

Enantiopure synthesis of 7-(1-pyrindanyl) propargyl ethers as rasagiline analogues *via* chemical or enzymatic resolution of 1-pyrindan- 7-ol

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Electronic Supplementary Information

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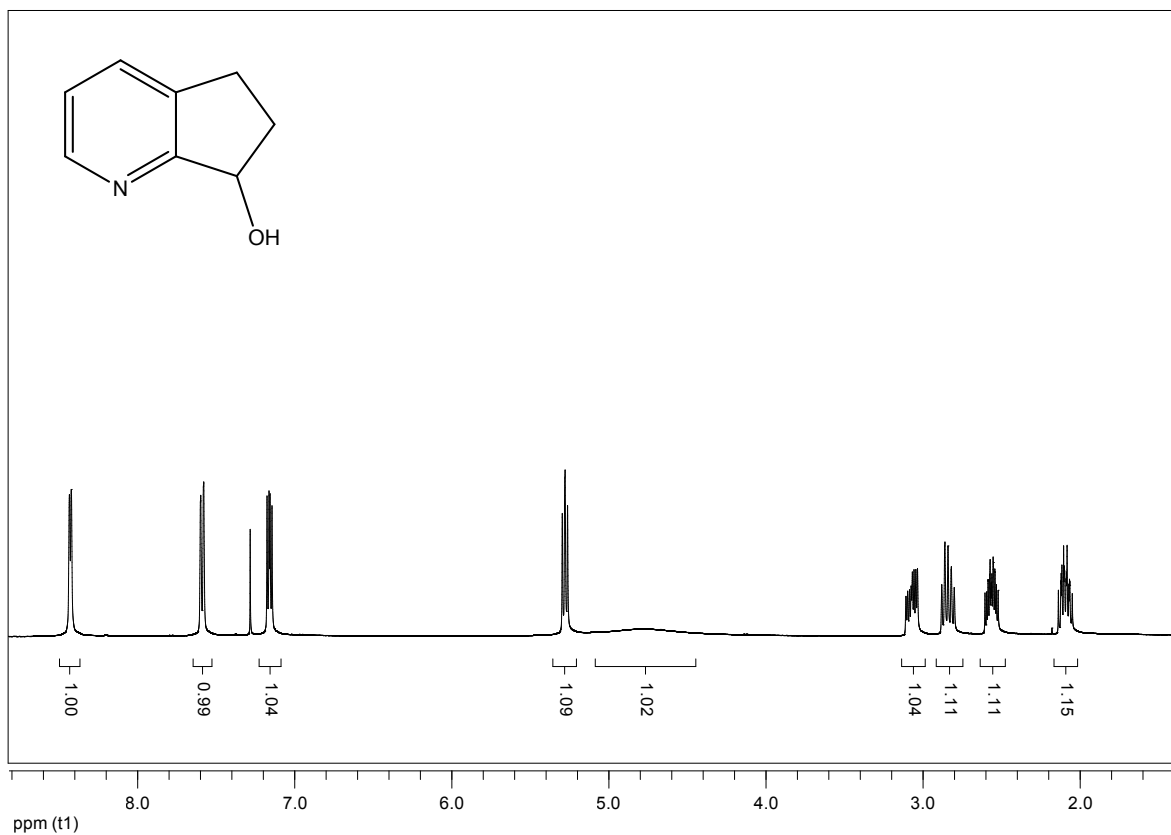


Figure 1. ¹H-NMR spectrum of compound (±)-3.

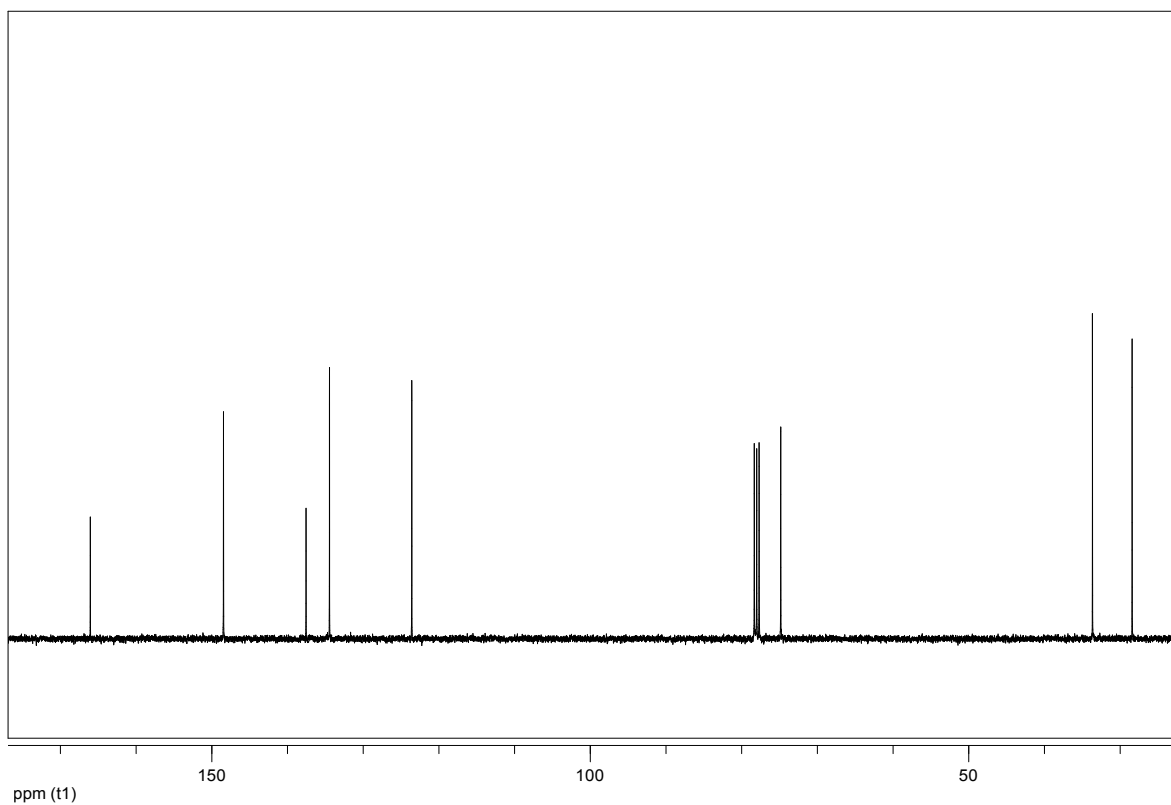


Figure 2. ¹³C-NMR spectrum of compound (±)-3.

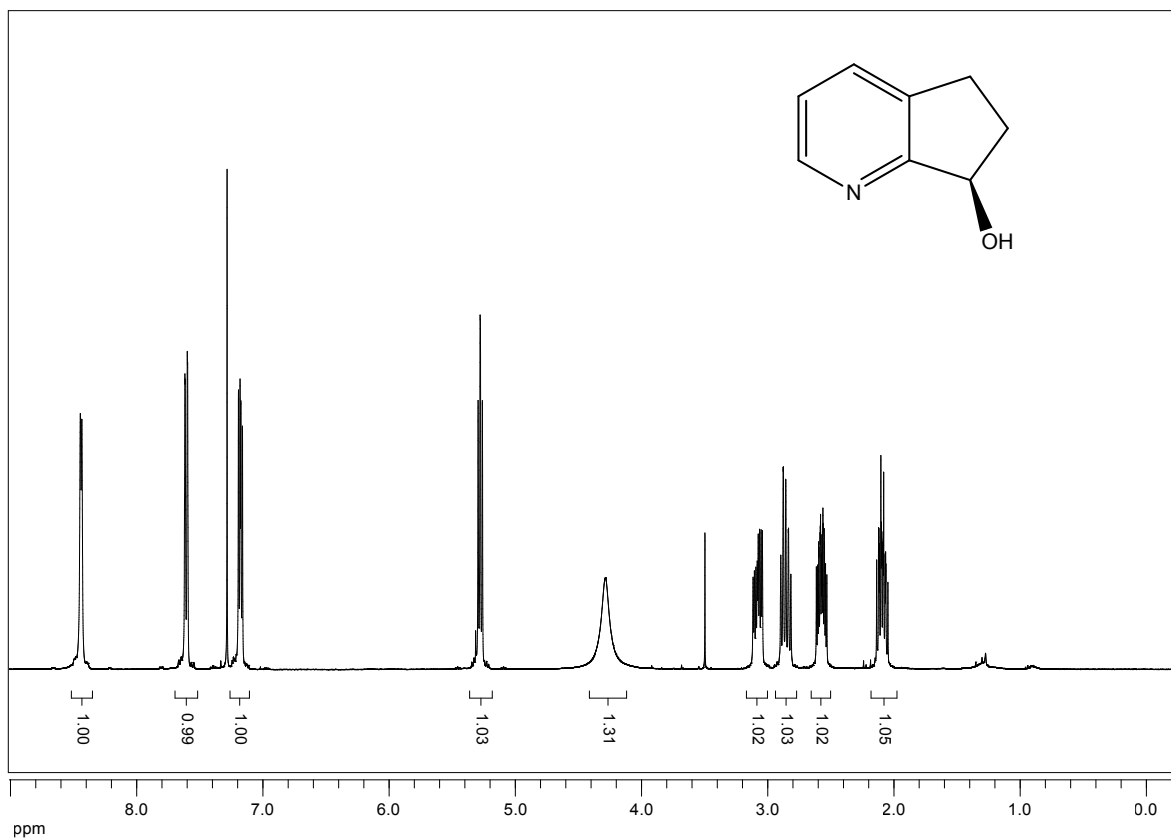


Figure 3. ¹H-NMR spectrum of compound (+)-3.

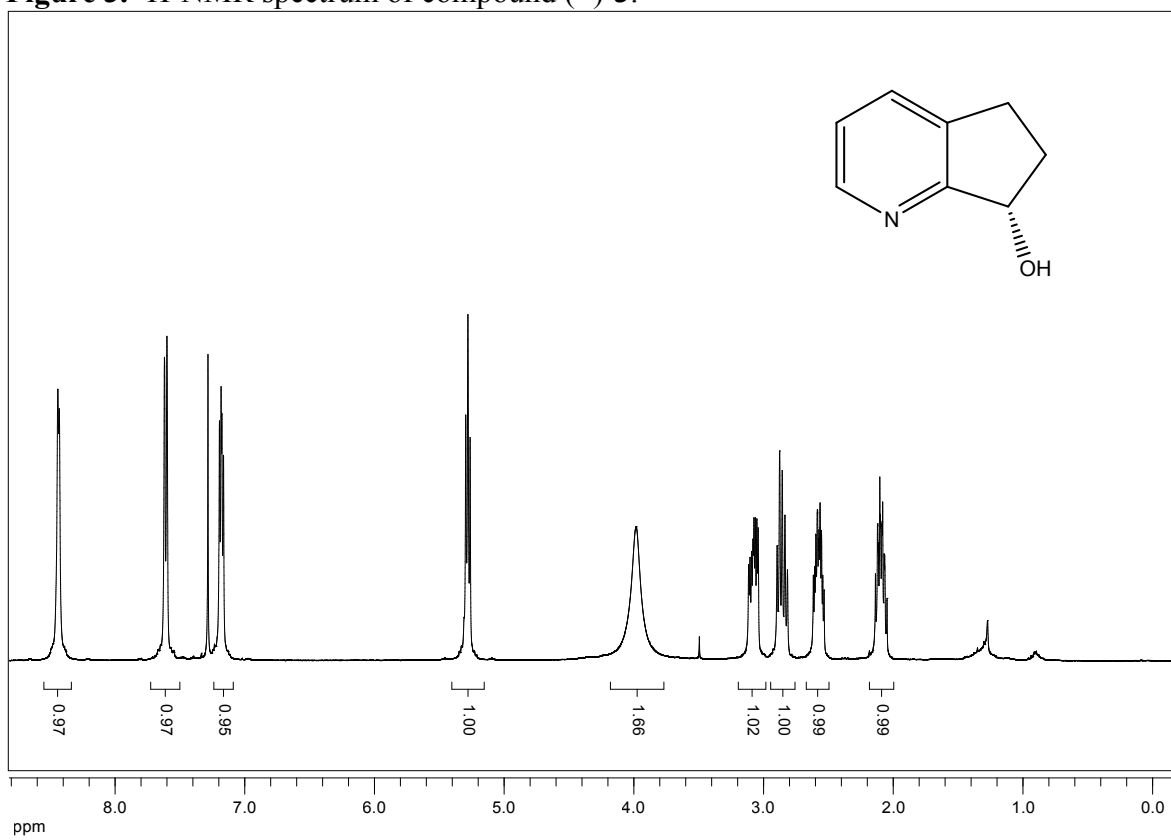


Figure 4. ¹H-NMR spectrum of compound (-)-3.

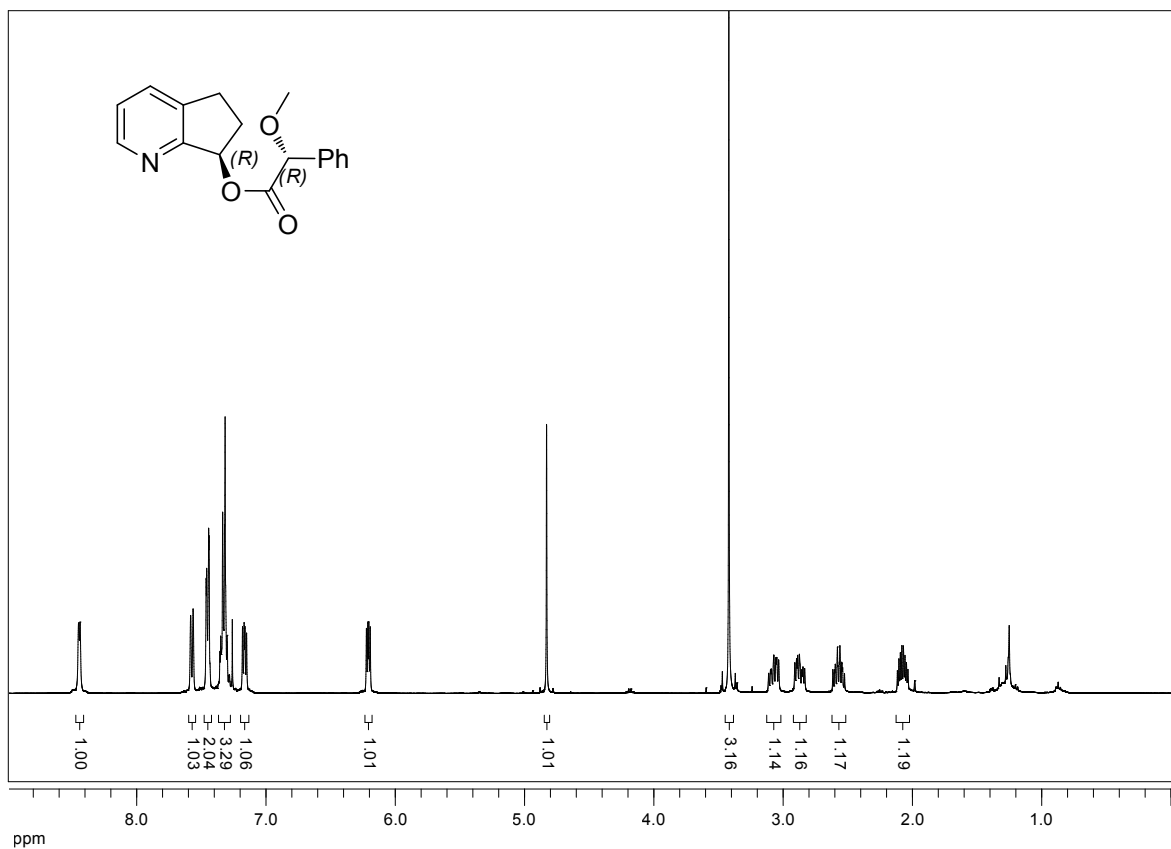


Figure 5. ¹H-NMR spectrum of compound 7a.

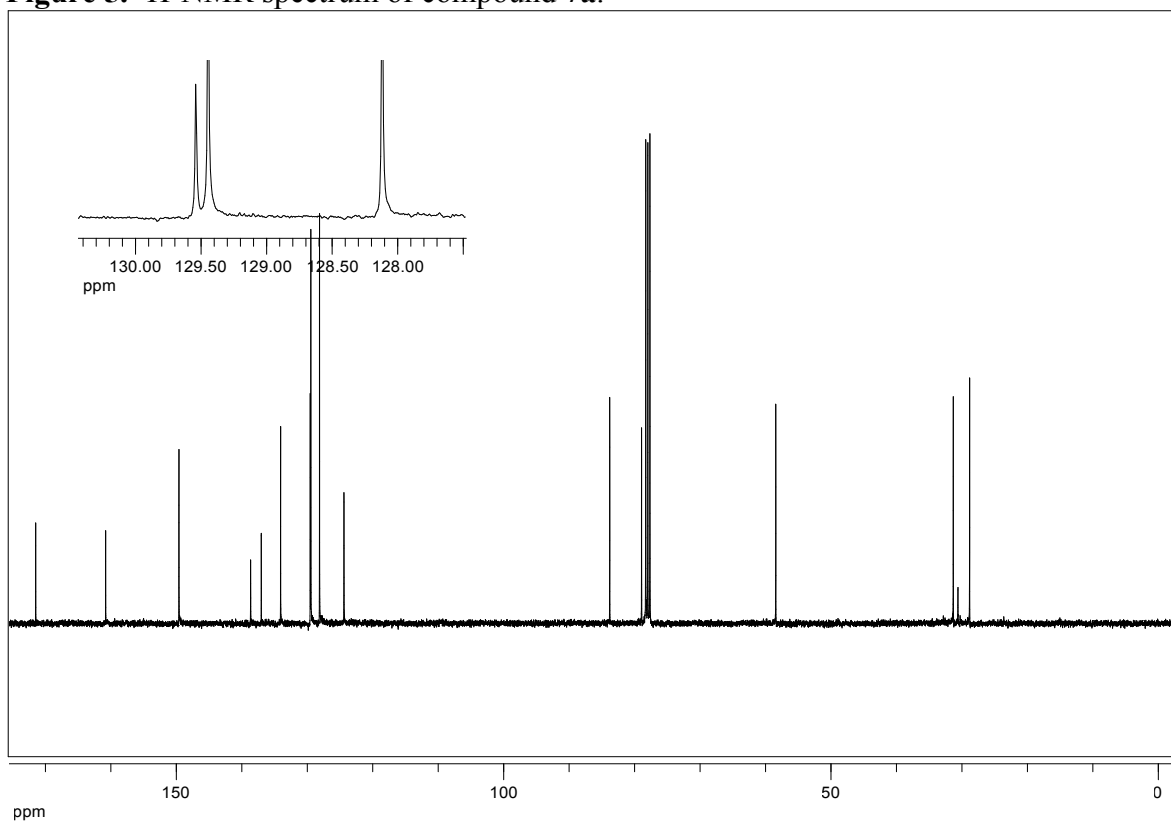


Figure 6. ¹³C-NMR spectrum of compound 7a/8a.

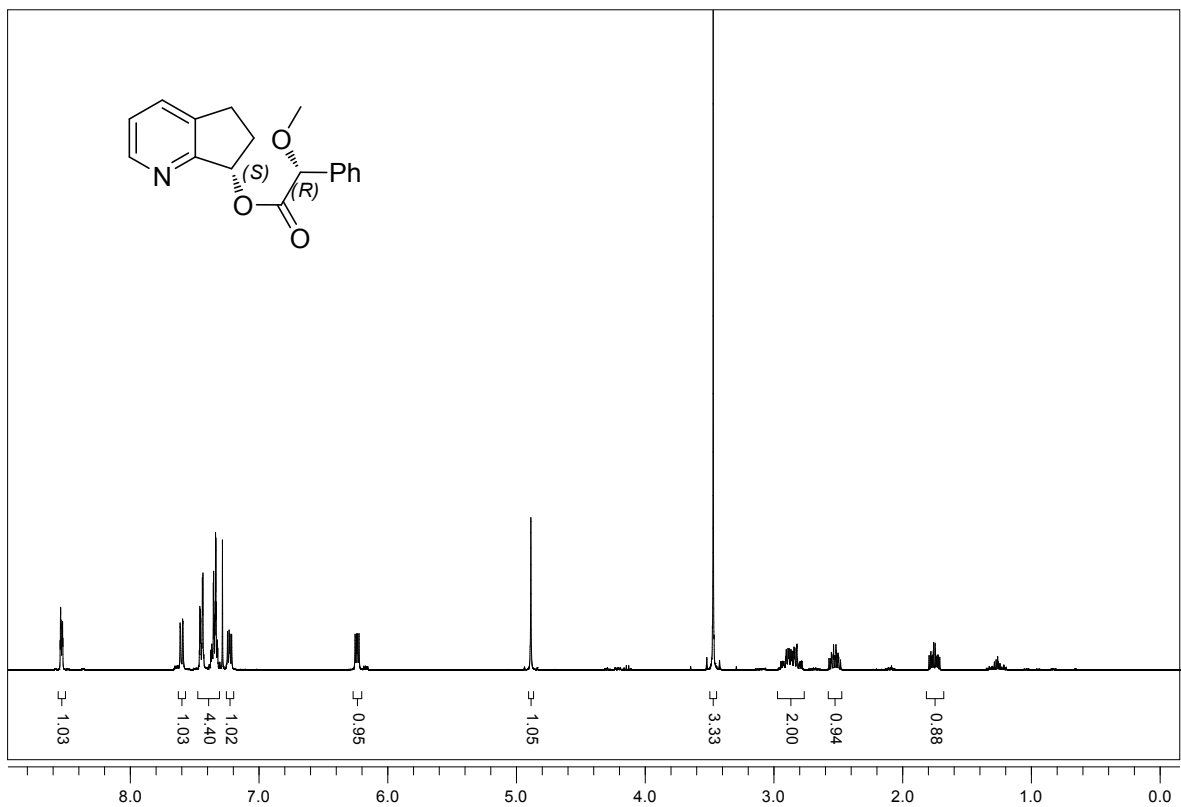


Figure 7. $^1\text{H-NMR}$ spectrum of compound **7b**.

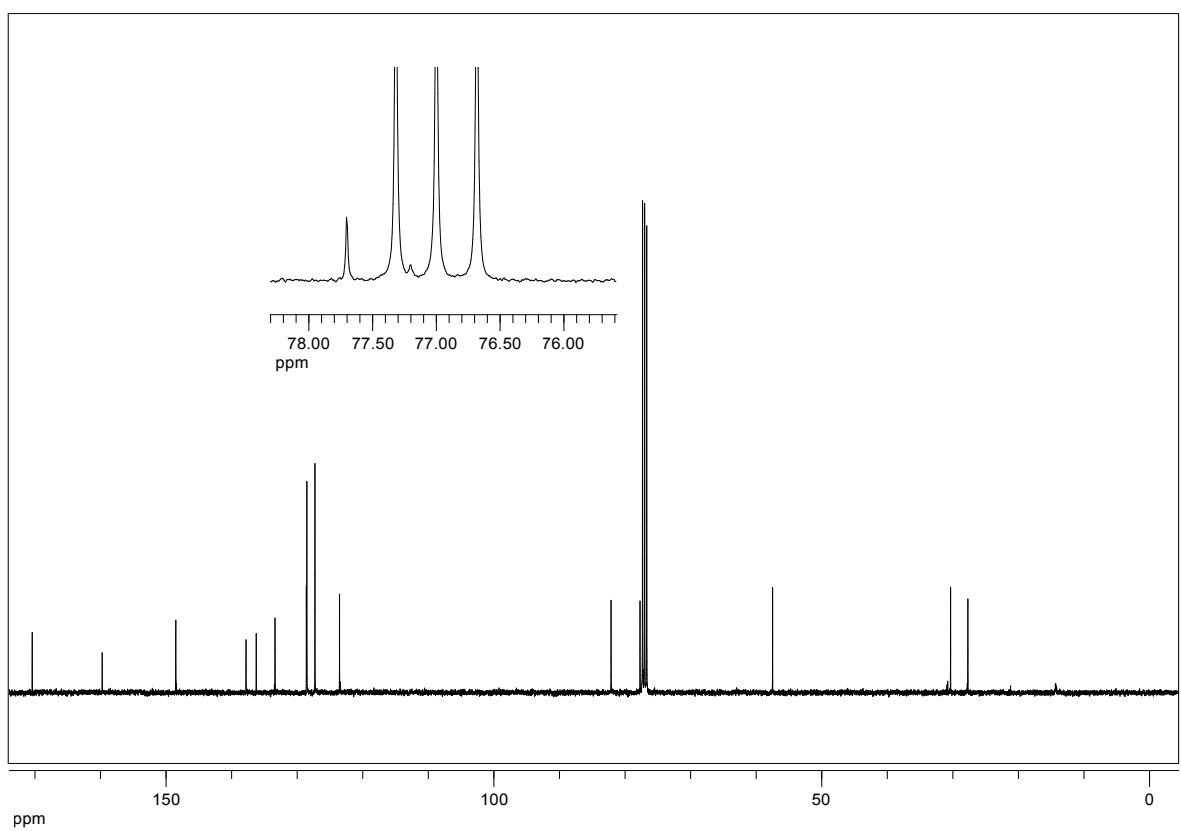


Figure 8. $^{13}\text{C-NMR}$ spectrum of compound **7b/8b**.

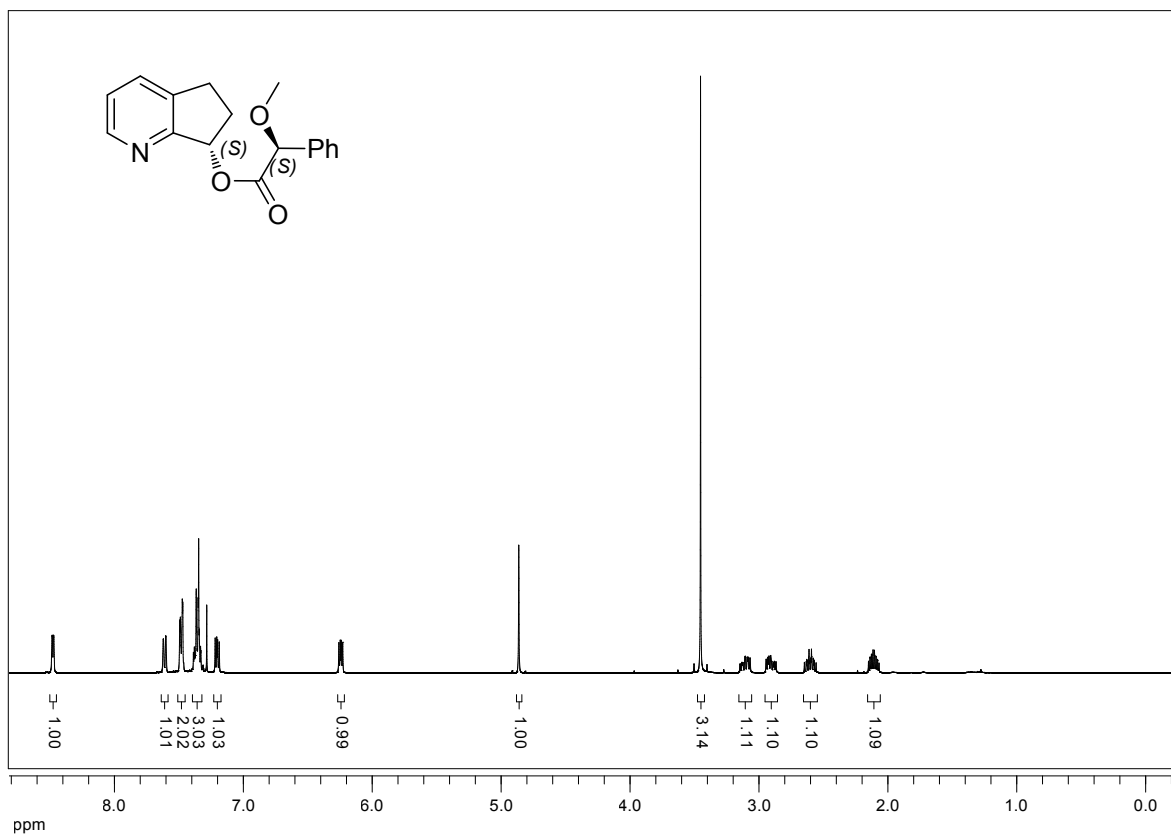


Figure 9. $^1\text{H-NMR}$ spectrum of compound **8a**.

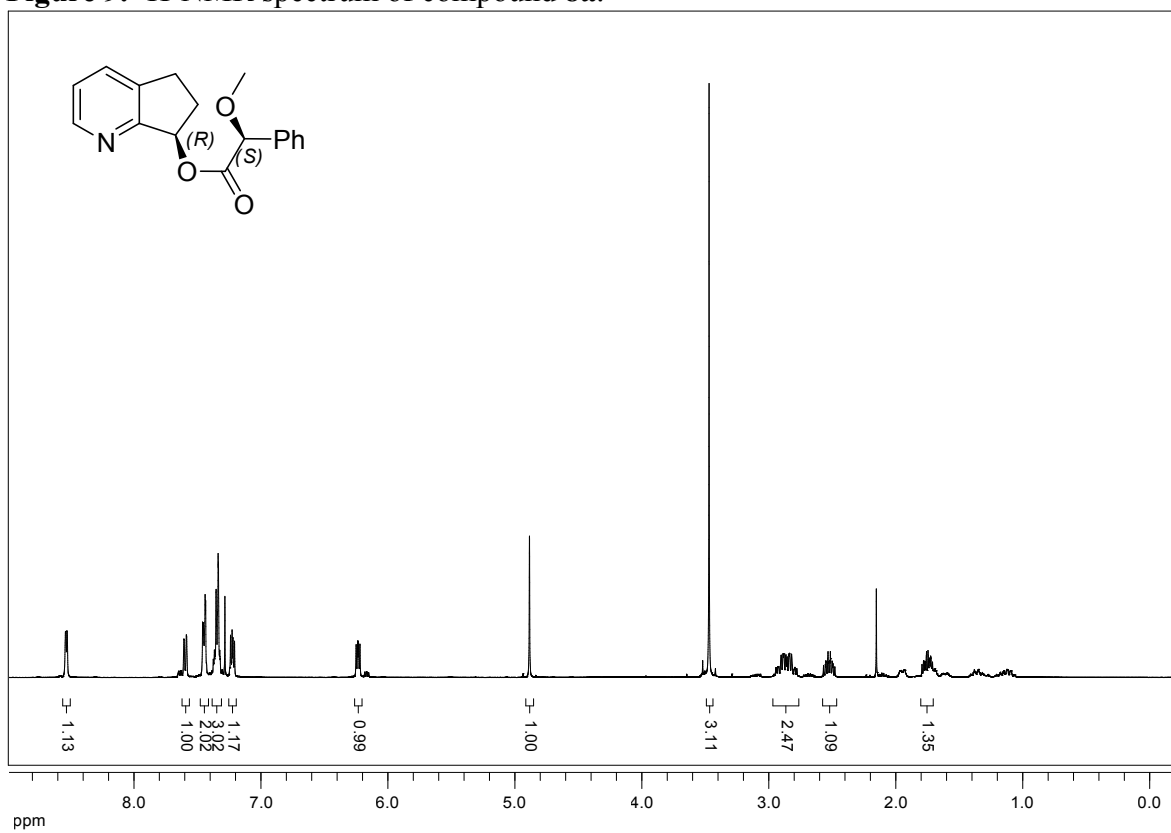


Figure 10. $^1\text{H-NMR}$ spectrum of compound **8b**.

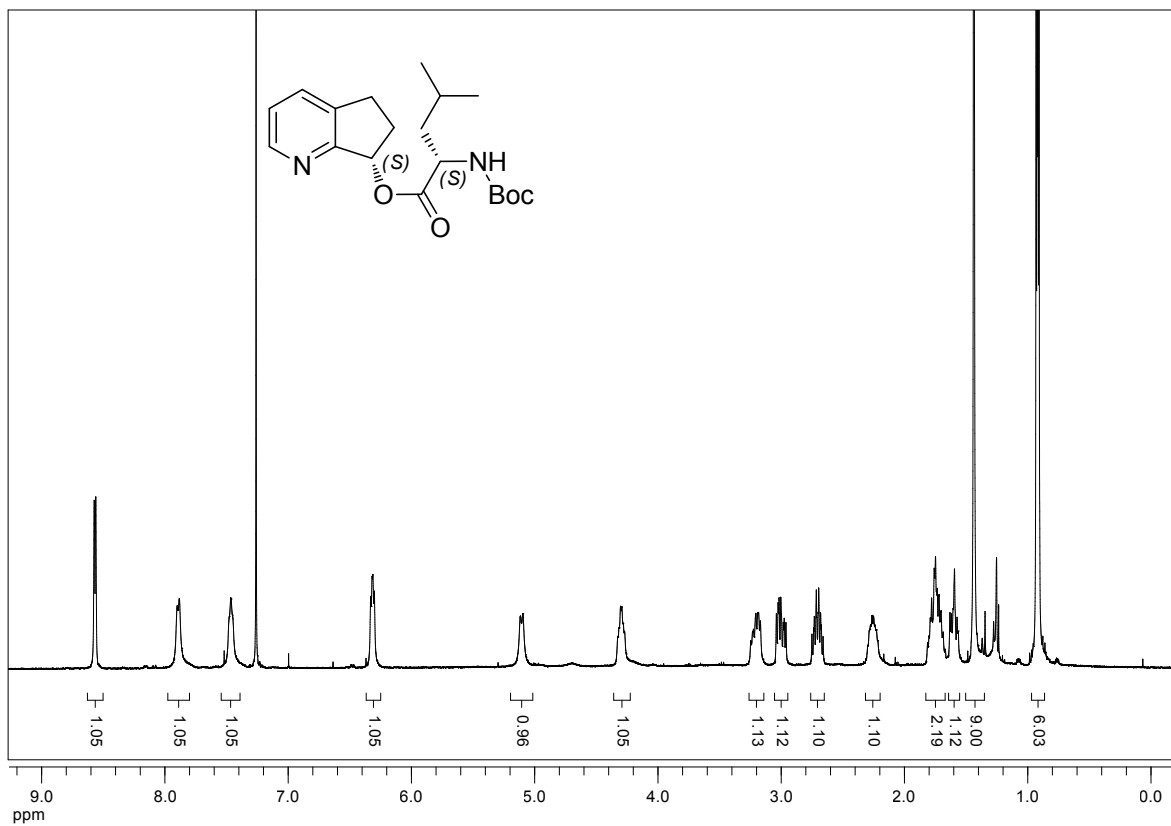


Figure 11. ¹H-NMR spectrum of compound 9.

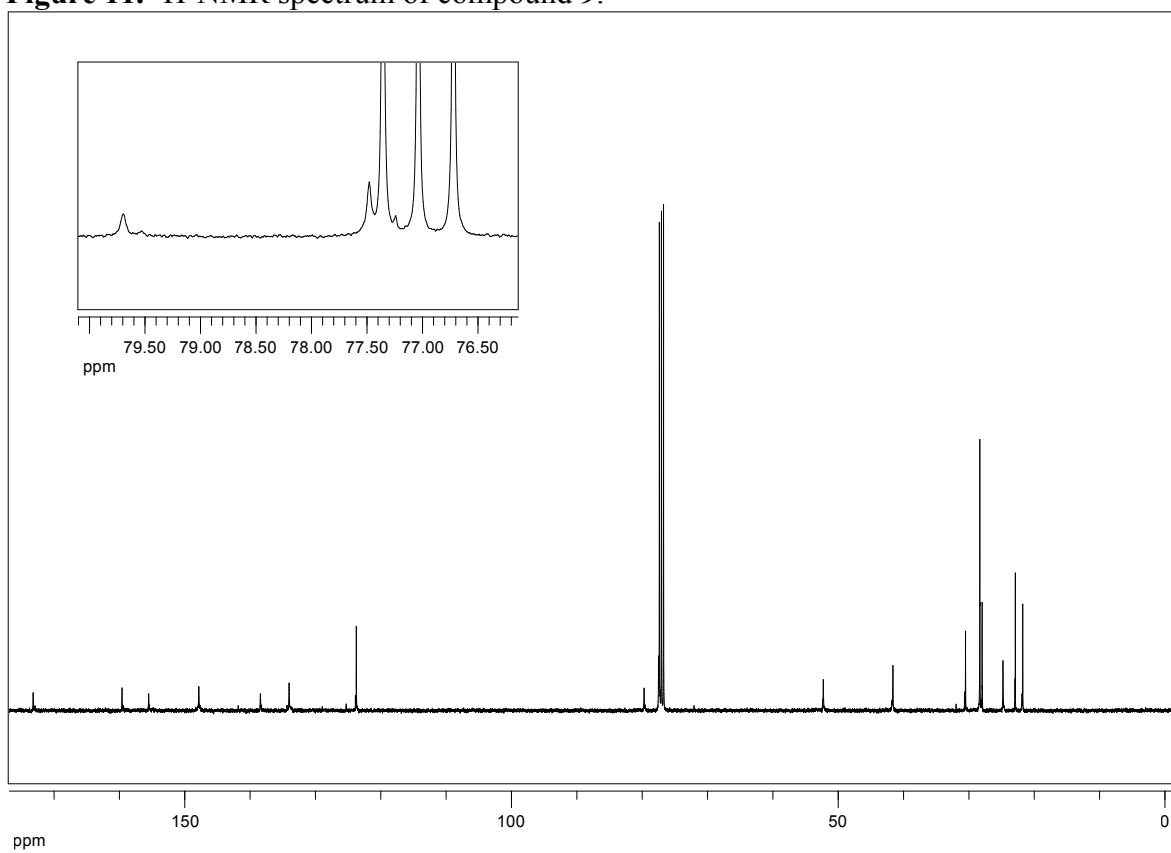


Figure 12. ¹³C-NMR spectrum of compound 9.

Table 1. Crystal data and structure refinement for **9**.

Identification code	9	
Empirical formula	C ₁₉ H ₂₈ N ₂ O ₄	
Formula weight	348.43	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2 ₁	
Unit cell dimensions	a = 11.1067(5) Å	α = 90°
	b = 8.7283(3) Å	β = 113.959(2)°
	c = 11.1168(5) Å	γ = 90°
Volume	984.83(7) Å ³	
Z	2	
Density (calculated)	1.175 Mg/m ³	
Absorption coefficient	0.082 mm ⁻¹	
F(000)	376	
Crystal size	0.800 x 0.540 x 0.170 mm ³	
Theta range for data collection	2.005 to 33.196°	
Index ranges	-17 ≤ h ≤ 16, -13 ≤ k ≤ 13, -16 ≤ l ≤ 17	
Reflections collected	46898	
Independent reflections	7403 [R(int) = 0.0377]	
Completeness to theta = 25.242°	100.0 %	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	7403 / 1 / 234	
Goodness-of-fit on F ²	1.025	
Final R indices [I > 2σ(I)]	R1 = 0.0386, wR2 = 0.0900	
R indices (all data)	R1 = 0.0464, wR2 = 0.0940	
Absolute structure parameter	0.2(2)	
Largest diff. peak and hole	0.297 and -0.224 e.Å ⁻³	

The crystallographic data for the structure **9** have been deposited at the Cambridge Crystallographic Data Center as supplementary publication number CCDC 1404442.

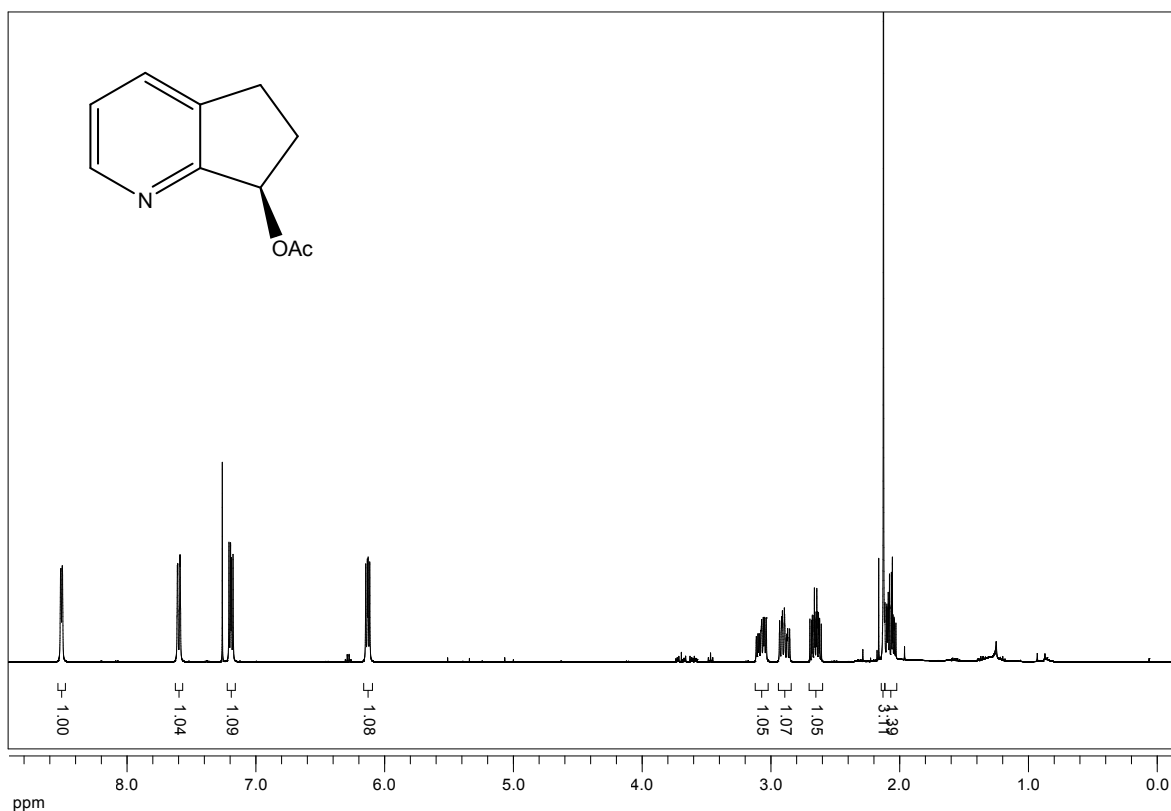


Figure 13. $^1\text{H-NMR}$ spectrum of compound (-)-6.

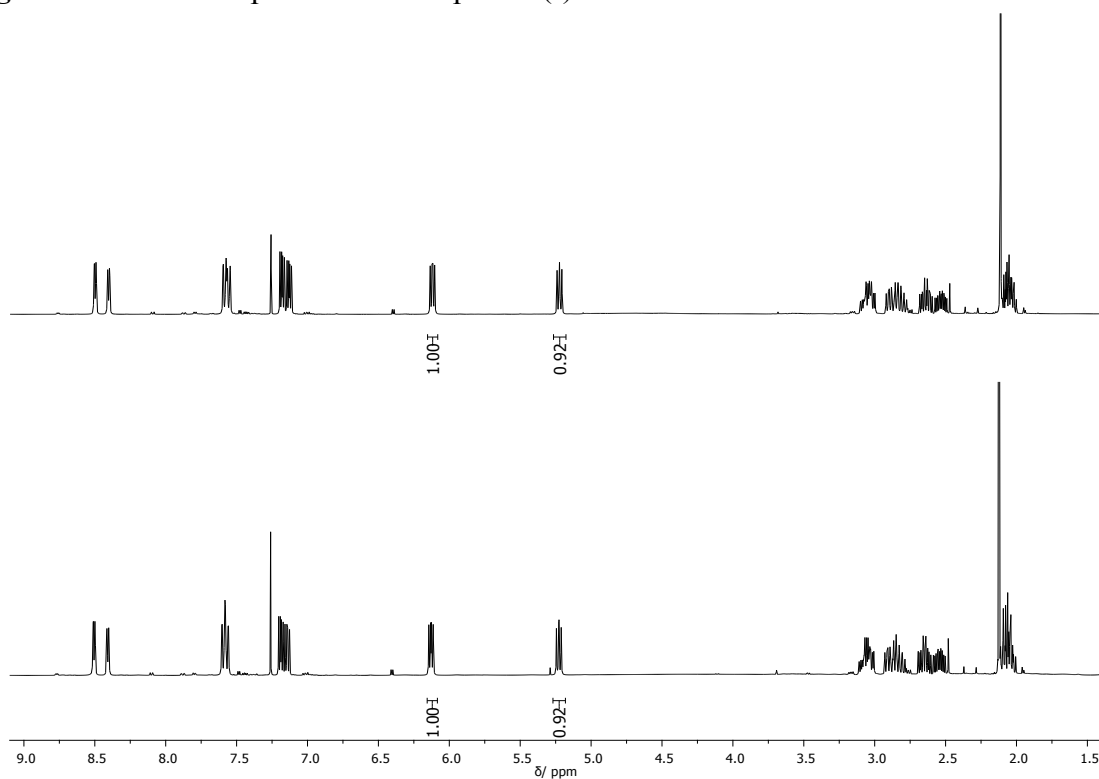


Figure 14. $^1\text{H-NMR}$ spectra of a filtered and dried sample from the reaction mixture of the enzymatic hydrolysis reaction after 18 h (above) and 48 h (bottom). The alcohol **3**/acetate **6** ratio found by spectra integration is 48:52.

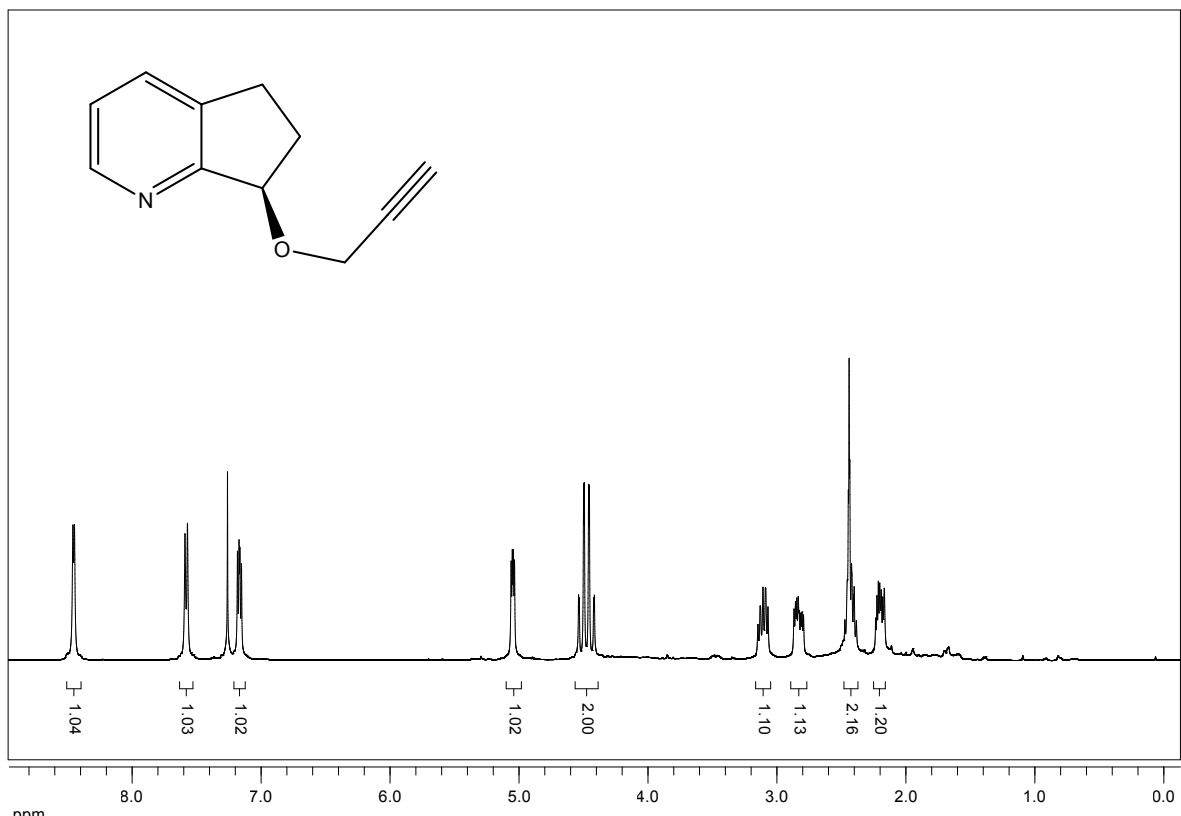


Figure 15. ¹H-NMR spectrum of compound (-)-2.

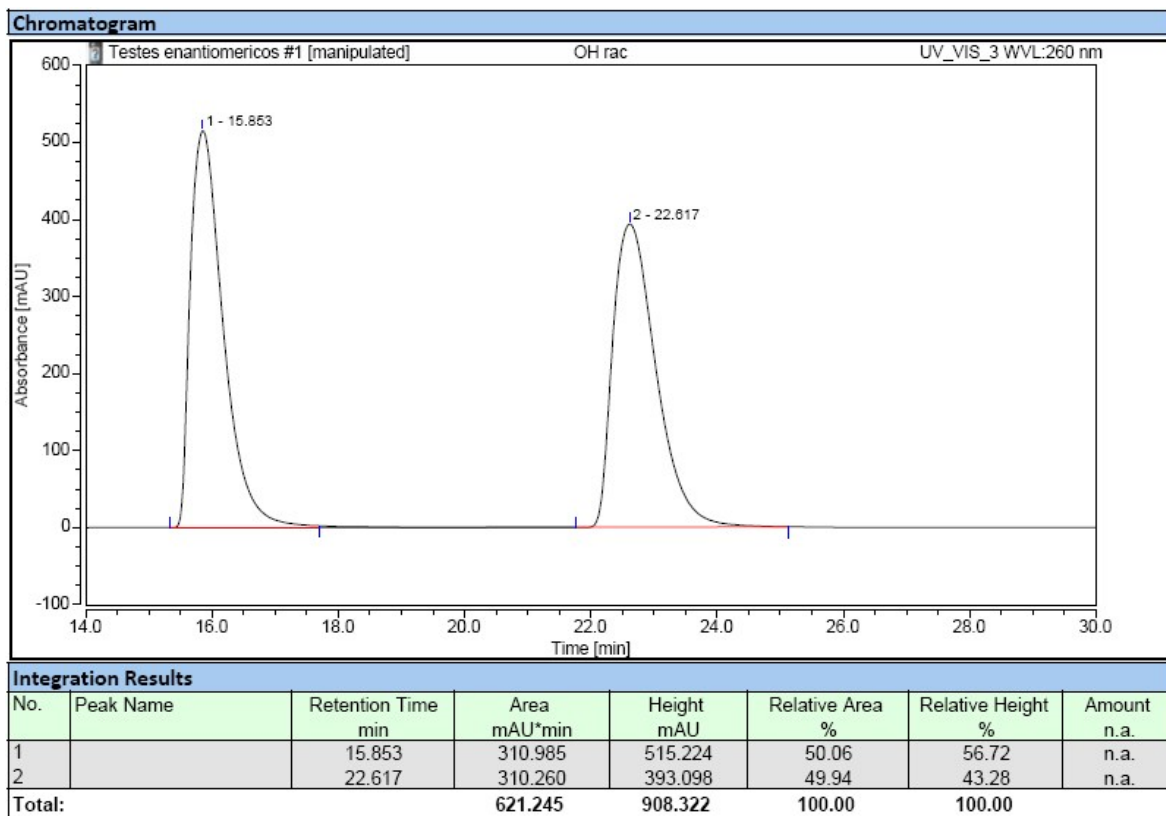


Figure 16. Chromatogram of racemic 3.

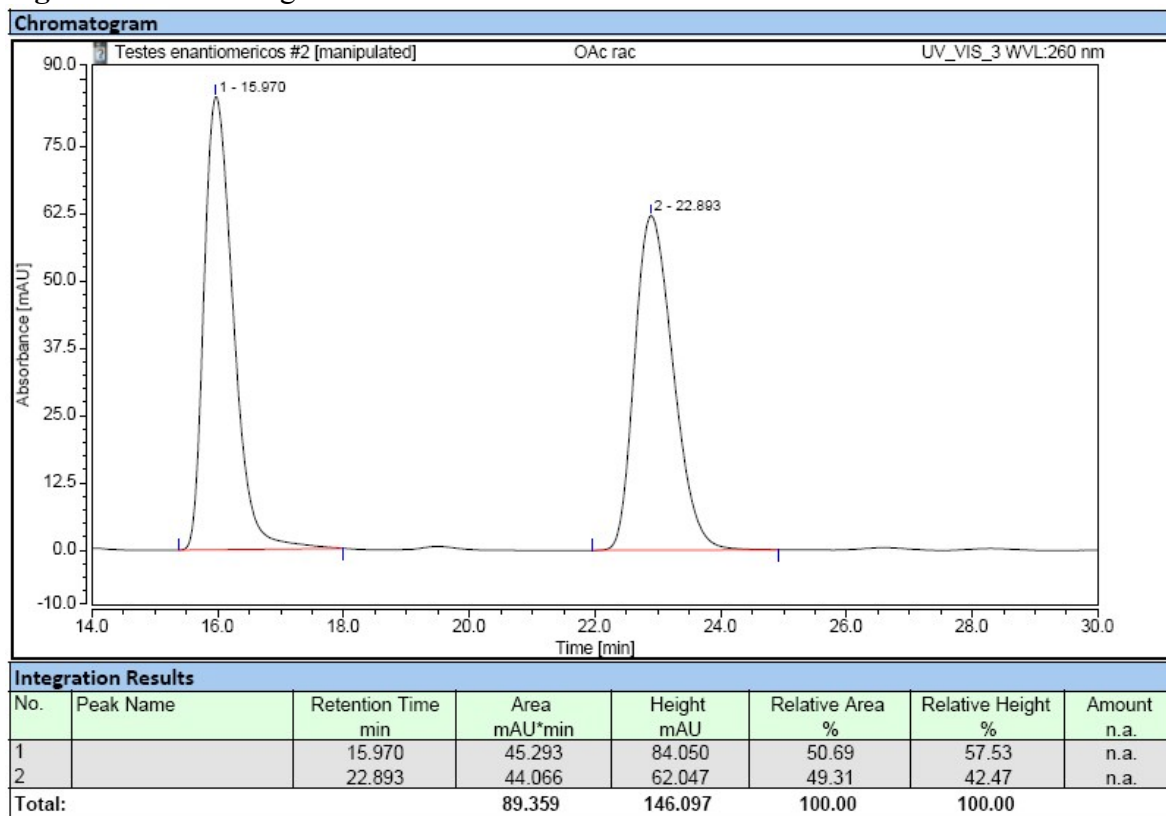


Figure 16. Chromatogram of racemic 6.

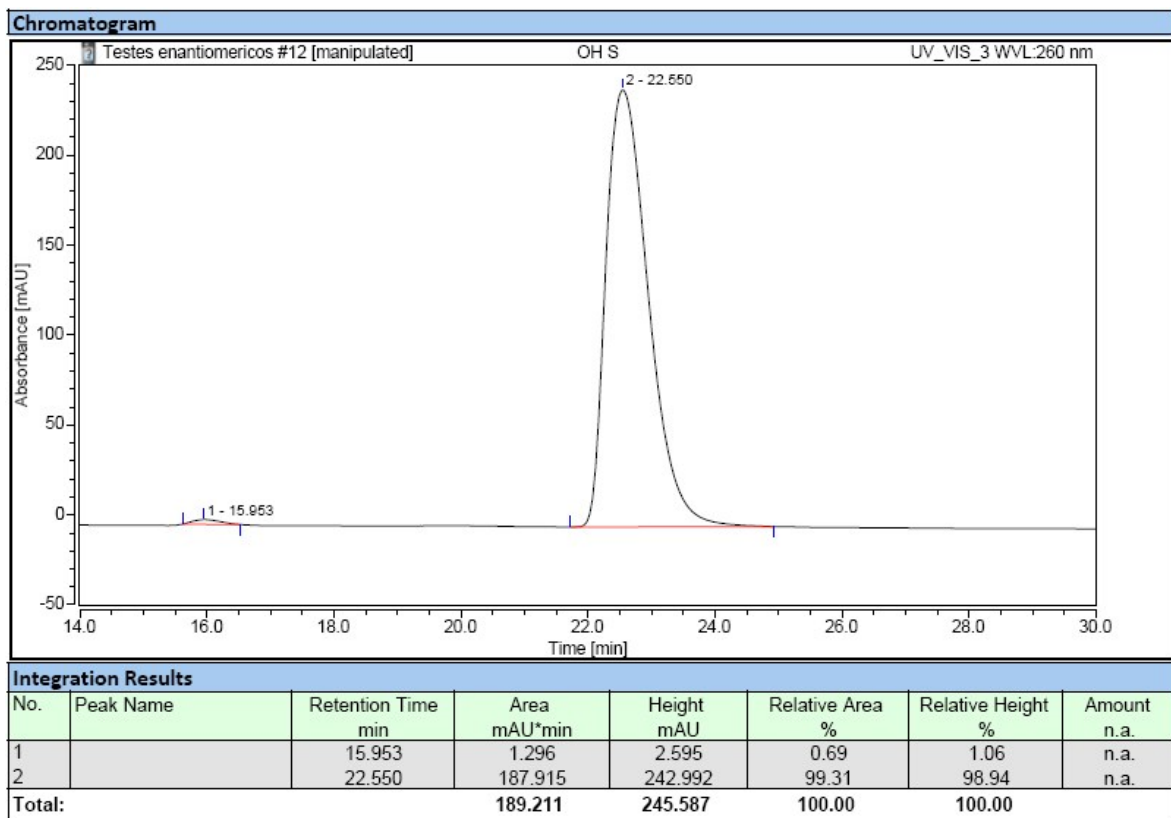


Figure 17. Chromatogram of (*S*)-3 obtained from chemical resolution.

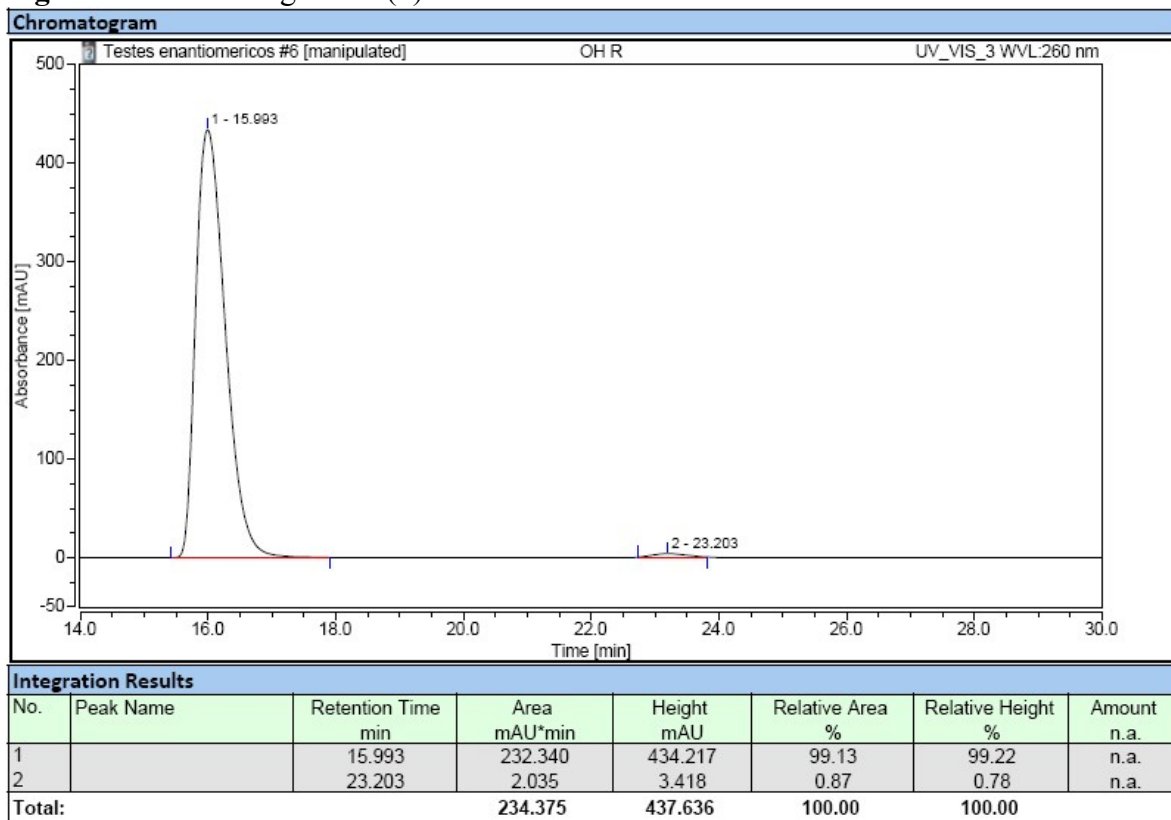


Figure 18. Chromatogram of (*R*)-3 obtained from enzymatic hydrolysis resolution.

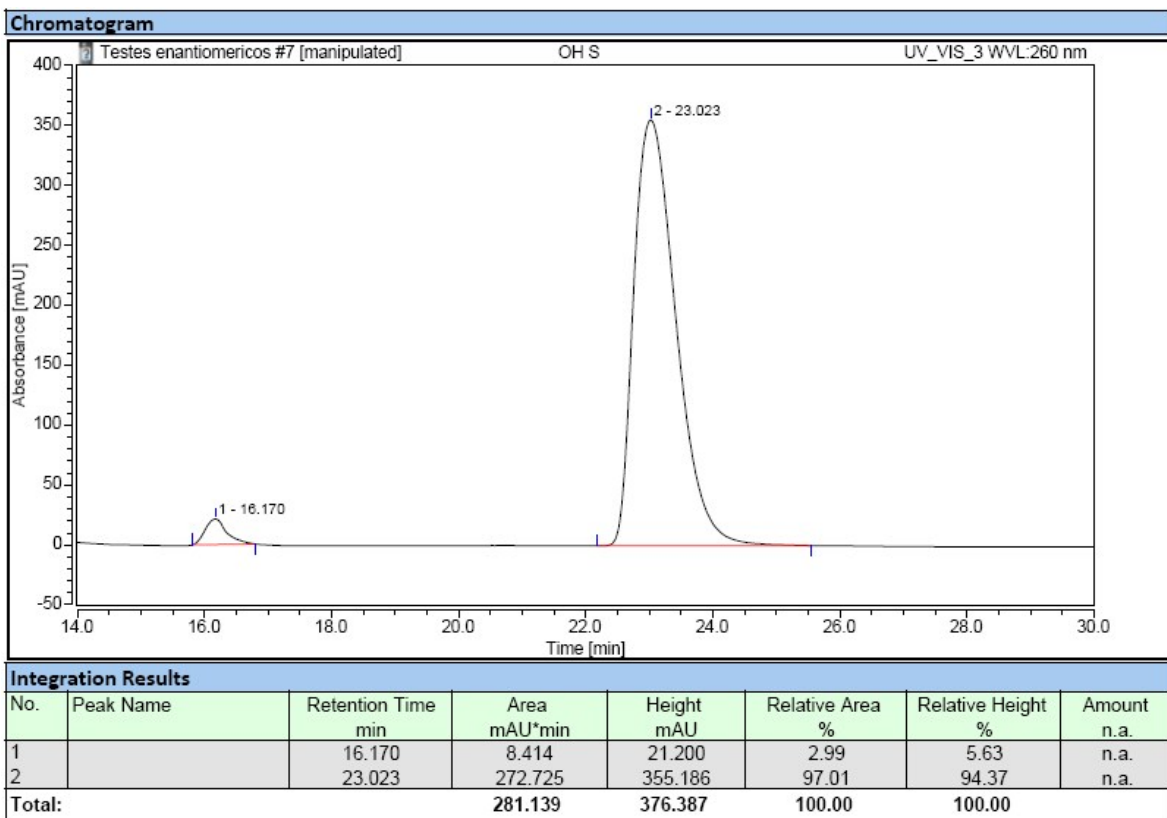


Figure 19. Chromatogram of (*S*)-3 obtained from enzymatic transesterification resolution.

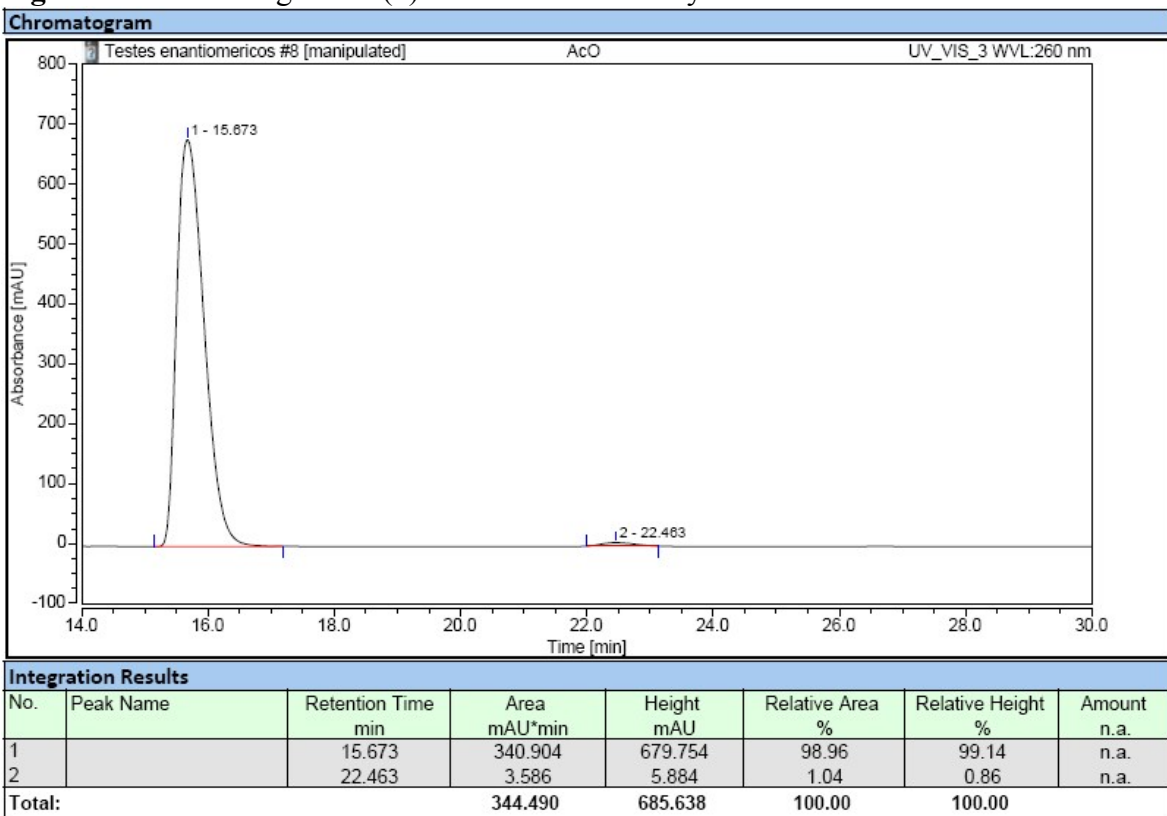


Figure 20. Chromatogram of (*R*)-6 obtained from enzymatic transesterification resolution.

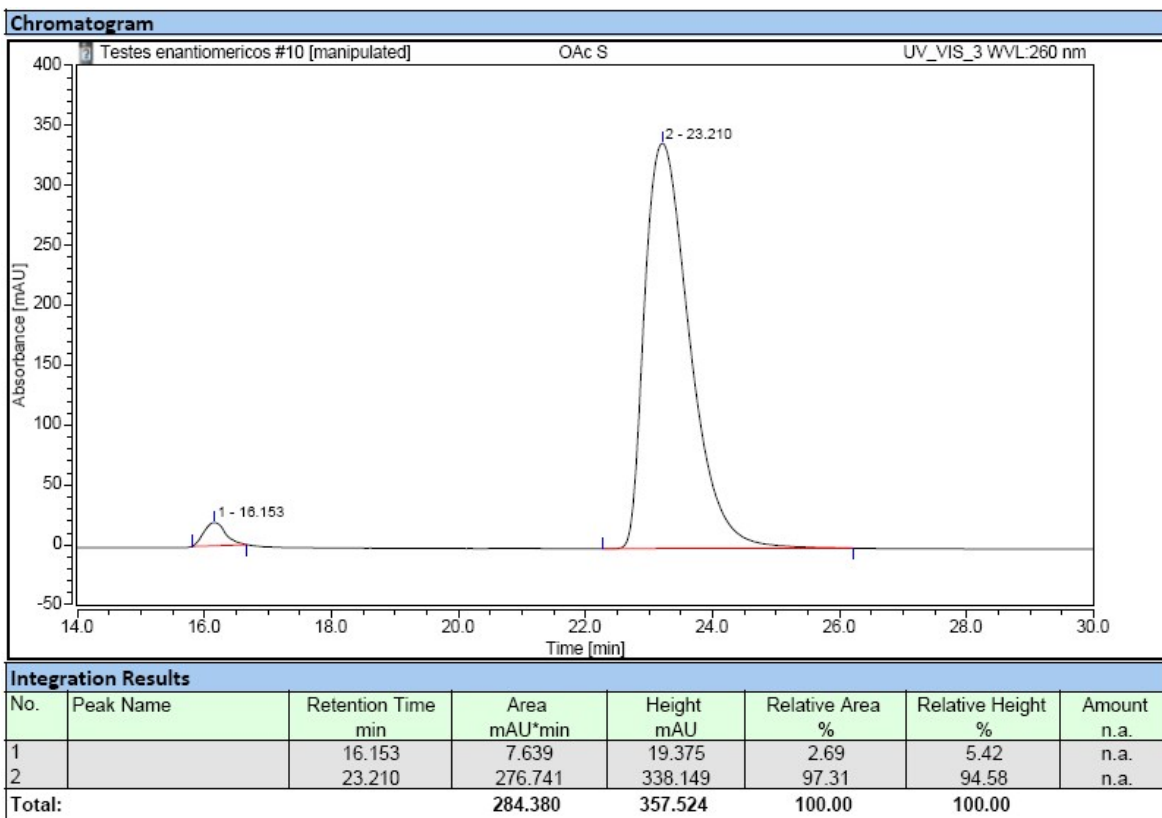


Figure 21. Chromatogram of (*S*)-**6** obtained from enzymatic hydrolysis resolution.