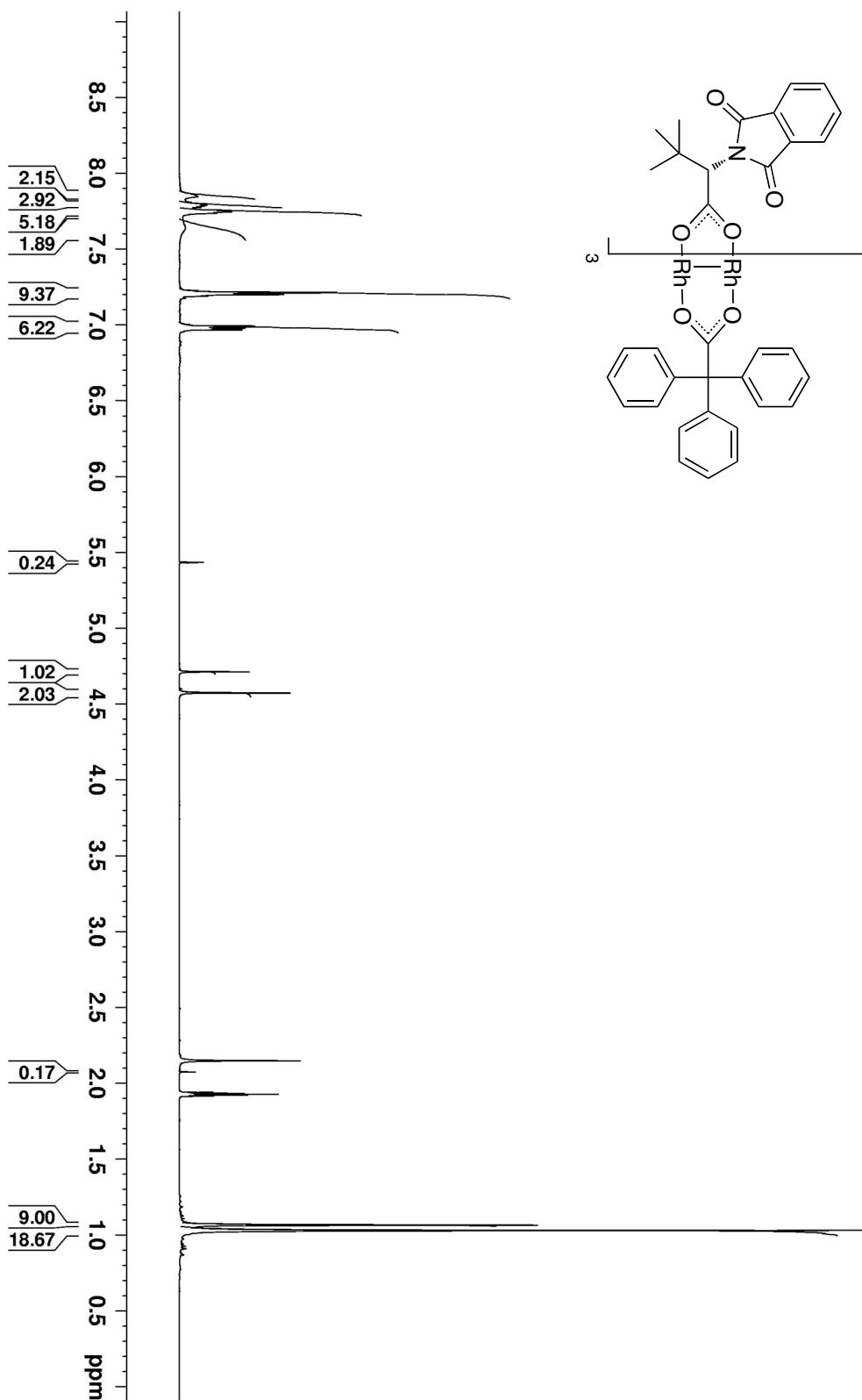
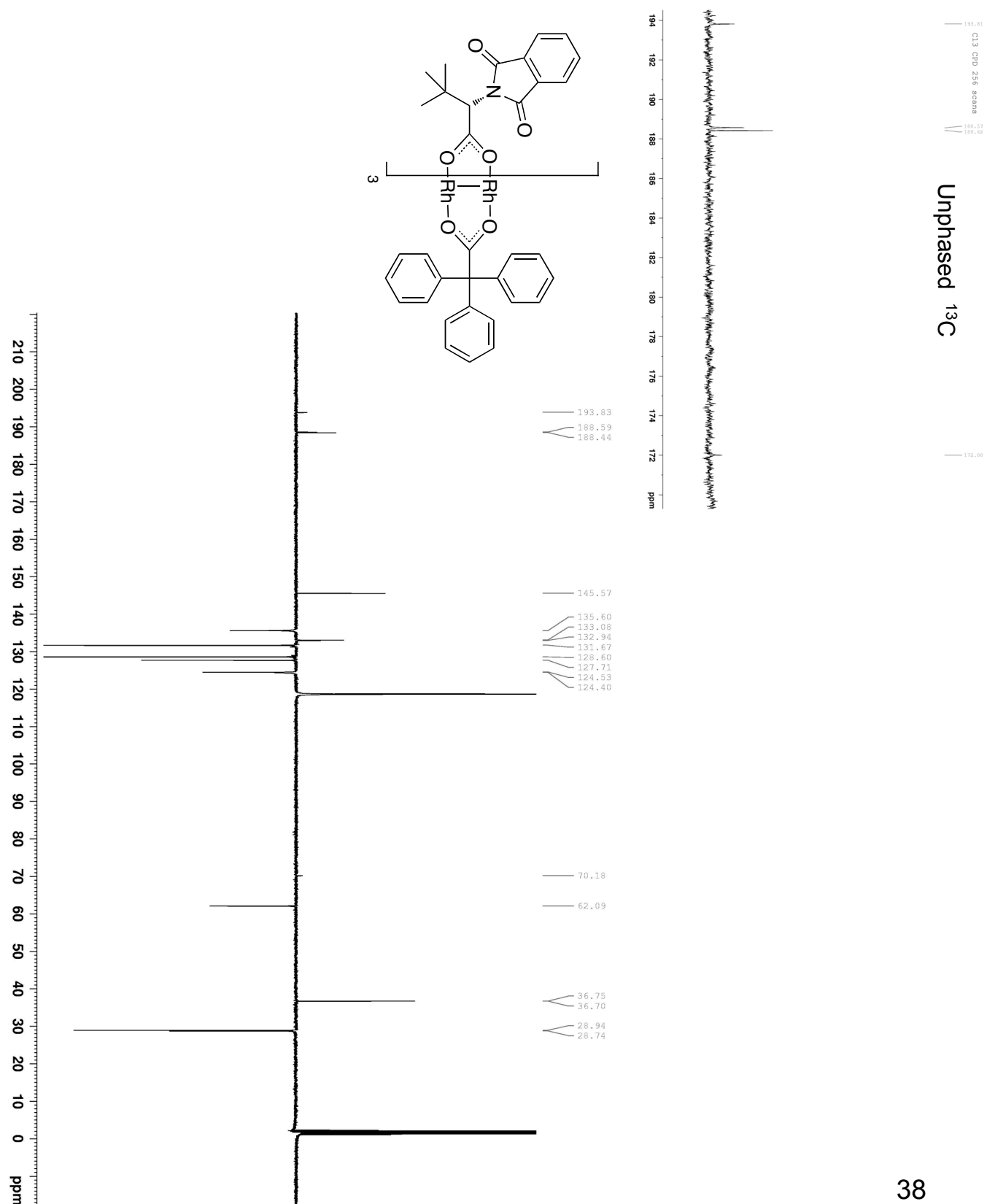


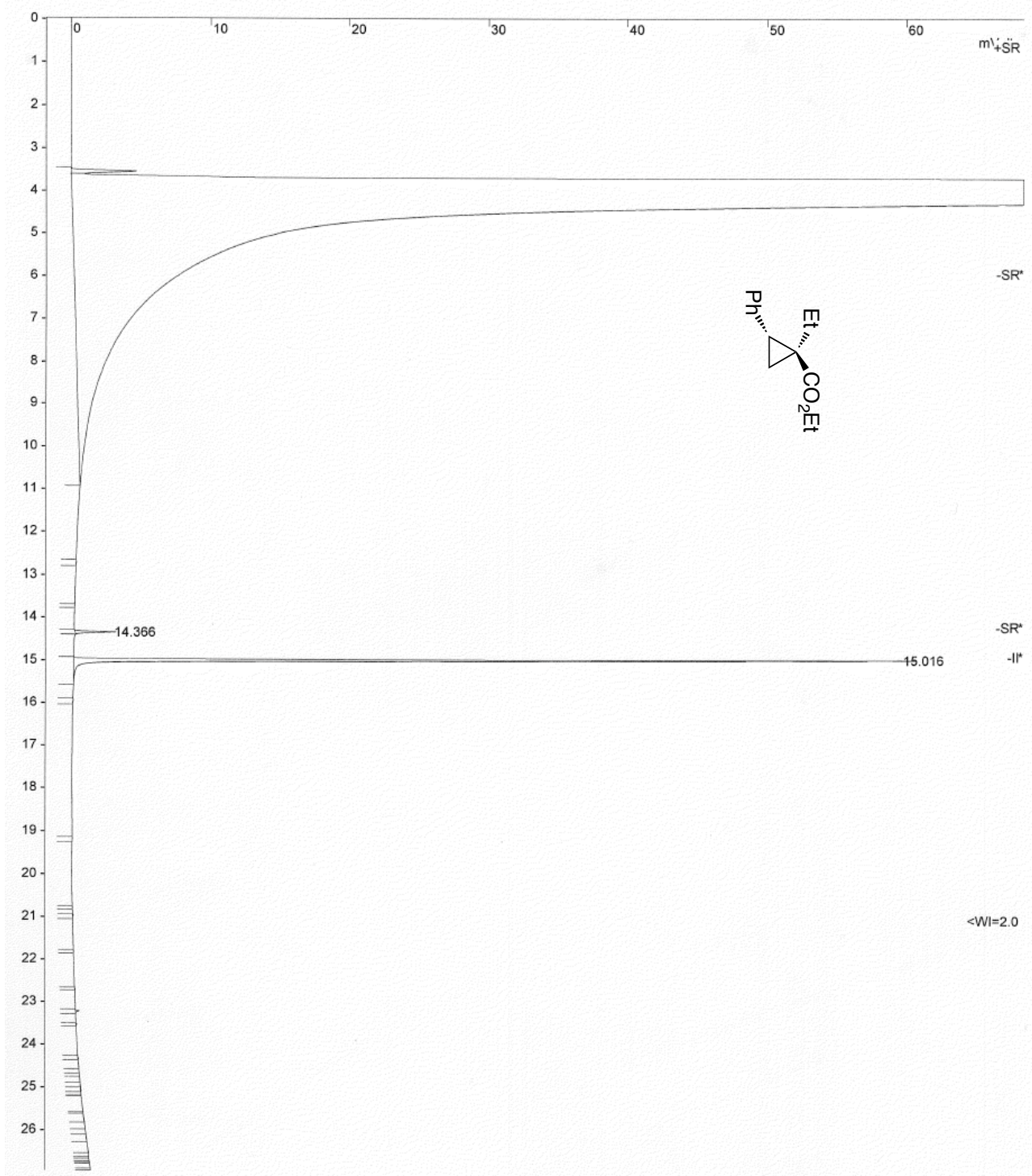
^1H NMR spectrum of A (400 MHz, CD_3CN)



¹³C NMR spectrum of A (100 MHz, CD₃CN)

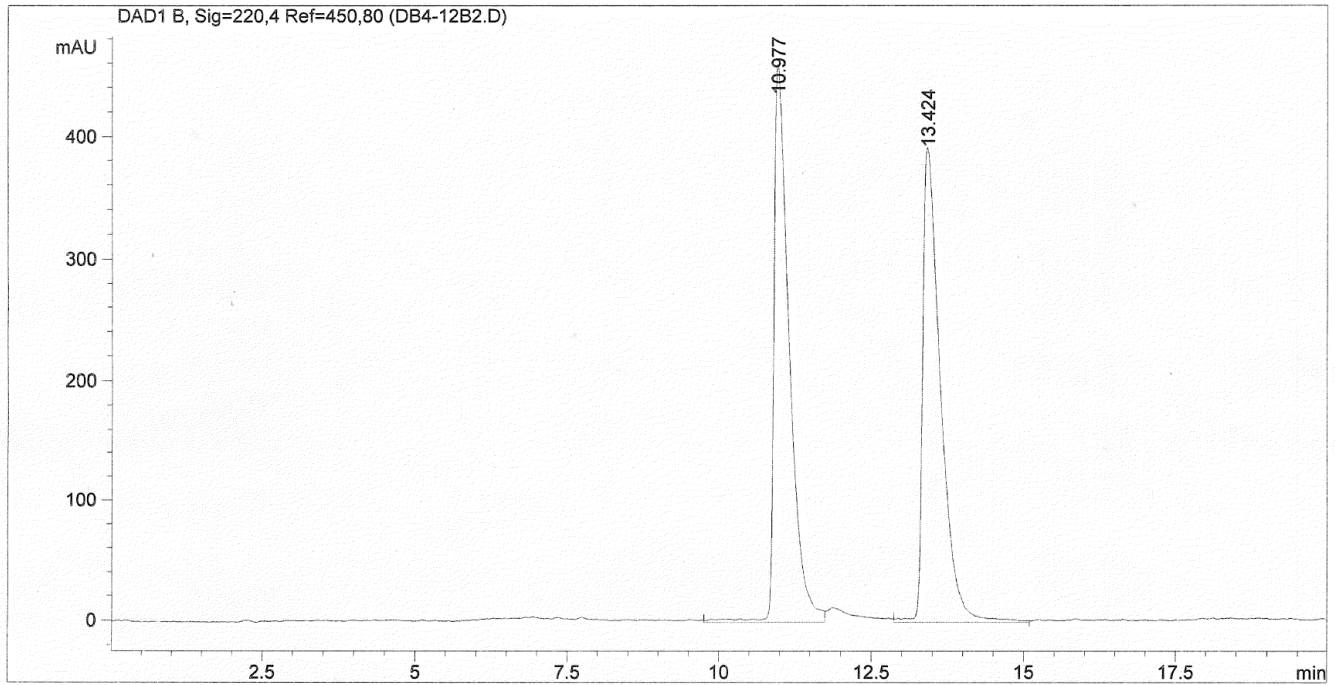


GC trace of 1 (Hewlett Packard HP5 column, 200 °C injection, 50–300 °C over 27 min)



| 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 | | | |

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



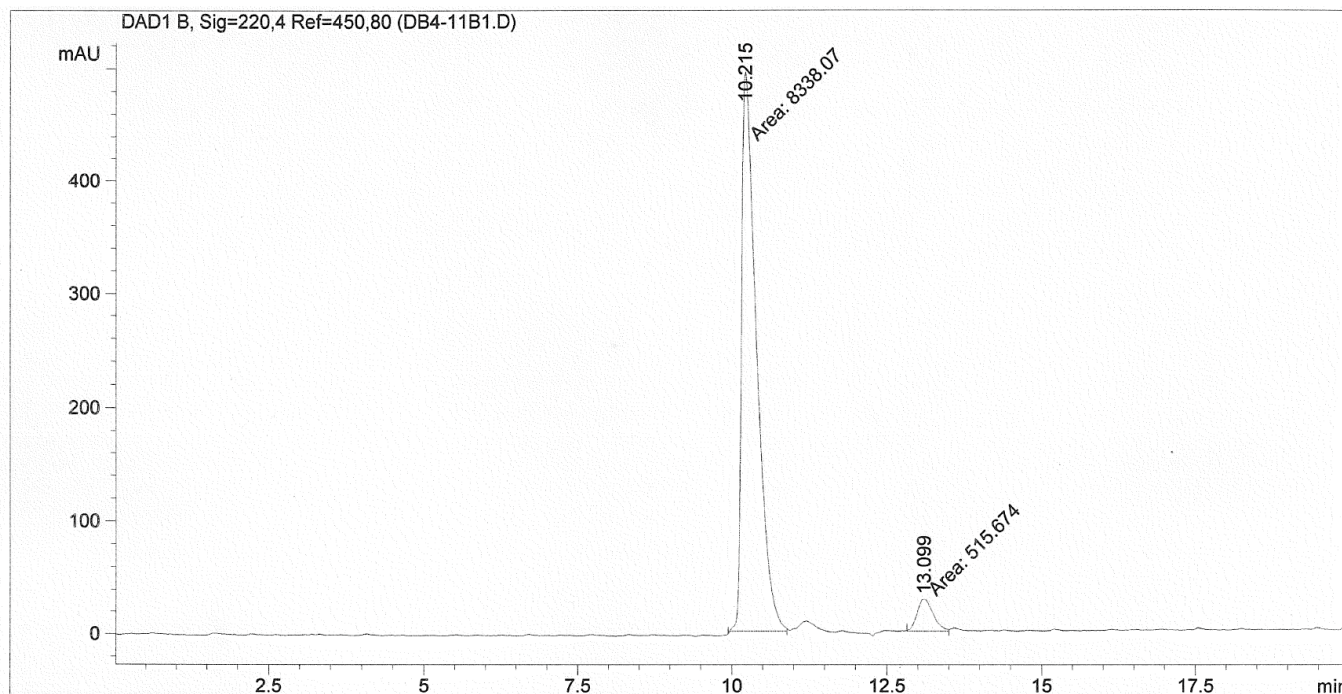
=====
Area Percent Report
=====

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 | 10.977 | 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)



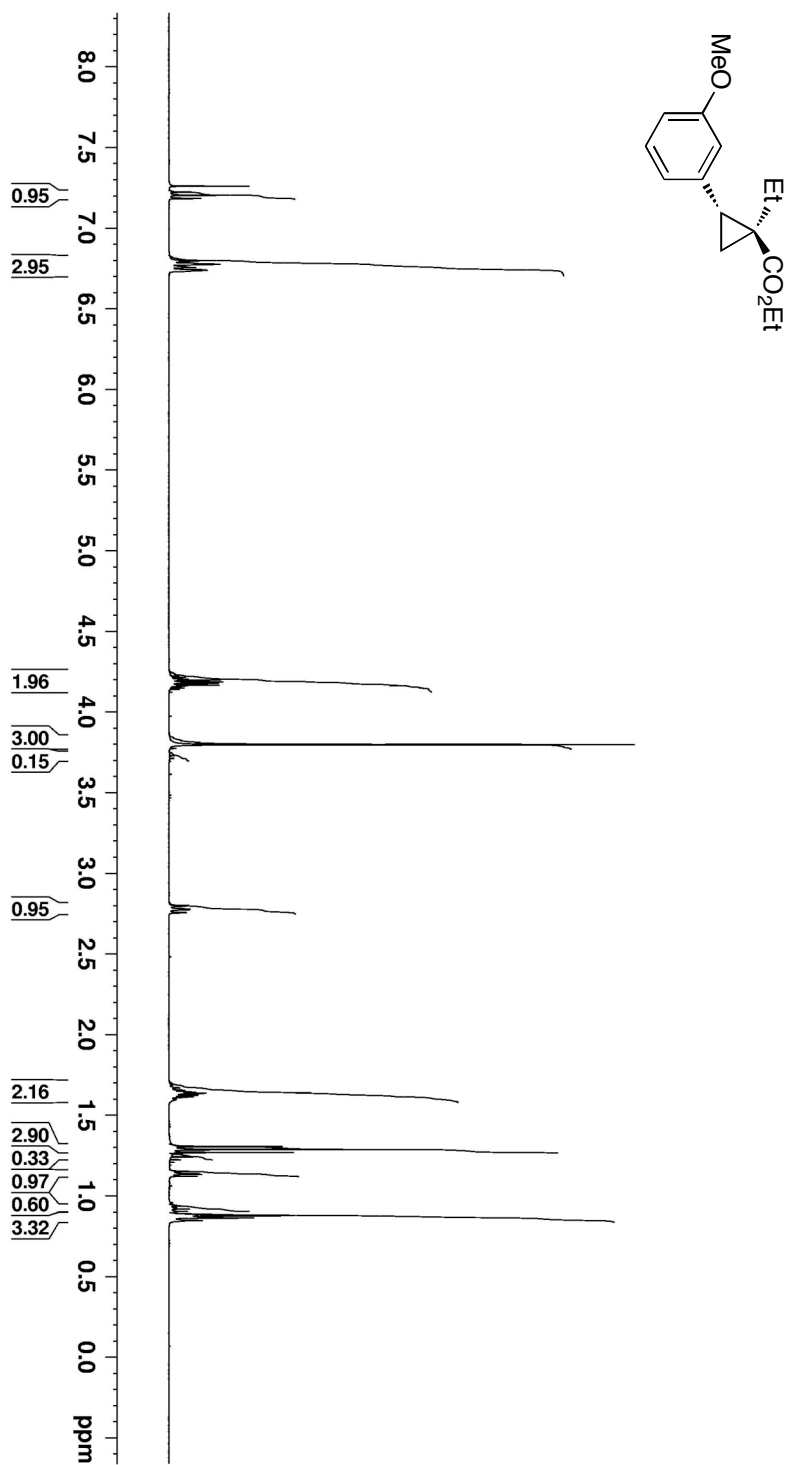
=====
Area Percent Report
=====

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

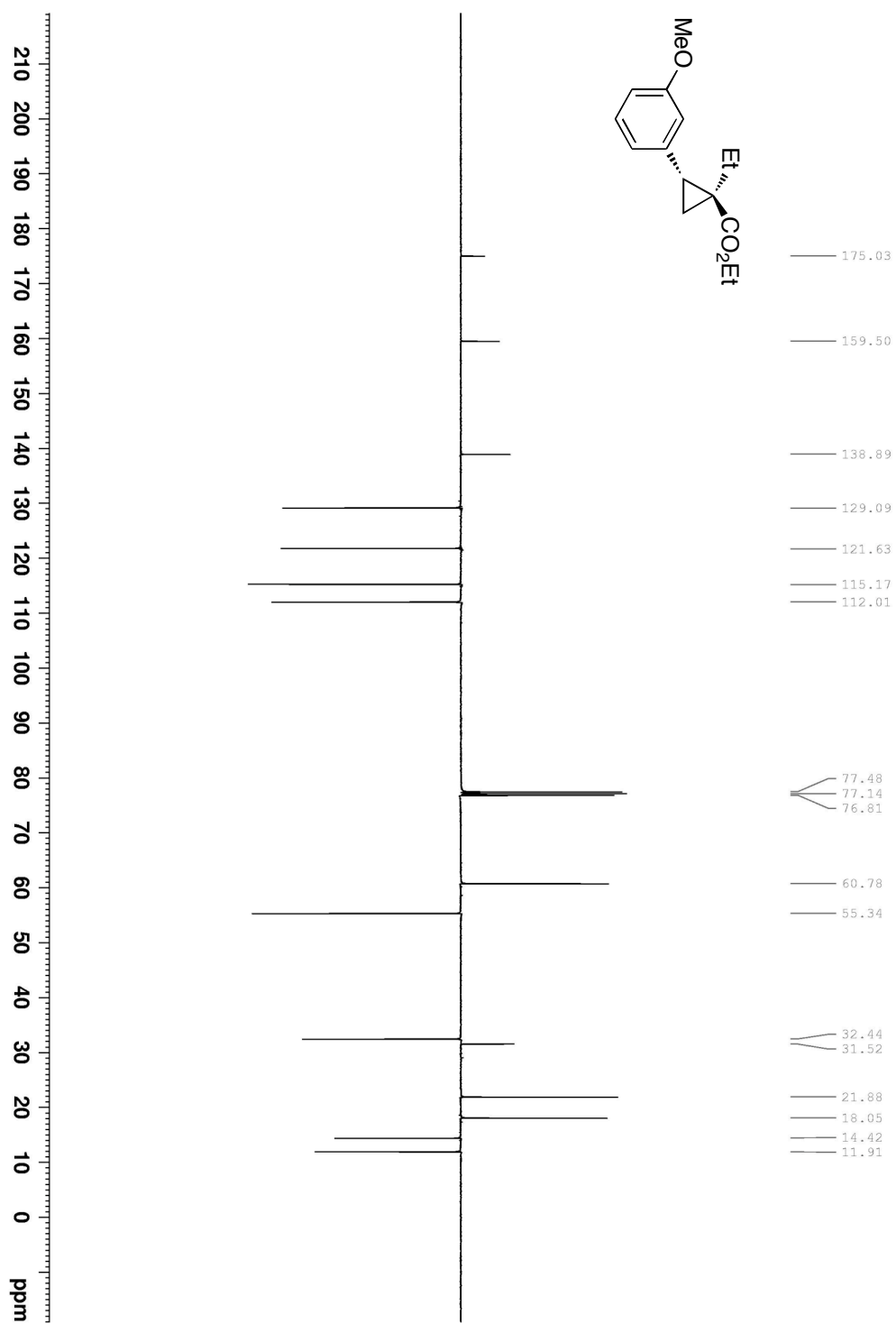
Signal 1: DAD1 B, Sig=220,4 Ref=450,80

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

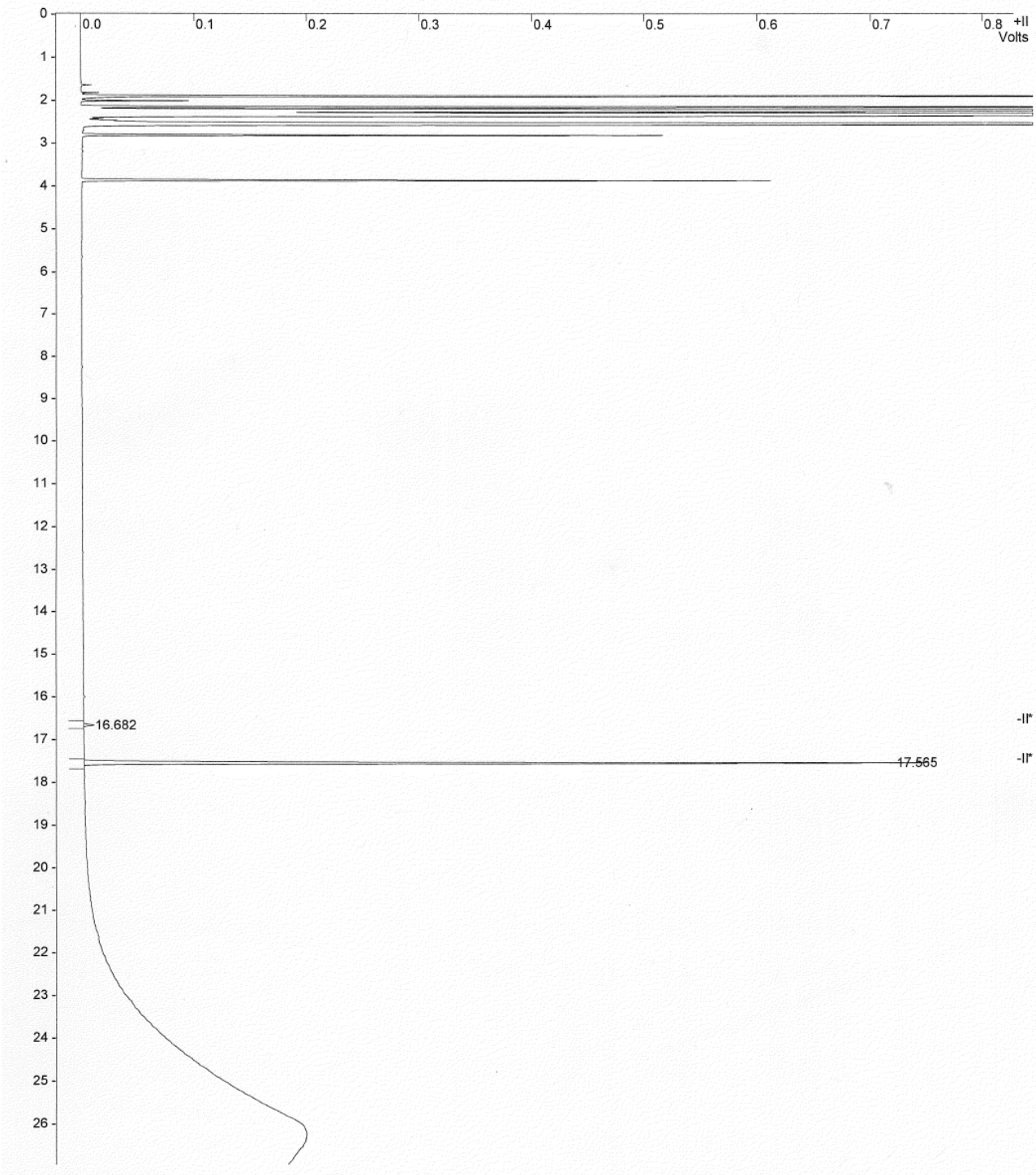
¹H NMR spectrum of **2** (400 MHz, CDCl₃)



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

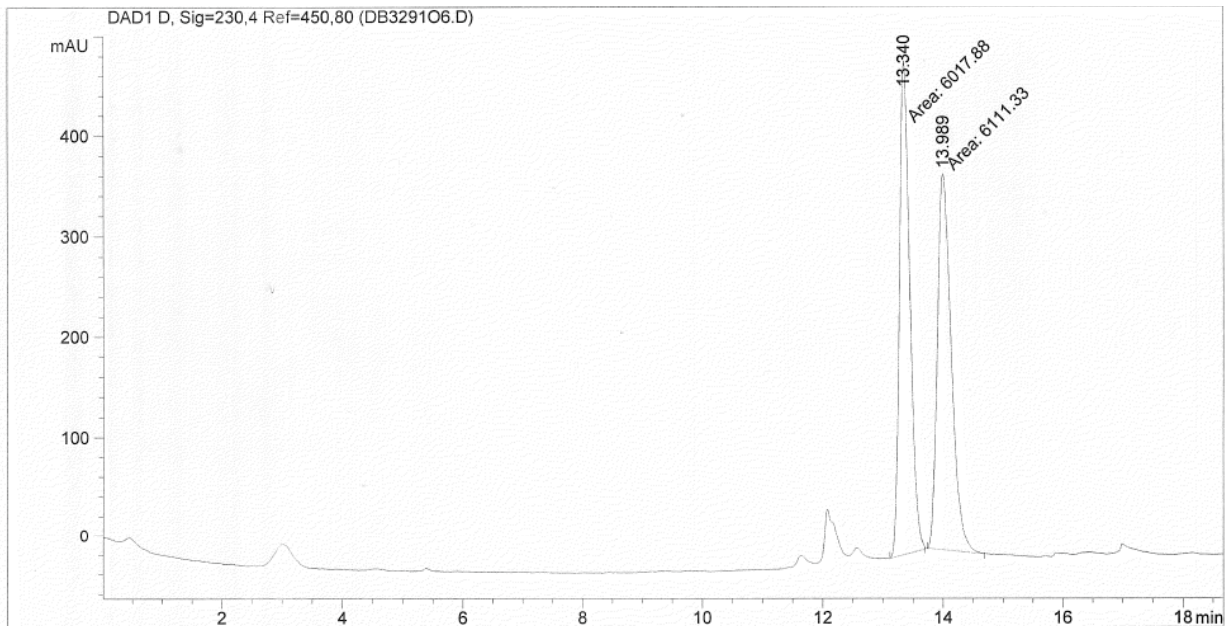


GC trace of 2 (Hewlett Packard HP1 column, 200 °C injection, 50–300 °C over 27 min)



| 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)



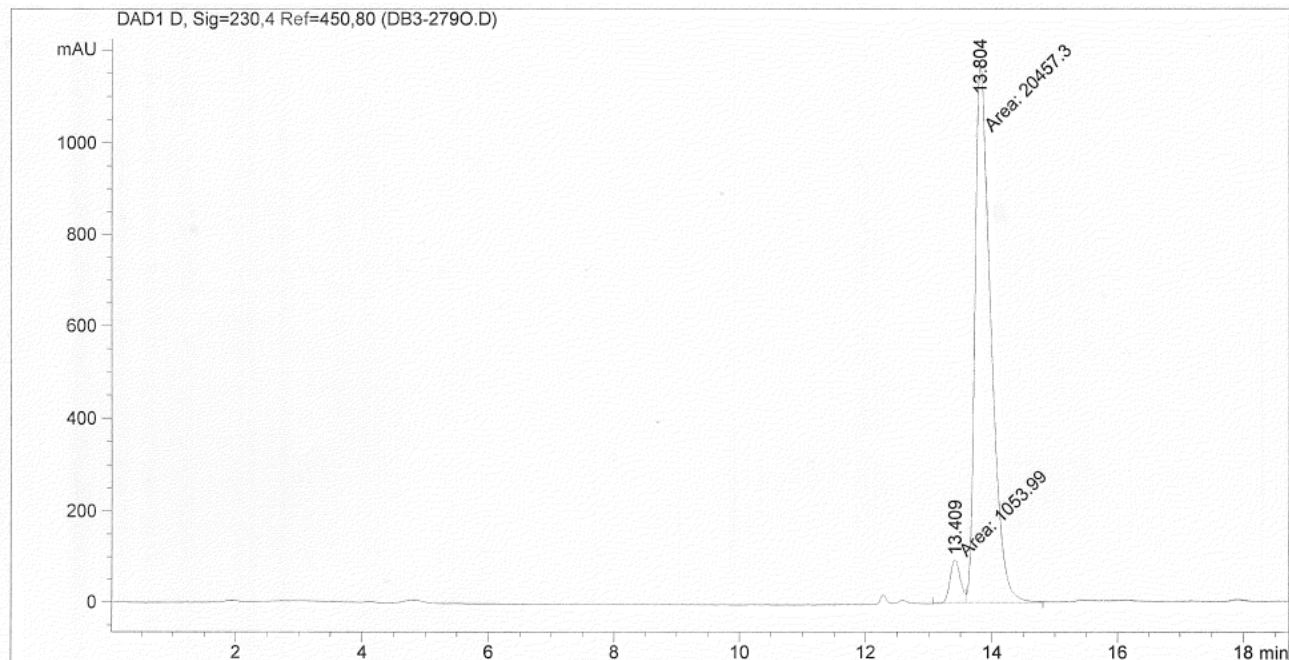
=====
 Area Percent Report
 =====

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 | 13.340 | MM | 0.2022 | 6017.88184 | 496.11420 | 49.6148 |
| 2 | 13.989 | MM | 0.2701 | 6111.32715 | 377.05579 | 50.3852 |

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



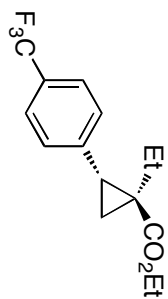
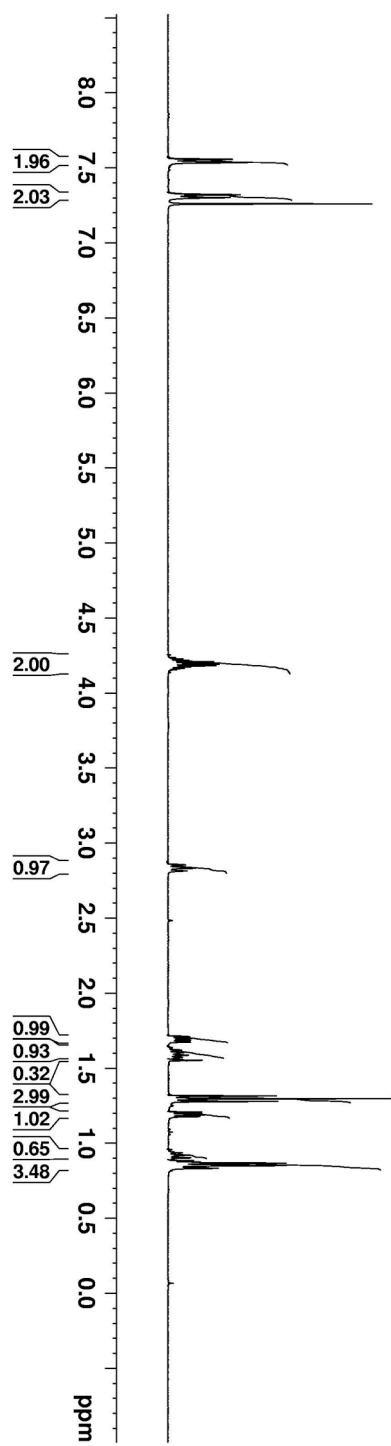
=====
 Area Percent Report
 =====

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

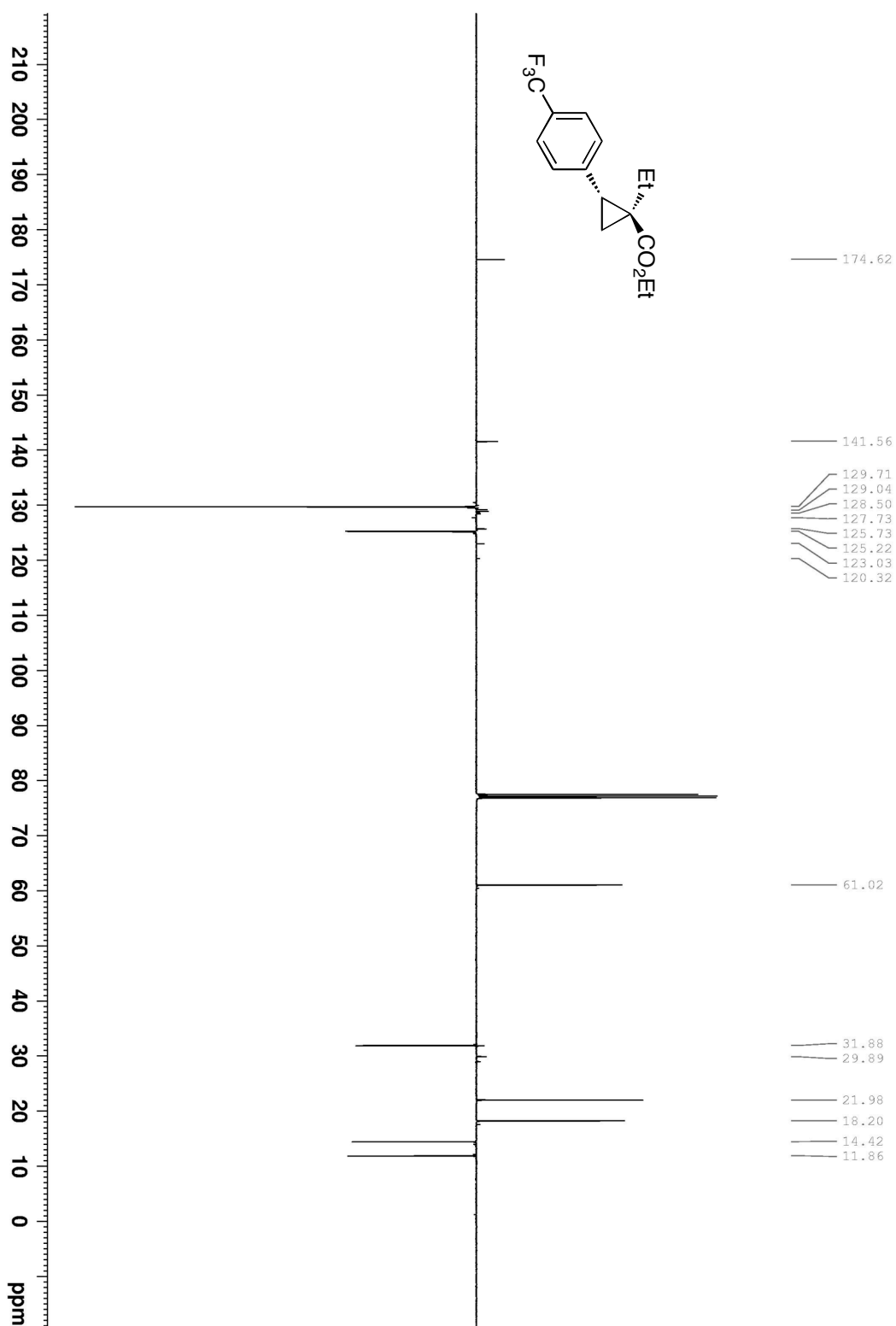
Signal 1: DAD1 D, Sig=230,4 Ref=450,80

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

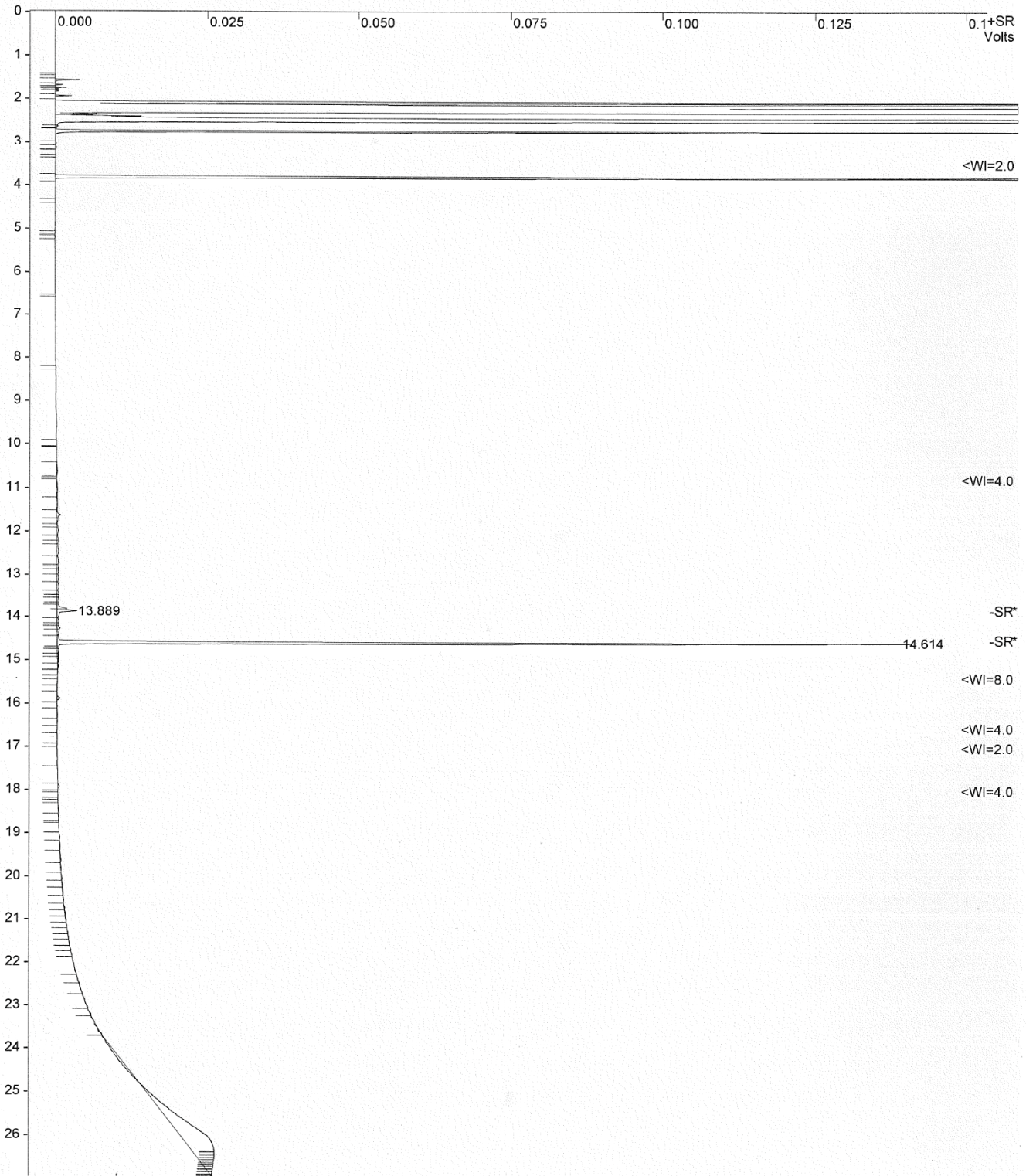
^1H NMR spectrum of **3** (400 MHz, CDCl_3)



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

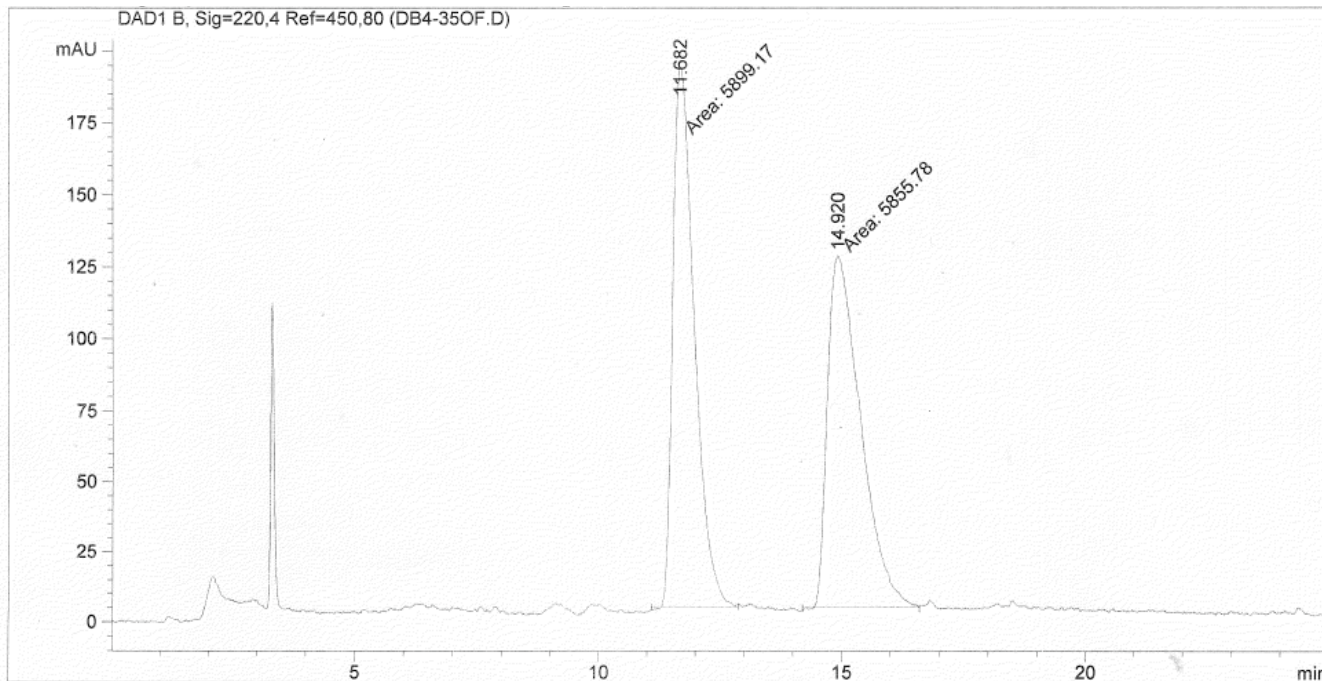


GC trace of 3 (Hewlett Packard HP1 column, 200 °C injection, 50–300 °C over 27 min)



| Peak No. | Peak Name | Result () | Ret. Time (min) | Time Offset (min) | Area (counts) | Sep. Code | Width 1/2 (sec) | Status Codes |
|----------|-----------|------------|-----------------|-------------------|---------------|-----------|-----------------|--------------|
| 1 | | 3.2369 | 13.889 | 0.000 | 11094 | VV | 2.8 | |
| 2 | | 96.7631 | 14.614 | 0.000 | 331638 | VB | 2.2 | |
| Totals: | | 100.0000 | | 0.000 | 342732 | | | |

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



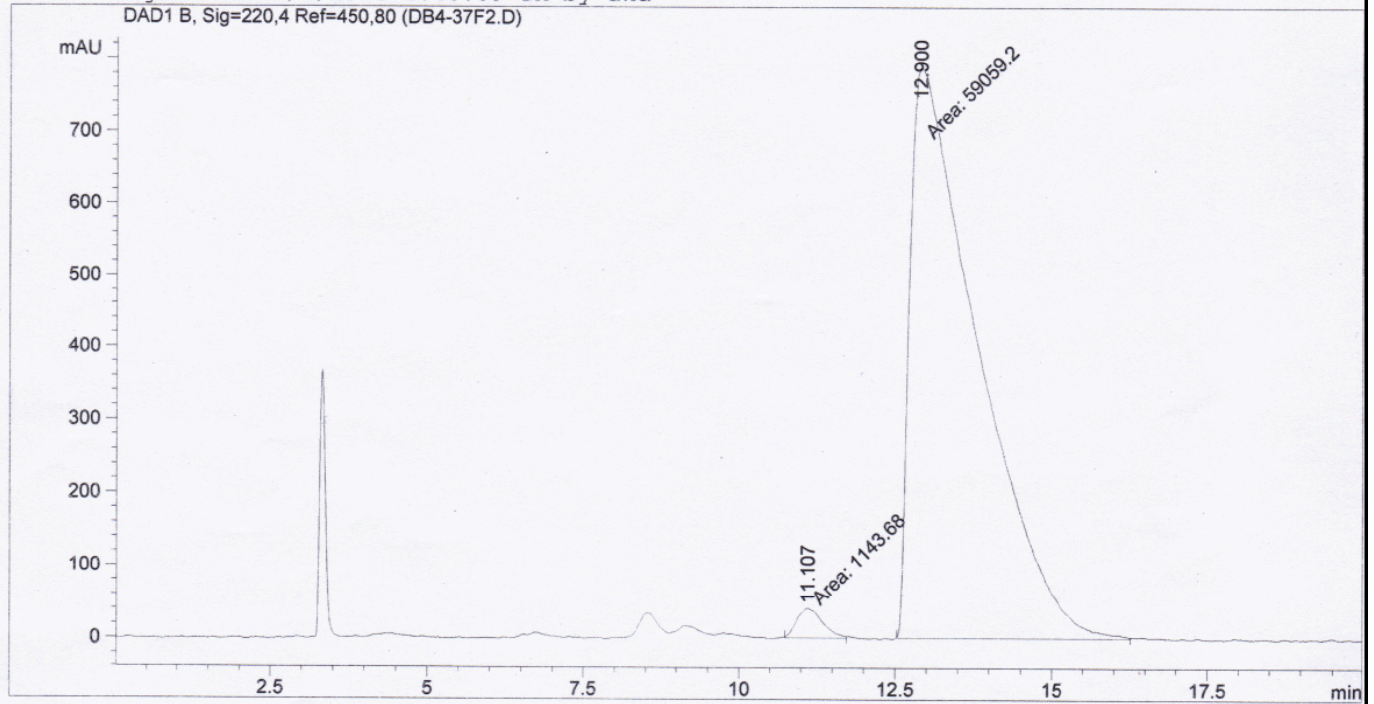
=====
 Area Percent Report
 =====

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 | 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)



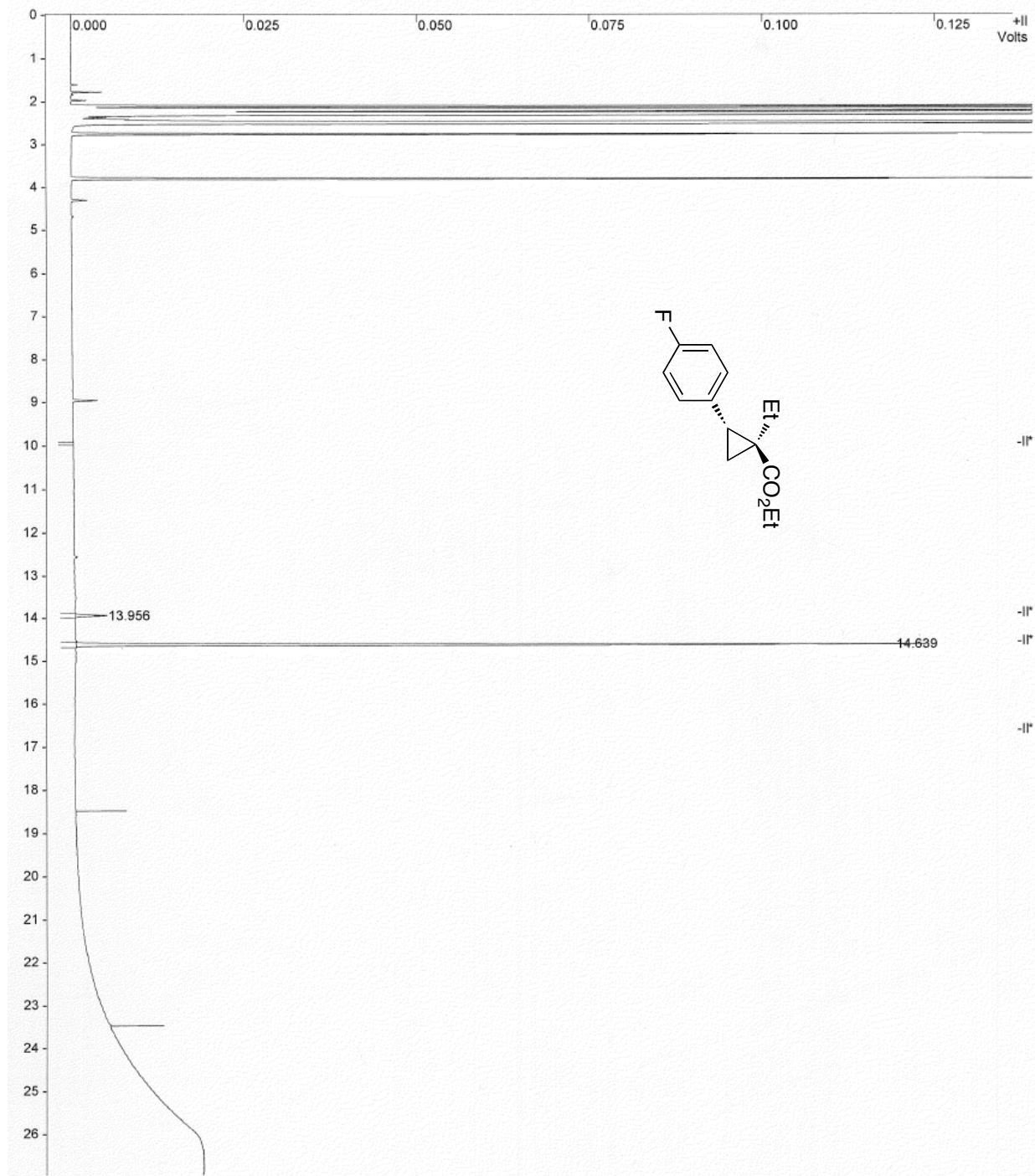
=====
 Area Percent Report
 =====

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

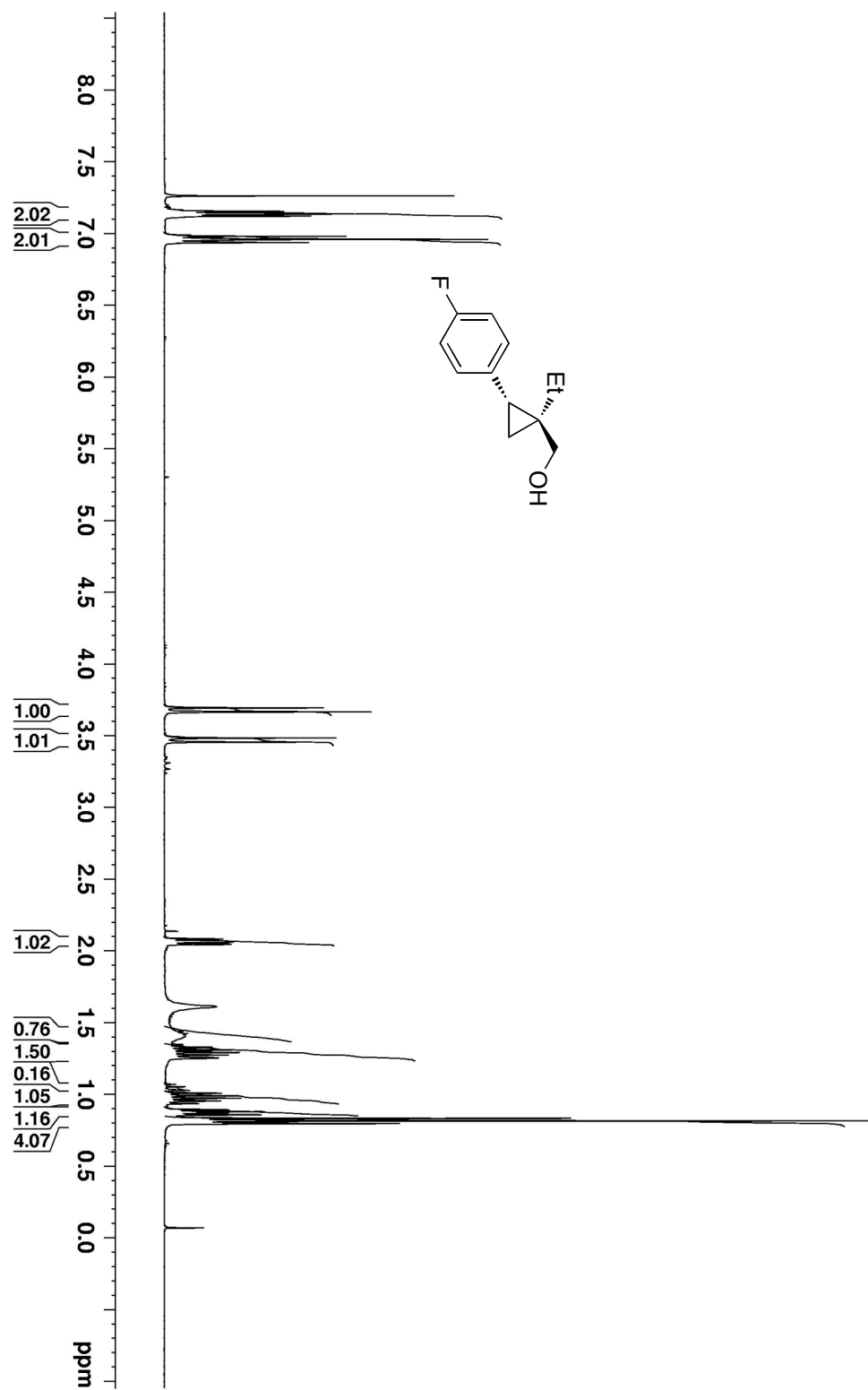
| Peak # | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area % |
|--------|---------------|------|-------------|--------------|--------------|---------|
| 1 | 11.107 | MM | 0.4773 | 1143.68164 | 39.93412 | 1.8997 |
| 2 | 12.900 | MM | 1.2516 | 5.90592e4 | 786.45551 | 98.1003 |

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

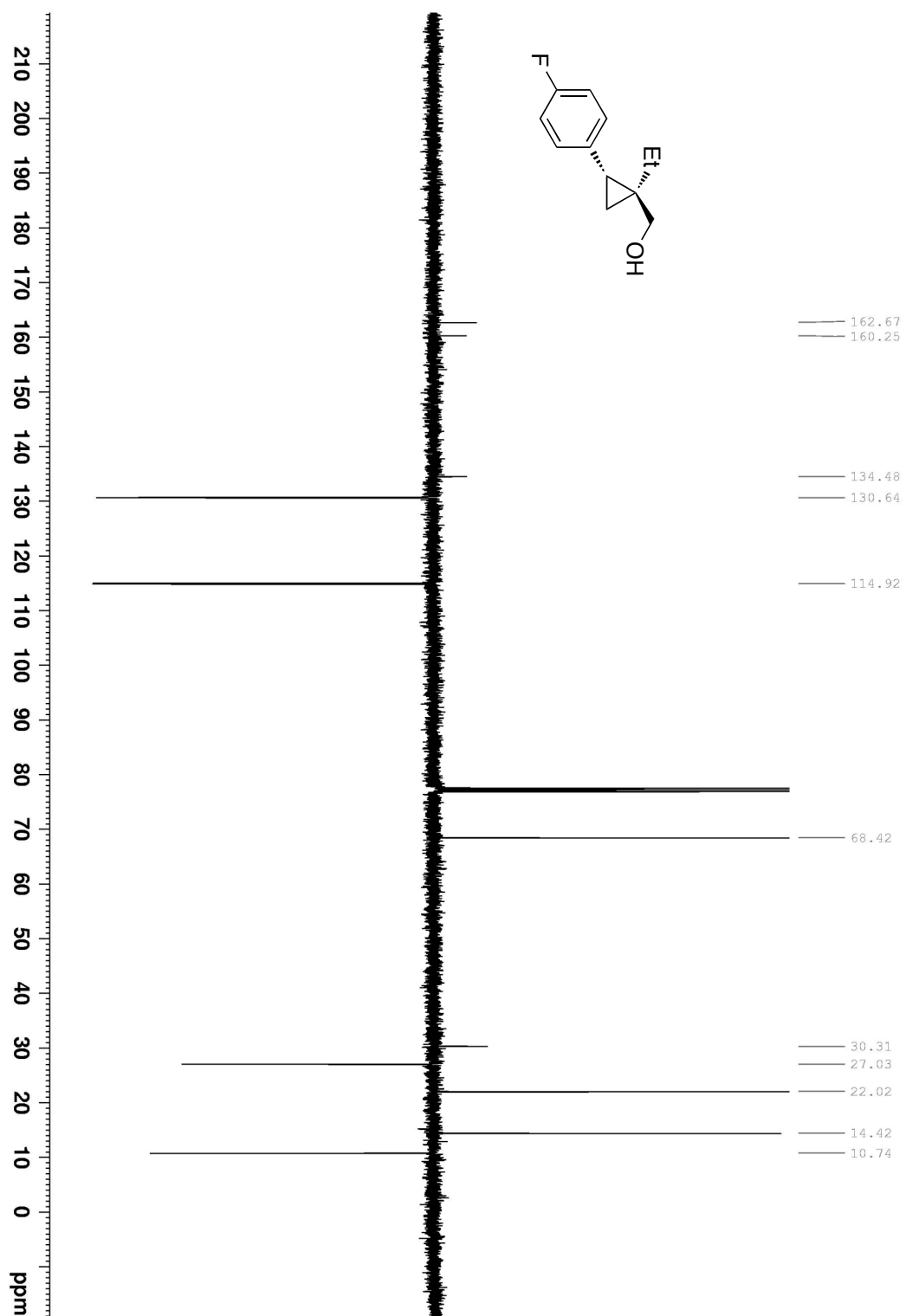


| Peak No. | Peak Name | Result (%) | Ret. Time (min) | Time Offset (min) | Area (counts) | Sep. Code | Width 1/2 (sec) | Status Codes |
|----------|-----------|------------|-----------------|-------------------|---------------|-----------|-----------------|--------------|
| 1 | | 3.4832 | 13.956 | 0.000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

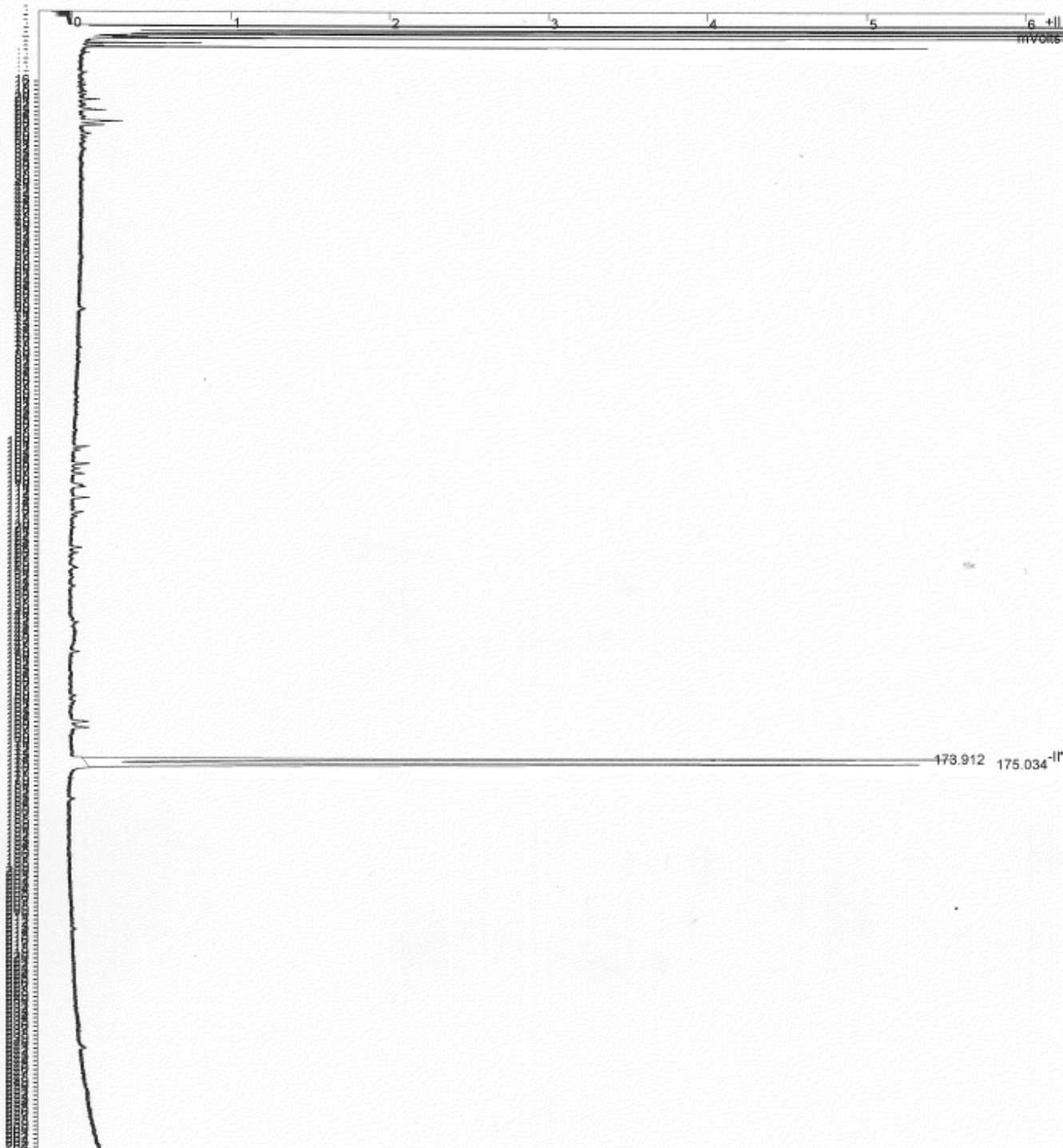
^1H NMR spectrum of **4a** (400 MHz, CDCl_3)



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

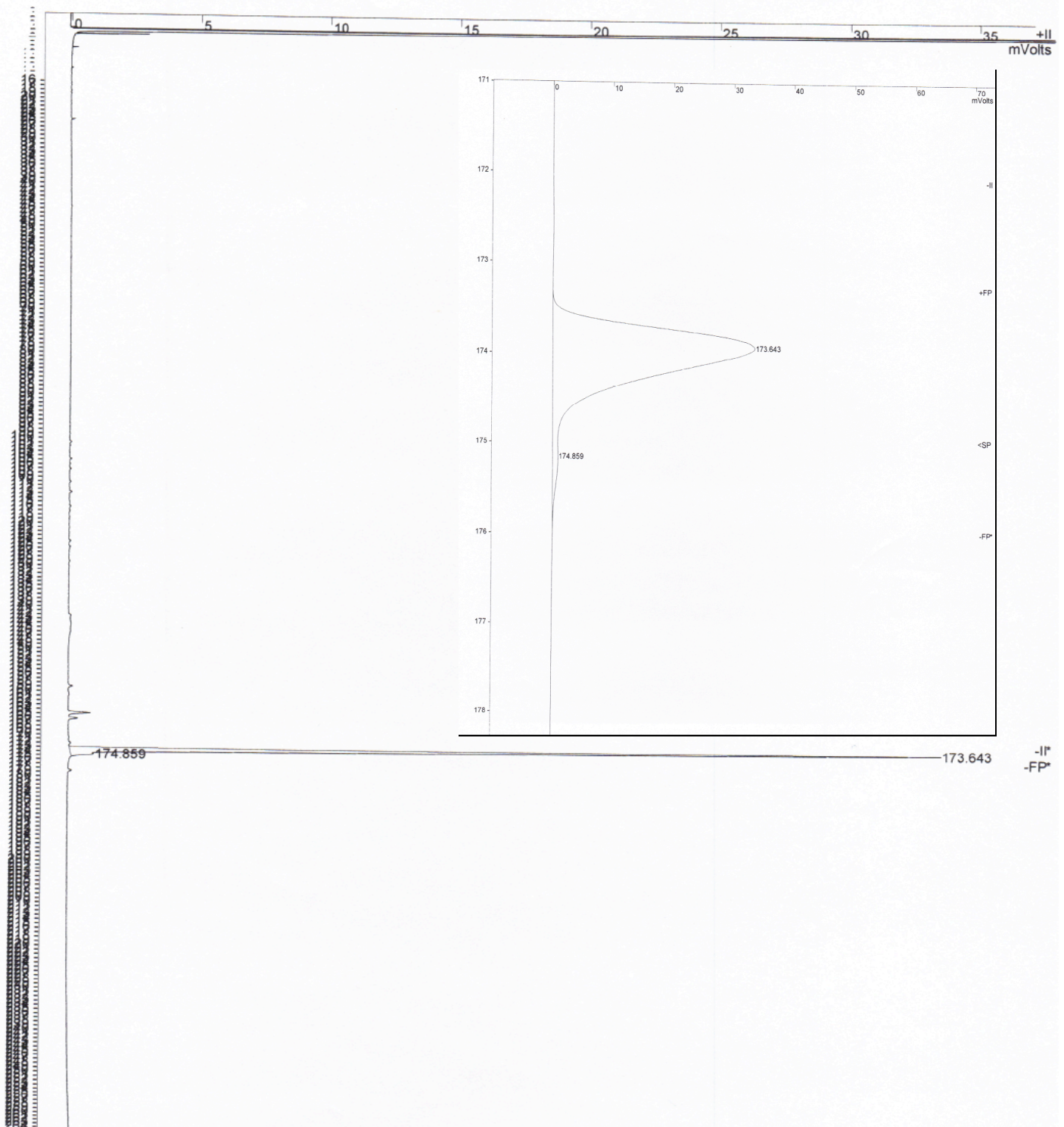


Chiral GC resolution of racemic **4a** (Chiraldex G-TA column, 200 °C injection, 50–180 °C over 135 min)



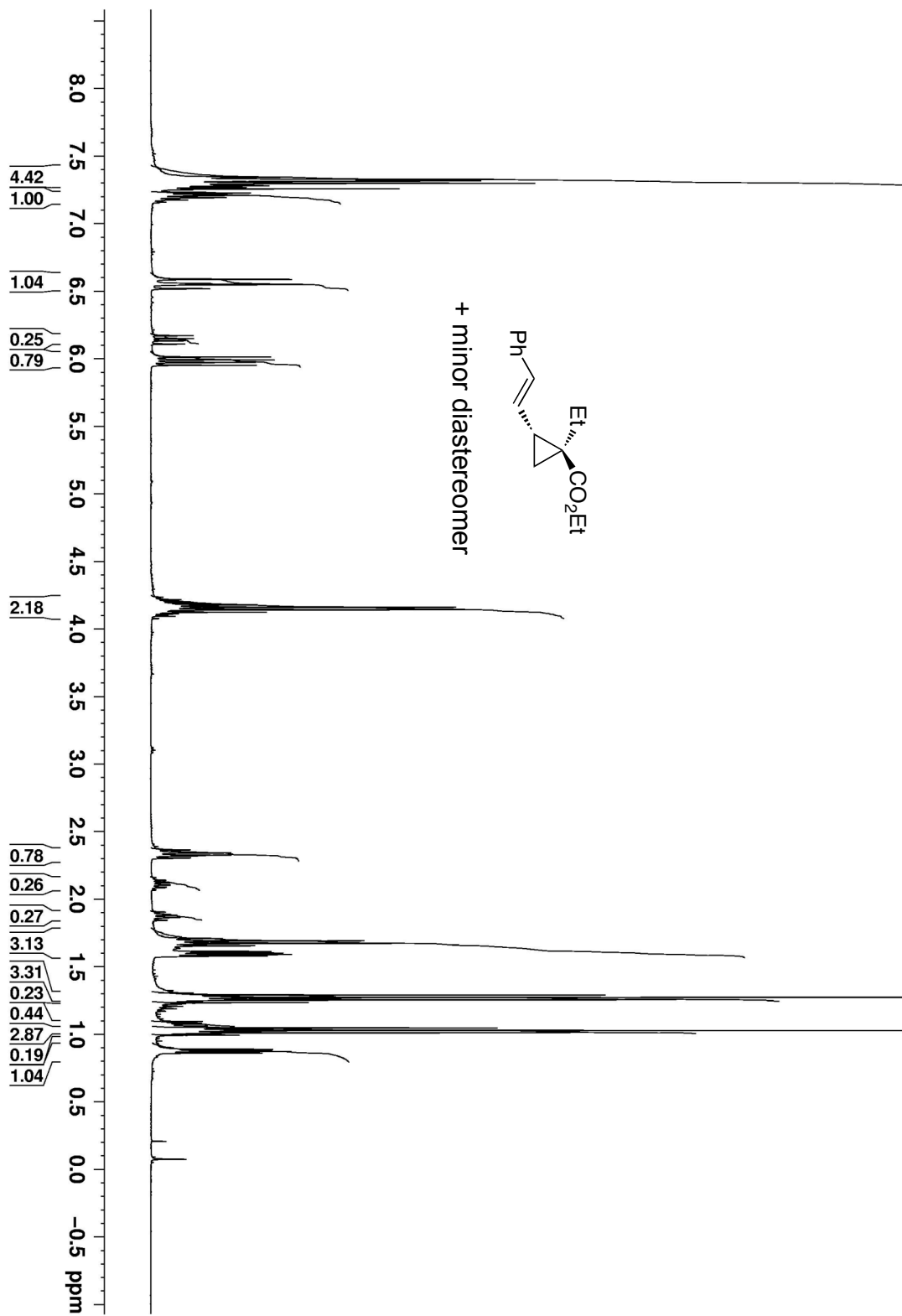
| Peak No. | Peak Name | Result () | Ret. Time (min) | Time Offset (min) | Area (counts) | Sep. Code | Width 1/2 (sec) | Status Codes |
|----------|-----------|------------|-----------------|-------------------|---------------|-----------|-----------------|--------------|
| 1 | | 49.6322 | 173.912 | 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)

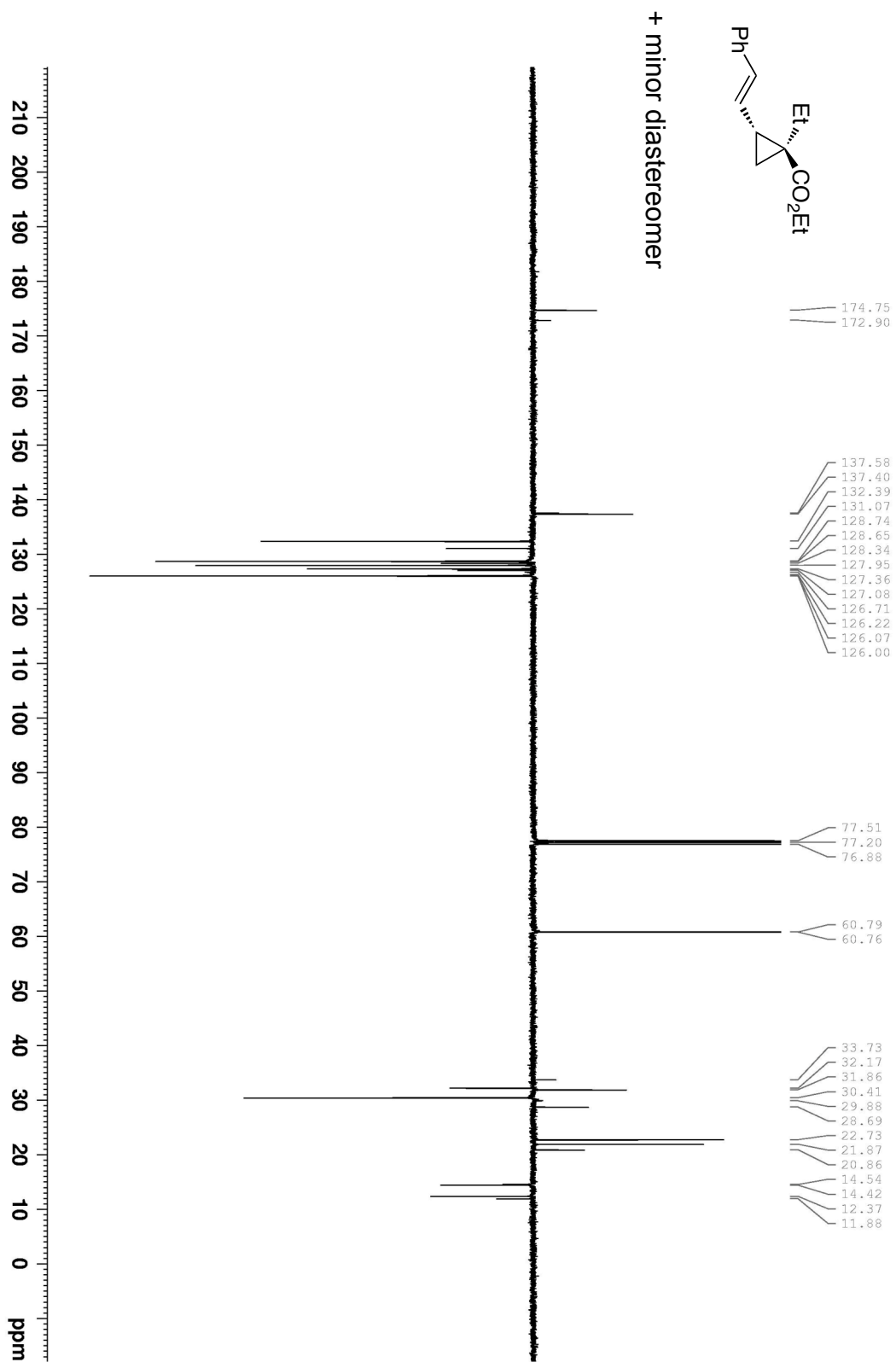


| Peak No. | Peak Name | Result () | Ret. Time (min) | Time Offset (min) | Area (counts) | Sep. Code | Width 1/2 (sec) | Status Codes |
|----------|-----------|------------|-----------------|-------------------|---------------|-----------|-----------------|--------------|
| 1 | | 97.5296 | 173.643 |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

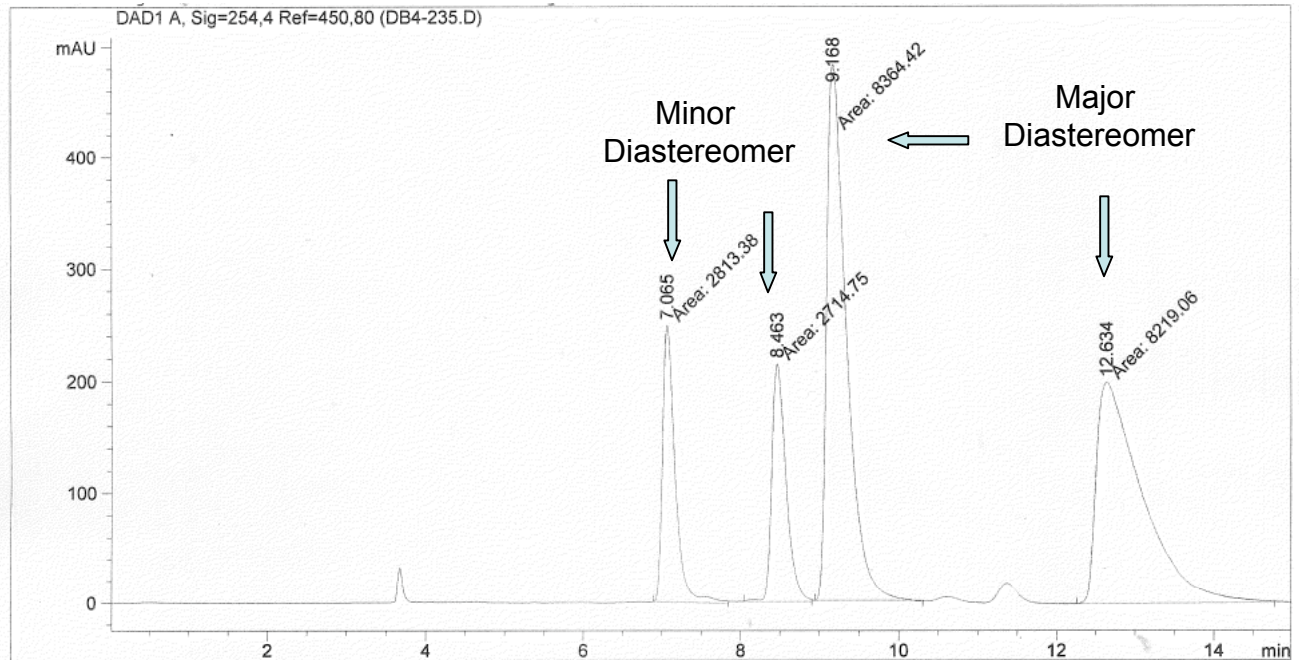
^1H NMR spectrum of **5** (400 MHz, CDCl_3)



^{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)



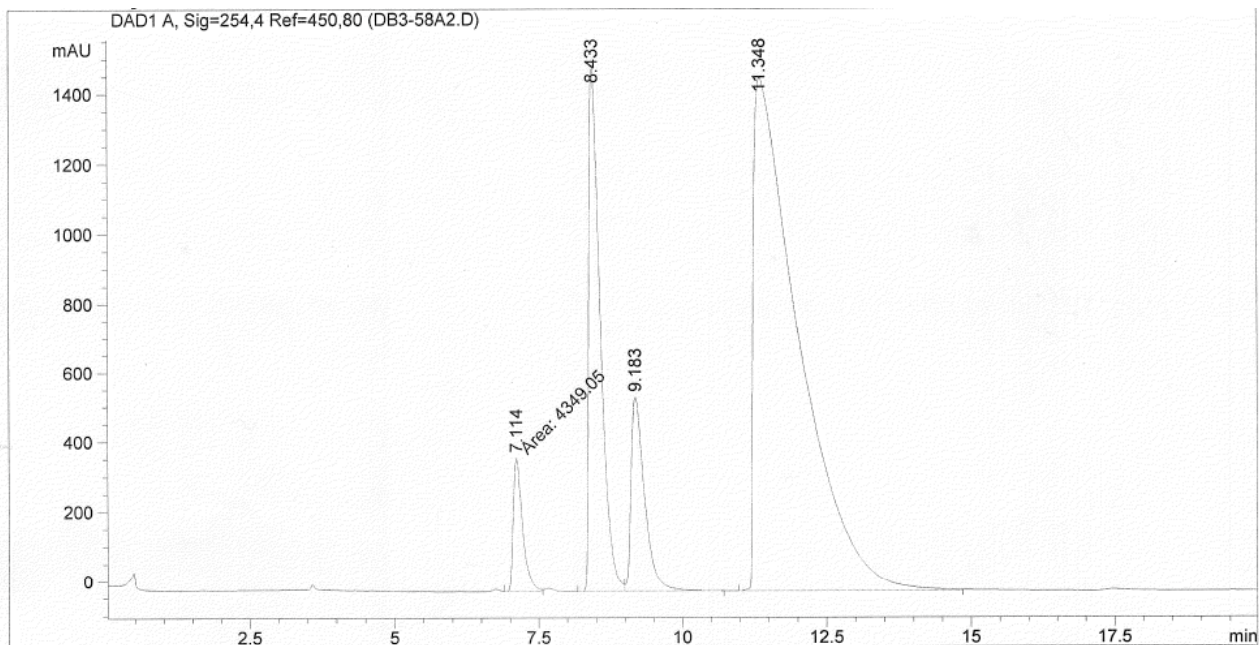
=====
 Area Percent Report
 =====

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)



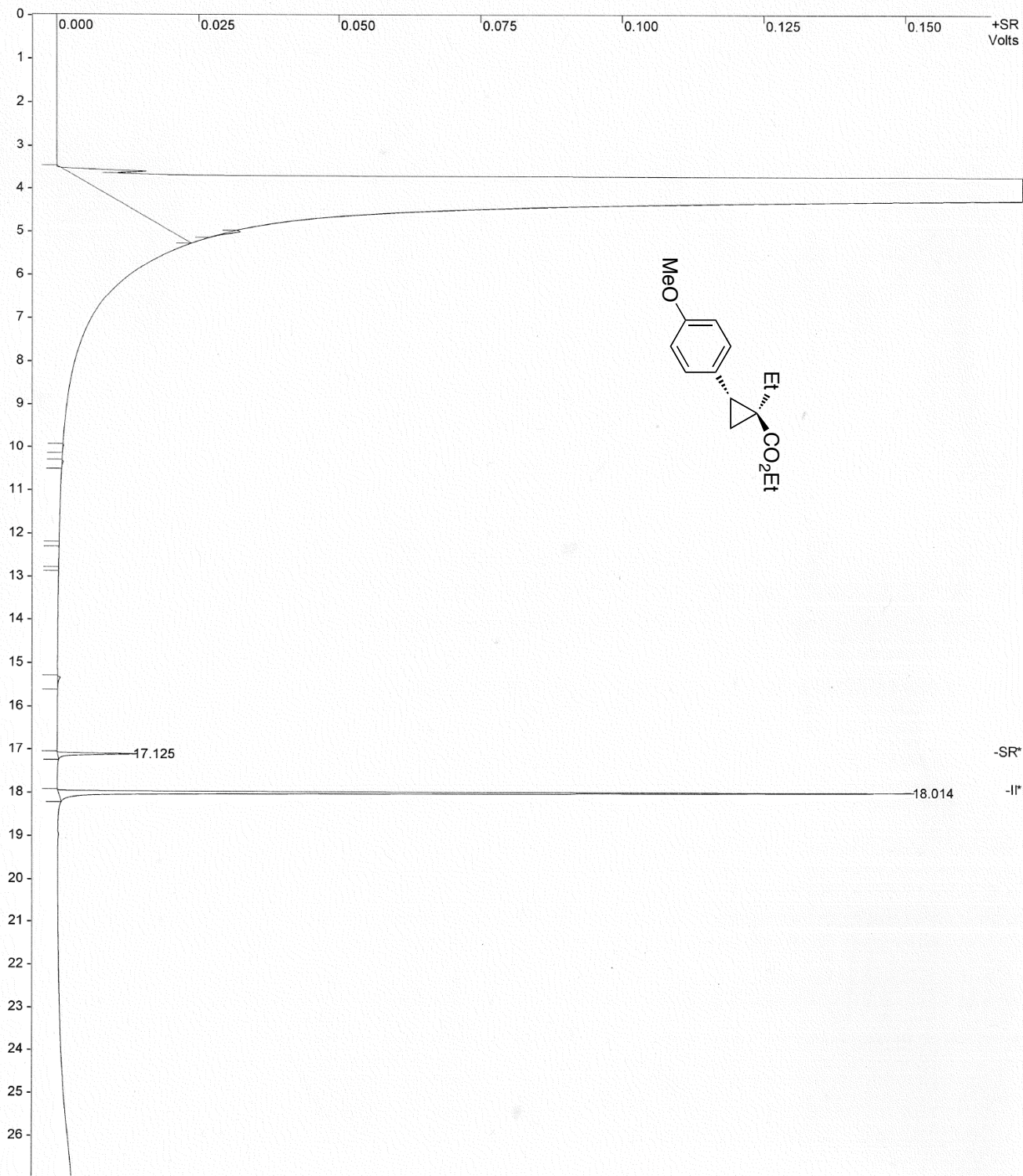
=====
Area Percent Report
=====

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 |

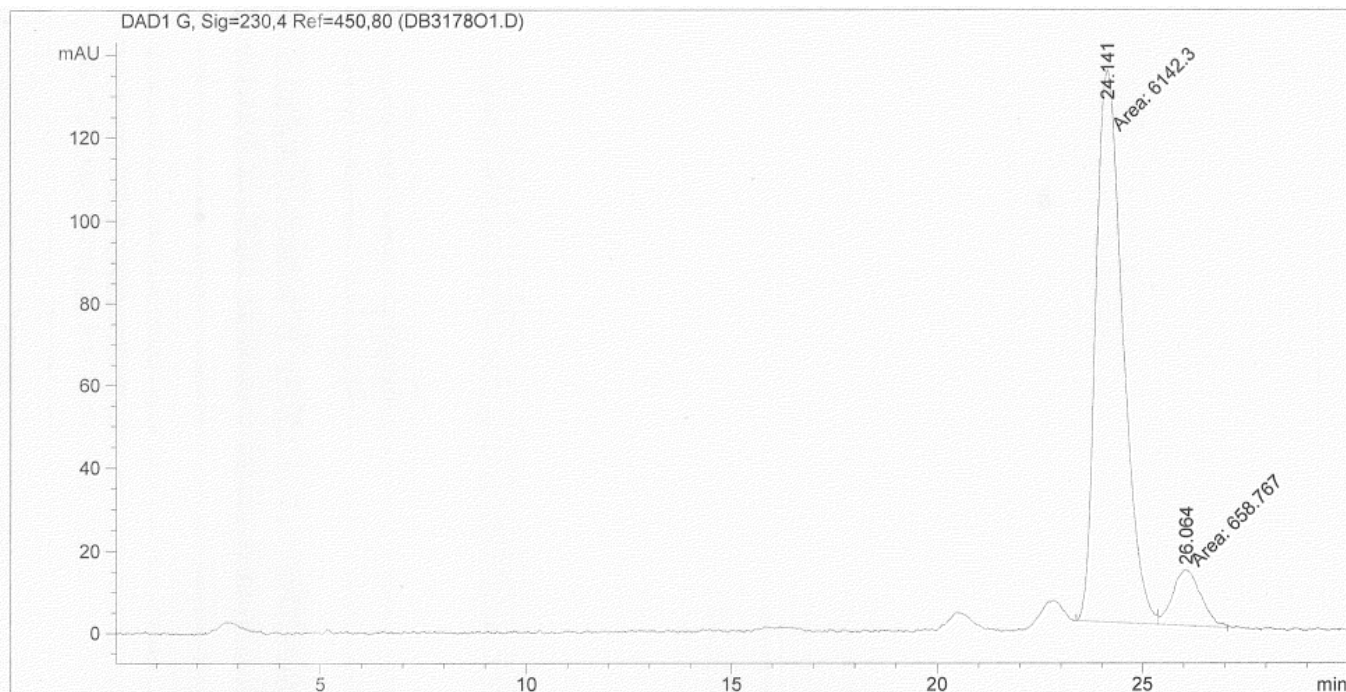
GC trace of **6** (Hewlett Packard HP1 column, 200 °C injection, 50–300 °C over 27 min)



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 $\text{Rh}_2(\text{S-PTTL})_3(\text{TPA})$



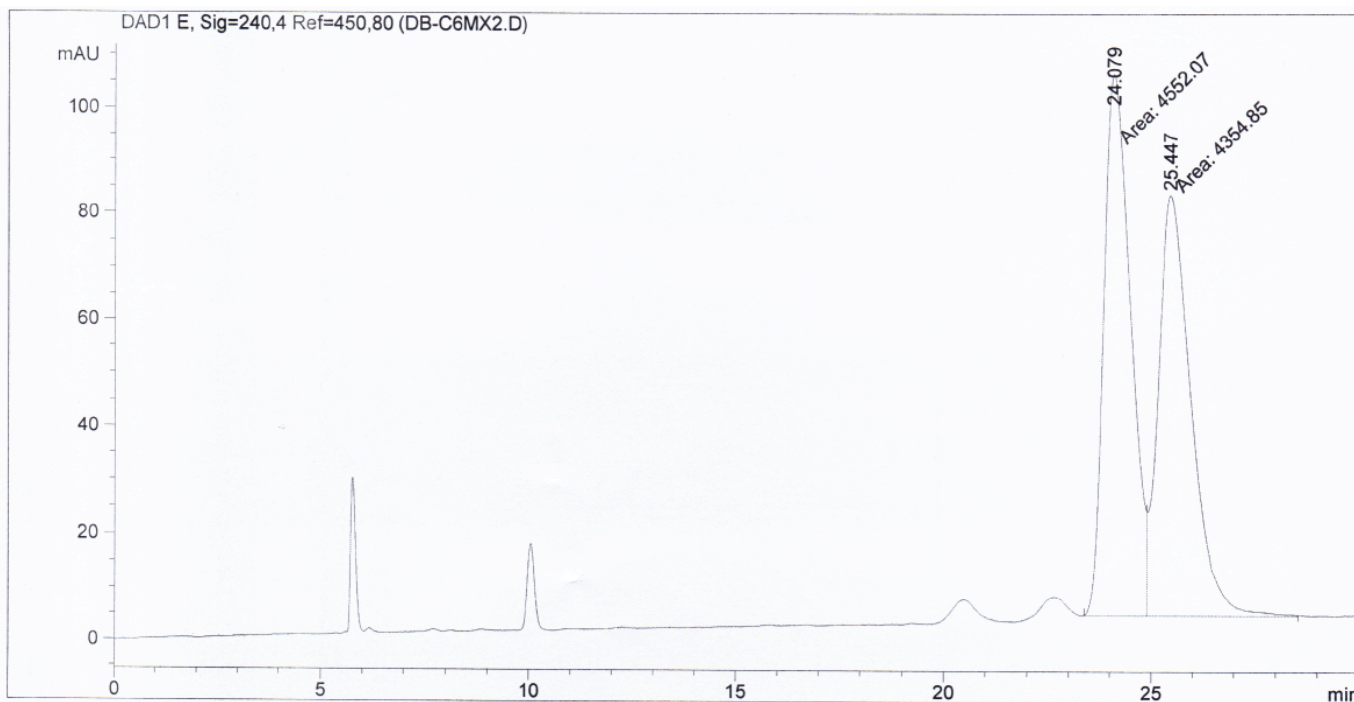
=====
Area Percent Report
=====

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)



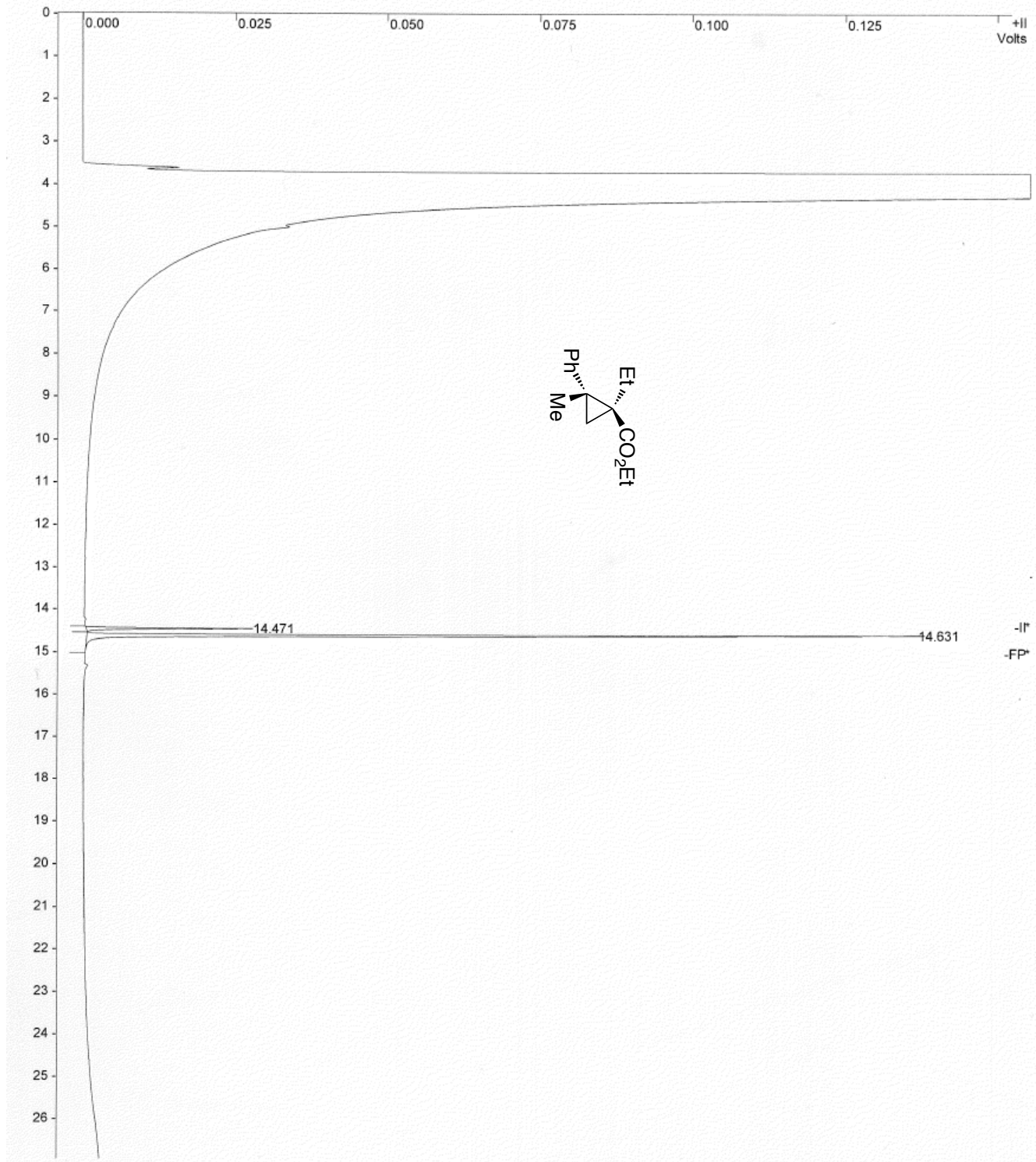
=====
 Area Percent Report
 =====

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

Signal 1: DAD1 E, Sig=240,4 Ref=450,80

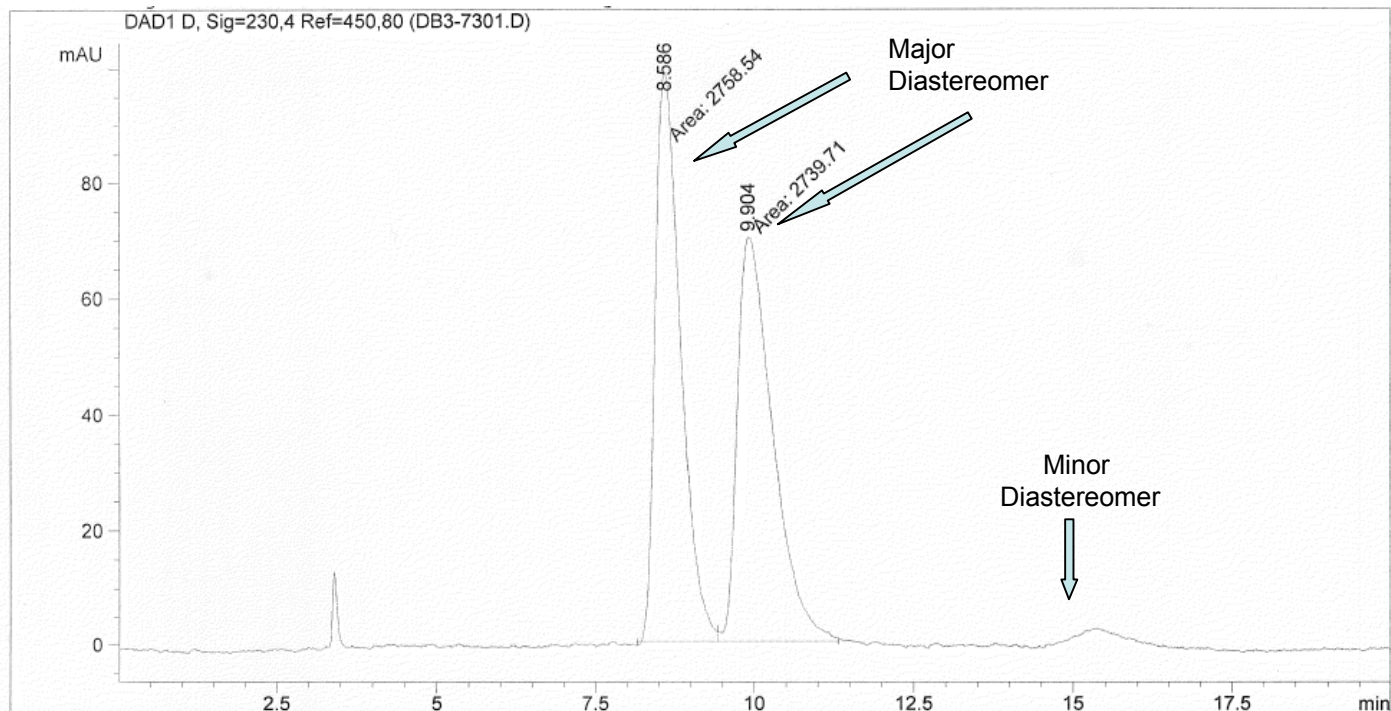
| Peak # | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area % |
|--------|---------------|------|-------------|--------------|--------------|---------|
| 1 | 24.079 | MF | 0.7484 | 4552.06738 | 101.37346 | 51.1071 |
| 2 | 25.447 | FM | 0.9224 | 4354.84619 | 78.68336 | 48.8929 |

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)



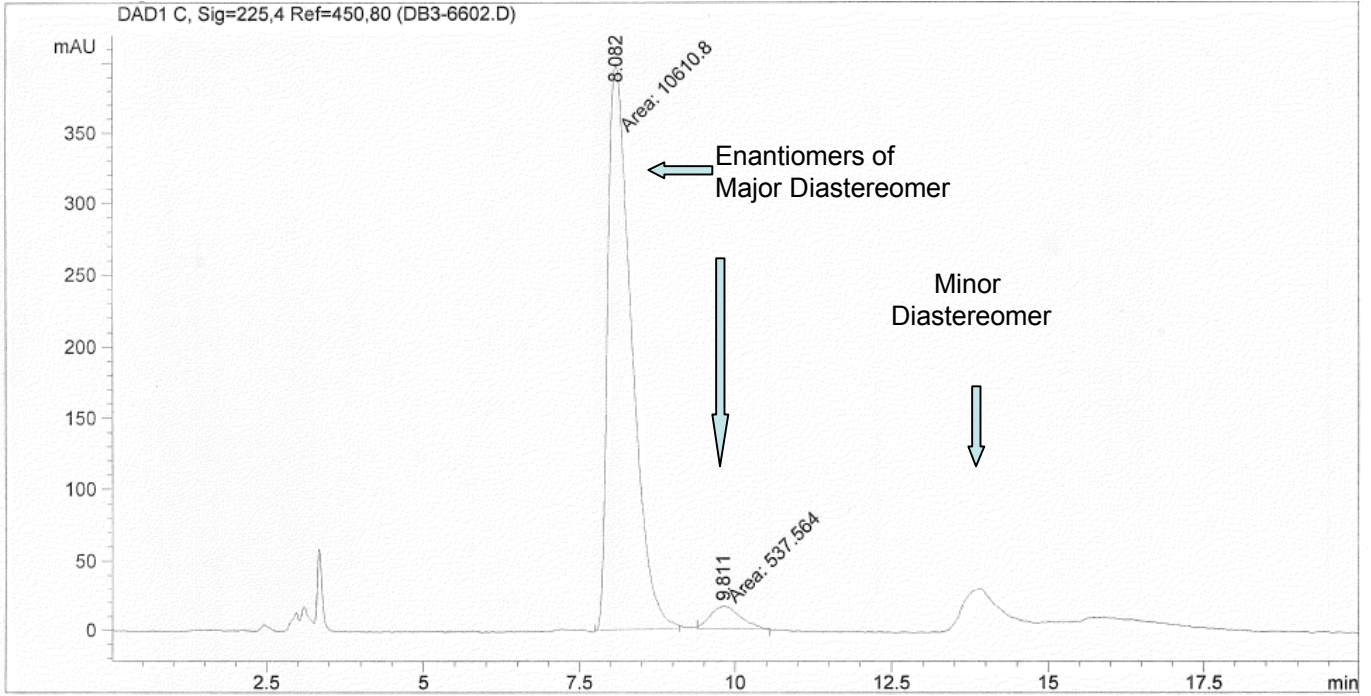
=====
 Area Percent Report
 =====

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)



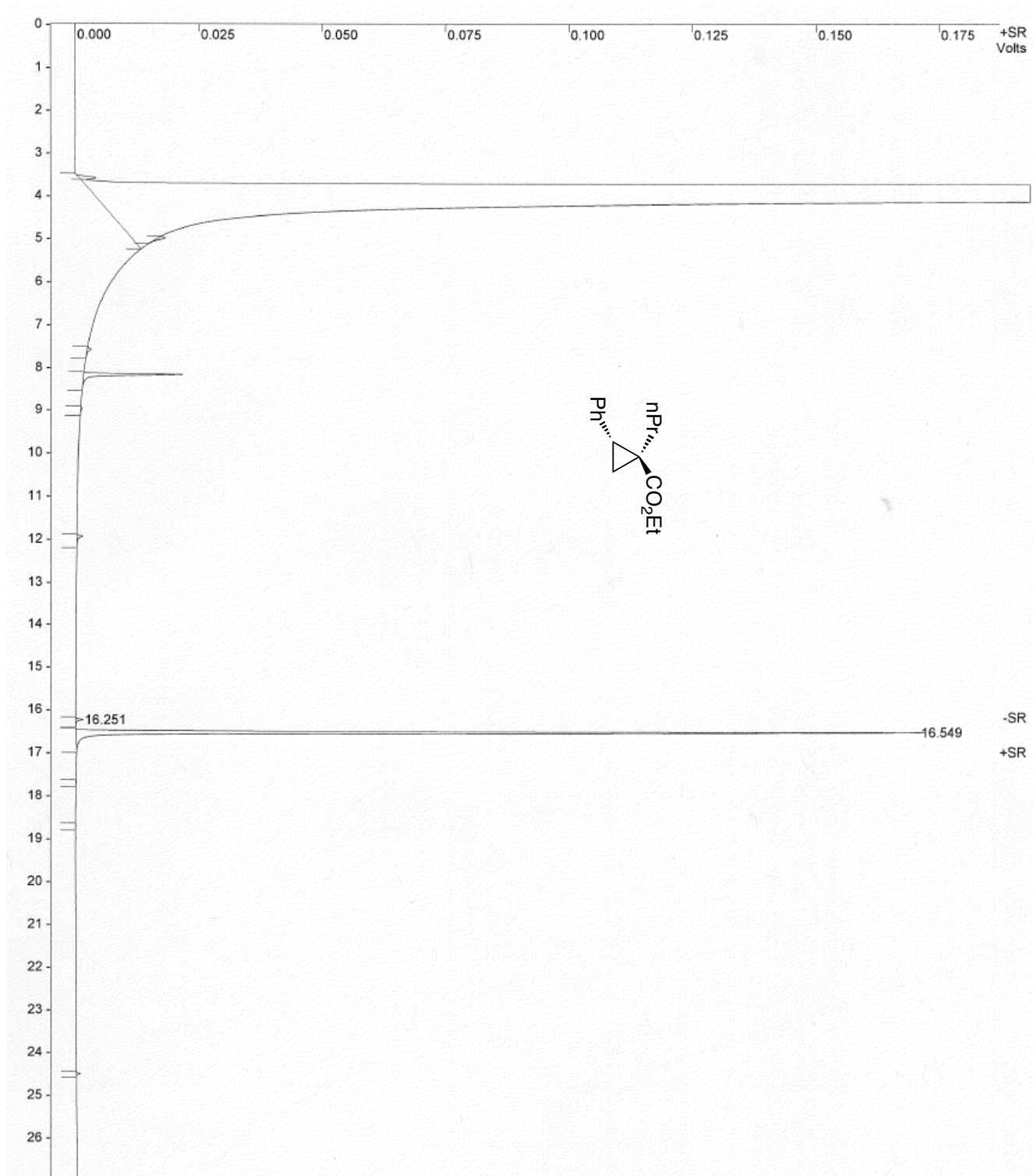
=====
 Area Percent Report
 =====

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

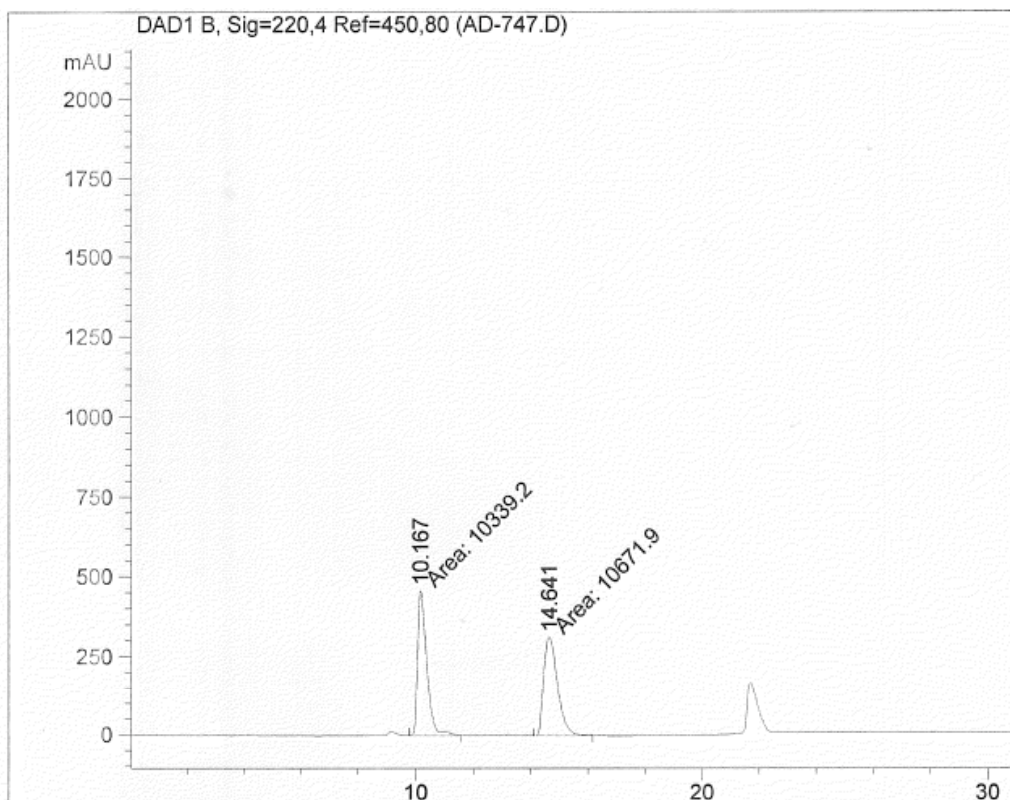
| Peak # | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area % |
|--------|---------------|------|-------------|--------------|--------------|---------|
| 1 | 8.082 | MM | 0.4431 | 1.06108e4 | 399.14804 | 95.1781 |
| 2 | 9.811 | MM | 0.5548 | 537.56384 | 16.14973 | 4.8219 |

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



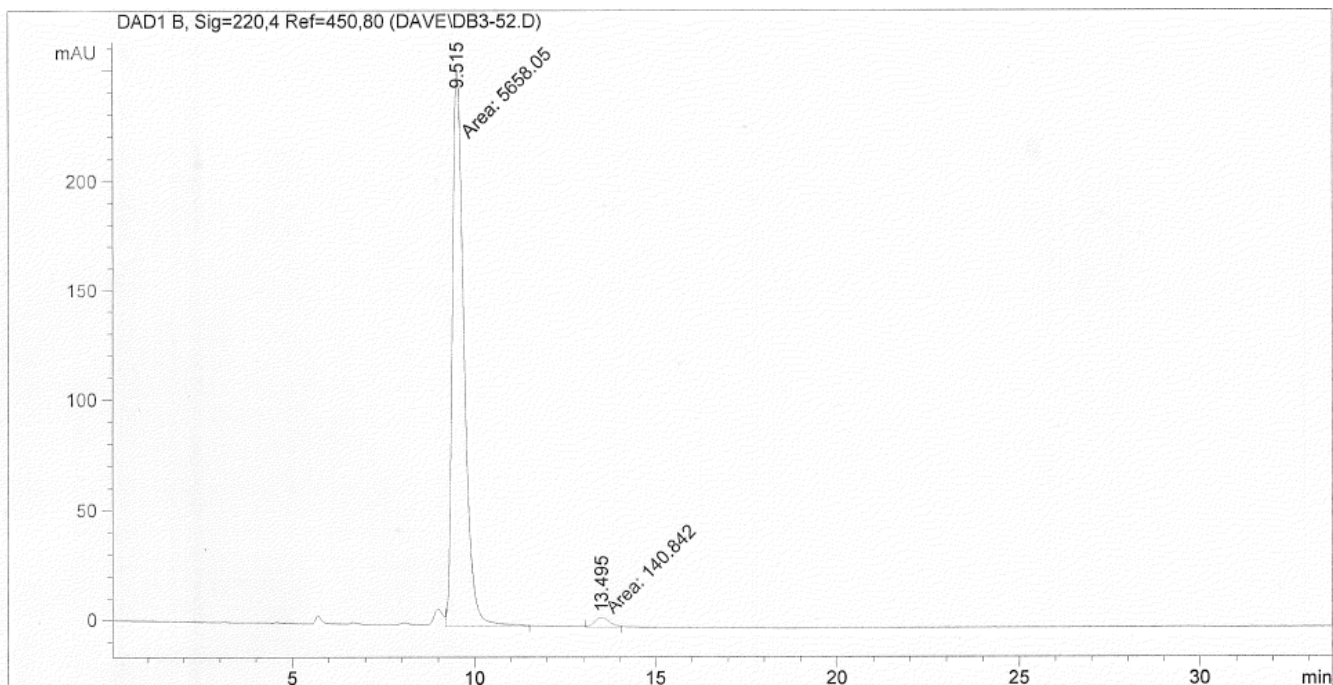
=====
 Area Percent Report
 =====

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)



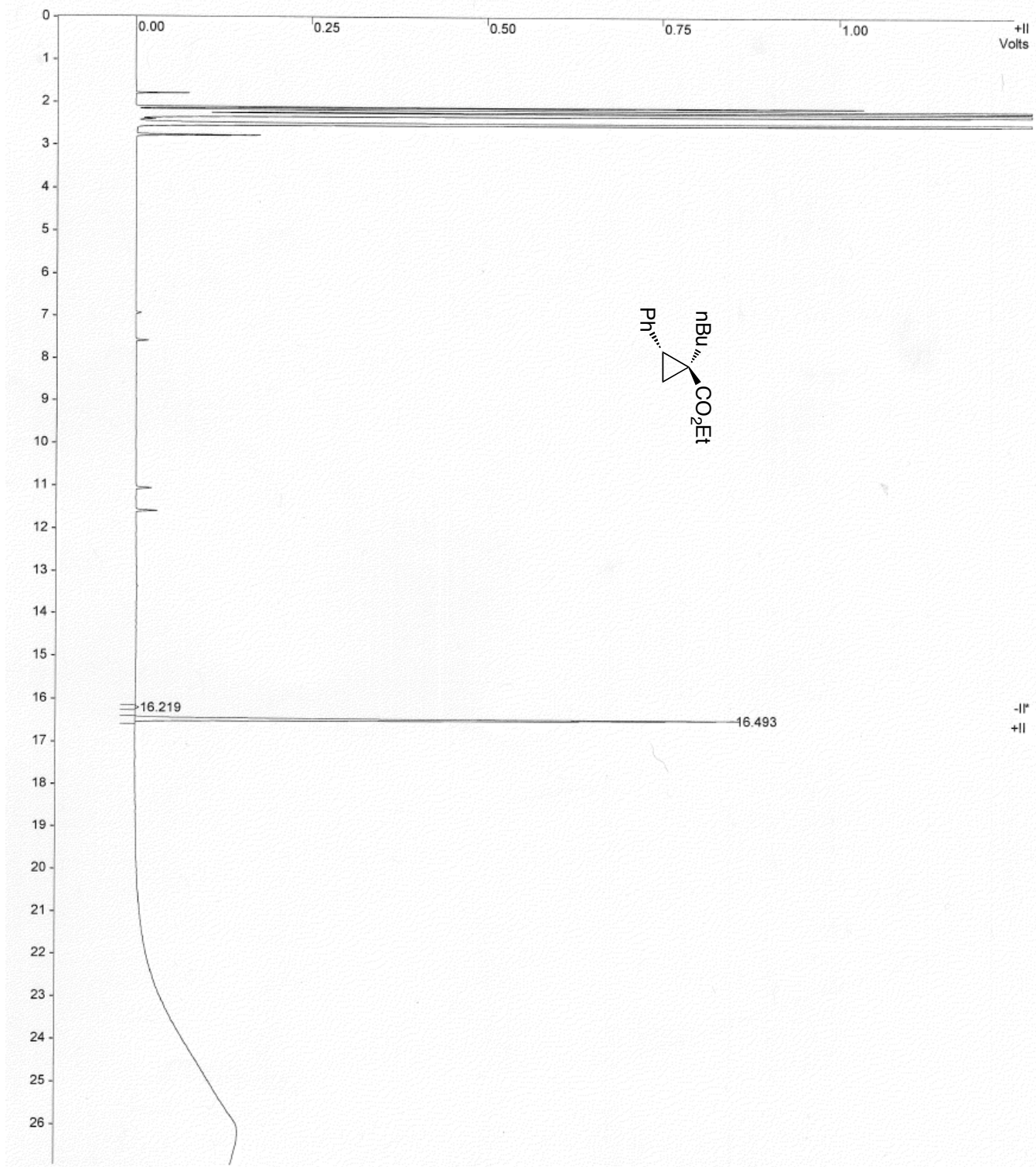
=====
 Area Percent Report
 =====

Sorted By : Signal
 Multiplier : 1.0000
 Dilution : 1.0000
 Sample Amount : 1.00000 [ng/ul] (not used in calc.)
 Use Multiplier & Dilution Factor with ISTDs

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

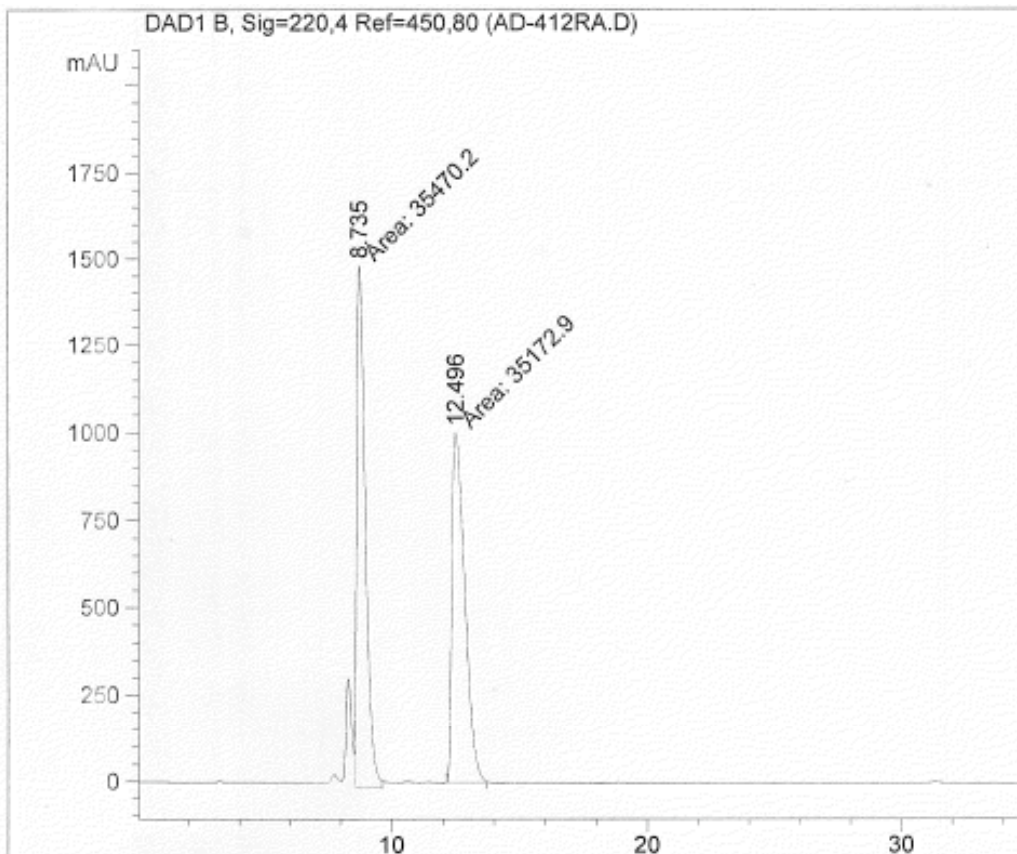
| Peak # | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area % |
|--------|---------------|------|-------------|--------------|--------------|---------|
| 1 | 9.515 | MM | 0.3726 | 5658.05420 | 253.08644 | 97.5712 |
| 2 | 13.495 | MM | 0.5026 | 140.84167 | 4.67056 | 2.4288 |

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



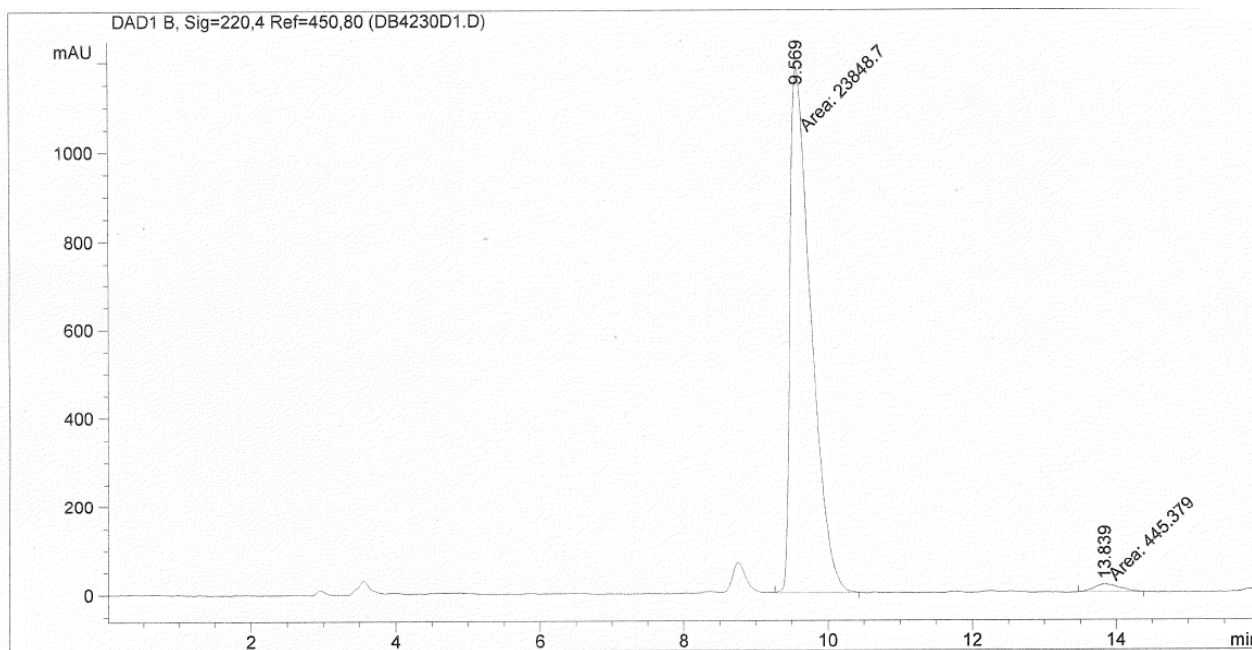
=====
 Area Percent Report
 =====

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 | 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)



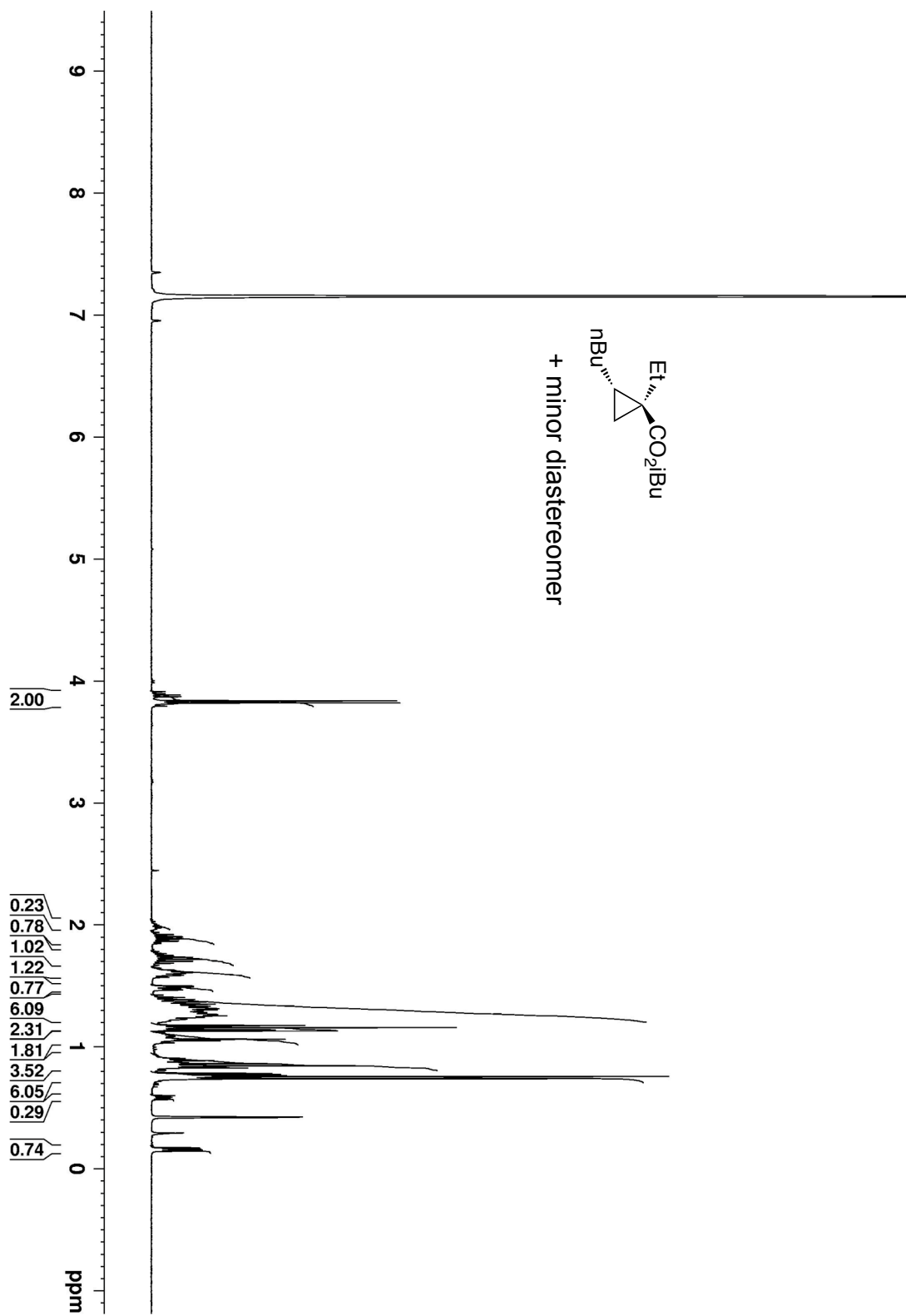
=====
 Area Percent Report
 =====

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

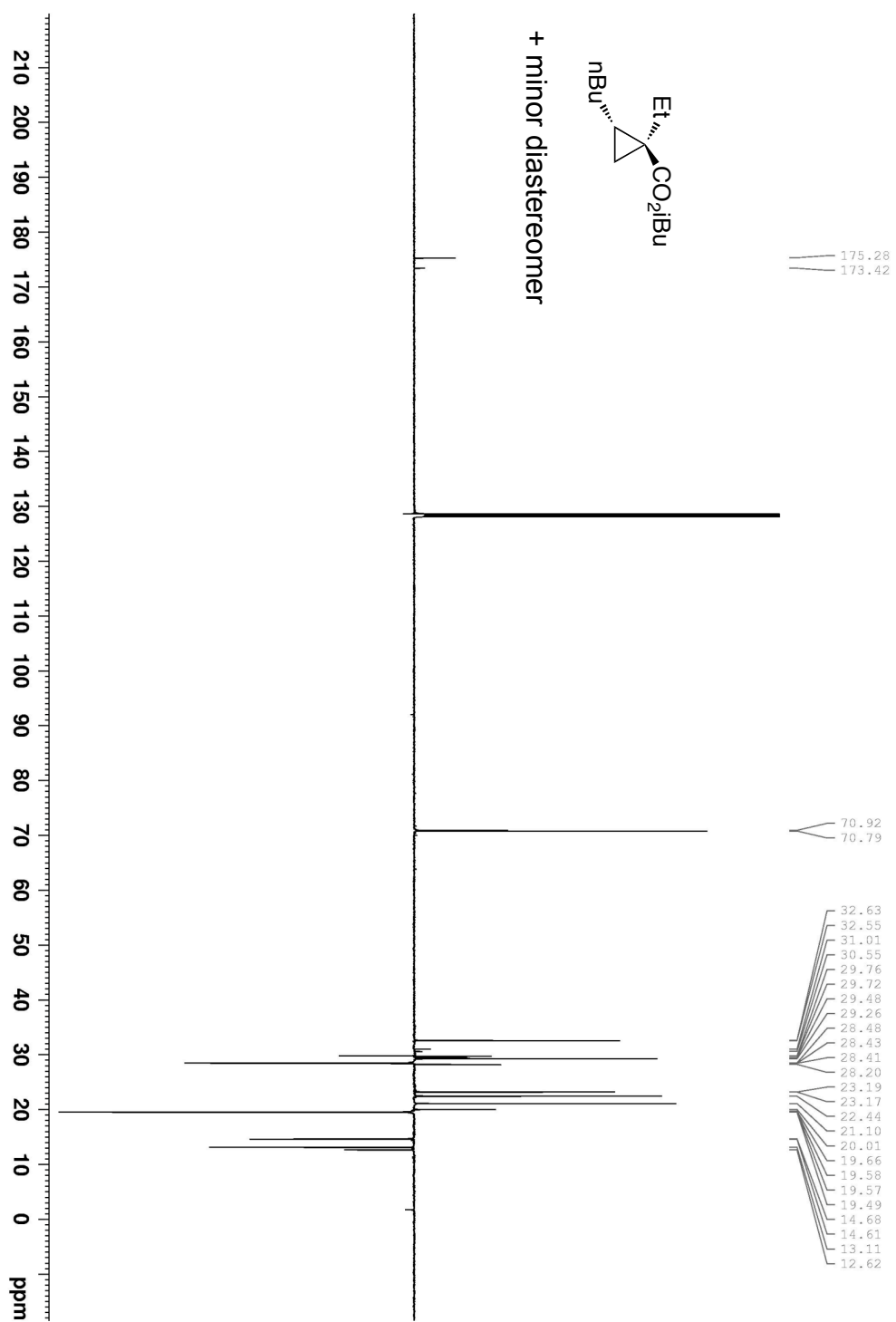
Signal 1: DAD1 B, Sig=220,4 Ref=450,80

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

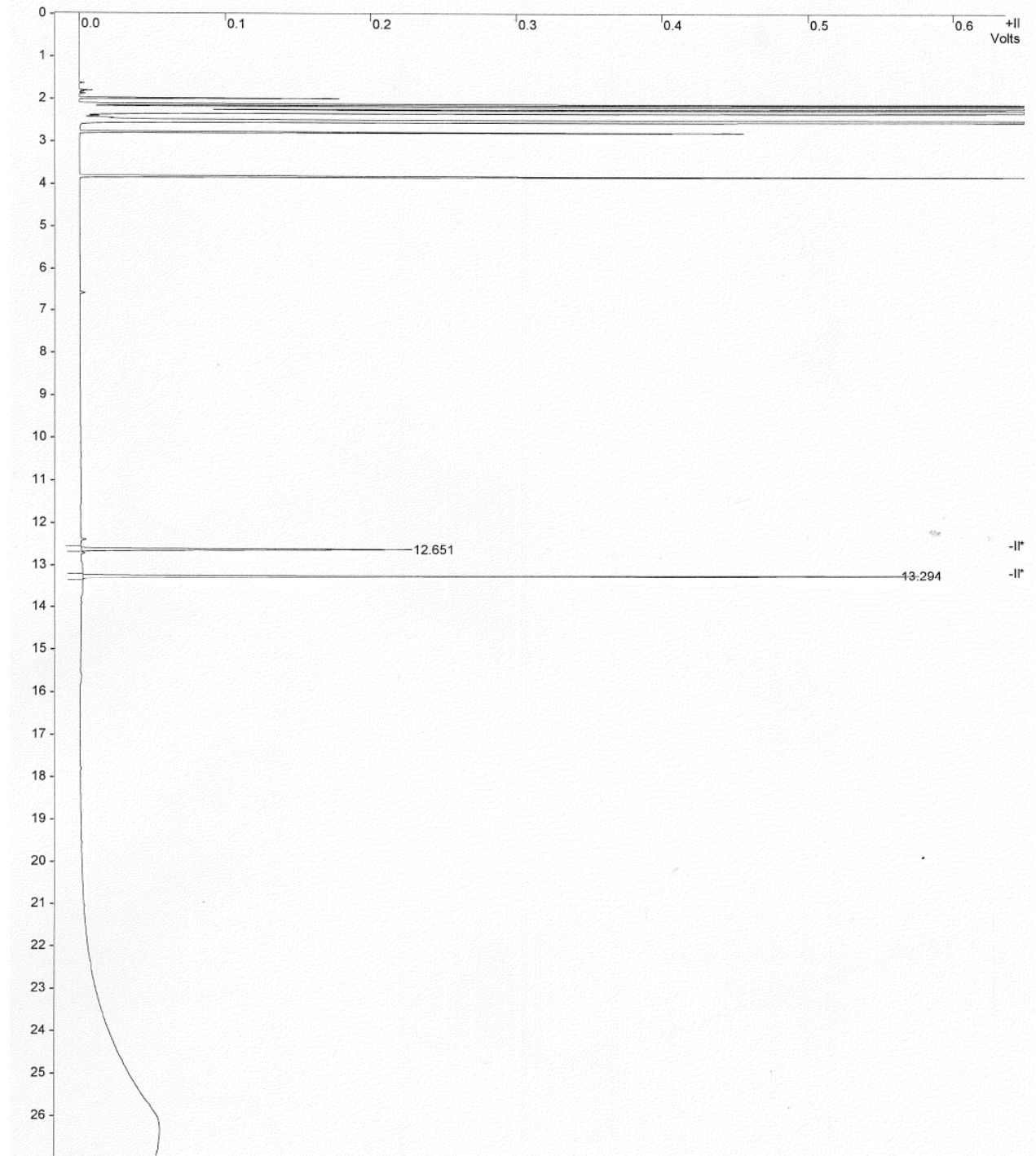
^1H NMR spectrum of **10** (400 MHz, C_6D_6)



^{13}C NMR spectrum of **10** (100 MHz, C_6D_6)

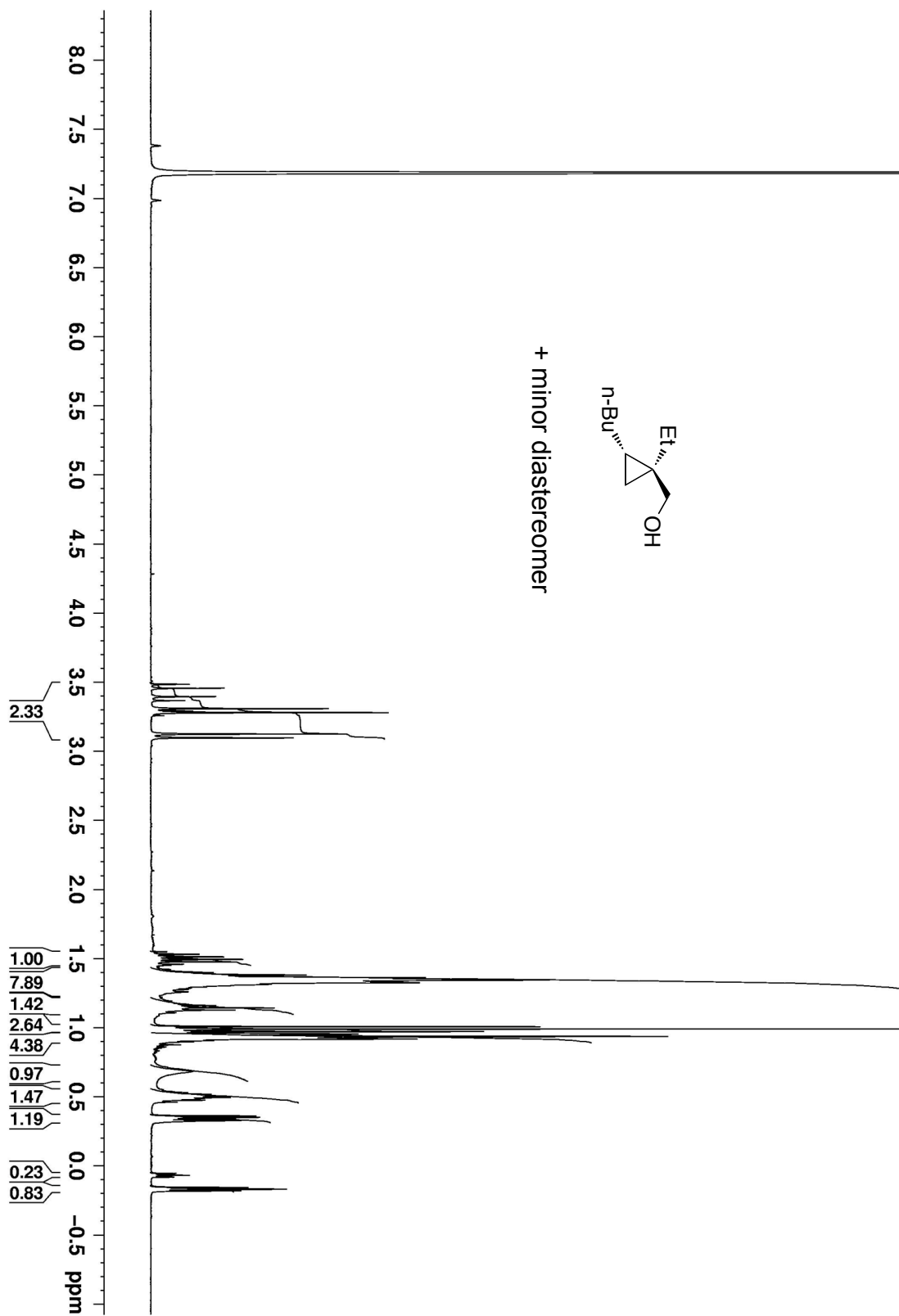


GC trace of **10** (Hewlett Packard HP1 column, 200 °C injection, 50–300 °C over 27 min)

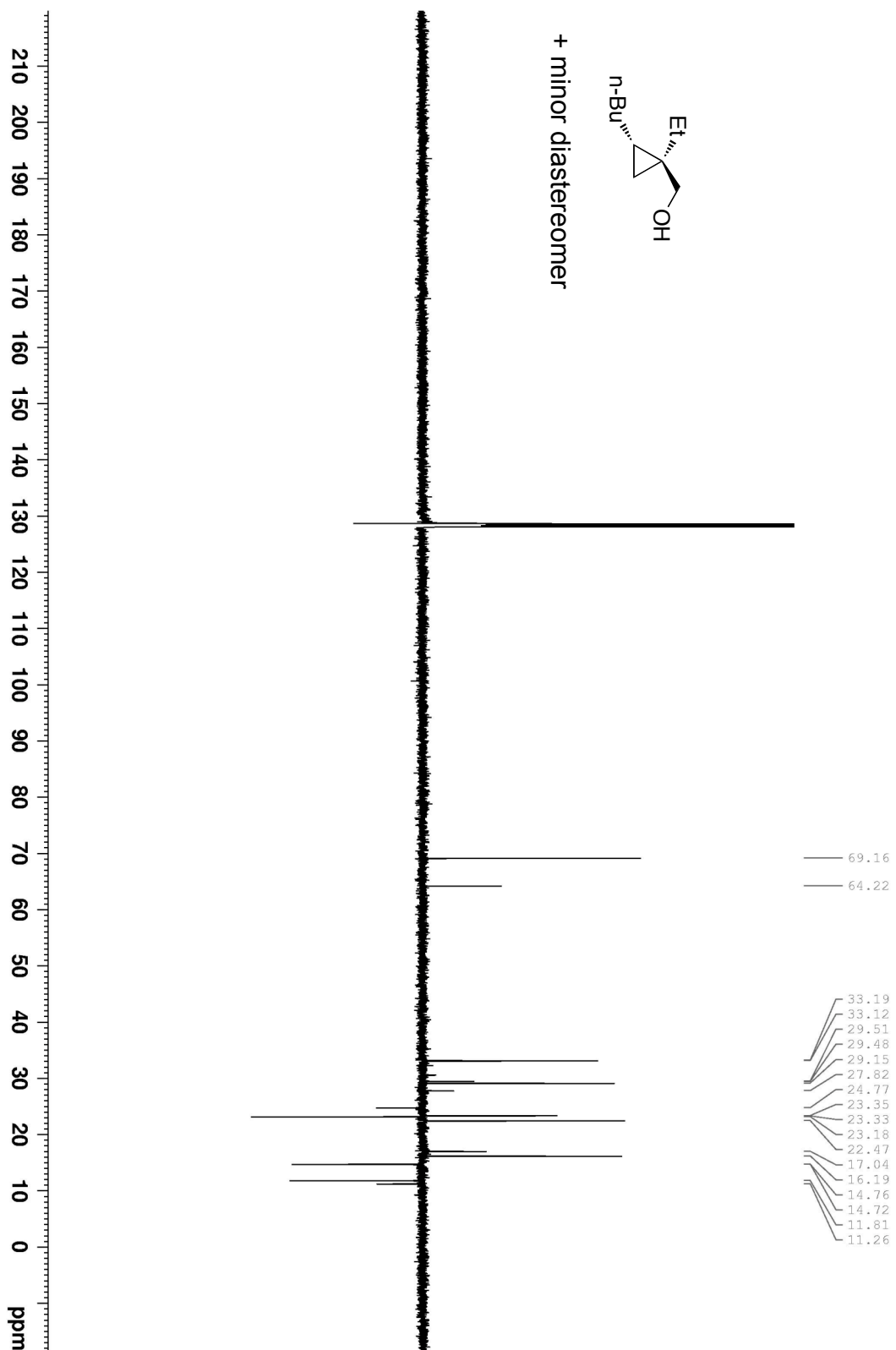


| Peak No. | Peak Name | Result () | Ret. Time (min) | Time Offset (min) | Area (counts) | Sep. Code | Width 1/2 (sec) | Status Codes |
|----------|-----------|------------|-----------------|-------------------|---------------|-----------|-----------------|--------------|
| 1 | | 28.1073 | 12.651 | 0.000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

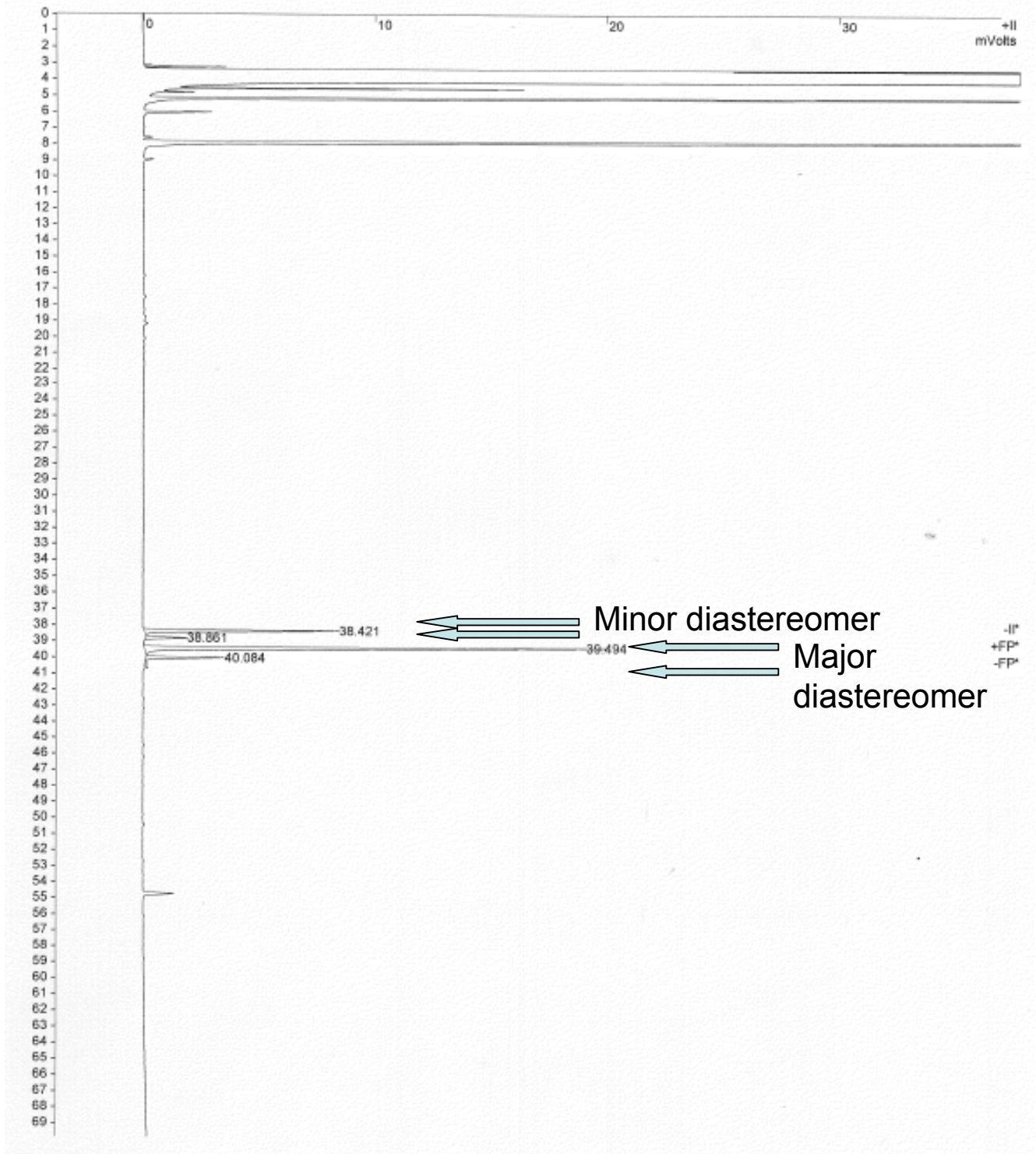
^1H NMR spectrum of **10a** (400 MHz, C_6D_6)



^{13}C NMR spectrum of **10a** (100 MHz, C_6D_6)

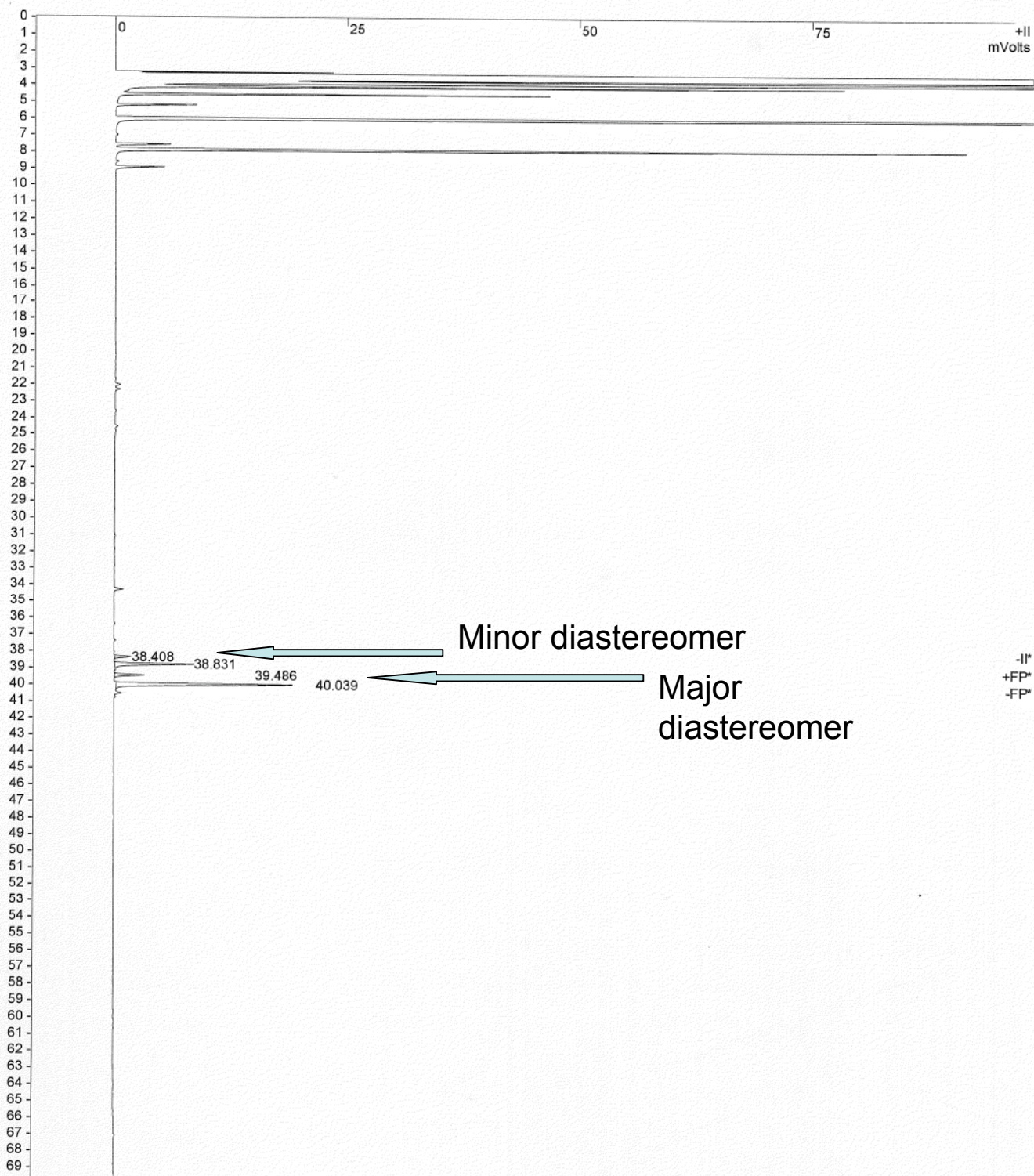


GC trace of Enantioselective **10a**



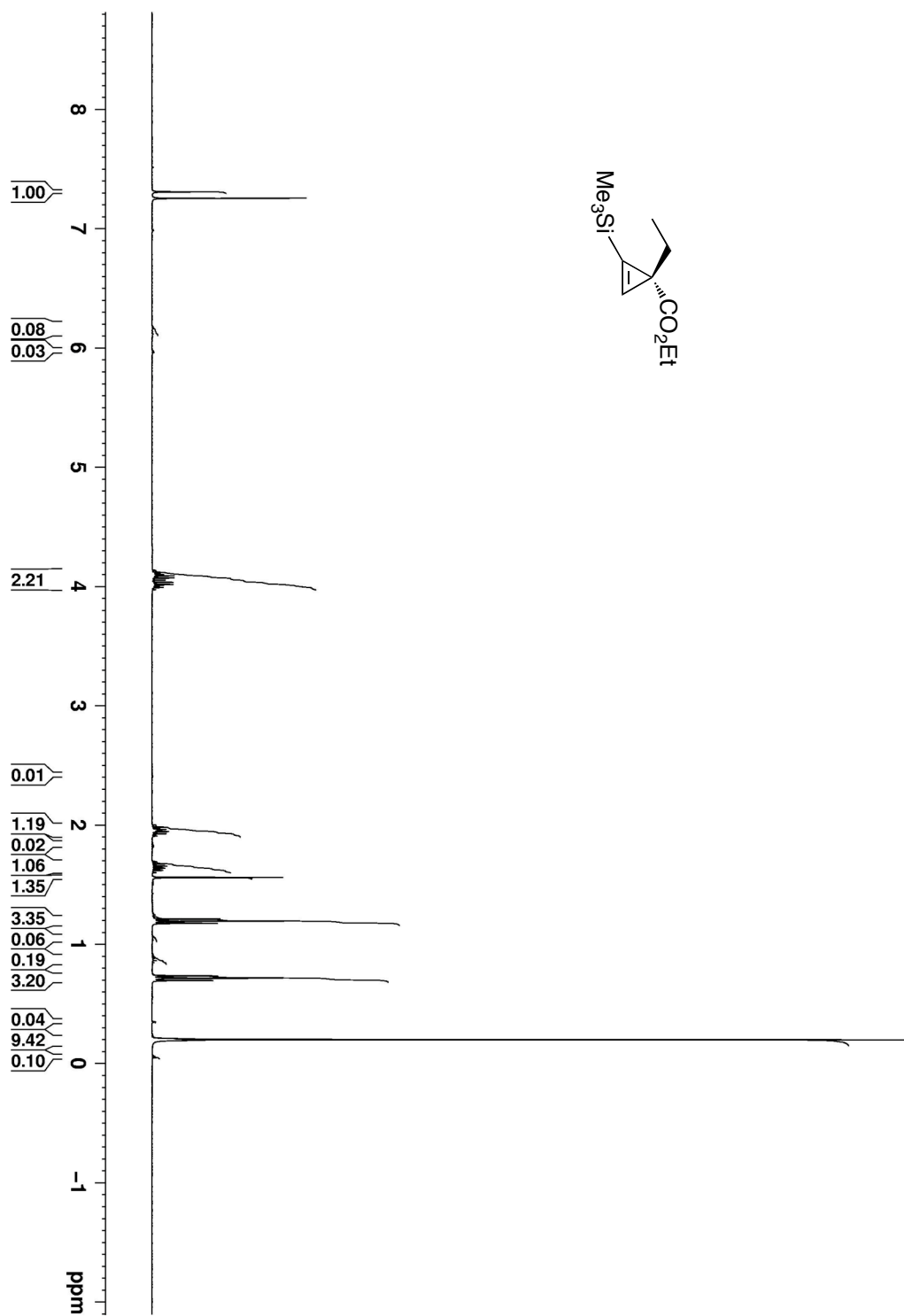
| Peak No. | Peak Name | Result () | Ret. Time (min) | Time Offset (min) | Area (counts) | Sep. Code | Width 1/2 (sec) | Status Codes |
|----------|-----------|------------|-----------------|-------------------|---------------|-----------|-----------------|--------------|
| 1 | | 24.4056 | 38.421 | 0.000 | 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 $\text{Rh}_2(\text{R-PTTL})_3(\text{TPA})$ catalyzed instead of with $\text{Rh}_2(\text{S-PTTL})_3(\text{TPA})$ to show the opposite trend in enantioselectivity.

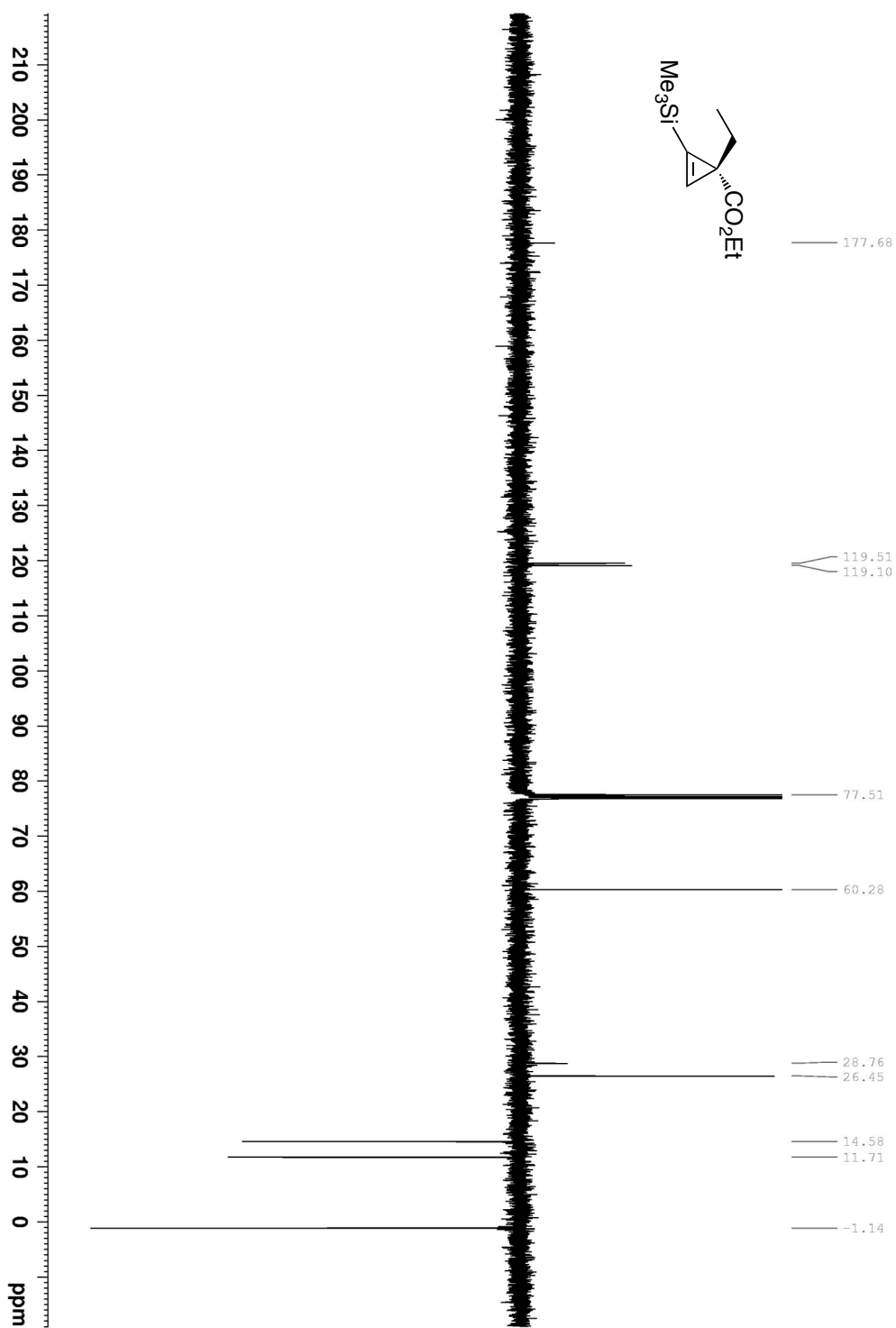


| Peak No. | Peak Name | Result () | Ret. Time (min) | Time Offset (min) | Area (counts) | Sep. Code | Width 1/2 (sec) | Status Codes |
|----------|-----------|------------|-----------------|-------------------|---------------|-----------|-----------------|--------------|
| 1 | | 4.6050 | 38.408 | 0.000 | 11153 | BB | 6.2 | |
| 2 | | 26.4122 | 38.831 | 0.000 | 63972 | BB | 6.7 | |
| 3 | | 9.4794 | 39.486 | 0.000 | 22960 | BB | 6.6 | |
| 4 | | 59.5035 | 40.039 | 0.000 | 144121 | BB | 7.0 | |
| Totals: | | 100.0001 | | 0.000 | 242206 | | | |

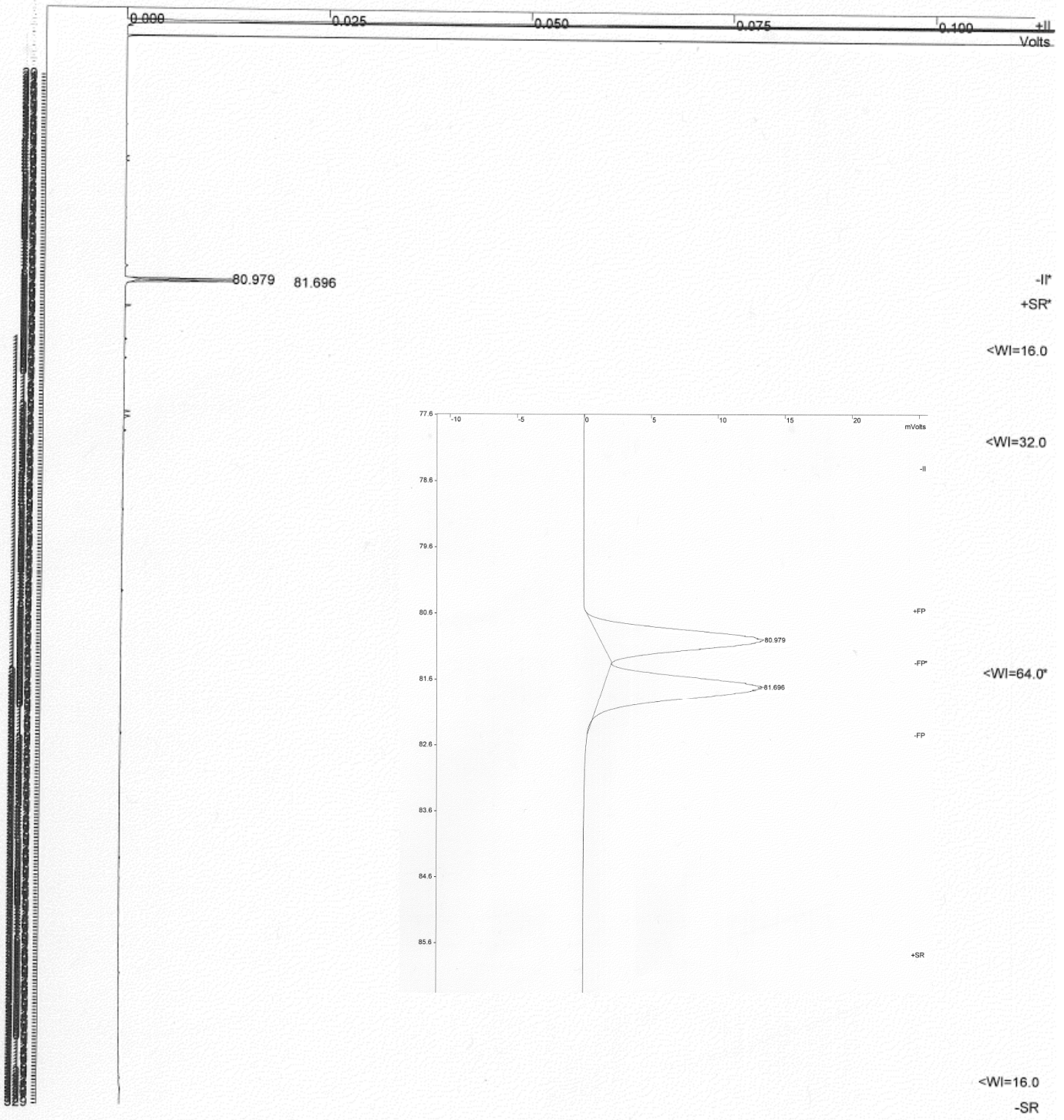
^1H NMR spectrum of **11** (400 MHz, CDCl_3)



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

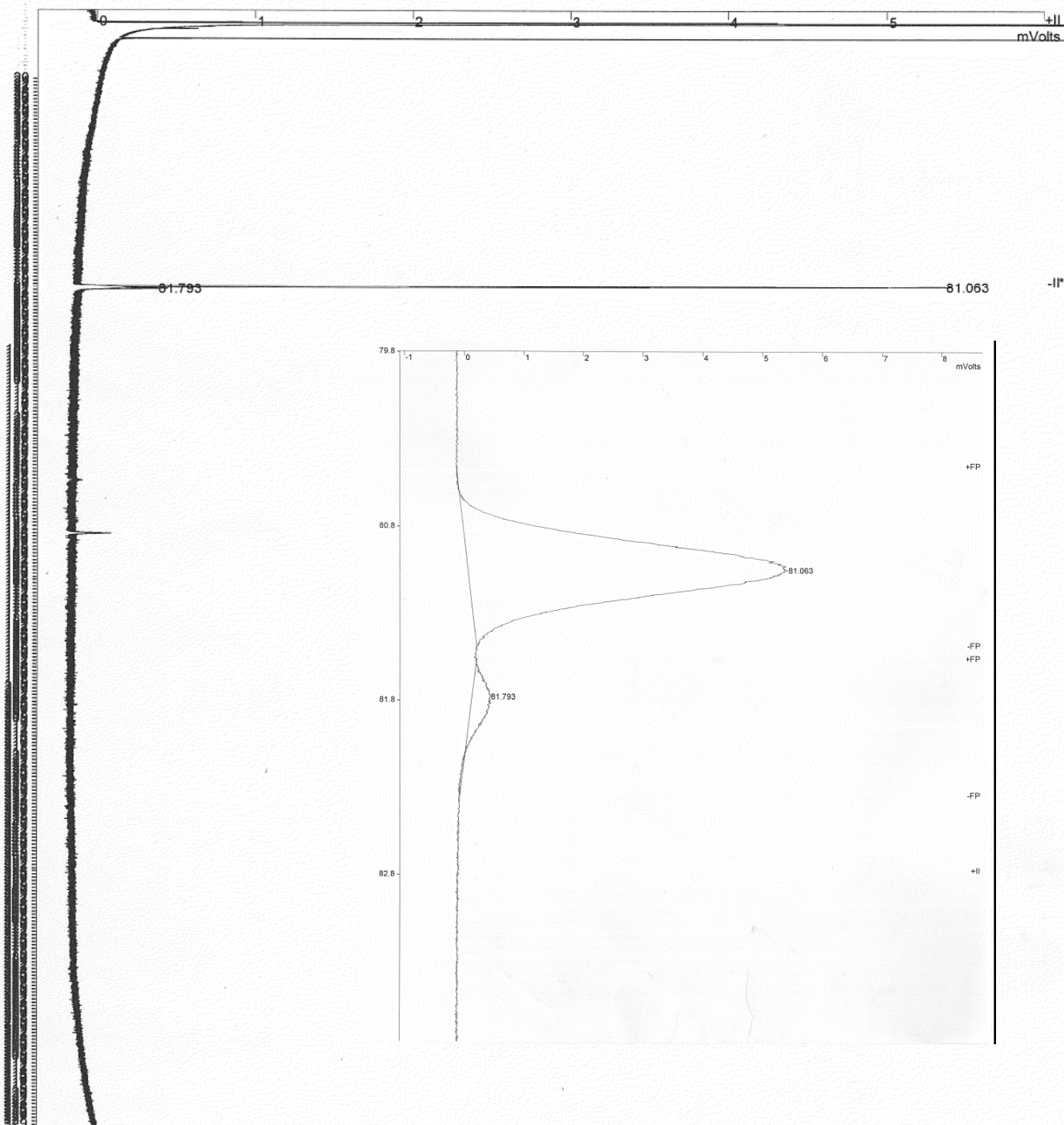


GC trace of racemic **11** (Chiralpak G-TA column, 200 °C injection, 50–180°C at 0.4°C per min., hold 20 min.)



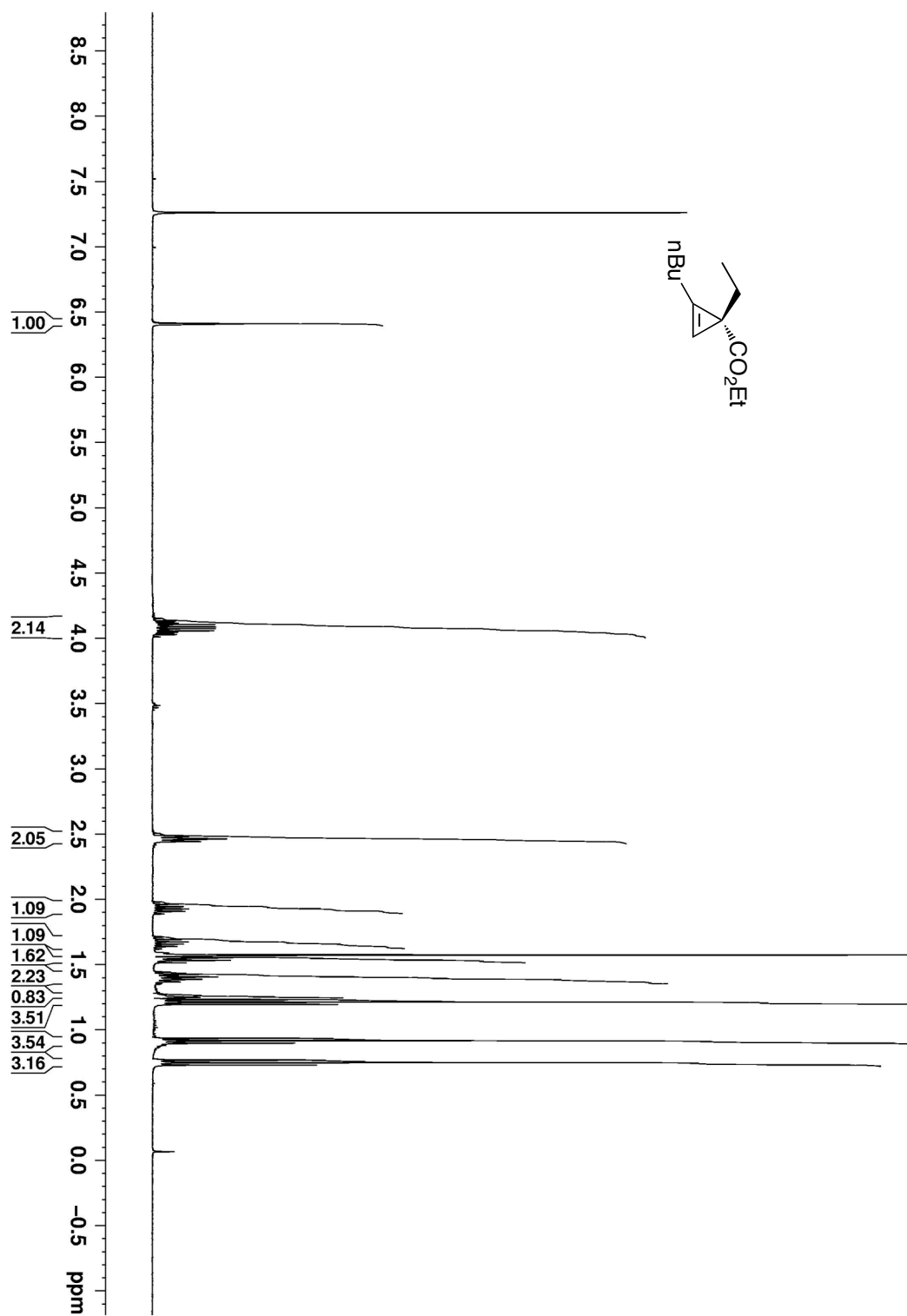
| Peak No. | Peak Name | Result () | Ret. Time (min) | Time Offset (min) | Area (counts) | Sep. Code | Width 1/2 (sec) | Status Codes |
|----------|-----------|------------|-----------------|-------------------|---------------|-----------|-----------------|--------------|
| 1 | | 48.8452 | 80.979 | 0.000 | 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.)

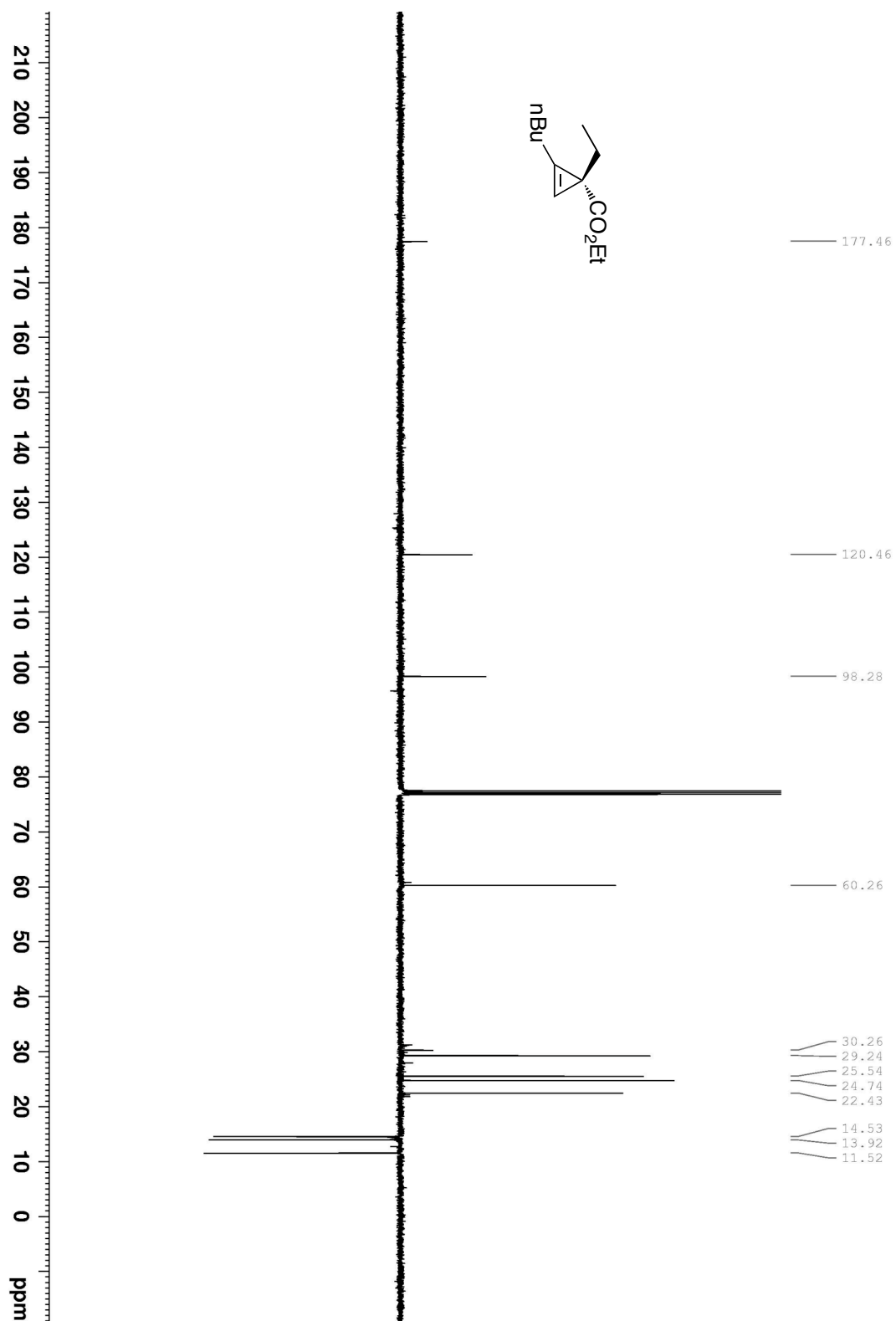


| Peak No. | Peak Name | Result () | Ret. Time (min) | Time Offset (min) | Area (counts) | Sep. Code | Width 1/2 (sec) | Status Codes |
|----------|-----------|------------|-----------------|-------------------|---------------|-----------|-----------------|--------------|
| 1 | | 95.8060 | 81.063 | 0.000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

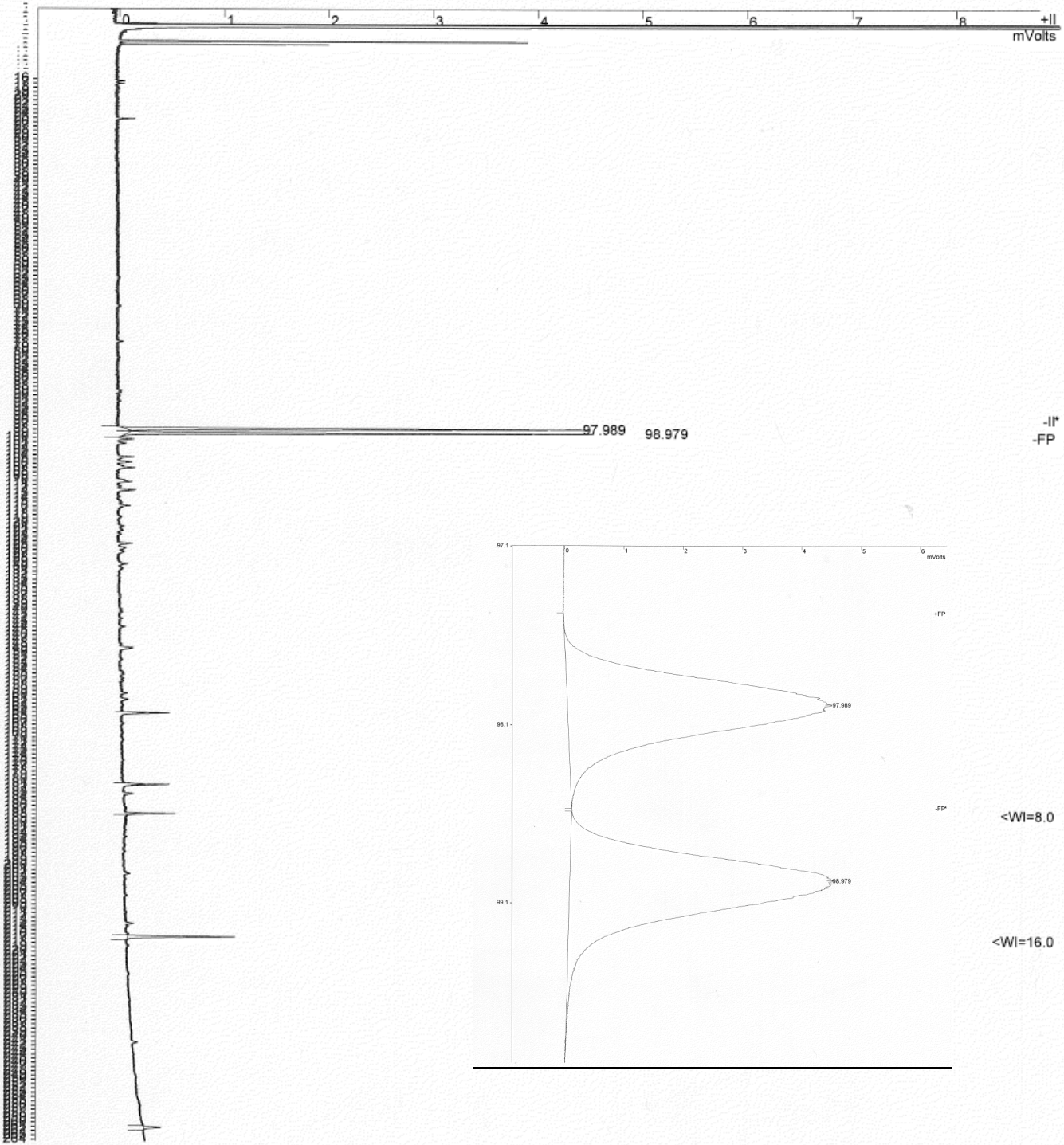
^1H NMR spectrum of **12** (400 MHz, CDCl_3)



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

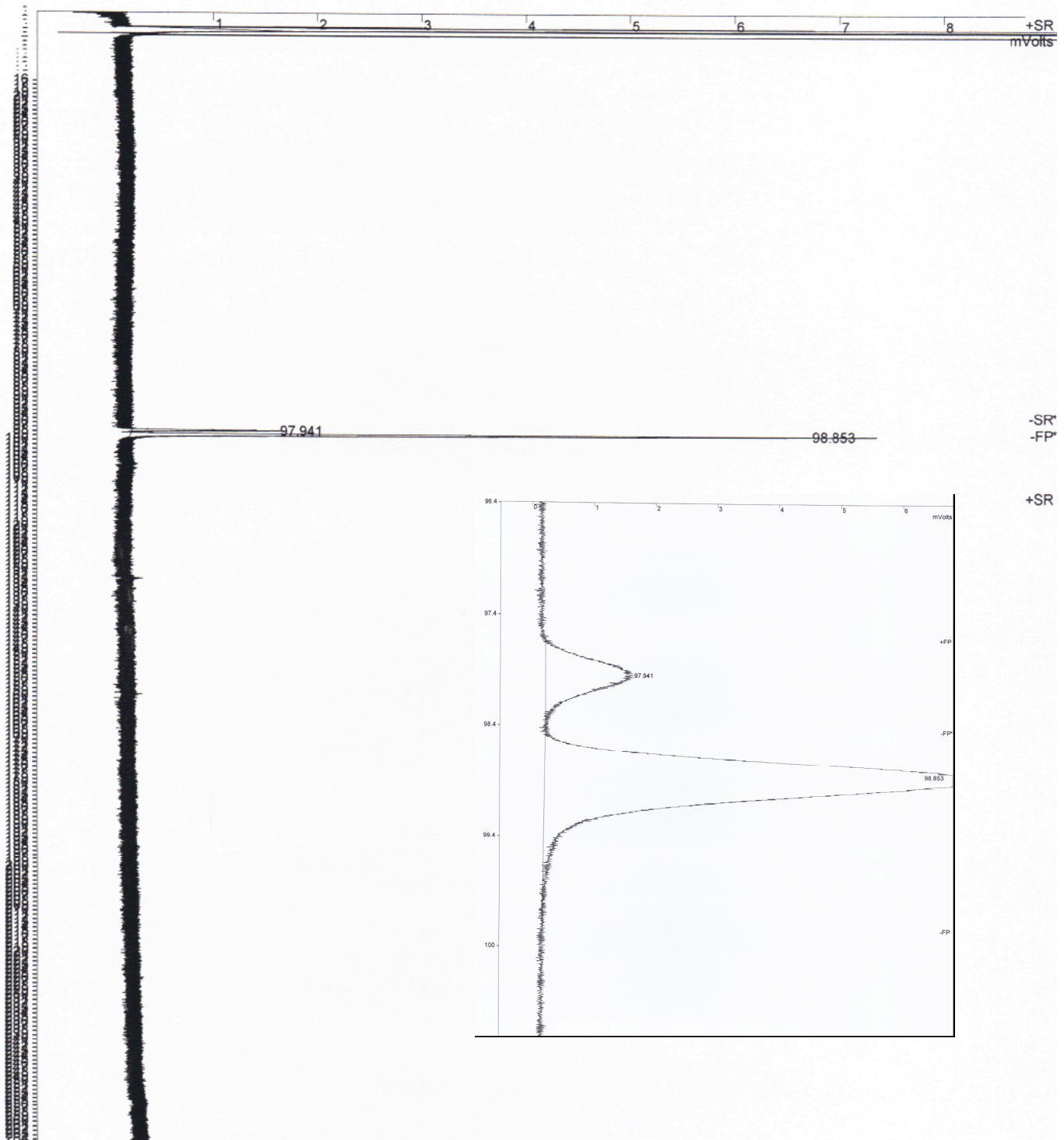


GC trace of racemic **12** (Chiralpak G-TA column, 200 °C injection, 50–180°C at 0.5°C per min., hold 20 min.)



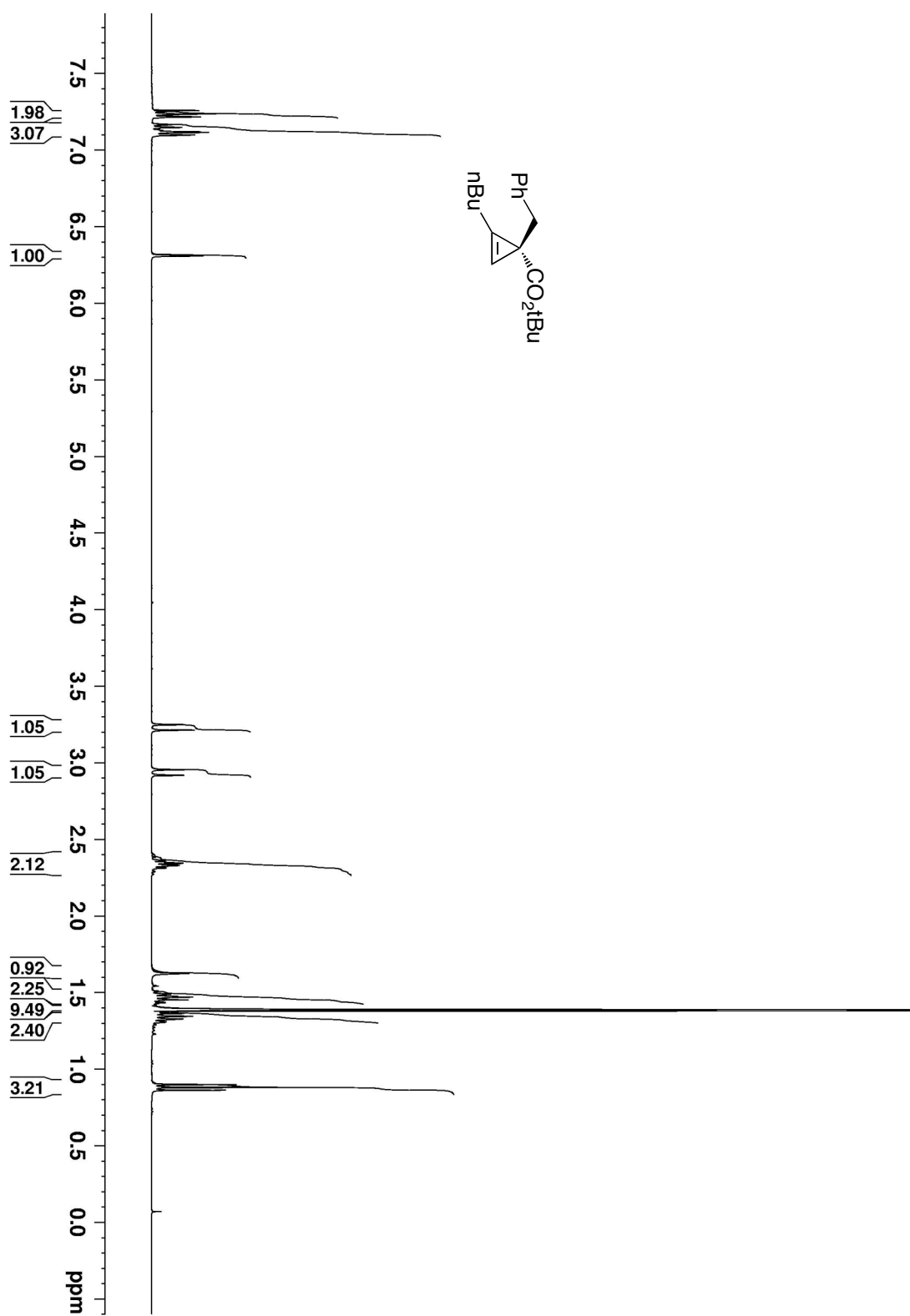
| Peak No. | Peak Name | Result () | Ret. Time (min) | Time Offset (min) | Area (counts) | Sep. Code | Width 1/2 (sec) | Status Codes |
|----------|-----------|------------|-----------------|-------------------|---------------|-----------|-----------------|--------------|
| 1 | | 49.4335 | 97.989 | 0.000 | 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.)

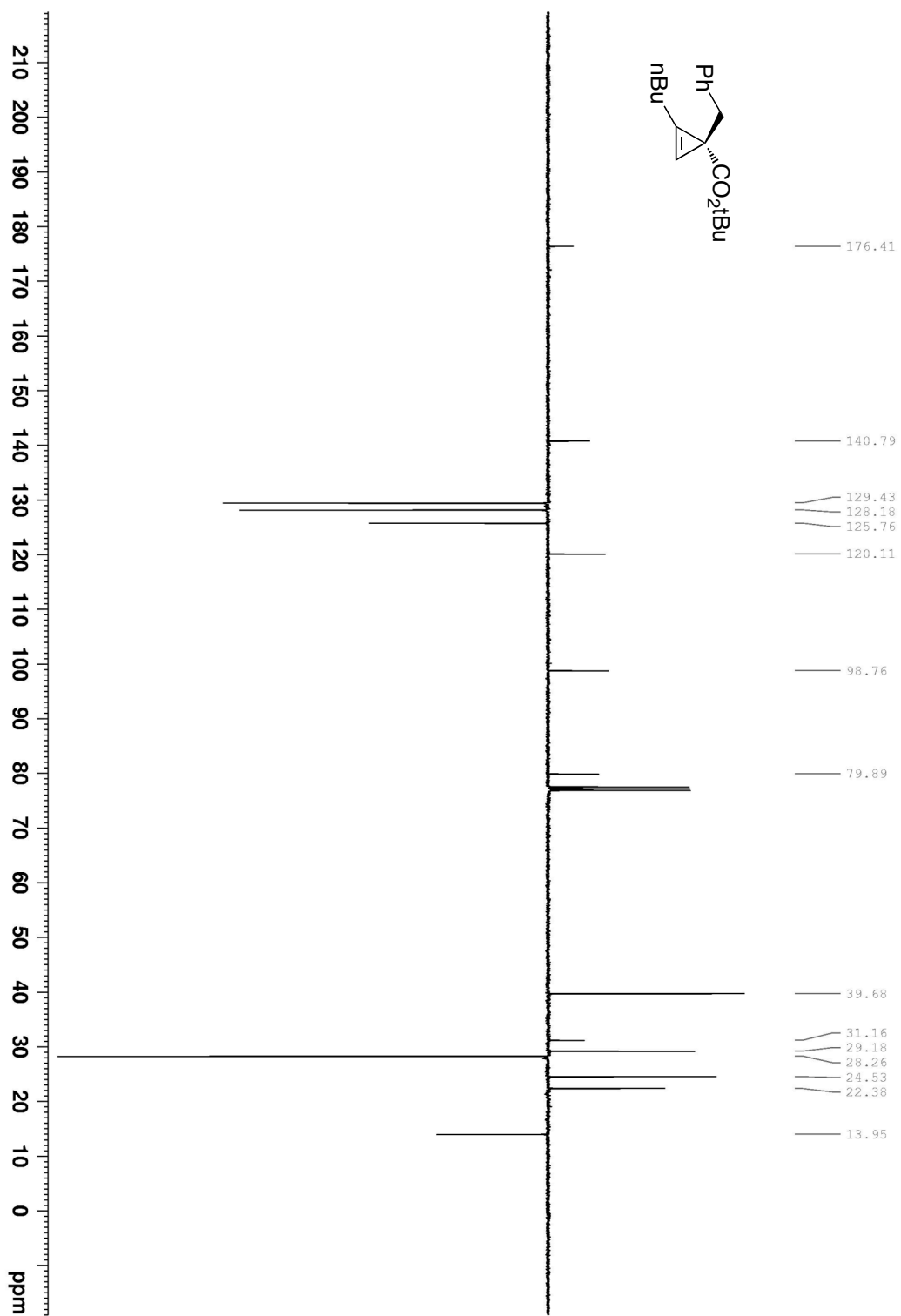


| Peak No. | Peak Name | Result () | Ret. Time (min) | Time Offset (min) | Area (counts) | Sep. Code | Width 1/2 (sec) | Status Codes |
|----------|-----------|------------|-----------------|-------------------|---------------|-----------|-----------------|--------------|
| 1 | | 14.0367 | 97.941 | 0.000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

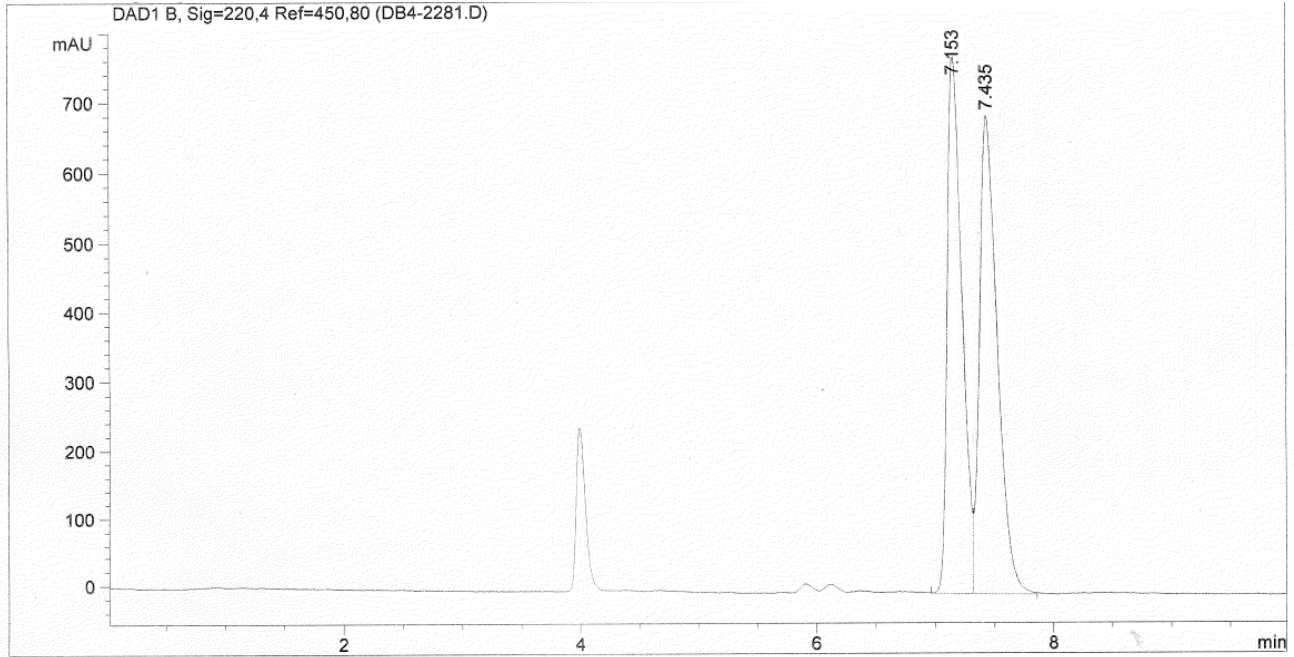
^1H NMR spectrum of **13** (400 MHz, CDCl_3)



^{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)



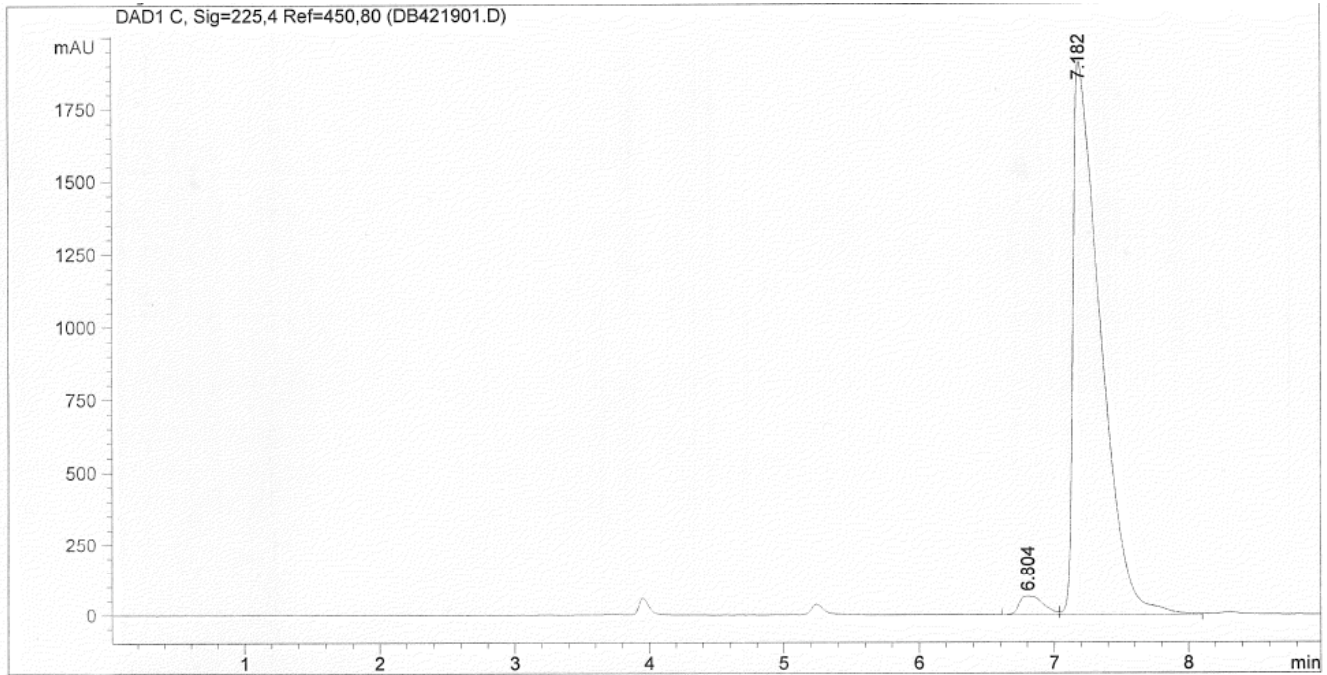
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 Area Percent Report
 =====

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)



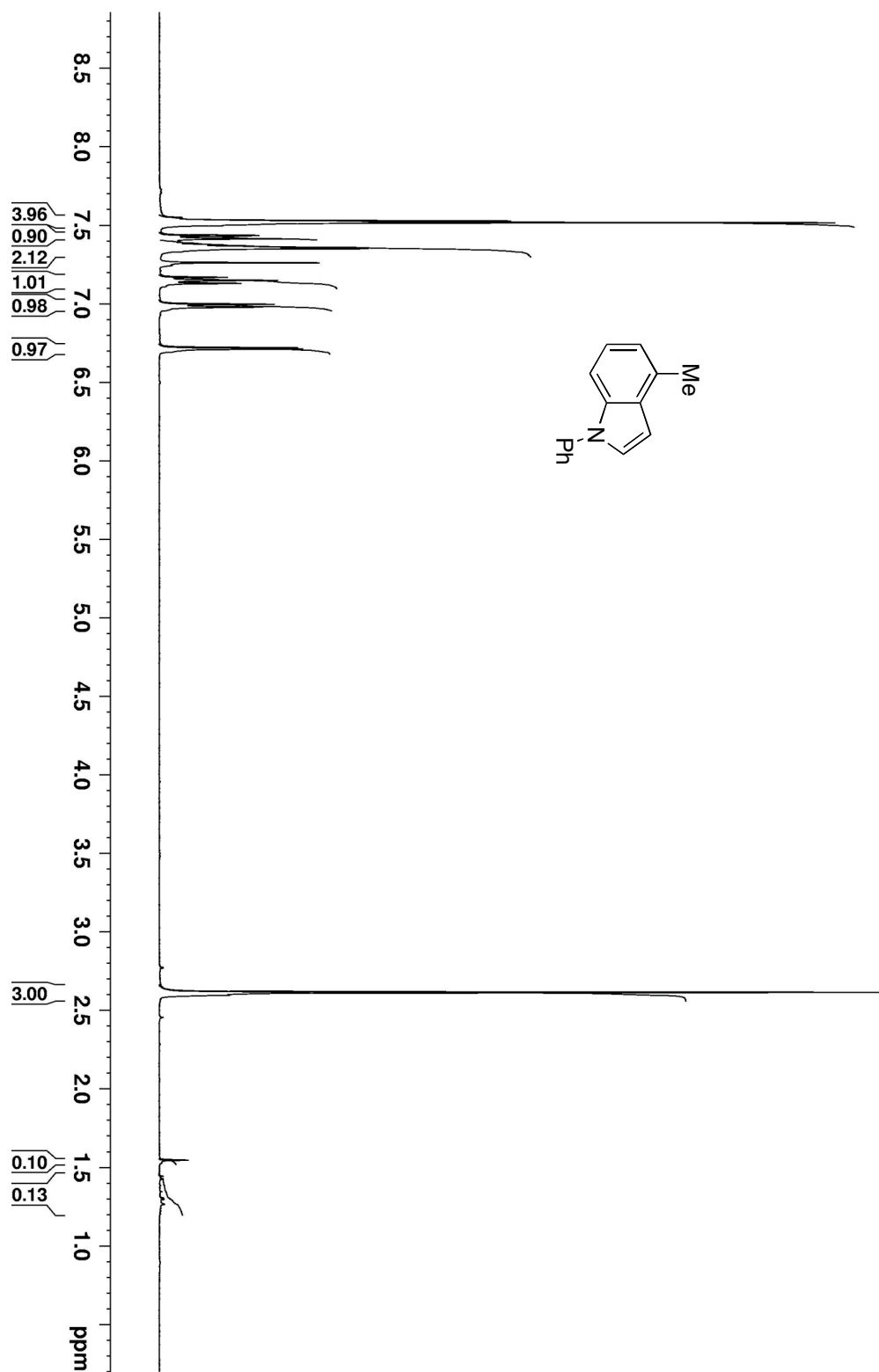
=====
Area Percent Report
=====

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

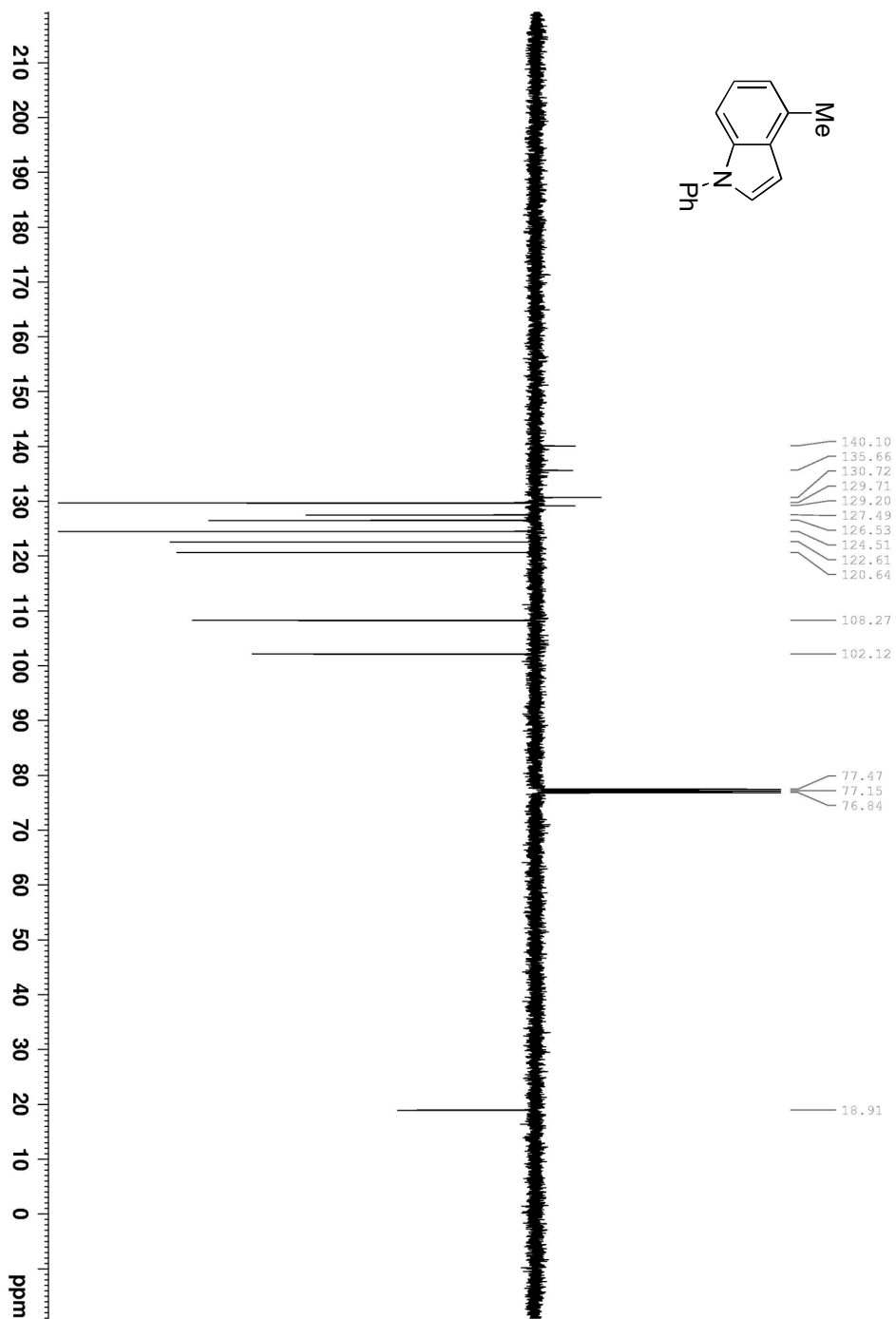
Signal 1: DAD1 C, Sig=225,4 Ref=450,80

| 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 |

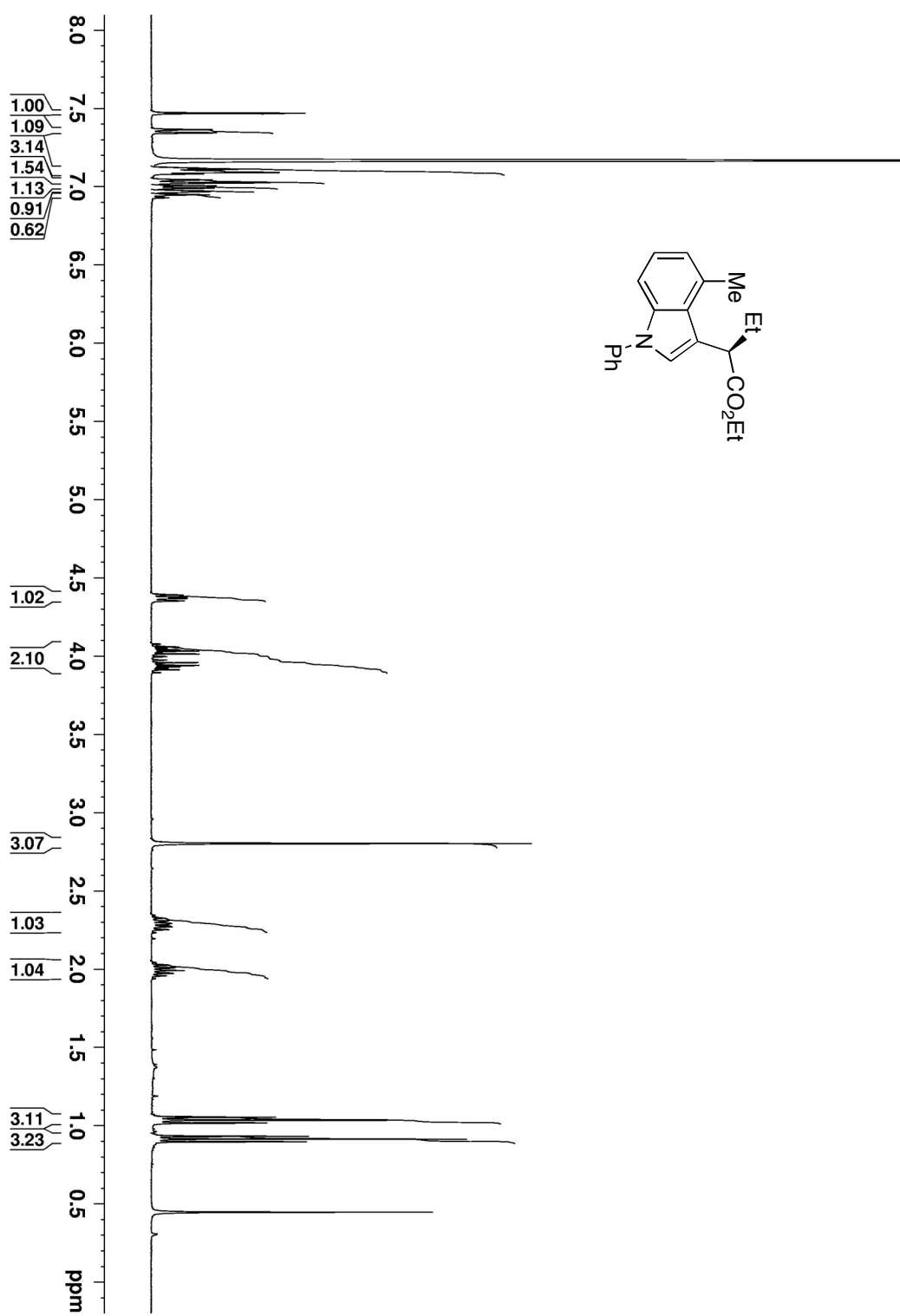
^1H NMR spectrum of **14** (400 MHz, CDCl_3)



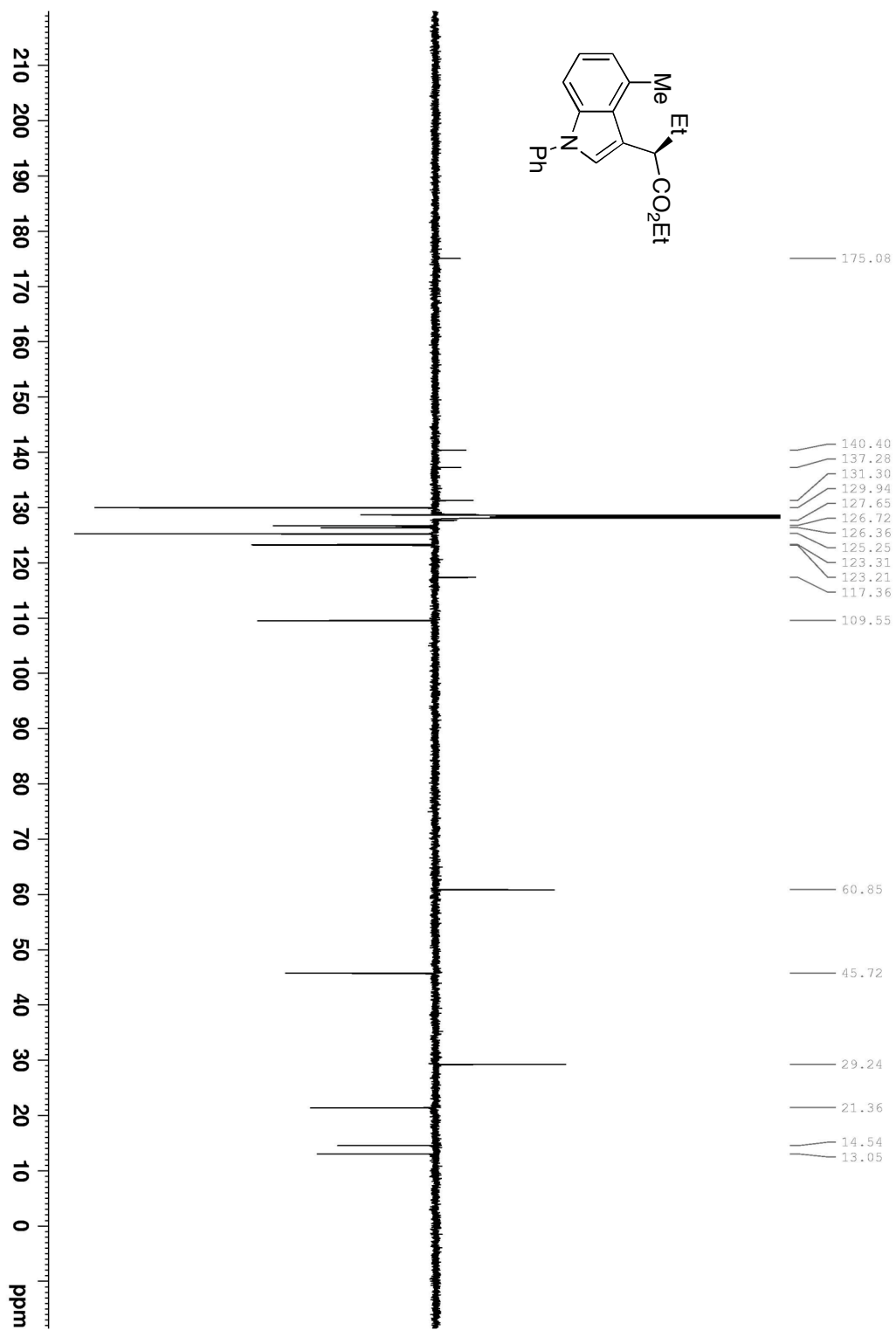
^{13}C NMR spectrum of **14** (100 MHz, CDCl_3)



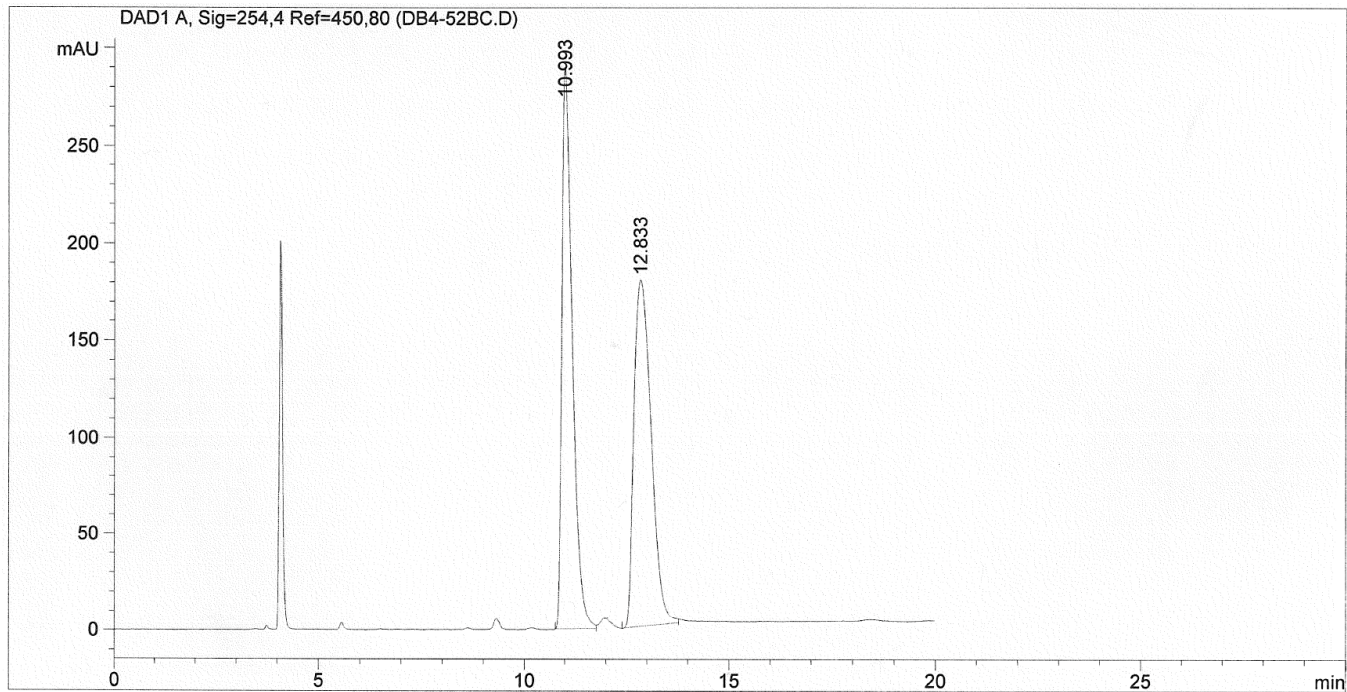
^1H NMR spectrum of **15** (400 MHz, C_6D_6)



^{13}C NMR spectrum of **15** (100 MHz, C_6D_6)



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



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 Area Percent Report
 =====

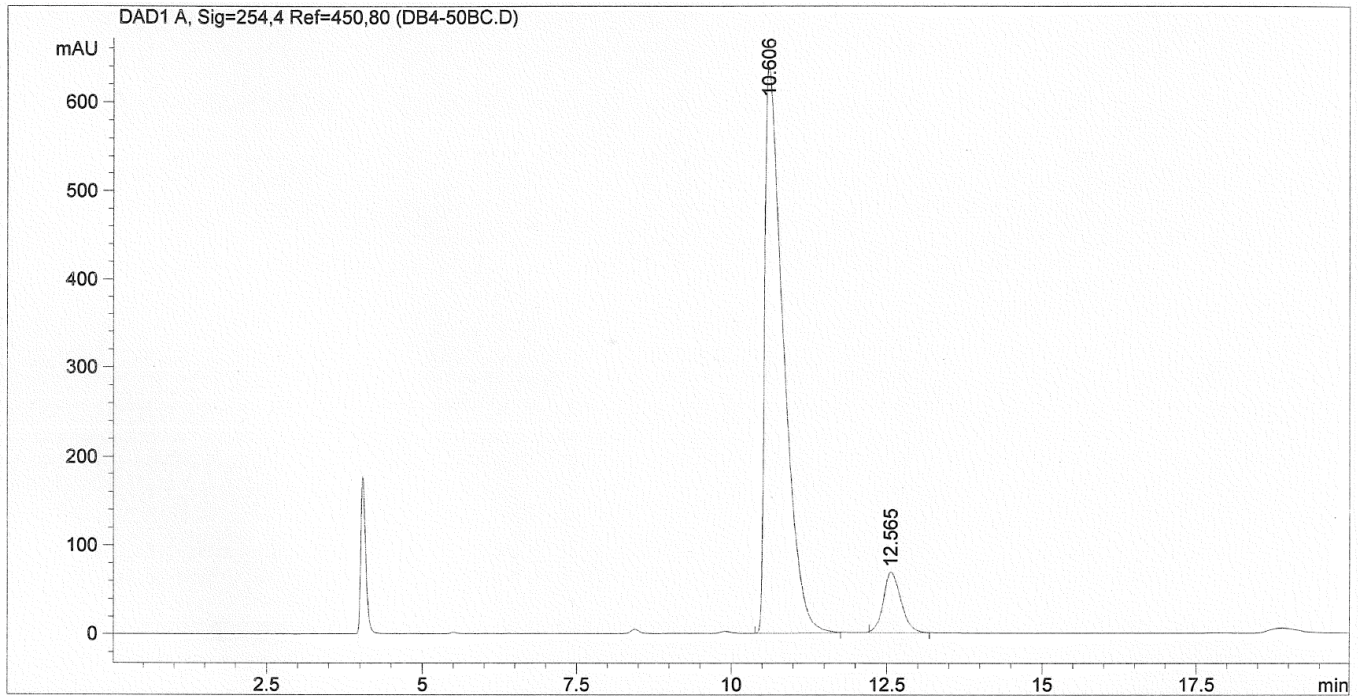
Sorted By : Signal
 Multiplier : 1.0000
 Dilution : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

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

| Peak # | RetTime [min] | Type | Width [min] | Area [mAU*s] | Height [mAU] | Area % |
|--------|---------------|------|-------------|--------------|--------------|---------|
| 1 | 10.993 | PV | 0.2676 | 5127.26123 | 289.21527 | 49.3078 |
| 2 | 12.833 | VB | 0.4551 | 5271.21533 | 179.24860 | 50.6922 |

Totals : 1.03985e4 468.46387

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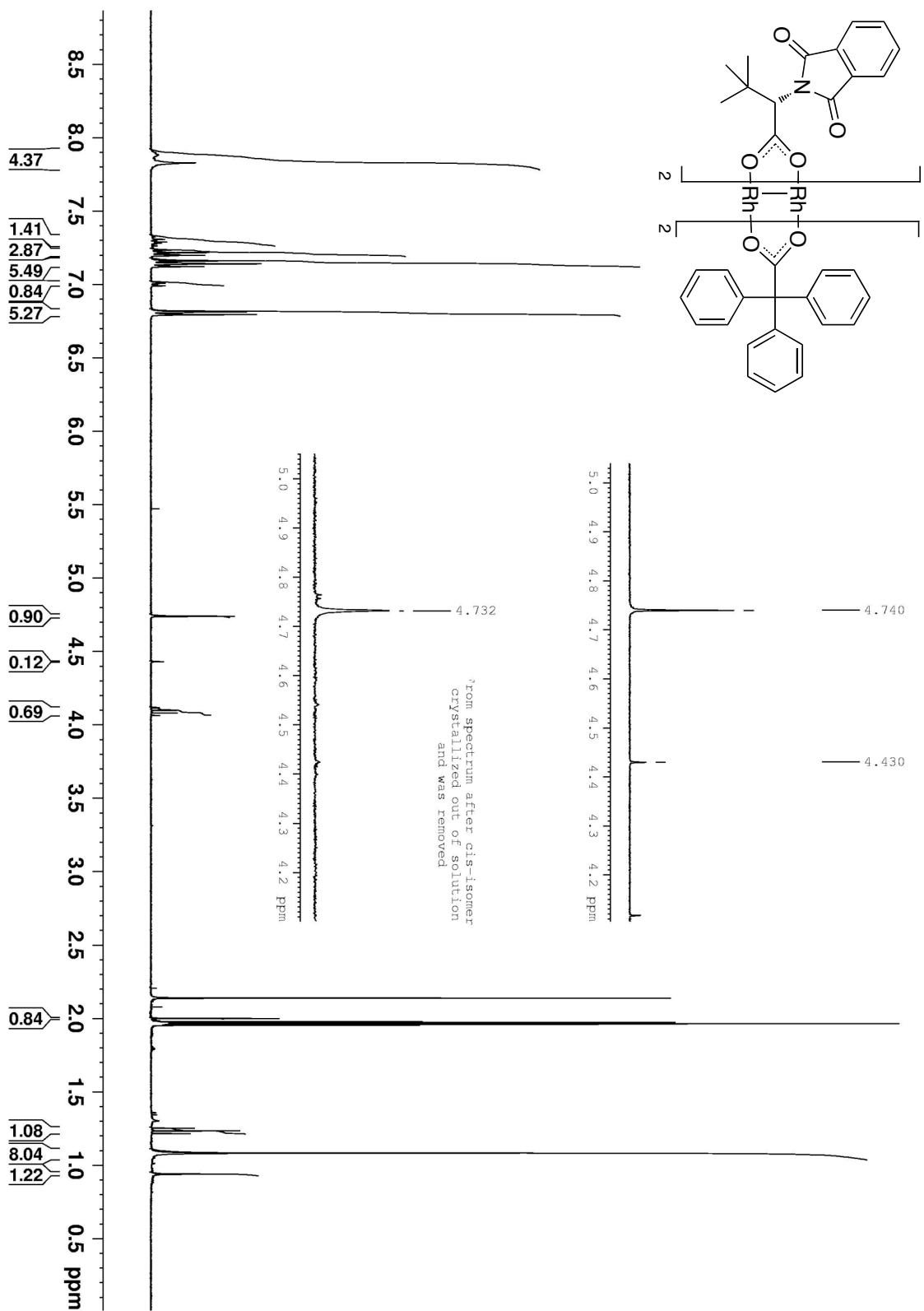
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

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 $\text{Rh}_2(\text{S-PTTL})_2(\text{TPA})_2$ + ethyl acetate adduct (400 MHz, CD_3CN)



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

