Supplementary Materials

Enantiomeric Variability of Distaminolyne A. Refinement of ECD and NMR Methods for Determining Optical Purity of 1-Amino-2-Alkanols

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Figure S1. ^{1}H NMR Spectrum of (*R*)-**5a** (CD_3OD, 500 MHz).

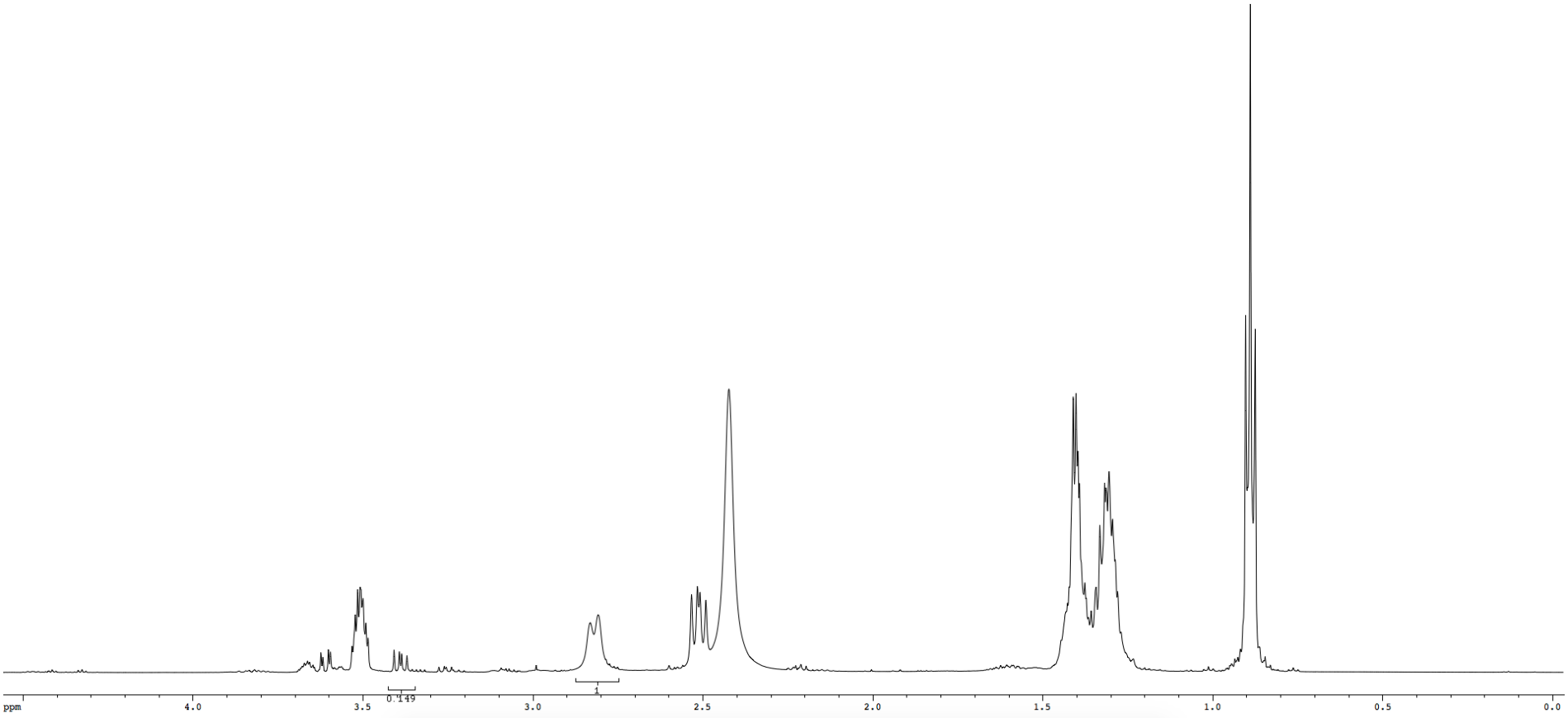


Figure S2. ^{13}C NMR Spectrum of (*R*)-**5a** (CD_3OD, 125 MHz).

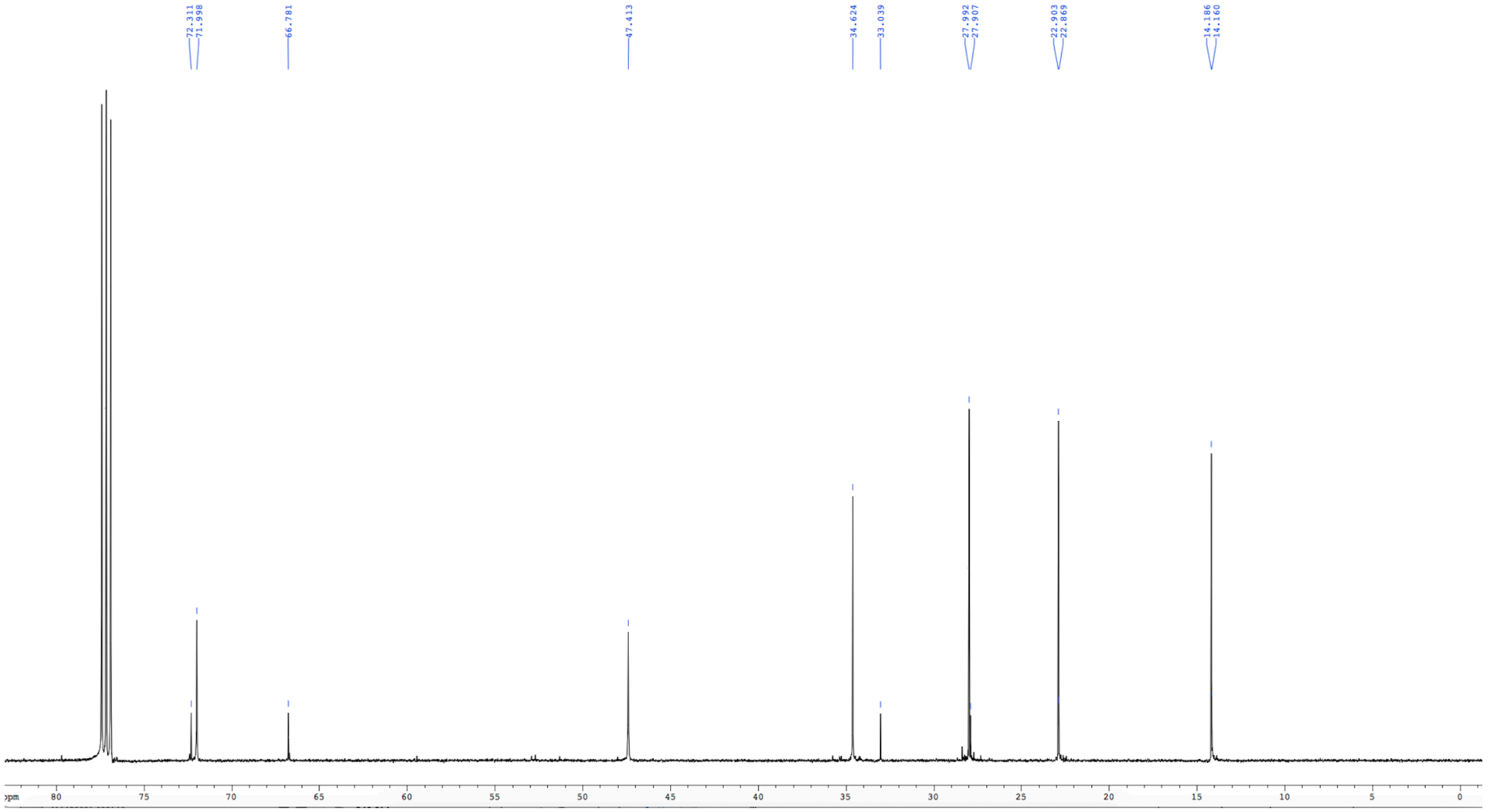


Figure S3. ^{1}H NMR Spectrum of (*S*)-**6a** ($CDCl_3$, 500 MHz).

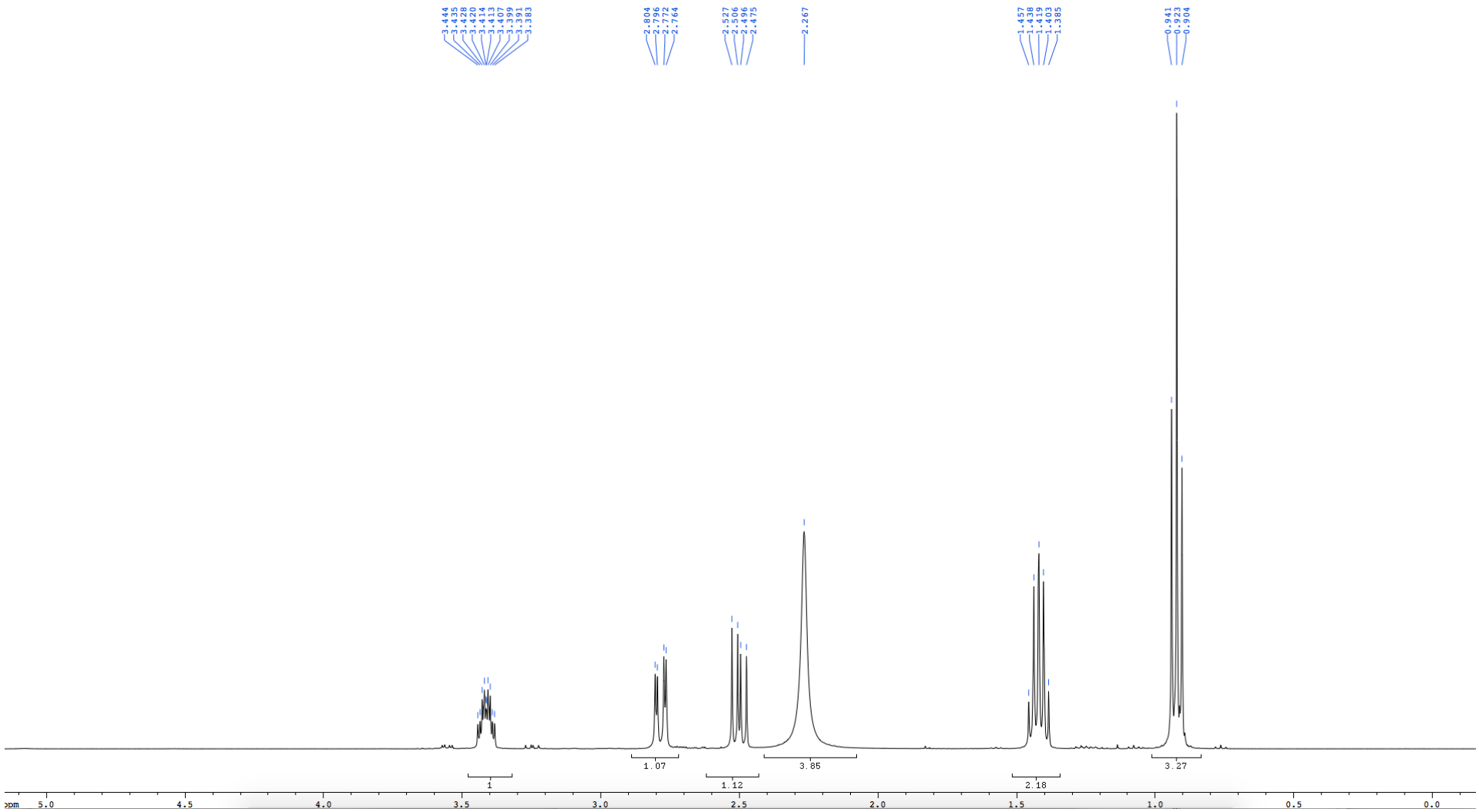


Figure S4. ^{13}C NMR Spectrum of (*S*)-**6a** ($CDCl_3$, 125 MHz)

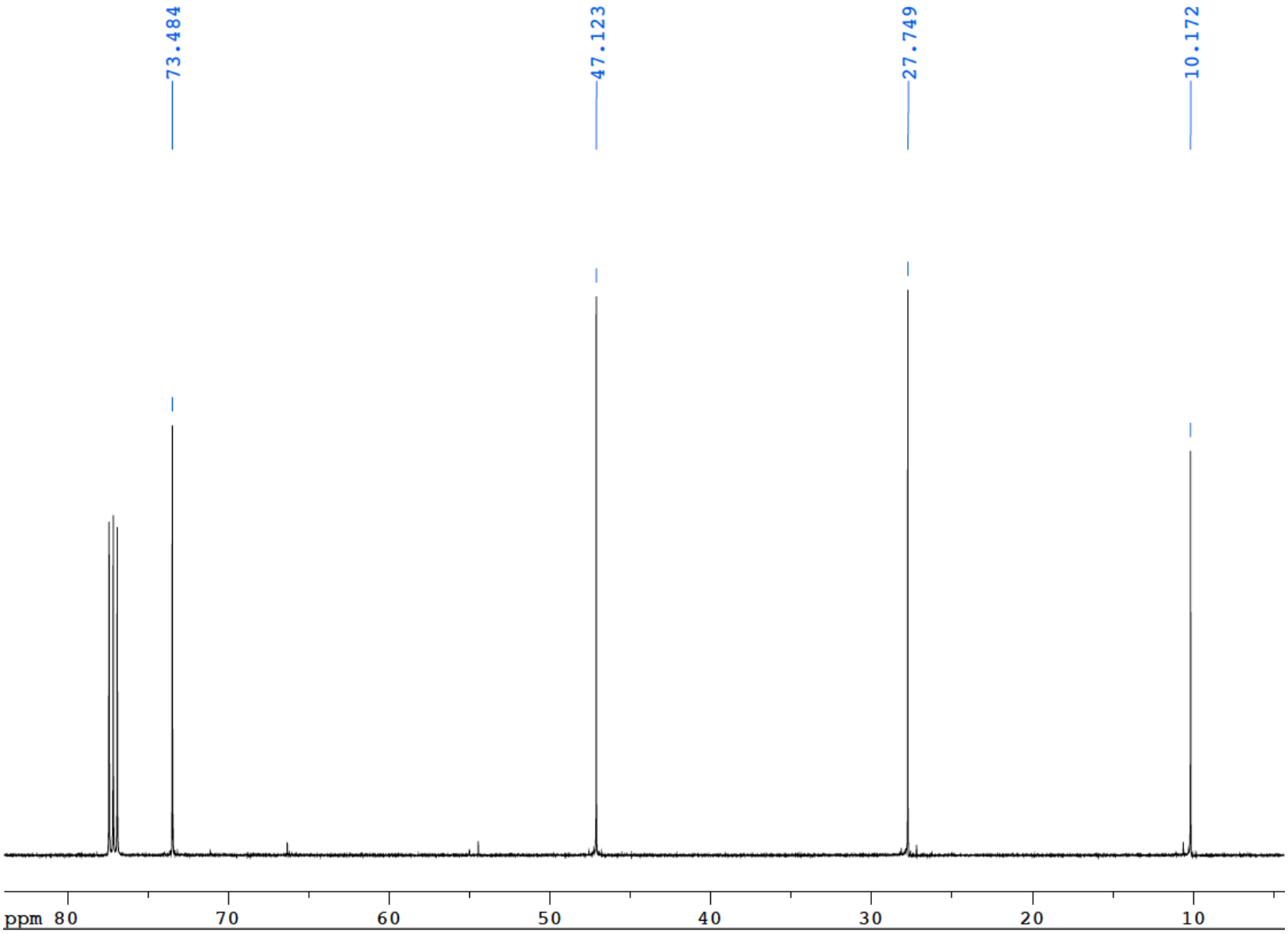


Figure S5. ^{1}H NMR Spectrum of (*R*)-**7b** ($CDCl_3$, 500 MHz).

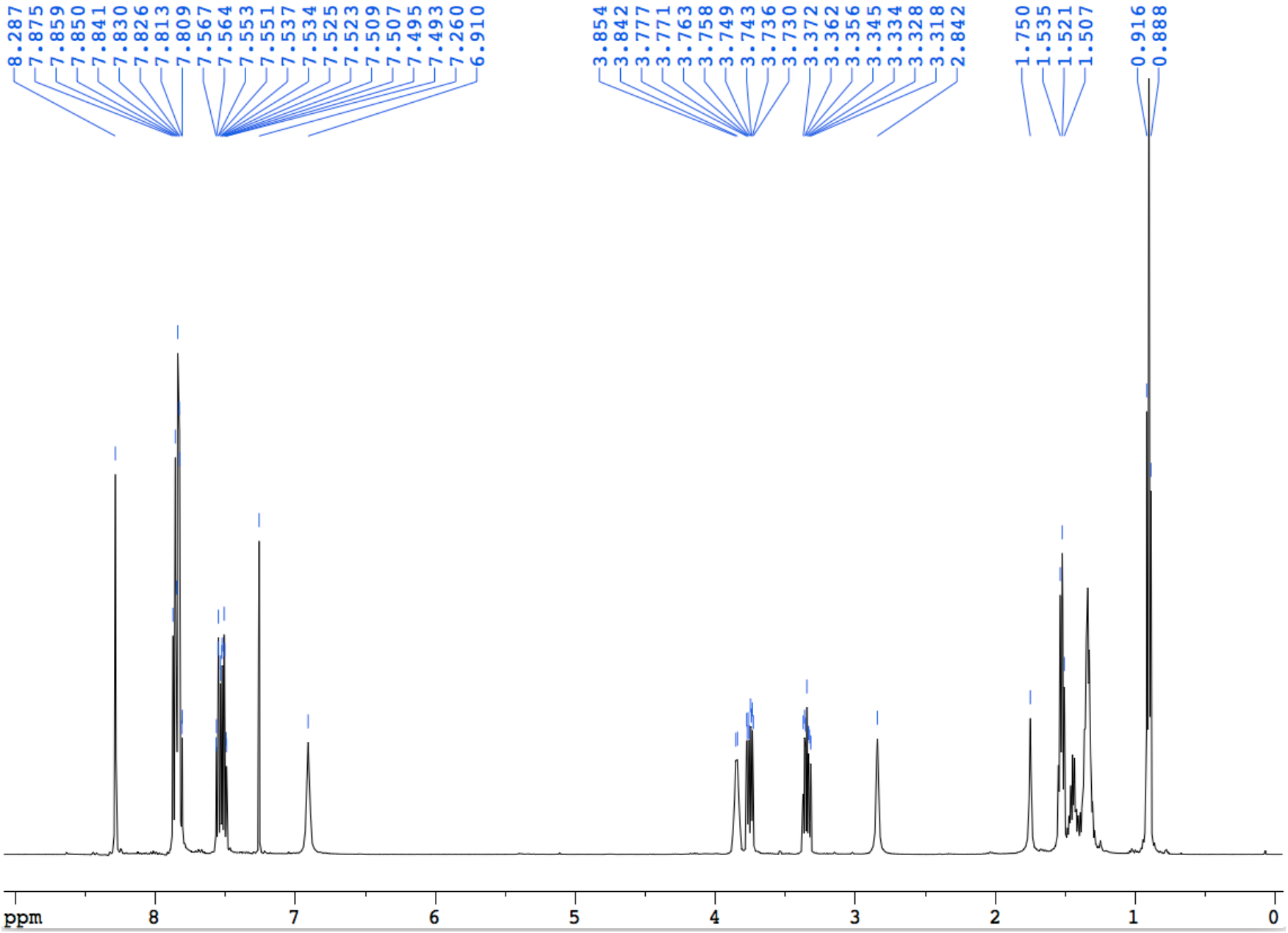


Figure S6. ^{13}C NMR Spectrum of (*R*)-**7b** ($CDCl_3$, 125 MHz).

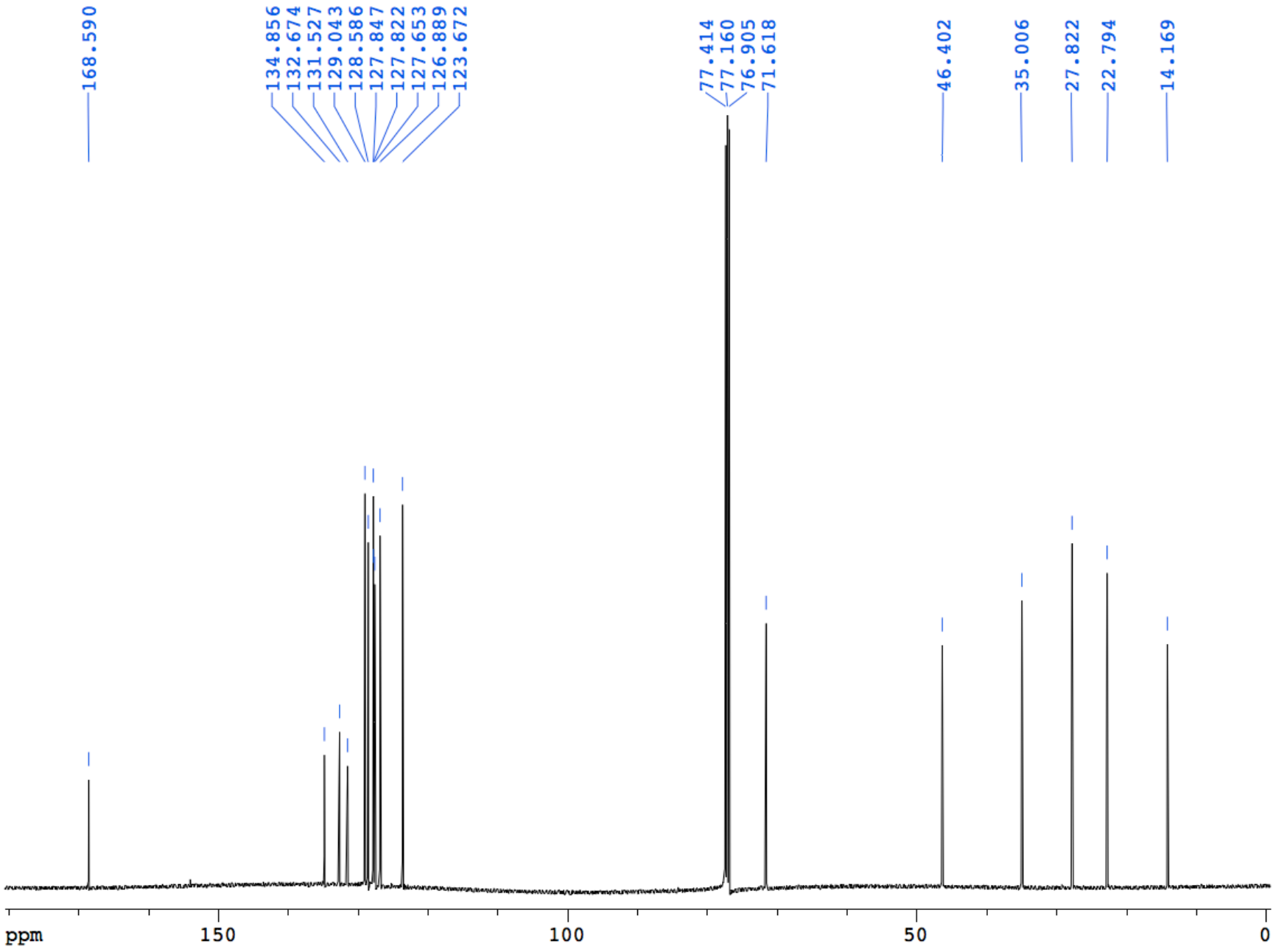


Figure S7. ^{1}H NMR Spectrum of **8** ($CDCl_3$, 500 MHz)

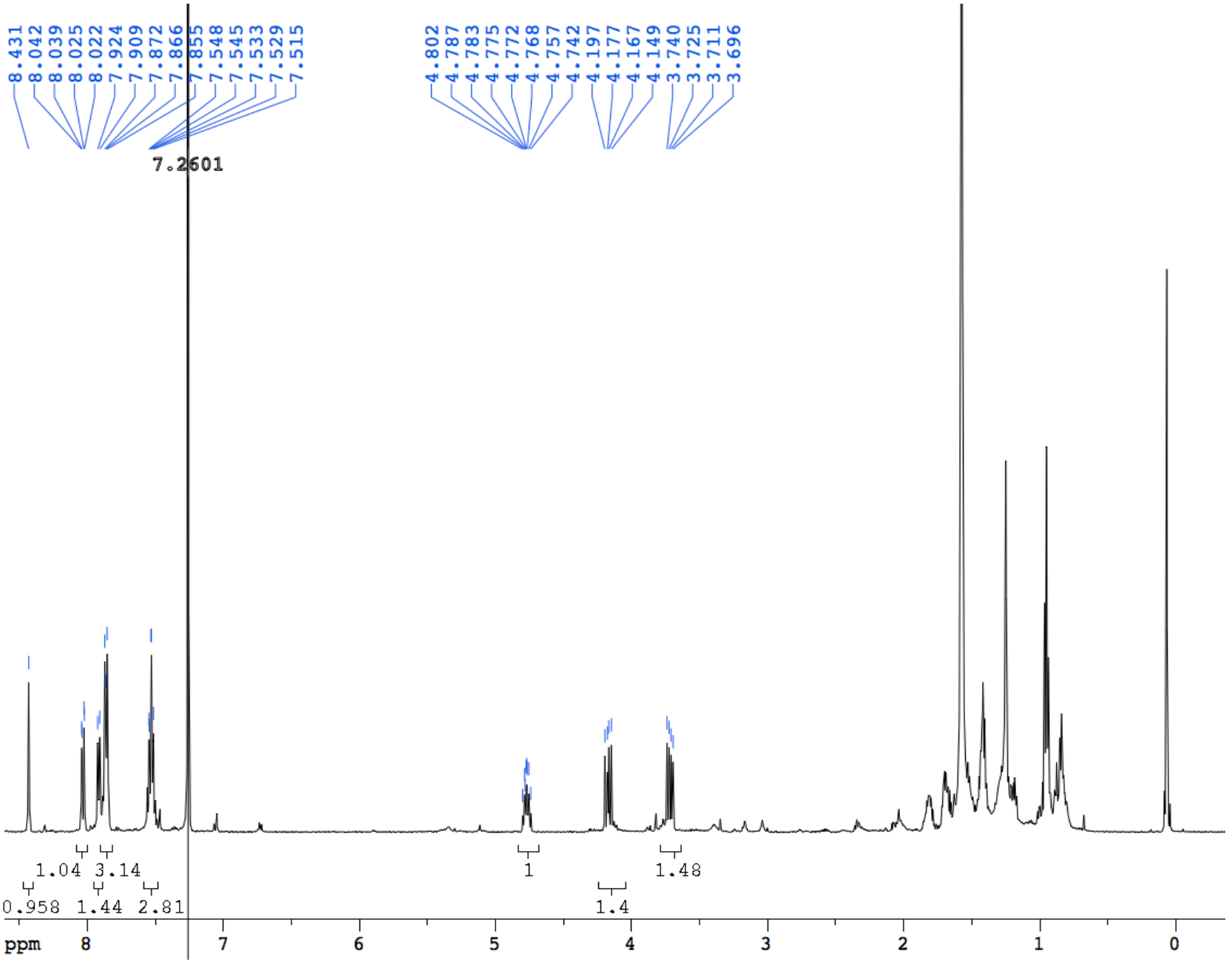


Figure S8. ^{1}H NMR Spectrum of (*R*)-**9b** ($CDCl_3$, 500 MHz) and expansions (a), (b), (c).

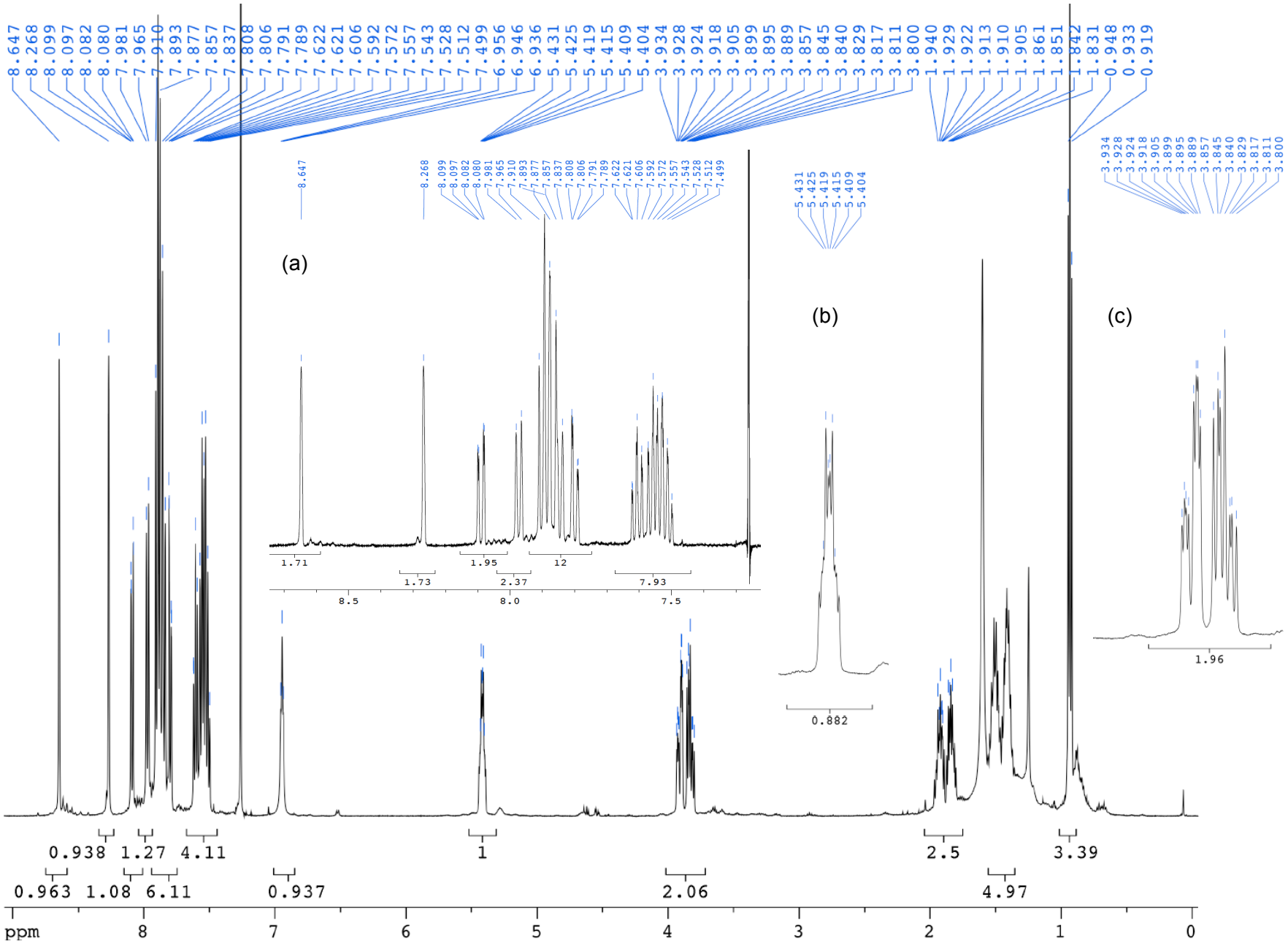


Figure S9. ^{13}C NMR Spectrum of (*R*)-**9b** ($CDCl_3$, 125 MHz).

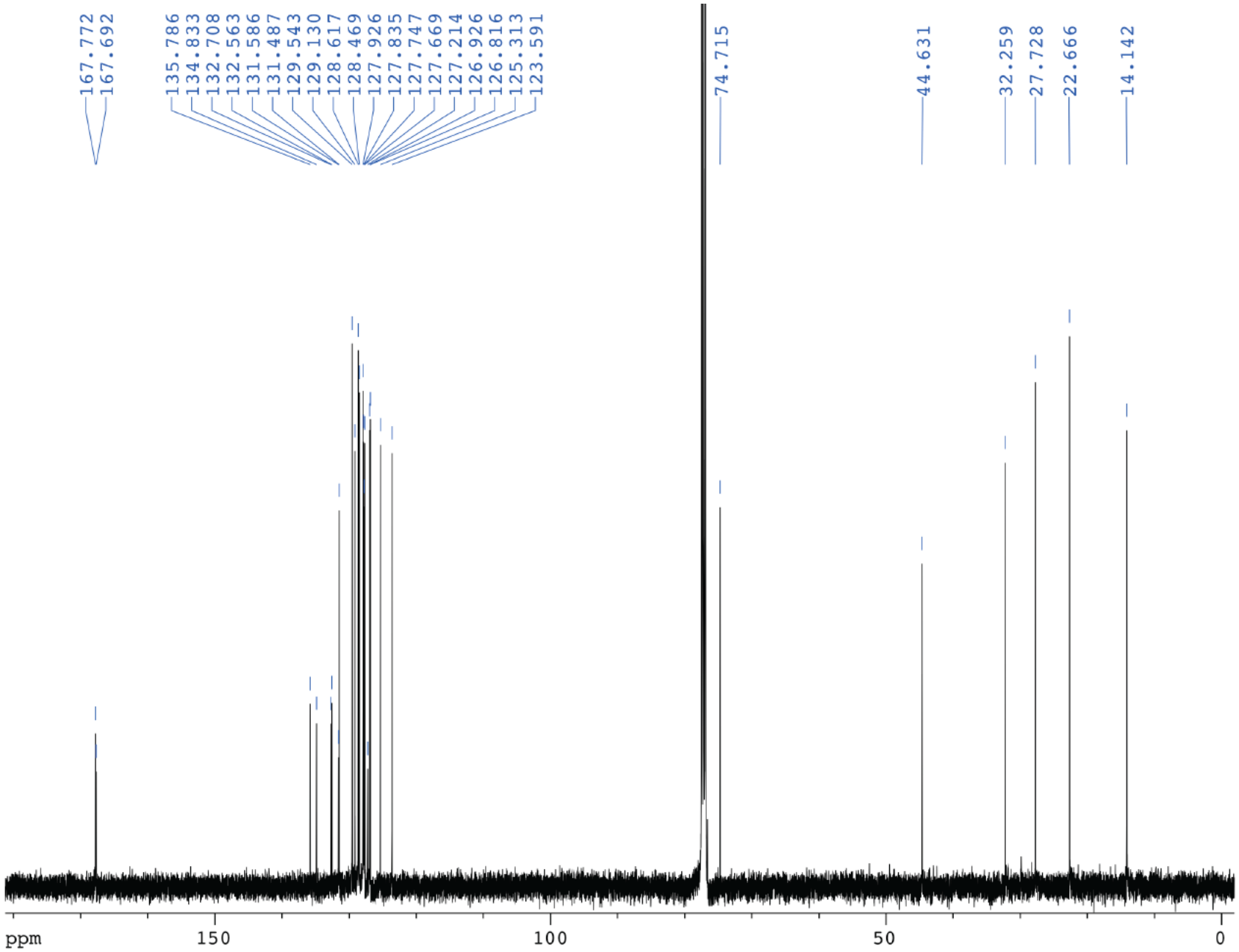


Figure S10. ^{1}H NMR Spectrum of (*S*)-**10a** ($CDCl_3$, 500 MHz)

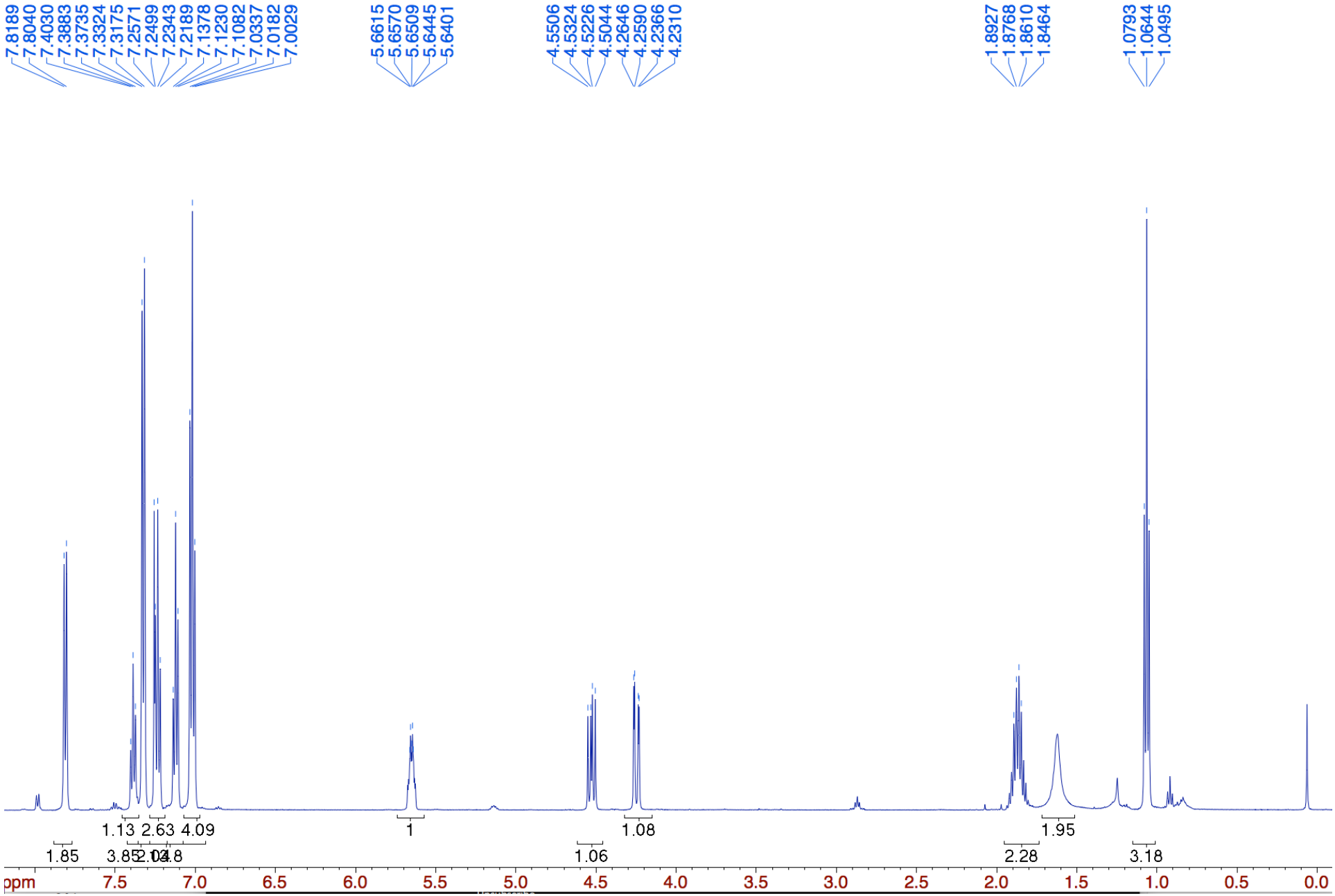


Figure S11. ^{13}C NMR Spectrum of (*S*)-**10a** ($CDCl_3$, 125 MHz).

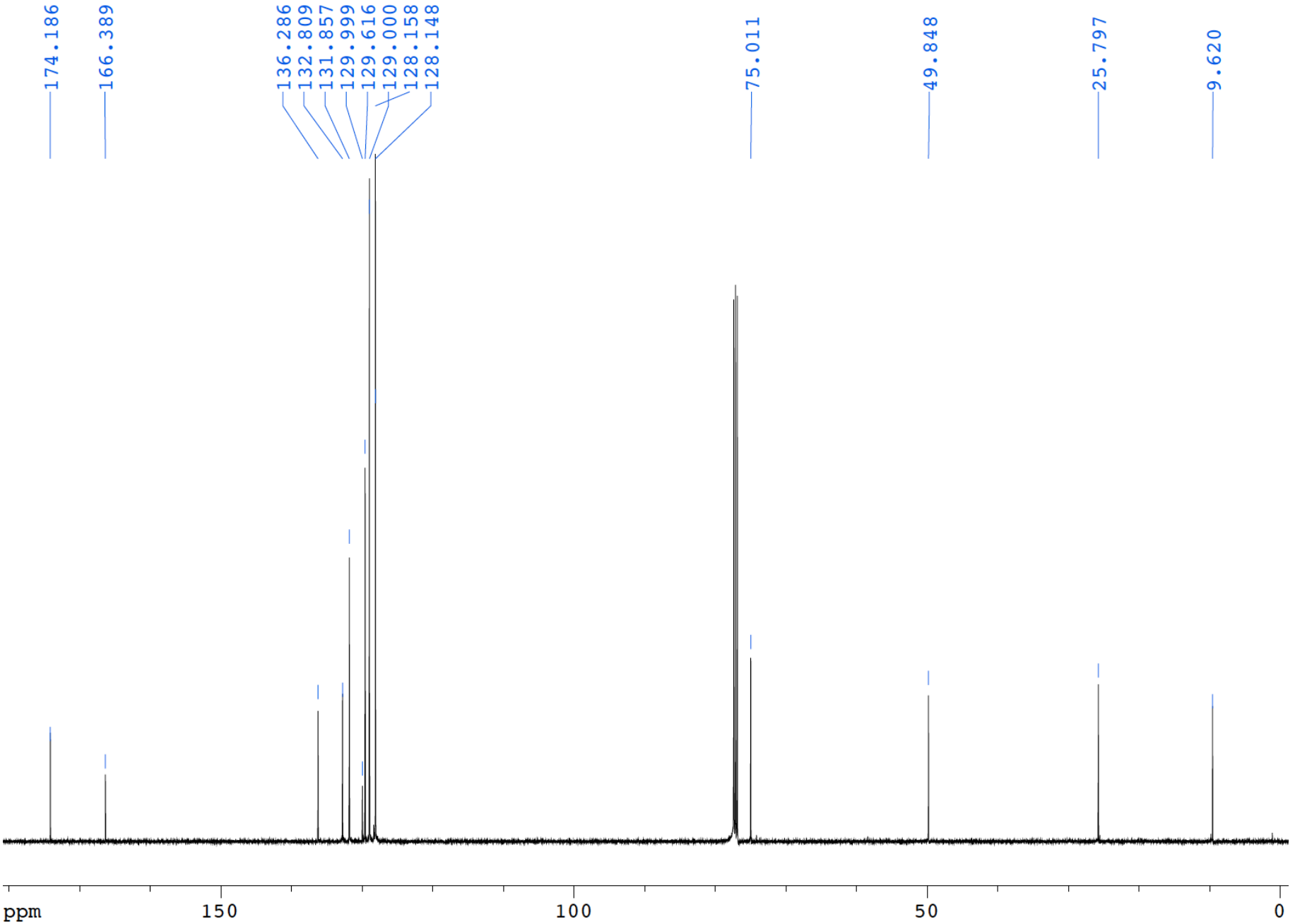


Figure S12. ^{1}H NMR Spectrum of (*R*)-**11b** ($CDCl_3$, 500 MHz).

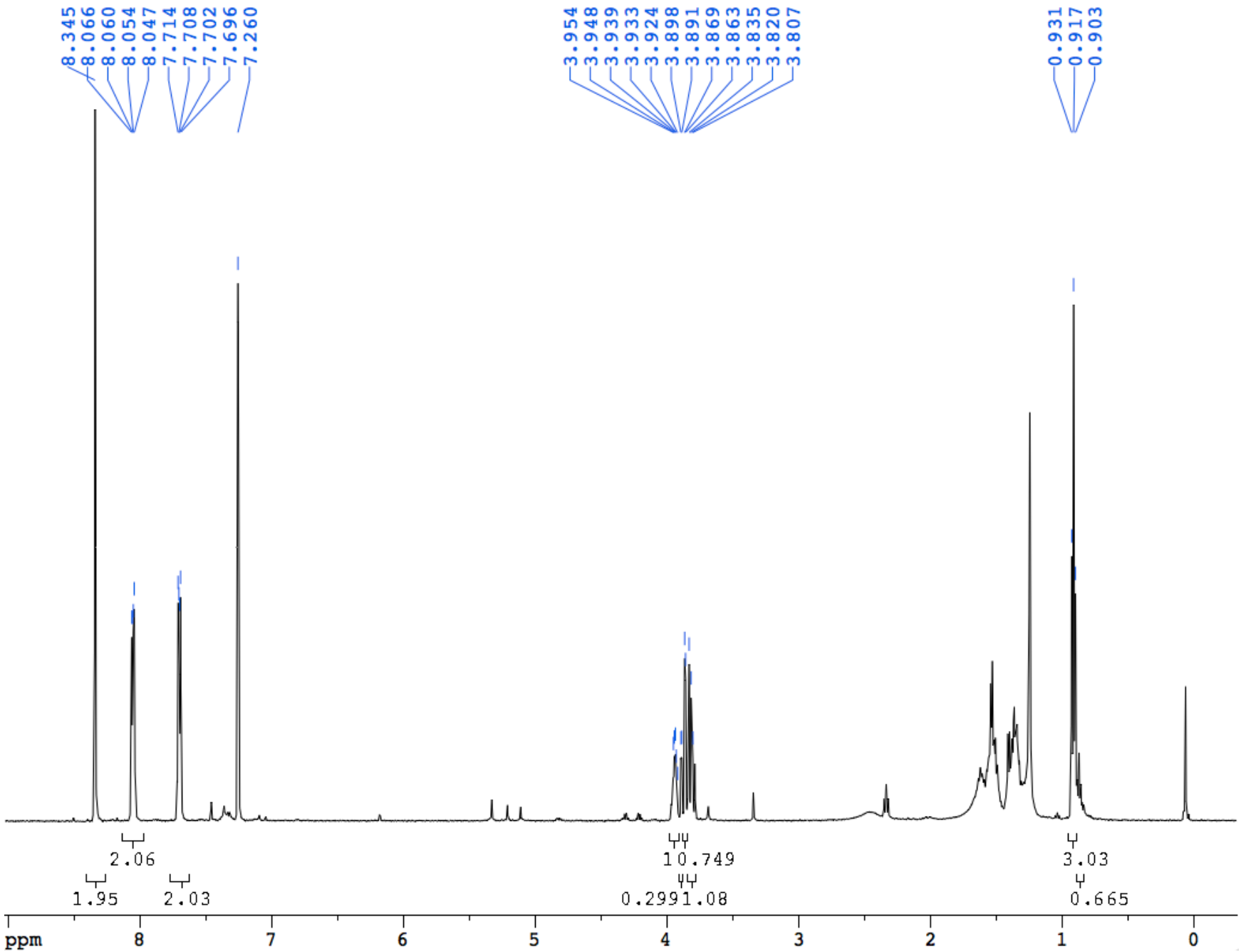


Figure S13. ^{13}C NMR Spectrum of (*R*)-**11b** ($CDCl_3$, 150 MHz).

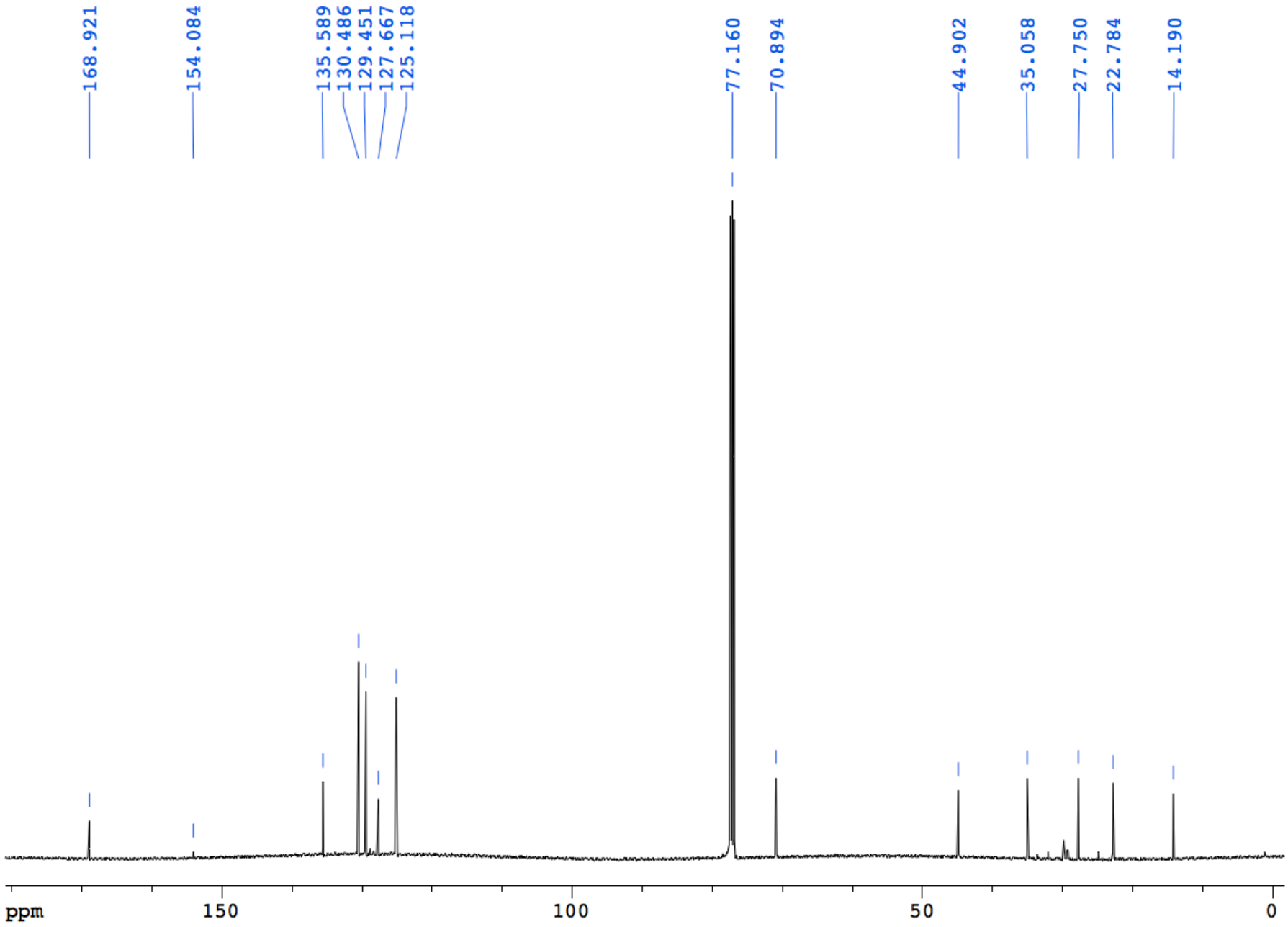


Figure S14. ^{1}H NMR Spectrum of (*R*)-**12b** ($CDCl_3$, 500 MHz).

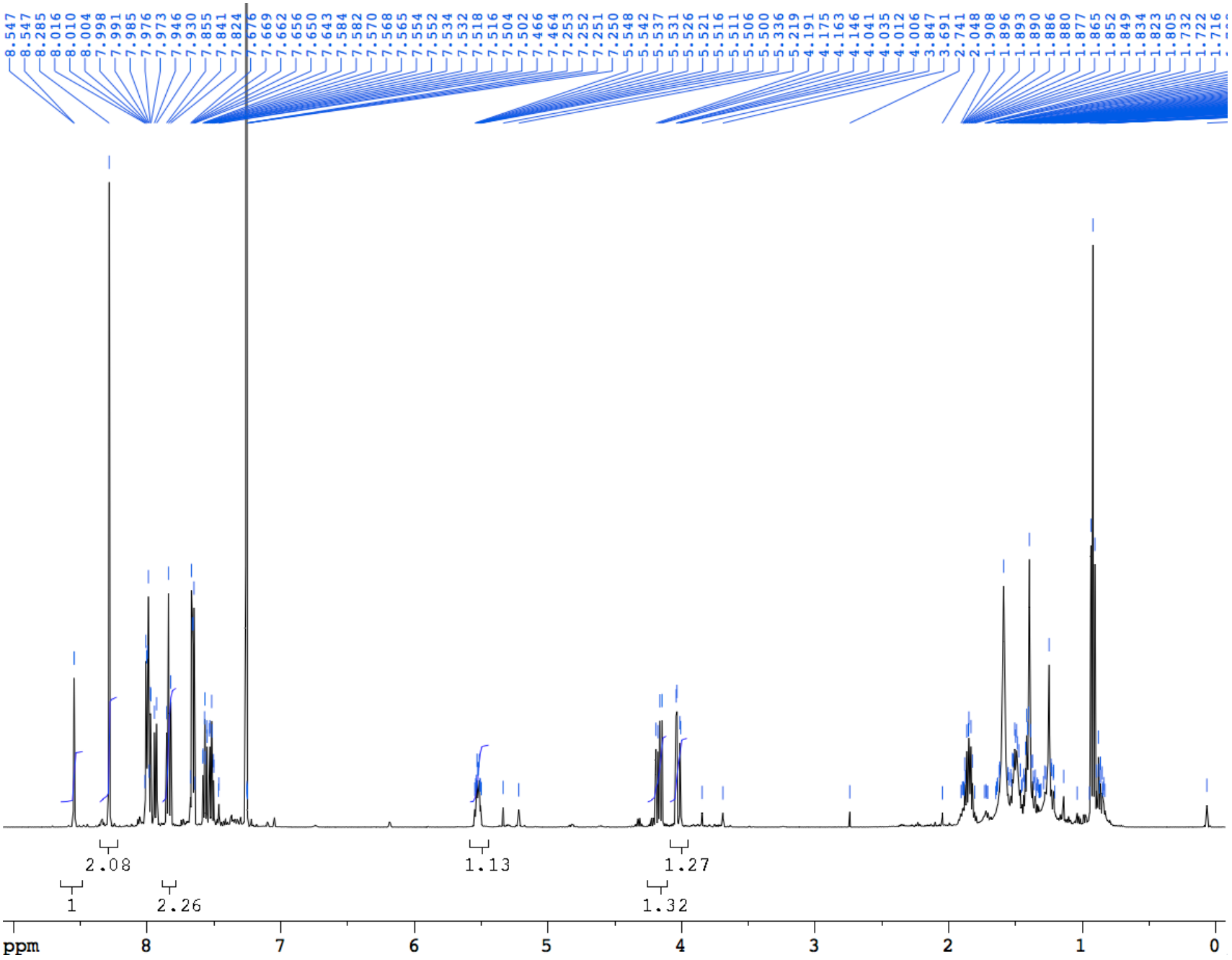


Figure S15. ^{1}H NMR Spectrum of (*R*)-**12b** ($CDCl_3$, 500 MHz) – Expansions (a) and (b)

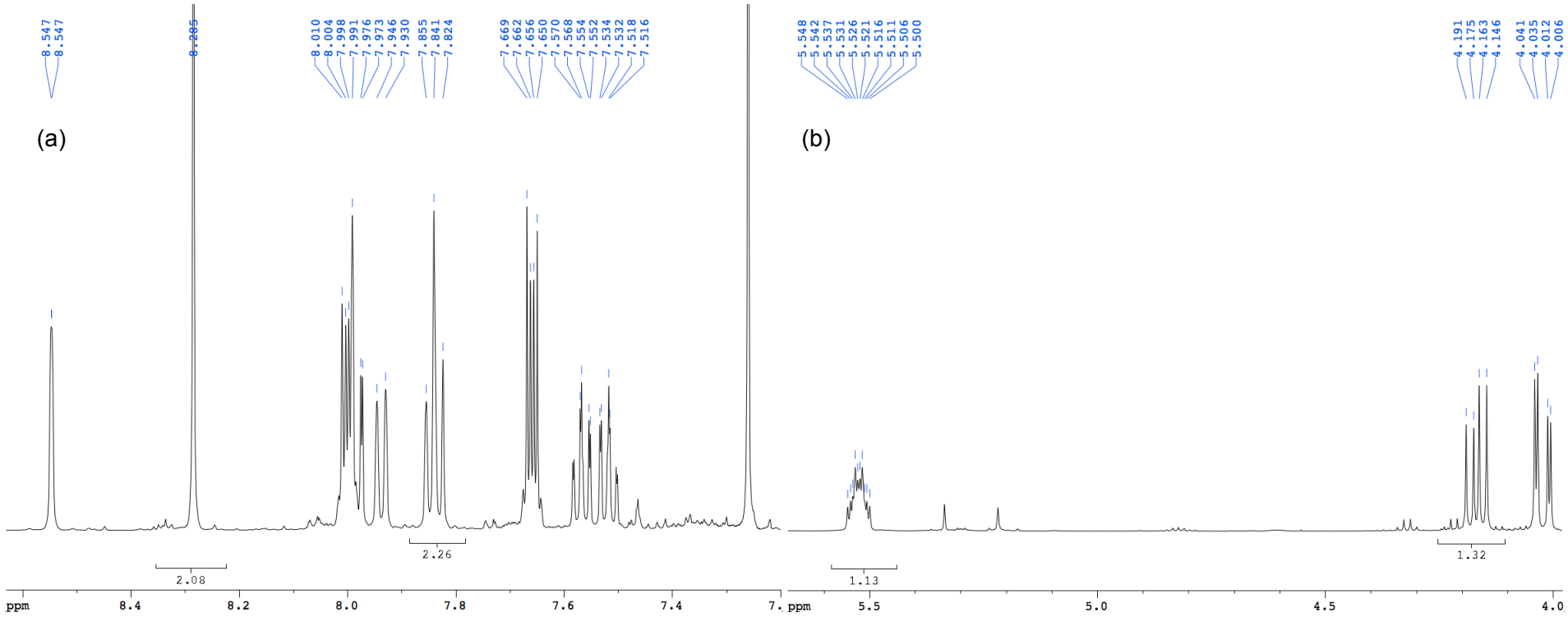


Figure S16. ^{1}H NMR Spectrum of (*S*)-**13a** ($CDCl_3$, 400 MHz)

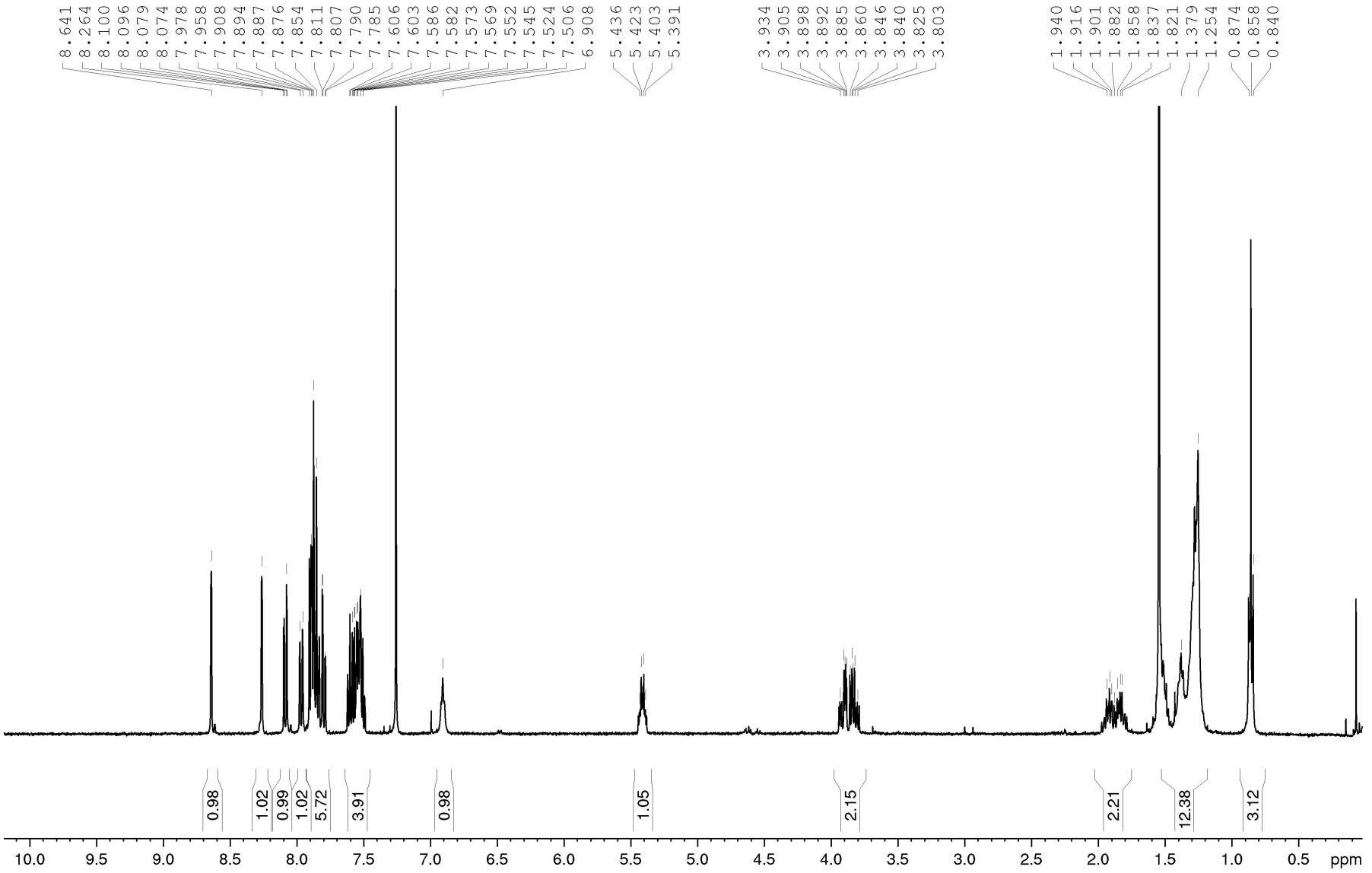
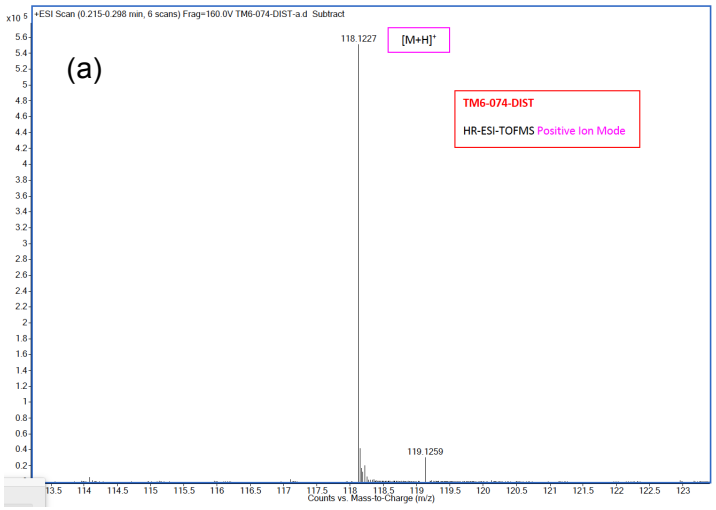
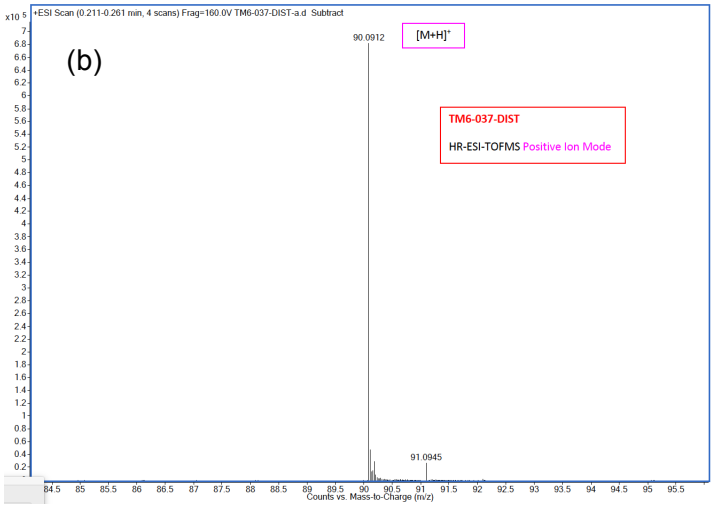


Figure S17. ESI HRMS of (a) (*R*)-**5b**, (b) (*S*)-**6a** and (c) (*R*)-**7b**.



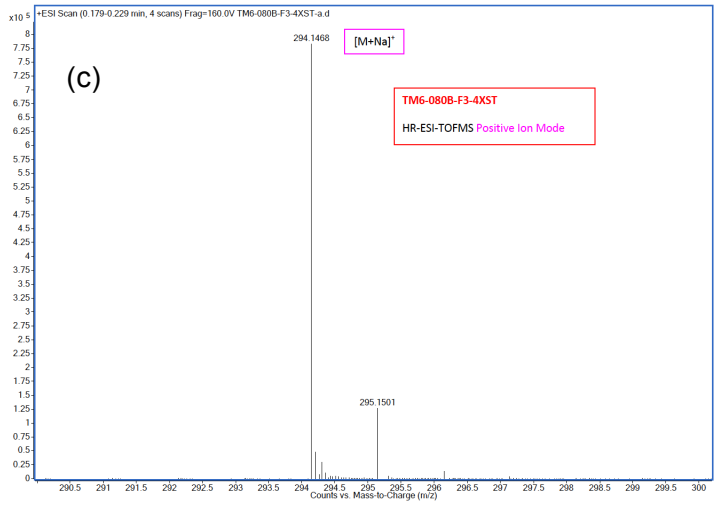
Search Results: Sample TM6-074-DIST

Mass Measured	Theo. Mass	Delta (ppm)	Composition
118.1227	118.1226	0.8	$[C_6H_{16}NO]^+$



Search Results: Sample TM6-037-DIST

Mass Measured	Theo. Mass	Delta (ppm)	Composition
90.0912	90.0913	-1.1	$[C_4H_{12}NO]^+$



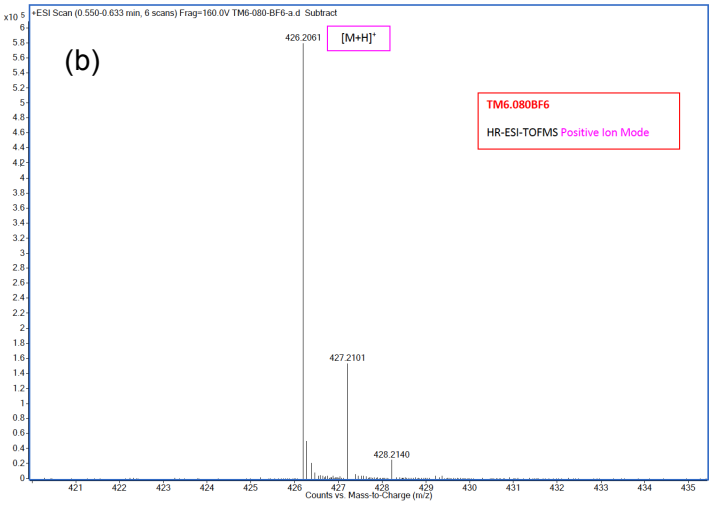
Search Results: Sample TM6-080B-F3-4XST

Mass Measured	Theo. Mass	Delta (ppm)	Composition
294.1468	294.1465	1.0	$[C_{17}H_{21}NO_2Na]^+$

Figure S18. ESI HRMS of (a) **8**, (b) (*R*)-**9b** and (c) (*S*)-**10a**.

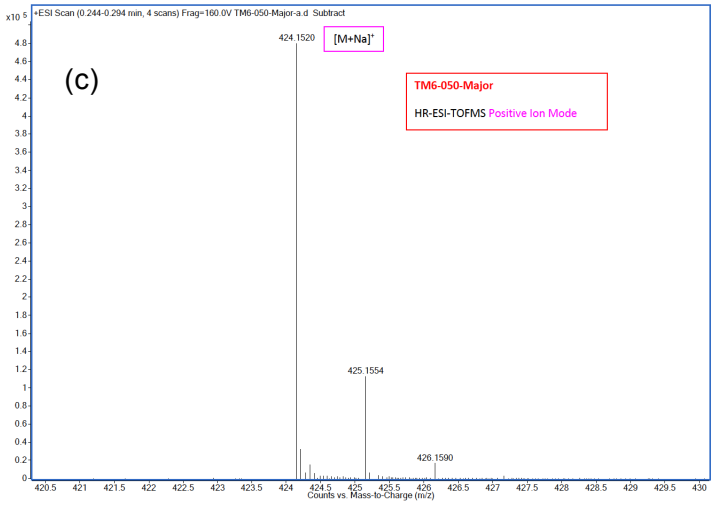
Search Results: Sample TM6-080B-F9

Mass Measured	Theo. Mass	Delta (ppm)	Composition
254.1539	254.1539	0.0	$[C_{17}H_{20}NO]^+$



Search Results: Sample TM6.080BF6

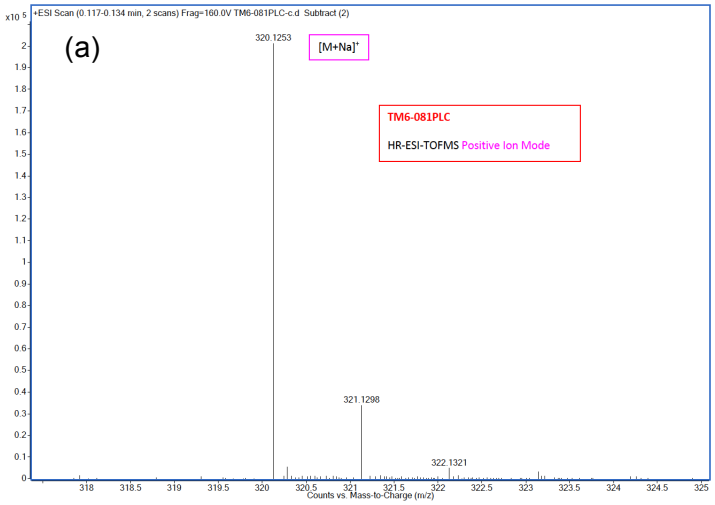
Mass Measured	Theo. Mass	Delta (ppm)	Composition
426.2061	426.2064	-0.7	$[C_{28}H_{28}NO_3]^+$



Search Results: Sample TM6-050-Major

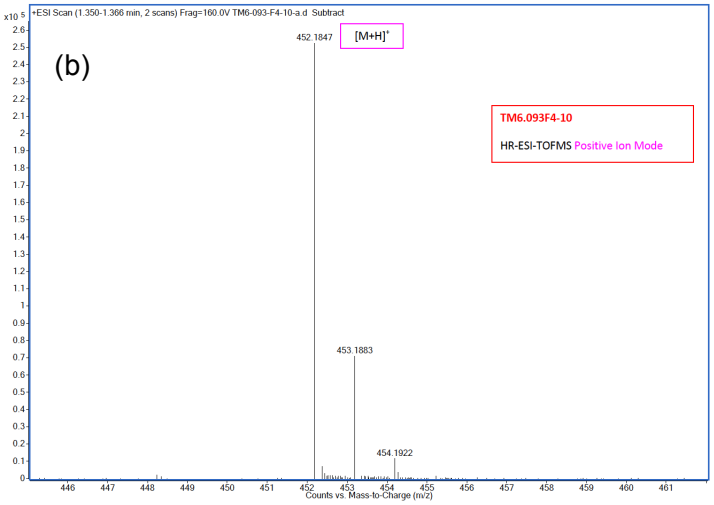
Mass Measured	Theo. Mass	Delta (ppm)	Composition
424.1520	424.1519	0.2	$[C_{25}H_{23}NO_4Na]^+$

Figure S19. ESI HRMS of (a) (*R*)-**11b**, (b) (*R*)-**12b** and (c) (*S*)-**13a**.



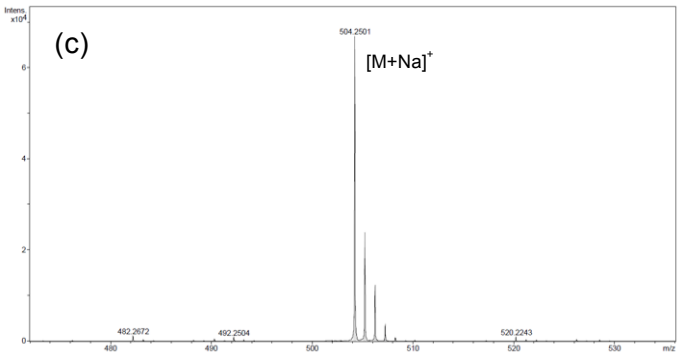
Search Results: Sample TM6-081PLC

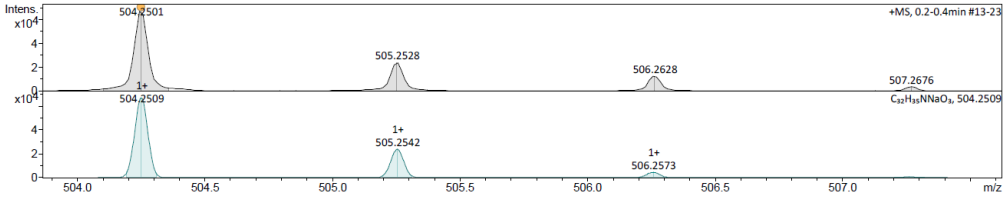
Mass Measured	Theo. Mass	Delta (ppm)	Composition
320.1253	320.1257	-1.3	$[C_{18}H_{19}NO_3Na]^+$



Search Results: Sample TM6.093F4-10

Mass Measured	Theo. Mass	Delta (ppm)	Composition
452.1847	452.1856	-2.0	$[C_{29}H_{26}NO_4]^+$





Meas. m/z	#	Ion Formula	m/z	err [ppm]	mSigma	# Sigma	Score	rdb	e⁻ Conf	N-Rule
504.2501	1	C32H32N4O2	504.2520	-3.7	62.5	1	100.00	19.0	odd	ok
	1	C32H32N4O2	504.2520	-3.7	62.5	1	100.00	19.0	odd	ok
	1	C32H35NNaO3	504.2509	-1.6	63.1	1	100.00	15.5	even	ok

Figure S20. ^{1}H NMR Spectrum of bis (*S*)-MPA derivative of (*S*)-**3a** ($CDCl_3$, 500 MHz).

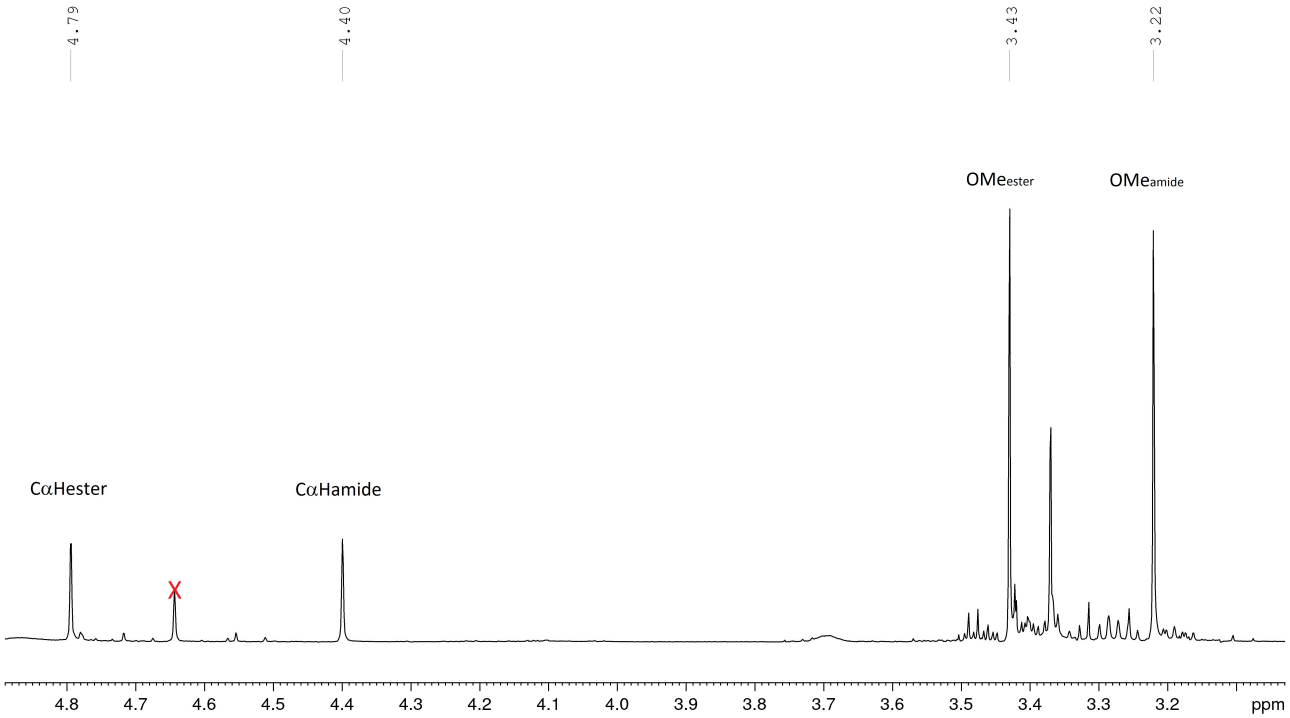


Figure S21. ^{1}H NMR Spectra of (a) *bis*-(*S*)-MPA **1c** and (b) *bis*-(*R*)-MPA **1d** highlighting CαH resonances and integrals ($CDCl_3$, 500 MHz).