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

Enantioselective Cascade Double Michael Addition of 3-Nitro-2*H*-Chromenes and Acyclic Enones: Efficient Synthesis of Functionalized Tricyclic Chroman Derivatives

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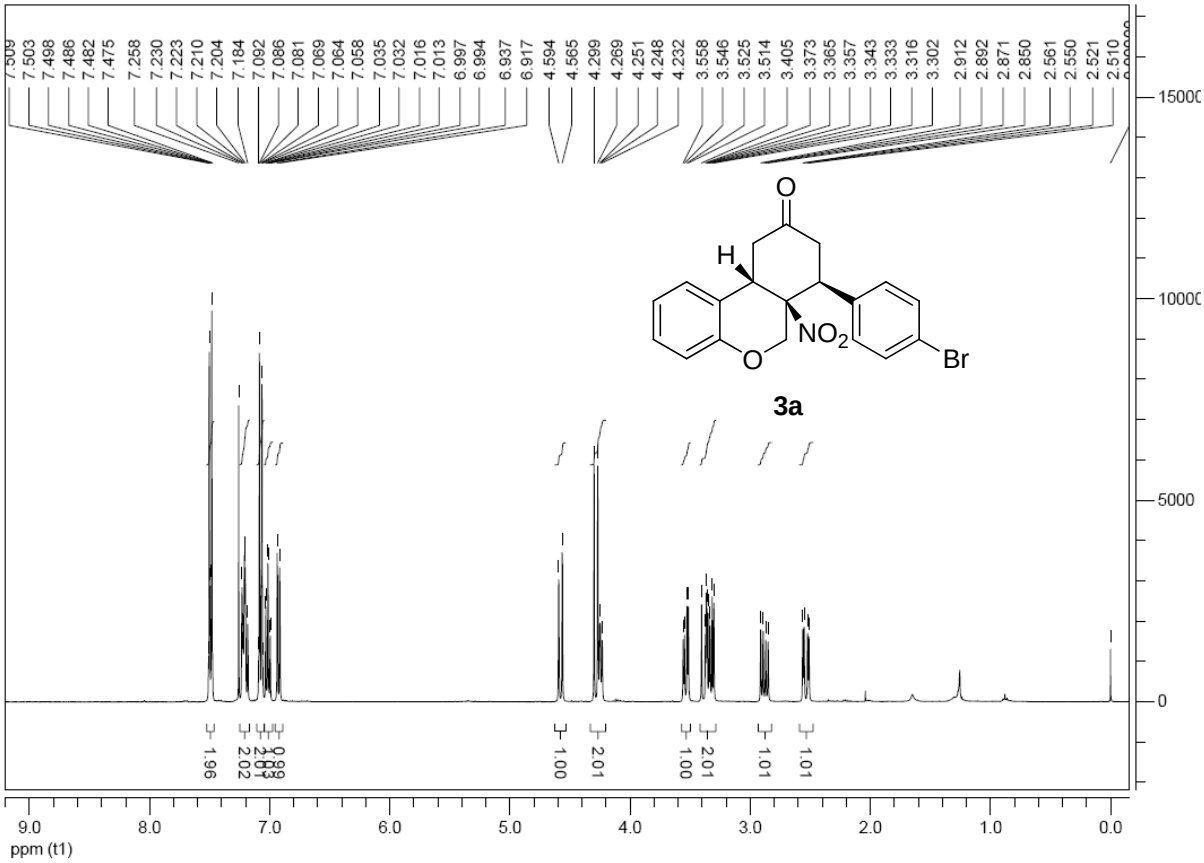
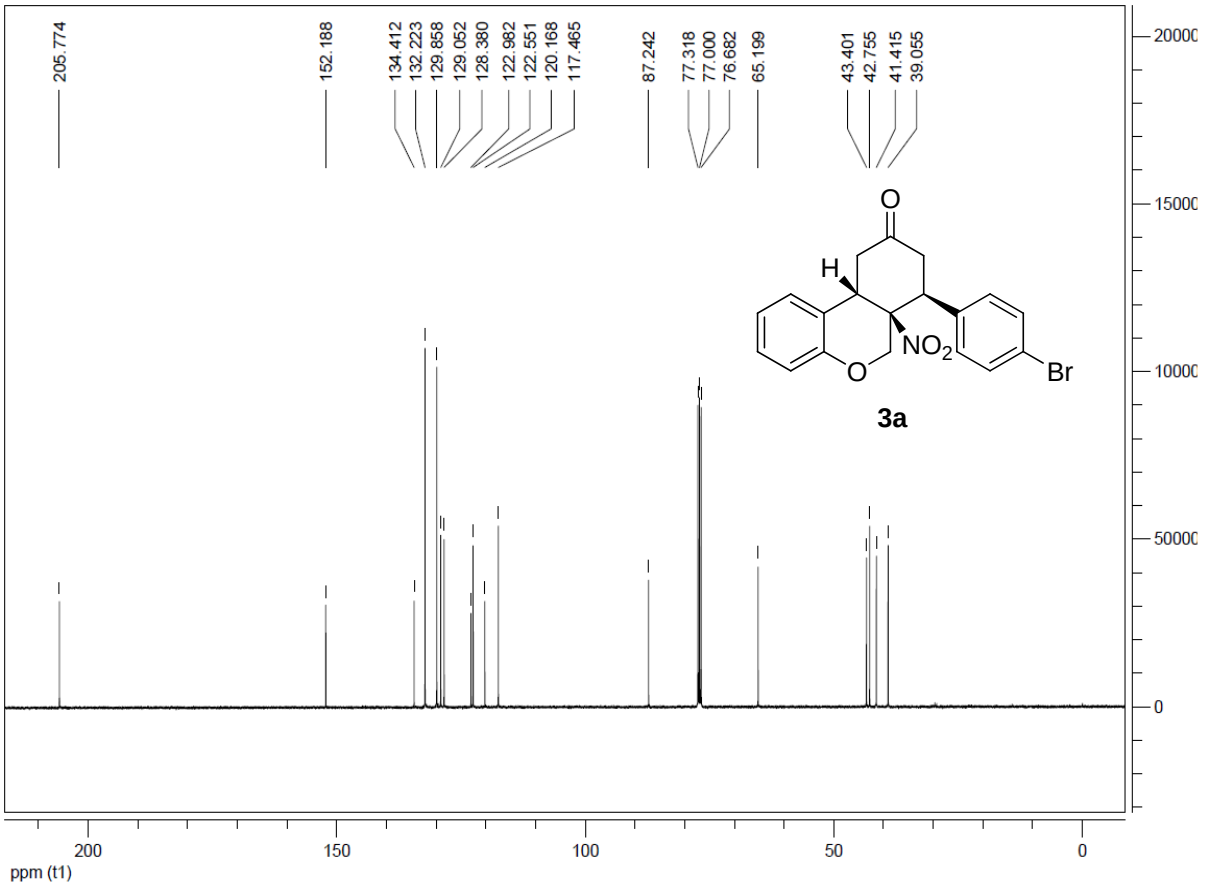
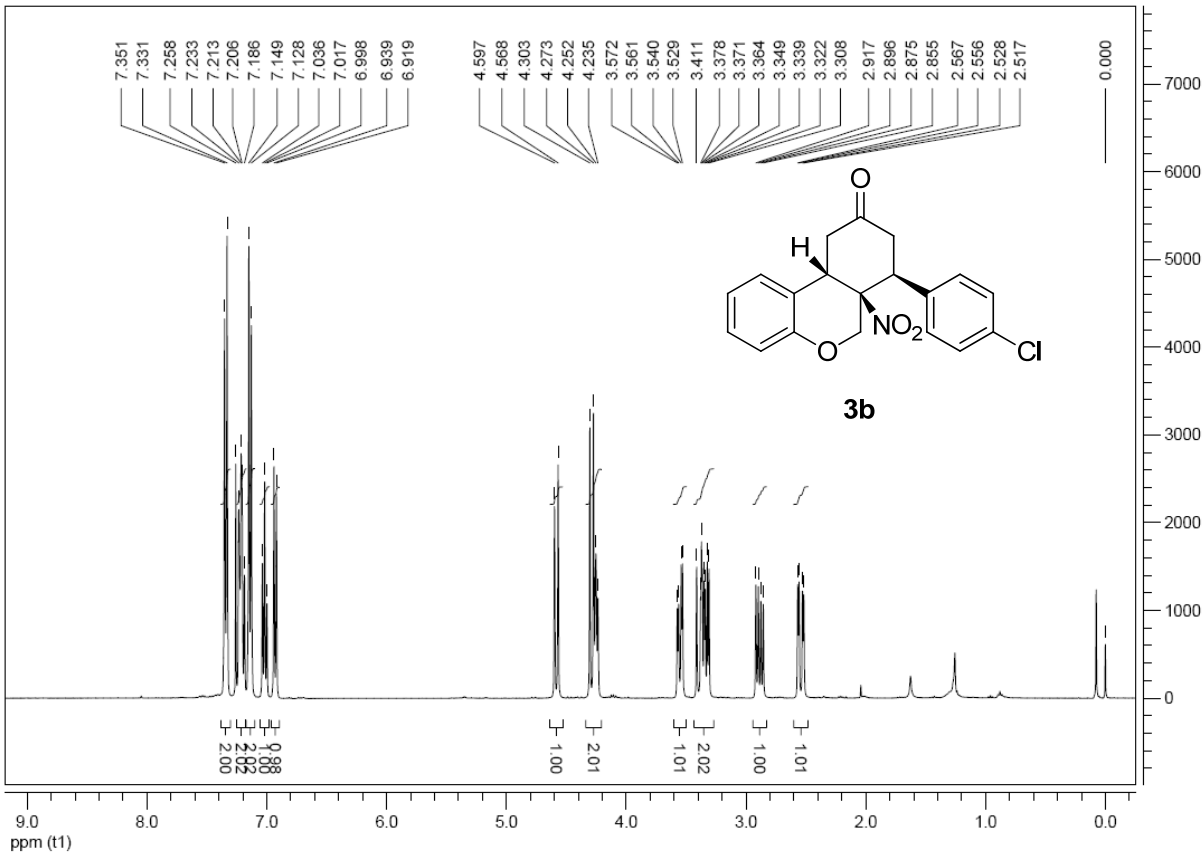
Supporting Information

Contents

Copies of ^{1}H and ^{13}C NMR spectra of products



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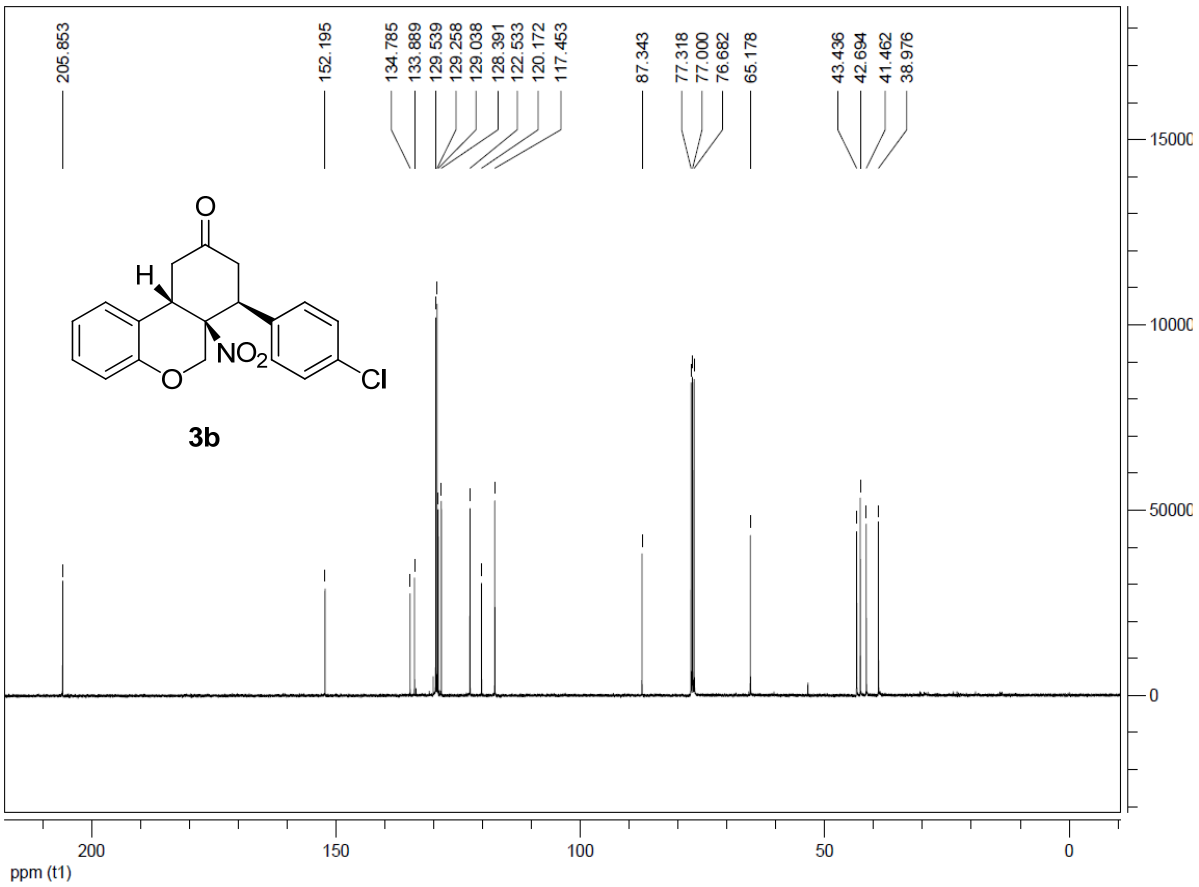
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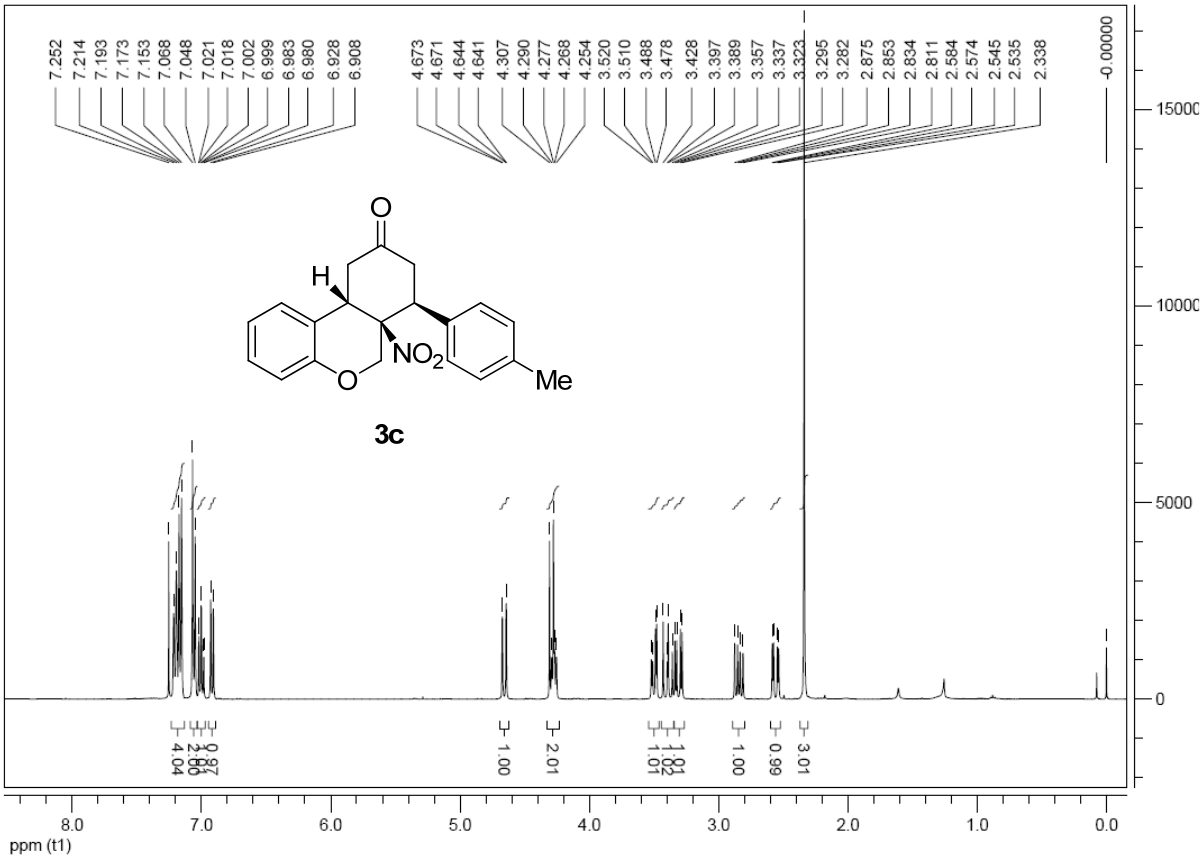
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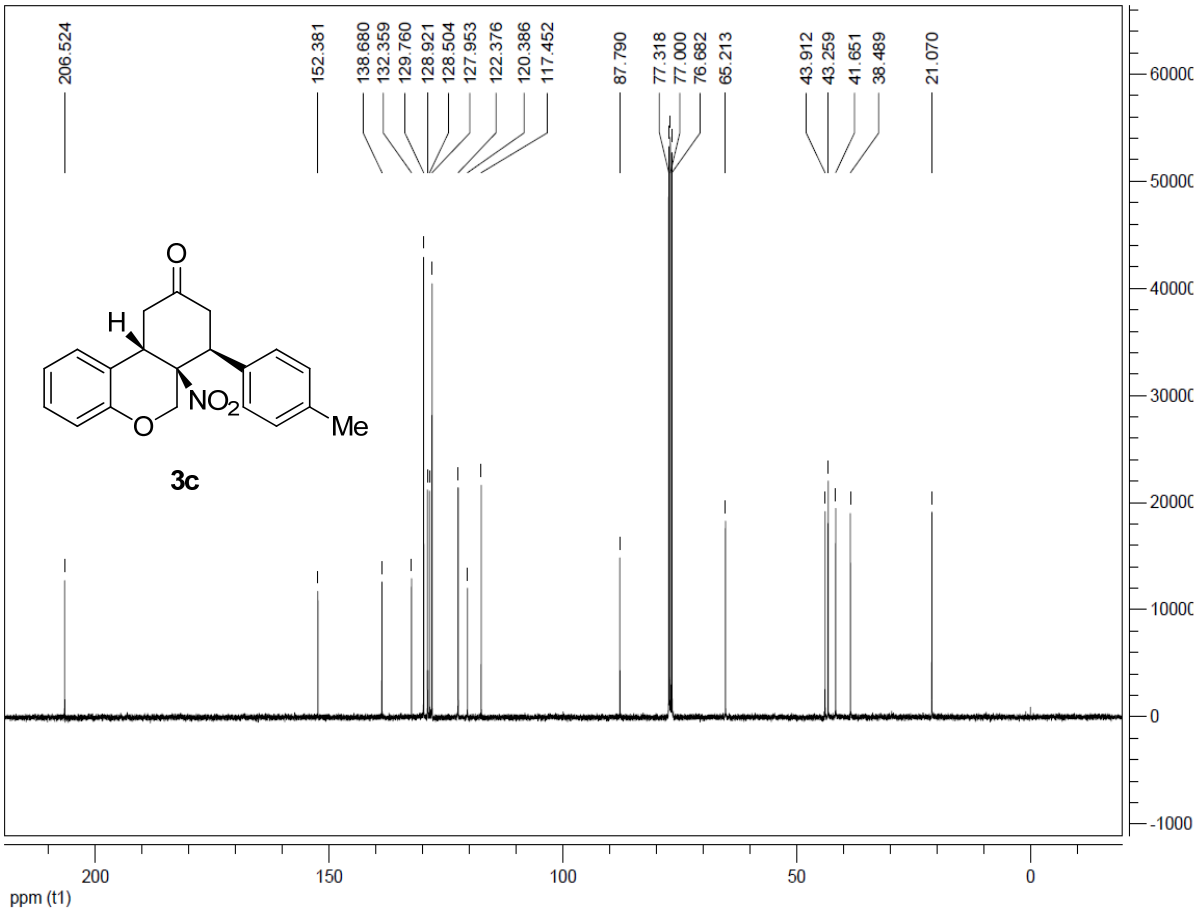
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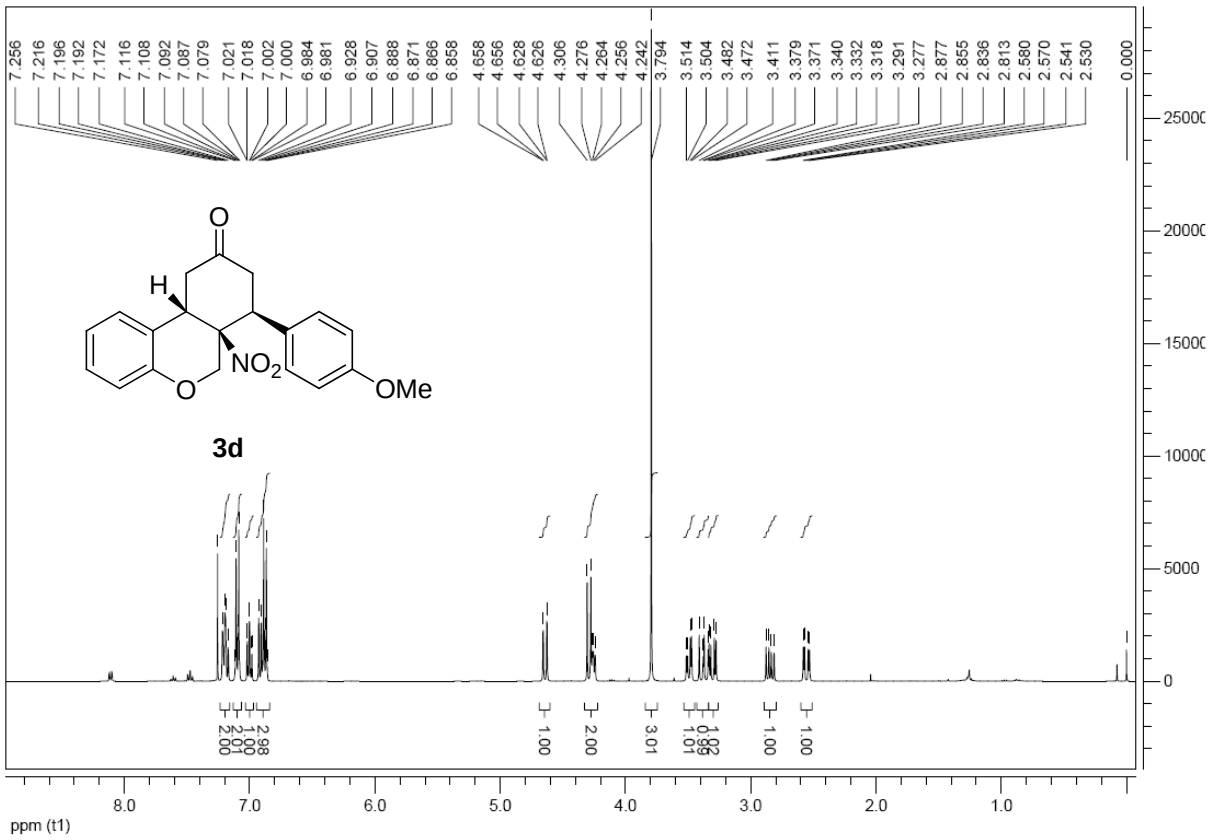
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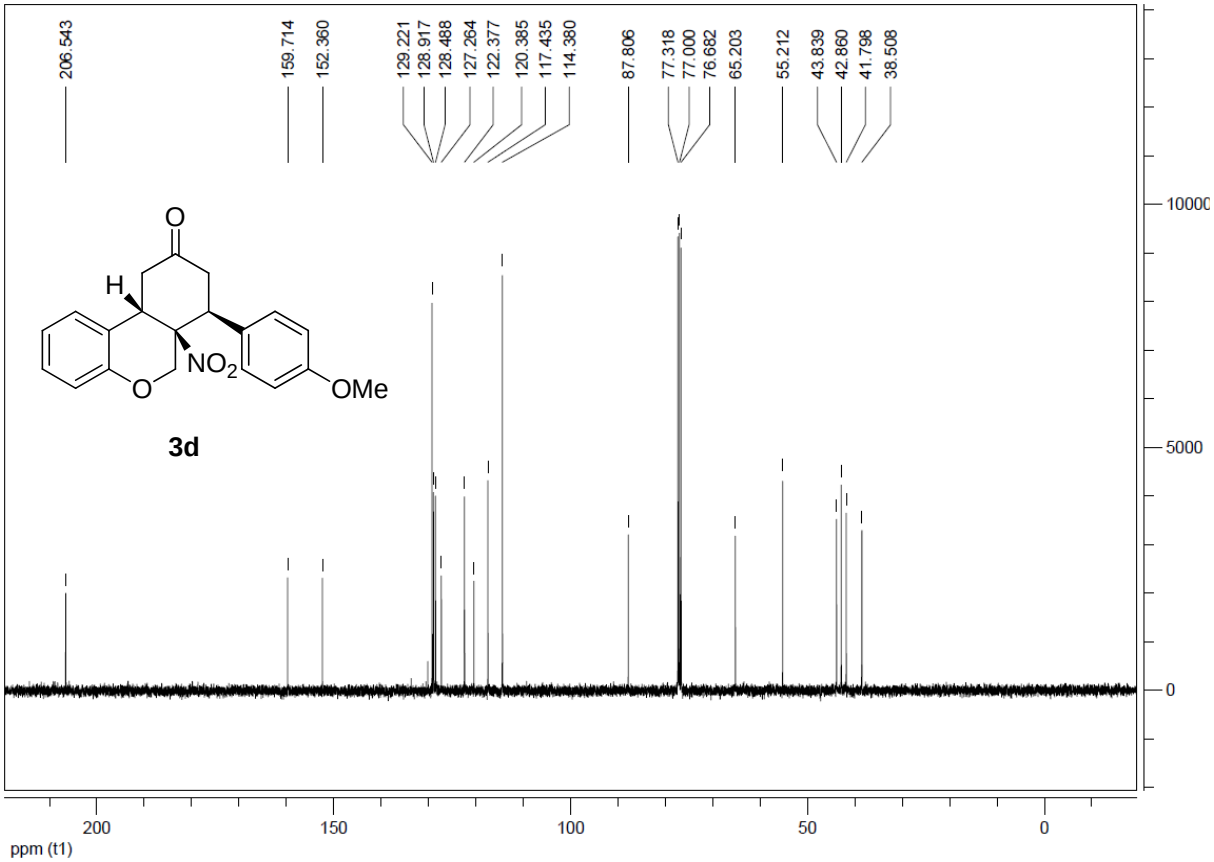
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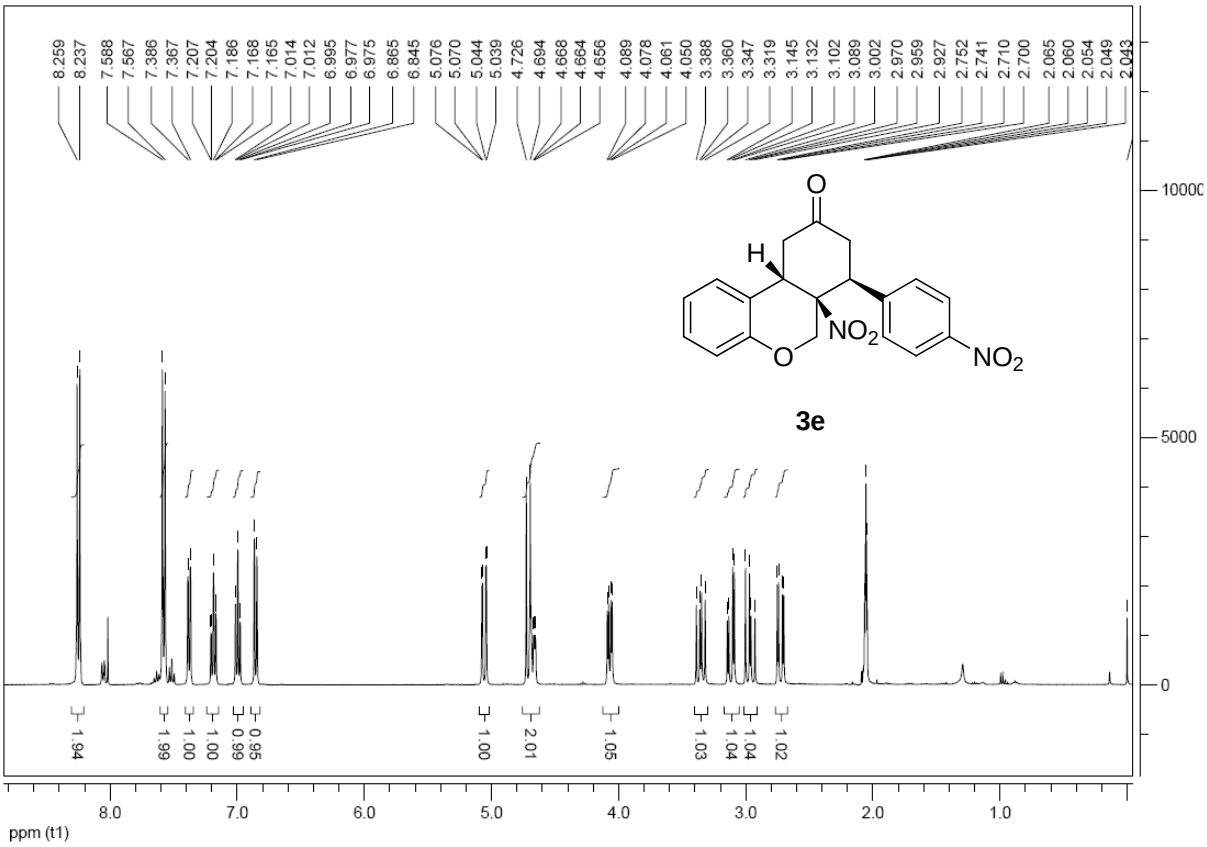
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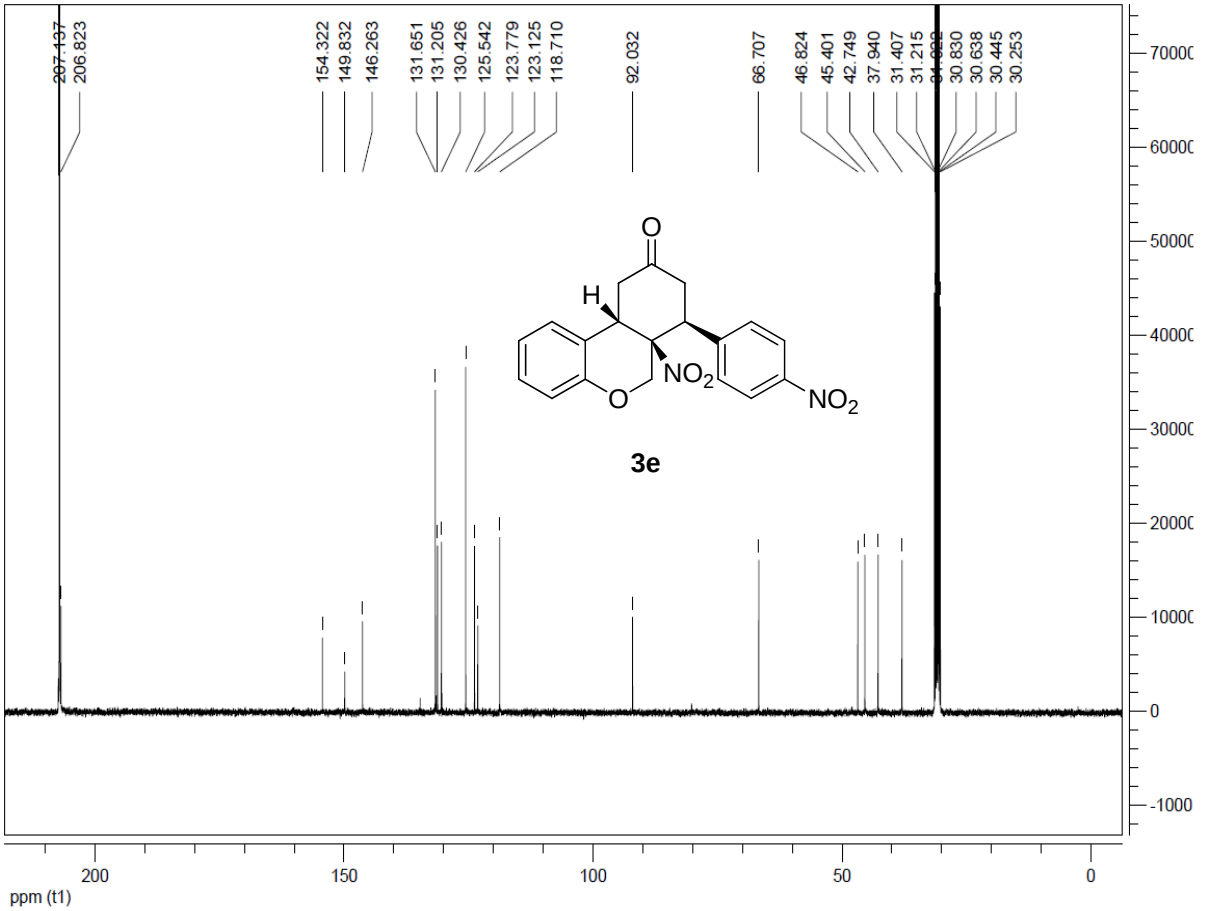
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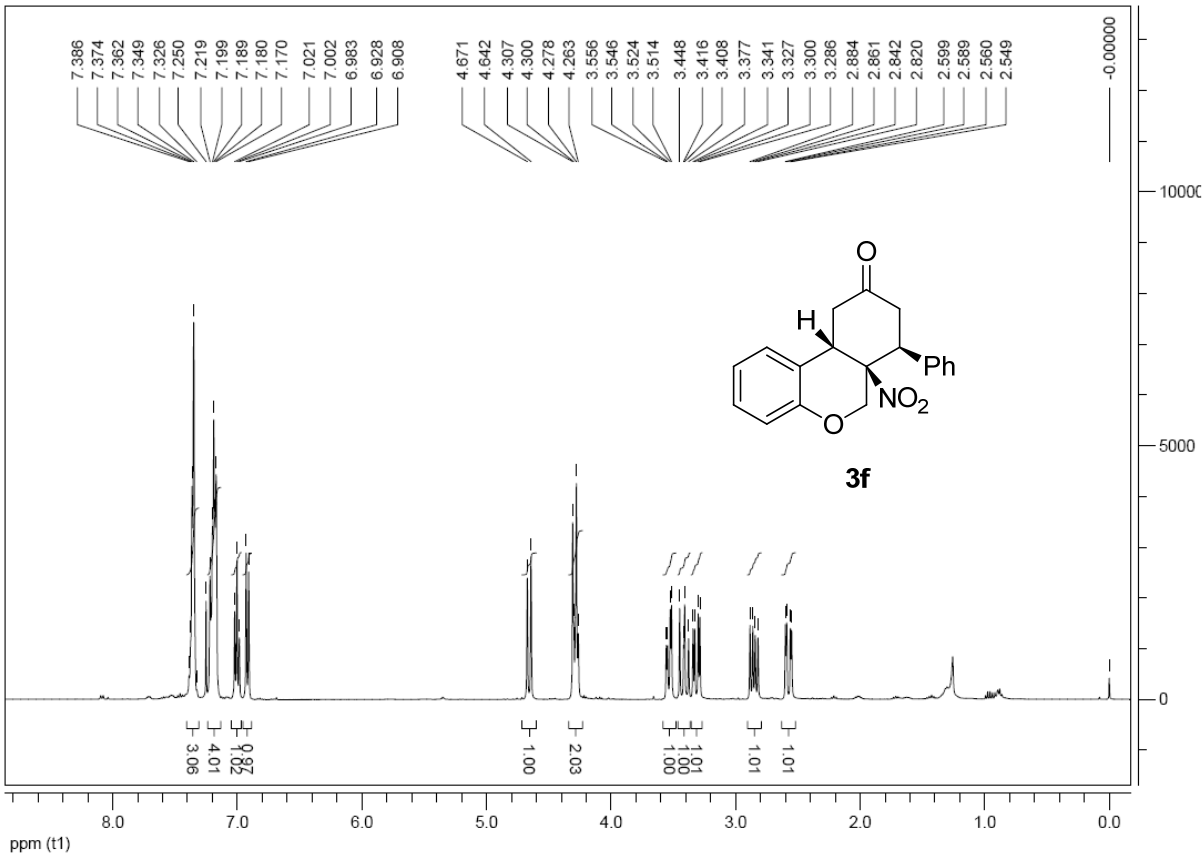
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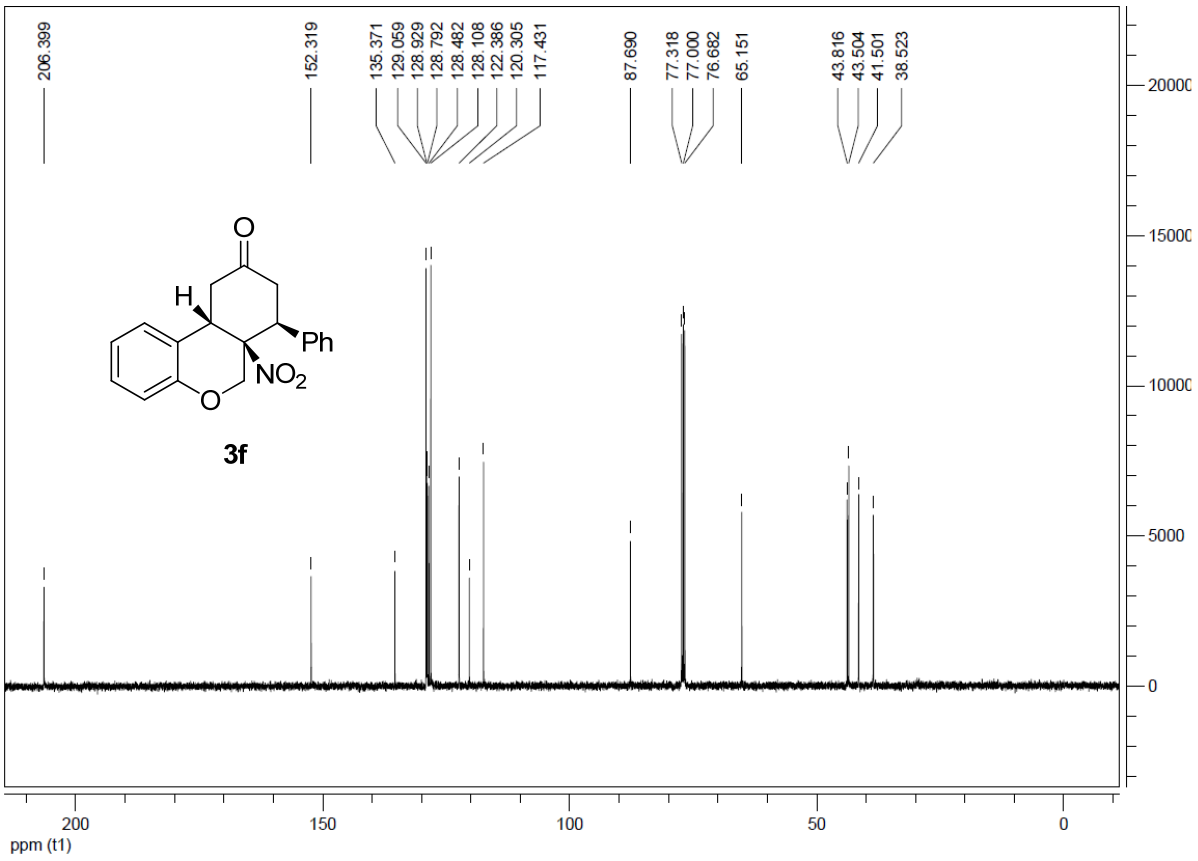
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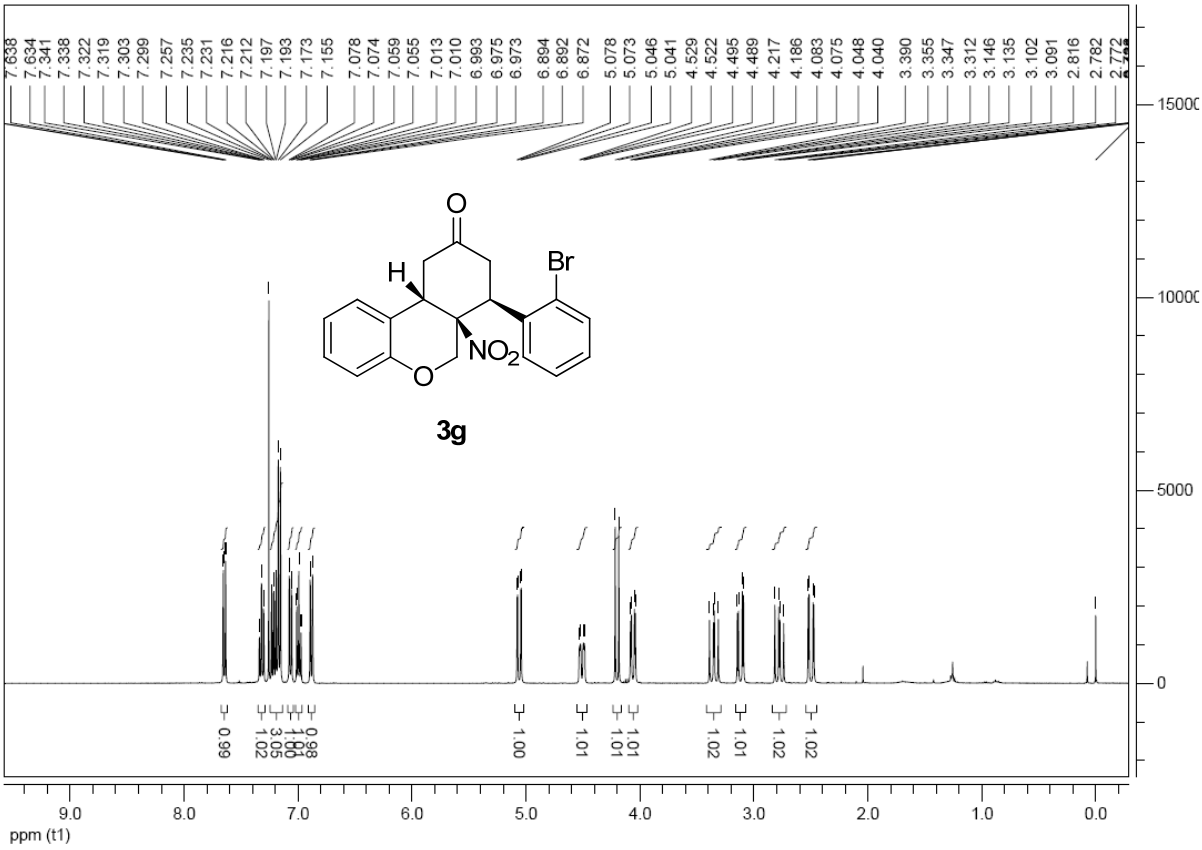
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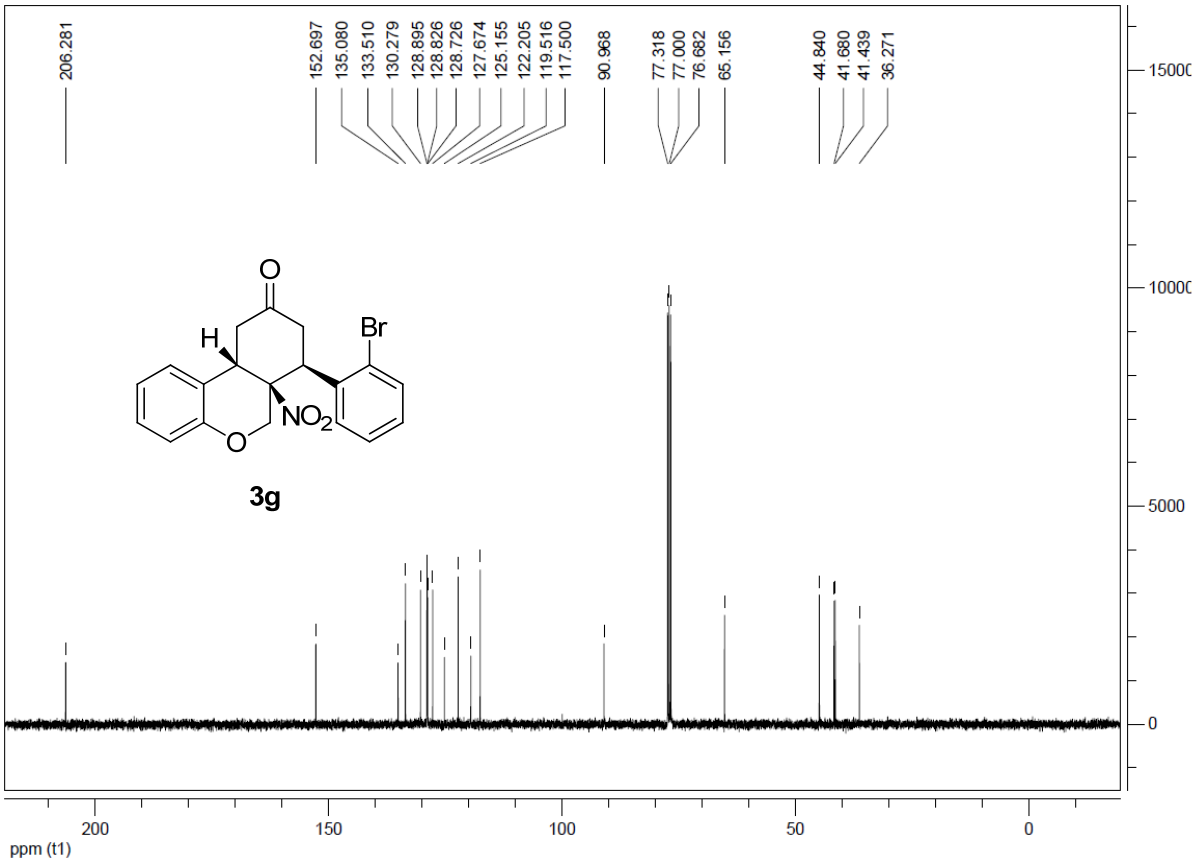
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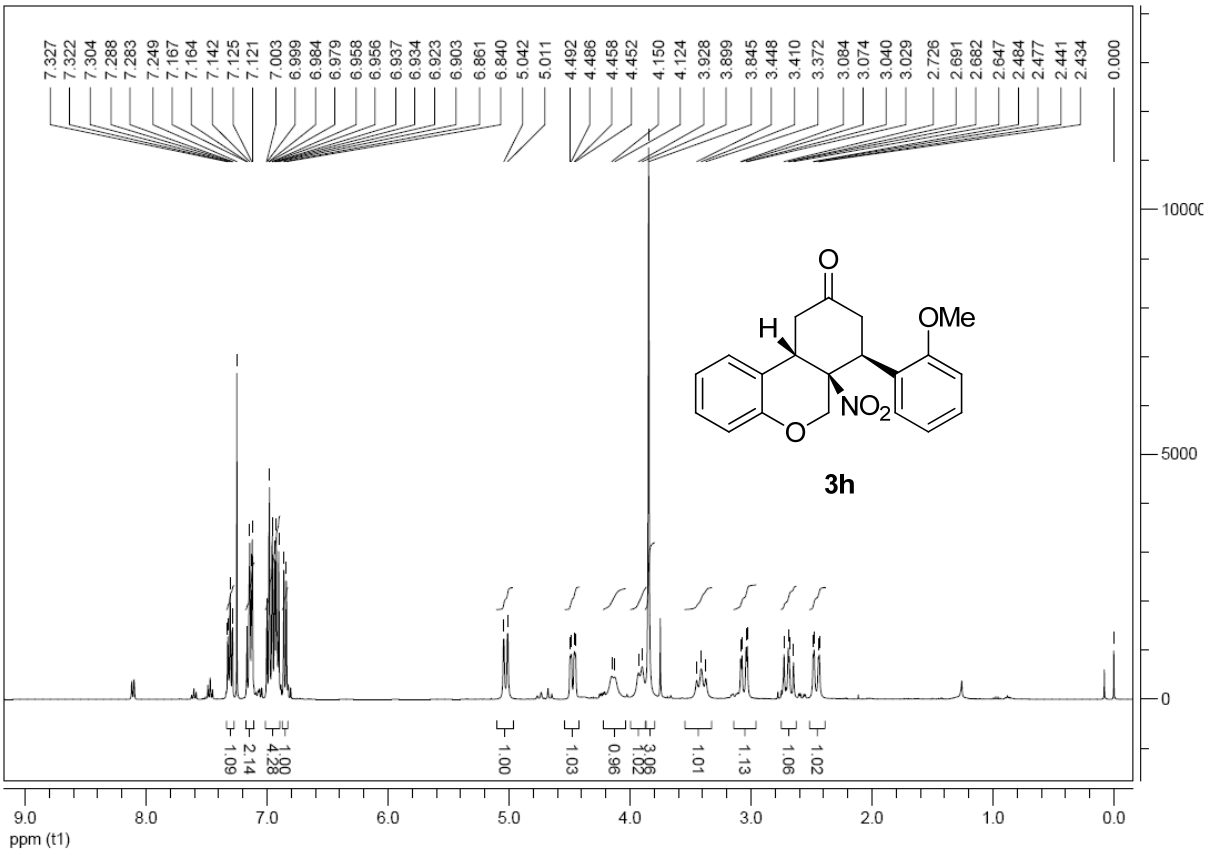
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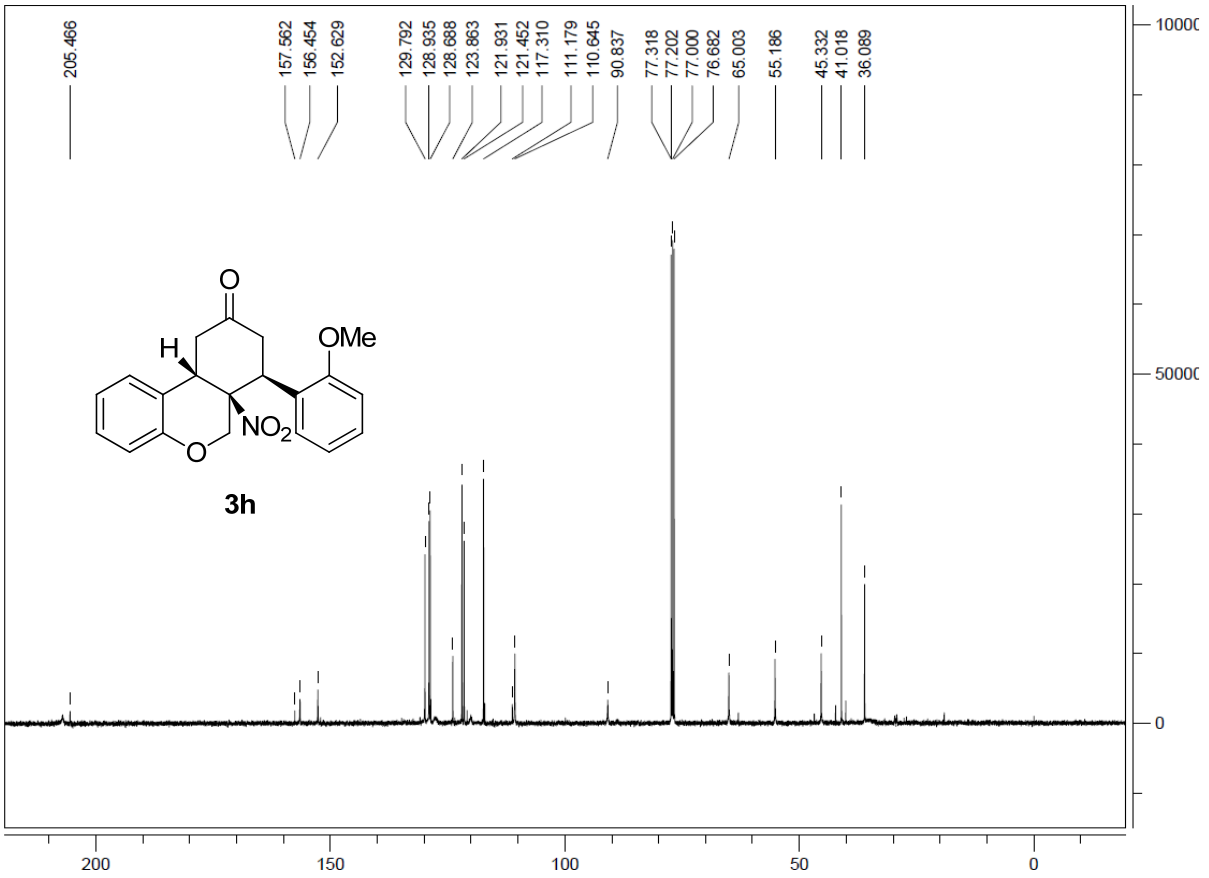
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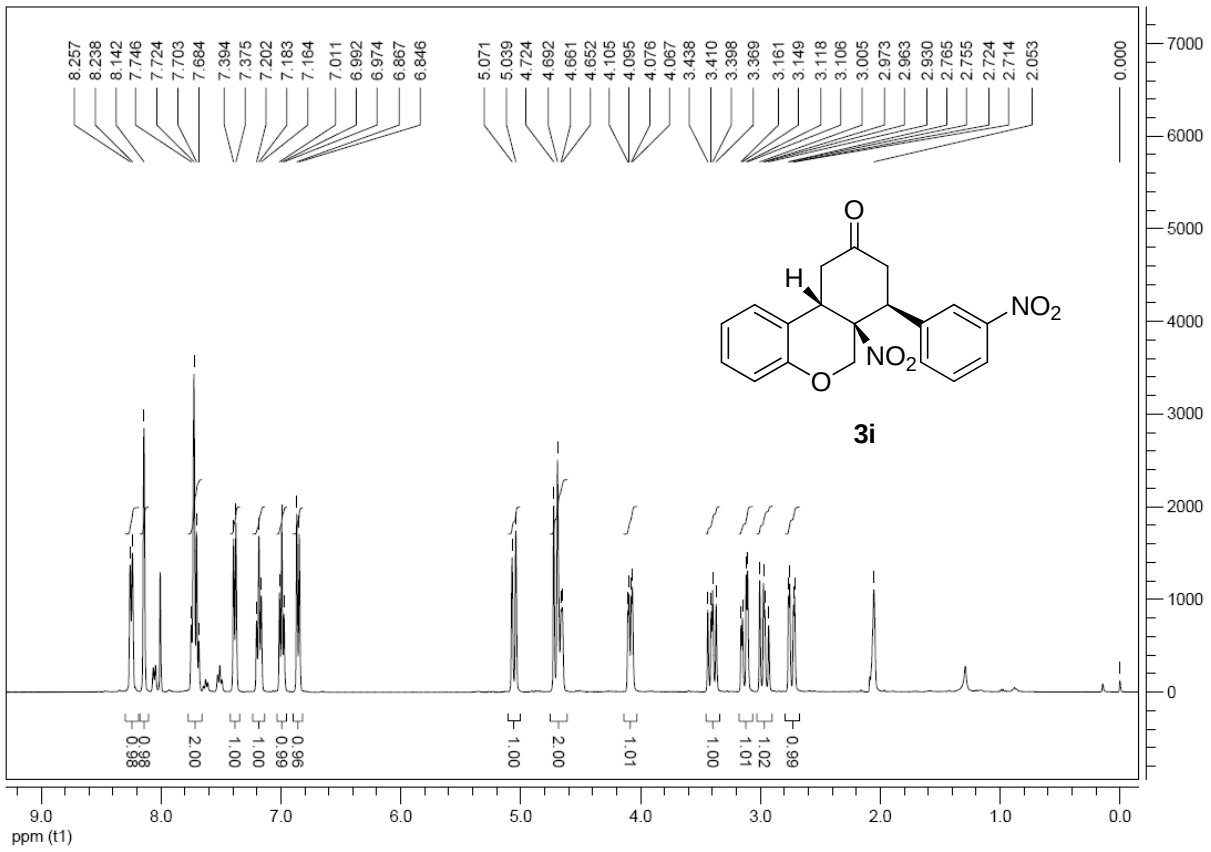
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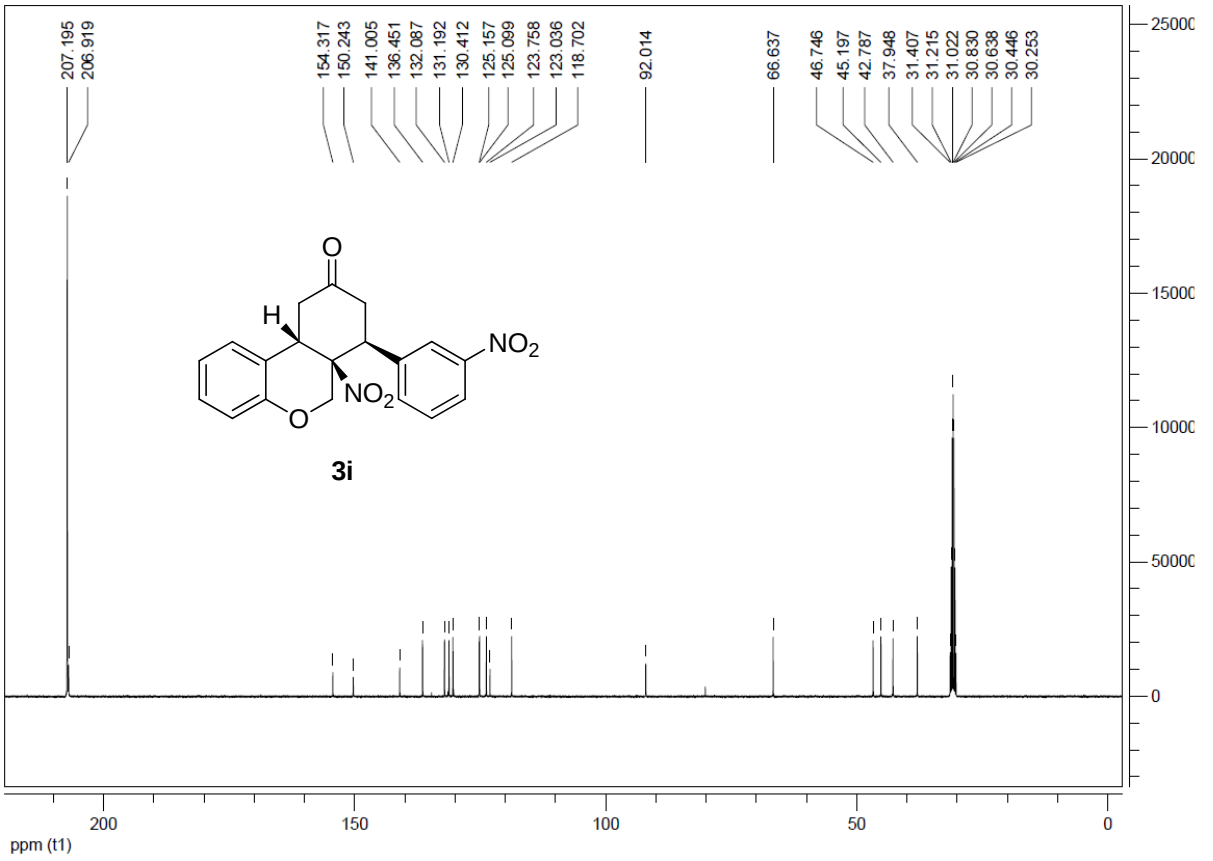
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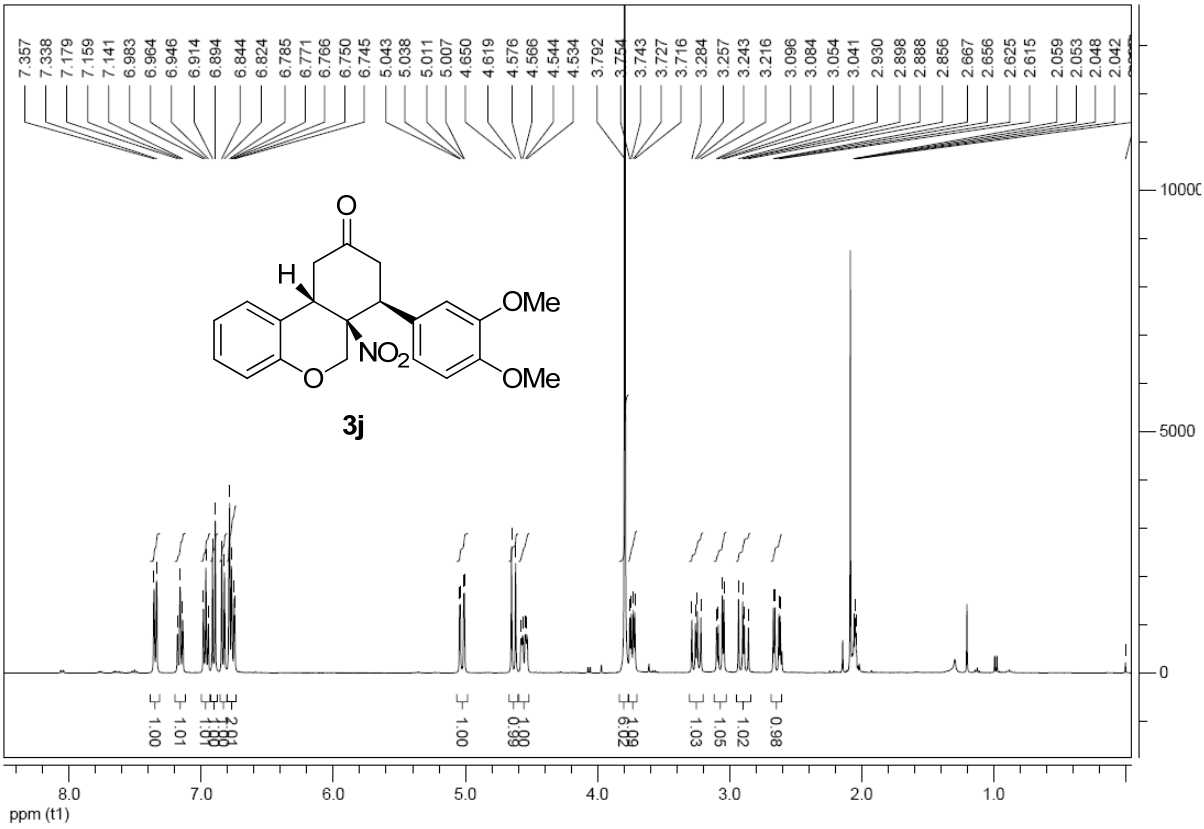
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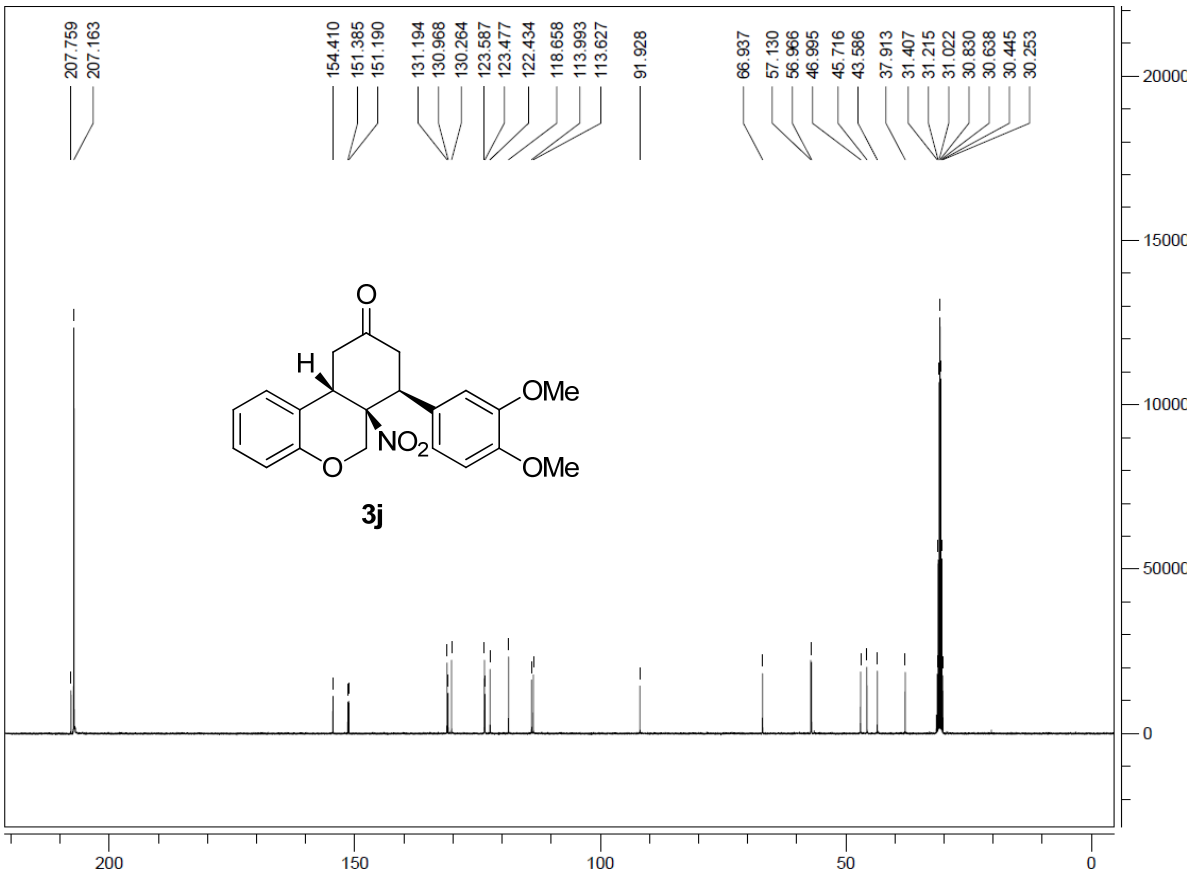
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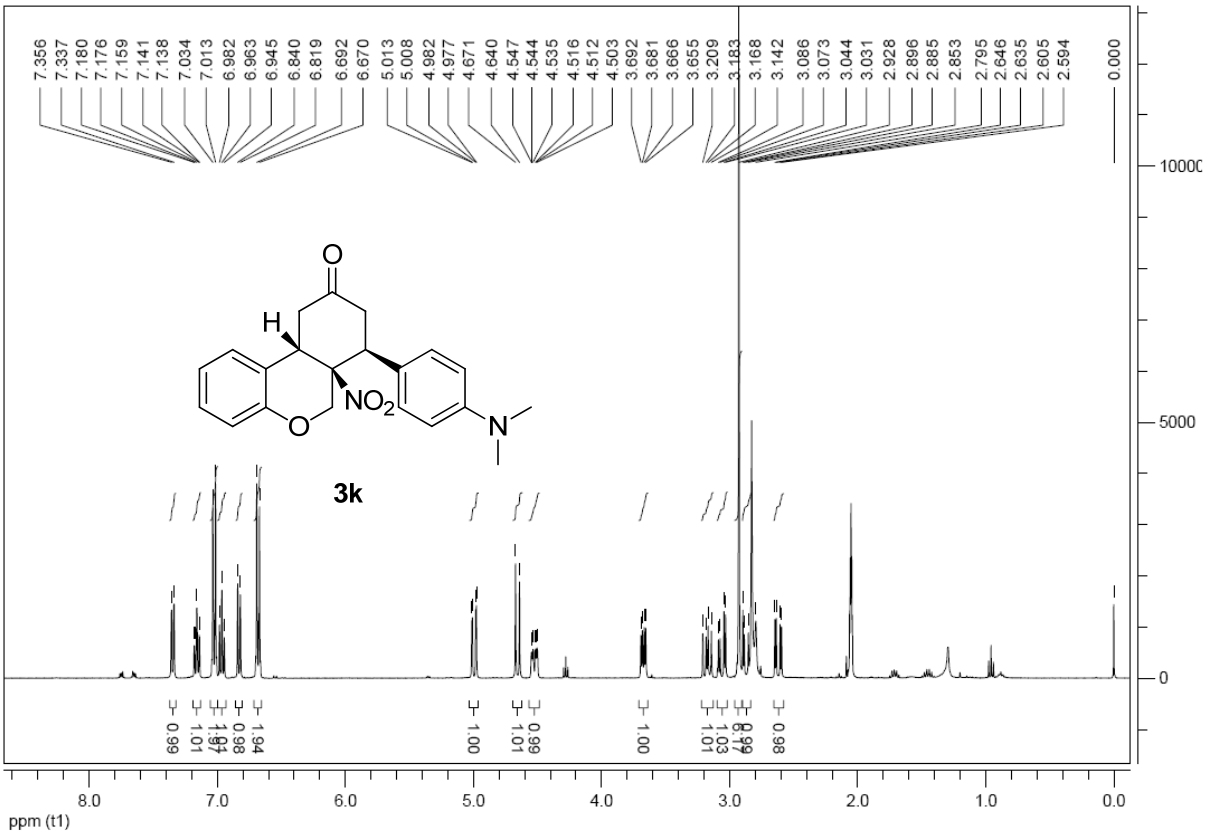
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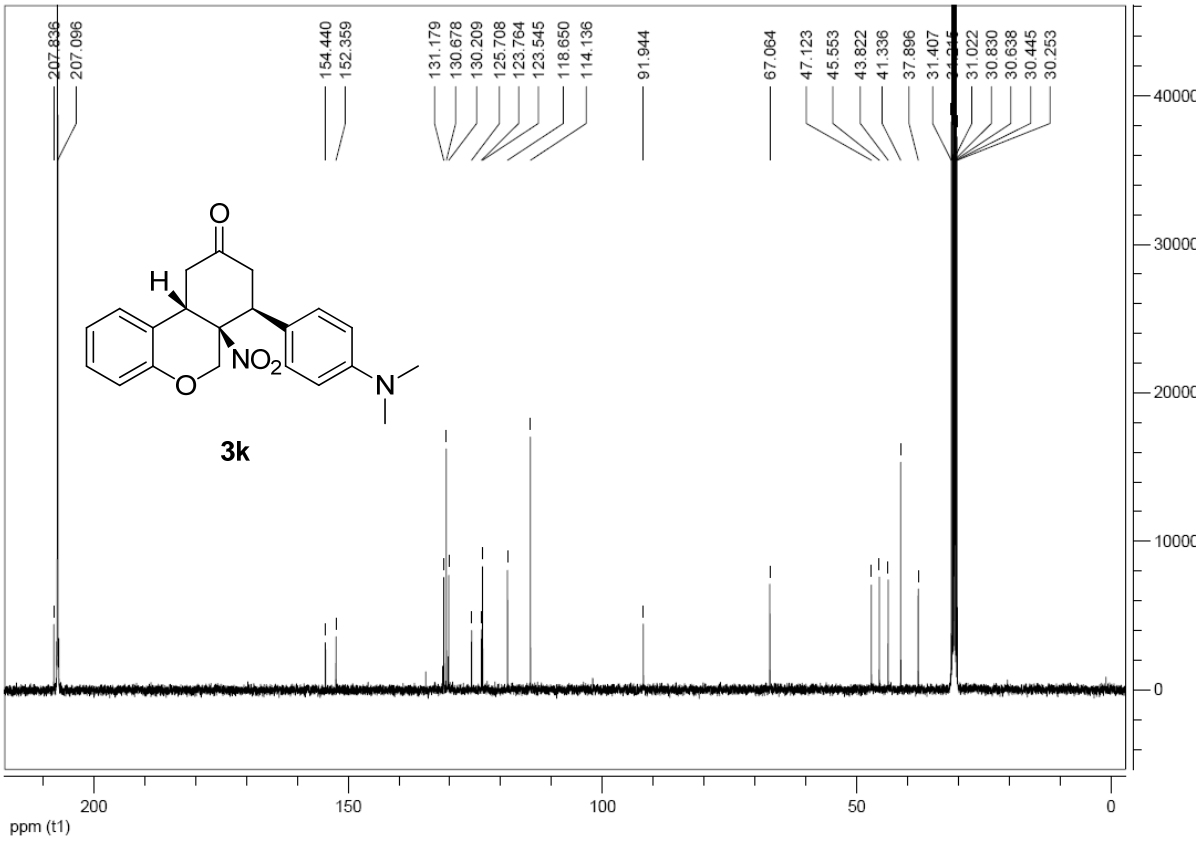
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152.359
131.179
130.678
130.209
125.708
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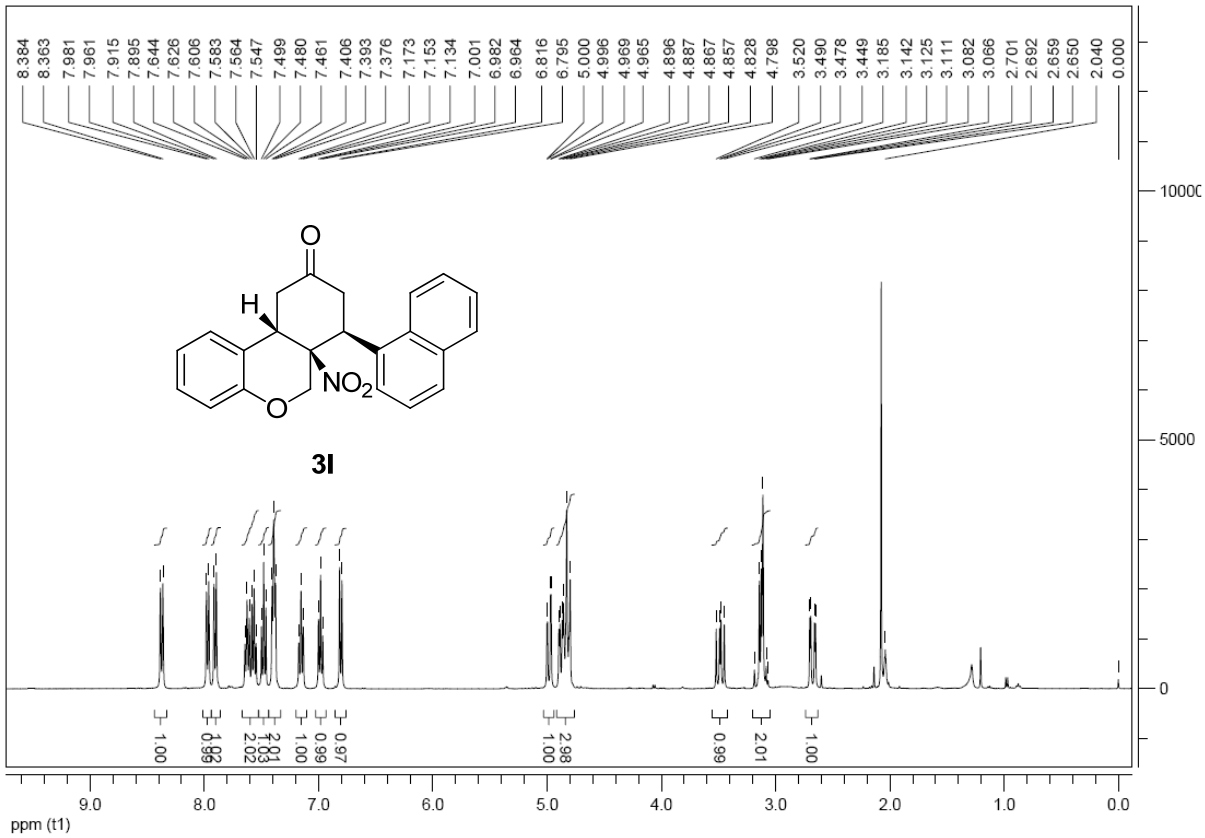
123.545
118.650
114.136
91.944
67.064
47.123
45.553
43.822
41.336
37.896
31.407
31.022
30.830
30.638
30.445
30.253
O
H
NO2
O
N
3k
40000
30000
20000
10000
0
200
150
100
50
0
ppm (t1)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8.363
7.981
7.961
7.915
7.895
7.644
7.626
7.606
7.583
7.564
7.547
7.499
7.480
7.461
7.406
7.393
7.376
7.173
7.153
7.134
7.001
6.982
6.964
6.816
6.795
5.000
4.996
4.969
4.965
4.896
4.887
4.867
4.857
4.828
4.798
3.520
3.490
3.478
3.449
3.185
3.142
3.125
3.111
3.082
3.066
2.701
2.692
2.659
2.650
2.040
0.000
O
H
NO2
O
3l
1.00
0.96
2.02
2.03
1.01
1.00
0.99
0.97
1.00
2.98
0.99
2.01
1.00
10000
5000
0
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7.0
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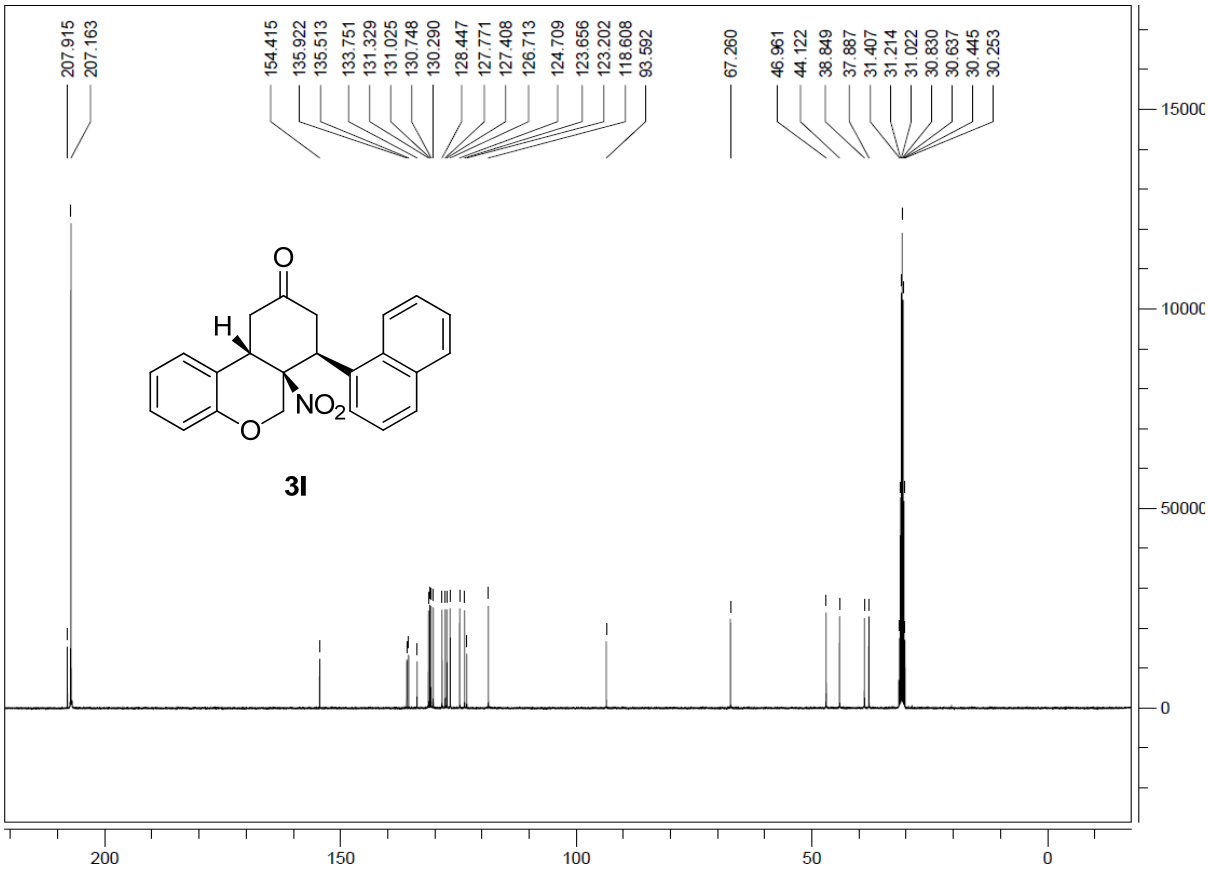
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)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131.329
131.025
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127.771
127.408
126.713
124.709
123.656
123.202
118.608
93.592
67.260
46.961
44.122
38.849
37.887
31.407
31.214
31.022
30.830
30.637
30.445
30.253
O
H
NO2
O
3l
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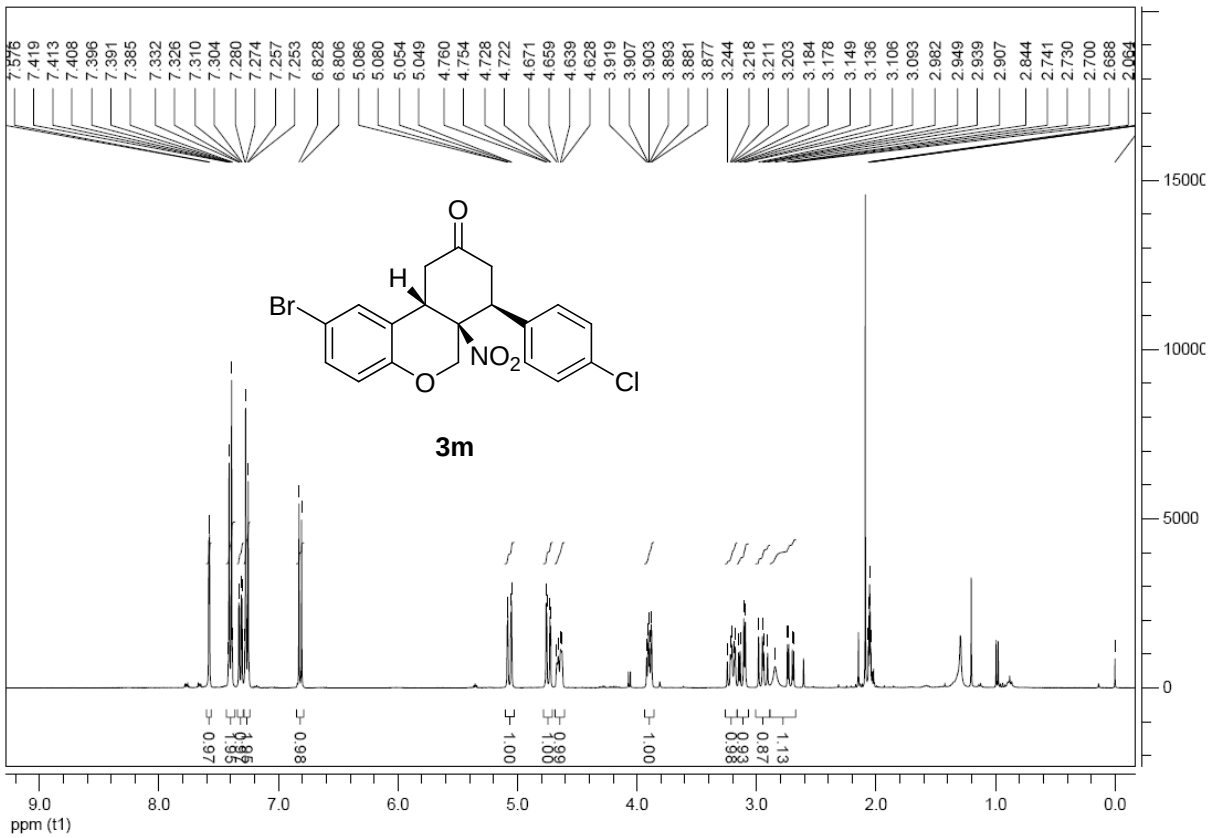
10000
5000
0
200
150
100
50
0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7.419
7.413
7.408
7.396
7.391
7.385
7.332
7.326
7.310
7.304
7.280
7.274
7.257
7.253
6.828
6.806
5.086
5.080
5.054
5.049
4.760
4.754
4.728
4.722
4.671
4.659
4.639
4.628
3.919
3.907
3.903
3.893
3.881
3.877
3.244
3.218
3.211
3.203
3.184
3.178
3.149
3.136
3.106
3.093
2.982
2.949
2.939
2.907
2.844
2.741
2.730
2.700
2.688
2.064
O
H
Br
NO2
O
Cl
3m
15000
10000
5000
0
0.97
1.95
0.98
1.00
0.99
1.00
0.88
0.87
1.13
9.0
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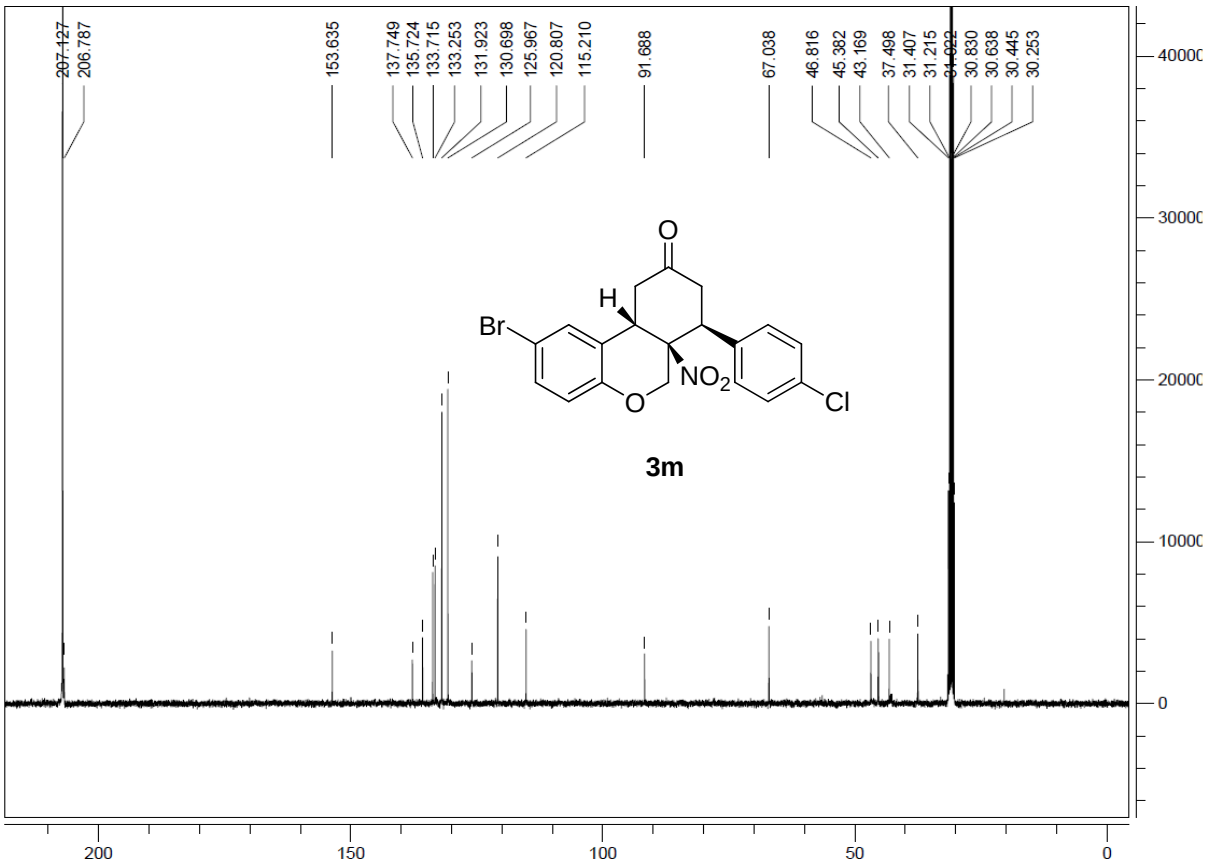
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)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206.787
153.635
137.749
135.724
133.715
133.253
131.923
130.698
125.967
120.807
115.210
91.688
67.038
46.816
45.382
43.169
37.498
31.407
31.215
30.830
30.638
30.445
30.253
O
H
Br
NO2
O
Cl
3m
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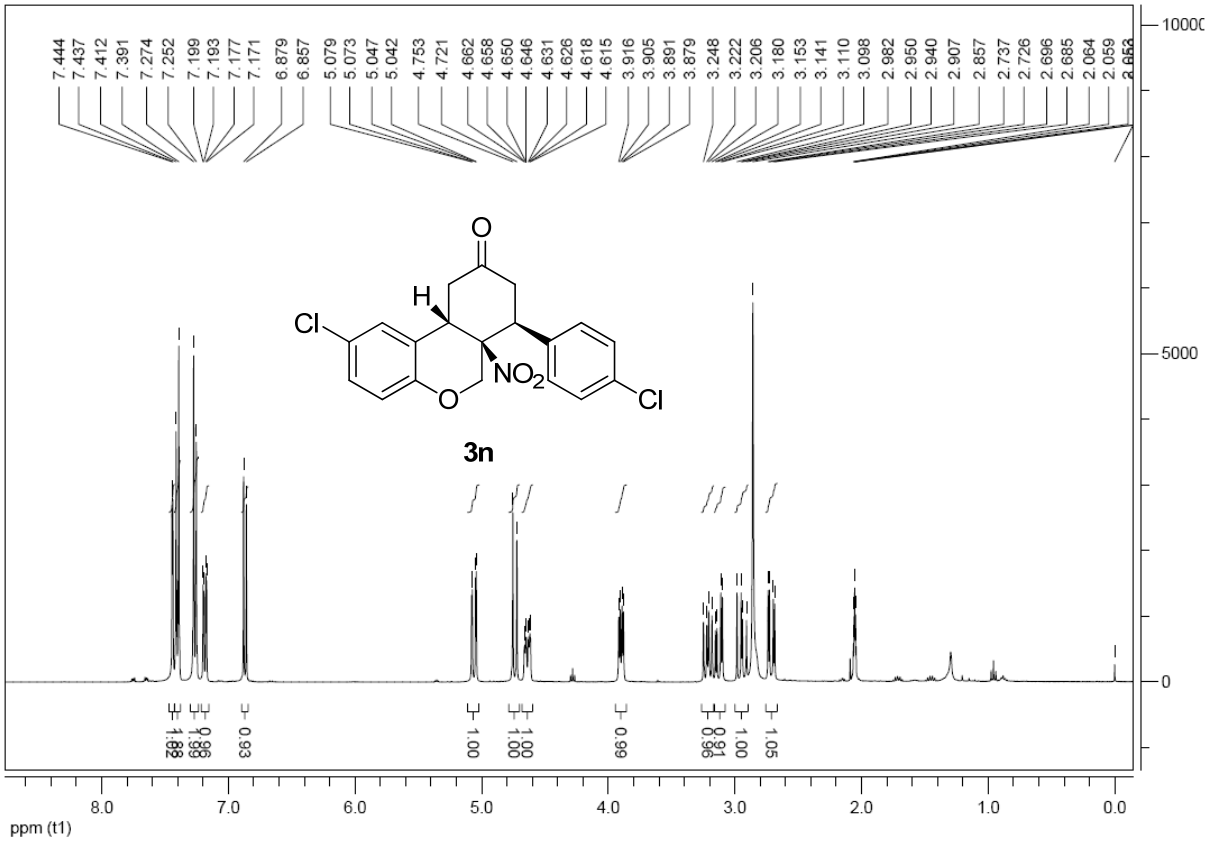
30000
20000
10000
0
200
150
100
50
0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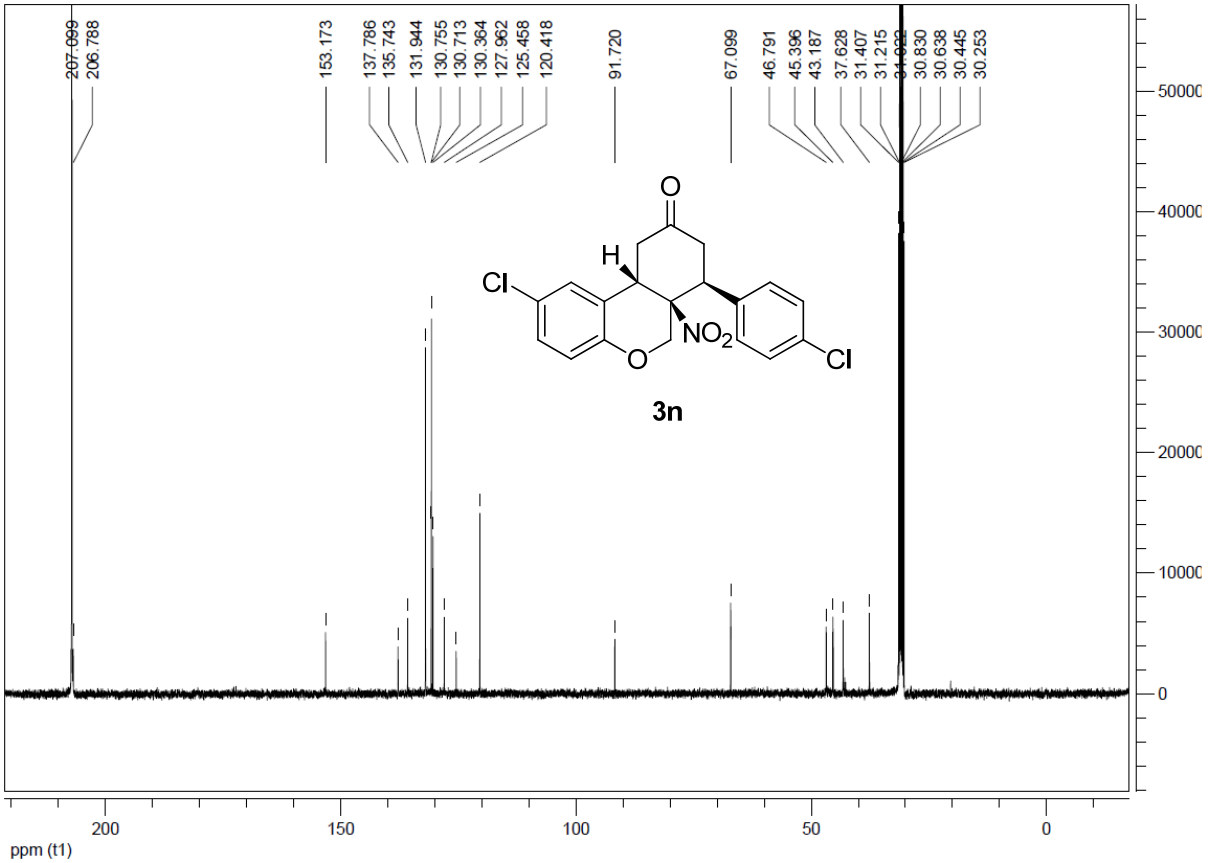
7.437
7.412
7.391
7.274
7.252
7.199
7.193
7.177
7.171
6.879
6.857
5.079
5.073
5.047
5.042
4.753
4.721
4.662
4.658
4.650
4.646
4.631
4.626
4.618
4.615
3.916
3.905
3.891
3.879
3.248
3.222
3.206
3.180
3.153
3.141
3.110
3.098
2.982
2.950
2.940
2.907
2.857
2.737
2.726
2.696
2.685
2.064
2.059
O
H
Cl
NO2
O
Cl
3n
0.93
1.00
1.00
0.99
0.96
1.00
1.05
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)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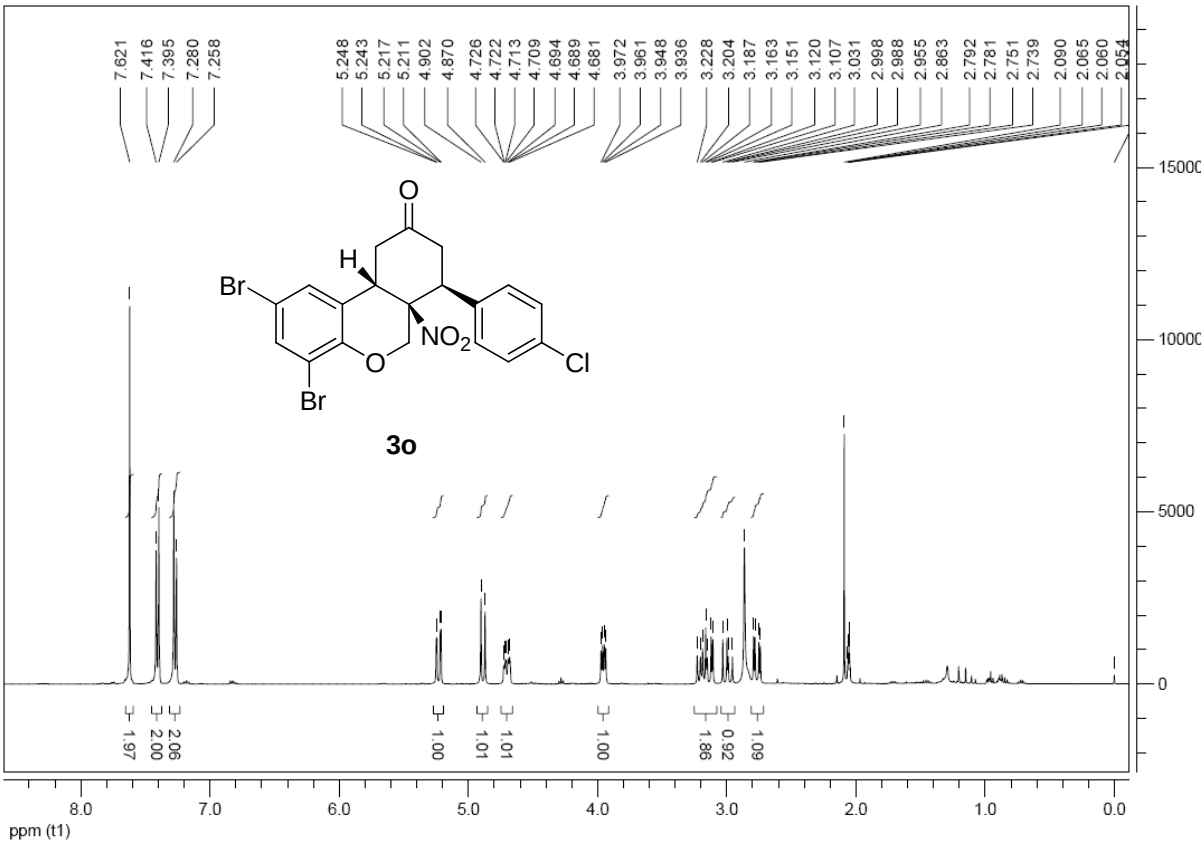
130.755
130.713
130.364
127.962
125.458
120.418
91.720
67.099
46.791
45.396
43.187
37.628
31.407
31.215
30.830
30.638
30.445
30.253
O
H
Cl
NO2
O
Cl
3n
200
150
100
50
0
ppm (t1)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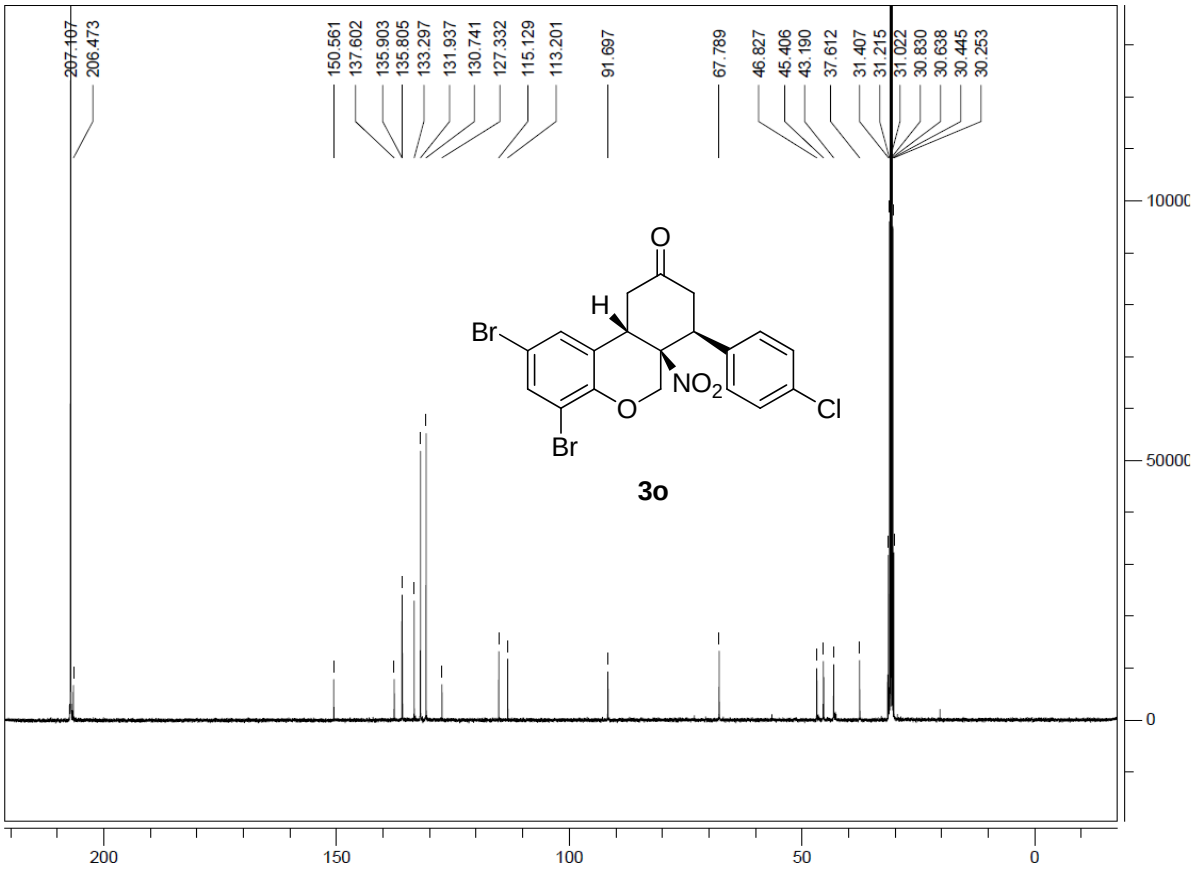
7.280
7.258
5.248
5.243
5.217
5.211
4.902
4.870
4.726
4.722
4.713
4.709
4.694
4.689
4.681
3.972
3.961
3.948
3.936
3.228
3.204
3.187
3.163
3.151
3.120
3.107
3.031
2.998
2.988
2.955
2.863
2.792
2.781
2.751
2.739
2.090
2.065
2.060
2.054
O
H
Br
NO2
O
Cl
Br
3o
1.97
2.00
2.06
1.00
1.01
1.01
1.00
1.86
0.92
1.09
15000
10000
5000
0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)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131.937
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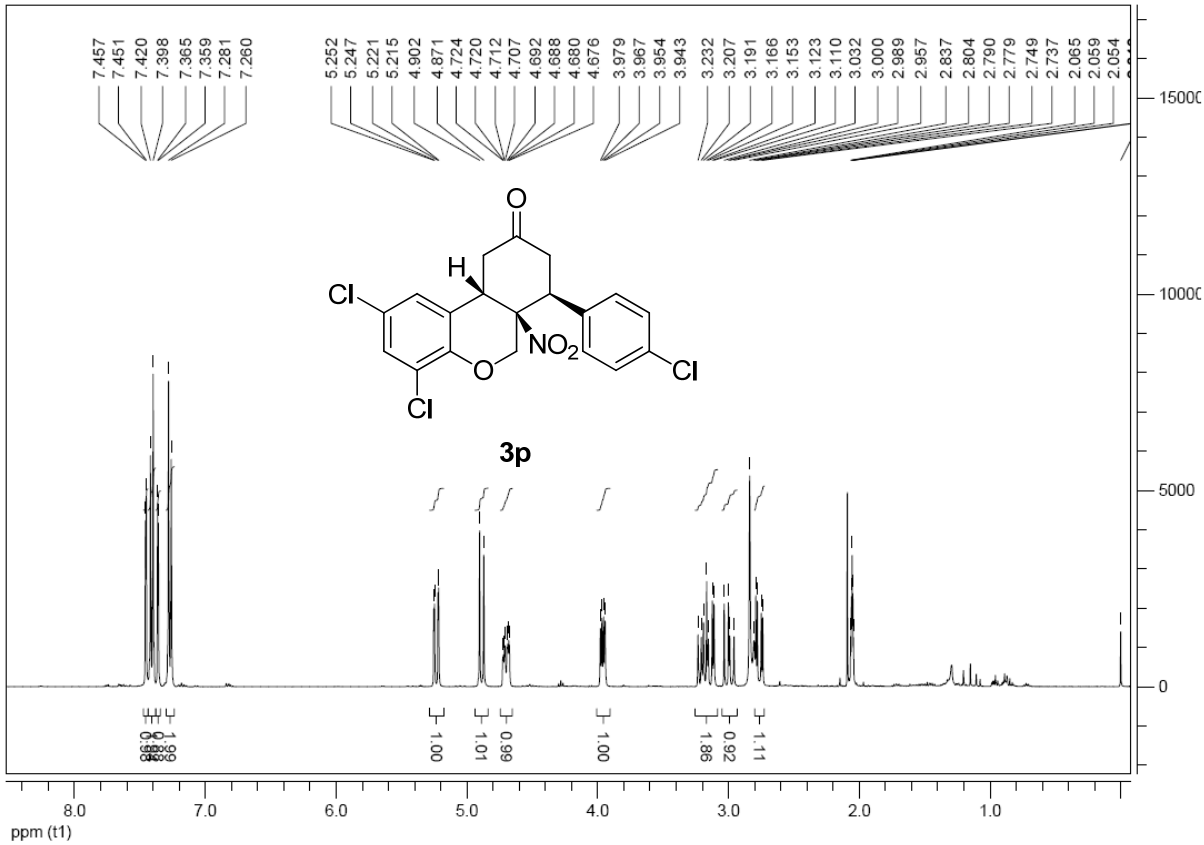
127.332
115.129
113.201
91.697
67.789
46.827
45.406
43.190
37.612
31.407
31.215
31.022
30.830
30.638
30.445
30.253
O
H
Br
NO2
O
Cl
Br
3o
10000
5000
0
200
150
100
50
0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7.398
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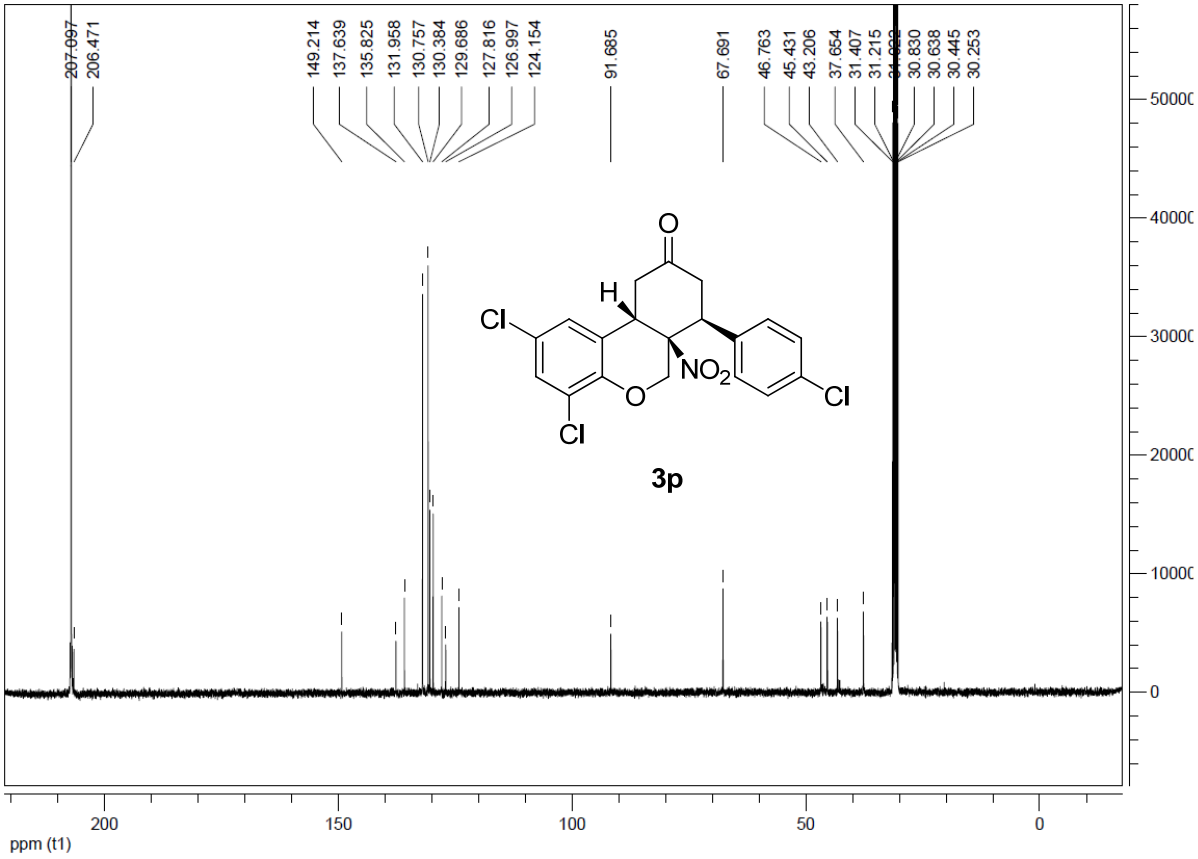
7.359
7.281
7.260
5.252
5.247
5.221
5.215
4.902
4.871
4.724
4.720
4.712
4.707
4.692
4.688
4.680
4.676
3.979
3.967
3.954
3.943
3.232
3.207
3.191
3.166
3.153
3.123
3.110
3.032
3.000
2.989
2.957
2.837
2.804
2.790
2.779
2.749
2.737
2.065
2.059
2.054
O
H
Cl
NO2
O
Cl
Cl
3p
1.00
1.01
0.99
1.00
1.86
0.92
1.11
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
ppm (t1)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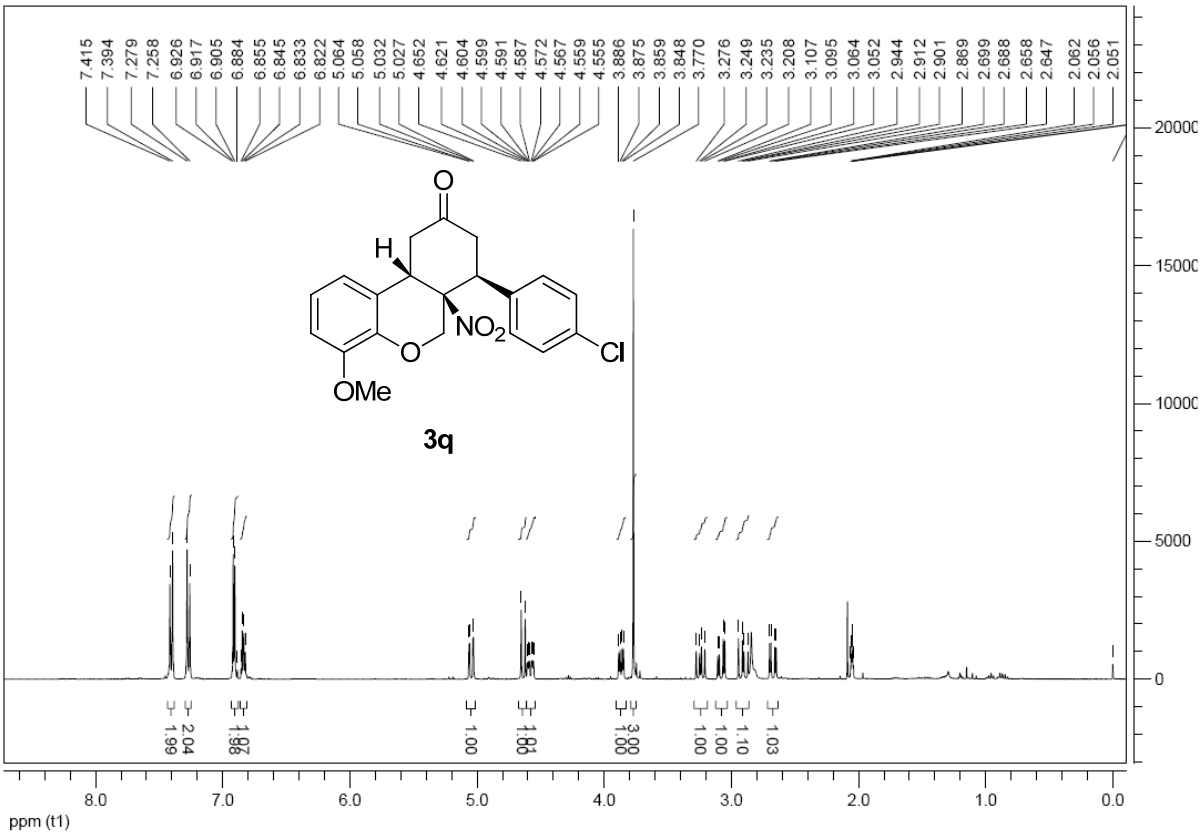
130.384
129.686
127.816
126.997
124.154
91.685
67.691
46.763
45.431
43.206
37.654
31.407
31.215
30.830
30.638
30.445
30.253
O
H
Cl
NO2
O
Cl
Cl
3p
200
150
100
50
0
ppm (t1)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7.258
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6.917
6.905
6.884
6.855
6.845
6.833
6.822
5.064
5.058
5.032
5.027
4.652
4.621
4.604
4.599
4.591
4.587
4.572
4.567
4.559
4.555
3.886
3.875
3.859
3.848
3.770
3.276
3.249
3.235
3.208
3.107
3.095
3.064
3.052
2.944
2.912
2.901
2.869
2.699
2.688
2.658
2.647
2.062
2.056
2.051
O
H
NO2
O
Cl
OMe
3q
1.99
2.04
1.96
1.00
1.80
3.00
1.00
1.10
1.03
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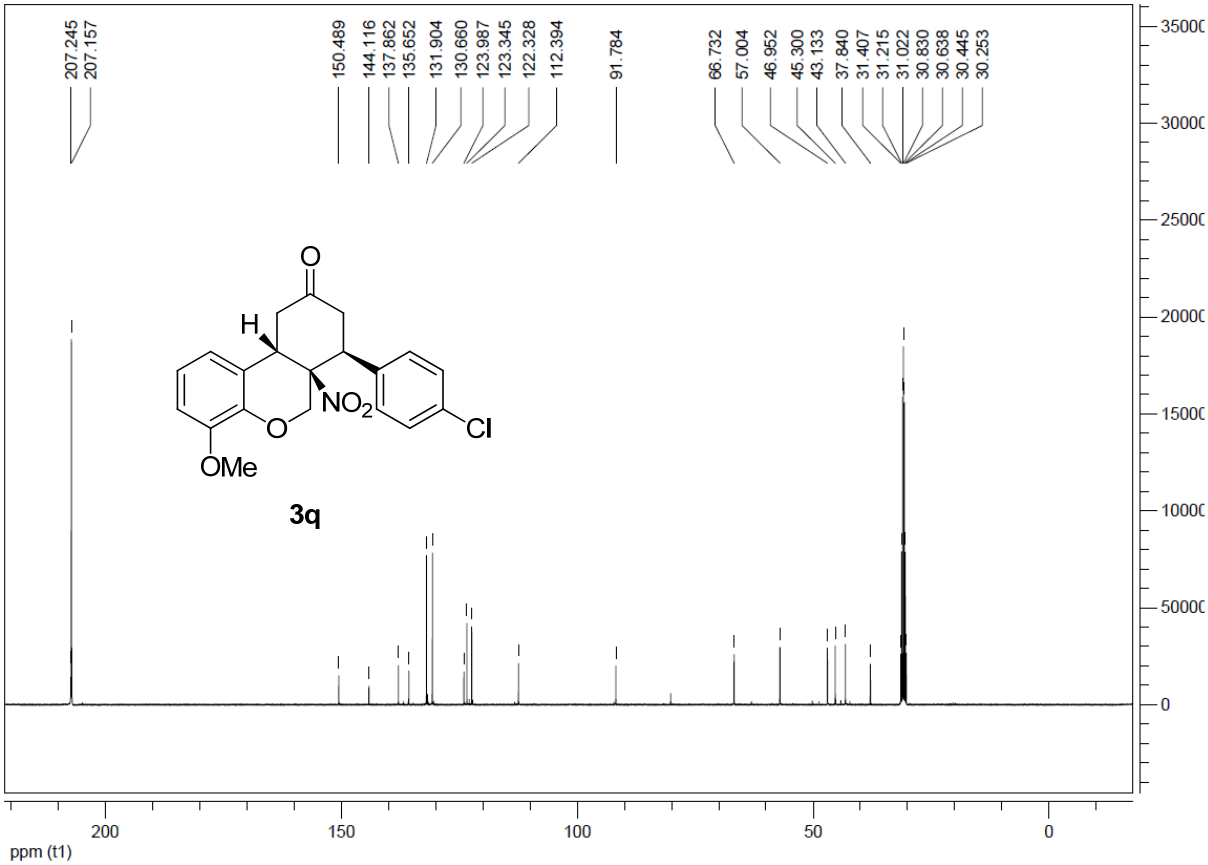
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)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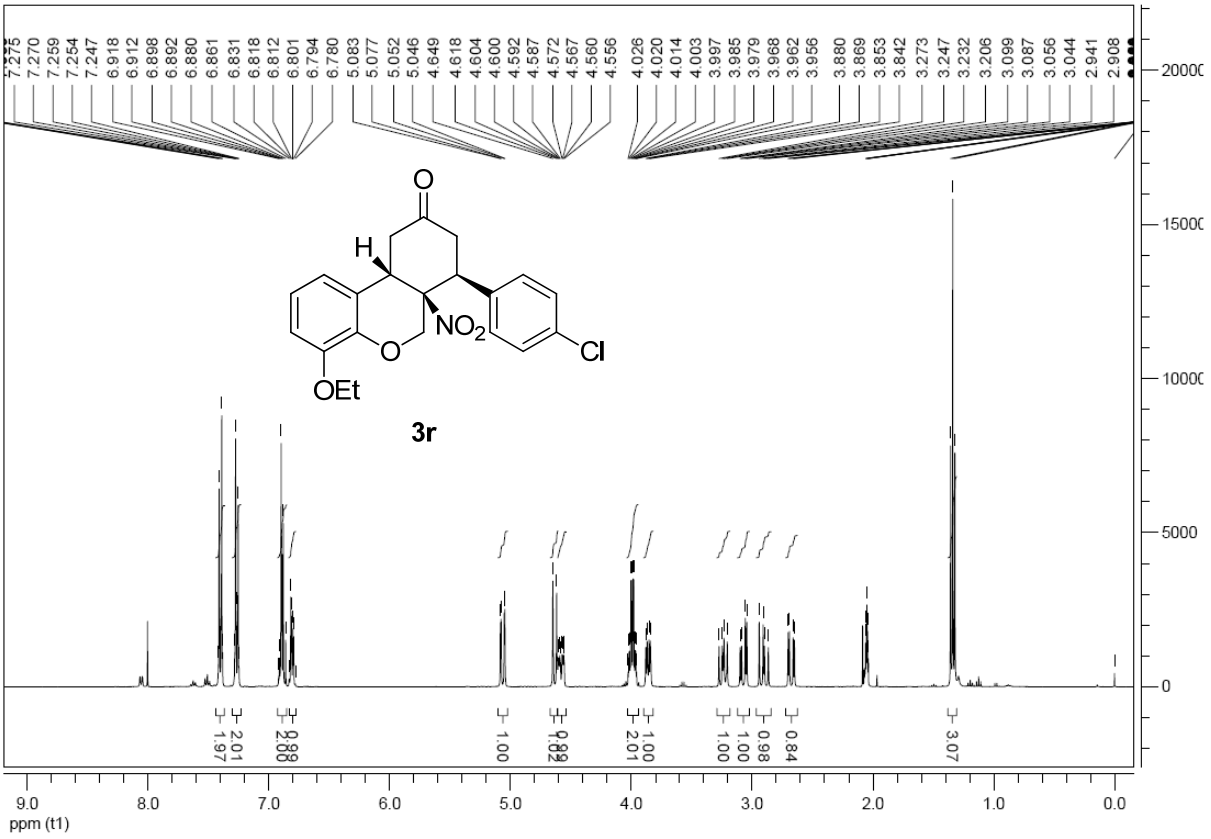
123.345
122.328
112.394
91.784
66.732
57.004
46.952
45.300
43.133
37.840
31.407
31.215
31.022
30.830
30.638
30.445
30.253
O
H
NO2
O
Cl
OMe
3q
200
150
100
50
0
ppm (t1)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6.898
6.892
6.880
6.861
6.831
6.818
6.812
6.801
6.794
6.780
5.083
5.077
5.052
5.046
4.649
4.618
4.604
4.600
4.592
4.587
4.572
4.567
4.560
4.556
4.026
4.020
4.014
4.003
3.997
3.985
3.979
3.968
3.962
3.956
3.880
3.869
3.853
3.842
3.273
3.247
3.232
3.206
3.099
3.087
3.056
3.044
2.941
2.908
O
H
NO2
O
Cl
OEt
3r
1.97
2.01
2.00
1.00
1.02
2.01
1.00
1.00
0.98
0.84
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7.0
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5.0
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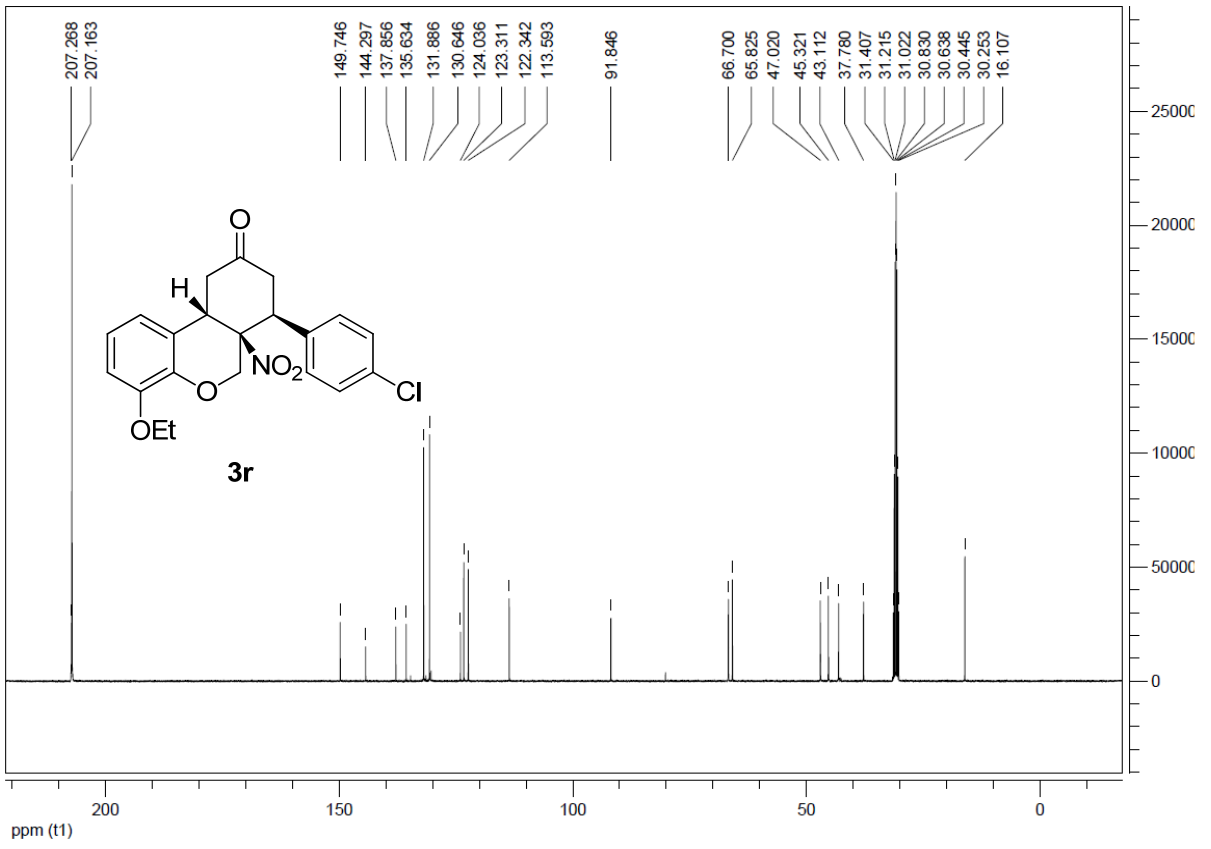
3.0
2.0
1.0
0.0
ppm (t1)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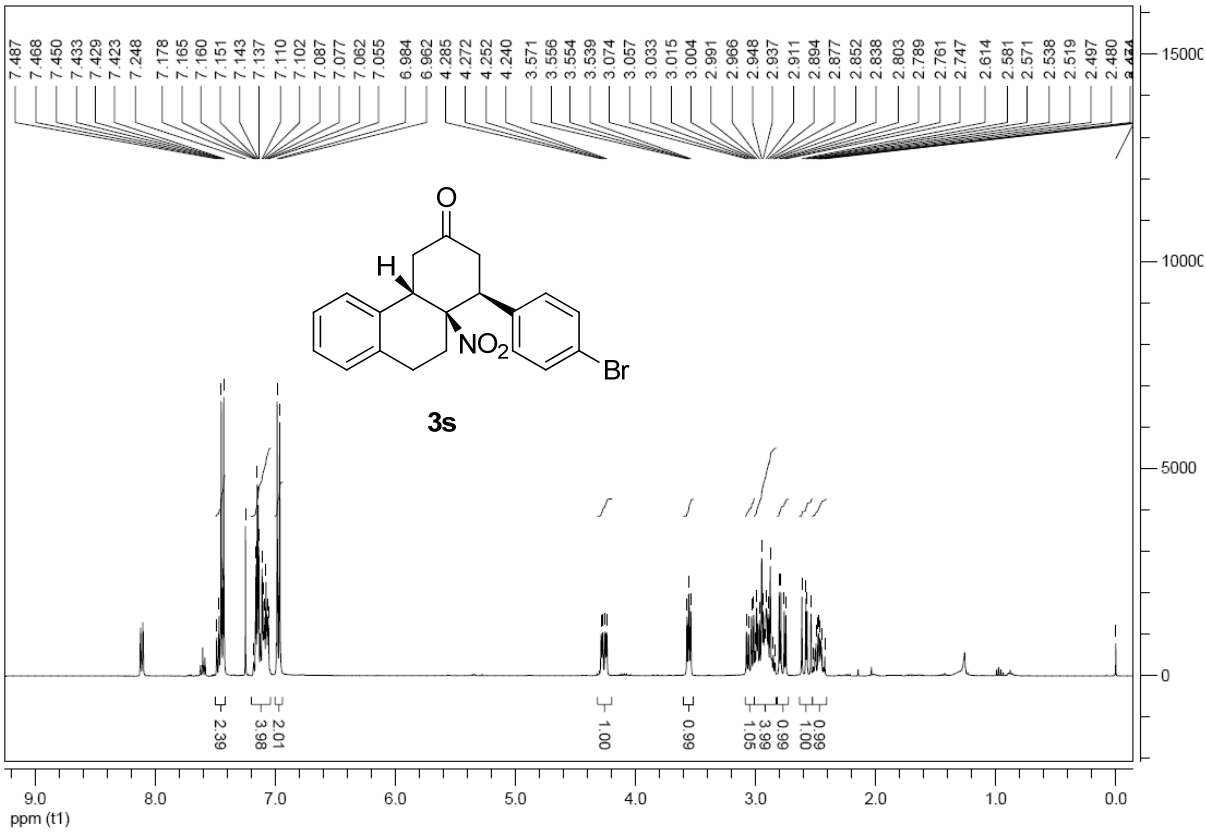
122.342
113.593
91.846
66.700
65.825
47.020
45.321
43.112
37.780
31.407
31.215
31.022
30.830
30.638
30.445
30.253
16.107
O
H
NO2
O
Cl
OEt
3r
200
150
100
50
0
ppm (t1)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7.433
7.429
7.423
7.248
7.178
7.165
7.160
7.151
7.143
7.137
7.110
7.102
7.087
7.077
7.062
7.055
6.984
6.962
4.285
4.272
4.252
4.240
3.571
3.556
3.554
3.539
3.074
3.057
3.033
3.015
3.004
2.991
2.966
2.948
2.937
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2.894
2.877
2.852
2.838
2.803
2.789
2.761
2.747
2.614
2.581
2.571
2.538
2.519
2.497
2.480
O
H
NO2
Br
3s
2.39
3.98
2.01
1.00
0.99
1.05
0.99
0.99
1.00
0.99
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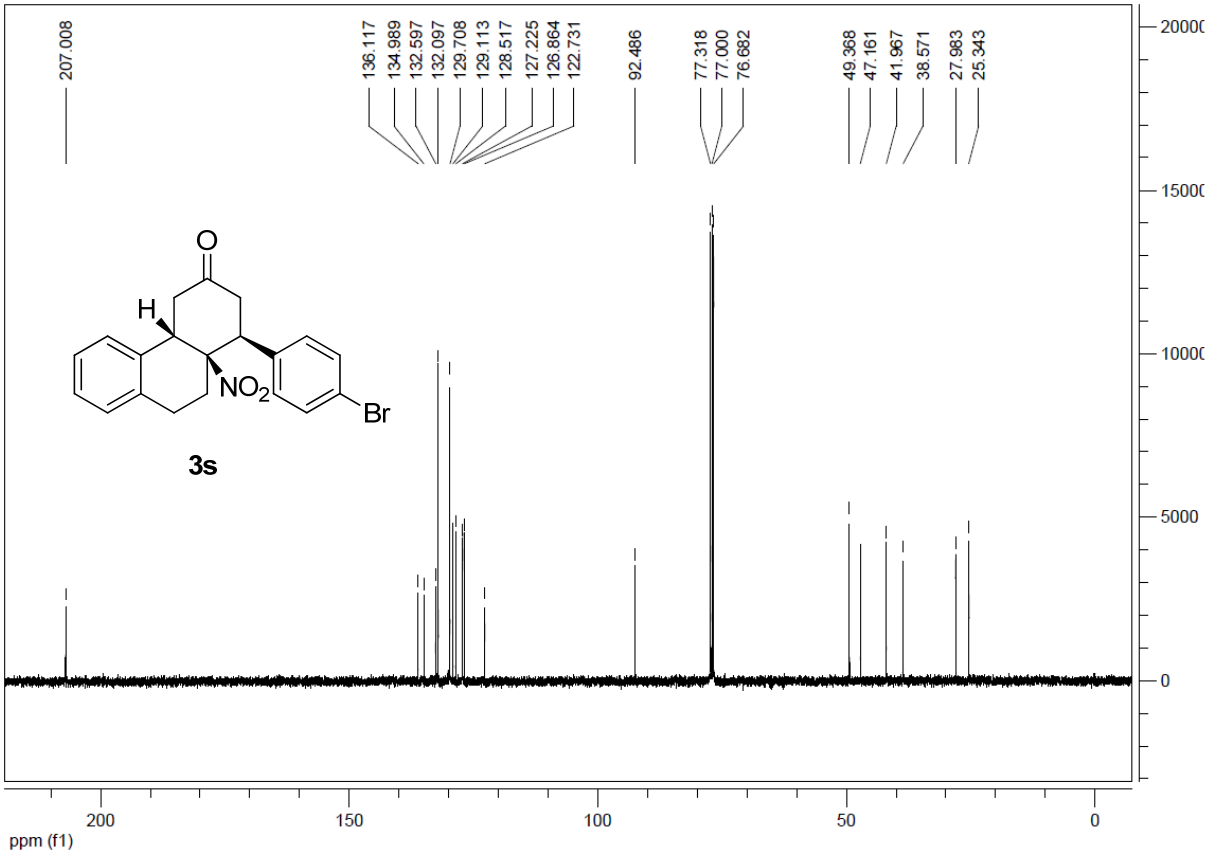
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)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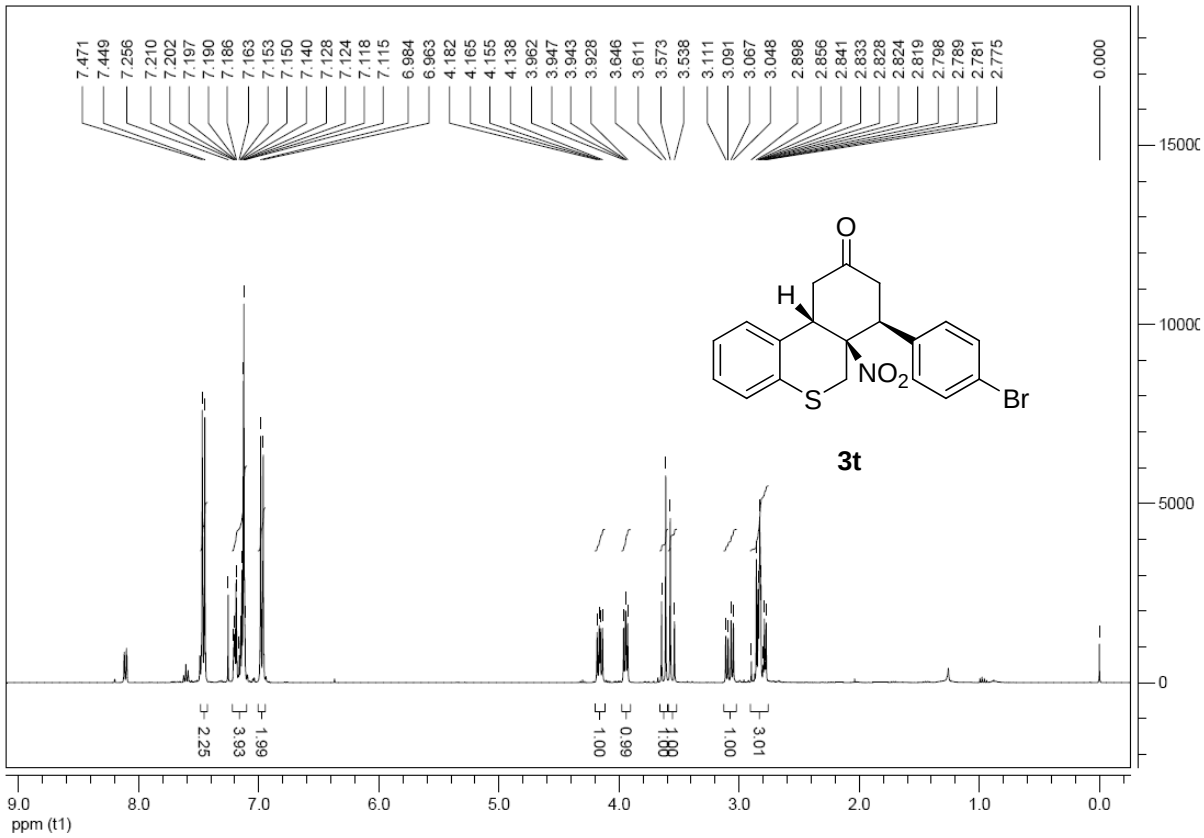
128.517
127.225
126.864
122.731
92.486
77.318
77.000
76.682
49.368
47.161
41.967
38.571
27.983
25.343
O
H
NO2
Br
3s
200
150
100
50
0
ppm (f1)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7.202
7.197
7.190
7.186
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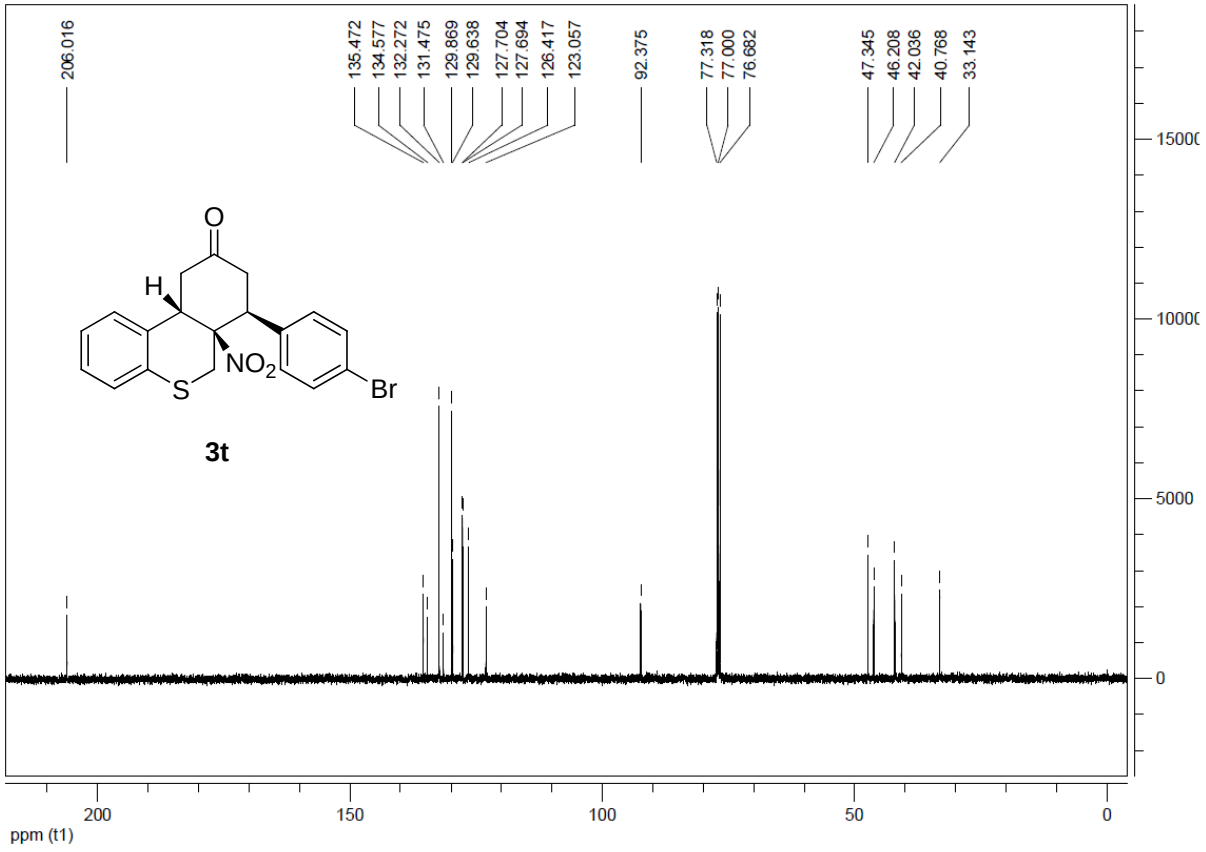
7.153
7.150
7.140
7.128
7.124
7.118
7.115
6.984
6.963
4.182
4.165
4.155
4.138
3.962
3.947
3.943
3.928
3.646
3.611
3.573
3.538
3.111
3.091
3.067
3.048
2.898
2.856
2.841
2.833
2.828
2.824
2.819
2.798
2.789
2.781
2.775
0.000
O
H
NO2
S
Br
3t
2.25
3.93
1.99
1.00
0.99
1.88
1.00
3.01
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)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129.869
129.638
127.704
127.694
126.417
123.057
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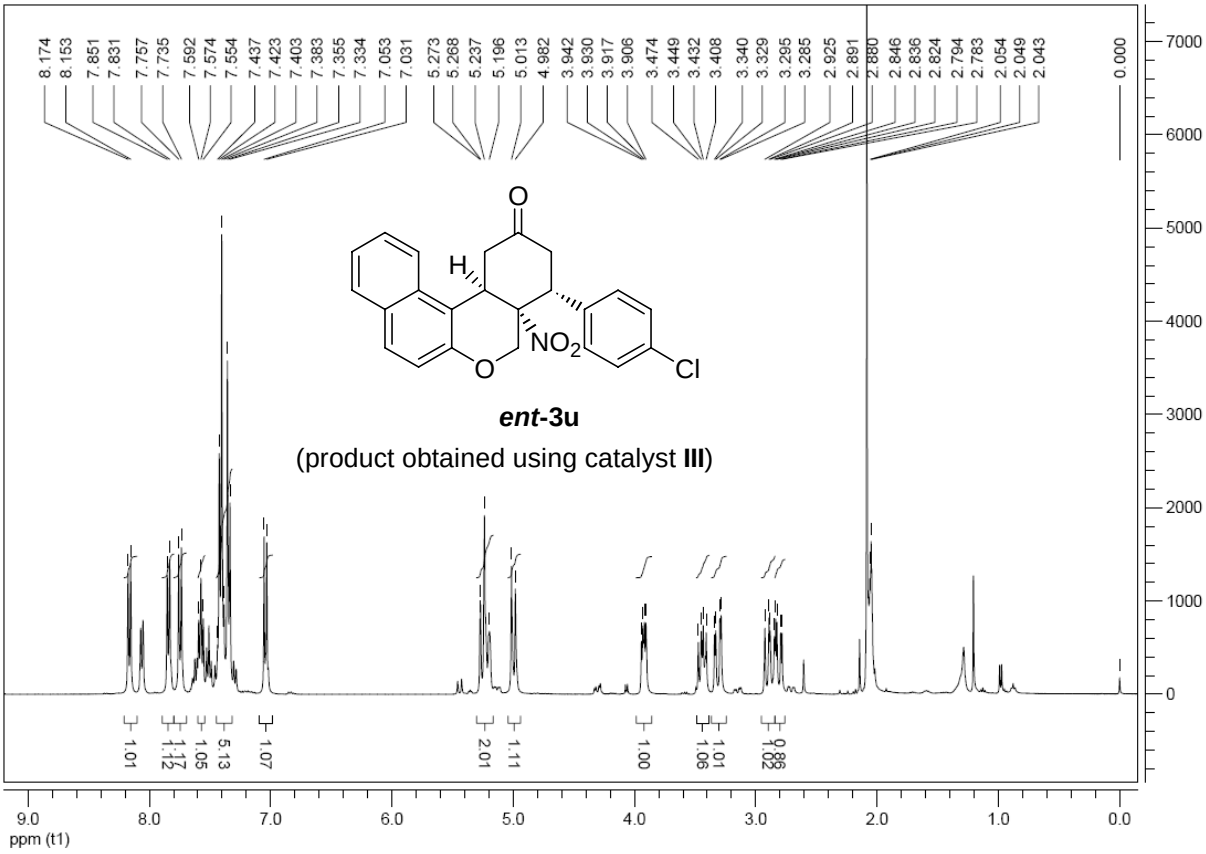
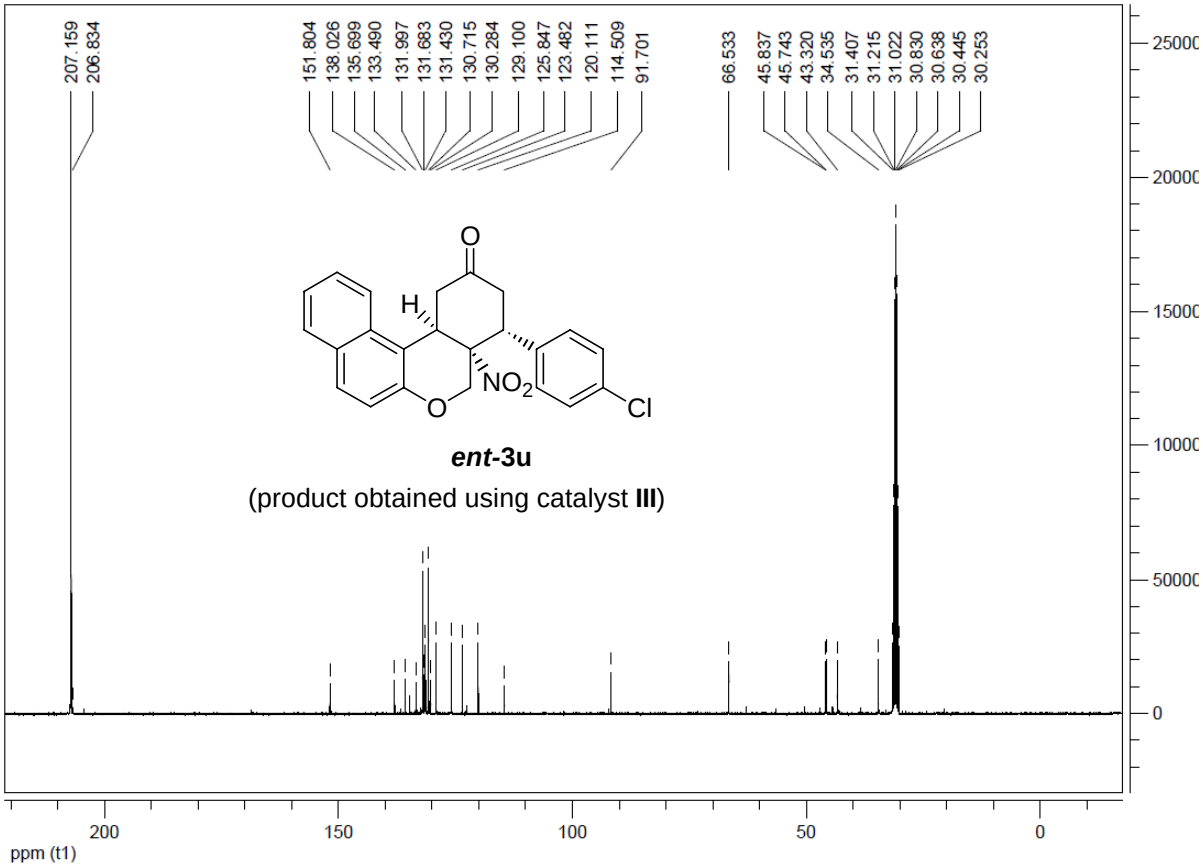
77.318
77.000
76.682
47.345
46.208
42.036
40.768
33.143
O
H
NO2
S
Br
3t
200
150
100
50
0
ppm (t1)



***ent*-3u**

(product obtained using catalyst **III**)

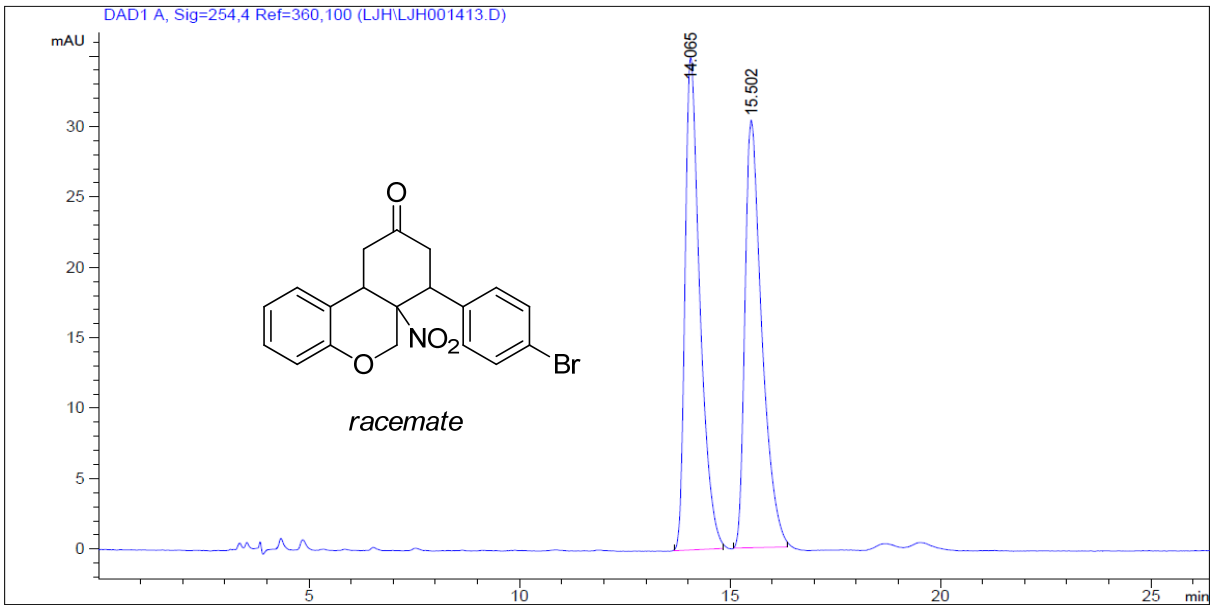


***ent*-3u**

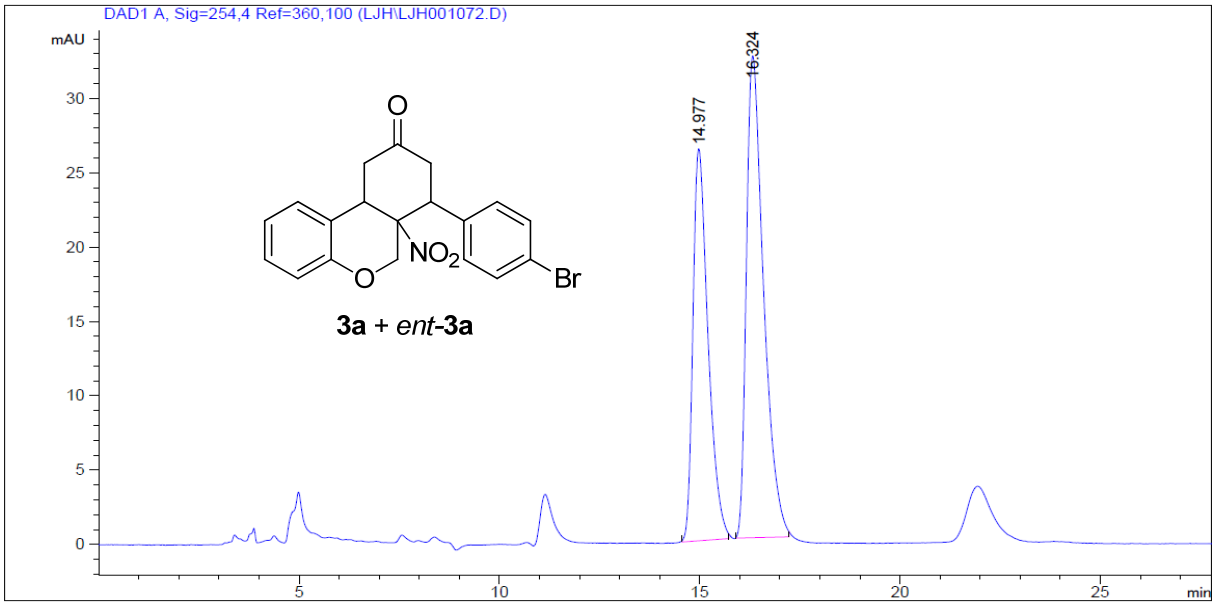
(product obtained using catalyst **III**)

Copies of HPLC chromatographs of products

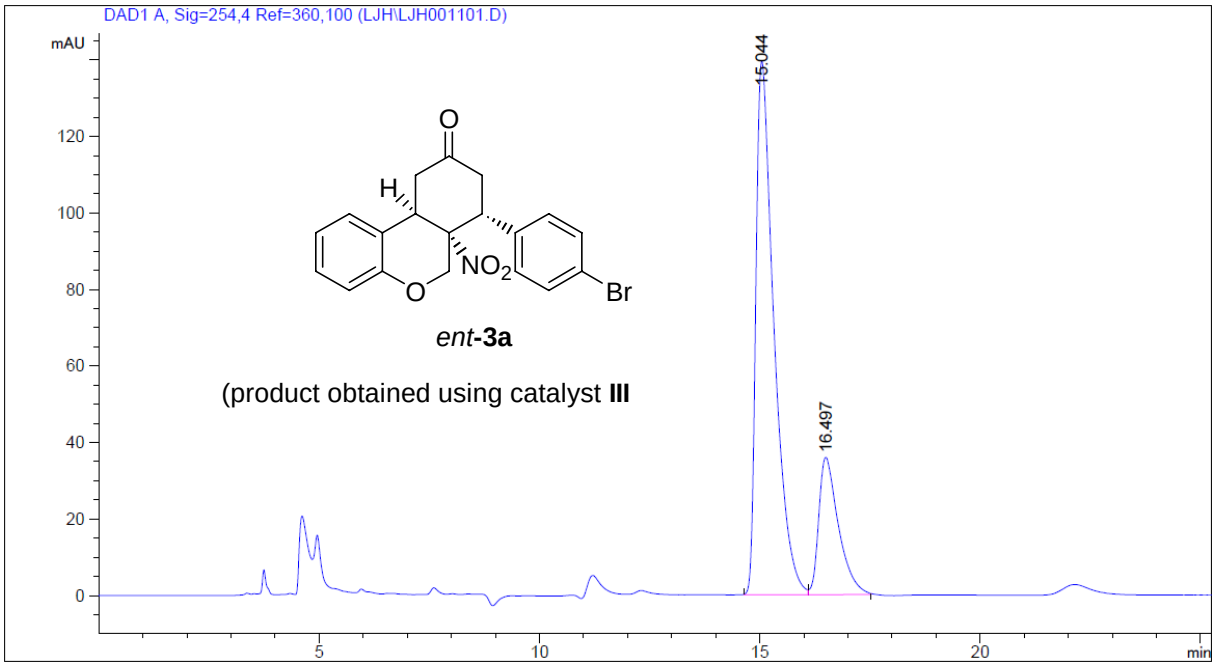
The enantioselectivity for the products **3b-u** obtained from catalyst **I** was determined by comparison of the chiral HPLC data with the product obtained from 1 eq. catalyst **I** plus 1 eq. catalyst **III**.



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.065	BB	0.3644	858.78888	34.97898	50.2431
2	15.502	BB	0.4147	850.47748	30.36698	49.7569



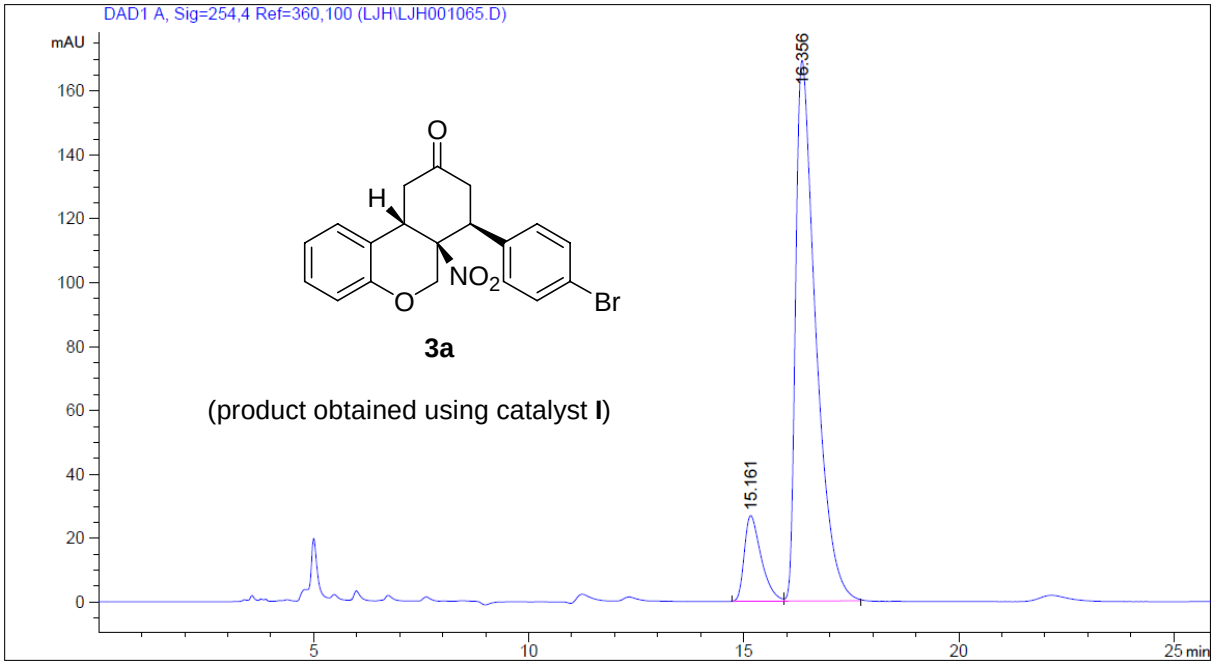
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.977	BB	0.3940	691.75928	26.37478	41.6846
2	16.324	BB	0.4463	967.74768	32.41551	58.3154



ent-**3a**

(product obtained using catalyst **III**

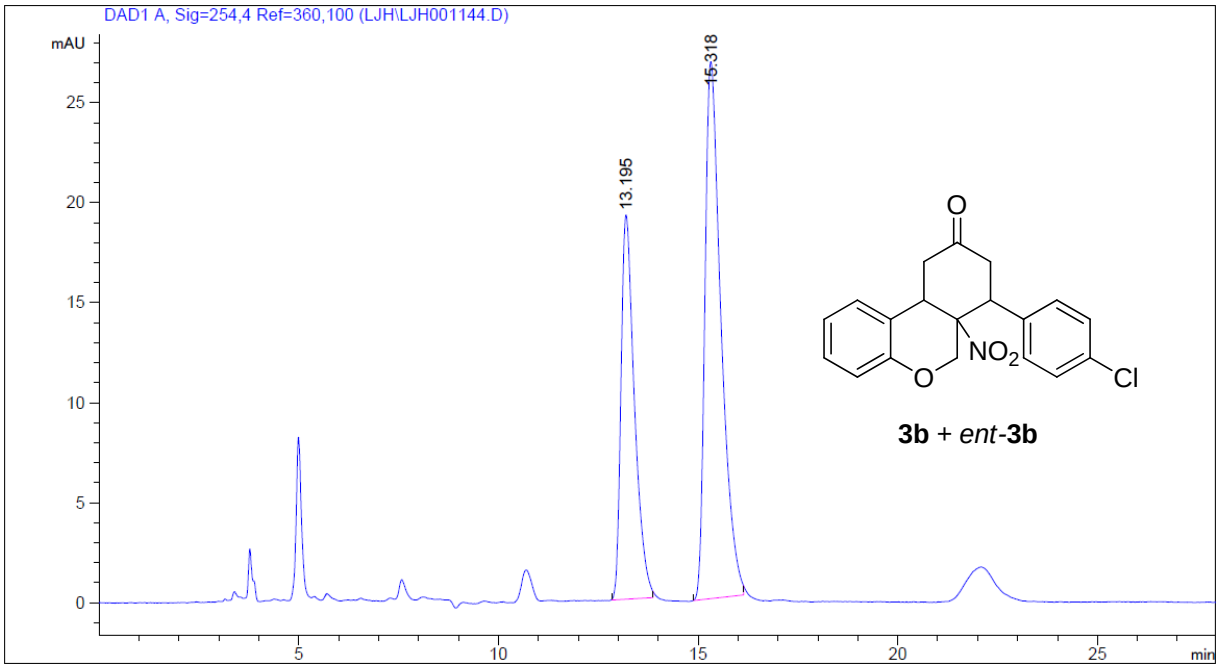
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.044	BB	0.4217	3967.77637	139.55348	78.0103
2	16.497	BB	0.4634	1118.44153	35.91027	21.9897



3a

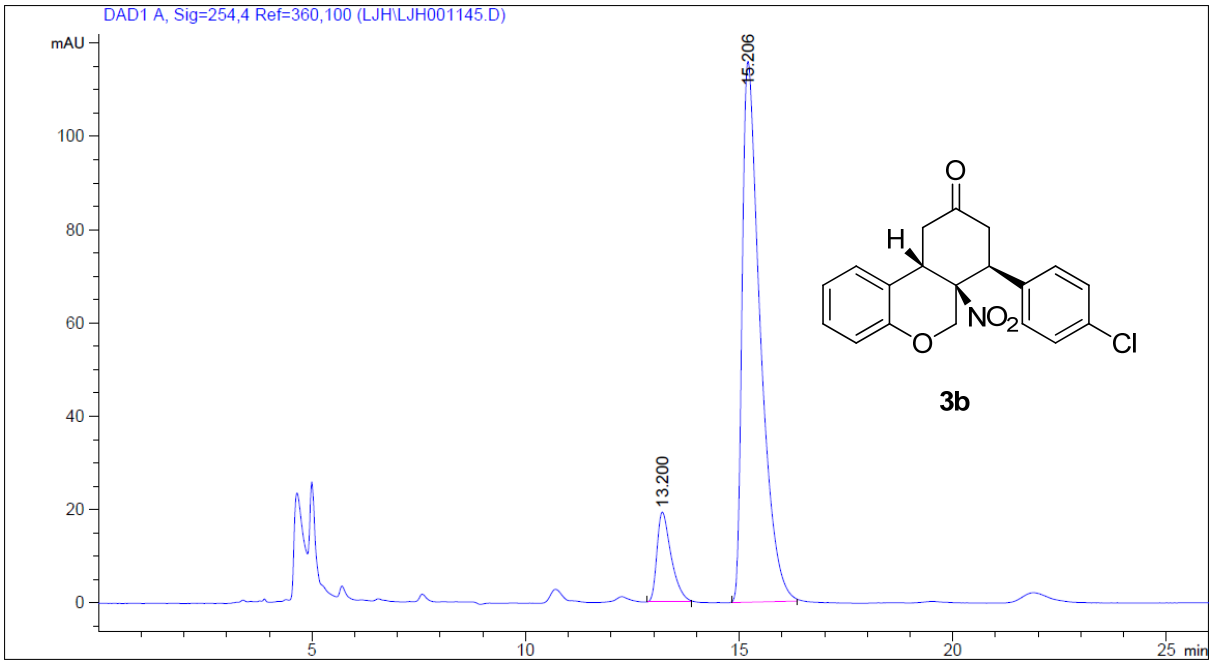
(product obtained using catalyst **I**)

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.161	BB	0.4103	732.23352	26.83512	11.7437
2	16.356	BB	0.4832	5502.89551	169.30943	88.2563



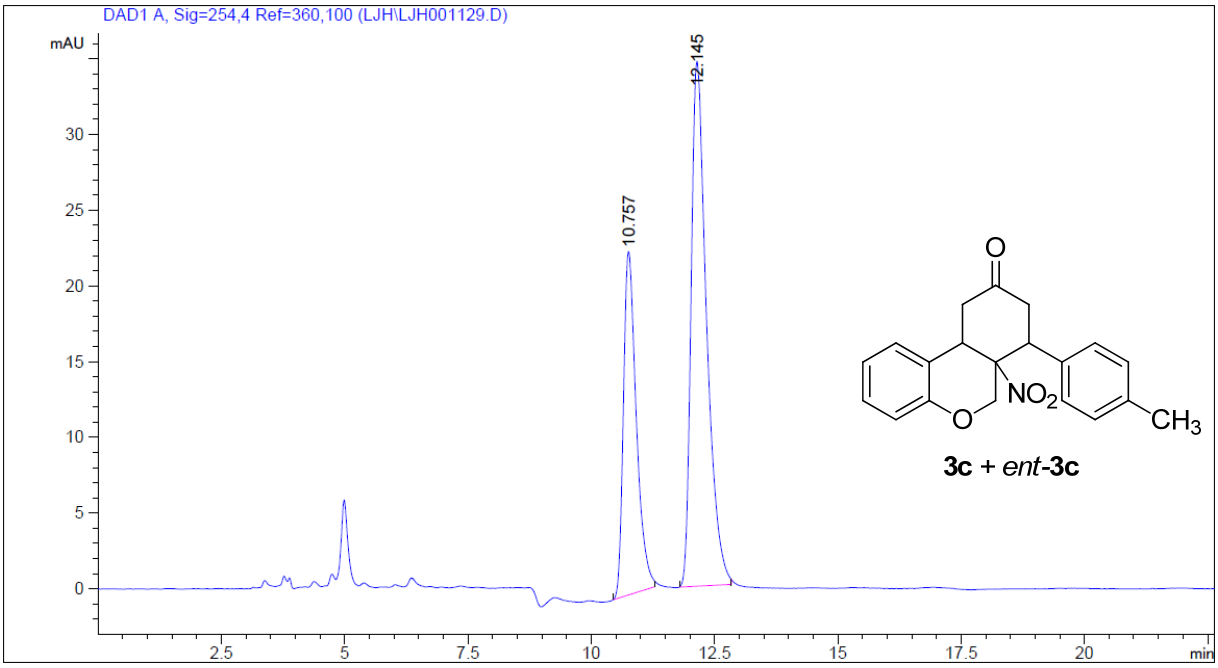
3b + *ent*-**3b**

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.195	BB	0.3511	450.02185	19.22274	37.3205
2	15.318	BB	0.4184	755.80981	26.85287	62.6795

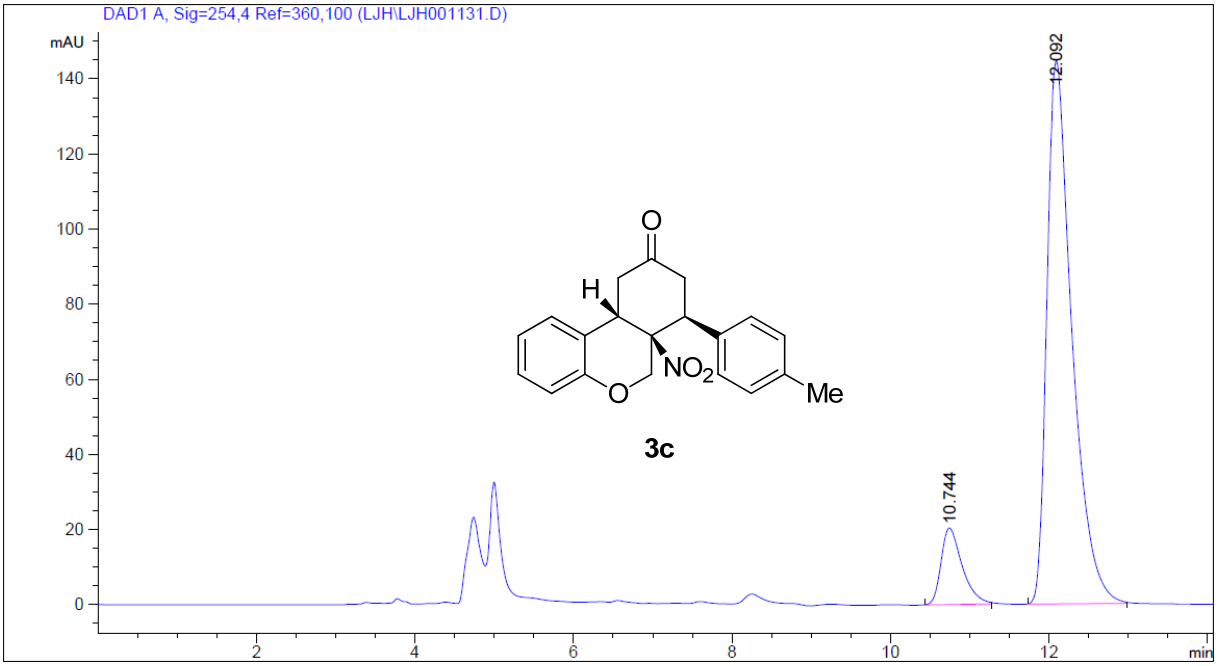


3b

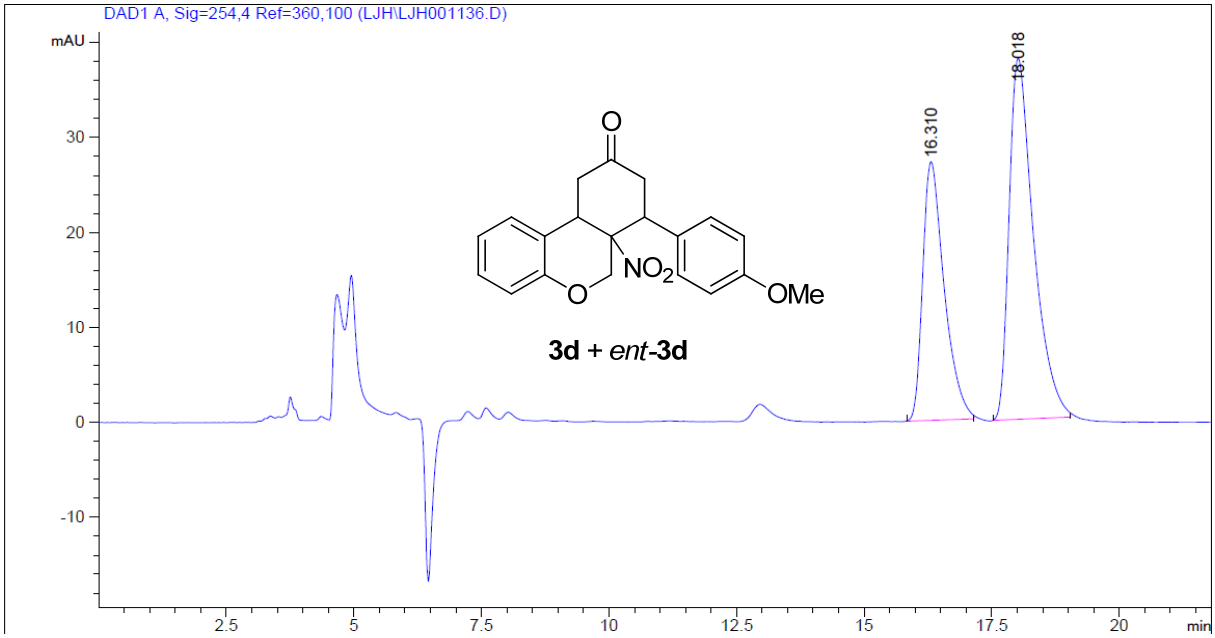
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.200	BB	0.3485	446.82642	19.27217	11.5308
2	15.206	BB	0.4371	3428.24219	115.89973	88.4692



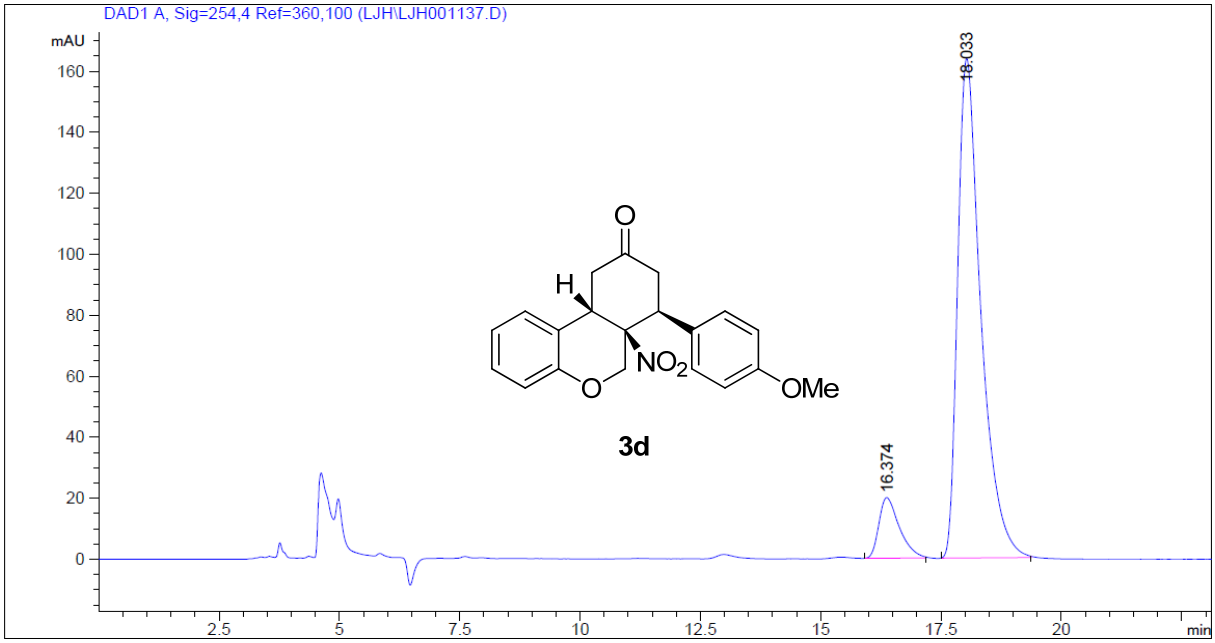
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.757	BB	0.2731	410.30487	22.67364	35.1935
2	12.145	BB	0.3243	755.54926	34.65741	64.8065



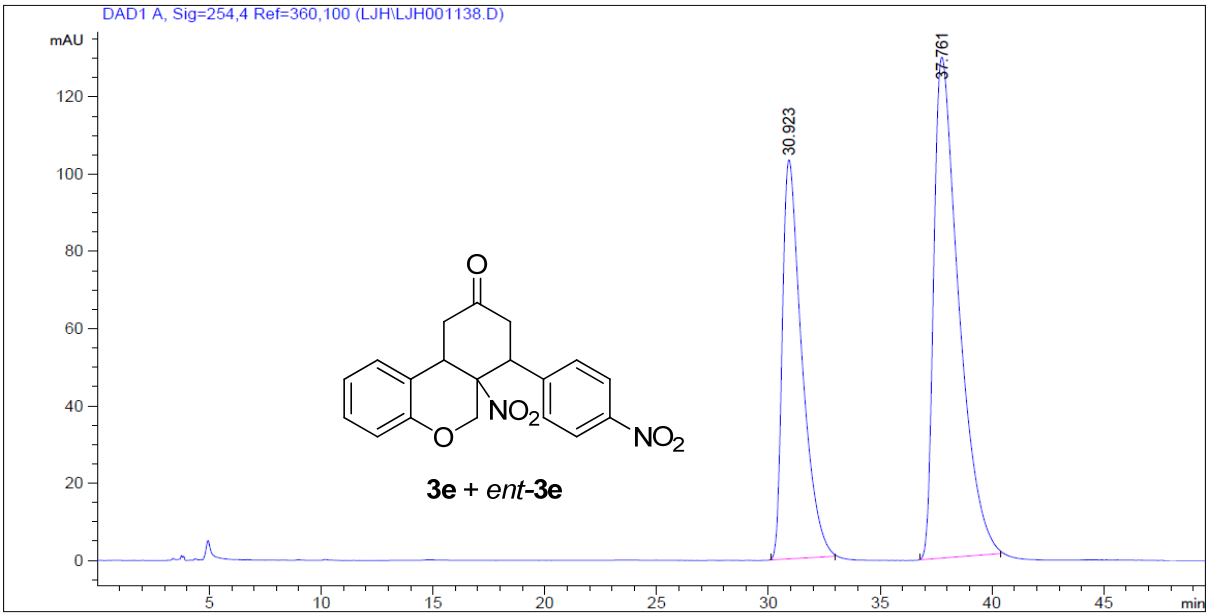
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.744	BB	0.2790	375.72214	20.38746	10.5843
2	12.092	BB	0.3258	3174.07568	144.74997	89.4157



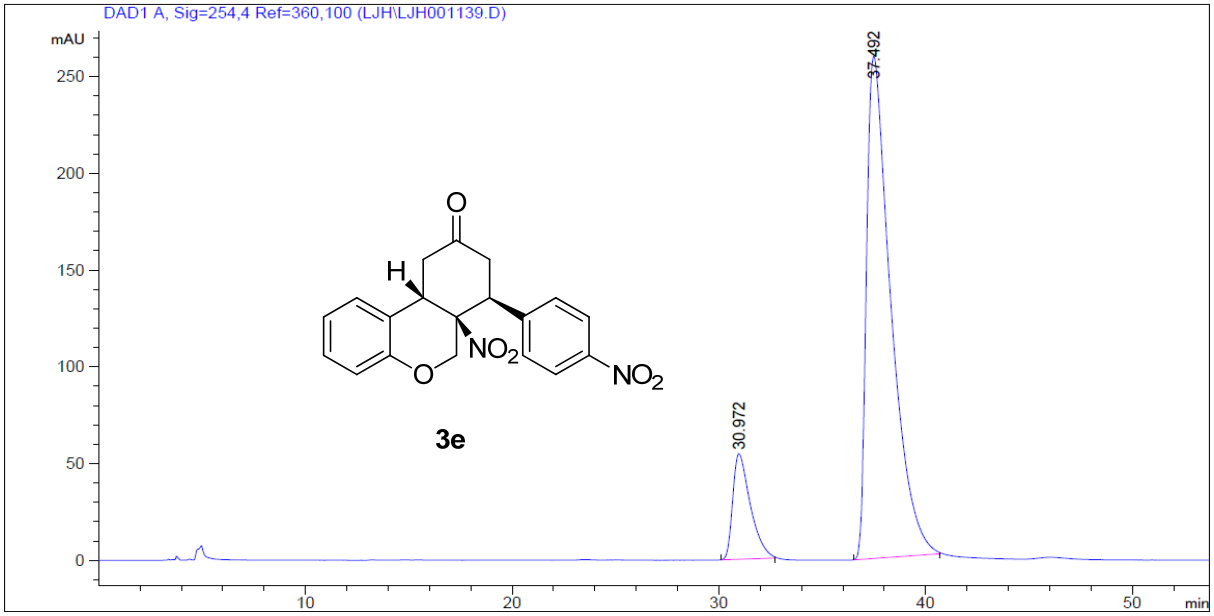
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.310	BB	0.4418	798.06610	27.24061	38.6390
2	18.018	BB	0.4968	1267.37695	38.02856	61.3610



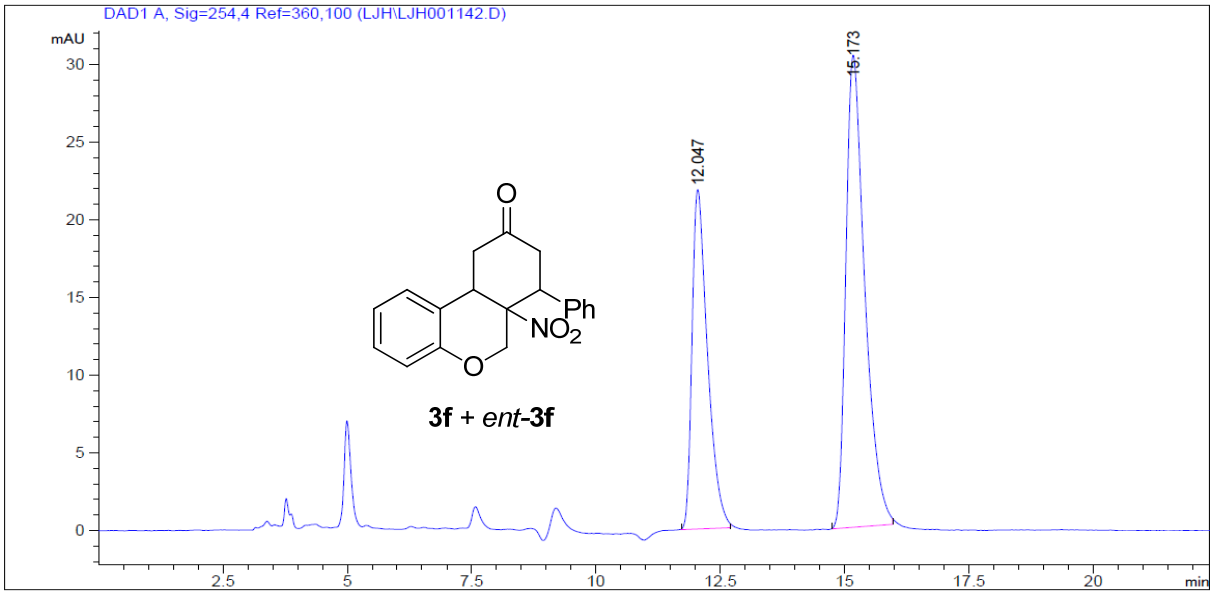
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.374	BB	0.4428	587.74713	19.88595	9.6591
2	18.033	BB	0.5016	5497.16211	163.76770	90.3409



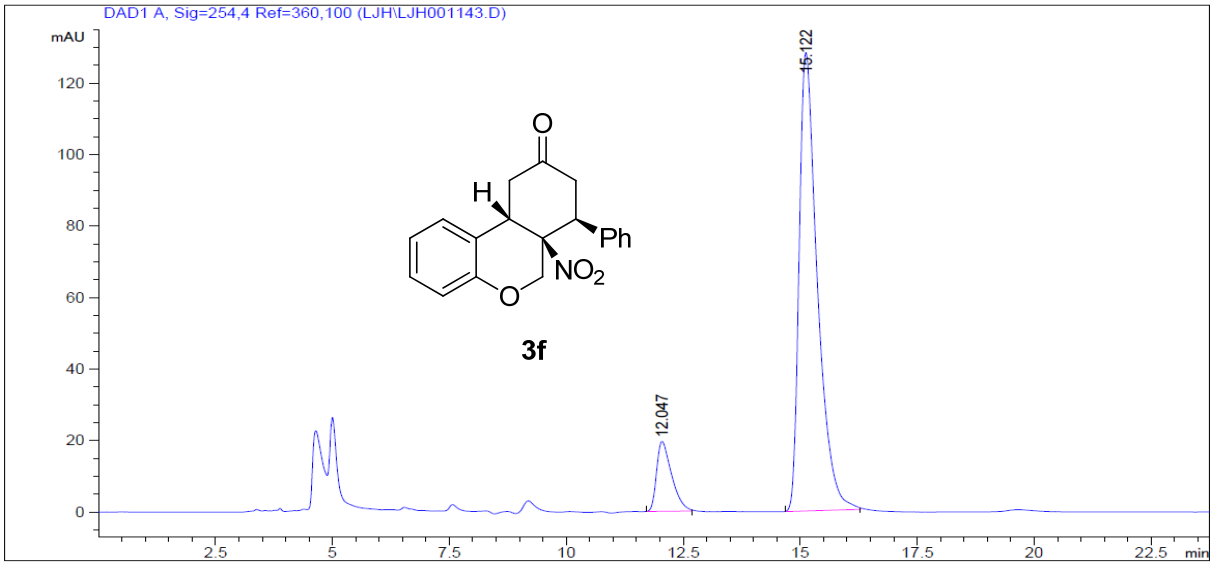
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	30.923	BB	0.9054	6283.67920	103.33691	38.4178
2	37.761	BB	1.1357	1.00725e4	129.74983	61.5822



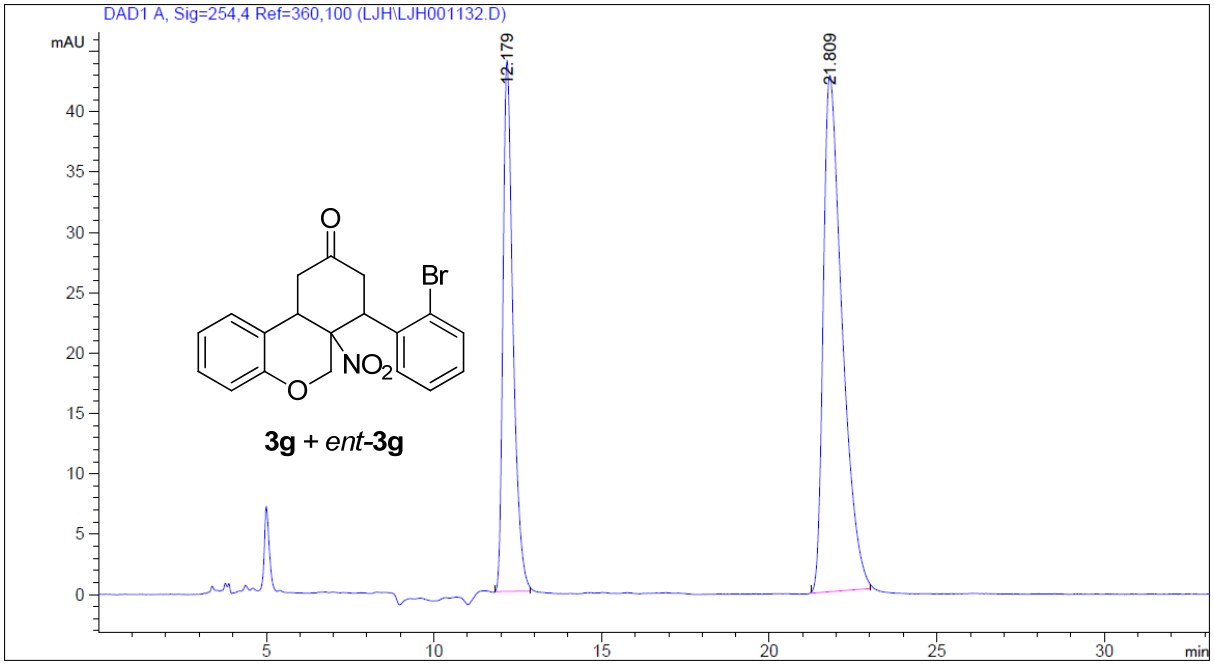
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	30.972	BB	0.8670	3228.42041	54.50412	13.1820
2	37.492	BB	1.1919	2.12626e4	259.49689	86.8180



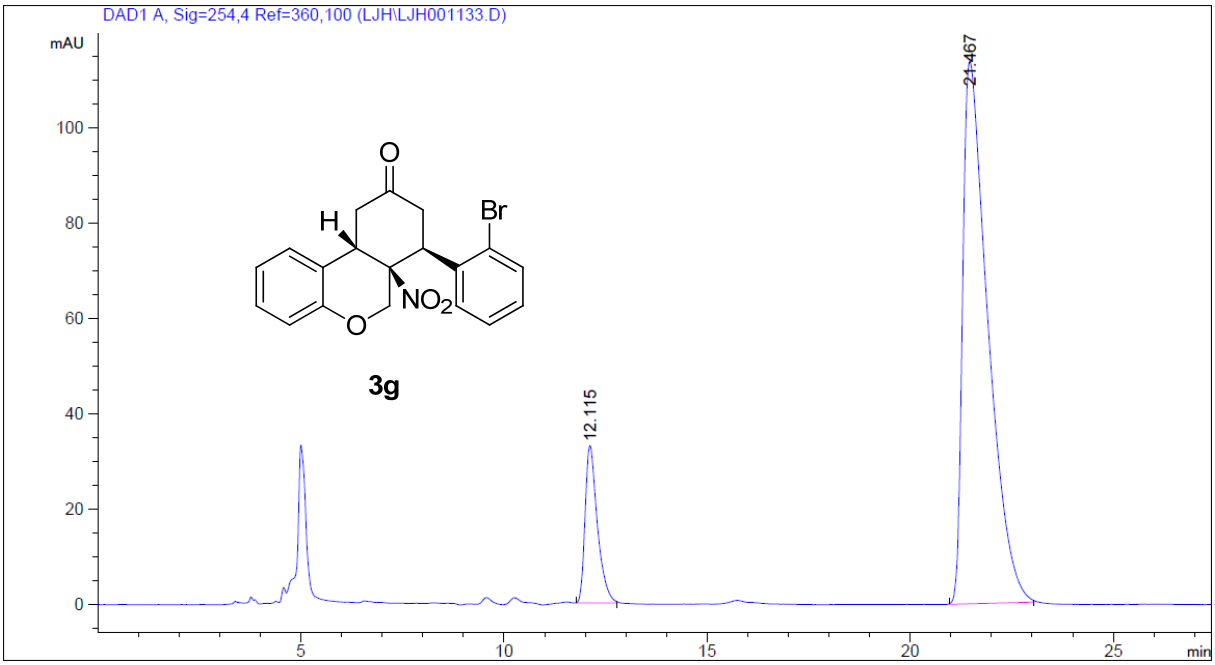
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.047	BB	0.3205	473.19794	21.86281	37.0624
2	15.173	BB	0.3984	803.56104	30.39947	62.9376



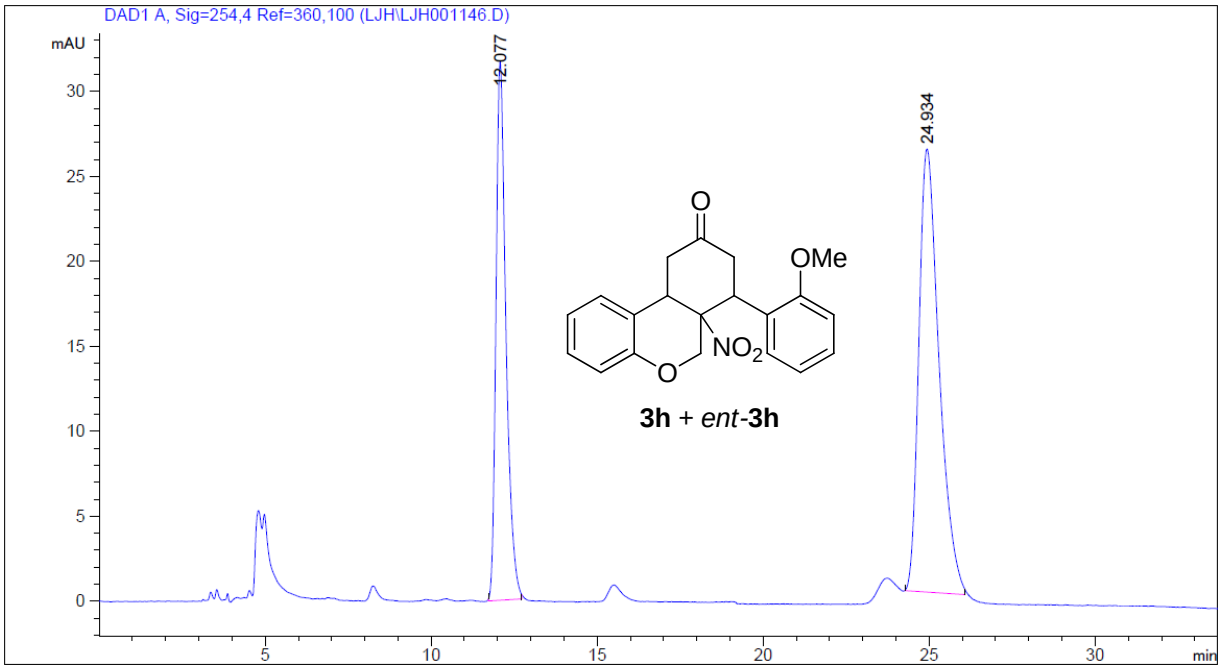
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.047	BB	0.3320	436.01117	19.56227	11.1518
2	15.122	BB	0.4019	3473.77148	128.27716	88.8482



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.179	BB	0.3113	918.17957	44.02082	35.3657
2	21.809	BB	0.5836	1678.06567	42.78381	64.6343

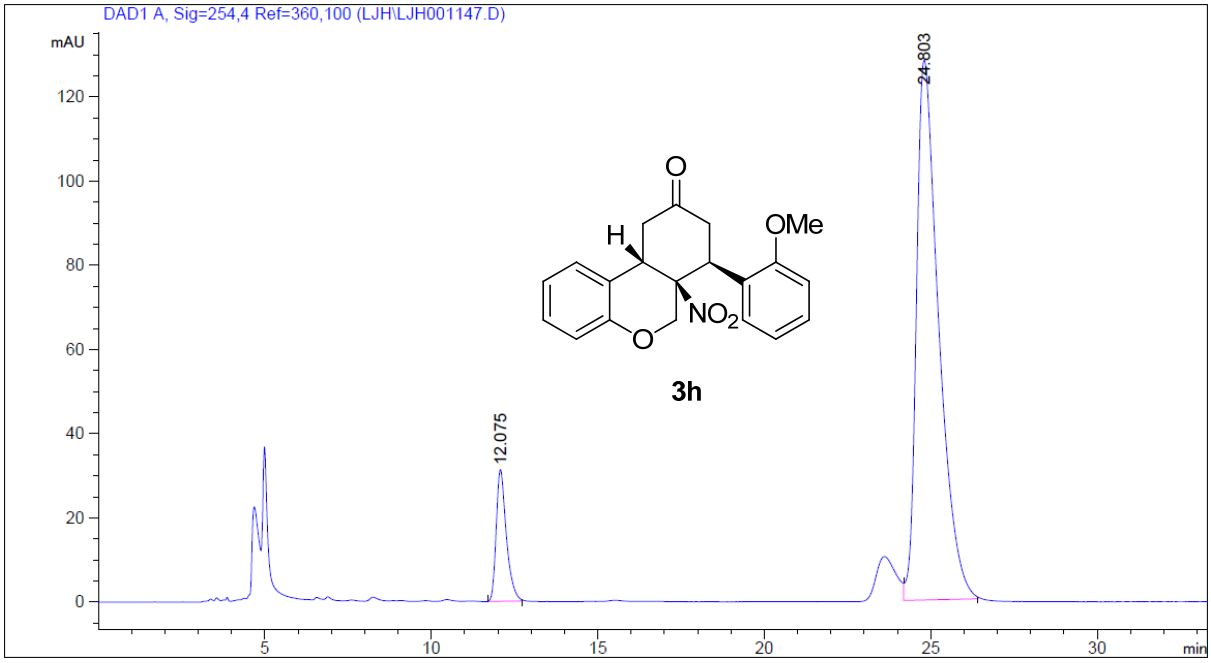


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.115	BB	0.3124	692.24274	33.03959	12.3811
2	21.467	BB	0.6278	4898.90283	113.89629	87.6189



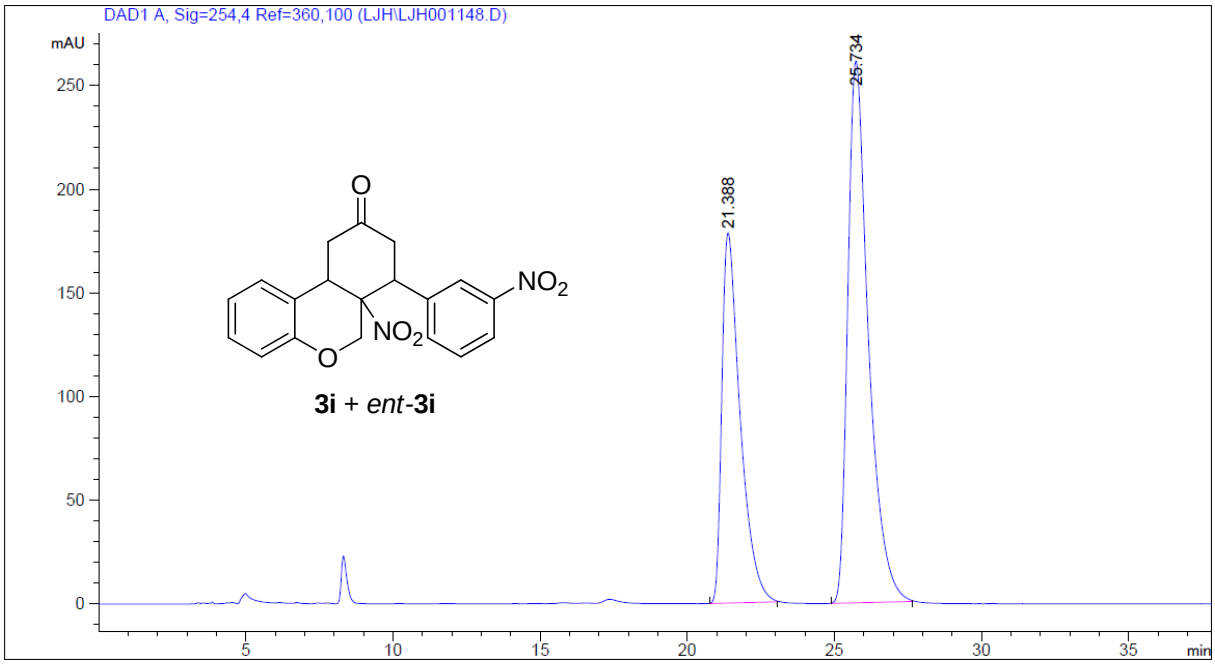
3h + *ent*-**3h**

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.077	BB	0.3134	661.56641	31.70617	37.4428
2	24.934	BB	0.6326	1105.30713	26.07461	62.5572



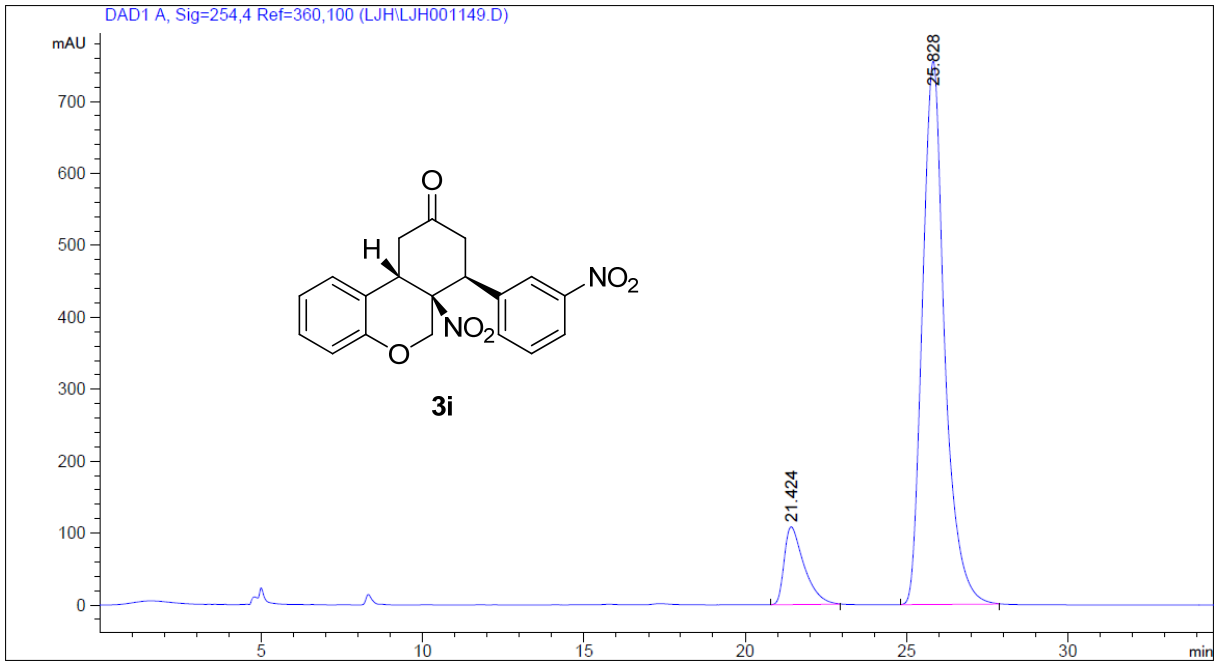
3h

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.075	BB	0.3171	662.54974	31.28623	10.0006
2	24.803	VB	0.6882	5962.55518	128.29031	89.9994



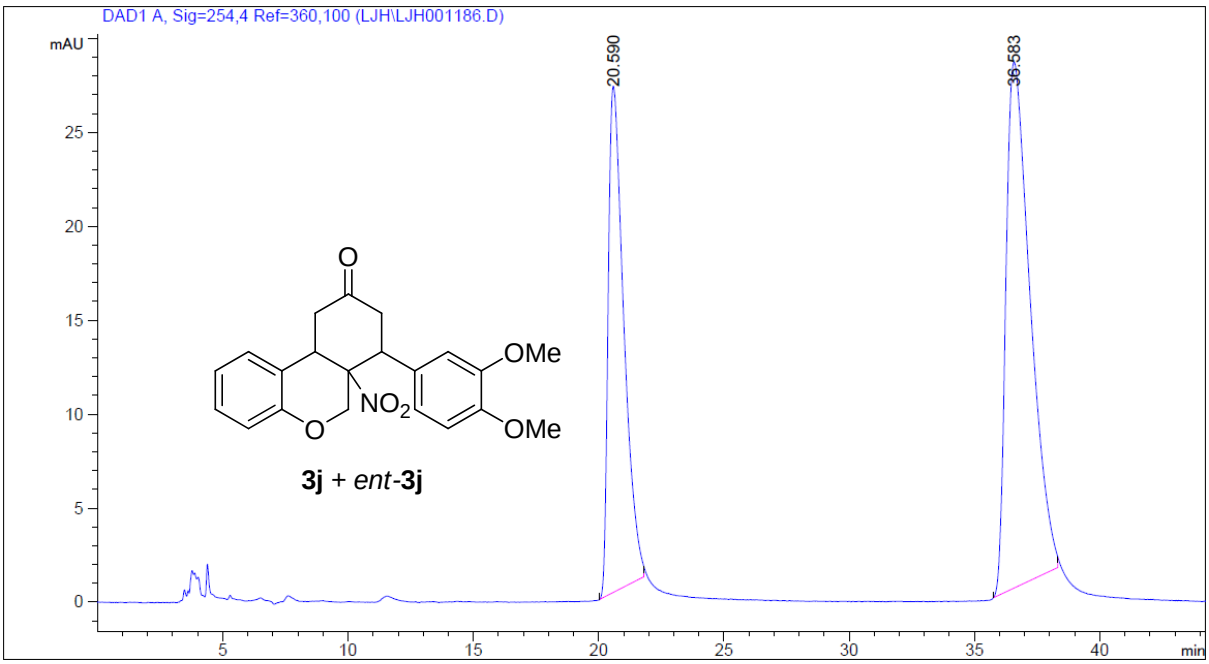
3i + *ent*-**3i**

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	21.388	BB	0.6433	7727.19336	178.42009	37.7168
2	25.734	BB	0.7320	1.27602e4	261.18729	62.2832

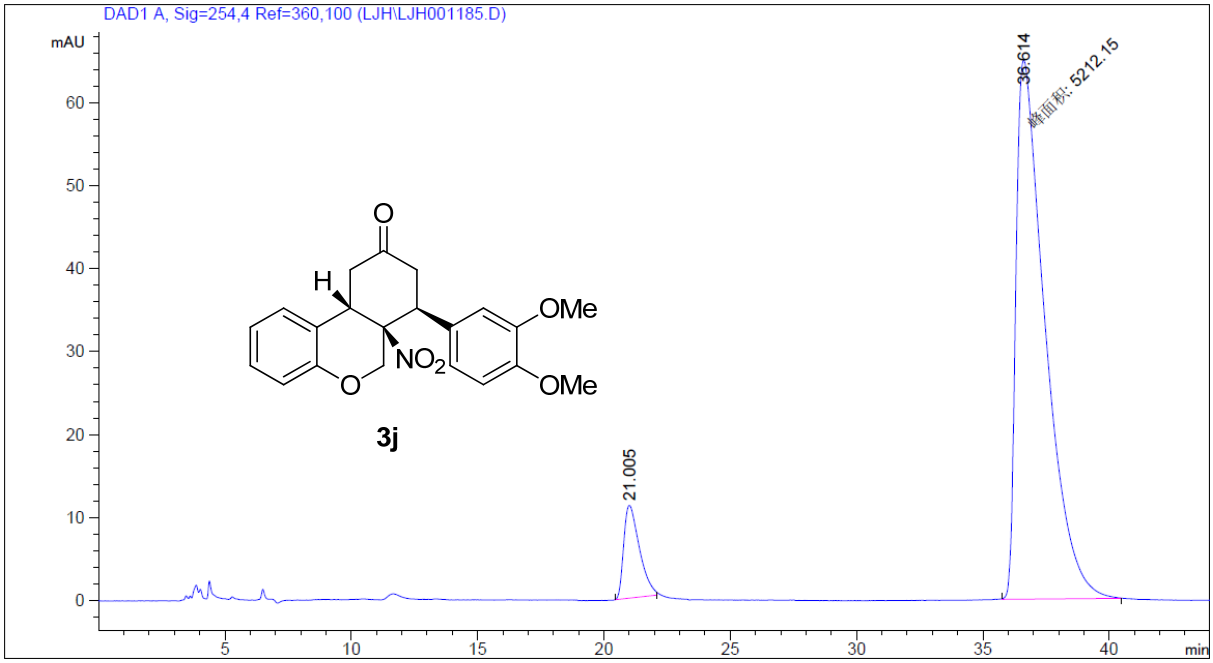


3i

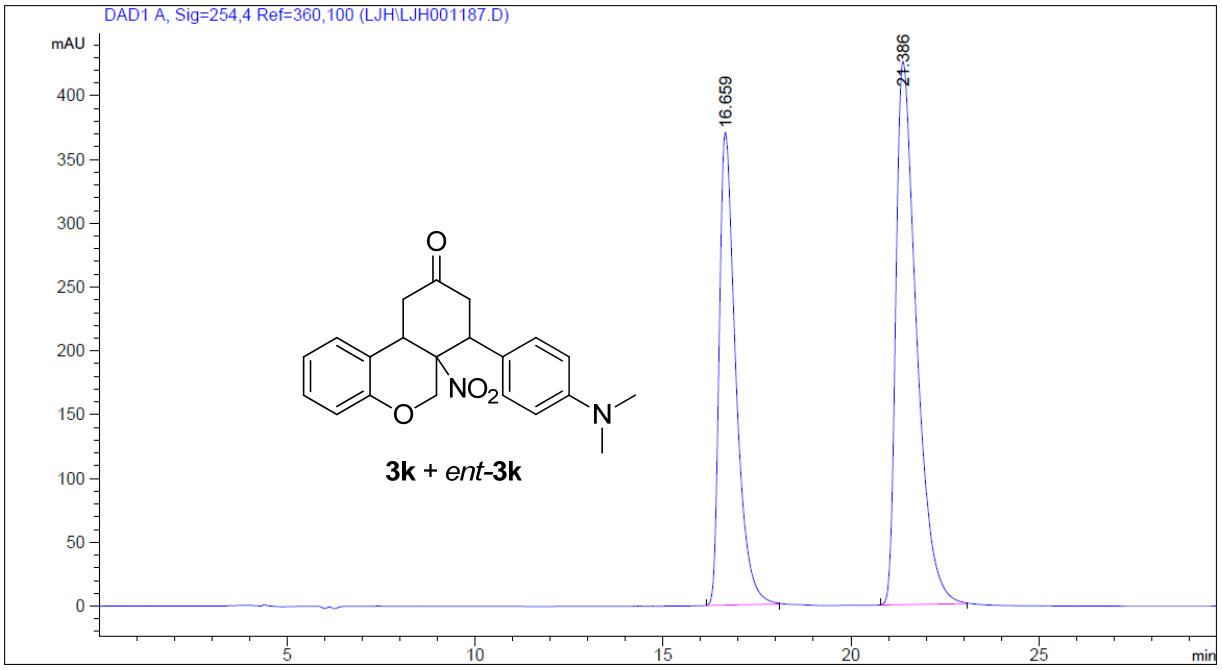
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	21.424	BB	0.6349	4641.74707	108.11337	11.4046
2	25.828	BB	0.7309	3.60589e4	755.37152	88.5954



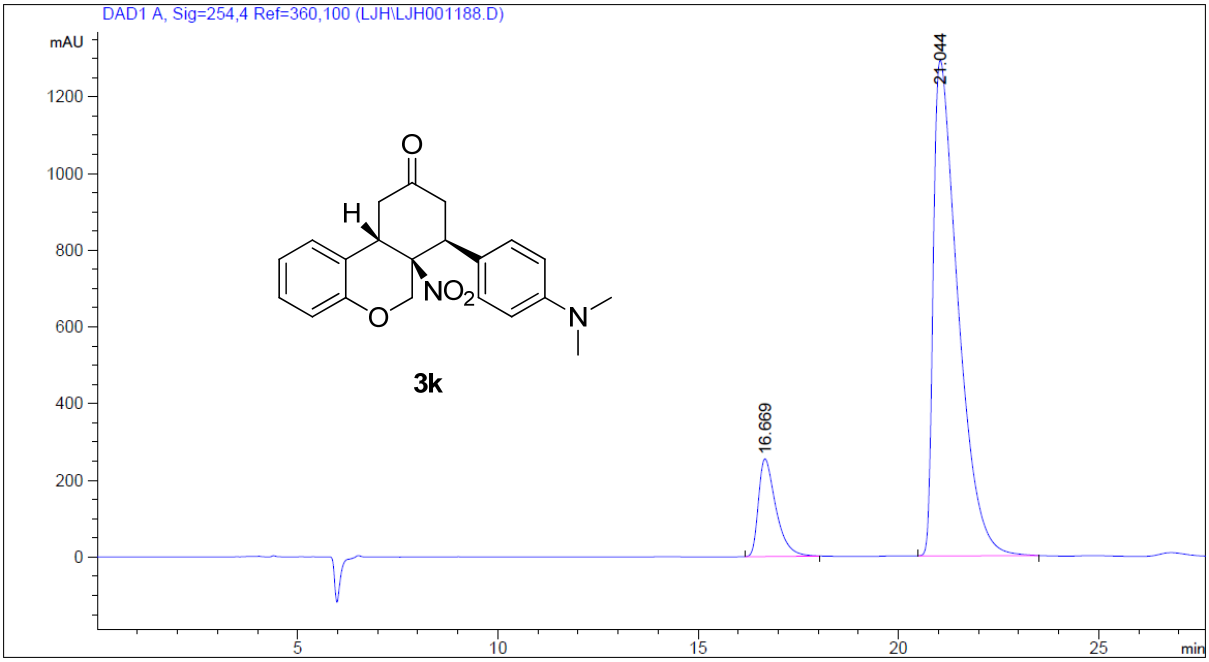
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.590	BB	0.6485	1165.43933	26.95165	38.2662
2	36.583	BB	0.9674	1880.16895	27.99984	61.7338



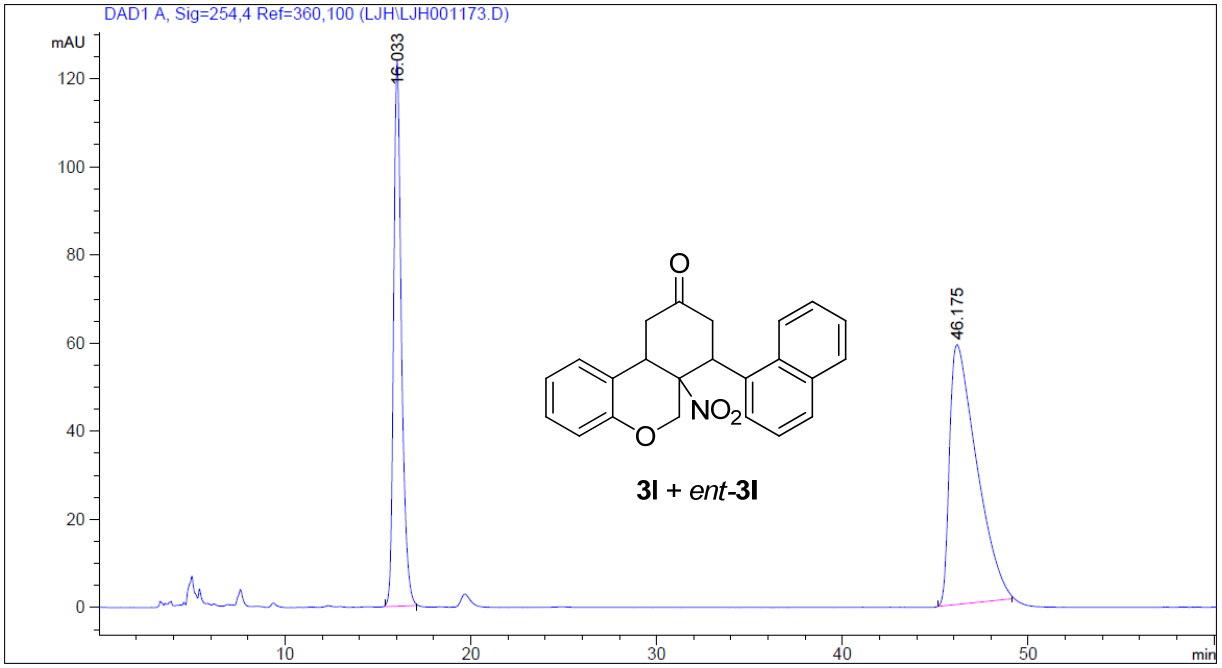
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	21.005	BB	0.6174	482.31274	11.17710	8.4698
2	36.614	MM T	1.3371	5212.15381	64.96638	91.5302



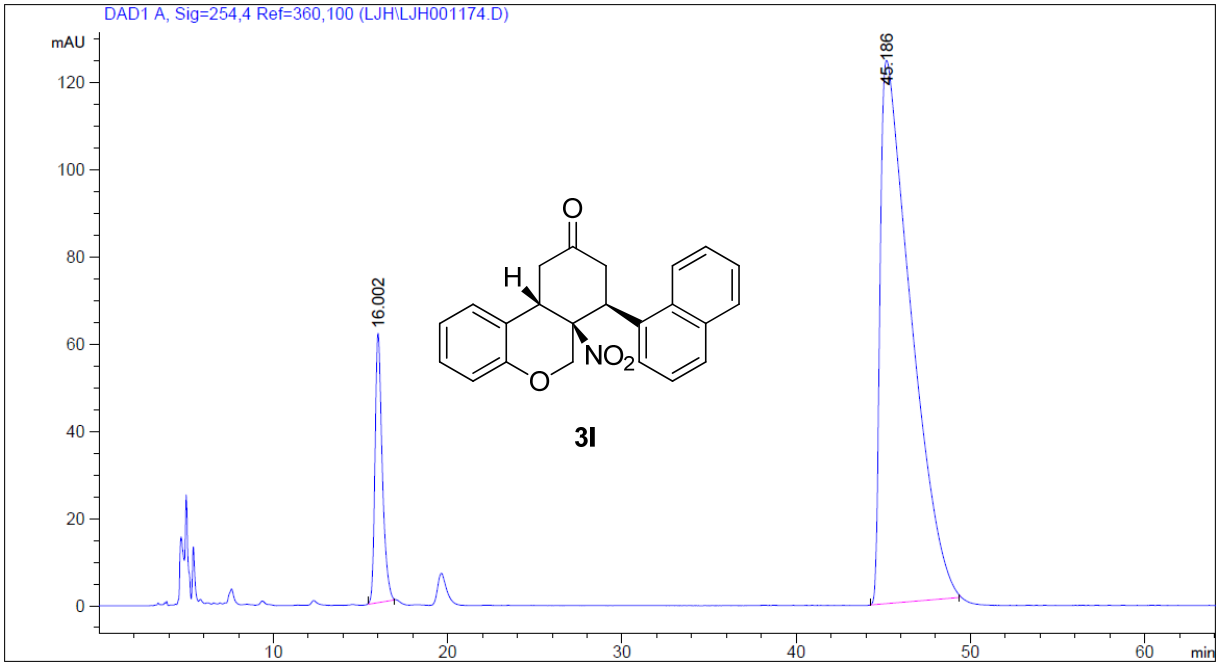
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.659	BB	0.4597	1.12965e4	370.53738	41.0675
2	21.386	BB	0.5722	1.62106e4	425.72293	58.9325



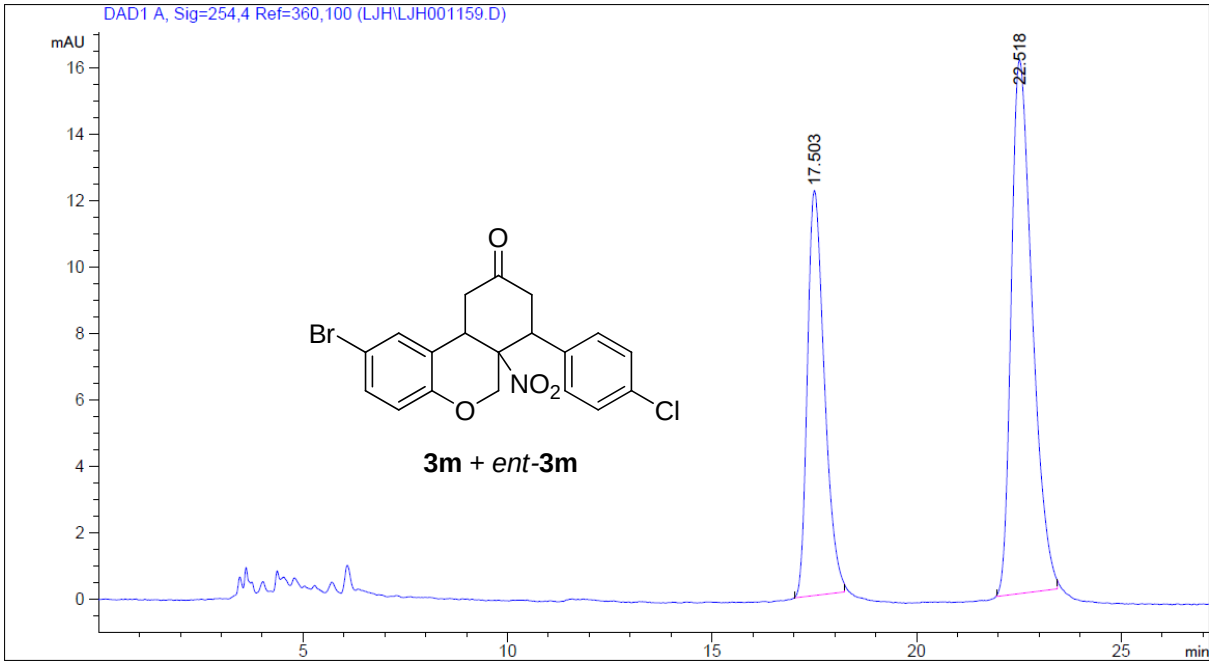
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.669	BB	0.4593	7756.69531	254.69623	12.3028
2	21.044	BB	0.6409	5.52913e4	1292.93970	87.6972



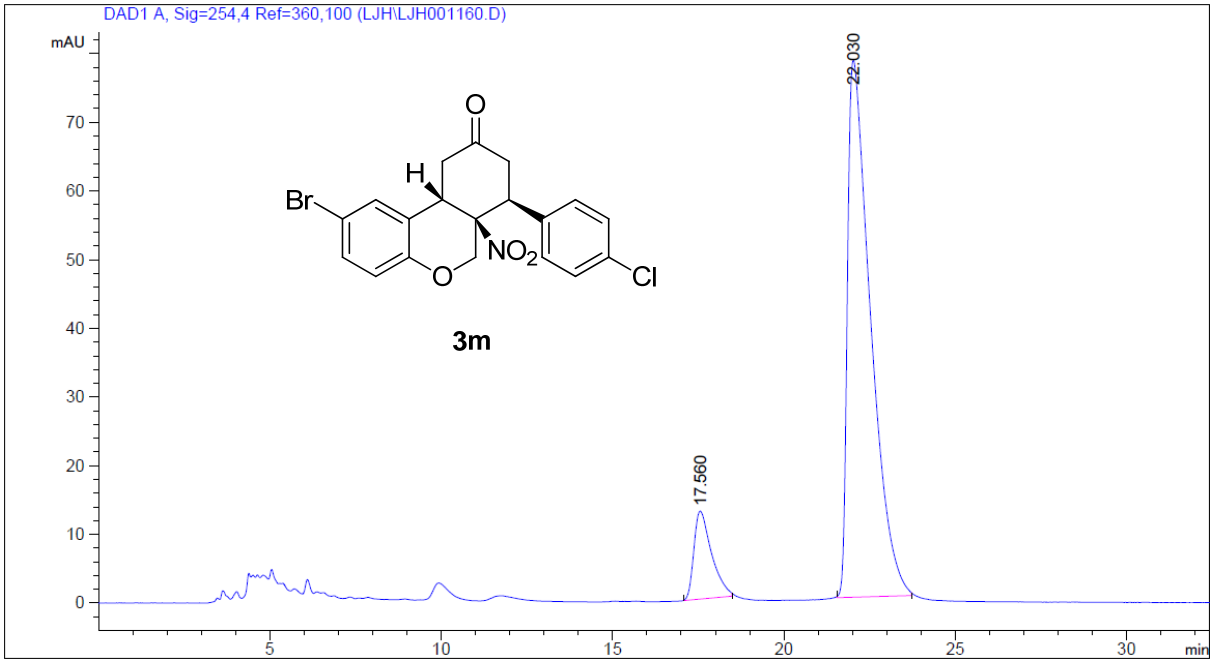
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.033	BB	0.4423	3632.94116	123.82288	37.7897
2	46.175	BB	1.3871	5980.62354	58.97387	62.2103



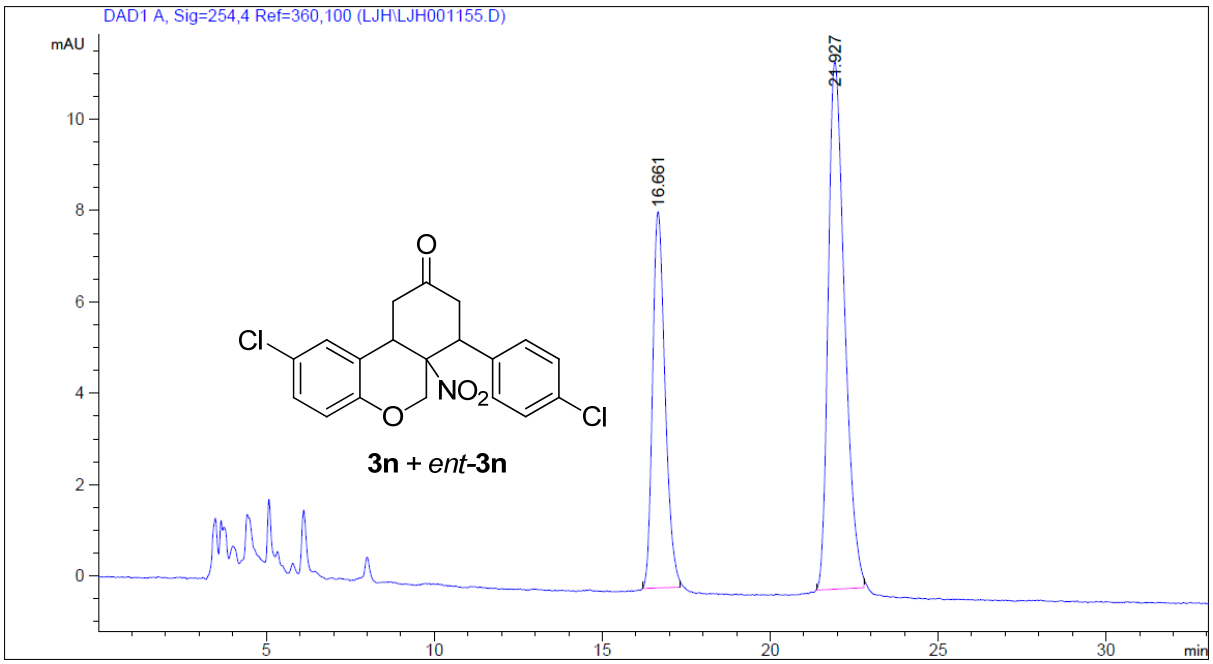
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.002	BB	0.4339	1789.86902	61.80561	10.4801
2	45.186	BB	1.6473	1.52888e4	124.61682	89.5199



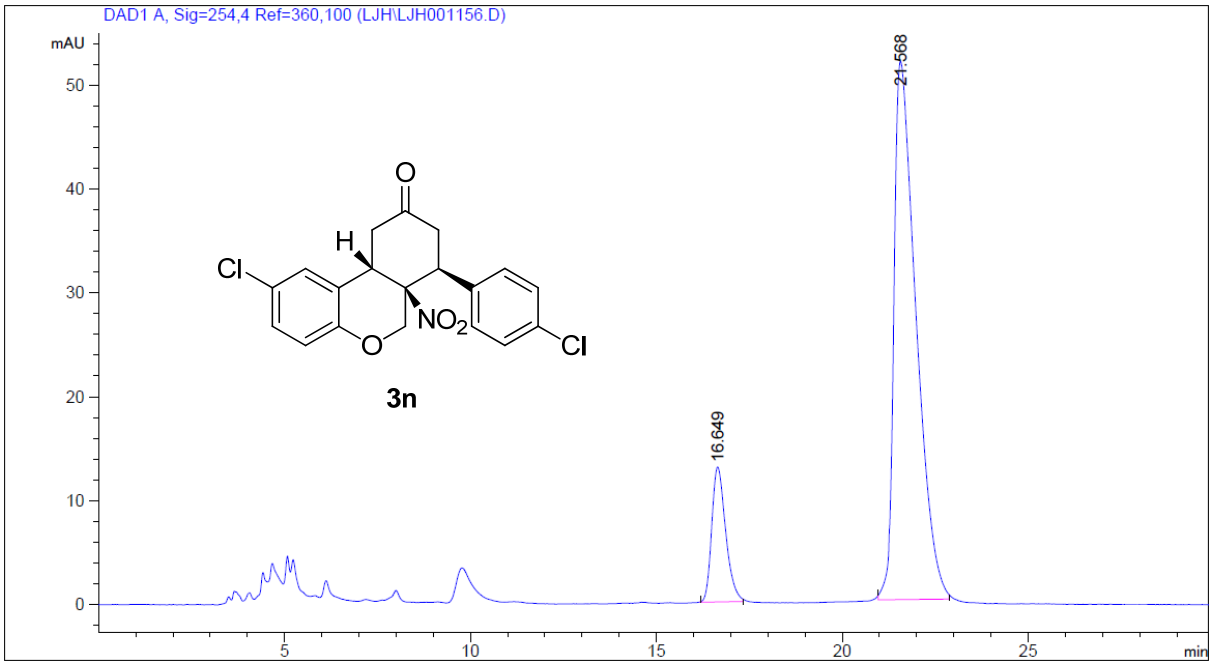
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.503	BB	0.4400	351.22867	12.19471	37.9675
2	22.518	BB	0.5230	573.84735	16.05537	62.0325



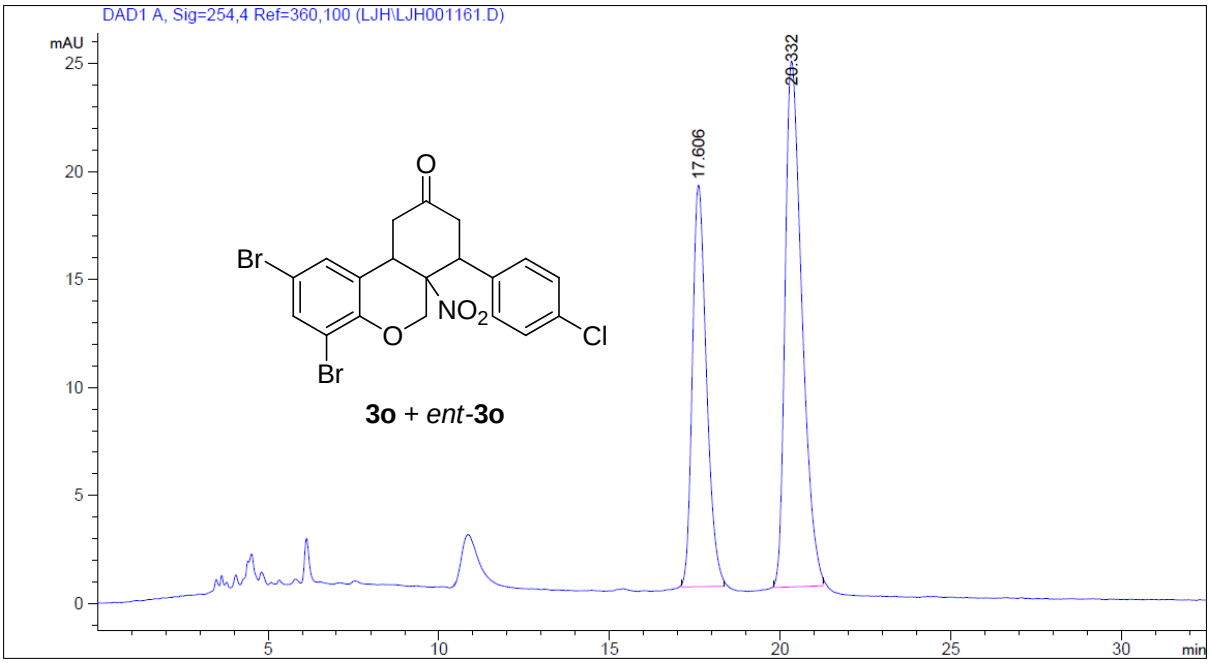
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.560	BB	0.5228	458.19385	12.82615	11.1396
2	22.030	BB	0.6731	3654.99976	78.22420	88.8604



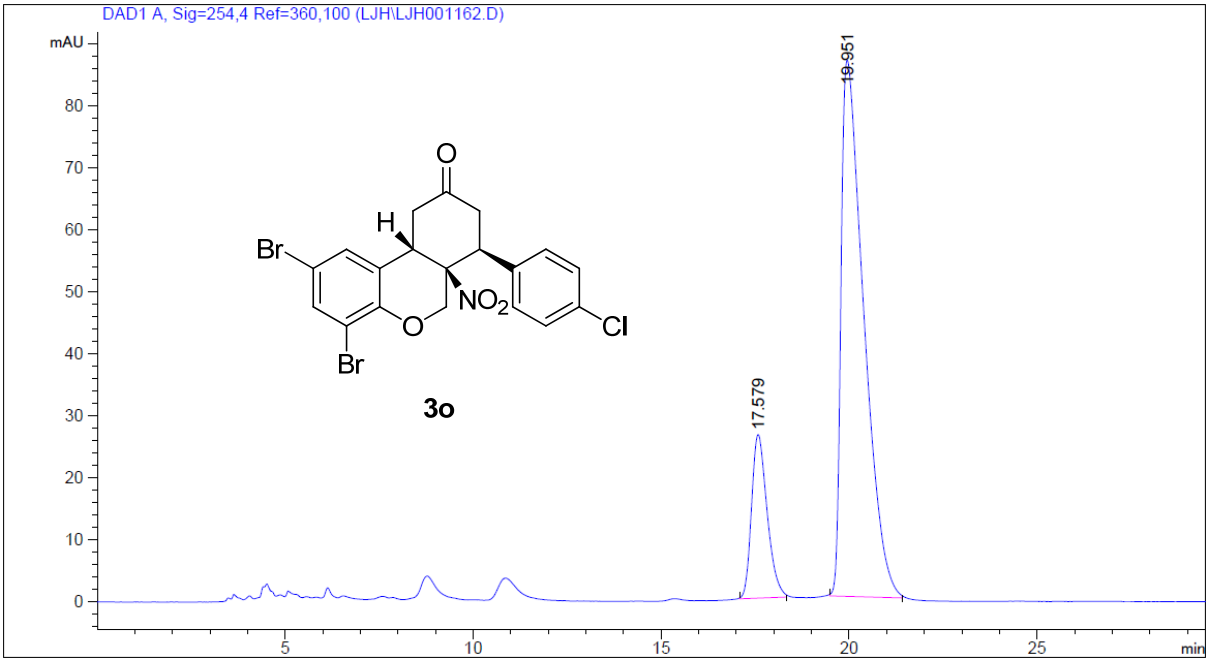
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.661	BB	0.3917	212.99754	8.23746	35.2237
2	21.927	BB	0.5080	391.70148	11.54146	64.7763



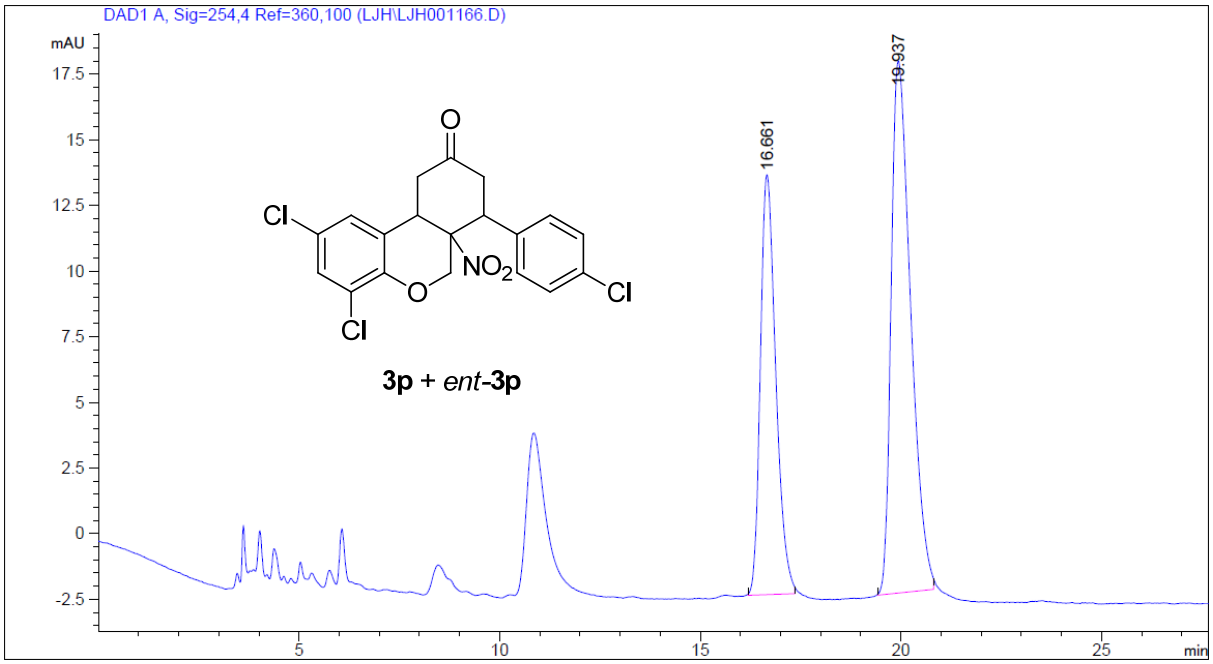
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.649	BB	0.4000	338.39282	12.98767	13.6989
2	21.568	BB	0.6041	2131.81860	51.80604	86.3011



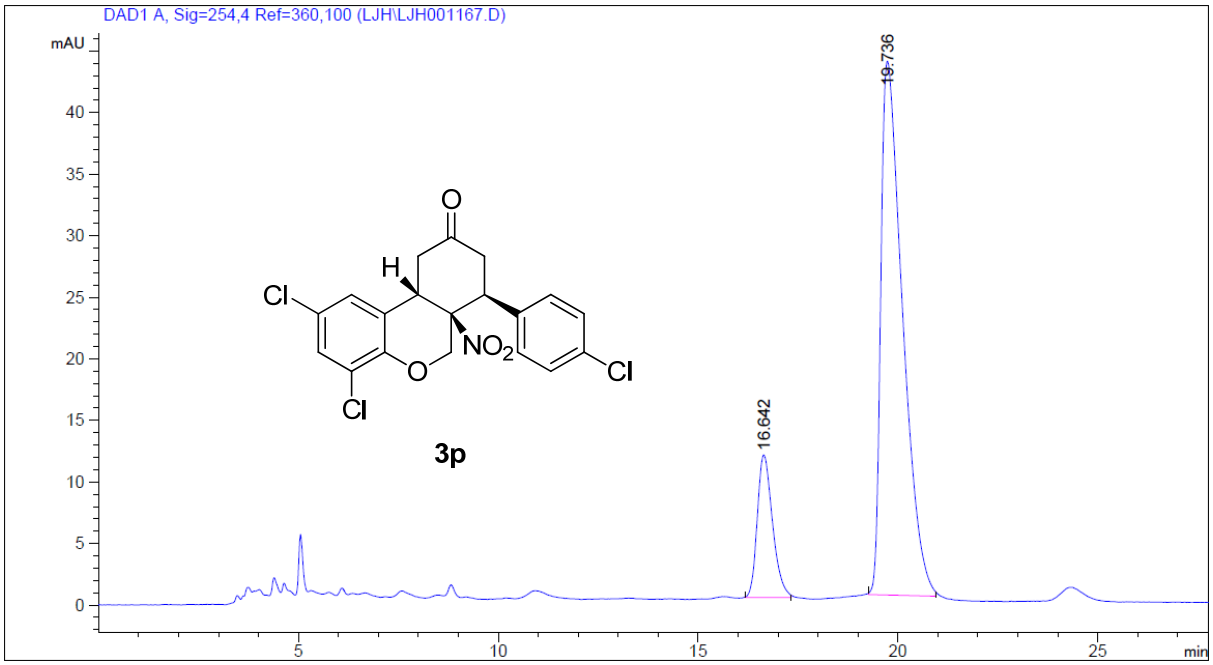
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.606	BB	0.4370	534.48517	18.61650	38.9633
2	20.332	BB	0.5149	837.27881	24.36364	61.0367



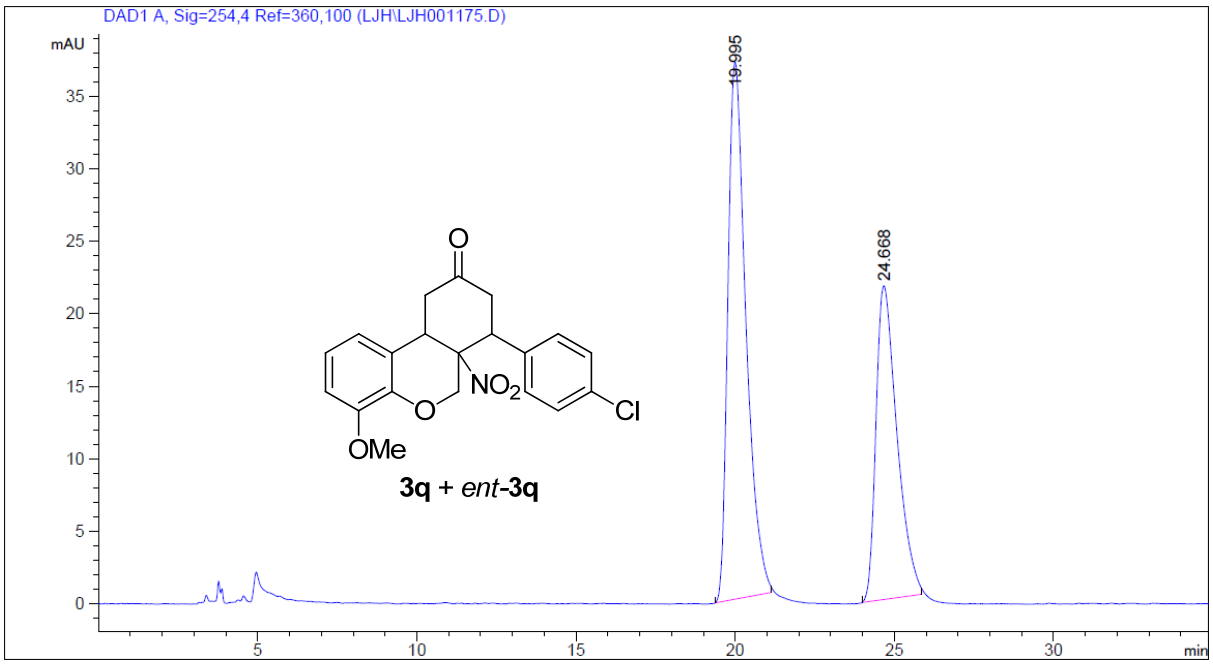
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.579	BB	0.4420	759.47894	26.37248	17.3558
2	19.951	BB	0.6120	3616.45972	86.46193	82.6442



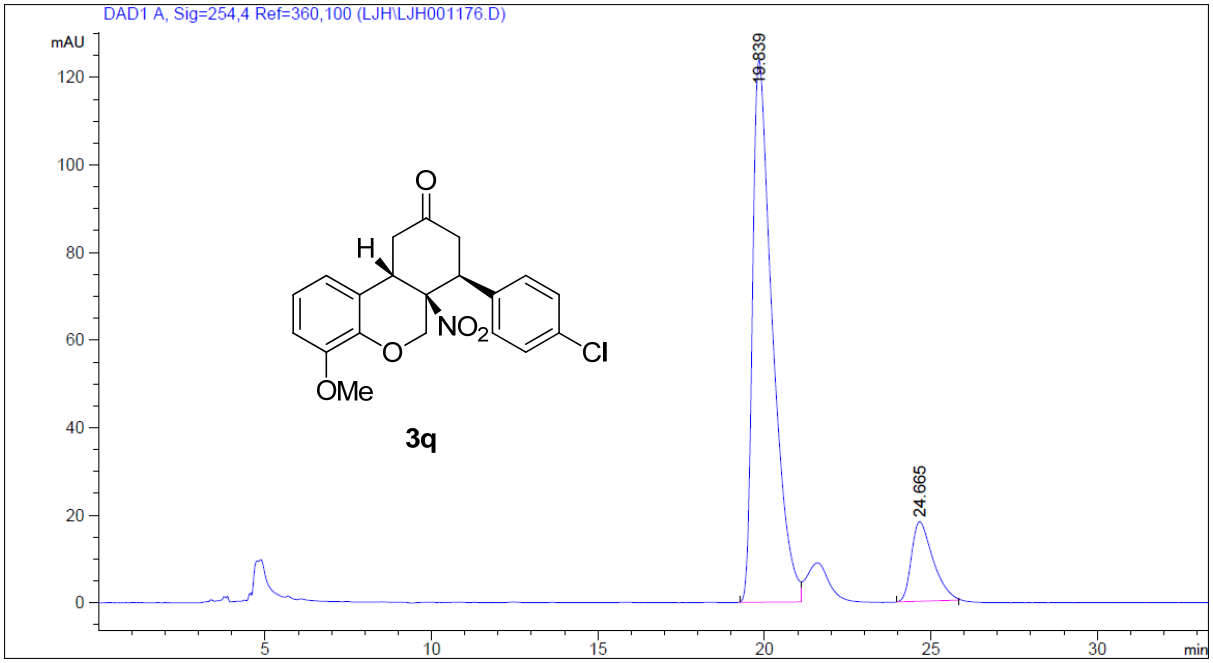
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.661	BB	0.4090	426.31653	15.99333	38.4347
2	19.937	BB	0.5132	682.88013	20.26348	61.5653



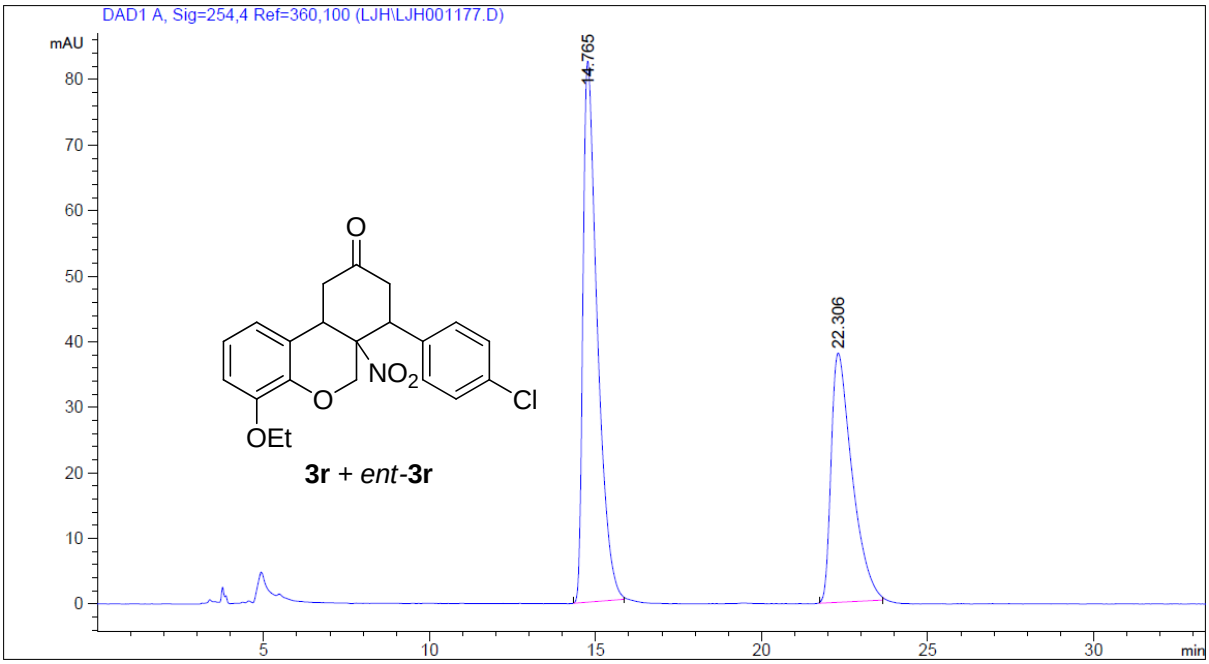
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.642	BB	0.4091	307.08994	11.59056	15.7796
2	19.736	BB	0.5529	1639.02454	43.36795	84.2204



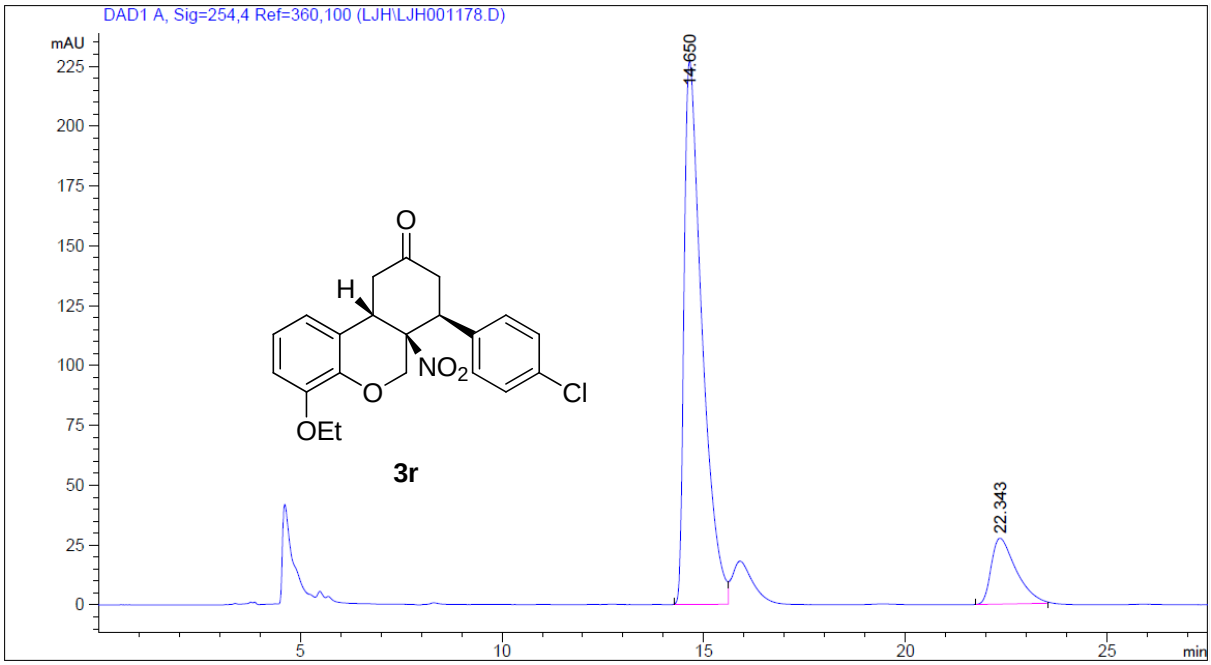
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.995	BB	0.5878	1459.69482	37.04039	59.6546
2	24.668	BB	0.6683	987.21570	21.63717	40.3454



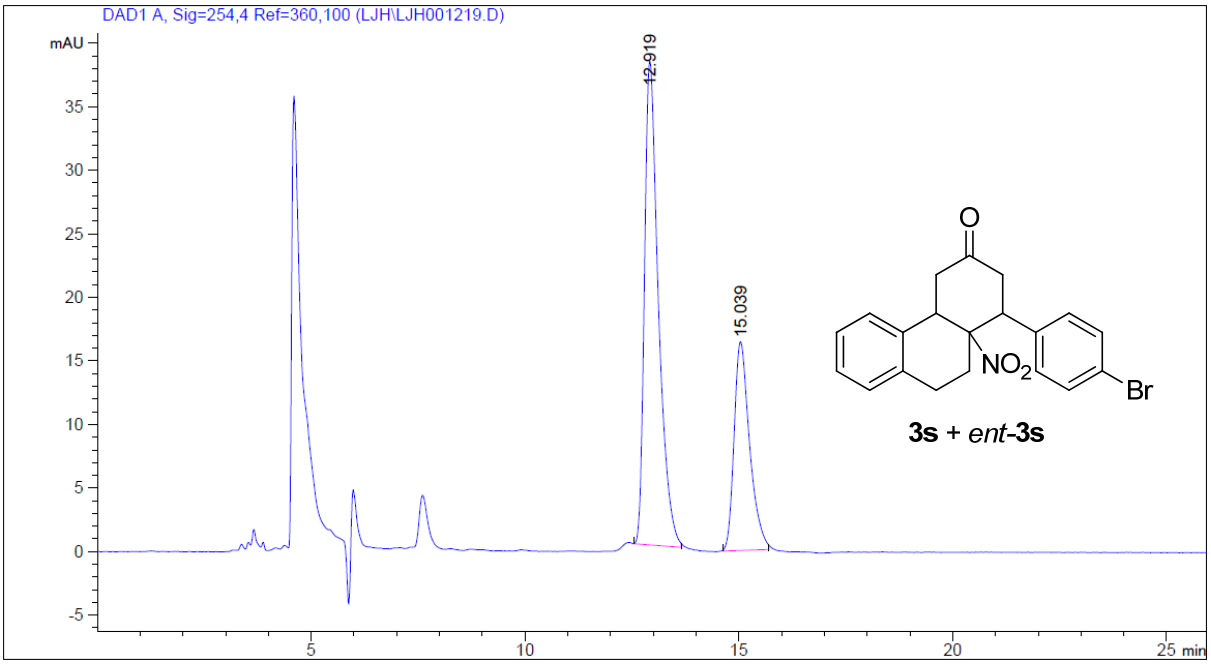
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.839	BB	0.6029	5146.85254	123.83667	86.2782
2	24.665	BB	0.6701	818.56287	18.15068	13.7218



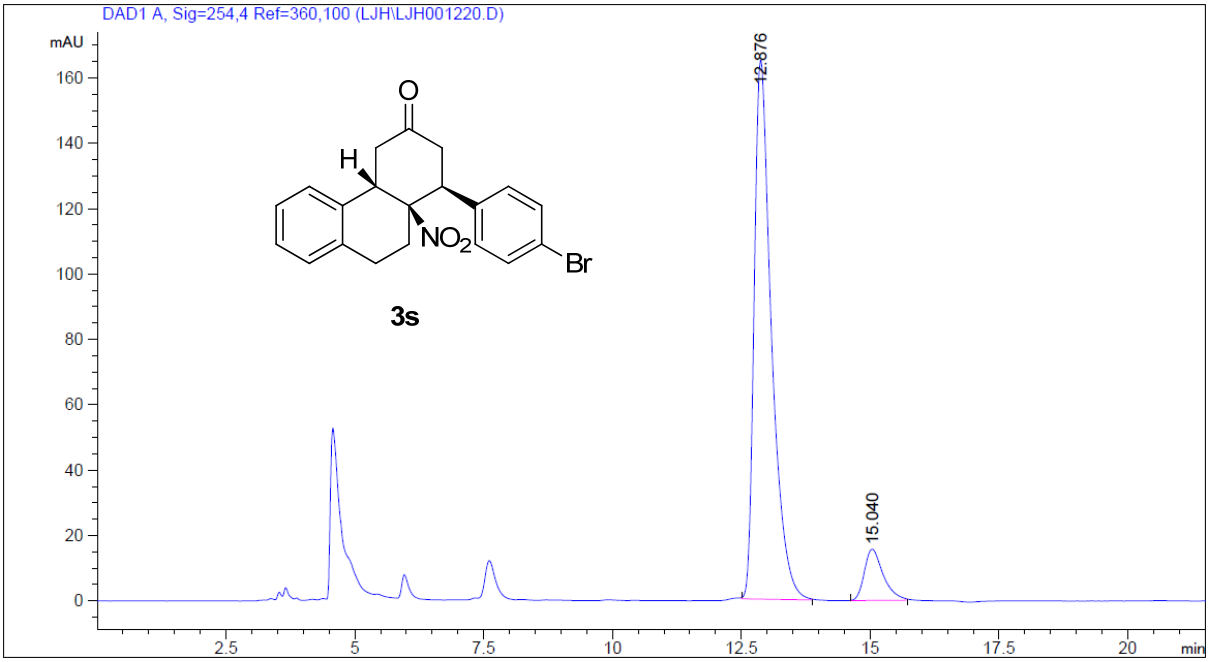
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.765	BB	0.4465	2479.80713	82.54687	59.7683
2	22.306	BB	0.6421	1669.22864	38.03167	40.2317



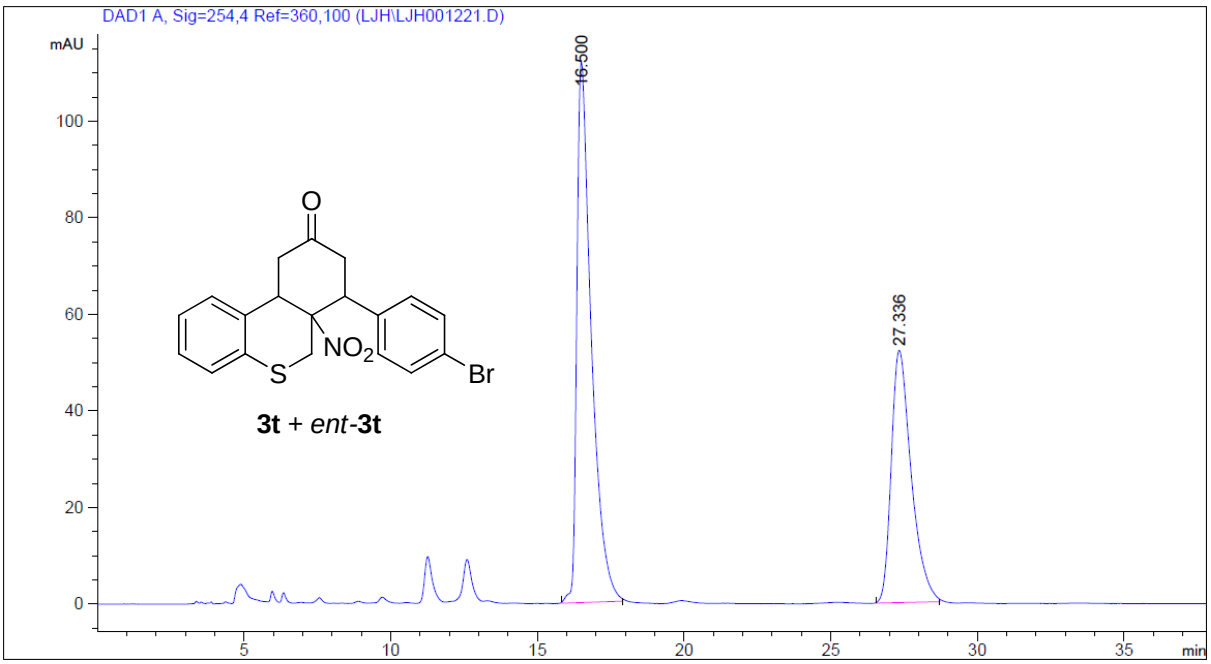
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.650	BV	0.4550	7022.62793	226.92572	85.6436
2	22.343	BB	0.6349	1177.20081	27.52623	14.3564



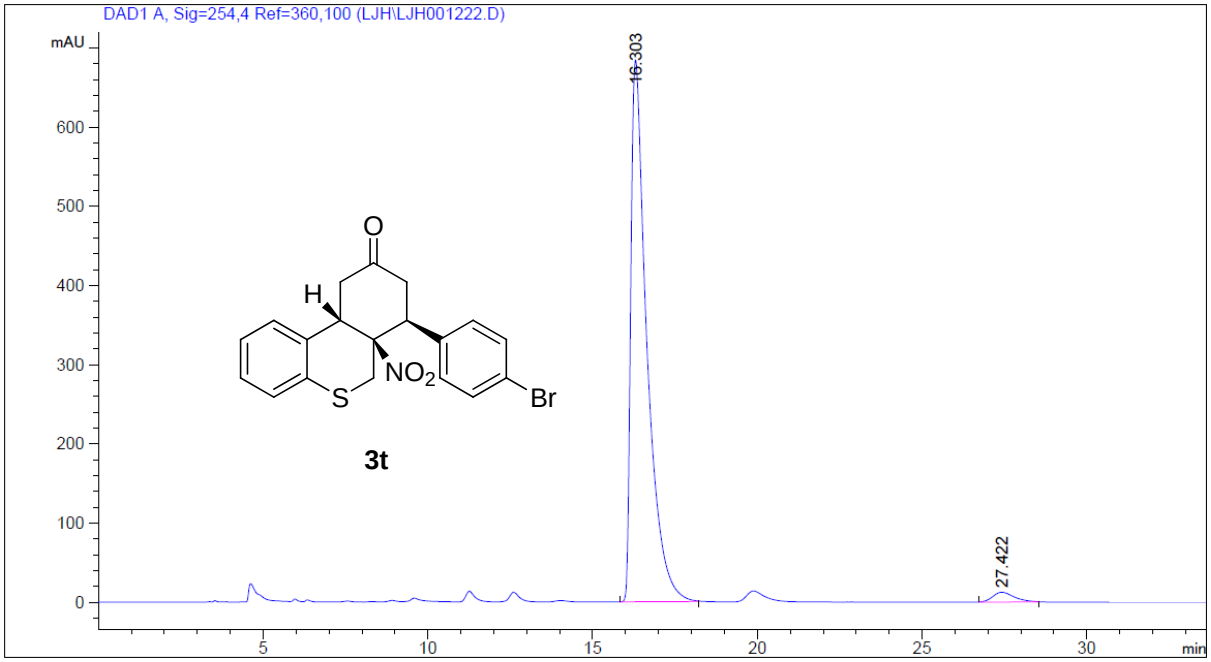
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.919	BB	0.3384	862.65649	38.06381	67.6160
2	15.039	BB	0.3774	413.16016	16.43094	32.3840



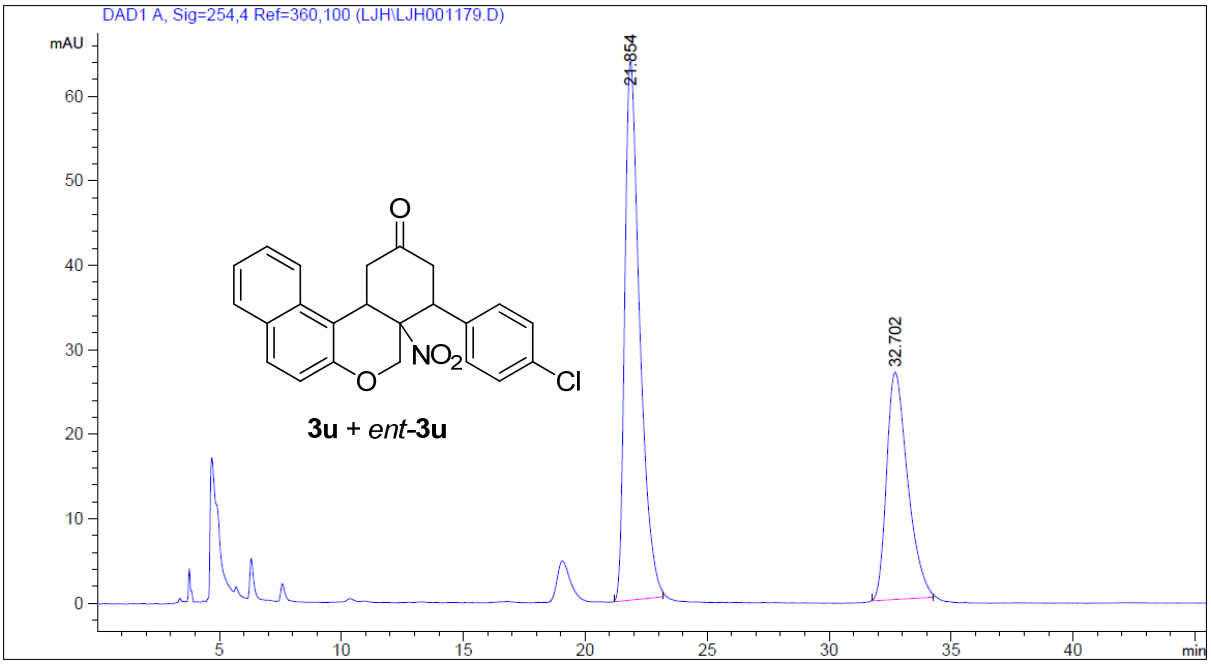
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.876	BB	0.3453	3836.12158	164.95131	90.5959
2	15.040	BB	0.3818	398.20151	15.70458	9.4041



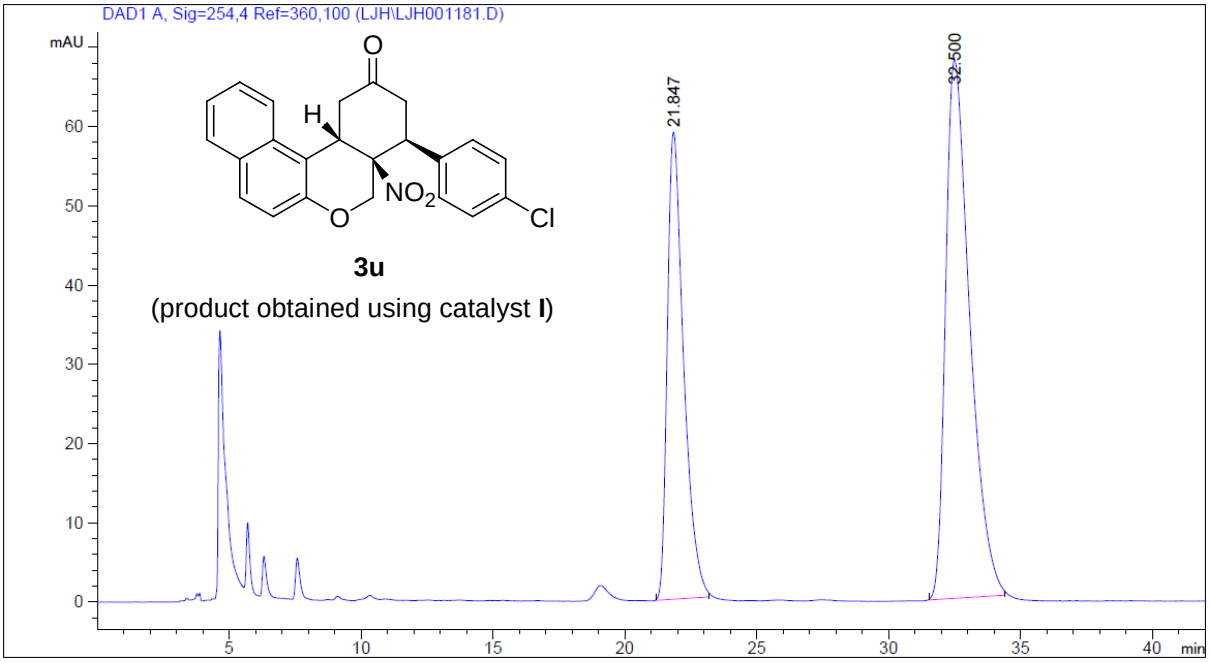
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.500	BB	0.5007	3843.54297	111.91351	60.9078
2	27.336	BB	0.7132	2466.88599	52.21719	39.0922



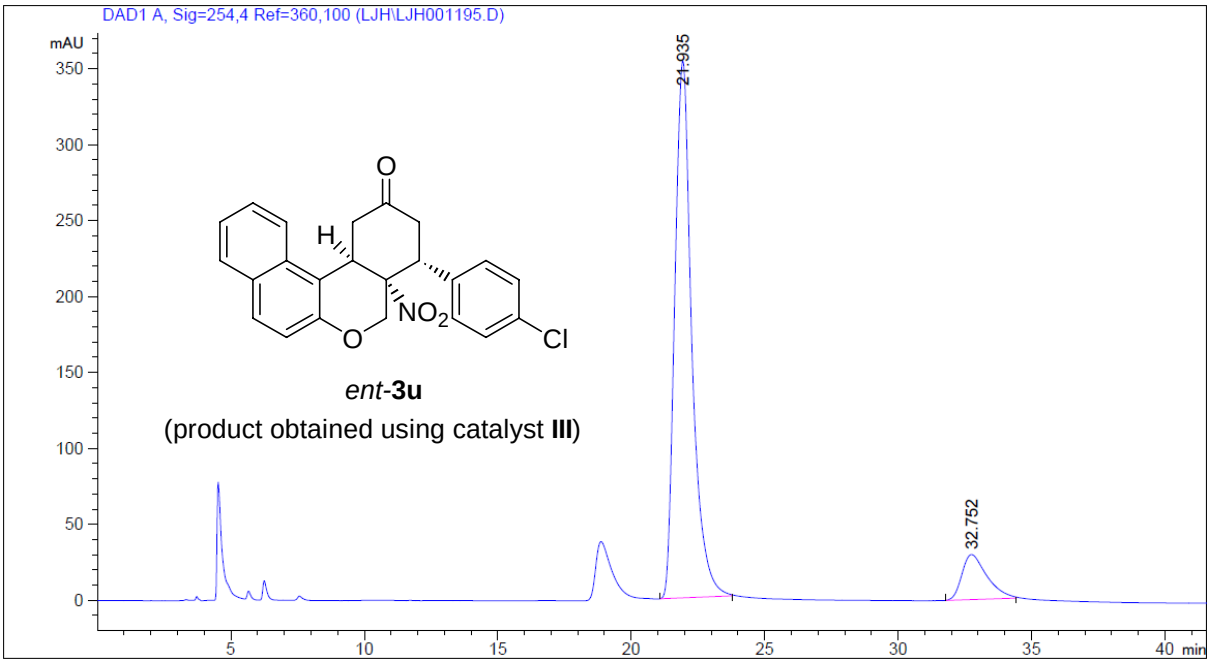
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.303	BB	0.4919	2.32241e4	684.32971	97.6283
2	27.422	BB	0.6738	564.19922	12.42386	2.3717



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	21.854	BB	0.6424	2736.17480	63.78766	62.8789
2	32.702	BB	0.8765	1615.32422	26.90358	37.1211



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	21.847	BB	0.6429	2530.90234	58.94249	36.9074
2	32.500	BB	0.9493	4326.52832	67.91057	63.0926



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(product obtained using catalyst **III**)

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	21.935	BB	0.6807	1.56547e4	353.47754	89.0541
2	32.752	BB	0.9645	1924.17493	29.59525	10.9459