One-Pot, Highly Efficient, Asymmetric Synthesis of Ring-Fused Piperidine Derivatives Bearing N,O- or N,N-Acetal Moieties

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Supporting Information

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1. Optimization of the Reaction Condition

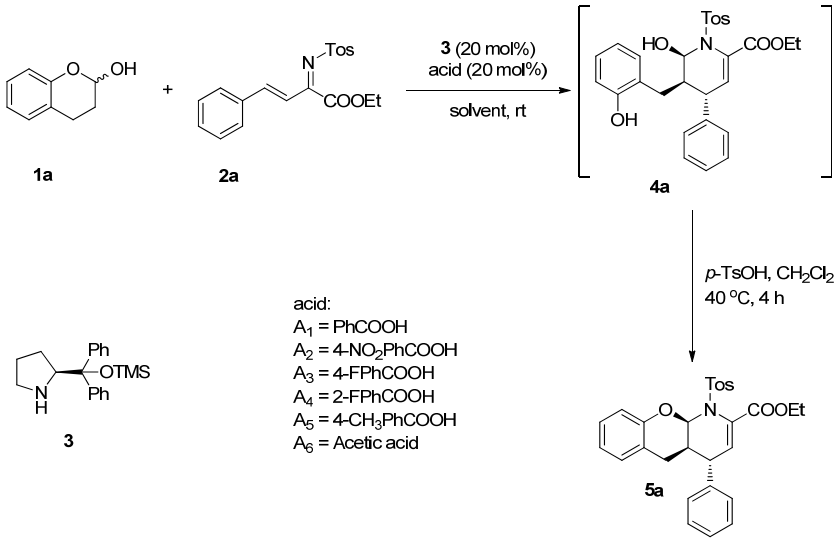


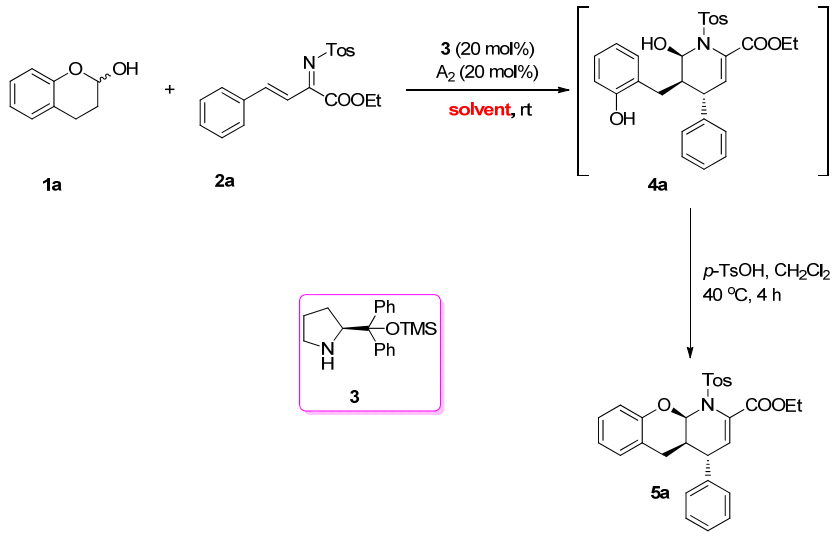
Table 1. Conditions screening[a]

Entry	Catalyst	Acid	Solvent	Time[b] (h)	Yield[c] (%)	ee[d] (%)
1	3	A_1	CH_3CN	12	72.2	99.2
2	3	A_2	CH_3CN	10	77.2	99.4
3	3	A_3	CH_3CN	12	80.3	99.1
4	3	A_4	CH_3CN	12	76.8	99.4
5	3	A_5	CH_3CN	12	70	99.3
6	3	A_6	CH_3CN	12	80.9	99.2
7[h]	3	A_2	CH_3CN/H_2O	12	74.6	98.8
8	3	A_2	Toluene	36	60.8	99.5
9	3	A_2	$CHCl_3$	>48	48.9	98.8
10	3	A_2	DMF	12	76.4	99.5
11[i]	3	A_2	DMF/ H_2O	12	77.3	99.2
12[e]	3	A_2	CH_3CN	18	65	99.4
13[f]	3	A_2	CH_3CN	20	60	99.4
14[g]	3	A_2	CH_3CN	16	85	99.4
15[k]	3	A_2	CH_3CN	16	76	99.4

[a] Unless otherwise specified, all reactions were carried out using **1a** (0.12 mmol, 1.2 equiv) and N-Tos-1-aza-1,3-butadienes **2a** (0.10 mmol, 1.0 equiv) in solvent (500 μL) with **3** (20 mol%) and acid (20 mol%) at 25 °C. After full conversion of the first step, the solvent was removed under vacuum and DCM (1.2 mL) and *p*-TsOH (5.0 equiv) were added to the reaction at 40 °C for 4 hours. When the reaction completed, the mixture was concentrated and the residue was purified by flash chromatography on silica gel to afford **5a**. The diastereoselectivity of **5a** is determined by 1H NMR. [b] For the first step. [c] Isolated yields of product **5a**. [d] Determined by HPLC analysis over chiral stationary phases of **5a**. [e] 10 mol% **3** was used. [f] 5 mol% **3** was used. [g] **5a** was obtained in one-pot, reaction time refer to two steps. [h/i] 10% H_2O was added. [k] After full conversion of the first step, the solvent was not removed and DCM (1.2 mL) and *p*-TsOH (5.0 equiv) were added to the reaction at 40 °C for 4 hours. Entry in ondine marks optimized conditions. *p*-TsOH = *p*-toluenesulfonic acid; TMS = trimethylsilyl; Tos = *p*-tolylsulfonyl.

2. Details of Reaction Conditions Study

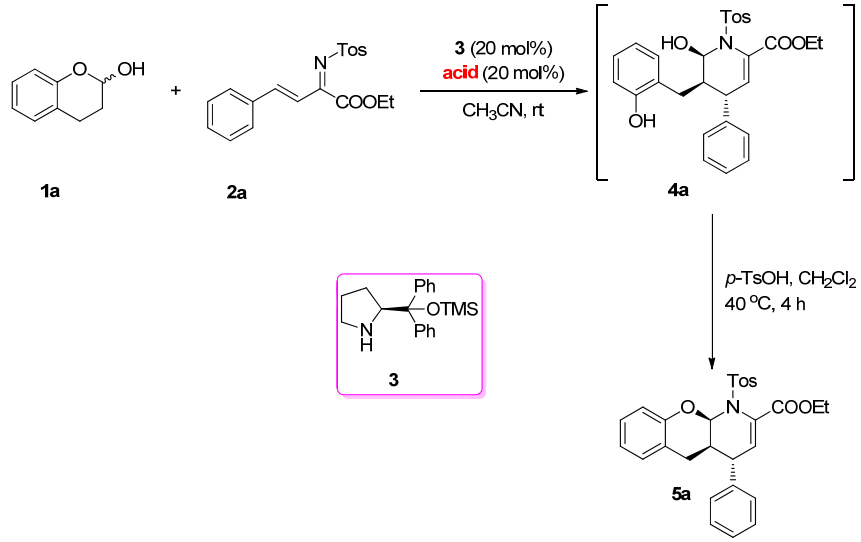
Table 2. Screening of solvents[a]



Entry	Solvent	Time (h)	Yield (%)[b]	5a ee (%)[c]
1	CH_3CN	10	77.2	99.4
2[d]	CH_3CN/H_2O	12	74.6	98.8
3	Toluene	36	60.8	99.5
4	$CHCl_3$	>48	48.9	98.8
5	DMF	12	76.4	99.5
6[e]	DMF/H_2O	12	77.3	99.2

[a] Unless otherwise specified, all reactions were carried out using **1a** (0.12 mmol, 1.2 equiv) and N-Tos-1-aza-1,3-butadienes **2a** (0.10 mmol, 1.0 equiv) in solvent (500 μL) with **3** (20 mol%) and A_2 (20 mol%) at 25 °C. After full conversion of the first step, the solvent was removed under vacuum and DCM (1.2 mL) and *p*-TsOH (5.0 equiv) were added to the reaction at 40 °C for 4 hours. When the reaction completed, the mixture was concentrated and the residue was purified by flash chromatography on silica gel to afford **5a**. [b] Isolated yields of product **5a**. [c] Determined by HPLC analysis over chiral stationary phases of **5a**. [d/e] 10% H_2O was added. Entry in ondine marks optimized conditions. DMF = Dimethylformamide; *p*-TsOH = *p*-toluenesulfonic acid; Tos = *p*-tolylsulfonyl.

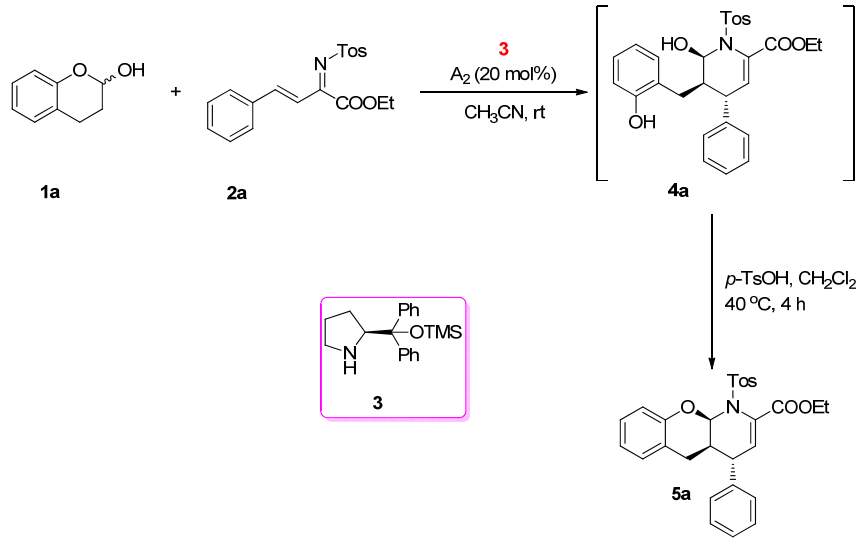
Table 3. Screening of acids[a]



Entry	Acid	Yield (%)[b]	4a ee (%)[c]
1	PhCOOH	72.2	99.2
2	4-NO_2PhCOOH	77.2	99.4
3	4-FPhCOOH	80.3	99.1
4	2-FPhCOOH	76.8	99.4
5	4-CH_3PhCOOH	70	99.3
6	acetic Acid	80.9	99.2

[a] Unless otherwise specified, all reactions were carried out using **1a** (0.12 mmol, 1.2 equiv) and N-Tos-1-aza-1,3-butadienes **2a** (0.10 mmol, 1.0 equiv) in CH_3CN (500 μL) with **3** (20 mol%) and acid (20 mol%) at 25 °C. After full conversion of the first step, the solvent was removed under vacuum and DCM (1.2 mL) and *p*-TsOH (5.0 equiv) were added to the reaction at 40 °C for 4 hours. When the reaction completed, the mixture was concentrated and the residue was purified by flash chromatography on silica gel to afford **5a**. [b] Isolated yields of product **5a**. [c] Determined by HPLC analysis over chiral stationary phases of **5a**. Entry in ondine marks optimized conditions. *p*-TsOH = *p*-toluenesulfonic acid.

Table 4. Screening of catalyst[a]

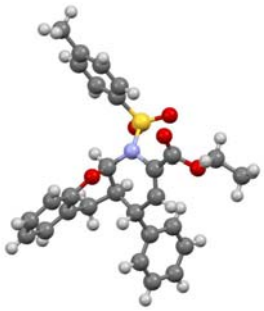


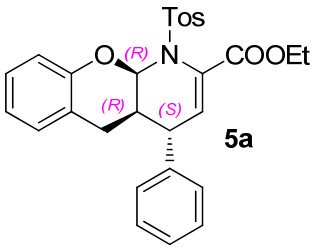
Entry	Catalyst	Time (h)	Yield (%)[b]	5a ee (%)[c]
1	5 mol %	20	60	99.4
2	10 mol %	18	65	99.4
3	20 mol %	10	77.2	99.4

[a] Unless otherwise specified, all reactions were carried out using **1a** (0.12 mmol, 1.2 equiv) and N-Tos-1-aza-1,3-butadienes **2a** (0.10 mmol, 1.0 equiv) in CH_3CN (500 μL) with **3** (mol%) and A_2 (20 mol%) at 25 °C. After full conversion of the first step, the solvent was removed under vacuum and DCM (1.2 mL) and *p*-TsOH (5.0 equiv) were added to the reaction at 40 °C for 4 hours. When the reaction completed, the mixture was concentrated and the residue was purified by flash chromatography on silica gel to afford **5a**. [b] Isolated yields of product **5a**. [c] Determined by HPLC analysis over chiral stationary phases of **5a**. Entry in ondine marks optimized conditions. *p*-TsOH = *p*-toluenesulfonic acid.

3. Single Crystal X-Ray Diffraction Data for Compounds 5a and 7

[CCDC 1429399 **(5a)** and CCDC 1432291 **(8)** contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.].





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Cell: a=9.9601(8) b=10.8355(7) c=23.6278(13)
alpha=90 beta=90 gamma=90

Temperature: 293 K

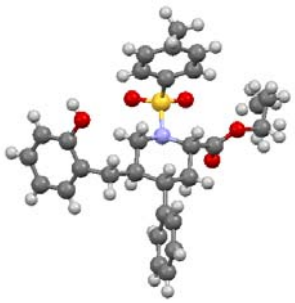
	Calculated	Reported
Volume	2550.0(3)	2550.0(3)
Space group	P 21 21 21	P212121
Hall group	P 2ac 2ab	?
Moiety formula	C28 H27 N O5 S	?
Sum formula	C28 H27 N O5 S	C28 H27 N O5 S
Mr	489.57	489.57
Dx,g cm-3	1.275	1.275
Z	4	4
Mu (mm-1)	1.443	1.443
F000	1032.0	1032.0
F000'	1036.35	
h,k,lmax	11,12,28	11,12,28
Nref	4562[2601]	3770
Tmin,Tmax	0.891,0.904	0.893,0.906
Tmin'	0.891	

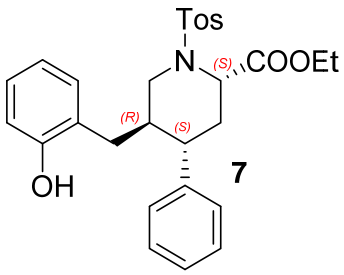
Correction method= # Reported T Limits: Tmin=0.893 Tmax=0.906 AbsCorr = MULTI-SCAN

Data completeness= 1.45/0.83 Theta(max)= 67.250

R(reflections)= 0.0479(3120) wR2(reflections)= 0.1383(3770)

S = 1.071 Npar= 319





Bond precision: C-C = 0.0033 A Wavelength=0.71073

Cell: a=10.0985(8) b=14.8716(11) c=17.4835(13)

alpha=90 beta=90 gamma=90

Temperature: 293 K

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Hall group	P 2ac 2ab	P 2ac 2ab
Moiety formula	C28 H31 N O5 S	C28 H31 N O5 S
Sum formula	C28 H31 N O5 S	C28 H31 N O5 S
Mr	493.60	493.60
Dx,g cm-3	1.249	1.249
Z	4	4
Mu (mm-1)	0.161	0.161
F000	1048.0	1048.0
F000'	1048.97	
h,k,lmax	12,17,20	12,17,20
Nref	4625[2625]	4623
Tmin,Tmax		
Tmin'		

Correction method= Not given

Data completeness= 1.76/1.00 Theta(max)= 25.010

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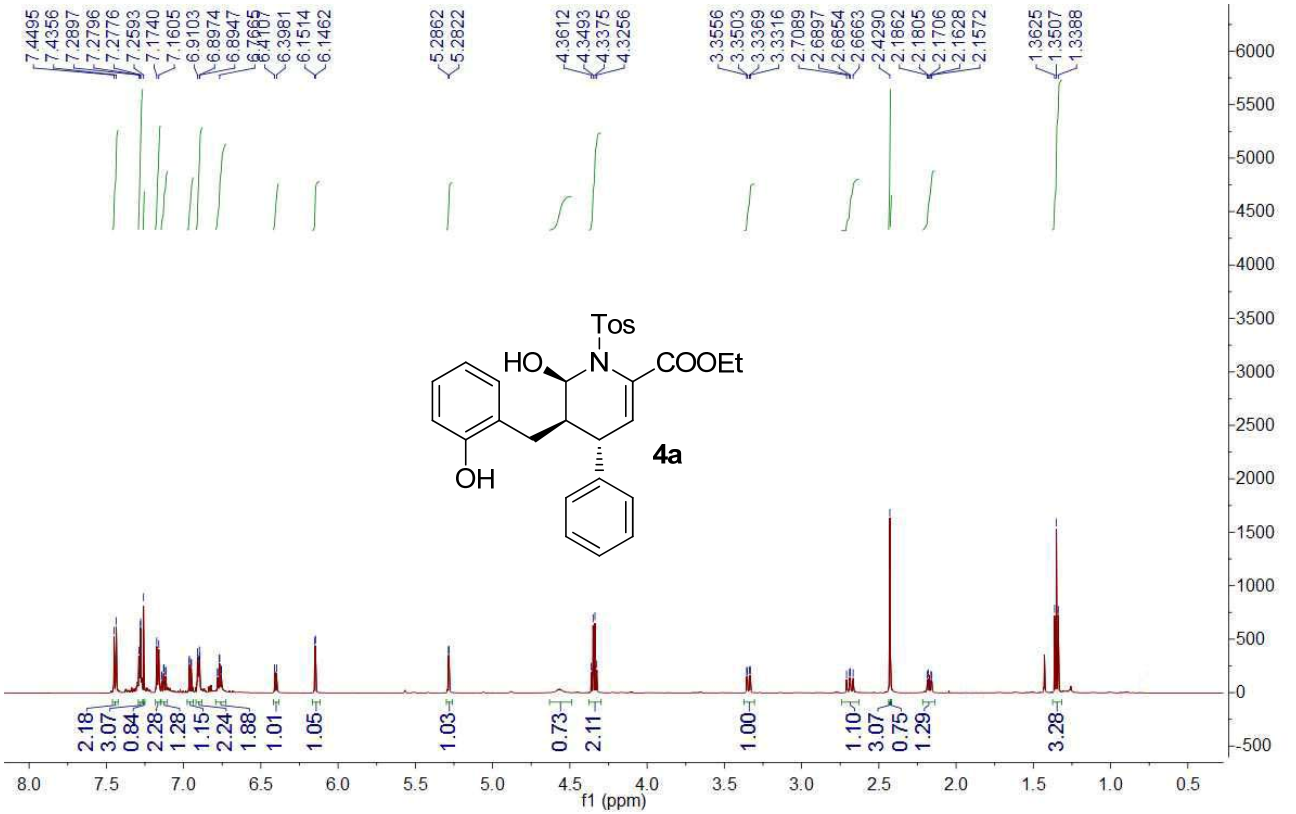
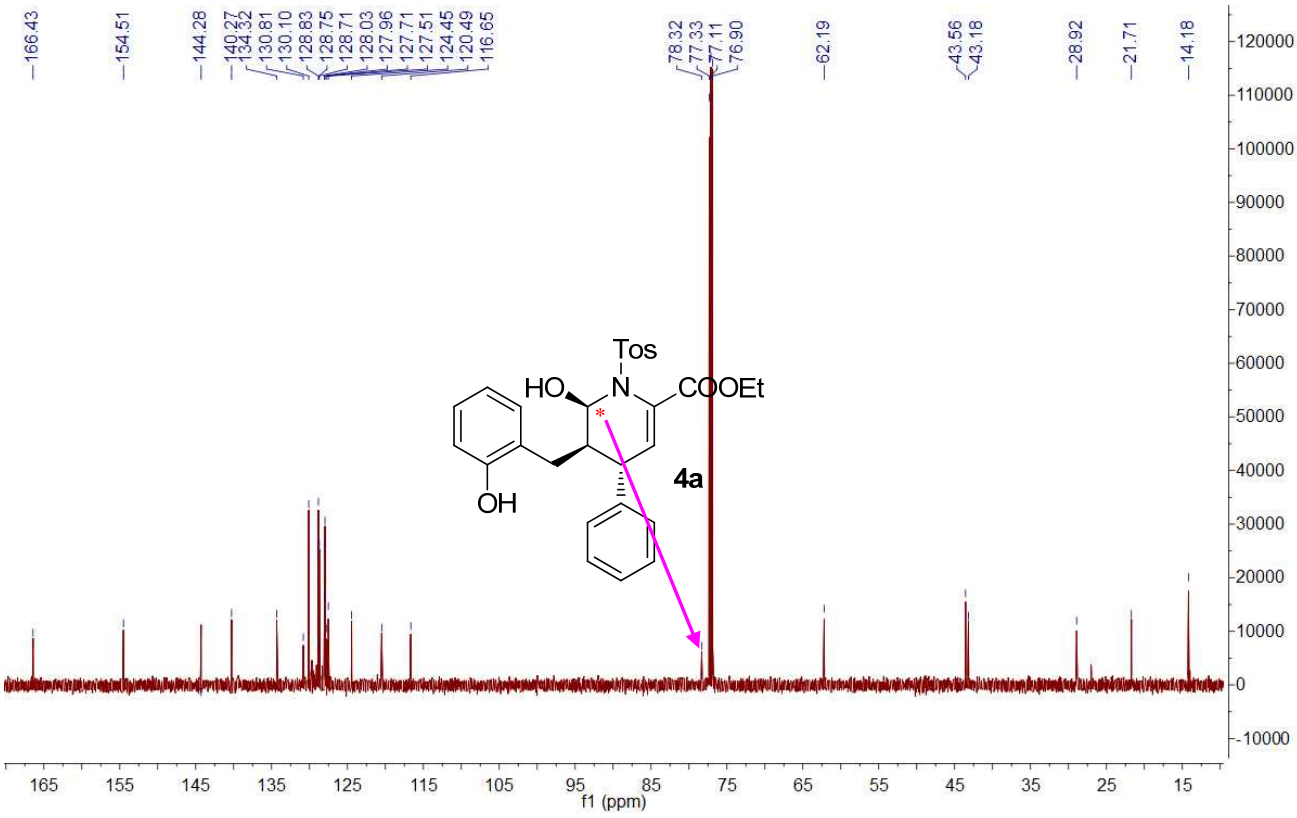
S = 1.043 Npar= 324

4. NMR spectra and HPLC Traces





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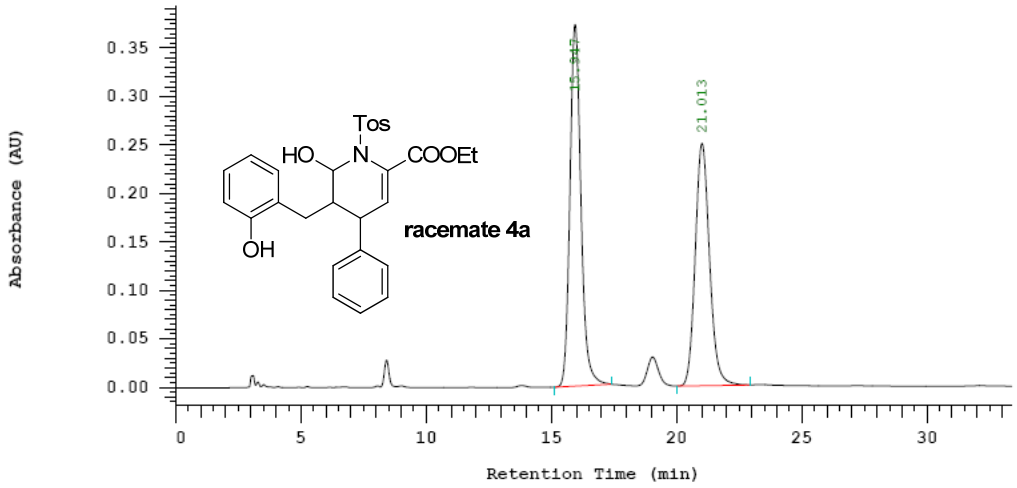

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Peak Quantitation: AREA

Calculation Method: AREA%

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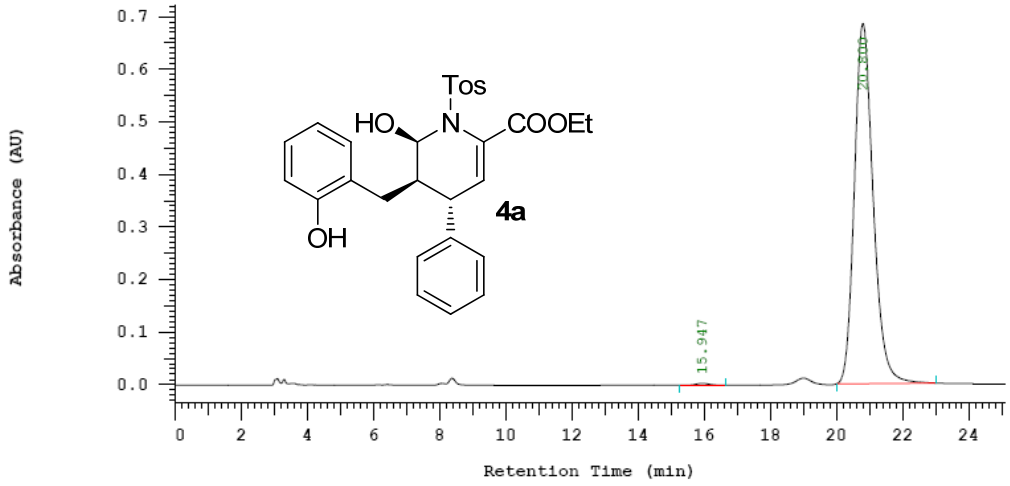
Chrom Type: Fixed WL Chromatogram, 210 nm



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Peak Quantitation: AREA

Calculation Method: AREA%

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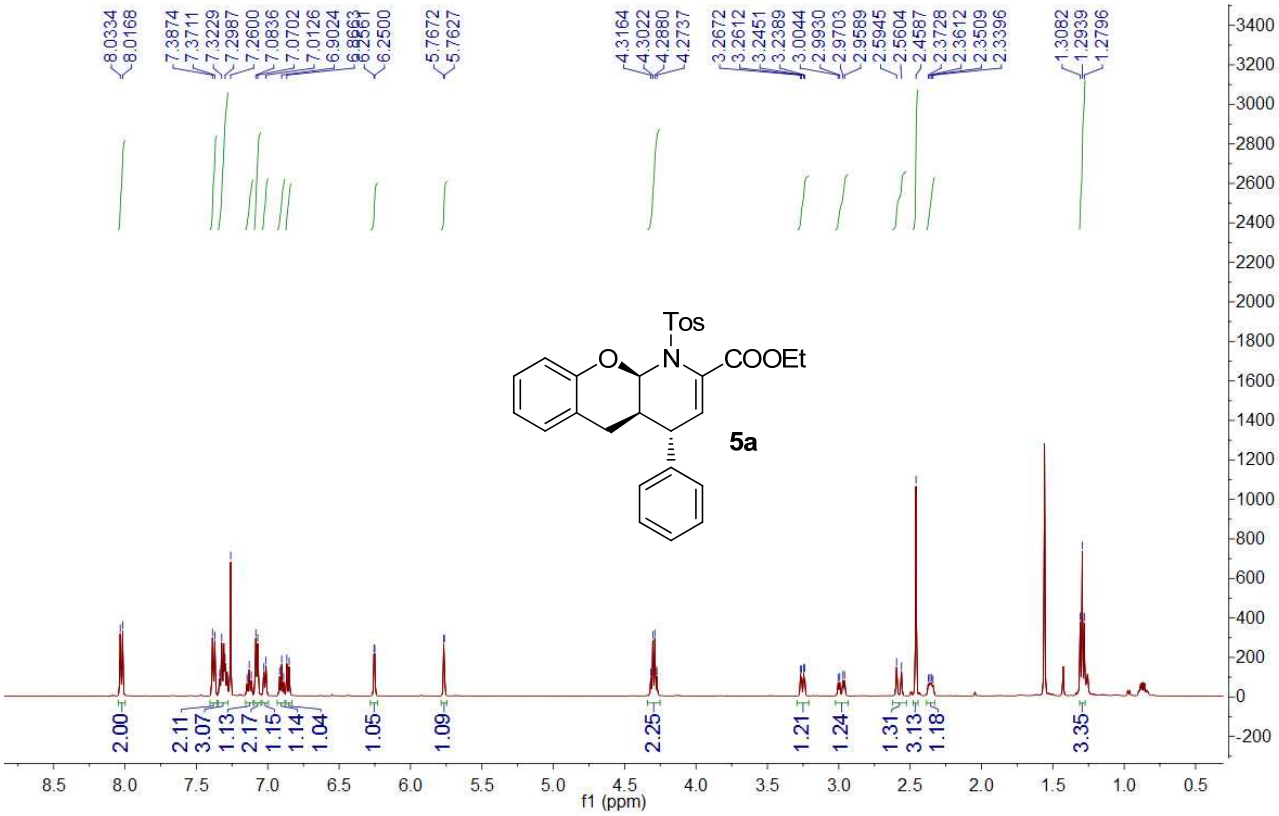
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4.2737
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3.2612
3.2451
3.2389
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2.5604
2.4587
2.3728
2.3612
2.3509
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1.3082
1.2939
1.2796
Tos
N
O
COOEt
5a
2.00
2.11
3.07
1.13
2.17
1.15
1.14
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1.09
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3.13
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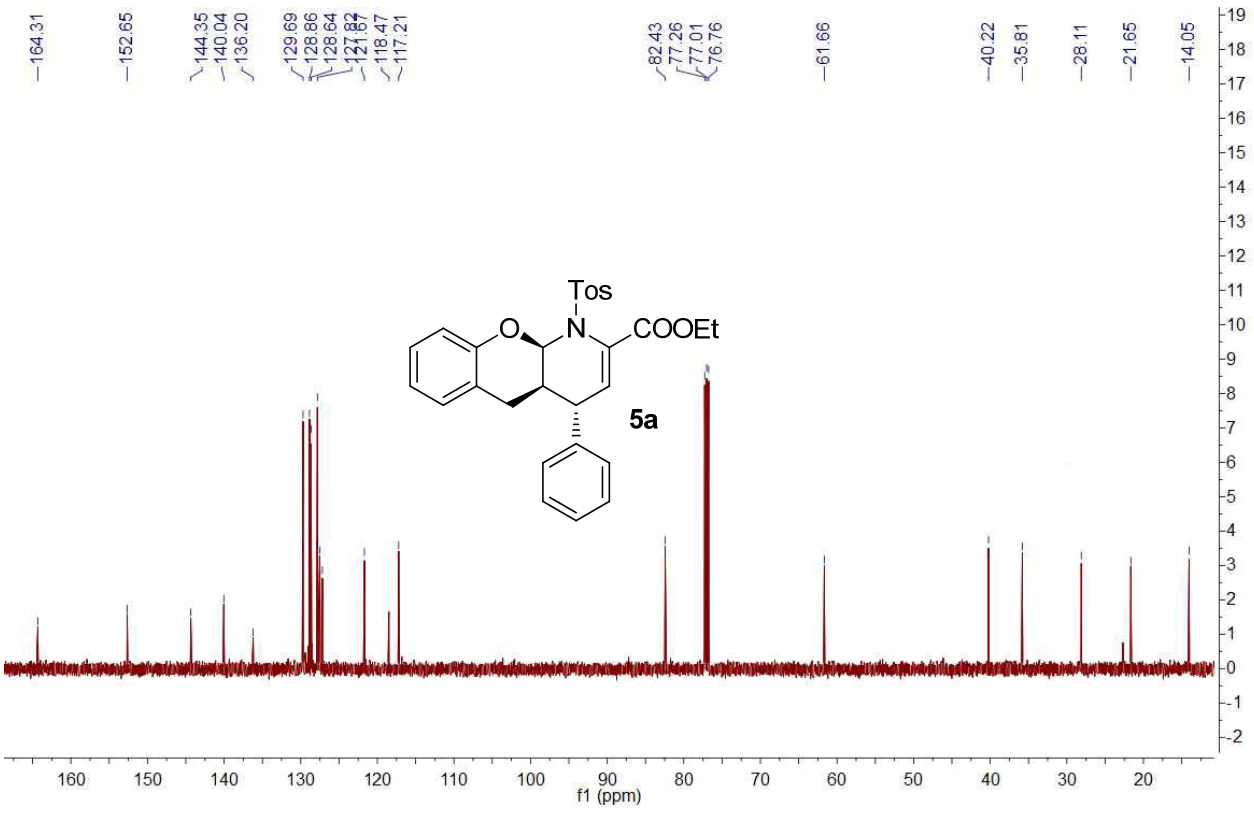
3.35
f1 (ppm)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77.26
77.01
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61.66
40.22
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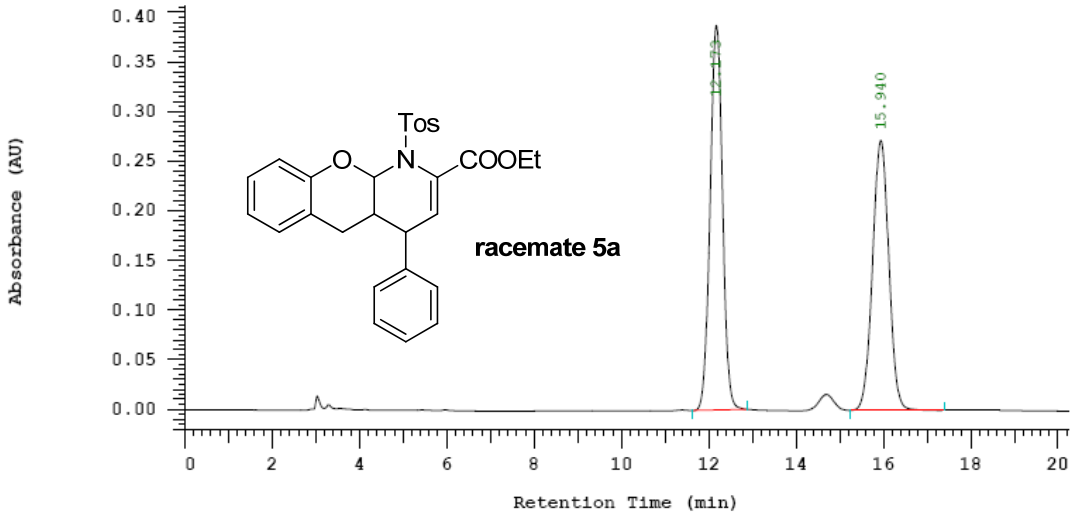
Tos
N
O
COOEt
5a
f1 (ppm)

Chrom Type: Fixed WL Chromatogram, 210 nm



Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
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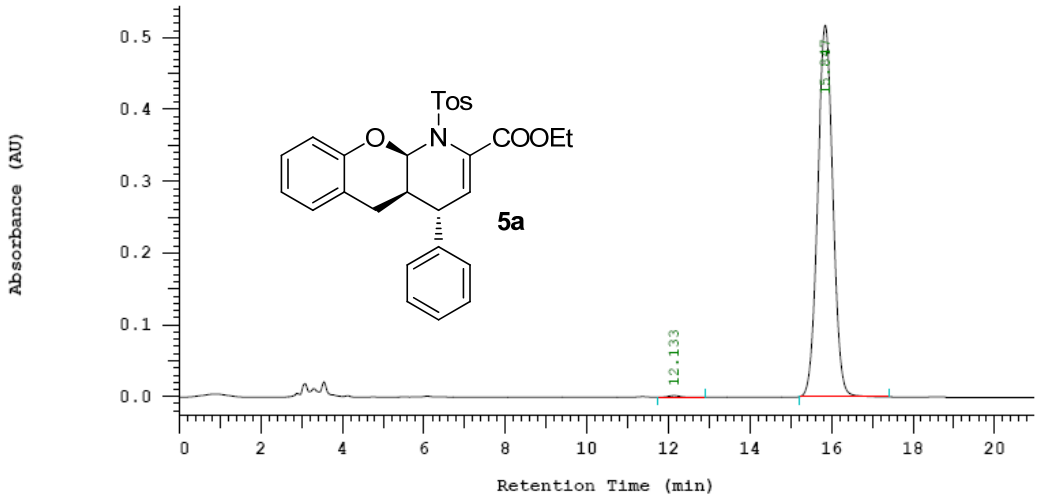
		7363160	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm



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Calculation Method: AREA%

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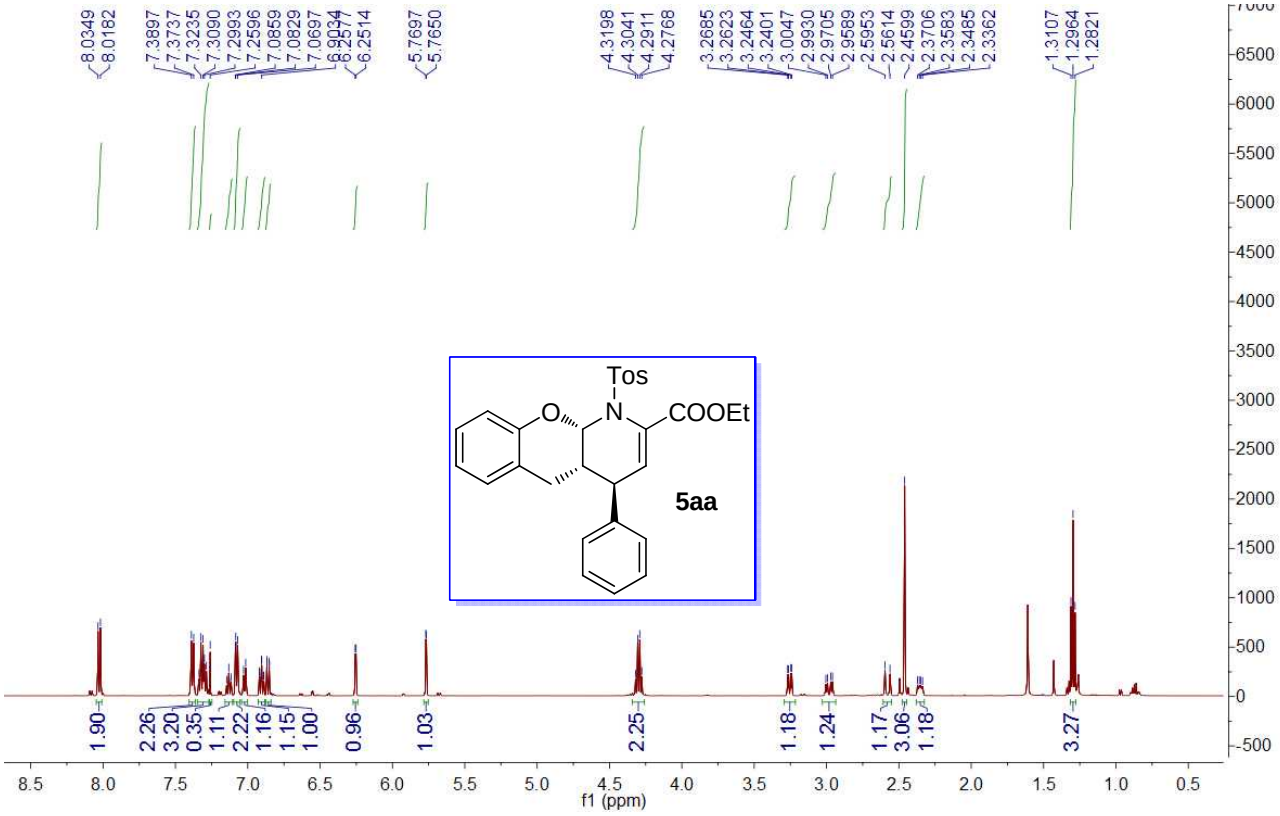
(*R*) catalyst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6.2514
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4.2768
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3.2623
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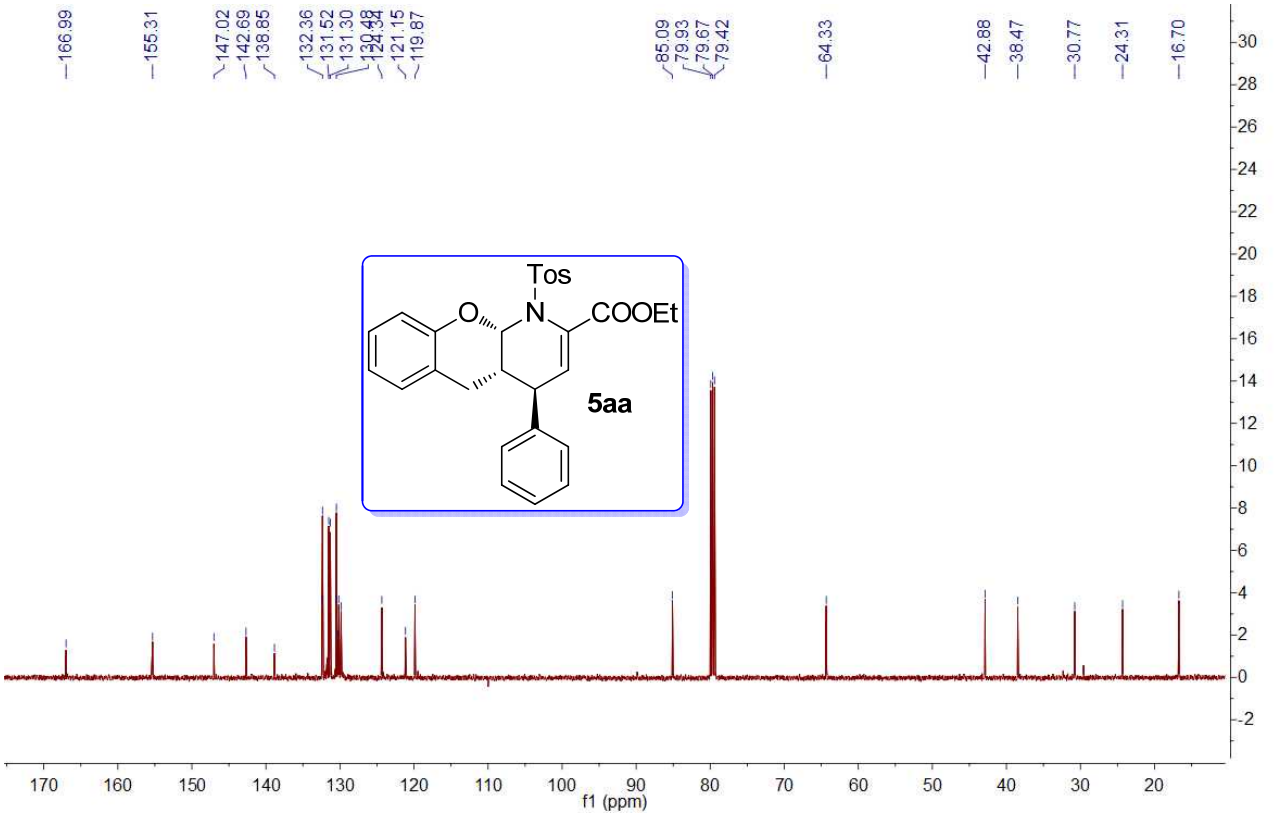
f1 (ppm)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Tos
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N
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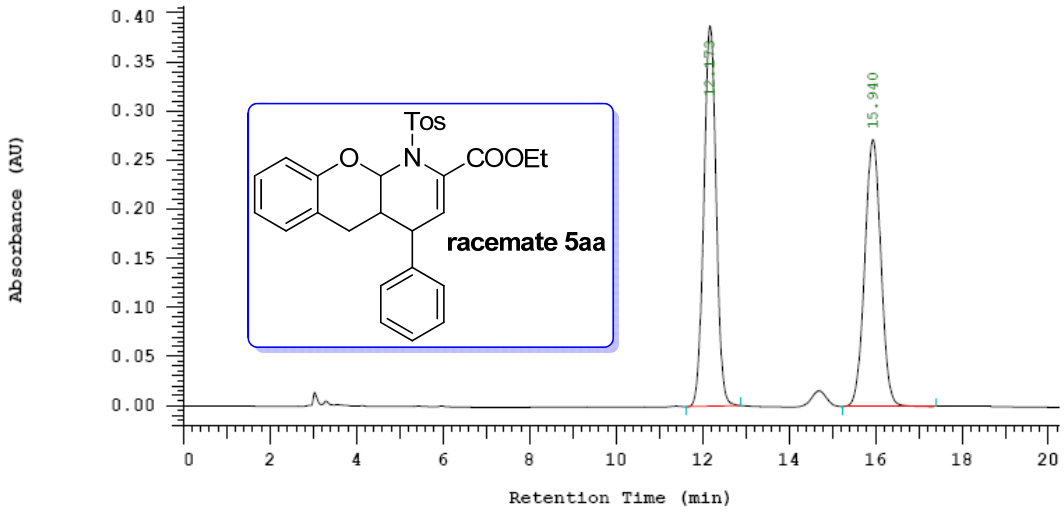
5aa
f1 (ppm)

Chrom Type: Fixed WL Chromatogram, 210 nm



Chrom Type: Fixed WL Chromatogram, 210 nm
Peak Quantitation: AREA
Calculation Method: AREA%

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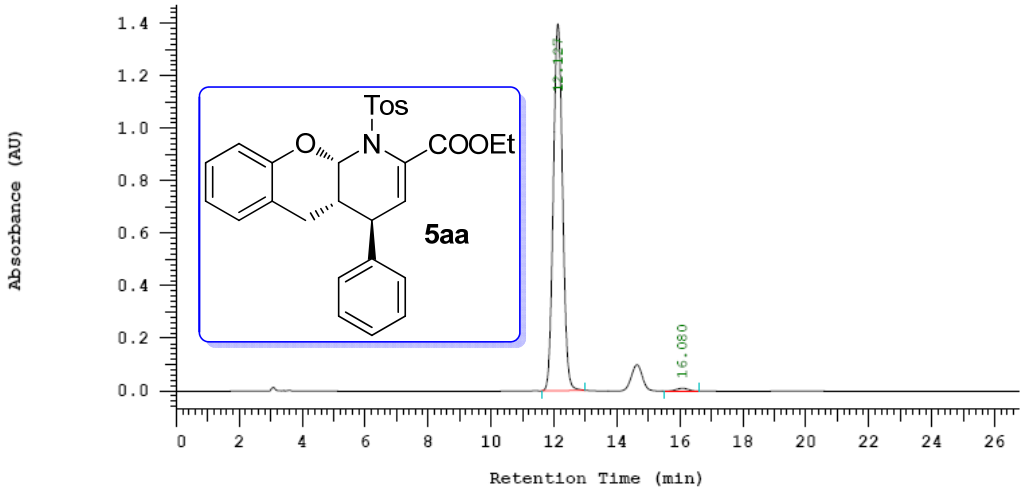
		7363160	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm



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Calculation Method: AREA%

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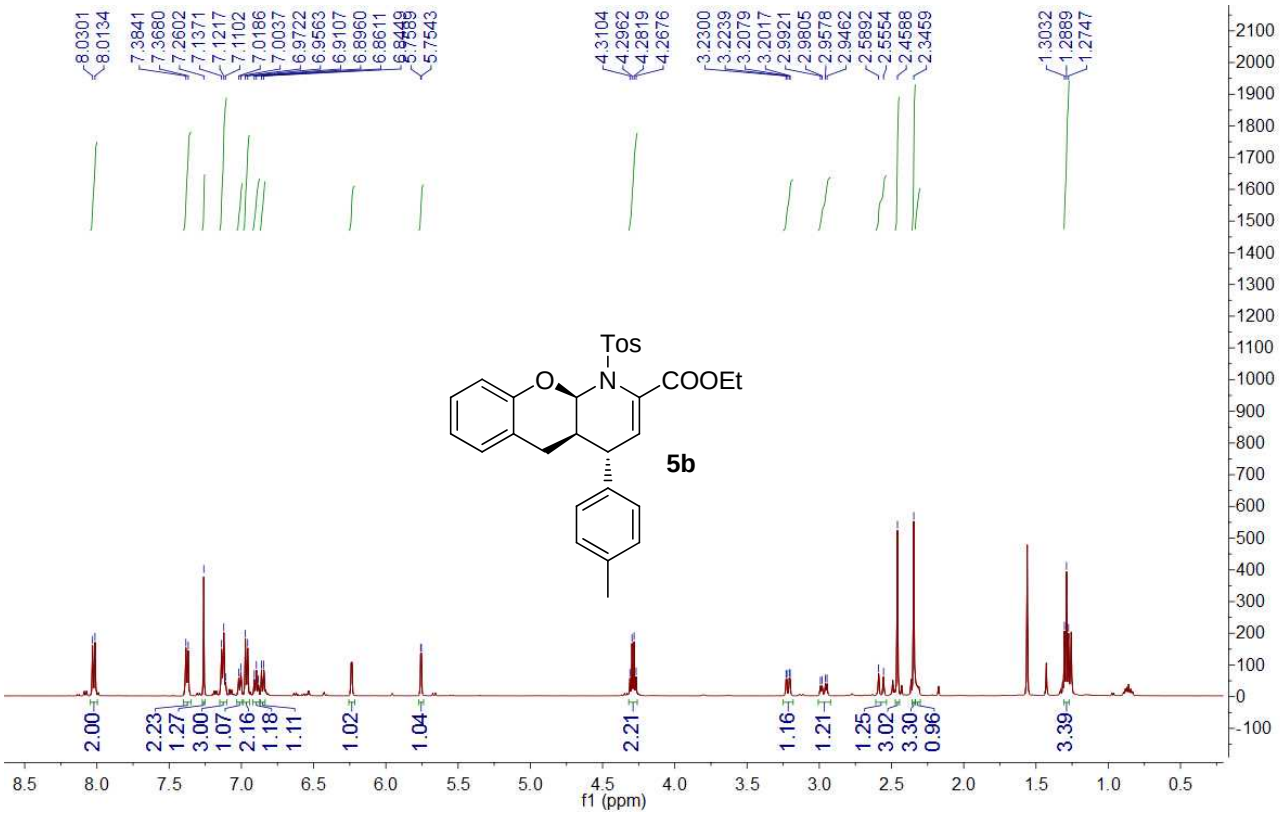
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6.8960
6.8611
5.7543
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4.2962
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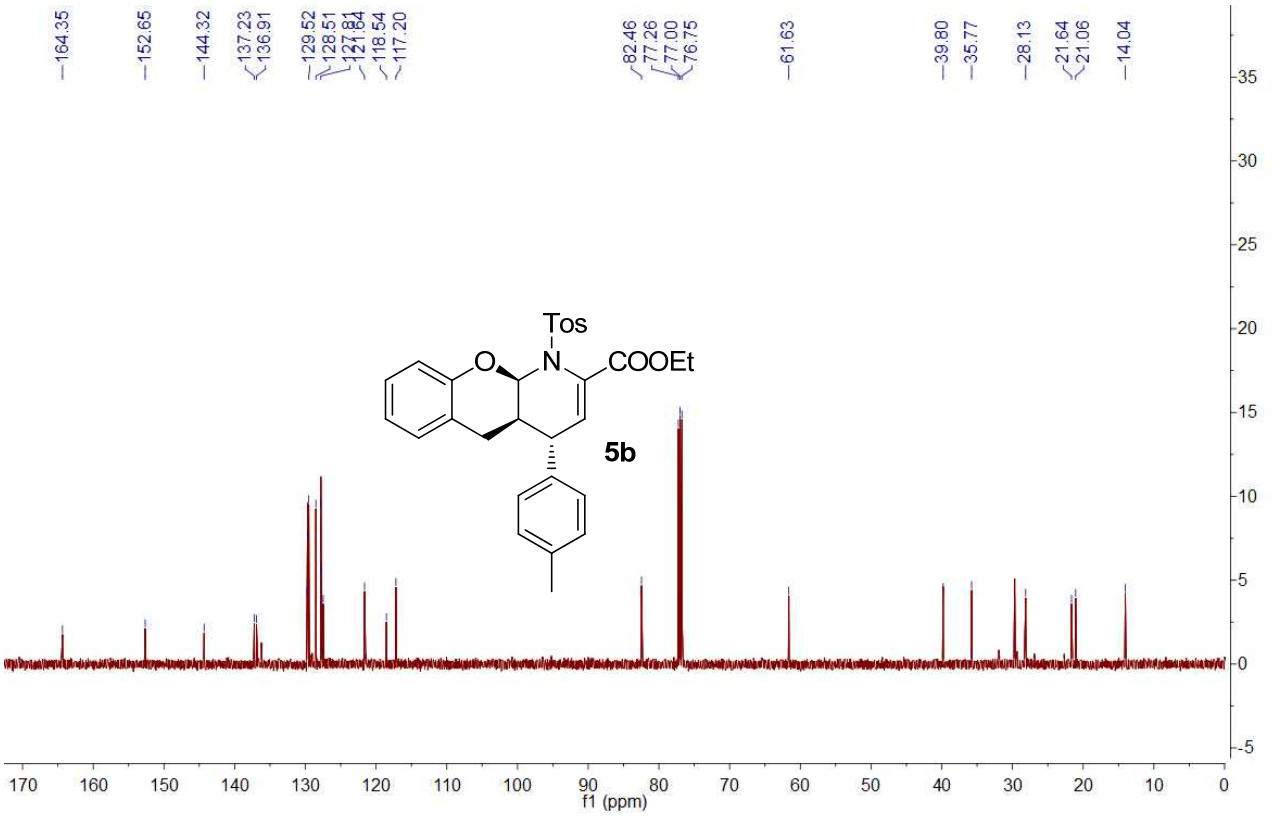
2.9578
2.9462
2.5892
2.5554
2.4588
2.3459
1.3032
1.2889
1.2747
Tos
N
O
COOEt
5b
2.00
2.23
1.27
3.00
1.07
2.16
1.18
1.11
1.02
1.04
2.21
1.16
1.21
1.25
3.02
3.30
0.96
3.39
f1 (ppm)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39.80
35.77
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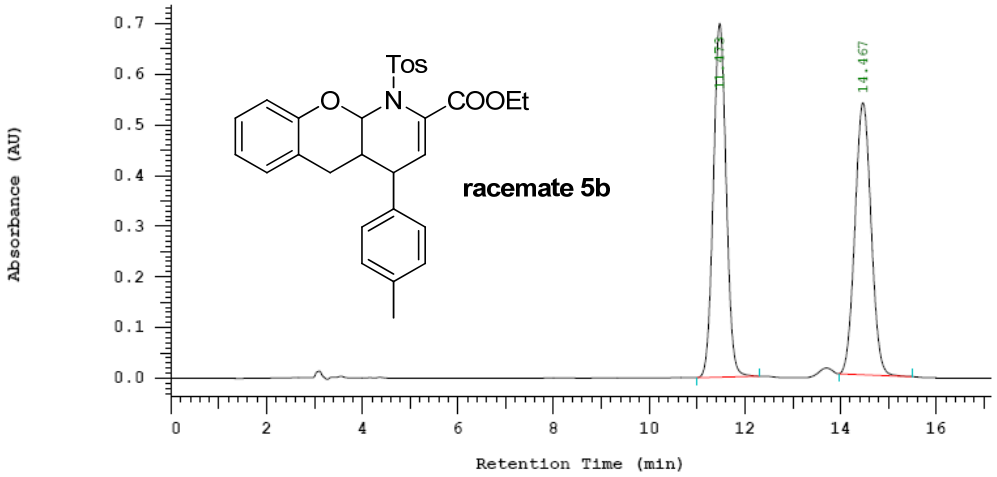
Tos
N
O
COOEt
5b
f1 (ppm)

Chrom Type: Fixed WL Chromatogram, 210 nm



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Peak Quantitation: AREA
Calculation Method: AREA%

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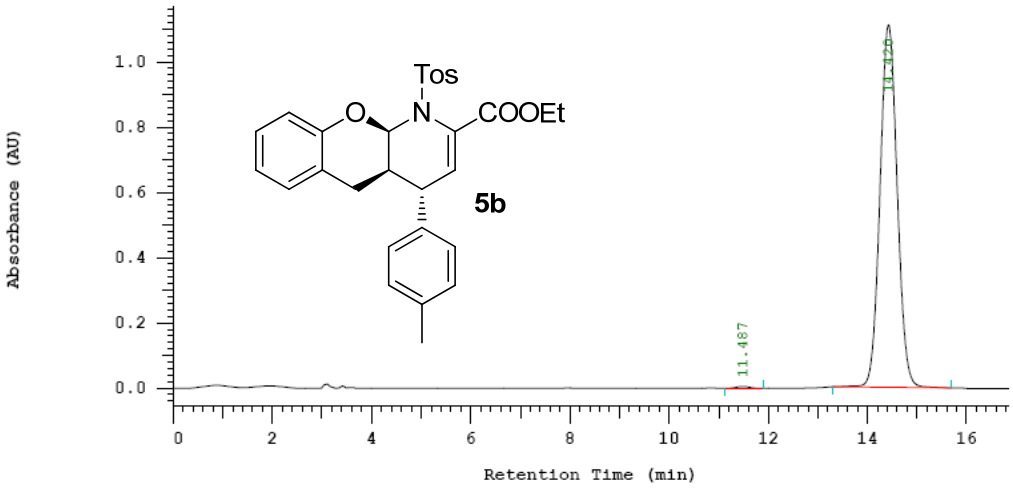

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Peak Quantitation: AREA
Calculation Method: AREA%

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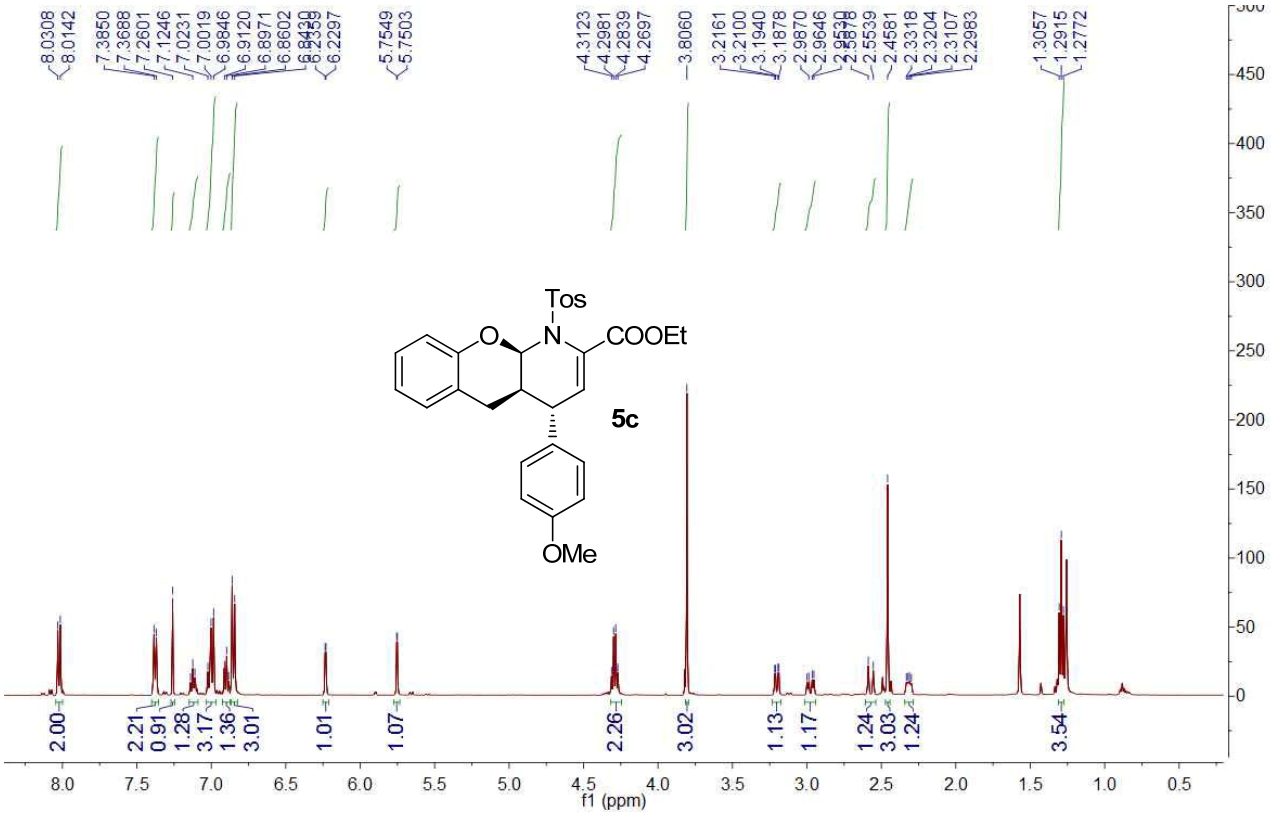
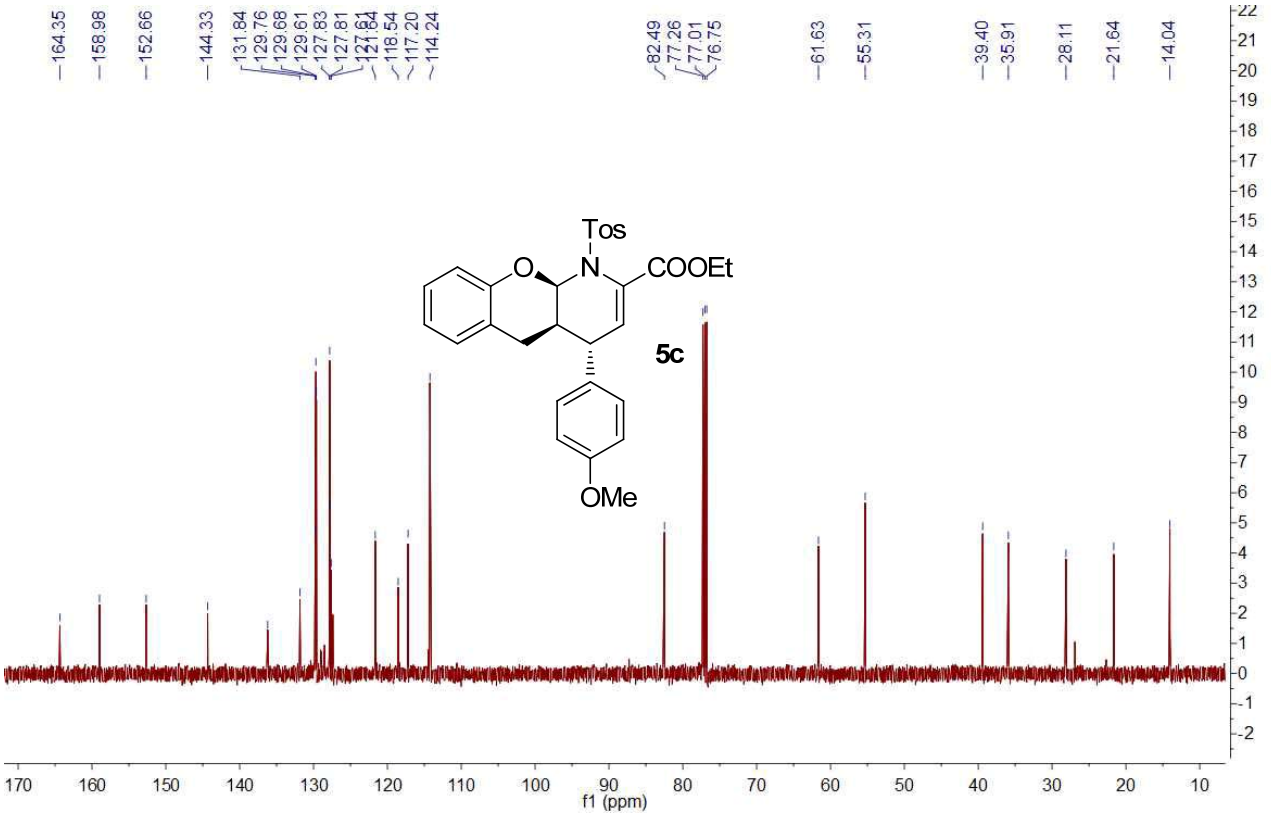
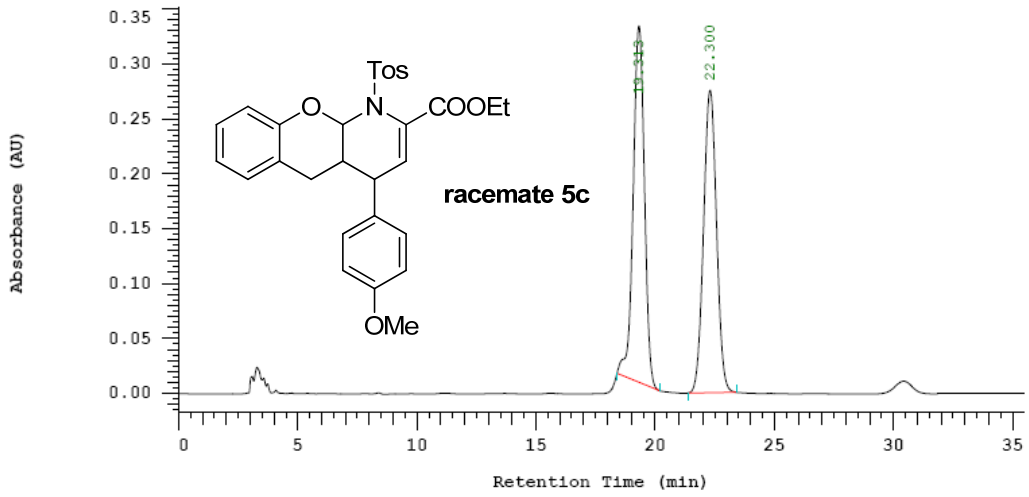
2	14.420	13349376	99.642	BB
		13397377	100.000	


8.0308
8.0142
7.3850
7.3688
7.2601
7.1246
7.0231
7.0019
6.9846
6.9120
6.8971
6.8802
6.2297
5.7549
5.7503
4.3123
4.2981
4.2839
4.2697
3.8060
3.2161
3.2100
3.1940
3.1878
2.9870
2.9646
2.5539
2.4581
2.3318
2.3204
2.3107
2.2983
1.3057
1.2915
1.2772
Tos
O
N
COOEt
5c
OMe
2.00
2.21
0.91
1.28
3.17
1.36
3.01
1.01
1.07
2.26
3.02
1.13
1.17
1.24
3.03
1.24
3.54
f1 (ppm)


164.35
158.98
152.66
144.33
131.84
129.76
129.68
129.61
127.83
127.81
118.54
117.20
114.24
82.49
77.26
77.01
76.75
61.63
55.31
39.40
35.91
28.11
21.64
14.04
Tos
O
N
COOEt
5c
OMe
f1 (ppm)

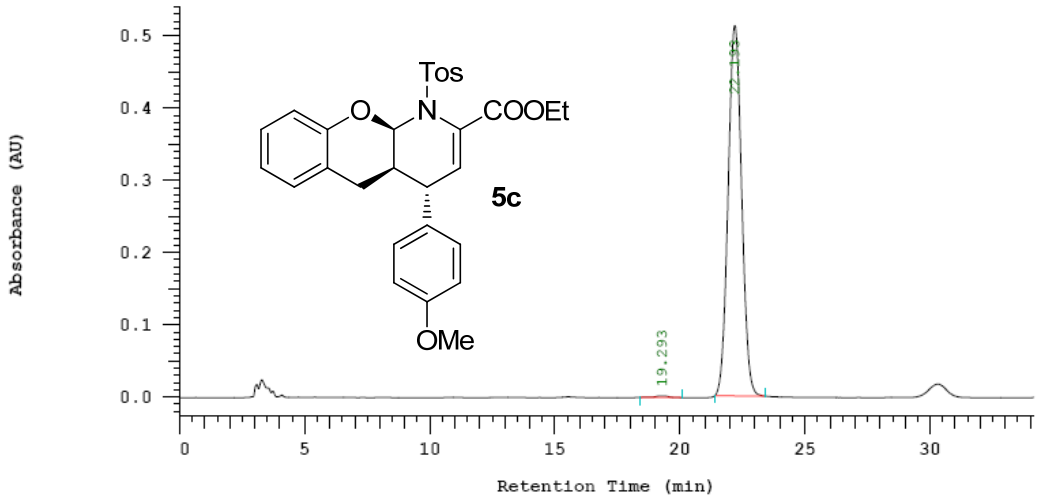
Chrom Type: Fixed WL Chromatogram, 210 nm



Chrom Type: Fixed WL Chromatogram, 210 nm
Peak Quantitation: AREA
Calculation Method: AREA%

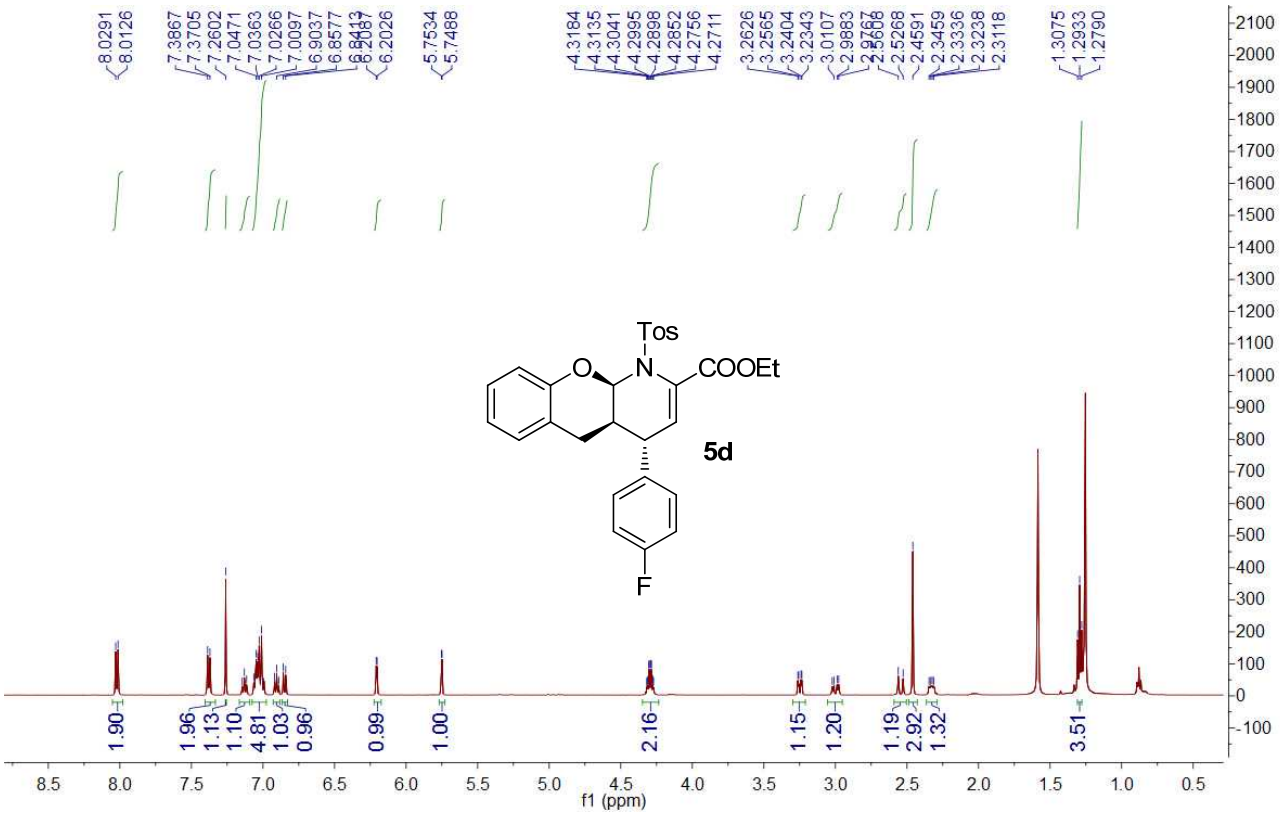
No.	RT	Area	Area %	BC
1	19.313	5281168	50.630	BB
2	22.300	5149762	49.370	BB
		10430930	100.000	

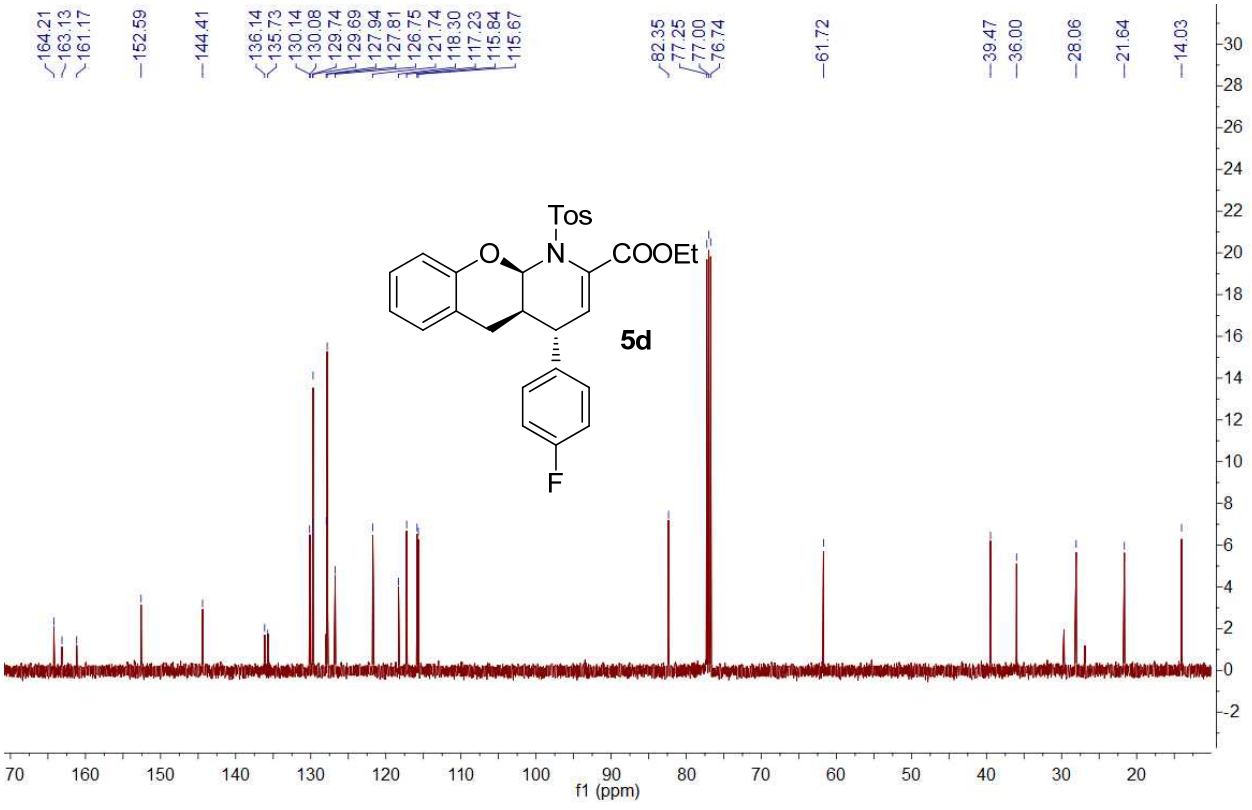
Chrom Type: Fixed WL Chromatogram, 210 nm



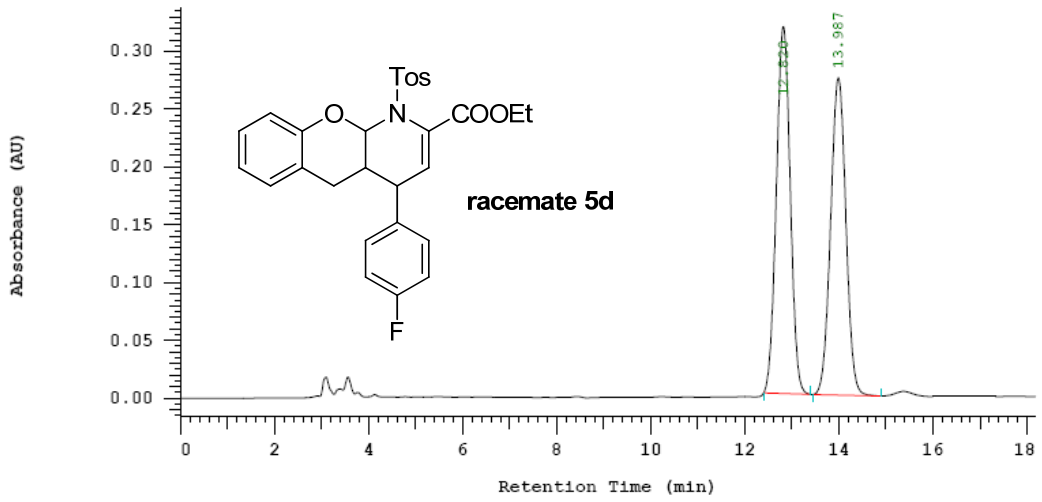
Chrom Type: Fixed WL Chromatogram, 210 nm
Peak Quantitation: AREA
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	19.293	34882	0.364	BB
2	22.193	9547754	99.636	BB
		9582636	100.000	


Tos
N
O
COOEt
5d
F
f1 (ppm)


Tos
N
O
COOEt
5d
F
f1 (ppm)

Chrom Type: Fixed WL Chromatogram, 210 nm



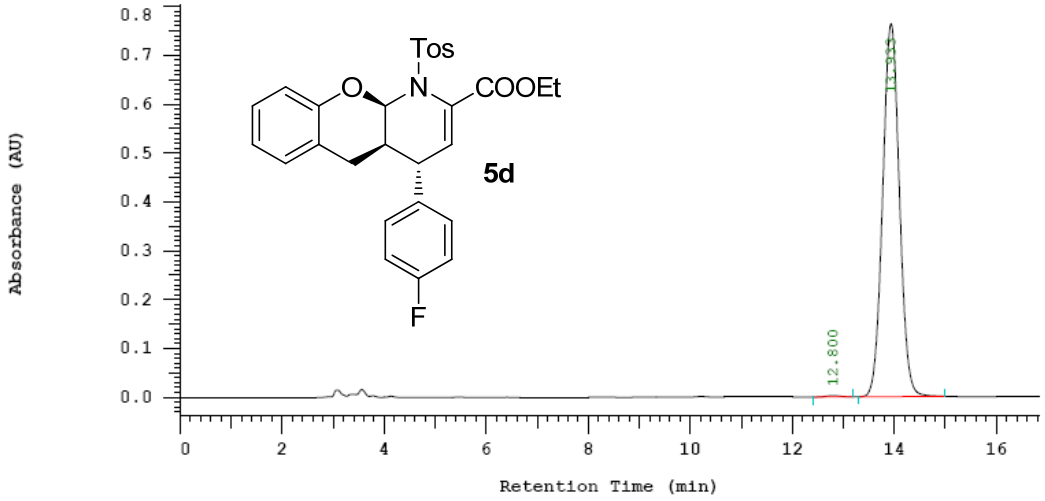
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	12.820	3250877	51.157	BB
2	13.987	3103866	48.843	BB
		6354743	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

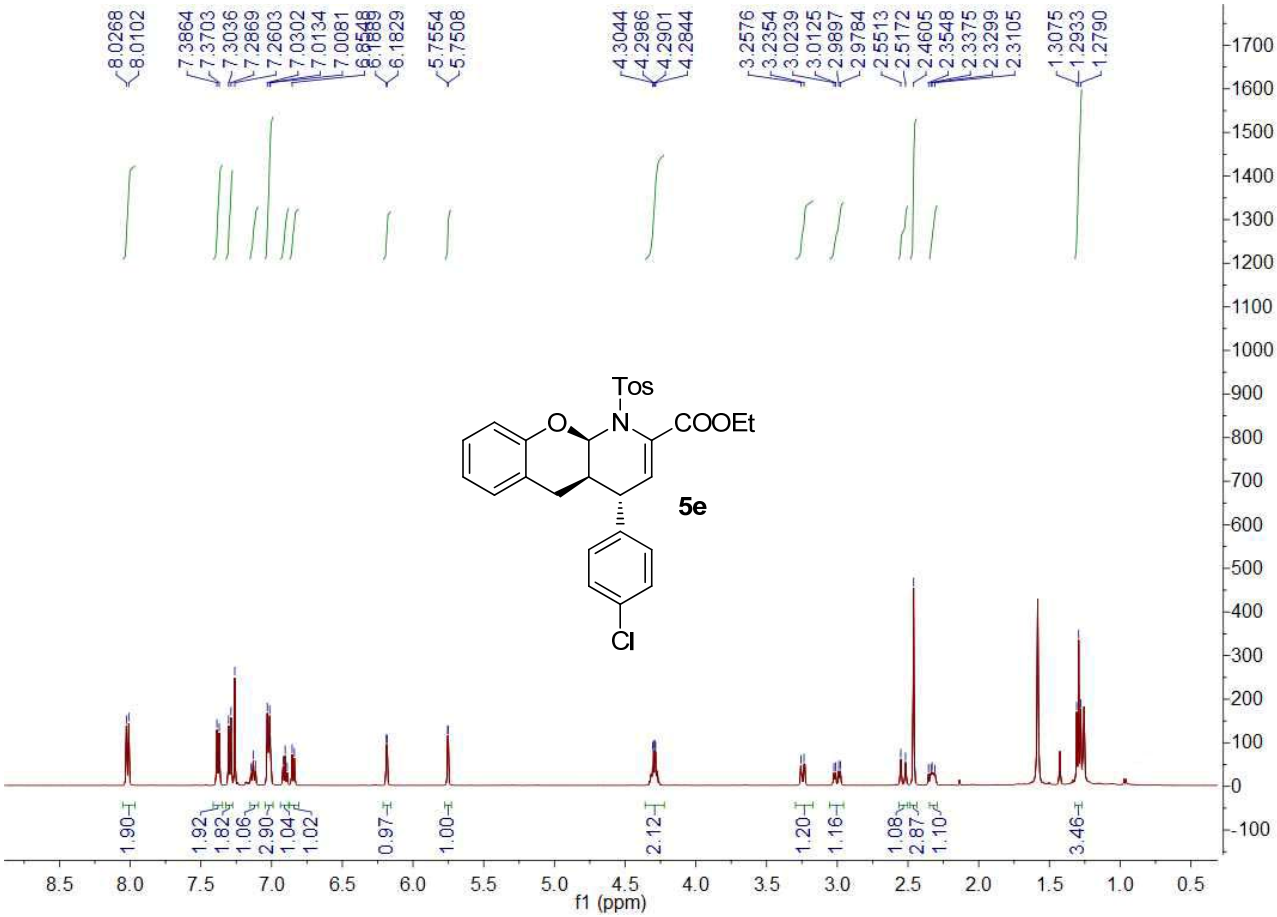


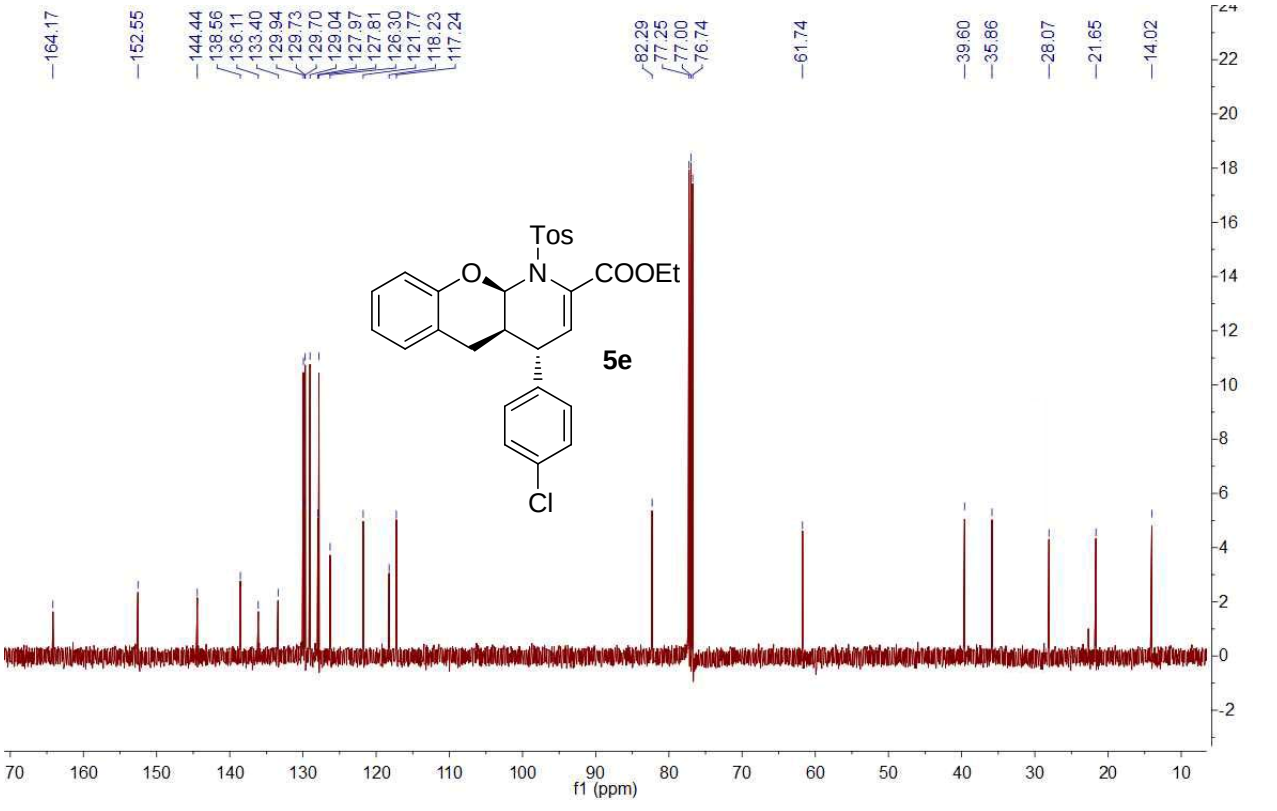
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

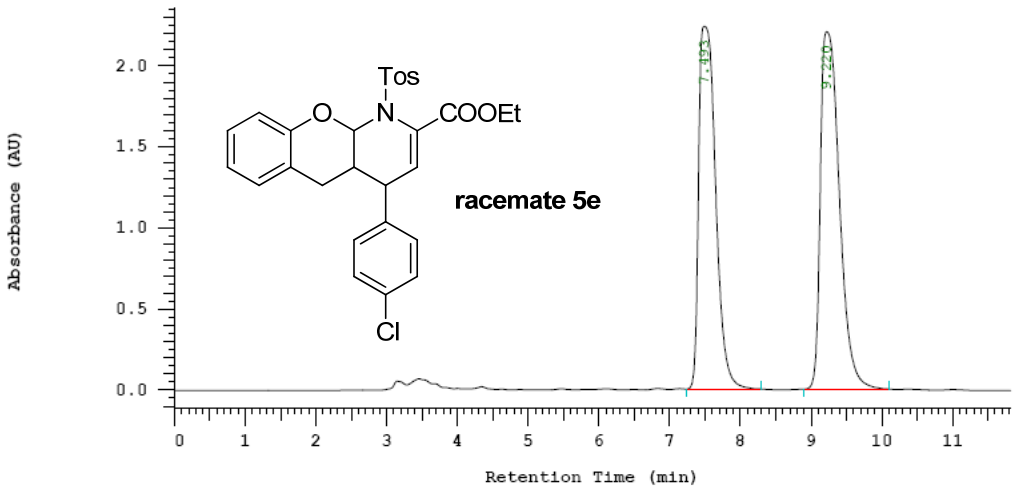
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	12.800	20833	0.239	BB
2	13.933	8706917	99.761	BB
		8727750	100.000	


8.0268
8.0102
7.3864
7.3703
7.3036
7.2869
7.2603
7.0302
7.0134
7.0081
6.1829
5.7554
5.7508
4.3044
4.2986
4.2901
4.2844
3.2576
3.2354
3.0239
3.0125
2.9897
2.9784
2.5513
2.5172
2.4605
2.3548
2.3375
2.3299
2.3105
1.3075
1.2933
1.2790
Tos
N
O
COOEt
5e
Cl
1.90
1.92
1.82
1.06
2.90
1.04
1.02
0.97
1.00
2.12
1.20
1.16
1.08
2.87
1.10
3.46
f1 (ppm)


164.17
152.55
144.44
138.56
136.11
133.40
129.94
129.73
129.70
129.04
127.97
127.81
126.30
121.77
118.23
117.24
82.29
77.25
77.00
76.74
61.74
39.60
35.86
28.07
21.65
14.02
Tos
N
O
COOEt
5e
Cl
f1 (ppm)

Chrom Type: Fixed WL Chromatogram, 210 nm



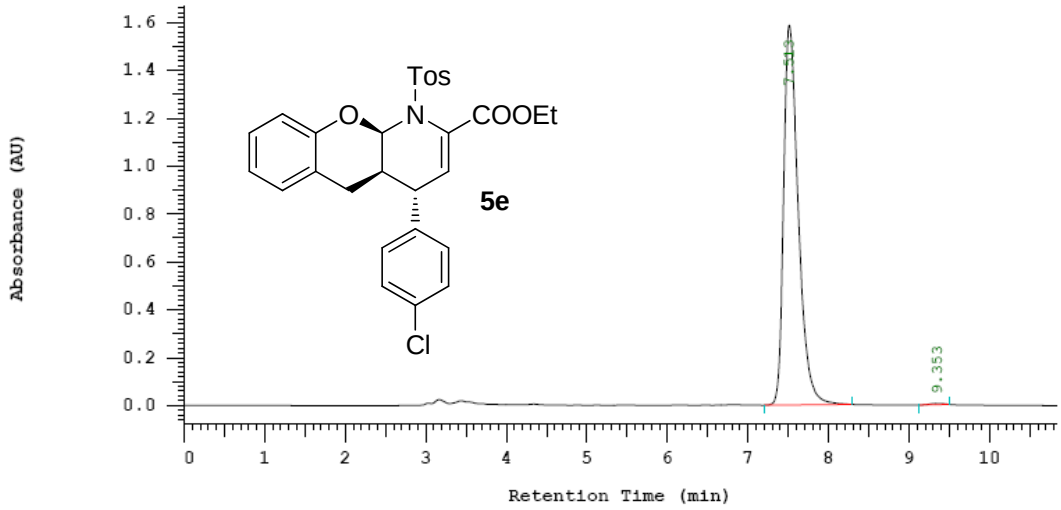
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	7.493	18605795	47.373	BB
2	9.220	20669086	52.627	BB
		39274881	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

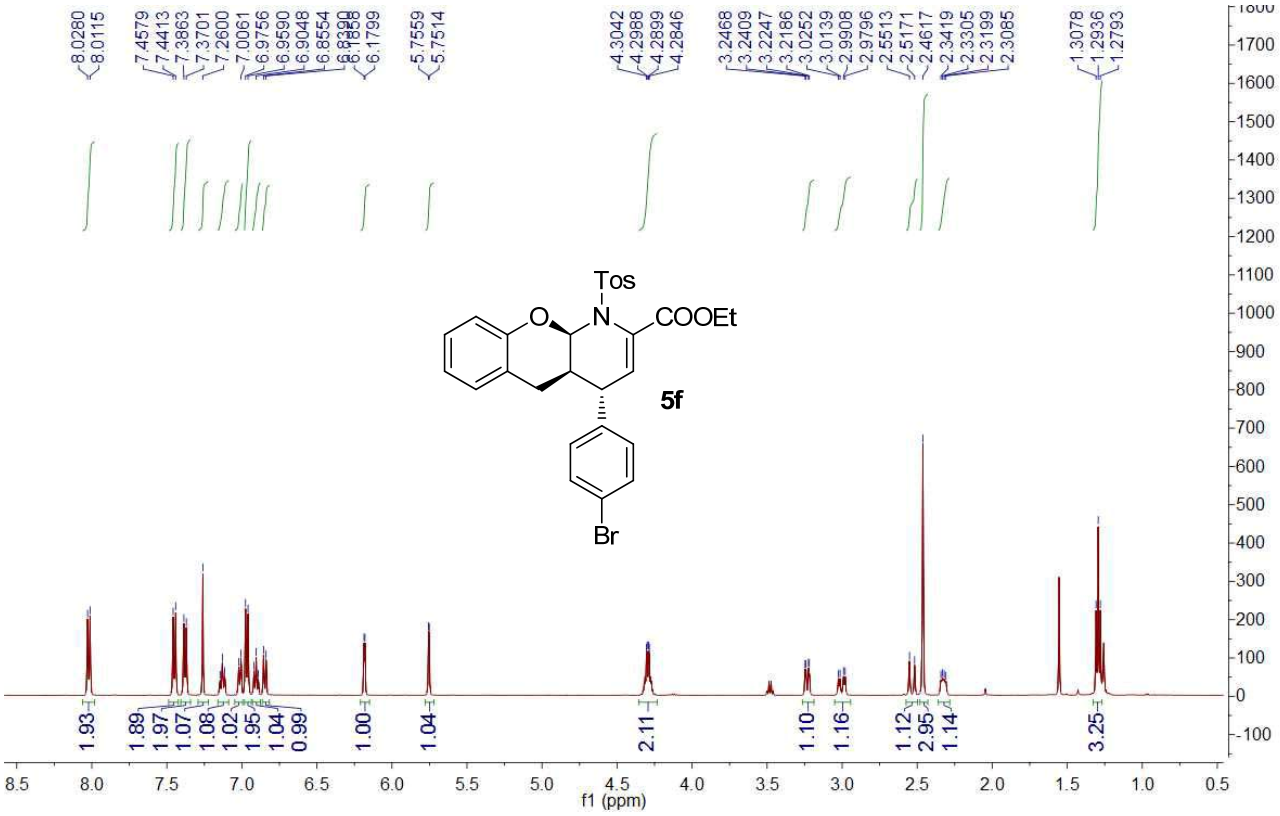


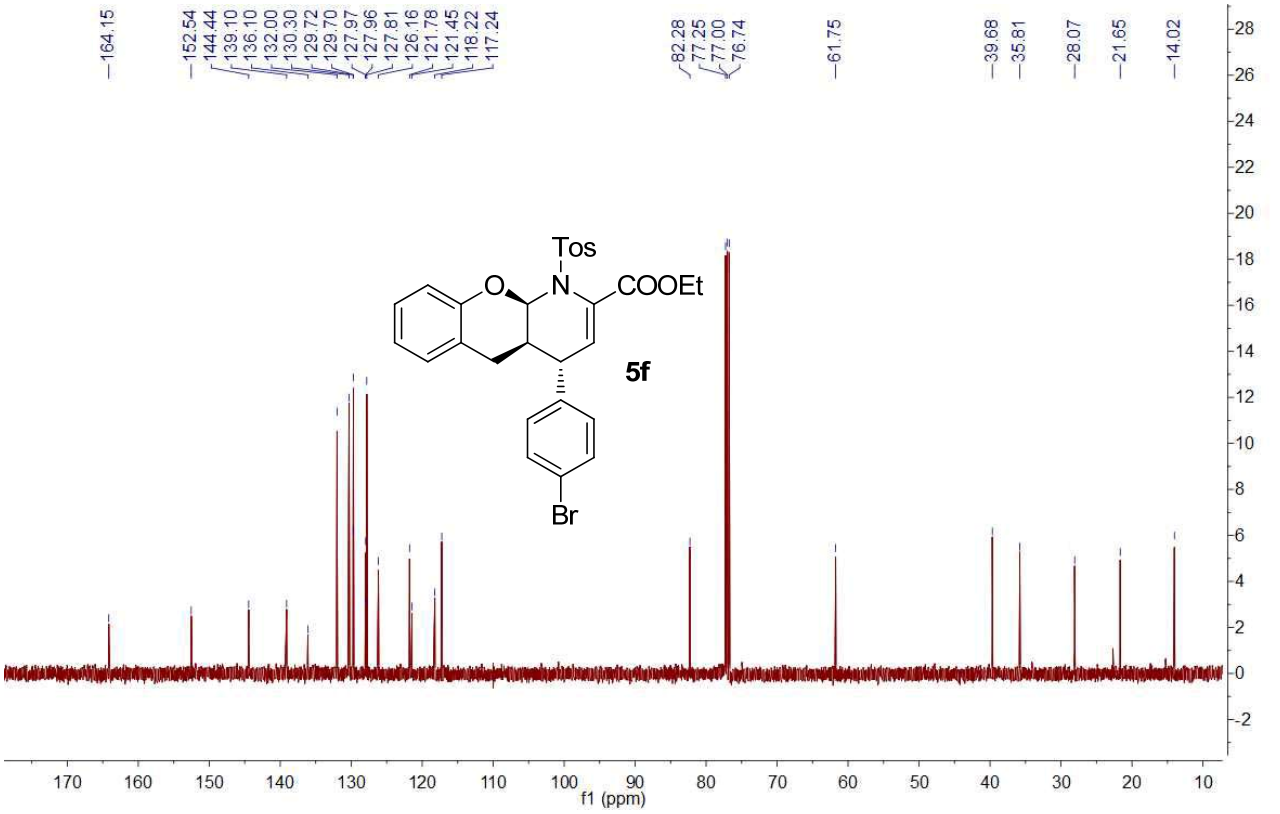
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

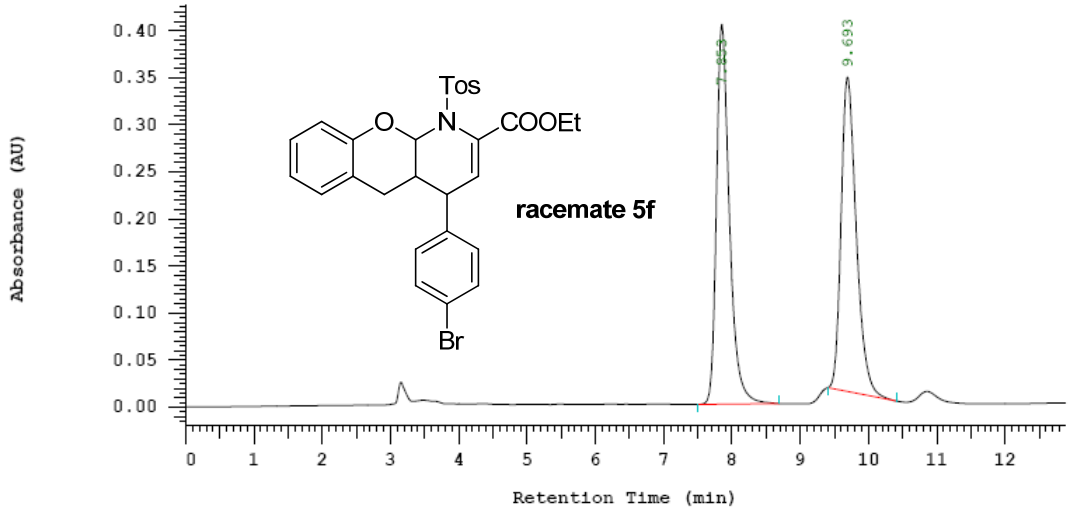
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	7.513	10005808	99.750	BB
2	9.353	25051	0.250	BB
		10030859	100.000	


8.0280
8.0115
7.4579
7.4413
7.3863
7.3701
7.2600
7.0061
6.9756
6.9590
6.9048
6.8554
6.1799
5.7559
5.7514
4.3042
4.2988
4.2899
4.2846
3.2468
3.2409
3.2247
3.2186
3.0252
3.0139
2.9908
2.9796
2.5513
2.5171
2.4617
2.3419
2.3305
2.3199
2.3085
1.3078
1.2936
1.2793
Tos
O
N
COOEt
5f
Br
1.93
1.89
1.97
1.07
1.08
1.02
1.95
1.04
0.99
1.00
1.04
2.11
1.10
1.16
1.12
2.95
1.14
3.25
f1 (ppm)


164.15
152.54
144.44
139.10
136.10
132.00
130.30
129.72
129.70
127.97
127.96
127.81
126.16
121.78
121.45
118.22
117.24
82.28
77.25
77.00
76.74
61.75
39.68
35.81
28.07
21.65
14.02
Tos
O
N
COOEt
5f
Br
f1 (ppm)

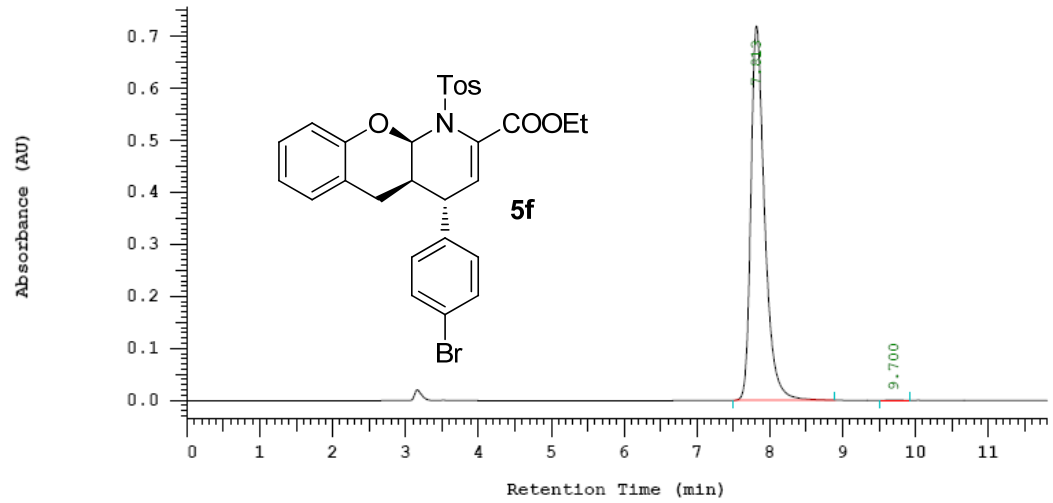
Chrom Type: Fixed WL Chromatogram, 210 nm



Chrom Type: Fixed WL Chromatogram, 210 nm
Peak Quantitation: AREA
Calculation Method: AREA%

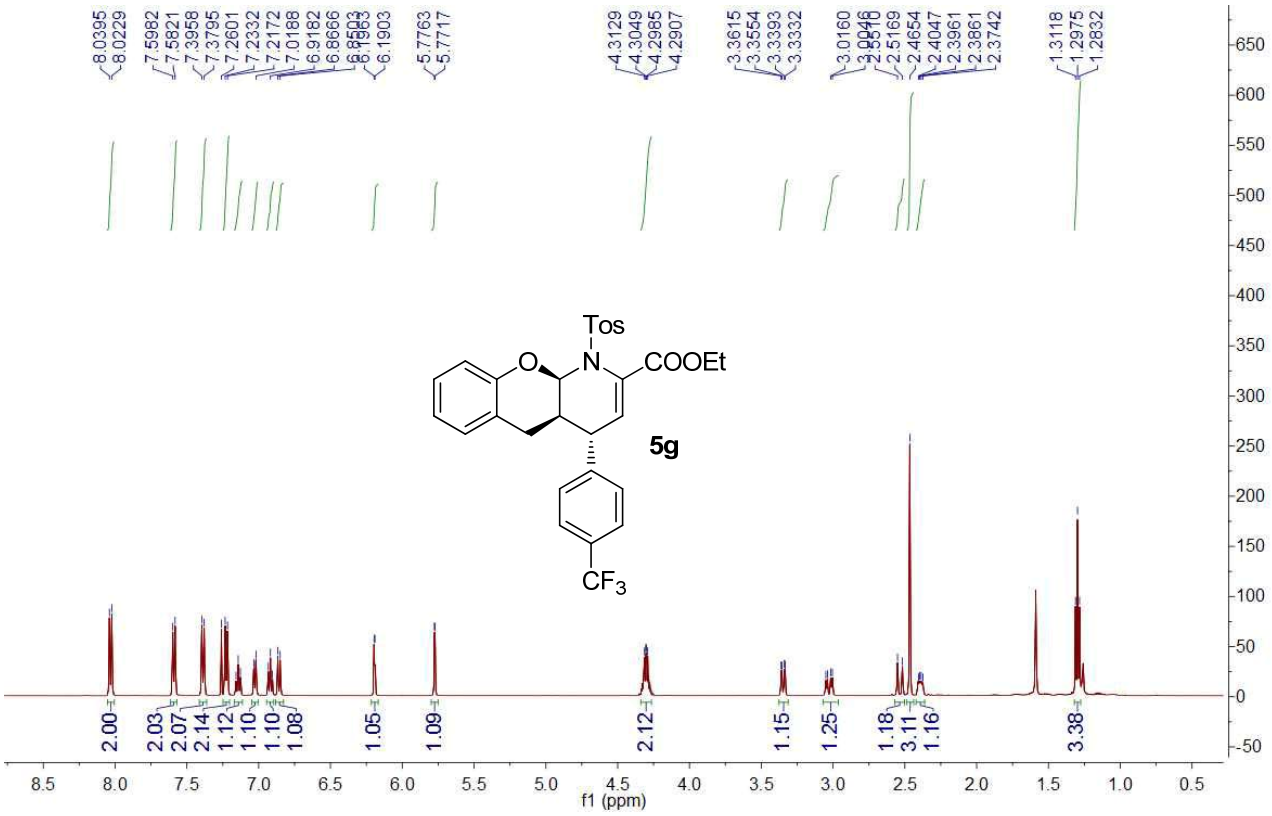
No.	RT	Area	Area %	BC
1	7.853	2677312	50.984	BB
2	9.693	2573936	49.016	BB
		5251248	100.000	

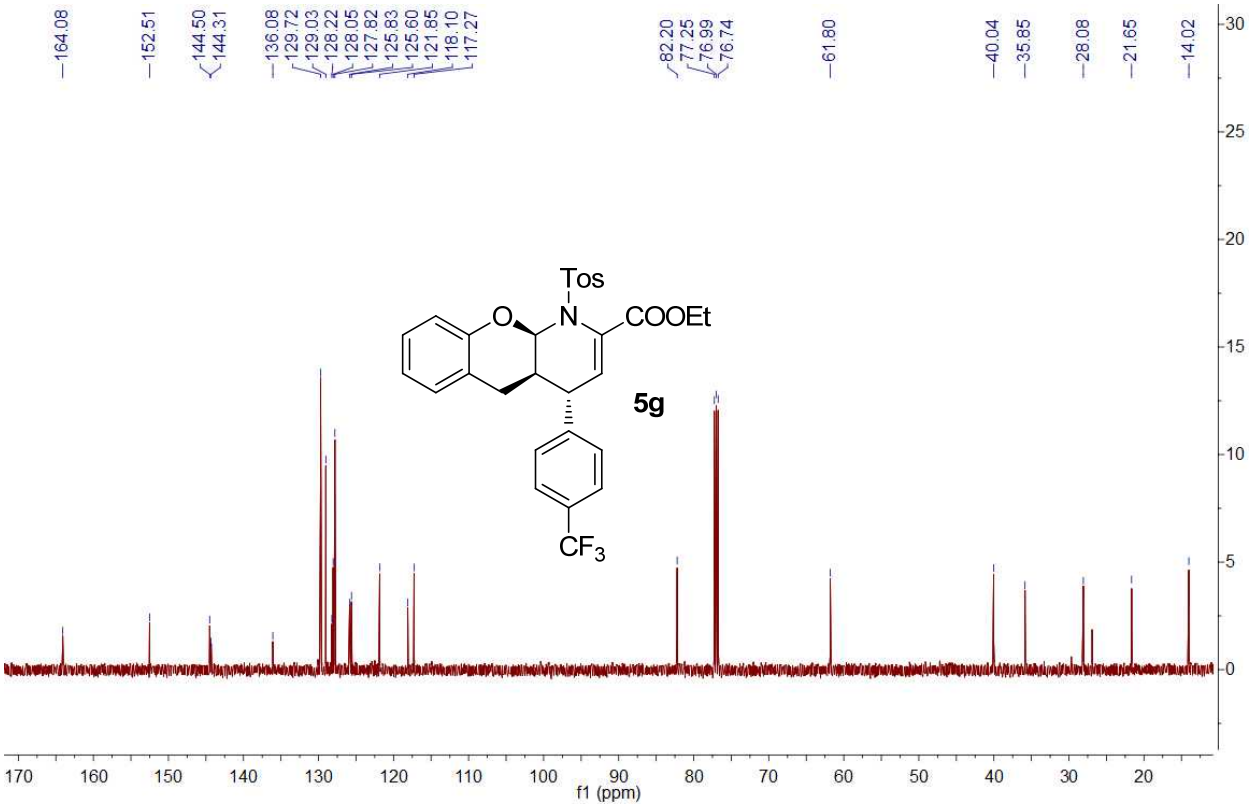
Chrom Type: Fixed WL Chromatogram, 210 nm



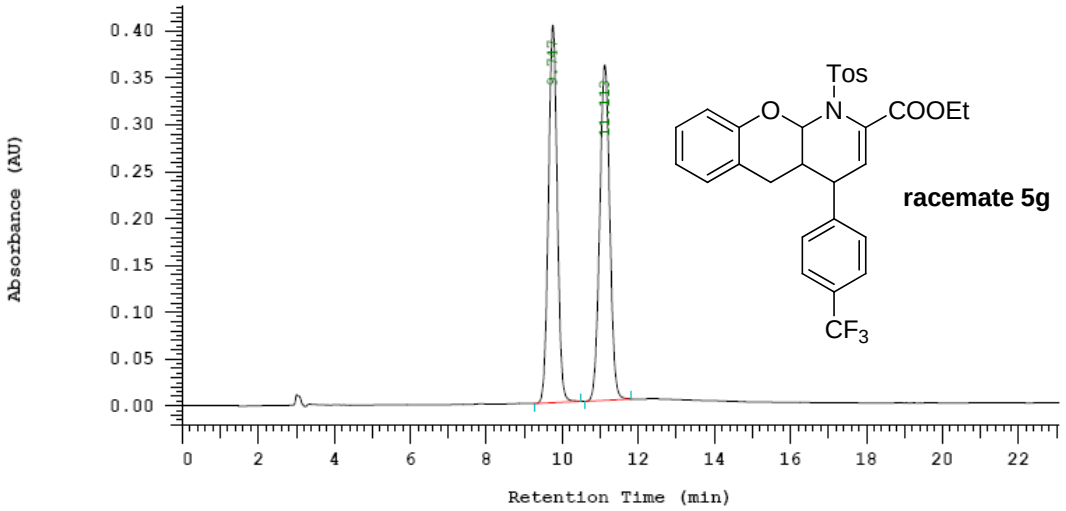
Chrom Type: Fixed WL Chromatogram, 210 nm
Peak Quantitation: AREA
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	7.813	4761893	99.926	BB
2	9.700	3541	0.074	BB
		4765434	100.000	


8.0395
8.0229
7.5982
7.5821
7.3958
7.3795
7.2601
7.2332
7.2172
7.0188
6.9182
6.8666
6.1903
5.7763
5.7717
4.3129
4.3049
4.2985
4.2907
3.3615
3.3554
3.3393
3.3332
3.0160
2.5169
2.4654
2.4047
2.3961
2.3861
2.3742
1.3118
1.2975
1.2832
Tos
N
O
COOEt
5g
CF3
2.00
2.03
2.07
2.14
1.12
1.10
1.10
1.08
1.05
1.09
2.12
1.15
1.25
1.18
3.11
1.16
3.38
f1 (ppm)


164.08
152.51
144.50
144.31
136.08
129.72
129.03
128.22
128.05
127.82
125.83
125.60
121.85
118.10
117.27
82.20
77.25
76.99
76.74
61.80
40.04
35.85
28.08
21.65
14.02
Tos
N
O
COOEt
5g
CF3
f1 (ppm)

Chrom Type: Fixed WL Chromatogram, 210 nm



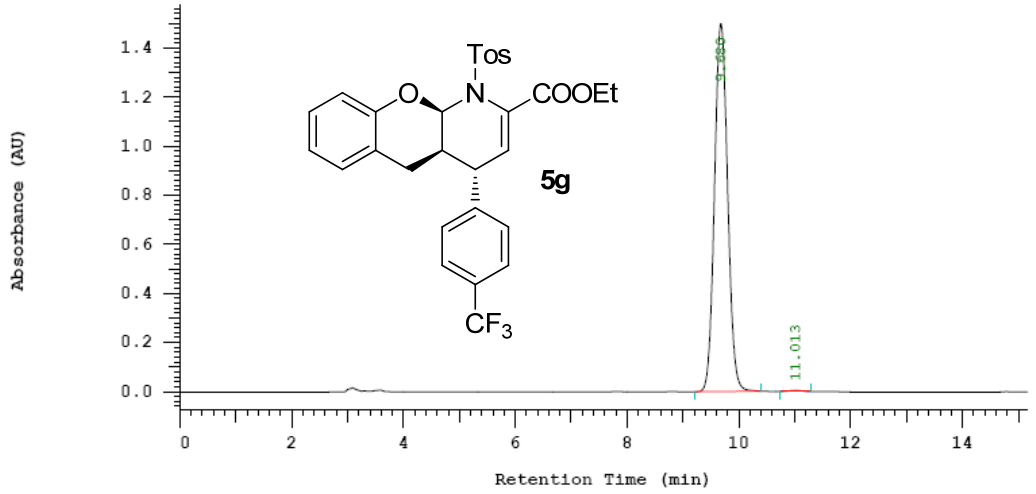
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	9.747	3219177	49.529	BB
2	11.113	3280415	50.471	BB
		6499592	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

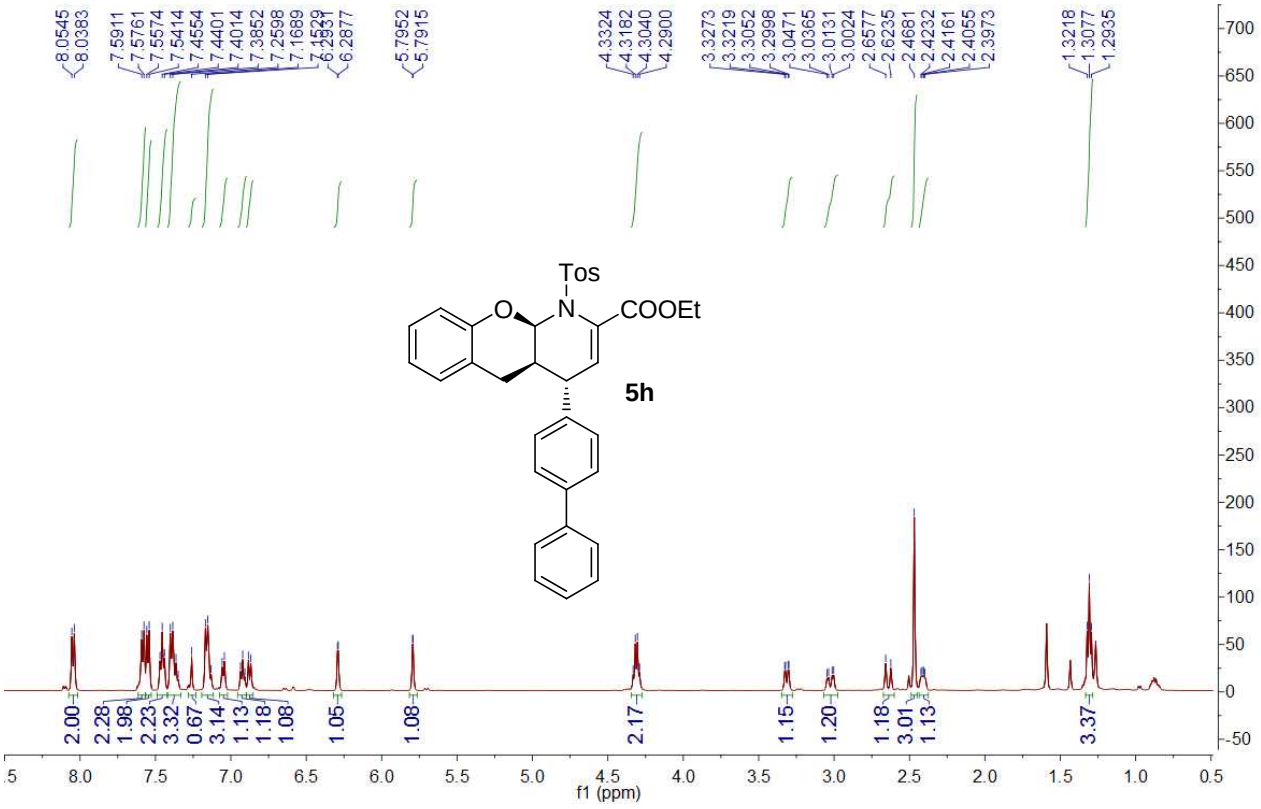


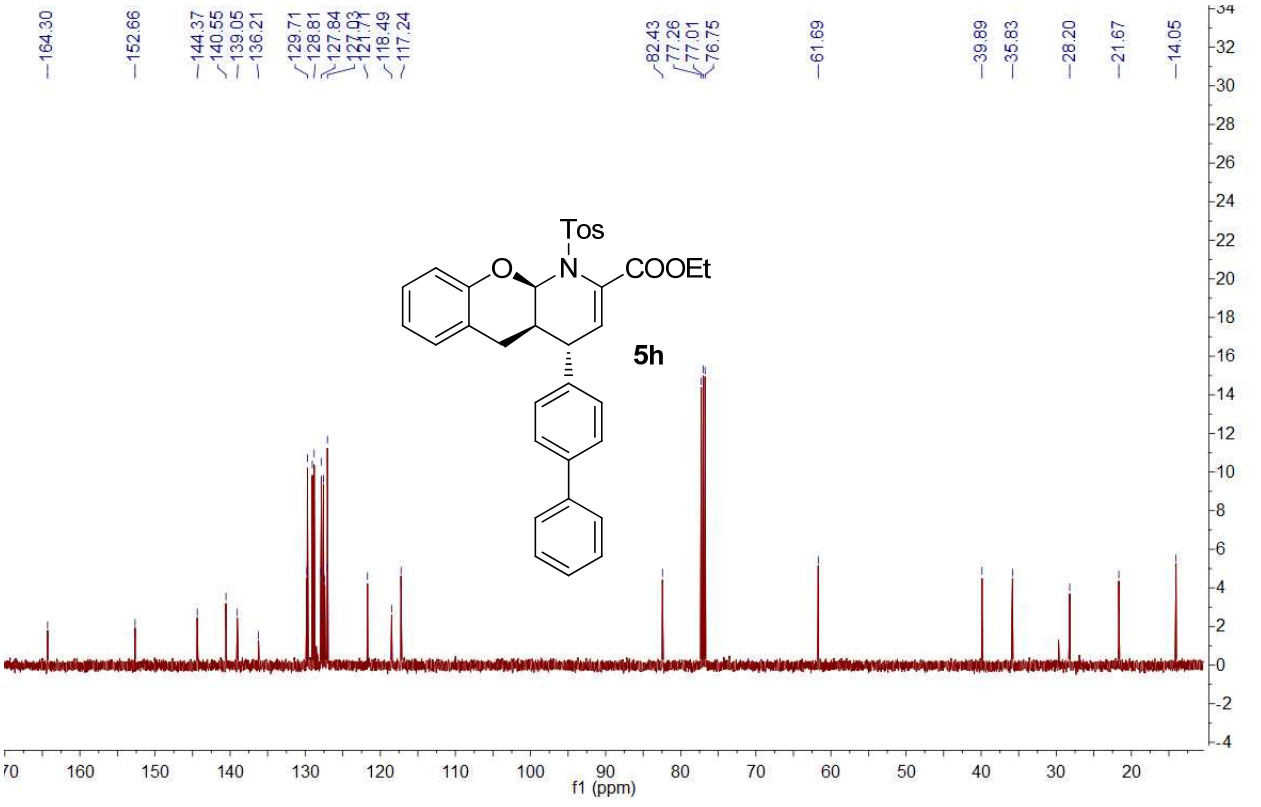
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

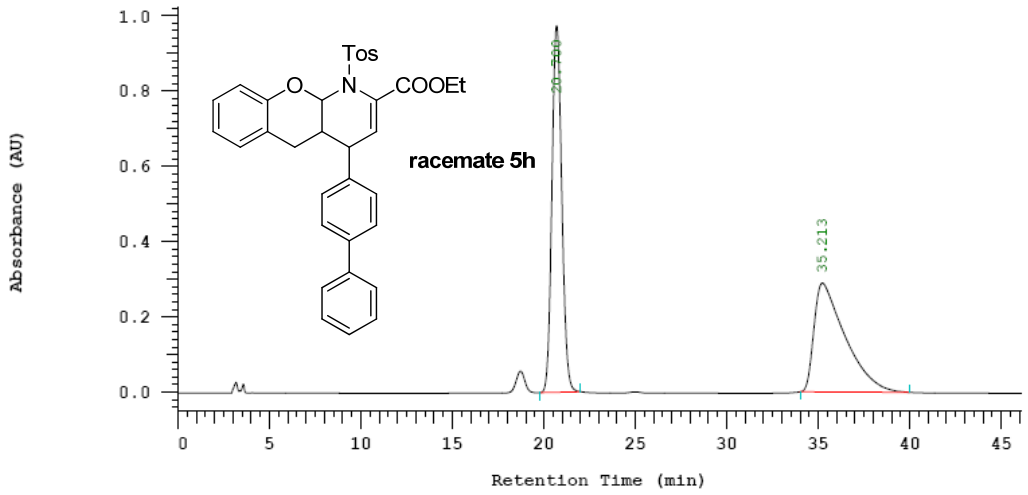
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	9.680	12183055	99.737	BB
2	11.013	32095	0.263	BB
		12215150	100.000	


8.0545
8.0383
7.5911
7.5761
7.5574
7.5414
7.4554
7.4401
7.4014
7.3852
7.2598
7.1689
6.2877
5.7952
5.7915
4.3324
4.3182
4.3040
4.2900
3.3273
3.3219
3.3052
3.2998
3.0471
3.0365
3.0131
3.0024
2.6577
2.6235
2.4681
2.4232
2.4161
2.4055
2.3973
1.3218
1.3077
1.2935
Tos
N
O
COOEt
5h
2.00
2.28
1.98
2.23
3.32
0.67
3.14
1.13
1.18
1.08
1.05
1.08
2.17
1.15
1.20
1.18
3.01
1.13
3.37
f1 (ppm)


164.30
152.66
144.37
140.55
139.05
136.21
129.71
128.81
127.84
121.71
118.49
117.24
82.43
77.26
77.01
76.75
61.69
39.89
35.83
28.20
21.67
14.05
Tos
N
O
COOEt
5h
f1 (ppm)

Chrom Type: Fixed WL Chromatogram, 210 nm



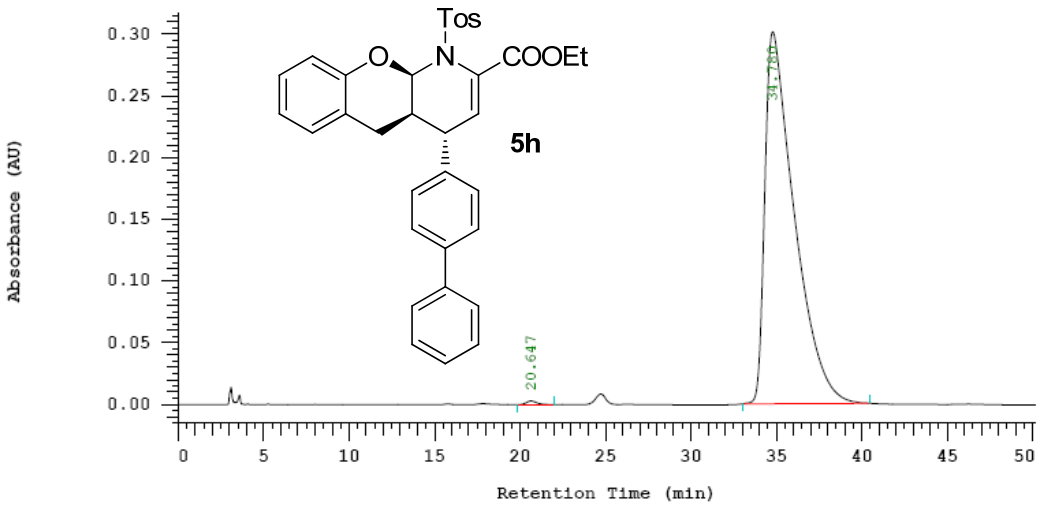
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	20.700	17961487	51.331	BB
2	35.213	17029859	48.669	BB
		34991346	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

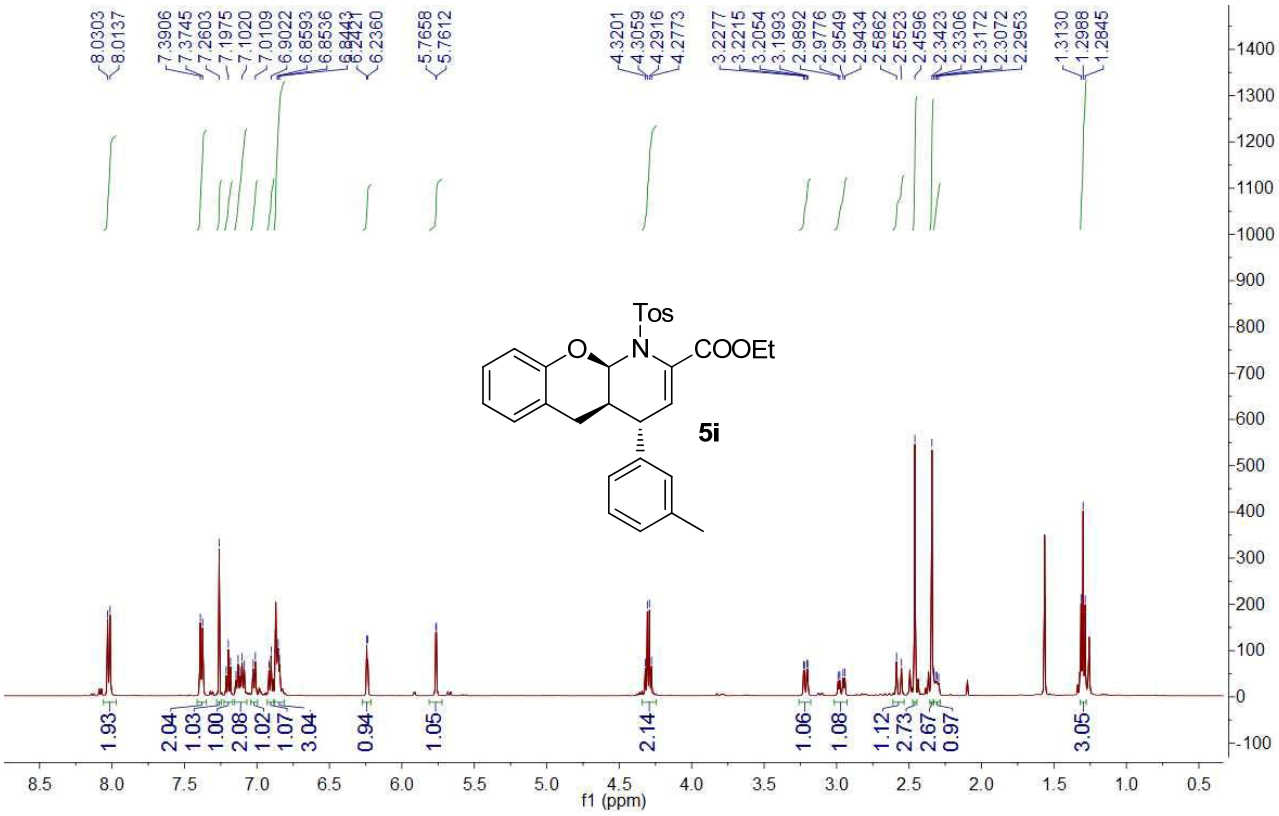


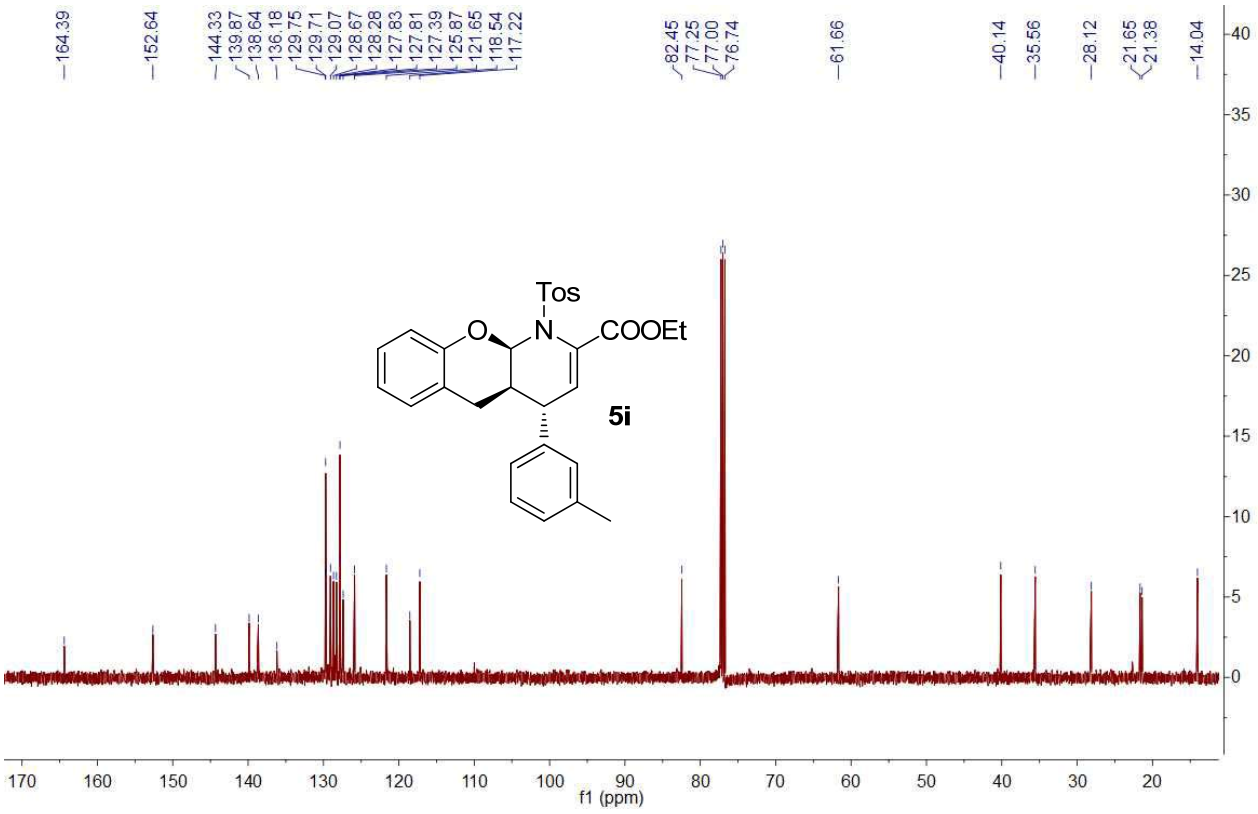
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

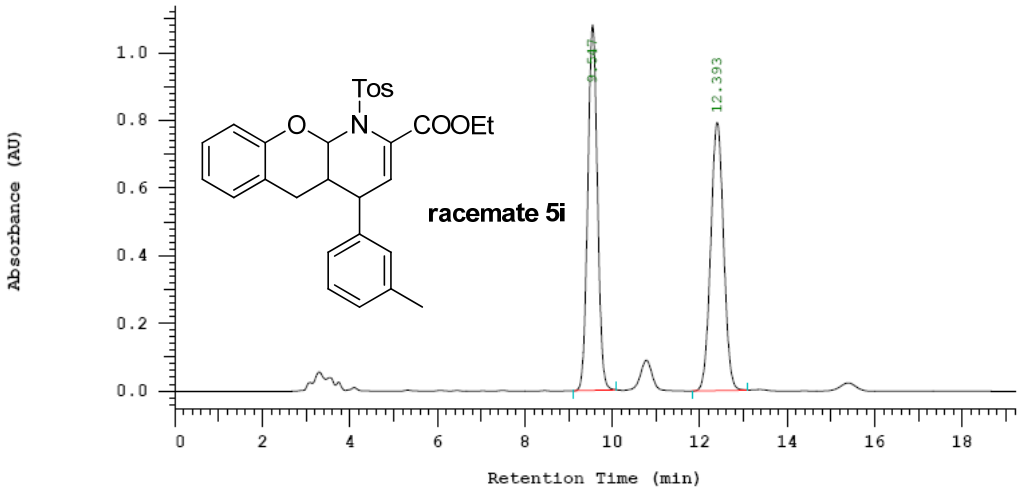
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	20.647	61132	0.340	BB
2	34.780	17929775	99.660	BB
		17990907	100.000	


8.0303
8.0137
7.3906
7.3745
7.2603
7.1975
7.1020
7.0109
6.9022
6.8593
6.8536
6.2360
5.7658
5.7612
4.3201
4.3059
4.2916
4.2773
3.2277
3.2215
3.2054
3.1993
2.9892
2.9776
2.9549
2.9434
2.5862
2.5523
2.4596
2.3423
2.3306
2.3172
2.3072
2.2953
1.3130
1.2988
1.2845
Tos
O
N
COOEt
5i
1.93
2.04
1.03
1.00
2.08
1.02
1.07
3.04
0.94
1.05
2.14
1.06
1.08
1.12
2.73
2.67
0.97
3.05
f1 (ppm)


164.39
152.64
144.33
139.87
138.64
136.18
129.75
129.71
129.07
128.67
128.28
127.83
127.81
127.39
125.87
121.65
118.54
117.22
82.45
77.25
77.00
76.74
61.66
40.14
35.56
28.12
21.65
21.38
14.04
Tos
O
N
COOEt
5i
f1 (ppm)

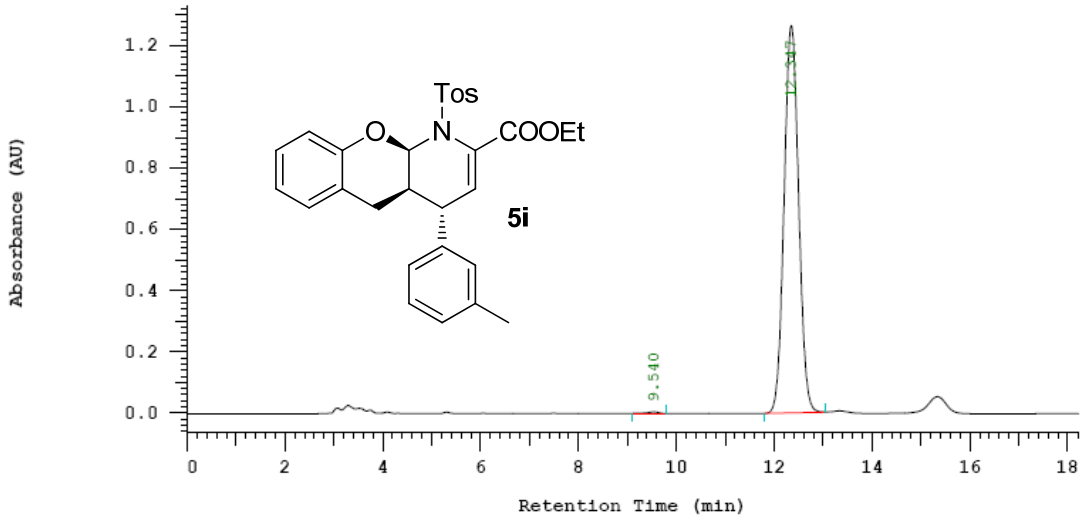
Chrom Type: Fixed WL Chromatogram, 210 nm



Chrom Type: Fixed WL Chromatogram, 210 nm
Peak Quantitation: AREA
Calculation Method: AREA%

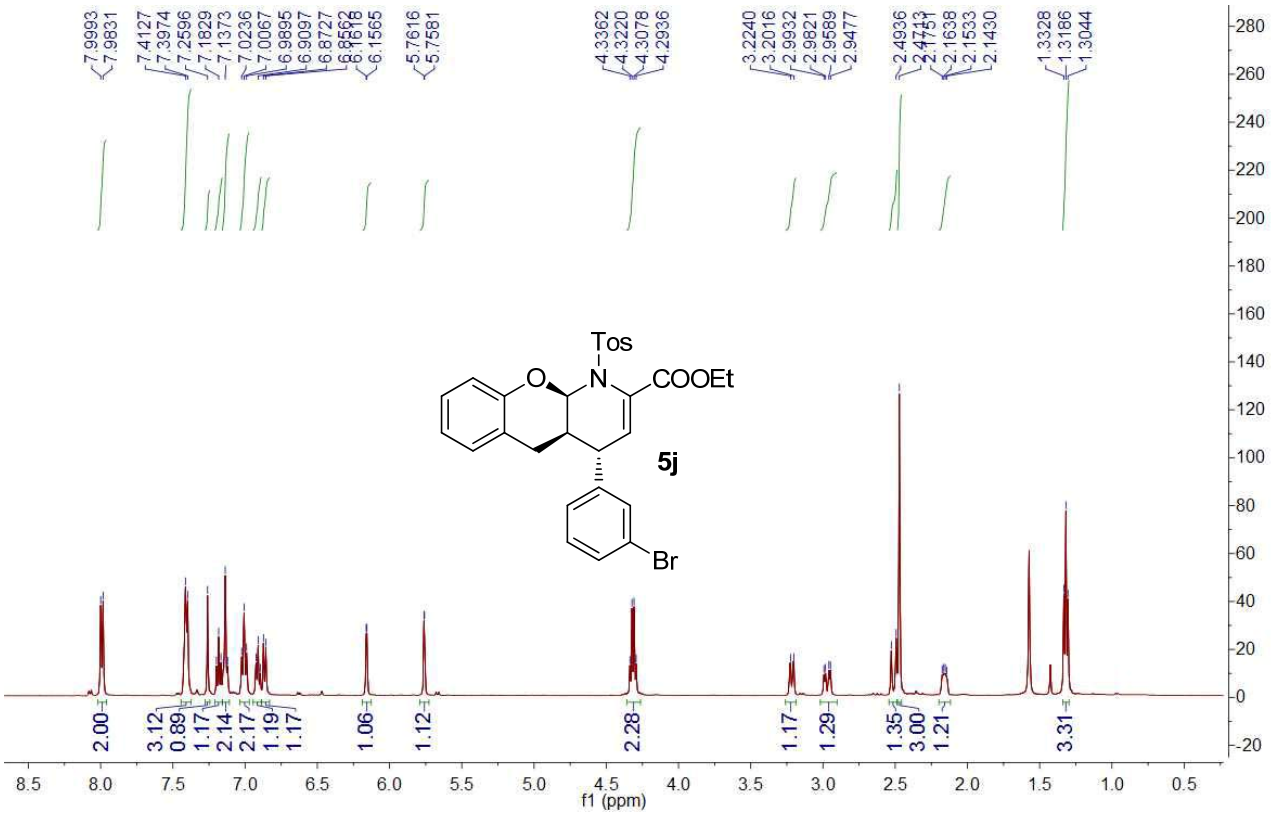
No.	RT	Area	Area %	BC
1	9.547	8377948	50.848	BB
2	12.393	8098570	49.152	BB
		16476518	100.000	

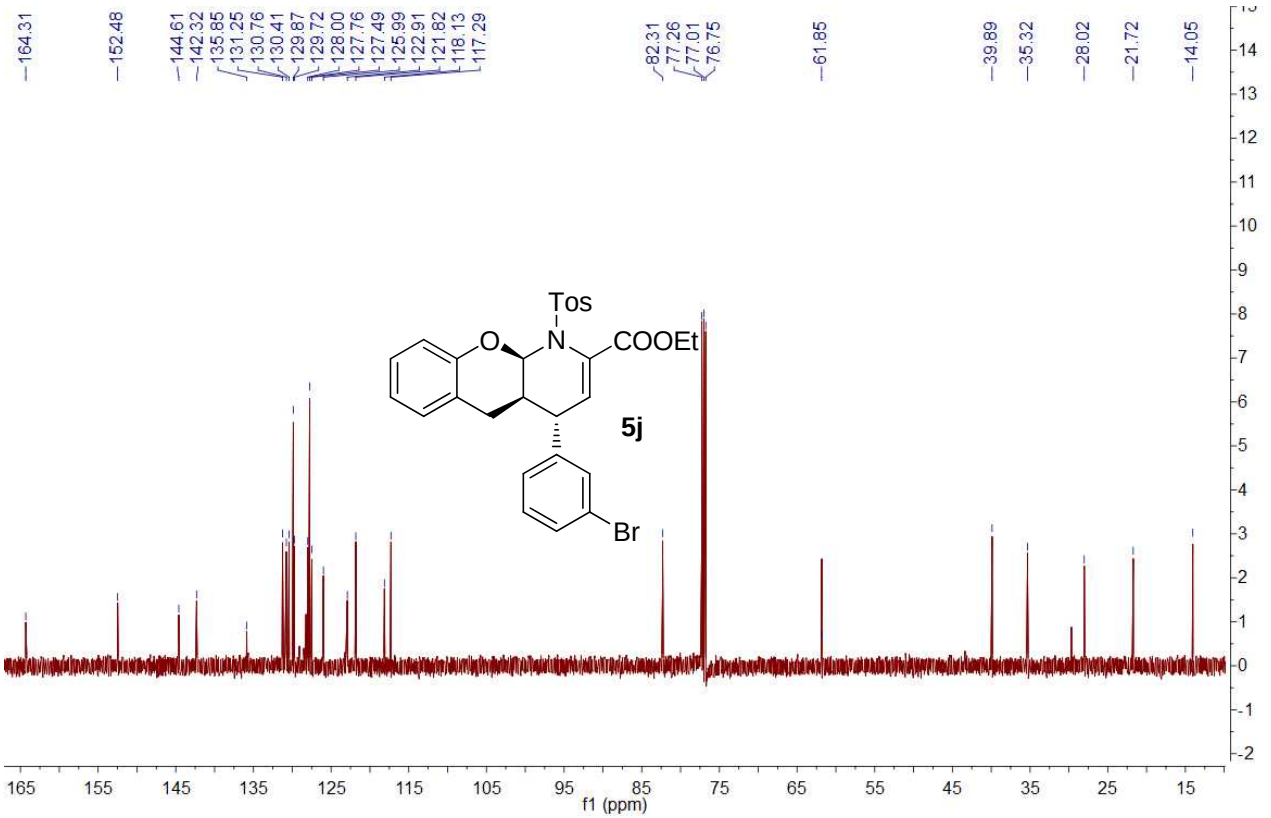
Chrom Type: Fixed WL Chromatogram, 210 nm



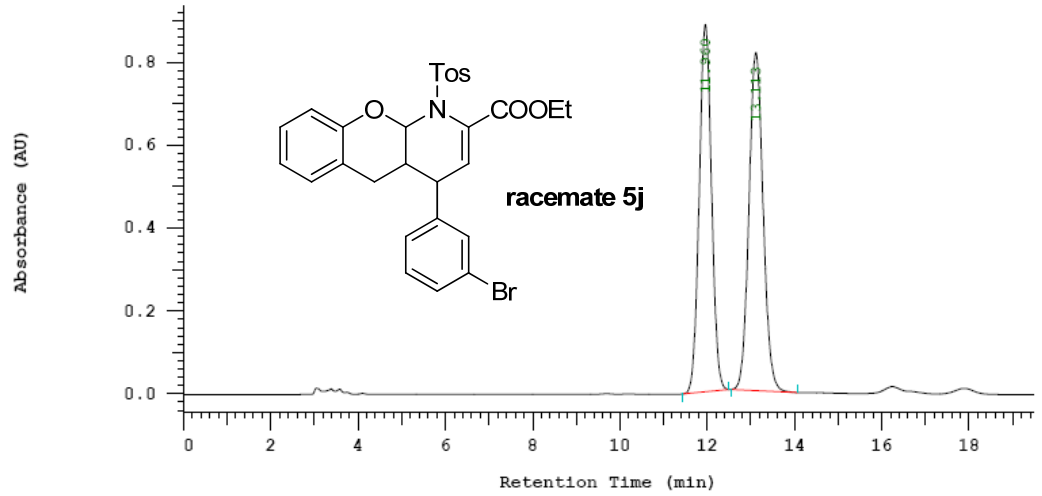
Chrom Type: Fixed WL Chromatogram, 210 nm
Peak Quantitation: AREA
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	9.540	39147	0.300	BB
2	12.347	13021592	99.700	BB
		13060739	100.000	


Tos
O
N
COOEt
5j
Br
f1 (ppm)


Tos
O
N
COOEt
5j
Br
f1 (ppm)

Chrom Type: Fixed WL Chromatogram, 210 nm



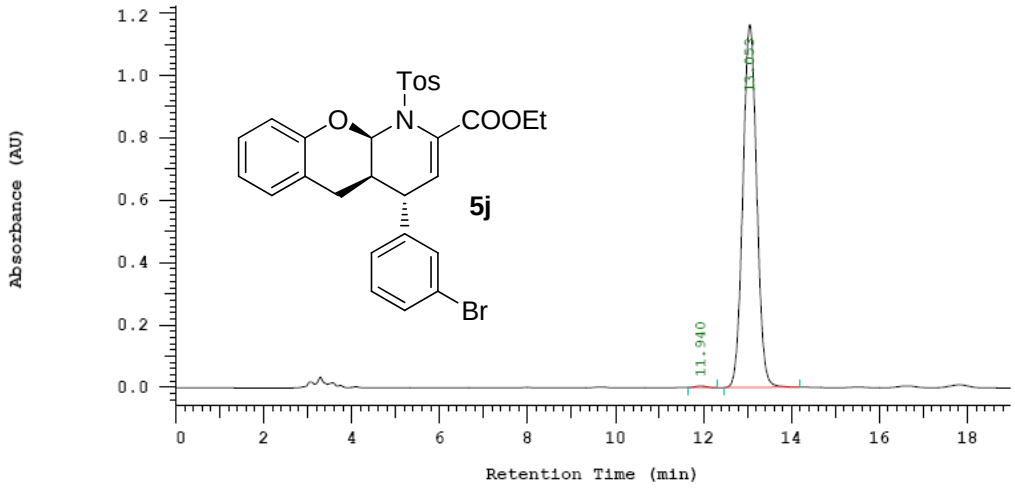
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	11.960	8695428	48.542	BB
2	13.113	9217805	51.458	BB
		17913233	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

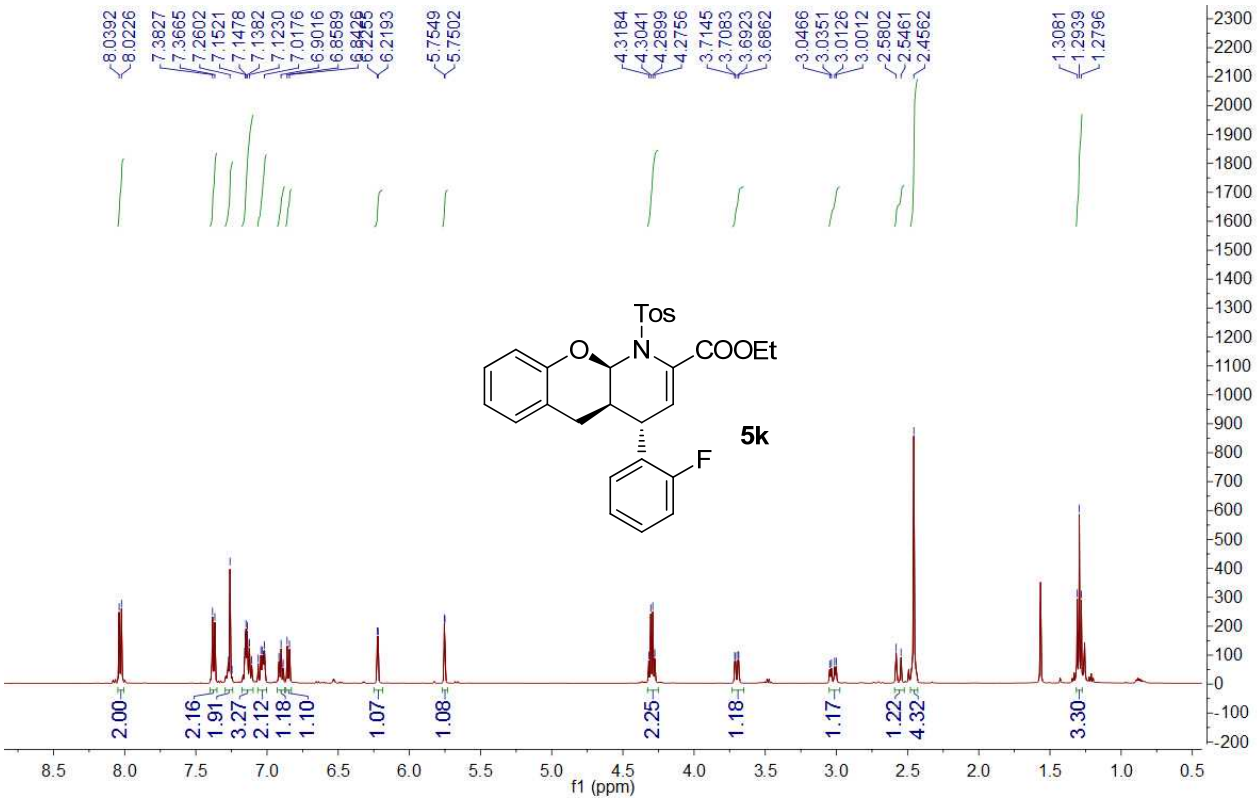


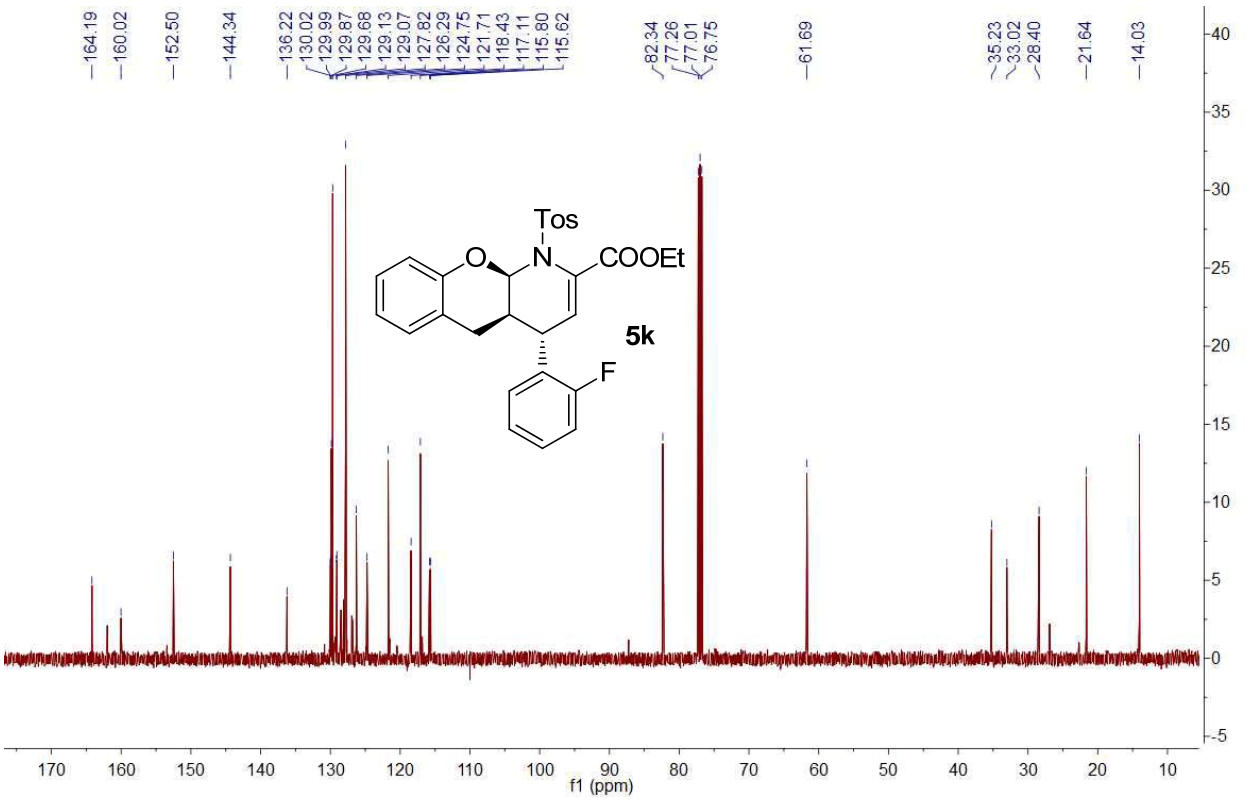
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

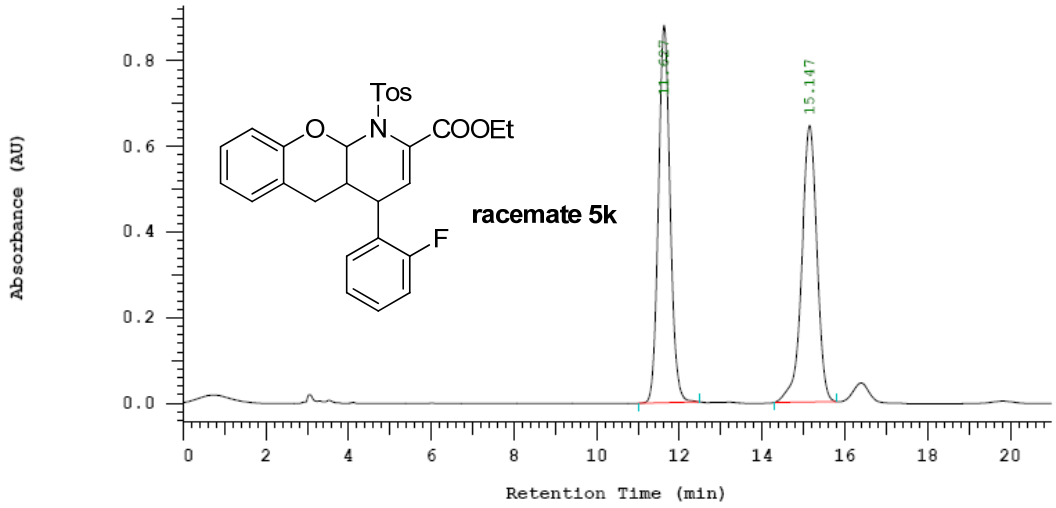
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	11.940	37103	0.291	BB
2	13.053	12703985	99.709	BB
		12741088	100.000	


8.0392
8.0226
7.3827
7.3665
7.2602
7.1521
7.1478
7.1382
7.1230
7.0176
6.9016
6.8589
6.2193
5.7549
5.7502
4.3184
4.3041
4.2899
4.2756
3.7145
3.7083
3.6923
3.6862
3.0466
3.0351
3.0126
3.0012
2.5802
2.5461
2.4562
1.3081
1.2939
1.2796
Tos
O
N
COOEt
5k
F
2.00
2.16
1.91
3.27
2.12
1.18
1.10
1.07
1.08
2.25
1.18
1.17
1.22
4.32
3.30
f1 (ppm)


164.19
160.02
152.50
144.34
136.22
130.02
129.99
129.87
129.68
129.13
129.07
127.82
126.29
124.75
121.71
118.43
117.11
115.80
115.62
82.34
77.26
77.01
76.75
61.69
35.23
33.02
28.40
21.64
14.03
Tos
O
N
COOEt
5k
F
f1 (ppm)

Chrom Type: Fixed WL Chromatogram, 210 nm



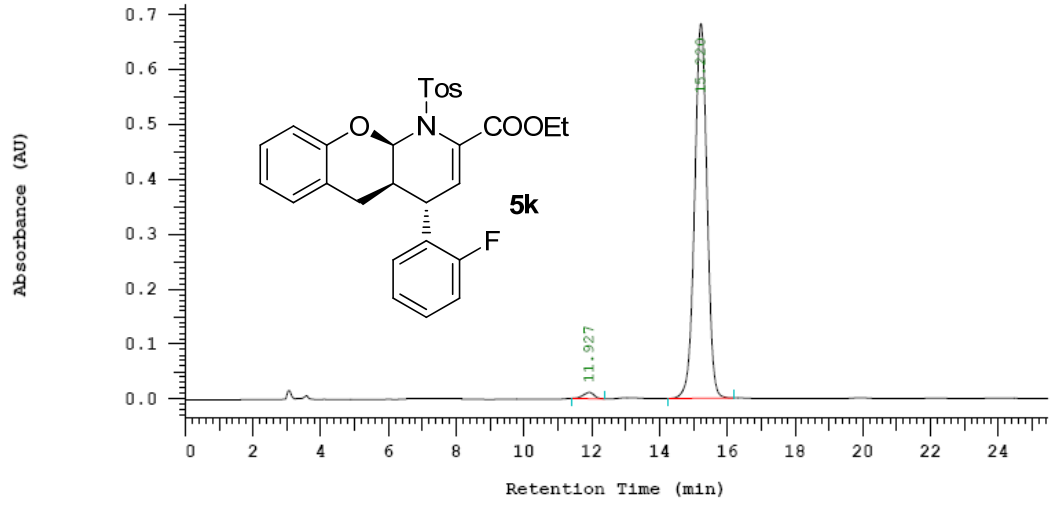
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	11.627	8642020	51.124	BB
2	15.147	8262076	48.876	BB
		16904096	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

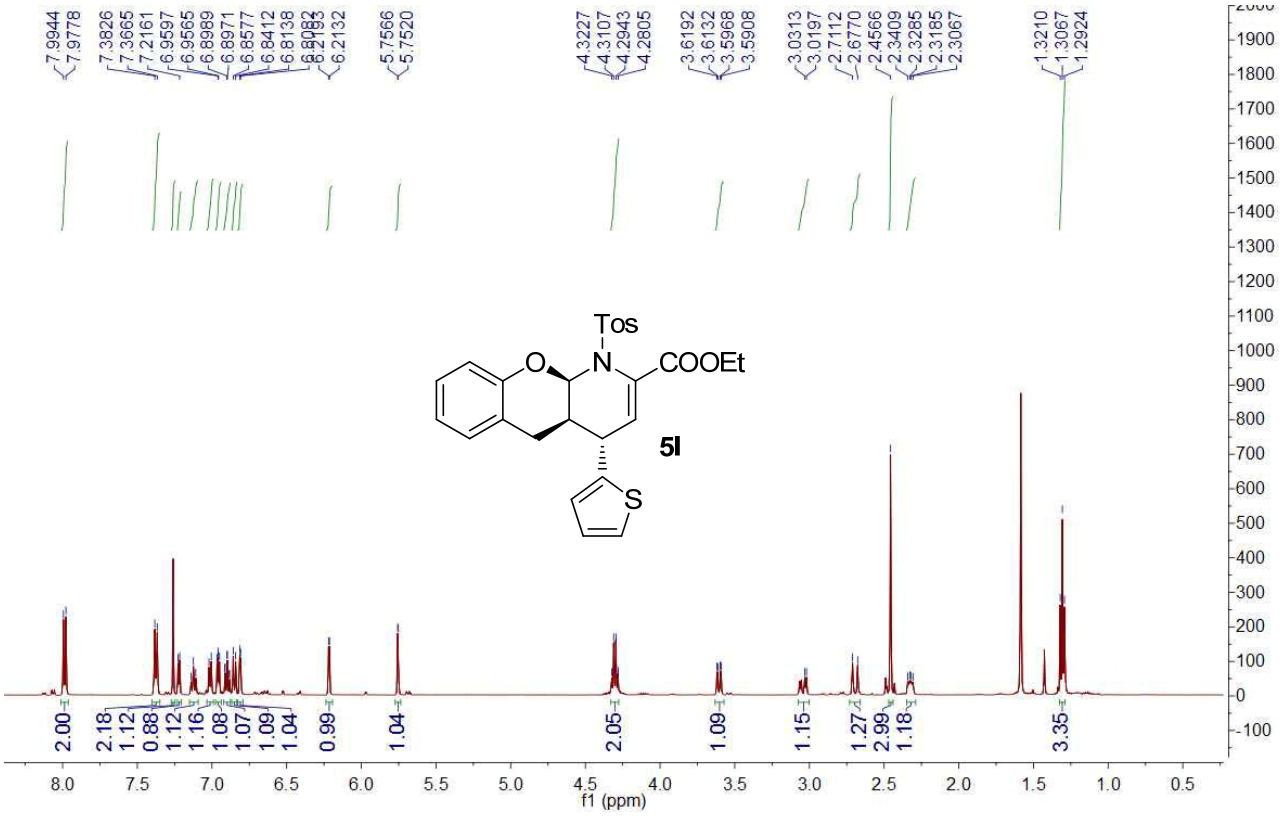


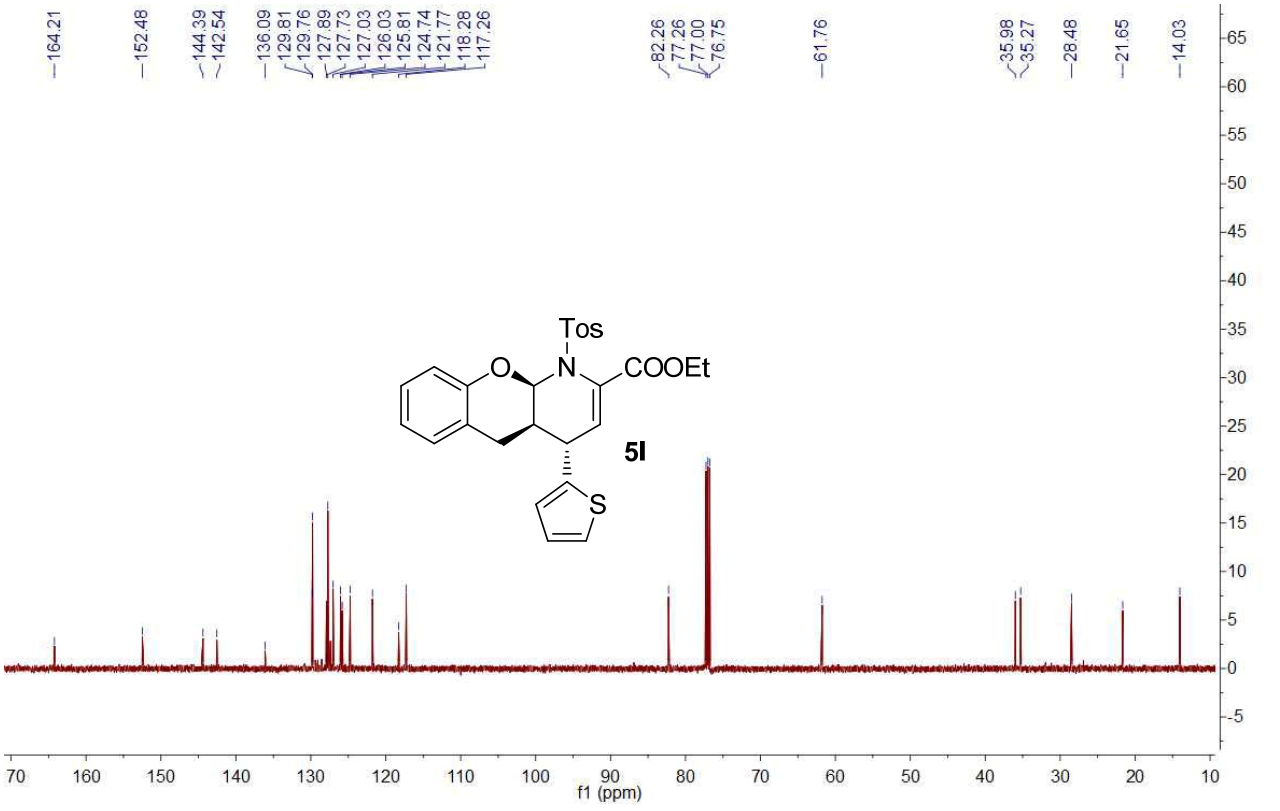
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

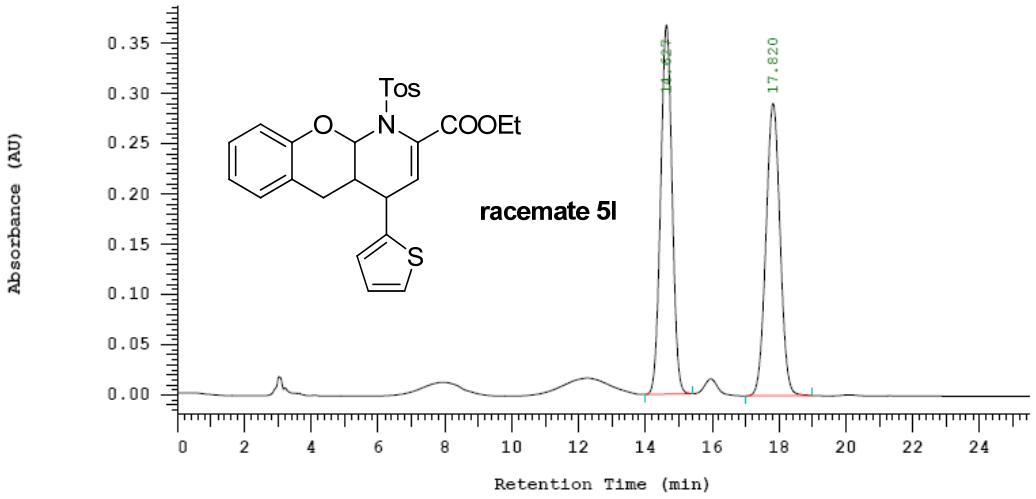
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	11.927	123101	1.405	BB
2	15.220	8637157	98.595	BB
		8760258	100.000	


7.9944
7.9778
7.3826
7.3665
7.2161
6.9597
6.9565
6.8989
6.8971
6.8577
6.8412
6.8138
6.2132
5.7566
5.7520
4.3227
4.3107
4.2943
4.2805
3.6192
3.6132
3.5968
3.5908
3.0313
3.0197
2.7112
2.6770
2.4566
2.3409
2.3285
2.3185
2.3067
1.3210
1.3067
1.2924
Tos
O
N
COOEt
S
5l
2.00
2.18
1.12
0.88
1.12
1.16
1.08
1.07
1.09
1.04
0.99
1.04
2.05
1.09
1.15
1.27
2.99
1.18
3.35
f1 (ppm)


164.21
152.48
144.39
142.54
136.09
129.81
129.76
127.89
127.73
127.03
126.03
125.81
124.74
121.77
118.28
117.26
82.26
77.26
77.00
76.75
61.76
35.98
35.27
28.48
21.65
14.03
Tos
O
N
COOEt
S
5l
f1 (ppm)

Chrom Type: Fixed WL Chromatogram, 210 nm



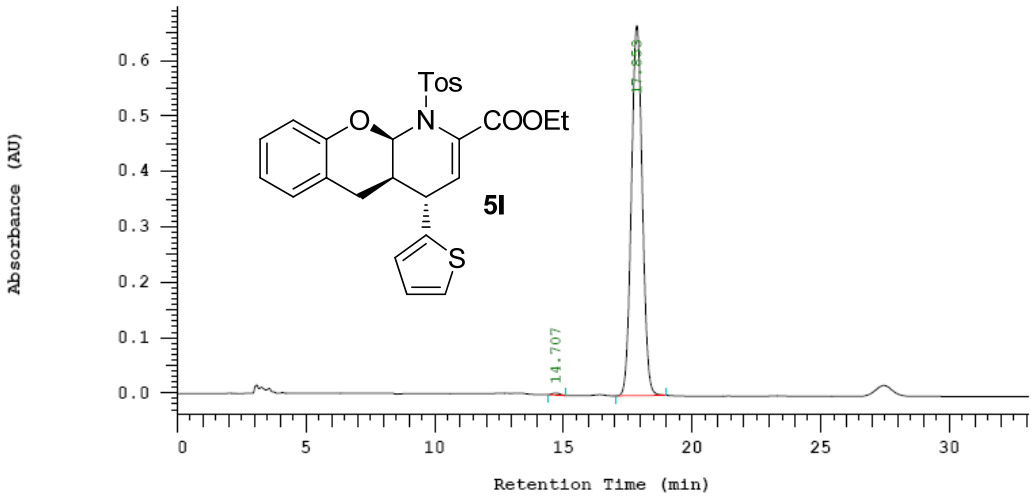
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	14.627	4347463	50.629	BB
2	17.820	4239375	49.371	BB
		8586838	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

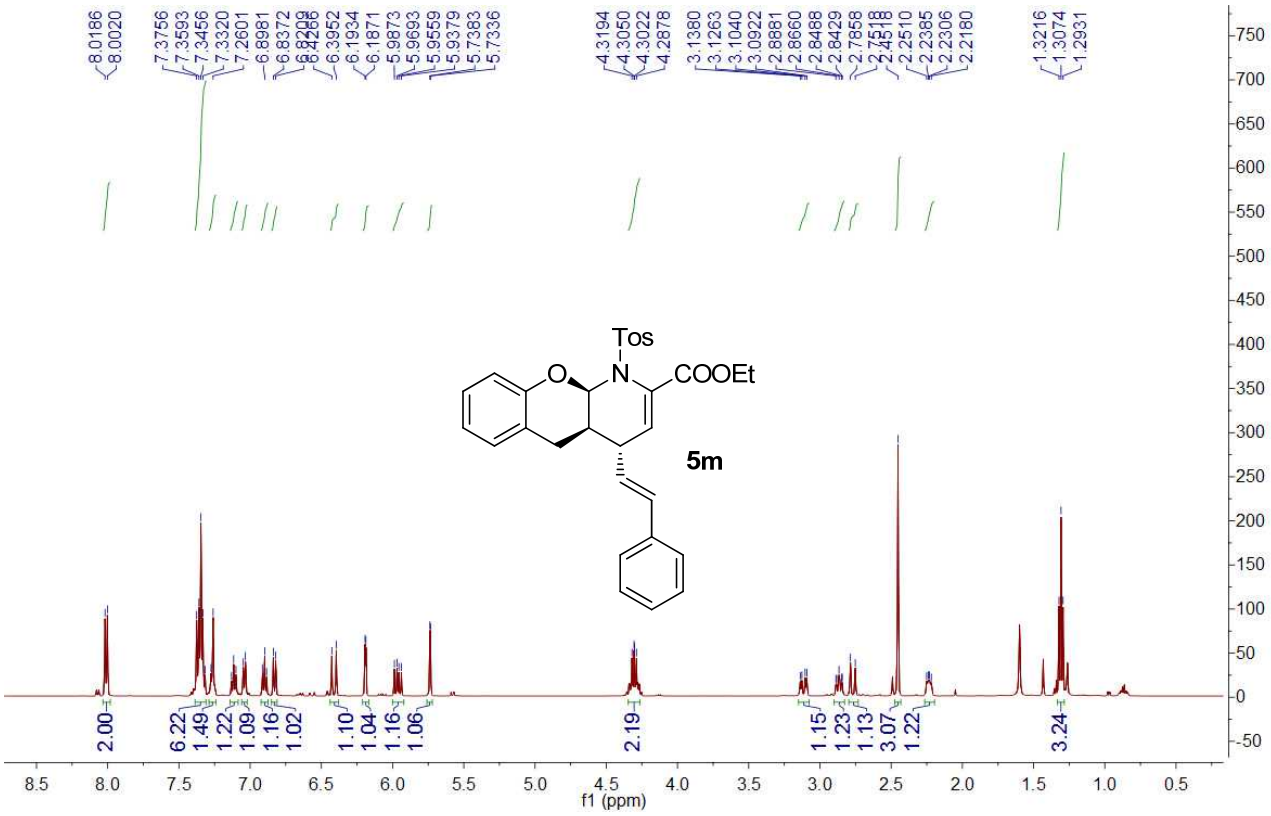


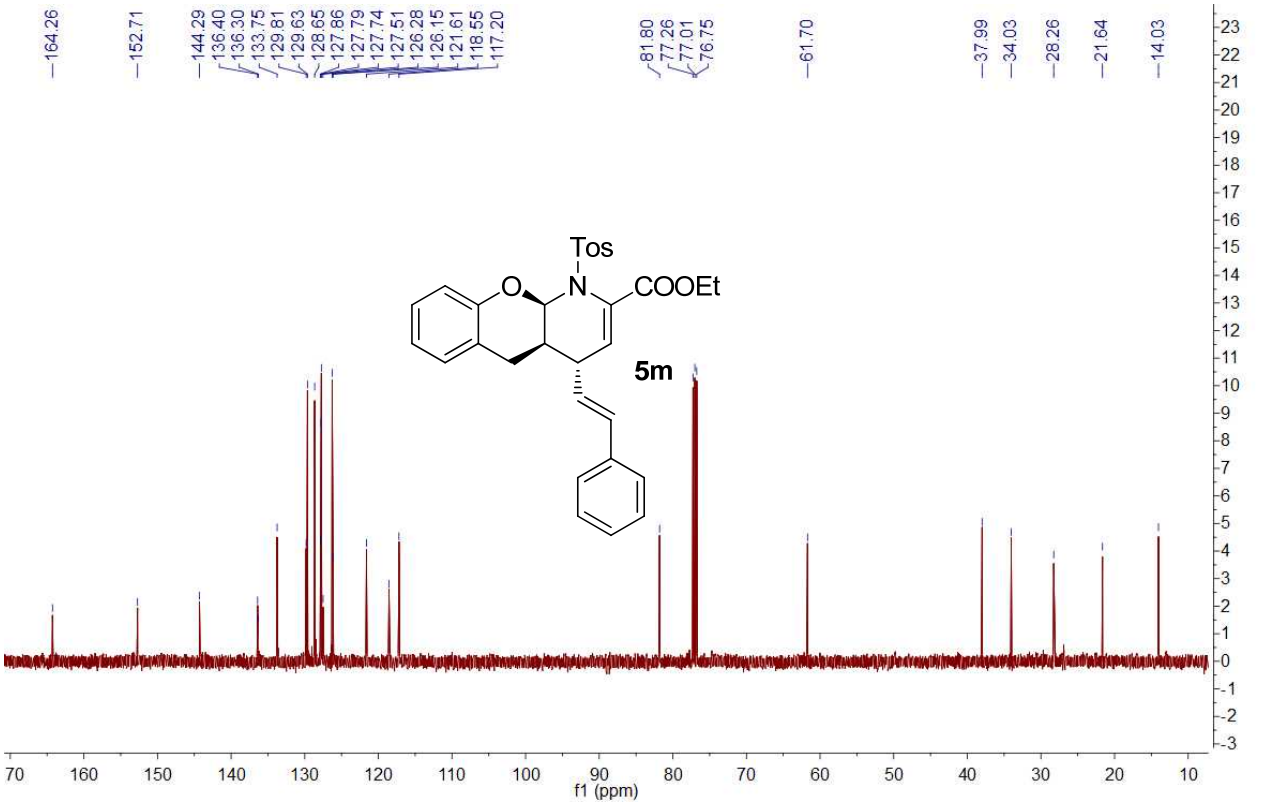
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

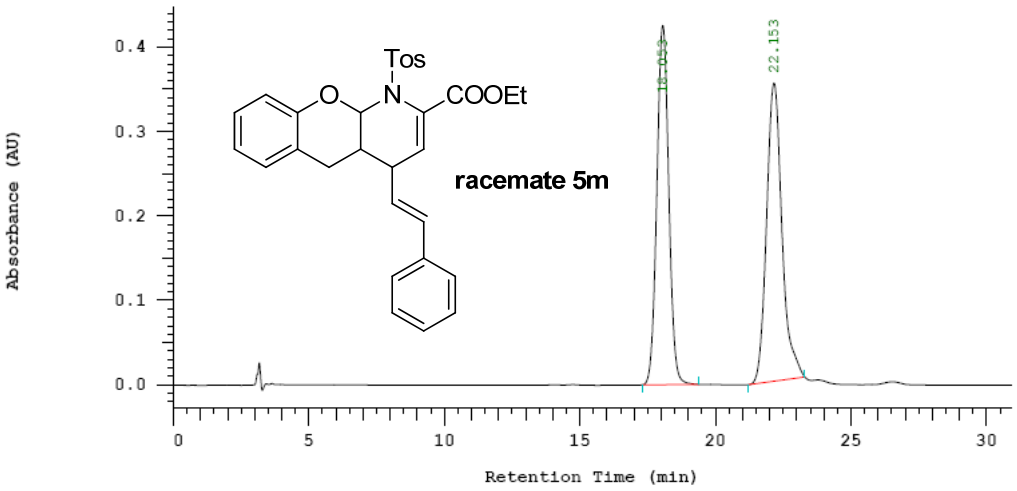
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	14.707	38343	0.391	BB
2	17.853	9766532	99.609	BB
		9804875	100.000	


8.0186
8.0020
7.3756
7.3593
7.3456
7.3320
7.2601
6.8981
6.8372
6.3952
6.1934
6.1871
5.9873
5.9693
5.9559
5.9379
5.7383
5.7336
4.3194
4.3050
4.3022
4.2878
3.1380
3.1263
3.1040
3.0922
2.8881
2.8660
2.8488
2.8429
2.7858
2.7518
2.2510
2.2385
2.2306
2.2180
1.3216
1.3074
1.2931
Tos
N
O
COOEt
5m
2.00
6.22
1.49
1.22
1.09
1.16
1.02
1.10
1.04
1.16
1.06
2.19
1.15
1.23
1.13
3.07
1.22
3.24
f1 (ppm)


164.26
152.71
144.29
136.40
136.30
133.75
129.81
129.63
128.65
127.86
127.79
127.74
127.51
126.28
126.15
121.61
118.55
117.20
81.80
77.26
77.01
76.75
61.70
37.99
34.03
28.26
21.64
14.03
Tos
N
O
COOEt
5m
f1 (ppm)

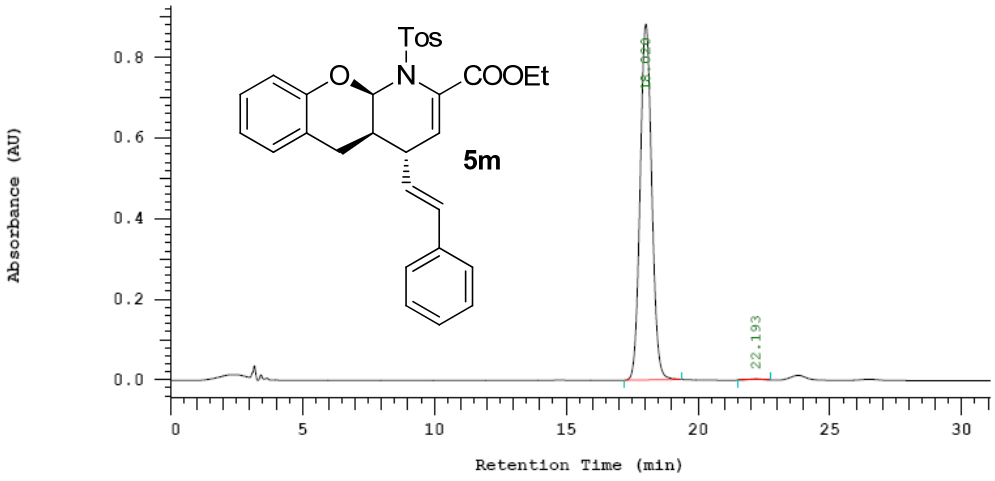
Chrom Type: Fixed WL Chromatogram, 210 nm



Chrom Type: Fixed WL Chromatogram, 210 nm
Peak Quantitation: AREA
Calculation Method: AREA%

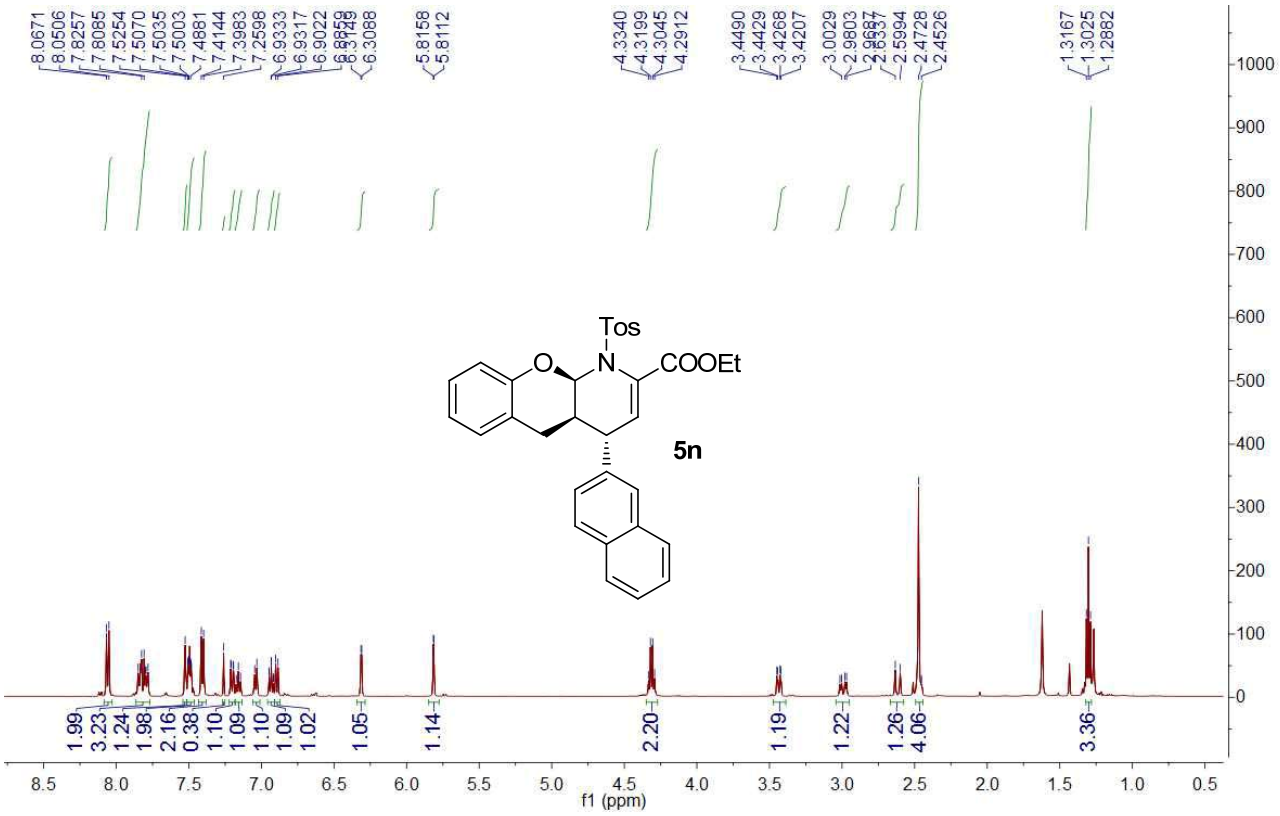
No.	RT	Area	Area %	BC
1	18.053	6426009	47.998	BB
2	22.153	6962140	52.002	BB
		13388149	100.000	

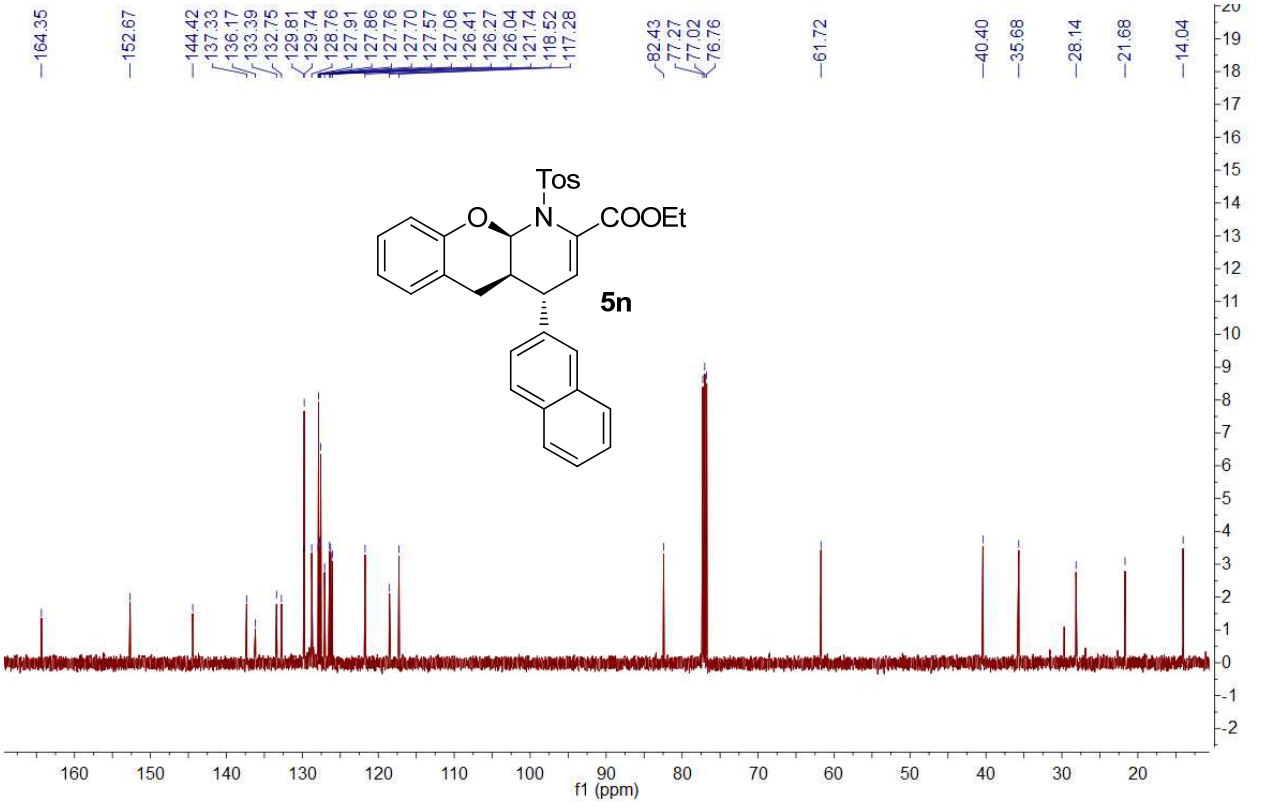
Chrom Type: Fixed WL Chromatogram, 210 nm



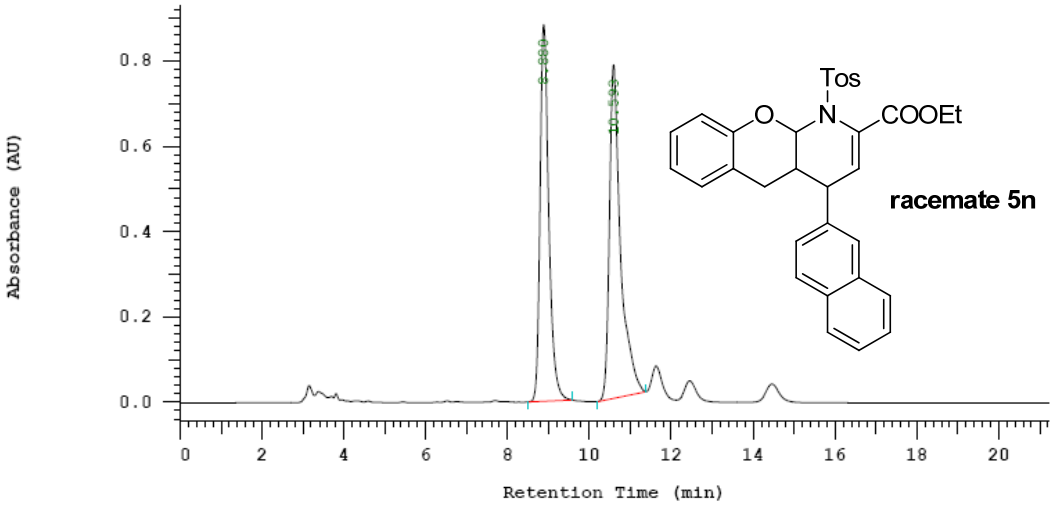
Chrom Type: Fixed WL Chromatogram, 210 nm
Peak Quantitation: AREA
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	18.020	13369424	99.664	BB
2	22.193	45103	0.336	BB
		13414527	100.000	


8.0671
8.0506
7.8257
7.8085
7.5254
7.5070
7.5035
7.5003
7.4881
7.4144
7.3983
7.2598
6.9333
6.9317
6.9022
6.8859
6.8749
6.3088
5.8158
5.8112
4.3340
4.3199
4.3045
4.2912
3.4490
3.4429
3.4268
3.4207
3.0029
2.9803
2.6337
2.5994
2.4728
2.4526
1.3167
1.3025
1.2882
Tos
O
N
COOEt
5n
1.99
3.23
1.24
1.98
2.16
0.38
1.10
1.09
1.10
1.09
1.02
1.05
1.14
2.20
1.19
1.22
1.26
4.06
3.36
f1 (ppm)


164.35
152.67
144.42
137.33
136.17
133.39
132.75
129.81
129.74
128.76
127.91
127.86
127.76
127.70
127.57
127.06
126.41
126.27
126.04
121.74
118.52
117.28
82.43
77.27
77.02
76.76
61.72
40.40
35.68
28.14
21.68
14.04
Tos
O
N
COOEt
5n
f1 (ppm)

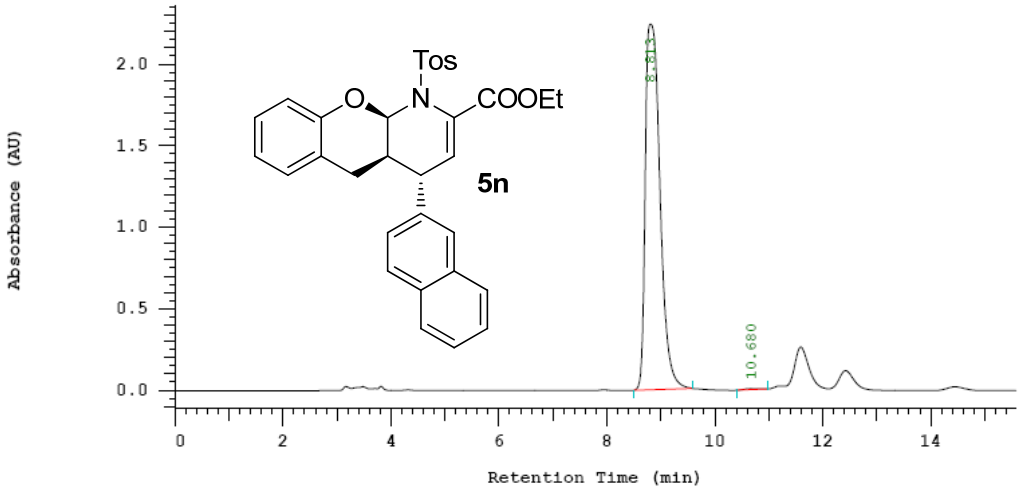
Chrom Type: Fixed WL Chromatogram, 210 nm



Chrom Type: Fixed WL Chromatogram, 210 nm
Peak Quantitation: AREA
Calculation Method: AREA%

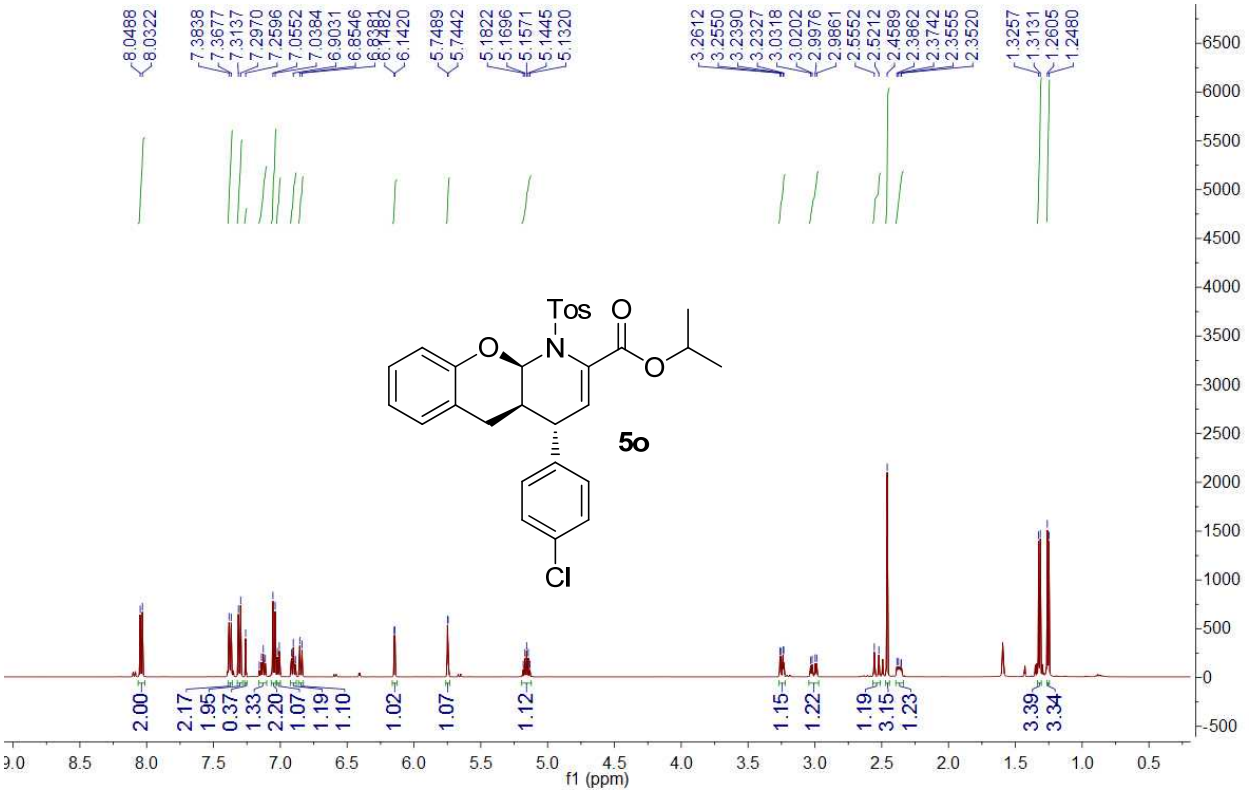
No.	RT	Area	Area %	BC
1	8.880	6519772	46.596	BB
2	10.593	7472288	53.404	BB
		13992060	100.000	

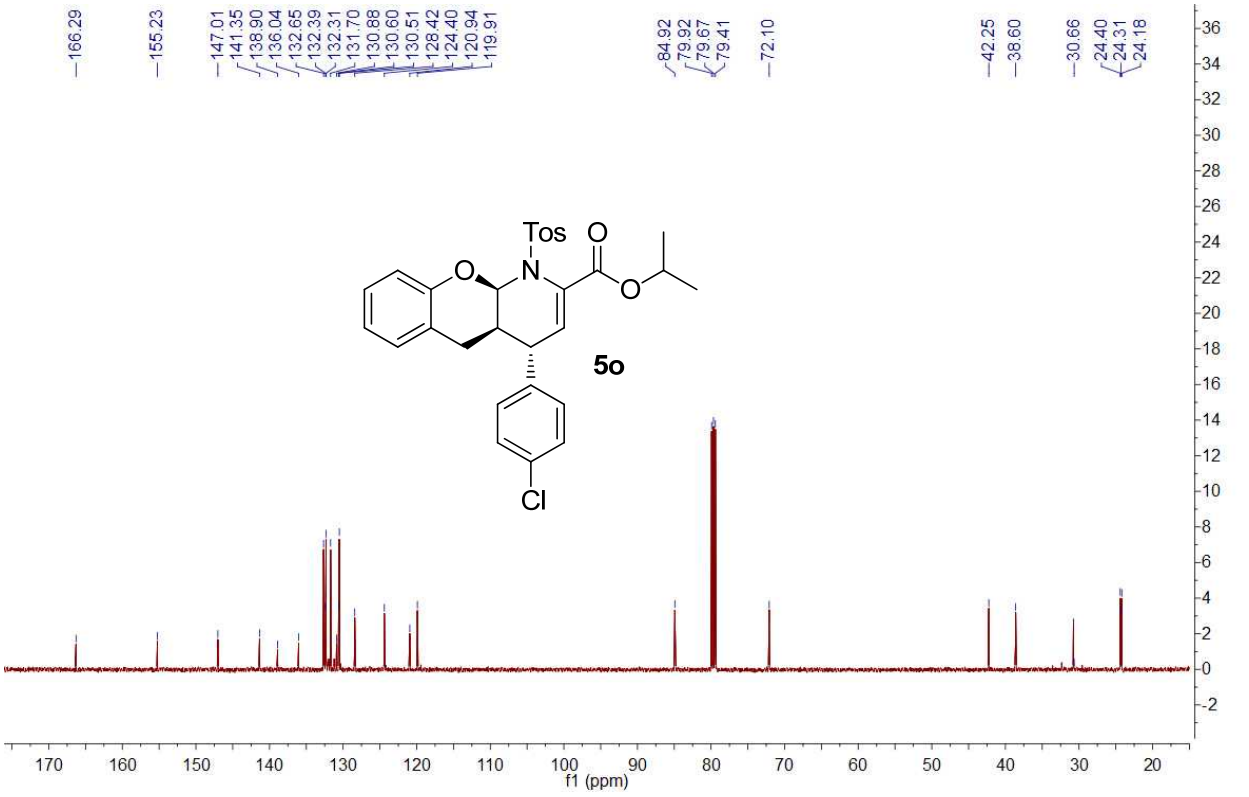
Chrom Type: Fixed WL Chromatogram, 210 nm



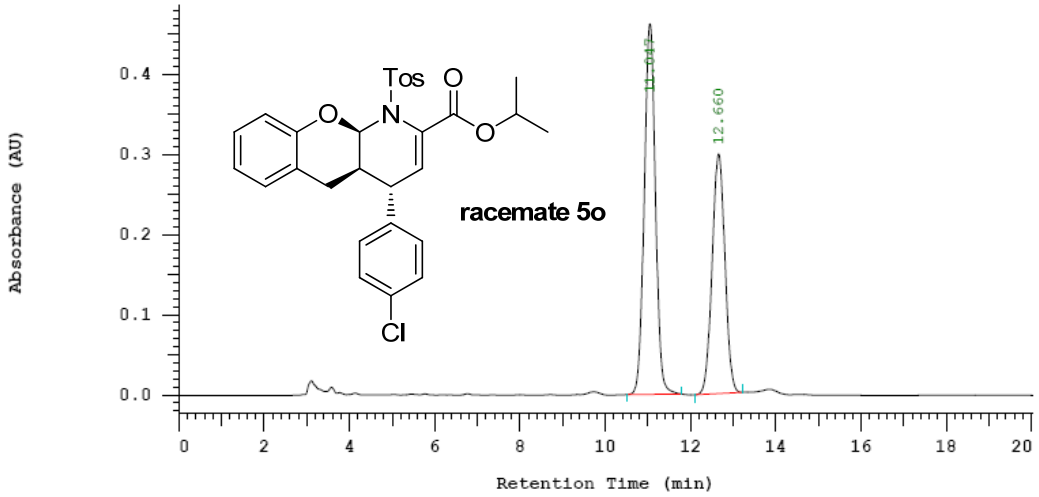
Chrom Type: Fixed WL Chromatogram, 210 nm
Peak Quantitation: AREA
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	8.813	21204568	99.741	BB
2	10.680	55010	0.259	BB
		21259578	100.000	


8.0488
8.0322
7.3838
7.3677
7.3137
7.2970
7.2596
7.0552
7.0384
6.9031
6.8546
6.8381
6.8282
6.1420
5.7489
5.7442
5.1822
5.1696
5.1571
5.1445
5.1320
3.2612
3.2550
3.2390
3.2327
3.0318
3.0202
2.9976
2.9861
2.5552
2.5212
2.4589
2.3862
2.3742
2.3555
2.3520
1.3257
1.3131
1.2605
1.2480
Tos
O
N
O
O
5o
Cl
2.00
2.17
1.95
0.37
1.33
2.20
1.07
1.19
1.10
1.02
1.07
1.12
1.15
1.22
1.19
3.15
1.23
3.39
3.34
f1 (ppm)


166.29
155.23
147.01
141.35
138.90
136.04
132.65
132.39
132.31
131.70
130.88
130.60
130.51
128.42
124.40
120.94
119.91
84.92
79.92
79.67
79.41
72.10
42.25
38.60
30.66
24.40
24.31
24.18
Tos
O
N
O
O
5o
Cl
f1 (ppm)

Chrom Type: Fixed WL Chromatogram, 210 nm



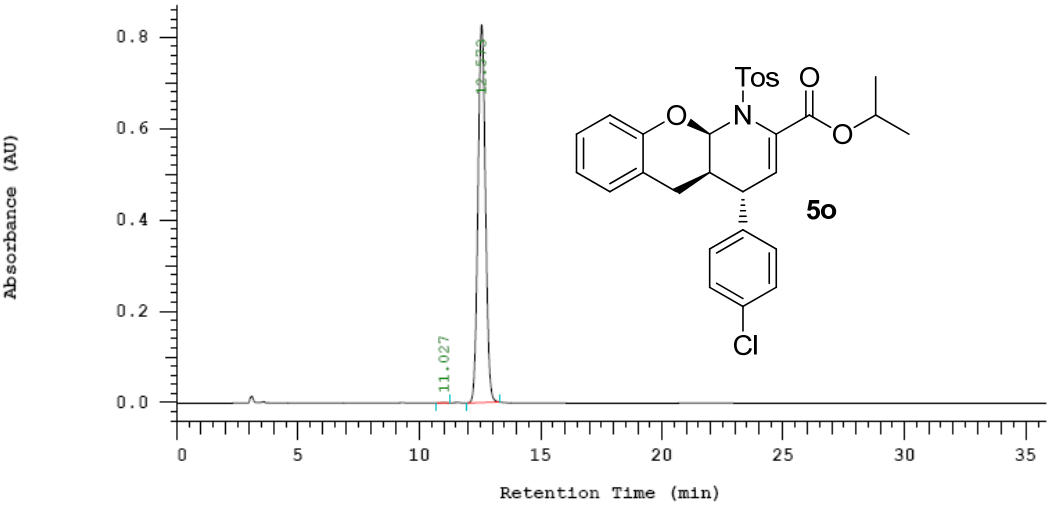
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	11.047	4230106	57.679	BB
2	12.660	3103770	42.321	BB
		7333876	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

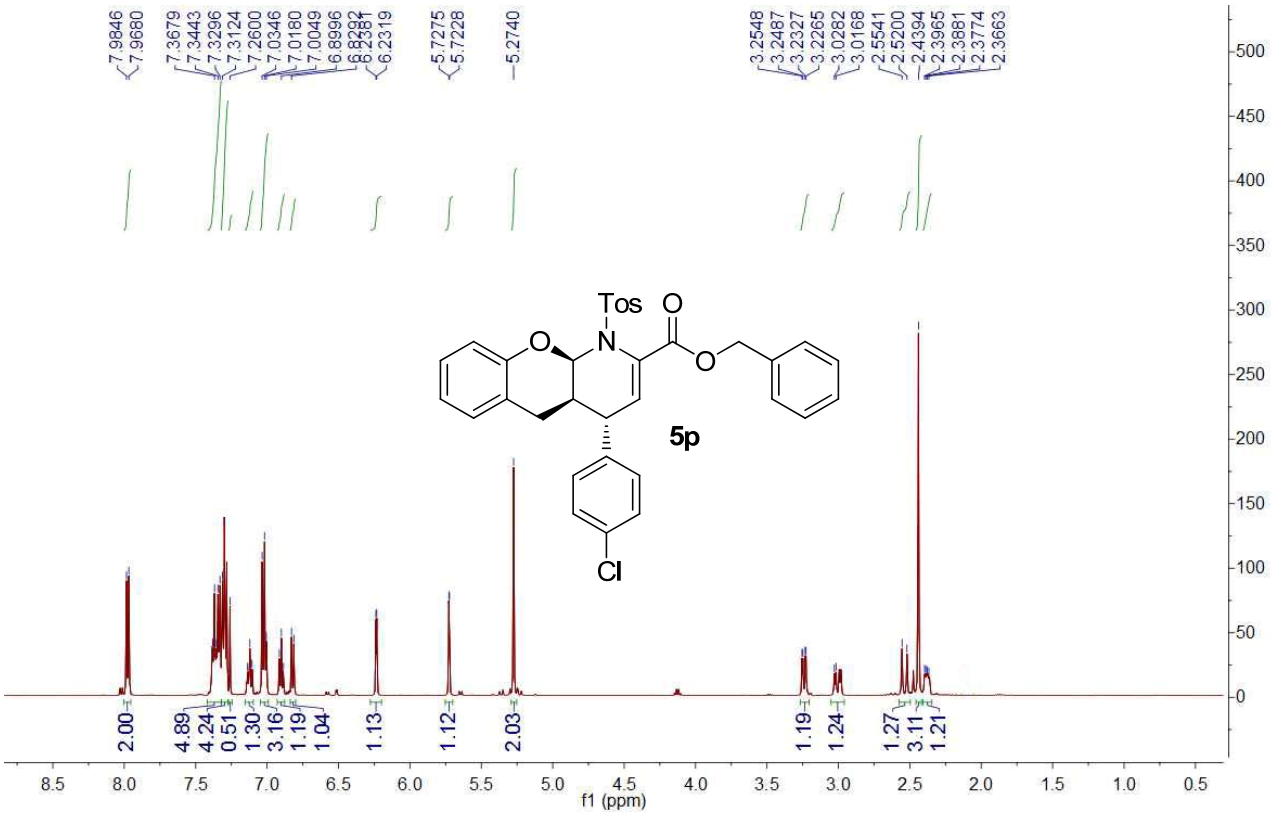


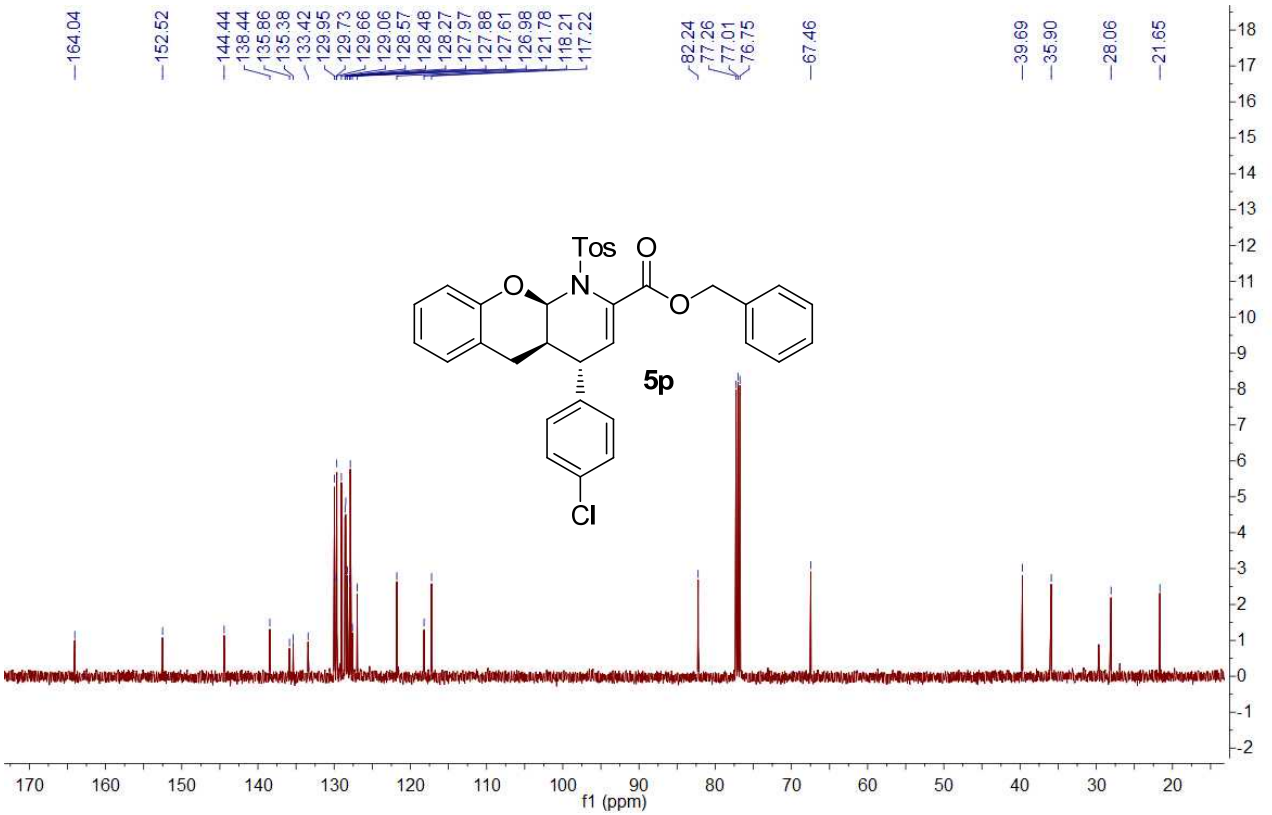
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

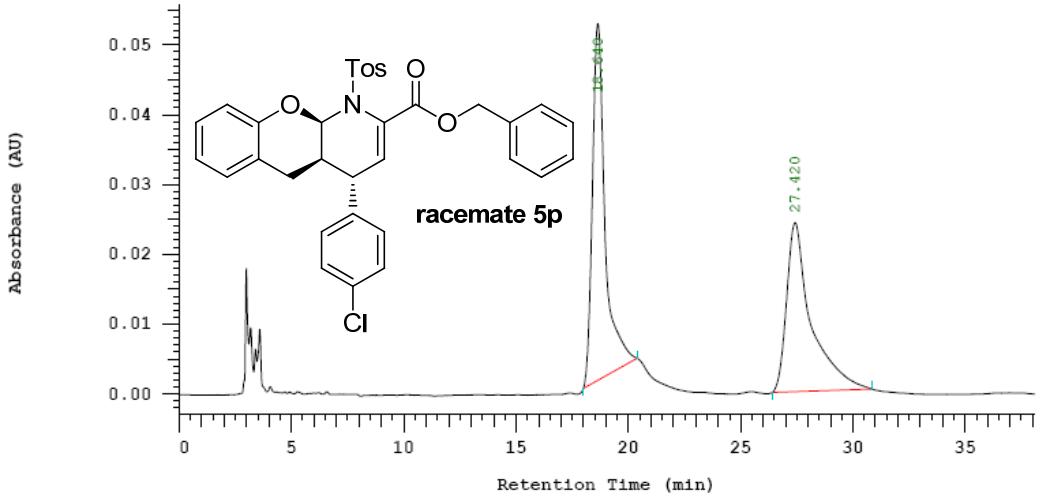
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	11.027	9209	0.106	BB
2	12.573	8704617	99.894	BB
		8713826	100.000	


7.9846
7.9680
7.3679
7.3443
7.3296
7.3124
7.2600
7.0346
7.0180
7.0049
6.8996
6.8292
6.2381
6.2319
5.7275
5.7228
5.2740
3.2548
3.2487
3.2327
3.2265
3.0282
3.0168
2.5541
2.5200
2.4394
2.3965
2.3881
2.3774
2.3663
Tos
O
N
O
O
5p
Cl
2.00
4.89
4.24
0.51
1.30
3.16
1.19
1.04
1.13
1.12
2.03
1.19
1.24
1.27
3.11
1.21
f1 (ppm)


164.04
152.52
144.44
138.44
135.86
135.38
133.42
129.95
129.73
129.66
129.06
128.57
128.48
128.27
127.97
127.88
127.61
126.98
121.78
118.21
117.22
82.24
77.26
77.01
76.75
67.46
39.69
35.90
28.06
21.65
Tos
O
N
O
O
5p
Cl
f1 (ppm)

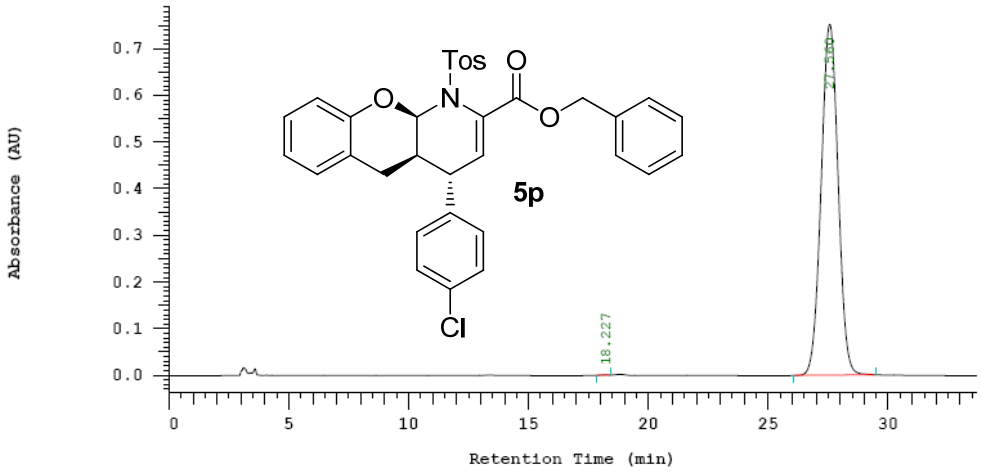
Chrom Type: Fixed WL Chromatogram, 210 nm



Chrom Type: Fixed WL Chromatogram, 210 nm
Peak Quantitation: AREA
Calculation Method: AREA%

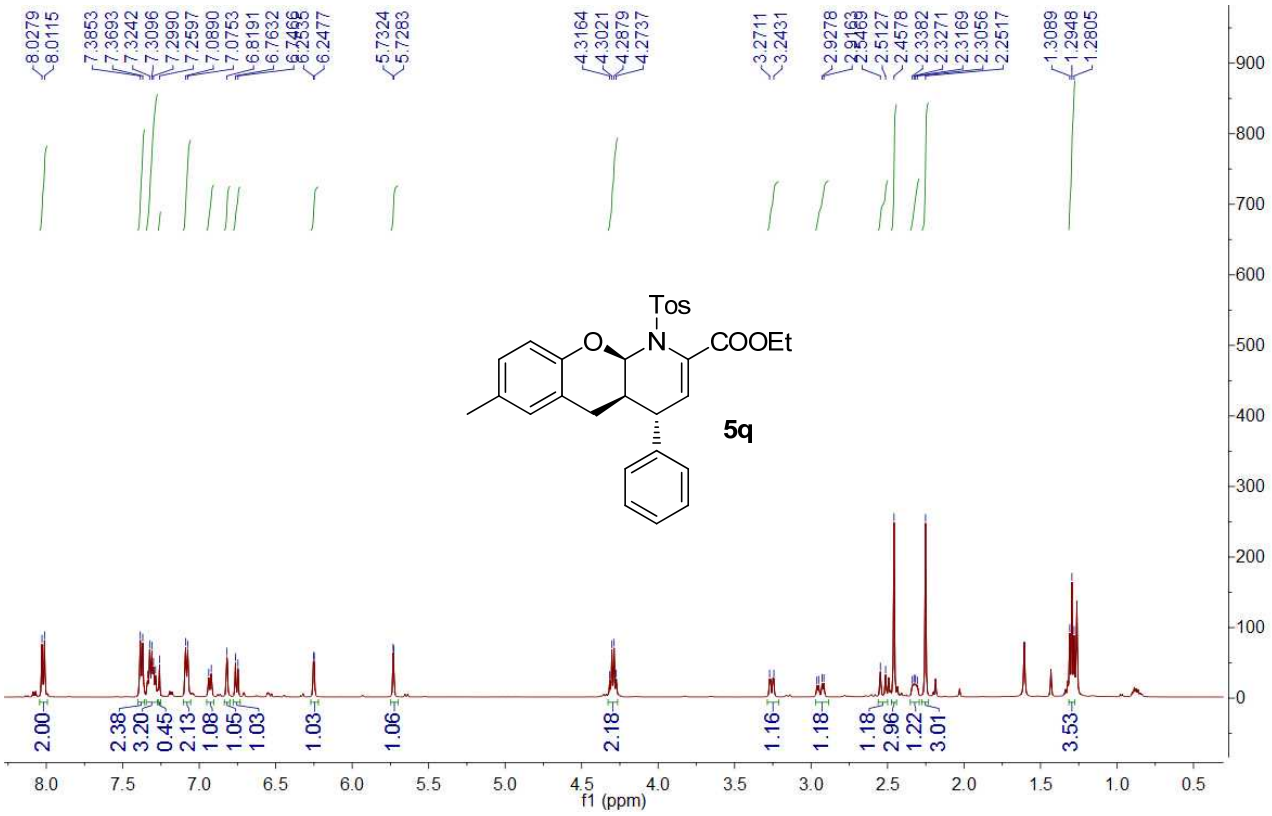
No.	RT	Area	Area %	BC
1	18.640	992879	52.017	BB
2	27.420	915882	47.983	BB
		1908761	100.000	

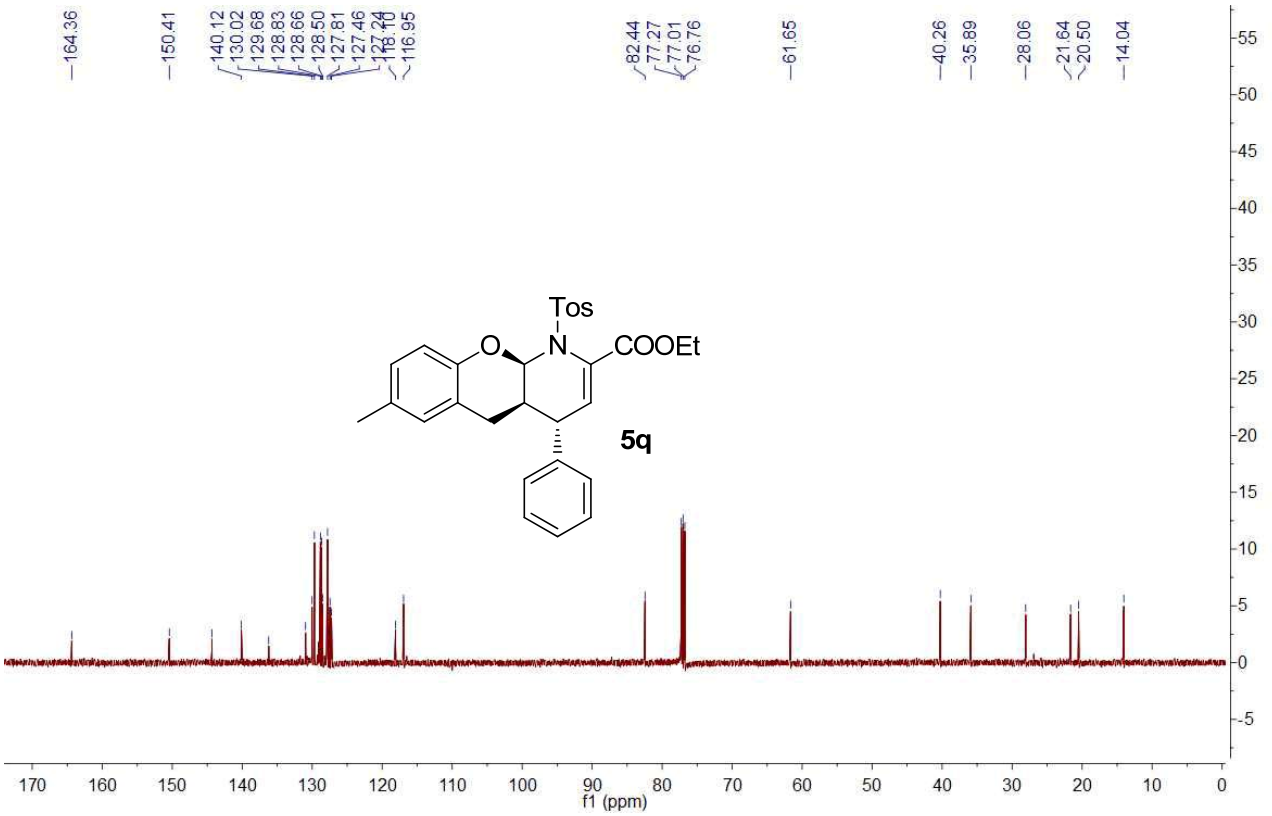
Chrom Type: Fixed WL Chromatogram, 210 nm



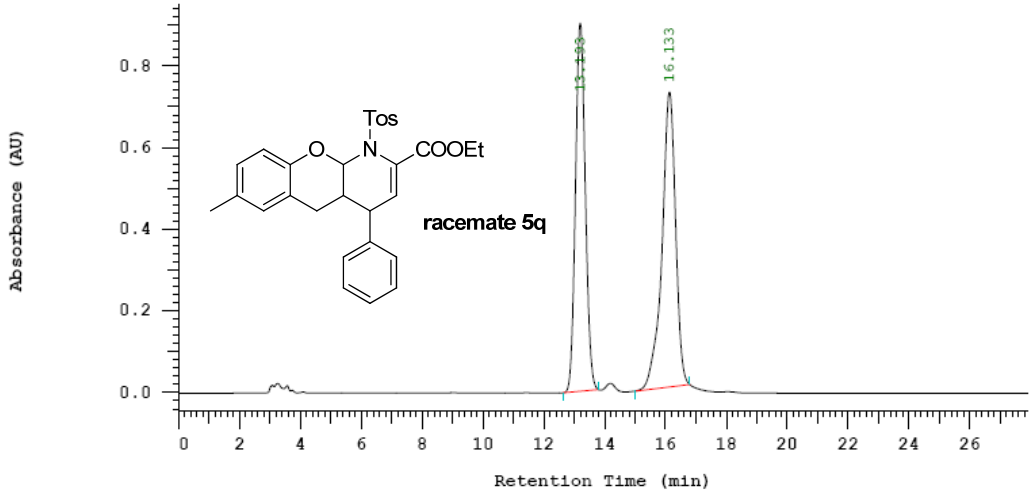
Chrom Type: Fixed WL Chromatogram, 210 nm
Peak Quantitation: AREA
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	18.227	5342	0.029	BB
2	27.560	18339617	99.971	BB
		18344959	100.000	


8.0279
8.0115
7.3853
7.3693
7.3242
7.3096
7.2990
7.2597
7.0890
7.0753
6.8191
6.7632
6.2477
5.7324
5.7283
4.3164
4.3021
4.2879
4.2737
3.2711
3.2431
2.9278
2.5127
2.4578
2.3382
2.3271
2.3169
2.3056
2.2517
1.3089
1.2948
1.2805
Tos
O
N
COOEt
5q
2.00
2.38
3.20
0.45
2.13
1.08
1.05
1.03
1.03
1.06
2.18
1.16
1.18
1.18
2.96
1.22
3.01
3.53
f1 (ppm)


164.36
150.41
140.12
130.02
129.68
128.83
128.66
128.50
127.81
127.46
116.95
82.44
77.27
77.01
76.76
61.65
40.26
35.89
28.06
21.64
20.50
14.04
Tos
O
N
COOEt
5q
f1 (ppm)

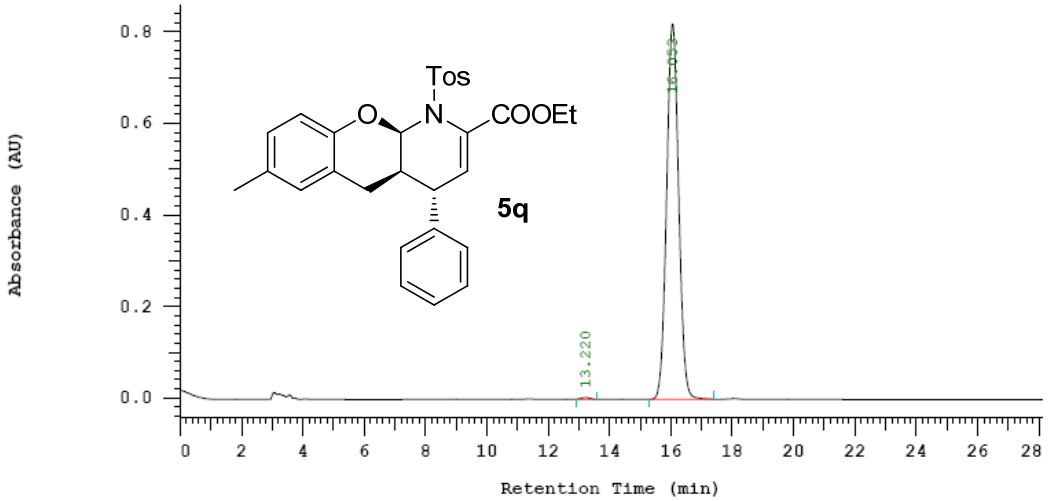
Chrom Type: Fixed WL Chromatogram, 210 nm



Chrom Type: Fixed WL Chromatogram, 210 nm
Peak Quantitation: AREA
Calculation Method: AREA%

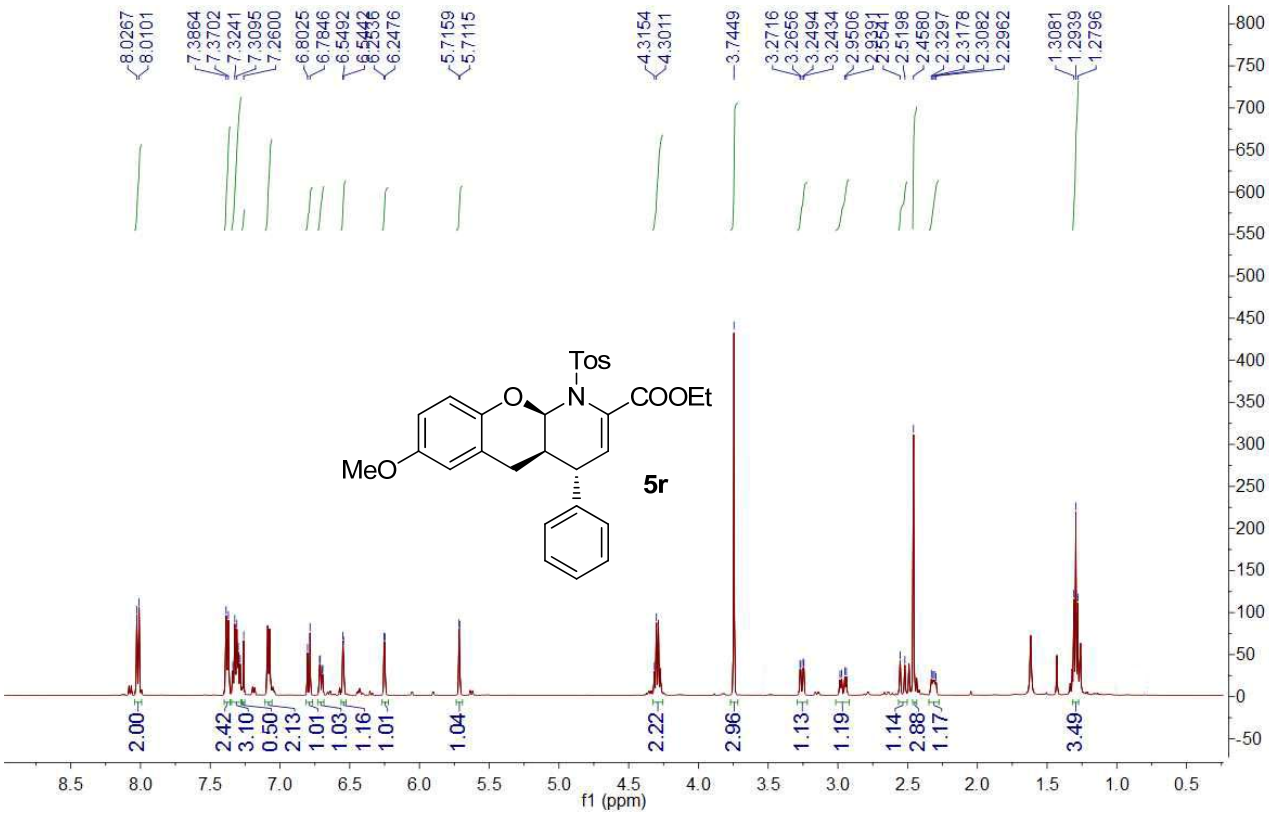
No.	RT	Area	Area %	BC
1	13.193	10029798	47.407	BB
2	16.133	11127019	52.593	BB
		21156817	100.000	

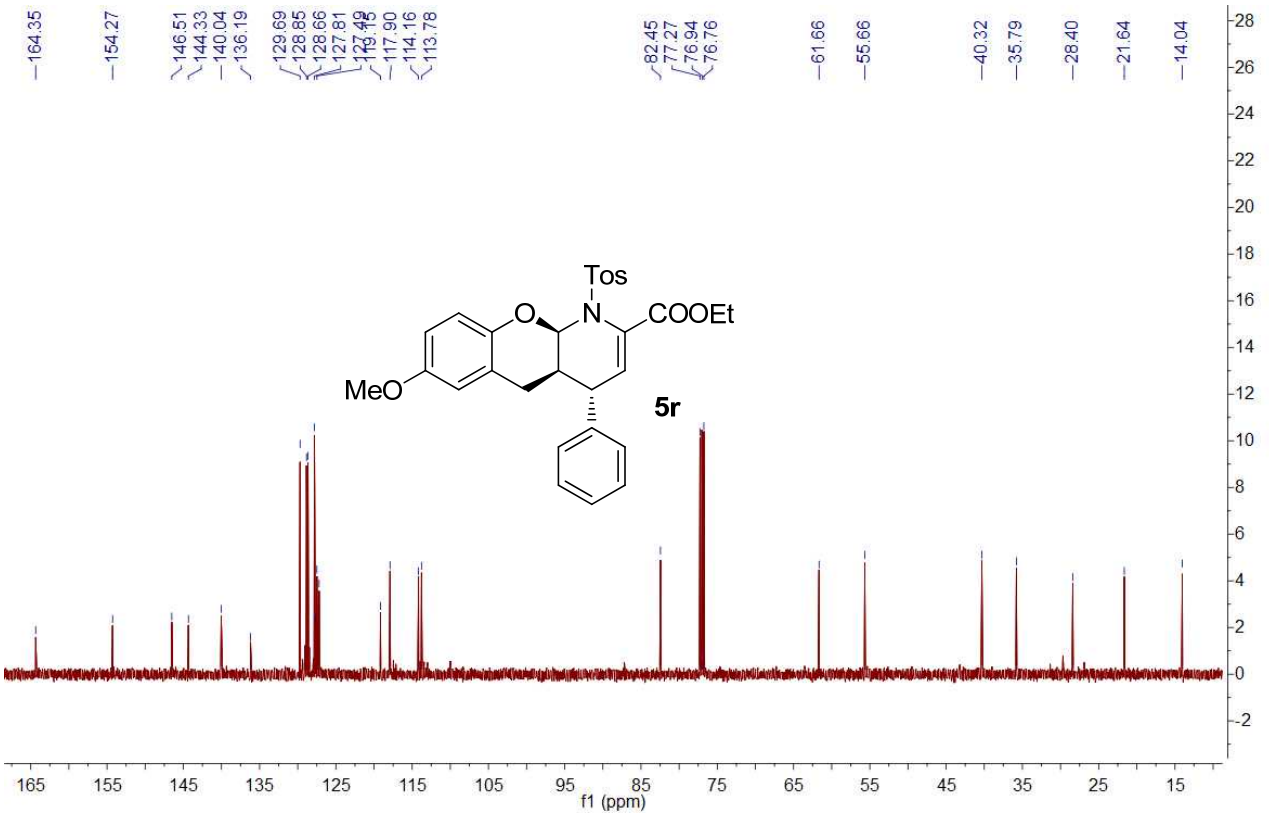
Chrom Type: Fixed WL Chromatogram, 210 nm



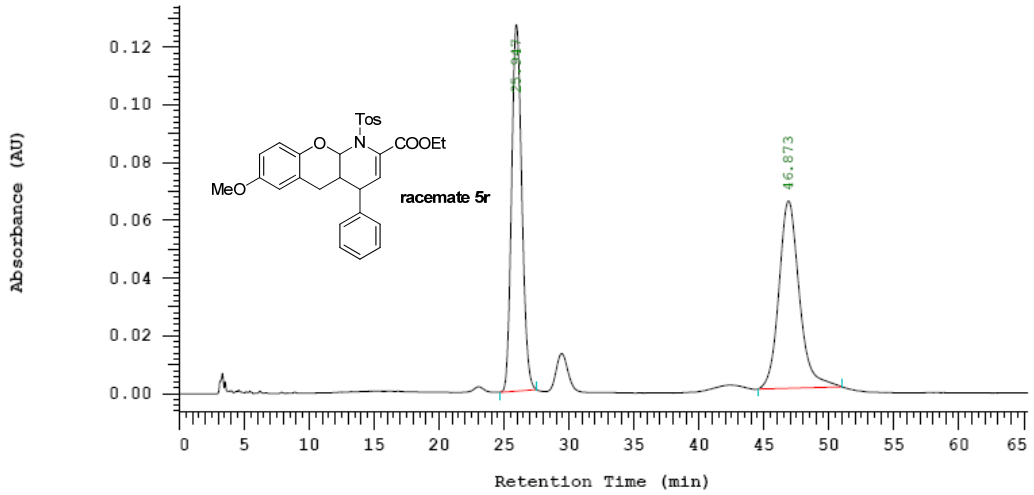
Chrom Type: Fixed WL Chromatogram, 210 nm
Peak Quantitation: AREA
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	13.220	34406	0.309	BB
2	16.053	11086575	99.691	BB
		11120981	100.000	


8.0267
8.0101
7.3864
7.3702
7.3241
7.3095
7.2600
6.8025
6.7846
6.5492
6.2536
6.2476
5.7159
5.7115
4.3154
4.3011
3.7449
3.2716
3.2656
3.2494
3.2434
2.9506
2.5541
2.5198
2.4580
2.3297
2.3178
2.3082
2.2962
1.3081
1.2939
1.2796
Tos
N
O
COOEt
MeO
5r
2.00
2.42
3.10
0.50
2.13
1.01
1.03
1.16
1.01
1.04
2.22
2.96
1.13
1.19
1.14
2.88
1.17
3.49
f1 (ppm)


164.35
154.27
146.51
144.33
140.04
136.19
129.69
128.85
128.66
127.81
117.90
114.16
113.78
82.45
77.27
76.94
76.76
61.66
55.66
40.32
35.79
28.40
21.64
14.04
Tos
N
O
COOEt
MeO
5r
f1 (ppm)

Chrom Type: Fixed WL Chromatogram, 210 nm



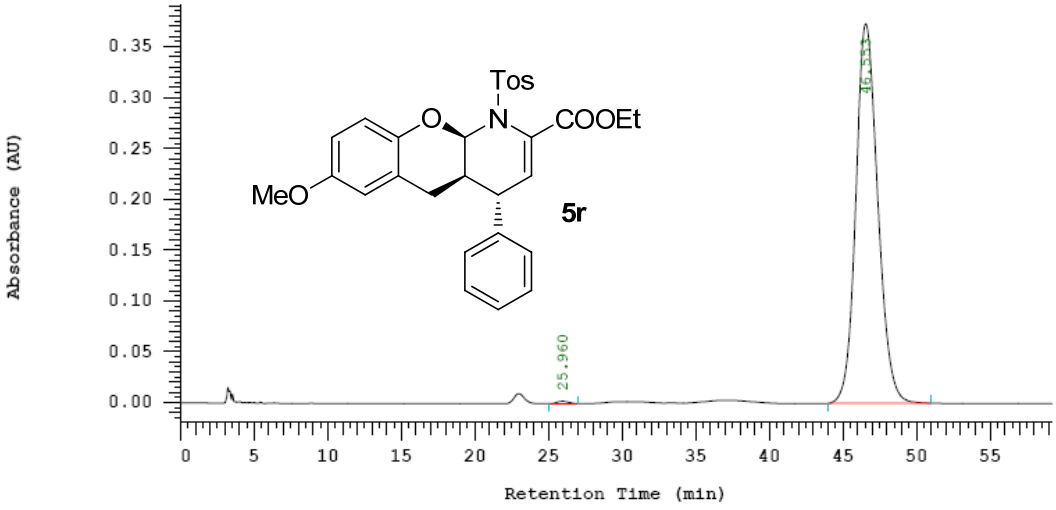
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	25.947	3418802	49.283	BB
2	46.873	3518307	50.717	BB
		6937109	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

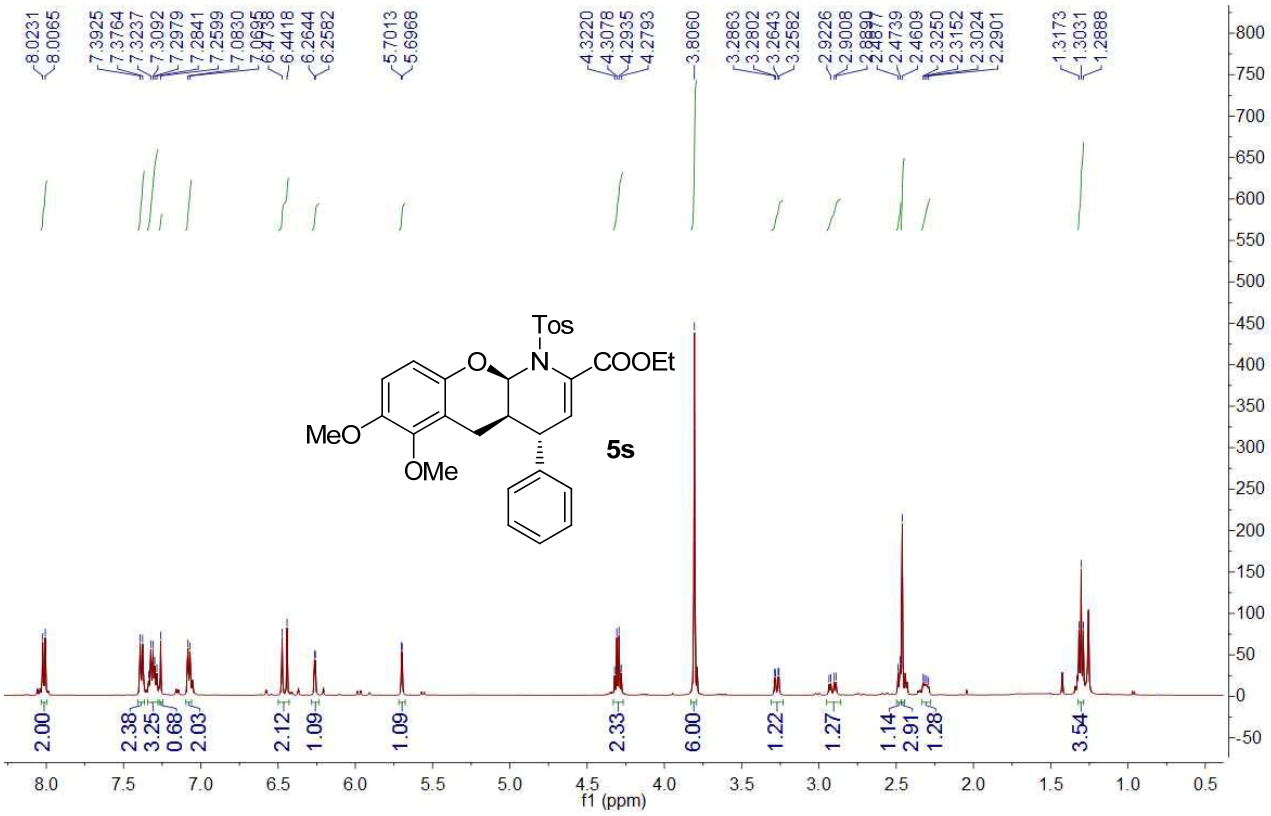


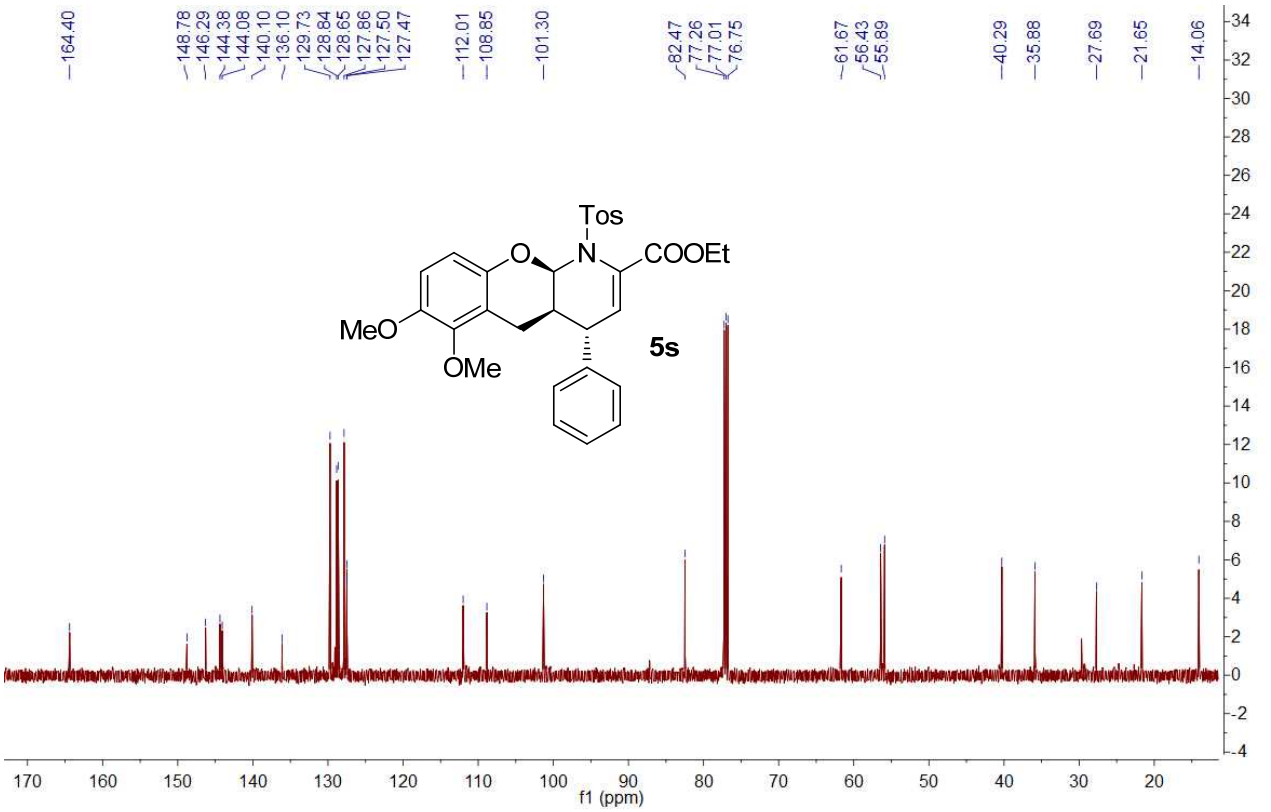
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

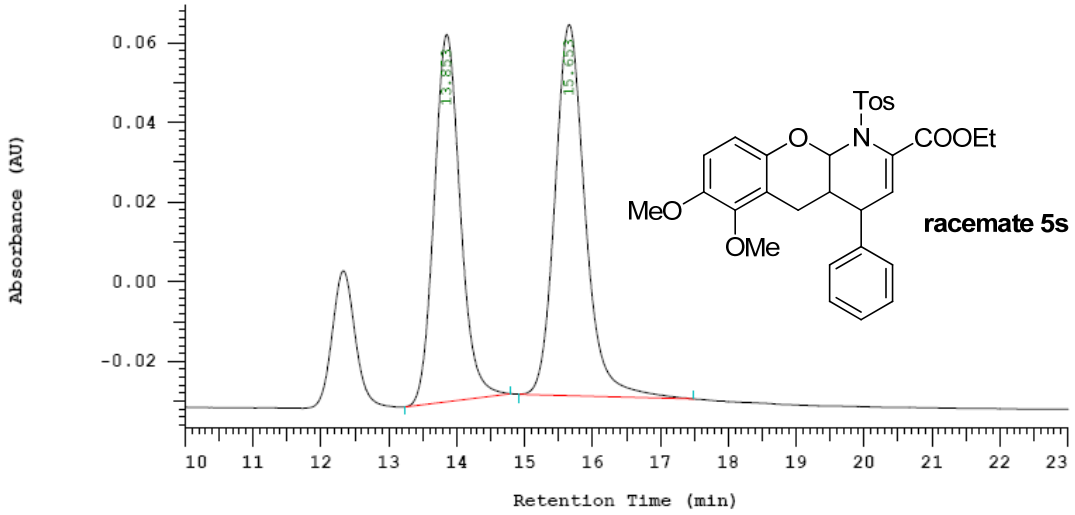
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	25.960	60382	0.313	BB
2	46.553	19243372	99.687	BB
		19303754	100.000	


8.0231
8.0065
7.3925
7.3764
7.3237
7.3092
7.2979
7.2841
7.2599
7.0830
7.0695
6.4738
6.4418
6.2644
6.2582
5.7013
5.6968
4.3220
4.3078
4.2935
4.2793
3.8060
3.2863
3.2802
3.2643
3.2582
2.9226
2.9008
2.4739
2.4609
2.3250
2.3152
2.3024
2.2901
1.3173
1.3031
1.2888
Tos
N
O
COOEt
MeO
OMe
5s
2.00
2.38
3.25
0.68
2.03
2.12
1.09
1.09
2.33
6.00
1.22
1.27
1.14
2.91
1.28
3.54
f1 (ppm)


164.40
148.78
146.29
144.38
144.08
140.10
136.10
129.73
128.84
128.65
127.86
127.50
127.47
112.01
108.85
101.30
82.47
77.26
77.01
76.75
61.67
56.43
55.89
40.29
35.88
27.69
21.65
14.06
Tos
N
O
COOEt
MeO
OMe
5s
f1 (ppm)

Chrom Type: Fixed WL Chromatogram, 210 nm



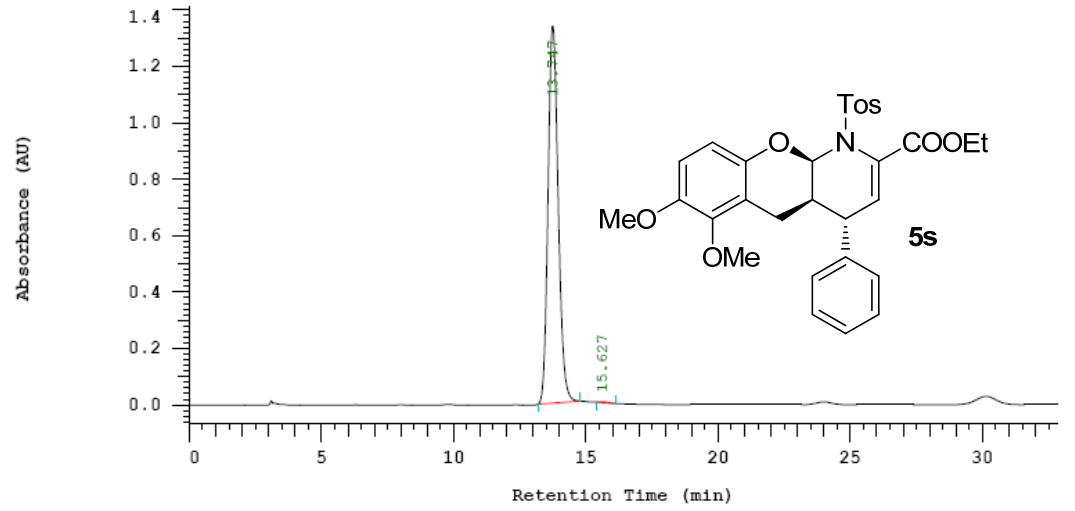
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	13.853	1231328	46.019	BB
2	15.653	1444384	53.981	BB
		2675712	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

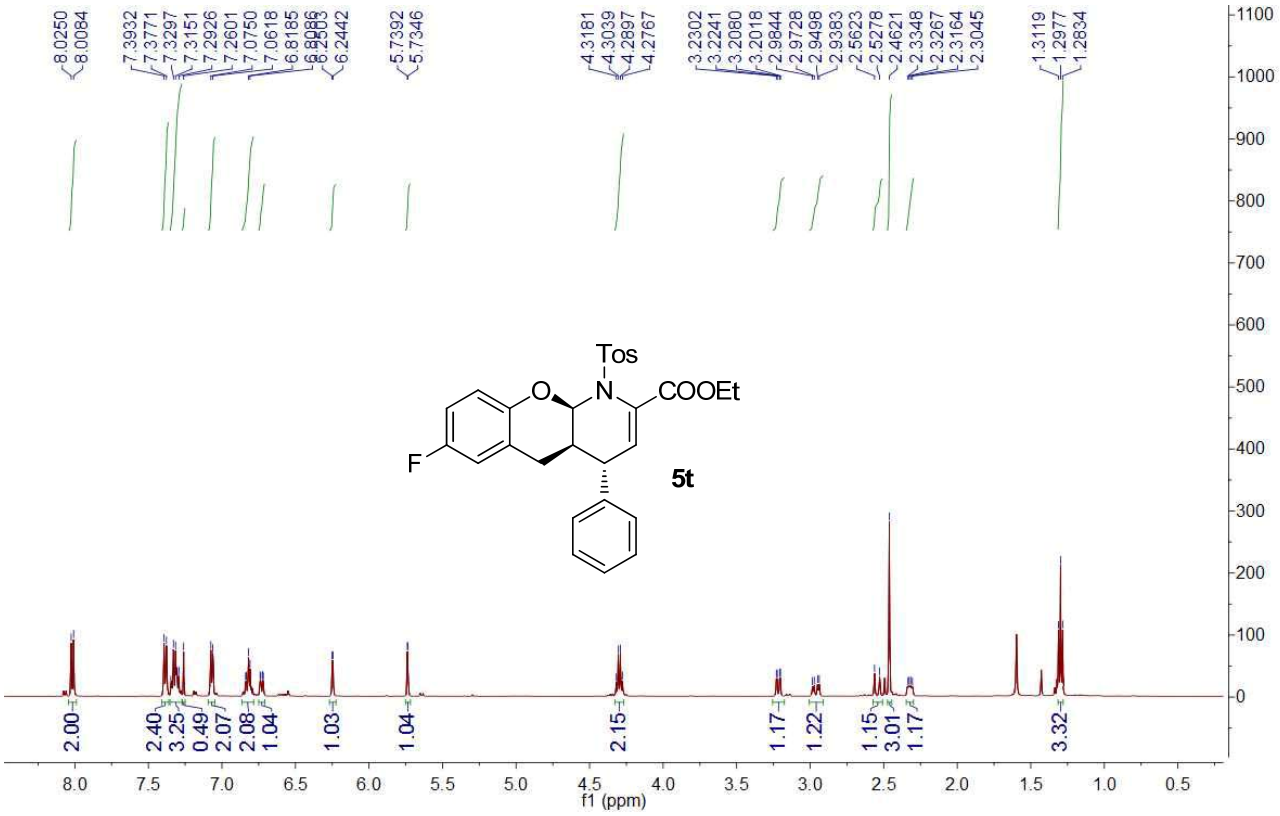


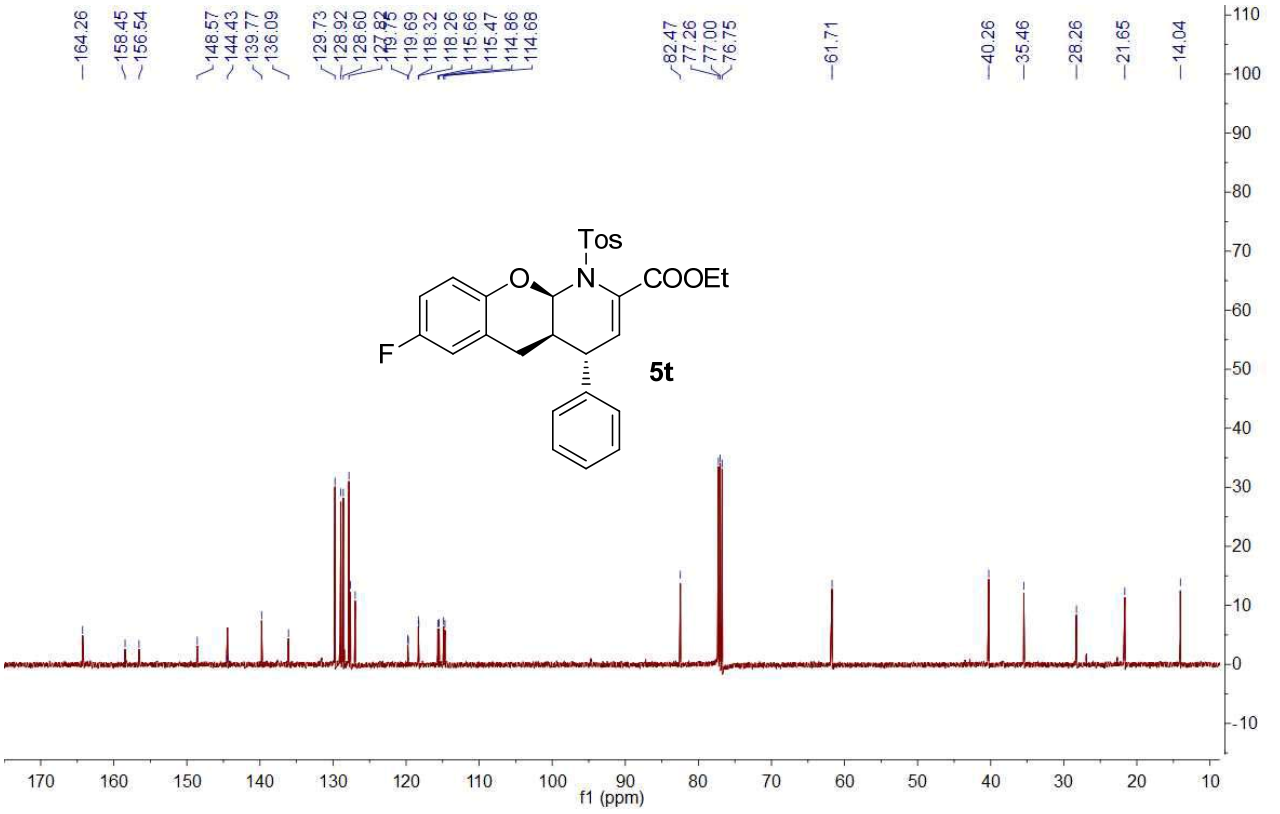
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

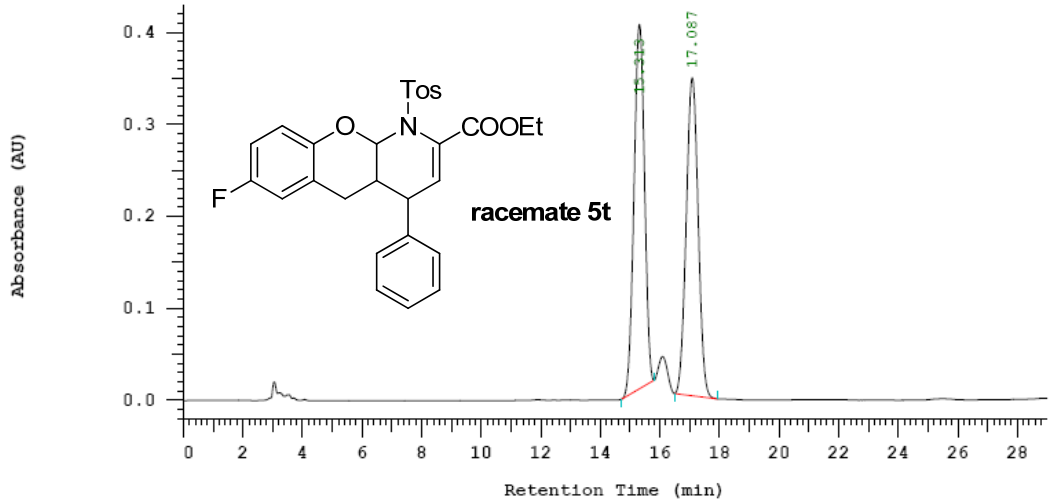
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	13.747	17627811	99.827	BB
2	15.627	30625	0.173	BB
		17658436	100.000	


8.0250
8.0084
7.3932
7.3771
7.3297
7.3151
7.2926
7.2601
7.0750
7.0618
6.8185
6.2503
6.2442
5.7392
5.7346
4.3181
4.3039
4.2897
4.2767
3.2302
3.2241
3.2080
3.2018
2.9844
2.9728
2.9498
2.9383
2.5623
2.5278
2.4621
2.3348
2.3267
2.3164
2.3045
1.3119
1.2977
1.2834
Tos
N
O
COOEt
F
5t
2.00
2.40
3.25
0.49
2.07
2.08
1.04
1.03
1.04
2.15
1.17
1.22
1.15
3.01
1.17
3.32
f1 (ppm)


164.26
158.45
156.54
148.57
144.43
139.77
136.09
129.73
128.92
128.60
119.69
118.32
118.26
115.66
115.47
114.86
114.68
82.47
77.26
77.00
76.75
61.71
40.26
35.46
28.26
21.65
14.04
Tos
N
O
COOEt
F
5t
f1 (ppm)

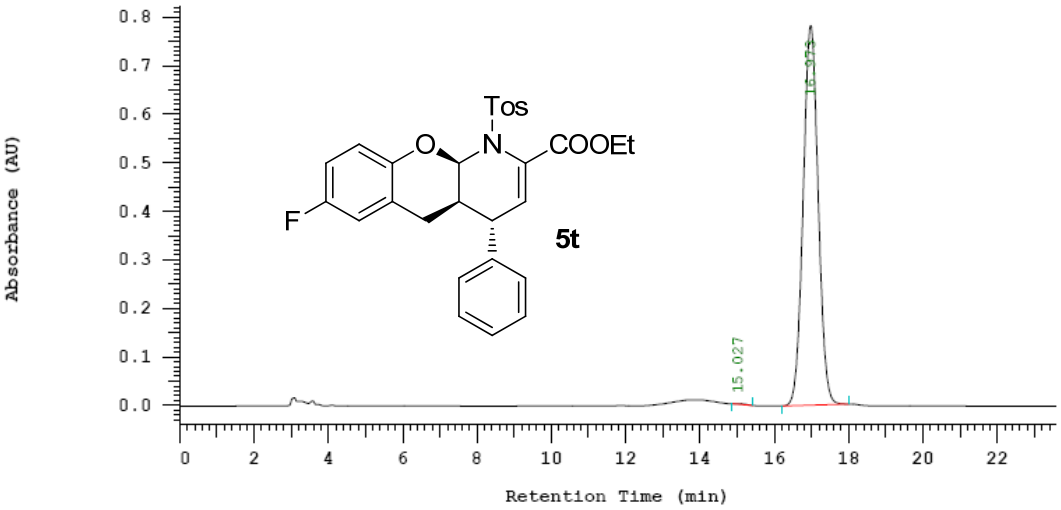
Chrom Type: Fixed WL Chromatogram, 210 nm



Chrom Type: Fixed WL Chromatogram, 210 nm
Peak Quantitation: AREA
Calculation Method: AREA%

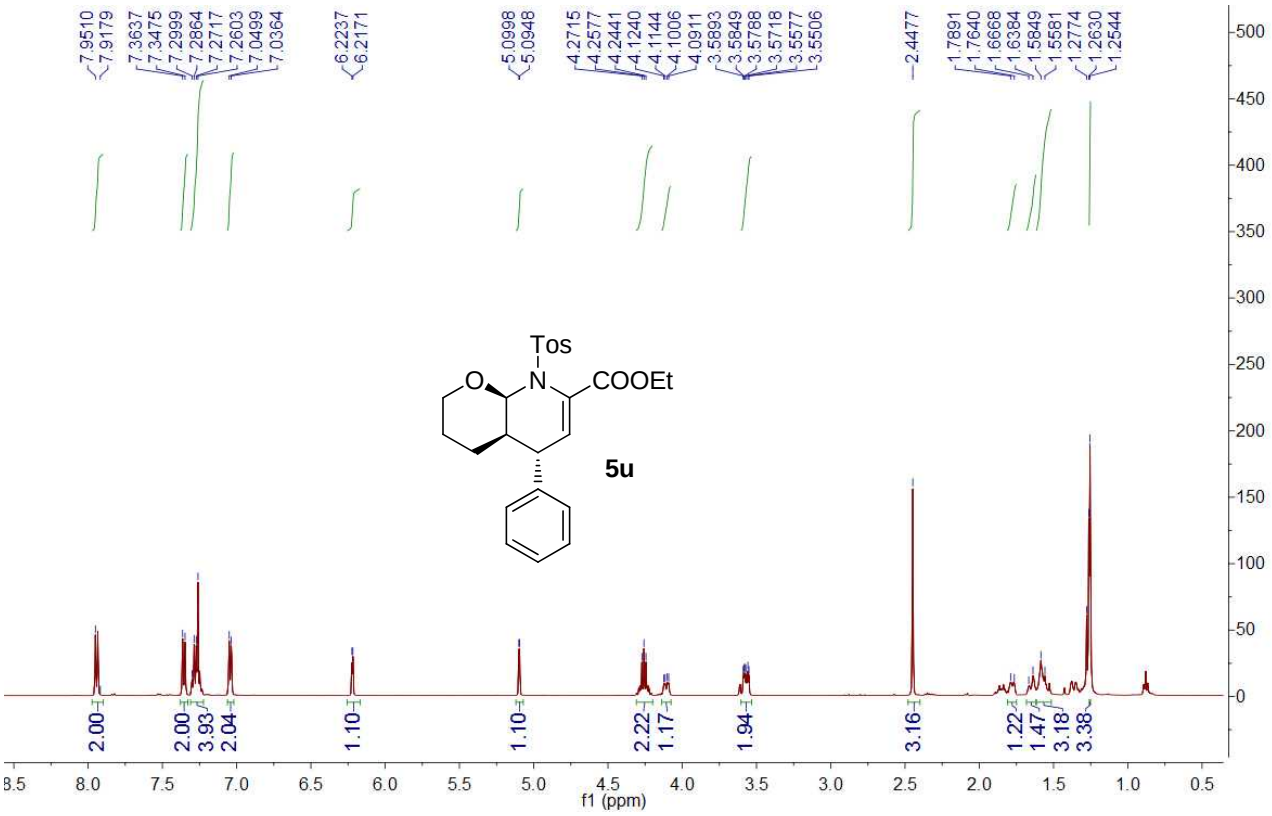
No.	RT	Area	Area %	BC
1	15.313	4801154	50.118	BB
2	17.087	4778492	49.882	BB
		9579646	100.000	

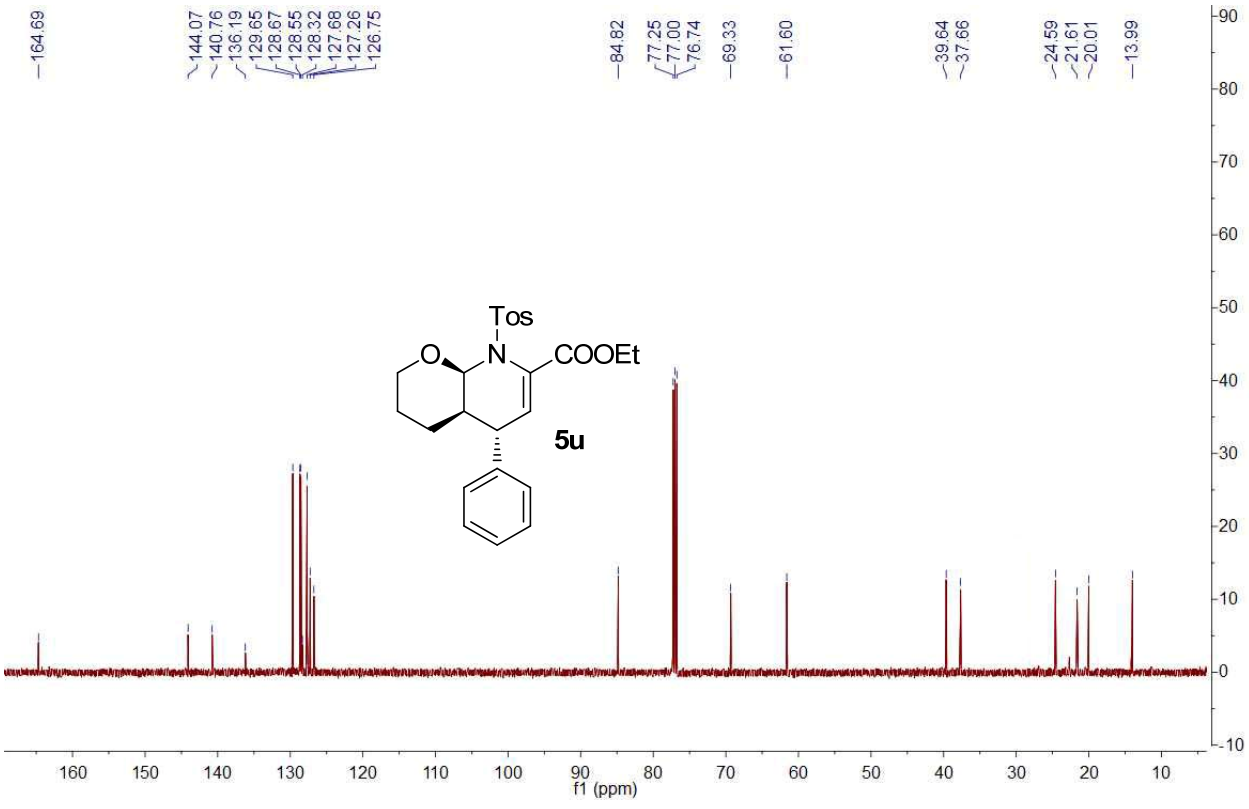
Chrom Type: Fixed WL Chromatogram, 210 nm



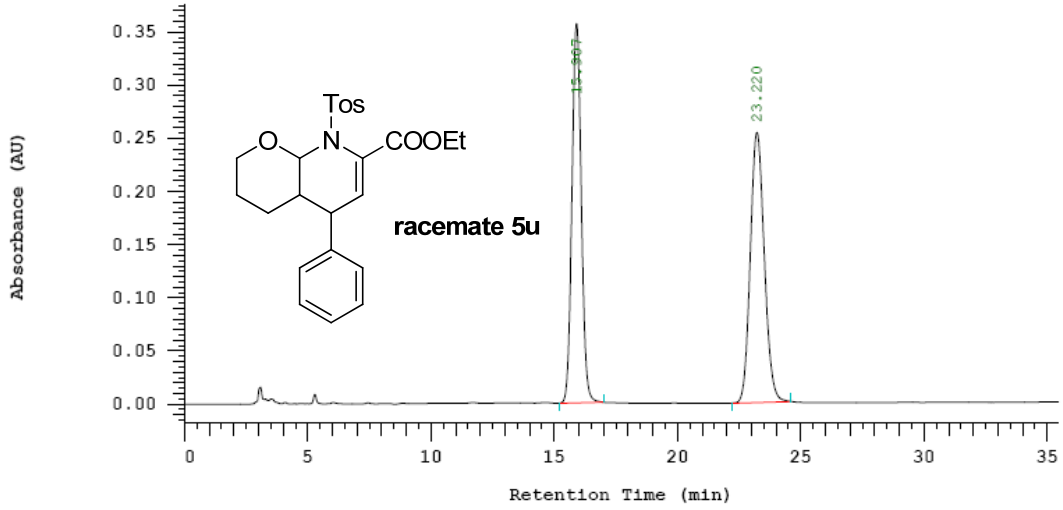
Chrom Type: Fixed WL Chromatogram, 210 nm
Peak Quantitation: AREA
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	15.027	13988	0.127	BB
2	16.973	10981186	99.873	BB
		10995174	100.000	


7.9510
7.9179
7.3637
7.3475
7.2999
7.2864
7.2717
7.2603
7.0499
7.0364
6.2237
6.2171
5.0998
5.0948
4.2715
4.2577
4.2441
4.1240
4.1144
4.1006
4.0911
3.5893
3.5849
3.5788
3.5718
3.5577
3.5506
2.4477
1.7891
1.7640
1.6668
1.6384
1.5849
1.5581
1.2774
1.2630
1.2544
Tos
O
N
COOEt
5u
2.00
2.00
3.93
2.04
1.10
1.10
2.22
1.17
1.94
3.16
1.22
1.47
3.18
3.38
f1 (ppm)


164.69
144.07
140.76
136.19
129.65
128.67
128.55
128.32
127.88
127.26
126.75
84.82
77.25
77.00
76.74
69.33
61.60
39.64
37.66
24.59
21.61
20.01
13.99
Tos
O
N
COOEt
5u
f1 (ppm)

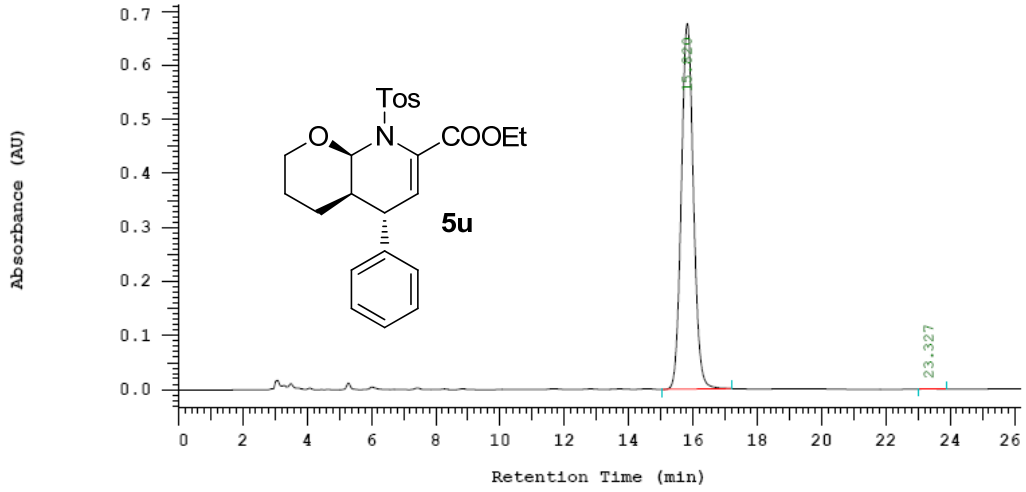
Chrom Type: Fixed WL Chromatogram, 210 nm



Chrom Type: Fixed WL Chromatogram, 210 nm
Peak Quantitation: AREA
Calculation Method: AREA%

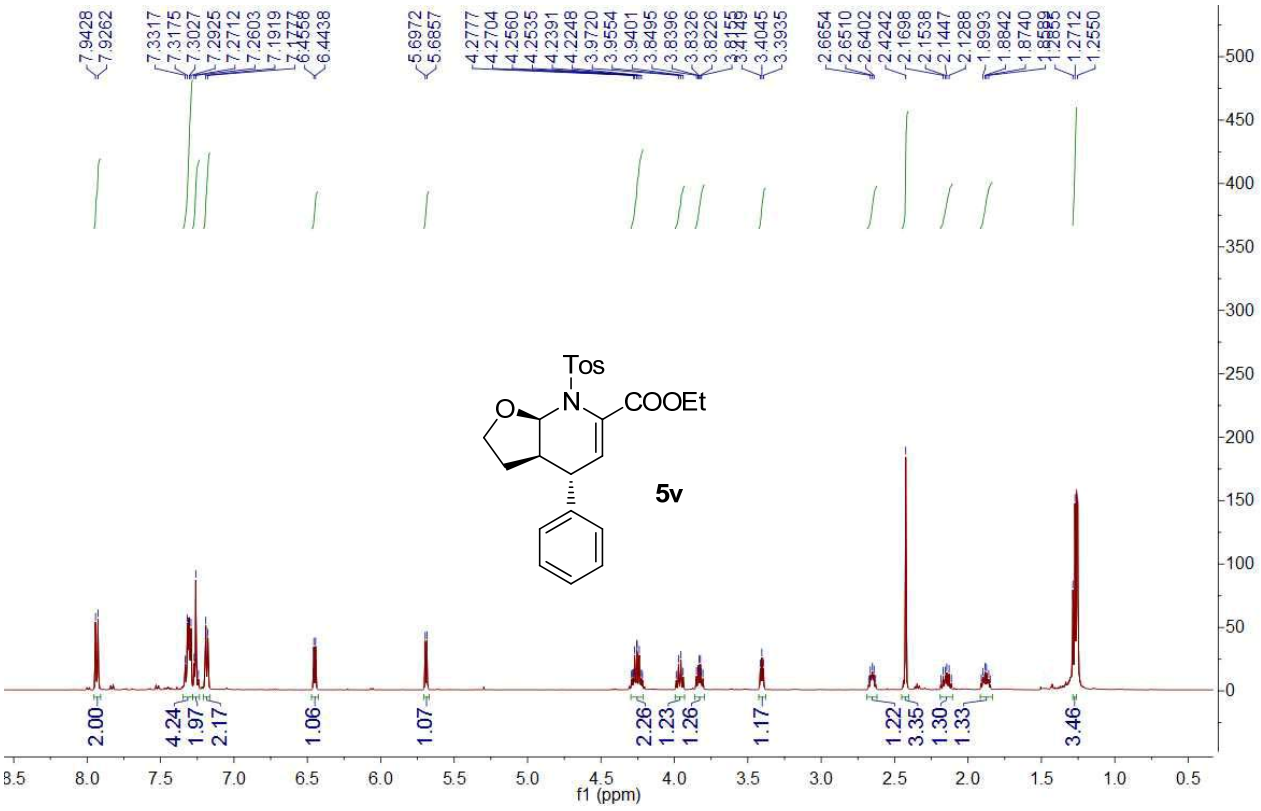
No.	RT	Area	Area %	BC
1	15.907	4659355	48.376	BB
2	23.220	4972233	51.624	BB
		9631588	100.000	

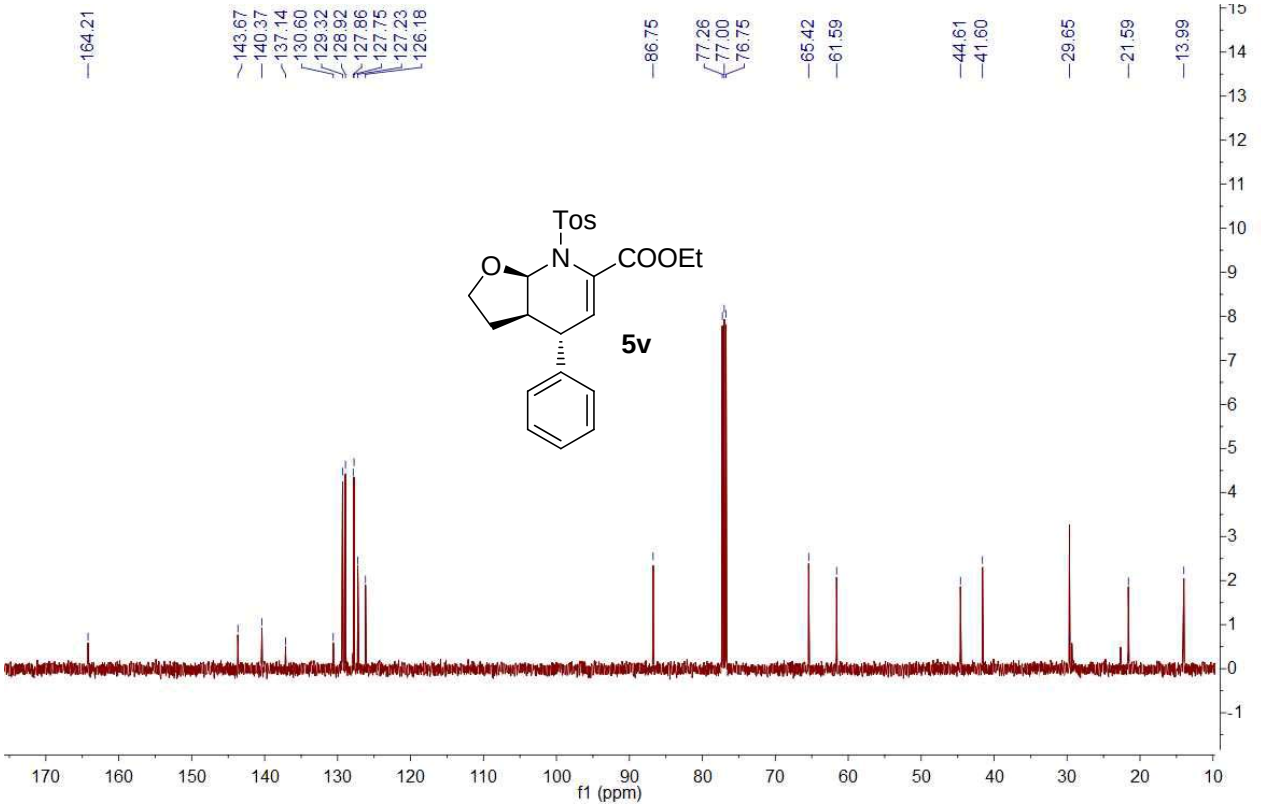
Chrom Type: Fixed WL Chromatogram, 210 nm



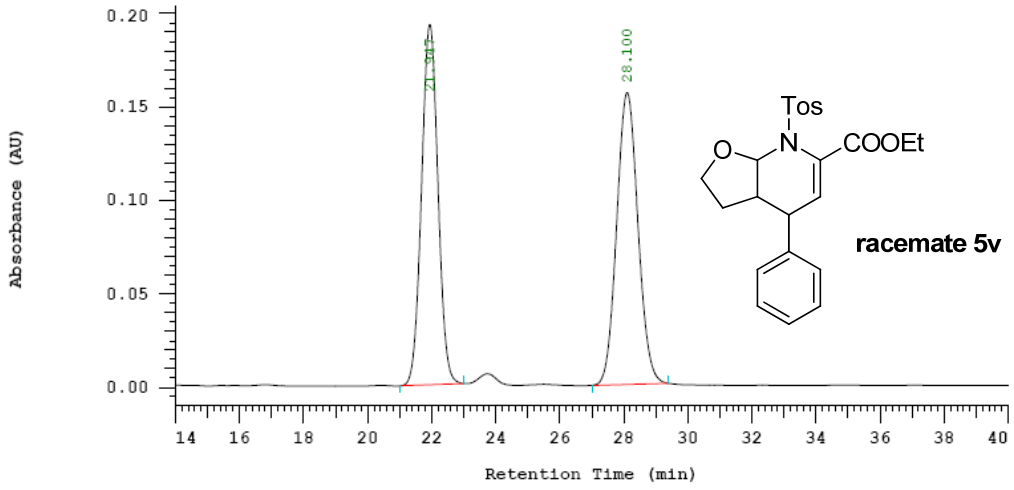
Chrom Type: Fixed WL Chromatogram, 210 nm
Peak Quantitation: AREA
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	15.820	8851317	99.924	BB
2	23.327	6772	0.076	BB
		8858089	100.000	


Tos
N
O
COOEt
5v
f1 (ppm)


Tos
N
O
COOEt
5v
f1 (ppm)

Chrom Type: Fixed WL Chromatogram, 210 nm



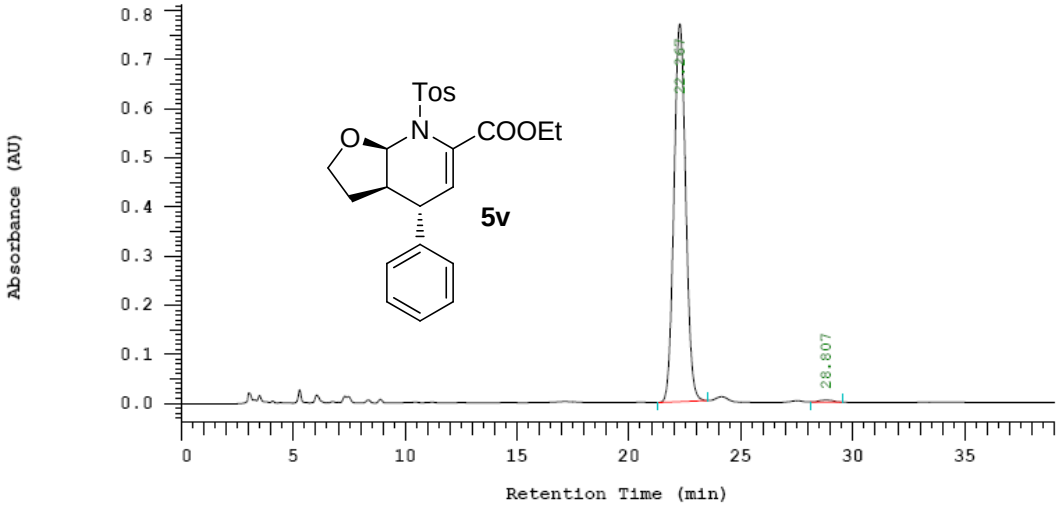
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	21.947	3405600	48.662	BB
2	28.100	3592918	51.338	BB
		6998518	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

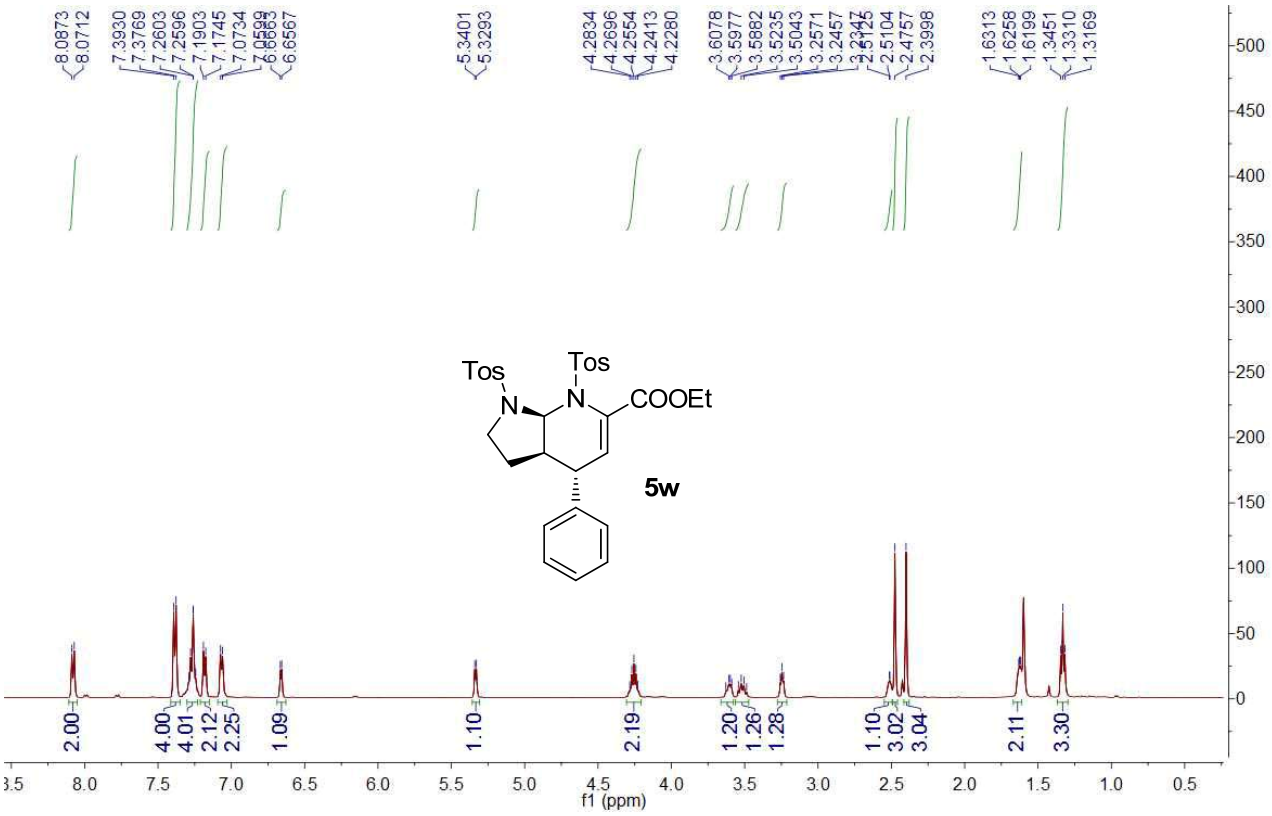


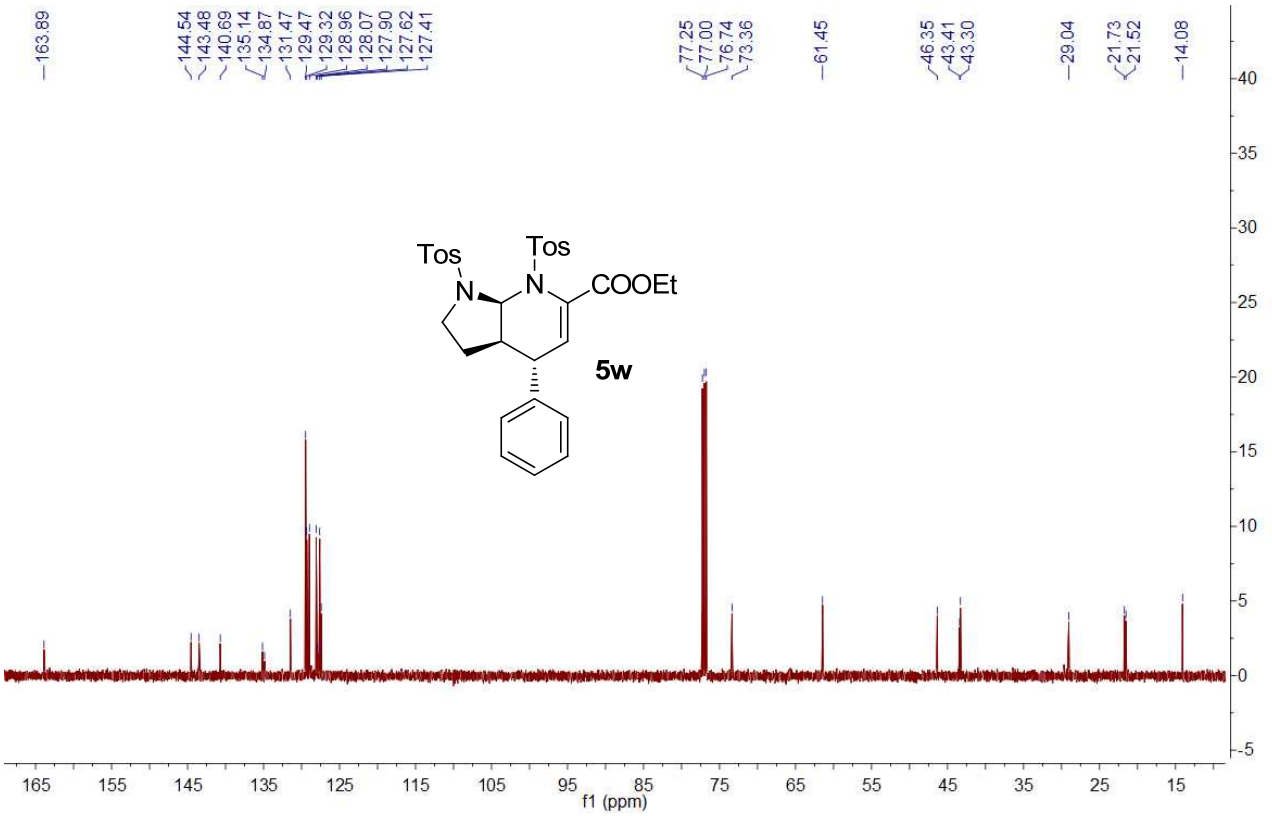
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

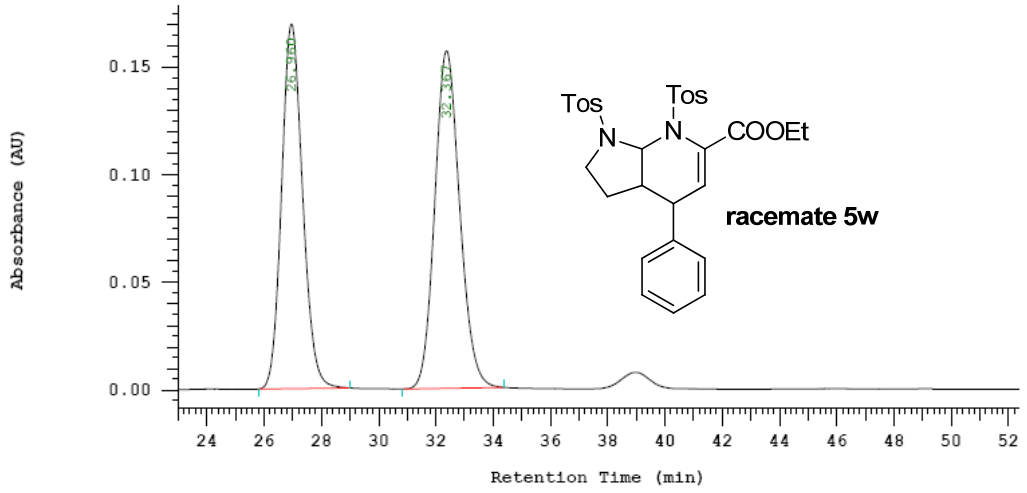
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	22.267	13867534	99.332	BB
2	28.807	93303	0.668	BB
		13960837	100.000	


8.0873
8.0712
7.3930
7.3769
7.2603
7.2596
7.1903
7.1745
7.0734
6.6567
5.3401
5.3293
4.2834
4.2696
4.2554
4.2413
4.2280
3.6078
3.5977
3.5882
3.5235
3.5043
3.2571
3.2457
2.5104
2.4757
2.3998
1.6313
1.6258
1.6199
1.3451
1.3310
1.3169
Tos
Tos
N
N
COOEt
5w
2.00
4.00
4.01
2.12
2.25
1.09
1.10
2.19
1.20
1.26
1.28
1.10
3.02
3.04
2.11
3.30
f1 (ppm)


163.89
144.54
143.48
140.69
135.14
134.87
131.47
129.47
129.32
128.96
128.07
127.90
127.62
127.41
77.25
77.00
76.74
73.36
61.45
46.35
43.41
43.30
29.04
21.73
21.52
14.08
Tos
Tos
N
N
COOEt
5w
f1 (ppm)

Chrom Type: Fixed WL Chromatogram, 210 nm



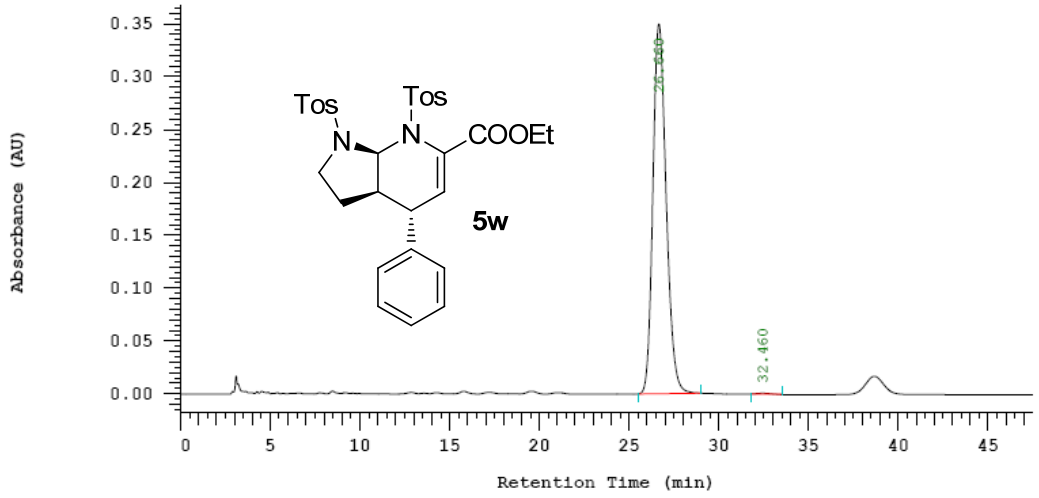
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	26.960	4291801	47.512	BB
2	32.367	4741311	52.488	BB
		9033112	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

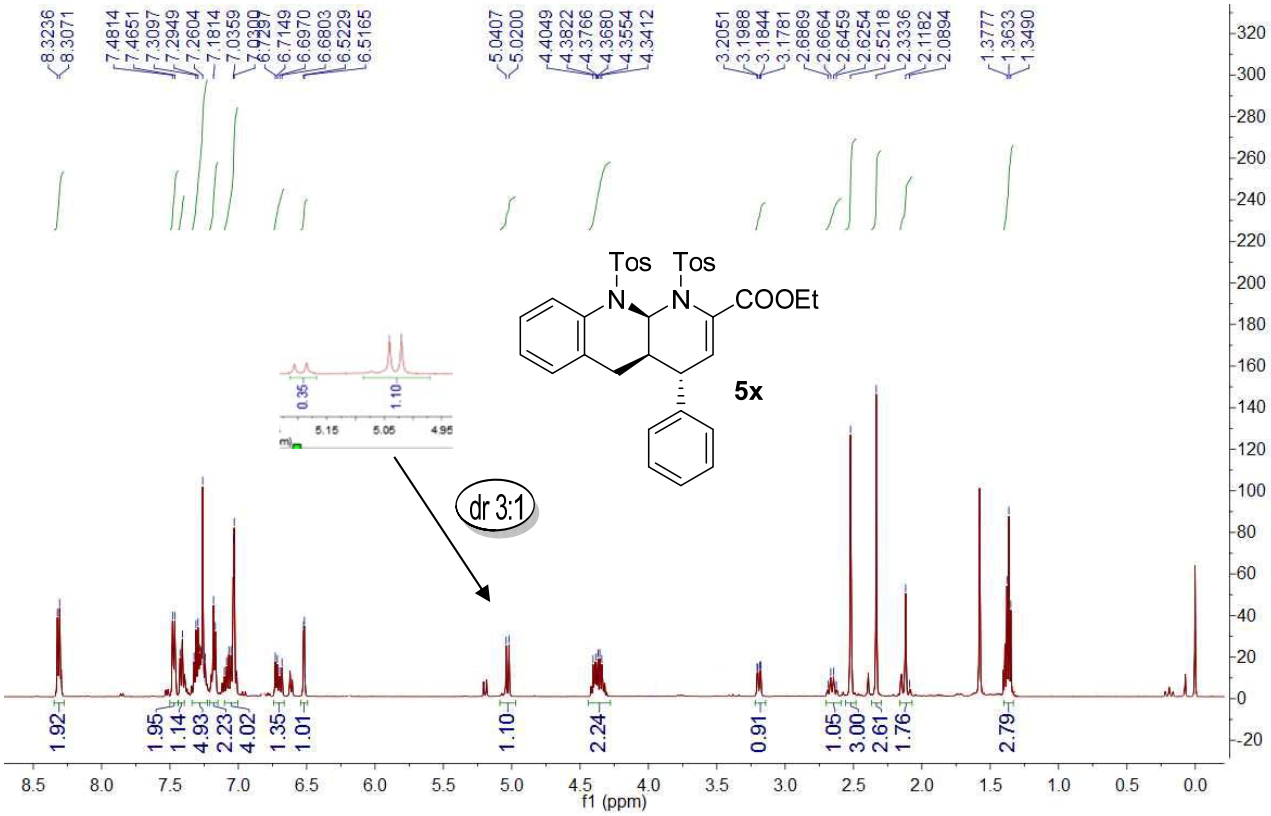


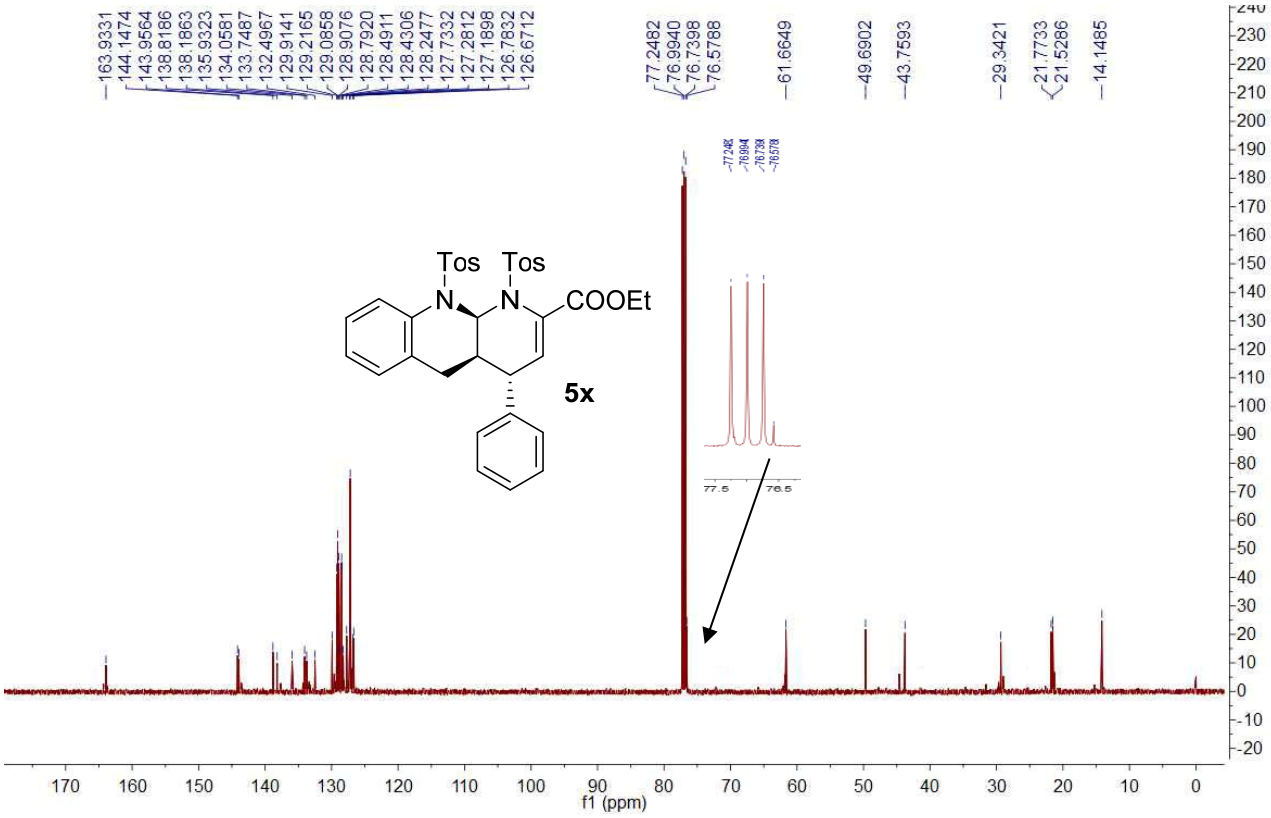
Chrom Type: Fixed WL Chromatogram, 210 nm

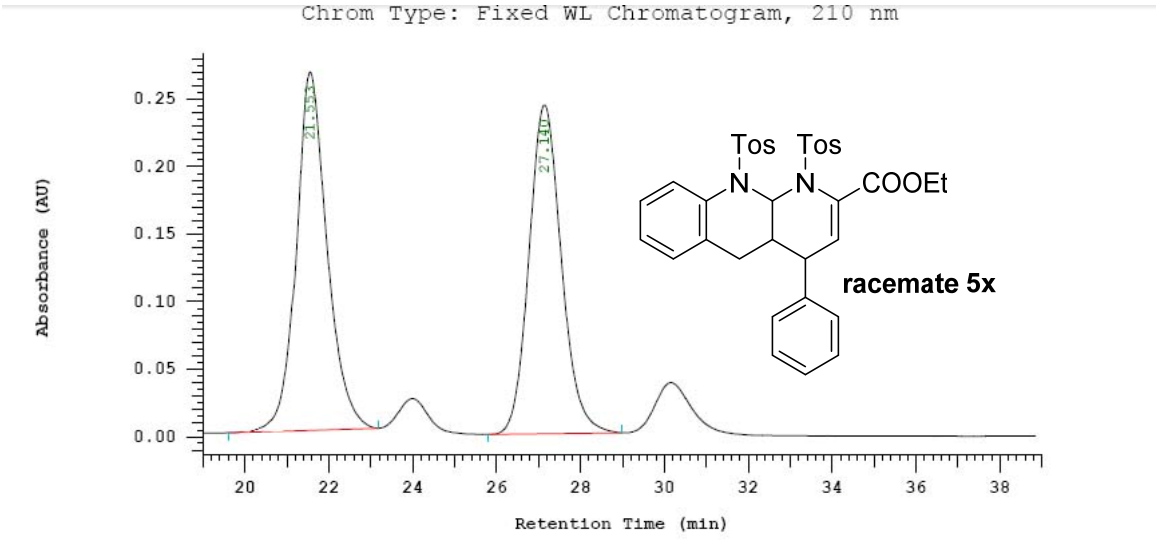
Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	26.660	8913444	99.799	BB
2	32.460	17927	0.201	BB
		8931371	100.000	


Tos
Tos
N
N
COOEt
5x
dr 3:1
f1 (ppm)


Tos
Tos
N
N
COOEt
5x
f1 (ppm)

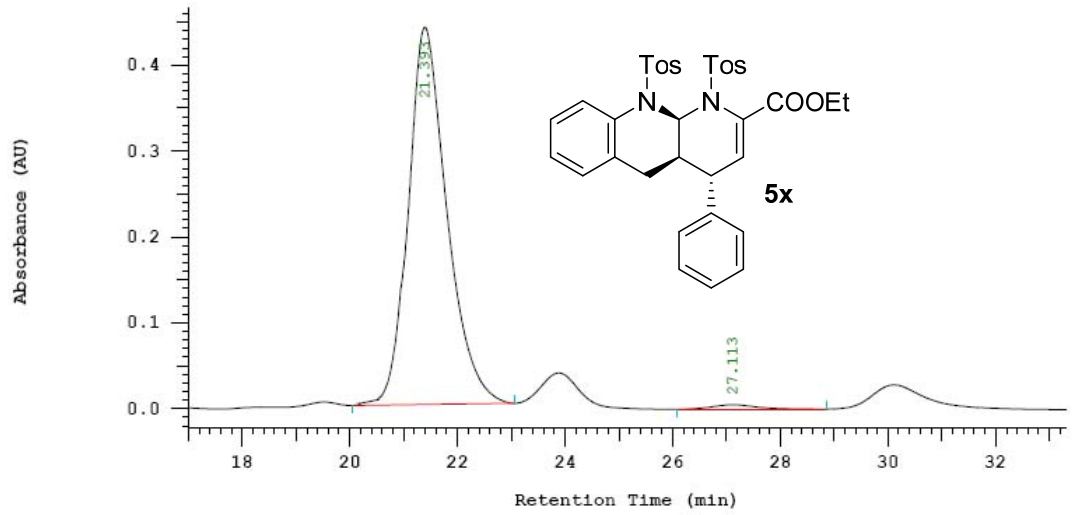


Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	21.553	7260308	52.697	BB
2	27.140	6517057	47.303	BB
		13777365	100.000	

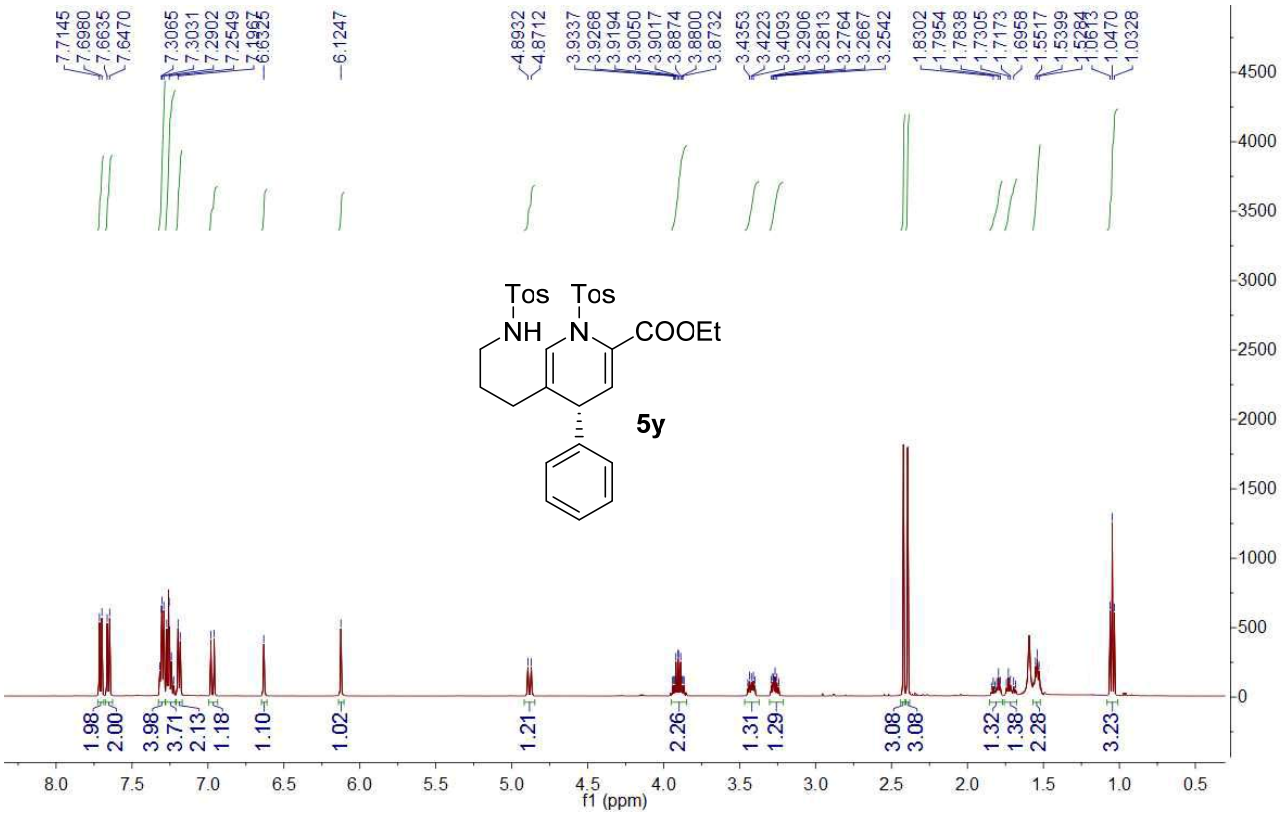


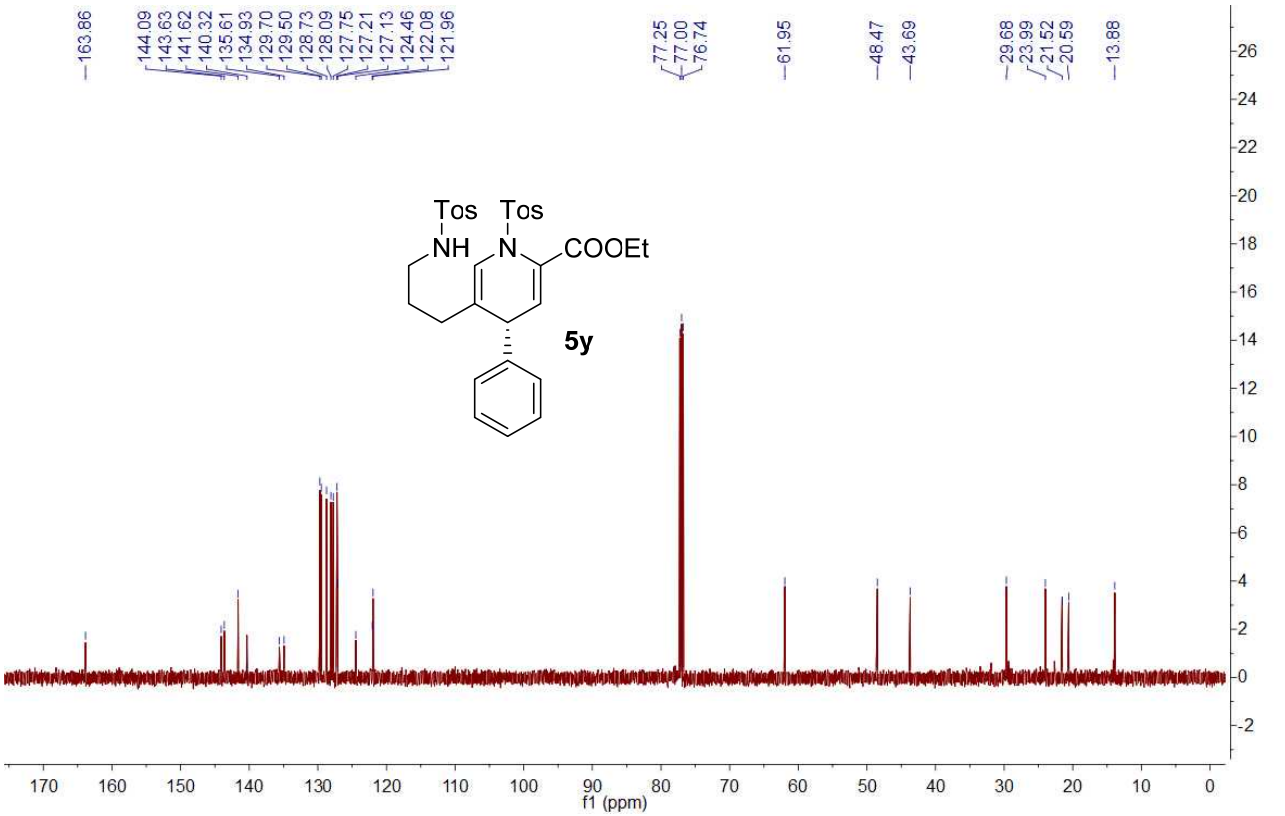
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

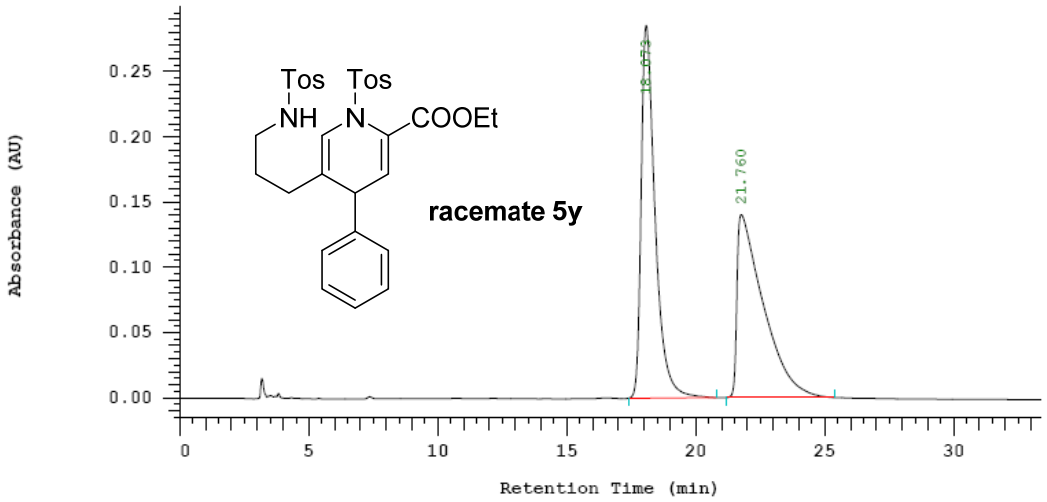
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	21.393	11656993	98.816	BB
2	27.113	139633	1.184	BB
		11796626	100.000	


7.7145
7.6980
7.6635
7.6470
7.3065
7.3031
7.2902
7.2549
6.6325
6.1247
4.8932
4.8712
3.9337
3.9268
3.9194
3.9050
3.9017
3.8874
3.8800
3.8732
3.4353
3.4223
3.4093
3.2906
3.2813
3.2764
3.2667
3.2542
1.8302
1.7954
1.7838
1.7305
1.7173
1.6958
1.5517
1.5399
1.0470
1.0328
Tos
Tos
NH
N
COOEt
5y
1.98
2.00
3.98
3.71
2.13
1.18
1.10
1.02
1.21
2.26
1.31
1.29
3.08
3.08
1.32
1.38
2.28
3.23
f1 (ppm)


163.86
144.09
143.63
141.62
140.32
135.61
134.93
129.70
129.50
128.73
128.09
127.75
127.21
127.13
124.46
122.08
121.96
77.25
77.00
76.74
61.95
48.47
43.69
29.68
23.99
21.52
20.59
13.88
Tos
Tos
NH
N
COOEt
5y
f1 (ppm)

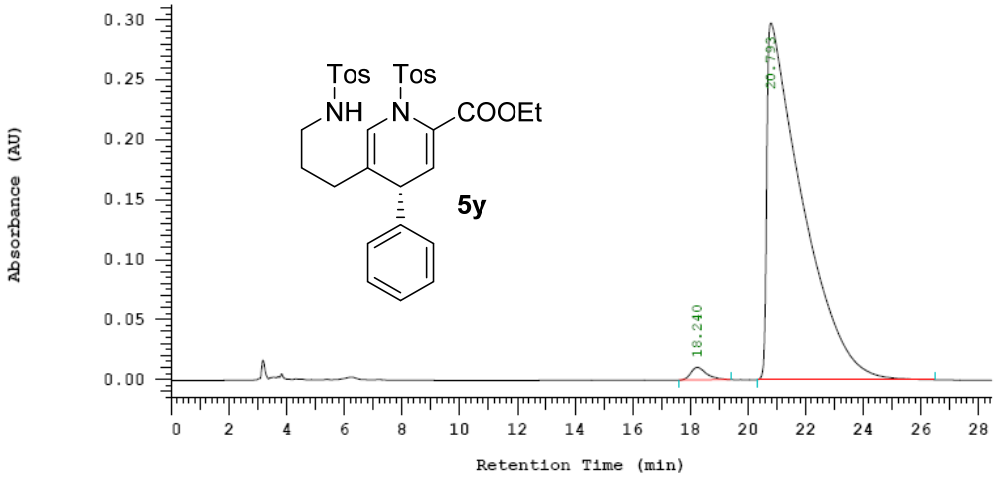
Chrom Type: Fixed WL Chromatogram, 210 nm



Chrom Type: Fixed WL Chromatogram, 210 nm
Peak Quantitation: AREA
Calculation Method: AREA%

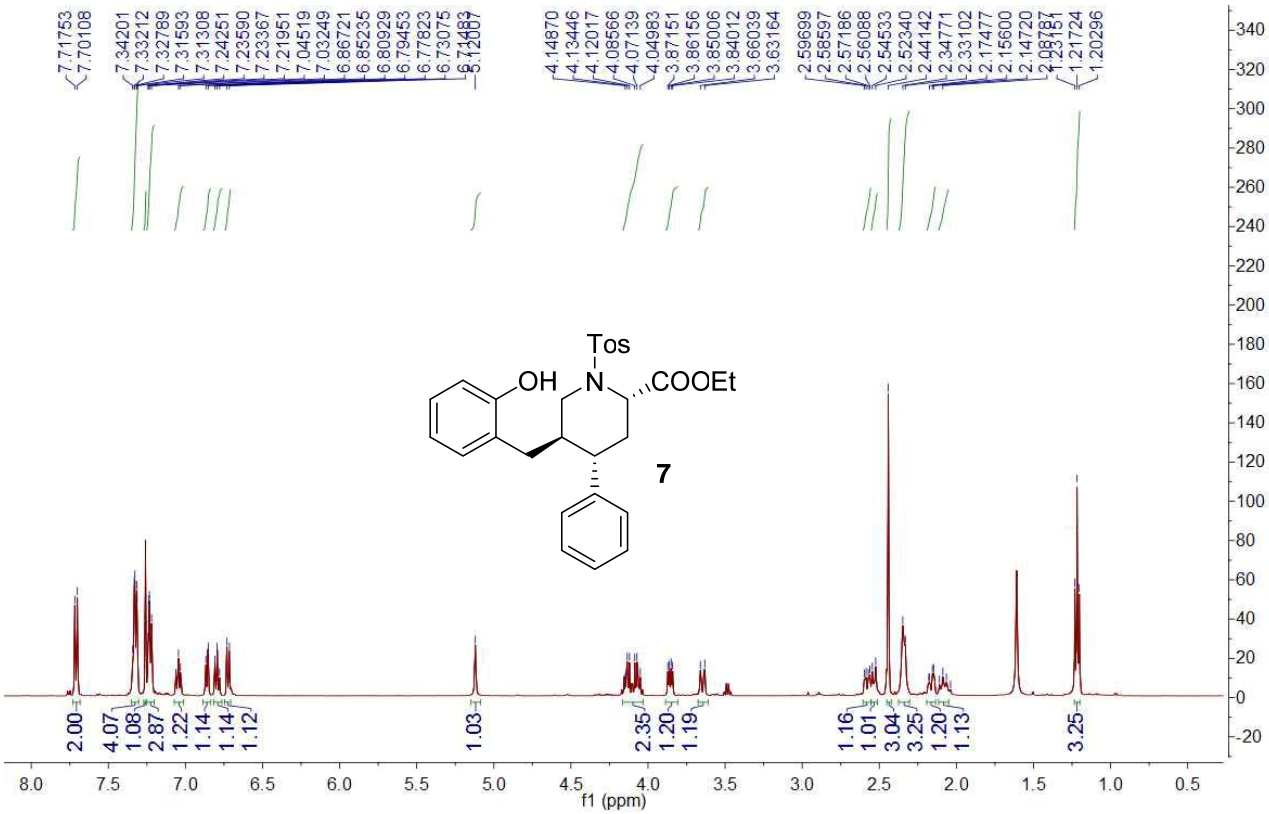
No.	RT	Area	Area %	BC
1	18.073	5323243	51.691	BB
2	21.760	4974977	48.309	BB
		10298220	100.000	

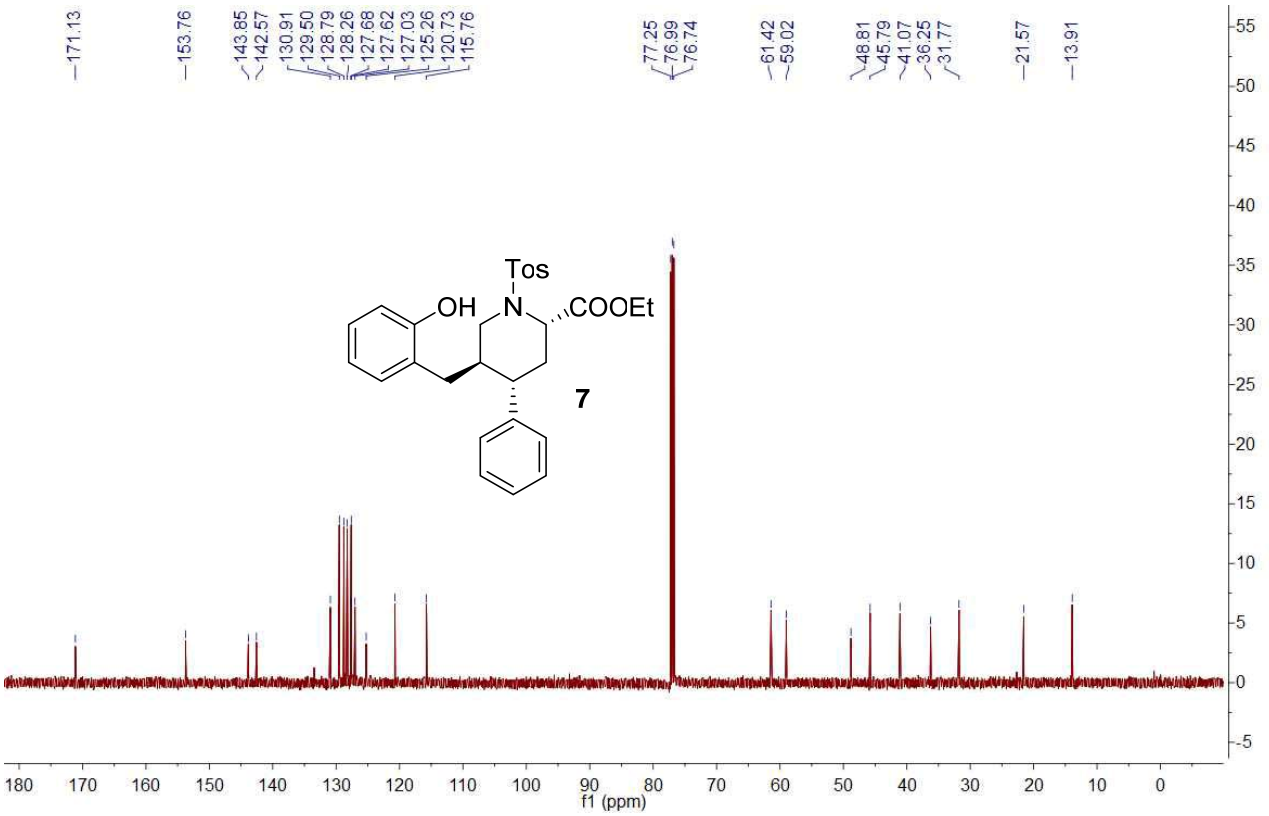
Chrom Type: Fixed WL Chromatogram, 210 nm

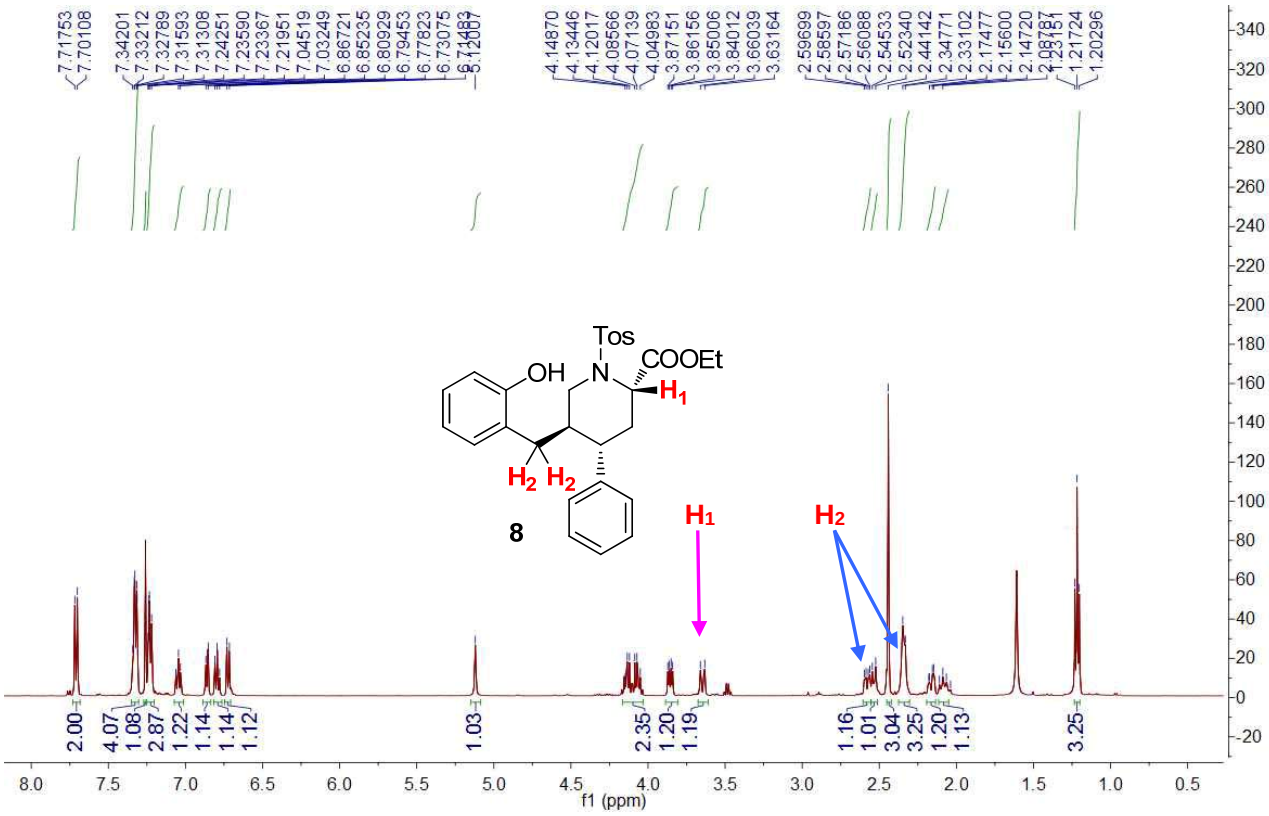


Chrom Type: Fixed WL Chromatogram, 210 nm
Peak Quantitation: AREA
Calculation Method: AREA%

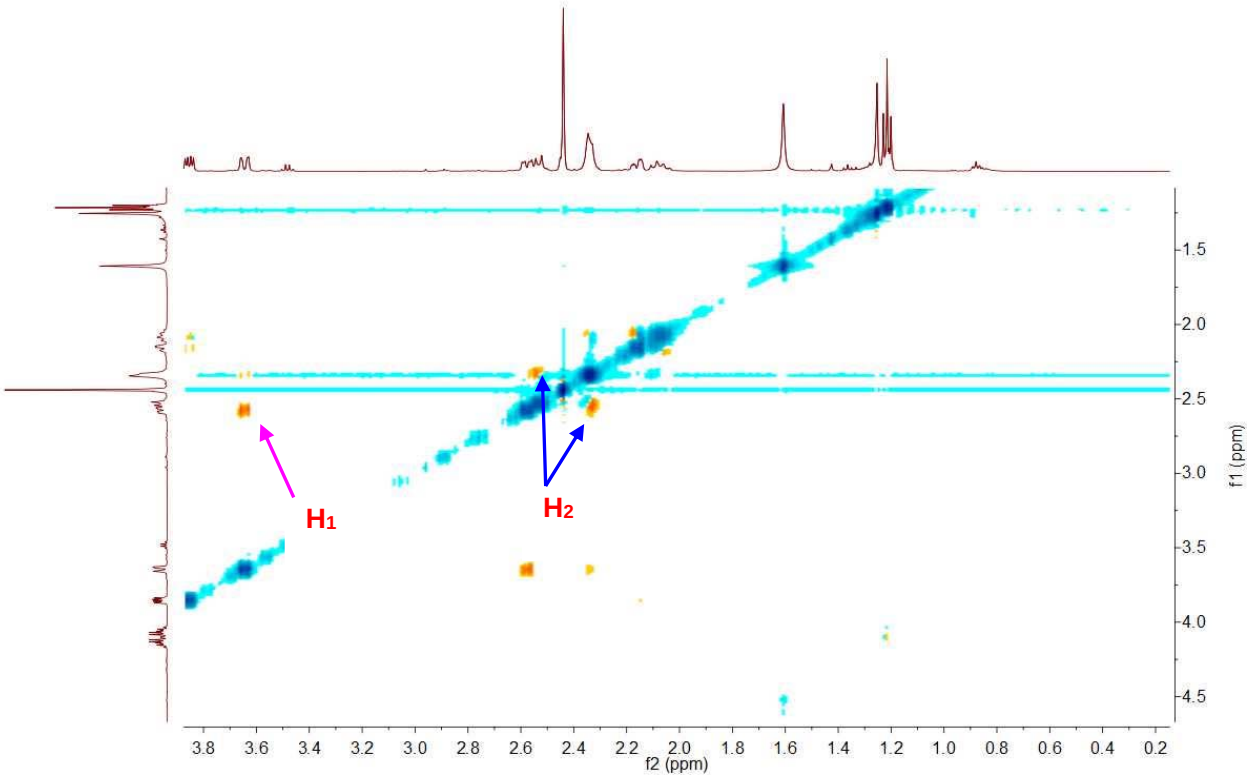
No.	RT	Area	Area %	BC
1	18.240	198817	1.542	BB
2	20.793	12698749	98.458	BB
		12897566	100.000	


Tos
OH
N
COOEt
7
f1 (ppm)

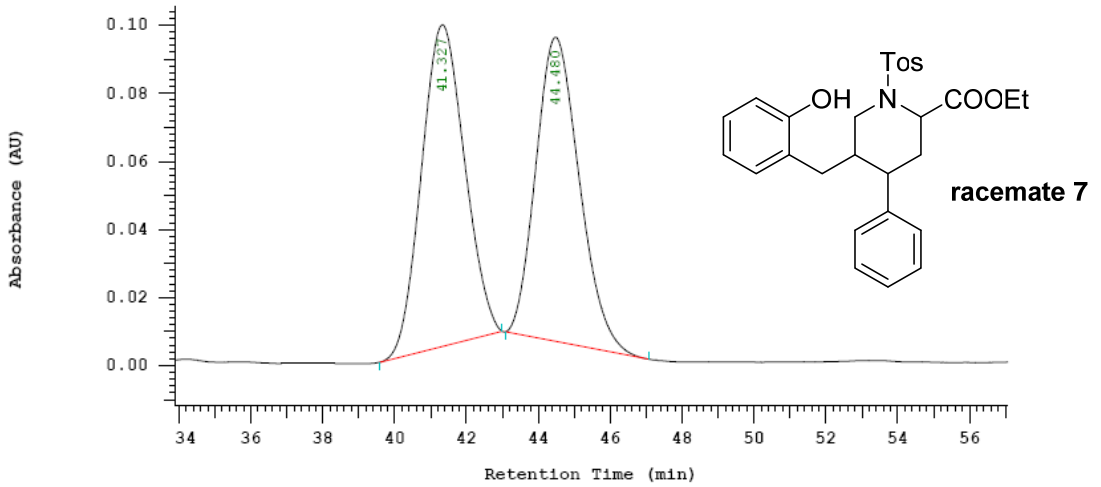

Tos
OH
N
COOEt
7
f1 (ppm)



$CDCl_3$-NOESY-7



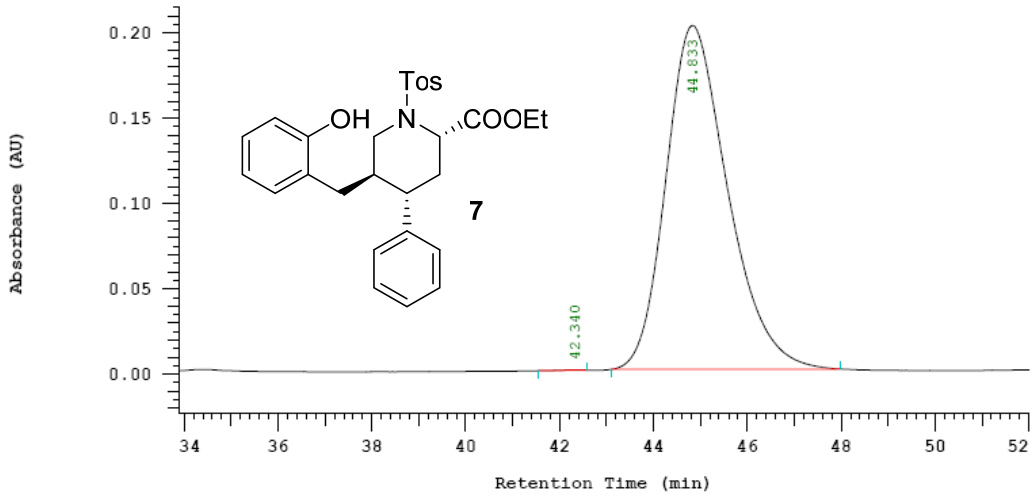
Chrom Type: Fixed WL Chromatogram, 210 nm



Chrom Type: Fixed WL Chromatogram, 210 nm
Peak Quantitation: AREA
Calculation Method: AREA%

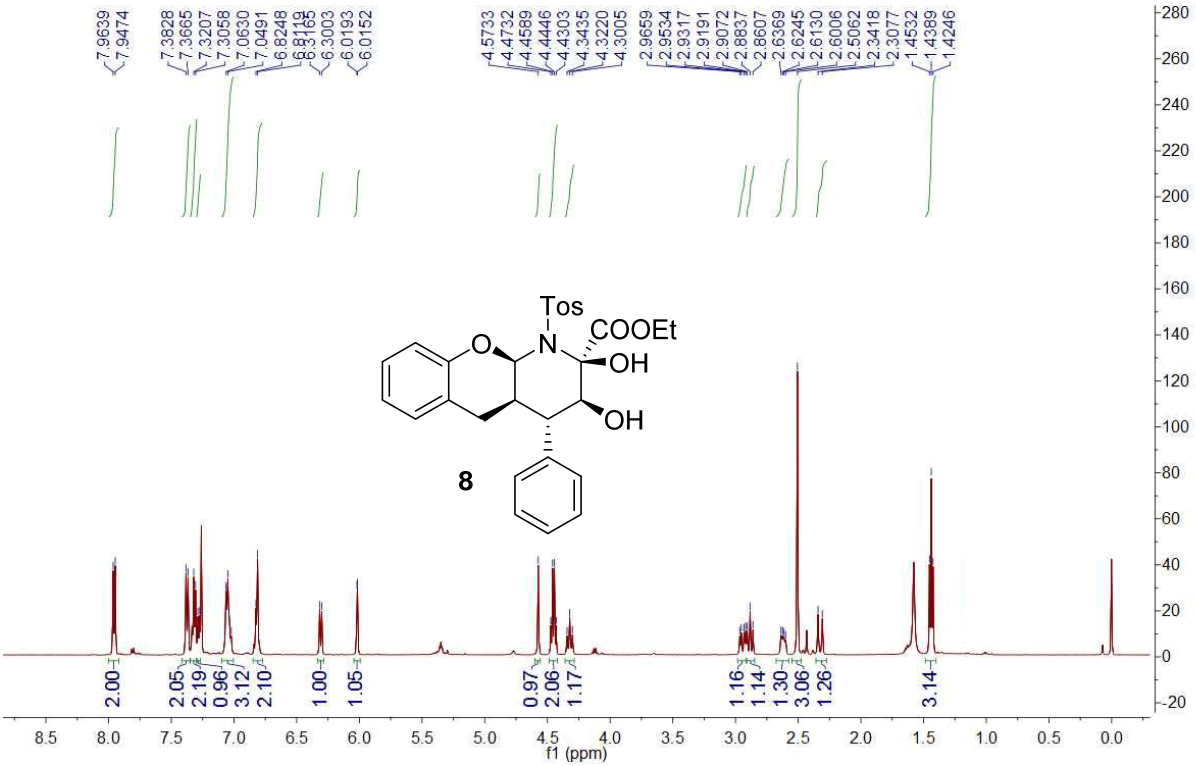
No.	RT	Area	Area %	BC
1	41.327	3926928	51.119	BB
2	44.480	3754985	48.881	BB
		7681913	100.000	

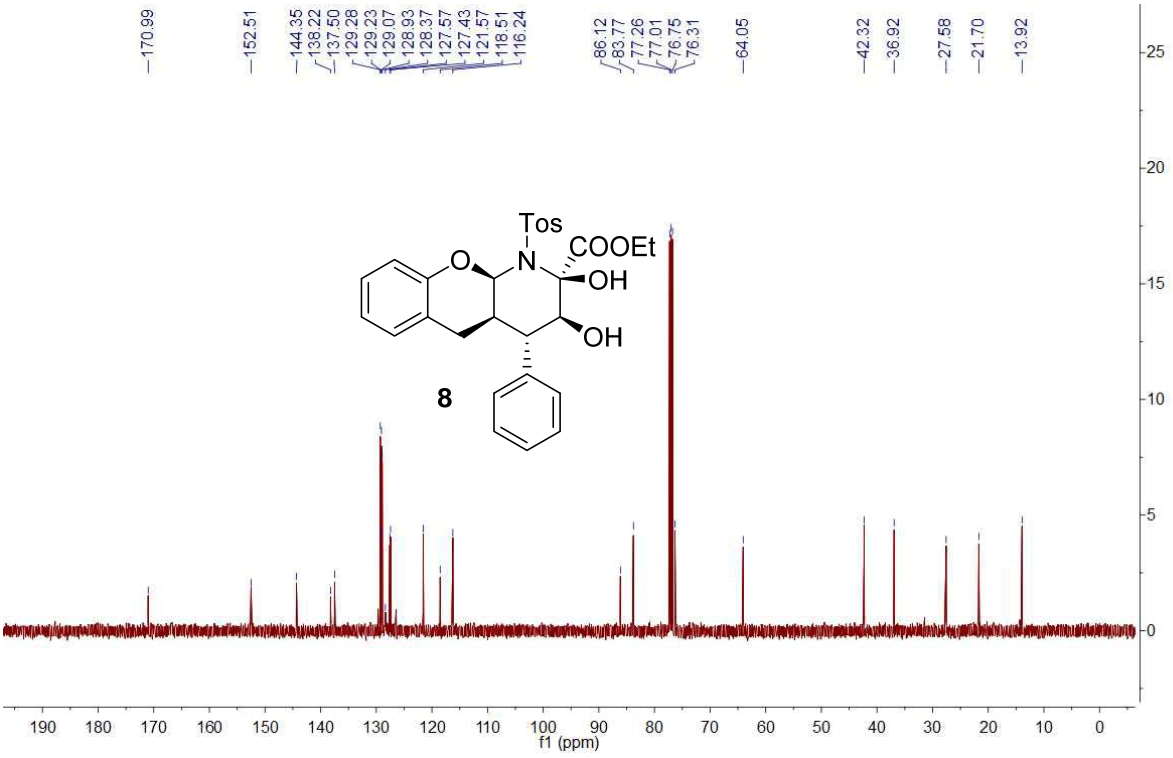
Chrom Type: Fixed WL Chromatogram, 210 nm



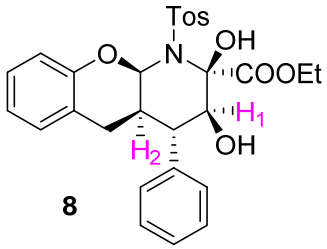
Chrom Type: Fixed WL Chromatogram, 210 nm
Peak Quantitation: AREA
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	42.340	4196	0.045	BB
2	44.833	9229070	99.955	BB
		9233266	100.000	

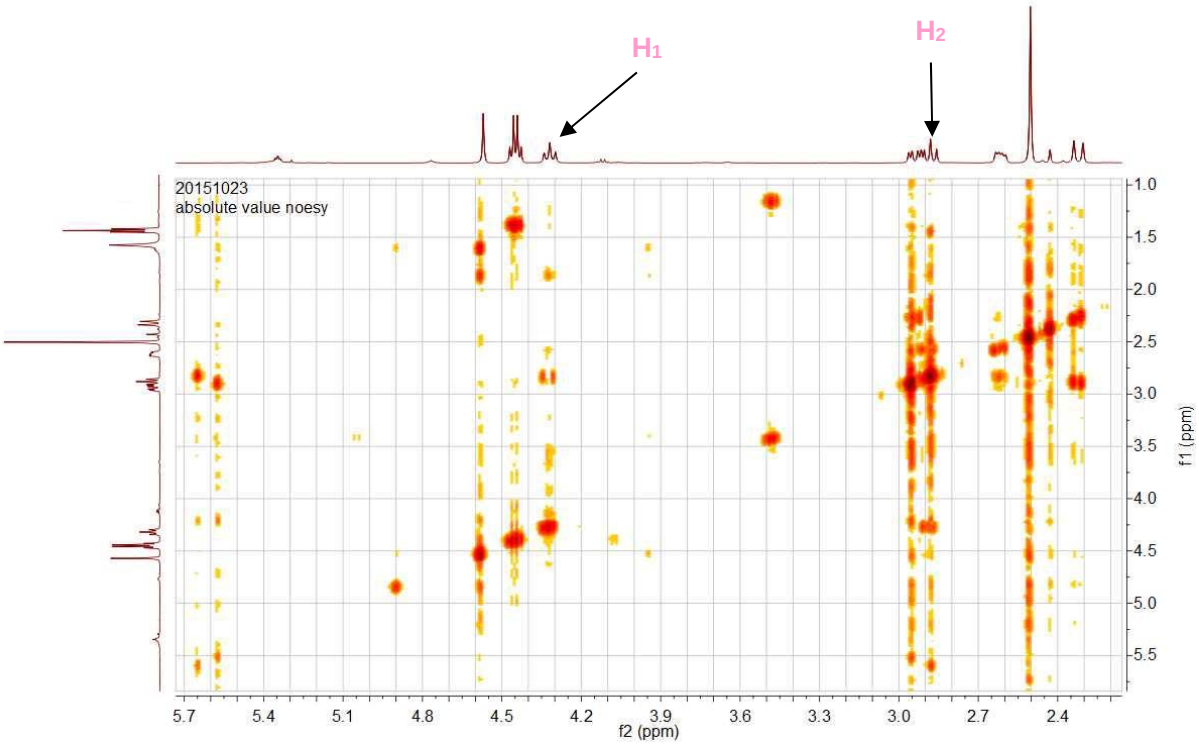

Tos
COOEt
OH
OH
O
N
8
f1 (ppm)


Tos
COOEt
OH
OH
O
N
8
f1 (ppm)

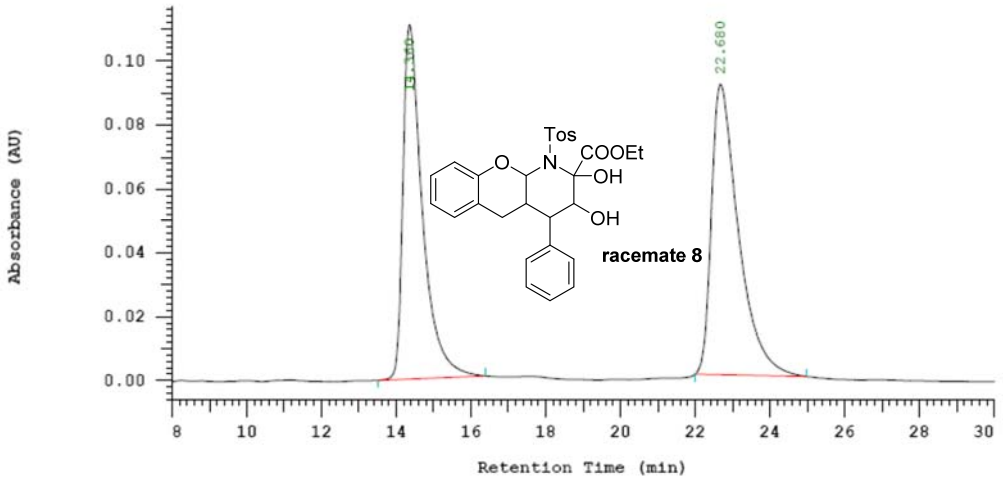
$CDCl_3$-NOESY-9



8



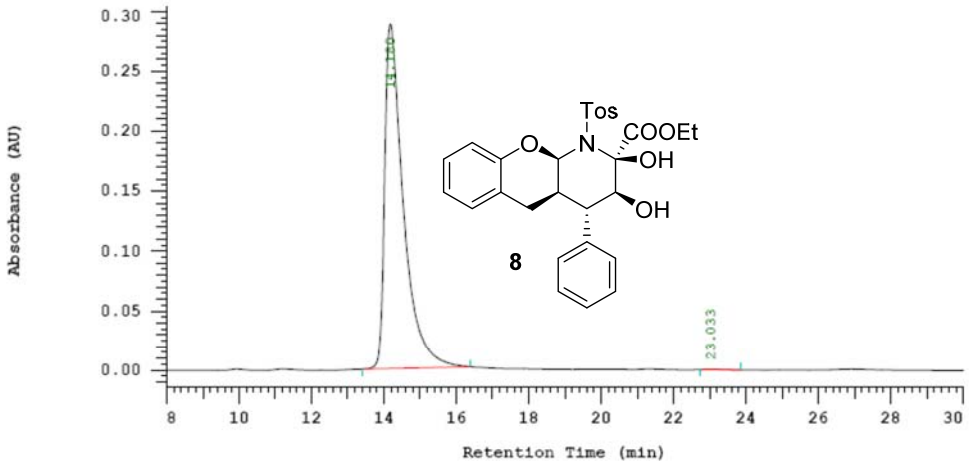
Chrom Type: Fixed WL Chromatogram, 231 nm



Chrom Type: Fixed WL Chromatogram, 231 nm
Peak Quantitation: AREA
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	14.360	1994165	46.575	BB
2	22.680	2287447	53.425	BB
		4281612	100.000	

Chrom Type: Fixed WL Chromatogram, 231 nm



Chrom Type: Fixed WL Chromatogram, 231 nm
Peak Quantitation: AREA
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	14.180	5173986	99.910	BB
2	23.033	4666	0.090	BB
		5178652	100.000	