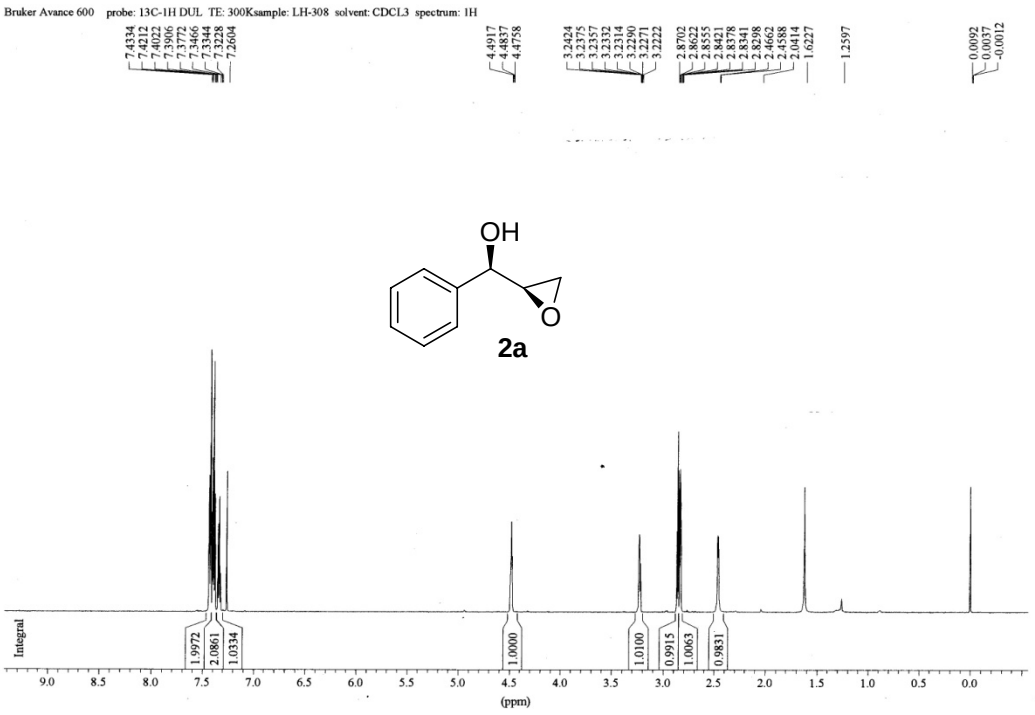
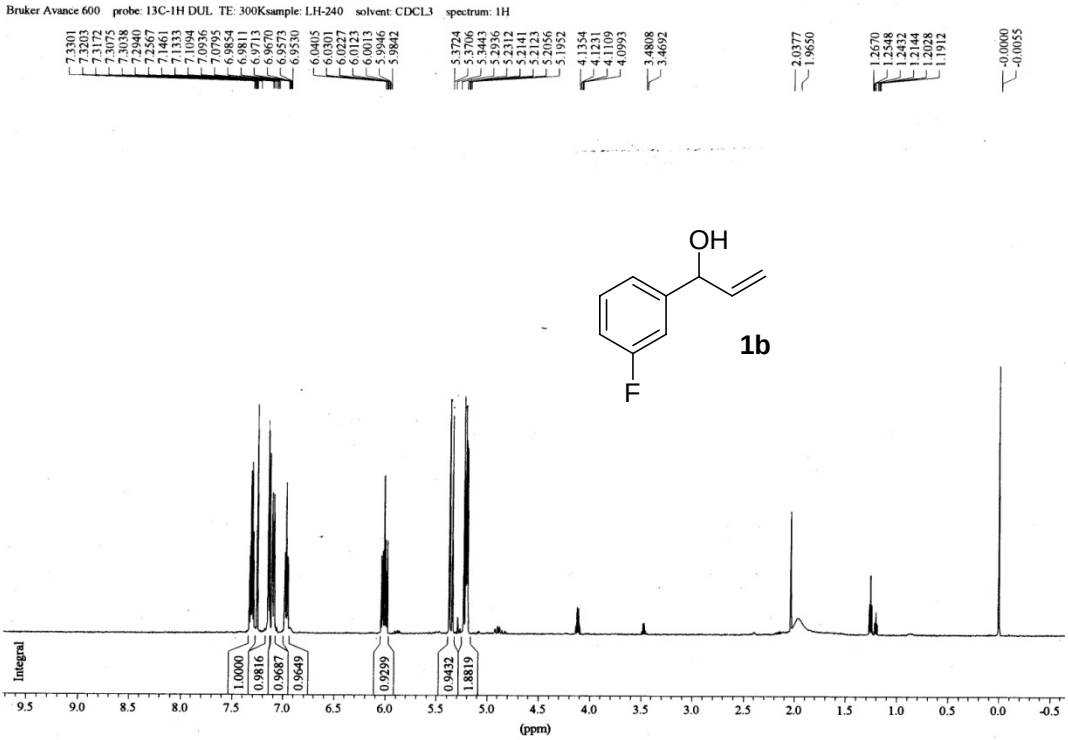
Selected 1HNMR, 13CNMR and HPLC

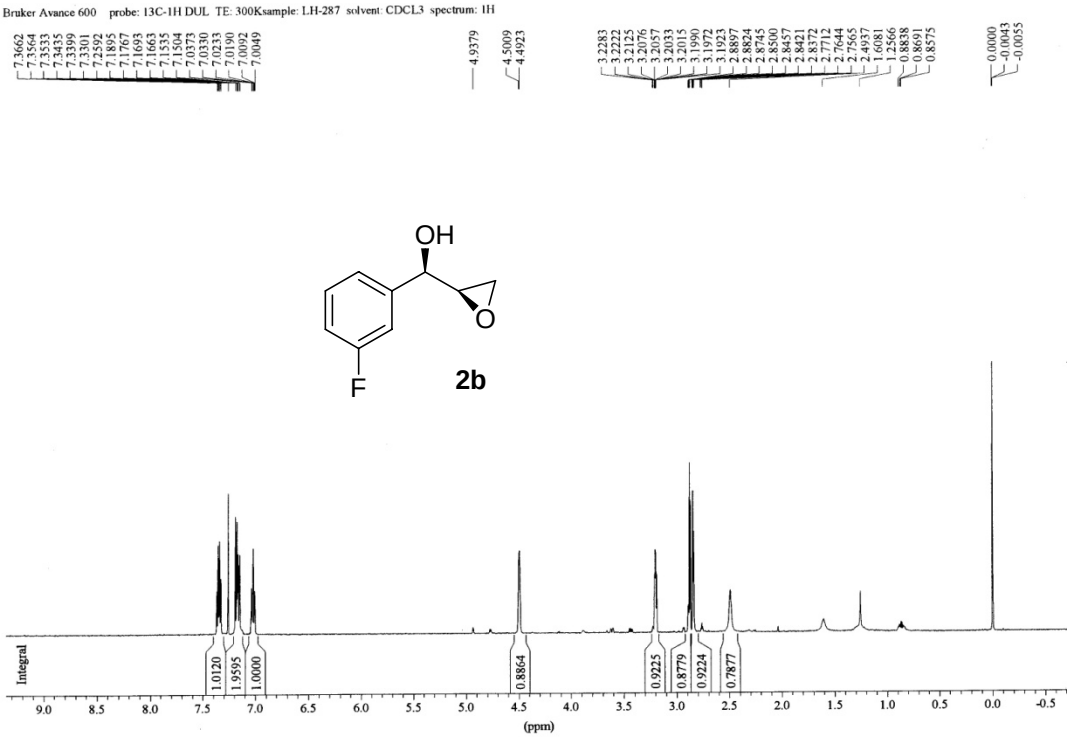
Highly Diastereo- and Enantioselective Epoxidation of Secondary Allylic Alcohols Catalyzed by Styrene Monooxygenase

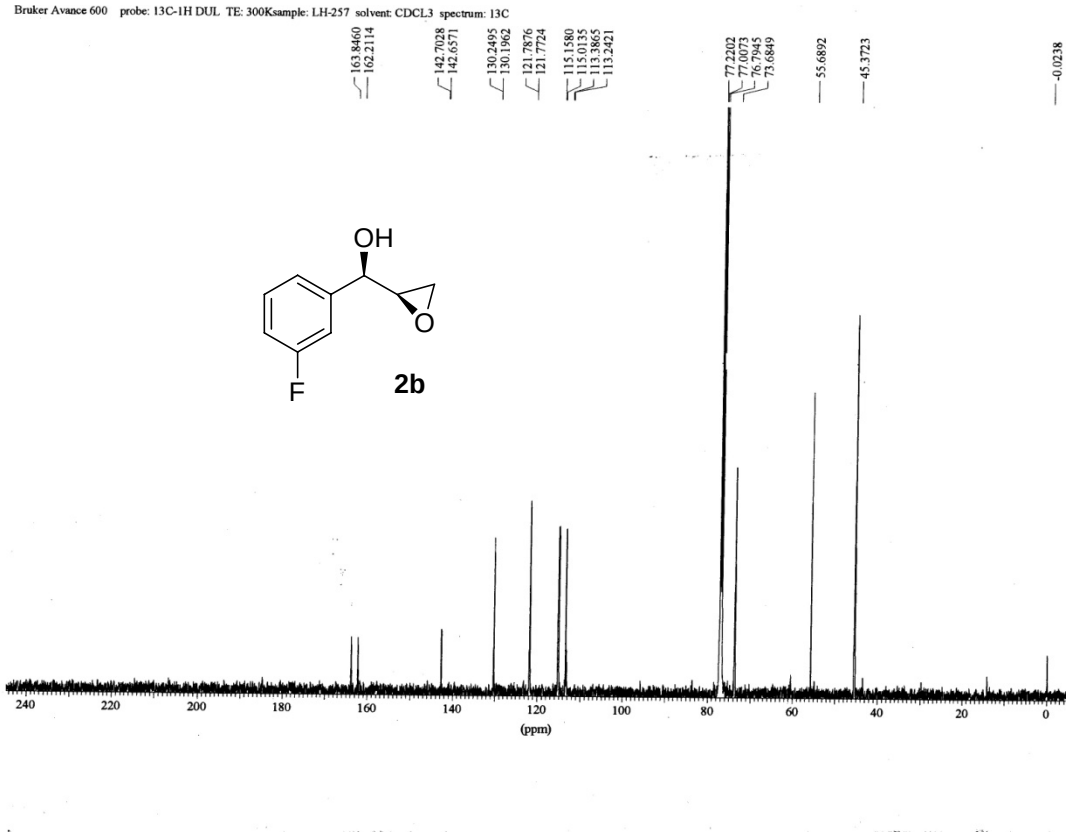
*Hui Lin, Yan Liu, and Zhong-Liu Wu**

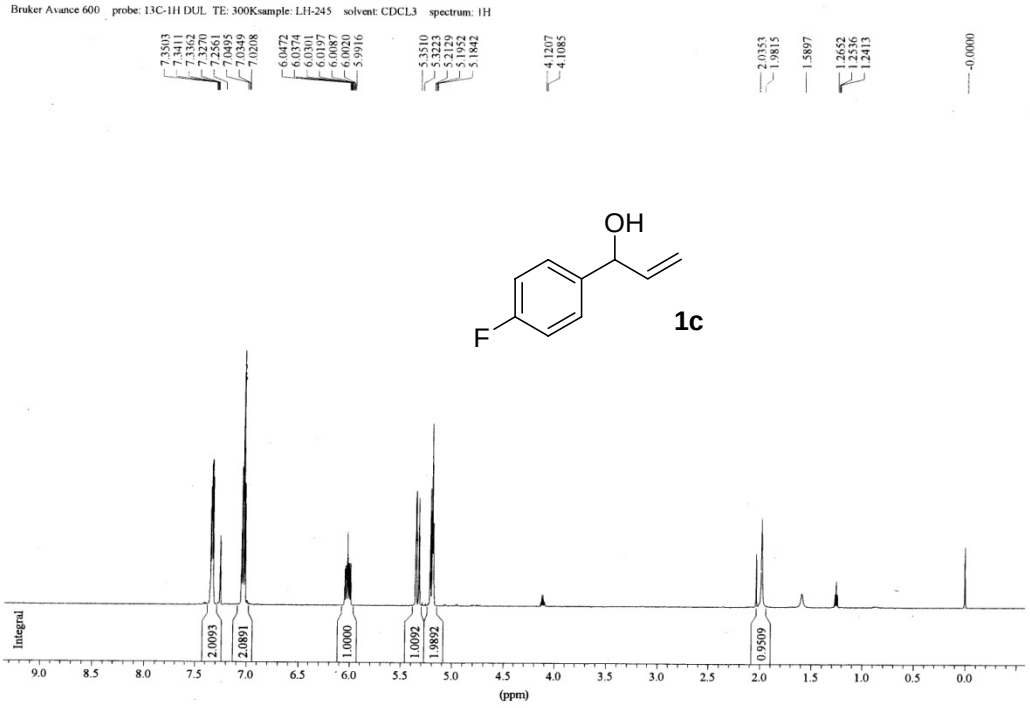
Selected ^{1}H NMR and ^{13}C NMR

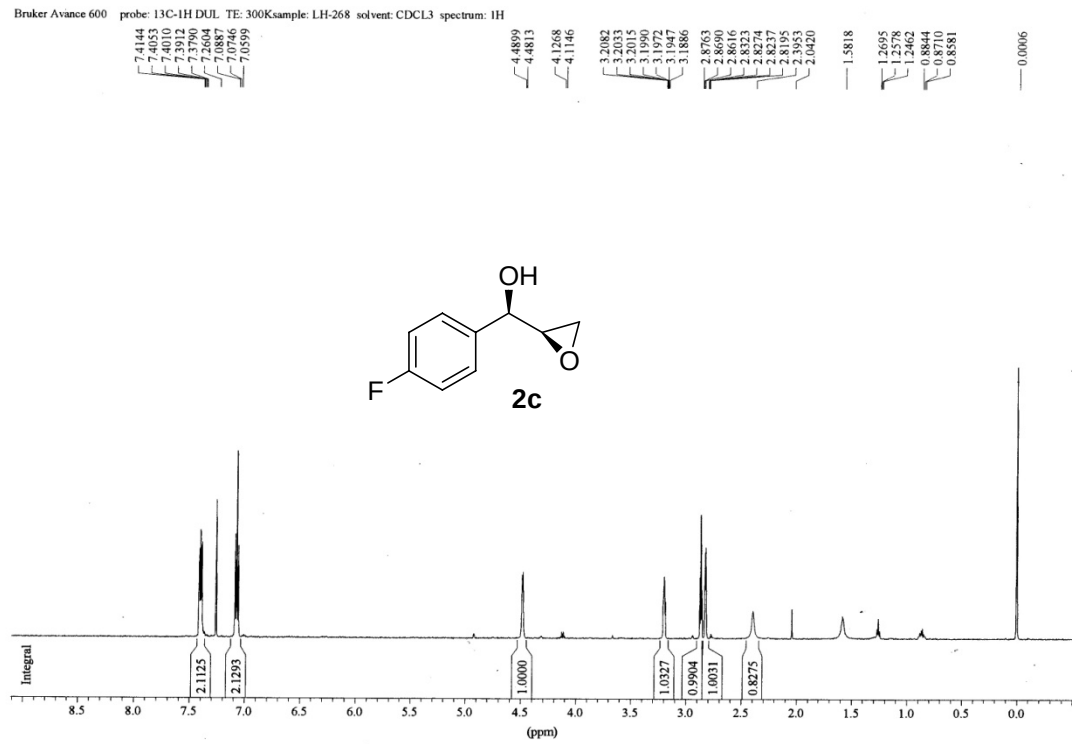


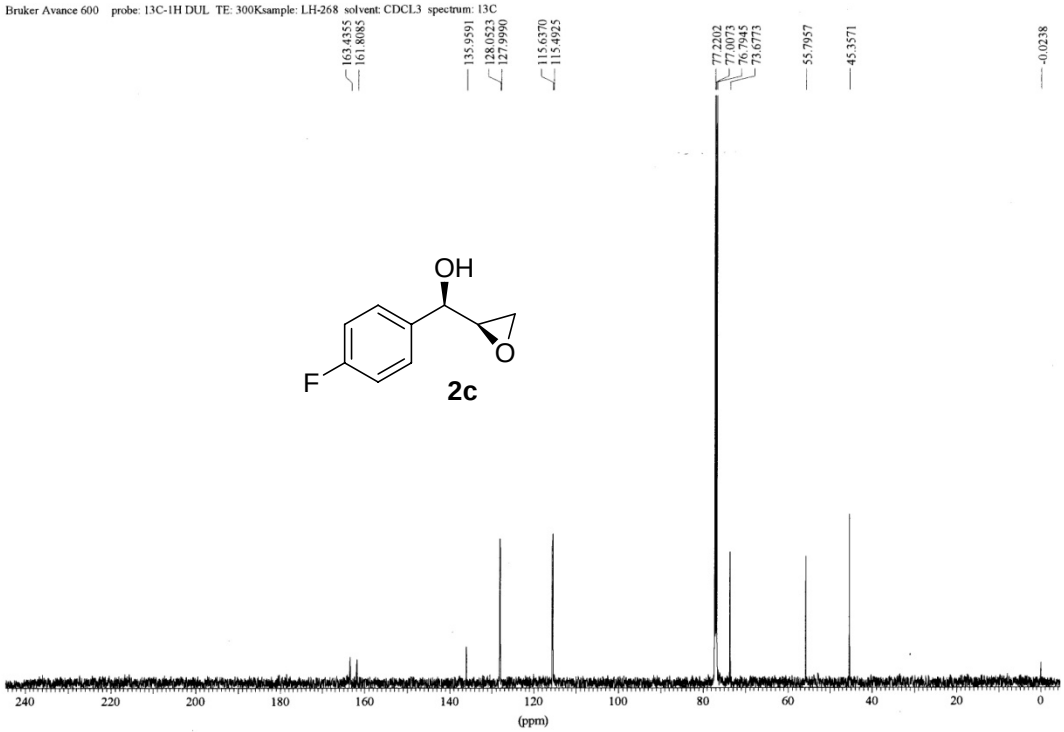


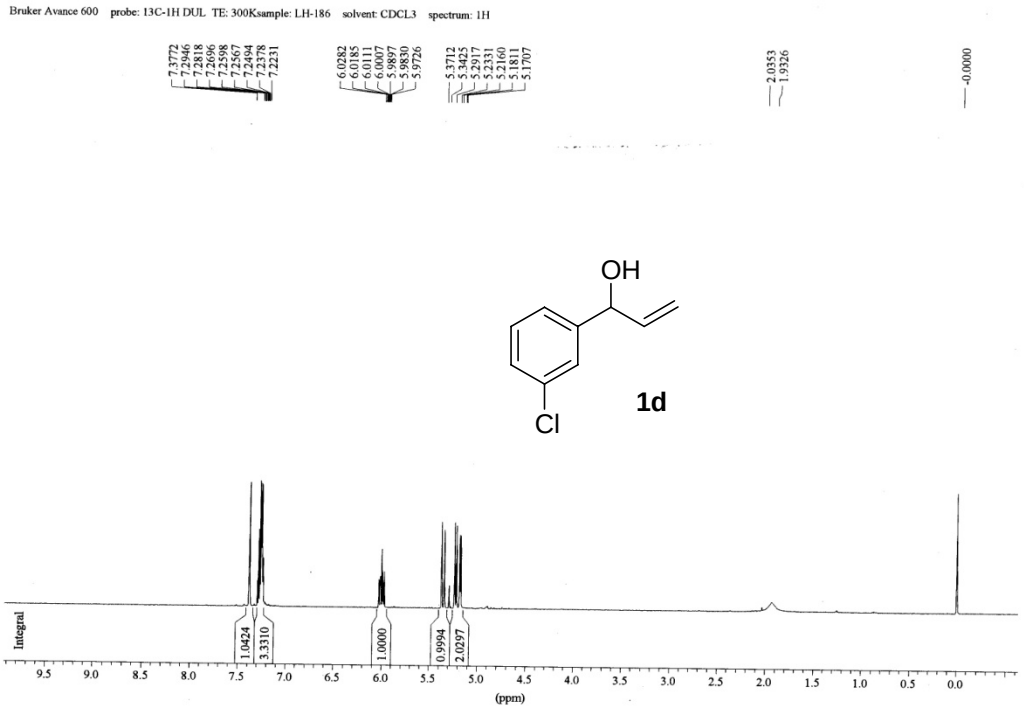

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-287 solvent: CDCL3 spectrum: 1H
7.3662
7.3564
7.3533
7.3435
7.3399
7.3301
7.2592
7.1895
7.1767
7.1693
7.1663
7.1535
7.1504
7.0373
7.0330
7.0233
7.0190
7.0092
7.0049
4.9379
4.5009
4.4923
3.2283
3.2222
3.2175
3.2076
3.2057
3.2033
3.2015
3.1990
3.1972
3.1923
2.8897
2.8824
2.8745
2.8500
2.8457
2.8421
2.8372
2.7712
2.7644
2.7564
2.4937
1.6081
1.2566
0.8838
0.8691
0.8575
0.0000
-0.0043
-0.0055
OH
O
F
2b
Integral
1.0120
1.9595
1.0000
0.8864
0.9225
0.8779
0.9224
0.7877
(ppm)


Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-257 solvent: CDCL3 spectrum: 13C
163.8460
162.2114
142.7028
142.6571
130.2495
130.1962
121.7876
121.7724
115.1580
115.0135
113.3865
113.2421
77.2202
77.0073
76.7945
73.6849
55.6892
45.3723
-0.0238
OH
O
F
2b
(ppm)

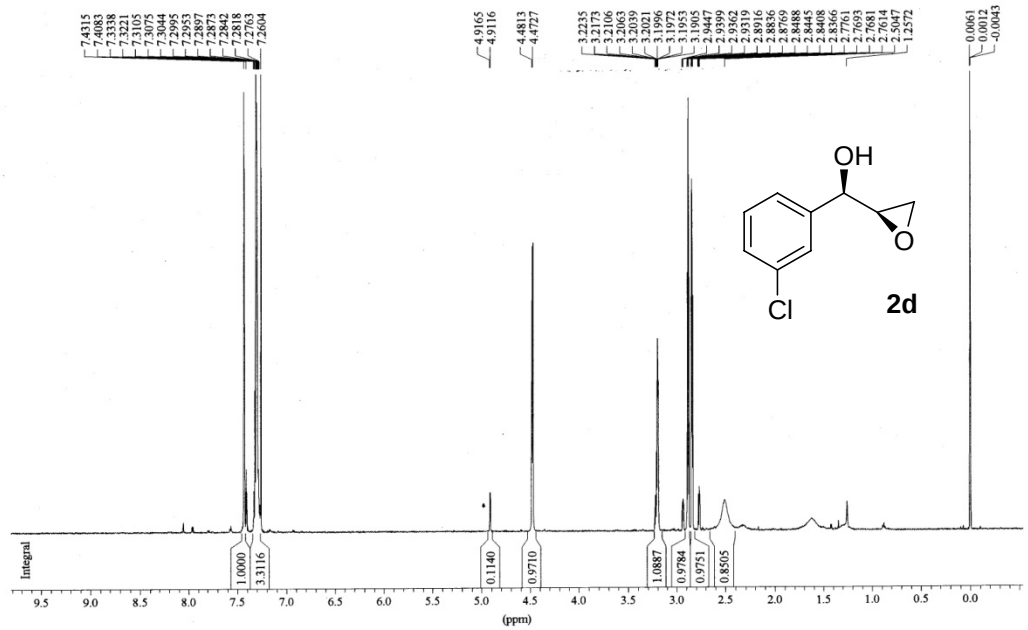

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-245 solvent: CDCL3 spectrum: 1H
OH
F
1c
Integral
(ppm)


Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-268 solvent: CDCL3 spectrum: 1H
OH
O
F
2c
Integral
(ppm)

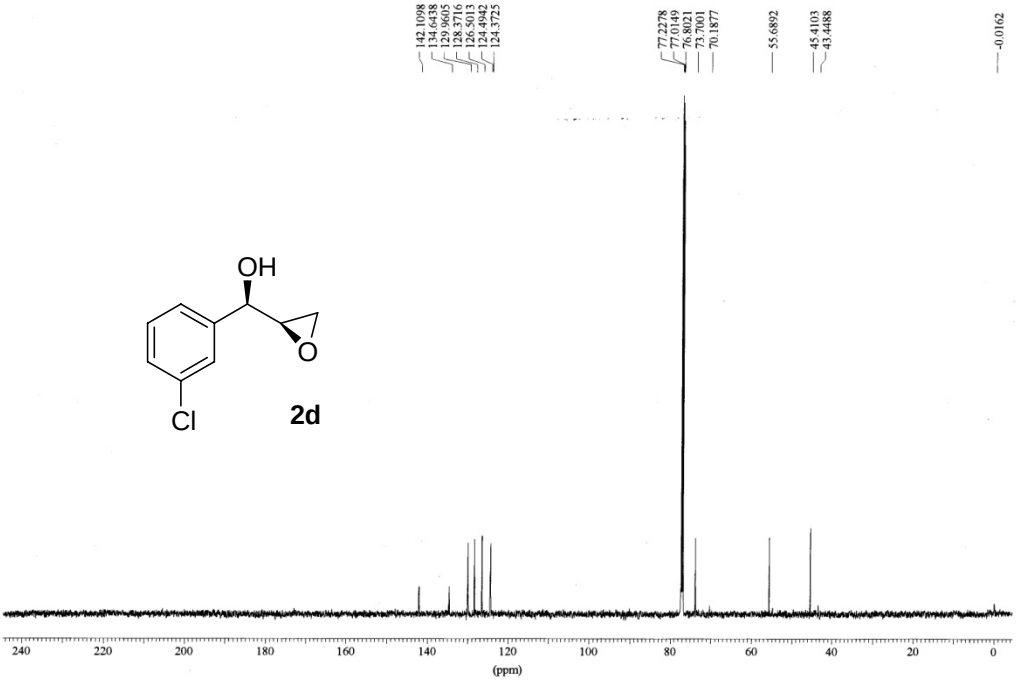

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-268 solvent: CDCL3 spectrum: 13C
163.4355
161.8085
135.9591
128.0523
127.9990
115.6370
115.4925
77.2202
77.0073
76.7945
73.6773
55.7957
45.3571
-0.0238
OH
O
F
2c
240
220
200
180
160
140
120
100
80
60
40
20
0
(ppm)

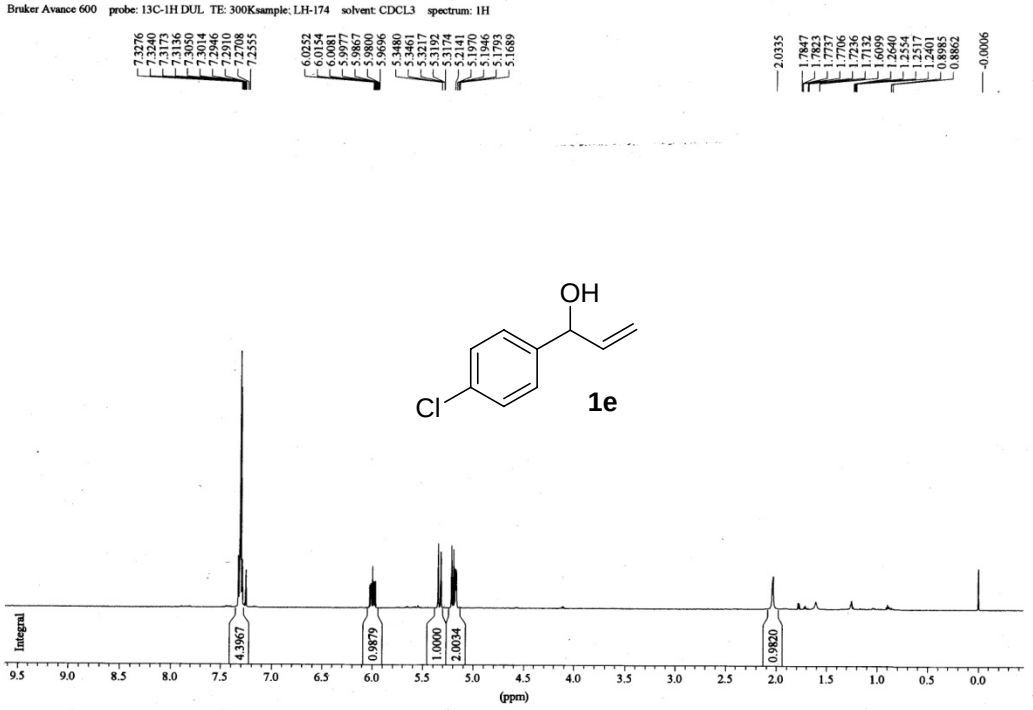

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-186 solvent: CDCL3 spectrum: 1H
7.3772
7.2946
7.2818
7.2696
7.2598
7.2567
7.2494
7.2378
7.2231
6.0282
6.0185
6.0111
6.0007
5.9897
5.9850
5.9726
5.3712
5.3425
5.2917
5.2331
5.2160
5.1811
5.1707
2.0353
1.9326
-0.0000
OH
Cl
1d
Integral
1.0424
3.3310
1.0000
0.9994
2.0297
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
(ppm)

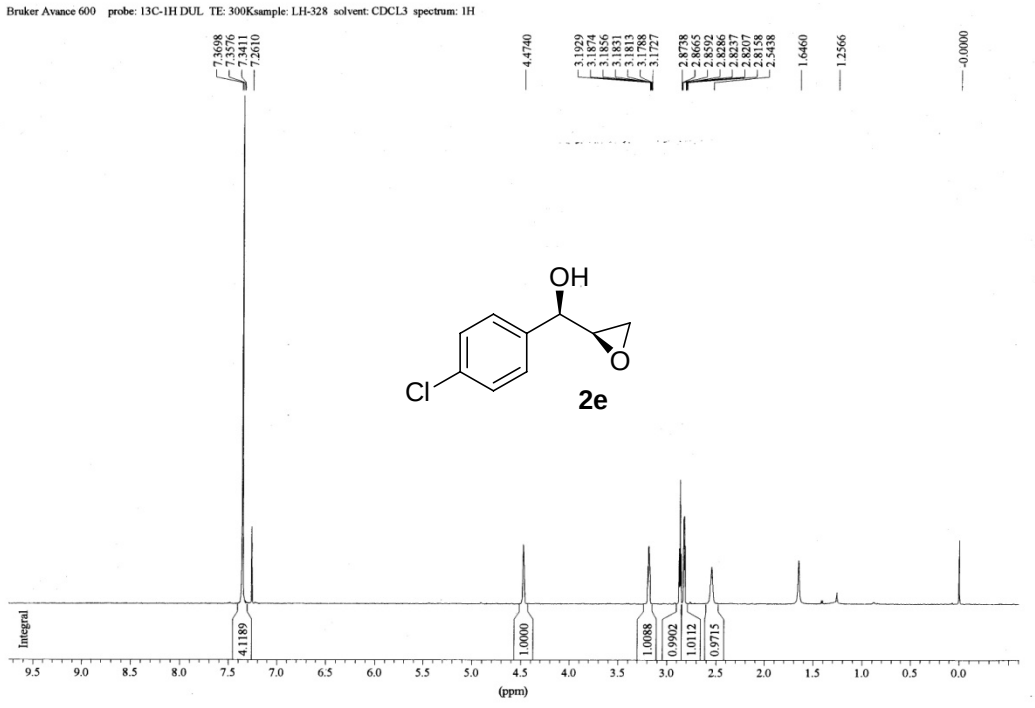
Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-324 solvent: CDCL3 spectrum: 1H



Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-324 solvent: CDCL3 spectrum: 13C




Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-174 solvent: CDCL3 spectrum: 1H
OH
Cl
1e
Integral
4.3967
0.9879
1.0000
2.0034
0.9820
(ppm)


Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-328 solvent: CDCL3 spectrum: 1H
OH
O
Cl
2e
Integral
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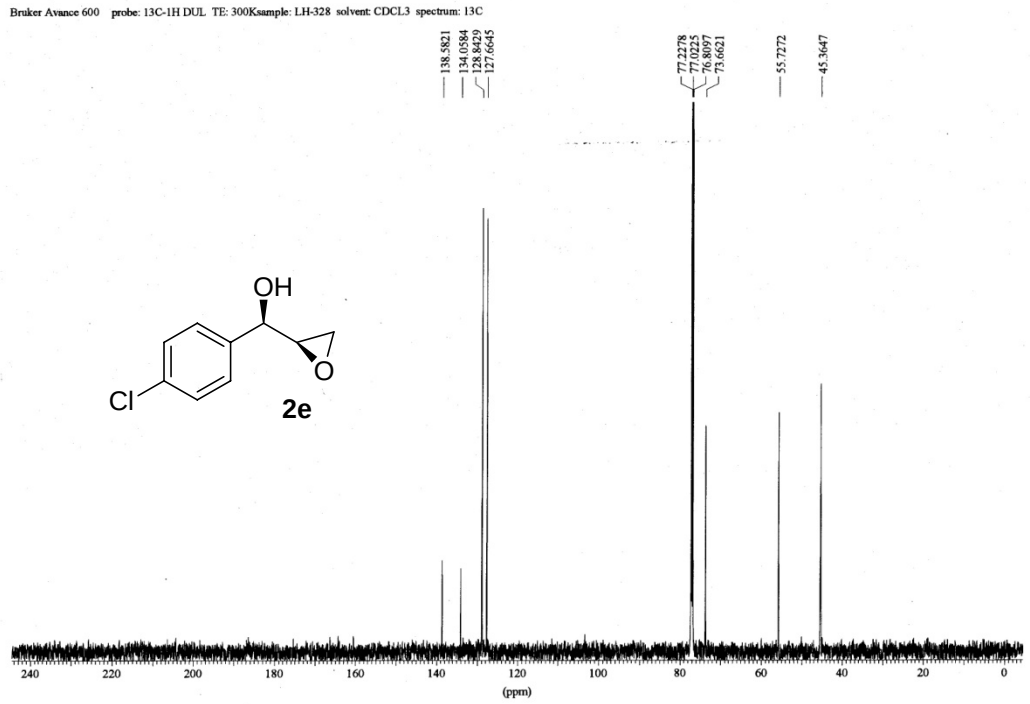
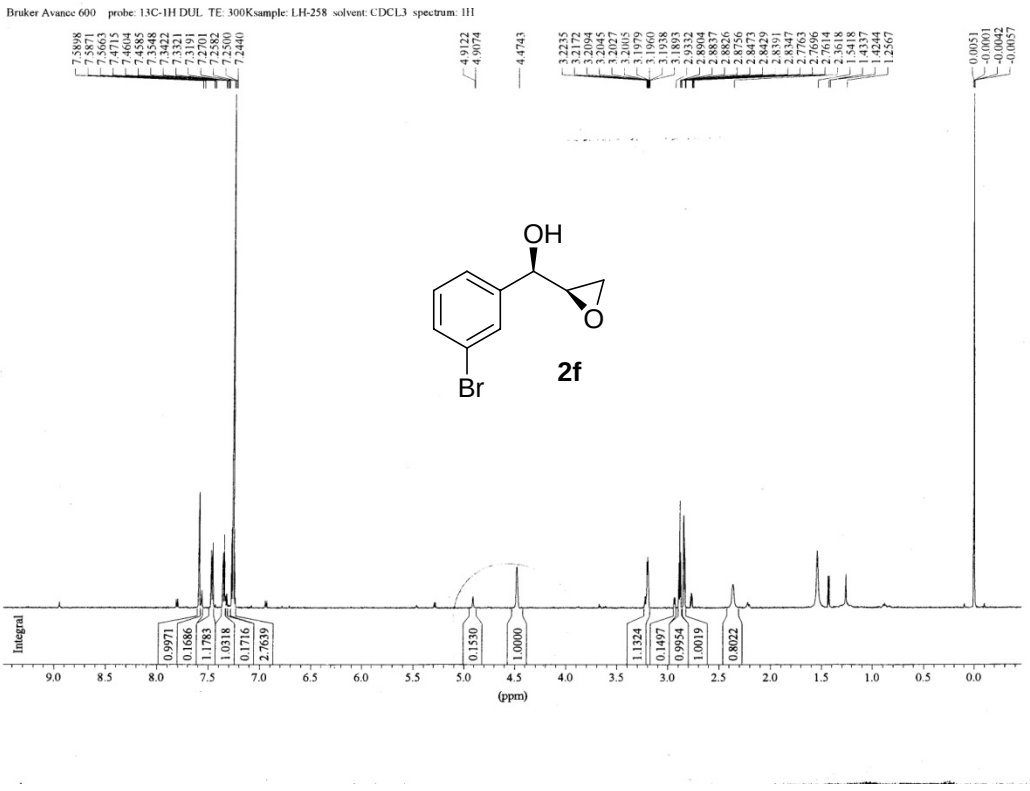
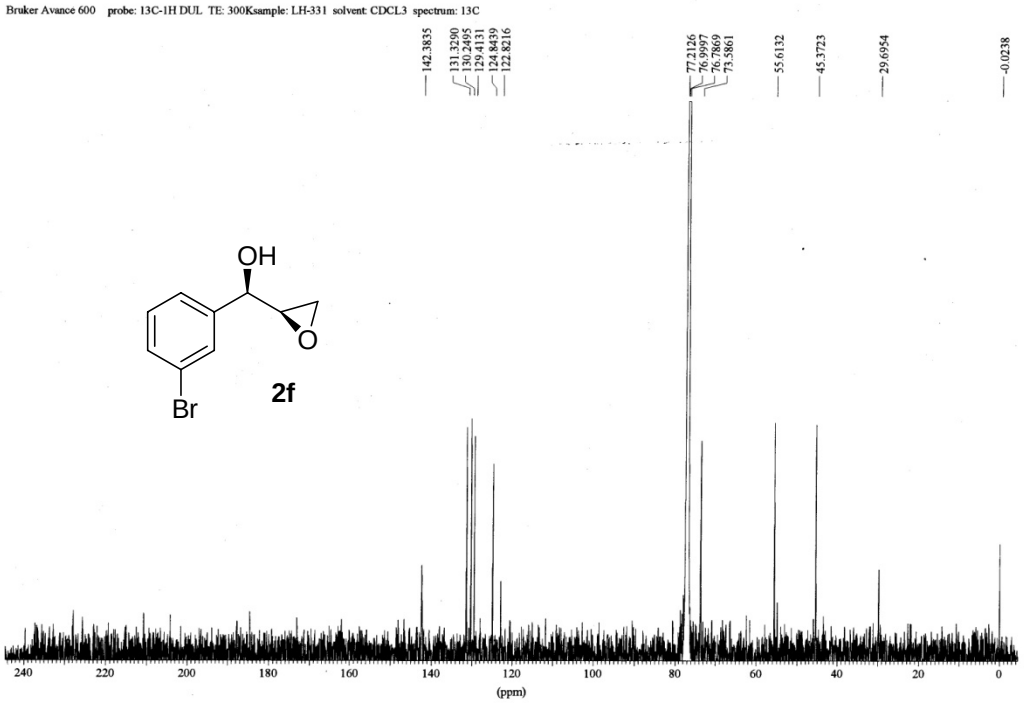
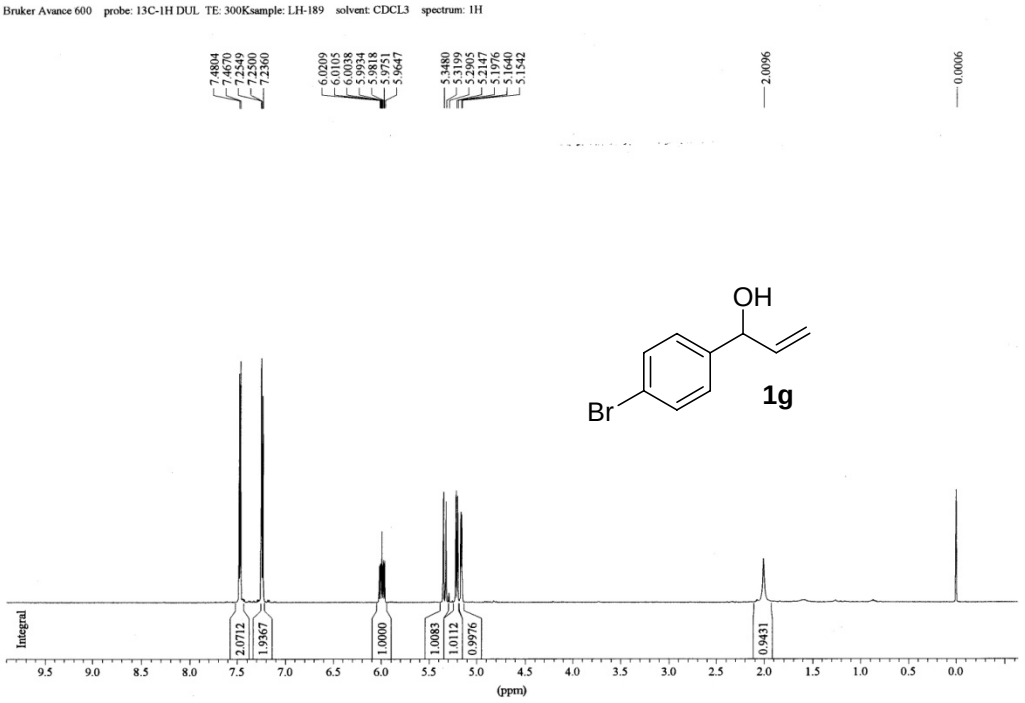
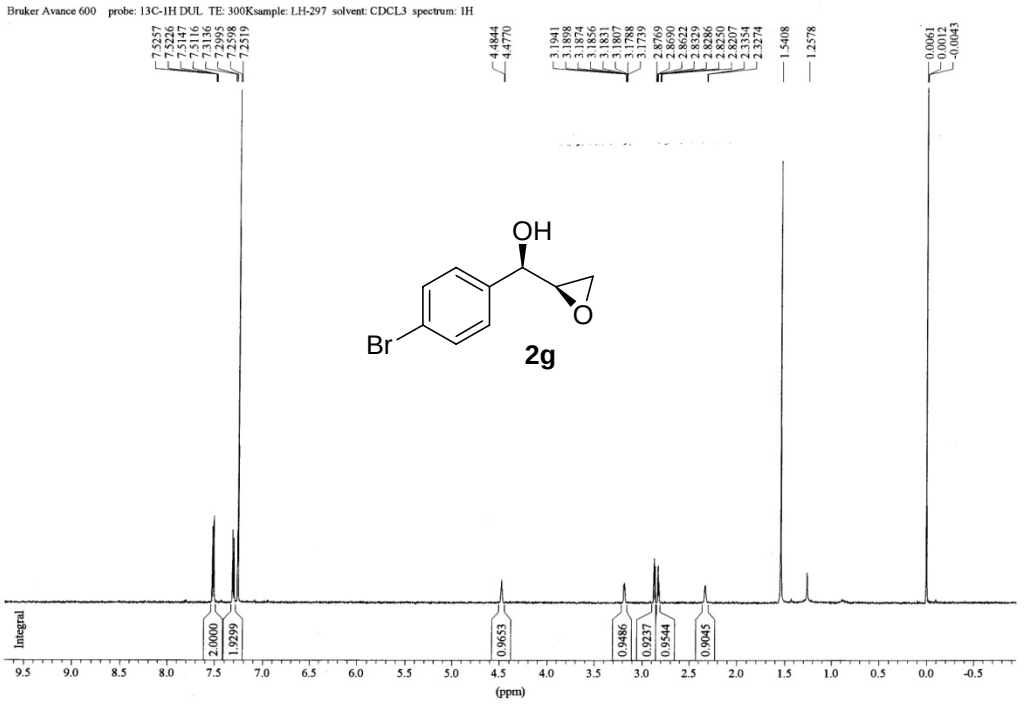
1.0000
1.0088
0.9902
1.0112
0.9715
(ppm)

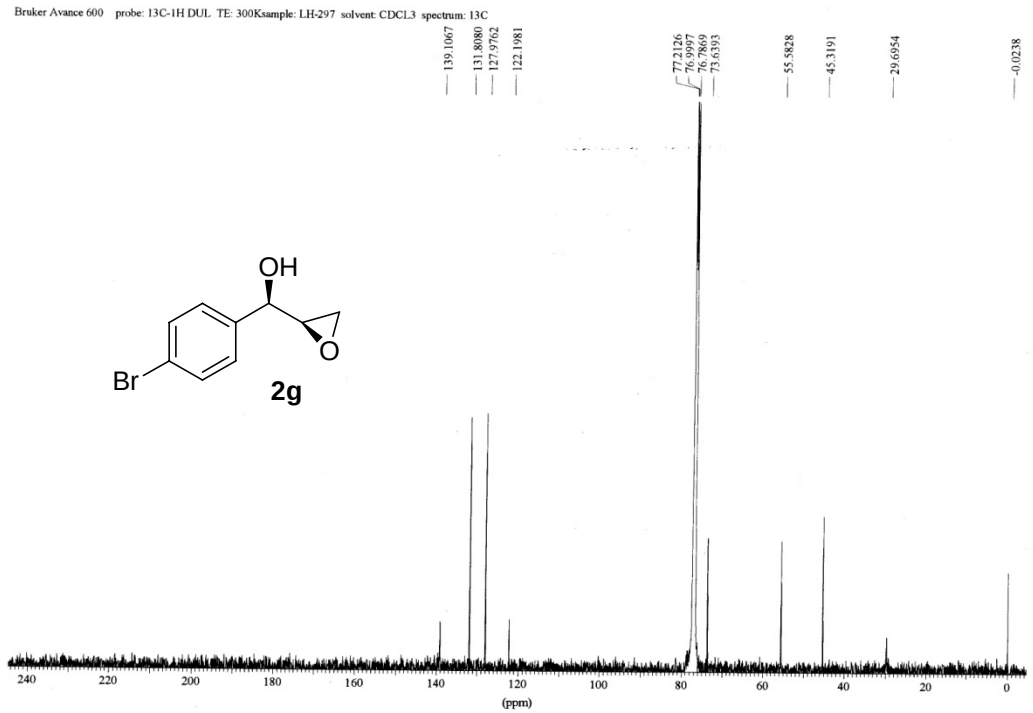

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-328 solvent: CDCL3 spectrum: 13C
138.5821
134.0584
128.8429
127.6645
77.2278
77.0225
76.8097
73.6621
55.7272
45.3647
OH
O
Cl
2e
240
220
200
180
160
140
120
100
80
60
40
20
0
(ppm)

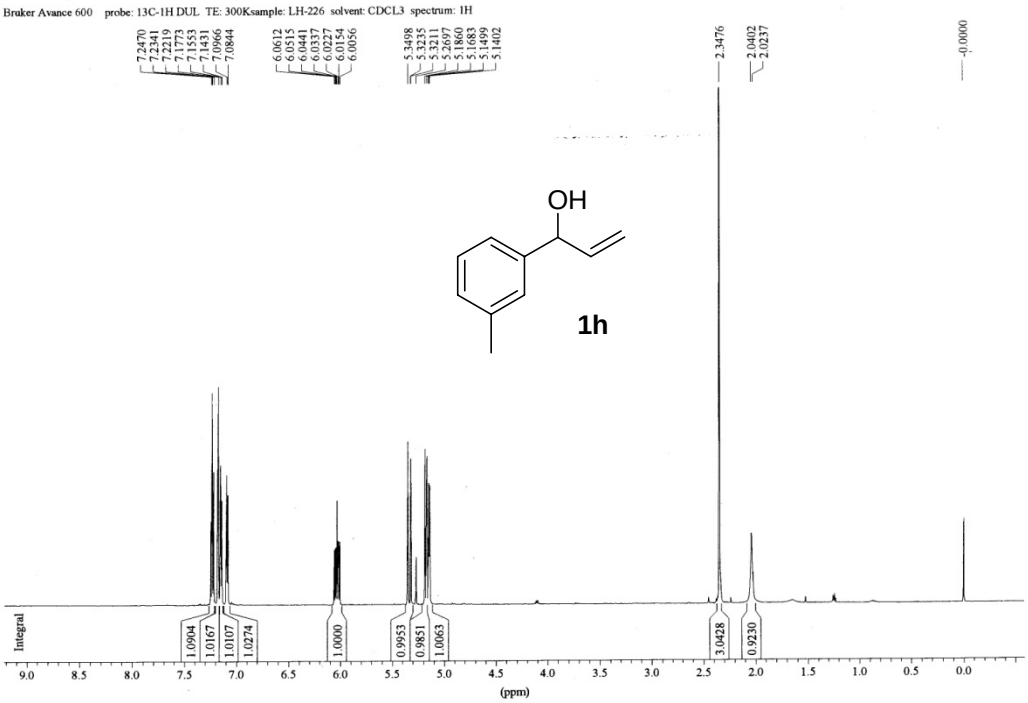

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-258 solvent: CDCL3 spectrum: 1H
7.5898
7.5871
7.5663
7.4715
7.4604
7.4585
7.3548
7.3422
7.3321
7.3191
7.2701
7.2582
7.2500
7.2440
4.9122
4.9074
4.4743
3.2235
3.2172
3.2094
3.2045
3.2027
3.1979
3.1960
3.1938
3.1893
2.9332
2.8904
2.8837
2.8826
2.8756
2.8473
2.8429
2.8391
2.8347
2.7763
2.7696
2.7614
2.3618
2.3448
1.4337
1.4244
1.2567
0.0051
-0.0001
-0.0042
-0.0057
OH
O
Br
2f
Integral
0.9971
0.1686
1.1783
1.0318
0.1716
2.7639
0.1530
1.0000
1.1324
0.1497
0.9954
1.0019
0.8022
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
(ppm)

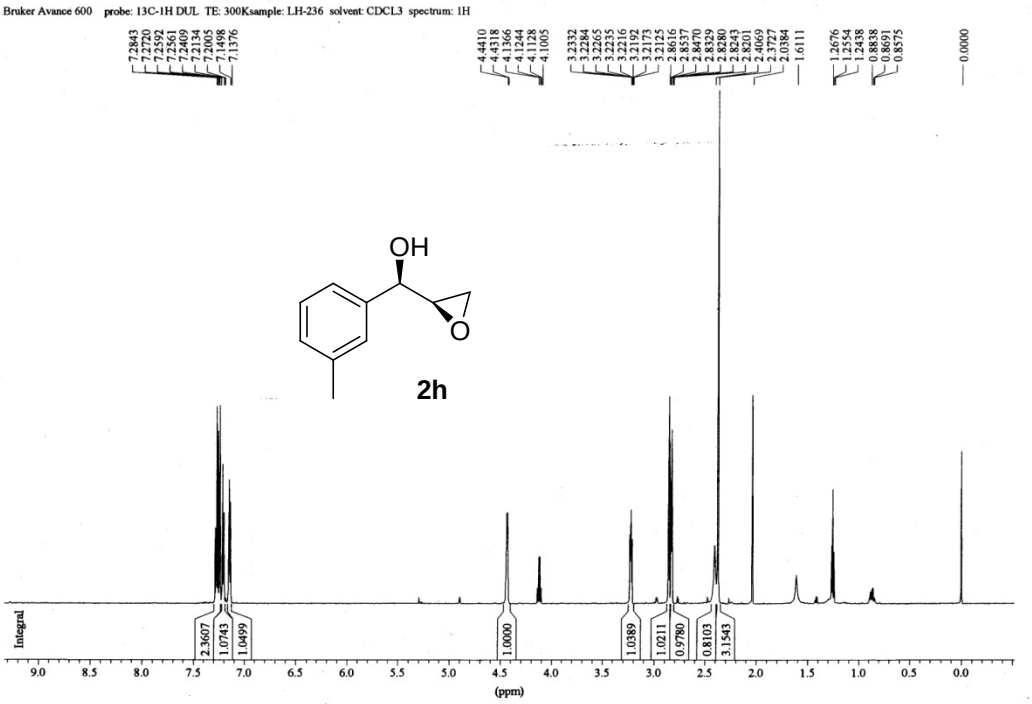

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-331 solvent: CDCL3 spectrum: 13C
142.3835
131.3290
130.2495
129.4131
124.8439
122.8216
77.3126
76.9997
76.7869
73.5861
55.6132
45.3723
29.6954
-0.0238
OH
O
Br
2f
240
220
200
180
160
140
120
100
80
60
40
20
0
(ppm)

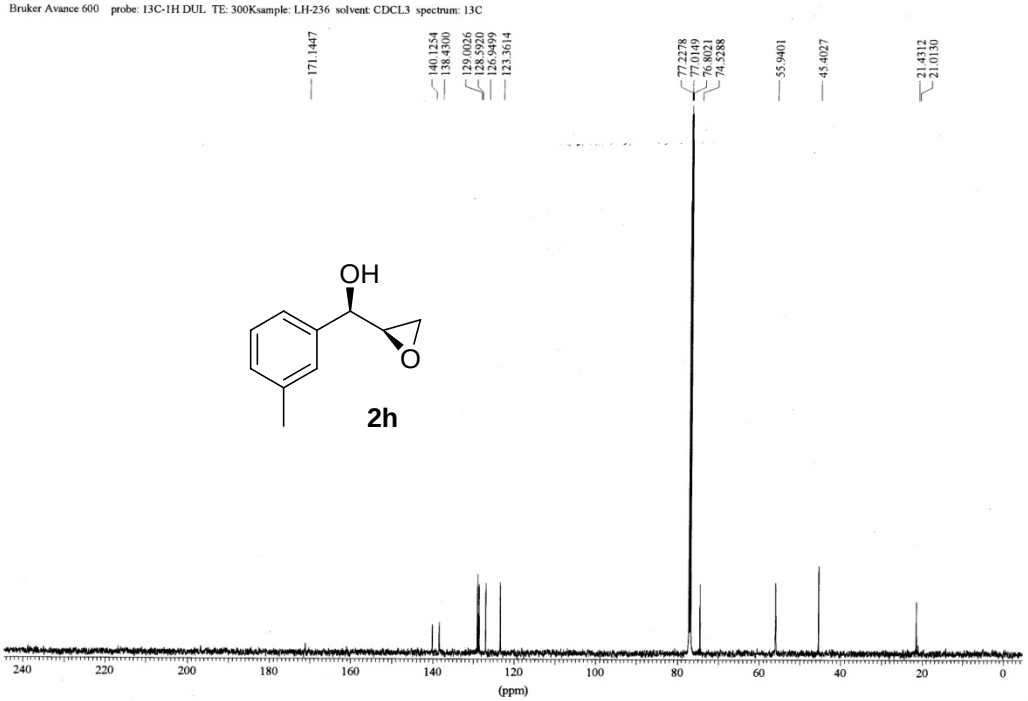

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-189 solvent: CDCL3 spectrum: 1H
7.4804
7.4670
7.2549
7.2500
7.2360
6.0209
6.0105
6.0038
5.9934
5.9818
5.9751
5.9647
5.3480
5.3199
5.2905
5.2147
5.1976
5.1640
5.1542
2.0096
0.0006
OH
Br
1g
Integral
2.0712
1.9367
1.0000
1.0083
1.0112
0.9976
0.9431
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
(ppm)

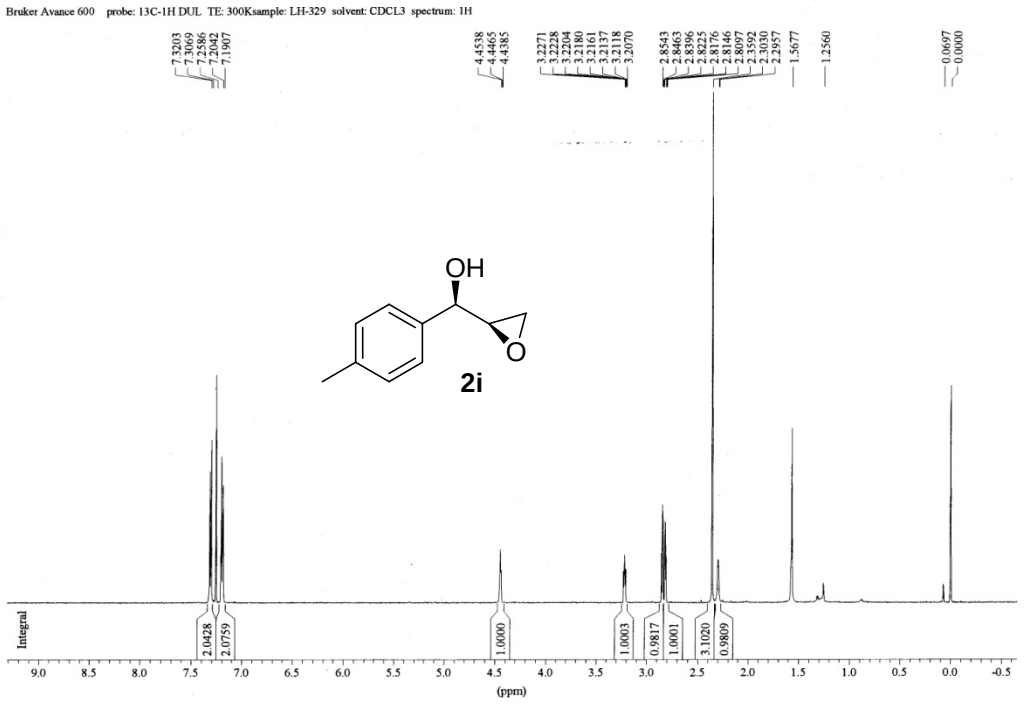

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-297 solvent: CDCL3 spectrum: 1H
OH
O
Br
2g
Integral
(ppm)

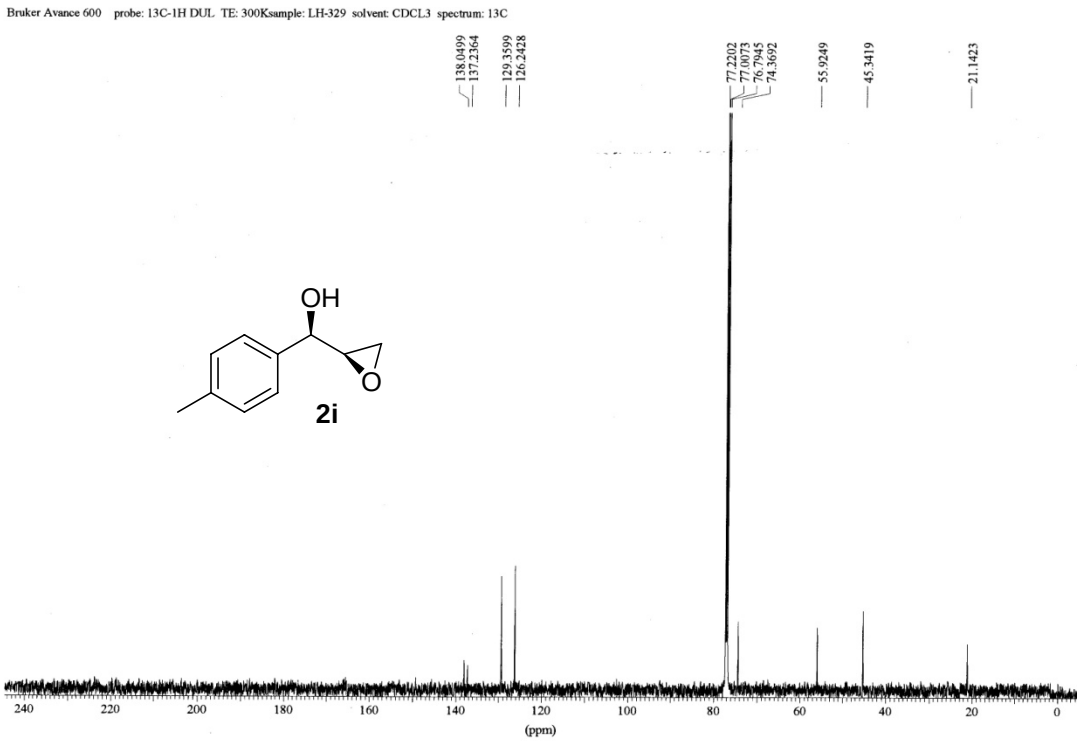

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-297 solvent: CDCL3 spectrum: 13C
OH
O
Br
2g
(ppm)

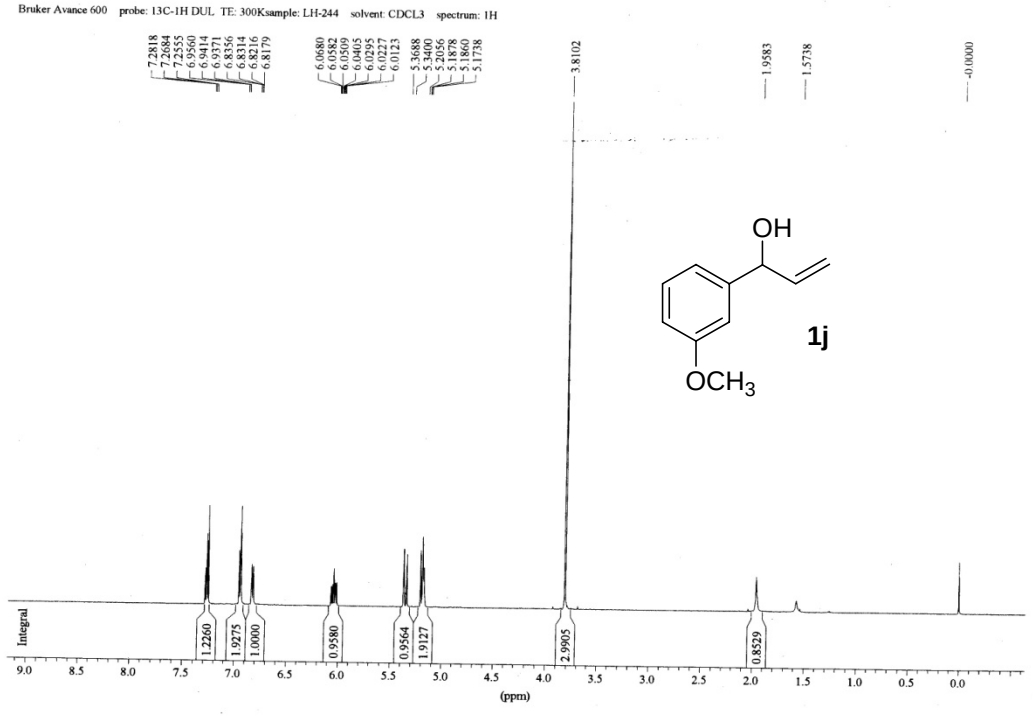

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-226 solvent: CDCL3 spectrum: 1H
OH
1h
Integral
(ppm)

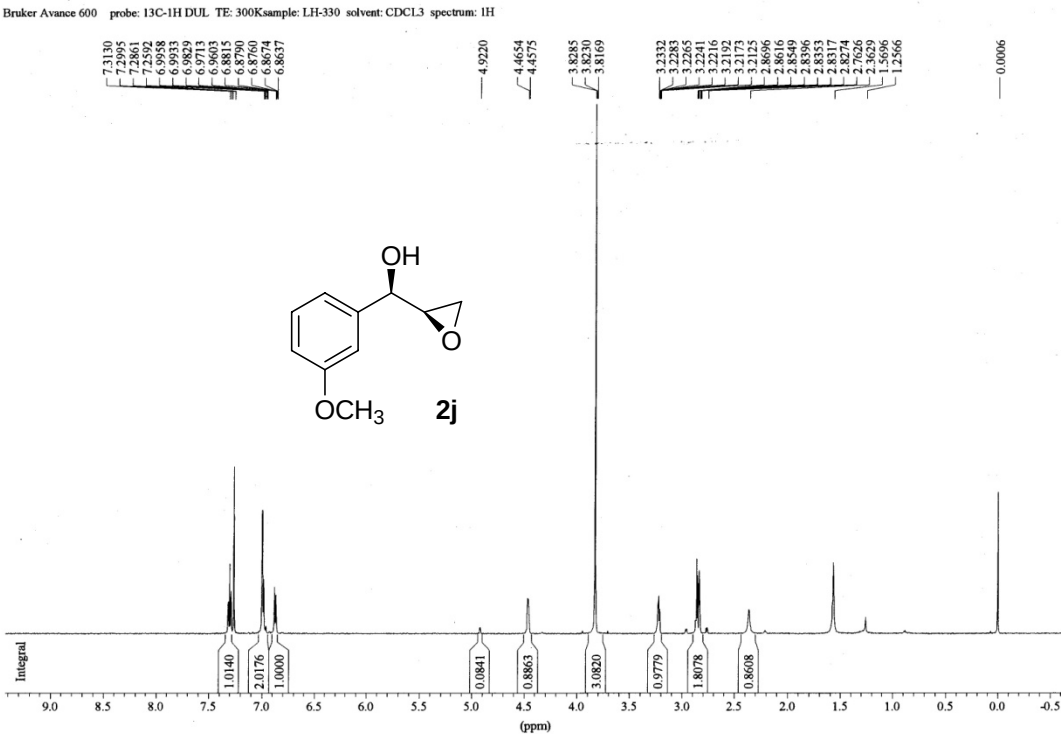

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-236 solvent: CDCL3 spectrum: 1H
OH
O
2h
Integral
(ppm)

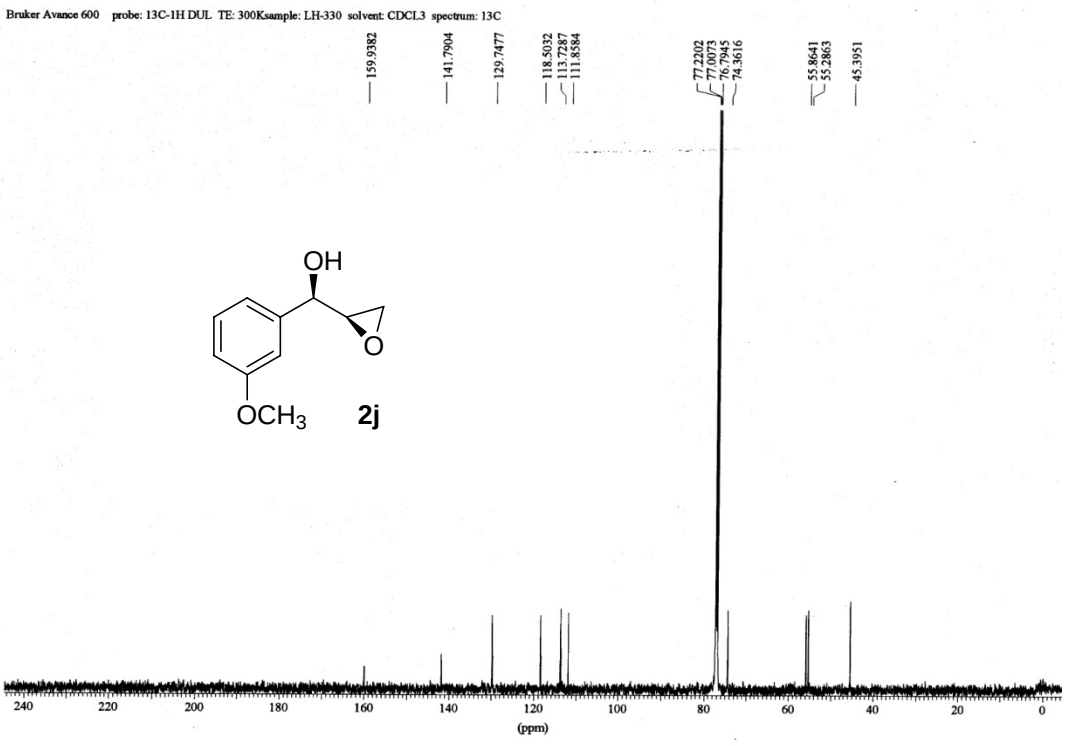

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-236 solvent: CDCL3 spectrum: 13C
171.1447
140.1254
138.4300
129.0026
128.5920
126.9499
123.3614
77.2278
77.0149
76.8021
74.5288
55.9401
45.4027
21.4312
21.0130
OH
O
2h
240
220
200
180
160
140
120
100
80
60
40
20
0
(ppm)

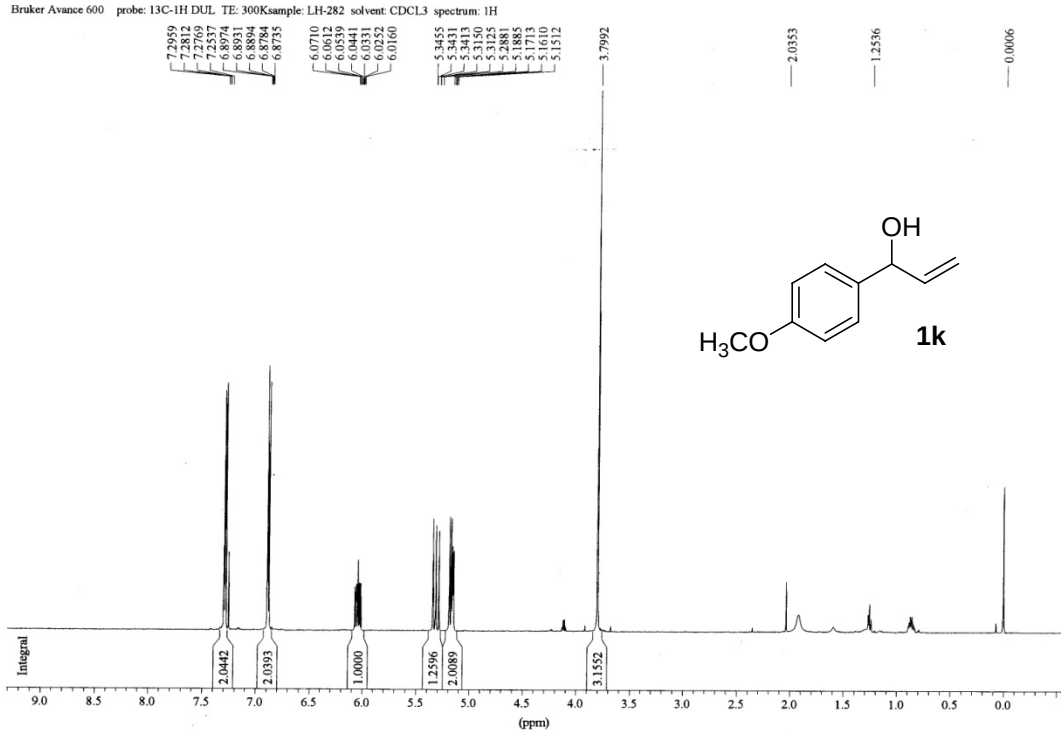

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-329 solvent: CDCL3 spectrum: 1H
7.3203
7.3069
7.2586
7.2042
7.1907
4.4538
4.4465
4.4385
3.2271
3.2228
3.2204
3.2180
3.2161
3.2137
3.2118
3.2070
2.8543
2.8463
2.8396
2.8225
2.8176
2.8146
2.8097
2.3592
2.3030
2.2957
1.5677
1.2560
0.0697
0.0000
OH
O
2i
Integral
2.0428
2.0759
1.0000
1.0003
0.9817
1.0001
3.1020
0.9809
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
(ppm)

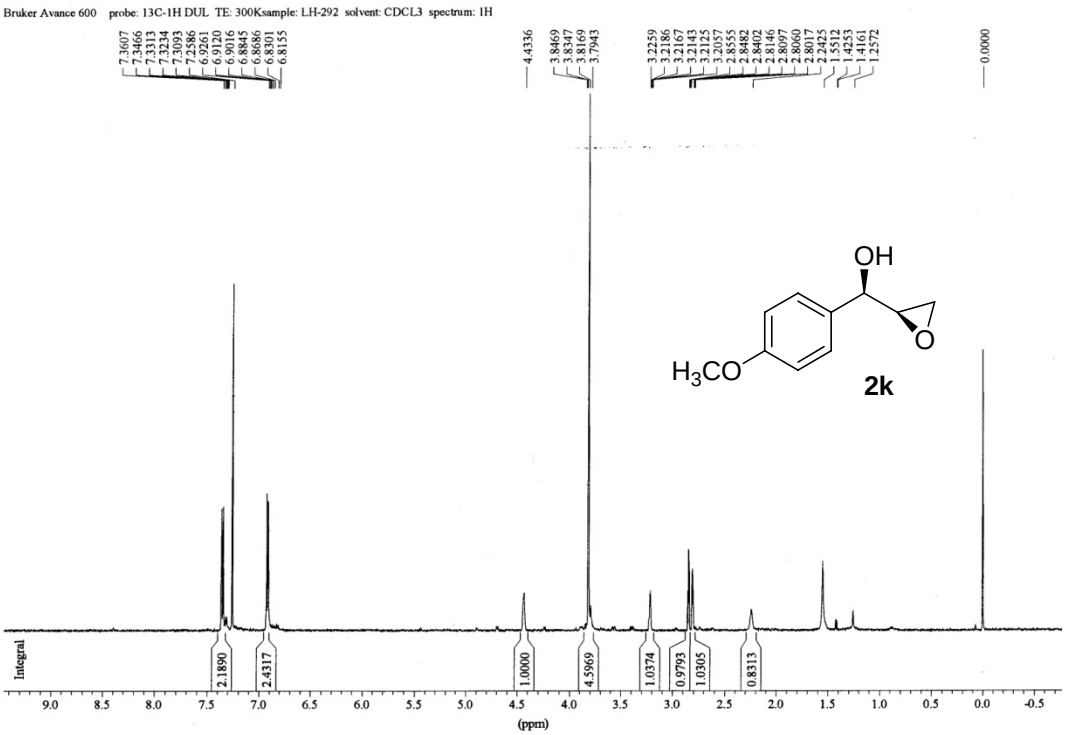

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-329 solvent: CDCL3 spectrum: 13C
138.0499
137.2364
129.3599
126.2428
77.2202
77.0073
76.7945
74.3692
55.9249
45.3419
21.1423
OH
O
2i
240
220
200
180
160
140
120
100
80
60
40
20
0
(ppm)

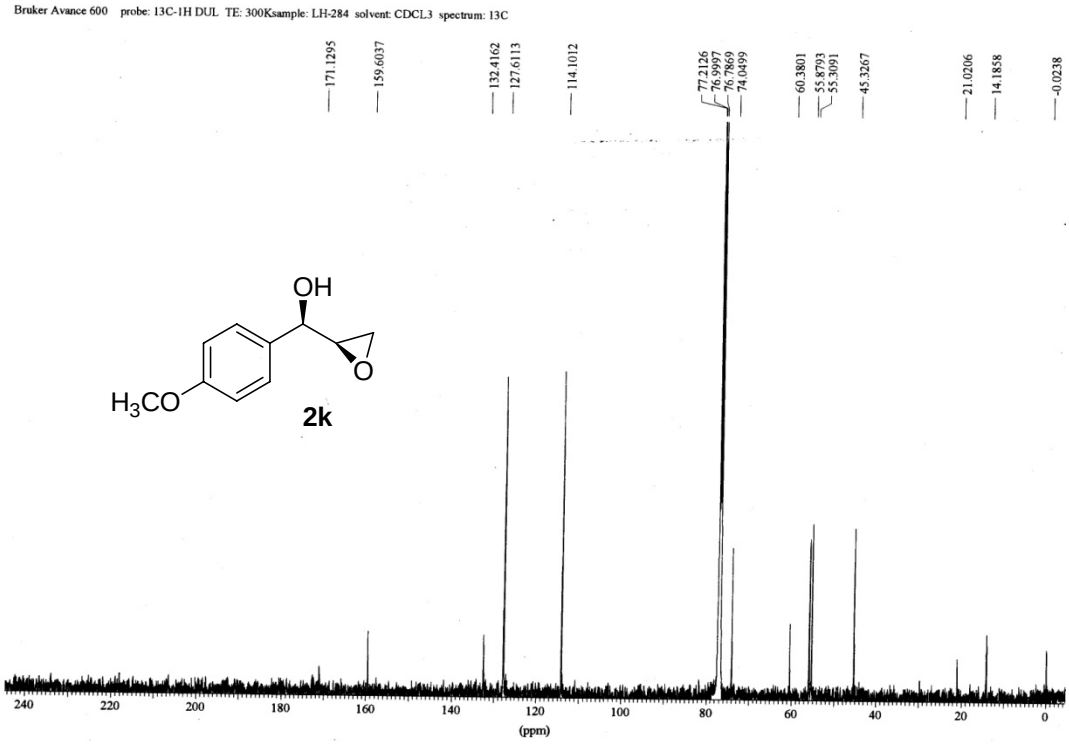

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-244 solvent: CDCL3 spectrum: 1H
7.2818
7.2684
7.2555
6.9560
6.9414
6.9371
6.8356
6.8314
6.8216
6.8179
6.0680
6.0582
6.0499
6.0405
6.0295
6.0227
6.0123
5.3688
5.3490
5.2056
5.1878
5.1860
5.1738
3.8102
1.9583
1.5738
-0.0000
OH
1j
OCH3
Integral
1.2260
1.9275
1.0000
0.9580
0.9564
1.9127
2.9905
0.8529
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
(ppm)

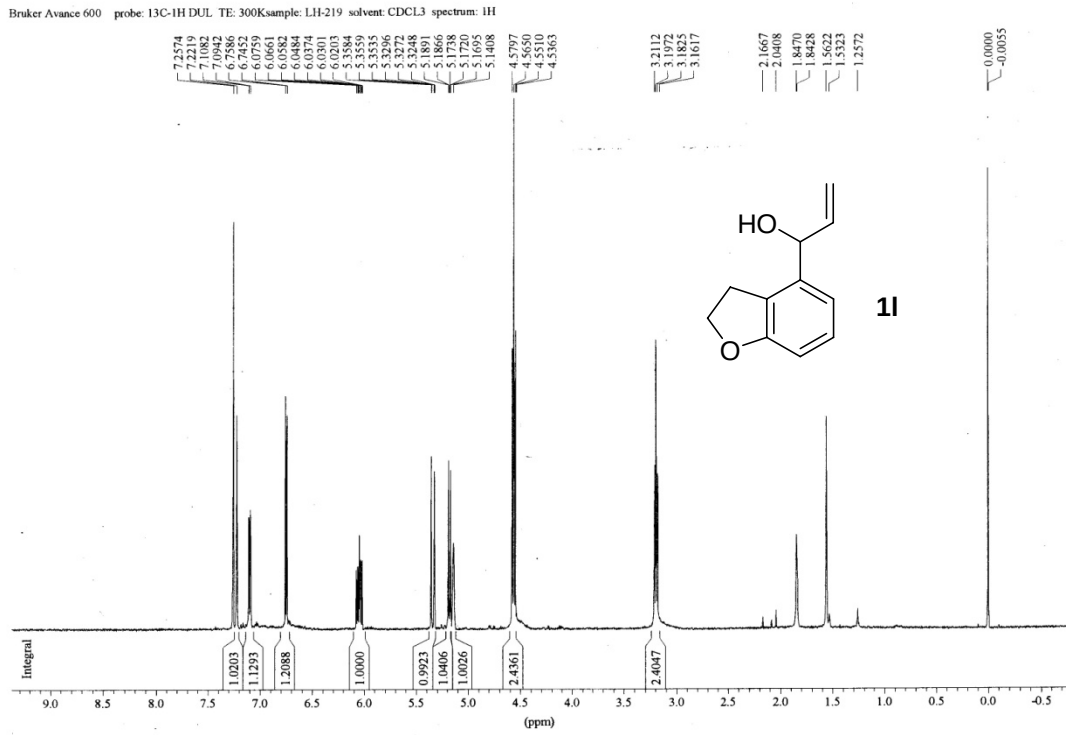

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-330 solvent: CDCL3 spectrum: 1H
OH
O
OCH3
2j
Integral
(ppm)

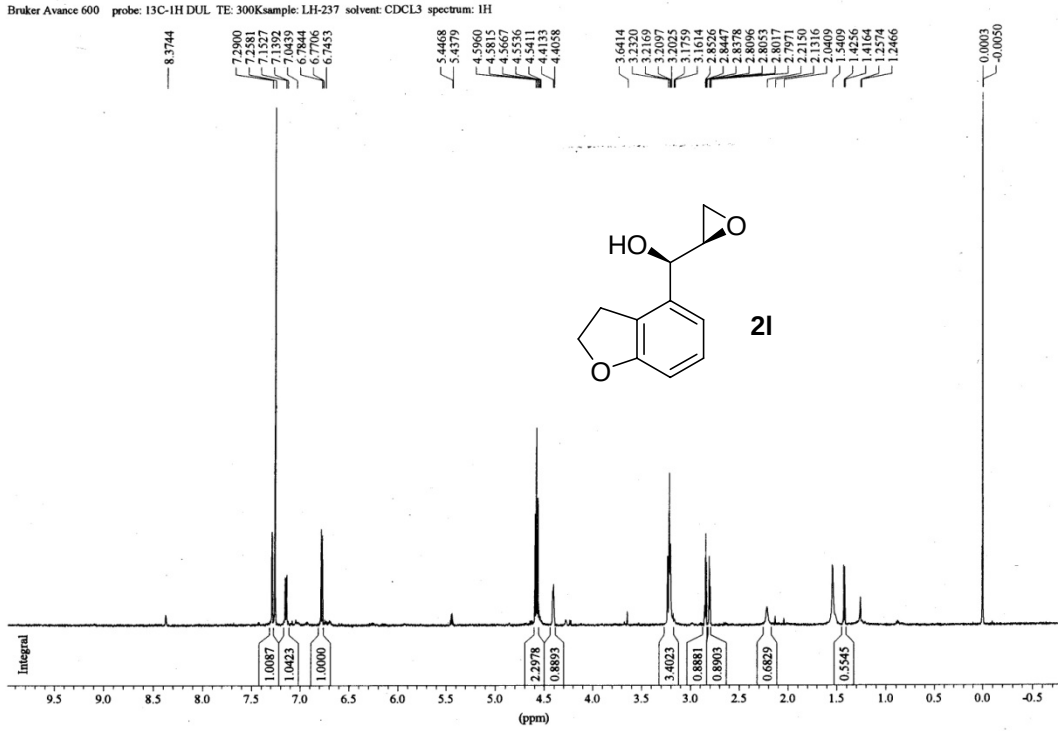

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-330 solvent: CDCL3 spectrum: 13C
OH
O
OCH3
2j
(ppm)

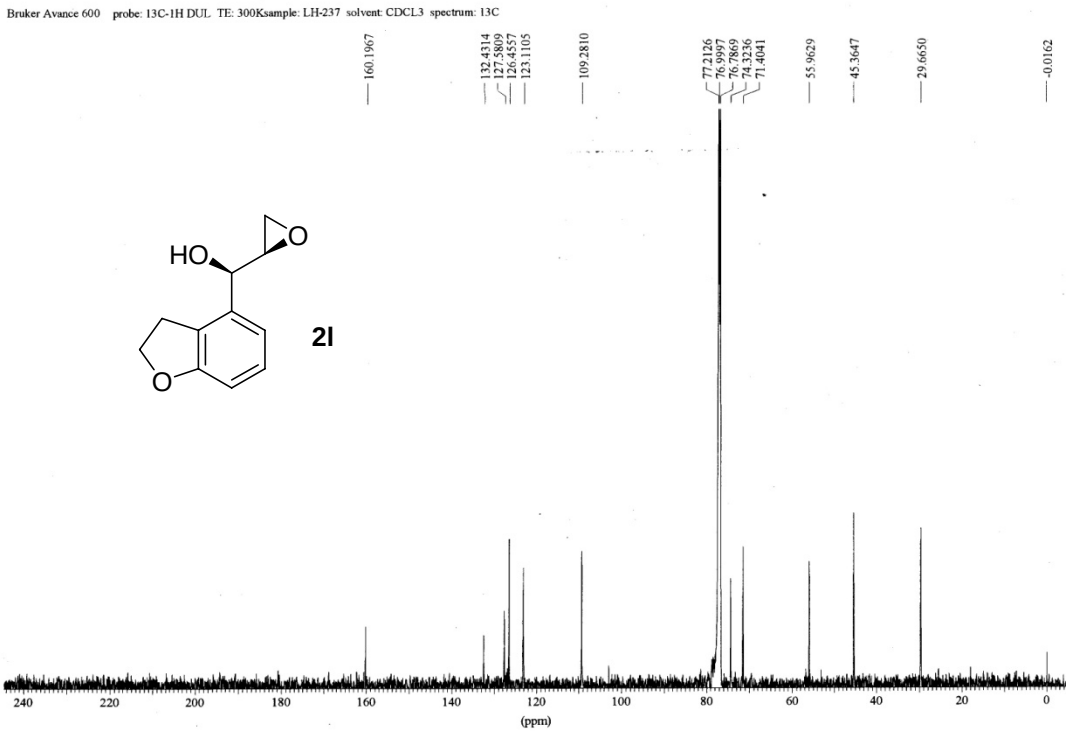

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-282 solvent: CDCL3 spectrum: 1H
OH
H3CO
1k
Integral
(ppm)

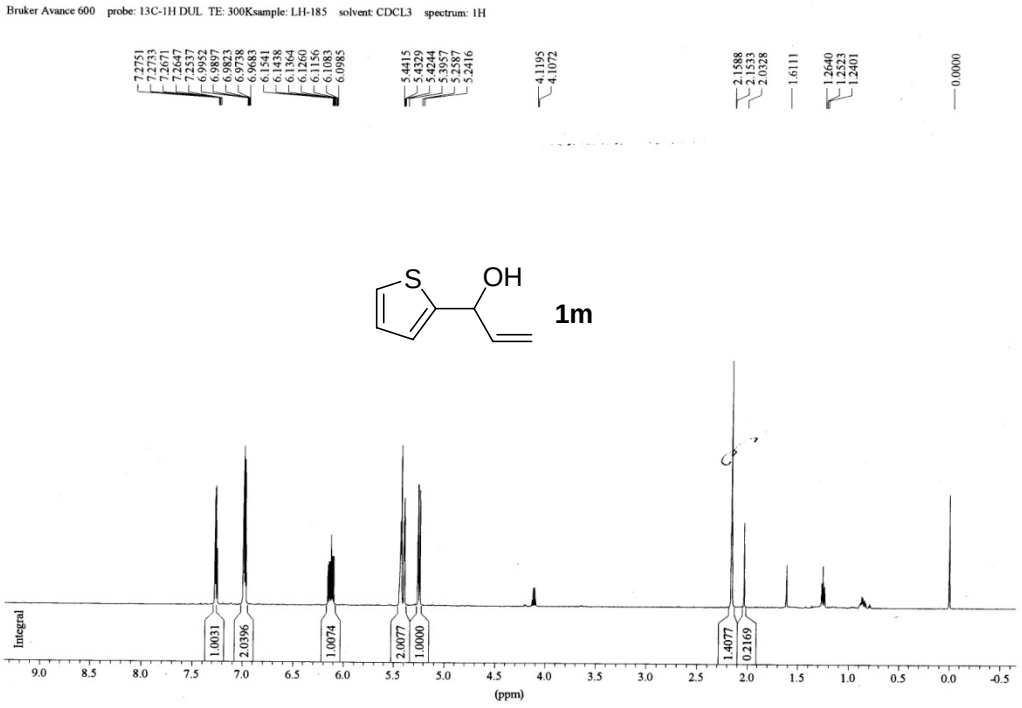

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-292 solvent: CDCL3 spectrum: 1H
OH
O
H3CO
2k
Integral
(ppm)

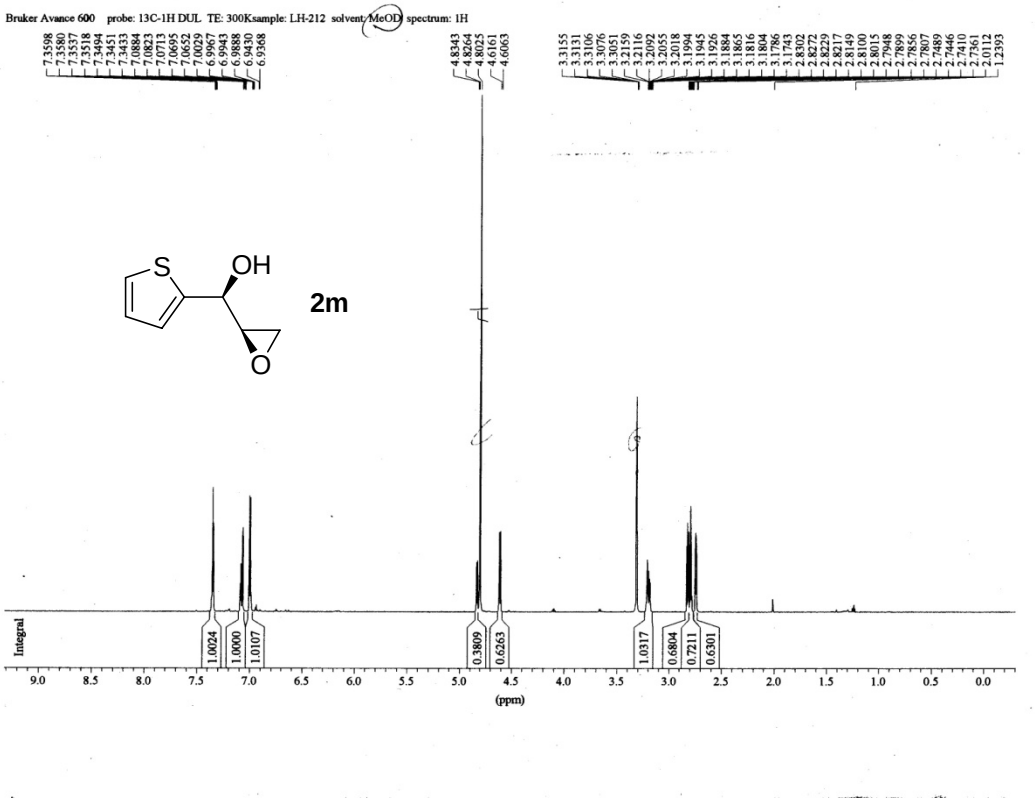

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-284 solvent: CDCL3 spectrum: 13C
171.1295
159.6037
132.4162
127.6113
114.1012
77.2126
76.9997
76.7869
74.0499
60.3801
55.8793
55.3091
45.3267
21.0206
14.1858
-0.0238
OH
O
H3CO
2k
240
220
200
180
160
140
120
100
80
60
40
20
0
(ppm)

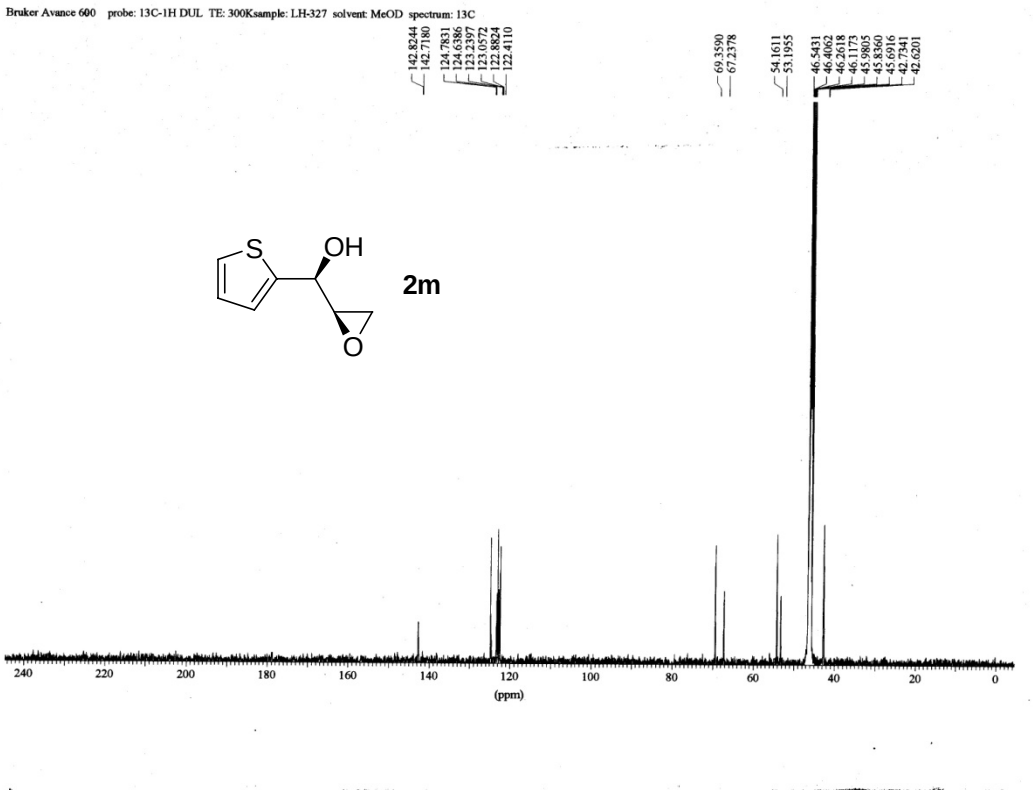

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-219 solvent: CDCL3 spectrum: 1H
7.2574
7.2219
7.1082
7.0942
6.7586
6.7452
6.0759
6.0661
6.0582
6.0484
6.0374
6.0301
6.0203
5.3584
5.3559
5.3535
5.3296
5.3272
5.3248
5.1891
5.1866
5.1738
5.1720
5.1695
5.1408
4.5797
4.5650
4.5510
4.5363
3.2112
3.1972
3.1825
3.1617
2.1667
2.0408
1.8470
1.8428
1.5622
1.5323
1.2572
0.0000
-0.0055
HO
O
1l
Integral
1.0203
1.1293
1.2088
1.0000
0.9923
1.0406
1.0026
2.4361
2.4047
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
(ppm)

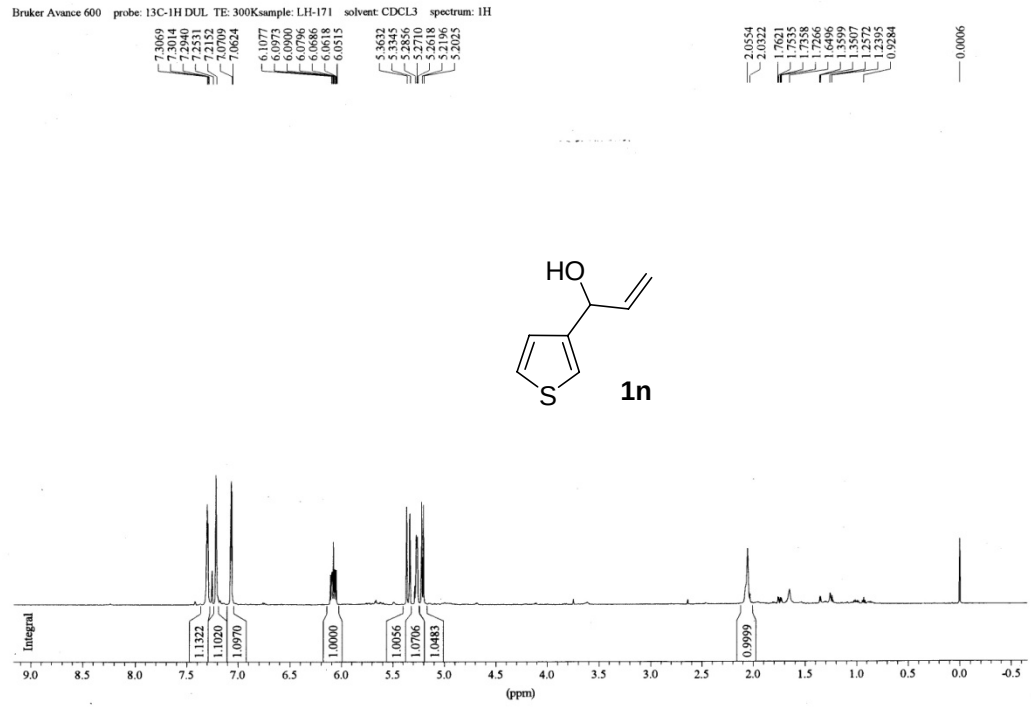

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-237 solvent: CDCL3 spectrum: 1H
HO
O
O
2l
Integral
1.0087
1.0423
1.0000
2.2978
0.8893
3.4023
0.8881
0.8903
0.6829
0.5545
(ppm)

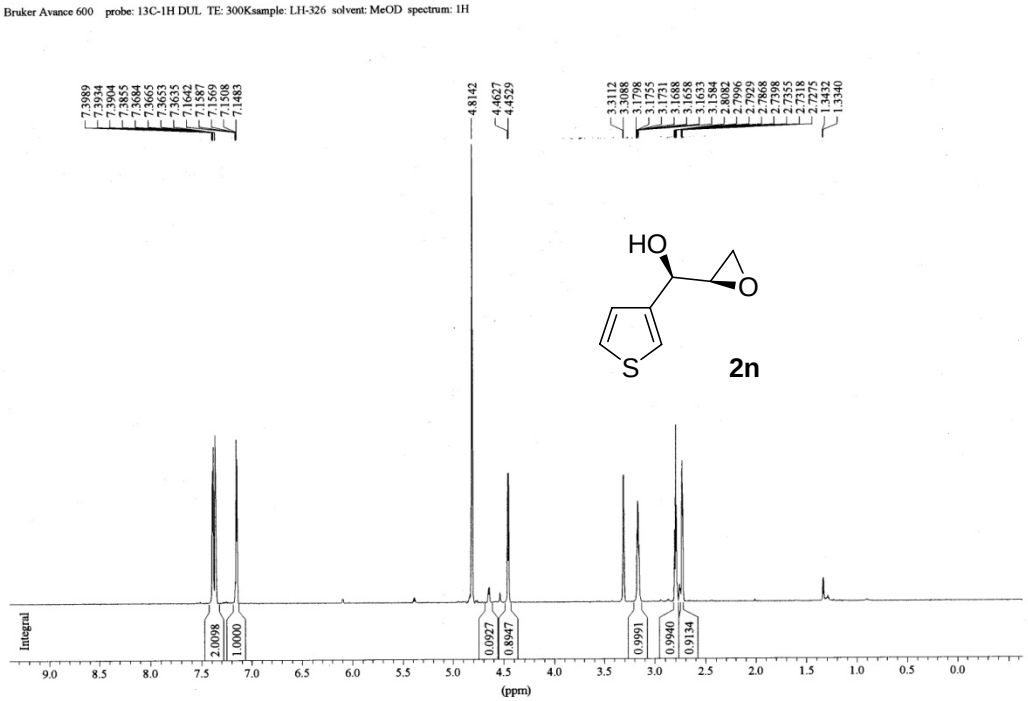

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-237 solvent: CDCL3 spectrum: 13C
160.1967
132.4314
127.5809
126.4557
123.1105
109.2810
77.2126
76.9997
76.7869
74.3236
71.4041
55.9629
45.3647
29.6650
-0.0162
HO
O
O
2l
(ppm)

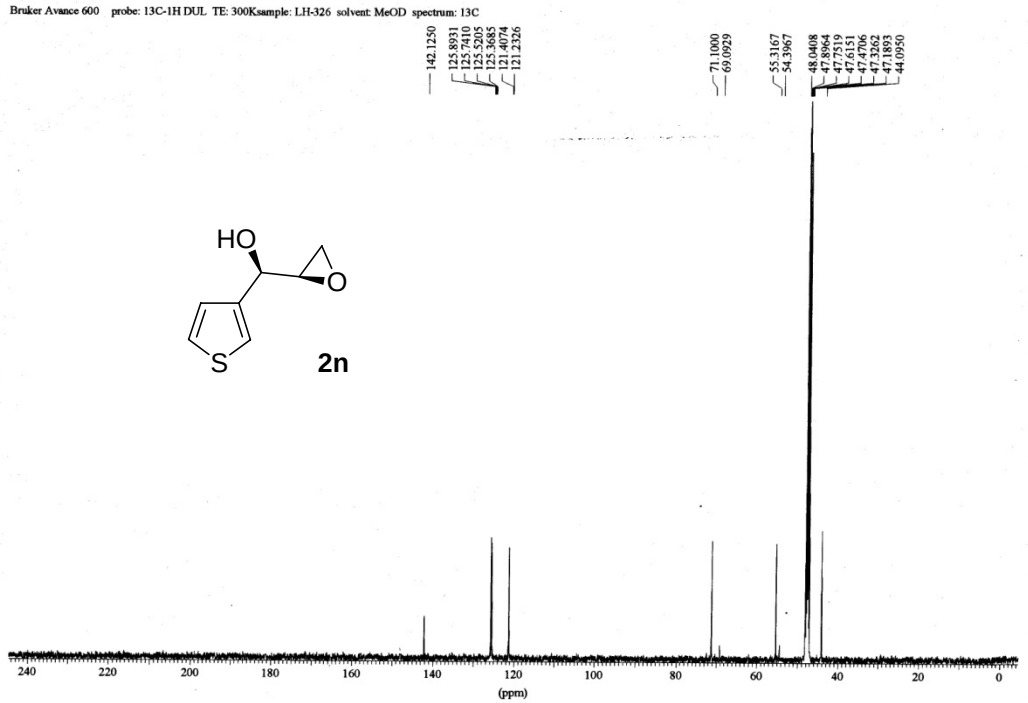

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-185 solvent: CDCL3 spectrum: 1H
7.2751
7.2733
7.2671
7.2647
7.2557
7.0952
6.9857
6.9823
6.9738
6.9683
6.1541
6.1438
6.1364
6.1260
6.1156
6.1053
6.0985
5.4415
5.4329
5.4244
5.3957
5.2587
5.2416
4.1195
4.1072
2.1588
2.1533
2.0328
1.6111
1.2640
1.2523
1.2401
0.0000
S
OH
1m
Integral
1.0031
2.0396
1.0074
2.0077
1.0000
1.4077
0.2169
(ppm)

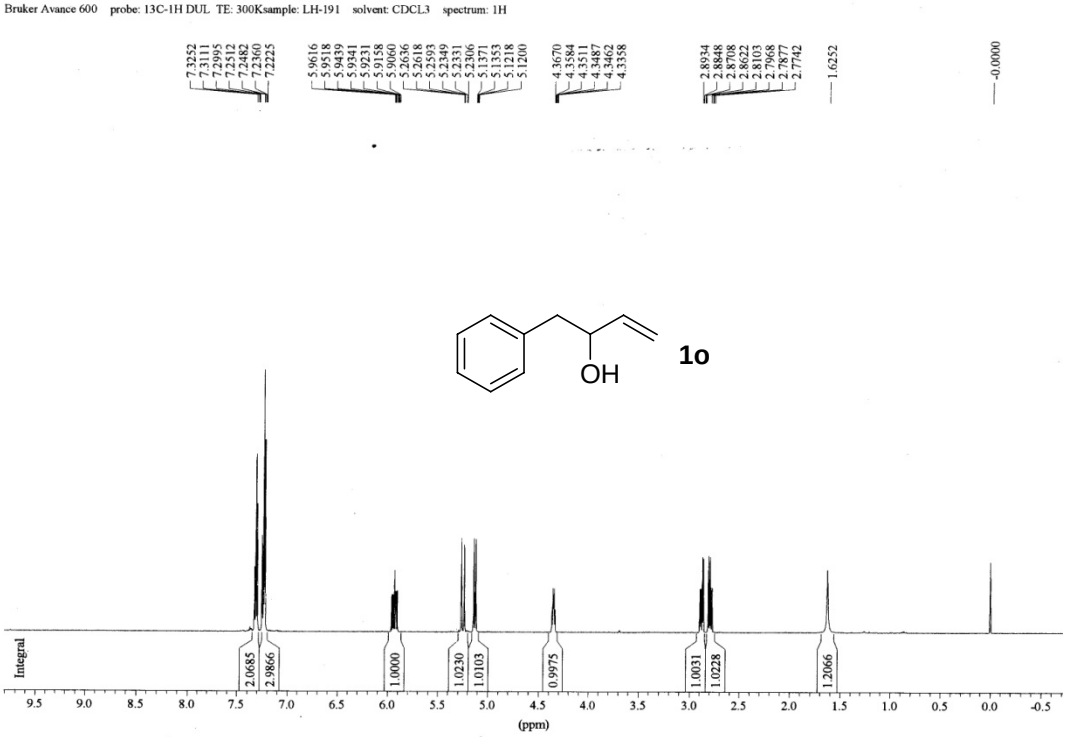

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-212 solvent: MeOD spectrum: 1H
7.3598
7.3580
7.3557
7.3518
7.3494
7.3451
7.3433
7.0884
7.0823
7.0713
7.0695
7.0652
7.0609
6.9967
6.9943
6.9888
6.9430
6.9368
4.8343
4.8264
4.8025
4.6161
4.6063
3.3155
3.3131
3.3106
3.3076
3.3051
3.2159
3.2116
3.2092
3.2055
3.2018
3.1994
3.1945
3.1926
3.1884
3.1865
3.1816
3.1804
3.1786
3.1743
3.1702
2.8272
2.8229
2.8217
2.8149
2.8100
2.8015
2.7948
2.7899
2.7855
2.7807
2.7489
2.7446
2.7410
2.7361
2.0112
1.2393
S
OH
O
2m
Integral
1.0024
1.0000
1.0107
0.3809
0.6263
1.0317
0.6804
0.7211
0.6301
(ppm)

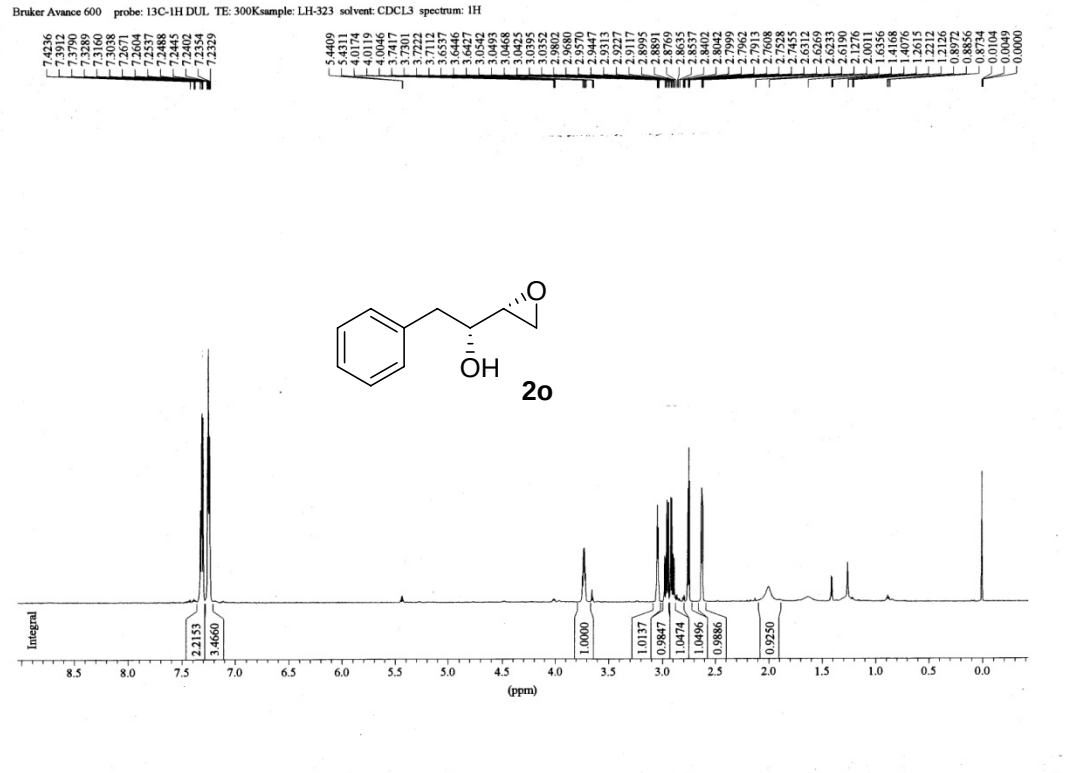

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-327 solvent: MeOD spectrum: 13C
142.8244
142.7180
124.7831
124.6386
123.2397
123.0572
122.8824
122.4110
69.3590
67.2378
54.1611
53.1955
46.5431
46.4062
46.2618
46.1173
45.9805
45.8360
45.6916
42.7341
42.6201
S
OH
O
2m
240
220
200
180
160
140
120
100
80
60
40
20
0
(ppm)

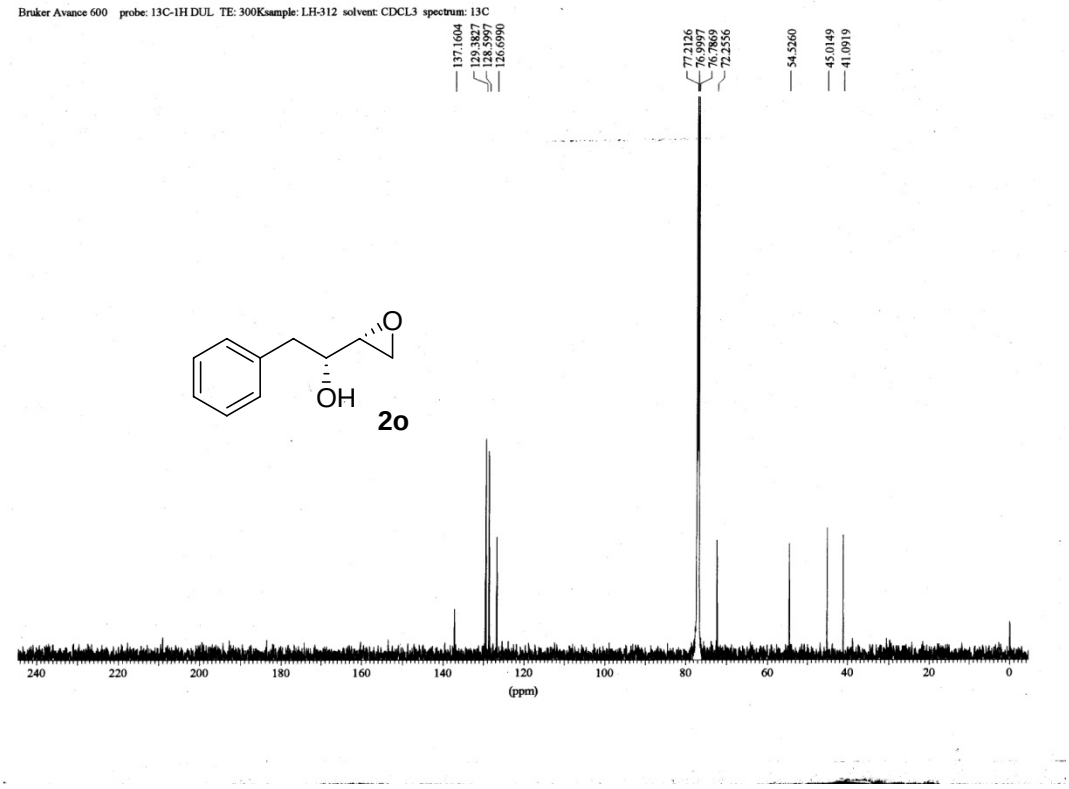

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-171 solvent: CDCL3 spectrum: 1H
7.3069
7.3014
7.2940
7.2513
7.2152
7.0709
7.0624
6.1077
6.0973
6.0900
6.0796
6.0686
6.0618
6.0515
5.3632
5.3345
5.2856
5.2710
5.2618
5.2196
5.2025
2.0554
2.0322
1.7621
1.7535
1.7358
1.7266
1.6496
1.6359
1.3597
1.2572
1.2395
0.9284
0.0006
HO
S
1n
Integral
1.1322
1.1020
1.0970
1.0000
1.0056
1.0706
1.0483
0.9999
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
(ppm)

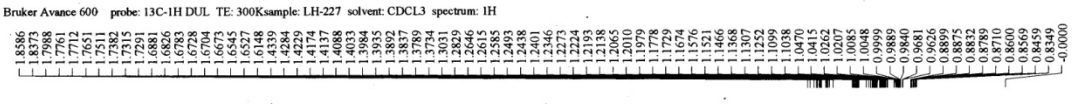

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-326 solvent: MeOD spectrum: 1H
HO
O
S
2n
Integral
2.0098
1.0000
0.0927
0.8947
0.9991
0.9940
0.9134
(ppm)

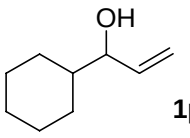

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-326 solvent: MeOD spectrum: 13C
HO
O
S
2n
(ppm)

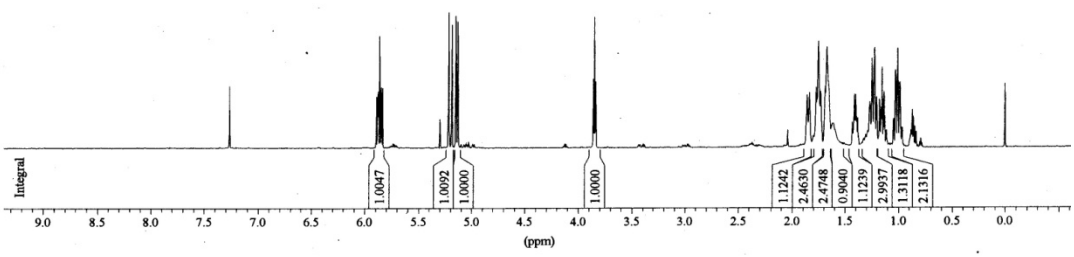

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-191 solvent: CDCL3 spectrum: 1H
OH
1o
Integral
2.0685
2.9866
1.0000
1.0230
1.0103
0.9975
1.0031
1.0228
1.2066
(ppm)

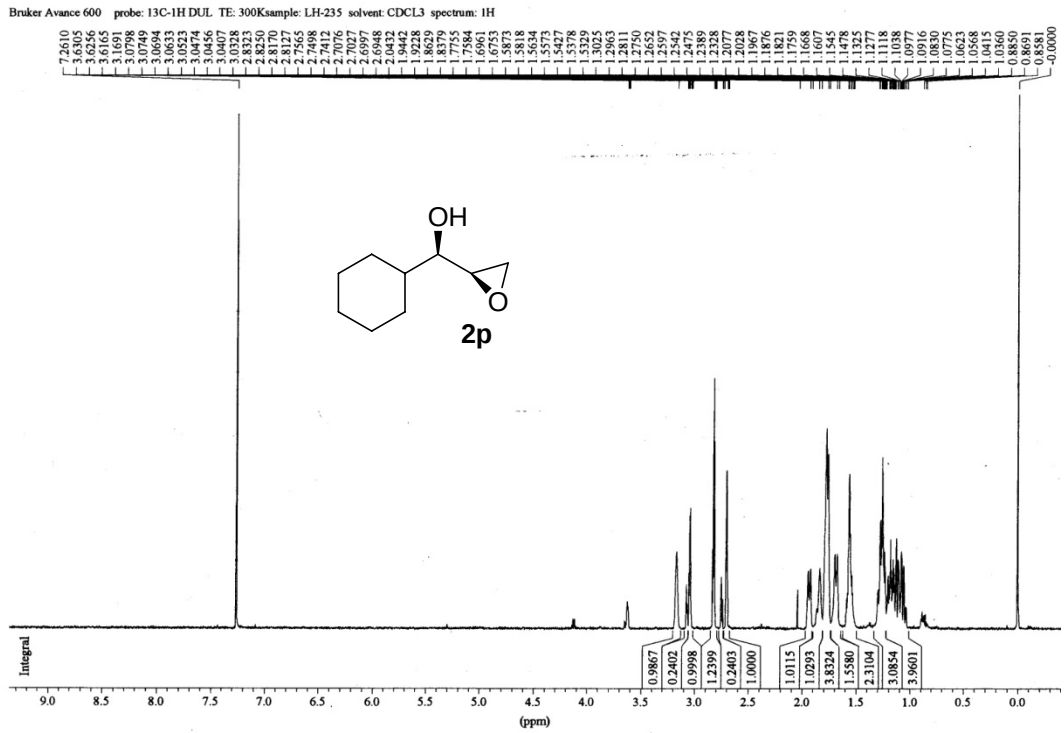

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-323 solvent: CDCL3 spectrum: 1H
O
OH
2o
Integral
2.2153
3.4660
1.0000
1.0137
0.9847
1.0474
1.0496
0.9886
0.9250
(ppm)

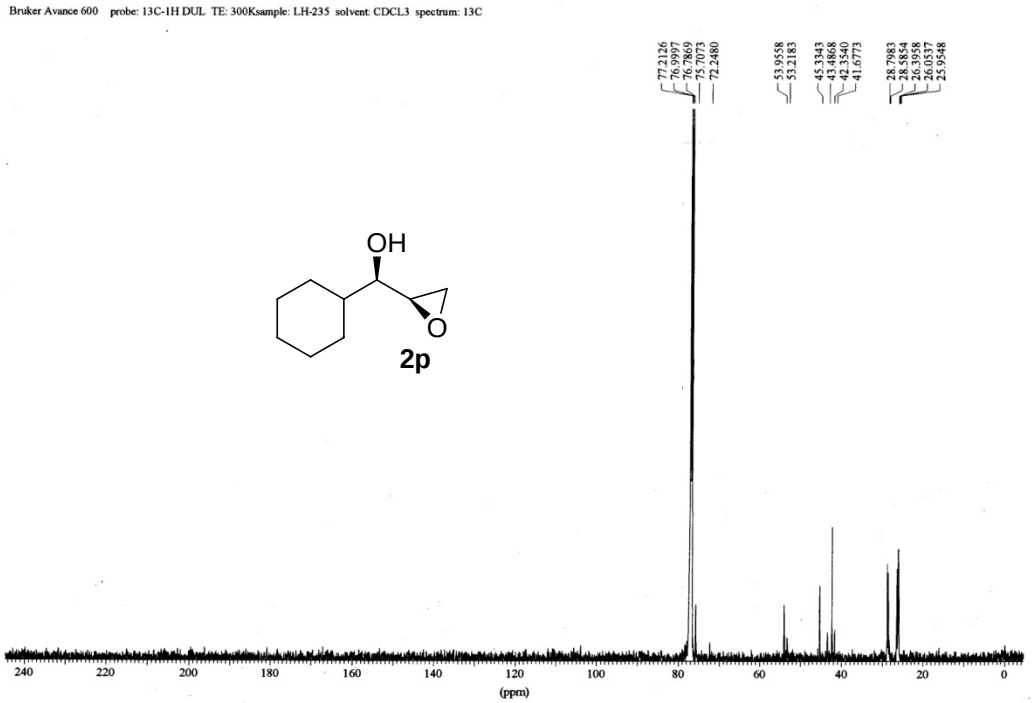

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-312 solvent: CDCL3 spectrum: 13C
137.1604
129.3827
128.5997
126.6990
77.2126
76.9997
76.7869
72.2356
54.5260
45.0149
41.0919
O
OH
2o
240
220
200
180
160
140
120
100
80
60
40
20
0
(ppm)


Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-227 solvent: CDCL3 spectrum: 1H

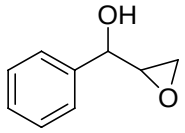

OH
1p


Integral
1.0047
1.0092
1.0000
1.0000
1.1242
2.4630
2.4748
0.9040
1.1239
2.9937
1.3118
2.1316
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
(ppm)


Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-235 solvent: CDCL3 spectrum: 1H
OH
O
2p
Integral
0.9867
0.2402
0.9998
1.2399
0.2403
1.0000
1.0115
1.0293
3.8324
1.5580
2.3104
3.0854
3.9601
(ppm)

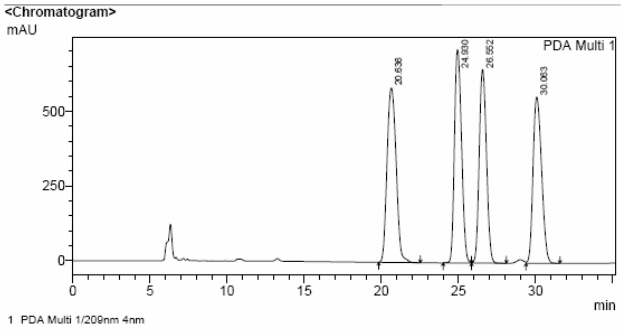

Bruker Avance 600 probe: 13C-1H DUL TE: 300Ksample: LH-235 solvent: CDCL3 spectrum: 13C
77.2126
76.9997
76.7869
75.7073
72.2480
53.9558
53.2183
45.3343
43.4868
42.3540
41.6773
28.7983
28.5854
26.3958
26.0537
25.9548
OH
O
2p
(ppm)

Chiral HPLC analysis of 2a ee value



==== Wu Group's Shimadzu LCsolution Analysis Report ====

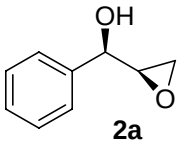
D:\...\LH\epoxidation\手性分析\epoxidation of unconjuncted olefins\oxiran-2-yl(phenyl)methanol-2.lcd
Acquired by : Admin
Sample Name : oxiran-2-yl(phenyl)methanol
Injection Volume : 5 uL
Data File Name : oxiran-2-yl(phenyl)methanol-2.lcd
Method File Name : B-90.lcm
Report File Name : 报告格式.lcr
Data Acquired : 2009-12-5 14:24:52
Description : oxiran-2-yl(phenyl)methanol
AS-H (4.6*250), 0.5 ml/min; 90% n-Hexane; oven 35; 1.9 MPa



PeakTable

PDA Ch1 209nm 4nm

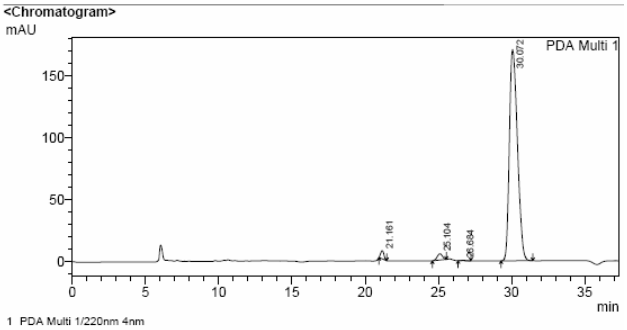
Peak#	Ret. Time	Area	Height	Area %
1	20.636	23923668	583302	26.797
2	24.930	22832664	713928	25.575
3	26.552	21114394	647092	23.650
4	30.063	21406620	555769	23.978
Total		89277346	2500092	100.000



2a from the wild type SMO

==== Wu Group's Shimadzu LCsolution Analysis Report ====

D:\...\epoxidation\手性分析\epoxidation of unconjuncted olefins\oxiran-2-yl(phenyl)methanol-6-p.lcd
Acquired by : Admin
Sample Name : oxiran-2-yl(phenyl)methanol-6-p
Injection Volume : 5 uL
Data File Name : oxiran-2-yl(phenyl)methanol-6-p.lcd
Method File Name : B-90.lcm
Report File Name : 报告格式.lcr
Data Acquired : 2009-12-5 15:21:37
Description : oxiran-2-yl(phenyl)methanol-6-p
AS-H (4.6*250), 0.5 ml/min; 90% n-Hexane; oven 35; 1.9 MPa



PeakTable

PDA Ch1 220nm 4nm

Peak#	Ret. Time	Area	Height	Area %
1	21.161	102875	6557	1.526
2	25.104	123475	5096	1.832
3	26.684	7488	318	0.111
4	30.072	6507605	170503	96.531
Total		6741442	182475	100.000