

Catalysis

An Alternative Enzymatic Route to the Ergogenic Ketone Body Ester (R)-3-Hydroxybutyl (R)-3-Hydroxybutyrate

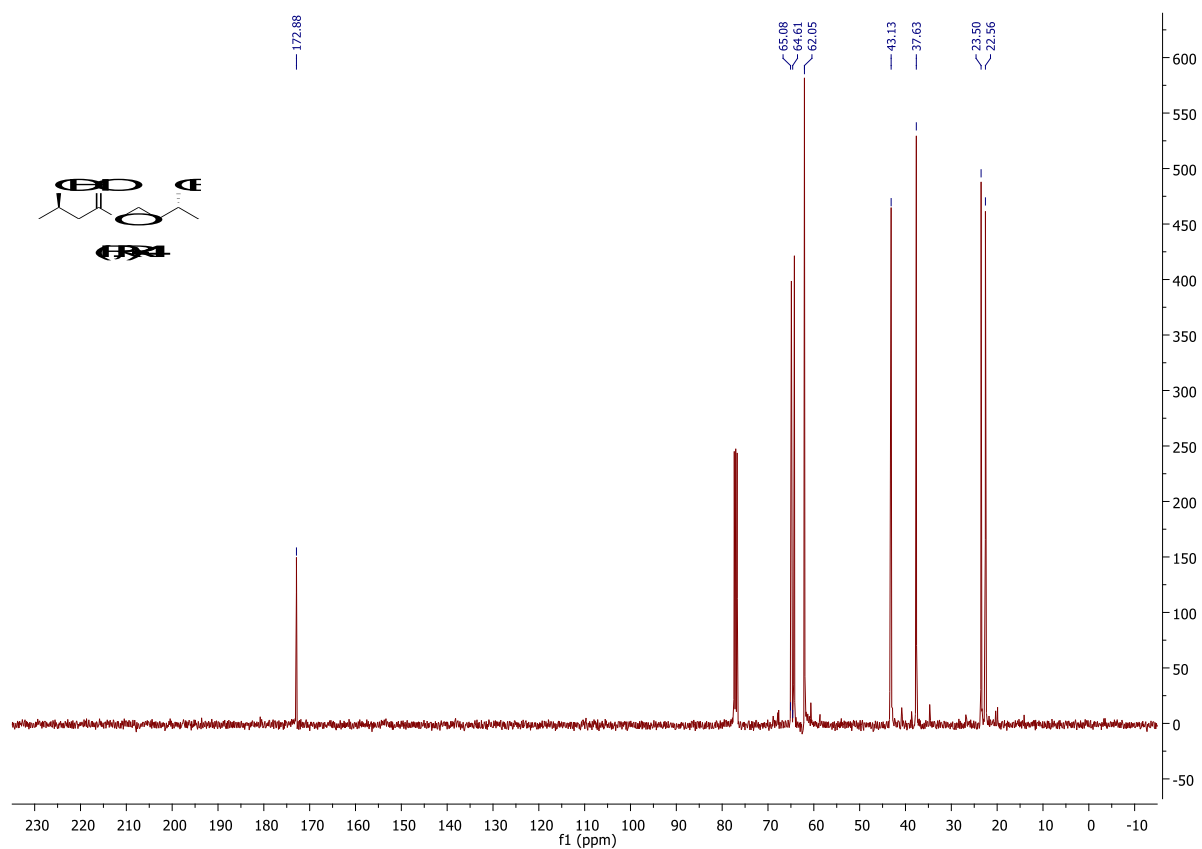
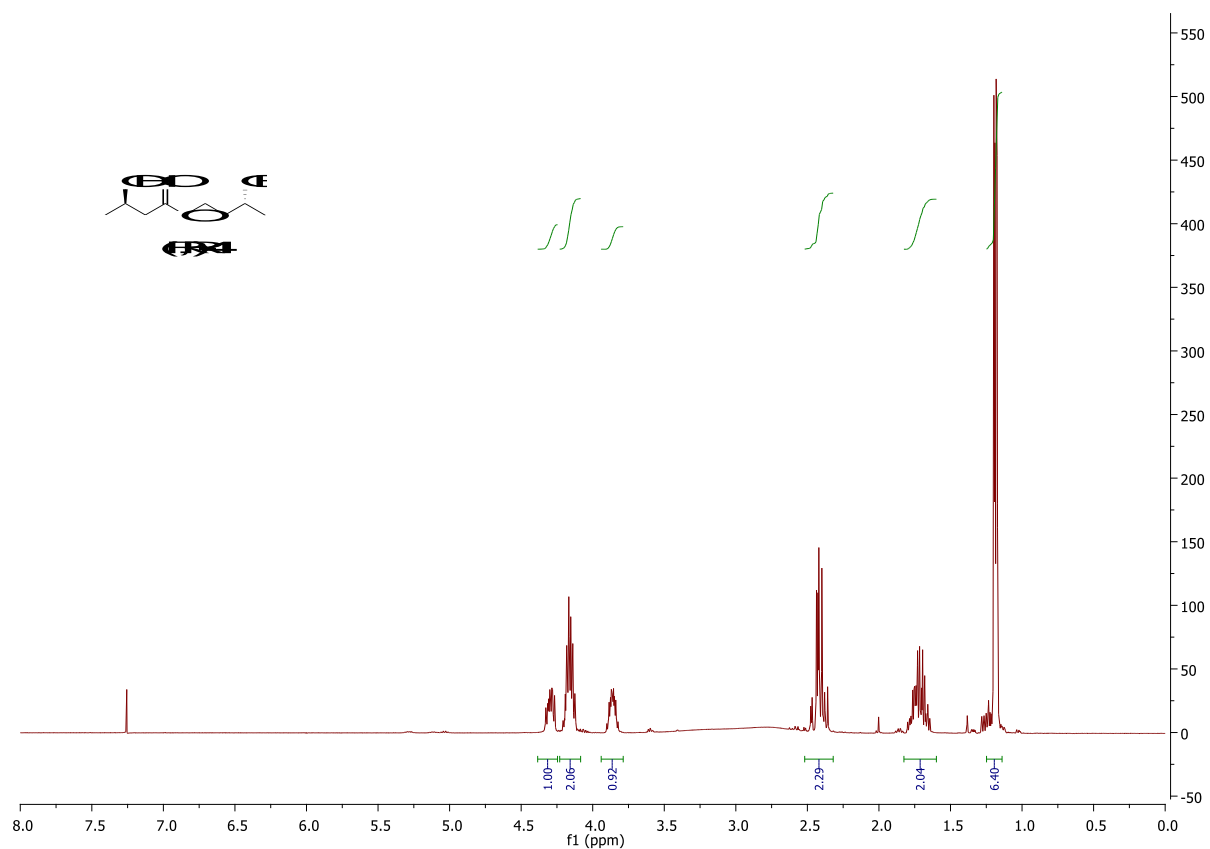
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Claudio Trapella1,..

Supplementary Materials

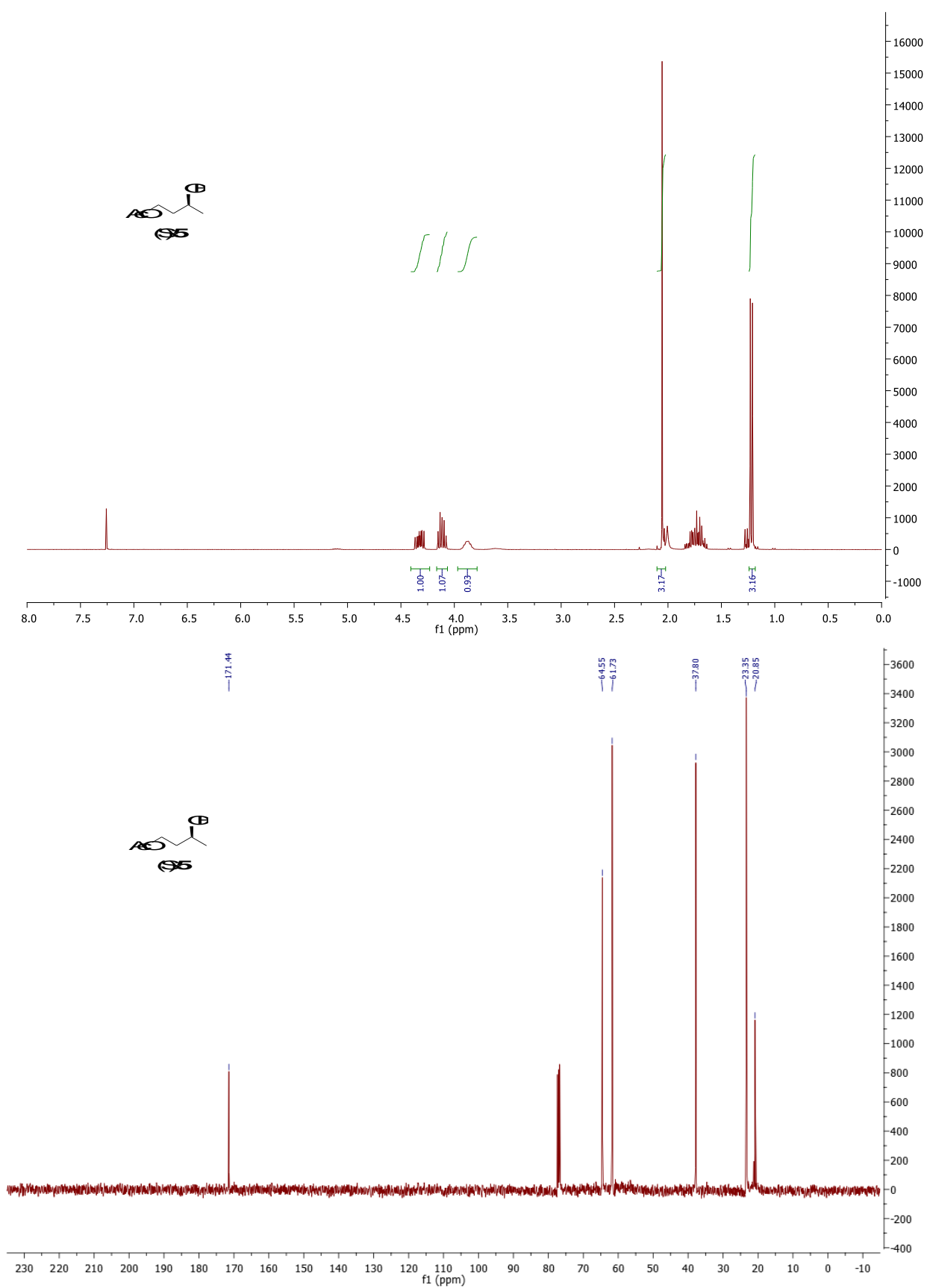
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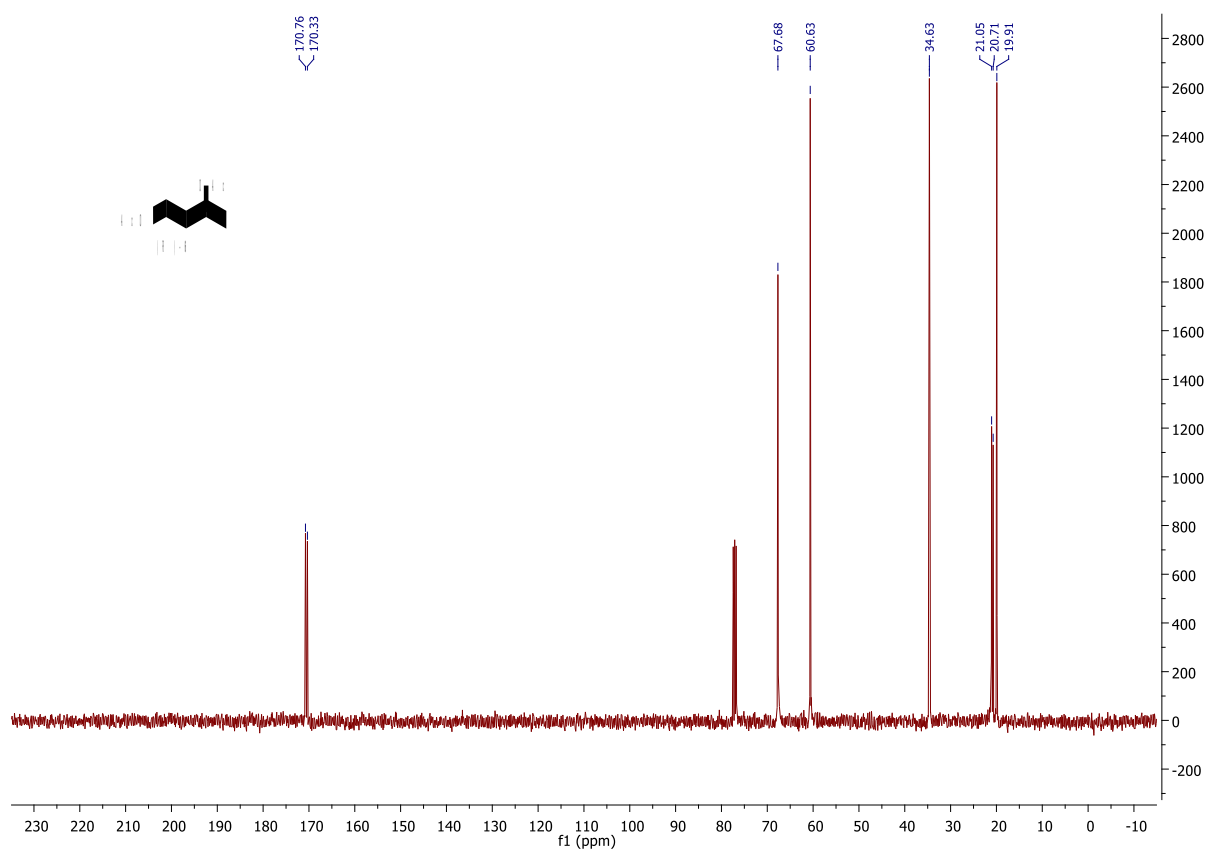
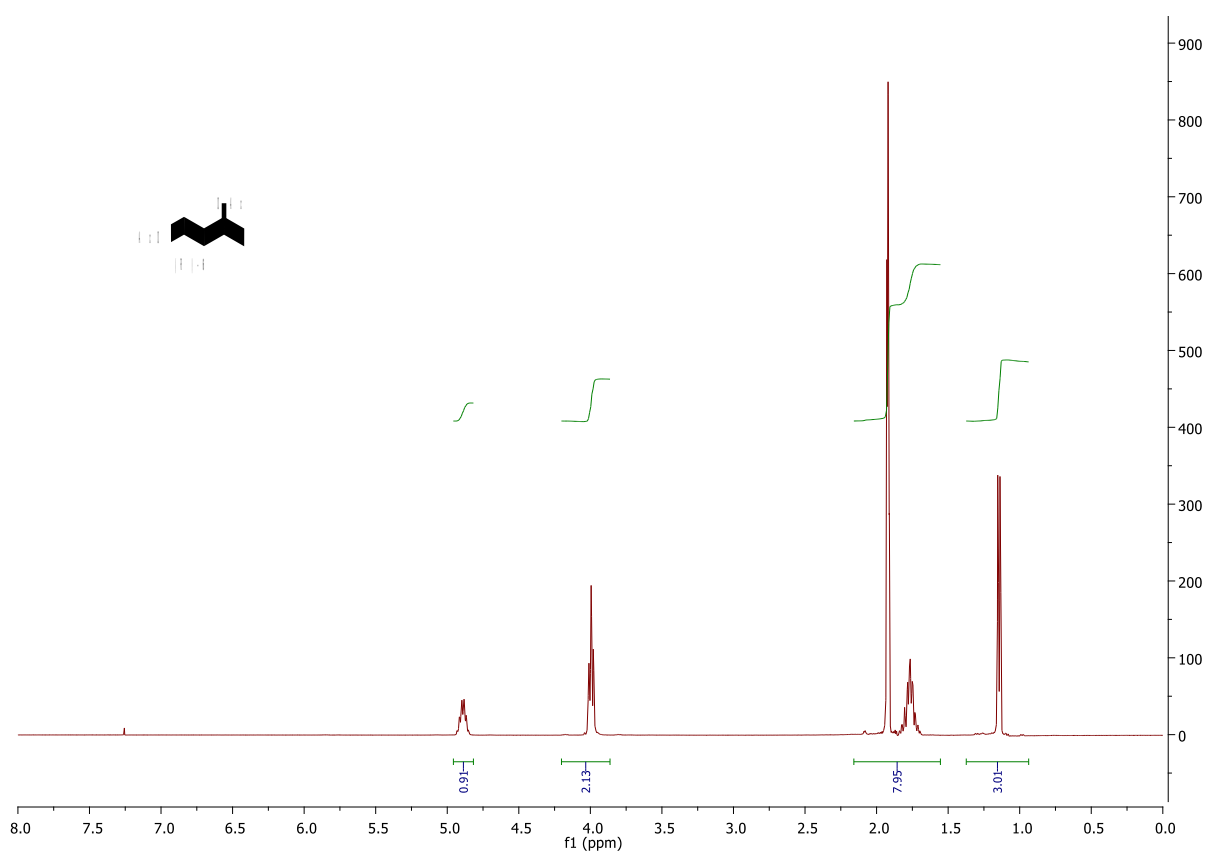
^1H - and ^{13}C -NMR spectra of compound 4



¹H- and ¹³C-NMR spectra of compound 5



^1H - and ^{13}C -NMR spectra of compound 6

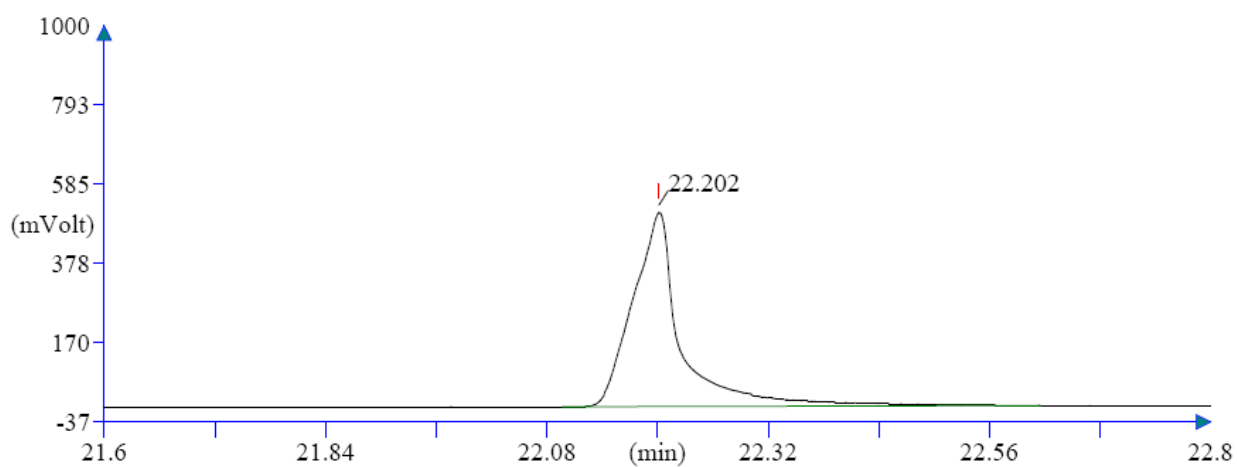


CC(C)[C@H](COc1ccc(C)cc1)C(=O)OC
(S)-**9**

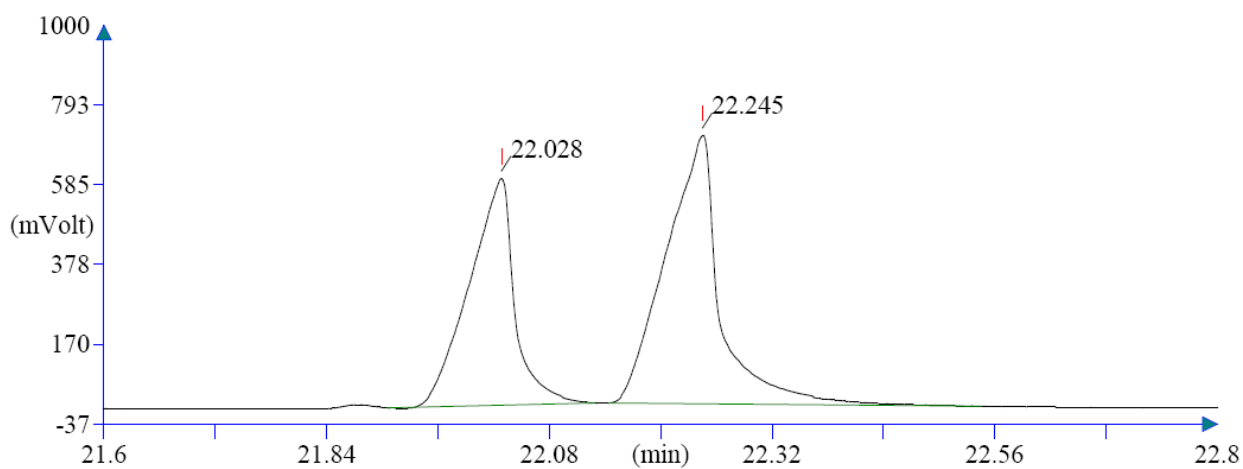
¹H NMR spectrum (CDCl₃) of (S)-**9**. The x-axis represents the chemical shift in ppm (f1), ranging from 0.0 to 8.0. The y-axis represents intensity, ranging from -100 to 1200. The spectrum shows several peaks corresponding to the structure of (S)-**9**. Key peaks include aromatic signals between 7.0 and 8.0 ppm, a methine signal around 4.7 ppm, a methoxy singlet around 3.8 ppm, a methylene doublet around 2.5 ppm, a large methine multiplet around 2.0 ppm, and a methyl singlet around 1.3 ppm. Integration values are provided below the peaks: 2.08, 2.18, 1.00, 1.16, 1.27, 3.56, 6.06, and 3.54. Green curly brackets indicate coupling patterns: doublets at 7.7 and 7.4, a doublet at 4.7, a doublet at 2.5, and a doublet at 1.3. A green line connects the peaks at 2.0 and 2.5.



Chiral phase GC of (R,R)-**4** and (R,R)/(R,S)-**4** mixture

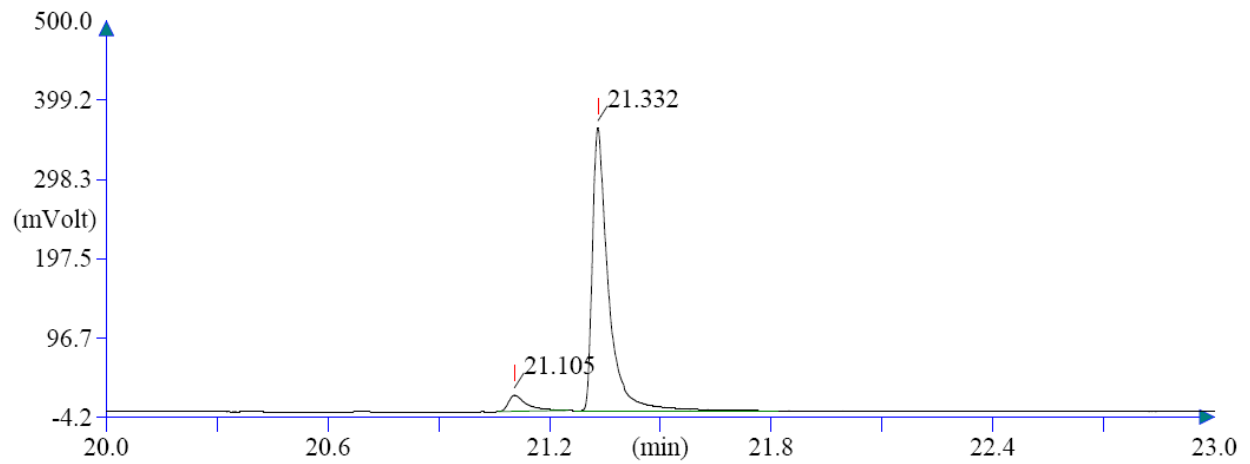


Retention Time (min)	Area (.1*uV*sec)	Area % (%)	Original Conc	Co
22.202	18626870	100.000	100.000	
	18626870		100.000	

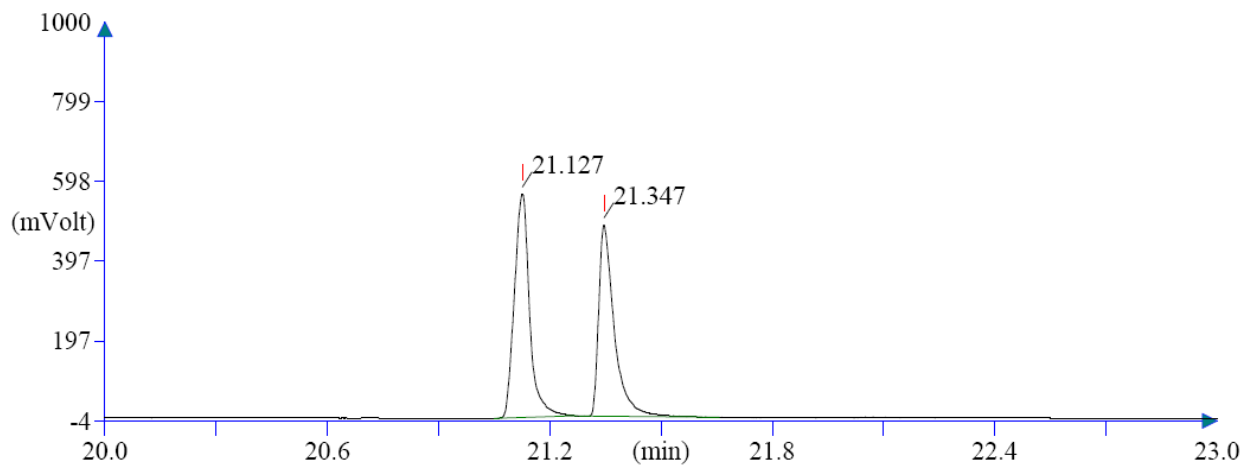


Retention Time (min)	Area (.1*uV*sec)	Area % (%)	Original Conc	Co
22.028	20120600	41.202	41.202	
22.245	28713300	58.798	58.798	
	48833900		100.000	

Chiral phase GC of acetylated (R)-2 from (S)-2 inversion

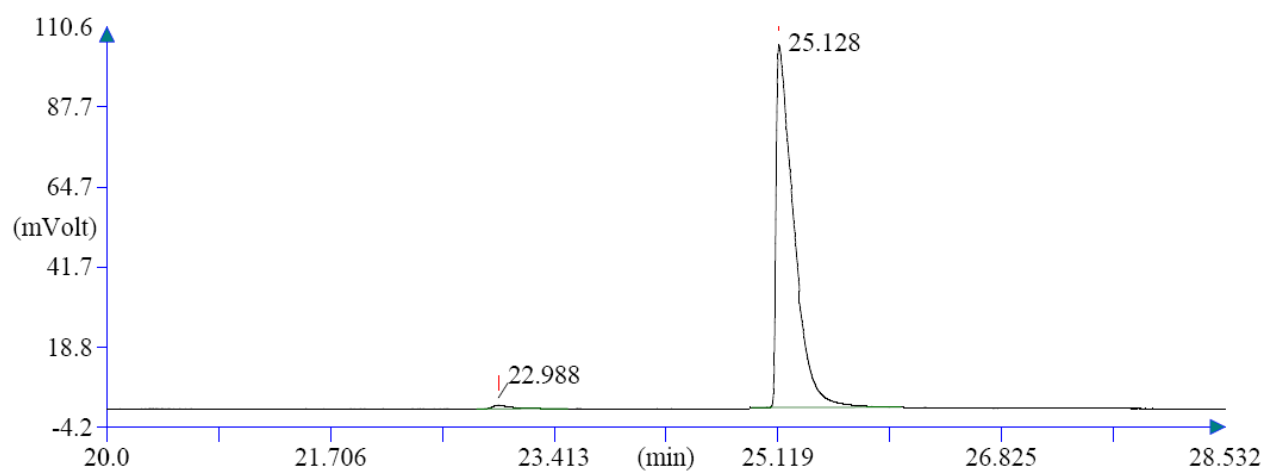


Retention Time (min)	Area (.1*uV*sec)	Area % (%)	Original Conc	Con
21.105	708121	6.096	6.096	
21.332	10908030	93.904	93.904	
	11616150		100.000	

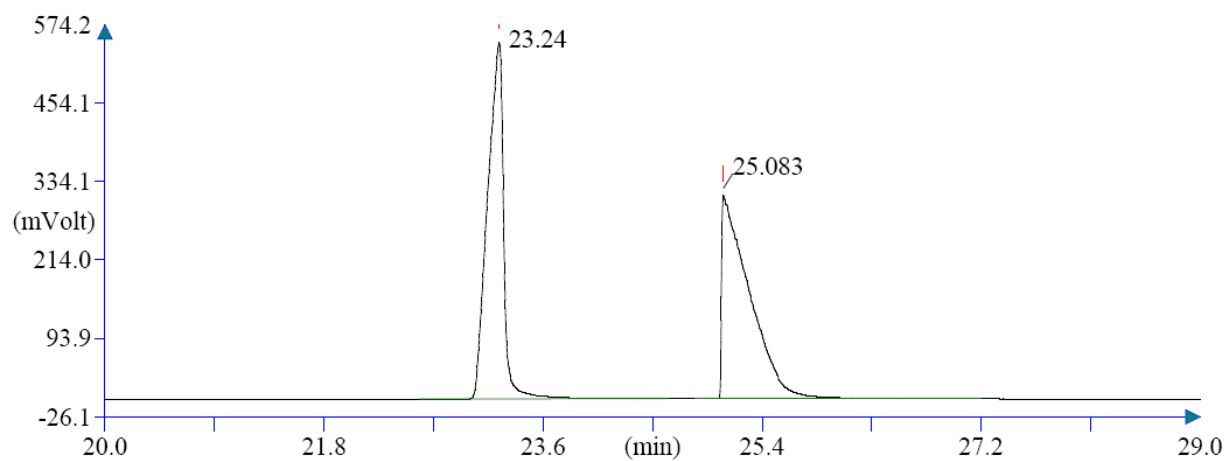


Retention Time (min)	Area (.1*uV*sec)	Area % (%)	Original Conc	Con
21.127	16122260	53.444	53.444	
21.347	14044540	46.556	46.556	
	30166800		100.000	

Chiral phase GC of acetylated (R)-3 from kinetic resolution rac-3

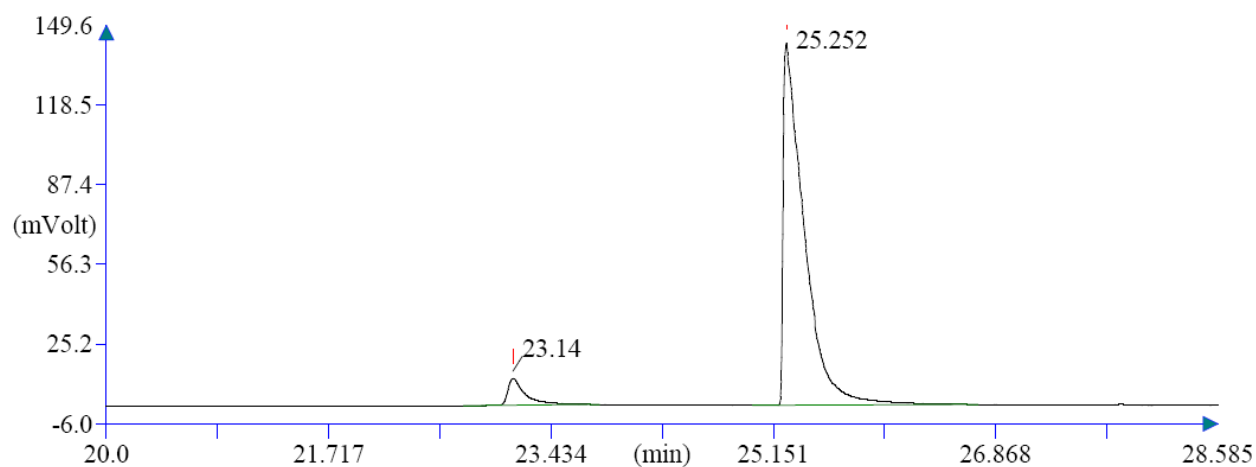


Retention Time (min)	Area (.1*uV*sec)	Area % (%)	Original Conc	Cor
22.988	108327	1.103	1.103	
25.128	9713536	98.897	98.897	
	9821863		100.000	

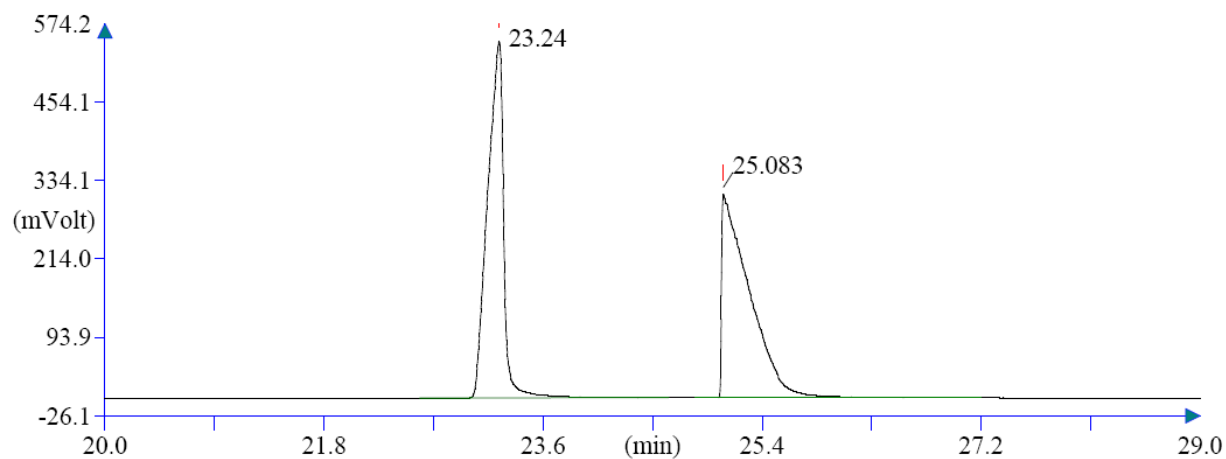


Retention Time (min)	Area (.1*uV*sec)	Area % (%)	Original Conc	Con
23.240	50110000	50.339	50.339	
25.083	49436020	49.661	49.661	
	99546020		100.000	

Chiral phase GC of (R)-6 from inversion of (S)-5



Retention Time (min)	Area (.1*uV*sec)	Area % (%)	Original Conc	Con
23.140	1013249	5.989	5.989	
25.252	15906180	94.011	94.011	
	16919430		100.000	



Retention Time (min)	Area (.1*uV*sec)	Area % (%)	Original Conc	Con
23.240	50110000	50.339	50.339	
25.083	49436020	49.661	49.661	
	99546020		100.000	