SUPPLEMENTARY INFORMATION:

Investigating the Reaction Mechanism and Organocatalytic Synthesis of α,α'-Dihydroxy Ketones

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Contents:

1. Formation of Mosher's esters.[1]

3,3,3-Trifluoro-2-methoxy-2-phenyl-propionic acid 3-hydroxy-2-oxo-3-p-tolyl-propyl ester (Mosher's ester of 3d). The reaction was carried out under anhydrous conditions. To a stirred solution of **3d** (0.010 g, 0.03 mmol) in CH_2Cl_2 (1 mL) was added 2,4,6-collidine (33 µL, 0.25 mmol) and (*S*)-MTPA chloride (10 µL, 0.04 mmol), and the reaction was stirred for 12 h at rt. The product was dry loaded onto silica gel and purified using flash chromatography (EtOAc/hexane, 1:4) to afford the titled compound as a colorless oil (0.012 g, 55%), (2*R*,3'*R*) major isomer. R_f 0.50 (EtOAc/hexane, 1:4); ^{1}H NMR (600 MHz; $CDCl_3$) δ 2.35 (3H, s, C*H*$_3$), 3.61 (3H, s, OC*H*$_3$), 4.70 (0.61H, d, *J* 17.4 Hz, C*H*HO (2*R*,3'*R*)), 4.85 (0.39H, d, *J* 17.4 Hz, C*H*HO (2S,3'*R*)), 4.89 (0.39H, d, *J* 17.4 Hz, CH*H*O (2S,3'*R*)), 5.00 (0.61H, d, *J* 17.4 Hz, CH*H*O (2*R*, 3'*R*)), 5.21 (1H, s, C*H*OH), 7.21 (4H, m, ArH), 7.42 (3H, m, Ph), 7.63 (2H, m, ArH); ^{13}C NMR (150 MHz; $CDCl_3$) δ 21.4, 55.9, 77.9, 127.3 (signal overlap), 128.6, 130.1 (signal overlap), 133.8, 139.6, 168.3, 201.0; ^{19}F NMR (282 MHz; $CDCl_3$) δ -72.2; *m/z* (CI) 397 (MH^+, 10%), 379 (100), 145 (84); *m/z* (HR-CI) calcd for MH^+ $C_{20}H_{20}F_3O_5$ 397.12628, found 397.12642.

3,3,3-Trifluoro-2-methoxy-2-phenyl-propionic acid 3-(4-fluoro-phenyl)-3-hydroxy-2-oxo-propyl ester (Mosher's ester of 3e). The reaction was carried out under anhydrous conditions. To a stirred solution of **3e** (0.008 g, 0.04 mmol) in CH_2Cl_2 (1 mL) was added 2,4,6-collidine (33 µL, 0.25 mmol) and (*S*)-MTPA chloride (10 µL, 0.04 mmol) and the reaction was stirred for 12 h at rt. The product was dry loaded onto silica gel and purified using flash chromatography (EtOAc/hexane, 1:4) to afford the titled compound as a colorless oil (0.010 g, 59%), (2*R*,3'*R*) major isomer. R_f 0.50 (EtOAc/hexane, 1:4); ^{1}H NMR (600 MHz; $CDCl_3$) δ 3.61 (3H, s, OC*H*$_3$), 4.74 (0.63H, d, *J* 16.8 Hz, C*H*HO (2*R*,3'*R*)), 4.88 (0.74H, m, CH_2O (2*S*,3'*R*)), 4.99 (0.63H, d, *J* 16.8 Hz, CH*H*O (2*R*,3'*R*)), 5.24 (1H, d, *J* 3.0 Hz, C*H*OH), 7.09 (3H, m, ArH), 7.42 (4H, m, ArH), 7.58 (2H, m, ArH); ^{13}C NMR (150 MHz; $CDCl_3$) δ 55.9, 66.4, 77.4, 114.3 (d, $^2J_{CF}$ 21.1 Hz), 116.6 (d, $^2J_{CF}$ 21.1 Hz), 122.9, 127.5 (signal overlap), 128.6 (signal overlap), 130.0, 131.2, 131.7, 139.1, 163.3 (d, $^1J_{CF}$ 246 Hz, *C*F), 166.2, 201.4; ^{19}F NMR (282 MHz; $CDCl_3$) δ -72.2, -112.4; *m/z* (HR-CI) calcd for MH^+ $C_{19}H_{17}O_5F_4$ 401.10060, found 401.10051.

3,3,3-Trifluoro-2-methoxy-2-phenyl-propionic acid 3-(4-chloro-phenyl)-3-hydroxy-2-oxo-propyl ester (Mosher's ester of 3f). The reaction was carried out under anhydrous conditions. To a stirred solution of **3f** (0.007 g, 0.04 mmol) in CH_2Cl_2 (1 mL) was added 2,4,6-collidine (33 µL, 0.25 mmol) and (*S*)-MTPA chloride (10 µL, 0.04 mmol), and the reaction was stirred for 12 h at rt. The product was dry loaded onto silica gel and purified using flash chromatography (EtOAc/hexane, 1:4) to afford the titled compound as a colourless oil (0.009 g, 60%), (2*R*,3'*R*) major isomer. R_f 0.50 (EtOAc/hexane, 1:4); ν_{max}(neat)/cm^{-1} 3420, 2930, 2855, 1734; ^{1}H NMR (600 MHz; $CDCl_3$) δ 3.60 (3H, s, OC*H*$_3$), 4.74 (0.70H, d, *J* 17.1 Hz, C*H*HO (2*R*,3'*R*)), 4.88 (0.60H, m, CH_2O (2S,3'*R*)), 4.99

(0.70H, d, *J* 17.1 Hz, CH*H*O (2*R*,3'*R*)), 5.22 (1H, d, *J* 3.0 Hz, C*H*OH), 7.29–7.42 (7H, m, ArH), 7.52 (2H, m, ArH); ^{13}C NMR (150 MHz; $CDCl_3$) δ 55.9, 66.5, 84.7 (d, $^2J_{CF}$ 28.5 Hz), 123.1 (d, $^1J_{CF}$ 285 Hz), 127.5, 128.6, 128.7, 129.7, 130.0, 131.7, 135.2, 135.6, 166.2, 201.4; ^{19}F NMR (282 MHz; $CDCl_3$) δ -72.2; *m/z* (HR-ES-) calcd for $[M-H]^-$ $C_{19}H_{15}{}^{35}ClF_3O_5$ 415.0560, found 415.0506.

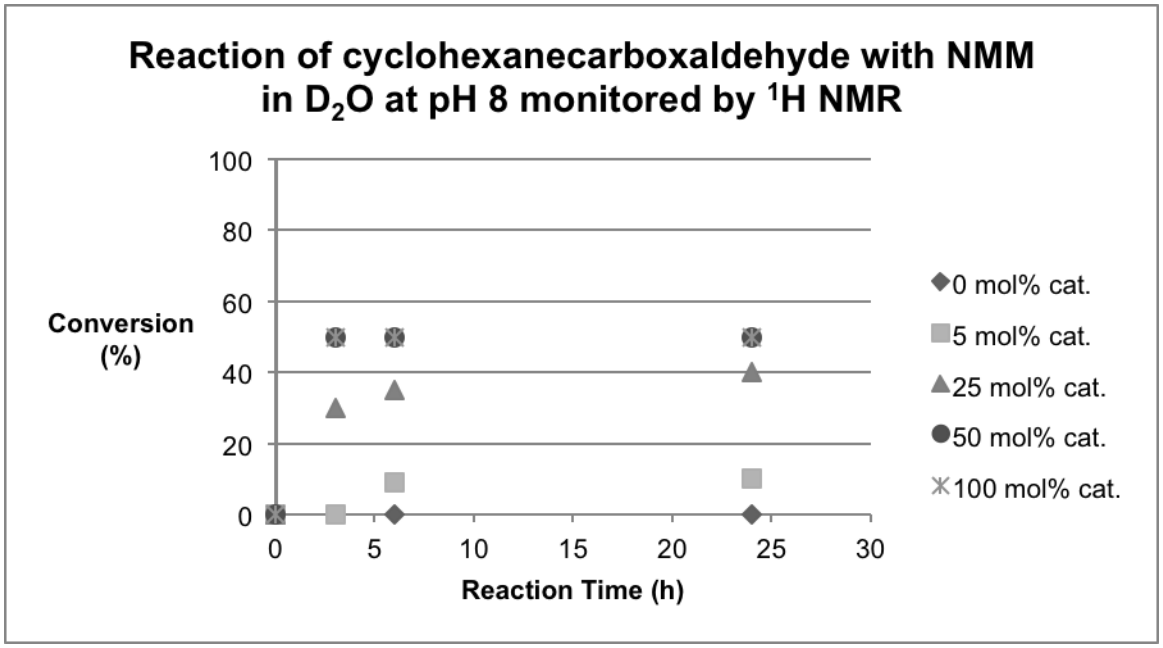
3,3,3-Trifluoro-2-methoxy-2-phenyl-propionic acid 3-(3-fluorophenyl)-3-hydroxy-2-oxo-propyl ester (Mosher's ester of 3i). The reaction was carried out under anhydrous conditions. To a stirred solution of **3i** (0.008 g, 0.04 mmol) in CH_2Cl_2 (1 mL) was added 2,4,6-collidine (33 µL, 0.25 mmol) and (*S*)-MTPA chloride (10 µL, 0.04 mmol) the reaction was stirred for 12 h at rt. The product was dry loaded onto silica gel and purified using flash chromatography (EtOAc/hexane, 1:4) to afford the titled compound as a colourless oil (0.010 g, 59%), and (2*R*,3'*R*) as the major isomer. R_f 0.50 (EtOAc/hexane, 1:4); ^{1}H NMR (600 MHz; $CDCl_3$) δ 3.61 (3H, s, OC*H*$_3$), 4.73 (0.63H, d, *J* 16.8 Hz, C*H*HO (2*R*,3'*R*)), 4.87 (0.75H, m, CH_2O (2S,3'*R*)), 4.99 (0.63H, d, *J* 16.8 Hz, CH*H*O (2*R*, 3'*R*)), 5.24 (1H, d, *J* 3.0 Hz, C*H*OH), 7.04–7.10 (2H, m, ArH), 7.29–7.43 (5H, m, ArH), 7.58 (2H, m, ArH); ^{13}C NMR (150 MHz; $CDCl_3$) δ 55.9, 66.4, 77.4, 114.2 (d, $^2J_{CF}$ 21.0 Hz), 116.5 (d, $^2J_{CF}$ 21.1 Hz), 127.5, 128.6 (signals superimposed), 130.0, 131.2, 131.7, 139.1, 163.3 (d, $^1J_{CF}$ 246 Hz, *C*F), 166.2, 201.4; ^{19}F NMR (282 MHz; $CDCl_3$) δ -72.2, -112.4; *m/z* (HR-ES+) calcd for MH^+ $C_{19}H_{17}F_4O_5$ 401.10060, found 401.10051.

3,3,3-Trifluoro-2-methoxy-2-phenyl-propionic acid 3-hydroxy-3-(3-methoxyphenyl)-2-oxo-propyl ester (Mosher's ester of 3k). The reaction was carried out under anhydrous conditions. To a stirred solution of **3k** (0.007 g, 0.04 mmol) in CH_2Cl_2 (1 mL) was added 2,4,6-collidine (33 µL, 0.25 mmol) and (*S*)-MTPA chloride (10 µL, 0.04 mmol) and the reaction was stirred for 12 h at rt. The product was dry loaded onto silica gel and purified using flash chromatography (EtOAc/hexane, 1:4) to afford the titled compound as a colorless oil (0.010 g, 67%), (2*R*,3'*R*) as the major isomer. R_f 0.50 (EtOAc/hexane, 1:4); ^{1}H NMR (600 MHz; $CDCl_3$) δ 3.61 (3H, s, OC*H*$_3$), 3.81 (3H, s, OC*H*$_3$), 4.71 (0.64H, d, *J* 17.1 Hz, C*H*HO (2*R*,3'*R*)), 4.87 (0.32H, d, *J* 17.1 Hz, C*H*HO (2S,3'*R*)), 4.92 (0.32H, d, *J* 17.1 Hz, C*H*HO (2S,3'*R*)), 5.03 (0.64H, d, *J* 17.1 Hz, CH*H*O (2*R*,3'*R*)), 5.22 (1H, s, C*H*OH), 6.86–6.92 (3H, m, ArH), 7.31 (1H, app. t, *J* 7.8 Hz, ArH), 7.41 (3H, m, ArH), 7.59 (2H, m, ArH); ^{13}C NMR (150 MHz; $CDCl_3$) δ 55.5, 55.9, 66.5, 77.6, 112.6, 115.2, 119.6, 127.5, 128.6 (signal overlap), 130.0, 130.6, 138.2, 160.4, 166.3, 201.6; ^{19}F NMR (282 MHz; $CDCl_3$) δ -72.2; *m/z* (HR-ES-) calcd for $[M-H]^-$ $C_{20}H_{18}F_3O_6$ 411.1056, found 411.1036.

3-[1-Hydroxy-2-oxo-3-(3,3,3-trifluoro-2-methoxy-2-phenyl-propionyloxy)-propyl]-benzoic acid methyl ester (Mosher's ester of 3l). The reaction was carried out under anhydrous conditions. To a stirred solution of **3l** (0.010 g, 0.04 mmol) in CH_2Cl_2 (1 mL) was added 2,4,6-collidine (33 µL, 0.25 mmol) and (*S*)-MTPA chloride (10 µL, 0.04 mmol) the reaction was stirred for 12 h at rt. The product was dry loaded onto silica gel and purified using flash chromatography (EtOAc/hexane,

1:4) to afford the titled compound as a colourless oil (0.013 g, 65%), (2*R*,3'*R*) as the major isomer. R_f 0.50 (EtOAc/hexane, 1:4); ^{1}H NMR (600 MHz; $CDCl_3$) δ 3.62 (3H, s, OC*H*$_3$), 3.80 (3H, s, OC*H*$_3$), 4.72 (0.83H, d, *J* 17.0 Hz, C*H*HO (2*R*,3'*R*)), 4.87 (0.17H, d, *J* 17.0 Hz, C*H*HO (2S,3'*R*)), 4.93 (0.17H, d, *J* 17.0 Hz, CH*H*O (2S,3'*R*)), 5.04 (0.83H, d, *J* 17.0 Hz, CH*H*O (2*R*,3'*R*)), 5.22 (1H, s, C*H*OH), 6.86–6.92 (3H, m, ArH), 7.31 (1H, app. t, *J* 7.8 Hz, ArH), 7.41 (3H, m, ArH), 7.59 (2H, m, ArH); ^{13}C NMR (150 MHz; $CDCl_3$) δ 55.5, 55.9, 66.5, 78.0, 112.6, 115.2, 119.6, 127.5, 127.6, 128.6 (signal overlap), 130.0, 130.6, 160.4, 166.3, 201.6; ^{19}F NMR (282 MHz; $CDCl_3$) δ -72.2; *m/z* (HR-ES+) calcd for MNa^+ $C_{21}H_{19}F_3O_7Na$ 463.0981, found 463.0975.

^{1}H NMR study of the reaction bewteen **1** and **2a** to give **3a** (%conversion, not isolated yield) at pH 8 using 0, 5, 25, 50, 100 mol% of NMM



Spectral Data

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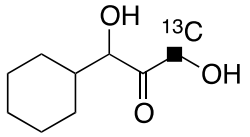
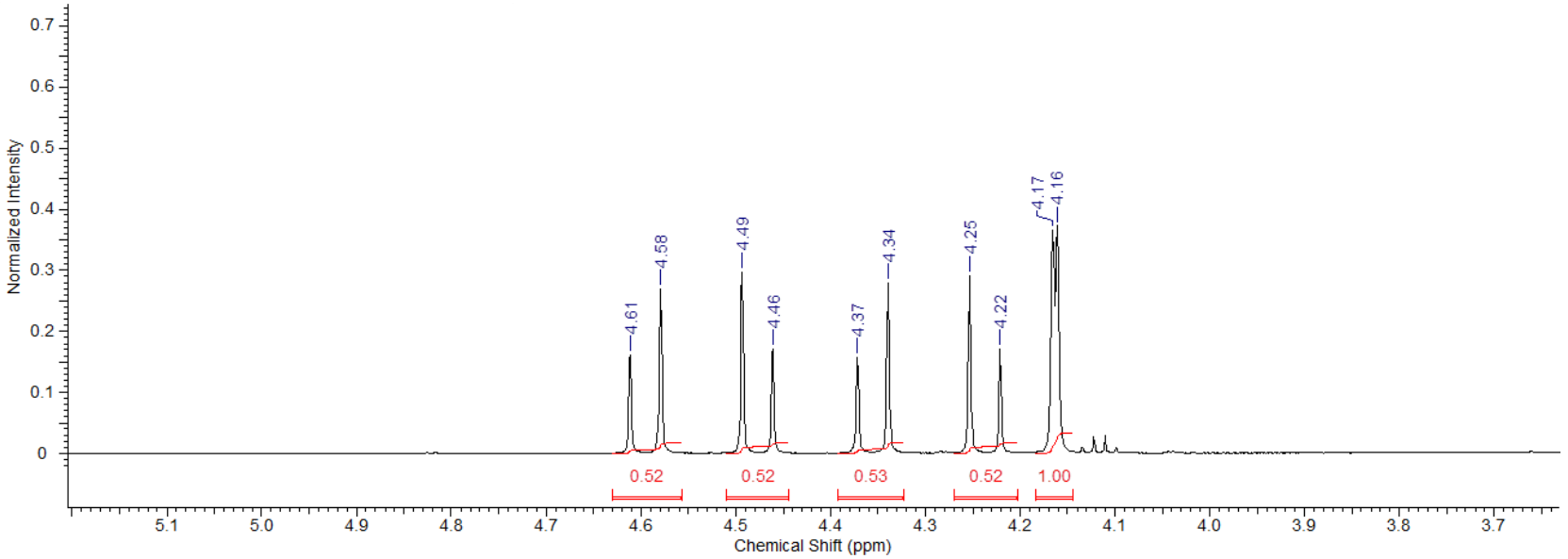
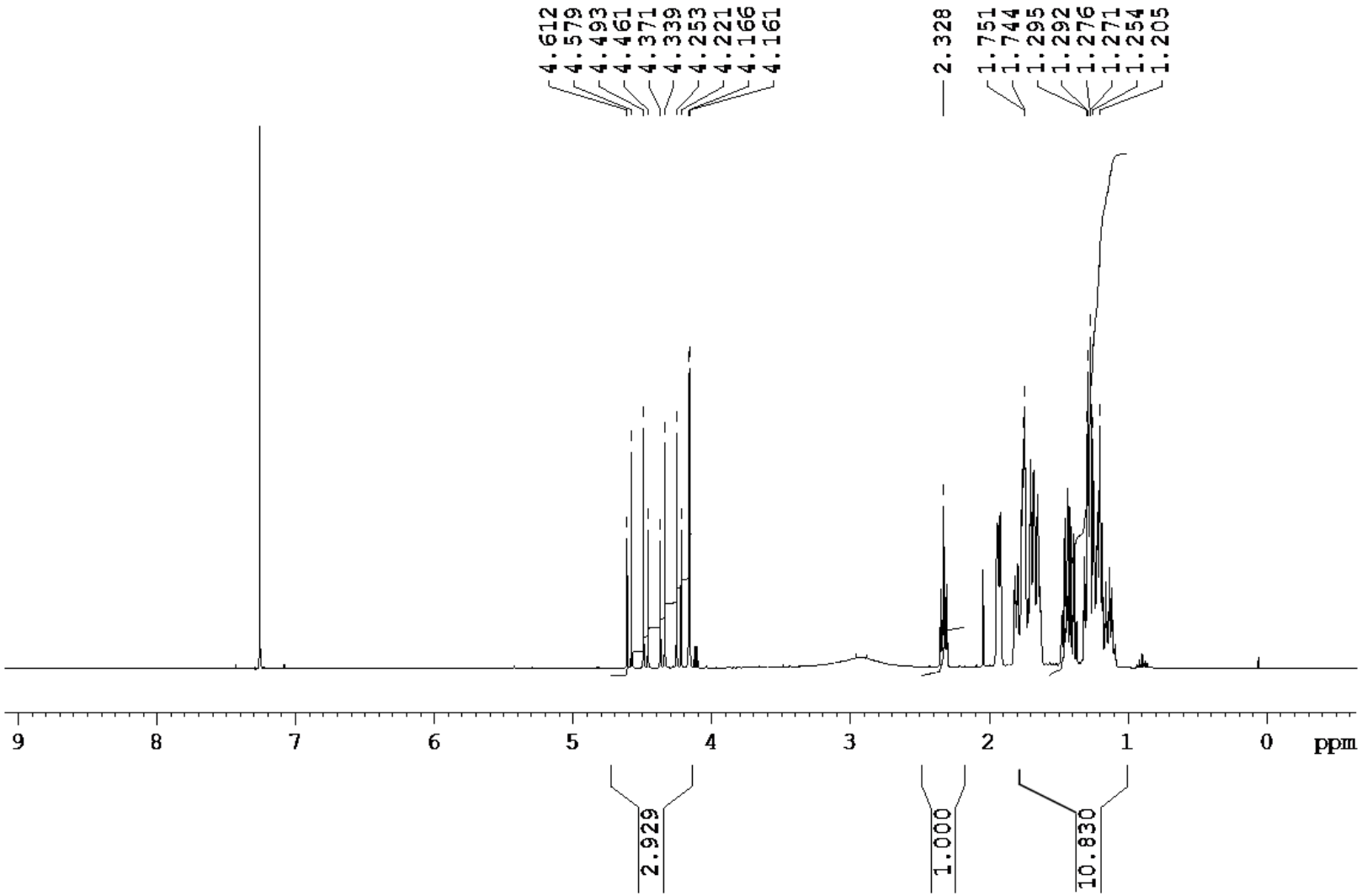
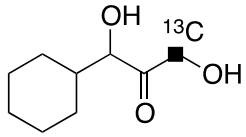
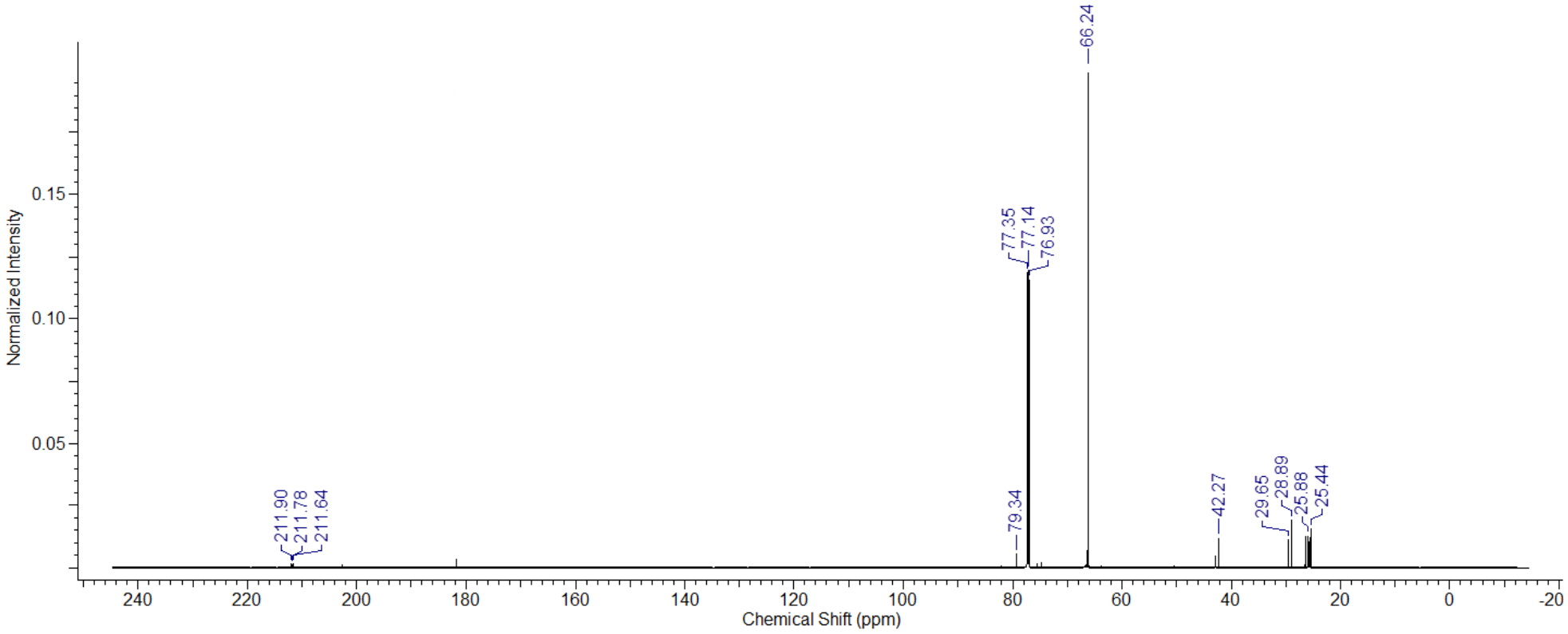




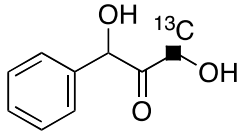


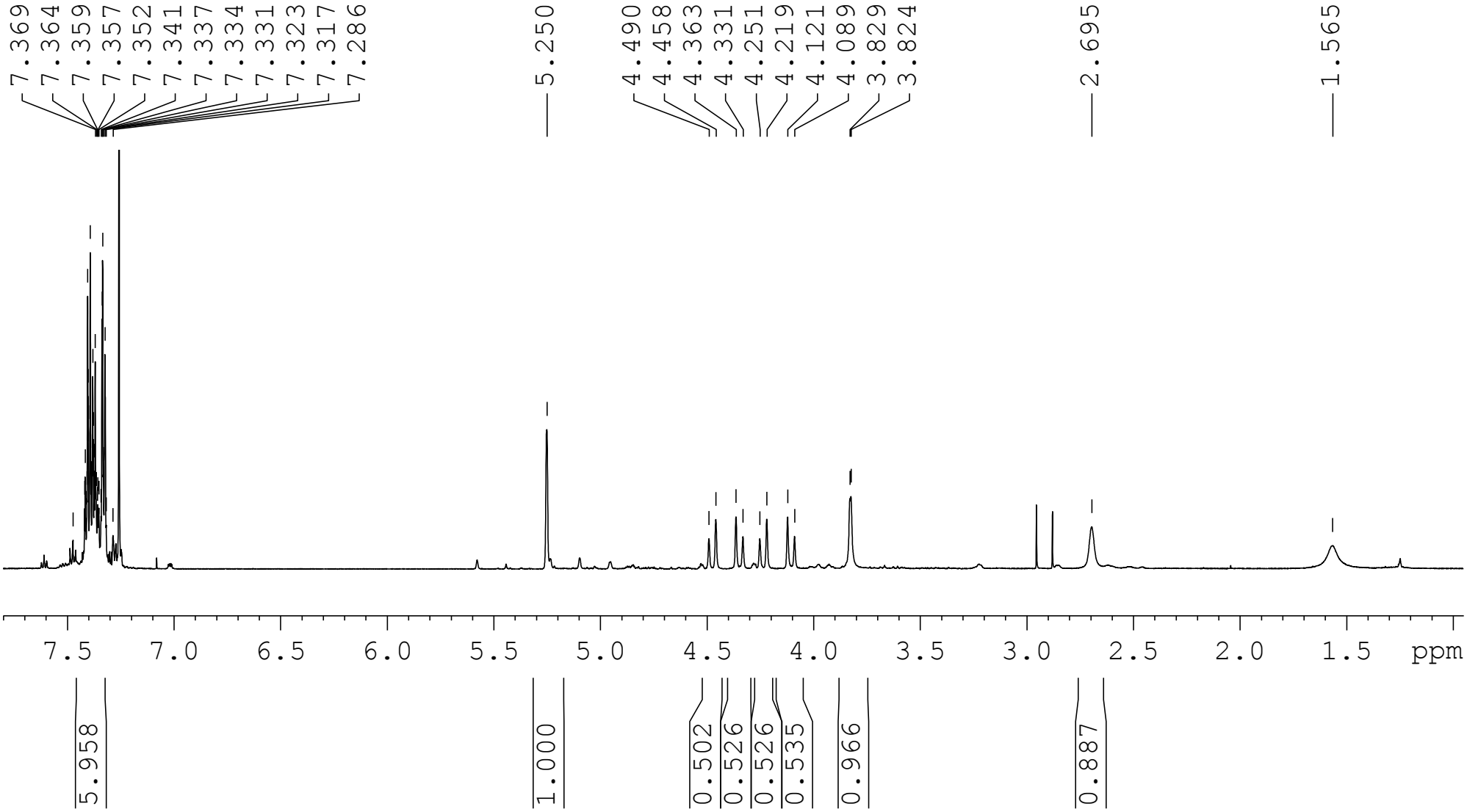
^{13}C NMR of [1-^{13}C]-cyclohexyl-1,3-dihydroxy-2-propanone [1-^{13}C]-3a



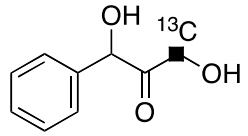


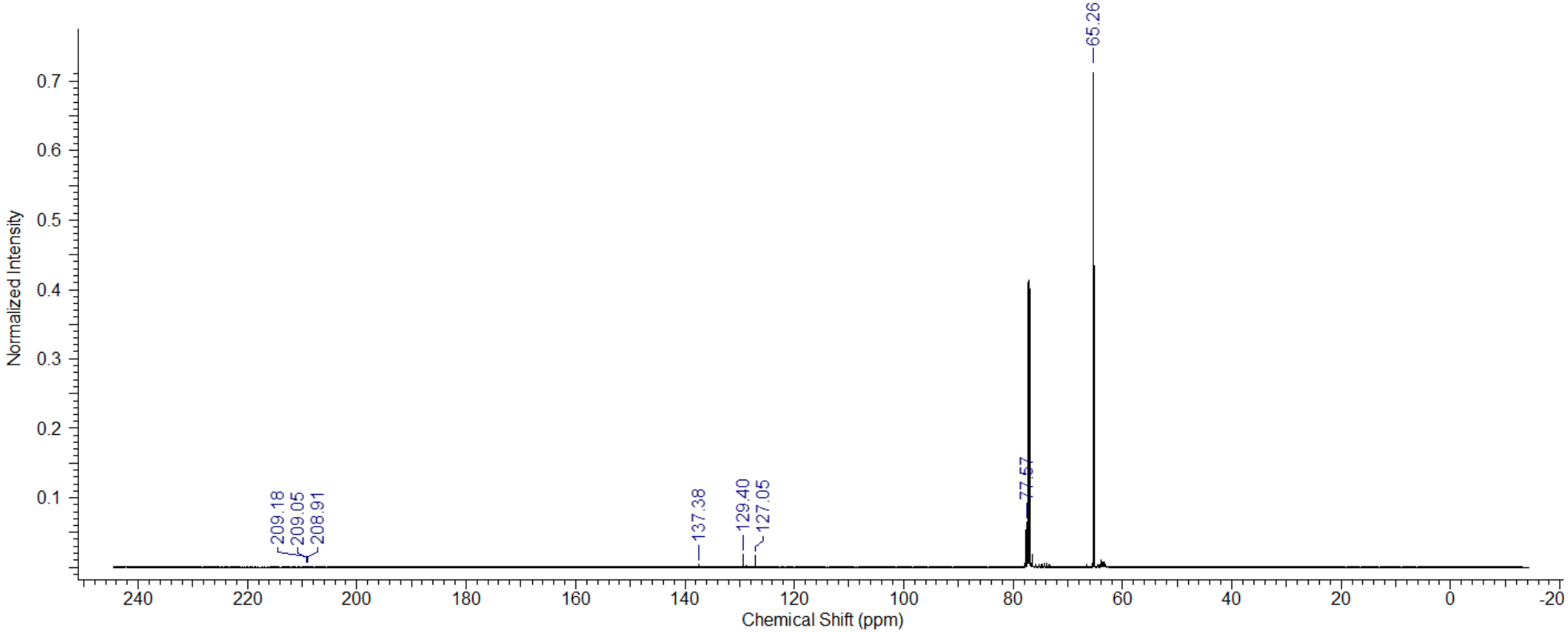
^{1}H NMR of [1-^{13}C]-3-dihydroxy-1-phenyl-2-propanone [1-^{13}C]-3b



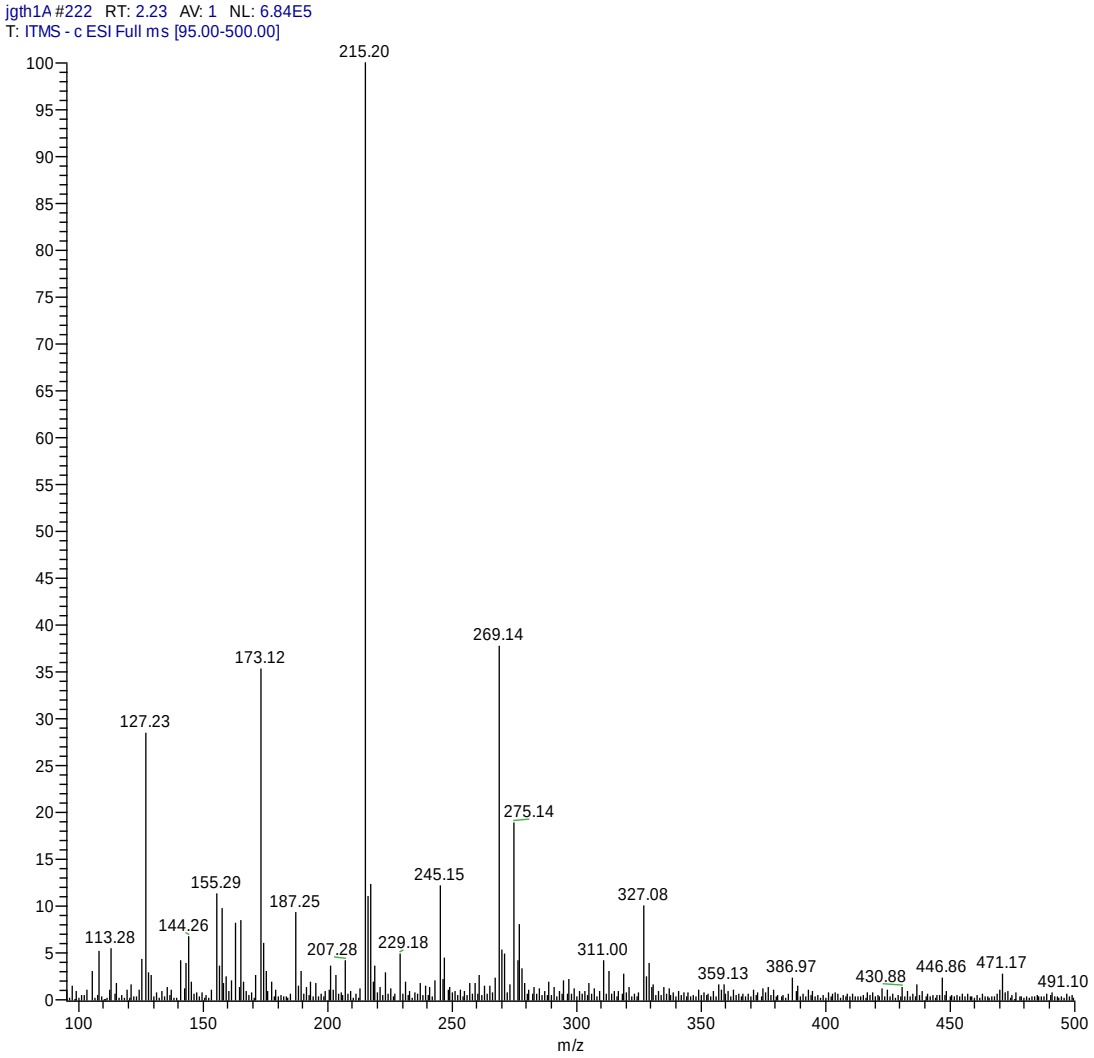


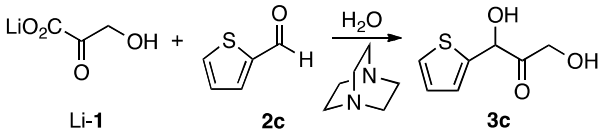
^{13}C NMR of [1-^{13}C]-3-dihydroxy-1-phenyl-2-propanone [1-^{13}C]-3b





ESI-(-)-MS spectrum for the reaction between 2c and Li-1 catalysed by DABCO





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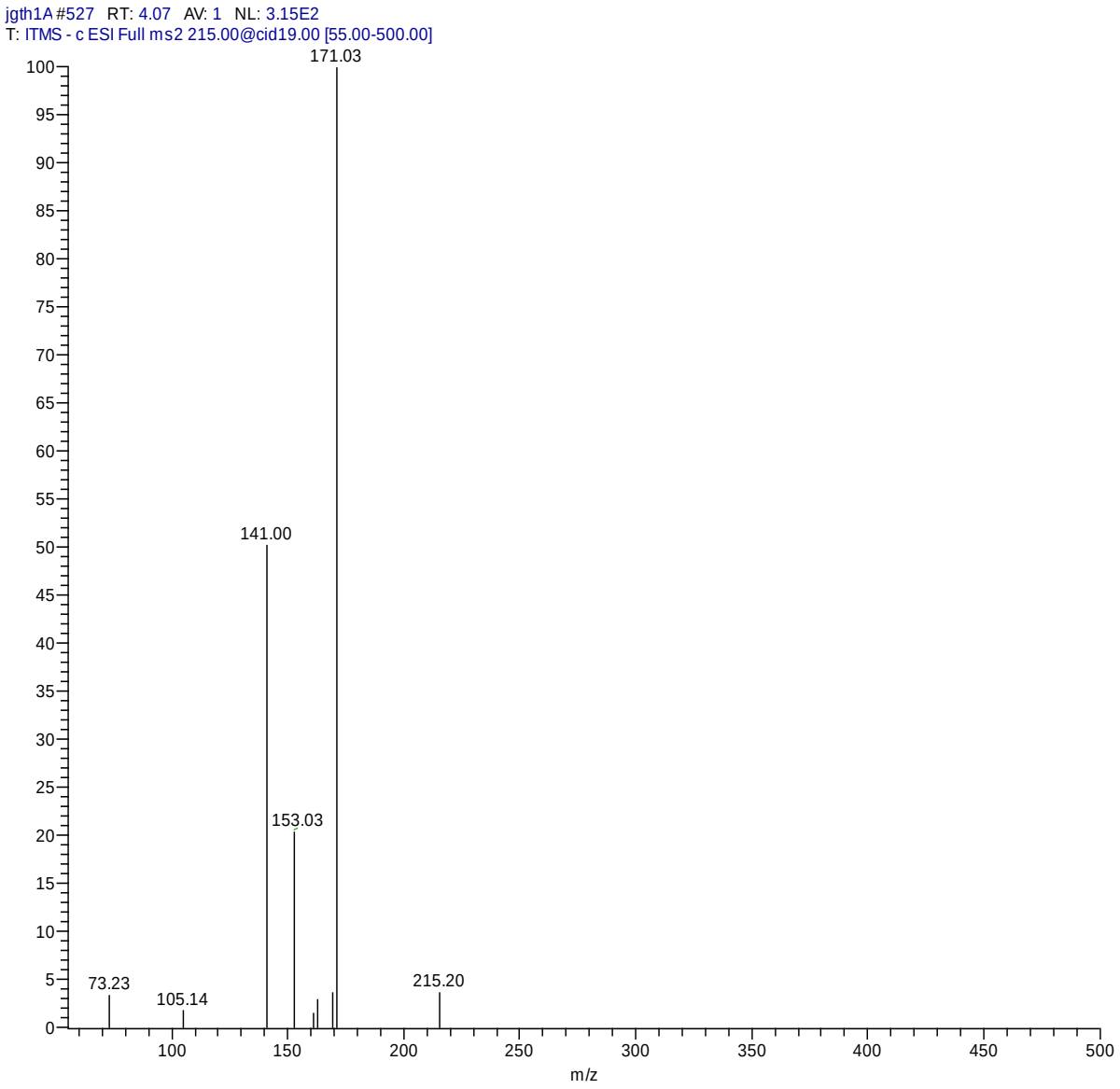

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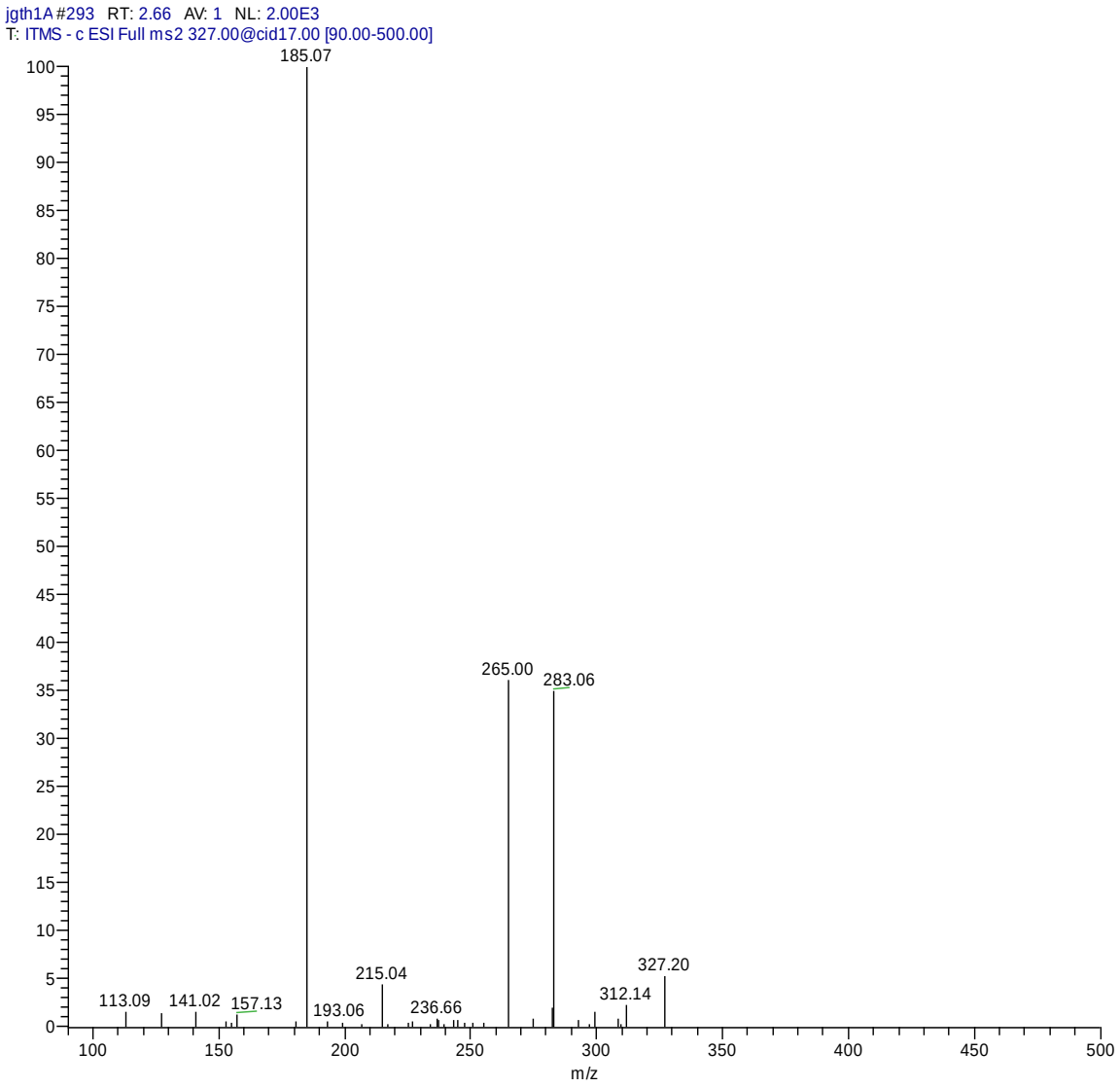
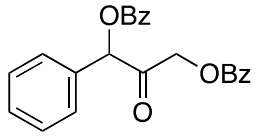


Chiral HPLC analysis of dibenzoylated 3b



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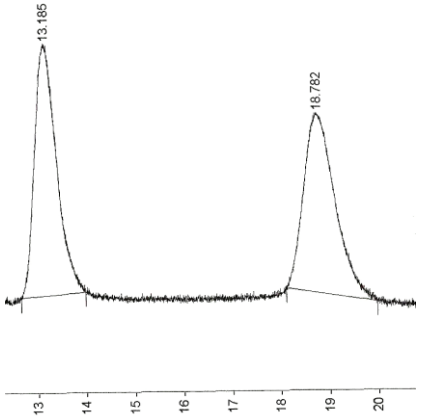

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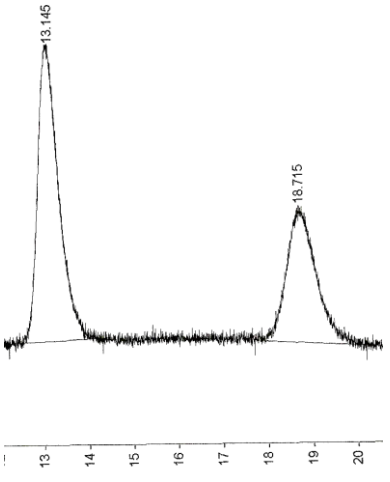
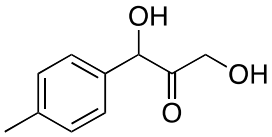
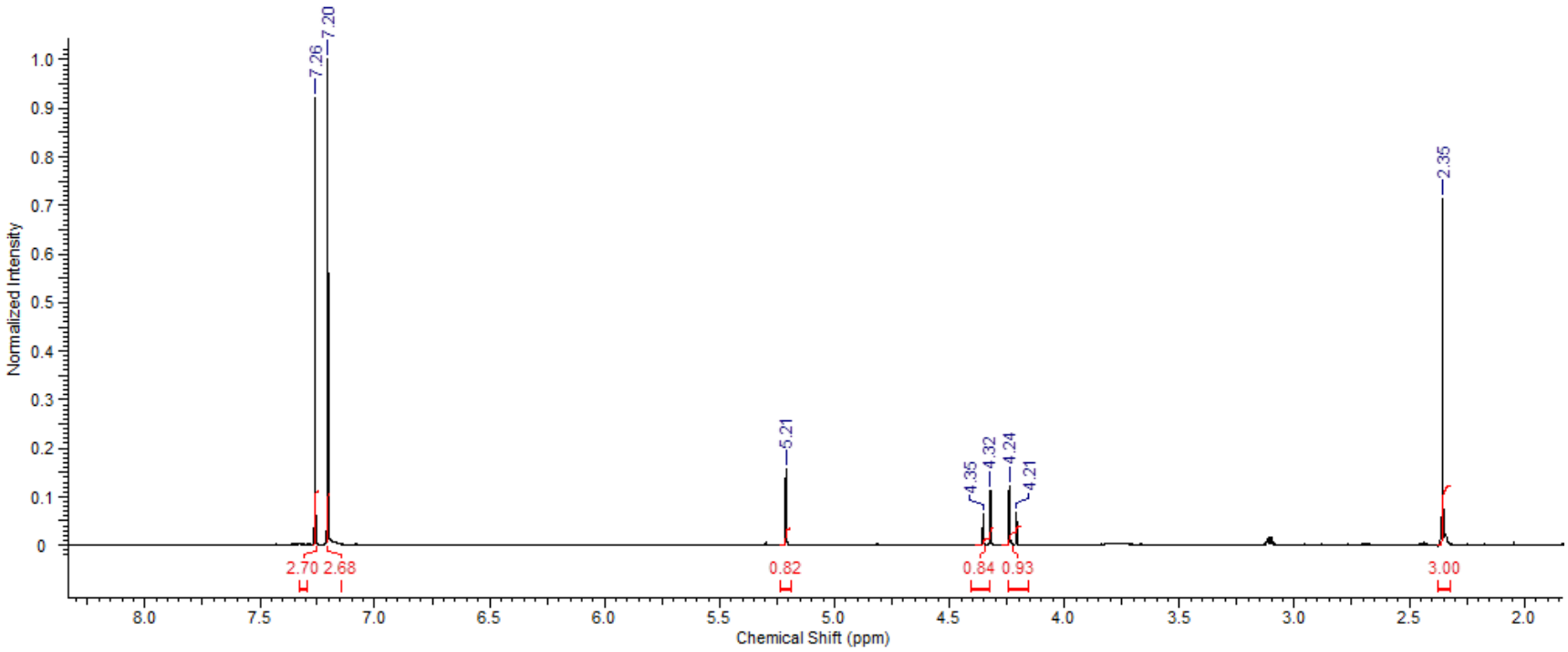


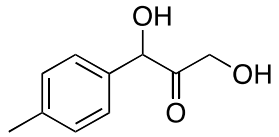
22% ee

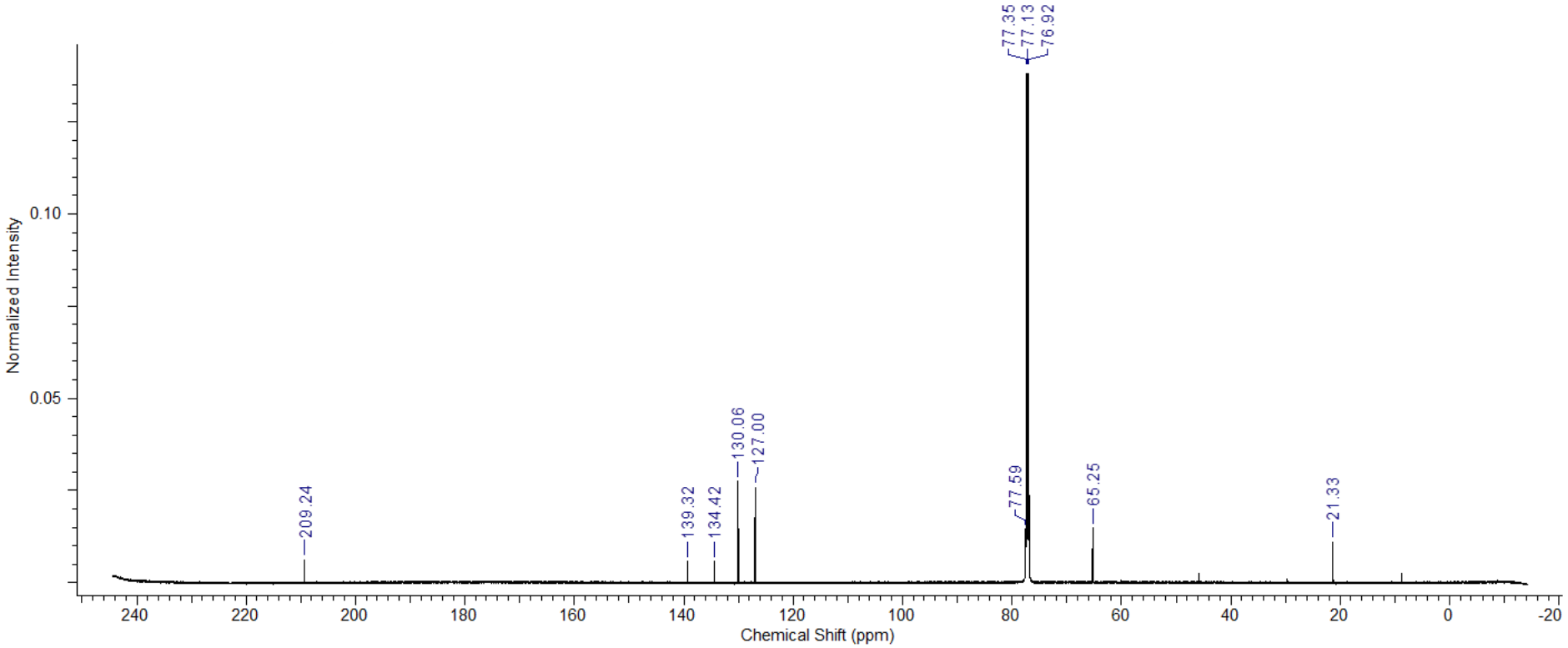


^{1}H NMR of 1,3-dihydroxy-1-(4-methylphenyl)-2-propanone (3d)

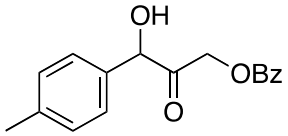


^{13}C NMR of 1,3-dihydroxy-1-(4-methylphenyl)-2-propanone (3d)





Chiral HPLC analysis of monobenzoylated 3d



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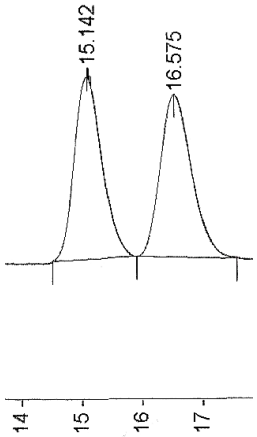

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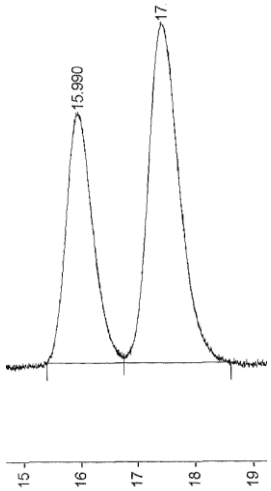
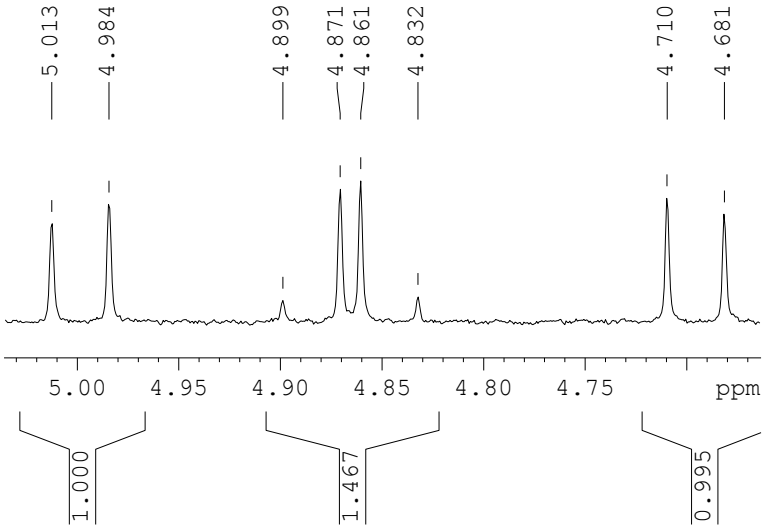
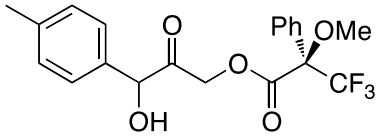


21% ee

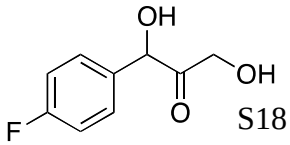
^{1}H NMR of key region of 3,3,3-Trifluoro-2-methoxy-2-phenyl-propionic acid 3-hydroxy-2-oxo-3-p-tolyl-propyl ester (Mosher's ester of 3d)

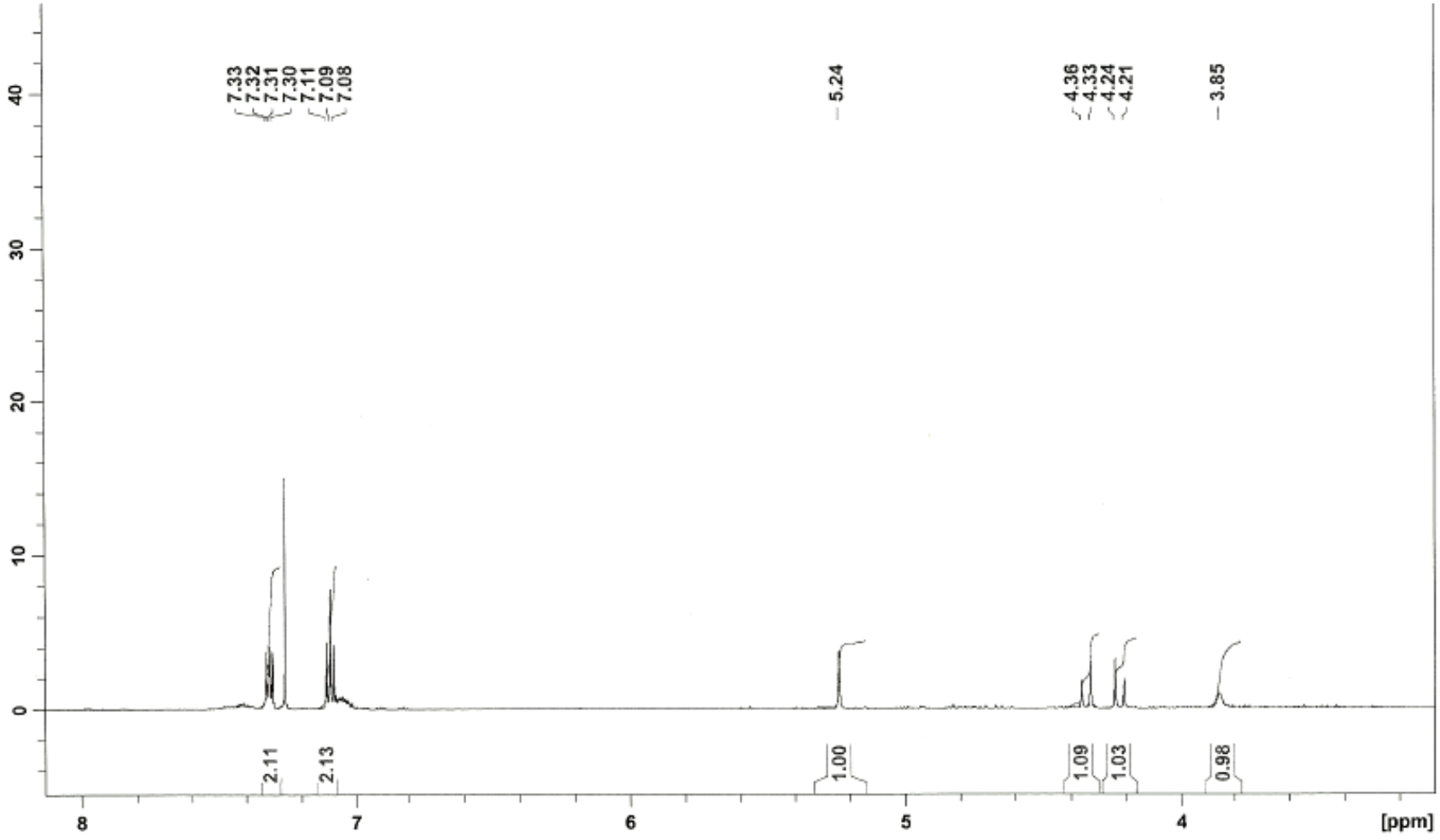




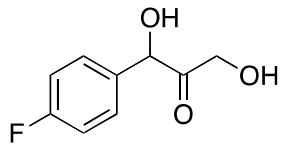
(3*R*)-major isomer

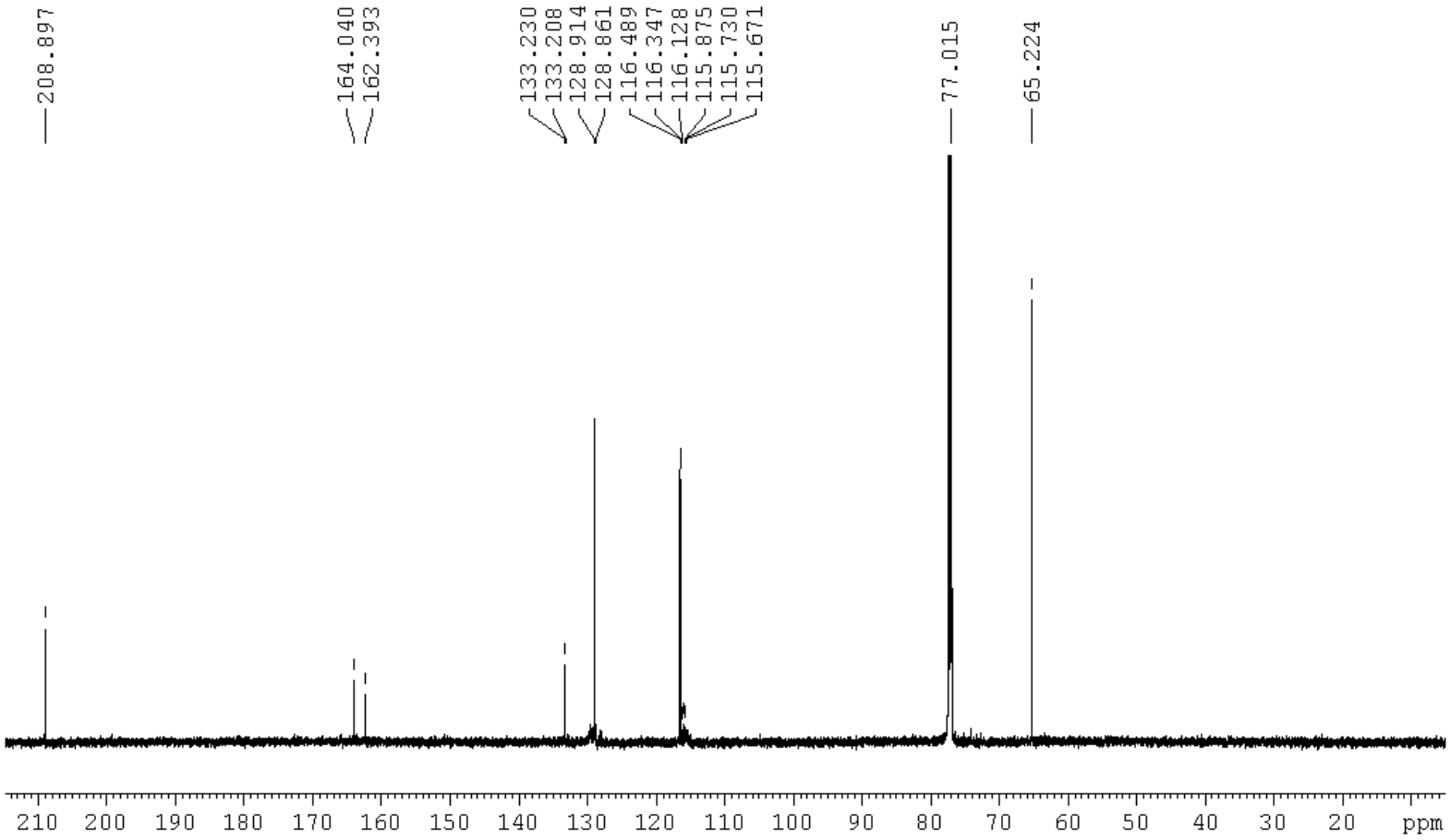
[1]H NMR of 1-(4-fluorophenyl)-1,3-dihydroxy-2-propanone (3e)

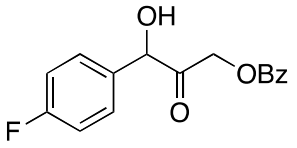




^{13}C NMR of 1-(4-fluorophenyl)-1,3-dihydroxy-2-propanone (3e)



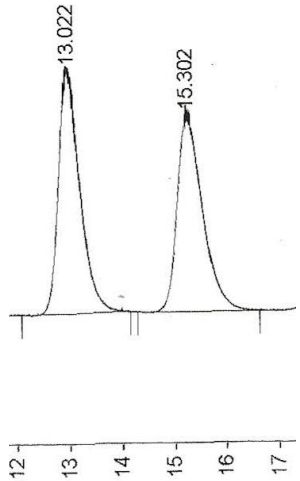




Chiral HPLC analysis of monobenzoylated 3e

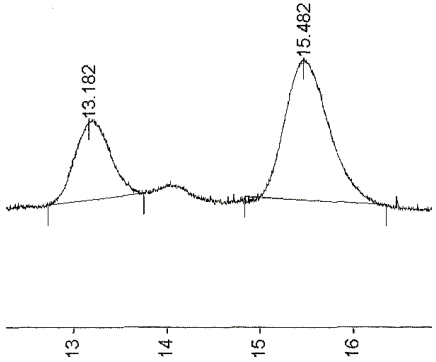
Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		0.4931	3.108	0.000	92849	BB	0.0	
2		50.3005	13.022	0.000	9471602	BB	24.8	
3		49.1529	15.302	0.000	9255510	BB	29.6	
4		0.0449	28.165	0.000	8459	BB	0.7	
5		0.0085	32.872	0.000	1607	BB	0.7	
	Totals:	99.9999		0.000	18830027			



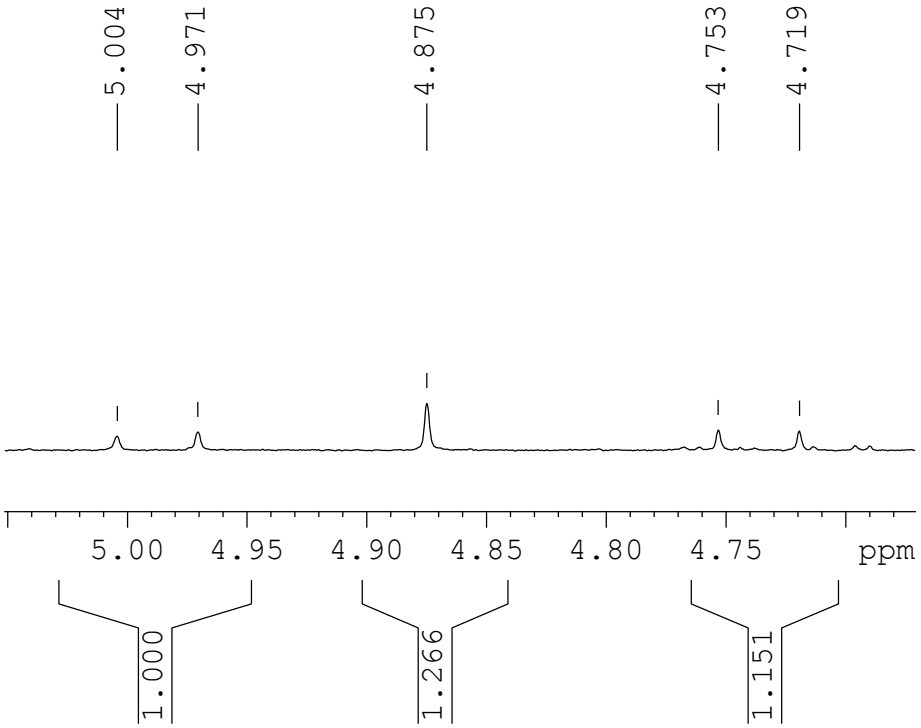
Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

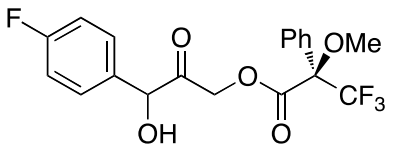
Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		31.8326	13.182	0.000	894459	BB	26.9	
2		68.1674	15.482	0.000	1915422	BB	29.7	
	Totals:	100.0000		0.000	2809881			



36% *ee*

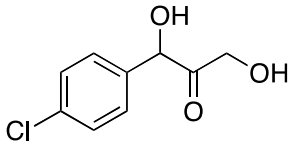
^{1}H NMR of key region of 1-(4-Fluorophenyl)-1-hydroxy-3-(2,2,2-trifluoro-1-methoxy-1-phenyl-ethoxy)-2-propanone (Mosher's ester of 3e)

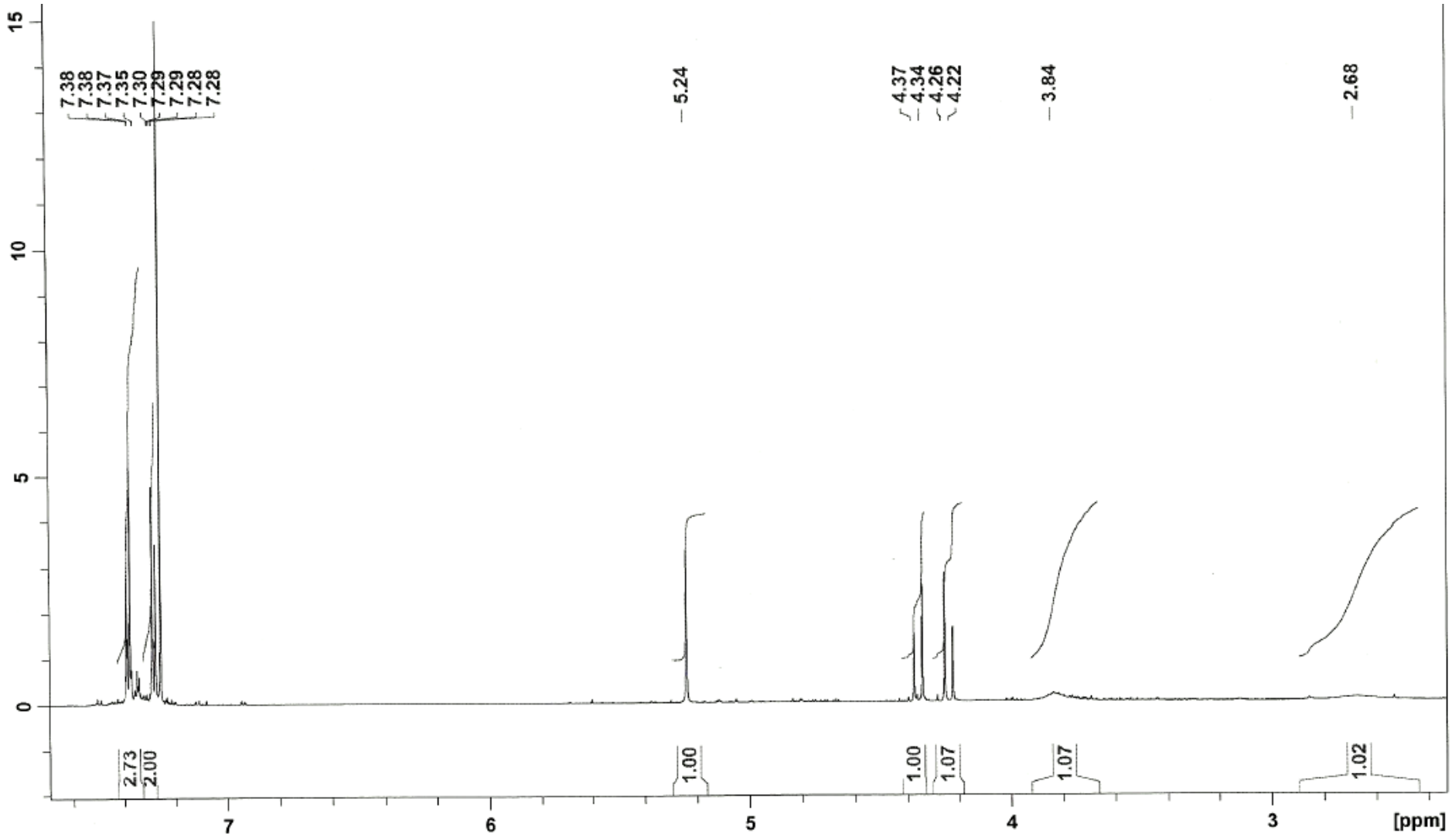




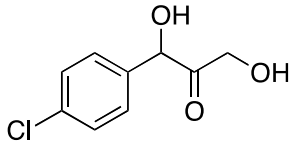
(3*R*)-major isomer

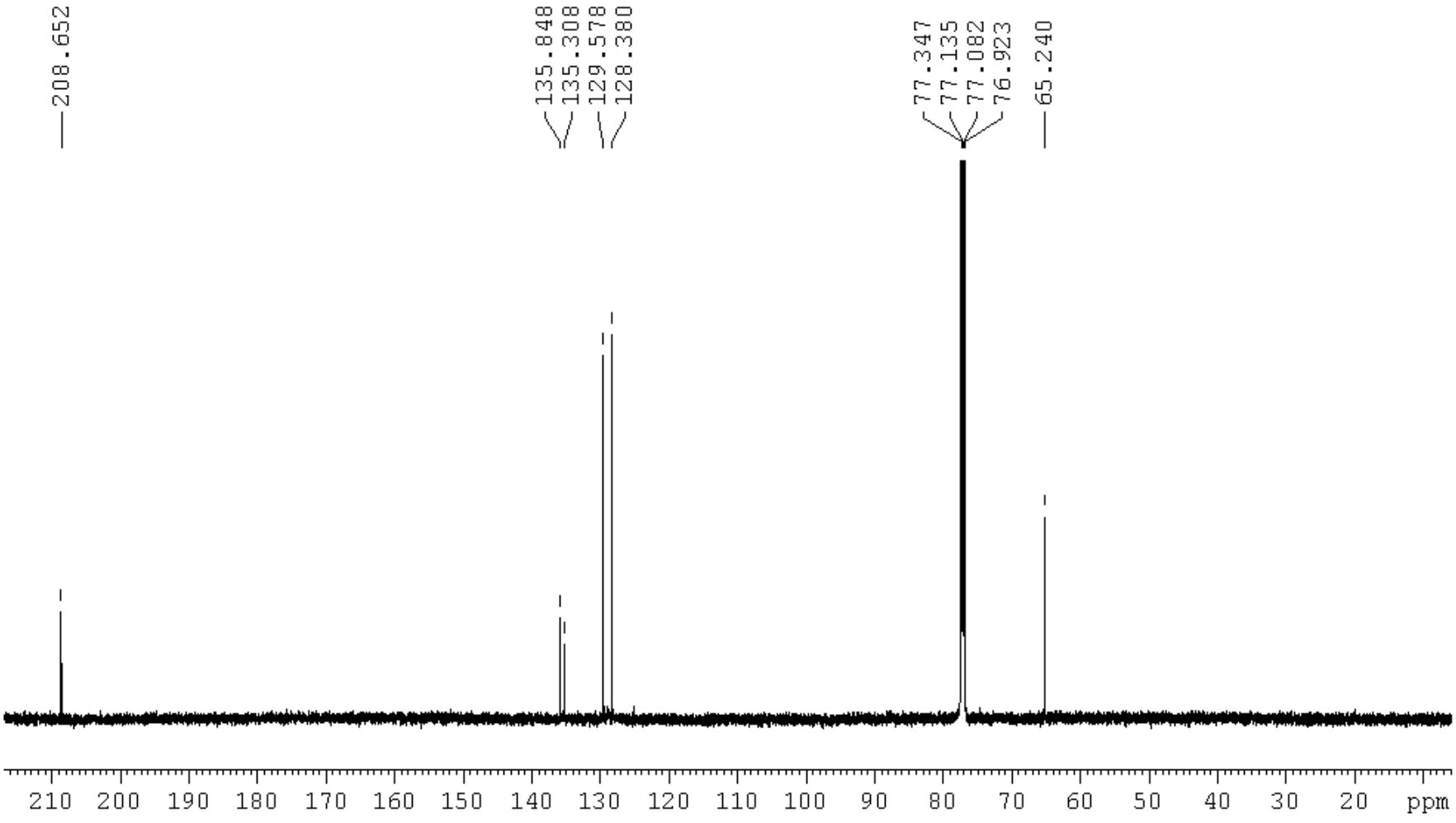
^{1}H NMR of 1-(4-chlorophenyl)-1,3-dihydroxy-2-propanone (3f)



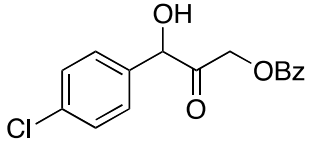


^{13}C NMR of 1-(4-chlorophenyl)-1,3-dihydroxypropan-2-one (3f)





Chiral HPLC analysis of monobenzoylated 3f



Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

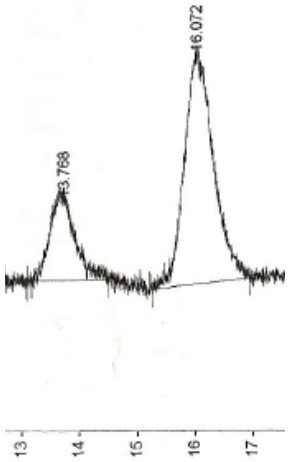
Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		49.7101	13.575	0.000	8584182	BB	29.6	
2		50.2899	15.878	0.000	8684309	BB	35.4	
	Totals:	100.0000		0.000	17268491			

Total Unidentified Counts : 17268492 counts

Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

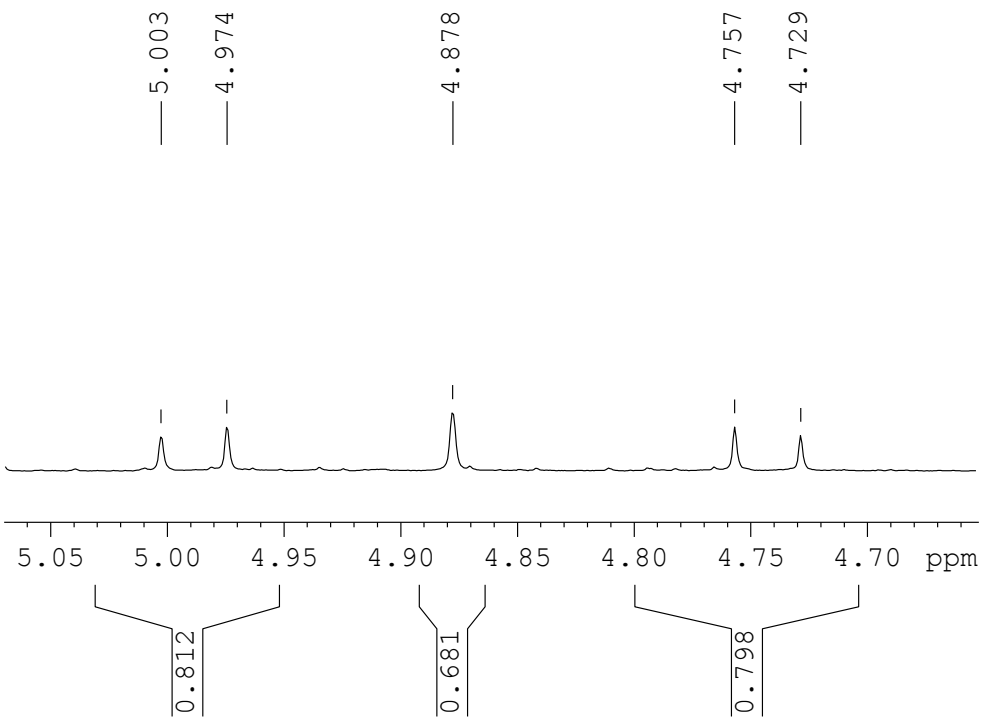
Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		24.8727	13.768	0.000	191790	BB	22.1	
2		75.1273	16.072	0.000	579295	BB	30.6	
	Totals:	100.0000		0.000	771085			

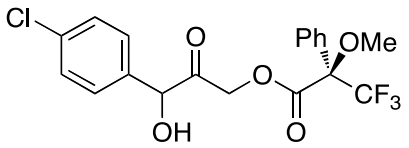
Total Unidentified Counts : 771085 counts



50% *ee*

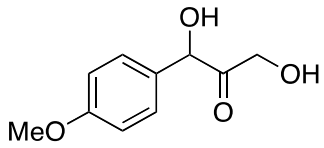
[1]H NMR of key region of 1-(4-chlorophenyl)-1-hydroxy-3-(2,2,2-trifluoro-1-methoxy-1-phenyl-ethoxy)-2-propanone (Mosher's ester of 3f)

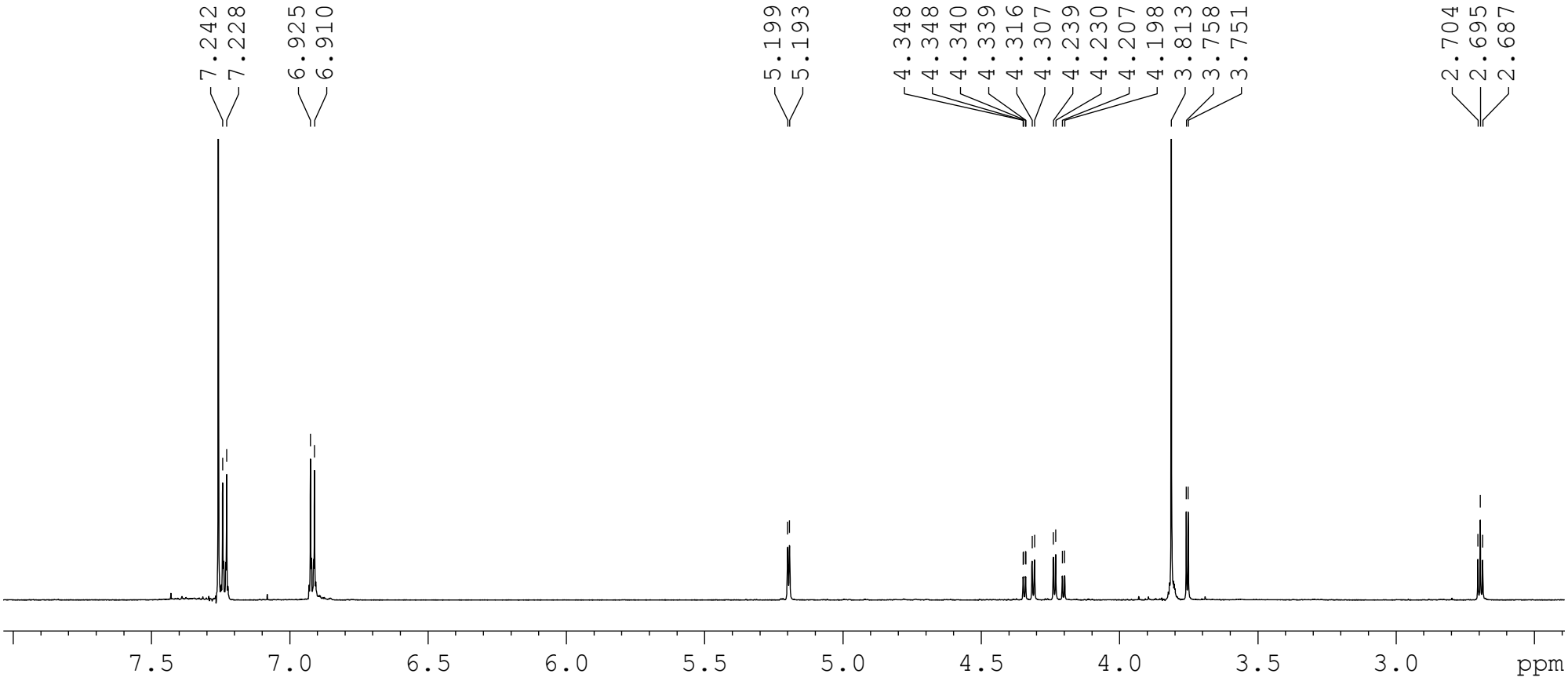




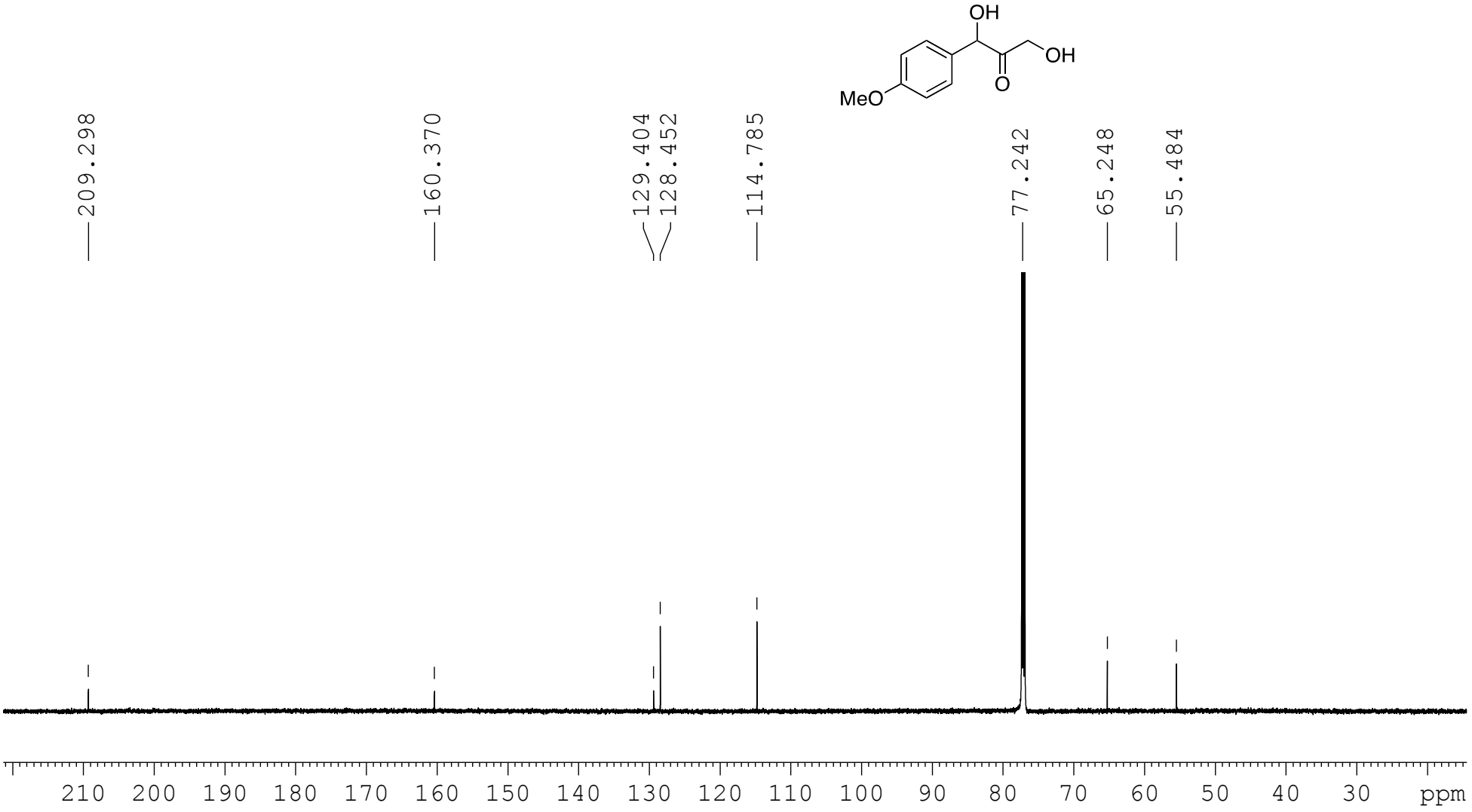
(3*R*)-major isomer

^{1}H NMR of 1,3-dihydroxy-1-(4-methoxyphenyl)-2-propanone (3g)

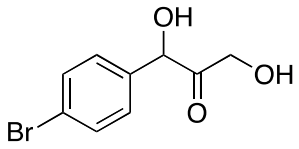


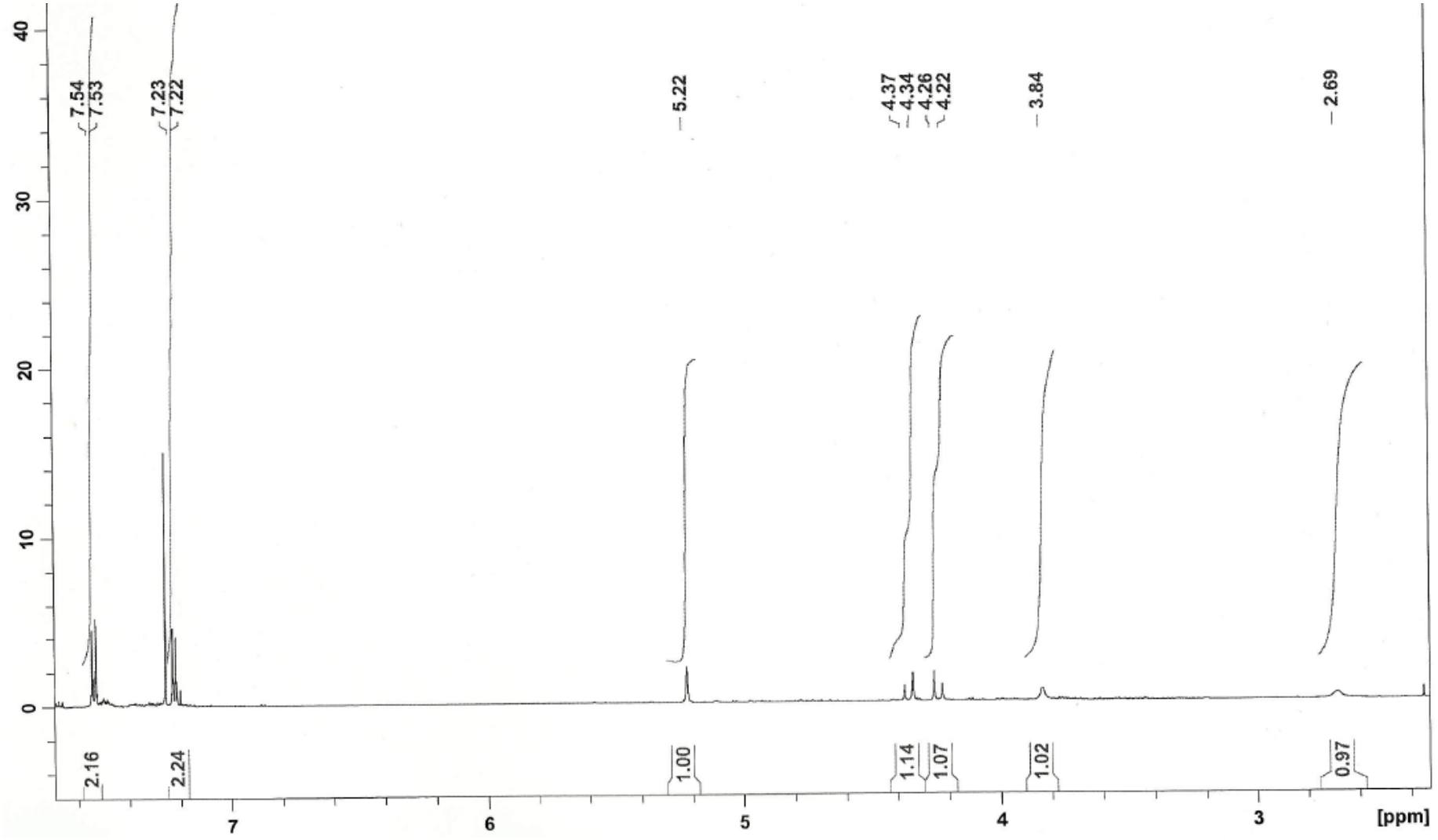


^{13}C NMR of 1,3-dihydroxy-1-(4-methoxyphenyl)-propan-2-one (3g)

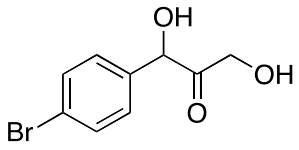


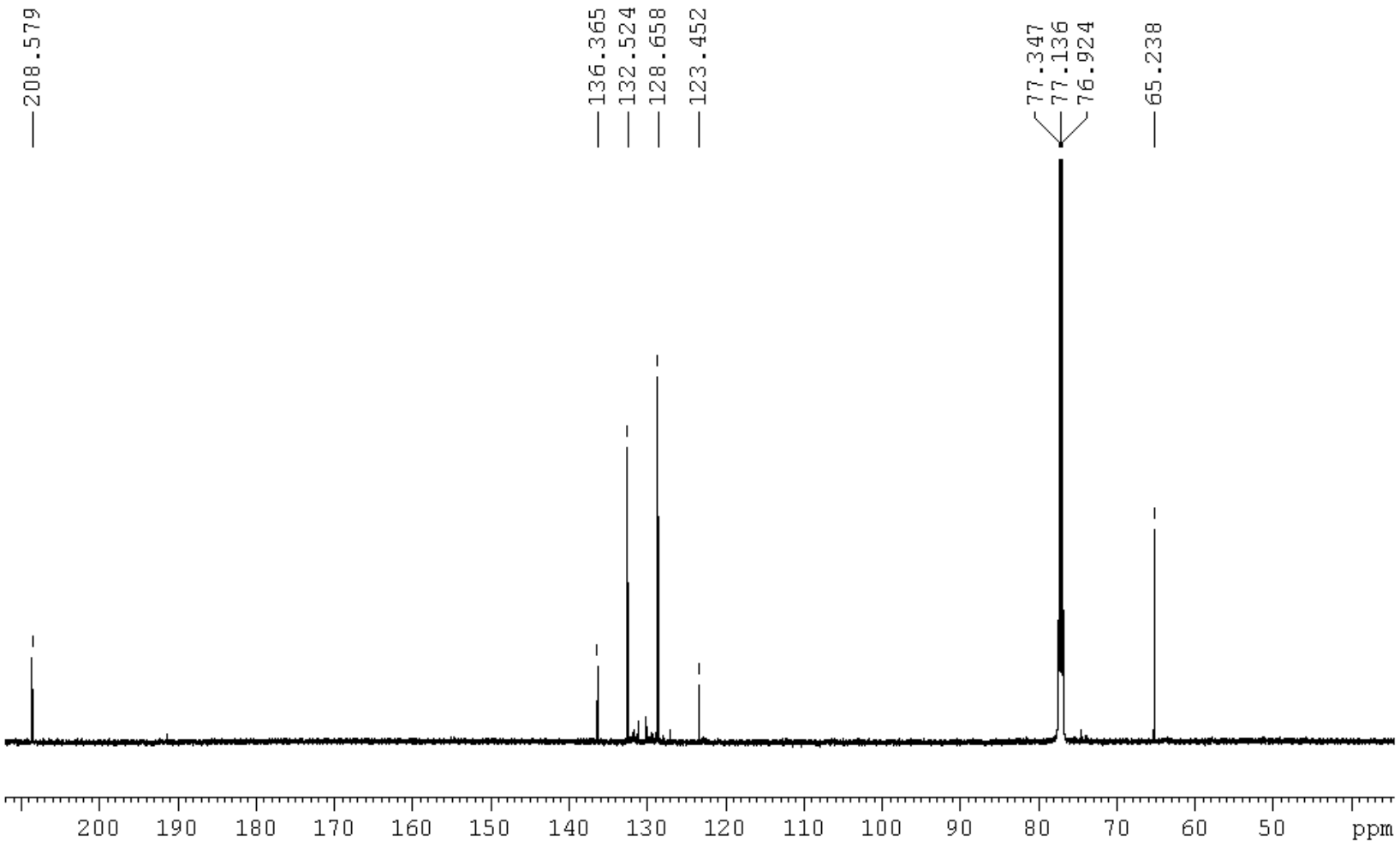
^{1}H NMR of 1-(4-bromophenyl)-1,3-dihydroxy-2-propanone (3h)



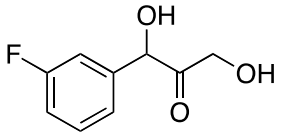


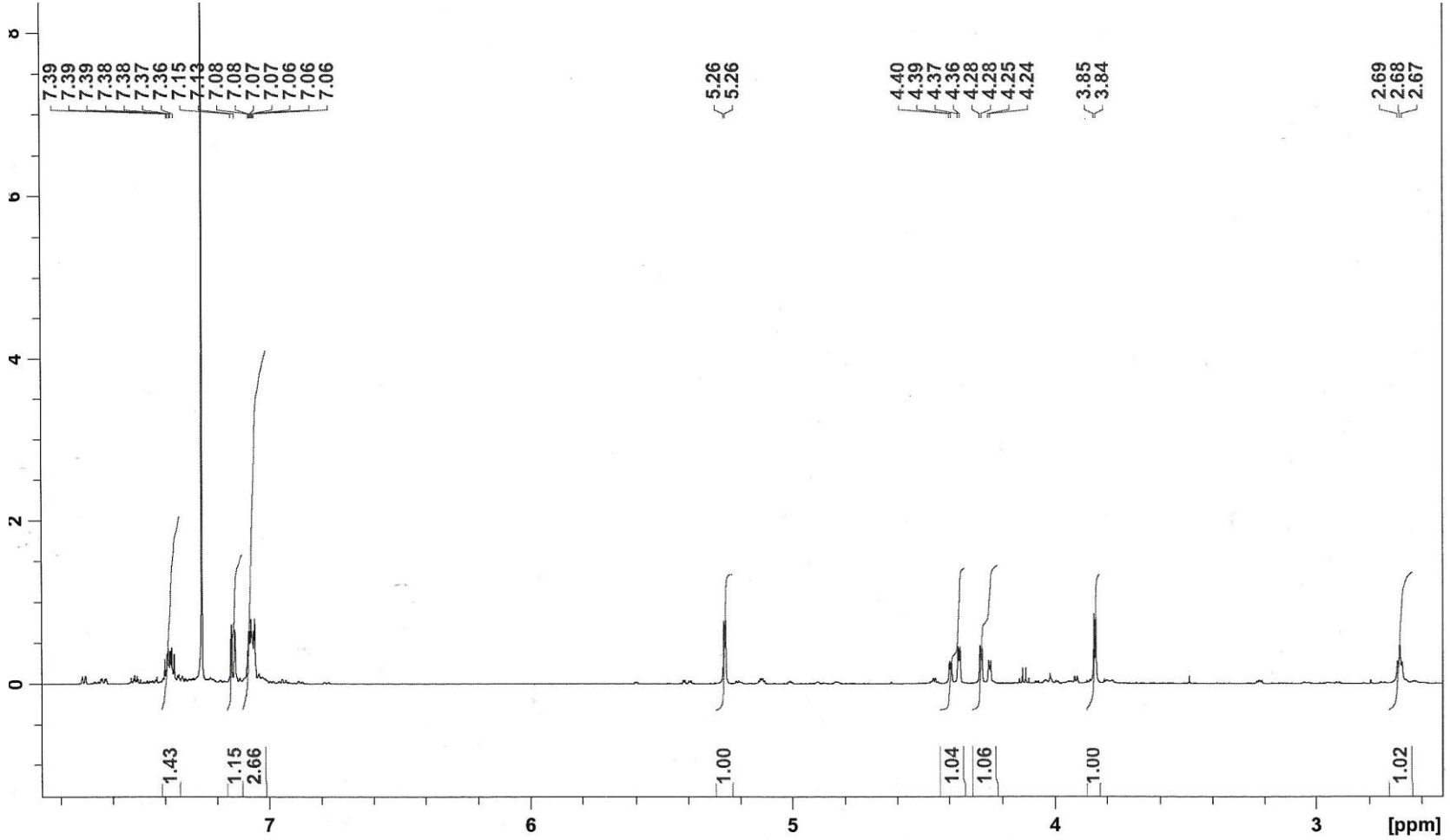
^{13}C NMR of 1-(4-bromophenyl)-1,3-dihydroxy-2-propanone (3h)



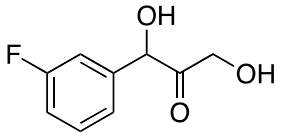


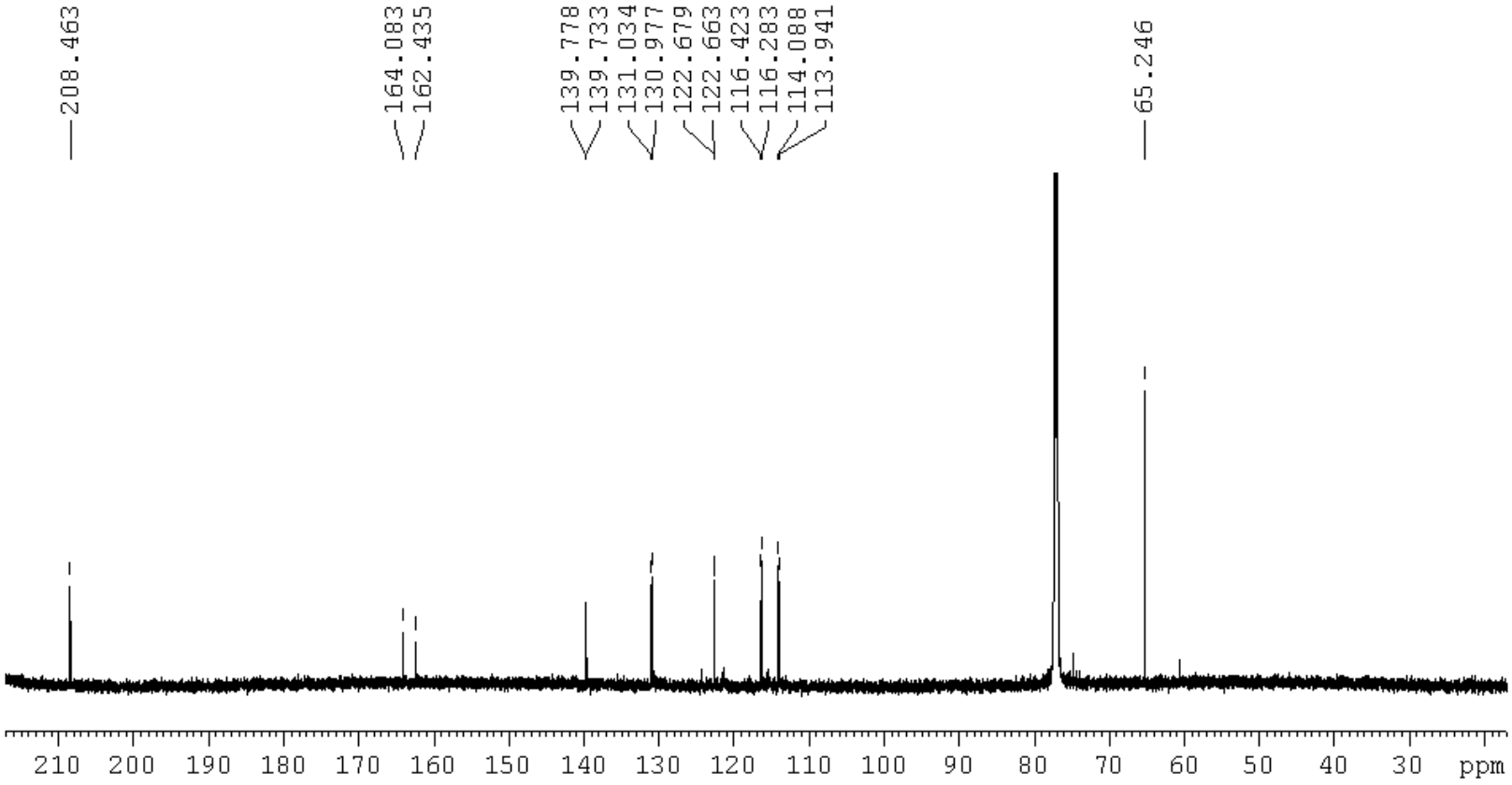
^{1}H NMR of 1-(3-fluorophenyl)-1,3-dihydroxy-2-propanone (3i)



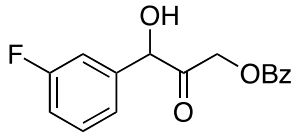


^{13}C NMR of 1-(3-fluorophenyl)-1,3-dihydroxy-2-propanone (3i)



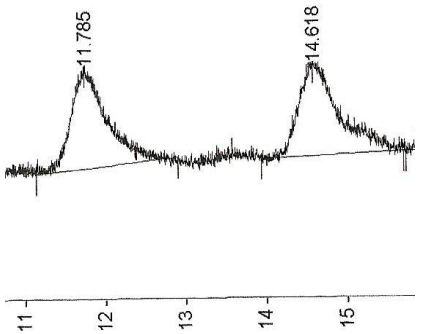


Chiral HPLC analysis of monobenzoylated 3i



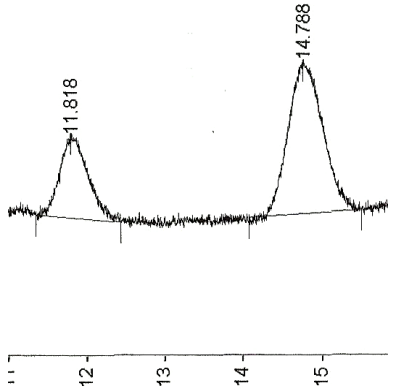
Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		3.6996	3.172	0.000	69223	BB	0.0	
2		24.2664	3.562	0.000	454047	BB	12.5	
3		30.0109	6.952	0.000	561531	BB	13.5	
4		16.6789	11.785	0.000	312078	BB	26.2	
5		17.1284	14.618	0.000	320489	BB	27.9	
6		6.4561	16.432	0.000	120800	BB	17.2	
7		1.7596	17.432	0.000	32925	BB	0.4	
	Totals:	99.9999		0.000	1871093			



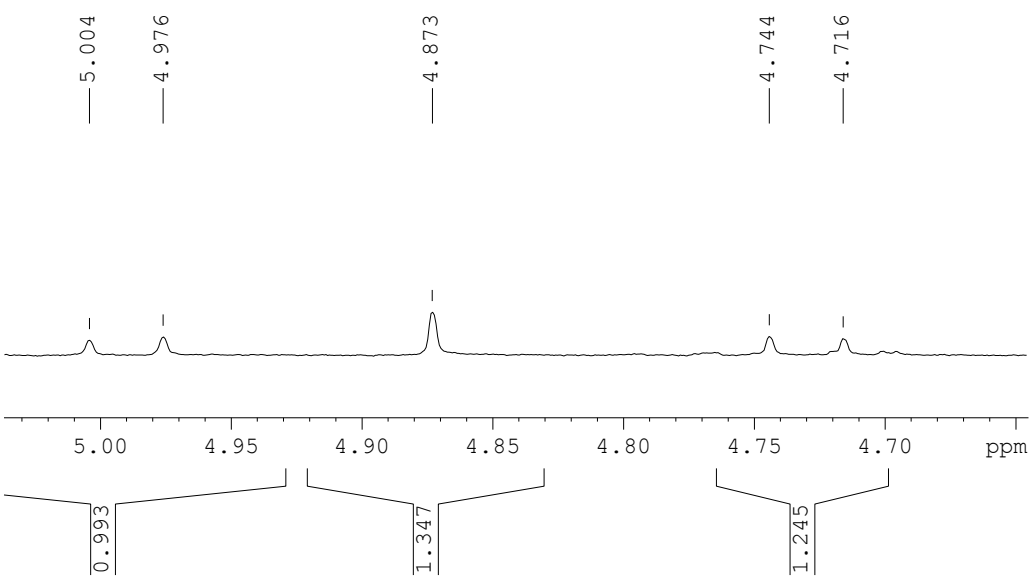
Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

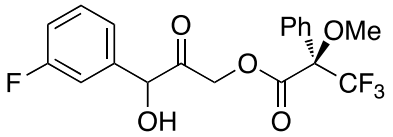
Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		31.1307	11.818	0.000	286947	BB	21.2	
2		68.8693	14.788	0.000	634803	BB	29.1	
	Totals:	100.0000		0.000	921750			



38% *ee*

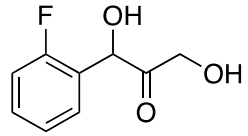
^{1}H NMR of key region of 3,3,3-trifluoro-2-methoxy-2-phenyl-propionic acid 3-(3-fluoro-phenyl)-3-hydroxy-2-oxo-propyl ester (Mosher's ester of 3i)

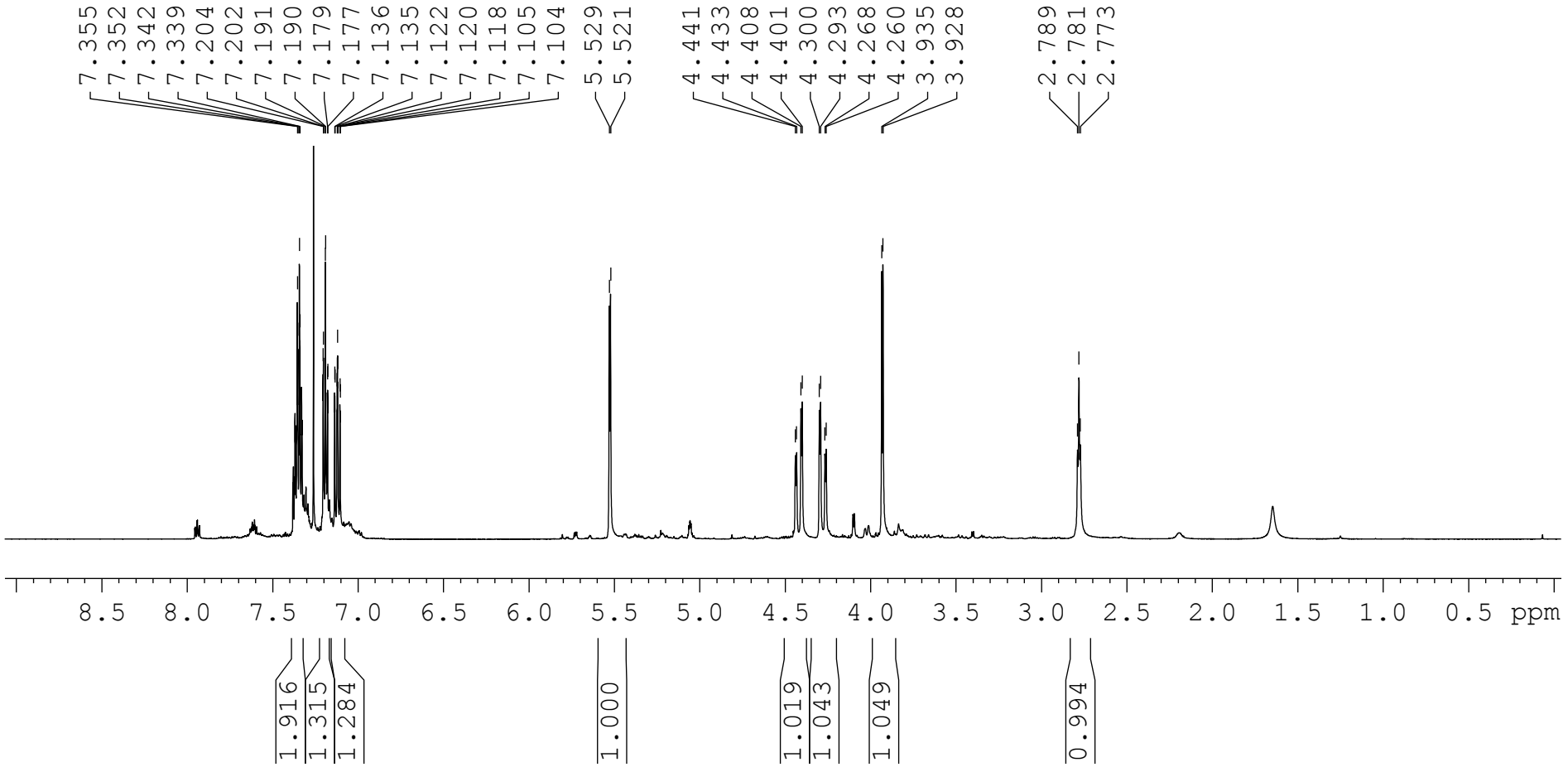




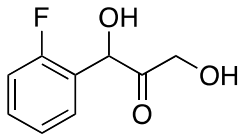
(3*R*)-major isomer

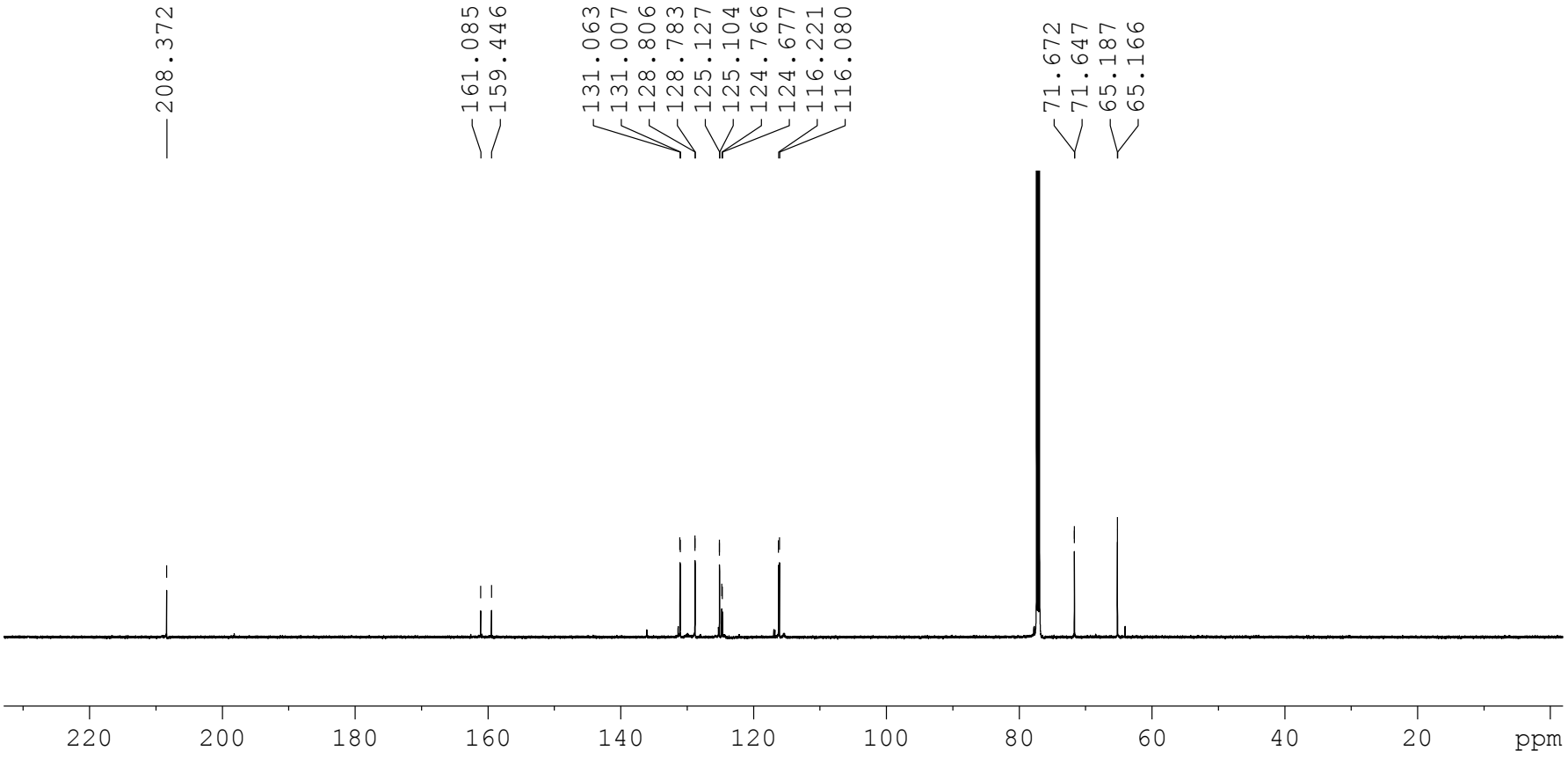
^{1}H NMR of 1-(2-fluorophenyl)-1,3-dihydroxy-2-propanone (3j)



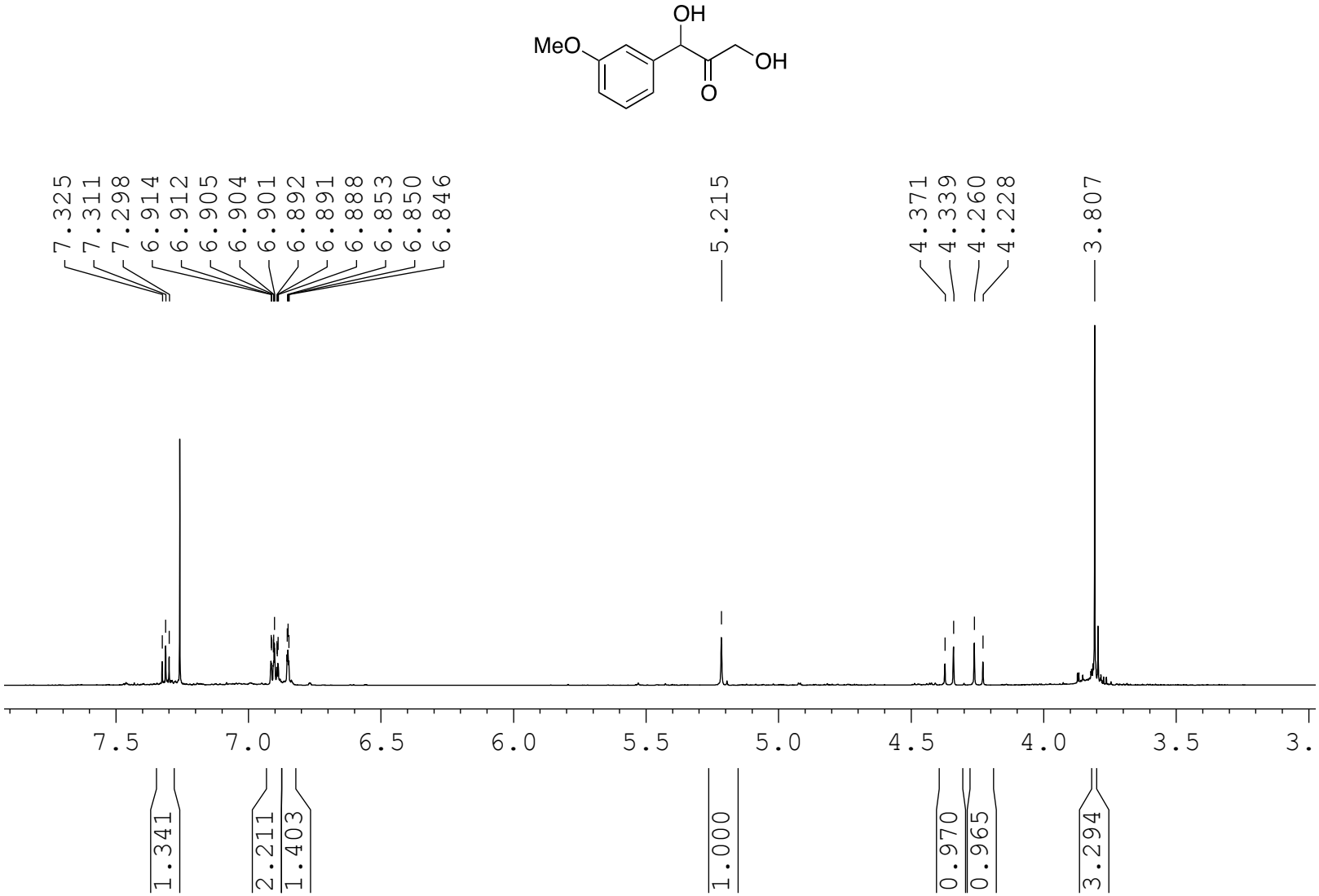


^{13}C NMR of 1-(2-fluorophenyl)-1,3-dihydroxy-2-propanone (3j)

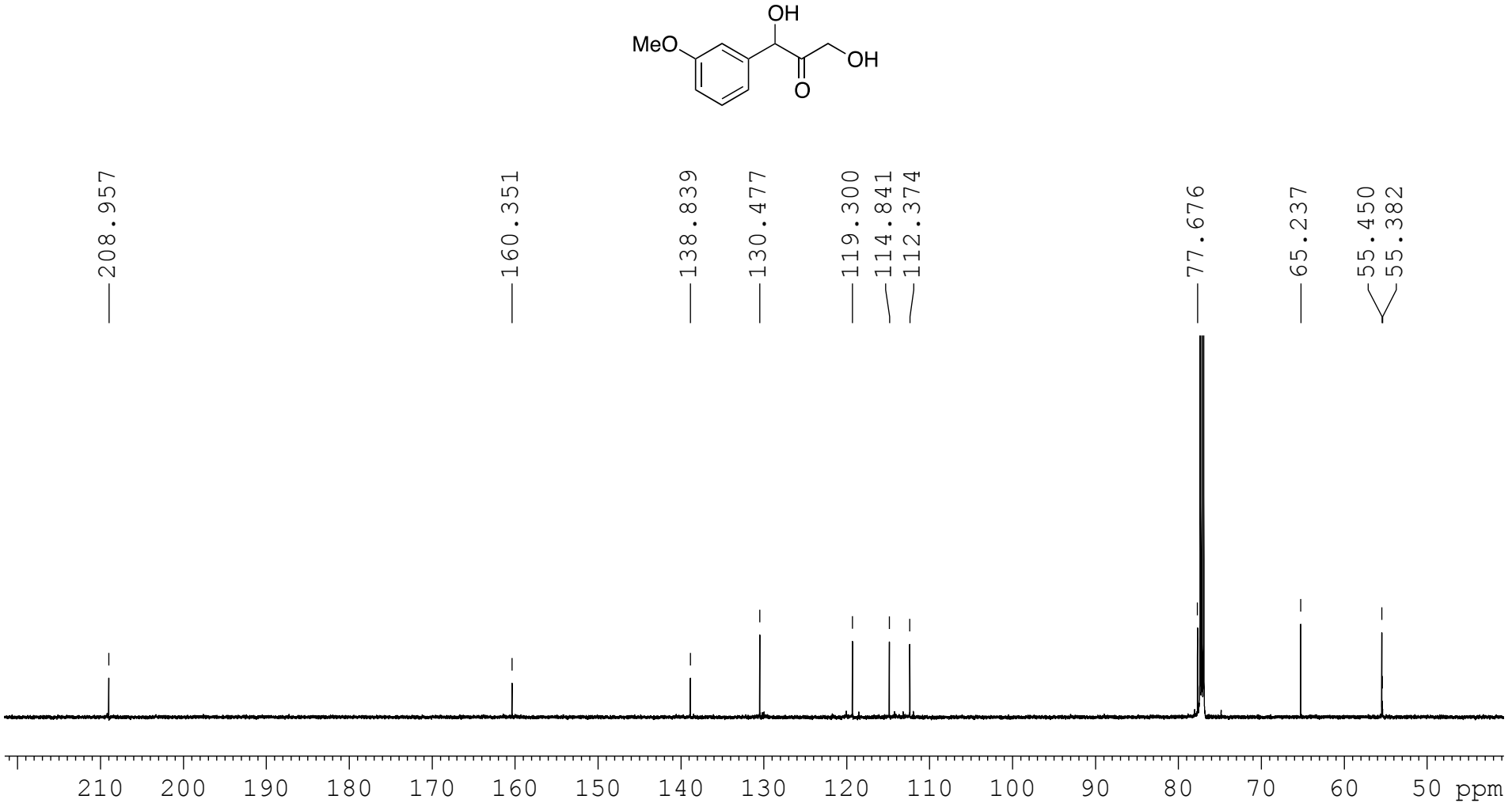




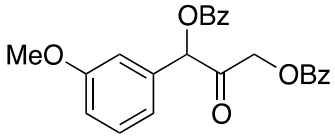
^{1}H NMR of 1,3-dihydroxy-1-(3-methoxyphenyl)-2-propanone (3k)



^{13}C NMR of 1,3-dihydroxy-1-(3-methoxyphenyl)-2-propanone (3k)



Chiral HPLC analysis of dibenzoylated 3k

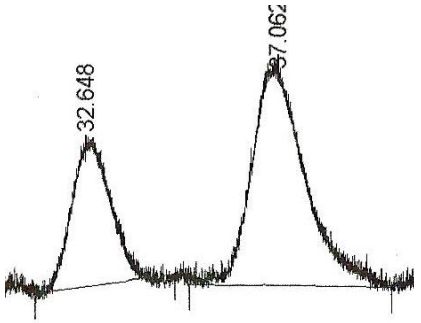


Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		31.8778	32.648	0.000	1012461	BB	57.4	
2		68.1222	37.062	0.000	2163611	BB	69.4	
	Totals:	100.0000		0.000	3176072			

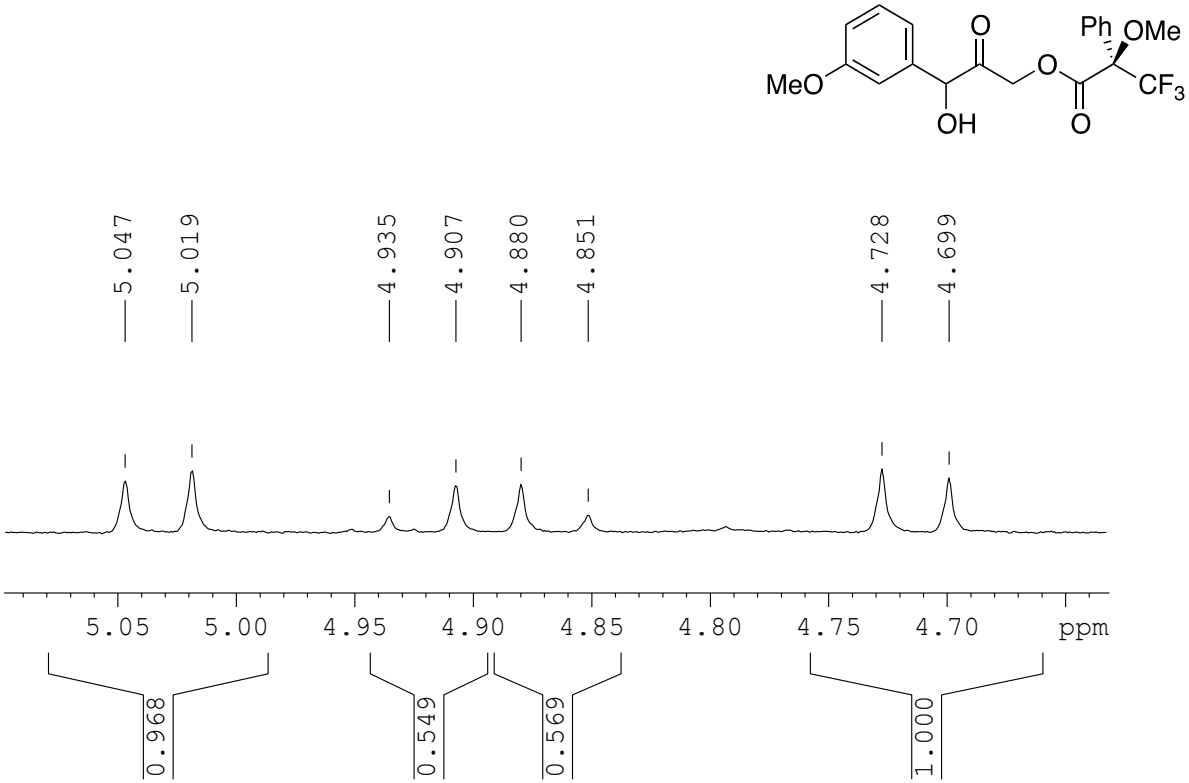
Total Unidentified Counts : 3176071 counts

Detected Peaks: 2 Rejected Peaks: 0 Identified Peaks: 0



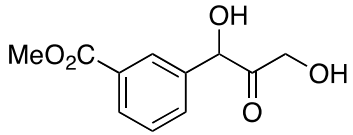
36% *ee*

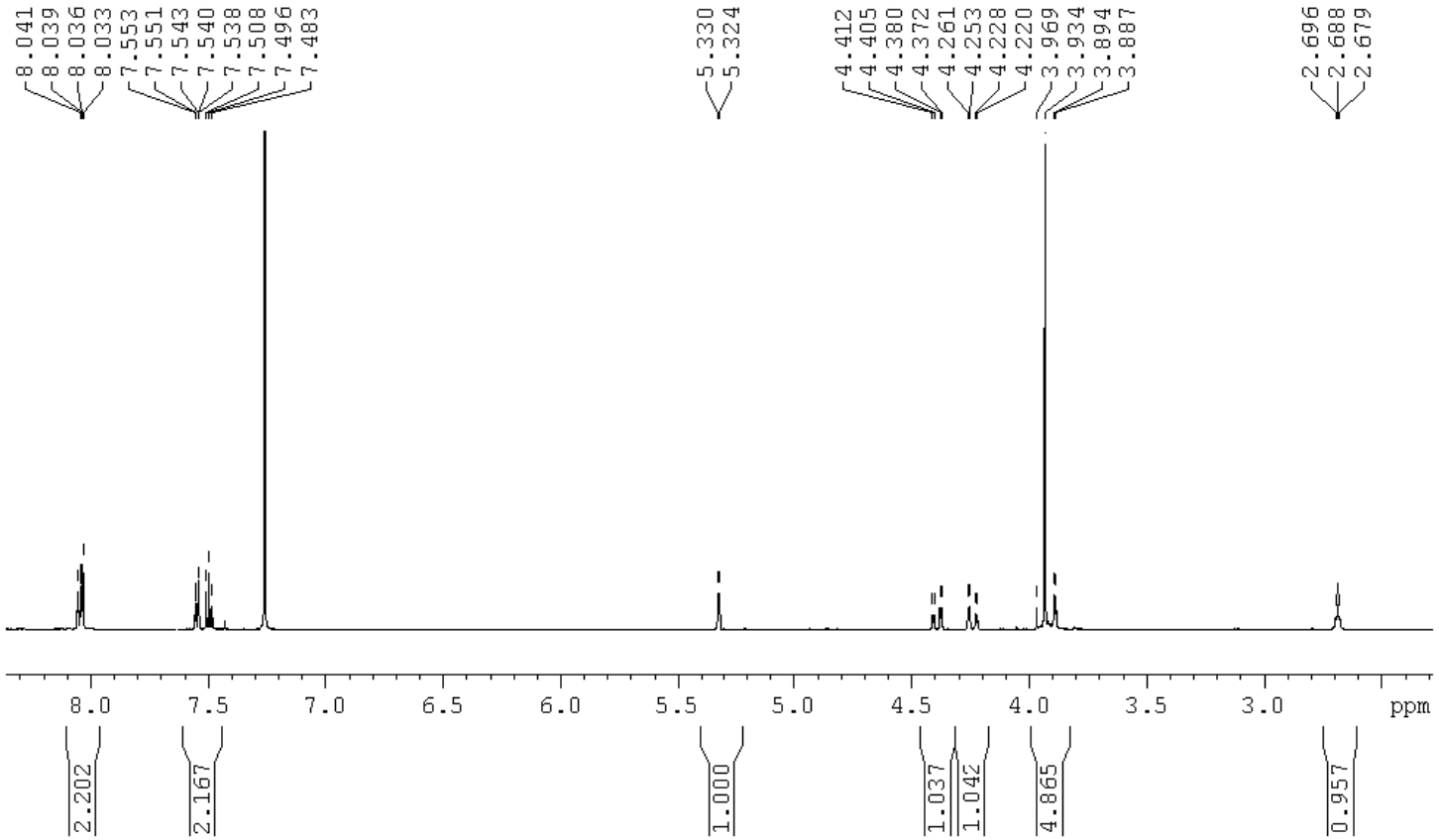
^{1}H NMR of key region of 3,3,3-trifluoro-2-methoxy-2-phenyl-propionic acid 3-hydroxy-3-(3-methoxyphenyl)-2-oxo-propyl ester (Mosher's ester of 3k)



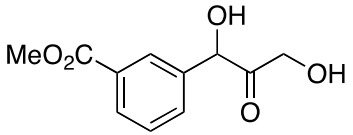
(3*R*)-major isomer

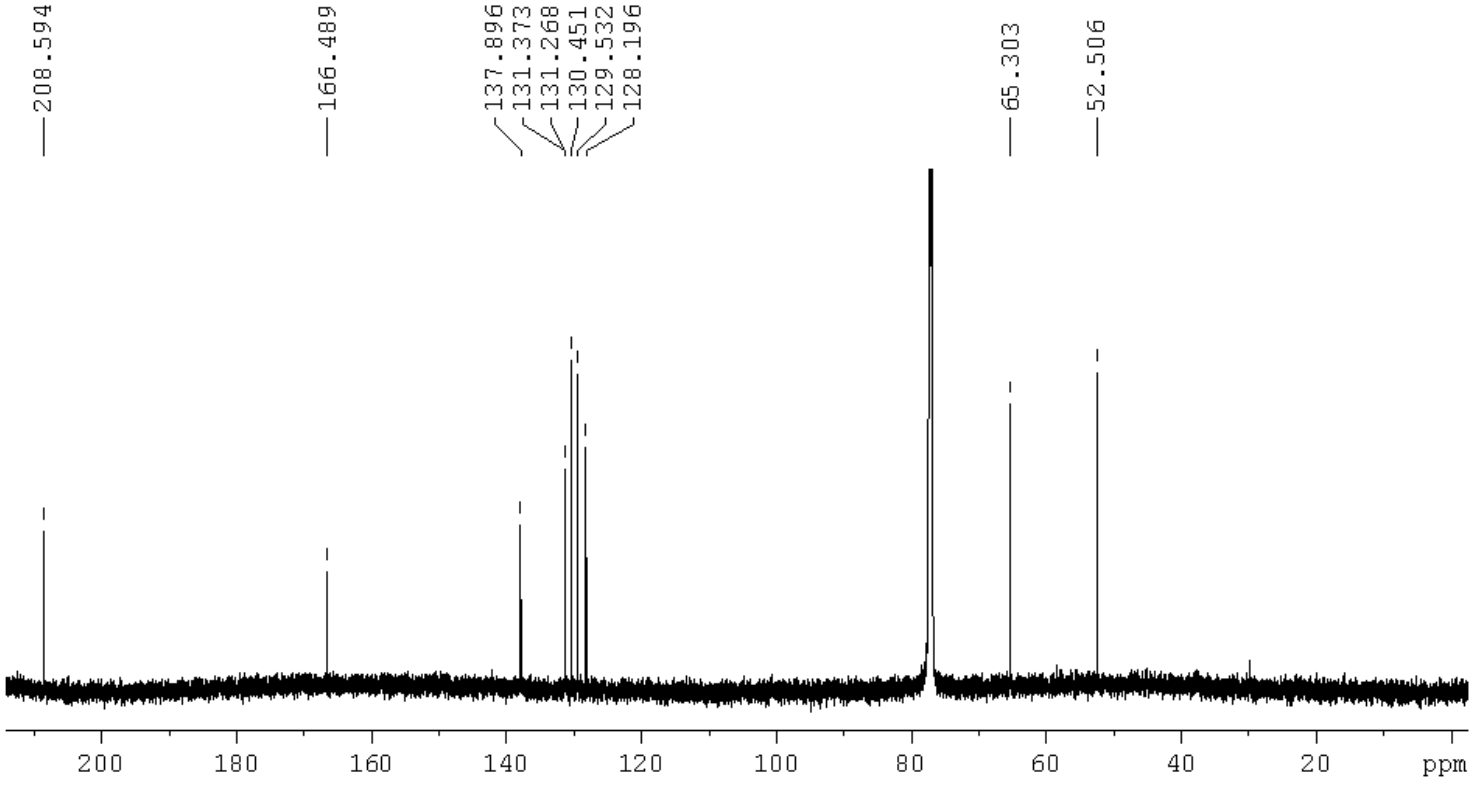
^{1}H NMR of 3-(1,3-dihydroxy-2-oxo-propyl)-benzoic acid methyl ester (3l)



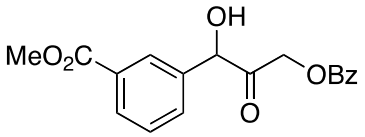


^{13}C NMR of 3-(1,3-dihydroxy-2-oxo-propyl)-benzoic acid methyl ester (3l)





Chiral HPLC analysis of monobenzoylated 3l



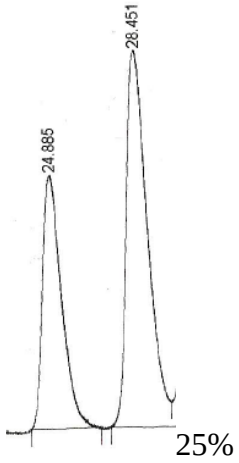
Run Mode : Analysis
Peak Measurement: Peak Area
Calculation Type: Percent

Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		4.0963	3.177	0.000	222044	BB	8.3	
2		10.1454	5.339	0.000	549941	BB	11.3	
3		26.6480	6.879	0.000	1444485	BB	14.0	
4		22.3614	24.450	0.000	1212124	BB	39.4	
5		36.7490	28.094	0.000	1212019	BB	55.7	
	Totals:	100.0001		0.000	5420613			

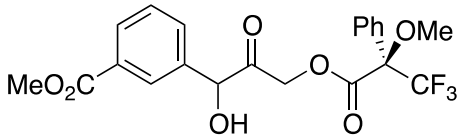
Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		0.0139	0.927	0.000	5716	BB	0.0	
2		0.4437	3.164	0.000	182245	BV	11.3	
3		0.0247	3.358	0.000	10160	VV	6.5	
4		0.7037	3.538	0.000	289006	VB	13.7	
5		0.2298	4.156	0.000	94396	BB	13.7	
6		27.1571	6.917	0.000	11153284	BB	22.5	
7		0.3003	12.953	0.000	123312	BB	19.3	
8		0.3483	14.028	0.000	143032	BB	21.7	
9		0.8107	16.432	0.000	332938	BB	32.0	
10		0.0294	18.477	0.000	12063	BB	2.8	
11		19.1927	24.885	0.000	7882375	BB	30.1	
12		31.6595	28.451	0.000	13019823	BV	55.4	
13		12.6452	30.642	0.000	5193323	VB	58.6	
14		0.8636	34.427	0.000	354677	BV	40.0	
15		2.2141	34.697	0.000	909321	VV	87.3	
16		3.3233	37.821	0.000	1364878	VB	73.7	
	Totals:	100.0000		0.000	41069549			

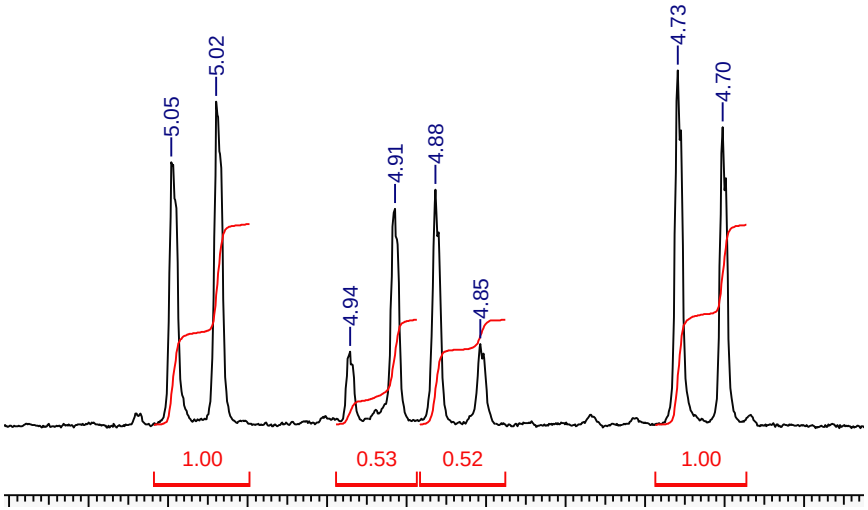
Total Unidentified Counts : 41069544 counts

Detected Peaks: 16 Rejected Peaks: 0 Identified Peaks: 0



^{1}H NMR of key region of 3-[1-hydroxy-2-oxo-3-(3,3,3-trifluoro-2-methoxy-2-phenyl-propionyloxy)-propyl]-benzoic acid methyl ester (Mosher's ester of 3l)





(3*R*)-major isomer

3. References

1. a) J. L. Galman and H. C. Hailes, *Tetrahedron: Asymmetry*, 2009, **20**, 1828–1831; b) A. Cázares, J. L. Galman, L. G. Crago, M. E. B. Smith, J. Strafford, L. Ríos-Solís, G. J. Lye, P. A. Dalby and H. C. Hailes, *Org. Biomol. Chem*., 2010, **6**, 1301–1309.