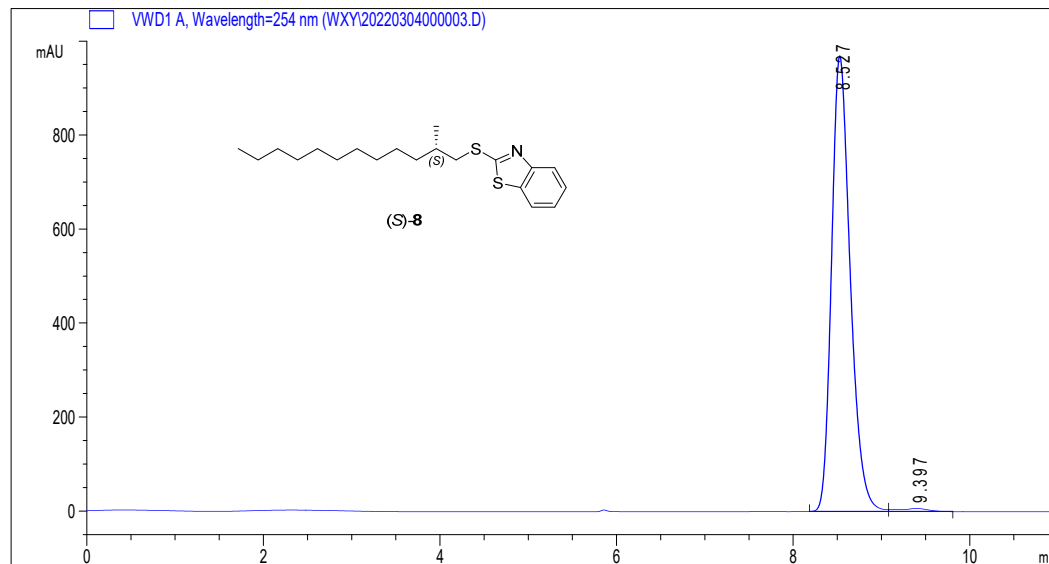
2. HPLC Chromatography of the Compounds

Figure S31. HPLC Chromatography of racemic 2-((2-methyldodecyl)thio)benzo[d]thiazole (*rac*-8) (Daicel Chiralcel OD-H column; *n*-hexane/*i*-propanol = 99:1, 0.7 mL/min, 254 nm)



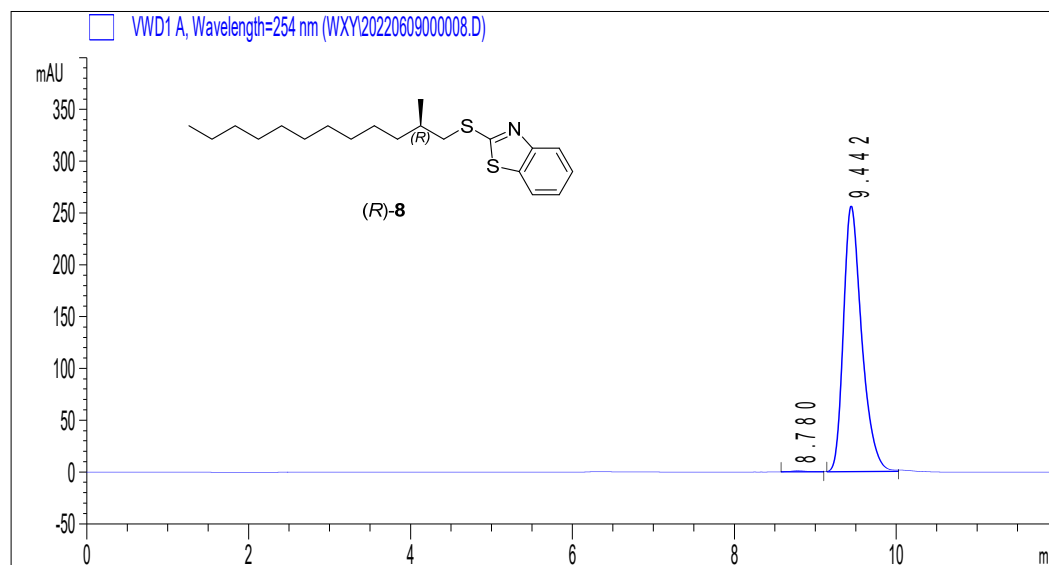
Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	8.721	BV	0.2900	3992.59229	204.86427	49.1413
2	9.497	VB	0.3005	4132.12256	209.07364	50.8587
Totals:				8124.71484	413.93791	

Figure S32. HPLC Chromatography of (*S*)-2-((2-methyldodecyl)thio)benzo[d]thiazole ((*S*)-8) (Daicel Chiralcel OD-H column; *n*-hexane/*i*-propanol = 99:1, 0.7 mL/min, 254 nm)



Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	8.527	BV	0.2240	1.42628e4	969.64209	98.8850
2	9.397	VB	0.3263	160.82082	6.88656	1.1150
Totals:				1.44236e4	976.52865	

Figure S33. HPLC Chromatography of (*R*)-2-((2-methyldodecyl)thio)benzo[d]thiazole ((*R*)-**8**) (Daicel Chiralcel OD-H column; *n*-hexane/*i*-propanol = 99:1, 0.7 mL/min, 254 nm)



Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	8.780	BB	0.2182	16.30239	1.11812	0.4028
2	9.442	BB	0.2372	4031.32129	256.58481	99.5972
Totals:				4047.62368	257.70293	