Rhodium-Catalyzed Asymmetric Ring Opening Reaction of Oxabenzonorbornadienes with Amines Using ZnI_2 as Activator

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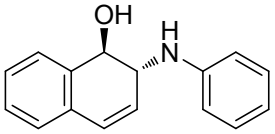
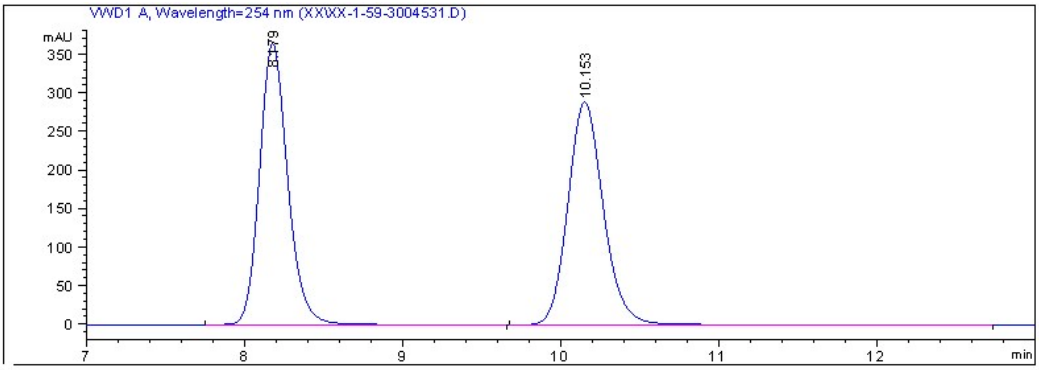
Supporting Information

Contents

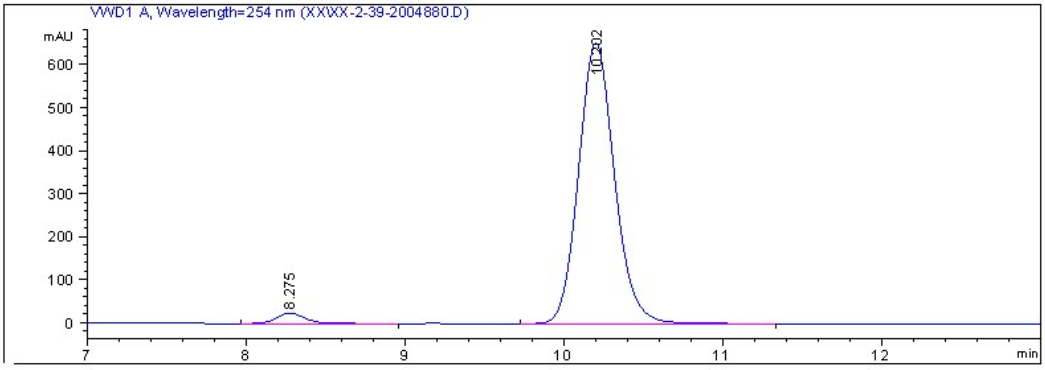
A: HPLC spectra of compounds

(*1R,2R*)-2-(phenylamino)-1,2-dihydronaphthalen-1-ol (3aa)



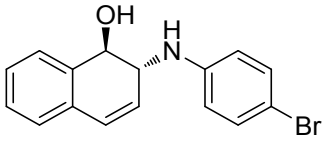


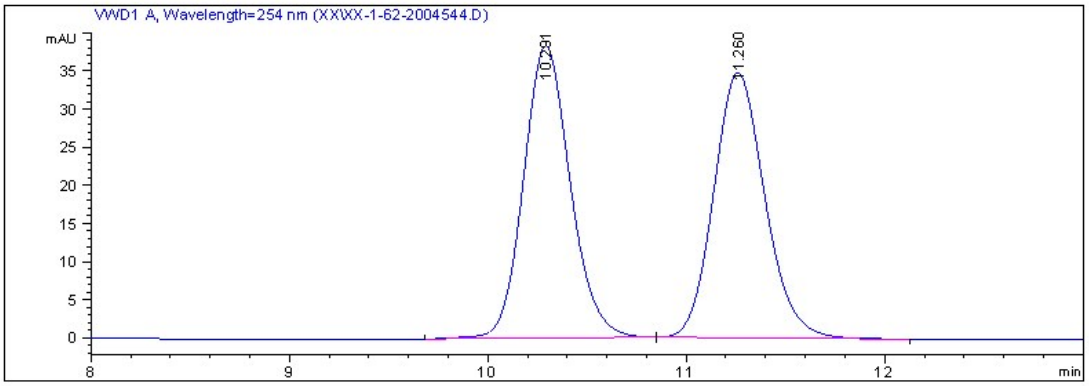
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.179	BB	0.1905	4515.59082	364.57715	50.0288
2	10.153	BBA	0.2422	4510.38965	288.86761	49.9712



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.275	BV	0.1943	307.58060	24.19364	2.9107
2	10.202	VV	0.2414	1.02595e4	652.56360	97.0893

(*1R*,*2R*)-2-((4-bromophenyl)amino)-1,2-dihydronaphthalen-1-ol (3ab)





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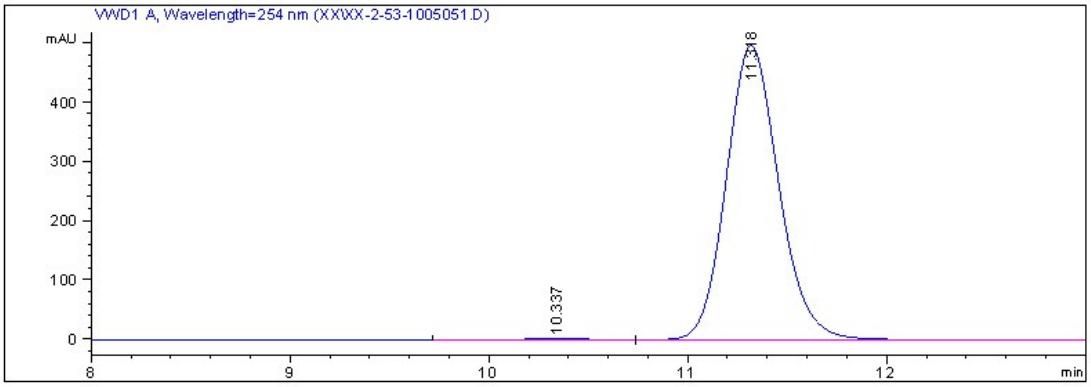
1	10.291	BB	0.2520	628.00580	38.36337	50.2401
2	11.260	BB	0.2758	622.00360	34.75885	49.7599



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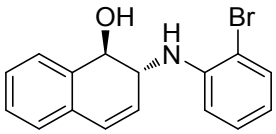
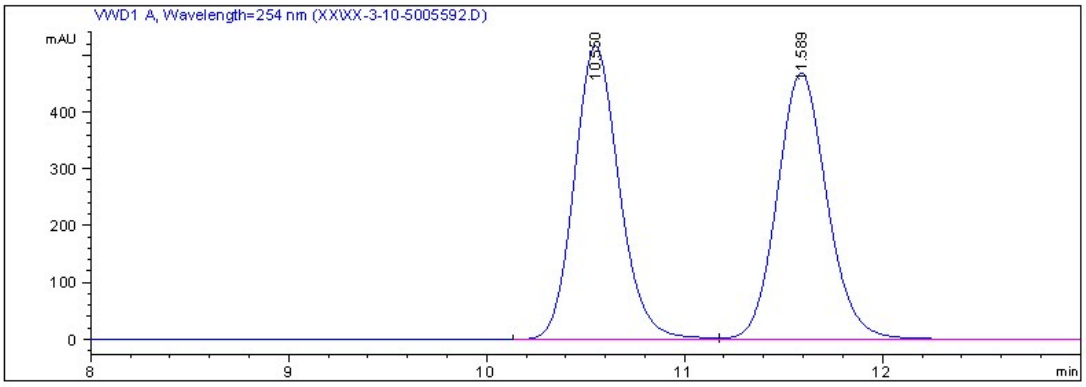
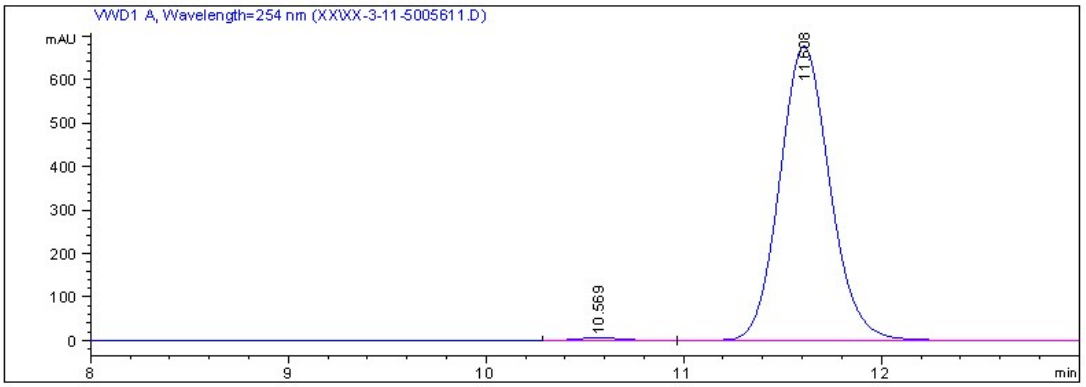
1	10.337	BB	0.2677	54.29538	3.05028	0.5919
2	11.318	BB	0.2833	9119.07031	496.54672	99.4081

(*1R,2R*)-2-((2-bromophenyl)amino)-1,2-dihydronaphthalen-1-ol (3ac)





Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.550	VV	0.2435	8168.61865	516.50897	49.8473
2	11.589	VBA	0.2703	8218.66211	469.40567	50.1527



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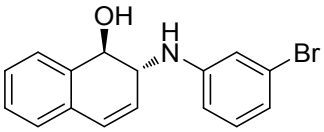
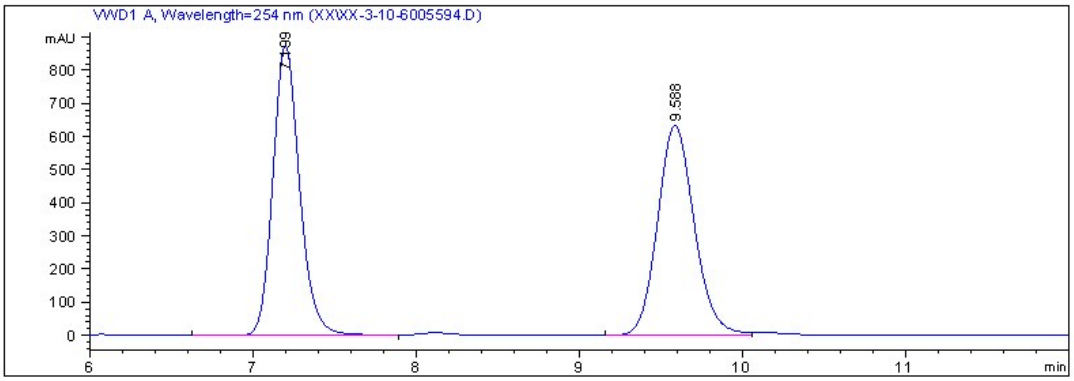
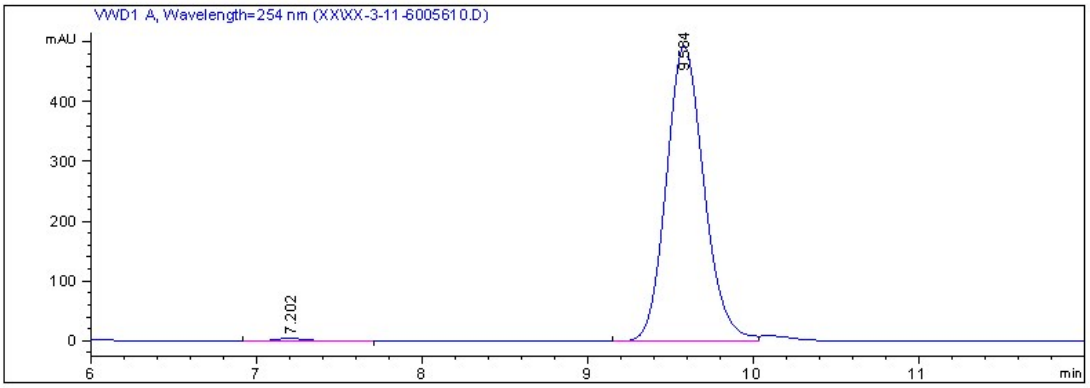
1	10.569	VB	0.2430	102.06426	6.50851	0.8467
2	11.608	BB	0.2734	1.19525e4	675.76282	99.1533

(*1R,2R*)-2-((3-bromophenyl)amino)-1,2-dihydronaphthalen-1-ol (3ad)





Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.199	BV	0.1742	9915.65137	874.80621	50.0102
2	9.588	BV	0.2414	9911.58984	634.11890	49.9898



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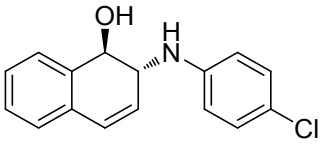
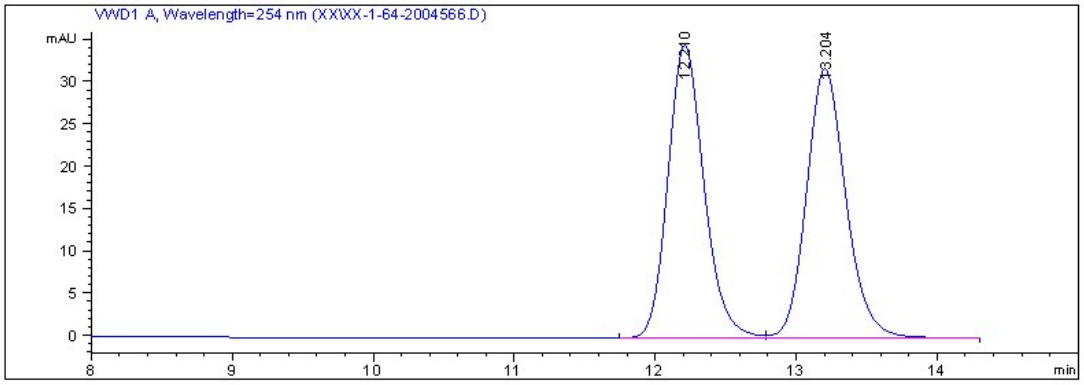
1	7.202	BB	0.1760	52.98172	4.61329	0.6746
2	9.584	BV	0.2443	7800.25830	493.71863	99.3254

(*1R,2R*)-2-((4-chlorophenyl)amino)-1,2-dihydronaphthalen-1-ol (3ae)





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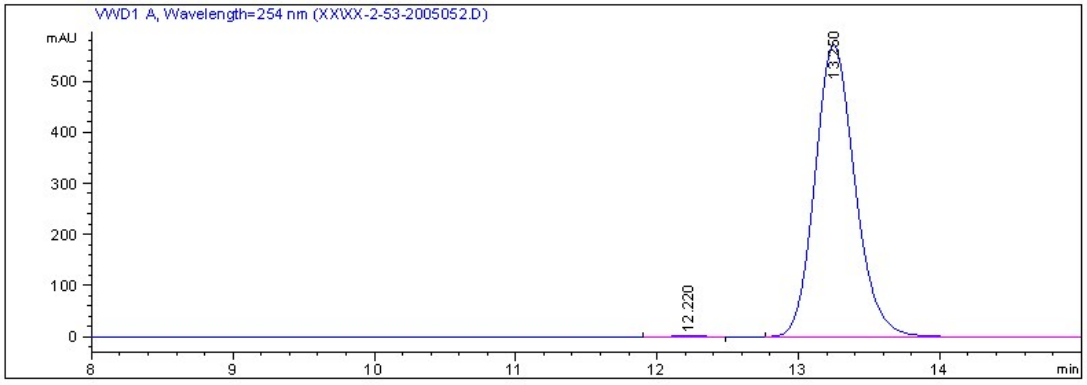
1	12.210	BV	0.2734	614.27893	34.54944	49.8984
2	13.204	VBA	0.2973	616.77948	31.79782	50.1016



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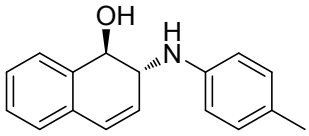
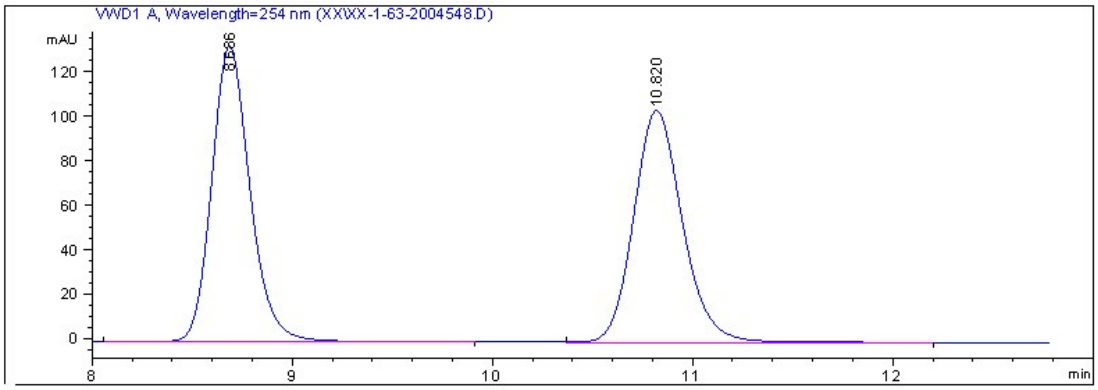
1	12.220	BV	0.2679	42.92094	2.50455	0.3780
2	13.250	VB	0.3031	1.13121e4	571.13660	99.6220

(*1R,2R*)-2-(p-tolylamino)-1,2-dihydronaphthalen-1-ol (3af)





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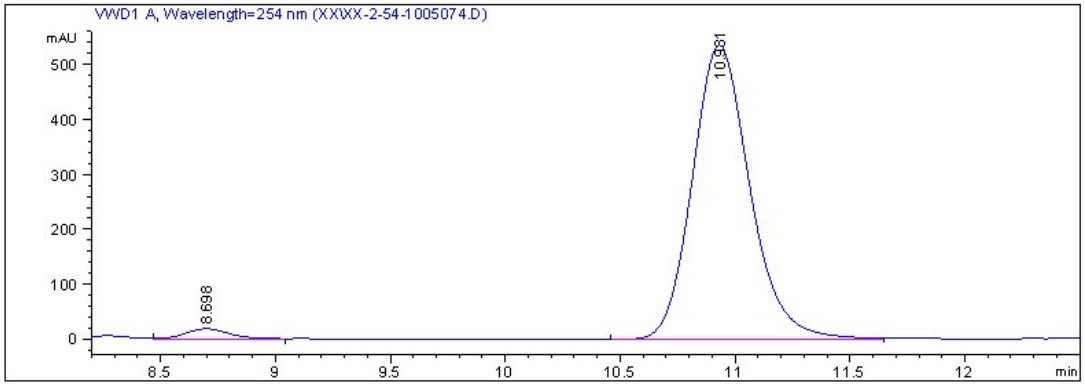
1	8.686	BB	0.2030	1765.29272	132.86456	50.1182
2	10.820	BB	0.2586	1756.96423	104.26015	49.8818



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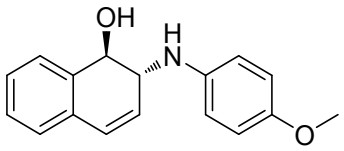
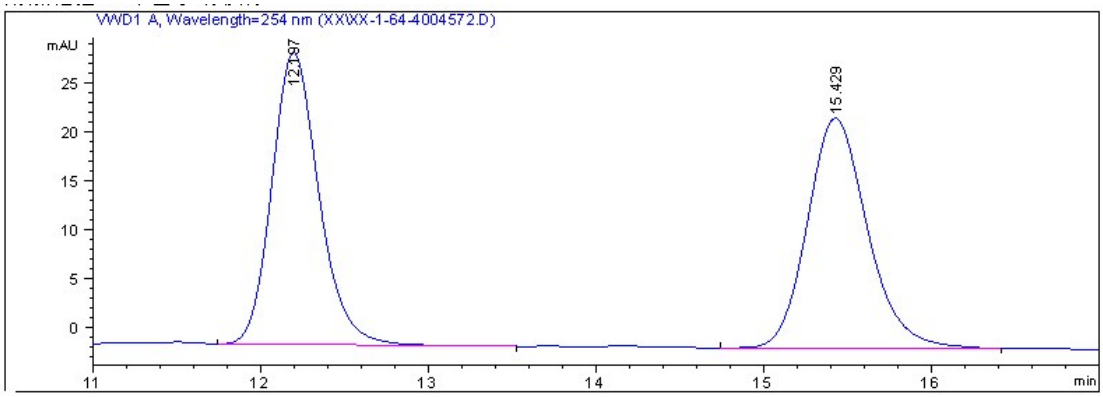
1	8.698	VV	0.2095	261.34195	18.99346	2.7268
2	10.931	BV	0.2669	9322.83691	536.12292	97.2732

(*1R,2R*)-2-((4-methoxyphenyl)amino)-1,2-dihydronaphthalen-1-ol (3ag)





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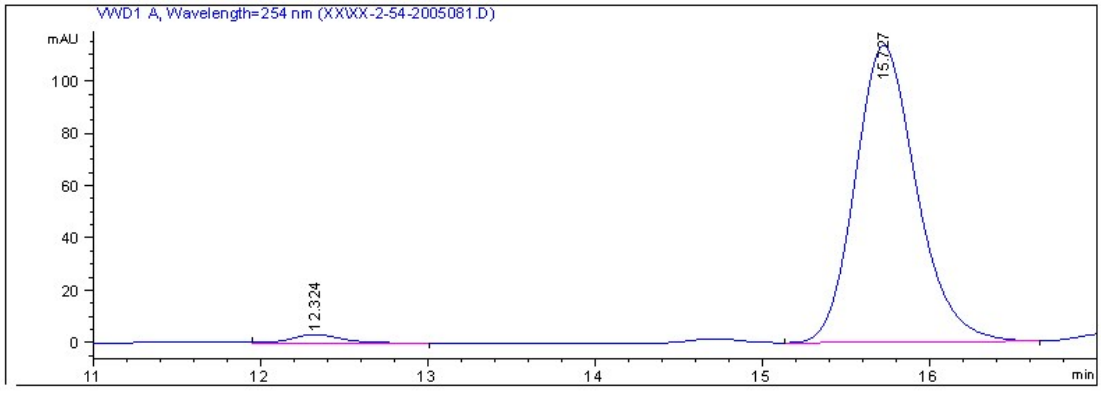
1	12.197	BB	0.2995	583.93176	29.94026	49.9163
2	15.429	BB	0.3821	585.89014	23.55910	50.0837



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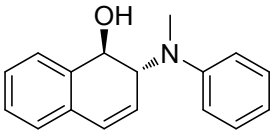
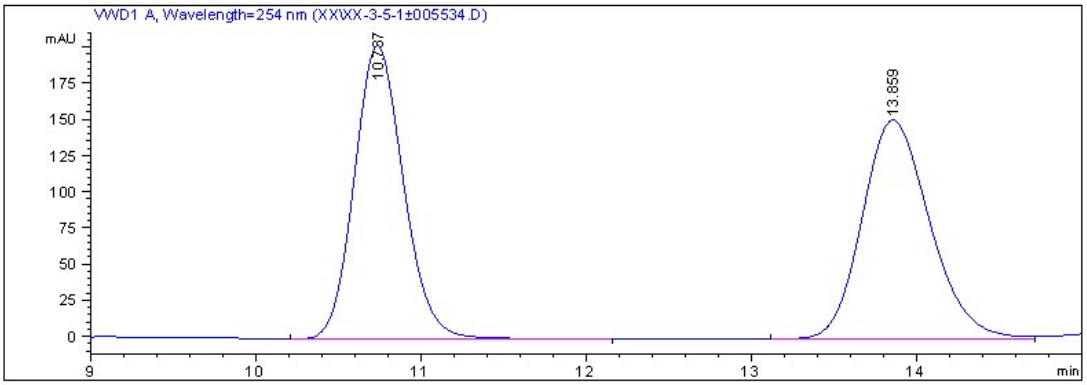
1	12.324	BB	0.3021	61.87652	3.12371	2.0982
2	15.727	BB	0.3913	2887.20776	113.30150	97.9018

(*1R,2R*)-2-(methyl(phenyl)amino)-1,2-dihydronaphthalen-1-ol (3ah)





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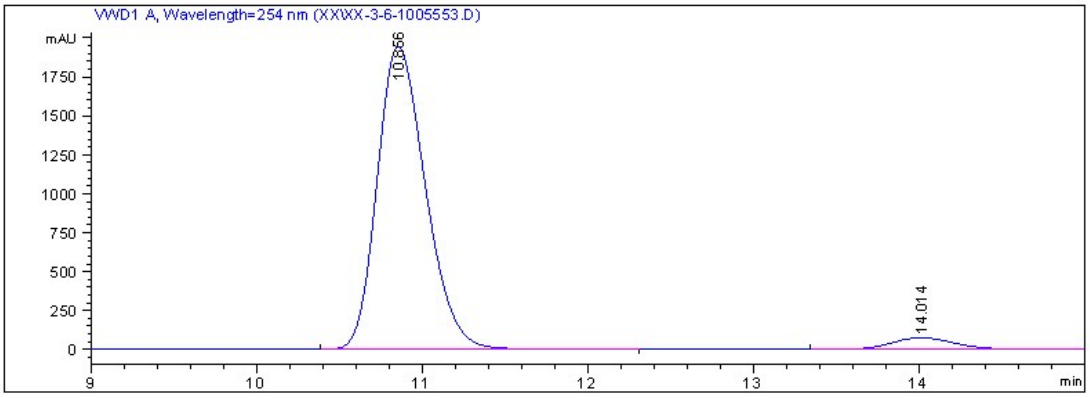
1	10.737	BB	0.3205	4201.07129	202.93895	49.2491
2	13.859	BV	0.4422	4329.18604	151.58702	50.7509



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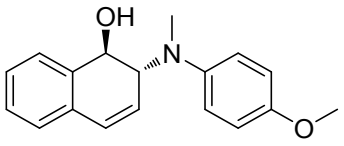
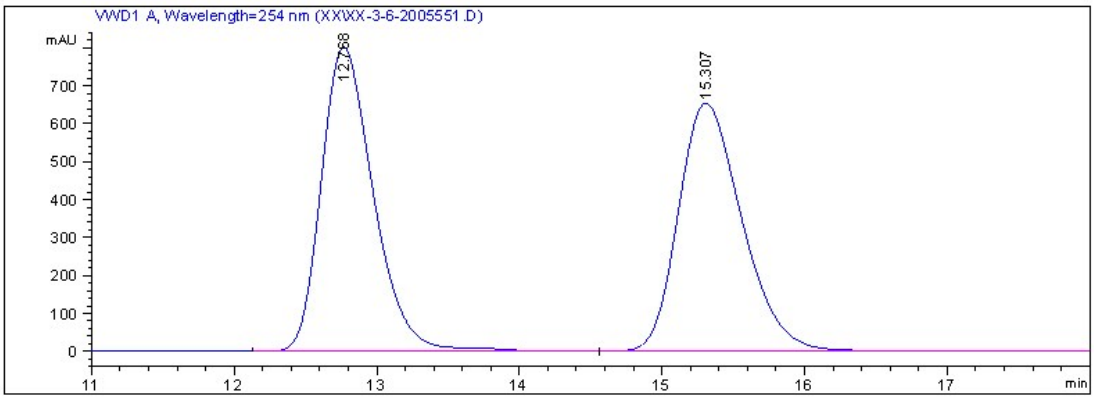
1	10.856	VB	0.3148	3.95112e4	1946.85400	95.0037
2	14.014	BB	0.4217	2077.90942	76.30031	4.9963

(*1R,2R*)-2-((4-methoxyphenyl)(methyl)amino)-1,2-dihydronaphthalen-1-ol (3ai)





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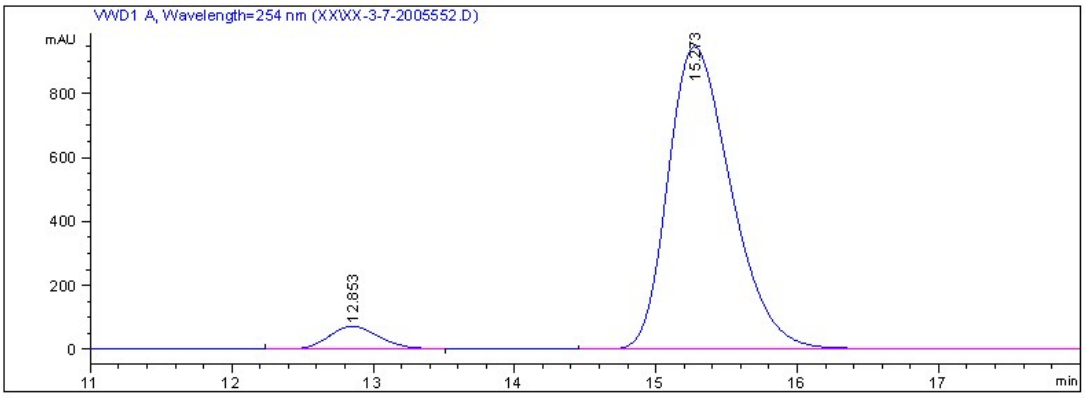
1	12.768	BB	0.3921	2.04077e4	801.43842	50.1658
2	15.307	BB	0.4797	2.02728e4	653.86774	49.8342



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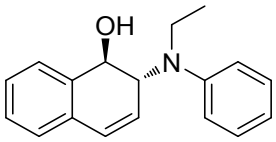
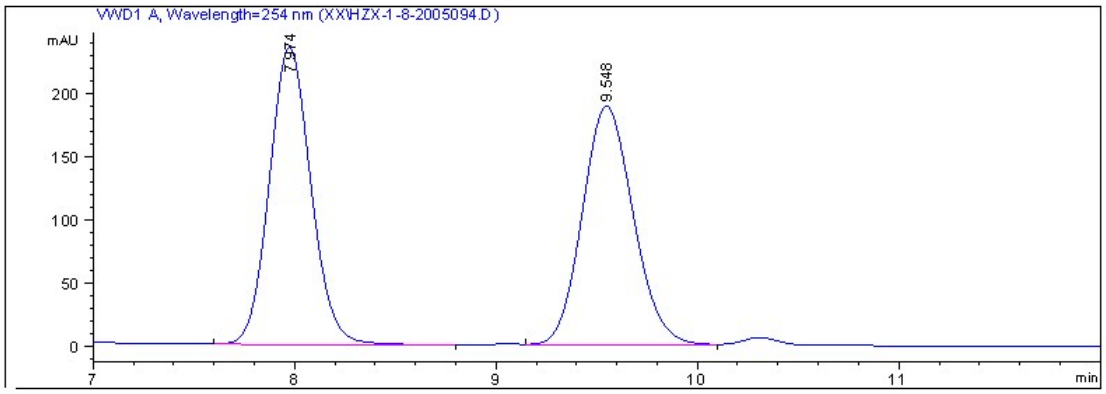
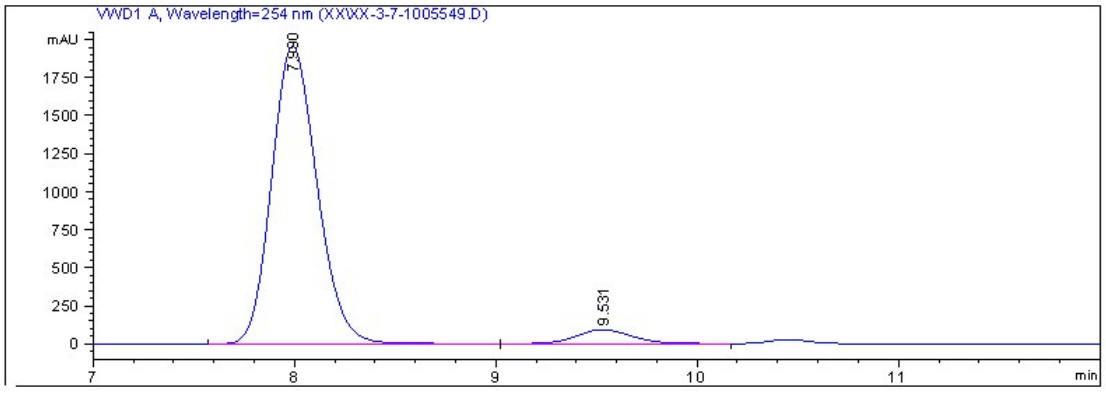
1	12.853	BV	0.3896	1836.55225	72.72127	5.8574
2	15.273	BBA	0.4838	2.95178e4	946.47217	94.1426

(*1R,2R*)-2-(ethyl(phenyl)amino)-1,2-dihydronaphthalen-1-ol (3aj)





Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.974	VB	0.2226	3373.09155	234.76782	50.3130
2	9.548	VV	0.2737	3331.12183	188.92914	49.6870



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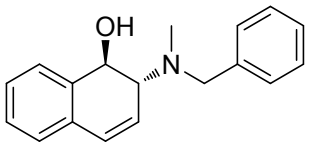
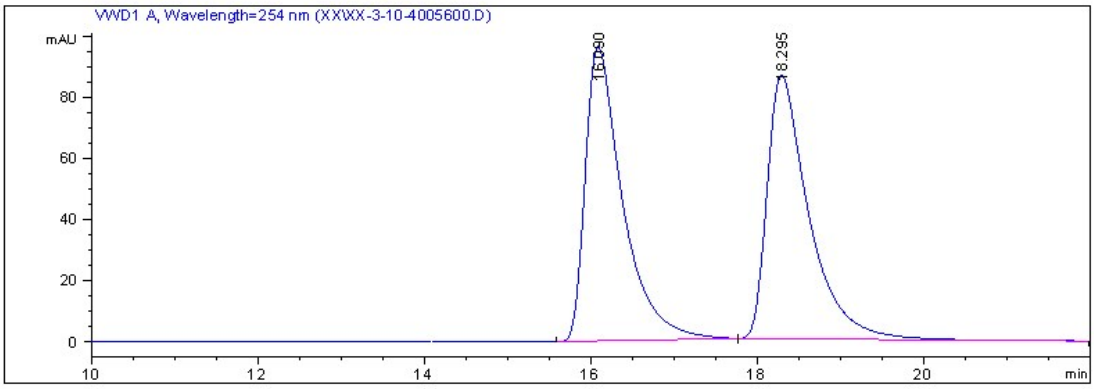
1	7.990	BB	0.2429	3.06474e4	1955.23560	94.5367
2	9.531	BV	0.3006	1771.11755	91.19701	5.4633

(*1R,2R*)-2-(benzyl(methyl)amino)-1,2-dihydronaphthalen-1-ol (3ak)





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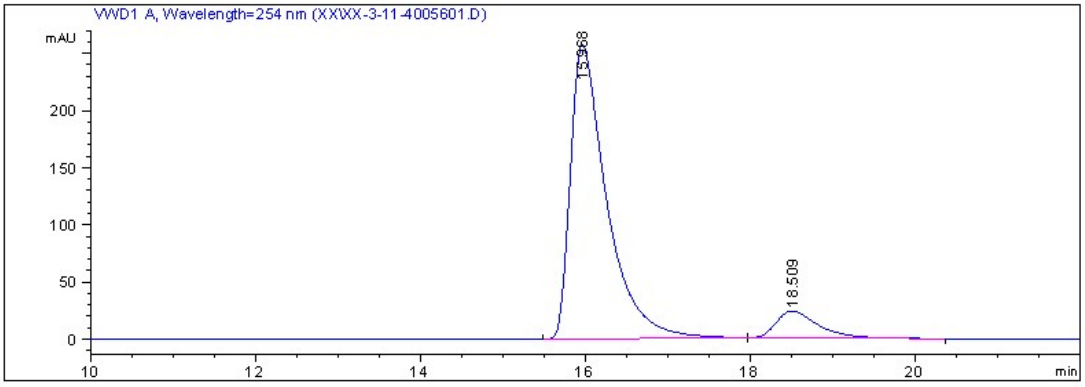
1	16.090	BB	0.4583	3000.18896	96.33494	49.9612
2	18.295	BB	0.5124	3004.84619	86.26593	50.0388



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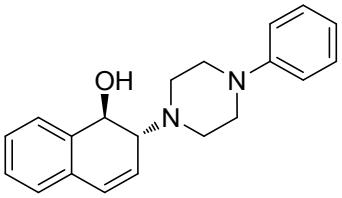
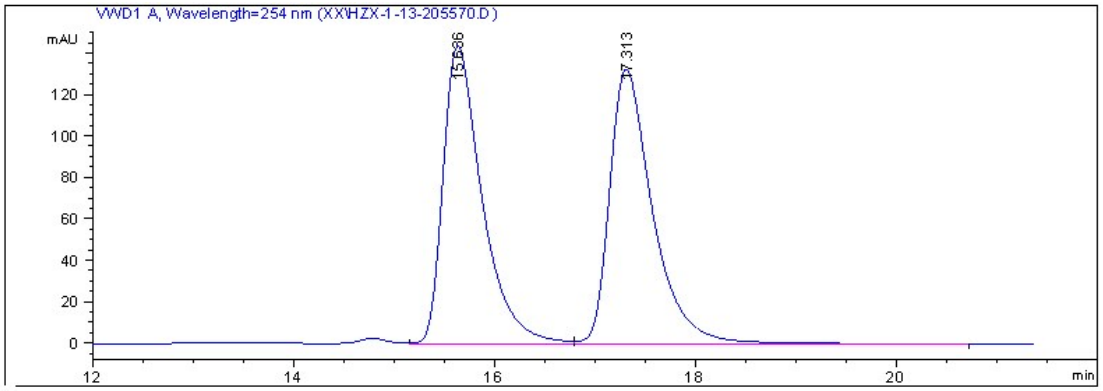
1	15.968	BB	0.4484	7846.17822	256.80942	90.3810
2	18.509	BB	0.5285	835.05157	23.55798	9.6190

(*1R,2R*)-2-(4-phenylpiperazin-1-yl)-1,2-dihydronaphthalen-1-ol (3al)





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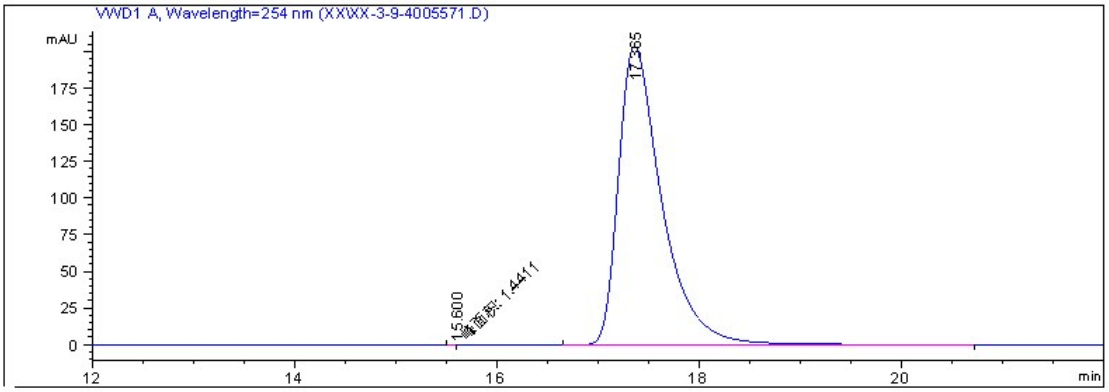
1	15.636	VV	0.4044	3868.77710	143.57152	49.5919
2	17.313	VB	0.4454	3932.44824	132.43578	50.4081



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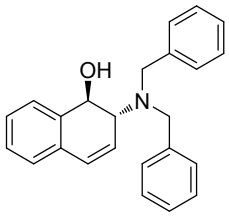
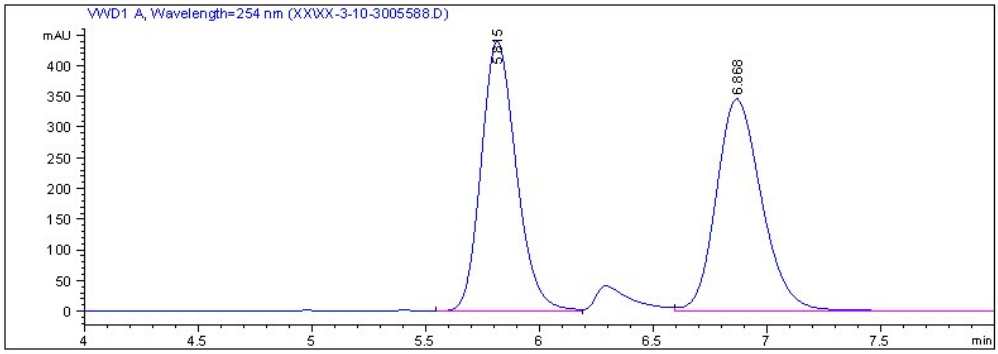
1	15.600	MM	0.0947	1.44110	2.53637e-1	0.0238
2	17.365	BB	0.4449	6056.88525	203.68887	99.9762

(*1R,2R*)-2-(dibenzylamino)-1,2-dihydronaphthalen-1-ol (3am)





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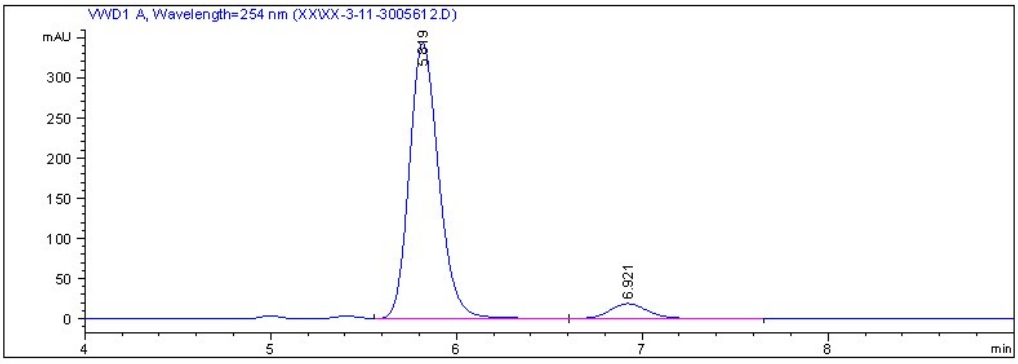
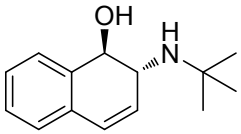
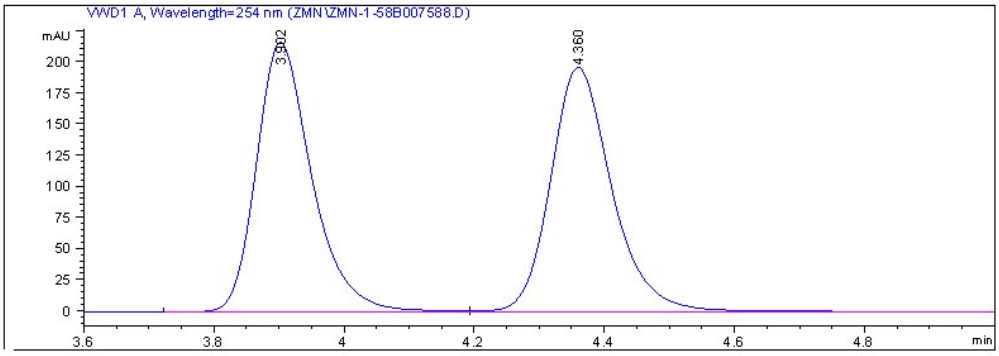
1	5.815	BV	0.1654	4713.65625	438.58847	49.6030
2	6.868	VB	0.2137	4789.11768	345.59076	50.3970



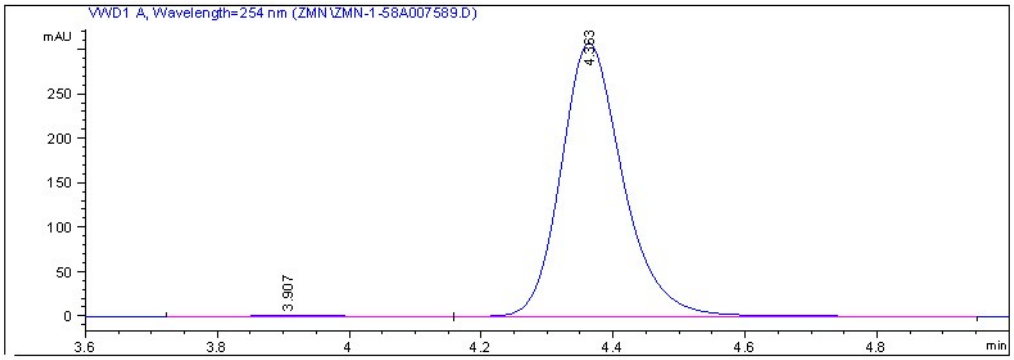
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.819	VB	0.1687	3753.78955	342.82809	93.6045
2	6.921	BB	0.2156	256.47766	18.40761	6.3955

(*1R,2R*)-2-(tert-butylamino)-1,2-dihydronaphthalen-1-ol (3an)



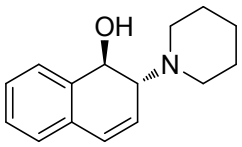


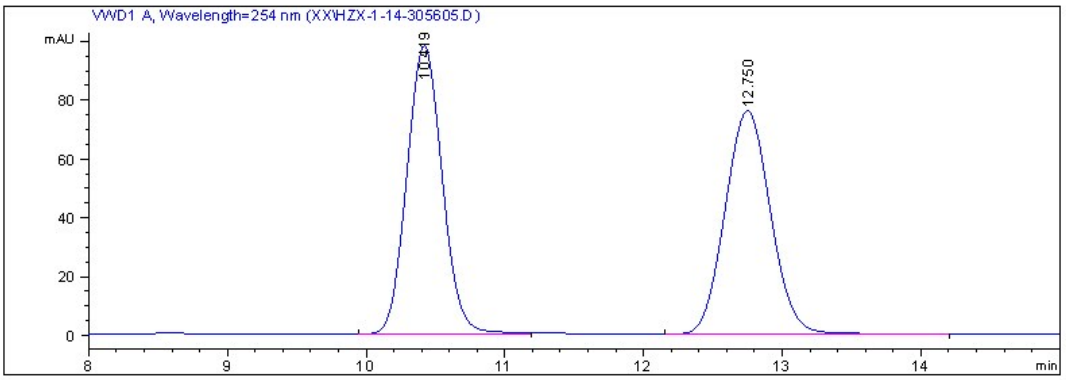
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.902	BV	0.0889	1279.77844	216.67751	49.8134
2	4.360	VB	0.0996	1289.36658	196.43668	50.1866



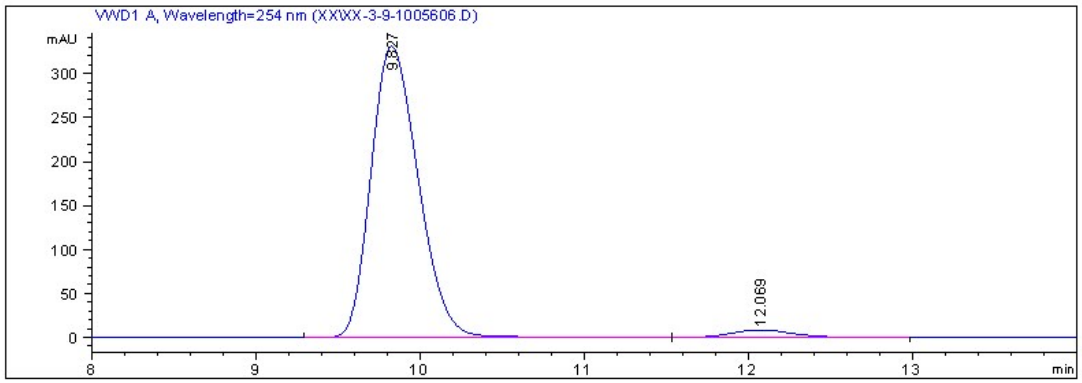
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.907	BV	0.1089	14.16421	1.85908	0.6995
2	4.363	VB	0.0992	2010.85779	308.04178	99.3005

(*1R,2R*)-2-(piperidin-1-yl)-1,2-dihydronaphthalen-1-ol (3ao)



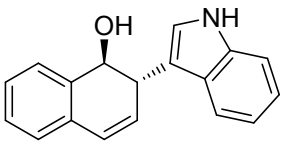


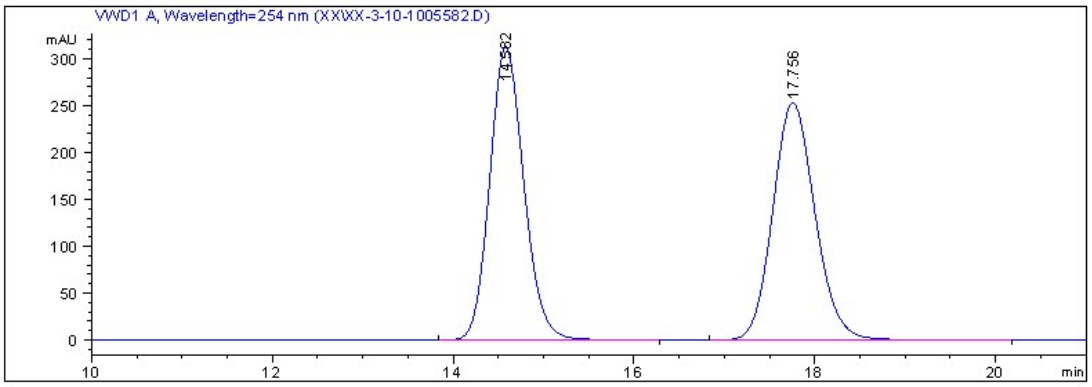
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.419	BV	0.2756	1735.03772	97.98779	50.1264
2	12.750	BB	0.3569	1726.29028	76.02316	49.8736



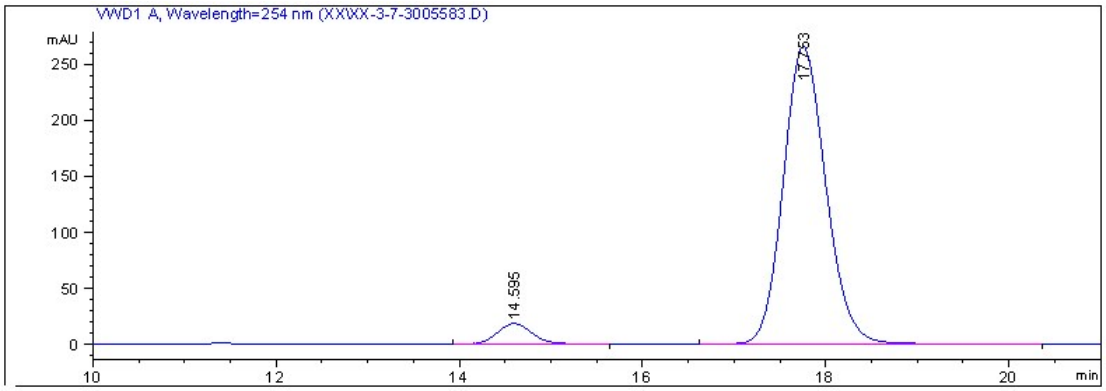
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.827	BB	0.3127	6602.52686	331.00534	96.8565
2	12.069	BB	0.3996	214.28645	8.37256	3.1435

(*1S,2S*)-2-(1*H*-indol-3-yl)-1,2-dihydronaphthalen-1-ol (3ap)



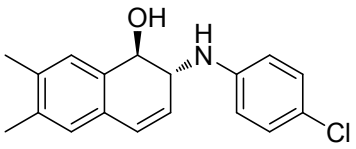


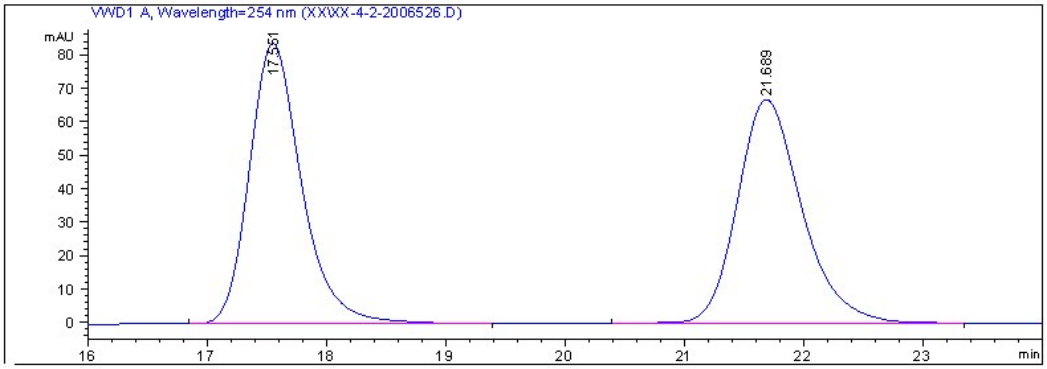
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.582	BB	0.4045	8193.72363	312.94376	49.9592
2	17.756	BB	0.5018	8207.12109	253.41916	50.0408



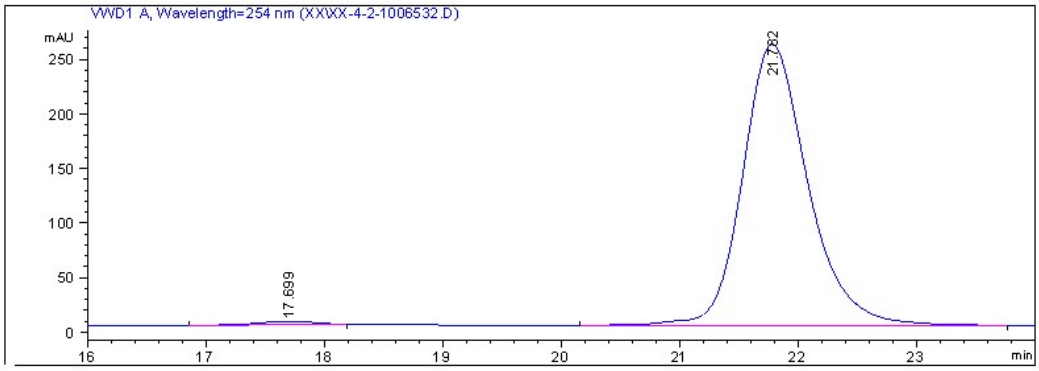
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.595	BB	0.4036	475.00696	18.25209	5.2386
2	17.753	BB	0.4990	8592.43262	265.86679	94.7614

(*1R,2R*)-2-((4-chlorophenyl)amino)-6,7-dimethyl-1,2-dihydronaphthalen-1-ol (3be)



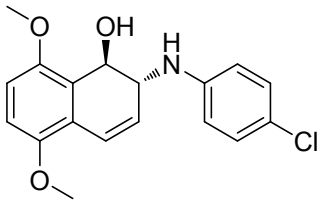


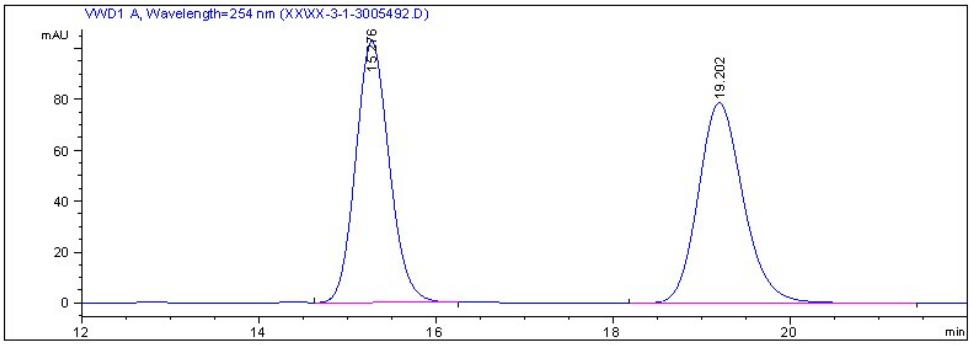
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.551	BB	0.4640	2551.56885	83.86168	50.1354
2	21.689	BB	0.5768	2537.78882	67.00917	49.8646



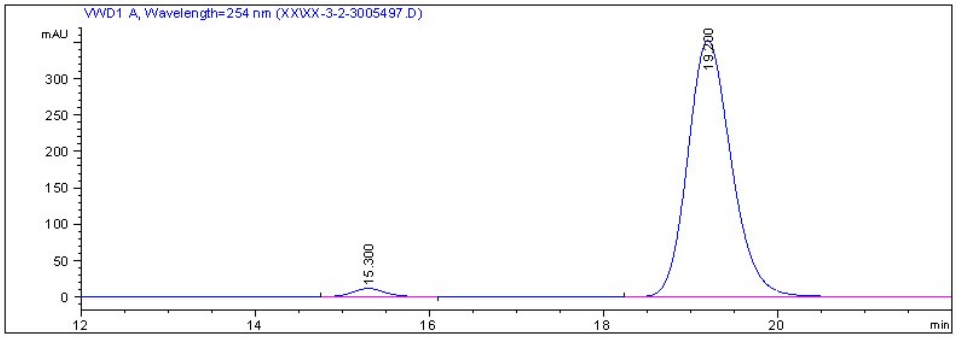
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.699	BB	0.5372	99.90675	2.95692	0.9991
2	21.782	BB	0.5828	9900.16895	257.90881	99.0009

(*1R,2R*)-2-((4-chlorophenyl)amino)-5,8-dimethoxy-1,2-dihydronaphthalen-1-ol (3ce)





Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.276	BB	0.4077	2708.41797	102.69289	49.3810
2	19.202	BB	0.5414	2776.32324	78.73471	50.6190



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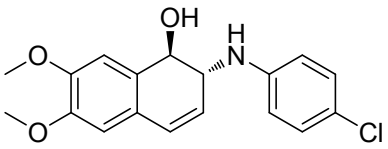
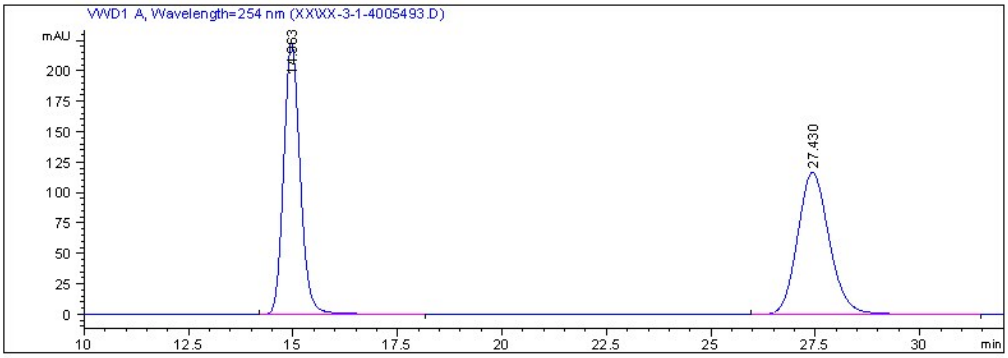
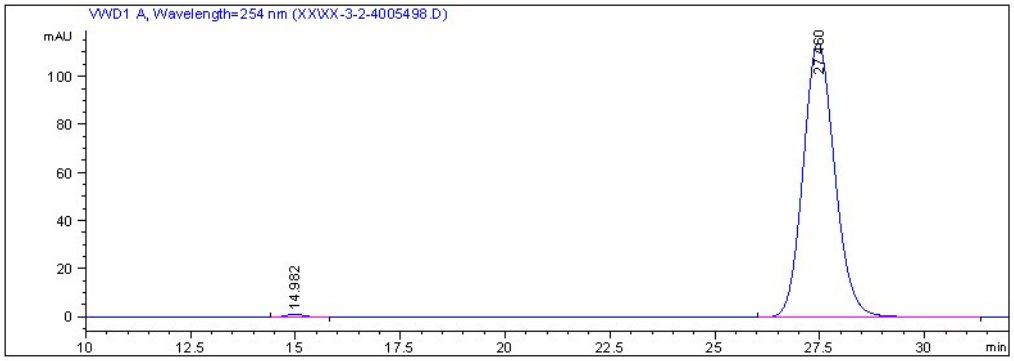
1	15.300	BB	0.4067	293.76254	11.21145	2.3369
2	19.200	BB	0.5354	1.22768e4	353.33633	97.6631

(*1R,2R*)-2-((4-chlorophenyl)amino)-6,7-dimethoxy-1,2-dihydronaphthalen-1-ol (3de)



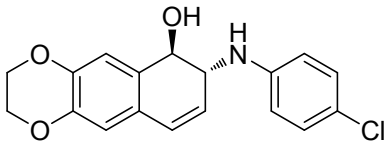


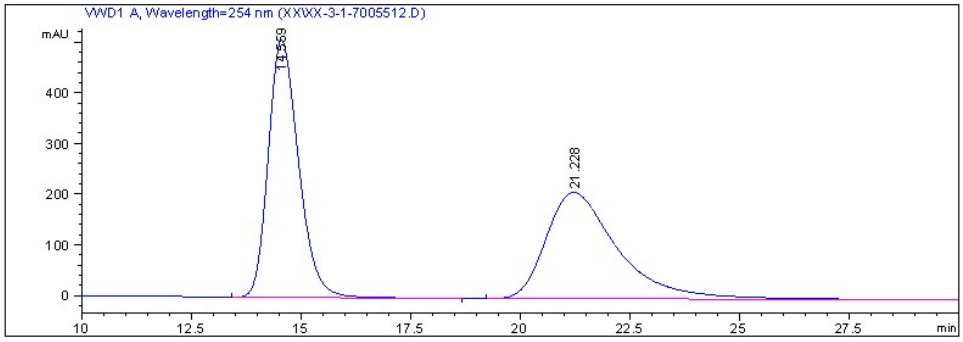
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.963	BB	0.4232	6130.11572	223.35675	49.8853
2	27.430	BB	0.8150	6158.29932	116.81683	50.1147



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.982	BB	0.4208	33.54596	1.23538	0.5588
2	27.460	BB	0.8105	5969.83887	114.08935	99.4412

(*6R,7R*)-7-((4-chlorophenyl)amino)-2,3,6,7-tetrahydronaphtho[2,3-*b*][1,4]dioxin-6-ol (3ee)





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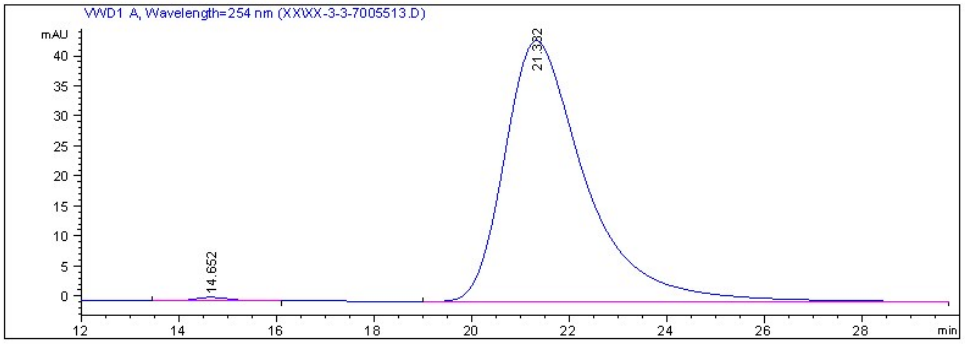
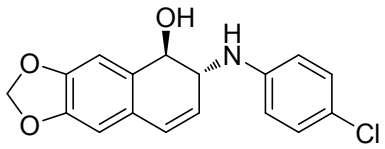
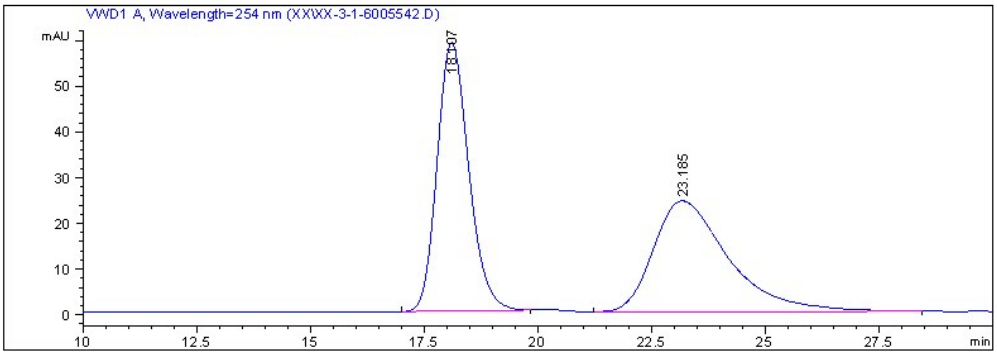
1	14.559	BB	0.7424	2.43784e4	507.47772	50.5465
2	21.228	BBA	1.6735	2.38513e4	209.92955	49.4535



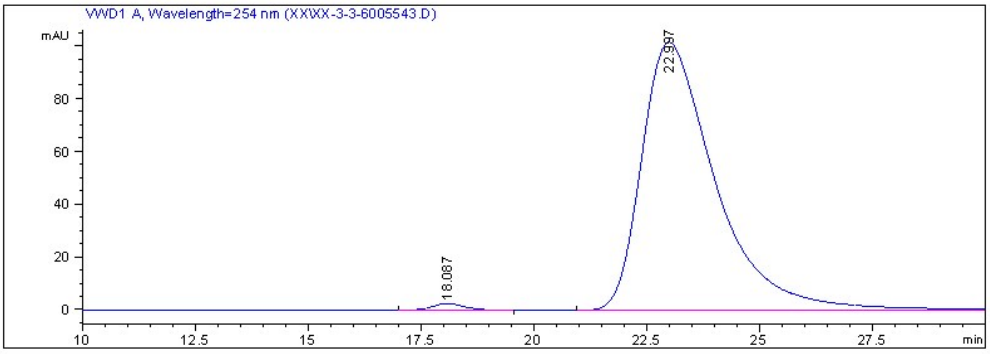
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.652	BB	0.7486	31.55239	6.29656e-1	0.6256
2	21.332	BBA	1.7358	5012.12695	43.44894	99.3744

(*5R,6R*)-6-((4-chlorophenyl)amino)-5,6-dihydronaphtho[2,3-d][1,3]dioxol-5-ol (3fe)



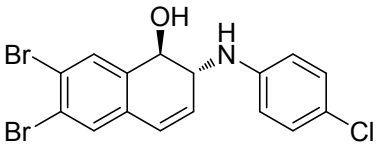


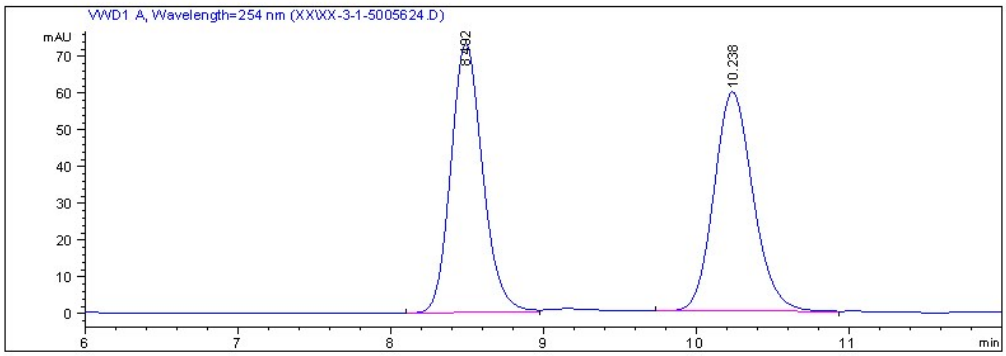
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.107	BB	0.7474	2835.15063	58.71103	50.5463
2	23.185	BB	1.7101	2773.86377	24.18402	49.4537



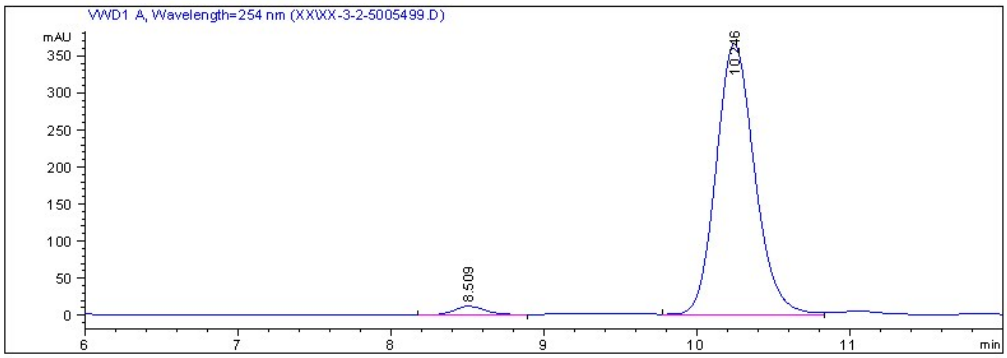
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.087	BB	0.7404	119.52572	2.43598	1.0203
2	22.997	BB	1.6927	1.15952e4	101.65601	98.9797

(*1R,2R*)-6,7-dibromo-2-((4-chlorophenyl)amino)-1,2-dihydronaphthalen-1-ol (3ge)



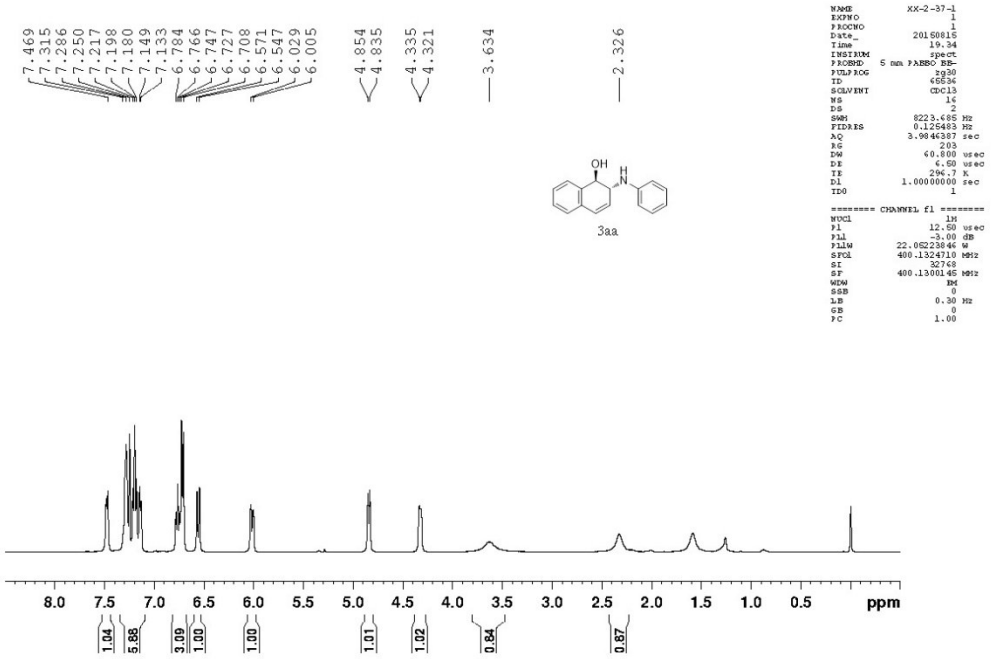


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.492	BV	0.2228	1058.95520	73.19112	49.9826
2	10.238	BB	0.2742	1059.69080	59.66092	50.0174



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.509	BV	0.2140	159.59262	11.49162	2.4426
2	10.246	BV	0.2696	6374.06543	365.29947	97.5574

B: Copies of ^{1}H and ^{13}C NMR spectra of ring-opening compounds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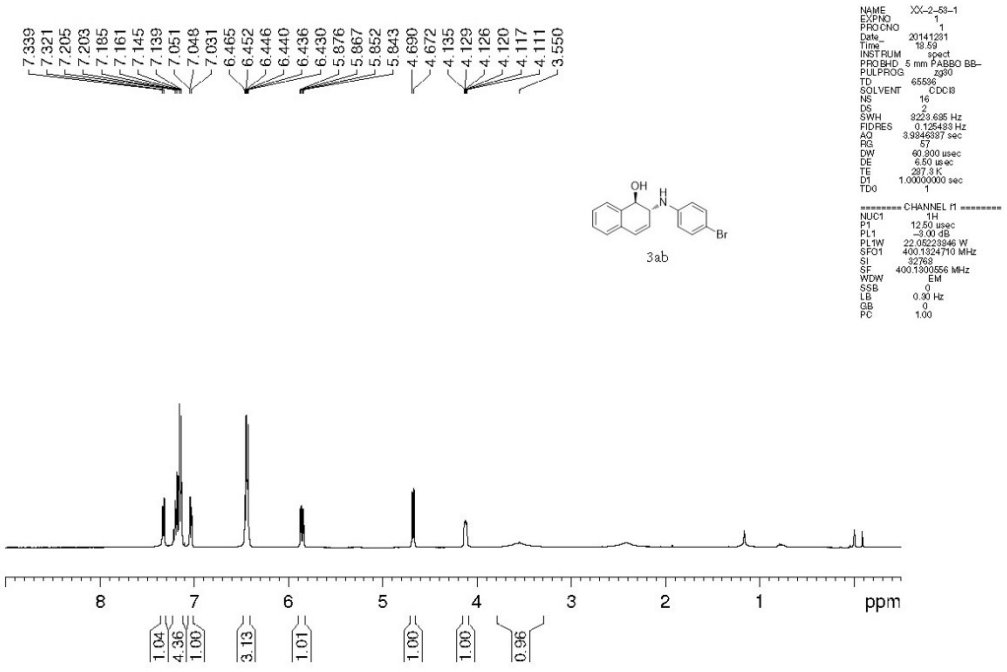
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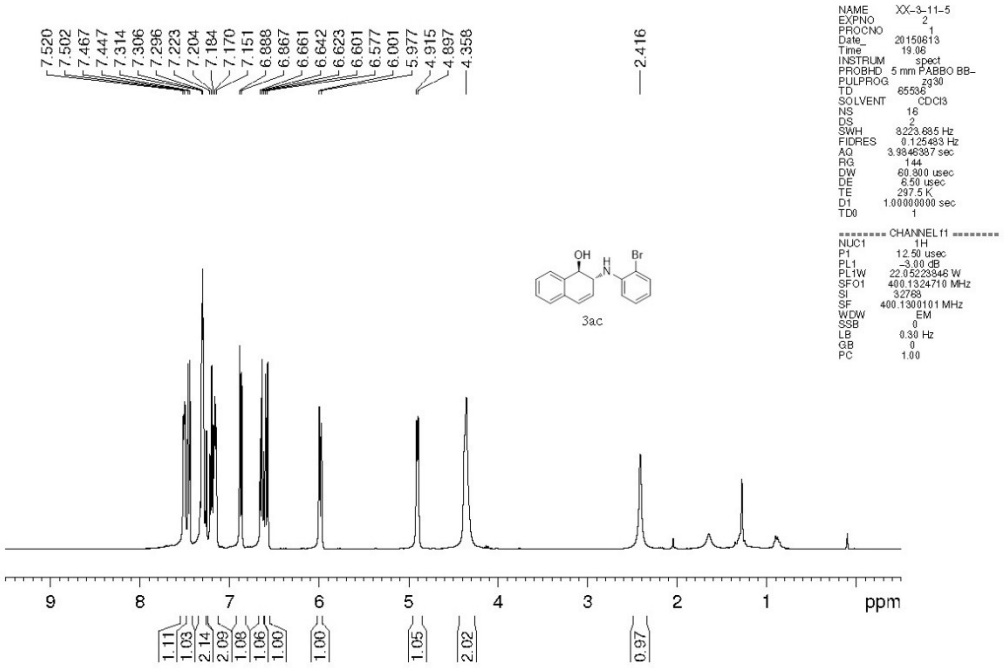
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DE 6.50 usec
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D1 1.00000000 sec
TD0 1
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P1 12.50 usec
PL1 -3.00 dB
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SFO1 400.1324710 MHz
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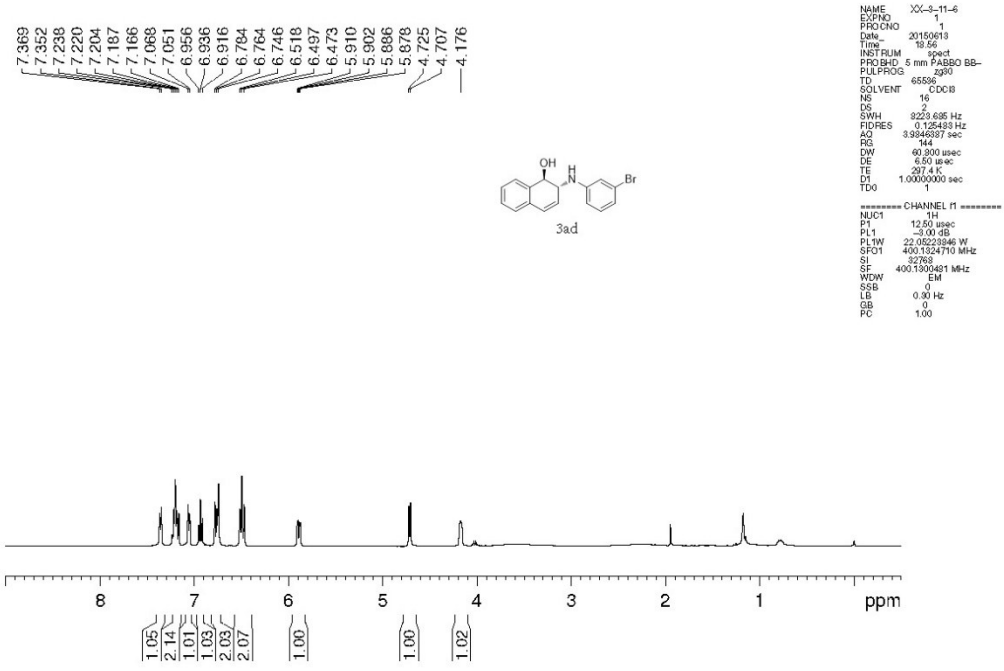
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DW 60.800 usec
DE 6.50 usec
TE 297.4 K
D1 1.00000000 sec
TD0 1
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PL1 -3.00 dB
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SFO1 400.1324710 MHz
SI 32768
SF 400.1300491 MHz
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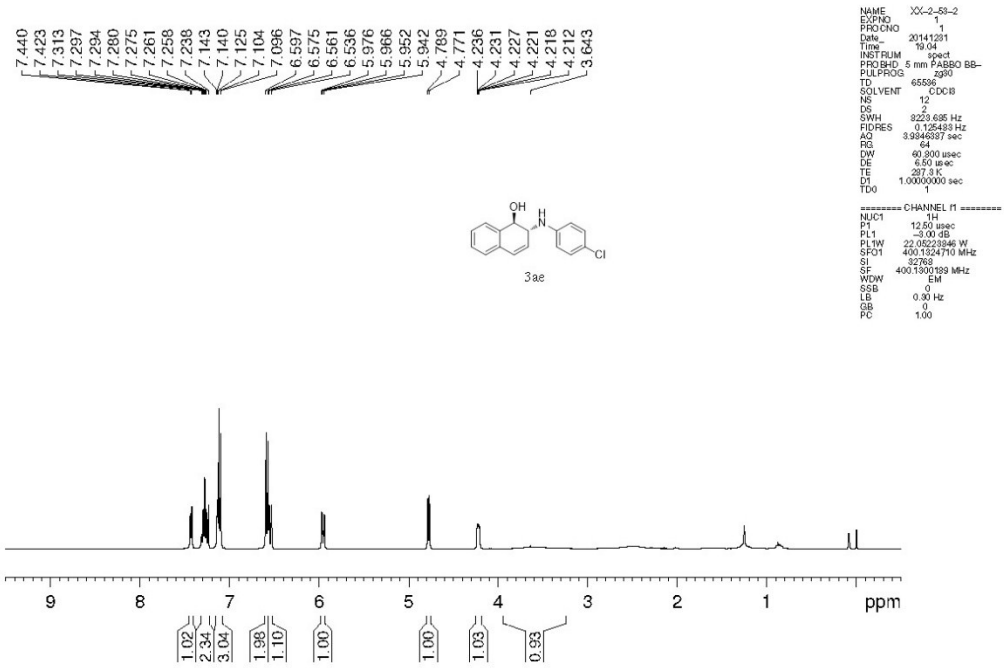
2.03
2.07
1.00
1.00
1.02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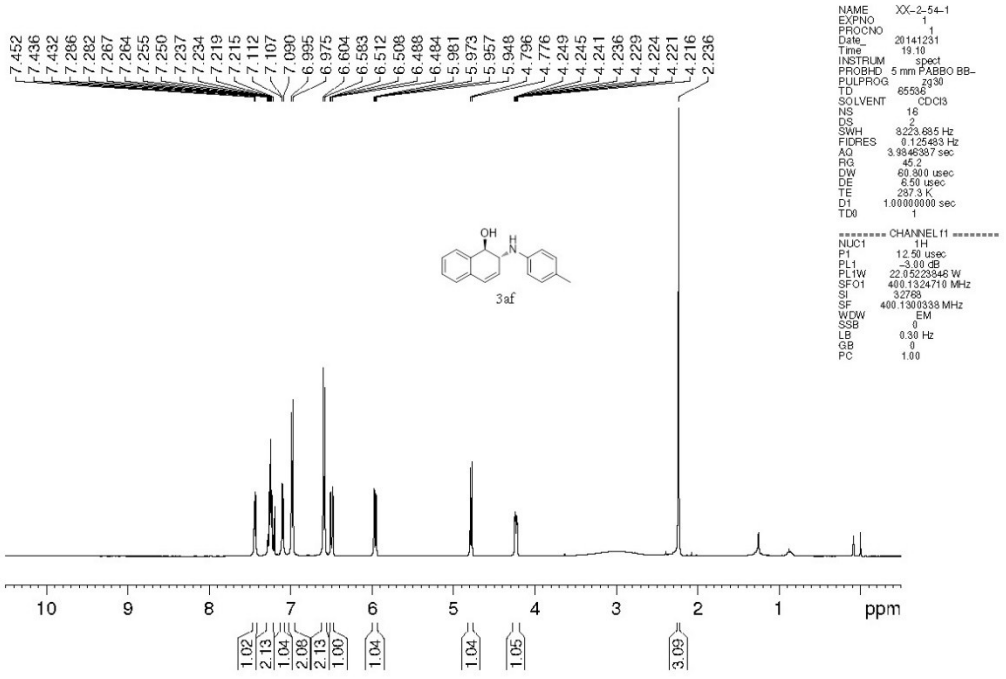
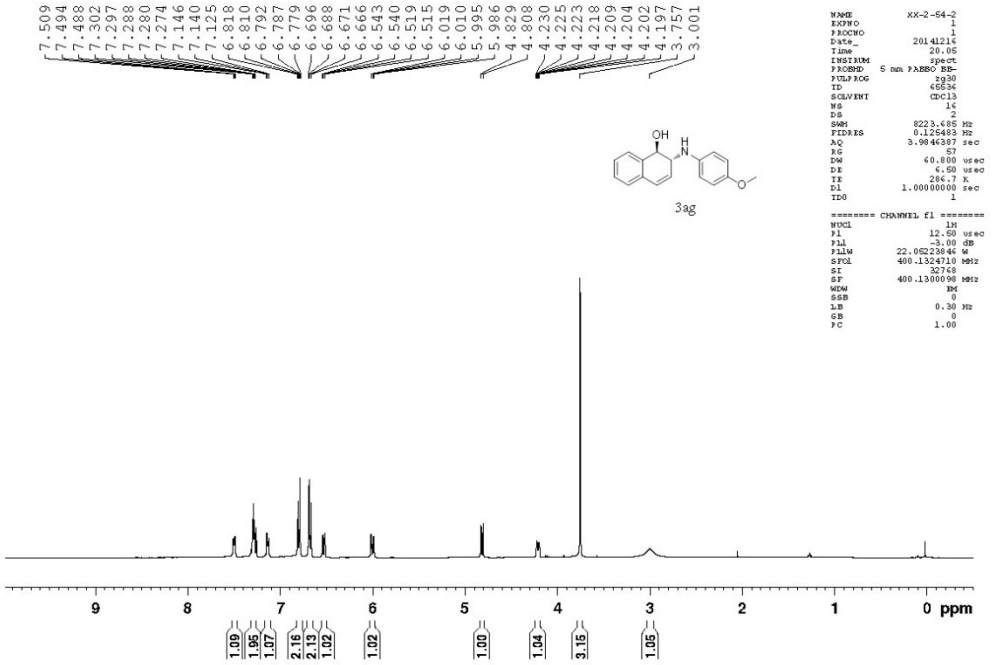
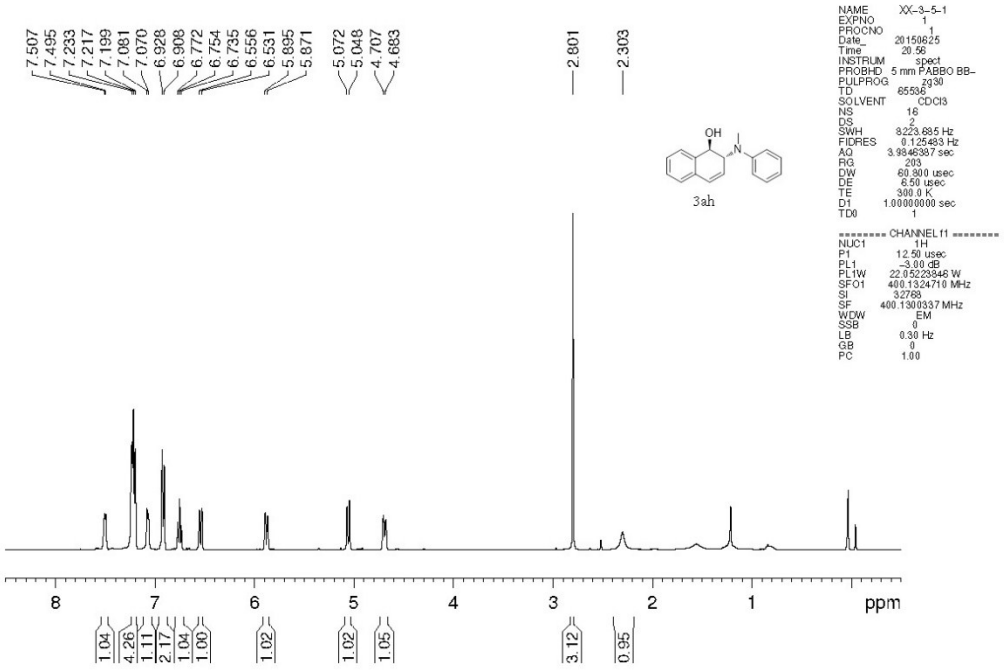
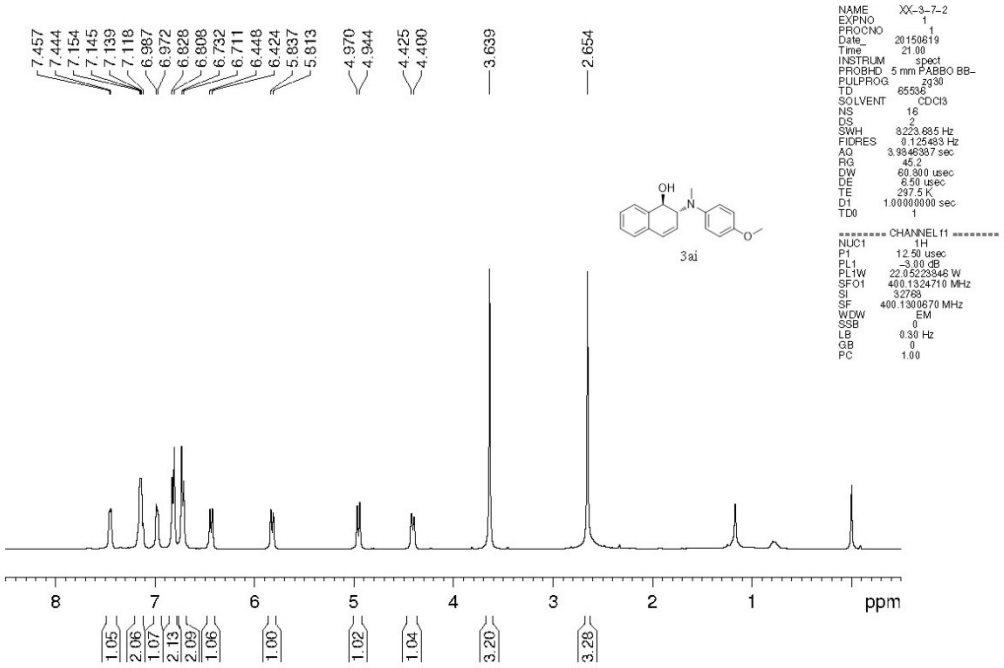
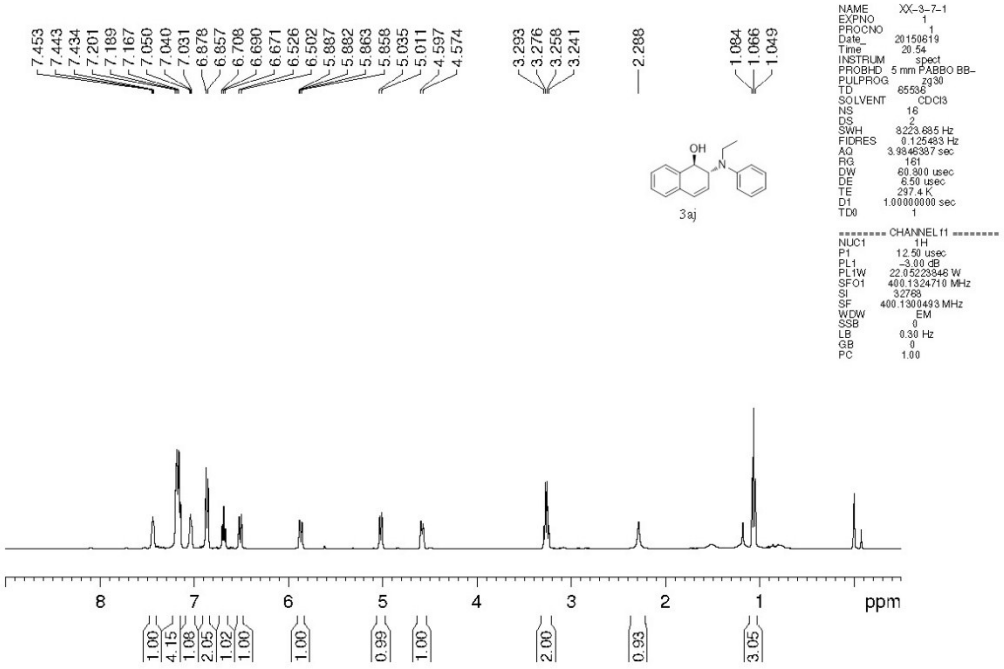
7.423
7.313
7.297
7.294
7.280
7.275
7.261
7.258
7.238
7.143
7.140
7.125
7.104
7.096
6.597
6.575
6.561
6.536
5.976
5.966
5.952
5.942
4.789
4.771
4.236
4.231
4.227
4.221
4.218
4.212
3.643
OH
H
N
Cl
3ae
NAME XX-2-53-2
EXPNO 1
PROCNO 1
Date_ 20141231
Time 19.04
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 12
DS 2
SWH 8229.685 Hz
FIDRES 0.125493 Hz
AQ 3.9846387 sec
RG 64
DW 60.800 usec
DE 6.50 usec
TE 287.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -3.00 dB
PL1W 22.05223846 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300199 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm
1.02
2.34
3.04
1.98
1.10
1.00
1.00
1.03
0.93


7.452
7.436
7.432
7.286
7.282
7.267
7.264
7.255
7.250
7.237
7.234
7.219
7.215
7.112
7.107
7.090
6.995
6.975
6.604
6.583
6.512
6.508
6.488
6.484
5.981
5.973
5.957
5.948
4.796
4.776
4.249
4.245
4.241
4.236
4.229
4.224
4.221
4.216
2.236
OH
H
N
3af
NAME XX-2-54-1
EXPNO 1
PROCNO 1
Date_ 20141231
Time 19.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 45.2
DW 60.800 usec
DE 6.50 usec
TE 287.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -3.00 dB
PL1W 22.05223846 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300338 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
1.02
2.13
1.04
2.08
2.13
1.00
1.04
1.04
1.05
3.09


7.509
7.494
7.488
7.302
7.297
7.288
7.280
7.274
7.146
7.140
7.125
6.818
6.810
6.792
6.787
6.779
6.696
6.688
6.671
6.666
6.543
6.540
6.519
6.515
6.019
6.010
5.995
5.986
4.829
4.808
4.230
4.225
4.223
4.218
4.209
4.204
4.202
4.197
3.757
3.001
OH
H
N
O
3ag
NAME XX-2-54-2
EXPNO 1
PROCNO 1
Date_ 20141214
Time 20.05
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 57
DW 60.800 usec
DE 6.50 usec
TE 286.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -3.00 dB
PL1W 22.05223846 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0 ppm
1.09
1.95
1.07
2.16
2.13
1.02
1.02
1.00
1.04
3.15
1.05


7.507
7.495
7.233
7.217
7.199
7.081
7.070
6.928
6.908
6.772
6.754
6.735
6.556
6.531
5.895
5.871
5.072
5.048
4.707
4.683
2.801
2.303
OH
N
3ah
NAME XX-3-5-1
EXPNO 1
PROCNO 1
Date_ 20150625
Time 20.56
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -3.00 dB
PL1W 22.05223846 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300337 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8
7
6
5
4
3
2
1
ppm
1.04
4.26
1.11
2.17
1.04
1.00
1.02
1.02
1.05
3.12
0.95


7.457
7.444
7.154
7.145
7.139
7.118
6.987
6.972
6.828
6.808
6.732
6.711
6.448
6.424
5.837
5.813
4.970
4.944
4.425
4.400
3.639
2.654
3ai
NAME XX-3-7-2
EXPNO 1
PROCNO 1
Date_ 20150619
Time 21.00
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 45.2
DW 60.800 usec
DE 6.50 usec
TE 297.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -3.00 dB
PL1W 22.05223848 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300670 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8
7
6
5
4
3
2
1
ppm
1.05
2.06
1.07
2.13
2.09
1.06
1.00
1.02
1.04
3.20
3.28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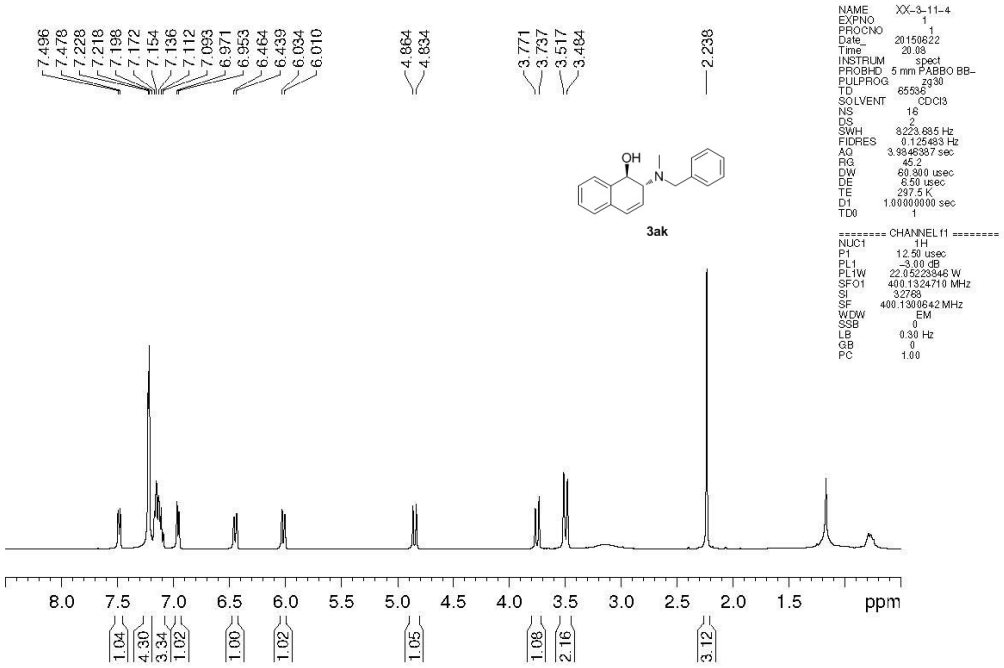
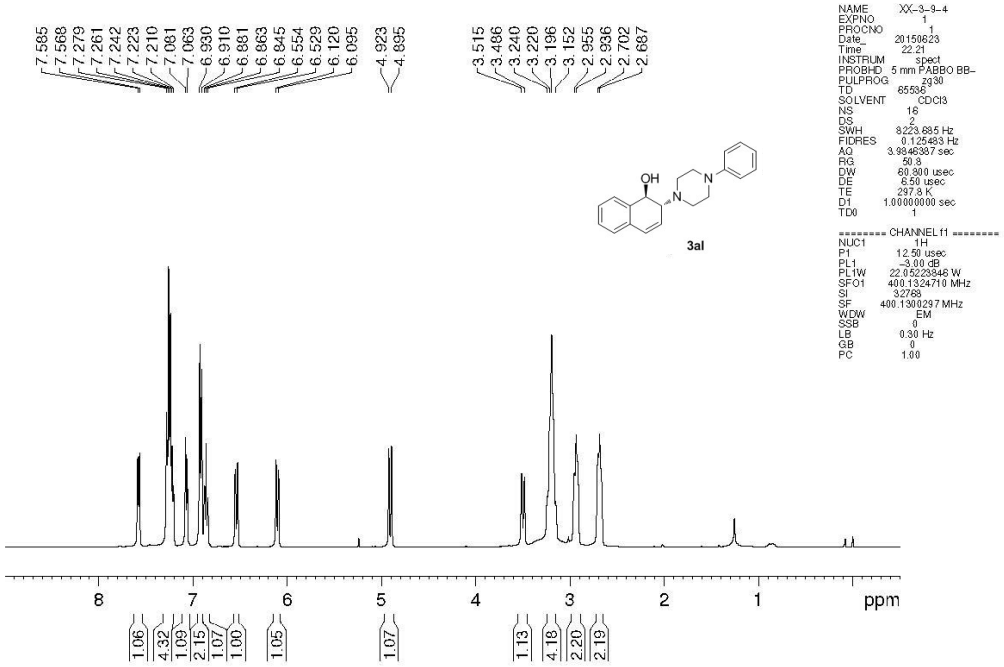
7.443
7.434
7.201
7.189
7.167
7.050
7.040
7.031
6.878
6.857
6.708
6.690
6.671
6.526
6.502
5.887
5.882
5.863
5.858
5.035
5.011
4.597
4.574
3.293
3.276
3.258
3.241
2.288
1.084
1.066
1.049
3aj
NAME XX-3-7-1
EXPNO 1
PROCNO 1
Date_ 20150619
Time 20.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 161
DW 60.800 usec
DE 6.50 usec
TE 297.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -3.00 dB
PL1W 22.05223848 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300493 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8
7
6
5
4
3
2
1
ppm
1.00
4.15
1.08
2.05
1.02
1.00
1.00
0.99
1.00
2.00
0.93
3.05


7.496
7.478
7.228
7.218
7.198
7.172
7.154
7.136
7.112
7.093
6.971
6.953
6.464
6.439
6.034
6.010
4.864
4.834
3.771
3.737
3.517
3.484
2.238
OH
N
3ak
NAME XX-3-11-4
EXPNO 1
PROCNO 1
Date_ 20150622
Time 20.09
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 45.2
DW 60.800 usec
DE 6.50 usec
TE 297.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -3.00 dB
PL1W 22.05223848 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300642 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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 ppm
1.04 4.30 3.34 1.02 1.00 1.02 1.05 1.08 2.16 3.12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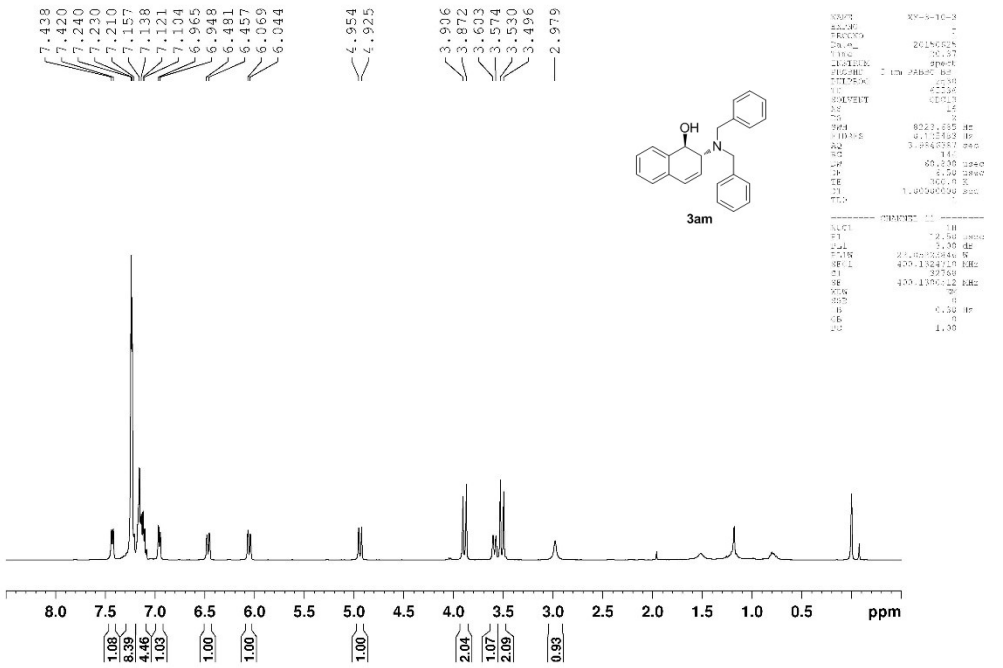
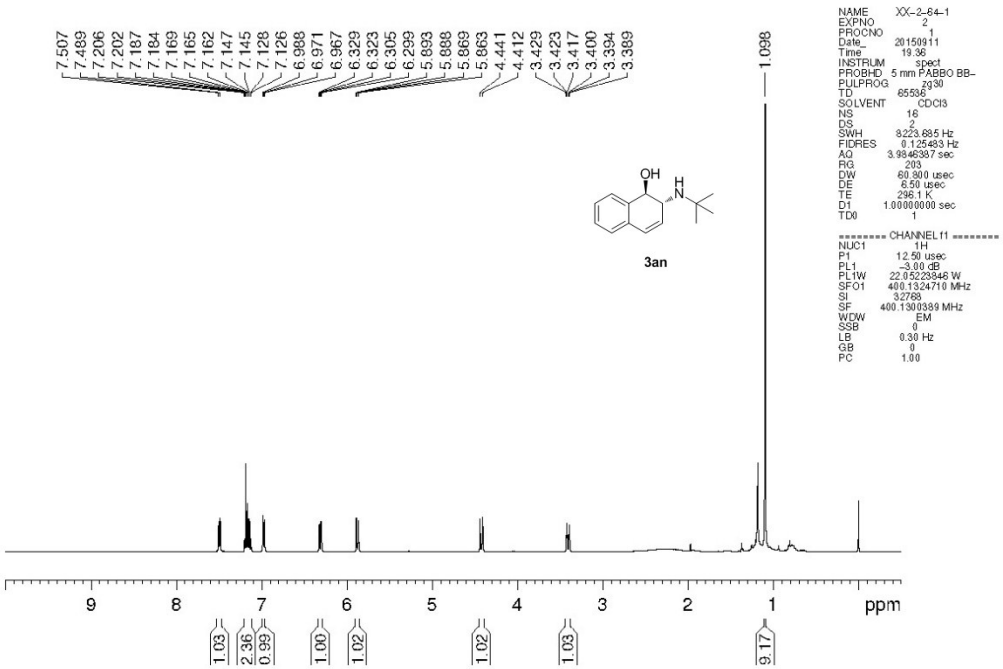
7.261
7.242
7.223
7.210
7.081
7.063
6.930
6.910
6.881
6.863
6.845
6.554
6.529
6.120
6.095
4.923
4.895
3.515
3.486
3.240
3.220
3.196
3.152
2.955
2.936
2.702
2.687
OH
N
N
3al
NAME XX-3-9-4
EXPNO 1
PROCNO 1
Date_ 20150623
Time 22.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 50.3
DW 60.800 usec
DE 6.50 usec
TE 297.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -3.00 dB
PL1W 22.05223846 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300297 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8 7 6 5 4 3 2 1 ppm
1.06 4.32 1.09 2.15 1.07 1.00 1.05 1.07 1.13 4.18 2.20 2.19


7.438
7.420
7.240
7.230
7.210
7.157
7.138
7.121
7.104
6.965
6.948
6.481
6.457
6.069
6.044
4.954
4.925
3.906
3.872
3.603
3.574
3.530
3.496
2.979
OH
N
3am
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
ppm
1.08
8.39
4.46
1.03
1.00
1.00
1.00
2.04
1.07
2.09
0.93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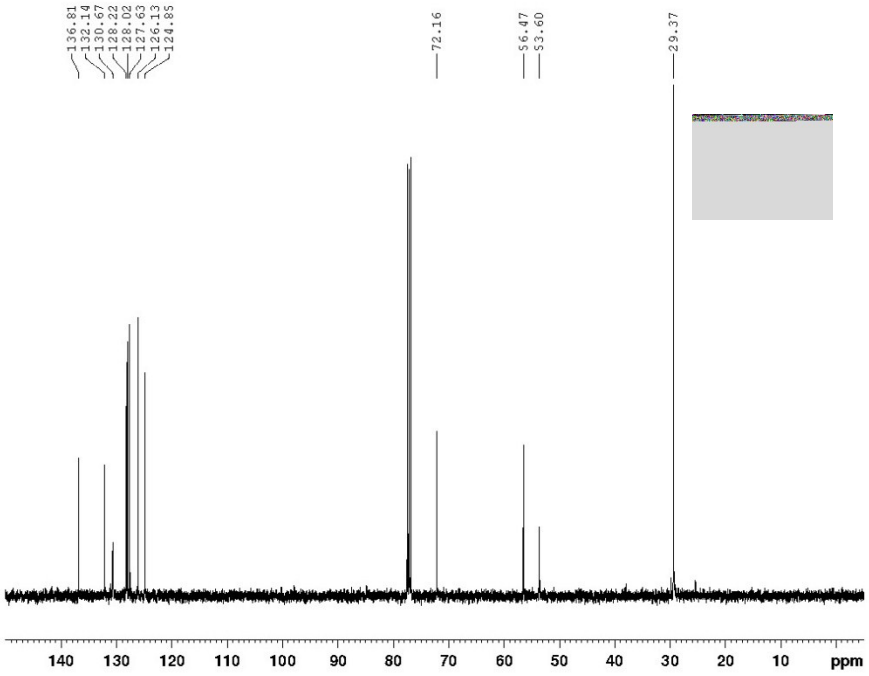
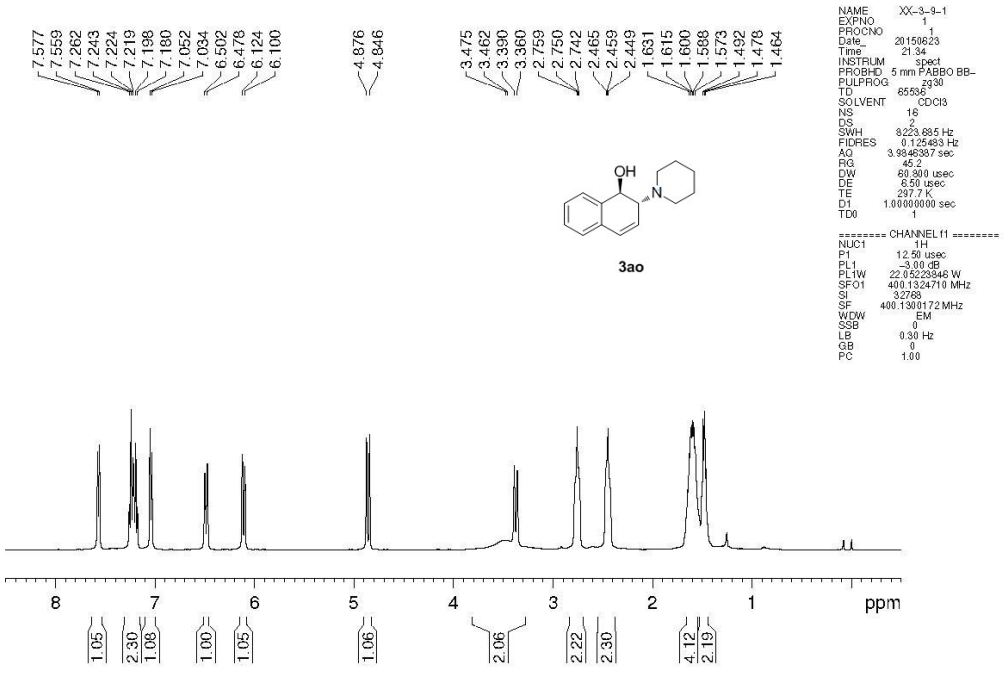
7.206
7.202
7.187
7.184
7.169
7.165
7.162
7.147
7.145
7.128
7.126
6.988
6.971
6.967
6.329
6.323
6.305
6.299
5.893
5.888
5.869
5.863
4.441
4.412
3.429
3.423
3.417
3.400
3.394
3.389
1.098
OH
H
N
3an
NAME XX-2-64-1
EXPNO 2
PROCNO 1
Date_ 20150911
Time 19.36
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846397 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 296.1 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 12.50 usec
PL1 -3.00 dB
PL1W 22.05223846 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300389 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm
1.03
2.36
0.99
1.00
1.02
1.02
1.03
9.17


136.81
132.14
130.67
128.22
128.02
127.63
126.13
124.85
72.16
56.47
53.60
29.37
140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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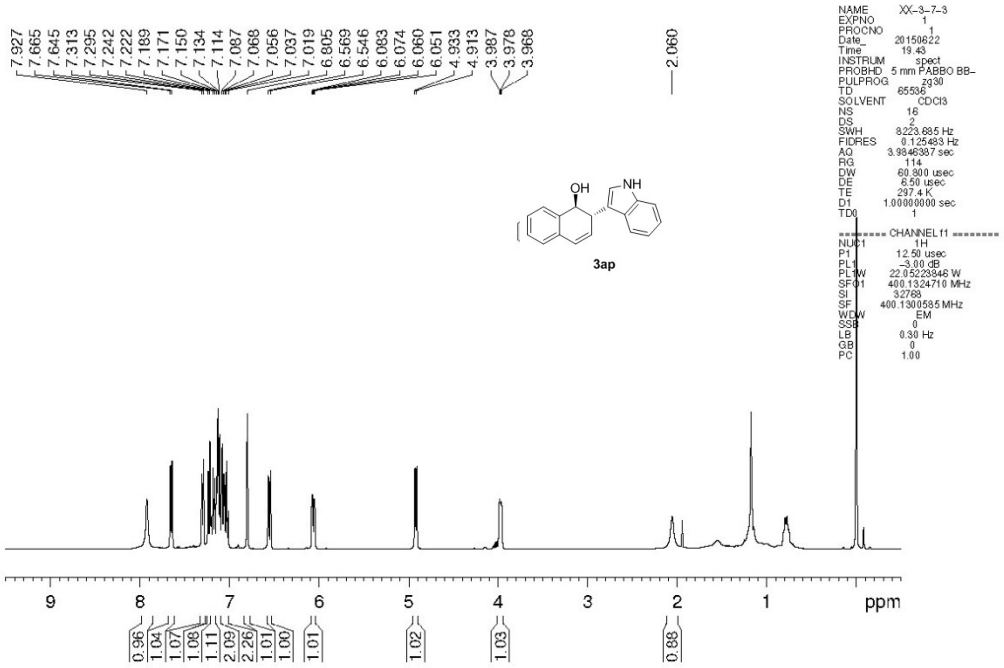
7.243
7.224
7.219
7.198
7.180
7.052
7.034
6.502
6.478
6.124
6.100
4.876
4.846
3.475
3.462
3.390
3.360
2.759
2.750
2.742
2.465
2.459
2.449
1.631
1.615
1.600
1.588
1.573
1.492
1.478
1.464
OH
N
3ao
NAME XX-3-9-1
EXPNO 1
PROCNO 1
Date_ 20150623
Time 21.34
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 45.2
DW 60.900 usec
DE 6.50 usec
TE 297.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -3.00 dB
PL1W 22.6223846 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300172 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8 7 6 5 4 3 2 1 ppm
1.05
2.30
1.08
1.00
1.05
1.06
2.06
2.22
2.30
4.12
2.19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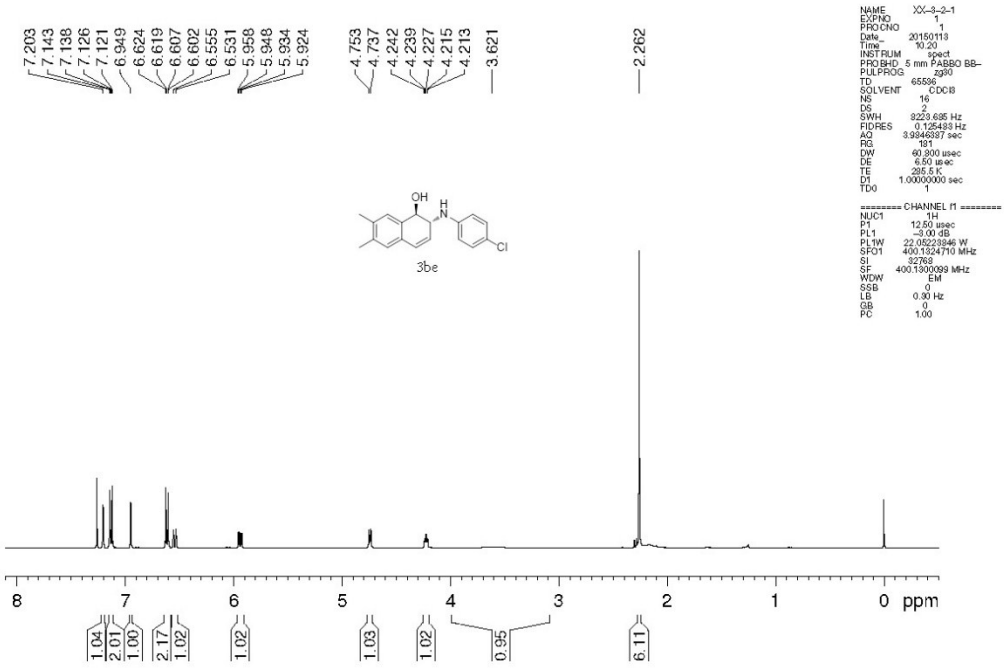
7.242
7.222
7.189
7.171
7.150
7.134
7.114
7.087
7.068
7.056
7.037
7.019
6.805
6.569
6.546
6.083
6.074
6.060
6.051
4.933
4.913
3.987
3.978
3.968
2.060
OH
NH
3ap
NAME XX-3-7-3
EXPNO 1
PROCNO 1
Date_ 20150622
Time 19.43
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 114
DW 60.800 usec
DE 6.50 usec
TE 297.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -3.00 dB
PL1W 22.05223846 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300585 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm
0.96
1.04
1.07
1.08
1.11
2.09
2.26
1.01
1.00
1.01
1.02
1.03
0.88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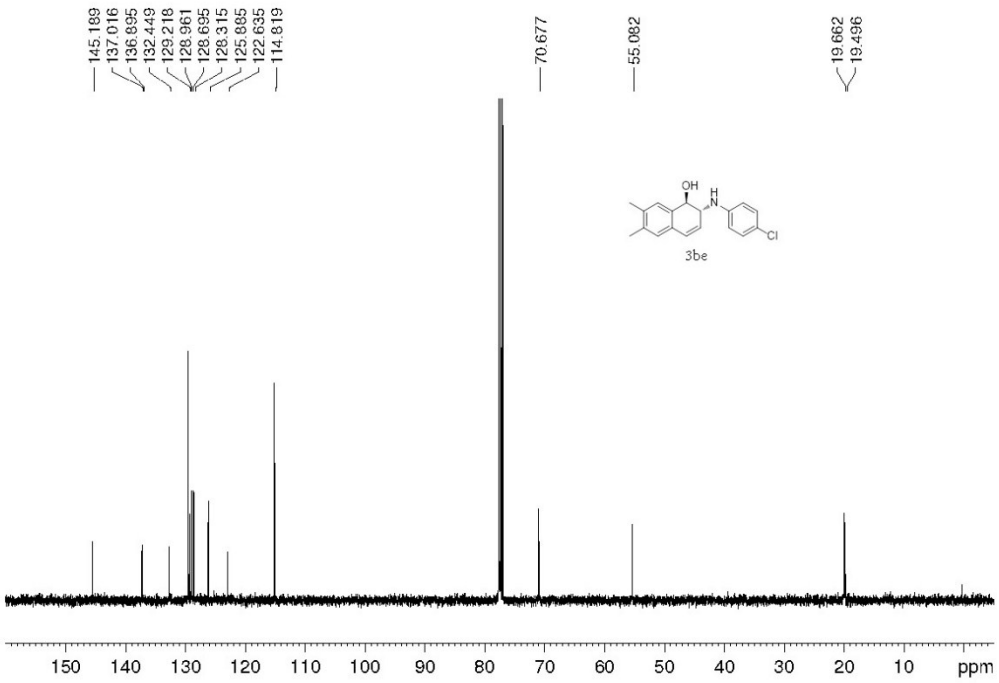
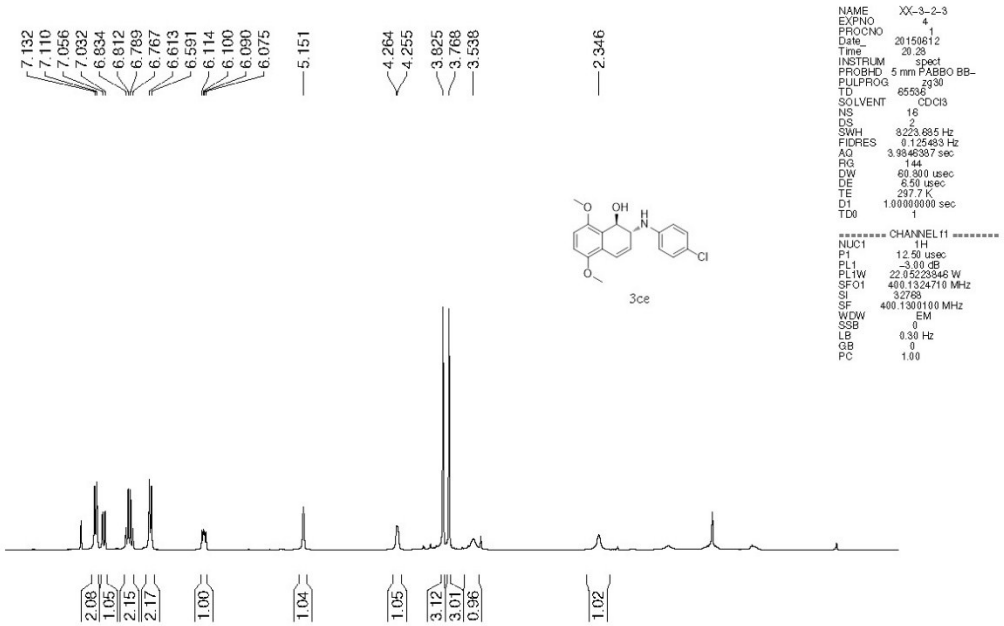
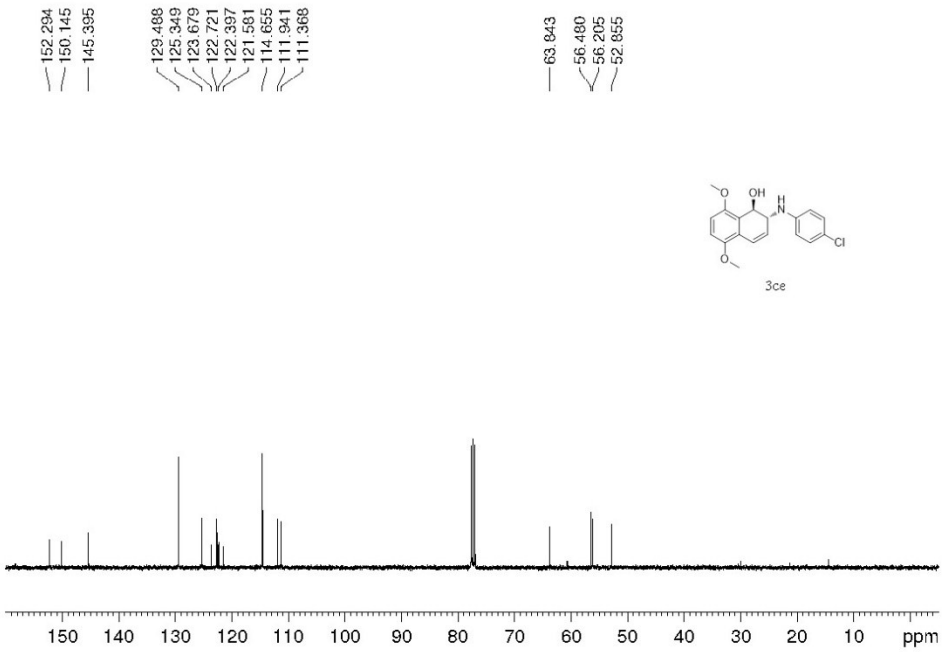
7.126
7.121
6.949
6.624
6.619
6.607
6.602
6.555
6.531
5.958
5.948
5.934
5.924
4.753
4.737
4.242
4.239
4.227
4.215
4.213
3.621
2.262
OH
H
N
Cl
3be
NAME XX-3-2-1
EXPNO 1
PROCNO 1
Date_ 20150113
Time 10.20
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 181
DW 60.800 usec
DE 6.50 usec
TE 295.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -3.00 dB
PL1W 22.05223846 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8
7
6
5
4
3
2
1
0 ppm
1.04
2.01
1.00
2.17
1.02
1.02
1.03
1.02
0.95
6.11

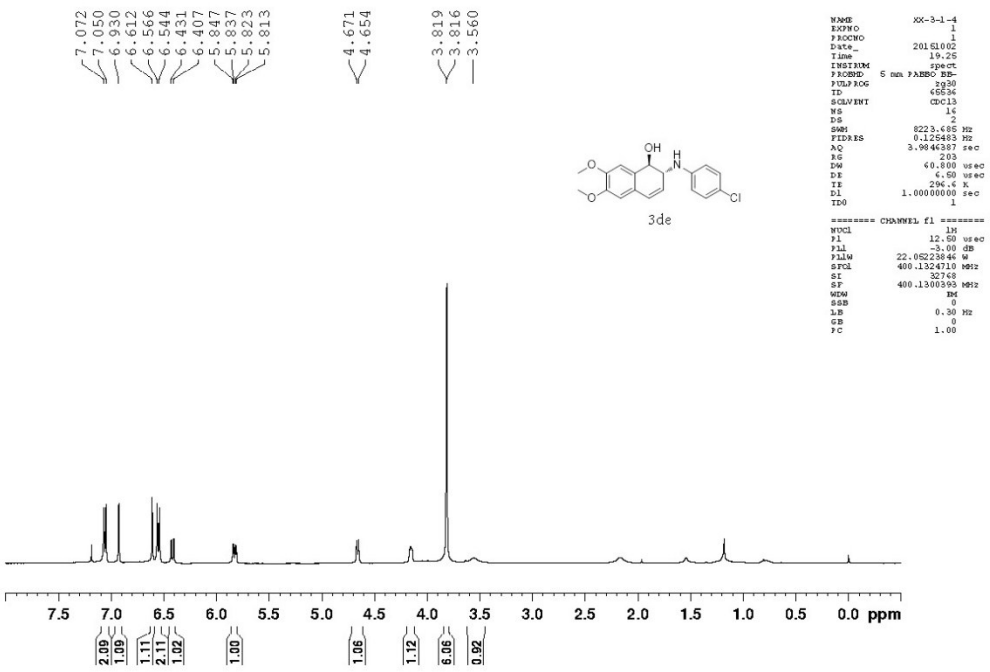




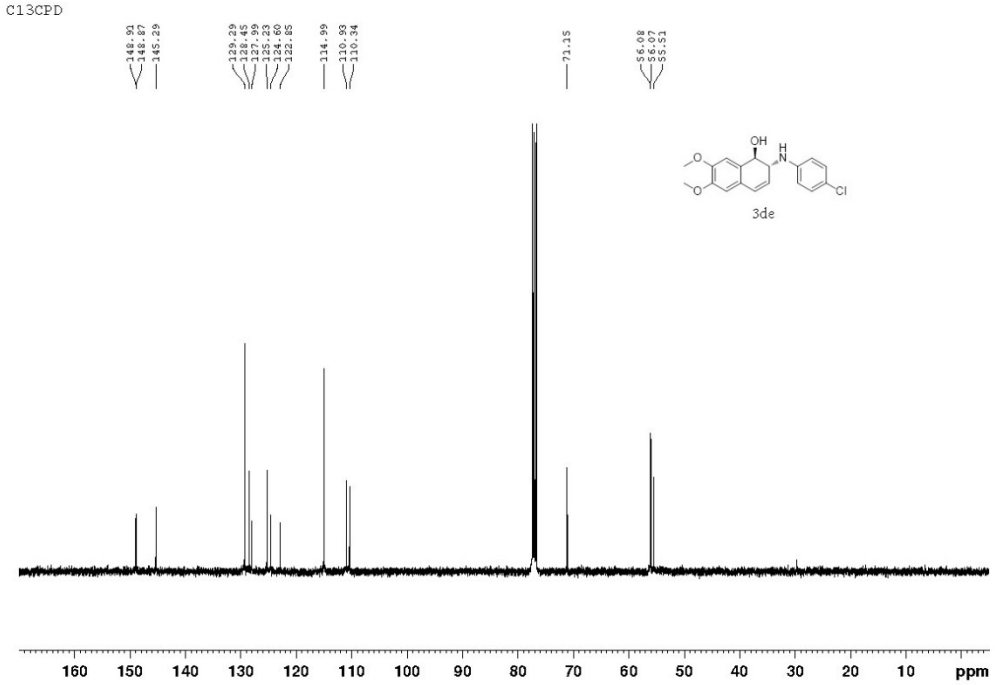
NAME XX-3-2-3
EXPNO 4
PROCNO 1
Date_ 20150612
Time 20.28
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125493 Hz
AQ 3.9846387 sec
RG 144
DW 60.800 usec
DE 6.50 usec
TE 297.7 K
D1 1.00000000 sec
TD0 1

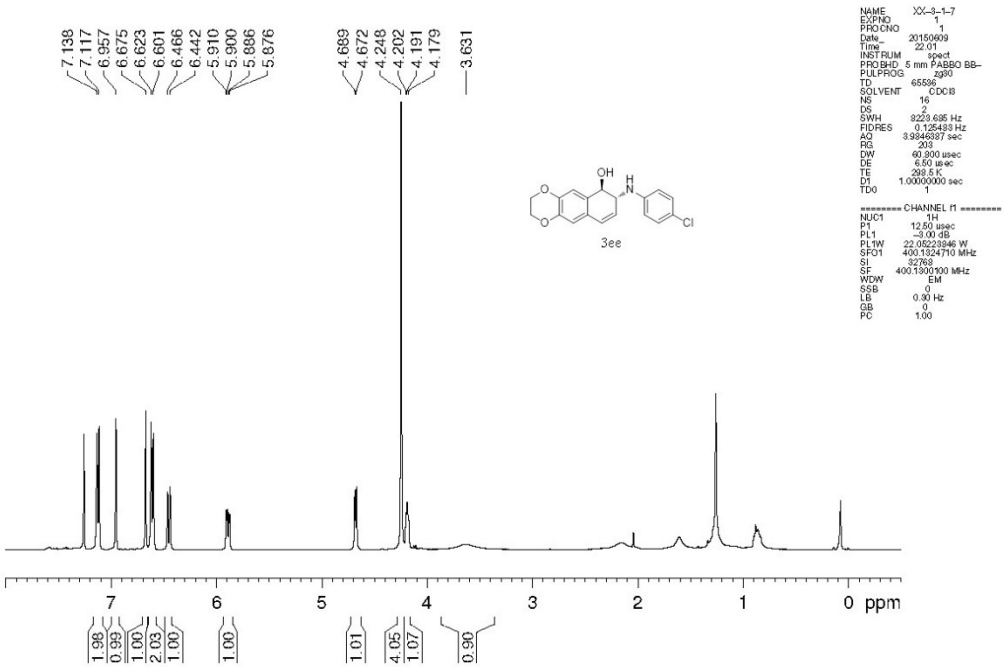
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -3.00 dB
PL1W 22.05223846 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300100 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

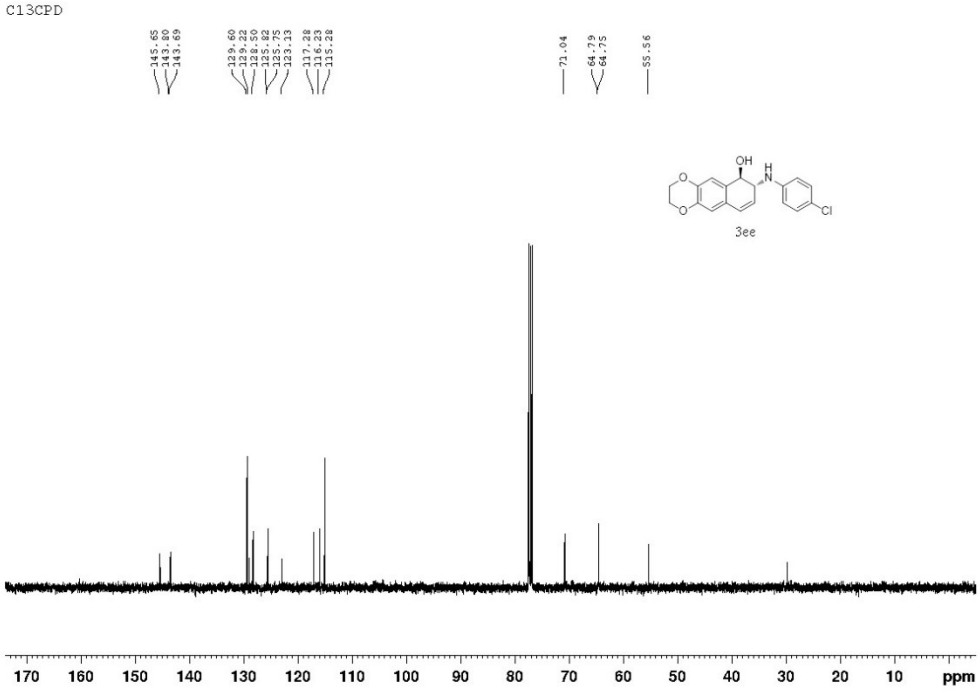


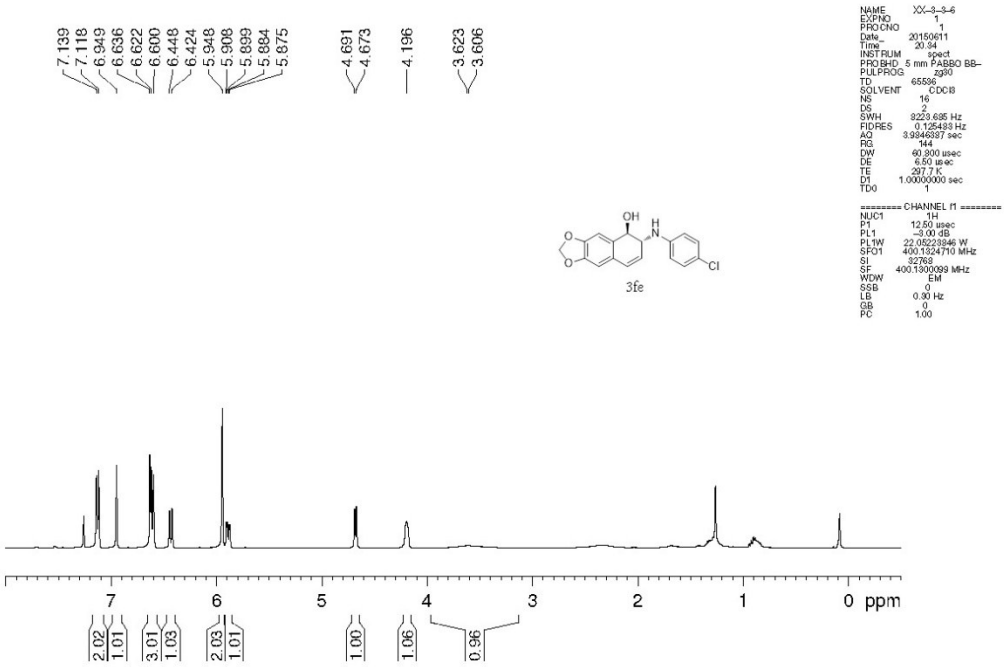


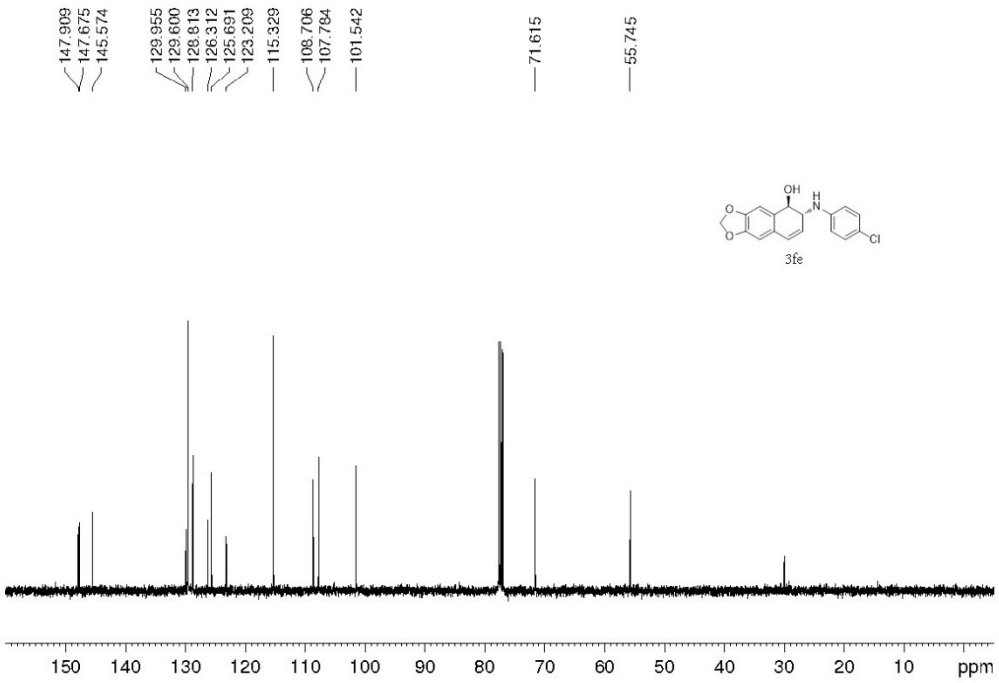

```
NAME                 XX-3-1-4
EXPNO                       1
PROCNO                      1
Date_             20161002
Time                    19.26
INSTRUM                spect
PROBHD     5 mm PABBO BB-
PULPROG                zg30
TD                     65536
SOLVENT                CDCl3
NS                         16
DS                          2
SWH                 8223.685 Hz
FIDRES              0.125483 Hz
AQ                  3.9844387 sec
RG                        203
DW                   60.800 usec
DE                    6.50 usec
TE                   296.4 K
D1                1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                    12.50 usec
PL1                   -3.00 dB
PLW1             22.05223846 W
SFO1             400.1324710 MHz
SI                      32768
SF               400.1300393 MHz
WDW                        EM
SSB                         0
LB                     0.30 Hz
GB                          0
PC                       1.00
```

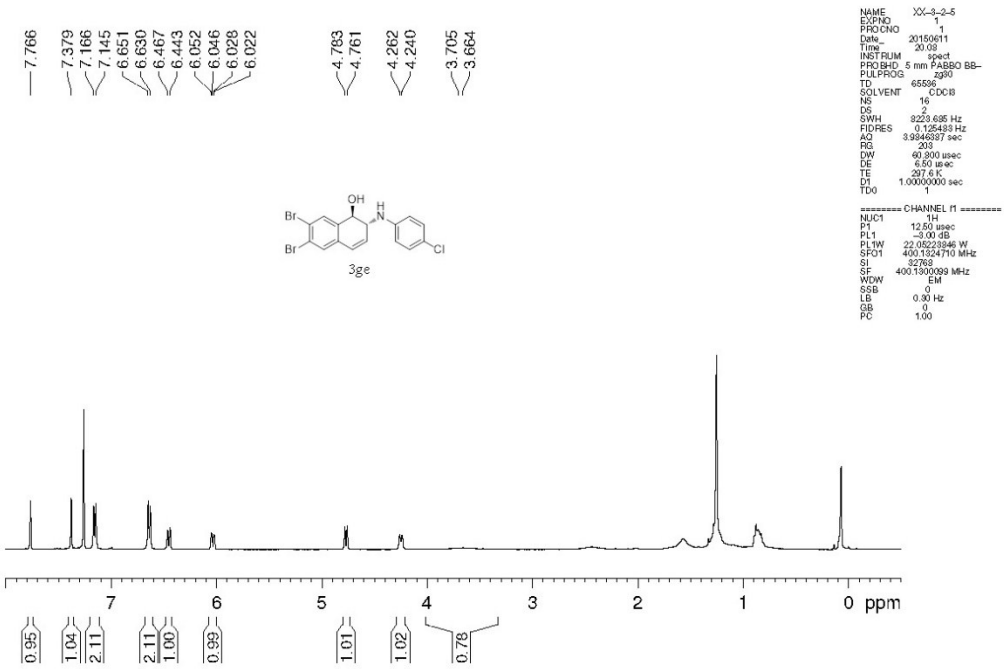

C13CPD
148.90
148.87
145.29
129.29
128.45
127.99
125.23
124.60
122.85
114.99
110.93
110.34
71.15
56.08
56.07
55.51
3de
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

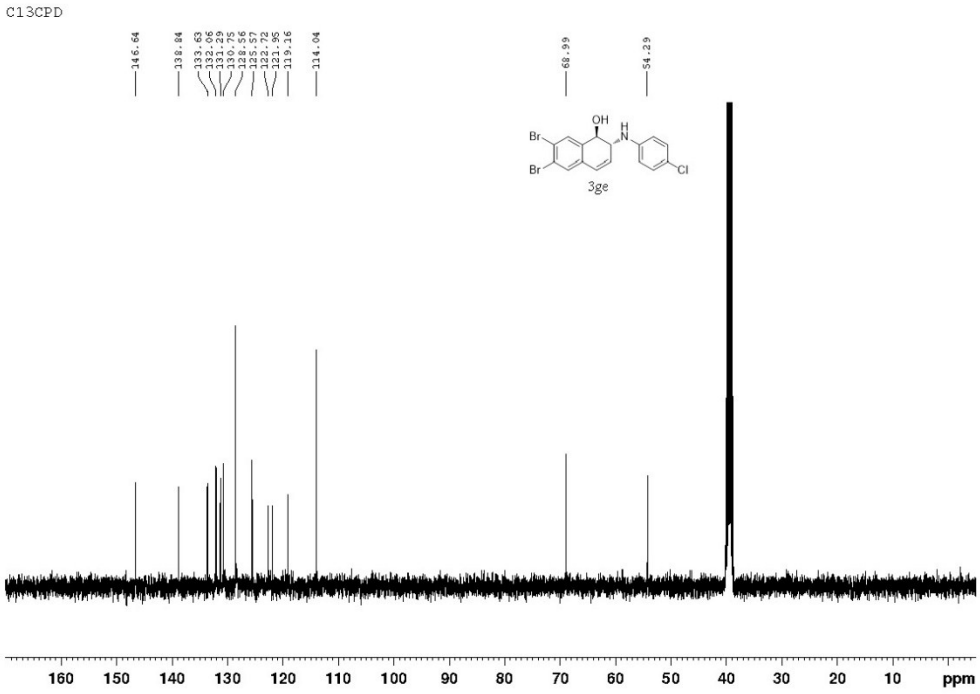

7.138
7.117
6.957
6.675
6.623
6.601
6.466
6.442
5.910
5.900
5.886
5.876
4.689
4.672
4.248
4.202
4.191
4.179
3.631
3ee
NAME XX-3-1-7
EXPNO 1
PROCNO 1
Date_ 20150609
Time 22.01
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 9223.685 Hz
FIDRES 0.125493 Hz
AQ 3.9846387 sec
RG 203
DW 60.900 usec
DE 6.50 usec
TE 298.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -3.00 dB
PL1W 22.05223846 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300100 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7 6 5 4 3 2 1 0 ppm
1.98
0.99
1.00
2.03
1.00
1.00
1.01
4.05
1.07
0.90


C13CPD
145.65
143.80
143.69
129.60
129.22
128.60
125.82
125.75
123.13
117.28
116.23
115.28
71.04
64.79
64.75
55.56
OH
H
N
O
O
Cl
3ee
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

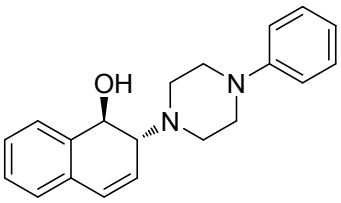

7.139
7.118
6.949
6.636
6.622
6.600
6.448
6.424
5.948
5.908
5.899
5.884
5.875
4.691
4.673
4.196
3.623
3.606
NAME XX-3-9-6
EXPNO 1
PROCNO 1
Date_ 20150611
Time 20.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 9223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9946897 sec
RG 144
DW 60.800 usec
DE 6.50 usec
TE 297.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -3.00 dB
PL1W 22.05223846 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
H
N
O
O
Cl
3fe
7 6 5 4 3 2 1 0 ppm
2.02
1.01
3.01
1.03
2.03
1.01
1.00
1.06
0.96


147.909
147.675
145.574
129.955
129.600
128.813
126.312
125.691
123.209
115.329
108.706
107.784
101.542
71.615
55.745
3fe
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


7.766
7.379
7.166
7.145
6.651
6.630
6.467
6.443
6.052
6.046
6.028
6.022
4.783
4.761
4.262
4.240
3.705
3.664
3ge
NAME XX-3-2-5
EXPNO 1
PROCNO 1
Date_ 20150611
Time 20.08
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 9223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9946987 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 297.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -3.00 dB
PL1W 22.05223946 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7 6 5 4 3 2 1 0 ppm
0.95
1.04
2.11
2.11
1.00
0.93
1.01
1.02
0.78


C13CPD
146.64
138.84
133.63
132.06
131.29
130.75
128.56
128.57
122.72
121.95
119.16
114.04
68.99
54.29
Br
Br
OH
H
N
Cl
3ge
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

C: X-ray crystallography of (1*R*,2*R*)-2-(4-phenylpiperazin-1-yl) -1,2-dihydronaphthalen-1-ol (3al)



3al