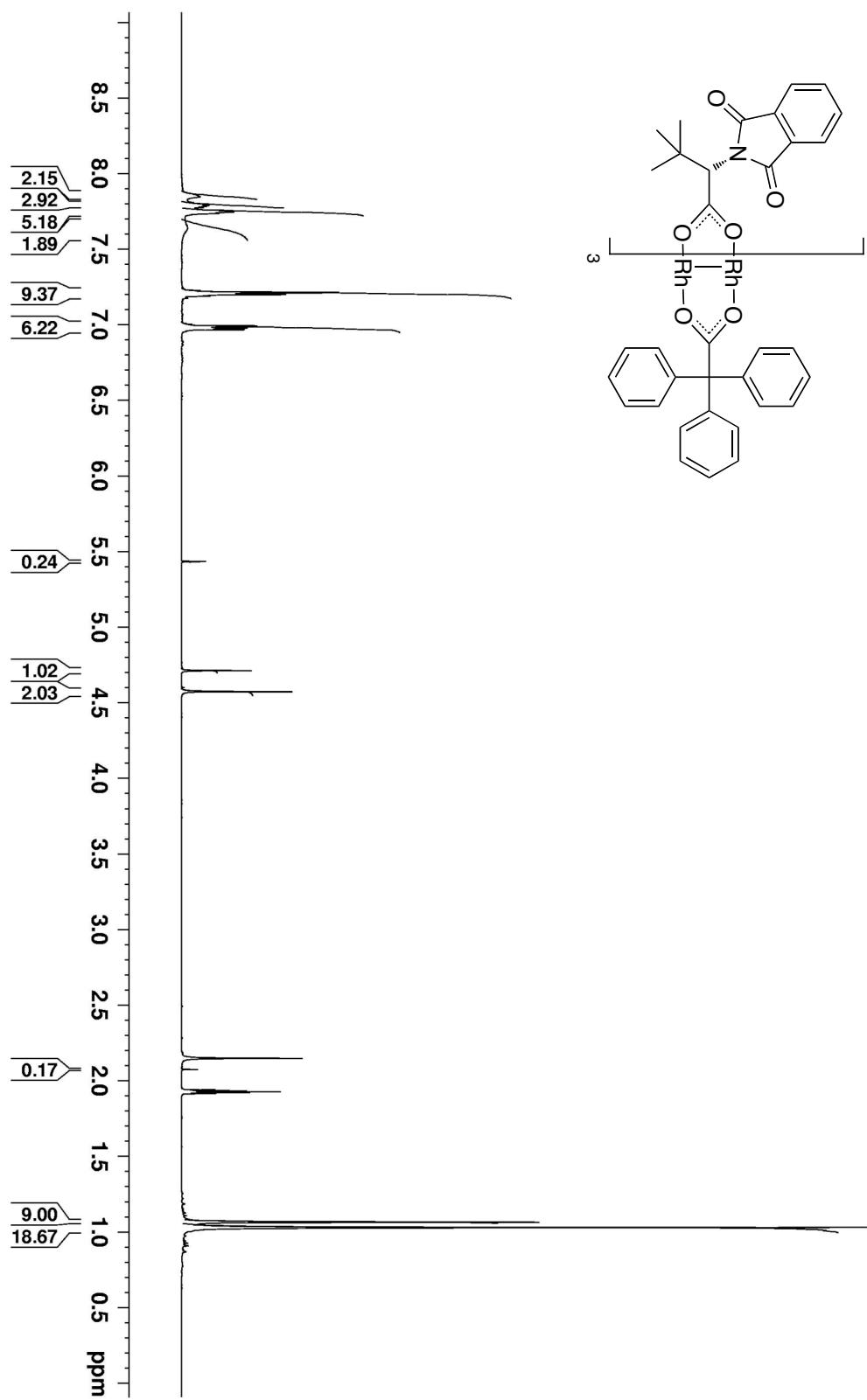
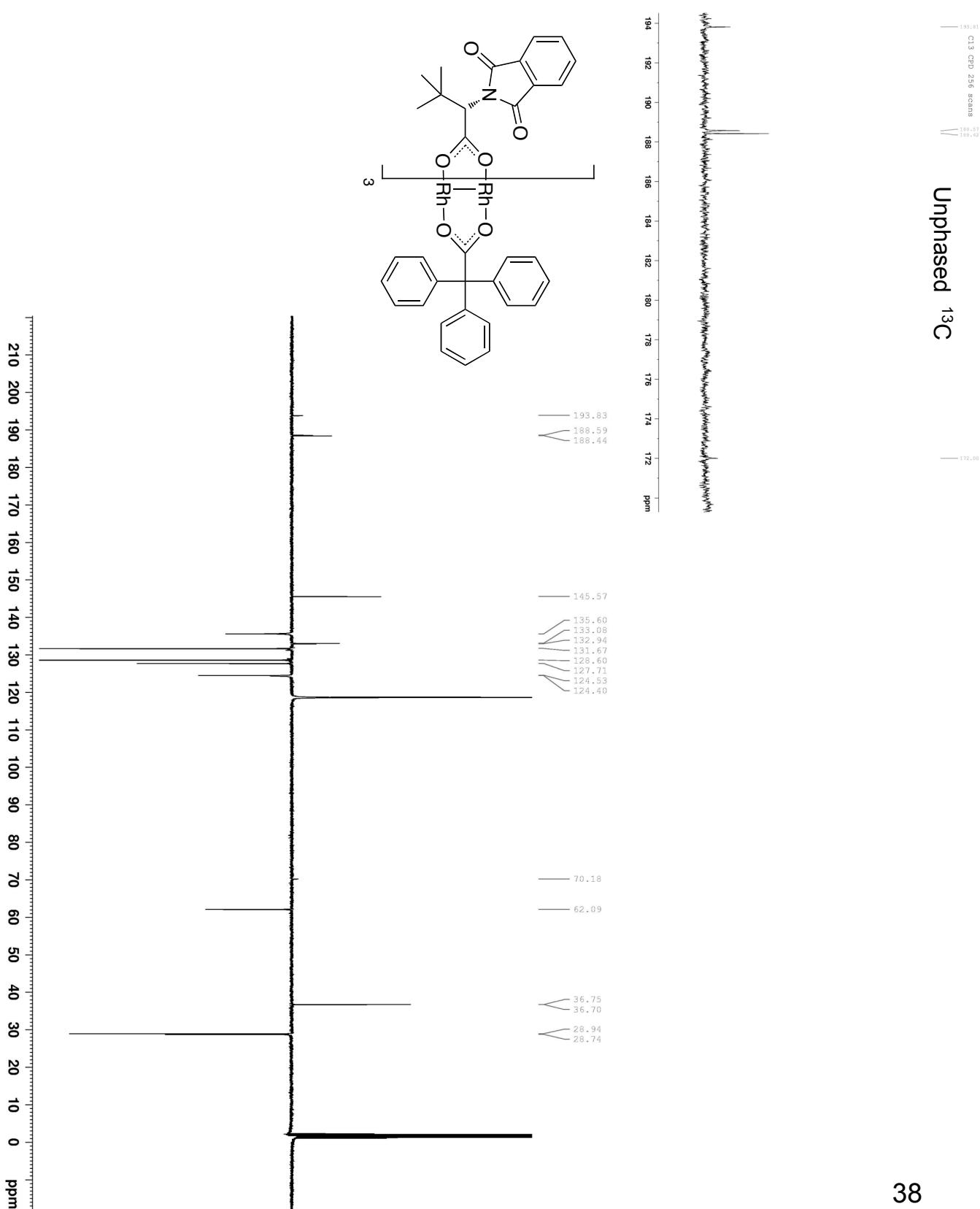


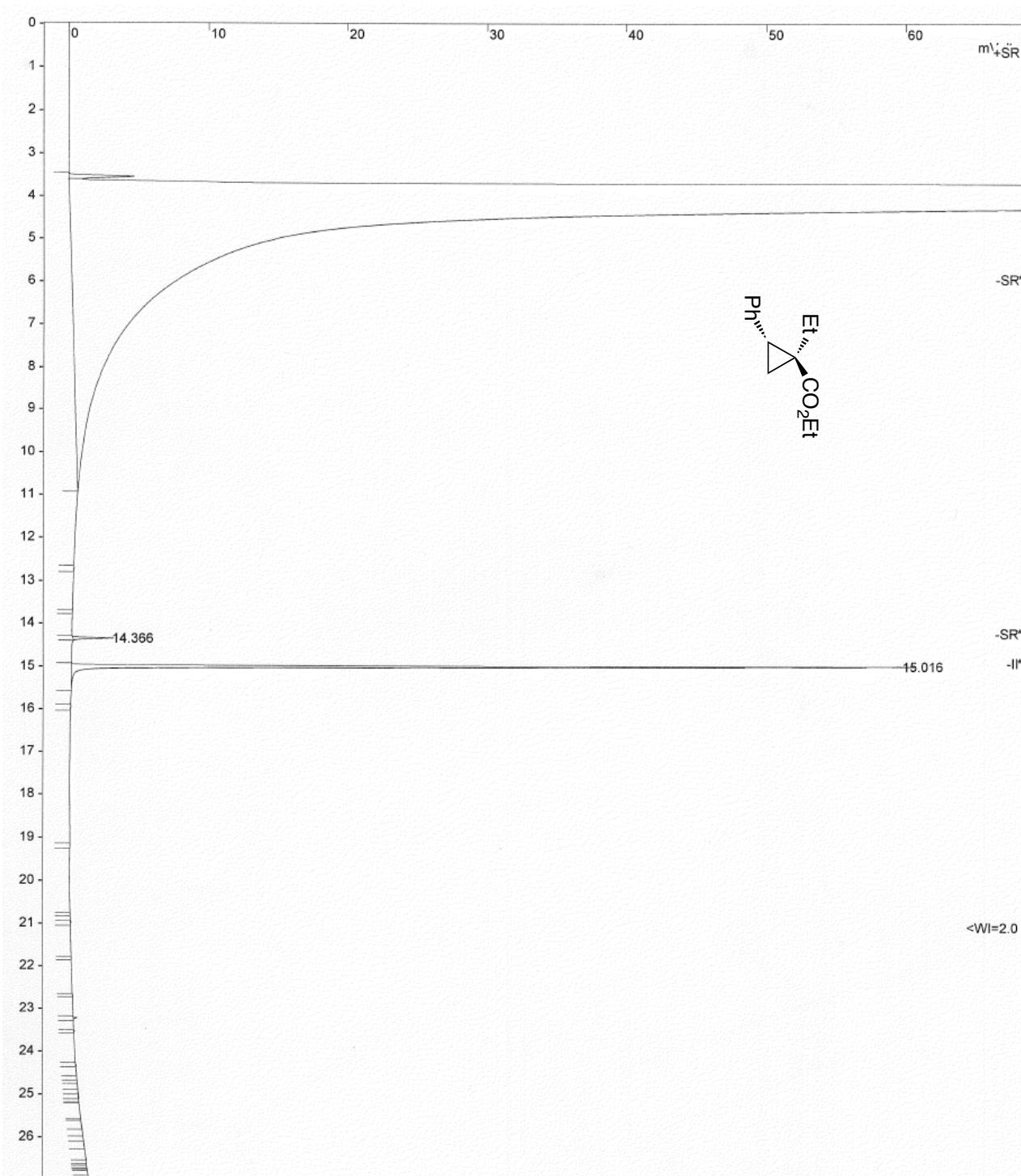
^1H NMR spectrum of A (400 MHz, CD_3CN)



^{13}C NMR spectrum of A (100 MHz, CD_3CN)

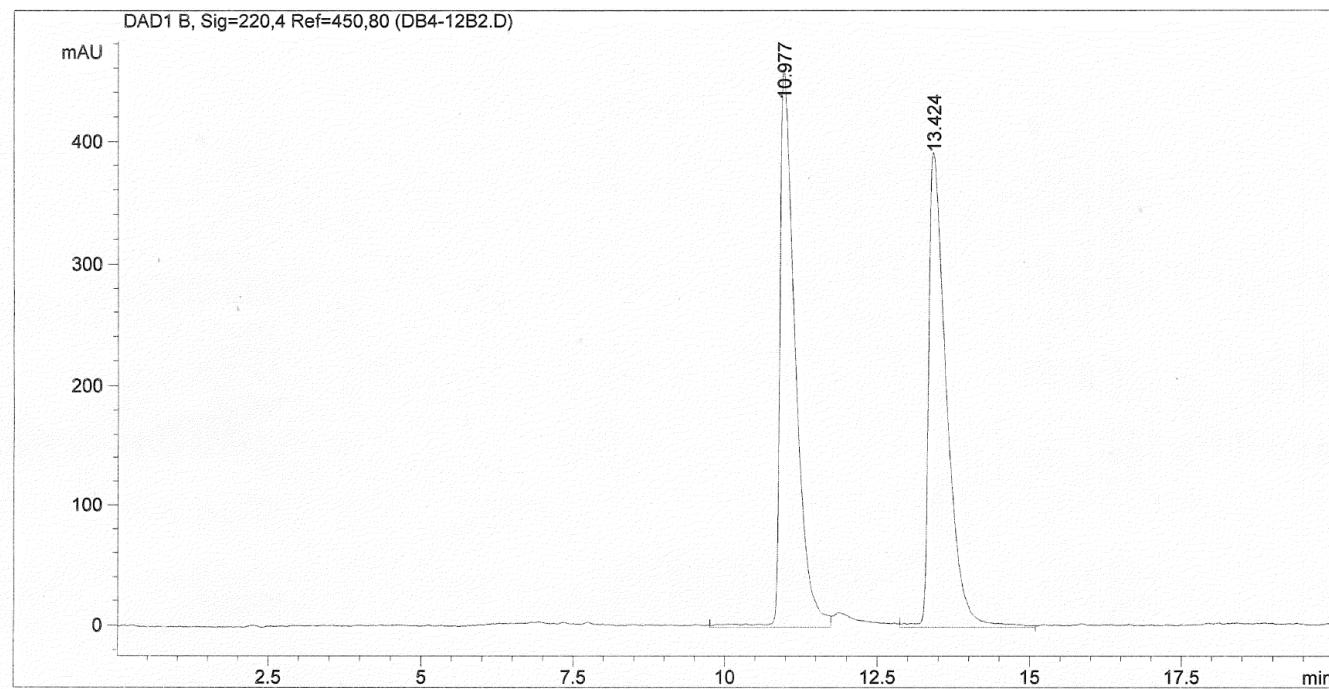


GC trace of **1** (Hewlett Packard HP5 column, 200 °C injection, 50–300 °C over 27 min)



Peak No.	Peak Name	Result (.)	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		4.2881	14.366	0.000	6865	BB	2.2	
2		95.7119	15.016	0.000	153238	BB	2.2	
Totals:		100.0000		0.000	160103			39

Racemic **1** (Chiracel IB column, 99.9:0.1 hexanes:isopropanol, 1 mL/min, 220nm)



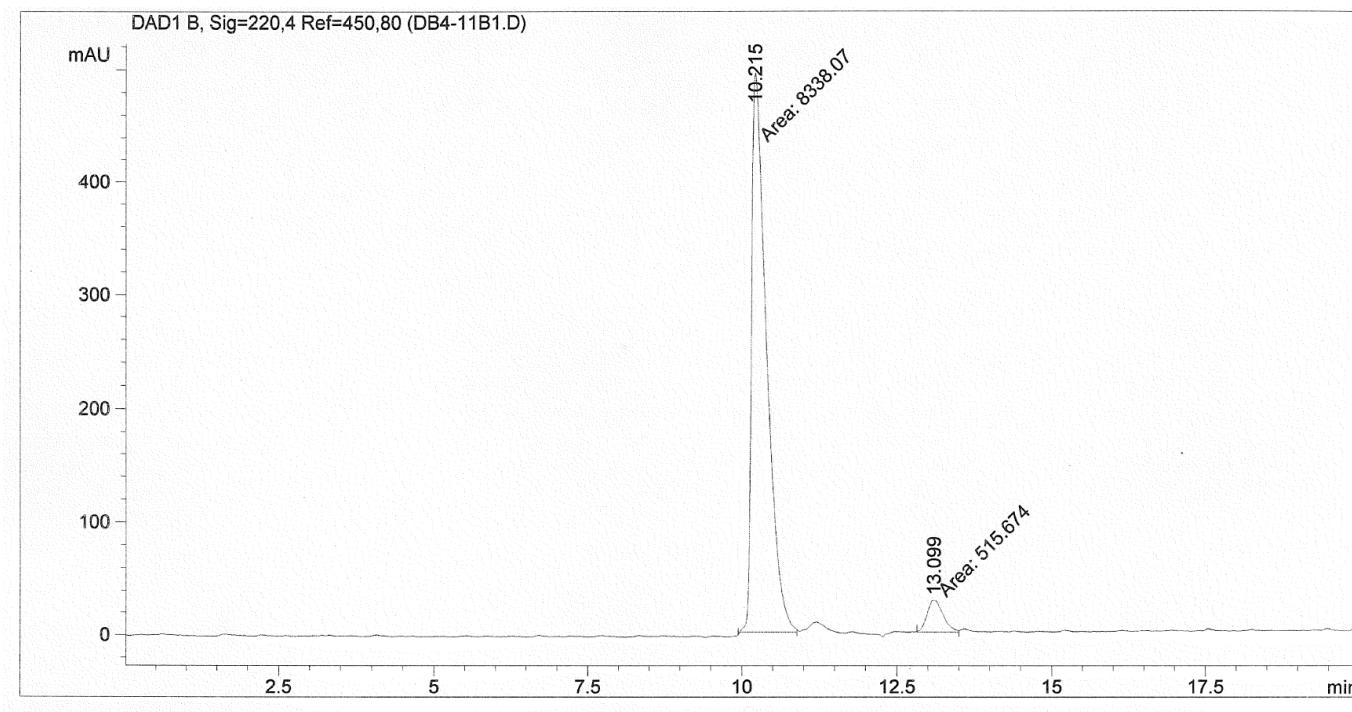
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Area Percent Report
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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 B, Sig=220,4 Ref=450,80

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.977	VV	0.2657	8153.20752	457.04047	50.3719
2	13.424	VB	0.3053	8032.83008	388.40359	49.6281

Enantiomeric excess of **1** (Chiracel IB column, 99.9:0.1 hexanes:isopropanol, 1 mL/min, 220nm)

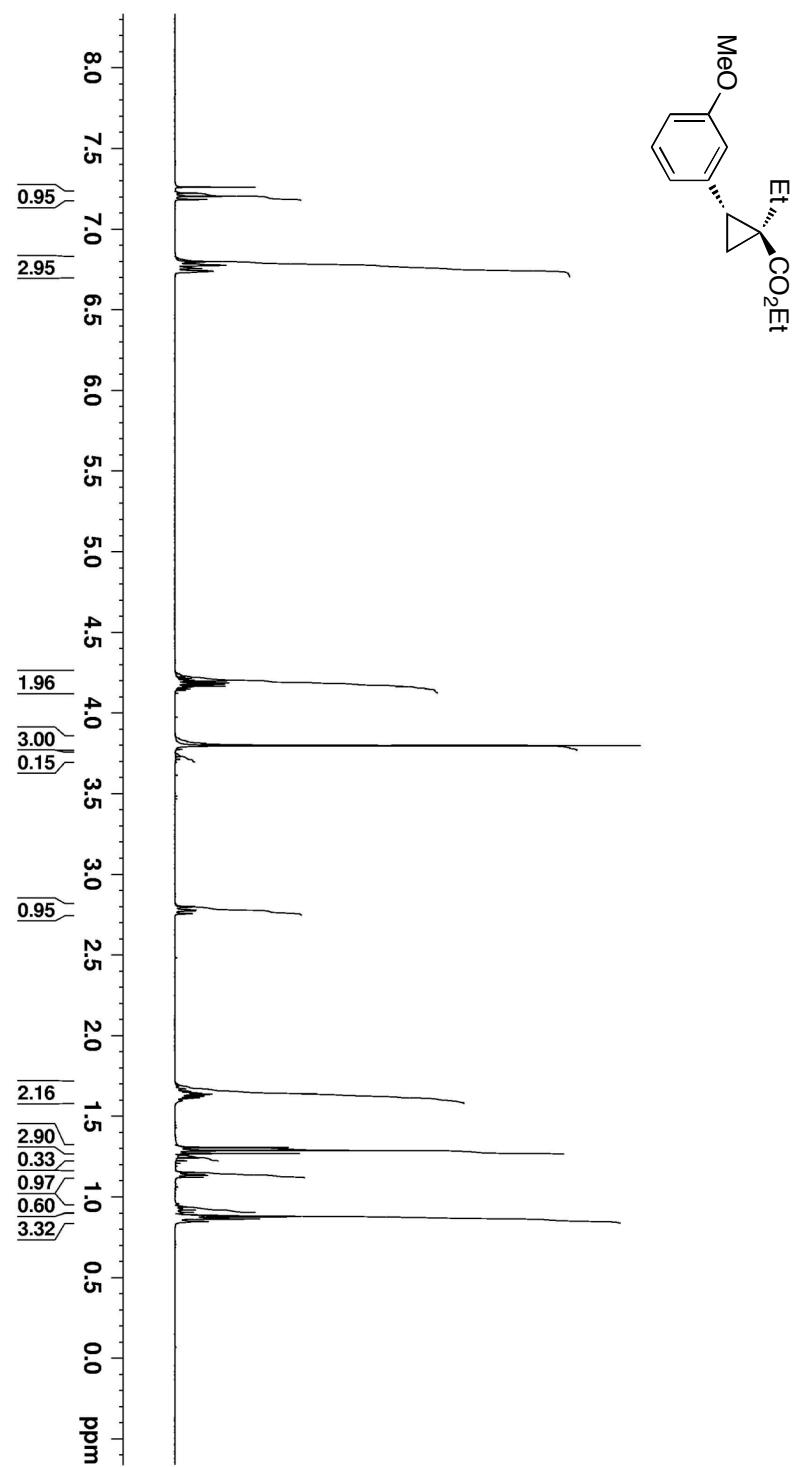


Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

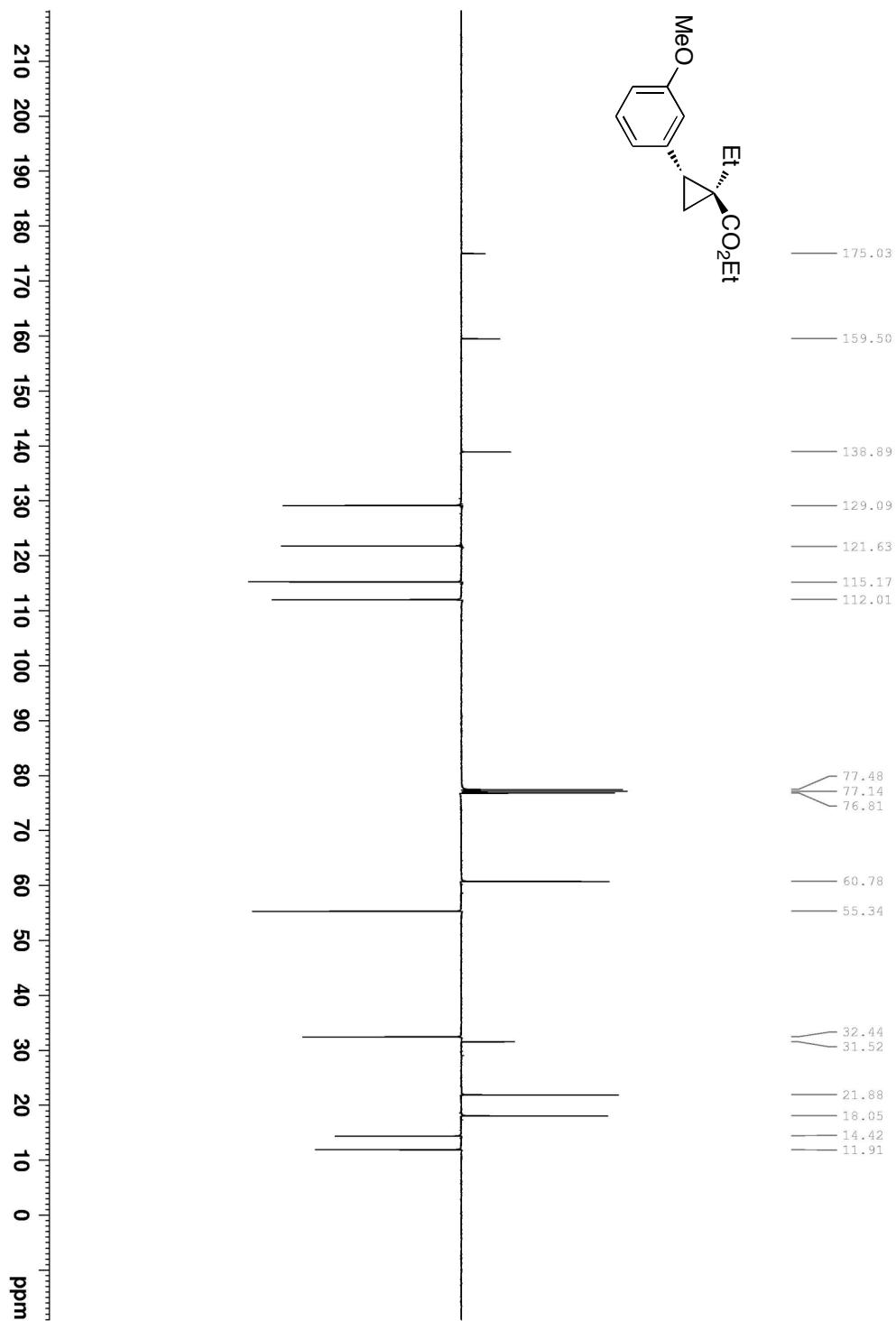
Signal 1: DAD1 B, Sig=220,4 Ref=450,80

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.215	MM	0.2808	8338.07227	494.97476	94.1756
2	13.099	MM	0.2966	515.67365	28.98072	5.8244

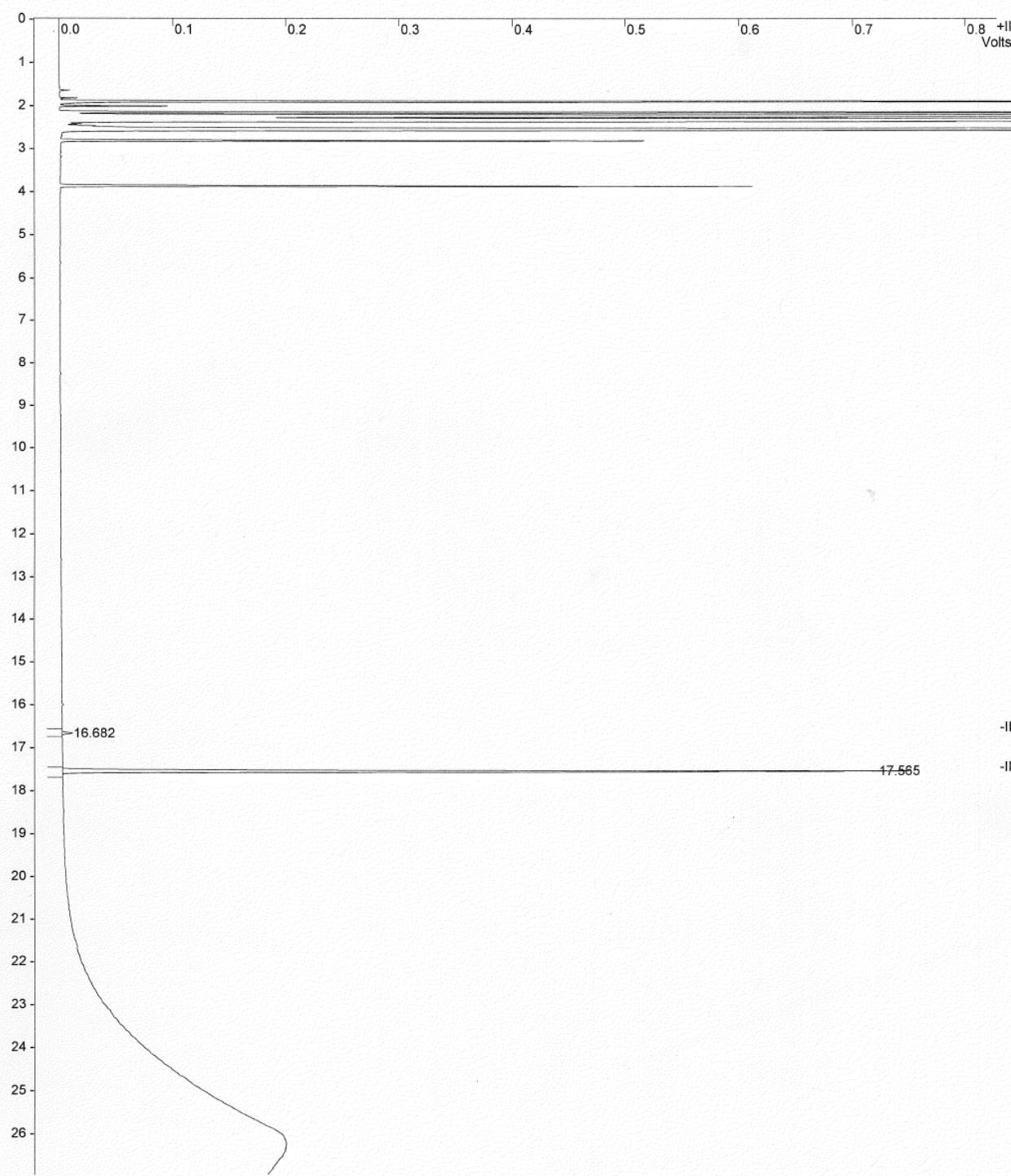
^1H NMR spectrum of **2** (400 MHz, CDCl_3)



^{13}C NMR spectrum of **2** (100 MHz, CDCl_3)

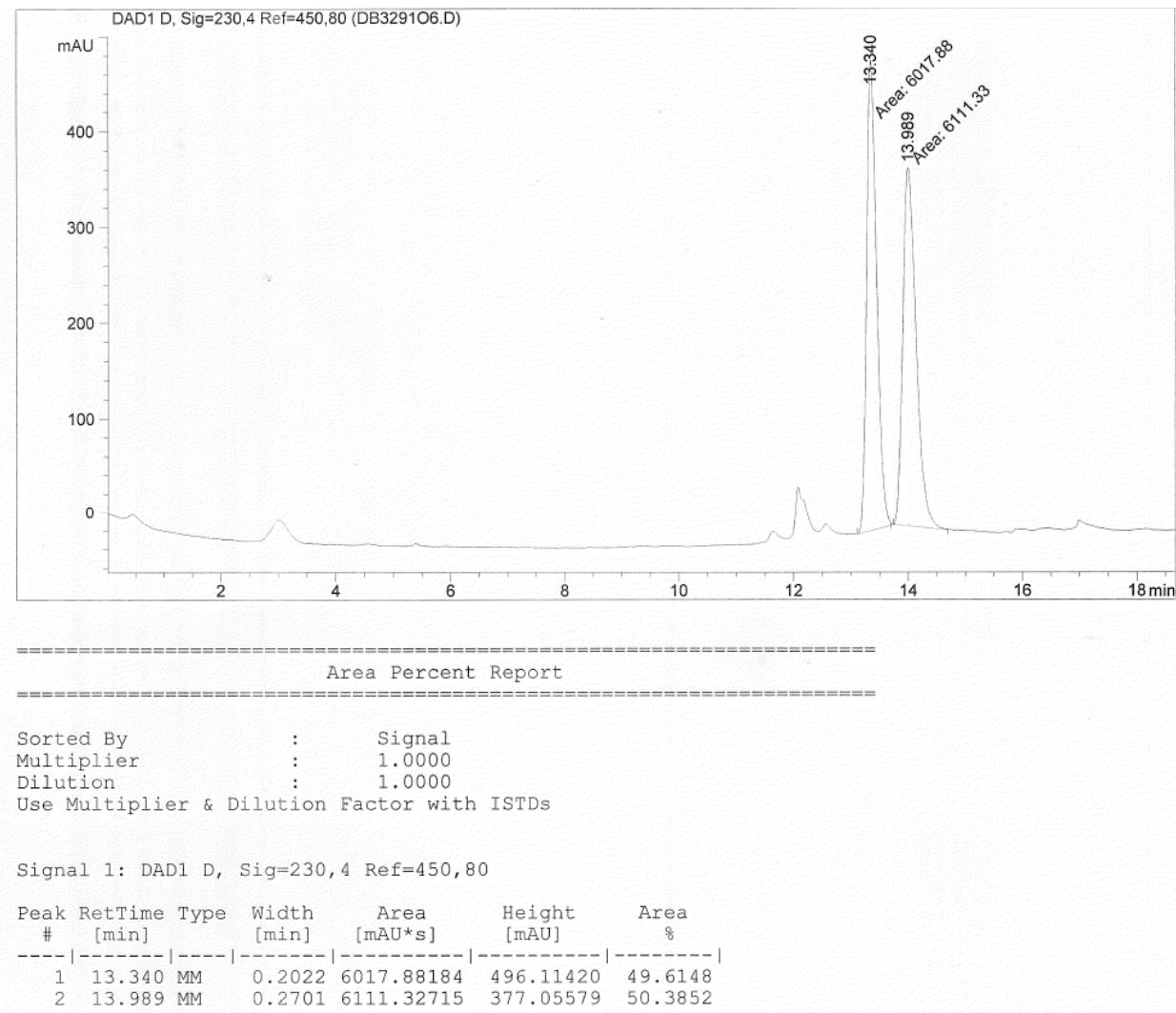


GC trace of **2** (Hewlett Packard HP1 column, 200 °C injection, 50–300 °C over 27 min)

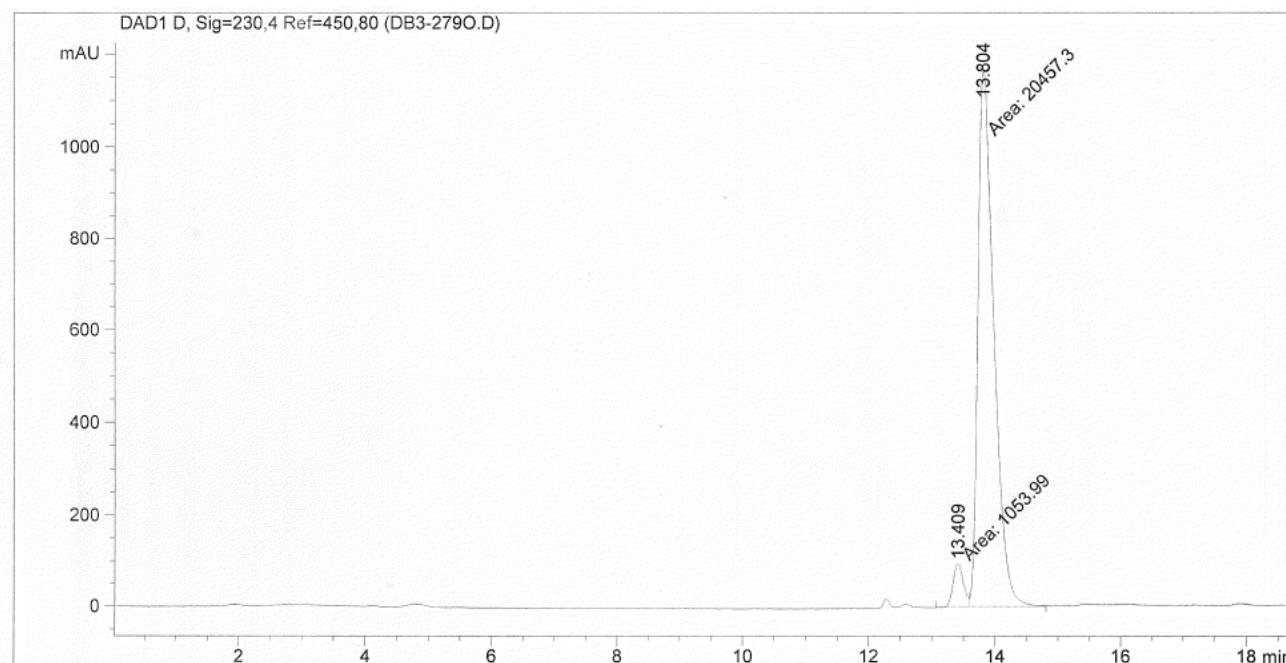


Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		1.3279	16.682	0.000	27307	BB	2.7	
2		98.6721	17.565	0.000	2029158	BB	2.5	
Totals:		100.0000		0.000	2056465			

HPLC resolution of racemic **2** (Chiracel OJ-H column, 99.9:0.1-90:10 hexanes:isopropanol over 120 min., 1 mL/min, 230 nm)



Enantiomeric excess of **2** (Chiracel OJ-H column, 99.9:0.1-90:10 hexanes:isopropanol over 120 min., 1 mL/min, 230 nm)



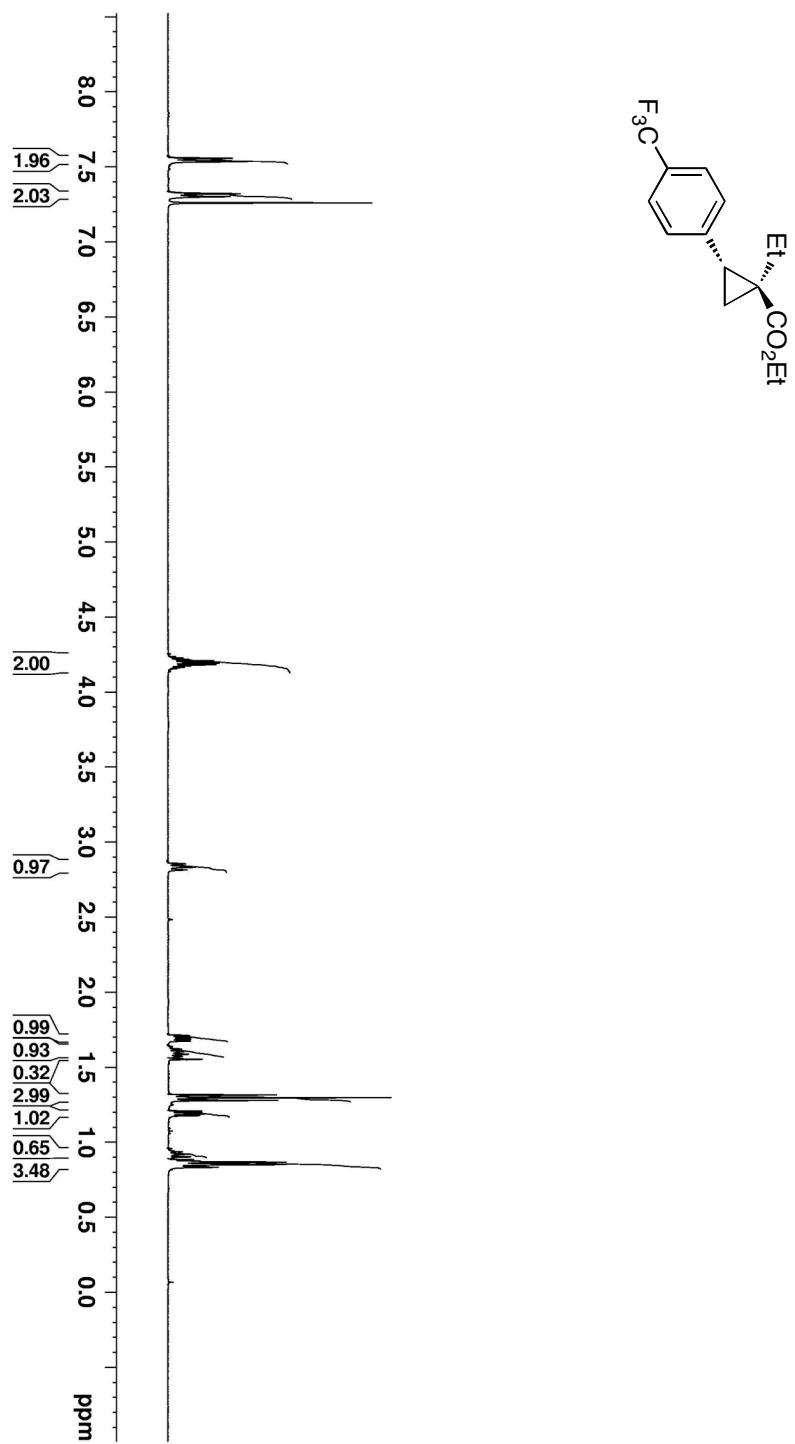
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Area Percent Report
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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

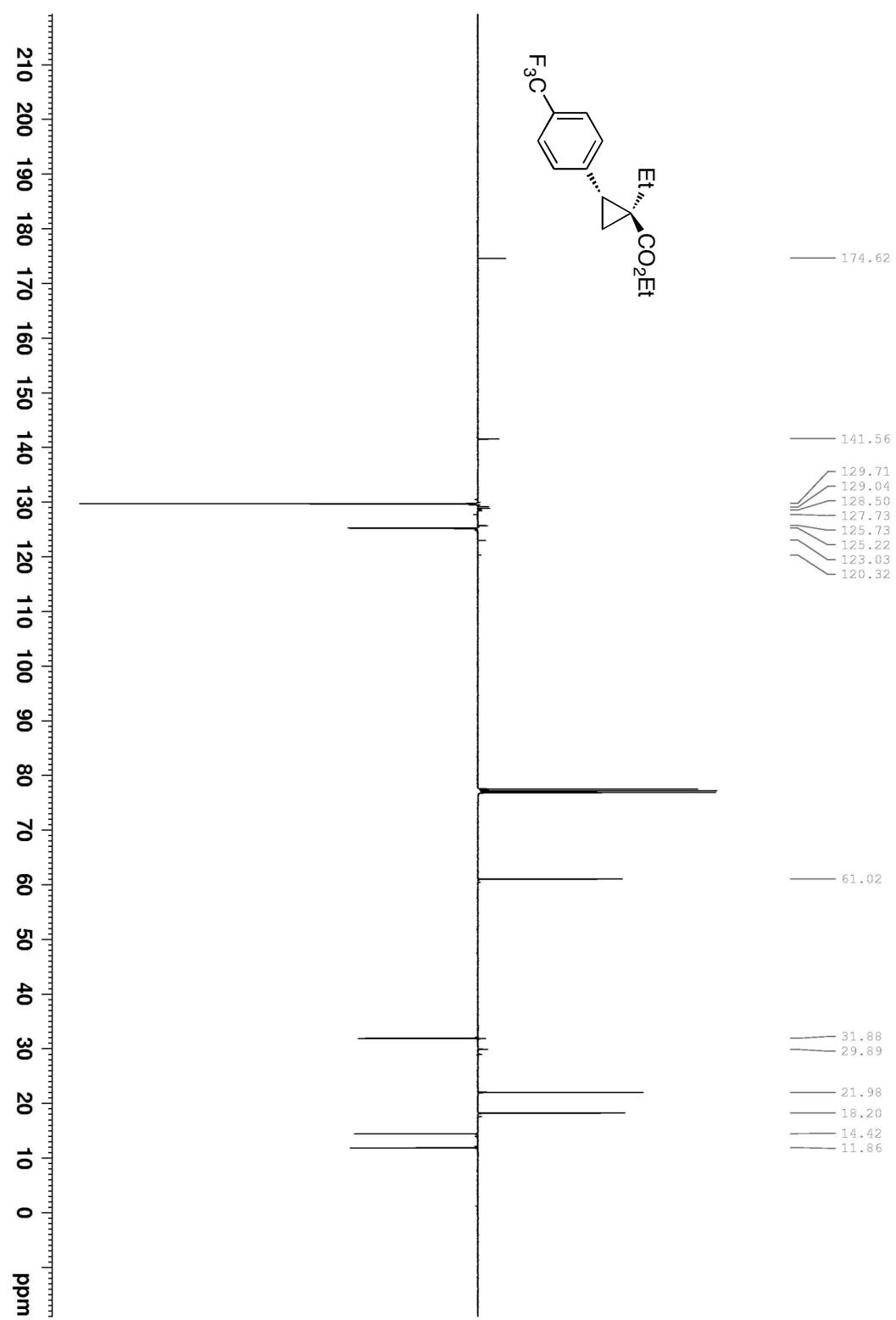
Signal 1: DAD1 D, Sig=230,4 Ref=450,80

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.409	MF	0.1868	1053.99170	94.05535	4.8997
2	13.804	FM	0.2919	2.04573e4	1167.98035	95.1003

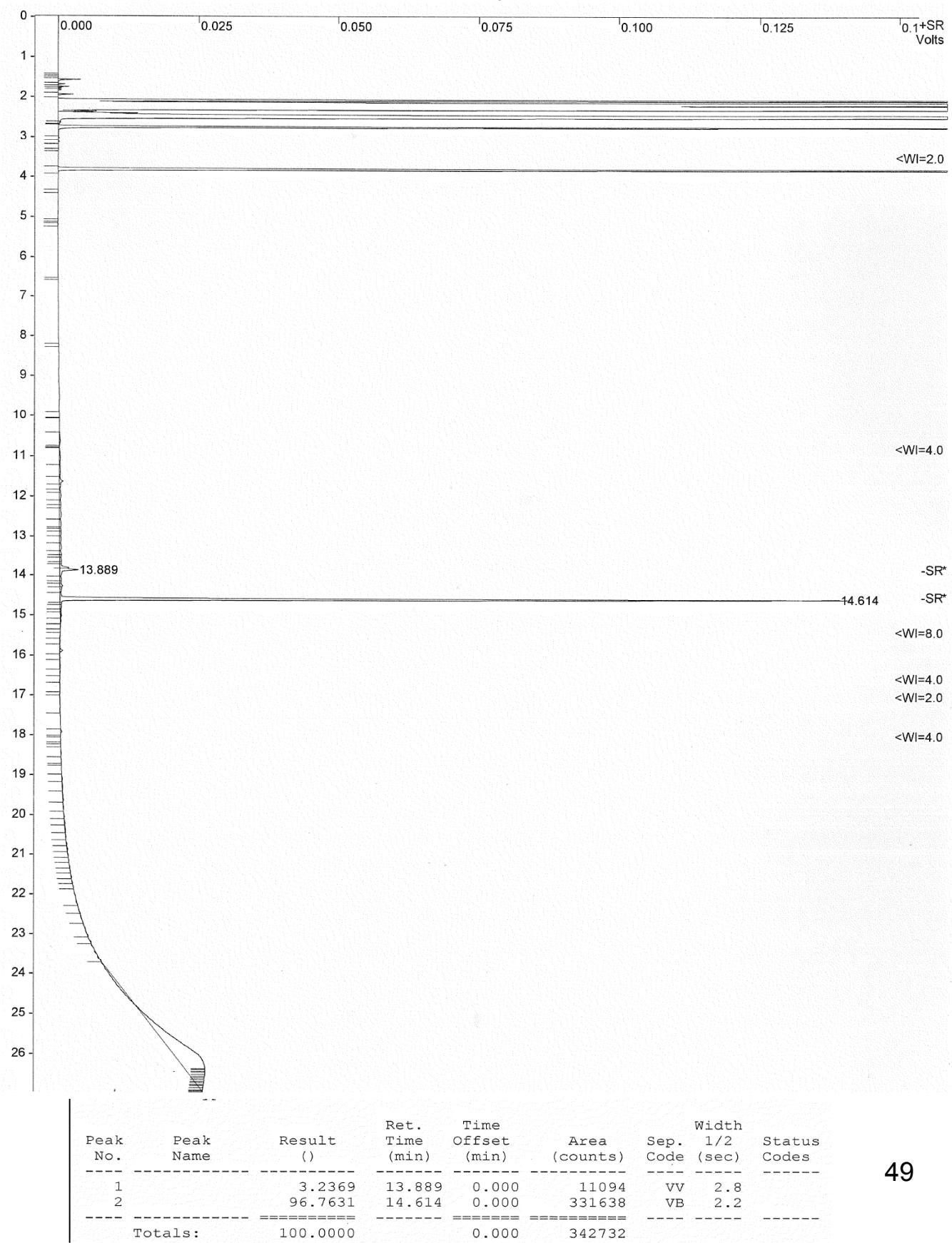
¹H NMR spectrum of **3** (400 MHz, CDCl₃)



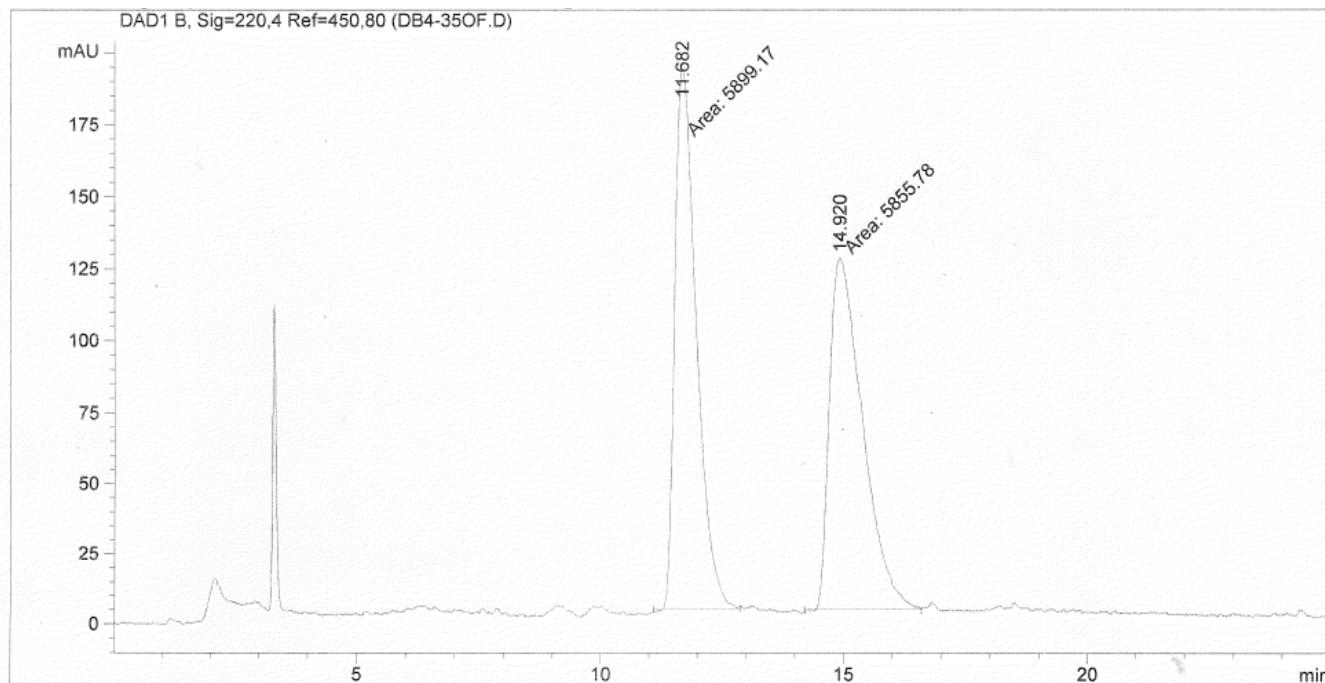
^{13}C NMR spectrum of **3** (100 MHz, CDCl_3)



GC trace of **3** (Hewlett Packard HP1 column, 200 °C injection, 50–300 °C over 27 min)



HPLC resolution of racemic **3** (Chiracel OF column, 99.9:0.1 hexanes:isopropanol, 1 mL/min, 220 nm)



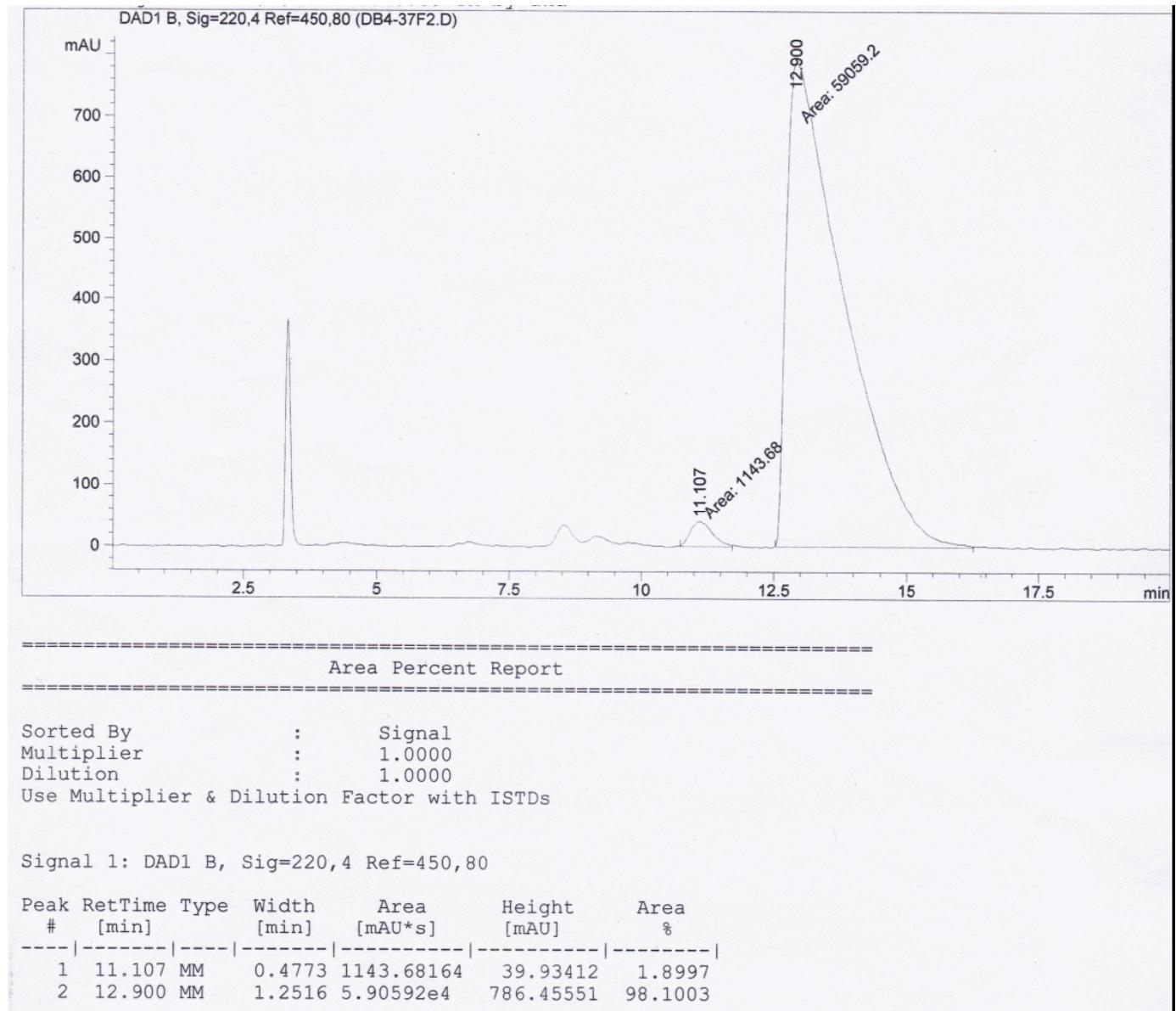
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Area Percent Report
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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

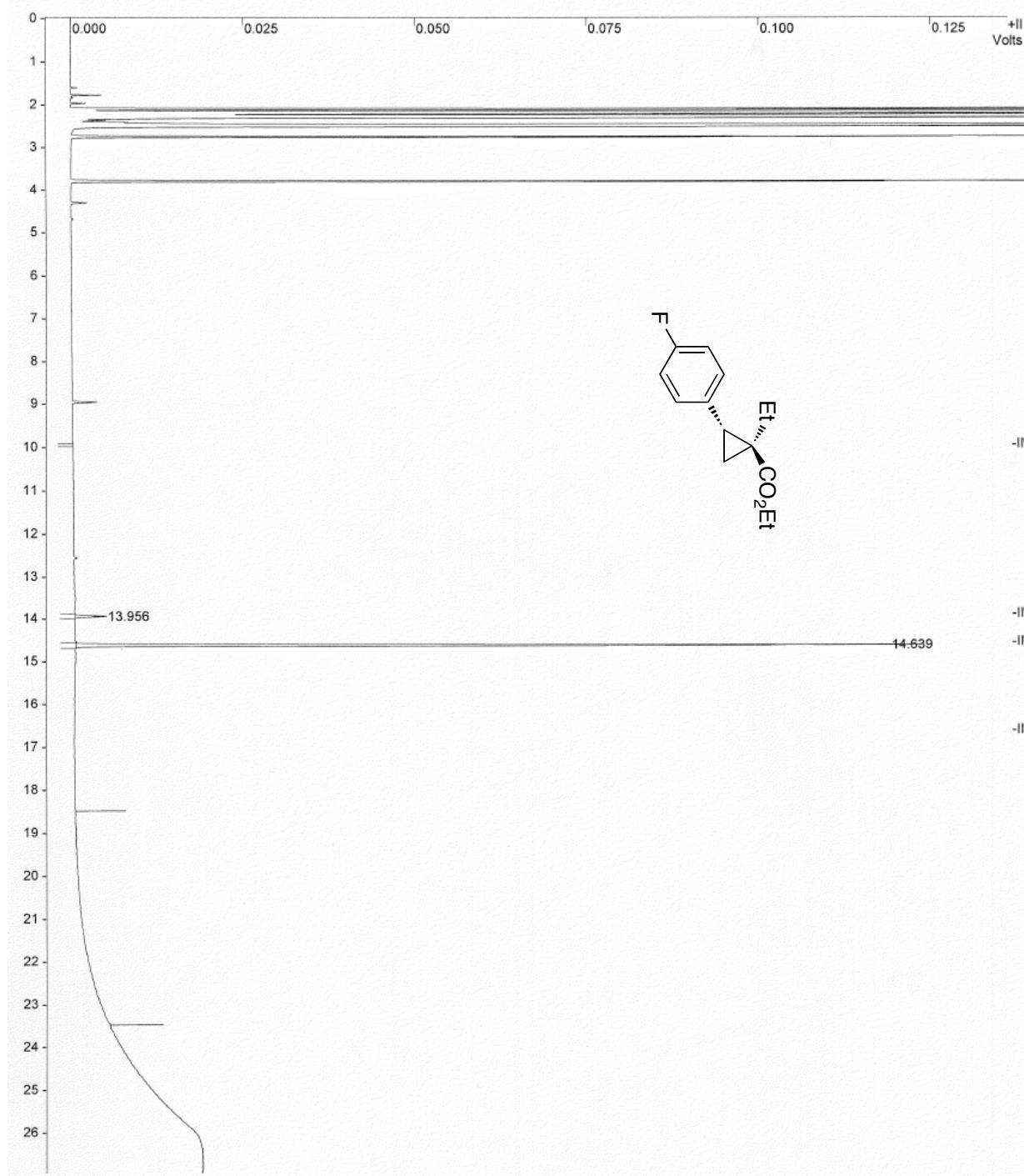
Signal 1: DAD1 B, Sig=220,4 Ref=450,80

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.682	MM	0.5189	5899.17432	189.46356	50.1846
2	14.920	MM	0.7882	5855.78027	123.81608	49.8154

Enantiomeric excess of **3** (Chiracel OF column, 99.9:0.1 hexanes:isopropanol, 1 mL/min,
220nm)

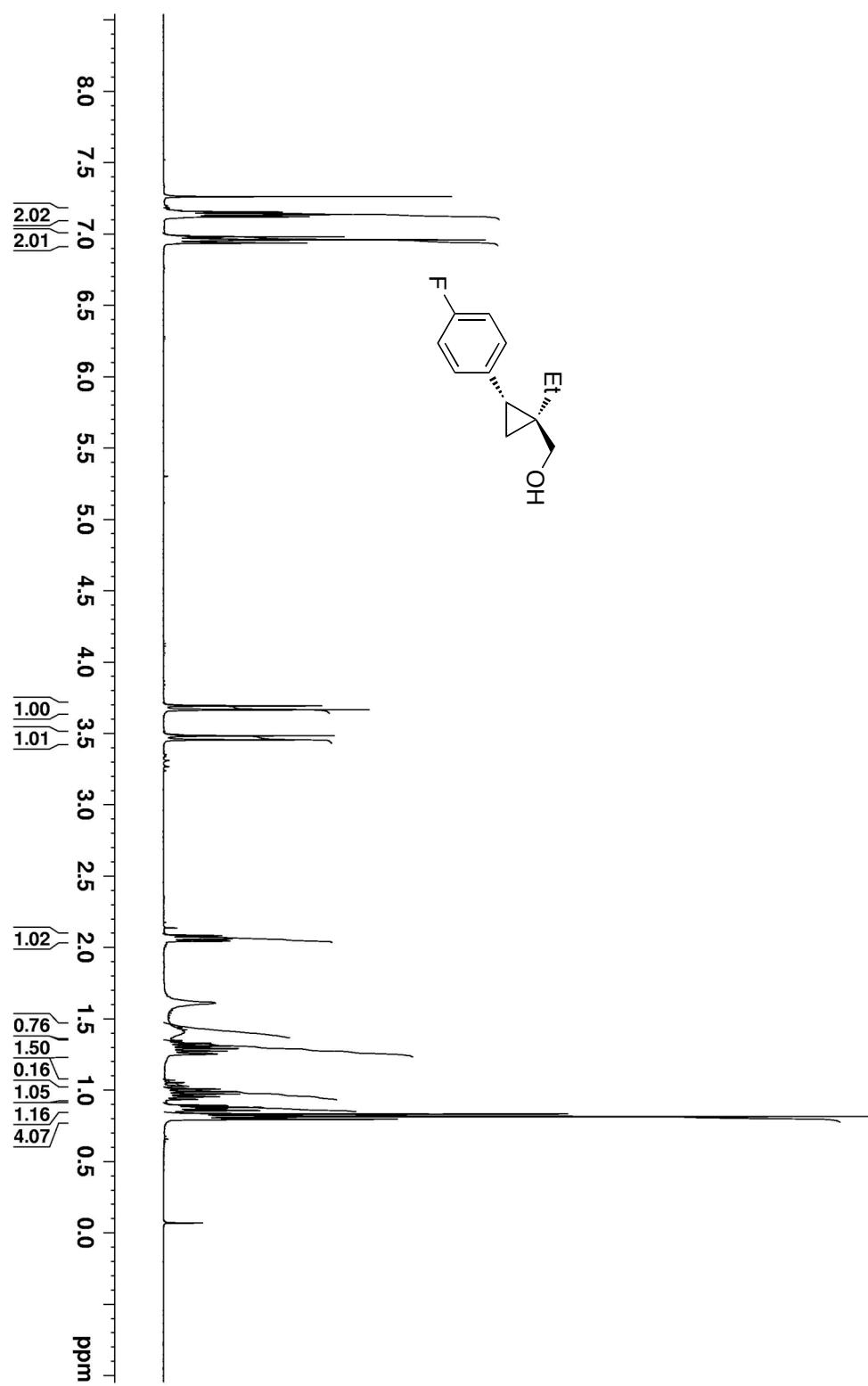


GC trace of **4** (Hewlett Packard HP1 column, 200 °C injection, 50–300 °C over 27 min)

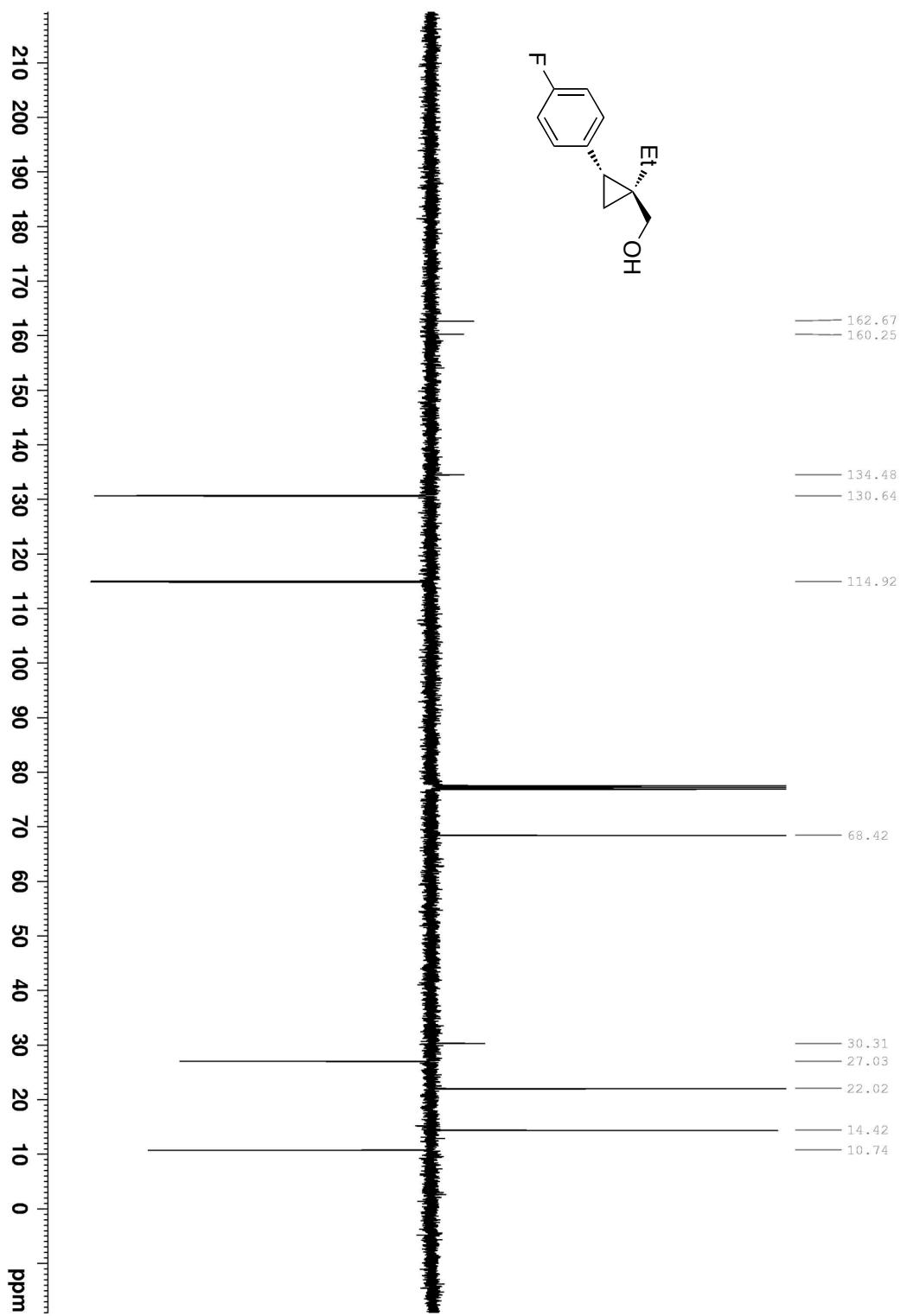


Peak No.	Peak Name	Result (')	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		3.4832	13.956	0.000	10384	BB	2.1	
2		96.5168	14.639	0.000	287717	BB	2.2	
Totals:		100.0000		0.000	298101			

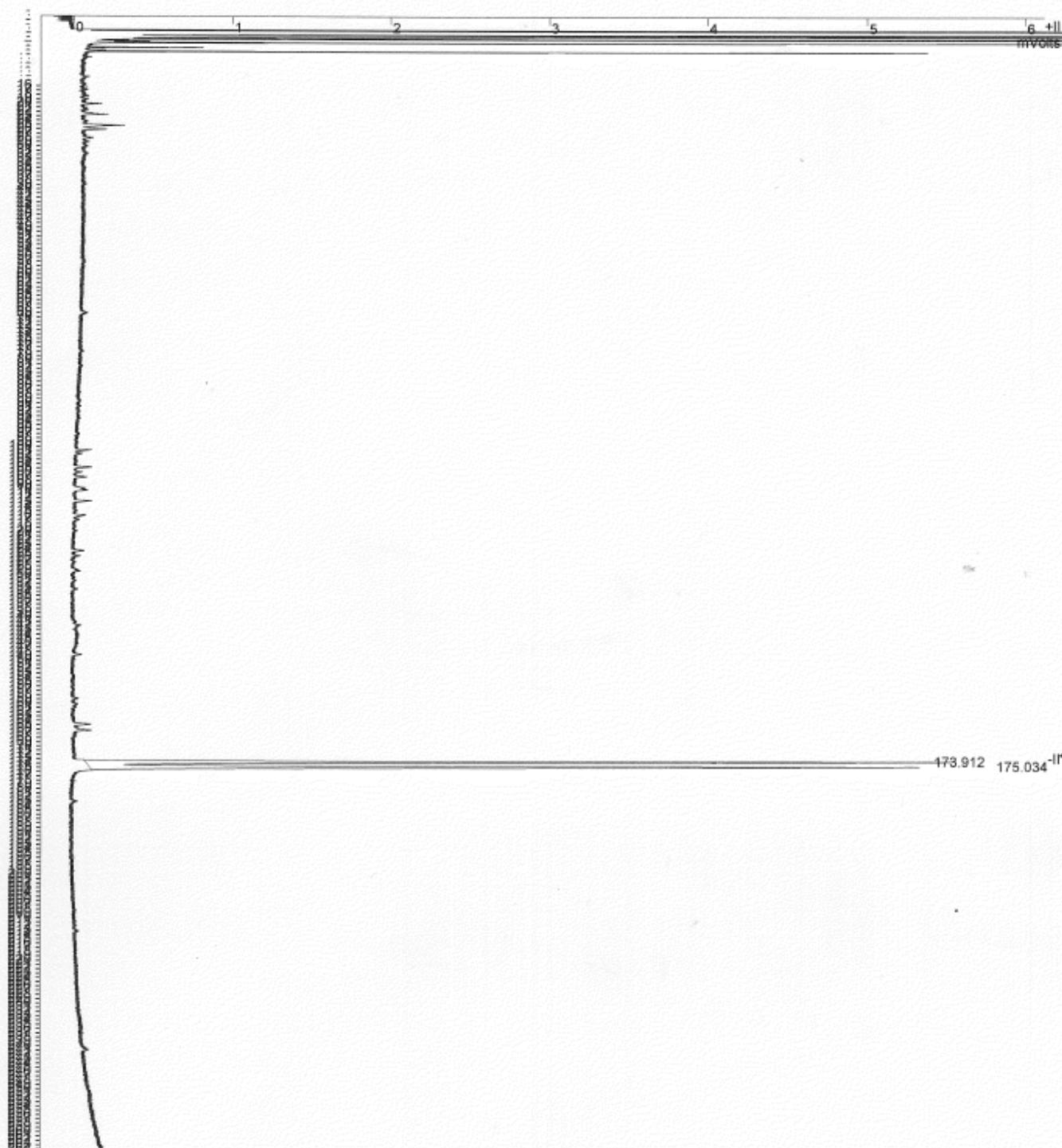
¹H NMR spectrum of **4a** (400 MHz, CDCl₃)



^{13}C NMR spectrum of **4a** (100 MHz, CDCl_3)

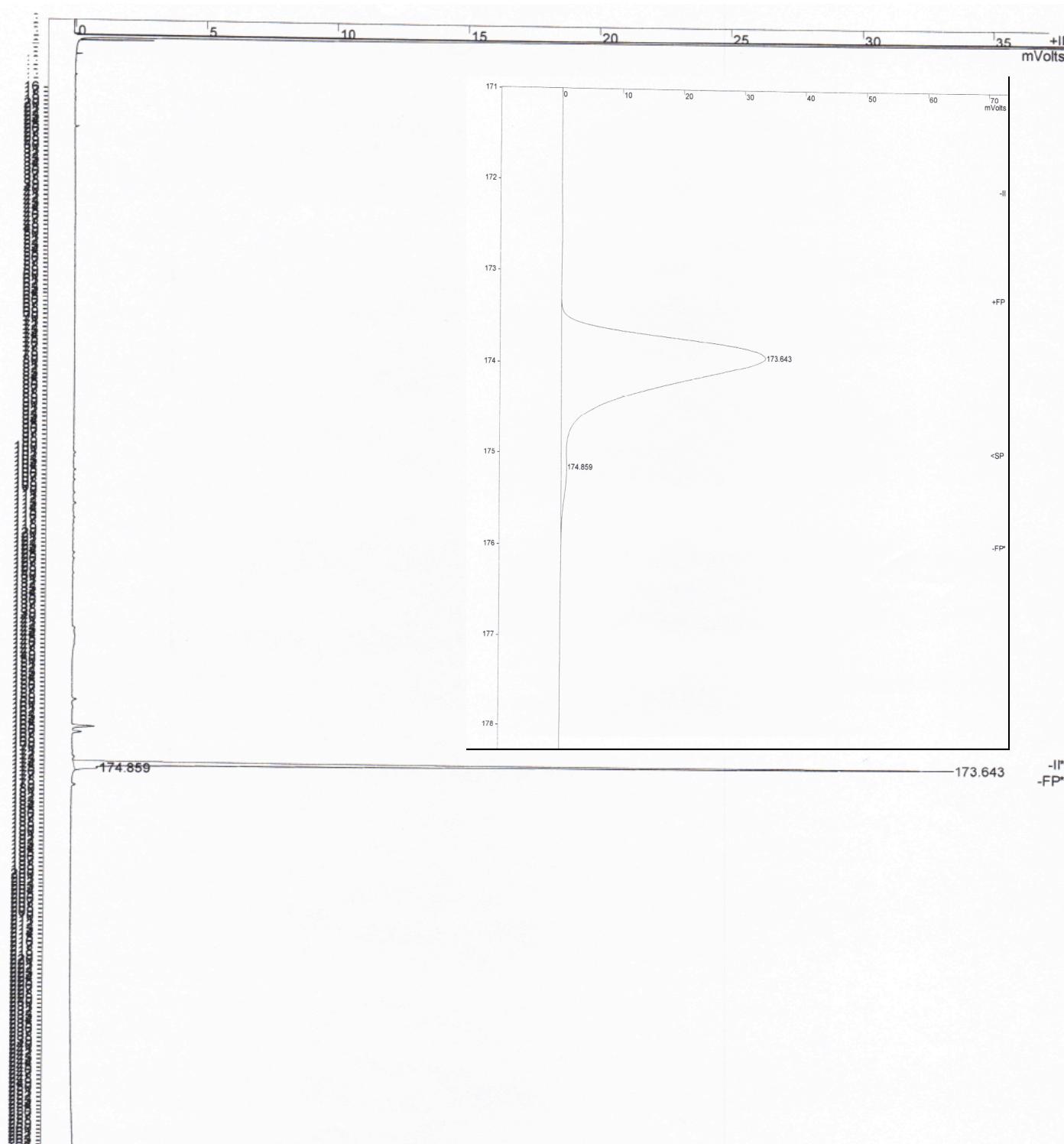


Chiral GC resolution of racemic **4a** (Chiraldex G-TA column, 200 °C injection, 50–180 °C over 135 min)



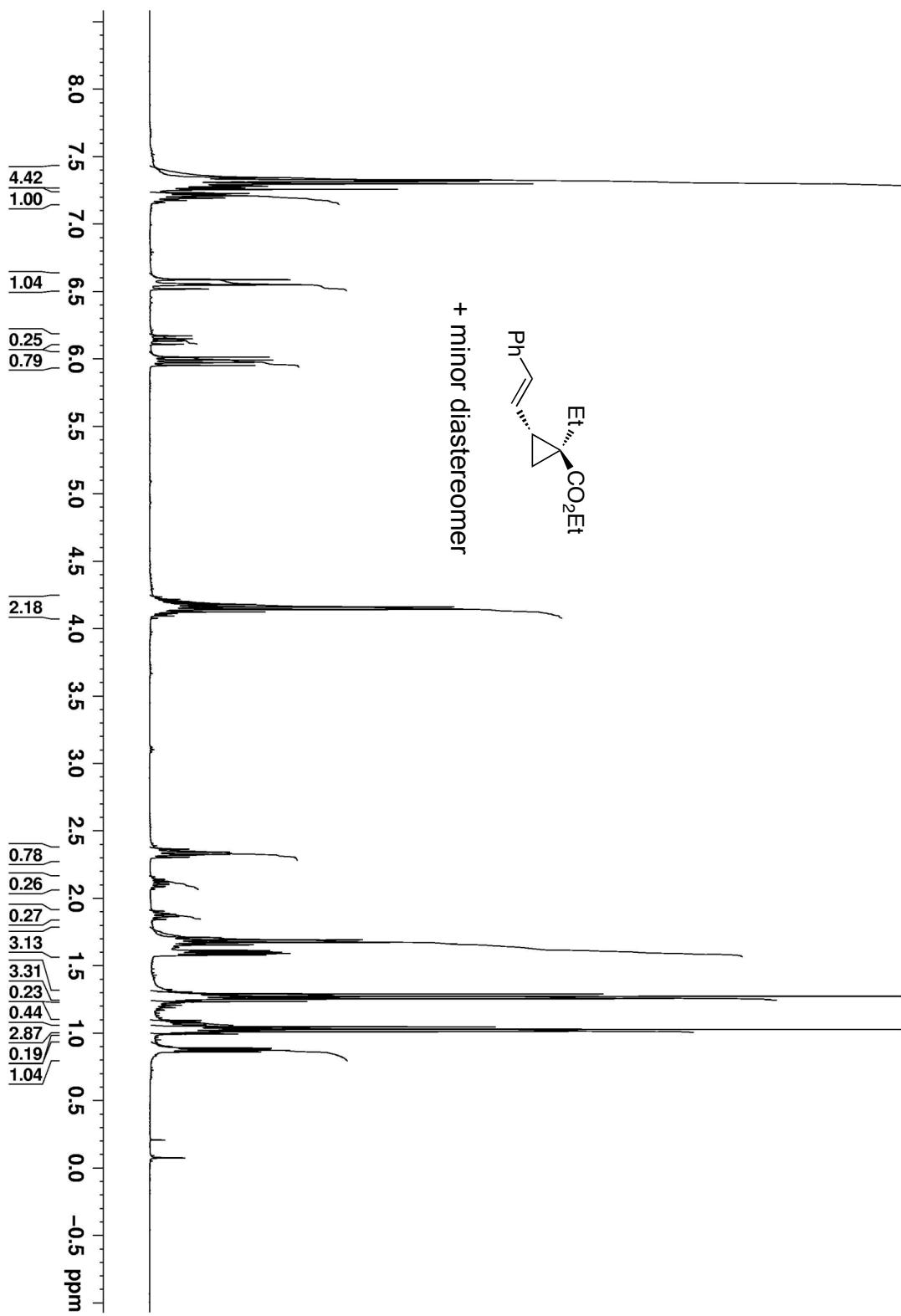
Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	1/2 (sec)	Status Codes
1		49.6322	173.912	0.000	147106	BV	24.7	
2		50.3678	175.034	0.000	149286	VB	26.4	
Totals:		100.0000		0.000	296392			

Enantiomeric excess of **4a** (Chiraldex G-TA column, 200 °C injection, 50–180 °C over 135 min)

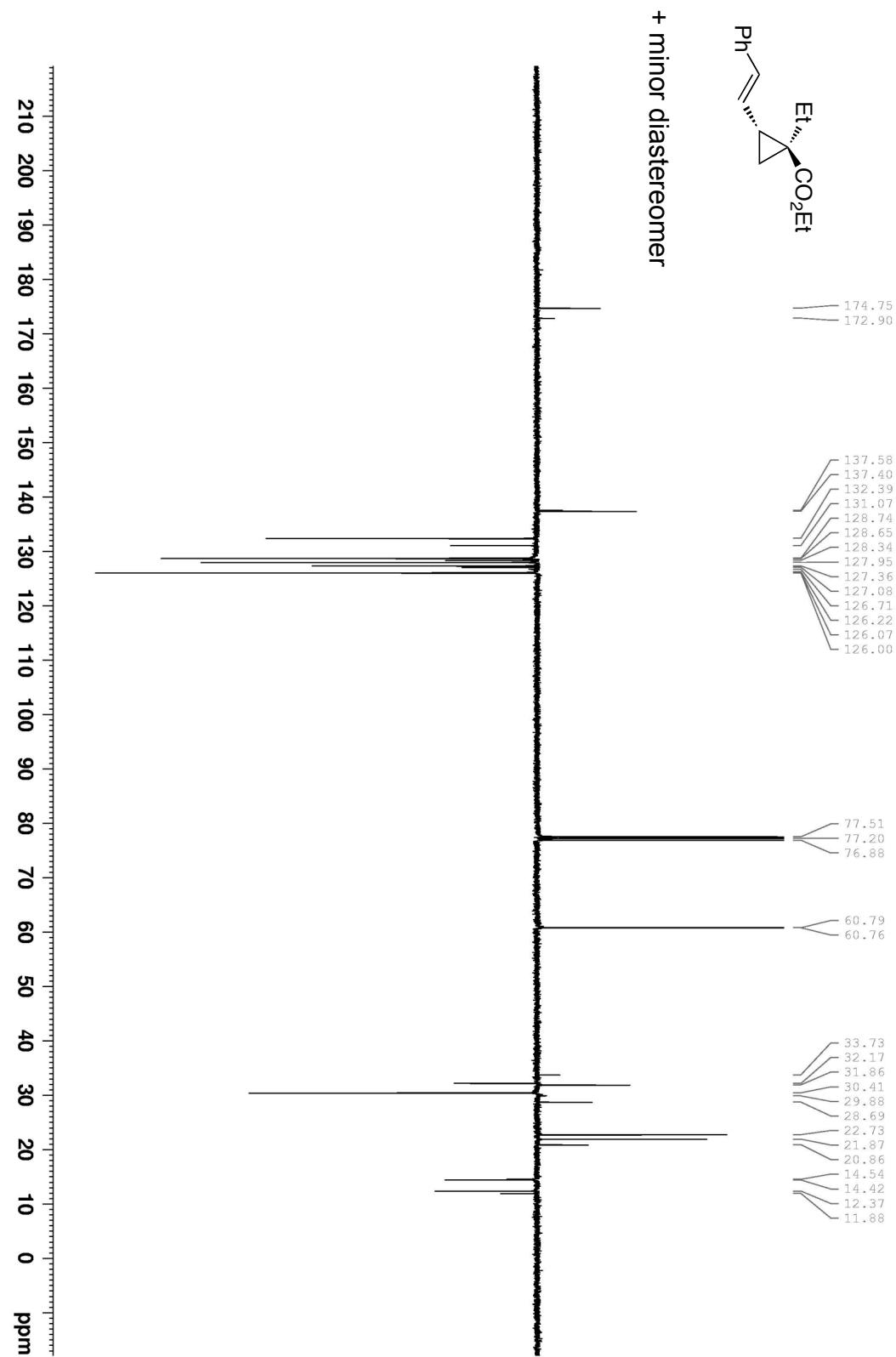


Peak No.	Peak Name	Result (')	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		97.5296	173.643	0.000	1161249	BV	0.0	
2		2.4704	174.859	0.000	29414	VB	4.2	
Totals:		100.0000		0.000	1190663			

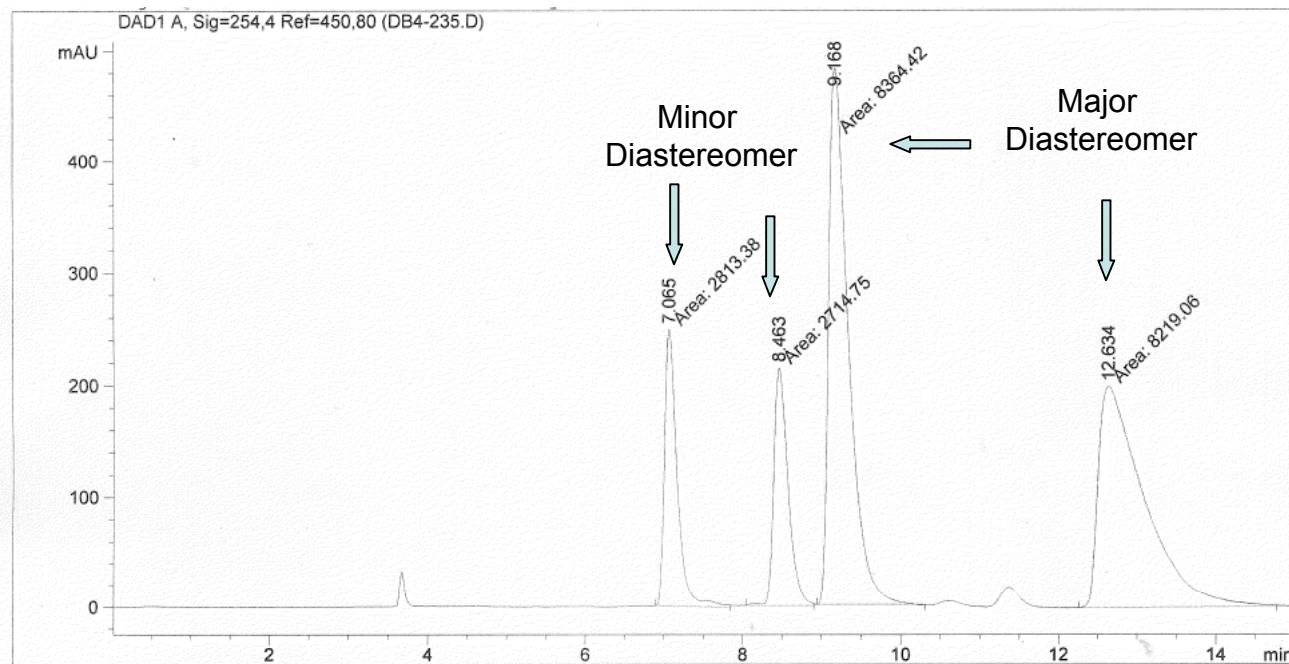
¹H NMR spectrum of **5** (400 MHz, CDCl₃)



^{13}C NMR spectrum of **5** (100 MHz, CDCl_3)



HPLC resolution of racemic **5** (Chiracel IA column, 99.9:0.1 hexanes:isopropanol, 1 mL/min, 254 nm)



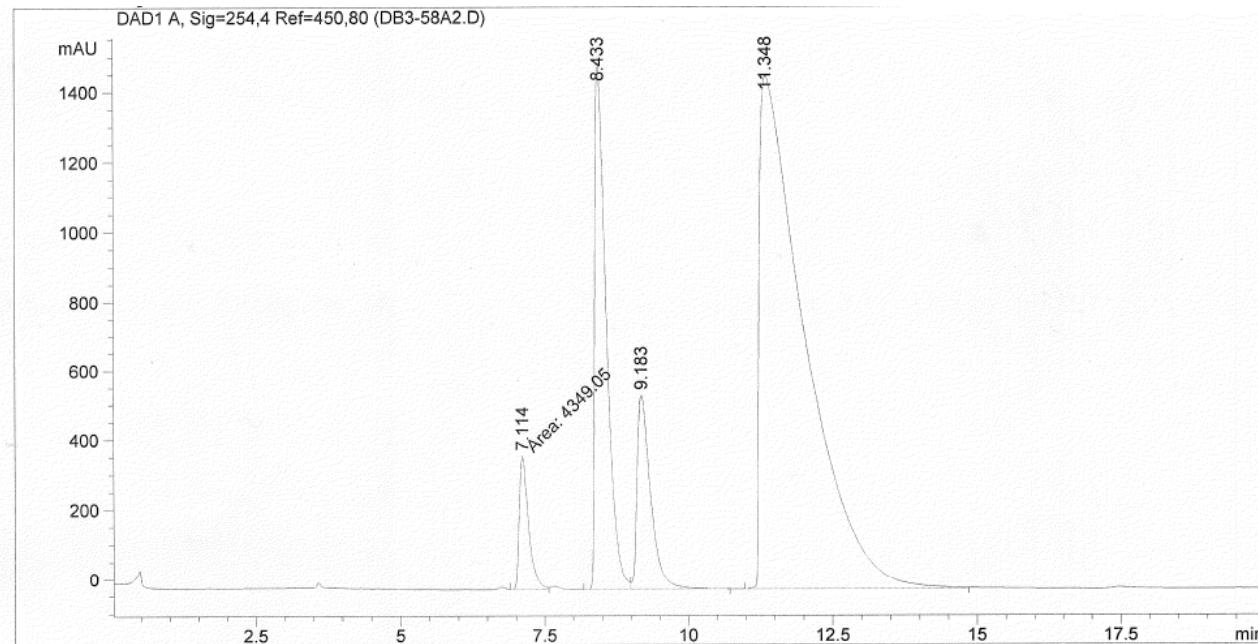
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Area Percent Report
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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=450,80

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.065	MM	0.1879	2813.38208	249.58528	12.7236
2	8.463	MM	0.2104	2714.75269	215.04797	12.2775
3	9.168	MM	0.2894	8364.41699	481.76370	37.8282
4	12.634	MM	0.6864	8219.05664	199.57355	37.1708

HPLC of **5** (Chiracel IA column, 99.9:0.1 hexanes:isopropanol, 1 mL/min,
254nm)



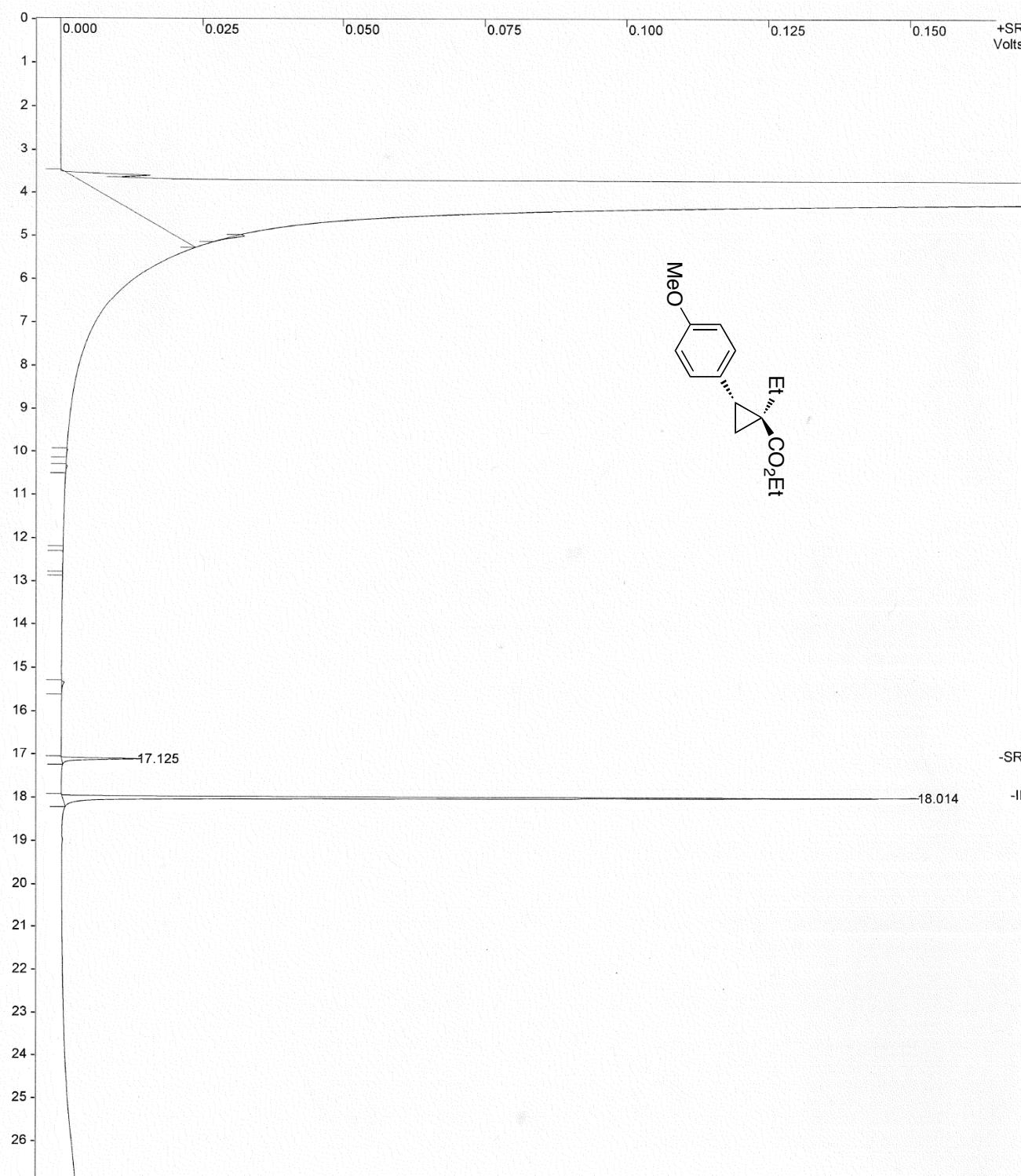
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Area Percent Report
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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=450,80

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.114	MM	0.1886	4349.04980	384.35138	3.7872
2	8.433	VV	0.2006	2.10179e4	1511.60791	18.3024
3	9.183	VB	0.2432	9198.65234	558.88684	8.0102
4	11.348	PV	0.6832	8.02710e4	1474.23840	69.9002

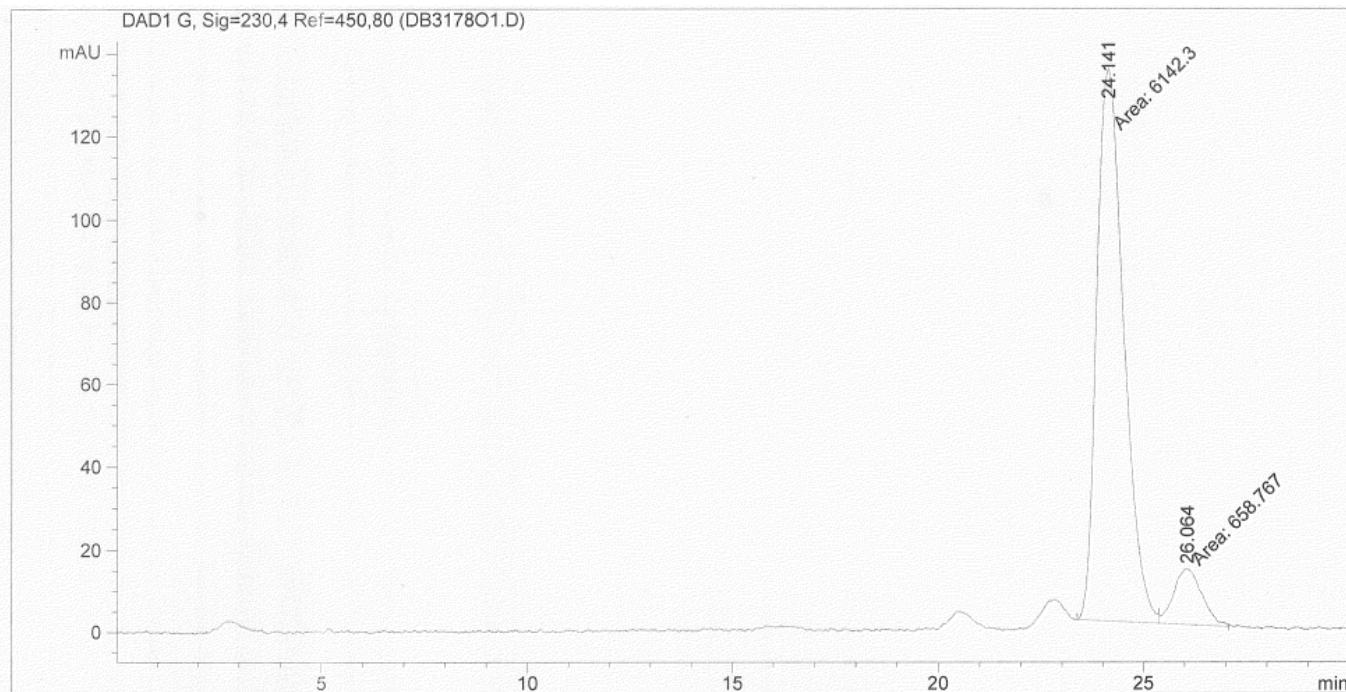
GC trace of **6** (Hewlett Packard HP1 column, 200 °C injection, 50–300 °C over 27 min)



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		8.3444	17.125	0.000	37759	BB	2.3	
2		91.6556	18.014	0.000	414746	BB	2.4	
Totals:		100.0000		0.000	452505			

Enantiomeric excess of **6** (Chiracel OJ-H column, 99.9:0.1 hexanes:isopropanol, 1 mL/min, 230nm) synthesized with Rh₂(S-PTTL)₃(TPA)



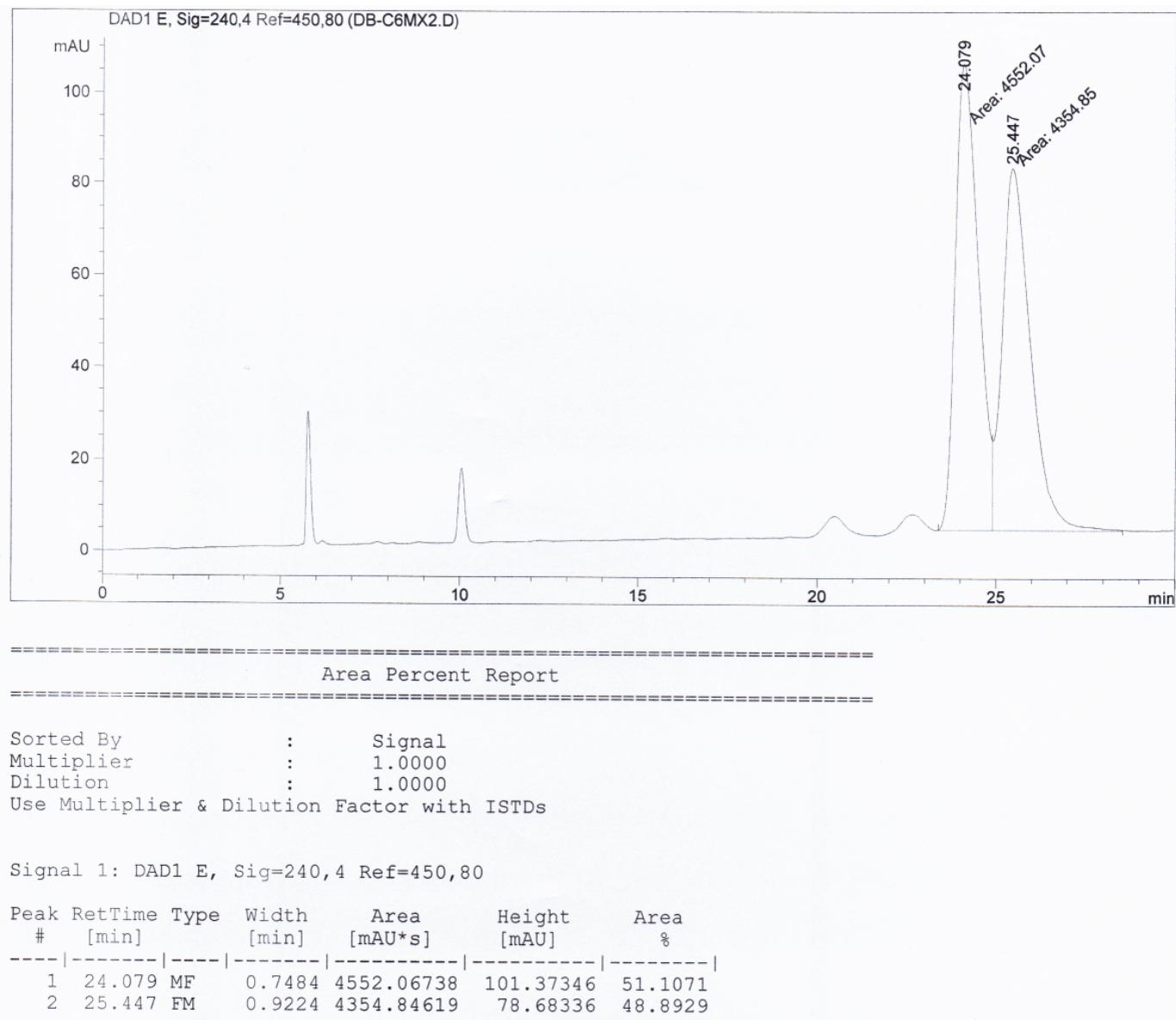
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Area Percent Report
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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

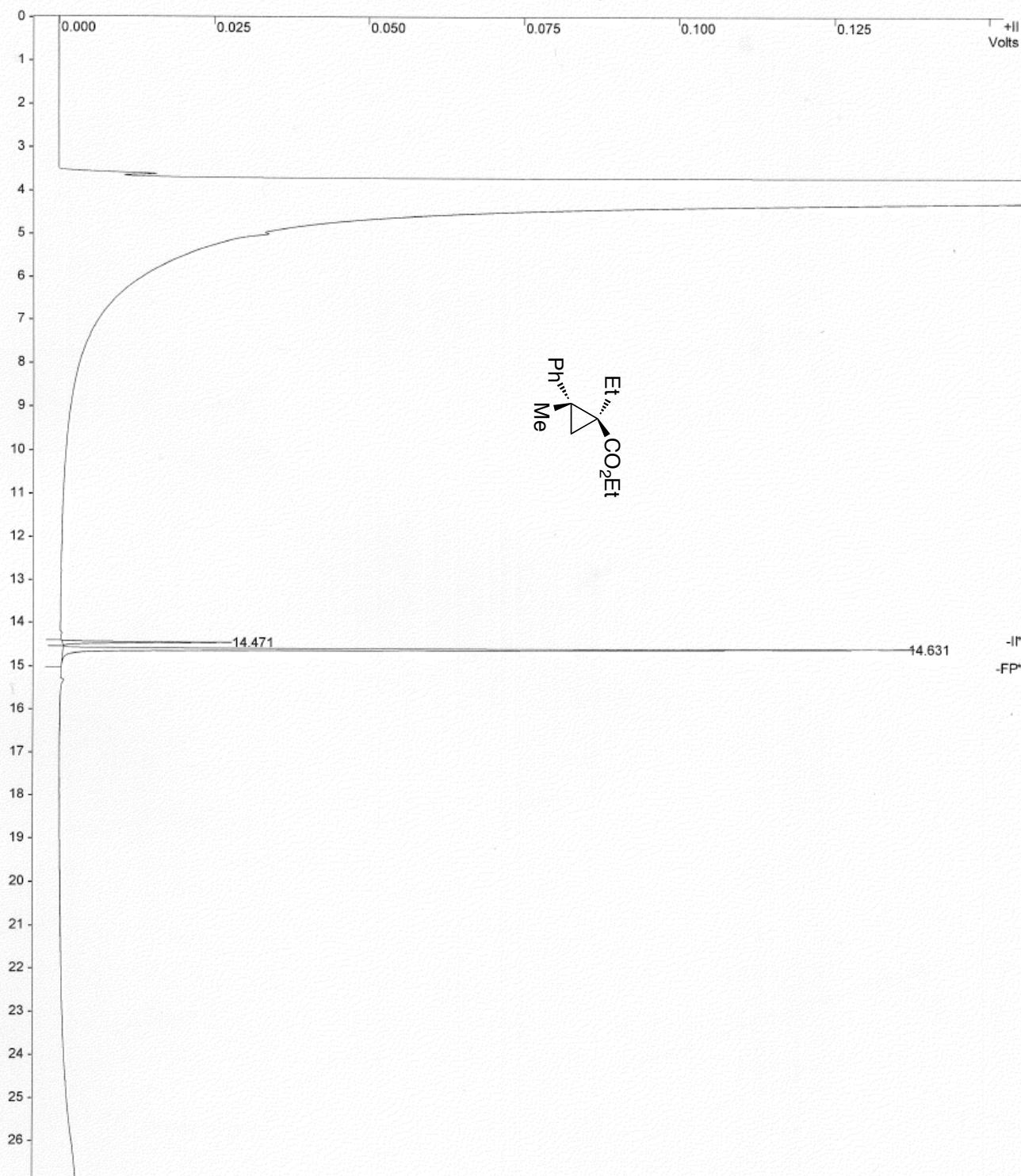
Signal 1: DAD1 G, Sig=230,4 Ref=450,80

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.141	MF	0.7666	6142.29736	133.53375	90.3138
2	26.064	FM	0.8047	658.76740	13.64360	9.6862

Coinjection of (1S, 2R) and (1R,2S) **6** to give near-racemate (Chiracel OJ-H column, 99.9:0.1 hexanes:isopropanol, 1 mL/min, 240nm)

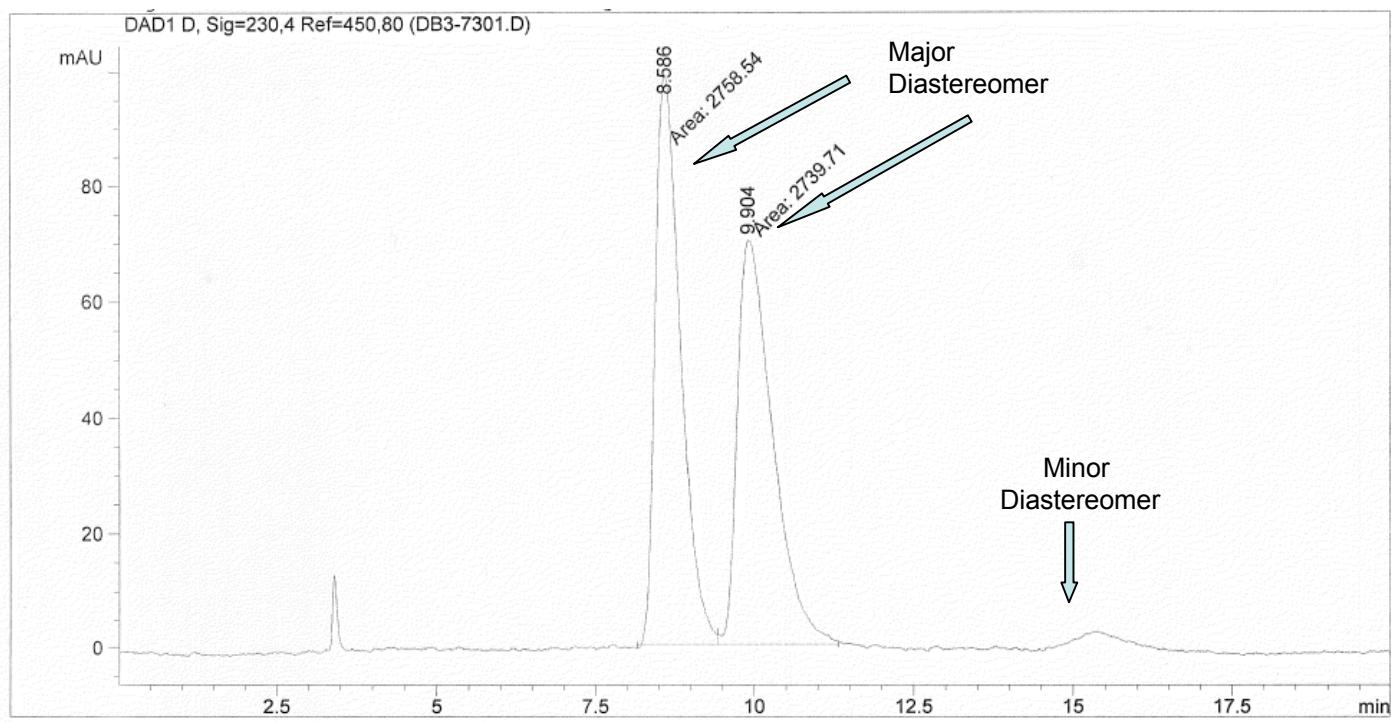


GC trace of **7** (Hewlett Packard HP1 column, 200 °C injection, 50–300 °C over 27 min)



Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		16.1973	14.471	0.000	66272	BB	2.2	
2		83.8027	14.631	0.000	342883	BB	2.2	
Totals:		100.0000		0.000	409155			

HPLC resolution of racemic **7** (Chiracel OF column, 99.9:0.1 hexanes:isopropanol, 1 mL/min, 230 nm)



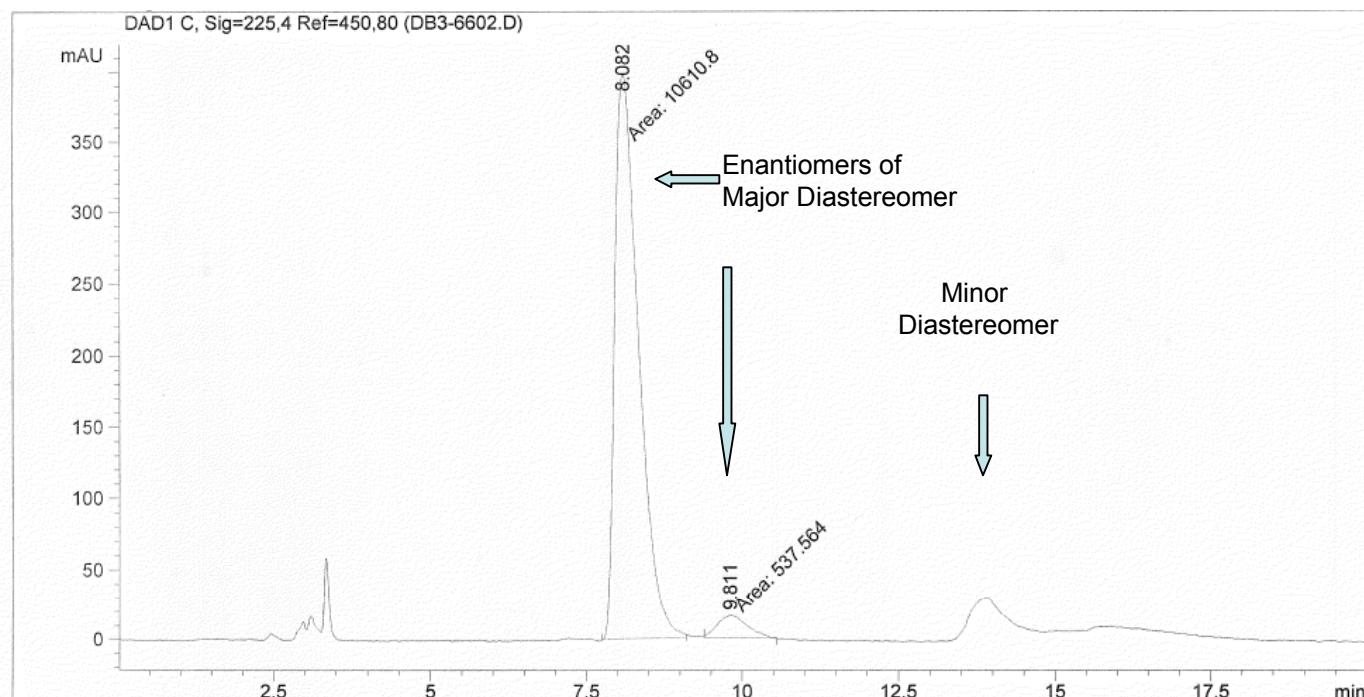
Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=230, 4 Ref=450, 80

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.586	MM	0.4649	2758.53784	98.90098	50.1712
2	9.904	MM	0.6531	2739.70898	69.91819	49.8288

Enantiomeric excess of **7** (Chiracel OF column, 99.9:0.1 hexanes:isopropanol, 1 mL/min,
220nm)



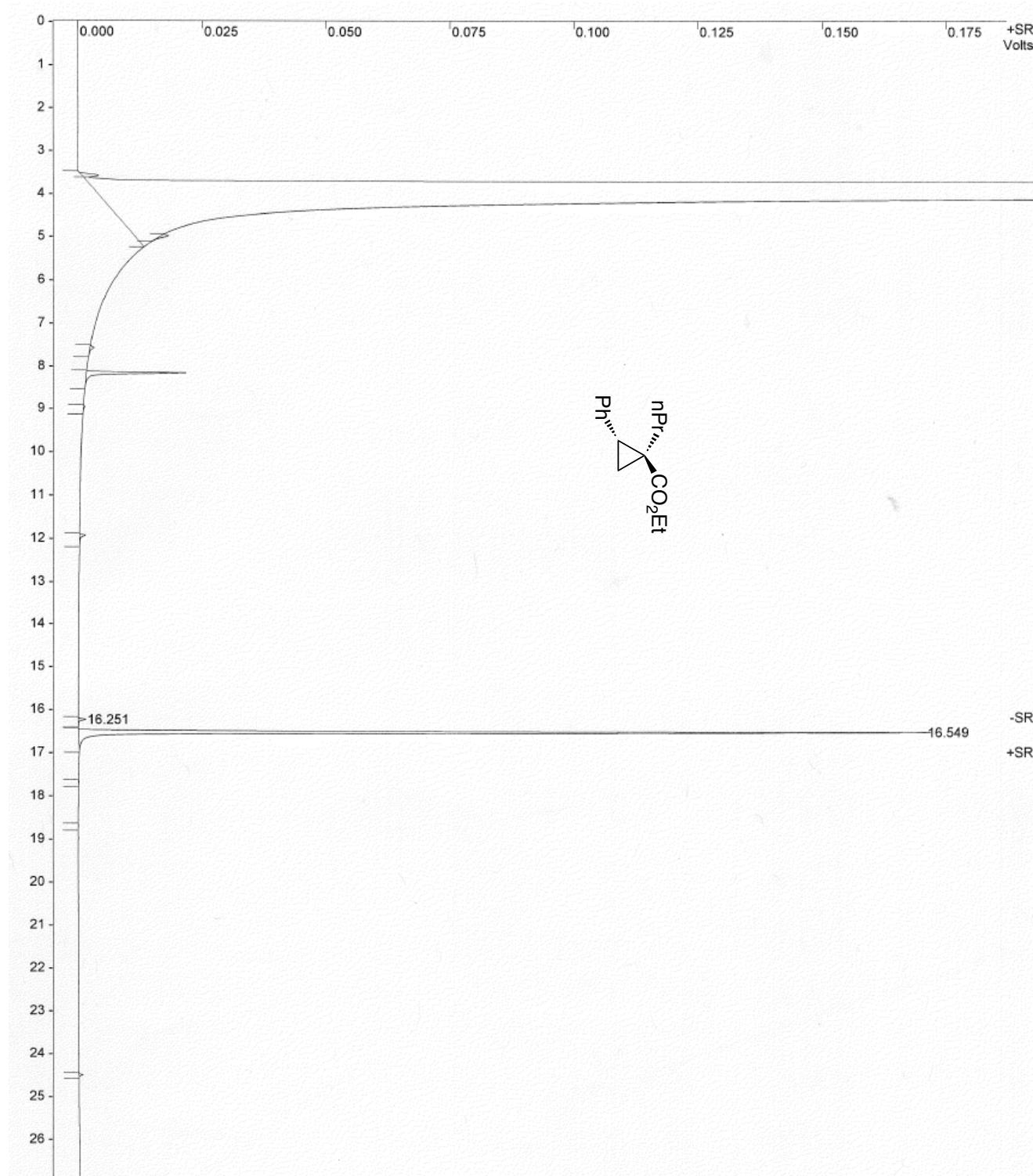
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Area Percent Report
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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 C, Sig=225,4 Ref=450,80

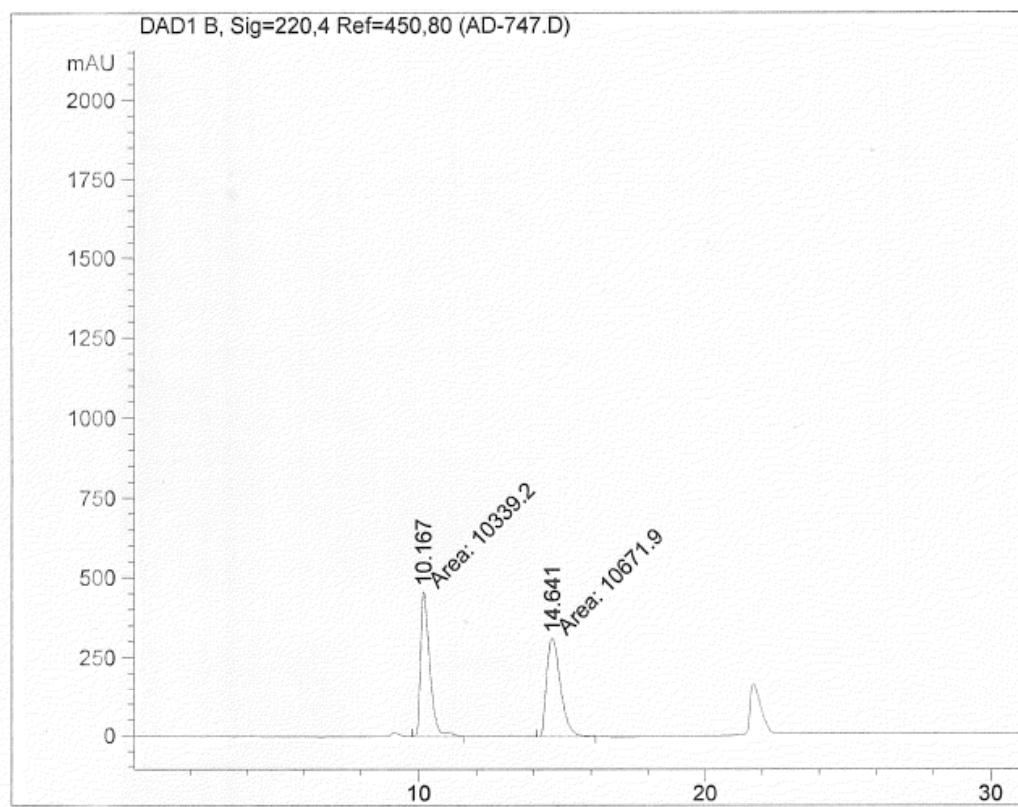
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.082	MM	0.4431	1.06108e4	399.14804	95.1781
2	9.811	MM	0.5548	537.56384	16.14973	4.8219

GC trace of **8** (Hewlett Packard HP5 column, 200 °C injection, 50–300 °C over 27 min)



Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		0.7813	16.251	0.000	4274	BB	2.4	
2		99.2187	16.549	0.000	542728	BB	2.8	
Totals:		100.0000		0.000	547002			

HPLC Resolution of racmic **8** (Chiracel OD column, 99.9:0.1 hexanes:isopropanol, 1 mL/min, 220nm)⁶



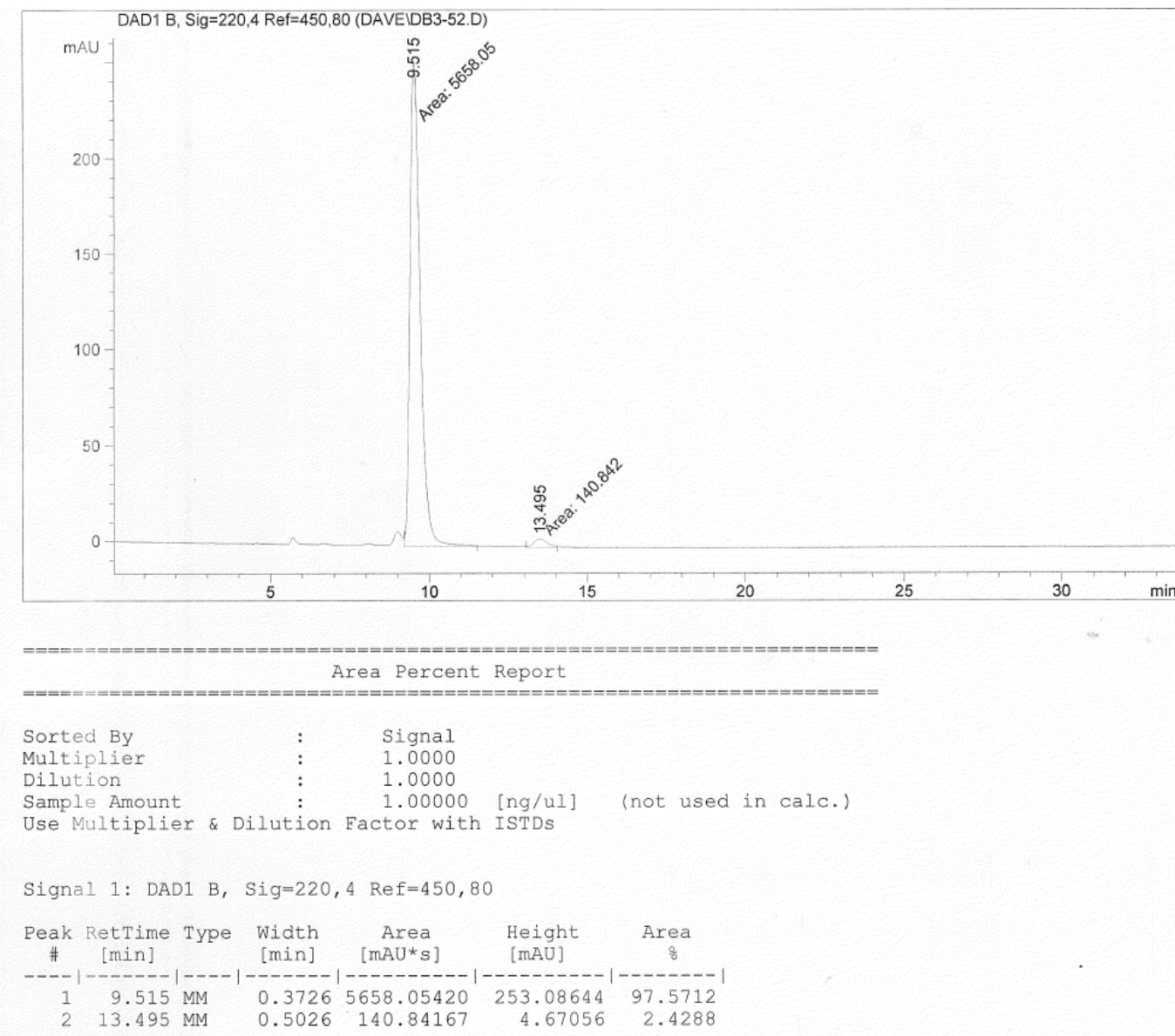
Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

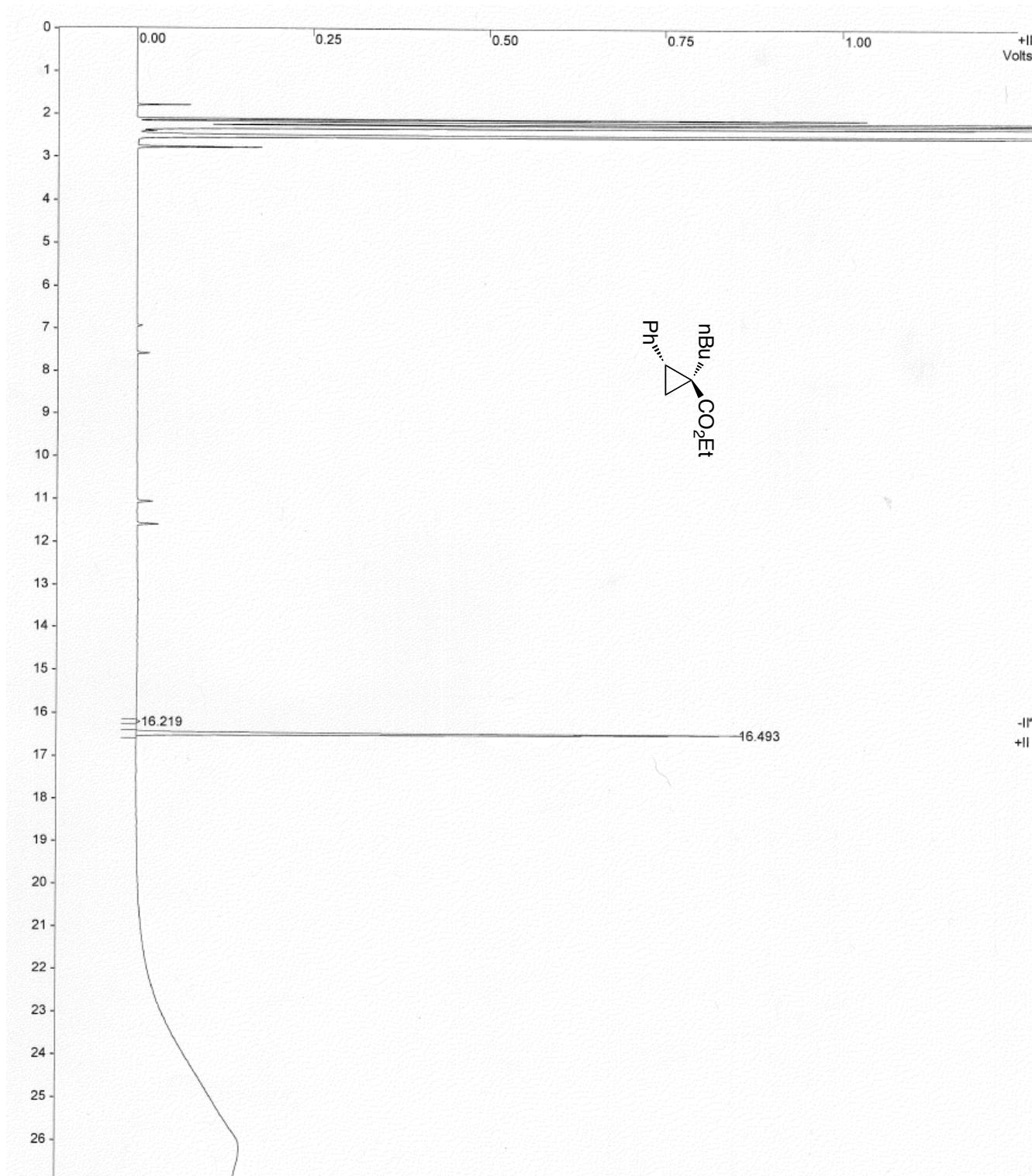
Signal 1: DAD1 B, Sig=220, 4 Ref=450, 80

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.167	MM	0.3759	1.03392e4	458.38092	49.2083
2	14.641	MM	0.5692	1.06719e4	312.50479	50.7917

Enantiomeric excess of **8** (Chiracel OD column, 99.9:0.1 hexanes:isopropanol, 1 mL/min, 220nm)

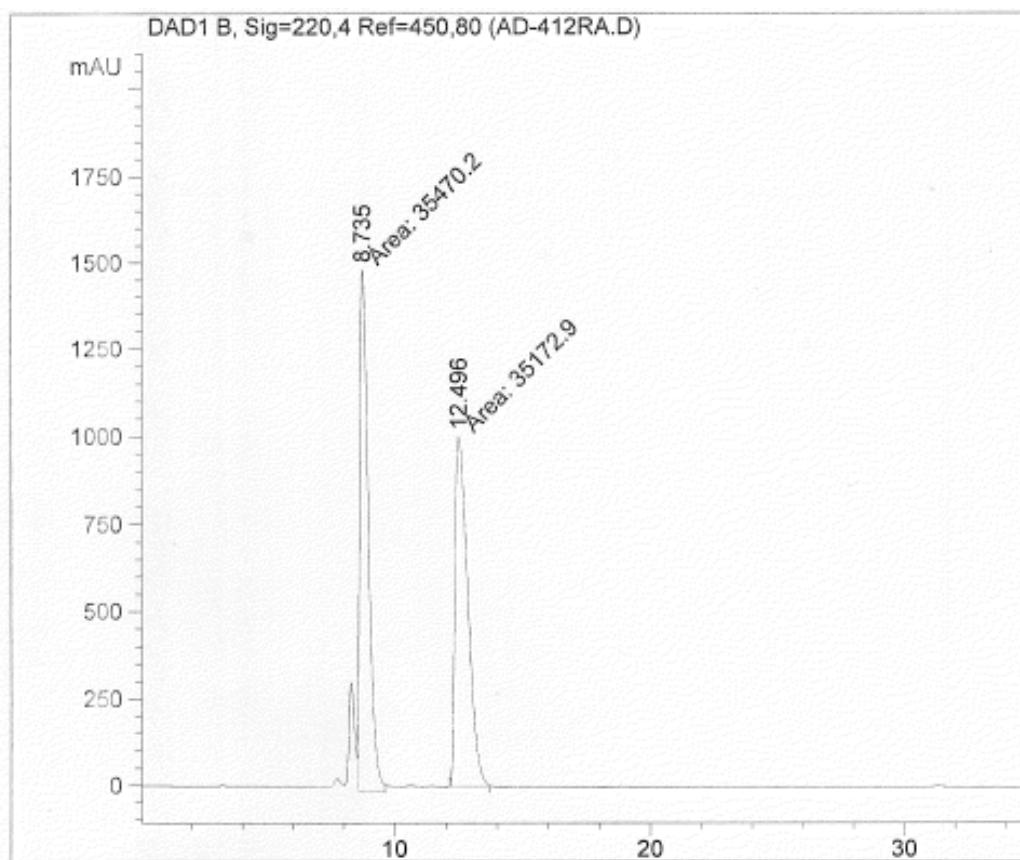


GC trace of **9** (Hewlett Packard HP5 column, 200 °C injection, 50–300 °C over 27 min)



Peak No.	Peak Name	Result (.)	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		0.5420	16.219	0.000	13334	BB	2.6	
2		99.4580	16.493	0.000	2446879	BB	2.7	
Totals:		100.0000		0.000	2460213			

HPLC Resolution of racemic **9** (Chiracel OD column, 99.9:0.1 hexanes:isopropanol, 1 mL/min, 220nm)⁶

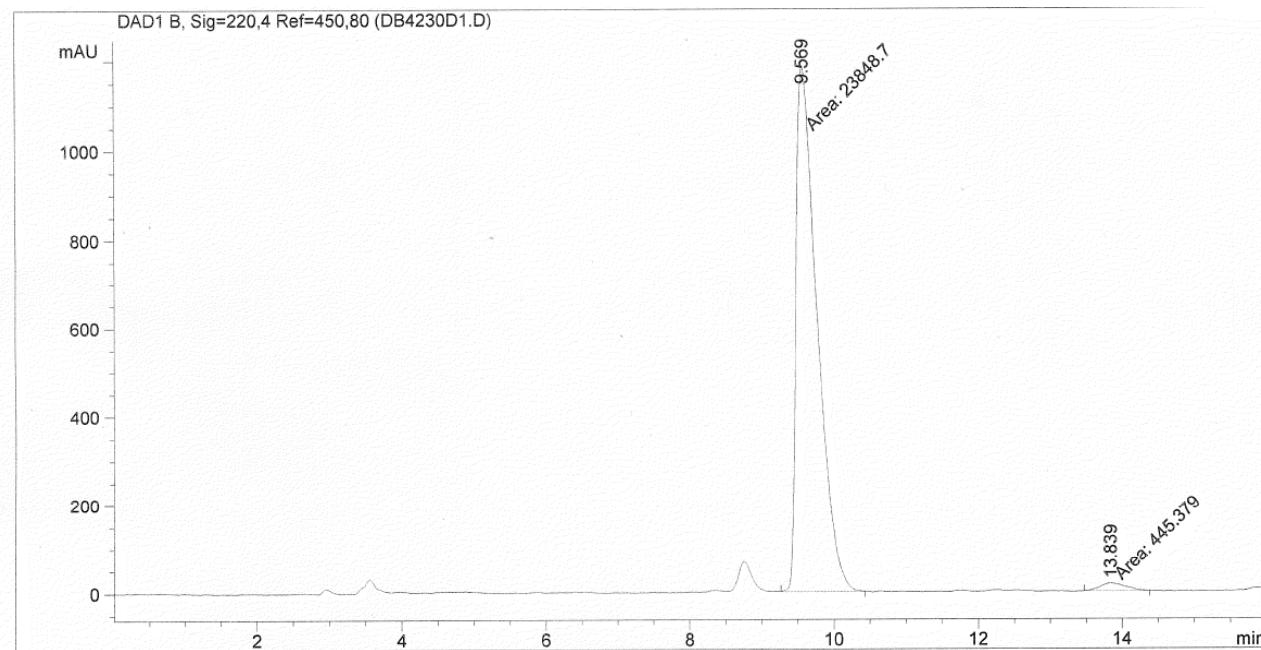


Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 B, Sig=220,4 Ref=450,80

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.735	FM	0.3950	3.54702e4	1496.61658	50.2105
2	12.496	MM	0.5842	3.51729e4	1003.45349	49.7895

Enantiomeric excess of **9** (Chiracel OD column, 99.9:0.1 hexanes:isopropanol, 1 mL/min, 220nm)



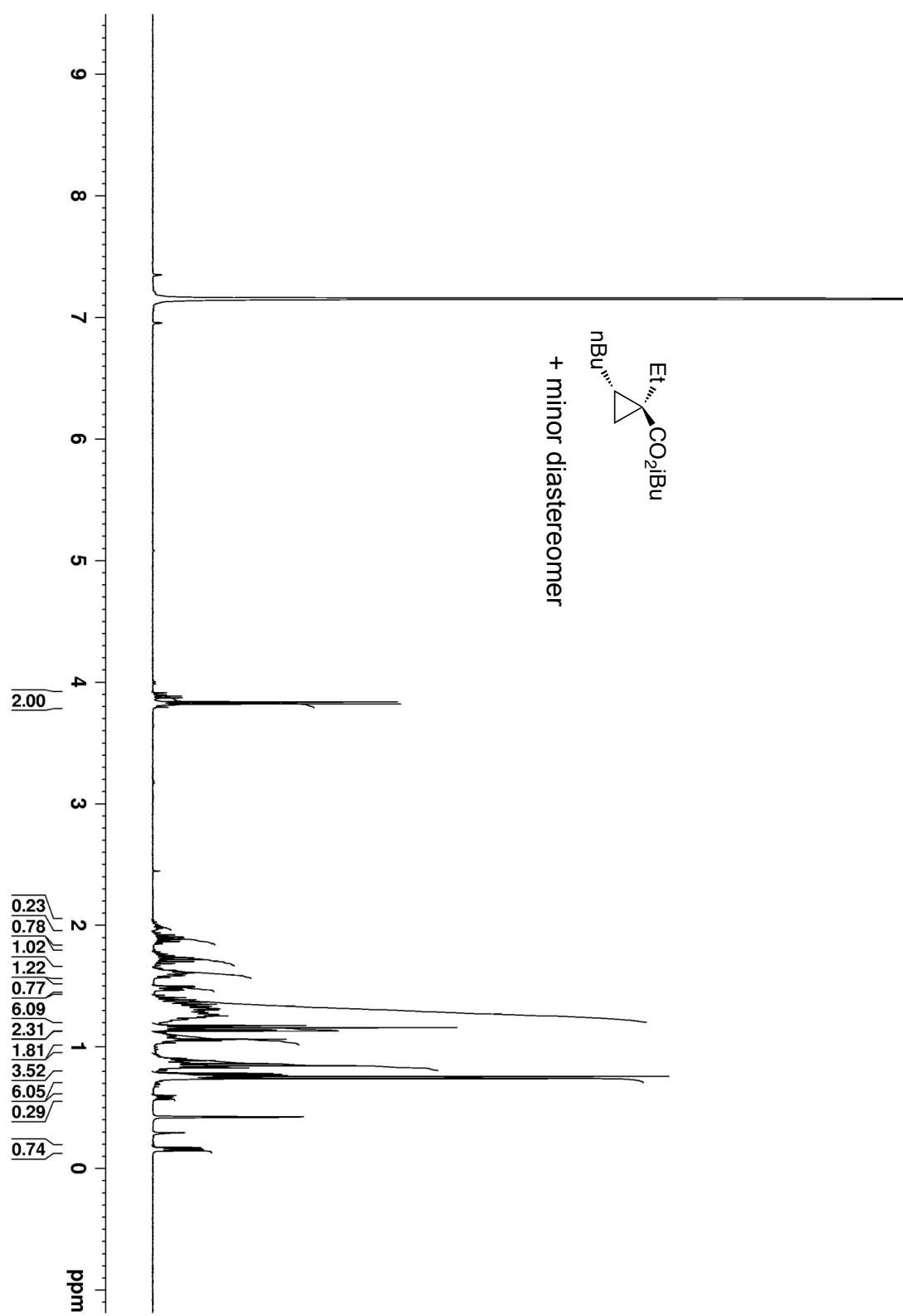
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Area Percent Report
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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

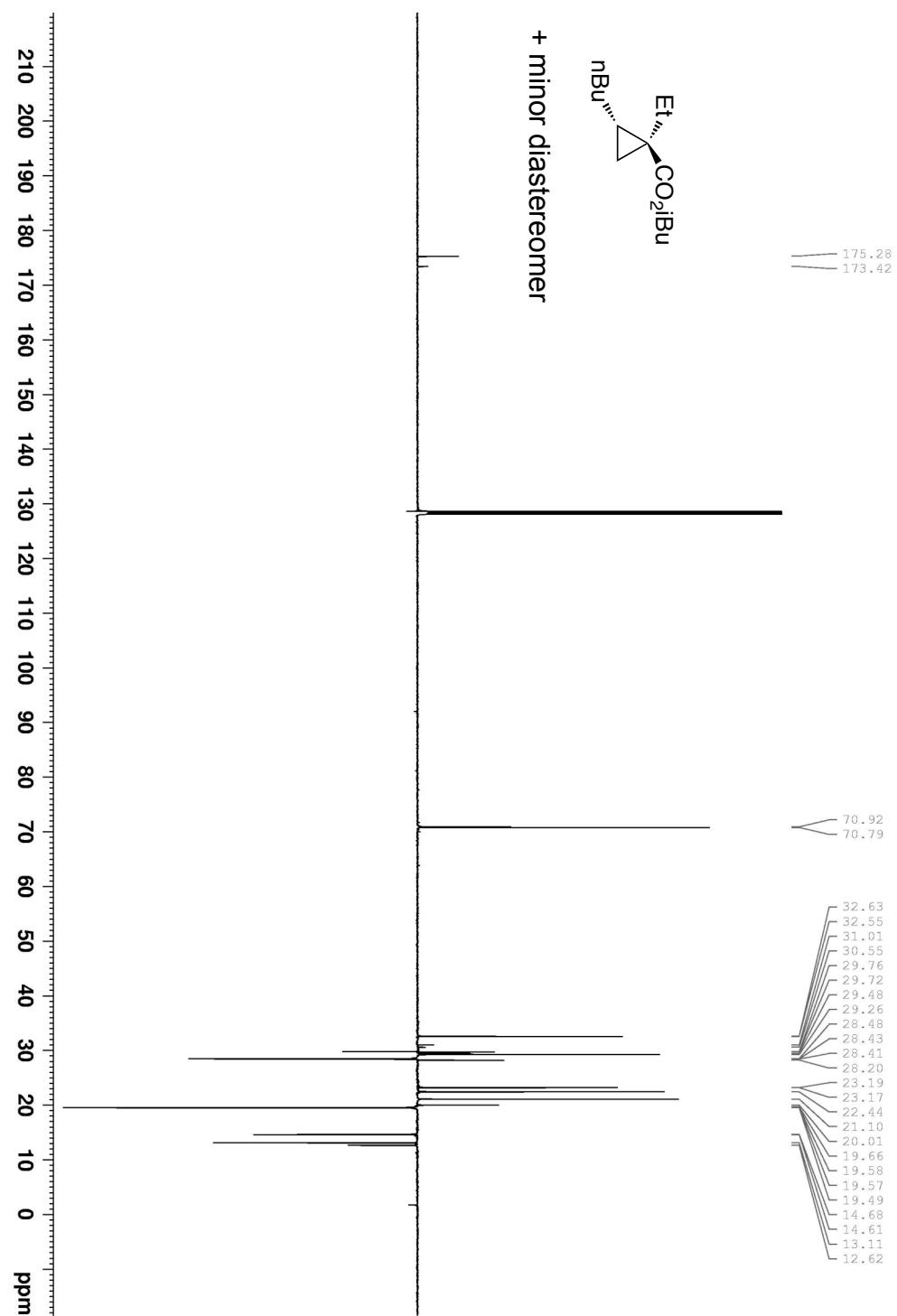
Signal 1: DAD1 B, Sig=220,4 Ref=450,80

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.569	MM	0.3349	2.38487e4	1187.02502	98.1667
2	13.839	MM	0.4232	445.37918	17.54071	1.8333

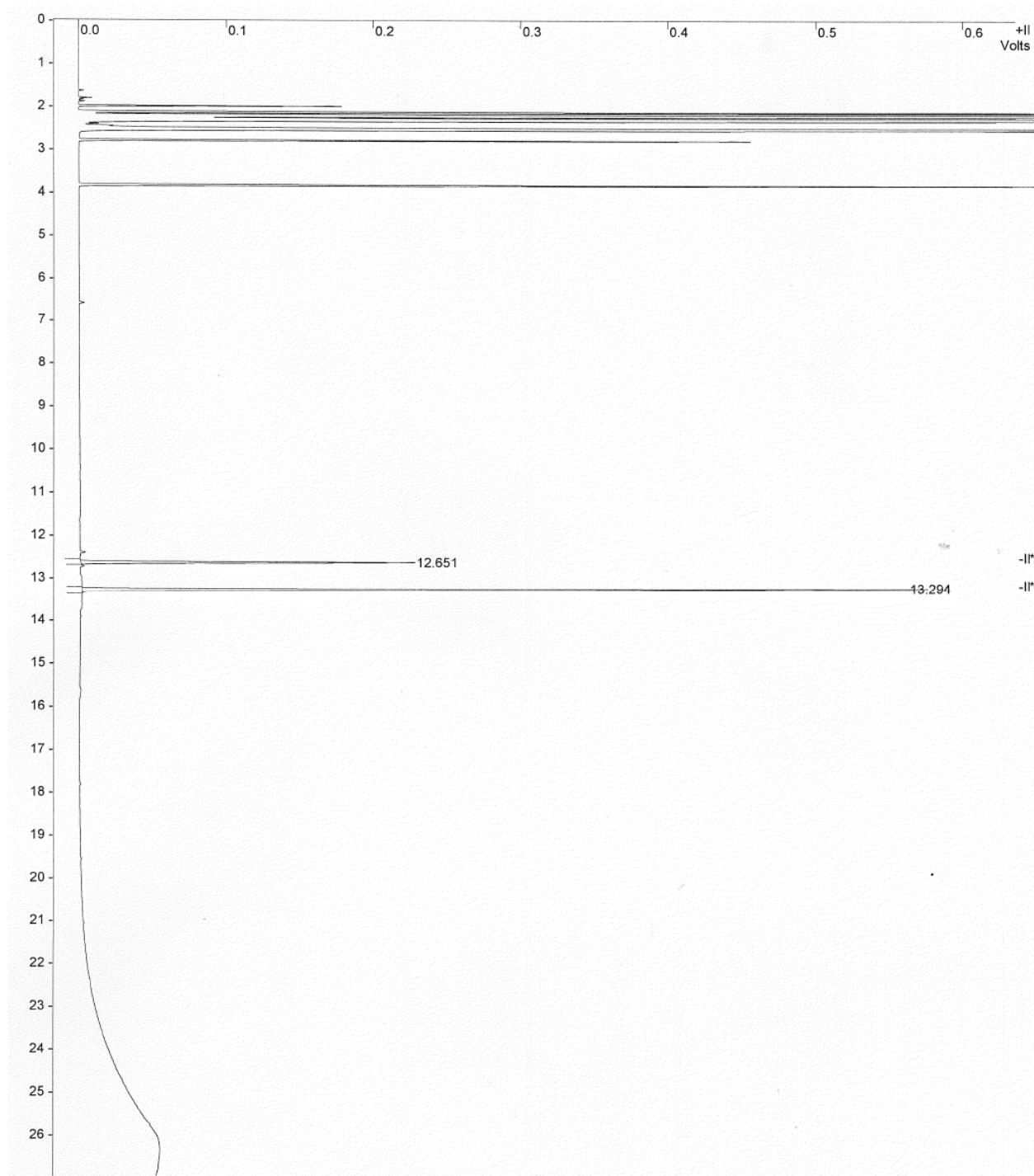
¹H NMR spectrum of **10** (400 MHz, C₆D₆)



¹³C NMR spectrum of **10** (100 MHz, C₆D₆)

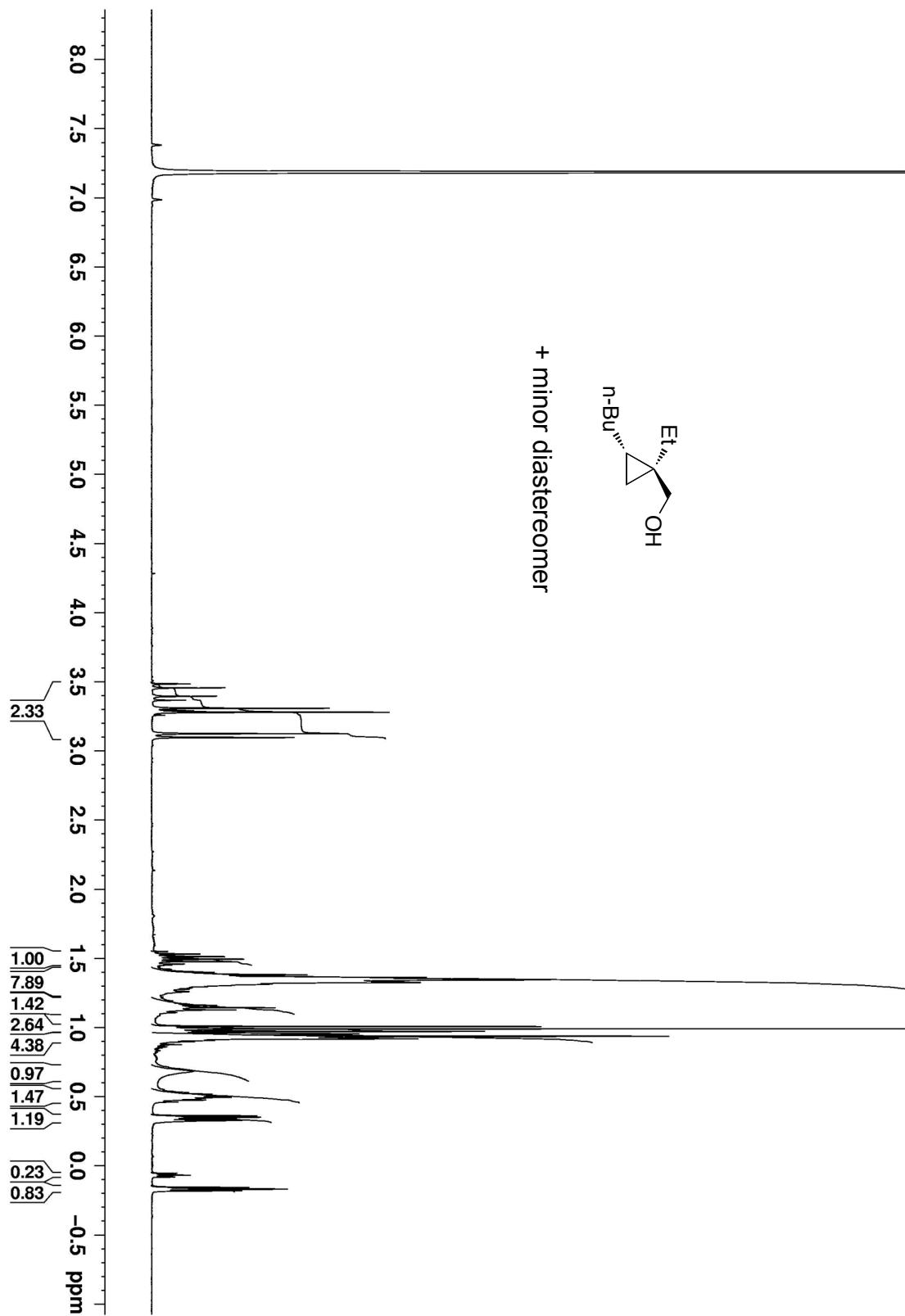


GC trace of **10** (Hewlett Packard HP1 column, 200 °C injection, 50–300 °C over 27 min)

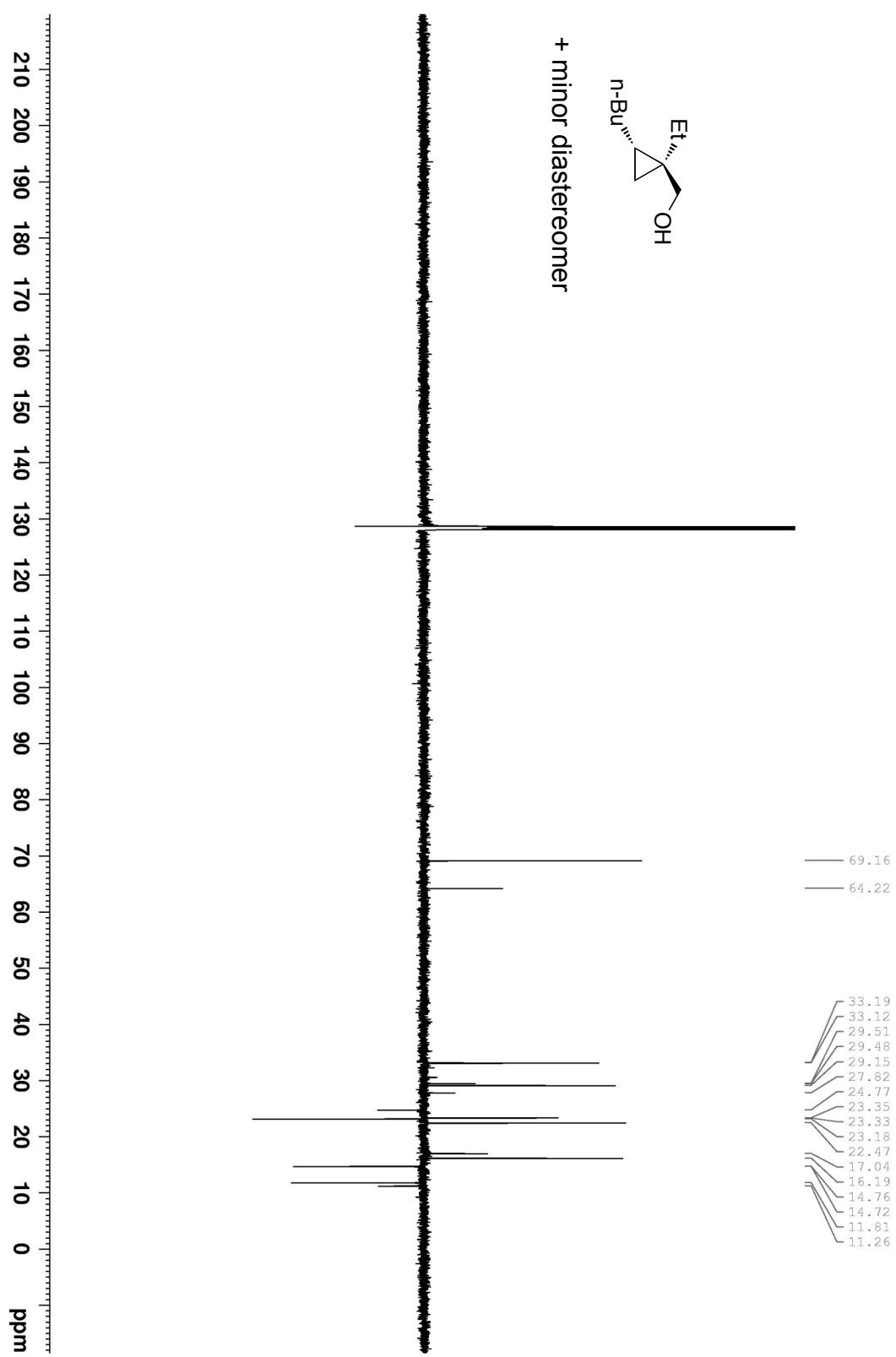


Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	1/2 (sec)	Status Codes
1		28.1073	12.651	0.000	516295	BB	2.1	
2		71.8927	13.294	0.000	1320577	BB	2.2	
Totals:		100.0000		0.000	1836872			

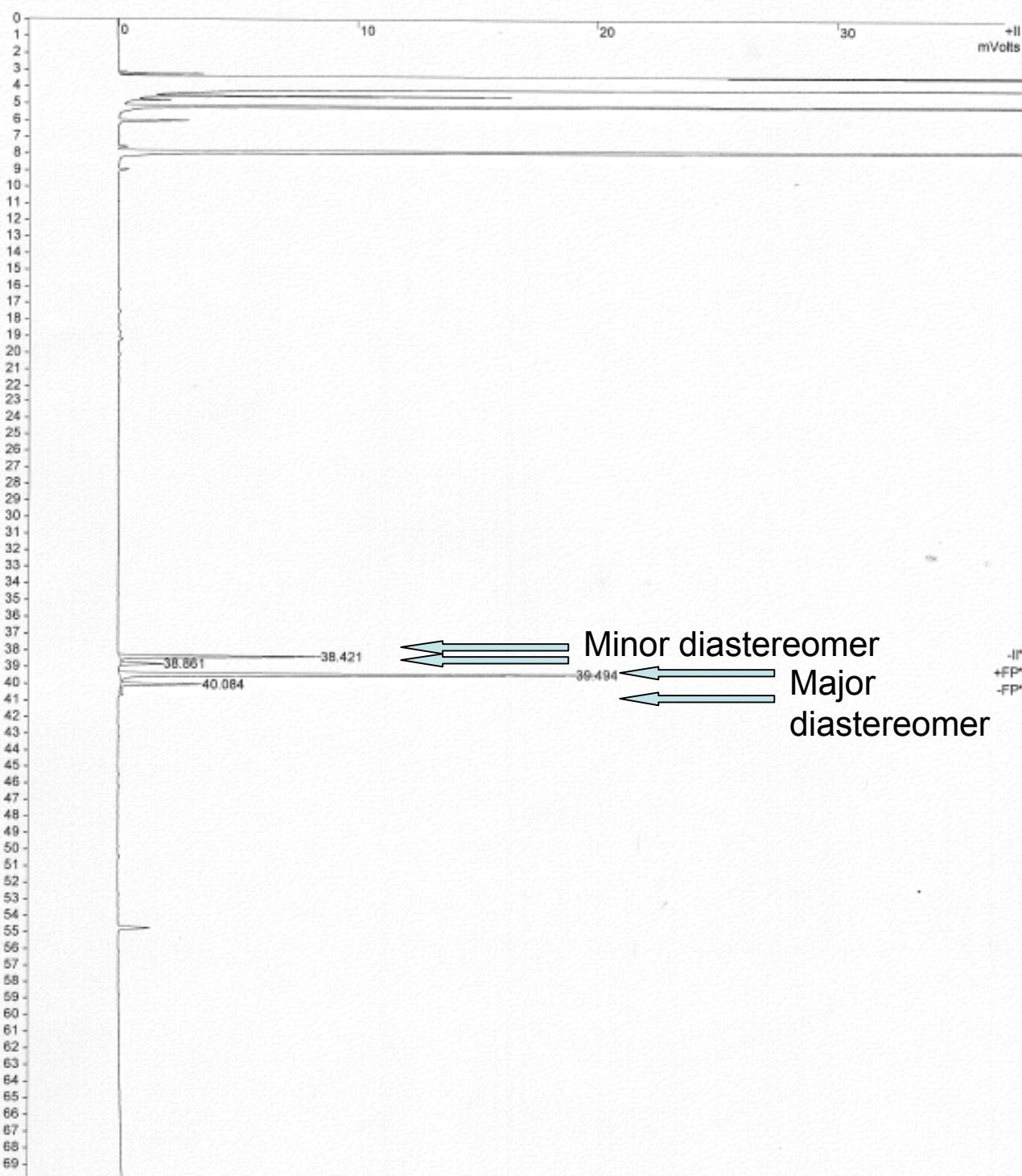
¹H NMR spectrum of **10a** (400 MHz, C₆D₆)



¹³C NMR spectrum of **10a** (100 MHz, C₆D₆)

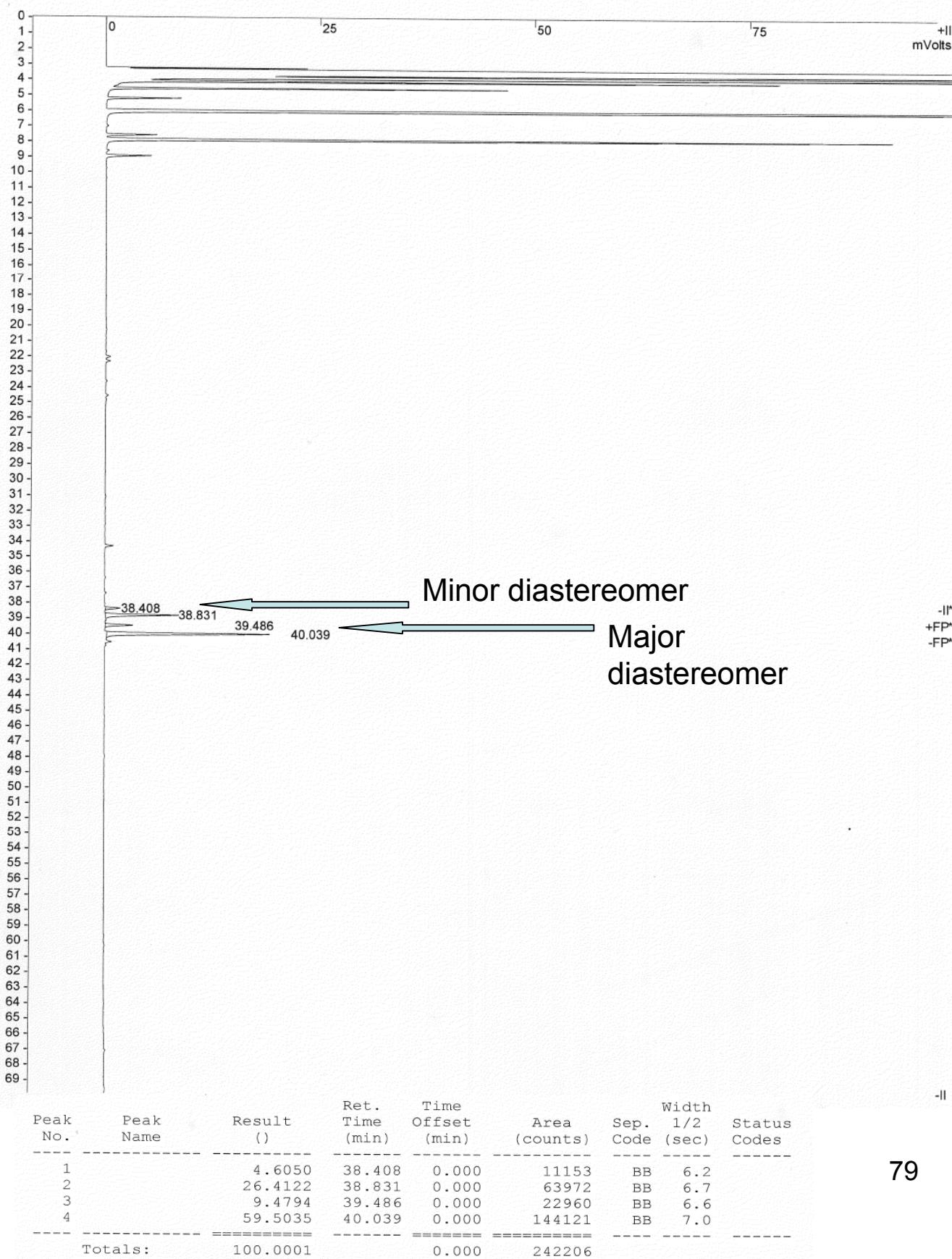


GC trace of Enantioselective **10a**

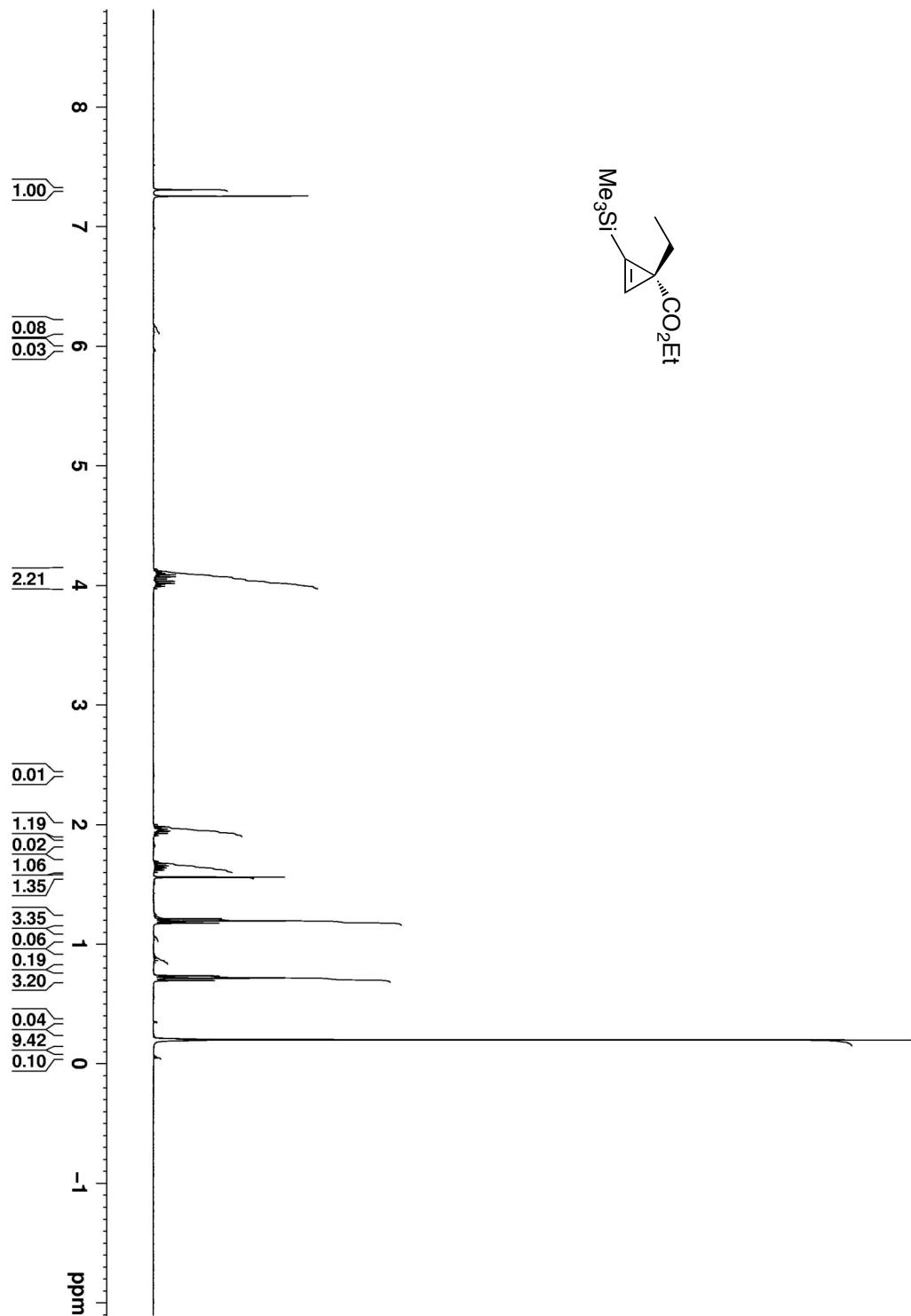


Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		24.4056	38.421	0.000	62228	BB	6.8	
2		5.2013	38.861	0.000	13262	BB	6.7	
3		60.6647	39.494	0.000	154679	BB	7.0	
4		9.7284	40.084	0.000	24805	BB	6.8	
Totals:		100.0000		0.000	254974			

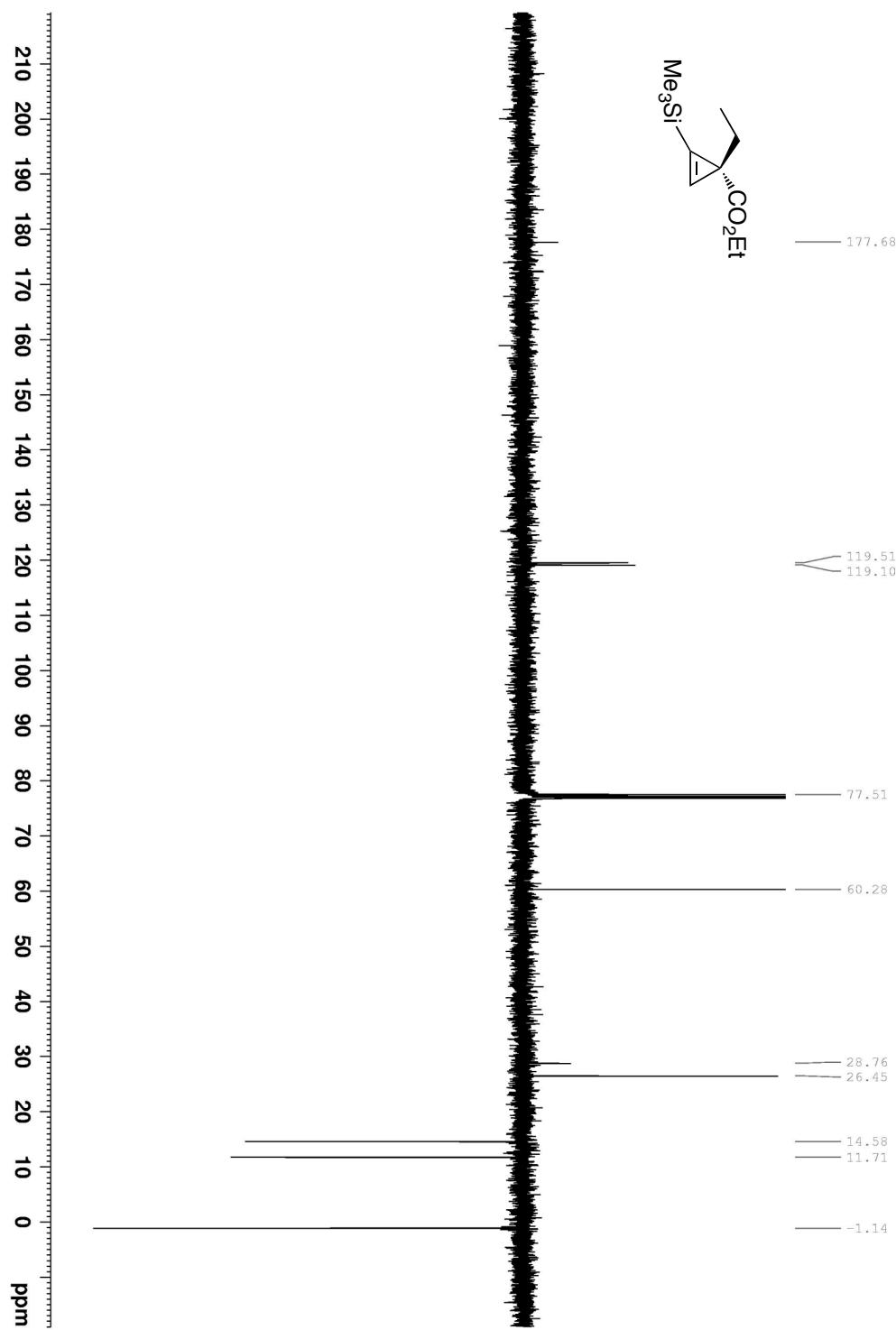
GC trace of **10a** catalyzed with Rh₂(R-PTTL)₃(TPA) catalyzed instead of with Rh₂(S-PTTL)₃(TPA) to show the opposite trend in enantioselectivity.



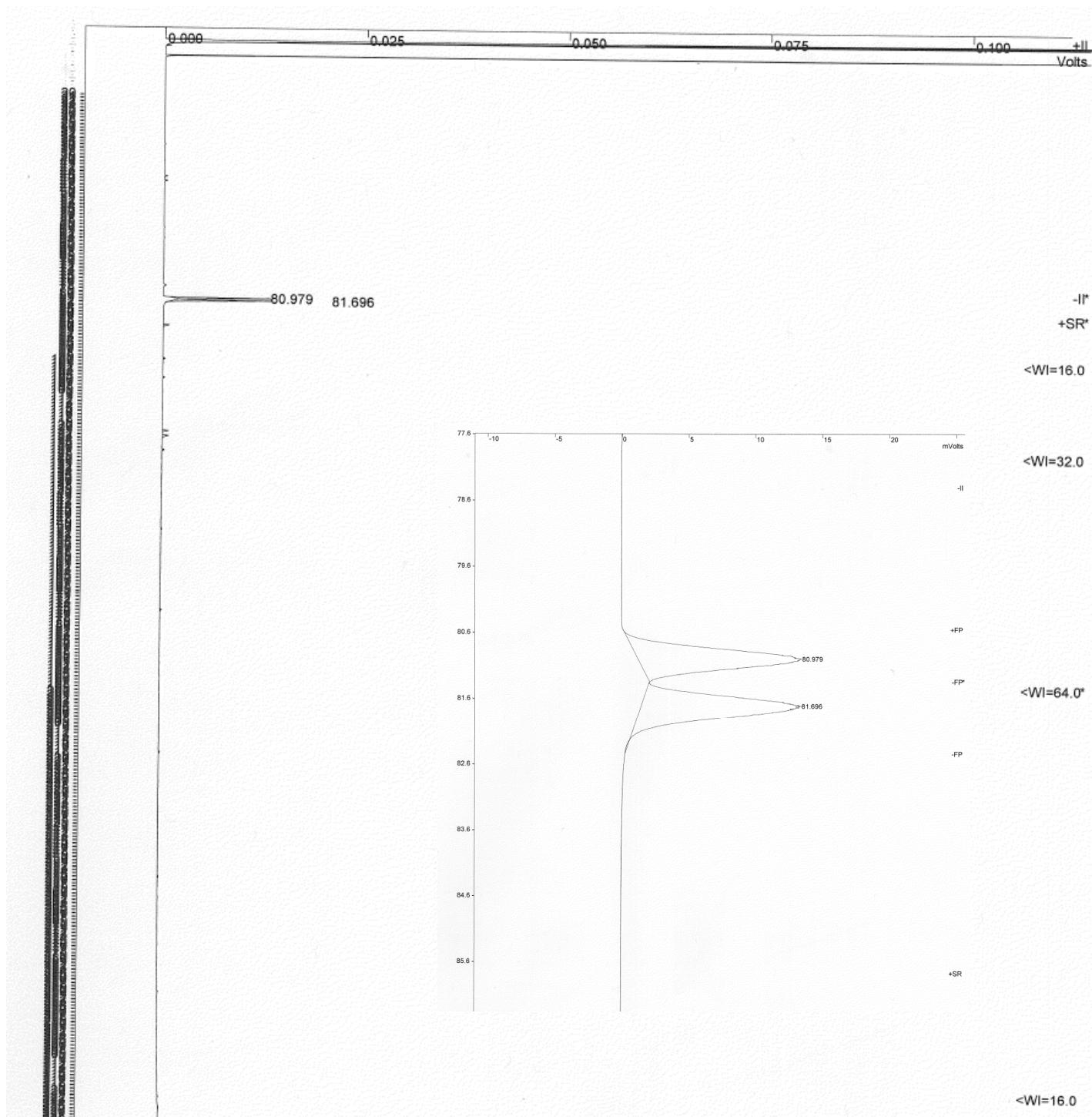
¹H NMR spectrum of **11** (400 MHz, CDCl₃)



^{13}C NMR spectrum of **11** (100 MHz, CDCl_3)

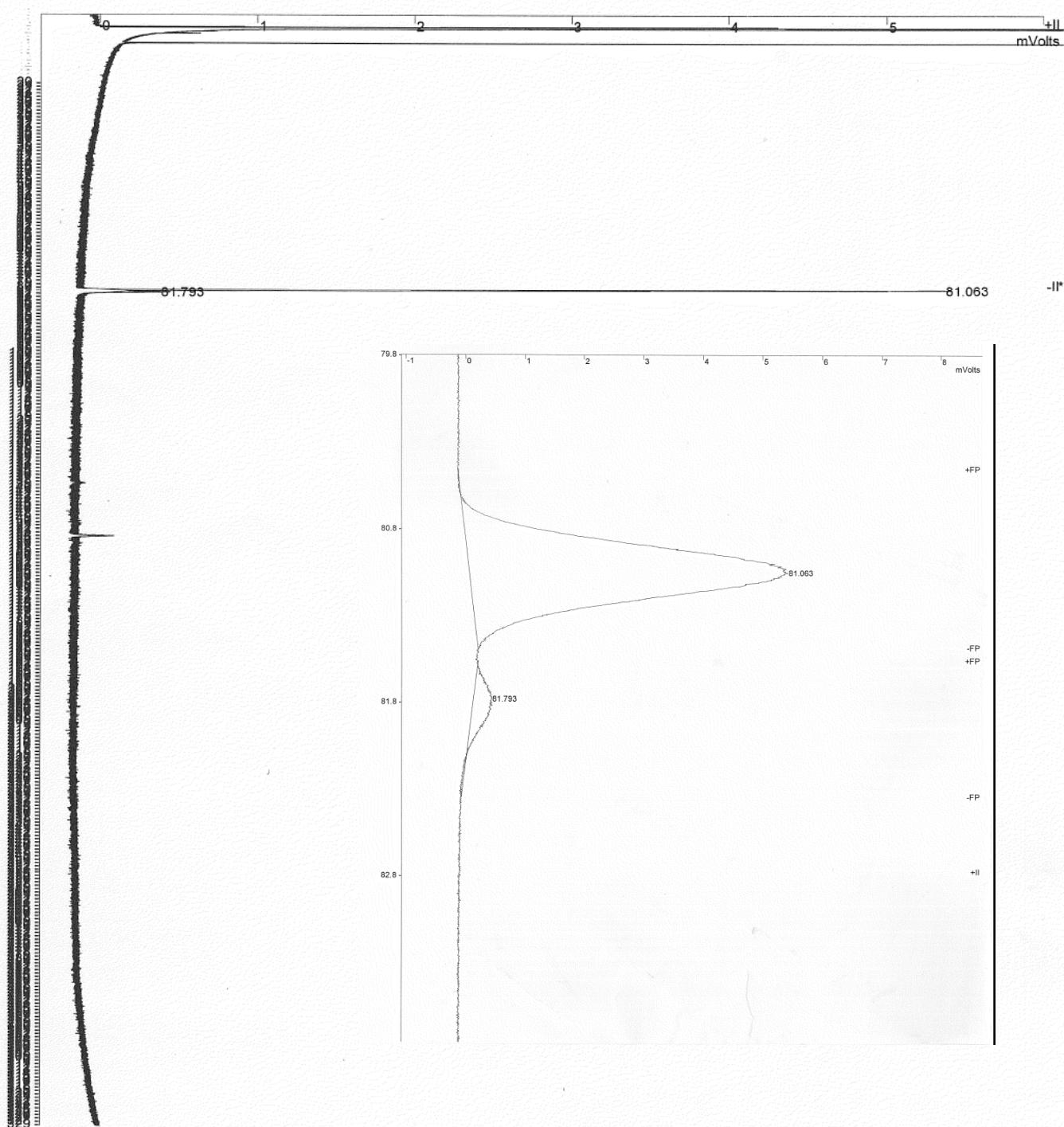


GC trace of racemic **11** (Chiralpak G-TA column, 200 °C injection, 50–180°C at 0.4°C per min., hold 20 min.)



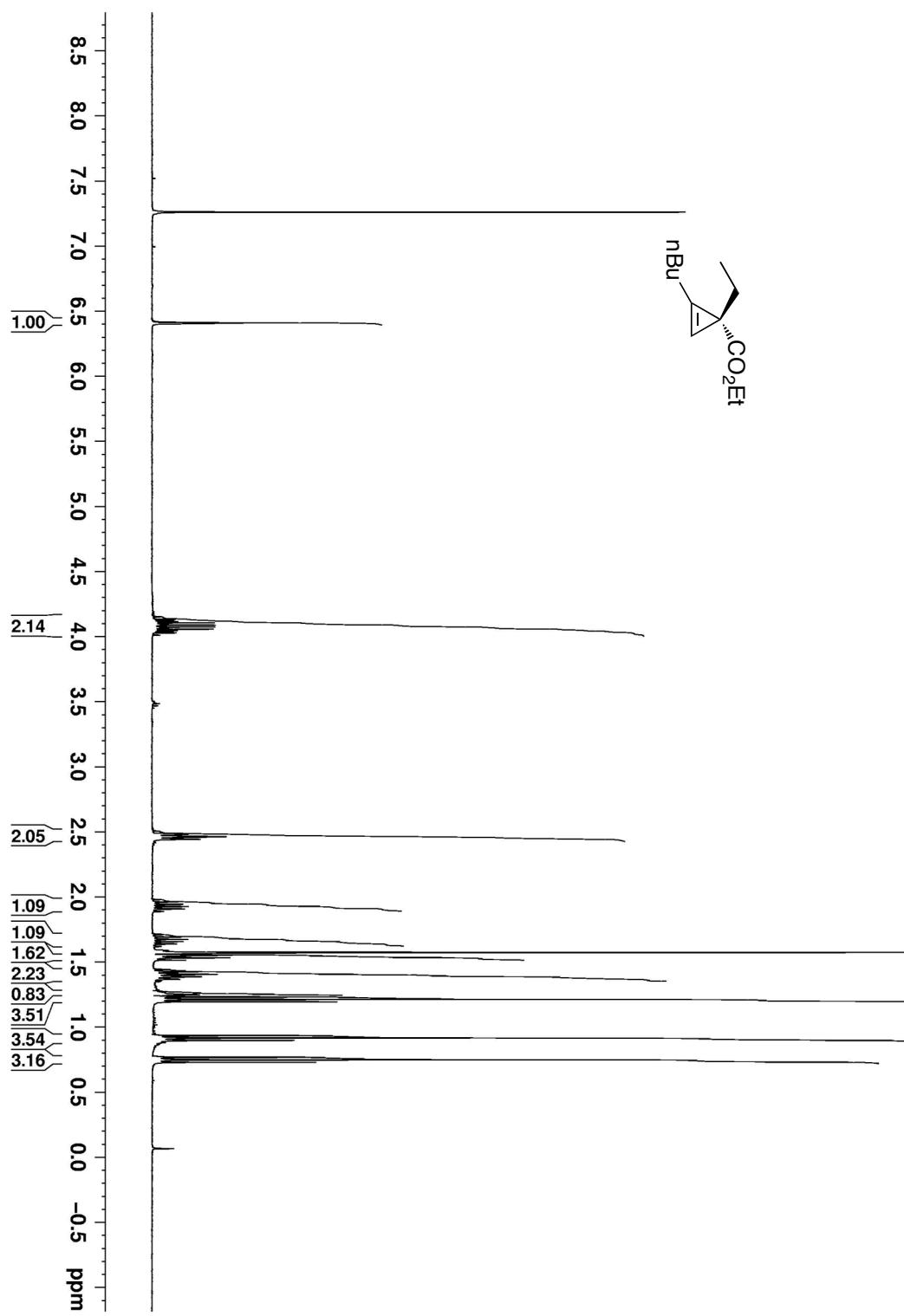
Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	1/2 (sec)	Status Codes
1		48.8452	80.979	0.000	245929	BB	20.8	
2		51.1548	81.696	0.000	257557	BB	19.8	
Totals:		100.0000		0.000	503486			

GC trace of enantioselective **11** (Chiralpak G-TA column, 200 °C injection, 50–180°C at 0.4°C per min., hold 20 min.)

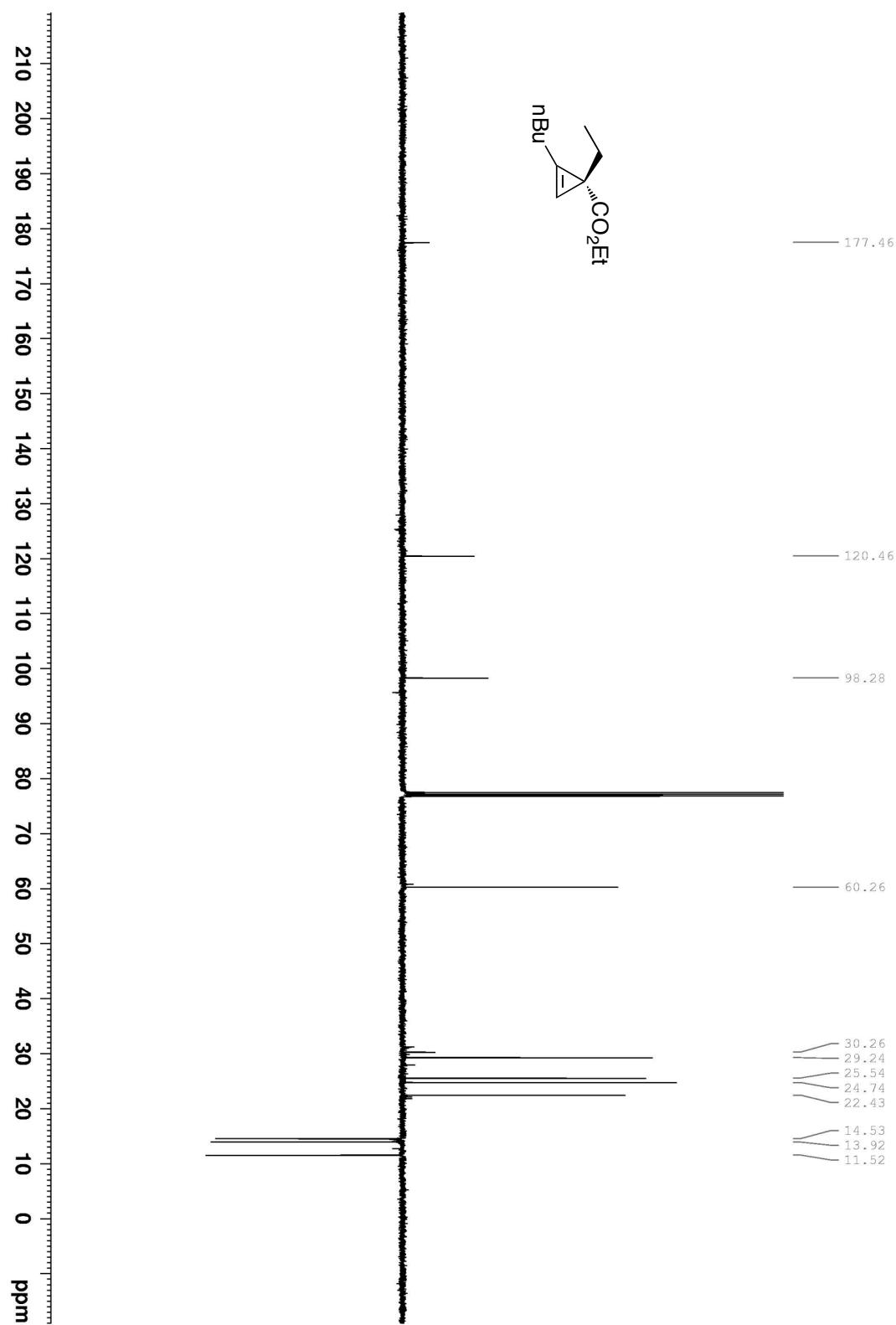


Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	1/2 sec	Status Codes
1		95.8060	81.063	0.000	114578	BB	20.7	
2		4.1940	81.793	0.000	5016	BB	12.5	
Totals:		100.0000		0.000	119594			

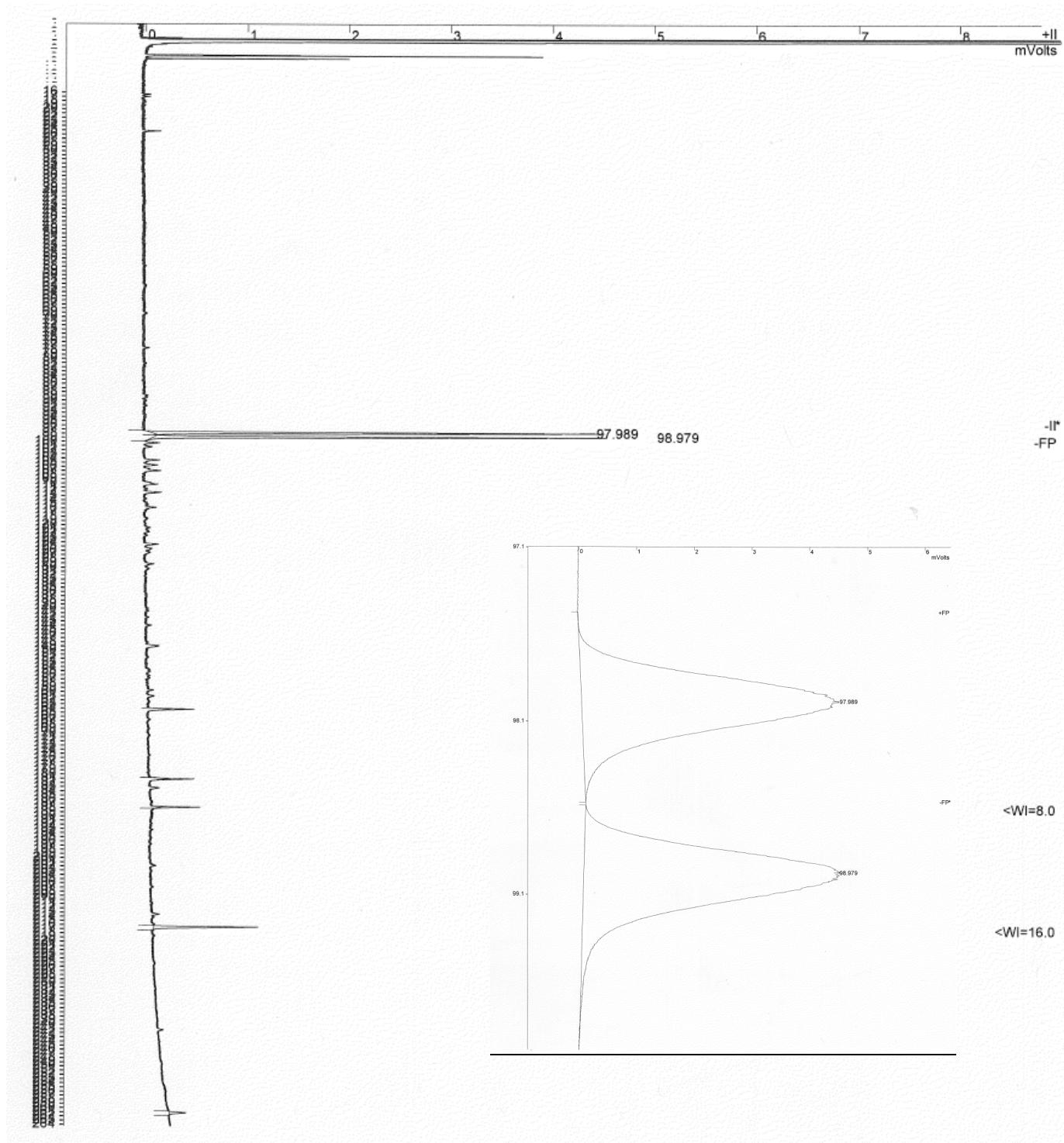
¹H NMR spectrum of **12** (400 MHz, CDCl₃)



^{13}C NMR spectrum of **12** (100 MHz, CDCl_3)

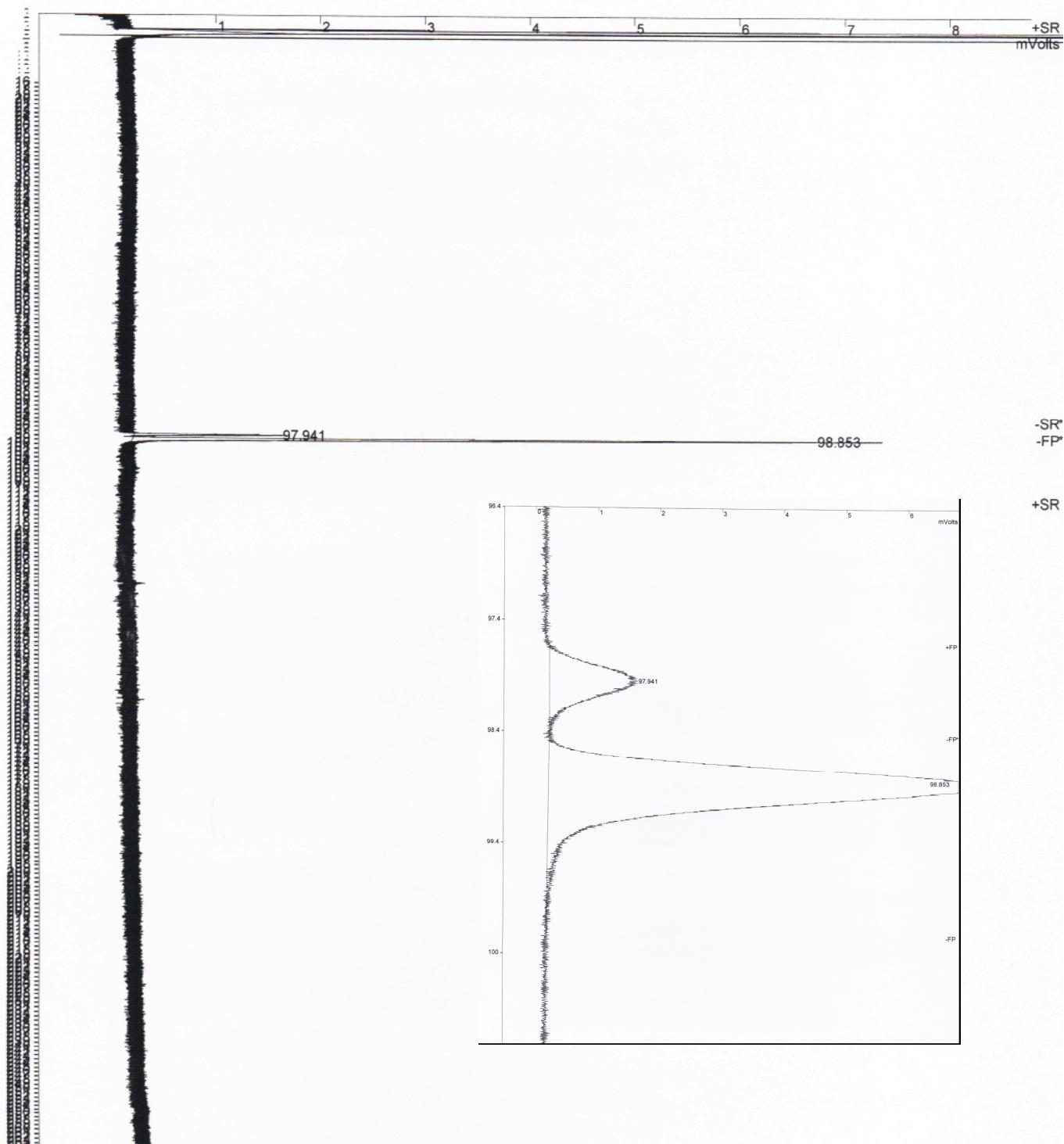


GC trace of racemic **12** (Chiralpak G-TA column, 200 °C injection, 50–180°C at 0.5°C per min., hold 20 min.)



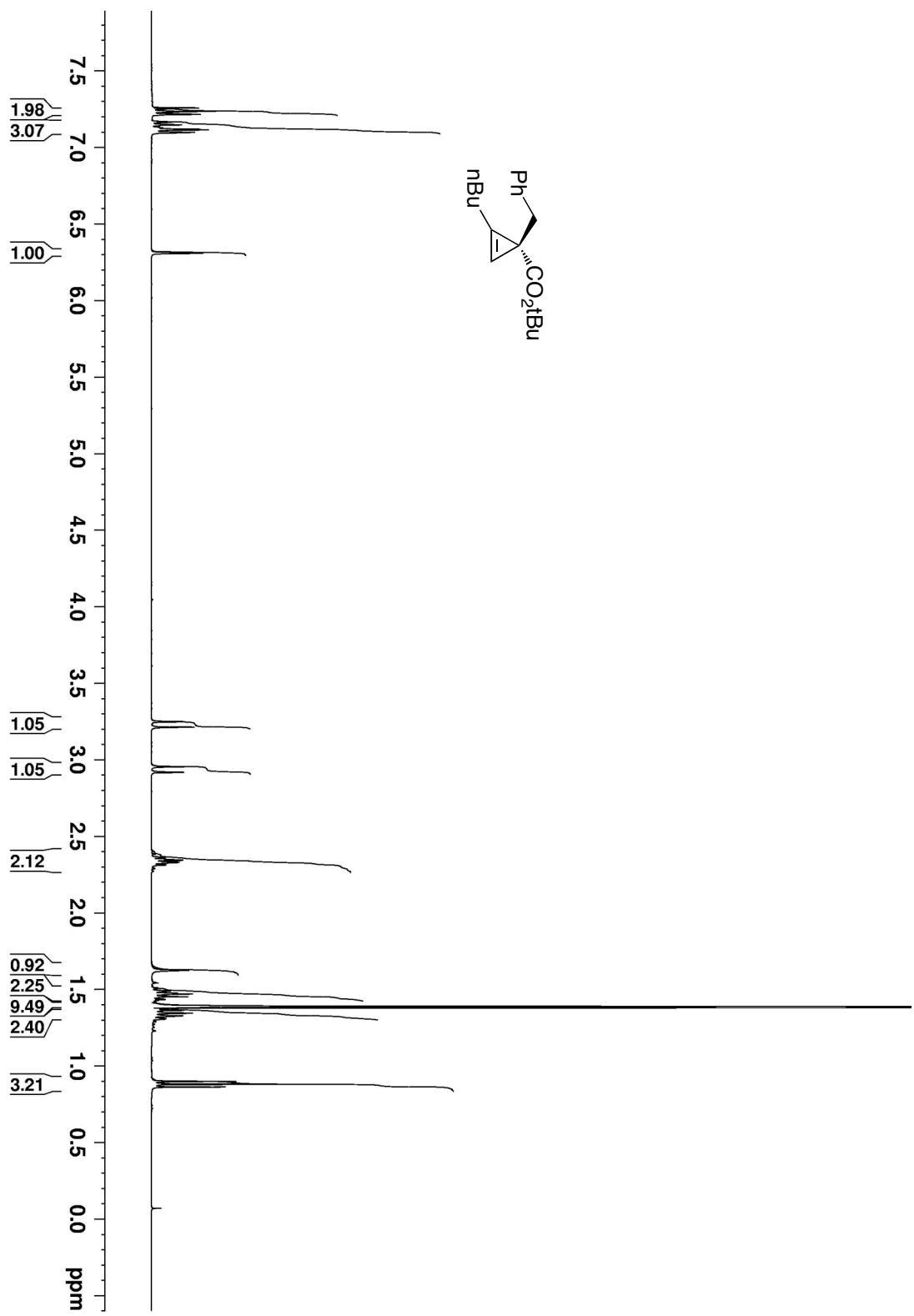
Peak No.	Peak Name	Result ()	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		49.4335	97.989	0.000	91249	BB	19.3	
2		50.5665	98.979	0.000	93340	BB	19.1	
Totals:		100.0000		0.000	184589			

Enantioselective GC trace of **12** (Chiralpak G-TA column, 200 °C injection, 50–180°C at 0.5°C per min., hold 20 min.)

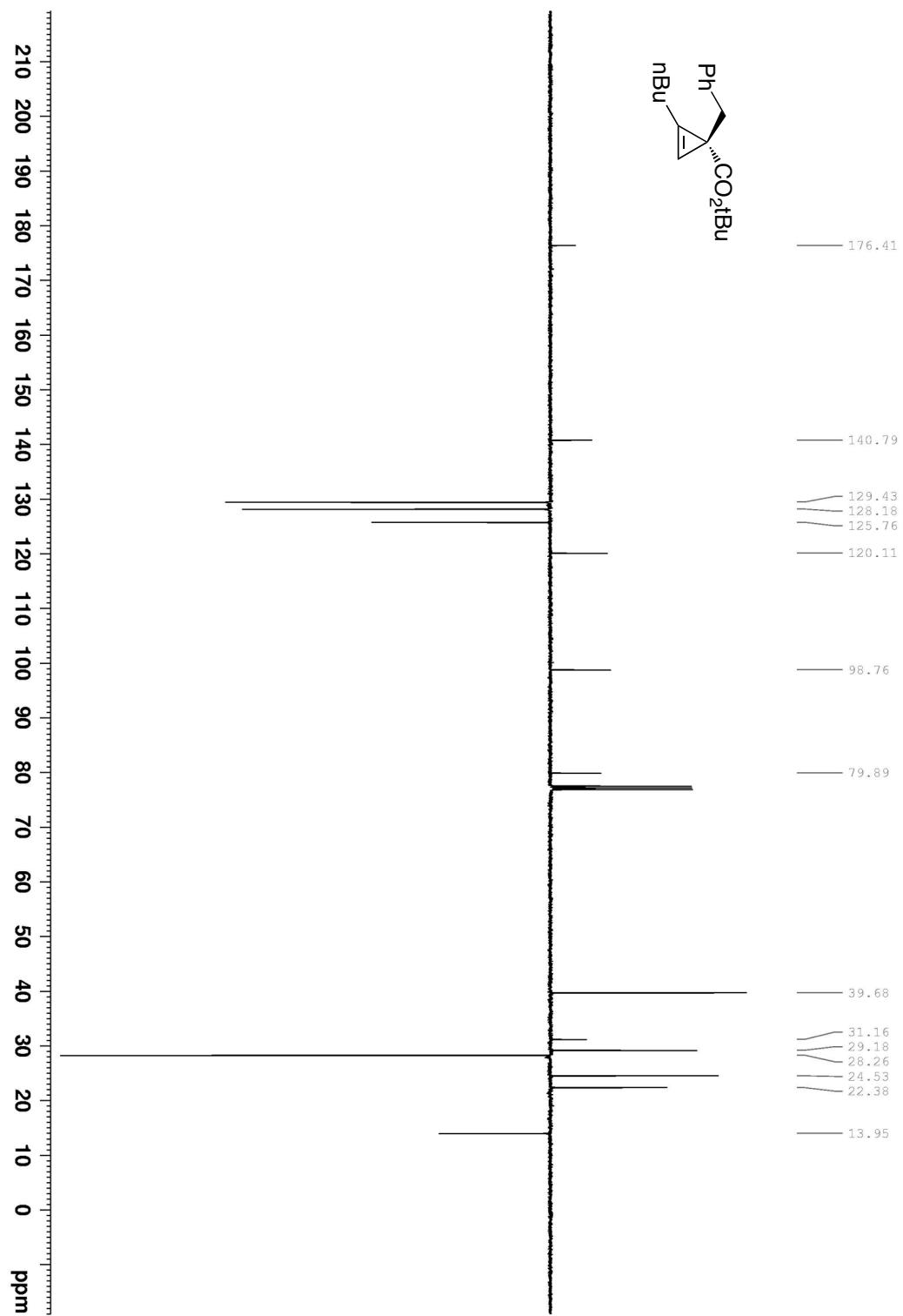


Peak No.	Peak Name	Result (')	Ret. Time (min)	Time Offset (min)	Area (counts)	Sep. Code	Width 1/2 (sec)	Status Codes
1		14.0367	97.941	0.000	26609	BB	17.3	
2		85.9633	98.853	0.000	162956	BB	20.8	
Totals:		100.0000		0.000	189565			

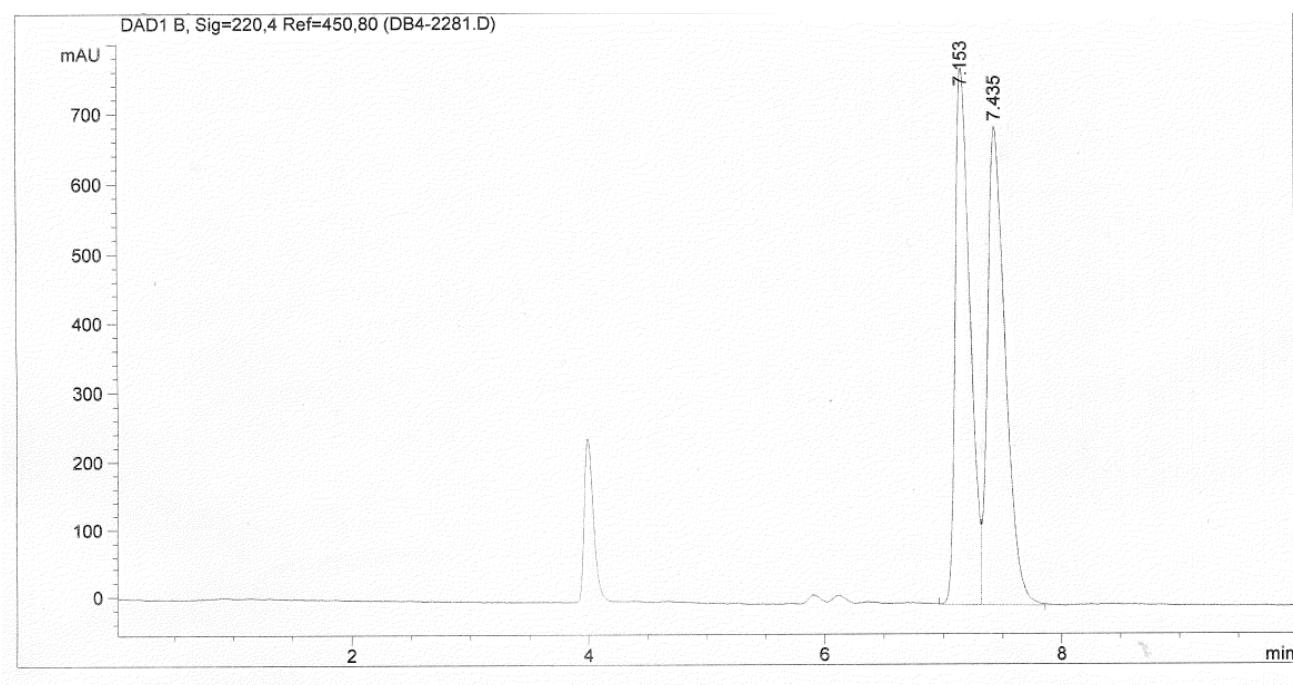
¹H NMR spectrum of **13** (400 MHz, CDCl₃)



^{13}C NMR spectrum of **13** (100 MHz, CDCl_3)



HPLC resolution of racemic **13** (Chiracel IB column, 99:1 hexanes:isopropanol, 1 mL/min, 220 nm)



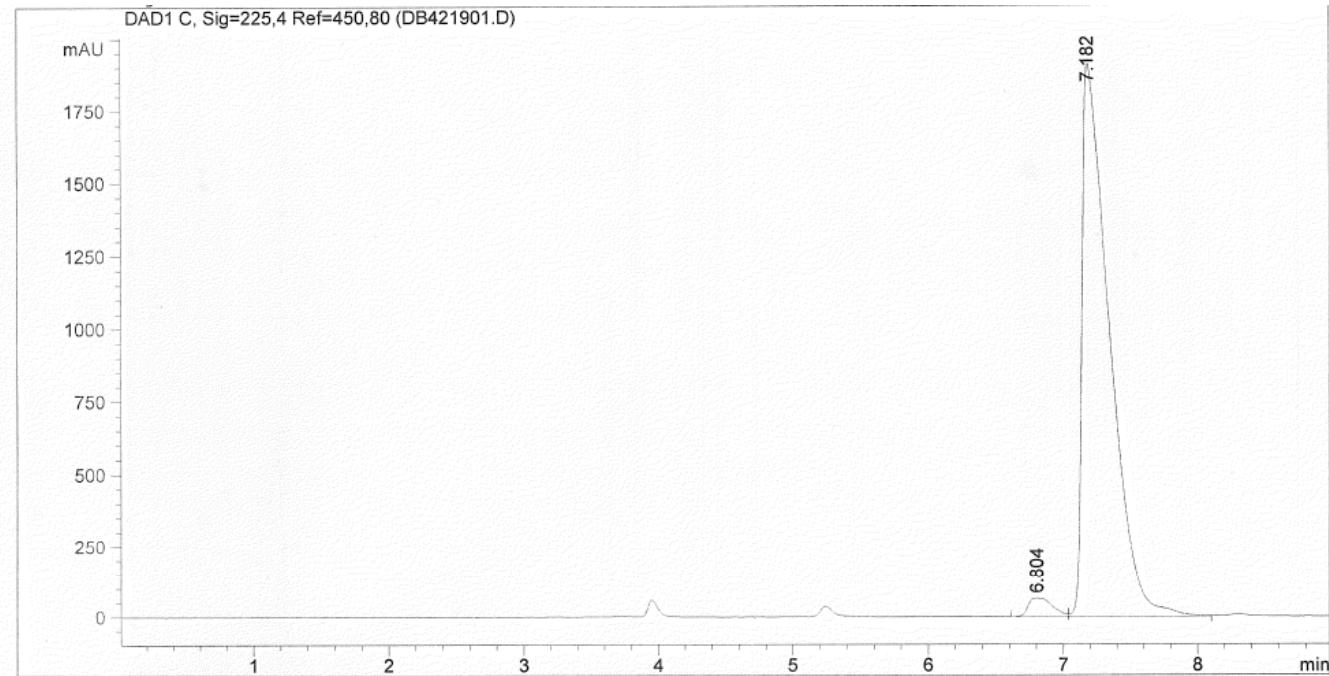
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Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 B, Sig=220,4 Ref=450,80

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.153	VV	0.1363	6742.84961	777.97626	48.0208
2	7.435	VV	0.1646	7298.66943	692.54535	51.9792

Enantiomeric excess of **13** (Chiracel IB column, 99:1 hexanes:isopropanol, 1 mL/min, 225nm)



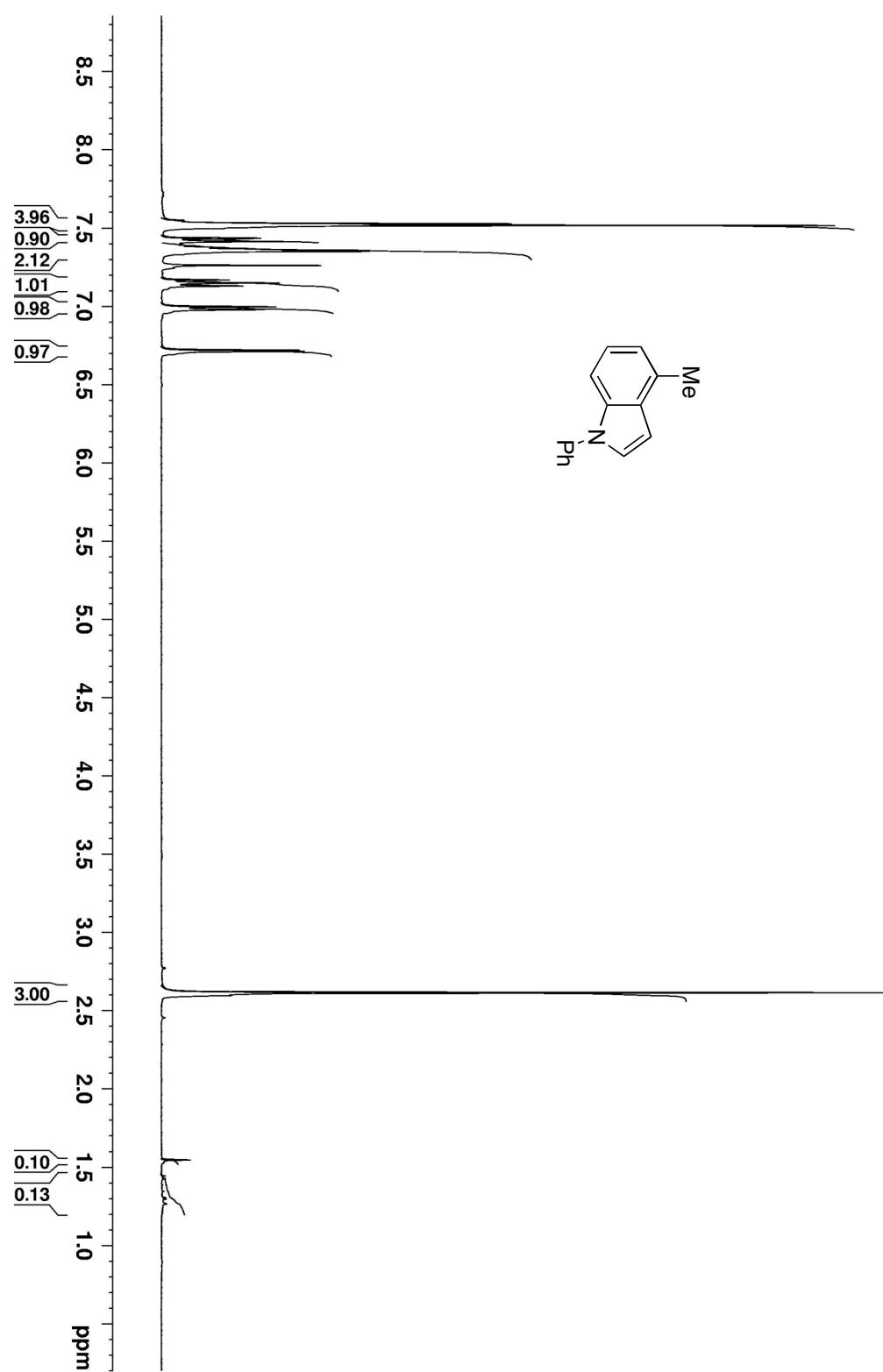
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Area Percent Report
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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

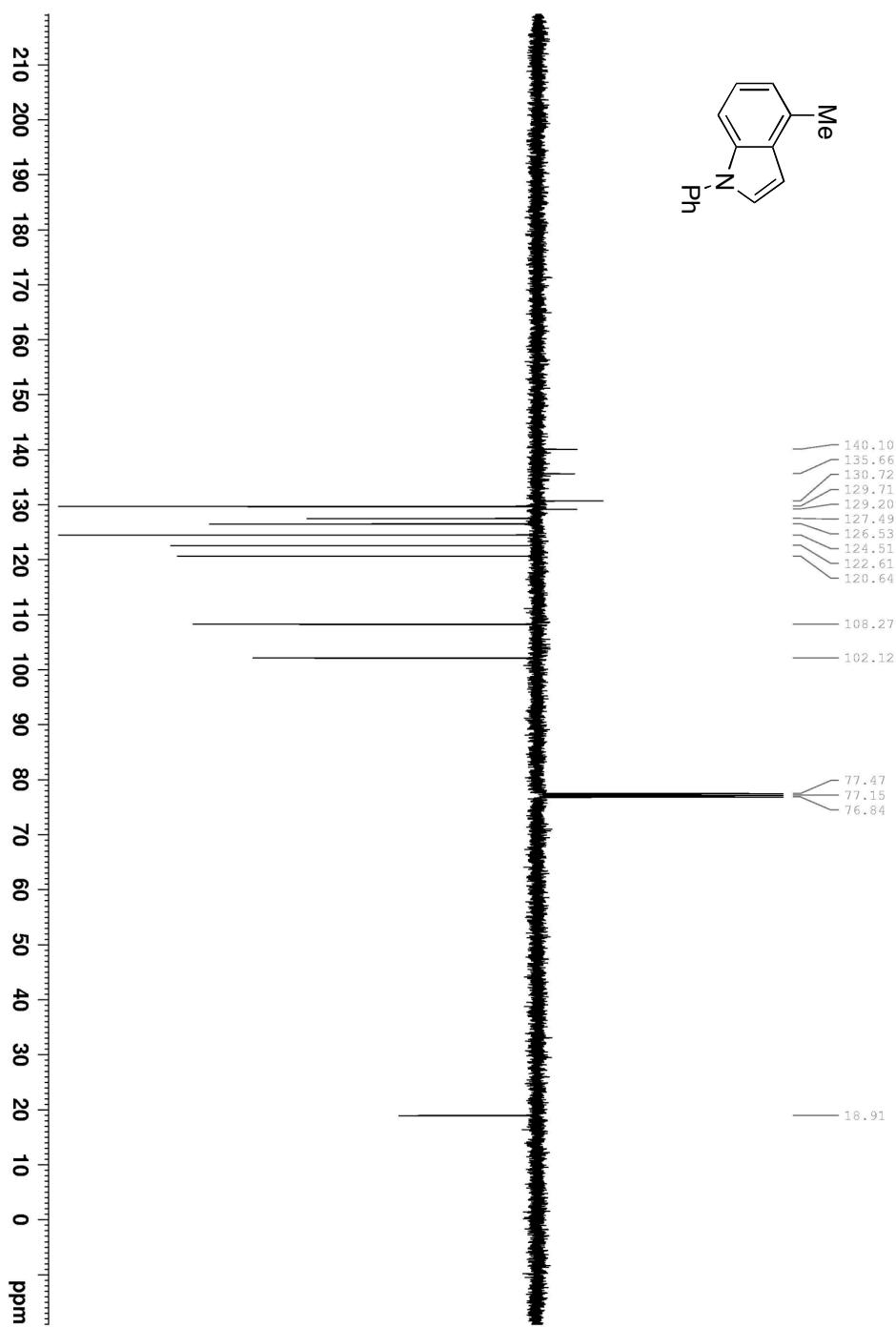
Signal 1: DAD1 C, Sig=225,4 Ref=450,80

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.804	VV	0.1724	809.45288	65.00059	2.9136
2	7.182	VV	0.1933	2.69728e4	1913.40662	97.0864

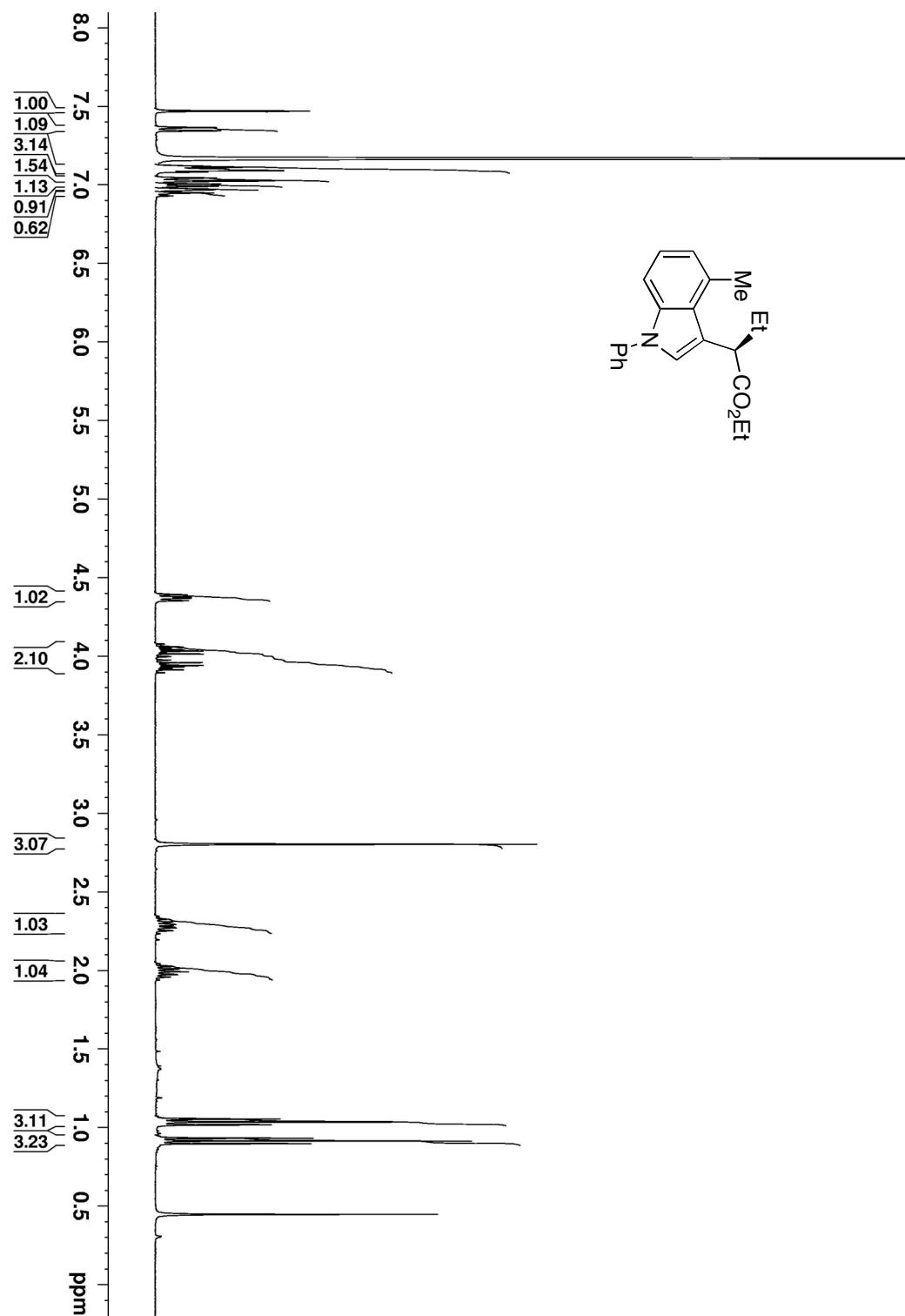
¹H NMR spectrum of **14** (400 MHz, CDCl₃)



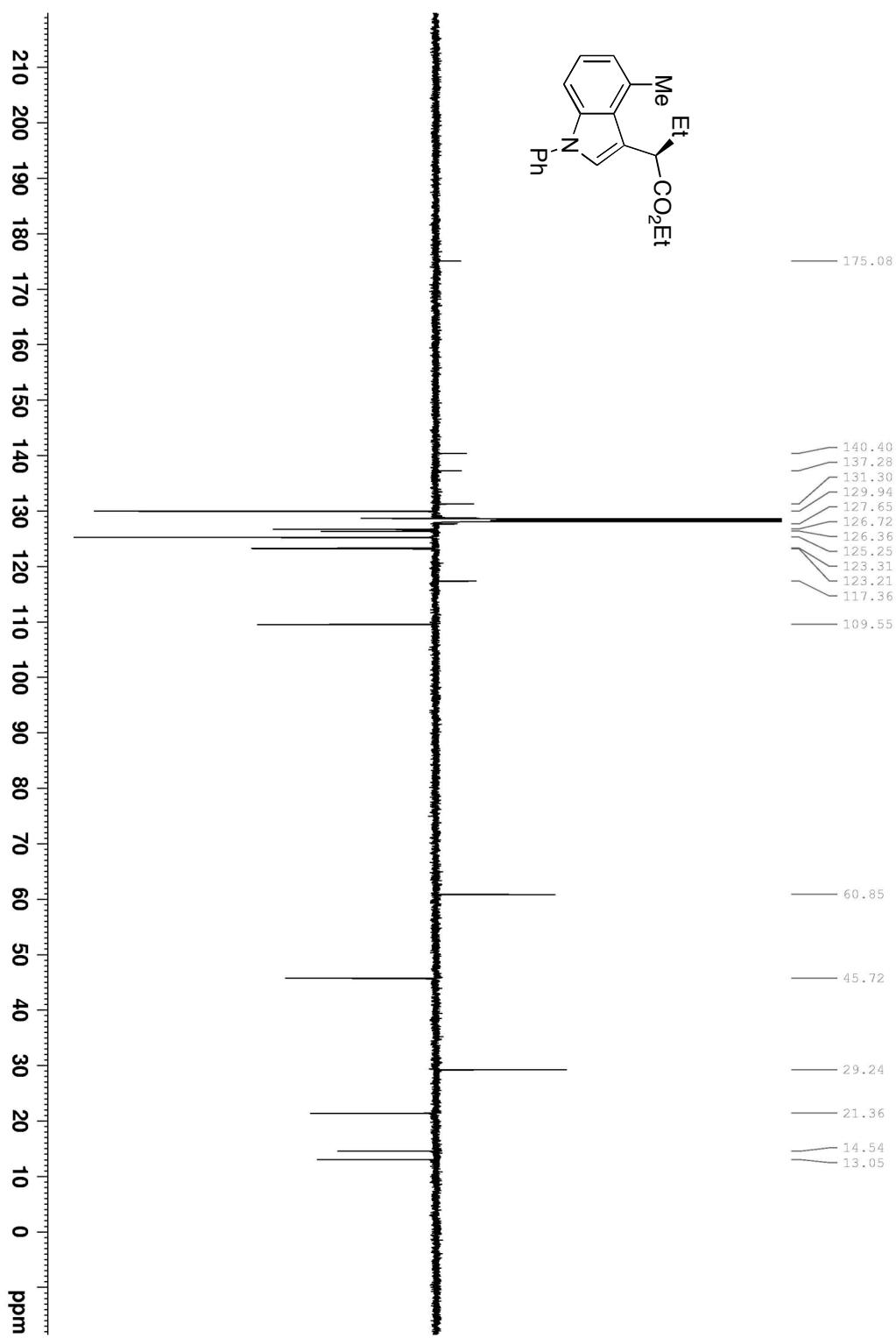
^{13}C NMR spectrum of **14** (100 MHz, CDCl_3)



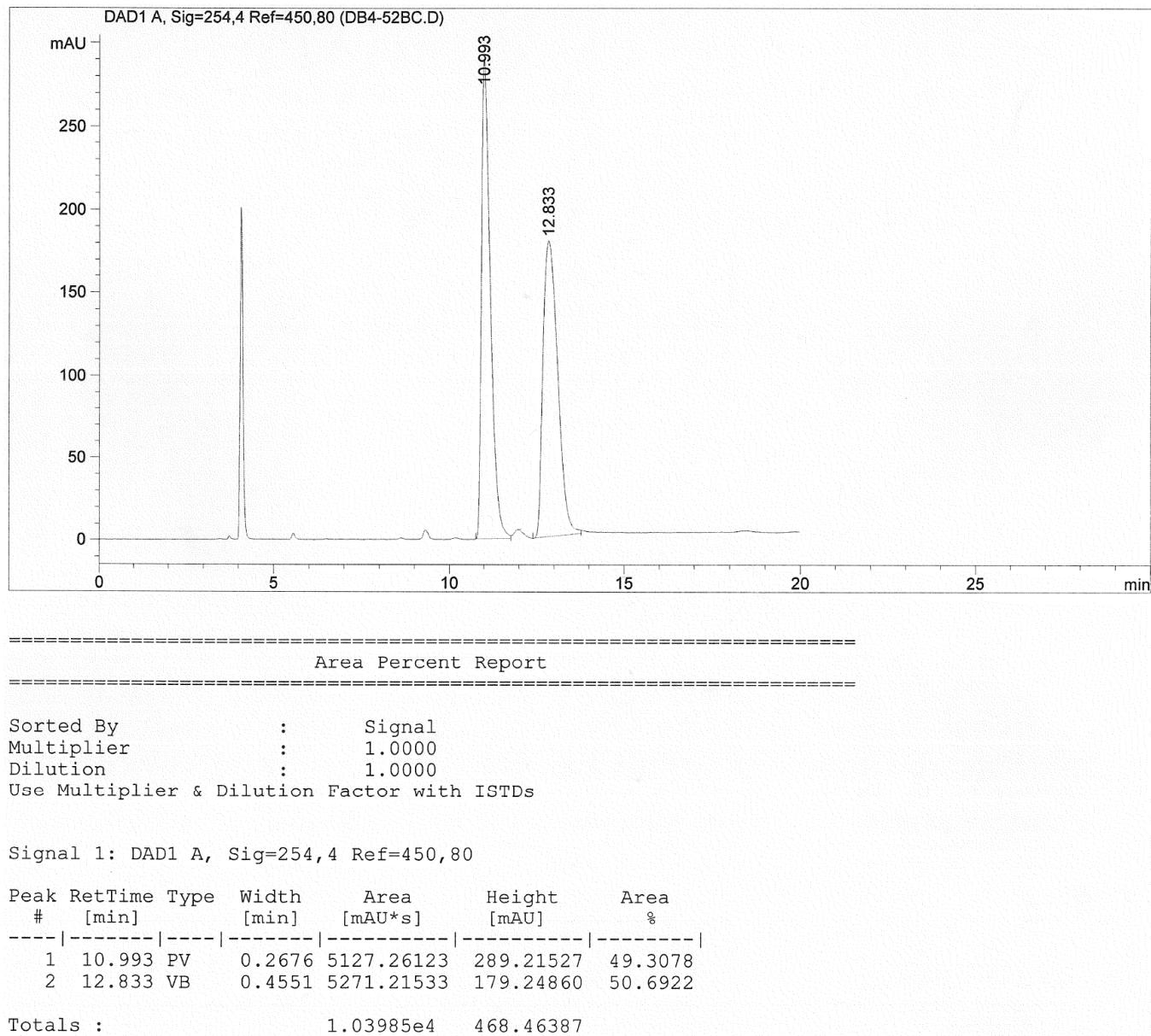
¹H NMR spectrum of **15** (400 MHz, C₆D₆)



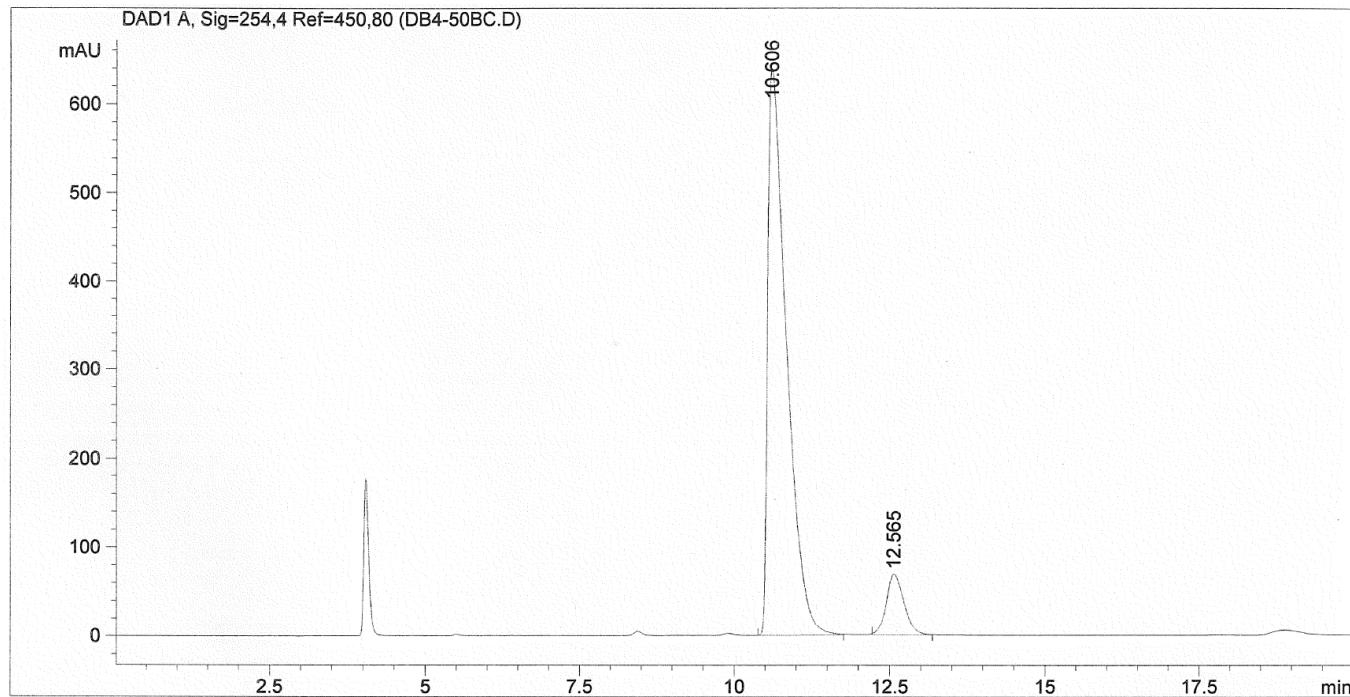
¹³C NMR spectrum of **15** (100 MHz, C₆D₆)



HPLC resolution of racemic **15** (Chiracel IB column, 99:1 hexanes:isopropanol, 1 mL/min, 254 nm)



Enantiomeric excess of **15** (Chiracel IB column, 99:1 hexanes:isopropanol, 1 mL/min,
254nm)



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Area Percent Report
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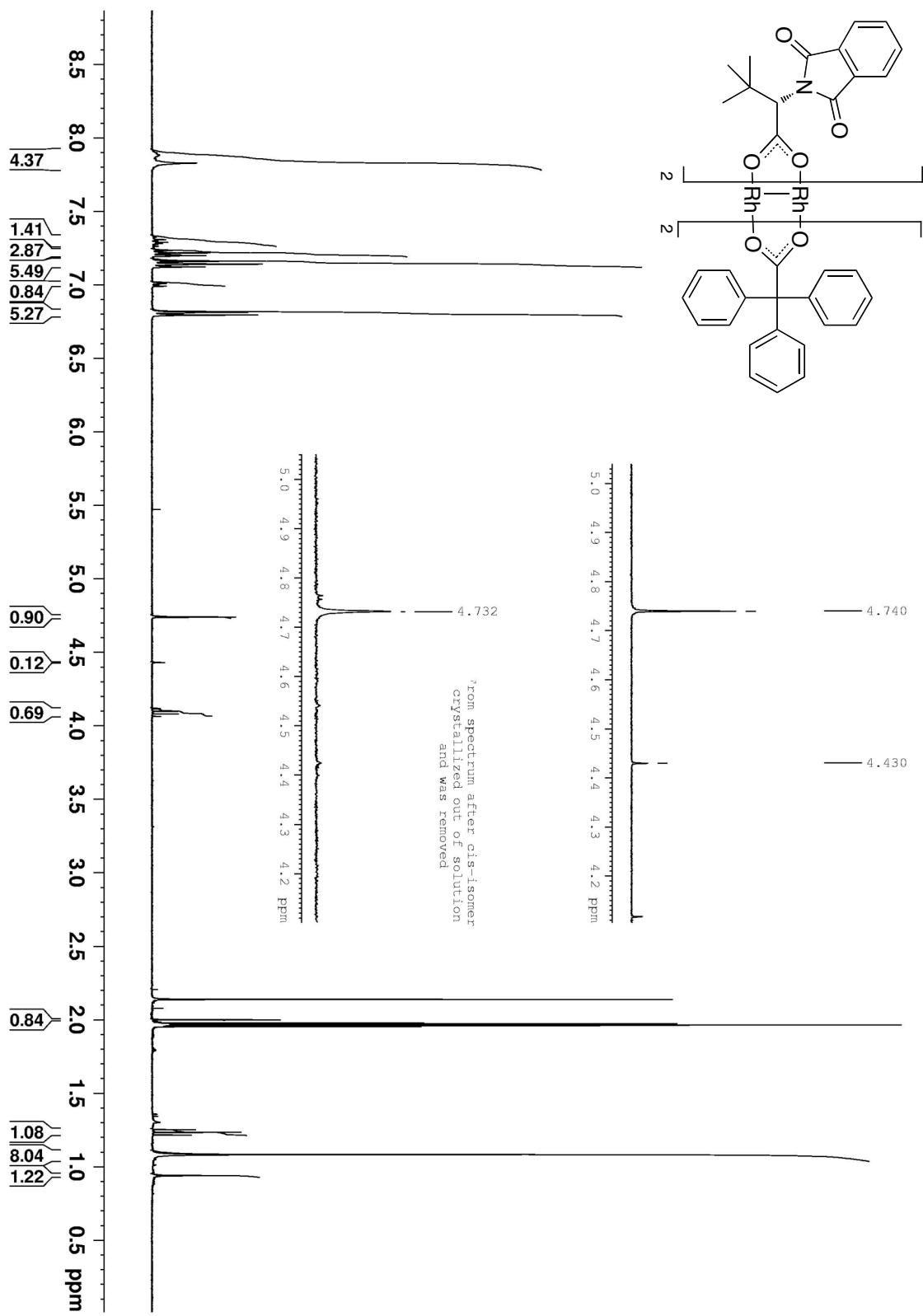
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=450,80

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.606	BB	0.2987	1.33472e4	637.49329	90.6329
2	12.565	BB	0.3072	1379.45874	68.88647	9.3671

Totals : 1.47266e4 706.37976

¹H NMR spectrum of Rh₂(S-PTTL)₂(TPA)₂ + ethyl acetate adduct (400 MHz, CD₃CN)



^{13}C NMR spectrum of $\text{Rh}_2(\text{S-PTTL})_2(\text{TPA})_2$ (100 MHz, CD_3CN)

