Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Supporting Information

NHC-Catalyzed [4+2] Annulation of 2-Bromo-2-Enal with Acylhydrazones: Enantioselective Synthesis of *δ*-Lactams

Baomin Sun,[‡a] Lijiu Gao,[‡a] Shide Shen,[b] Chenxia Yu,[a] Tuanjie Li,[a] Yuanwei Xie[a] and Changsheng Yao[*a]

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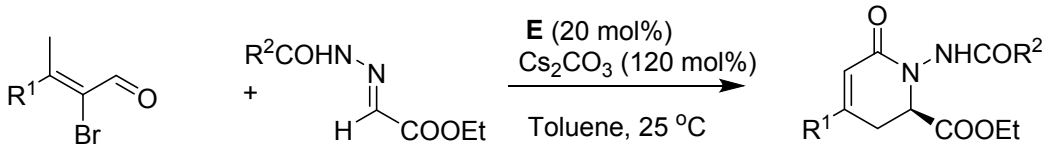
1. General methods

Common reagents and materials were purchased from commercial sources and purified by recrystallization or distillation. Melting points were determined in open capillaries and were uncorrected. IR spectra were taken on a FT-IR-Tensor 27 spectrometer in KBr pellets and reported in cm^{-1}. ^{1}H NMR spectra were measured on a Bruker DPX 400 MHz spectrometer in DMSO-d_6(100 MHz, ^{13}C NMR) or $CDCl_3$ with chemical shift (δ) given in ppm relative to TMS as internal standard. High-resolution mass spectra (HRMS) were obtained on a micrOTOF-QⅡ HRMS/MS instrument (Bruker) with the technique of electrospray ionization. Optical rotation values were measured with instruments operating at λ = 589 nm, corresponding to the sodium D line at the temperatures indicated.

2. Abstract

An asymmetric assembly of δ-lactam was realized via the NHC-catalyzed formal [4+2] annulation of acylhydrazones and 2-bromo-2-enals bearing γ-H. The advantages of this protocol include high enantioselectivity, good yields, mild reaction condition and potential biological significance of the final products.

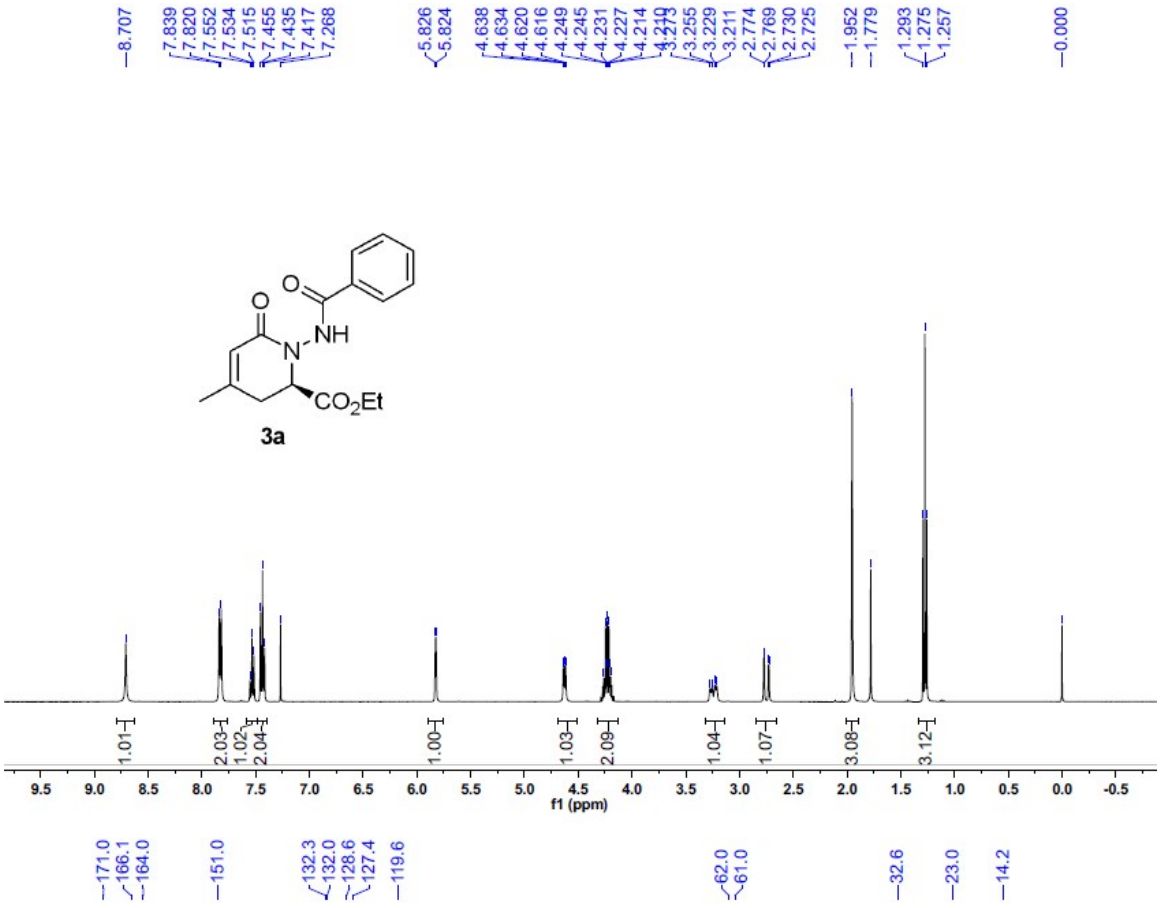
3. Experimental section



1 (2.25 equiv.) **2** **3**

An oven-dried 10-mL Schlenk tube equipped with a magnetic stir bar was charged with triazolium salt **E** (16.7 mg, 0.04 mmol), Cs_2CO_3 (78.2 mg, 0.24 mmol), α-bromo-α,β-unsaturated aldehyde **1** (0.45 mmol) and hydrazone **2** (0.2 mmol). The tube was closed with a septum. And the freshly distilled toluene (3 mL) was added into the mixture with a syringe. Then the mixture was stirred at 25 ^{o}C until completion (monitored by TLC). After removal of the solvent under reduced pressure, the resulting crude product was purified by column chromatography (silicagel, mixtures of petroleum ether/ethyl acetate, 2:1, v/v) to afford the desired product **3**.

4. NMR Spectra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4.634
4.620
4.616
4.249
4.245
4.231
4.227
4.214
3.255
3.229
3.211
2.774
2.769
2.730
2.725
1.952
1.779
1.293
1.275
1.257
0.000
O
O
NH
N
CO2Et
3a
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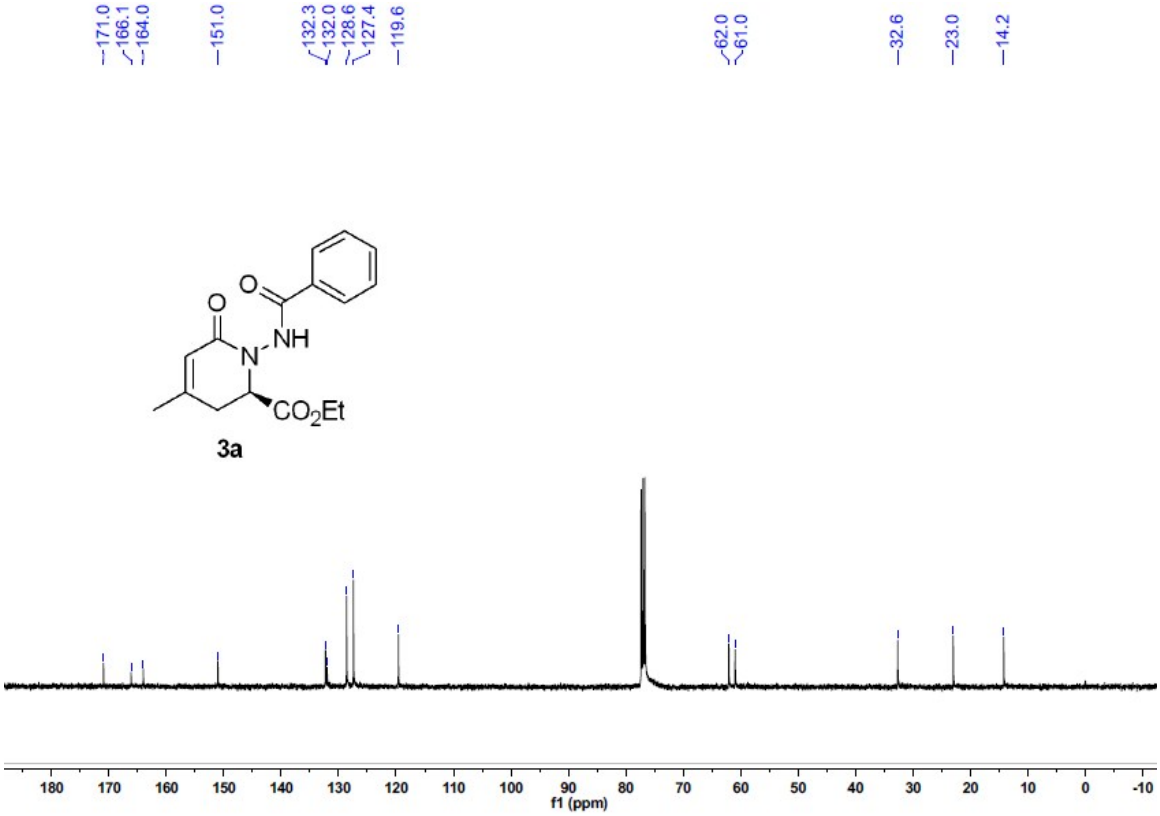
1.02
2.04
1.00
1.03
2.09
1.04
1.07
3.08
3.12
f1 (ppm)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127.4
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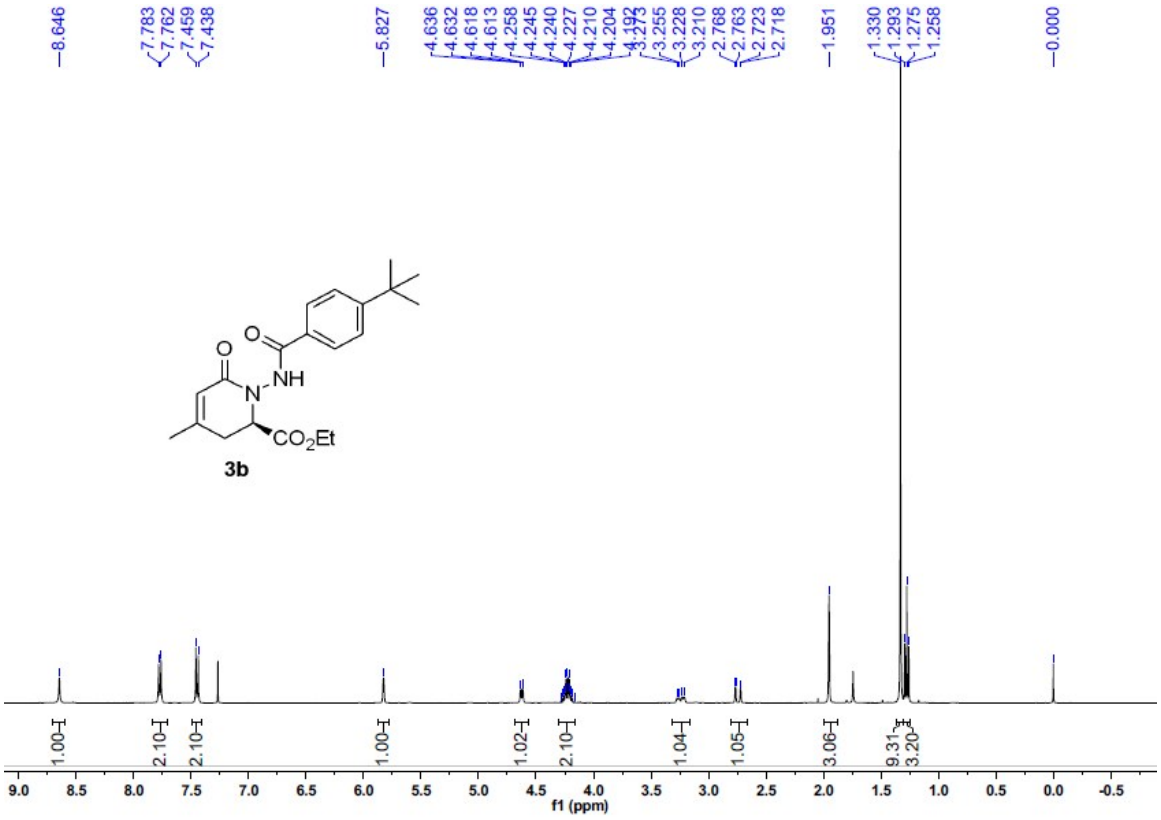
O
NH
N
CO2Et
3a
f1 (ppm)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4.227
4.210
4.204
3.273
3.255
3.228
3.210
2.768
2.763
2.723
2.718
1.951
1.330
1.293
1.275
1.258
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O
NH
N
CO2Et
3b
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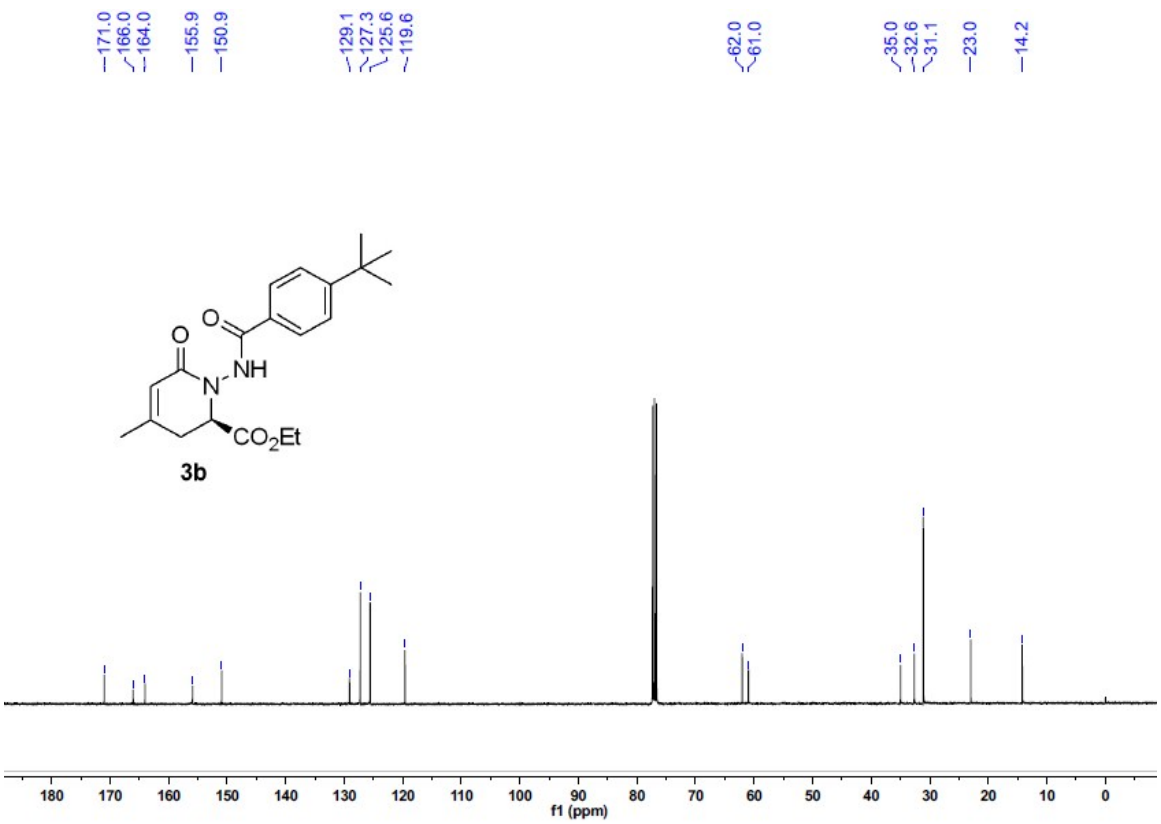
f1 (ppm)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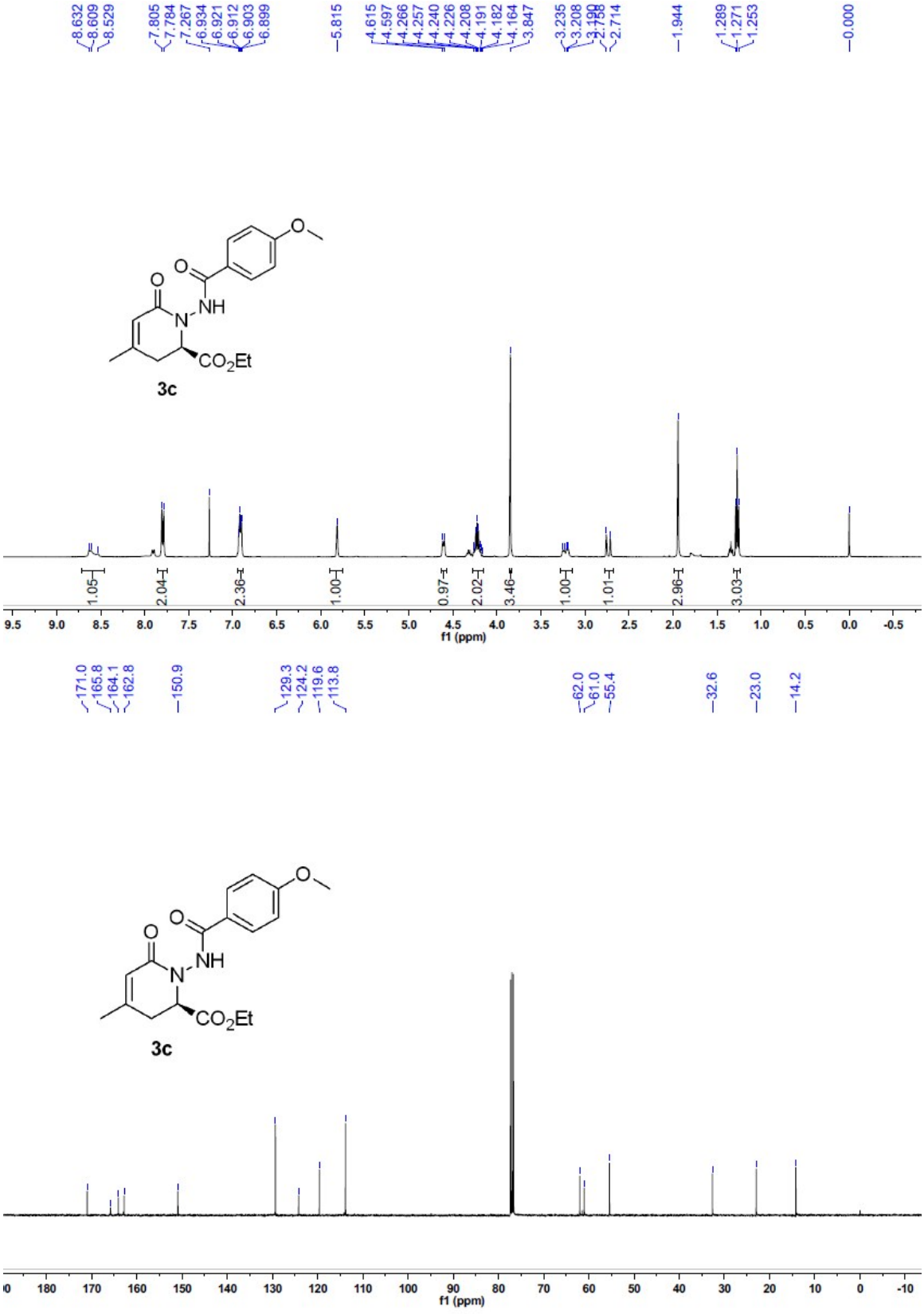
NH
N
CO2Et
3b
f1 (ppm)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3.208
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1.271
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NH
N
CO2Et
3c
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2.04
2.36
1.00
0.97
2.02
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1.00
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2.96
3.03
f1 (ppm)
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164.1
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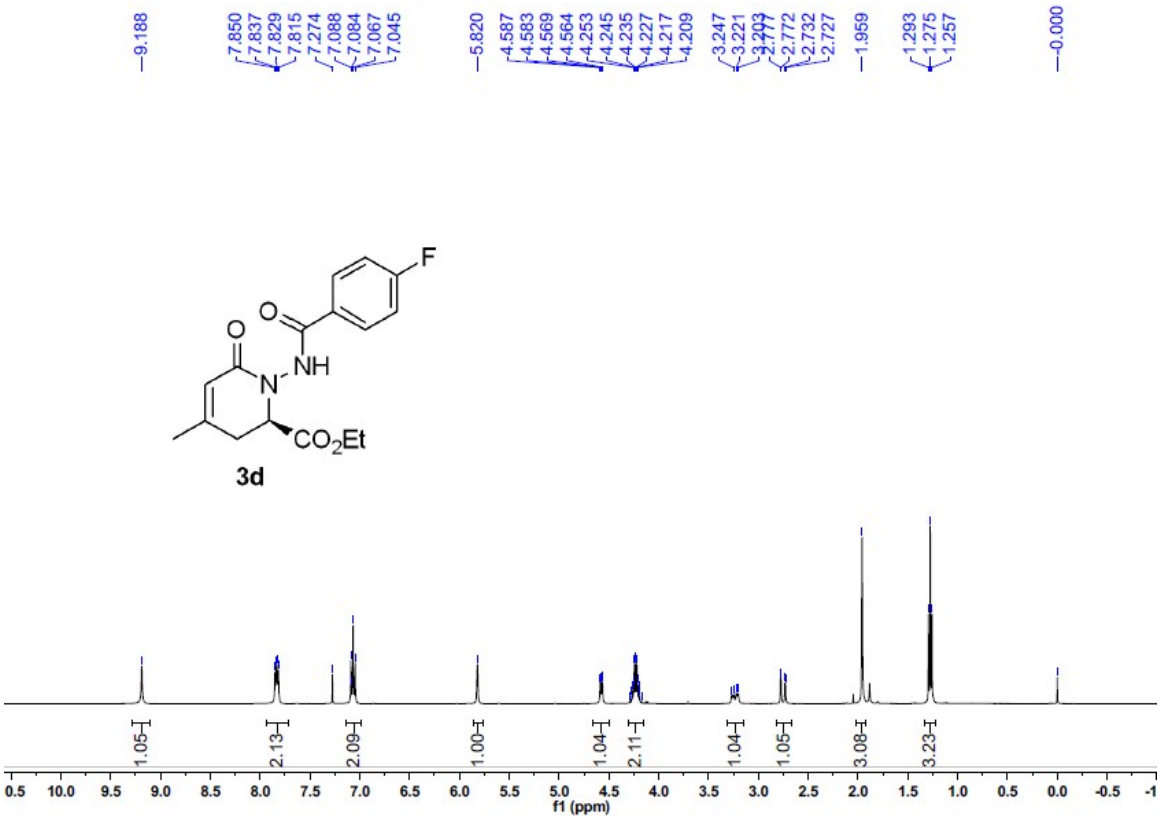
f1 (ppm)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4.564
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4.235
4.227
4.217
4.209
3.247
3.221
3.203
3.177
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O
NH
N
CO2Et
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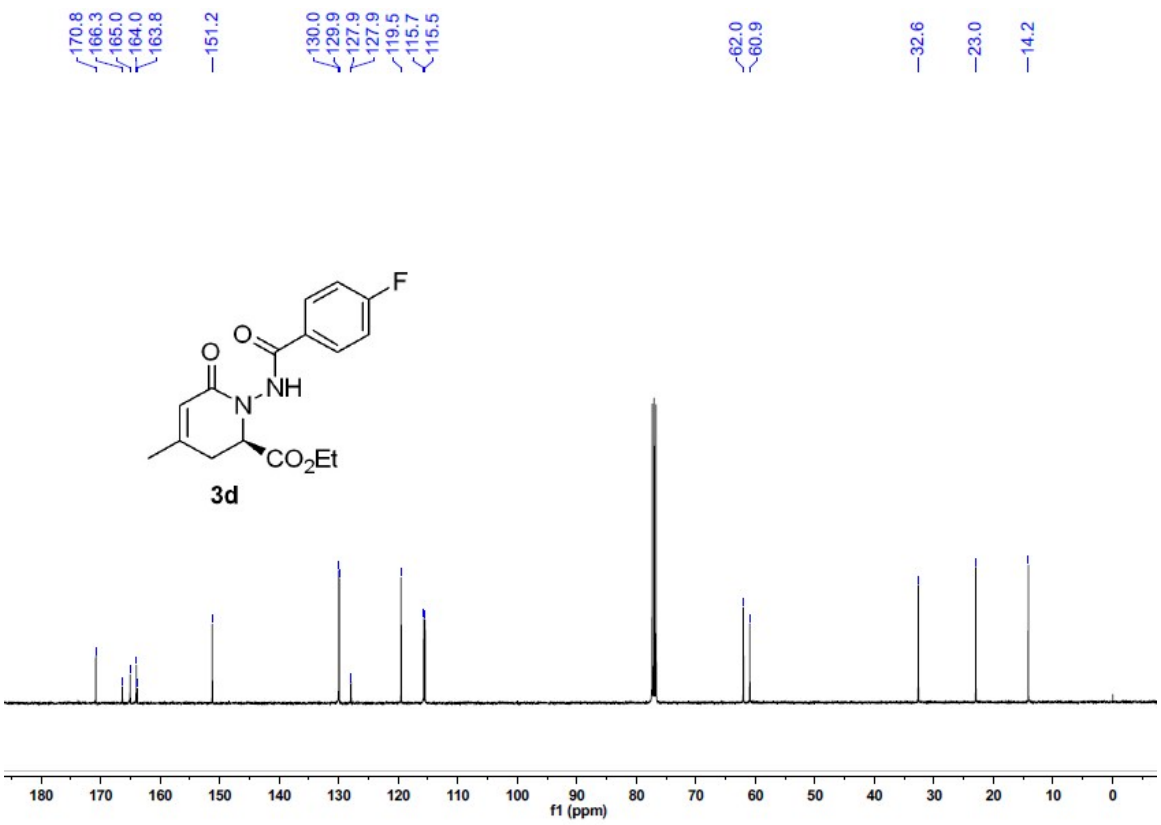
f1 (ppm)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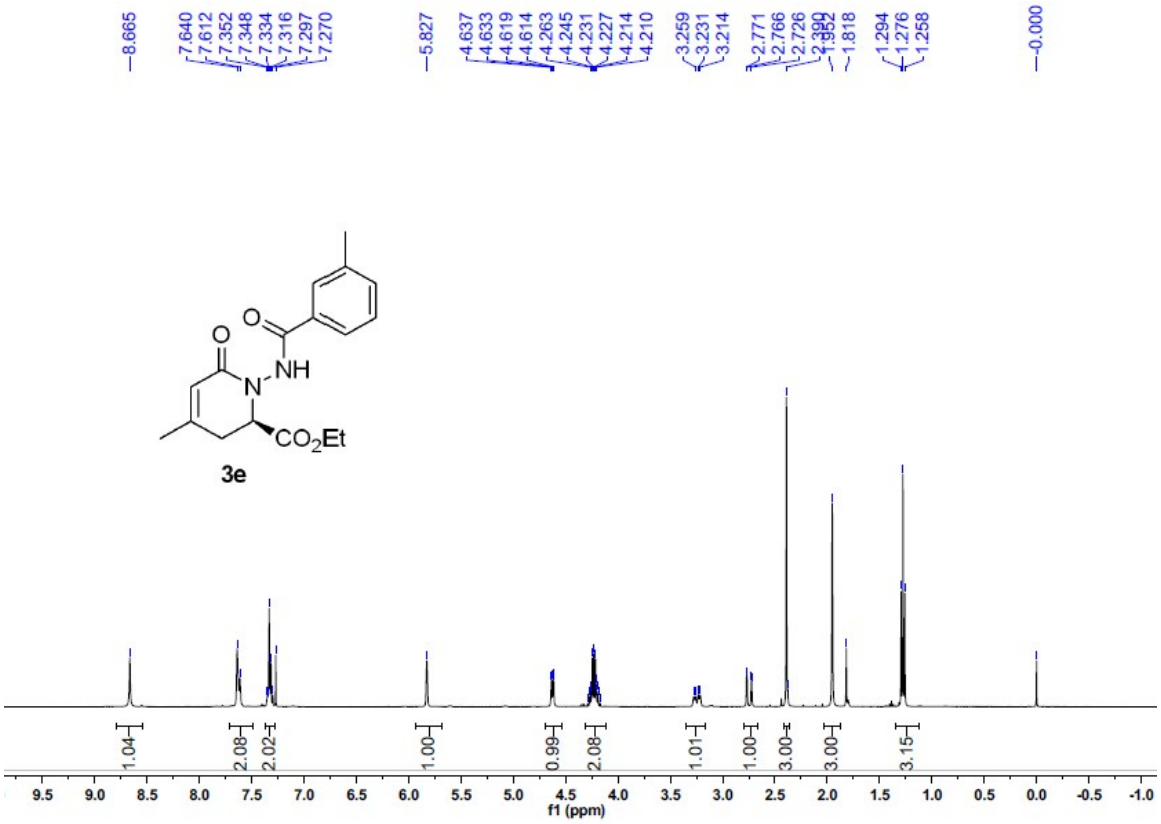
N
CO2Et
3d
f1 (ppm)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4.263
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4.231
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4.214
4.210
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3.231
3.214
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2.766
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1.258
0.000
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O
NH
N
CO2Et
3e
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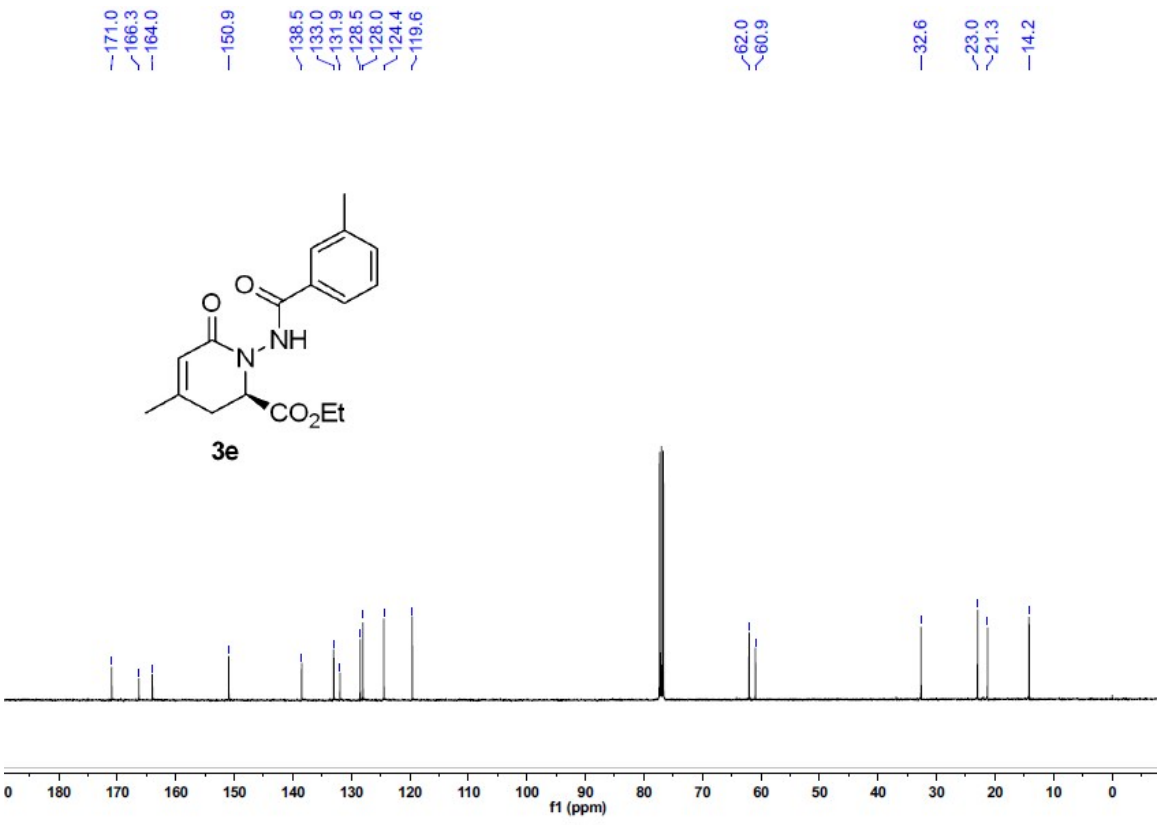
f1 (ppm)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128.5
128.0
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119.6
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60.9
32.6
23.0
21.3
14.2
O
O
NH
N
CO2Et
3e
f1 (ppm)


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7.095
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O
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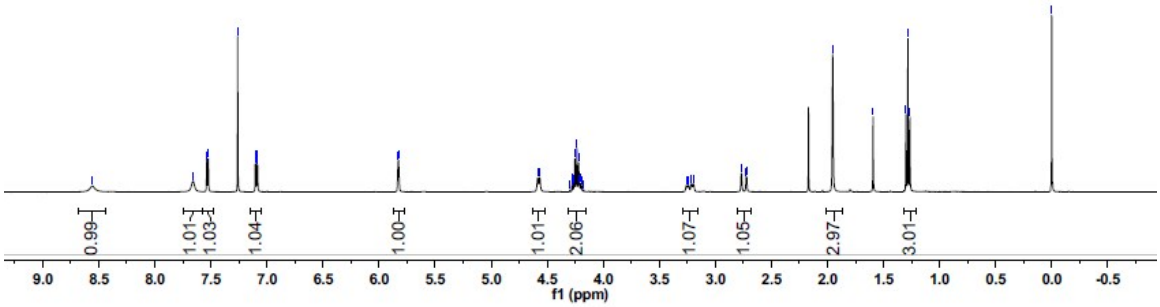
N
CO2Et

3f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f1 (ppm)


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151.4
146.2
134.0
131.3
129.6
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61.1
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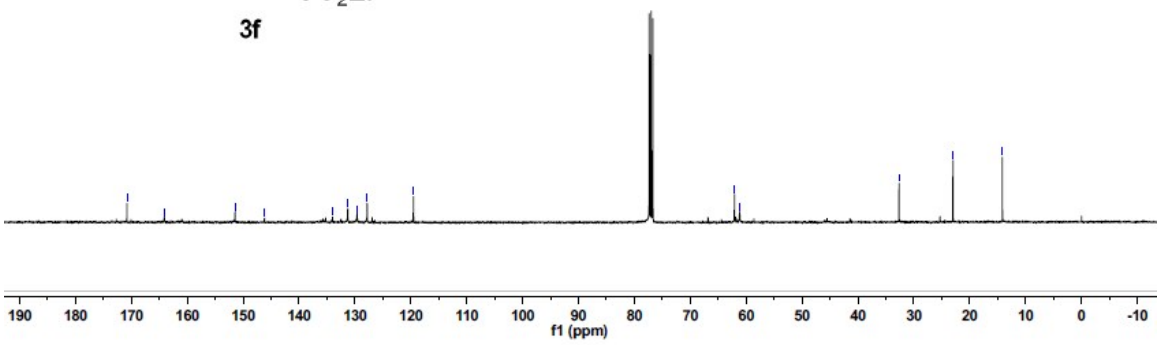
O
S
NH
N
CO2Et

3f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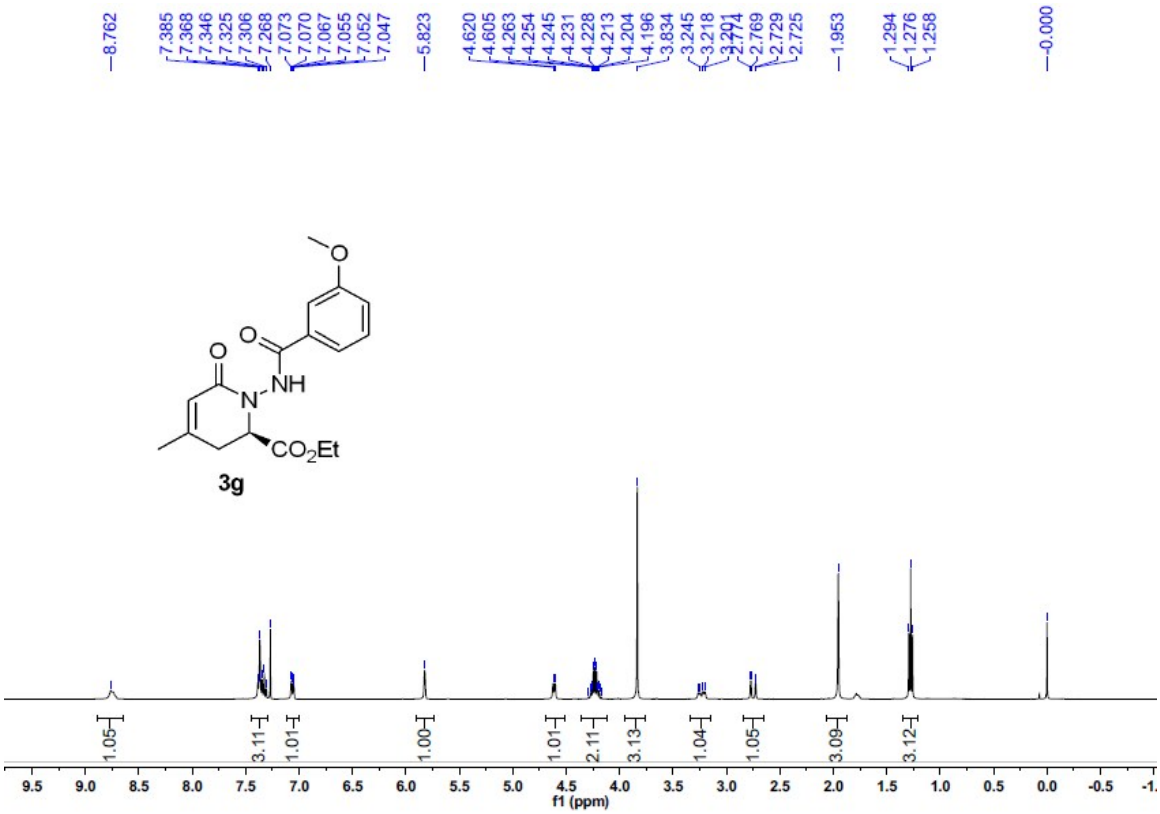
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5.823
4.620
4.605
4.263
4.254
4.245
4.231
4.228
4.213
4.204
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3.834
3.245
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2.725
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1.258
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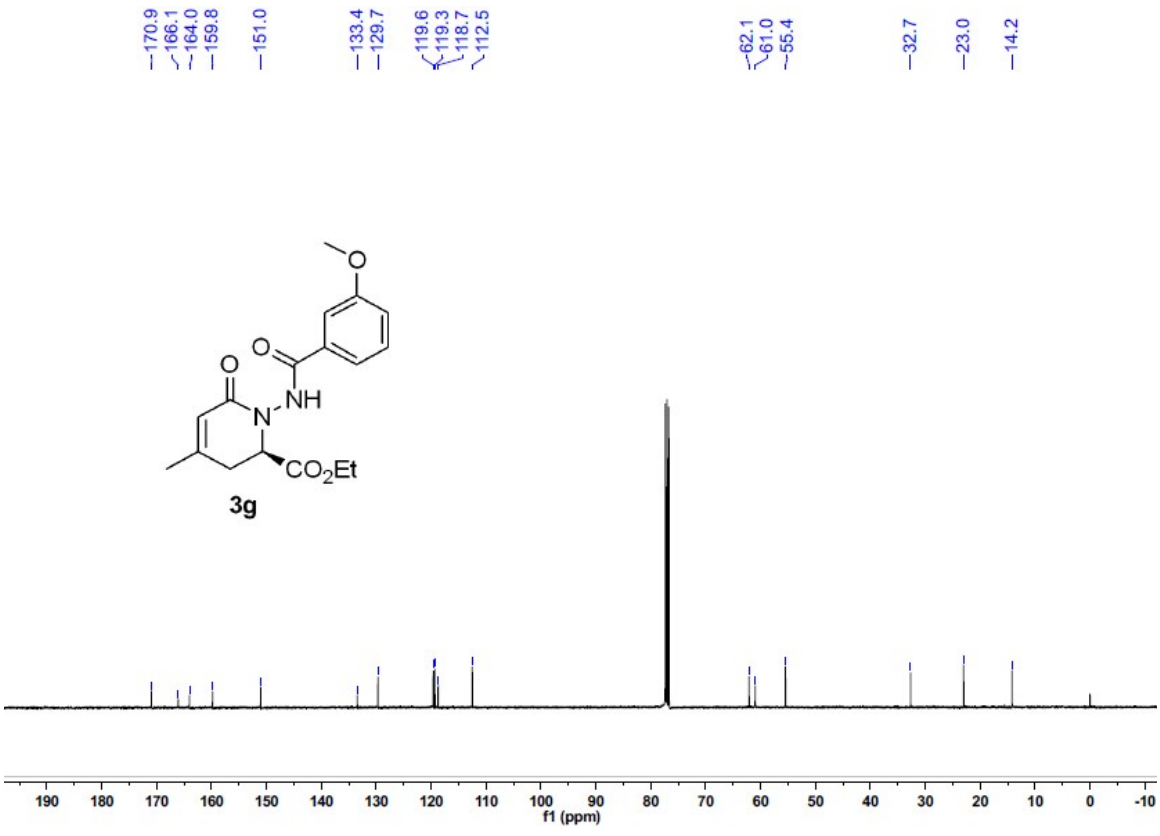
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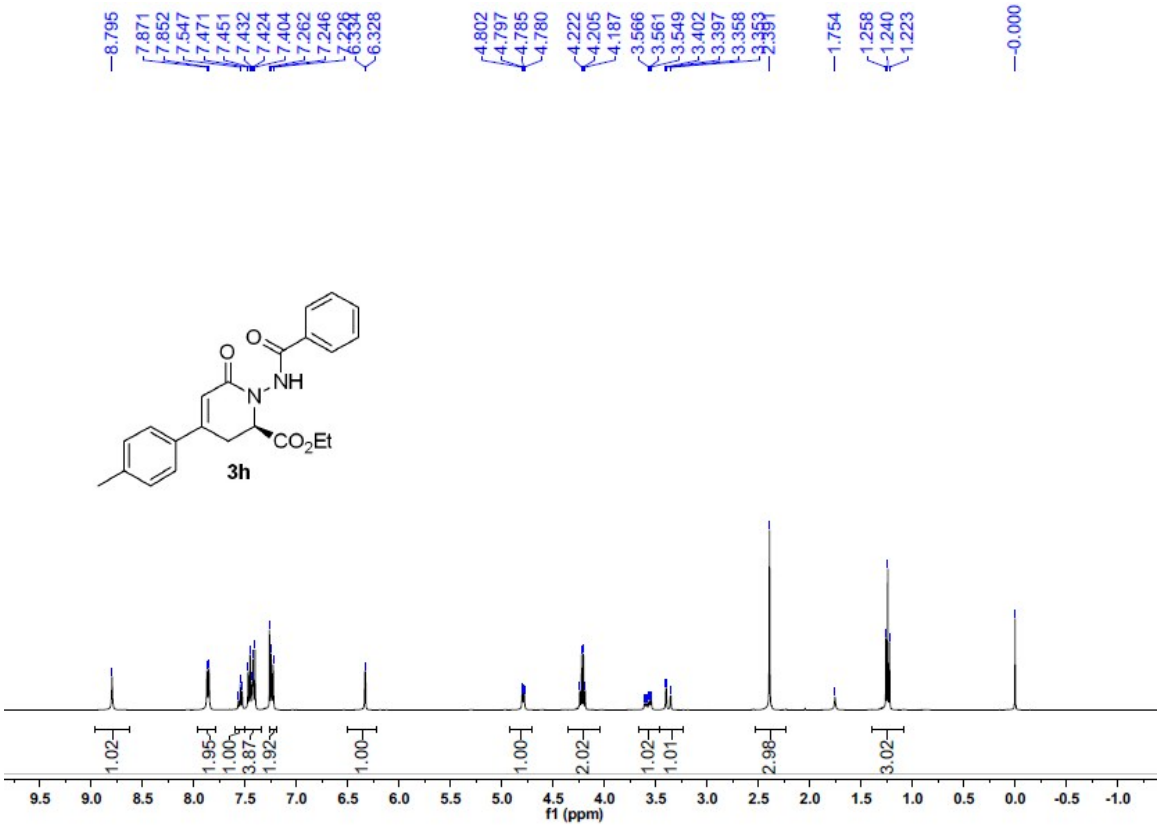
3g
f1 (ppm)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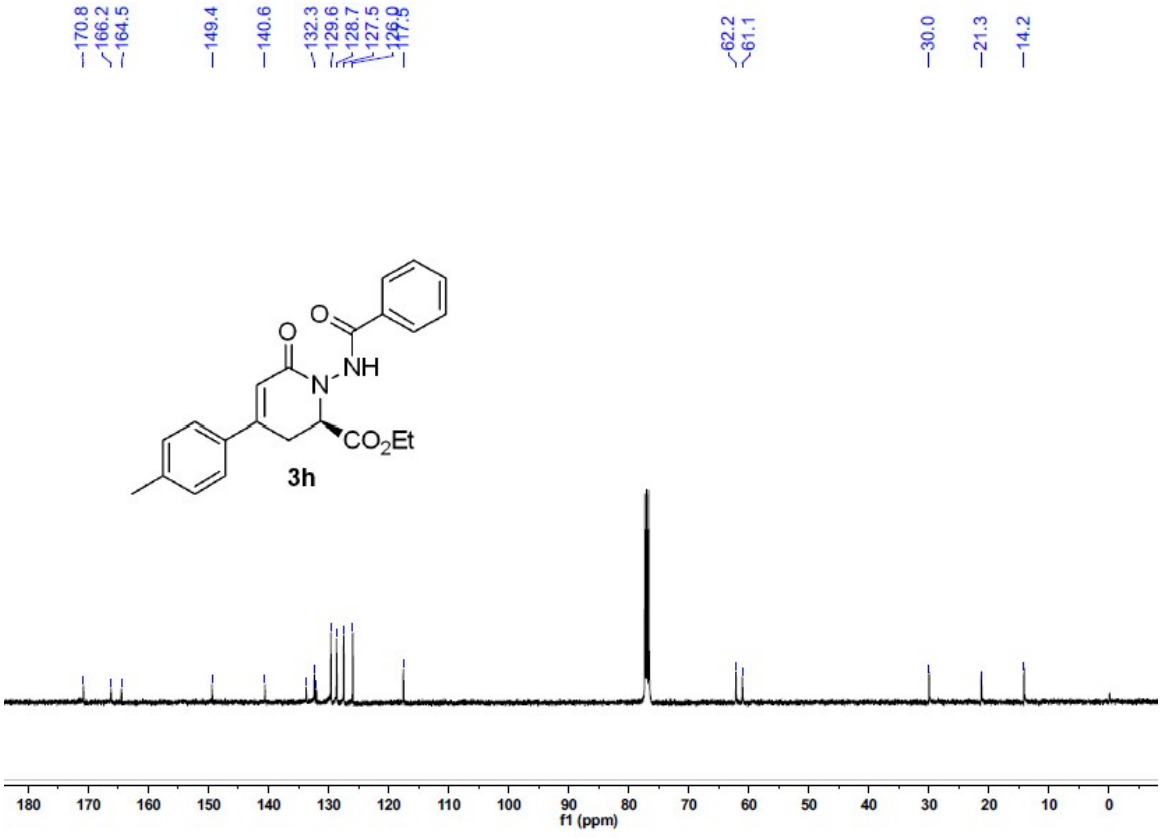
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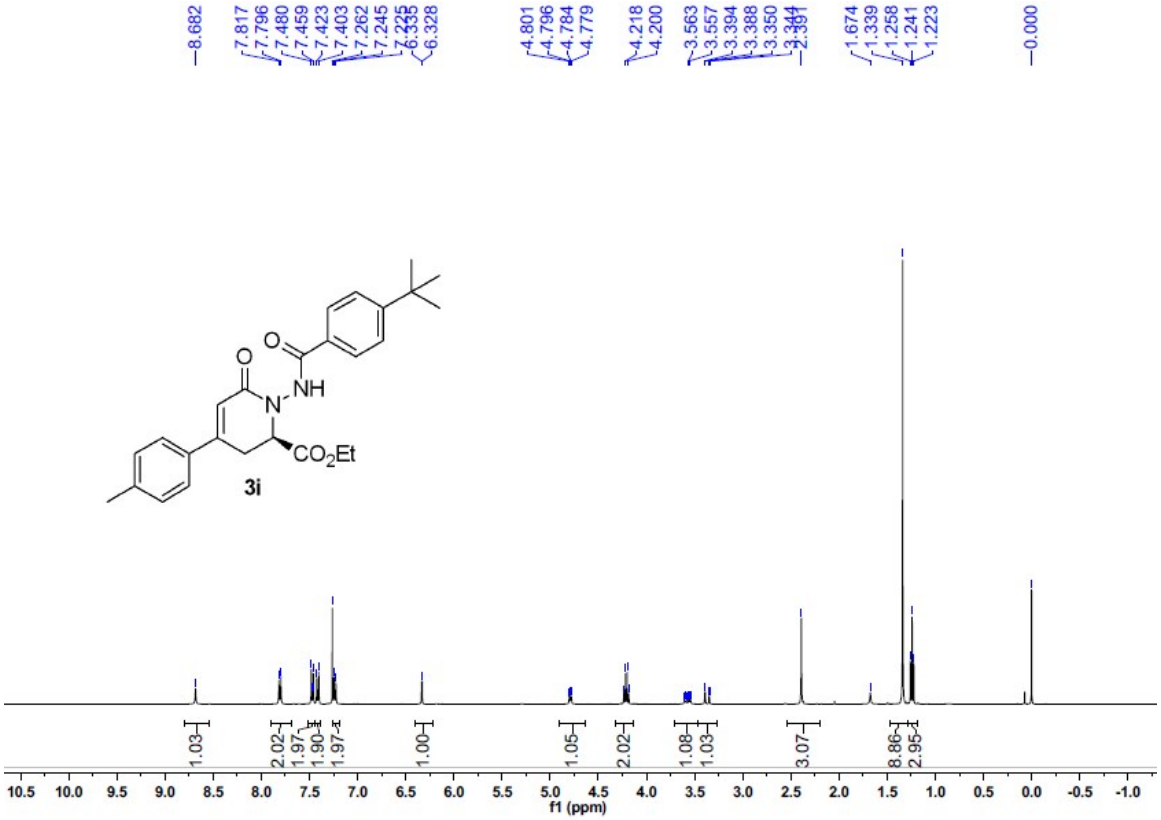
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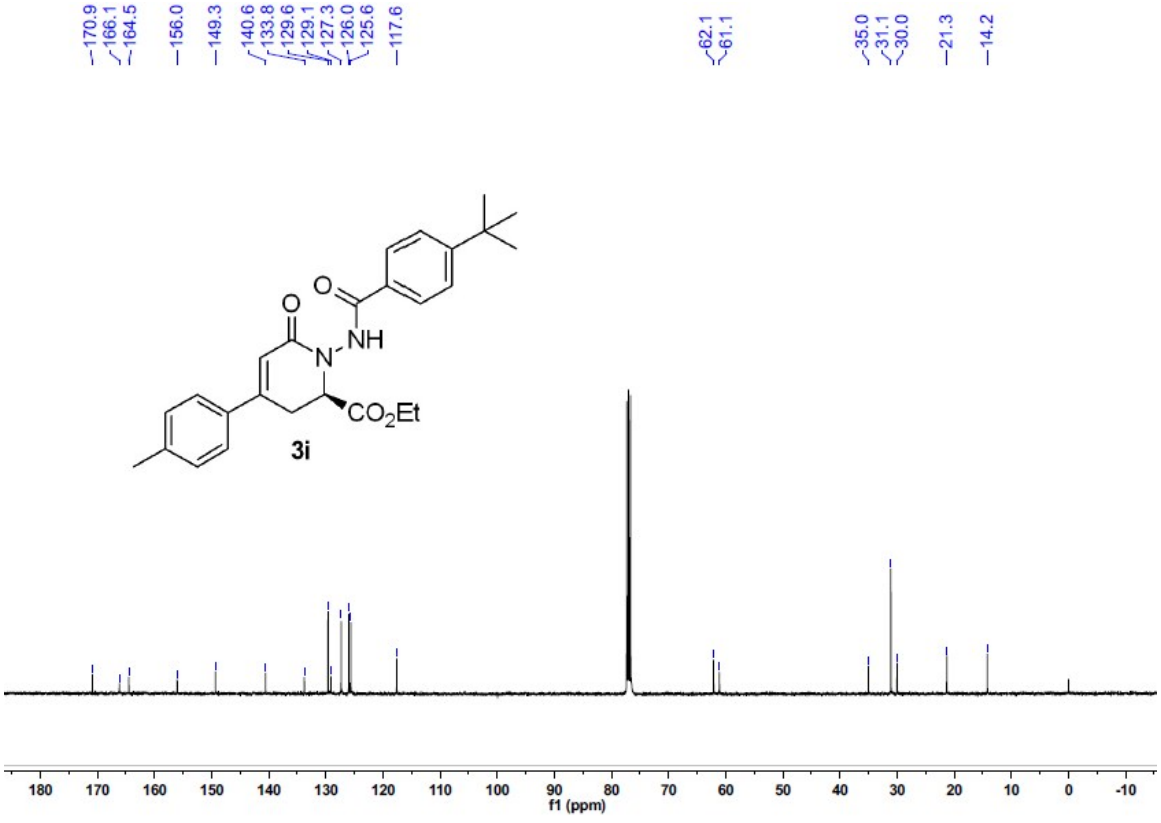
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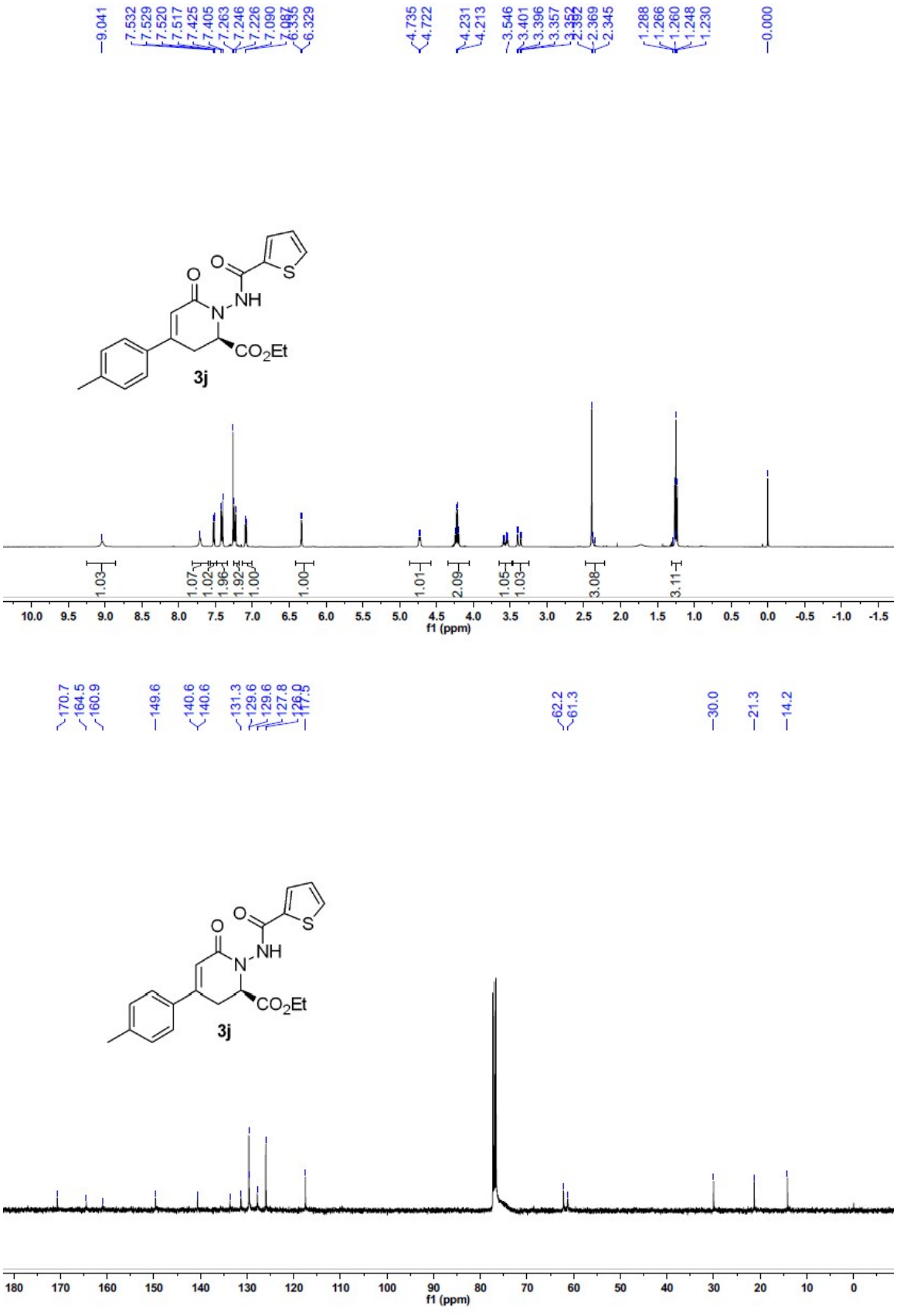
f1 (ppm)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1.00
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3.08
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f1 (ppm)
170.7
164.5
160.9
149.6
140.6
140.6
131.3
129.6
129.6
127.8
62.2
61.3
30.0
21.3
14.2
f1 (ppm)


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7.849
7.572
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7.455
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7.416
7.399
7.394
6.332
6.326
4.801
4.796
4.783
4.778
4.236
4.232
4.218
3.618
3.613
3.602
3.596
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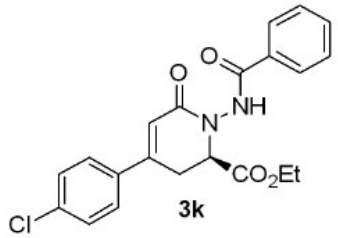
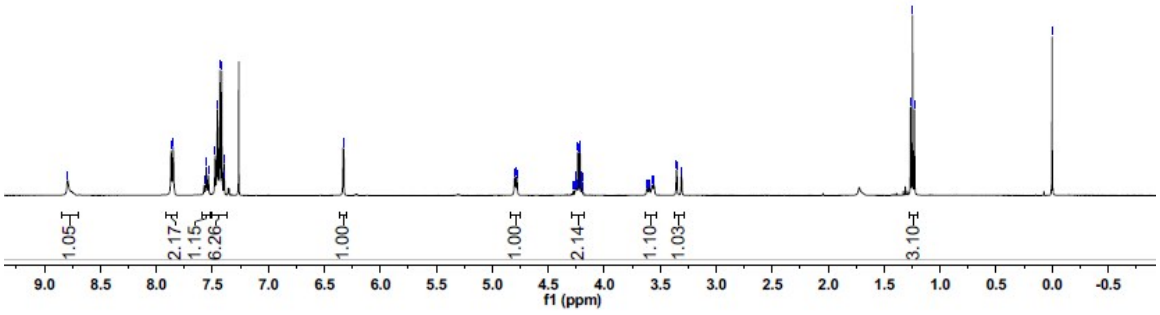

O
O
NH
N
CO2Et
Cl
3k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1.00
2.14
1.10
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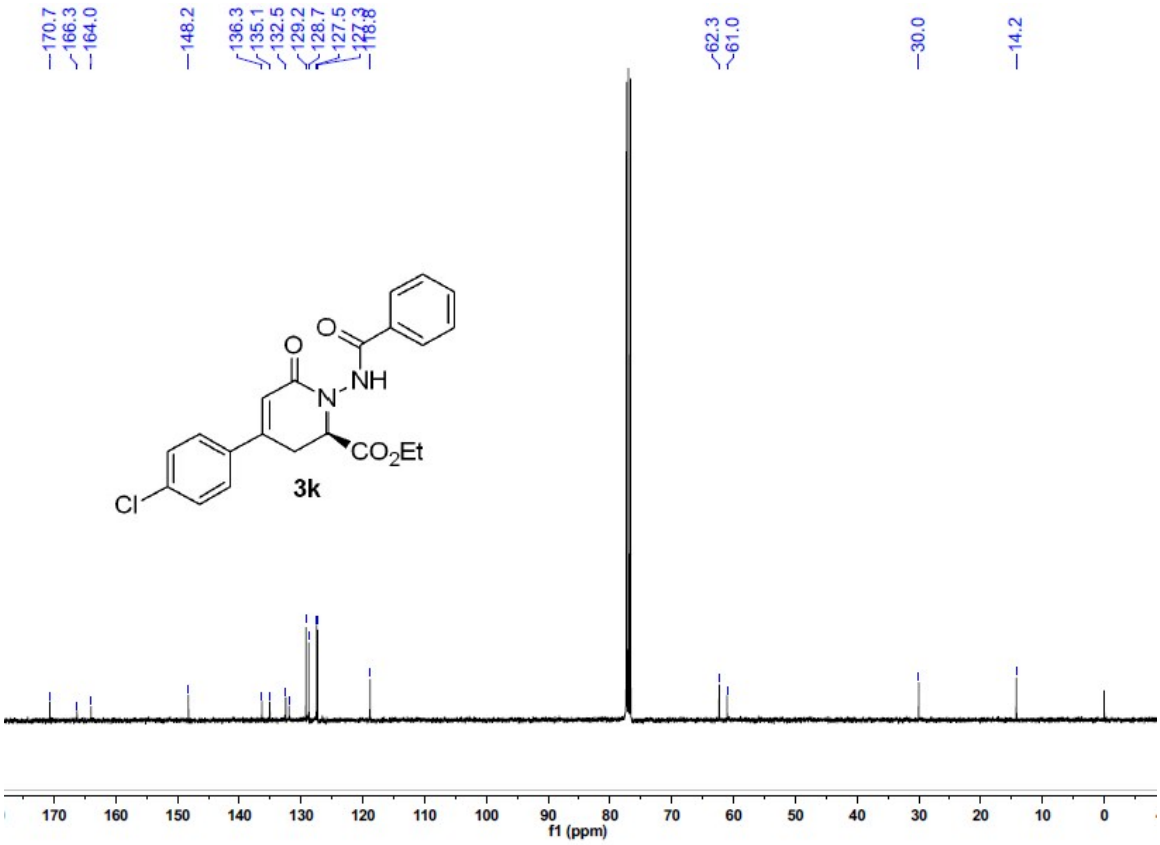
3.10
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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O
NH
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CO2Et
Cl
3k
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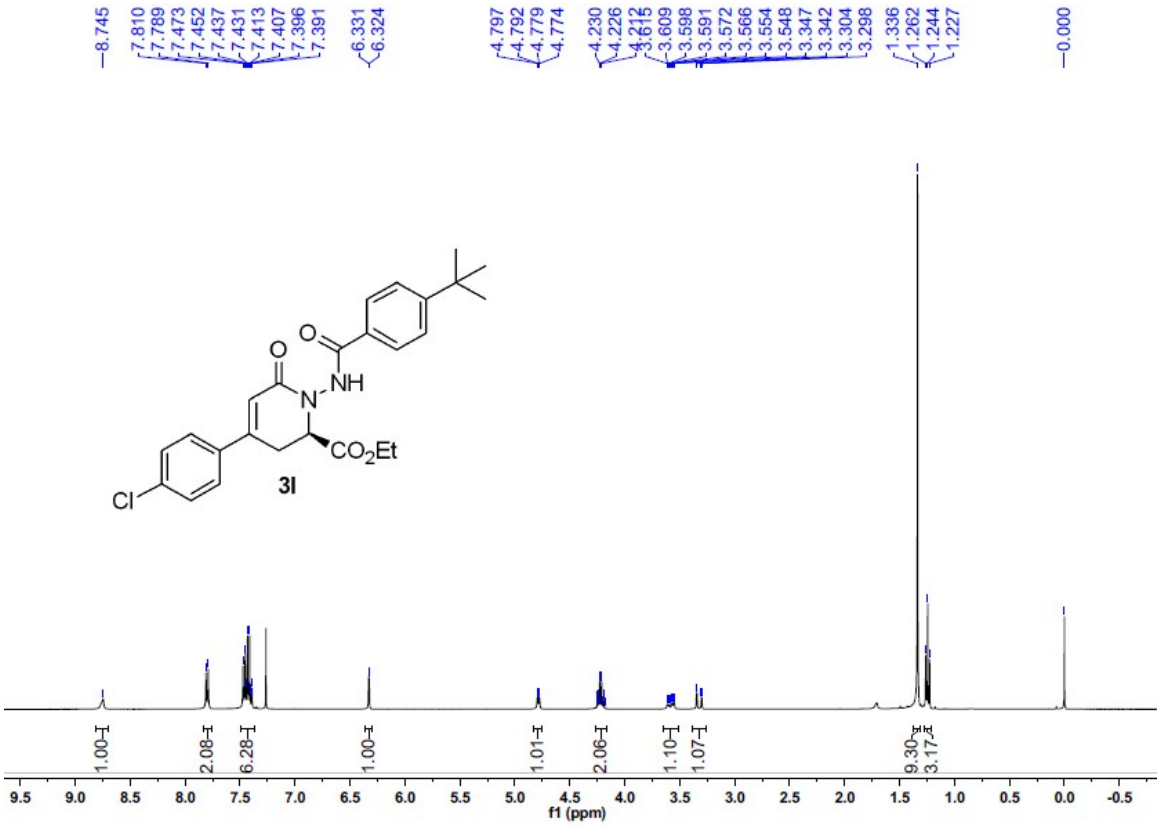
f1 (ppm)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4.222
4.215
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3.342
3.304
3.298
1.336
1.262
1.244
1.227
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NH
N
O
CO2Et
Cl
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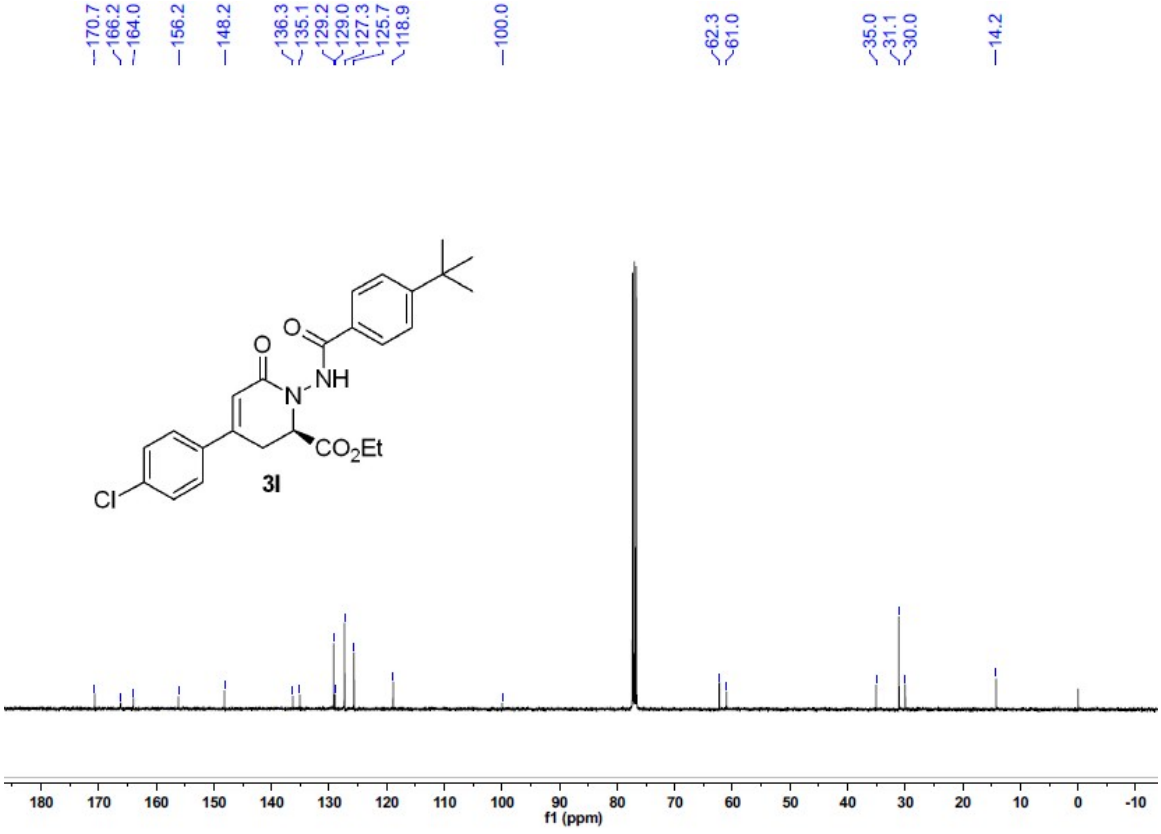
f1 (ppm)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156.2
148.2
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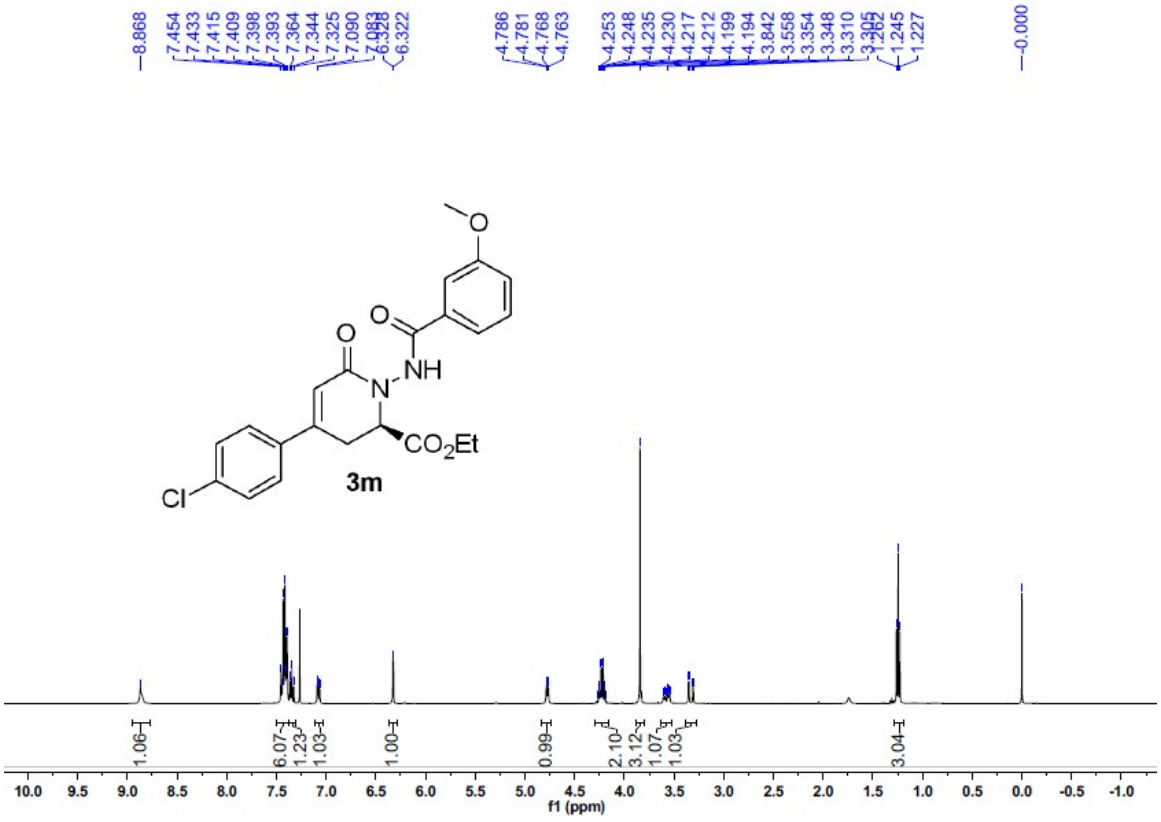
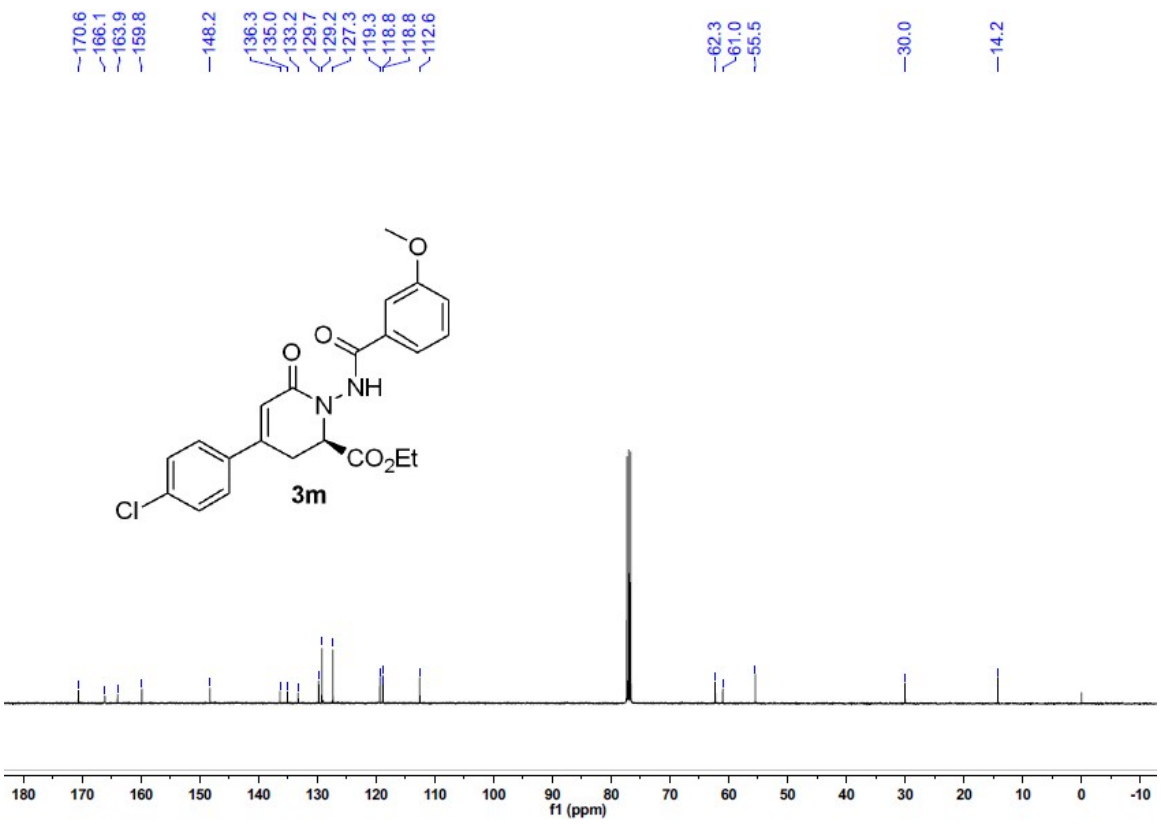
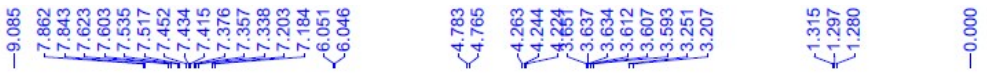
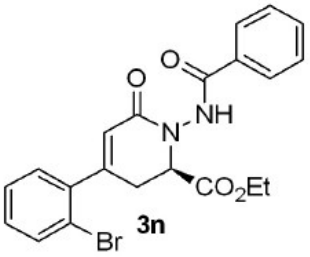
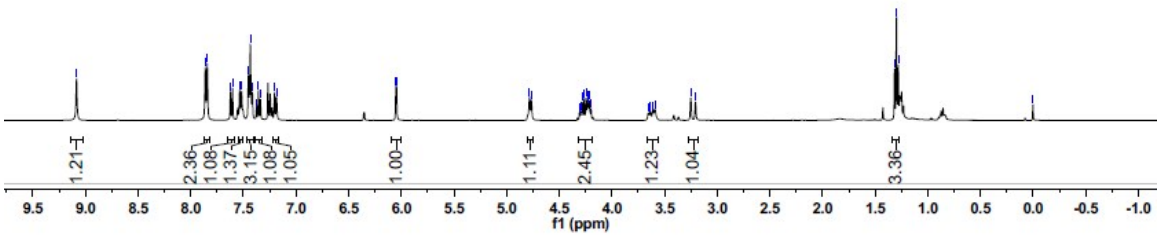
125.7
118.9
100.0
62.3
61.0
35.0
31.1
30.0
14.2
O
NH
N
O
CO2Et
Cl
3l
f1 (ppm)


8.868
7.454
7.433
7.415
7.409
7.398
7.393
7.364
7.344
7.325
7.090
6.328
6.322
4.786
4.781
4.768
4.763
4.253
4.248
4.235
4.230
4.217
4.212
4.199
4.194
3.842
3.558
3.354
3.348
3.310
1.245
1.227
0.000
O
O
NH
N
CO2Et
Cl
3m
1.06
6.07
1.23
1.03
1.00
0.99
2.10
3.12
1.07
1.03
3.04
f1 (ppm)

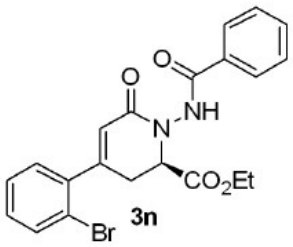

170.6
166.1
163.9
159.8
148.2
136.3
135.0
133.2
129.7
129.2
127.3
119.3
118.8
118.8
112.6
62.3
61.0
55.5
30.0
14.2
O
O
NH
N
CO2Et
Cl
3m
f1 (ppm)

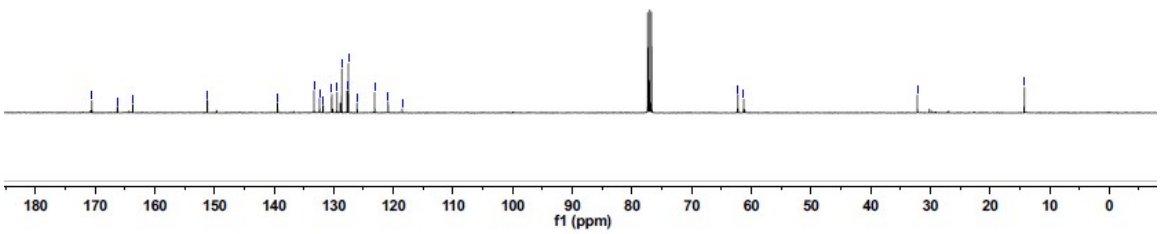

9.085
7.862
7.843
7.623
7.603
7.535
7.517
7.452
7.434
7.415
7.376
7.357
7.338
7.203
7.184
6.051
6.046
4.783
4.765
4.263
4.244
4.224
3.637
3.634
3.612
3.607
3.593
3.251
3.207
1.315
1.297
1.280
0.000


O
O
NH
N
CO2Et
Br
3n

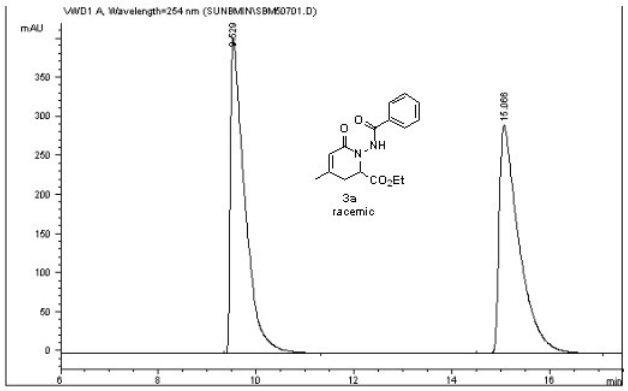

1.21
2.36
1.08
1.37
3.15
1.08
1.05
1.00
1.11
2.45
1.23
1.04
3.36
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


170.5
166.2
163.7
151.2
139.4
133.3
132.4
131.8
130.3
129.4
128.6
127.8
127.5
126.1
123.1
120.9
118.5
62.3
61.3
32.2
14.2

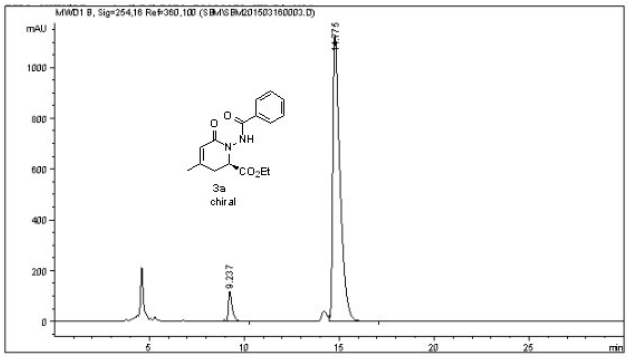

O
O
NH
N
CO2Et
Br
3n


180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

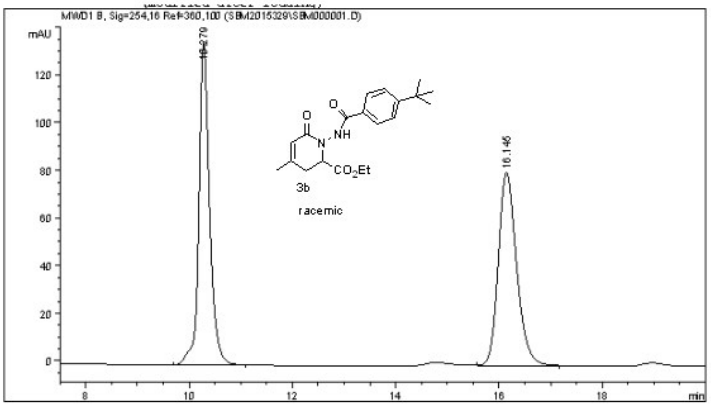
5. HPLC Spectra



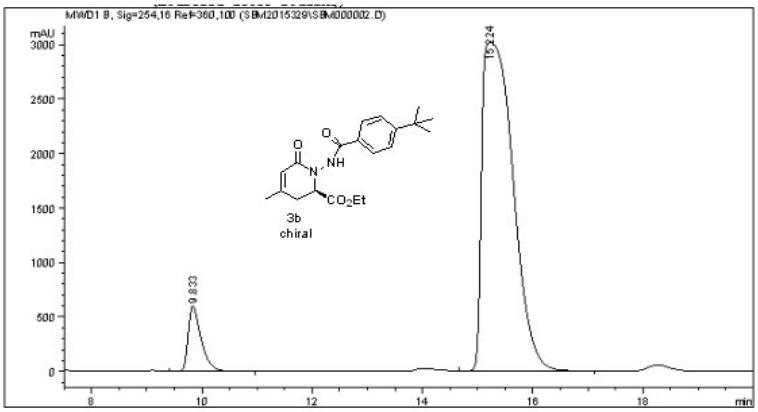

```
=====================================================================
                      Area Percent Report
=====================================================================

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


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1   9.529 PB     0.2915 8253.67090  402.76825  49.9614
   2  15.066 BB     0.4065 8266.42969  291.56198  50.0386

Totals :                  1.65201e4   694.33023
```



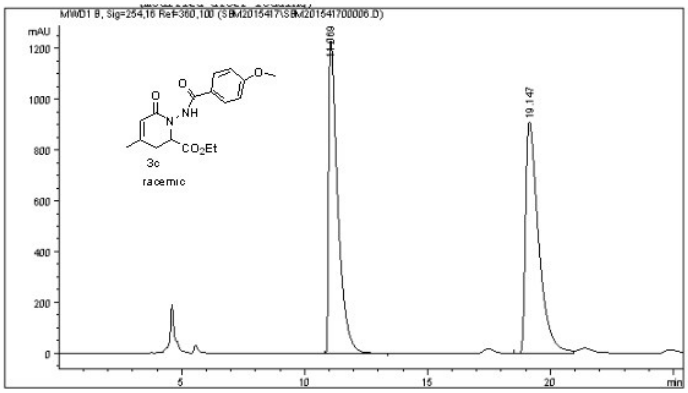

```
=====================================================================
                      Area Percent Report
=====================================================================

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


Signal 1: MWD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.237 BB     0.2046 1629.25891  116.88483   5.2045
   2  14.775 VB     0.3863 2.96758e4  1122.96851  94.7955

Totals :                  3.13050e4  1239.85334
```



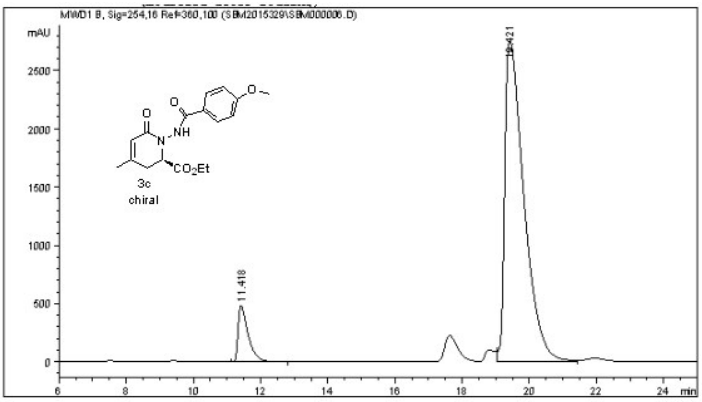

```
=====================================================================
                      Area Percent Report
=====================================================================

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


Signal 1: MWD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]      %
----|-------|----|-------|----------|----------|--------|
   1  10.279 BB    0.2147 1956.25232  135.26466  49.4599
   2  16.145 BB    0.3774 1998.97559   81.14372  50.5401

Totals :                  3955.22791  216.40839
```



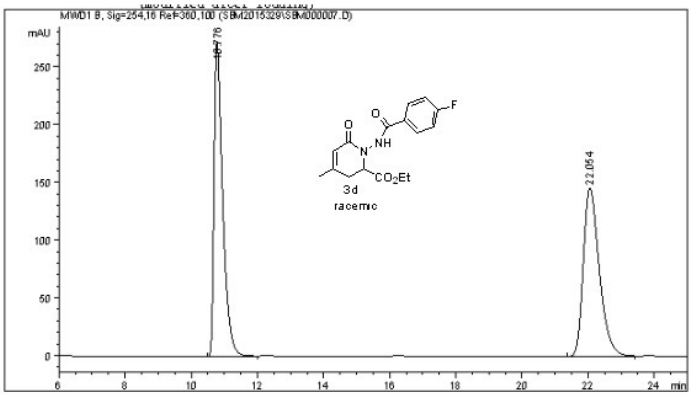

```
=====================================================================
                      Area Percent Report
=====================================================================

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


Signal 1: MWD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]      %
----|-------|----|-------|----------|----------|--------|
   1   9.833 VB    0.2437 9836.29199  598.59888   7.6450
   2  15.224 VV    0.5148 1.18827e5  3021.71460  92.3550

Totals :                  1.28664e5  3620.31348
```



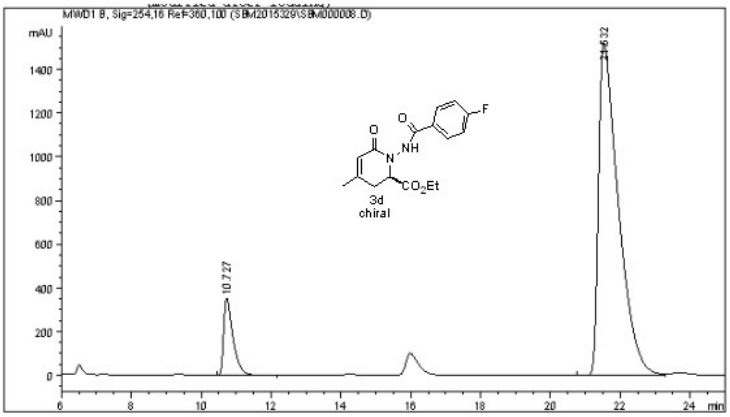

```
=====================================================================
                      Area Percent Report
=====================================================================

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


Signal 1: MWD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  11.069 BB    0.3764 3.16462e4  1229.07520  48.5074
   2  19.147 BV    0.5503 3.35937e4   910.74097  51.4926

Totals :                  6.52399e4  2139.81616
```



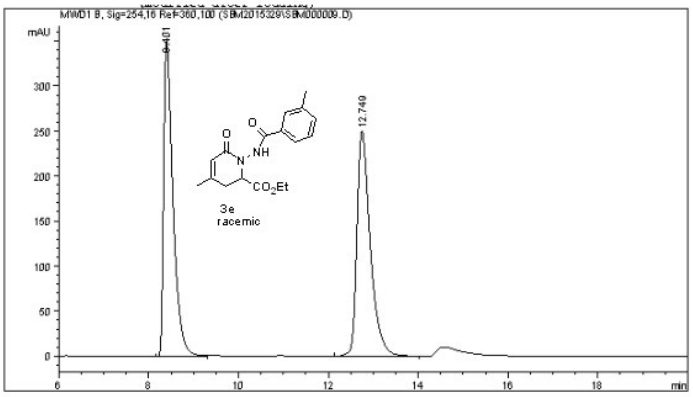

```
=====================================================================
                      Area Percent Report
=====================================================================

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


Signal 1: MWD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  11.418 BB    0.3022 9992.89453  485.44186   8.3847
   2  19.421 VV    0.5950 1.09187e5  2751.23291  91.6153

Totals :                  1.19180e5  3236.67477
```



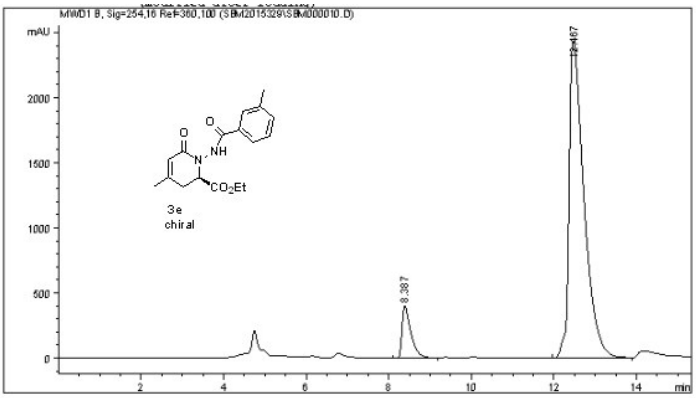

```
=====================================================================
                    Area Percent Report
=====================================================================

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


Signal 1: MWD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  10.776 BB    0.2691 4925.35889  272.21667  50.1954
   2  22.054 BB    0.5103 4887.00928  146.06912  49.8046

Totals :                  9812.36816  418.28580
```



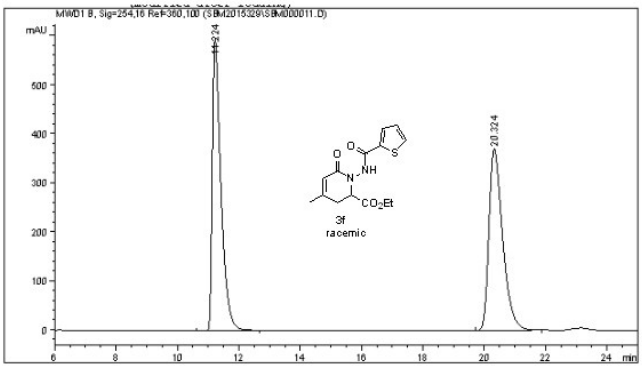

```
=====================================================================
                    Area Percent Report
=====================================================================

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


Signal 1: MWD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  10.727 VB    0.2741 6533.79980  352.76584   9.8326
   2  21.532 VV    0.5795 5.99165e4  1521.35327  90.1674

Totals :                  6.64503e4  1874.11911
```



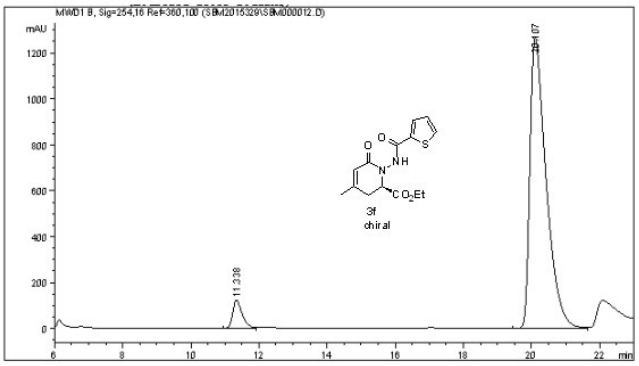

```
================================================================
                    Area Percent Report
================================================================

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


Signal 1: MWD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]    [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.401 BV    0.2153 5076.07373  349.79114  49.5338
   2  12.749 BB    0.3092 5171.61768  250.12096  50.4662

Totals :                  1.02477e4   599.91209
```



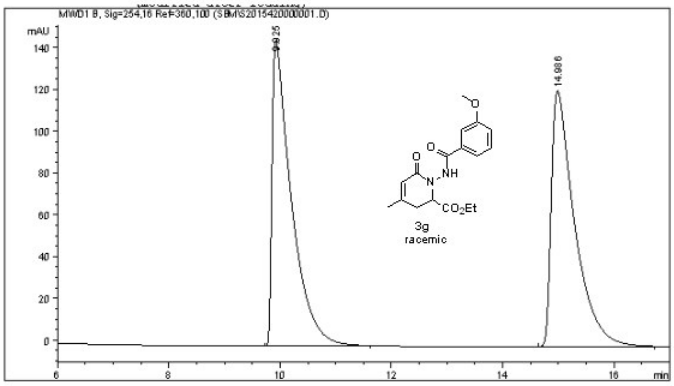

```
================================================================
                    Area Percent Report
================================================================

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


Signal 1: MWD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]    [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.387 VV    0.2168 5926.20459  399.99872   8.8450
   2  12.467 VV    0.3747 6.10741e4  2434.66382  91.1550

Totals :                  6.70003e4  2834.66254
```



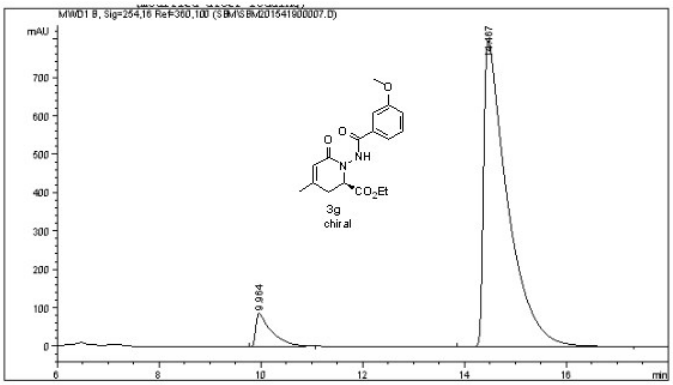

```
=====================================================================
                    Area Percent Report
=====================================================================

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


Signal 1: MWD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  11.224 BB    0.2891 1.15909e4   595.49945  50.2445
   2  20.324 BB    0.4697 1.14781e4   370.18542  49.7555

Totals :                  2.30690e4   965.68488
```



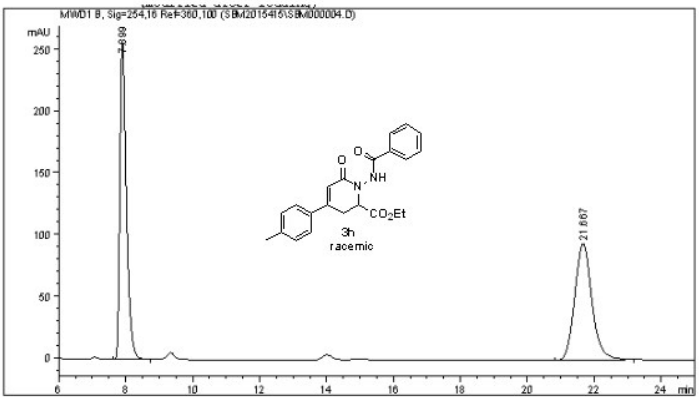

```
=====================================================================
                    Area Percent Report
=====================================================================

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


Signal 1: MWD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  11.338 BV    0.2873 2387.16040  123.59637   5.3969
   2  20.107 VV    0.4911 4.18446e4  1267.66016  94.6031

Totals :                  4.42317e4  1391.25653
```



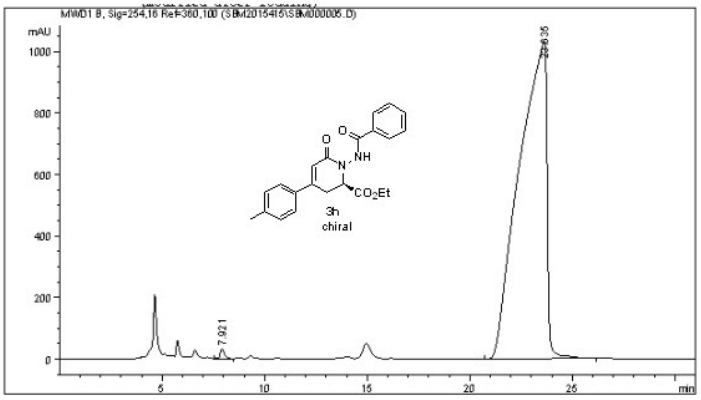

```
=====================================================================
                      Area Percent Report
=====================================================================

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


Signal 1: MWD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  9.925 BB    0.3344 3516.92993  146.30910  49.8011
   2 14.986 BB    0.4196 3545.02539  122.50875  50.1989

Totals :                  7061.95532  268.81785
```



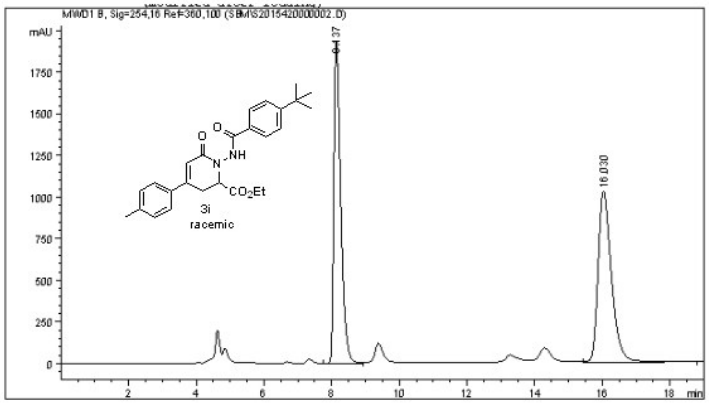

```
=====================================================================
                      Area Percent Report
=====================================================================

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


Signal 1: MWD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  9.964 BB    0.2838 1810.79346   86.66051   6.5343
   2 14.467 BB    0.4525 2.59013e4   798.31549  93.4657

Totals :                  2.77121e4   884.97601
```




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

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


Signal 1: MWD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.899 BB    0.2031 3462.56030  257.13708  50.6799
   2  21.667 BB    0.5494 3369.65088   94.19396  49.3201

Totals :                  6832.21118  351.33105
```




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

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


Signal 1: MWD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.921 VV    0.2263  511.80624   33.12492   0.5273
   2  23.635 BB    1.1879 9.65518e4  1031.59058  99.4727

Totals :                  9.70636e4  1064.71550
```



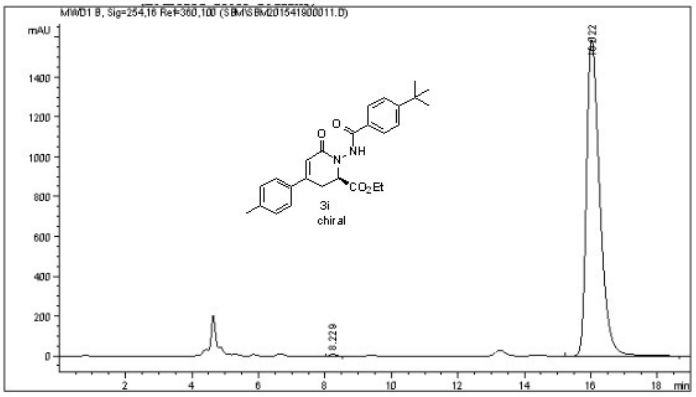
Area Percent Report

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

Signal 1: MWD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.137	VV	0.2138	2.74540e4	1931.45520	49.2902
2	16.030	BB	0.4190	2.82446e4	1026.68567	50.7098

Totals : 5.56986e4 2958.14087



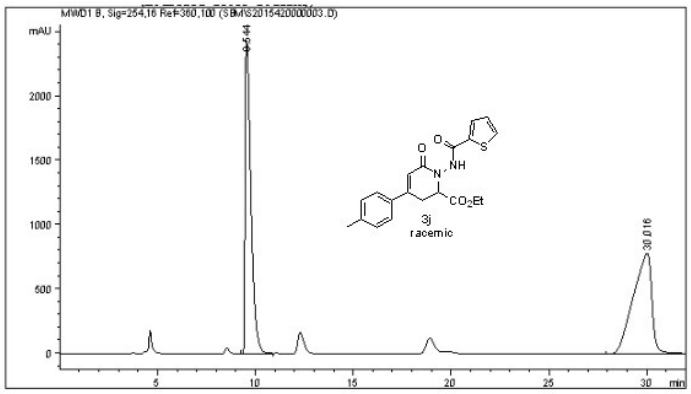
Area Percent Report

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

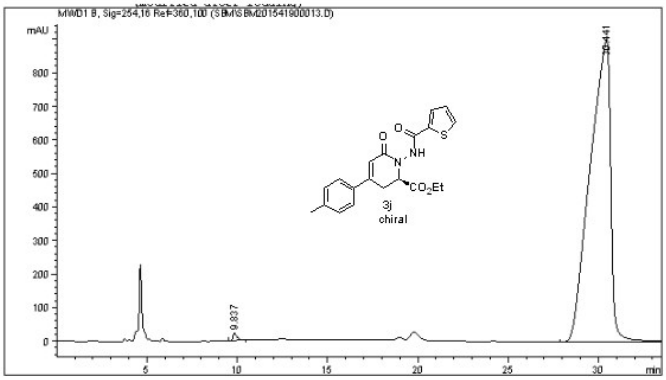
Signal 1: MWD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.229	VV	0.1772	177.72806	12.55518	0.4014
2	16.022	BB	0.4239	4.41022e4	1588.54382	99.5986

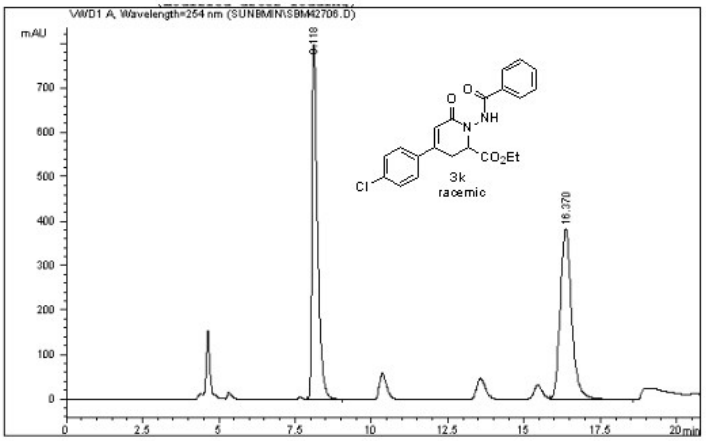
Totals : 4.42799e4 1601.09900



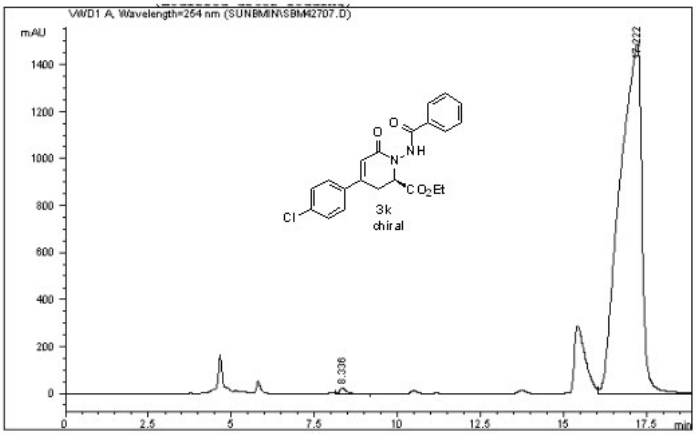

```
=====================================================================
                      Area Percent Report
=====================================================================

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


Signal 1: MWD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]      %
----|-------|----|-------|----------|----------|--------|
   1   9.544 BV    0.3067 4.98935e4  2438.53003  49.0525
   2  30.016 BBA   1.1053 5.18211e4   781.79852  50.9475

Totals :                  1.01715e5  3220.32855
```



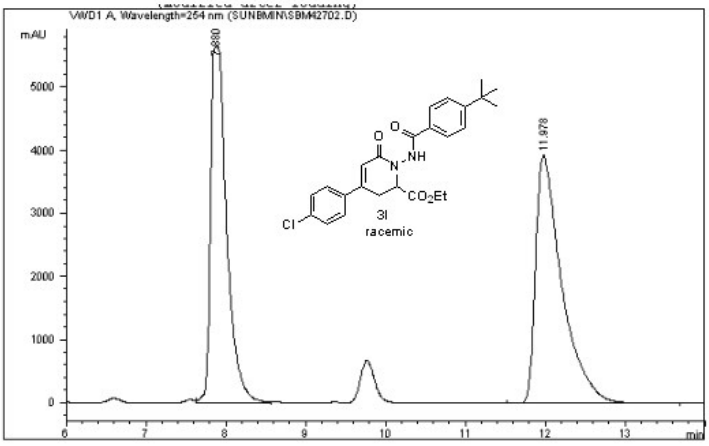

```
=====================================================================
                      Area Percent Report
=====================================================================

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


Signal 1: MWD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]      %
----|-------|----|-------|----------|----------|--------|
   1   9.837 VB    0.2203  338.24966   22.12740   0.4692
   2  30.441 VBA   1.1761 7.17487e4   901.93622  99.5308

Totals :                  7.20870e4   924.06362
```



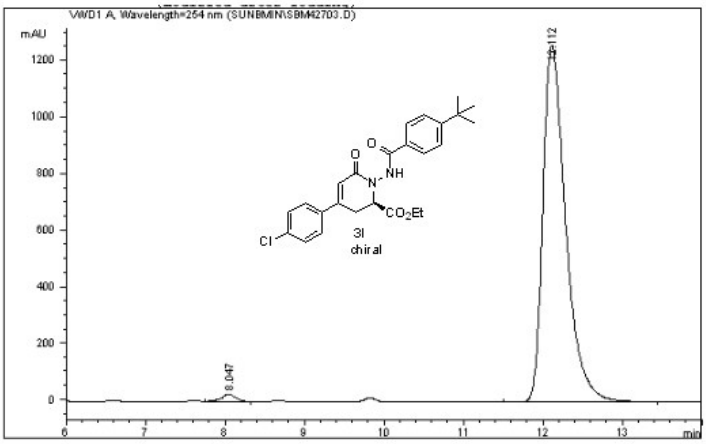

```
================================================================================
                          Area Percent Report
================================================================================

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


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1   8.118 VV     0.1899 1.02136e4   798.77344  50.1519
   2  16.370 VB     0.4046 1.01517e4   383.89285  49.8481

Totals :                  2.03653e4  1182.66629
```



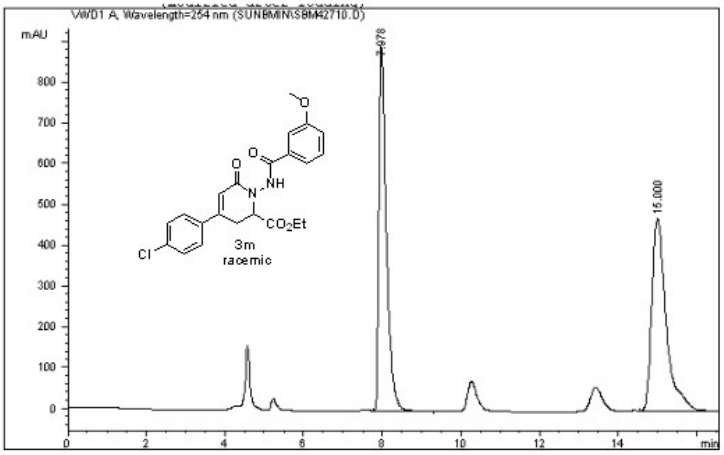

```
================================================================================
                          Area Percent Report
================================================================================

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


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1   8.336 VB     0.2303  405.57346   25.96082   0.5866
   2  17.222 VBA    0.8150 6.87324e4  1489.37012  99.4134

Totals :                  6.91380e4  1515.33093
```



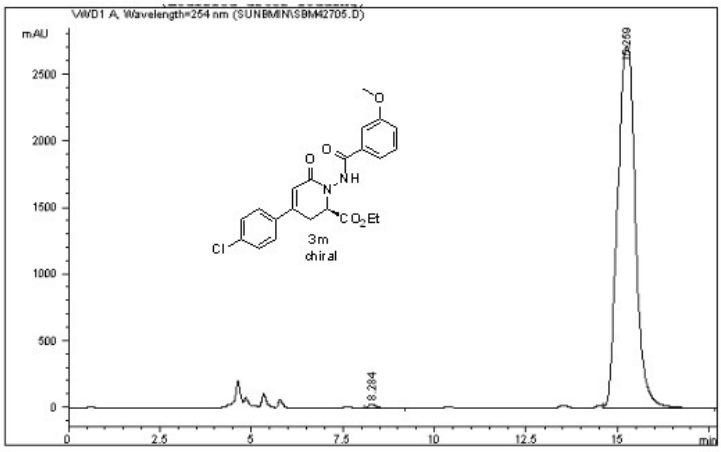

```
=====================================================================
                      Area Percent Report
=====================================================================

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


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1   7.880 VV    0.2080 7.96160e4  5651.54004  47.0605
   2  11.978 VV    0.3335 8.95619e4  3919.90039  52.9395

Totals :                  1.69178e5  9571.44043
```



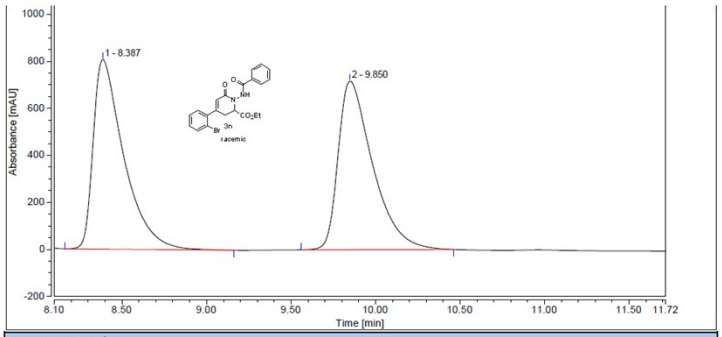

```
=====================================================================
                      Area Percent Report
=====================================================================

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


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1   8.047 VV    0.2126  395.16156   27.06244   1.5484
   2  12.112 PB    0.3022 2.51261e4  1261.84644  98.4516

Totals :                  2.55212e4  1288.90887
```




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

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


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1   7.978 VB    0.1944 1.17706e4   894.05591  49.0292
   2  15.000 VBA   0.3870 1.22368e4   474.09323  50.9708

Totals :                  2.40074e4  1368.14914
```

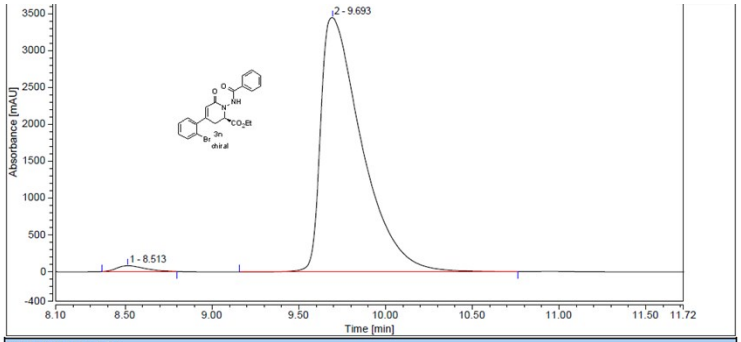



```
=====================================================================
                      Area Percent Report
=====================================================================

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


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1   8.284 VP    0.2694  523.50281   28.92497   0.5632
   2  15.259 VB    0.5026 9.24262e4  2720.81470  99.4368

Totals :                  9.29497e4  2749.73967
```



Integration Results

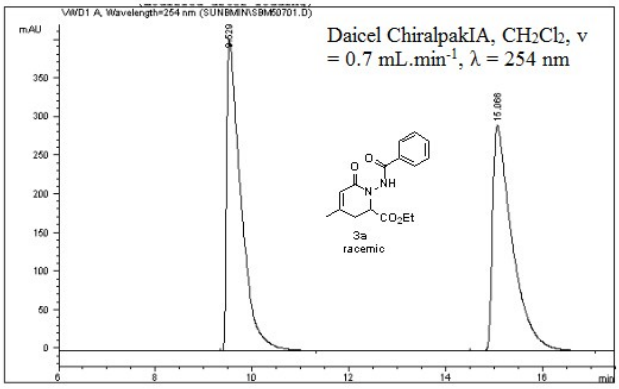
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		8.387	168.914	807.069	49.88	52.93	n.a.
2		9.850	169.695	717.624	50.12	47.07	n.a.
Total:			**338.609**	**1524.692**	**100.00**	**100.00**	



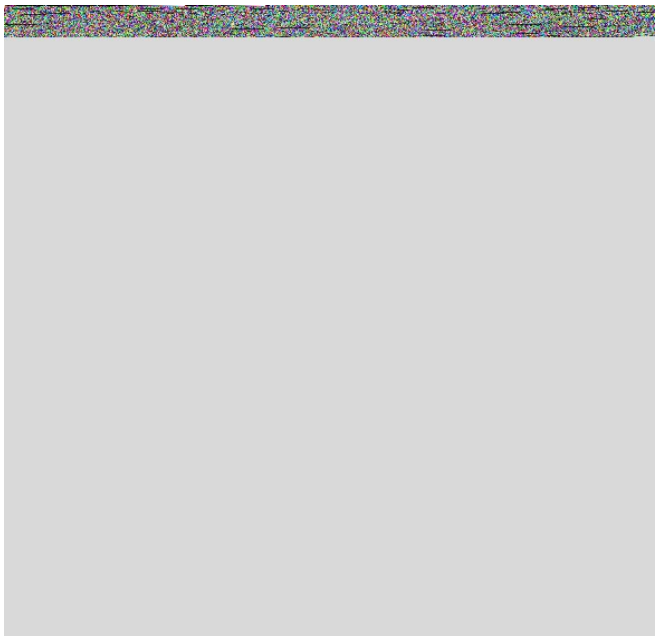
Integration Results

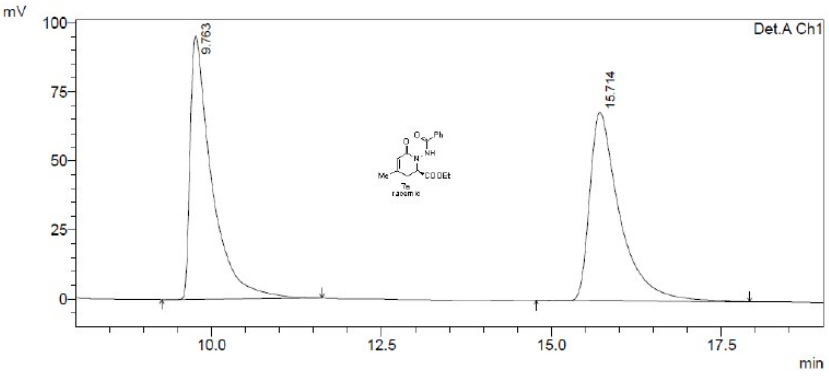
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		8.513	15.003	80.297	1.56	2.27	n.a.
2		9.693	949.781	3452.298	98.44	97.73	n.a.
Total:			**964.784**	**3532.594**	**100.00**	**100.00**	

6. Determination of the absolute configuration by comparison

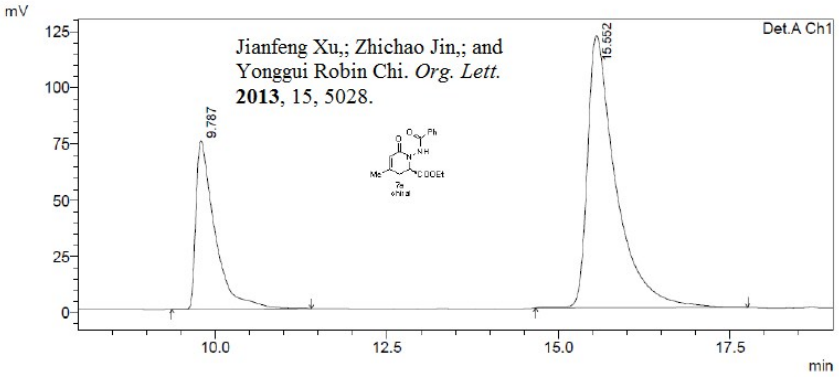



```
=====================================================================
                    Area Percent Report
=====================================================================

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


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1   9.529 PB    0.2915 8253.67090  402.76825  49.9614
   2  15.066 BB    0.4065 8266.42969  291.56198  50.0386

Totals :                  1.65201e4   694.33023
```





Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.763	2095405	95317	49.957	58.274
2	15.714	2099036	68250	50.043	41.726
Total		4194441	163567	100.000	100.000



Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.787	1452698	74906	29.289	38.187
2	15.552	3507144	121252	70.711	61.813
Total		4959841	196158	100.000	100.000