Supporting Information

A highly concise and practical route to clavaminols, sphinganine and (+)-spisulosine *via* Indium mediated allylation of α-hydrazino aldehyde and a theoretical insight into the stereochemical aspect of the reaction

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Experimental Section

General Methods: All reactions were carried out under argon or nitrogen in oven-dried glassware using standard gas-light syringes, cannulas and septa. Solvents and reagents were purified and dried by standard methods prior to use. Optical rotations were measured at room temperature. IR spectra were recorded on an FT-IR instrument. ^{1}H NMR spectra were recorded on 200 MHz, 300 MHz and 500 MHz and are reported in parts per million (δ) downfield relative to $CDCl_3$ as internal standard and ^{13}C NMR spectra were recorded at 50 MHz, 75 MHz and 125 MHz and assigned in parts per million (δ) relative to $CDCl_3$. Column chromatography was performed on silica gel (100-200 and 230-400 mesh) using a mixture of petroleum ether and ethyl acetate as the eluent.

Theoretical Study

All the calculation are done using Gaussian09 .The Cartesian coordinates, IR frequencies and total energy with ZPE correction for all the optimized saddle points are listed below.

TS Anti

Cartesian coordinate

C	-0.13465200	0.57518600	0.17961700
C	1.15279300	-0.14603300	0.57466300
N	-1.08786200	-0.33585100	-0.44488700
H	-0.60398000	0.90714800	1.11526400
O	2.26232700	0.40216400	0.51112000
H	1.03228000	-1.14212200	1.01039900
C	-0.75976400	-1.24126600	-1.45231400
O	-1.58803800	-1.89649000	-2.09697000
O	0.59285000	-1.30768600	-1.60688400
C	1.16483300	-2.27939600	-2.61221000
C	1.49181500	-3.60154500	-1.97076600
C	0.59598200	-4.68149900	-2.05846800
C	0.90577700	-5.90811800	-1.45884600
C	2.11366000	-6.06531200	-0.76611200
C	3.01386800	-4.99426200	-0.67756100
C	2.70491500	-3.76960900	-1.27942700
In	4.80535200	-0.14936300	0.86578100
C	4.66267200	1.02005400	2.91117900
C	5.74167900	2.01960200	2.87836700
C	7.03417400	1.86191700	3.26130300
N	-2.43993000	-0.09016400	-0.23210900
C	-8.41005500	1.11501300	2.01441100
C	-9.29659500	0.58385300	1.06769000
C	-8.91729300	-0.52615200	0.30069800
C	-7.65338200	-1.10083000	0.47859600
C	-6.75721500	-0.57211500	1.42317800
C	-7.14682500	0.53870600	2.19083700
C	-5.39365900	-1.17824900	1.60060100
O	-4.46341100	-0.46880900	0.65427800
C	-3.14810200	-0.84279900	0.67620400
O	-2.66776000	-1.71884100	1.41344200
O	0.08986900	1.70941600	-0.68446800
C	0.28645800	4.10523300	-1.01000600
C	-0.95054200	4.68143300	-1.34826000
C	-1.02428300	5.68912000	-2.31655500
C	0.14124500	6.13136400	-2.95819500
C	1.37863700	5.56389000	-2.62660000
C	1.44930100	4.55544700	-1.65743100

C	0.36150300	3.00319200	0.01208900
H	0.43532500	-2.37503200	-3.41370300
H	2.05033100	-1.75687700	-2.96359500
H	-0.33899300	-4.55970100	-2.59352000
H	0.21054900	-6.73623100	-1.53339300
H	2.35396100	-7.01550100	-0.30316500
H	3.95176300	-5.11486600	-0.14782500
H	3.40536200	-2.94323700	-1.21445100
H	3.65895500	1.44594200	2.98705600
H	4.80568700	0.22979500	3.65887600
H	5.47390000	2.97790400	2.42719800
H	7.76533300	2.65357600	3.13251800
H	7.38797600	0.94214500	3.72246000
H	-2.91770800	0.55304200	-0.84949400
H	-8.70224600	1.97021200	2.61271400
H	-10.27588900	1.02821300	0.93197900
H	-9.60235400	-0.94238900	-0.42875800
H	-7.36213400	-1.96171500	-0.11394600
H	-6.46315800	0.94902600	2.92650600
H	-5.00272500	-1.03760900	2.60783600
H	-5.37184500	-2.23687200	1.34358300
H	-1.85305800	4.34248900	-0.85038600
H	-1.98271800	6.12895800	-2.56747600
H	0.08541400	6.91345700	-3.70670500
H	2.28230300	5.90507700	-3.11845800
H	2.40819400	4.11766300	-1.40074300
H	-0.39693400	3.12916200	0.79162400
H	1.35035300	2.95021900	0.46905300

IR FREQENCIES

-15.7844	5.8961	9.7361
10.3780	12.4054	13.5181
14.5210	17.2456	19.4271
19.8887	28.7576	34.3008
37.3491	38.6503	42.2938
48.3150	64.5103	68.1406
69.9181	80.7926	94.0452
99.6157	122.0362	135.7915
151.9365	166.5560	186.7191
193.7524	199.1910	223.5472
239.5862	252.6497	280.5818
295.1369	329.8380	331.6750
338.4293	341.4643	350.1305
370.7740	385.5548	407.4986
417.5628	418.9122	420.0519
421.2457	468.9837	496.6367
516.3965	521.2186	534.7343

580.8659	591.4813	605.6315
619.8269	627.6694	645.4756
645.6890	646.0341	669.3491
681.2057	711.4921	713.1591
713.2919	720.6415	721.8971
743.9815	748.7461	770.5037
772.8277	788.6045	799.3925
829.2386	840.5947	847.8070
850.1796	856.0096	870.5269
871.9828	872.9990	876.7662
919.7269	922.1200	936.8201
956.8427	962.8257	964.4066
967.2613	980.5233	985.3535
987.8859	999.4724	1002.4259
1003.3986	1003.6664	1003.7010
1015.3168	1015.4301	1016.1701
1018.8703	1020.6343	1020.6356
1038.9978	1043.6731	1043.7678
1043.9731	1044.3311	1063.6487
1108.8193	1109.4207	1110.1279
1152.3040	1198.5709	1199.1130
1200.0152	1200.0646	1212.0742
1214.0582	1214.3922	1216.3270
1232.4248	1234.1342	1236.9785
1239.3951	1242.0916	1243.7271
1270.5683	1295.7066	1325.7247
1325.9912	1326.0858	1329.6594
1348.0601	1380.9852	1382.2363
1385.0227	1389.0508	1397.3357
1406.9525	1415.1566	1432.4294
1450.9195	1479.5820	1484.1256
1484.9931	1485.0200	1504.4474
1517.2276	1523.6058	1526.6523
1527.4020	1529.1249	1532.0688
1597.5014	1600.8115	1601.4903
1601.7555	1617.4816	1621.3526
1622.0040	1623.0846	1635.9718
1646.1656	2987.0760	2991.5194
3005.7019	3040.5823	3047.4373
3054.7910	3072.3329	3077.8642
3081.8850	3082.5397	3114.1081
3137.1866	3137.4182	3138.4071
3140.4033	3141.1924	3144.4722
3147.8430	3150.4023	3151.2204
3154.3555	3160.6291	3162.0272
3163.8978	3165.7705	3173.3949

3174.5034 3175.1699 3567.4902

ENERGY with ZPE correction= -480.136895 a.u.

TS Syn

Cartesian coordinate

C	1.78579000	-2.11199500	-1.75768900
C	2.32856500	-1.44013100	-3.02194500
N	0.53947400	-1.46427900	-1.25630400
H	1.49731800	-3.14055400	-1.99533700
O	1.71222200	-1.49306200	-4.09154500
H	3.29207900	-0.93315300	-2.93584500
C	0.51999100	-0.10975100	-0.90632800
O	-0.35051200	0.43084800	-0.20271600
O	1.60416000	0.53770100	-1.41584500
C	1.69009100	2.02004400	-1.26954000
C	3.10609600	2.47105400	-1.01876200
C	3.44085700	3.78095200	-1.40591100
C	4.71164700	4.29794400	-1.14013300
C	5.66965900	3.50082100	-0.49982800
C	5.34629700	2.18848900	-0.13017400
C	4.06791600	1.67183500	-0.38268800
In	-2.58774600	2.30664300	-0.36142000
C	-2.81282600	1.94785100	1.95193500
C	-3.13961600	3.21212800	2.63357900
C	-2.31523700	4.14570000	3.17399300
O	2.83003600	-2.05886700	-0.78351800
C	3.57177000	-2.51291800	1.48471300
C	3.14894600	-1.48400700	2.34500100
C	3.98878200	-1.01605100	3.36115600
C	5.26320800	-1.57592700	3.53137100
C	5.69082300	-2.60560600	2.68324000
C	4.84935400	-3.06879400	1.66327100
C	2.67014800	-2.98785300	0.37685000
N	-0.67971400	-2.05295200	-1.59190300
C	-5.54878400	-0.17529100	-0.43754400
C	-5.93094300	-0.07618900	0.90823200
C	-5.61248200	-1.10662100	1.79881200
C	-4.90342600	-2.23047900	1.34729300
C	-4.51836900	-2.33560400	0.00208000
C	-4.85555000	-1.30309300	-0.89045000
C	-3.77606600	-3.55273400	-0.50655900
O	-2.60634800	-3.14720400	-1.34671600
C	-1.44931200	-2.76549700	-0.70347200

O	-1.15785600	-3.05412000	0.46495900
H	1.29046700	2.42446400	-2.19953600
H	1.01031800	2.28388300	-0.45841400
H	2.70507200	4.39603400	-1.91393300
H	4.95279000	5.31177700	-1.43950400
H	6.65560500	3.89913900	-0.29281700
H	6.08875200	1.56611400	0.35630600
H	3.82482200	0.65675700	-0.09390100
H	-3.60734600	1.20393500	2.04582600
H	-1.84528800	1.50861200	2.21811300
H	-4.20537100	3.46066100	2.63614900
H	-2.71110700	5.07259400	3.57591300
H	-1.23582300	4.01737500	3.21545700
H	2.16111700	-1.05405300	2.21801600
H	3.65200200	-0.22208400	4.01746600
H	5.91501600	-1.21309700	4.31817700
H	6.67404300	-3.04271000	2.81424900
H	5.18343700	-3.86672100	1.00820200
H	1.62173300	-2.97144100	0.68224800
H	2.93858500	-3.99172700	0.03695200
H	-1.07779800	-1.81511800	-2.49246500
H	-5.79579800	0.61606200	-1.13494000
H	-6.46180500	0.80089300	1.26199100
H	-5.90176300	-1.03226200	2.84081000
H	-4.65006100	-3.01917900	2.04660300
H	-4.56883400	-1.38258600	-1.93284500
H	-3.42499800	-4.18925300	0.30458900
H	-4.37352600	-4.14129200	-1.20045400

IR FREQENCIES

35.5327	-8.4193	4.4384
8.5573	10.8908	15.2143
17.0574	19.8405	23.2171
25.2930	26.5817	33.9859
37.7917	38.6926	43.5786
52.8829	57.9571	62.7261
70.7222	80.1473	80.8512
95.2518	123.0401	140.8466
157.7402	163.8295	180.0704
184.4664	200.0632	214.0012
227.5464	240.8424	260.7133
278.2083	299.6201	328.8424
333.1913	339.9064	365.6039
383.1091	402.6894	412.3143

416.6153	419.4535	421.1749
434.1594	459.3662	484.2412
502.4472	524.9703	546.1703
580.6394	596.2229	602.5876
627.1805	632.8909	641.3657
644.4696	646.4655	646.7027
664.4945	676.3699	698.2937
712.2320	714.2071	717.2946
736.2886	756.4961	758.0660
765.0474	775.9140	804.2889
818.1862	835.0485	839.3797
841.0067	868.1625	870.4401
873.5872	895.9244	902.6888
924.1026	926.2176	935.9334
941.7095	958.3065	970.9466
976.3565	987.7263	992.6618
995.9829	1001.8358	1005.4596
1012.6924	1013.7857	1014.2625
1015.5962	1017.5820	1018.0425
1018.5003	1024.5602	1038.4305
1042.7563	1043.0092	1044.1408
1048.0737	1053.5877	1081.4969
1104.1425	1105.2859	1110.5959
1125.6934	1173.6684	1198.9875
1199.0980	1199.9751	1206.3418
1212.7679	1213.7342	1215.2707
1217.0387	1221.3457	1230.8063
1239.3875	1246.7576	1270.8102
1282.2760	1297.1506	1315.3896
1322.3546	1327.1750	1329.5877
1335.3353	1370.0339	1370.8234
1383.1316	1384.3542	1400.5165
1402.5162	1423.6988	1434.3284
1456.5105	1481.8989	1482.6716
1483.2581	1484.6478	1502.7287
1516.2589	1526.7948	1527.0872
1528.7796	1531.1745	1532.6318
1594.9595	1599.8769	1601.4622
1602.6271	1603.9833	1619.1094
1622.7466	1625.2919	1646.7963
1661.7494	3005.5425	3015.7458
3032.0496	3032.5687	3048.8372
3055.7825	3065.8824	3074.8152
3080.0925	3083.3988	3096.9477
3122.1131	3134.6468	3136.3433
3139.2673	3140.9662	3145.2797

3148.5989	3149.6378	3154.9414
3154.9735	3161.1196	3165.6493
3166.9375	3169.3046	3173.0459
3175.1743	3177.8168	3542.8638

ENERGY with ZPE correction= -480.122491 a.u.

Anti Product

Cartesian coordinate

C	-2.80995200	-1.50264800	-0.67716500
C	-3.71168700	-1.82888900	0.49551200
C	-5.15880300	-1.27745000	0.44030500
C	-5.92283200	-1.73426100	-0.77926500
C	-7.04506800	-2.46276900	-0.75211100
O	-3.13319400	-1.34966400	1.75788000
N	-1.85956900	-0.44449800	-0.63830200
C	1.93811700	4.63342100	1.22679900
C	2.50095900	5.09451900	0.02911400
C	1.80996600	4.92563000	-1.17872000
C	0.56036100	4.29547800	-1.18739300
C	-0.00972400	3.82721000	0.00895900
C	0.68820600	4.00296100	1.21582600
C	-1.34579800	3.13992800	-0.00534600
O	-1.09413300	1.67290400	-0.23048600
C	-2.19366900	0.88296200	-0.39107600
O	-3.36637100	1.29173000	-0.35352300
N	-0.50964600	-0.79543200	-0.78981600
C	5.95077400	-1.38667700	-0.03566100
C	6.29463600	-2.52848800	-0.77192700
C	5.36309200	-3.56331000	-0.93314800
C	4.09044200	-3.45443600	-0.36113000
C	3.73644300	-2.31103000	0.37551500
C	4.67715400	-1.27926600	0.53499500
C	2.36487900	-2.18737700	0.97492600
O	1.47712300	-1.55653600	-0.06941500
C	0.17851200	-1.32838900	0.26648400
O	-0.32148900	-1.59016300	1.38319200
H	-2.58356900	-2.26504500	-1.41264100
H	-3.77612200	-2.92572900	0.52691700
H	-5.66790200	-1.60871800	1.35084100
H	-5.09309900	-0.18659000	0.45827900
H	-5.51391100	-1.42439900	-1.74023700
H	-7.56119700	-2.75174400	-1.66184800
H	-7.49113900	-2.79140000	0.18305200
H	-2.15196200	-1.47742700	1.74852400

H	2.46788300	4.76660000	2.16291500
H	3.46752300	5.58503200	0.03729000
H	2.24037700	5.28558200	-2.10602600
H	0.02451900	4.16918500	-2.12231000
H	0.25169500	3.64979400	2.14409300
H	-1.87394200	3.23416200	0.94298600
H	-1.98496200	3.48830200	-0.81592600
H	-0.04134700	-0.56270200	-1.65463500
H	6.67057000	-0.58701200	0.09489800
H	7.28141100	-2.61337700	-1.21230300
H	5.62786200	-4.44967100	-1.49784100
H	3.37155600	-4.25760000	-0.48388500
H	4.41295200	-0.39573600	1.10634500
H	1.92814400	-3.15351700	1.22557300
H	2.34762200	-1.53738300	1.84861100

IR FREQENCIES

11.4581	16.2475	20.7989
25.8795	27.6355	30.9277
42.3834	52.8453	61.4403
69.4139	83.3352	86.2926
101.4400	118.9462	121.9076
136.5403	150.4859	190.4759
200.3221	230.8042	245.9195
253.7542	298.8409	333.2117
341.2371	344.2254	375.4183
379.3519	399.6064	405.6091
420.8752	421.3210	456.5167
484.2334	497.1188	515.1844
537.4239	559.9726	582.5675
589.8863	617.3076	645.8310
646.0932	653.3463	658.2100
690.2565	713.8761	715.7634
723.7074	741.7566	745.5612
772.4259	785.6545	804.4975
840.6643	852.4337	870.7375
871.2123	876.9150	892.6577
919.7388	938.9979	960.4839
962.2584	972.4710	978.7789
979.4046	983.6516	990.5380
1002.2251	1003.4185	1010.5917
1016.0667	1016.5714	1018.6696
1020.2244	1043.9860	1044.4161
1045.3372	1069.3351	1108.8222
1109.7747	1127.1044	1158.8073
1199.2541	1200.4954	1207.9993
1213.7715	1215.8382	1238.4389

1241.0038	1242.0766	1243.1320
1244.6202	1266.9250	1301.3212
1313.8281	1325.8403	1325.9501
1335.0030	1347.1508	1371.5629
1382.3349	1384.0768	1397.2728
1407.3297	1412.9937	1426.3362
1471.8772	1484.7260	1485.1980
1500.1252	1507.5148	1522.0384
1522.1764	1528.8076	1529.5122
1589.2695	1601.8311	1601.8365
1608.2831	1623.1981	1623.2874
1669.2731	2950.8802	3014.6981
3056.3994	3058.3815	3069.7693
3090.4676	3100.3713	3115.8603
3119.2893	3138.6272	3138.9528
3141.6029	3142.0272	3151.5106
3151.7105	3162.1317	3162.4661
3169.4164	3174.5697	3174.7792
3178.2454	3399.6107	3580.8245

ENERGY with ZPE correction= -232.094295 a.u.

Syn Product

Cartesian coordinate

C	-3.36448600	-1.48722500	-0.89205600
C	-4.26625600	-0.29531400	-0.86897700
C	-4.43369500	0.36662900	0.52857200
C	-5.51566100	1.42009000	0.54965600
C	-6.59246500	1.38756900	1.34369700
O	-3.90440000	0.72832600	-1.88919100
N	-1.28315800	-0.23492500	-0.70120000
C	2.12374400	5.32382400	-0.52464300
C	3.43731100	4.94785500	-0.83635700
C	3.89411100	3.66453600	-0.50591000
C	3.03839700	2.76000900	0.13281000
C	1.71846000	3.12809800	0.44556800
C	1.26888000	4.41715000	0.11317900
C	0.79610900	2.14993200	1.11571300
O	0.13580300	1.34138800	0.02953700
C	-0.73592200	0.36770600	0.42089900
O	-1.01511200	0.08423000	1.59418700
N	-1.99052900	-1.44245400	-0.56487300
C	4.22114100	-2.23224300	1.39920800
C	4.99626300	-2.21384600	0.23168000
C	4.44700600	-2.65669500	-0.97940900

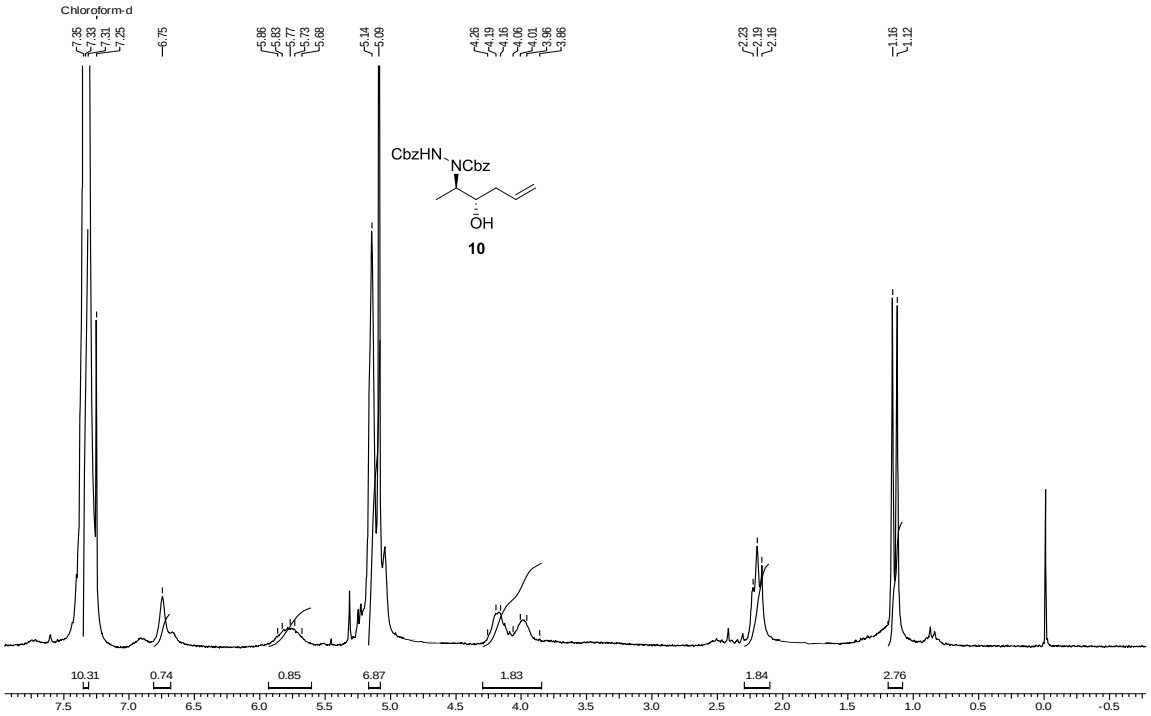
C	3.12487500	-3.11380300	-1.02214600
C	2.33965400	-3.13124400	0.14328800
C	2.89897700	-2.68930900	1.35435900
C	0.91522500	-3.60615300	0.09280200
O	0.05693400	-2.41012900	-0.22251100
C	-1.28549900	-2.62113600	-0.30417000
O	-1.84320300	-3.72521100	-0.17163000
H	-3.74964100	-2.48306800	-1.04929500
H	-5.24337700	-0.64116600	-1.20504800
H	-4.66427100	-0.42146400	1.25368200
H	-3.47556800	0.80407500	0.83130300
H	-5.38372300	2.24816500	-0.14307100
H	-7.34149900	2.17232400	1.32125000
H	-6.76207300	0.57698500	2.04772100
H	-3.03101600	1.11178000	-1.66865400
H	-0.82079700	-0.08502200	-1.59172500
H	1.76864400	6.31679400	-0.77483200
H	4.10041500	5.64988600	-1.32859100
H	4.91084900	3.37223700	-0.74158900
H	3.39403700	1.76810300	0.39069600
H	0.25303200	4.71055200	0.35617700
H	1.32564200	1.44976300	1.76087500
H	0.00434700	2.64213900	1.67954700
H	4.64492800	-1.89554000	2.33823200
H	6.02088500	-1.86193800	0.26623900
H	5.04564800	-2.64874800	-1.88284100
H	2.70163500	-3.45964000	-1.95932700
H	2.30079900	-2.70557900	2.25921400
H	0.74626900	-4.34043500	-0.69415800
H	0.57345800	-4.00597000	1.04699400

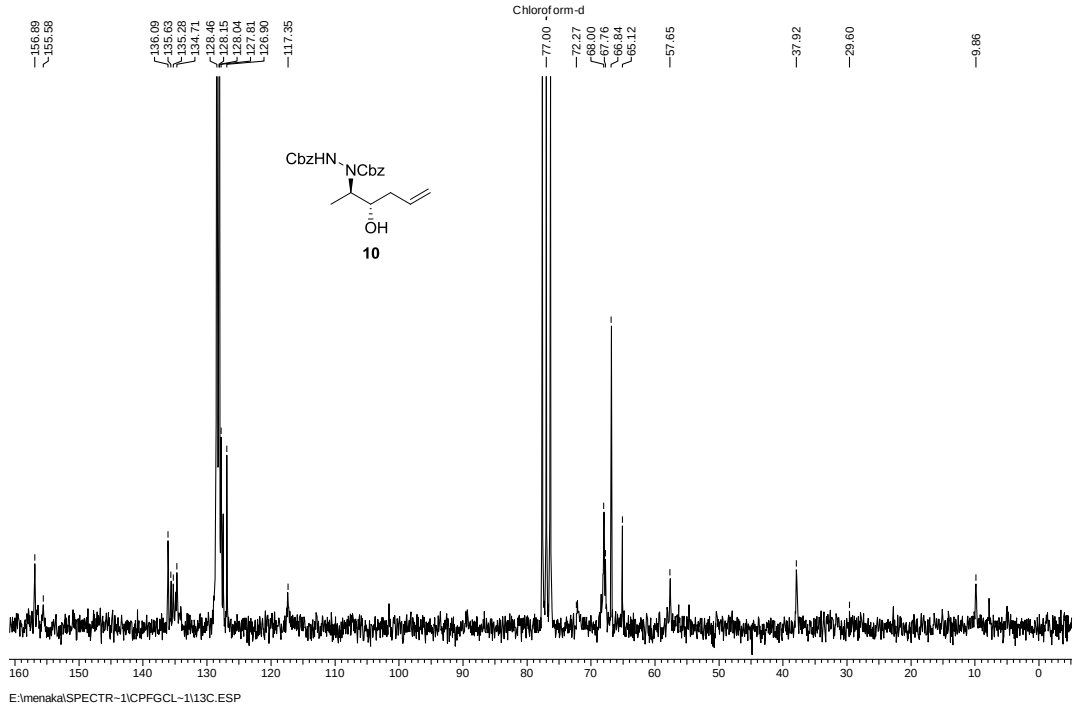
IR FREQENCIES

7.0729	12.9507	15.5601
20.5673	29.9287	32.6133
41.6832	50.5094	55.2998
67.5081	81.3251	84.8072
97.1032	104.2288	126.2964
128.6177	149.7481	183.9829
195.9103	218.0515	223.7967
261.4760	274.4675	303.8785
324.7321	340.8373	343.1718
359.4434	380.6263	384.7849
404.3195	419.8448	421.5470
441.8303	489.1160	494.0368
517.5714	553.9543	578.8098
581.9202	625.9385	640.4876
645.6877	645.7752	658.3308

710.4720	714.9851	715.5143
735.9223	740.8327	757.8201
773.3293	776.0111	797.2697
833.8008	849.8993	862.5586
870.2192	873.5106	881.4704
917.4316	933.8024	955.0053
961.8174	965.1943	974.5059
979.4568	979.6062	991.1298
1001.6879	1004.9231	1015.5097
1016.1979	1019.4289	1019.9346
1021.6511	1038.5492	1043.7723
1044.1254	1045.2568	1109.4198
1109.9913	1118.9218	1137.5088
1191.4399	1199.2966	1199.7678
1213.0559	1214.4983	1215.1764
1238.9047	1240.2702	1241.7408
1243.0507	1277.6158	1311.4536
1317.3462	1325.8249	1325.8511
1334.1131	1337.7173	1366.2592
1382.2791	1383.2322	1406.5743
1409.8090	1413.9392	1435.1547
1471.0274	1485.1215	1485.1467
1493.6740	1504.4741	1521.7201
1522.0119	1528.1527	1528.8872
1594.9096	1601.6713	1602.0367
1622.9441	1623.5339	1638.7938
1666.6723	3003.5634	3041.6816
3056.0843	3056.6009	3076.3107
3093.2346	3116.0337	3116.7552
3117.3613	3138.8201	3138.9777
3141.9864	3142.0617	3151.8937
3151.9455	3162.2697	3162.3474
3174.7021	3174.7511	3178.8669
3220.0826	3527.9782	3619.9099

ENERGY with ZPE correction= -232.093891 a.u.


Chloroform-d
7.35
7.33
7.31
7.25
6.75
5.86
5.83
5.77
5.73
5.68
5.14
5.09
4.26
4.19
4.16
4.06
4.01
3.96
3.86
2.23
2.19
2.16
1.16
1.12
CbzHN
NCbz
OH
10
10.31
0.74
0.85
6.87
1.83
1.84
2.76


Chloroform-d
156.89
155.58
136.09
135.63
135.28
134.71
128.46
128.15
128.04
127.81
127.61
126.90
117.35
77.00
72.27
68.00
67.76
66.84
65.12
57.65
37.92
29.60
9.86
CbzHN
NCbz
OH
10
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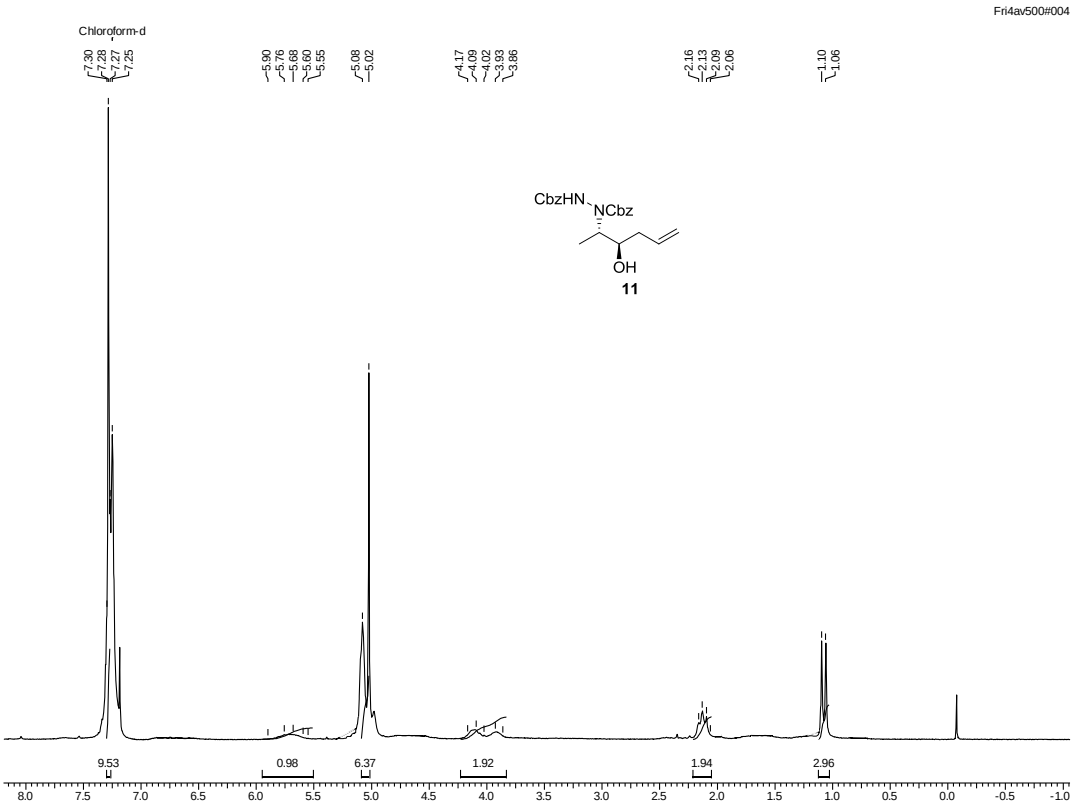
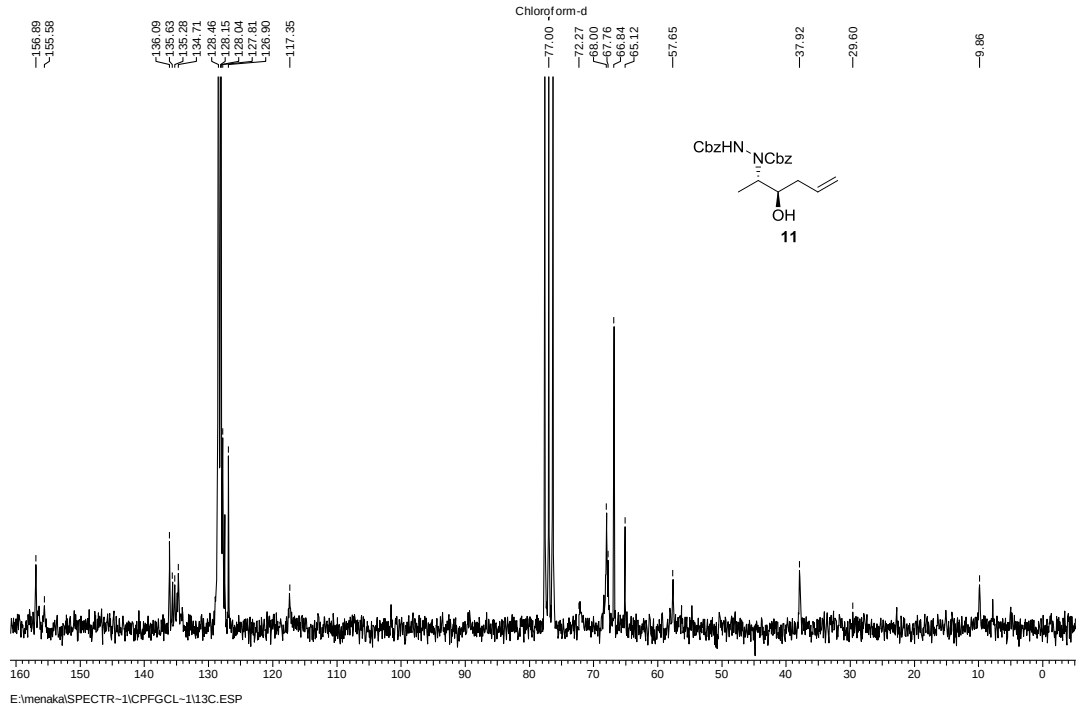
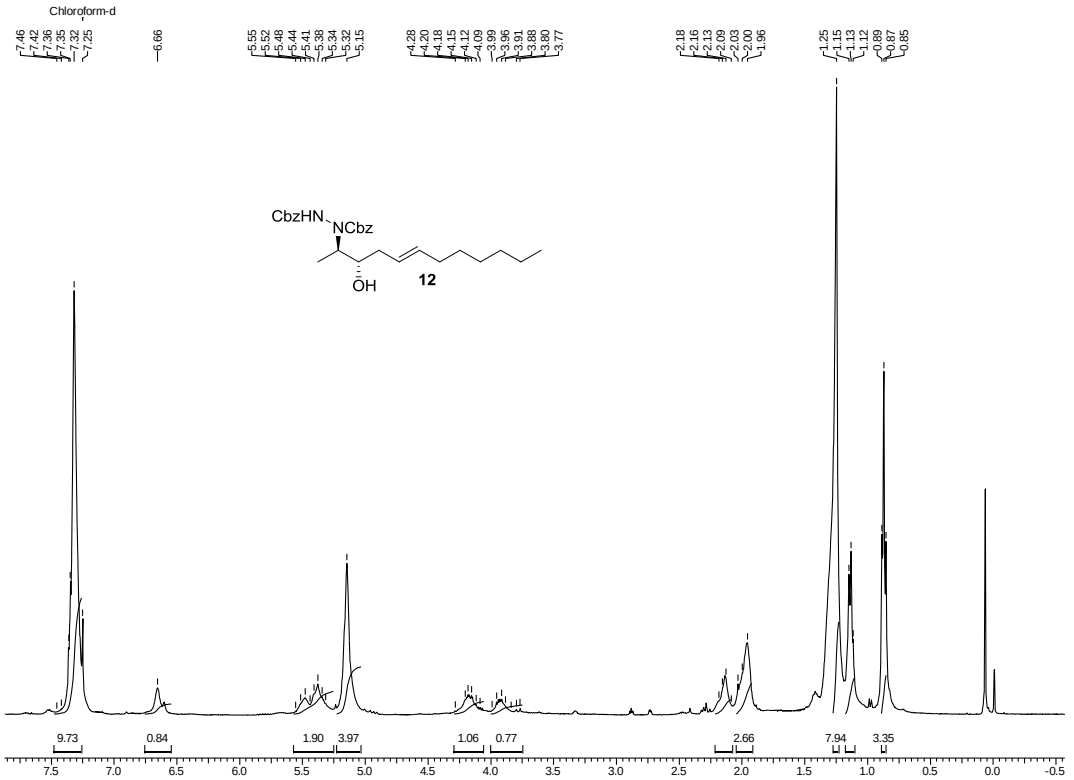
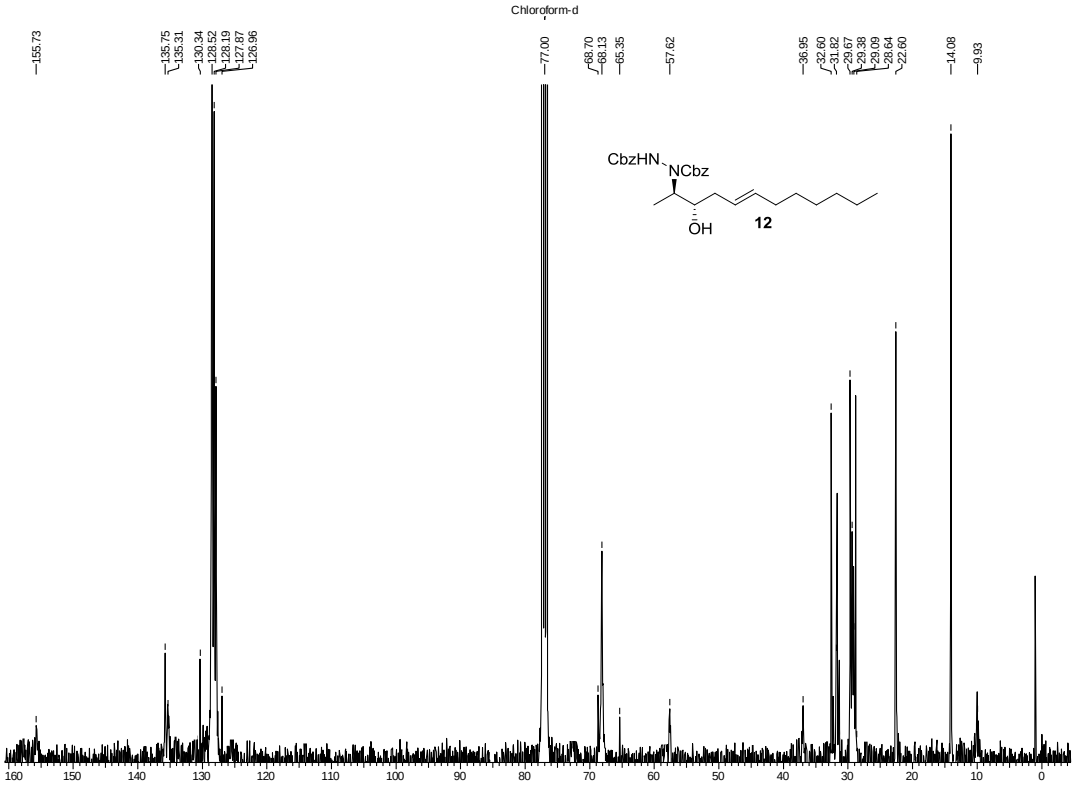
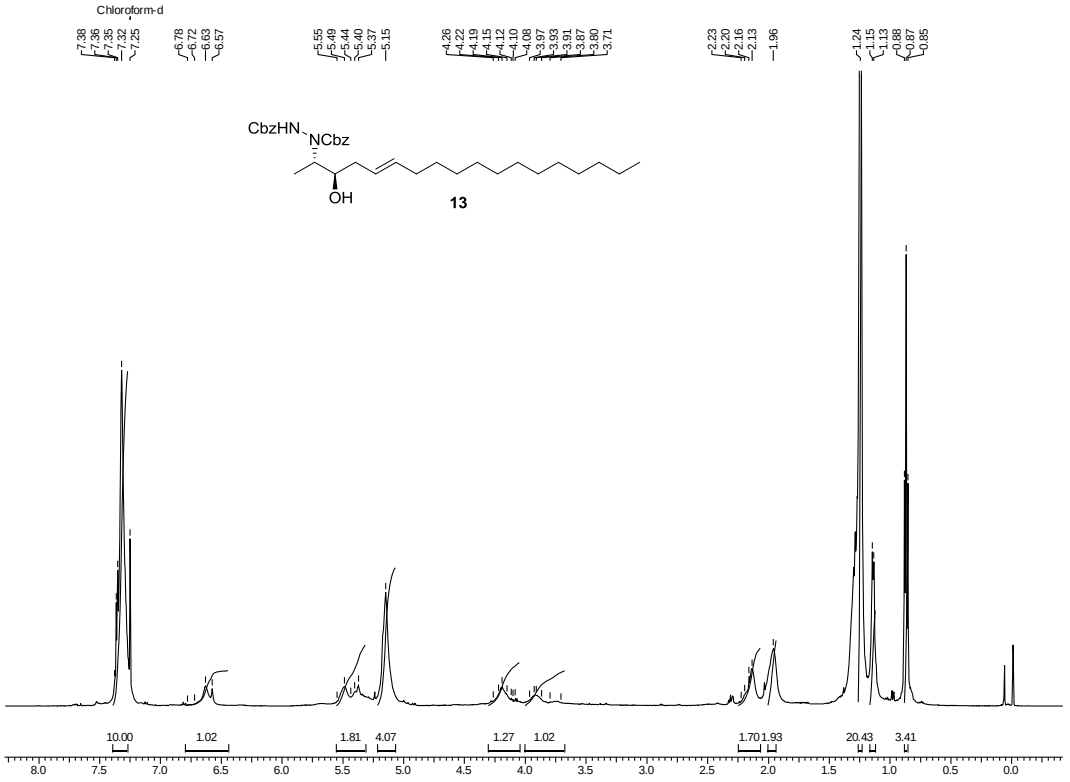
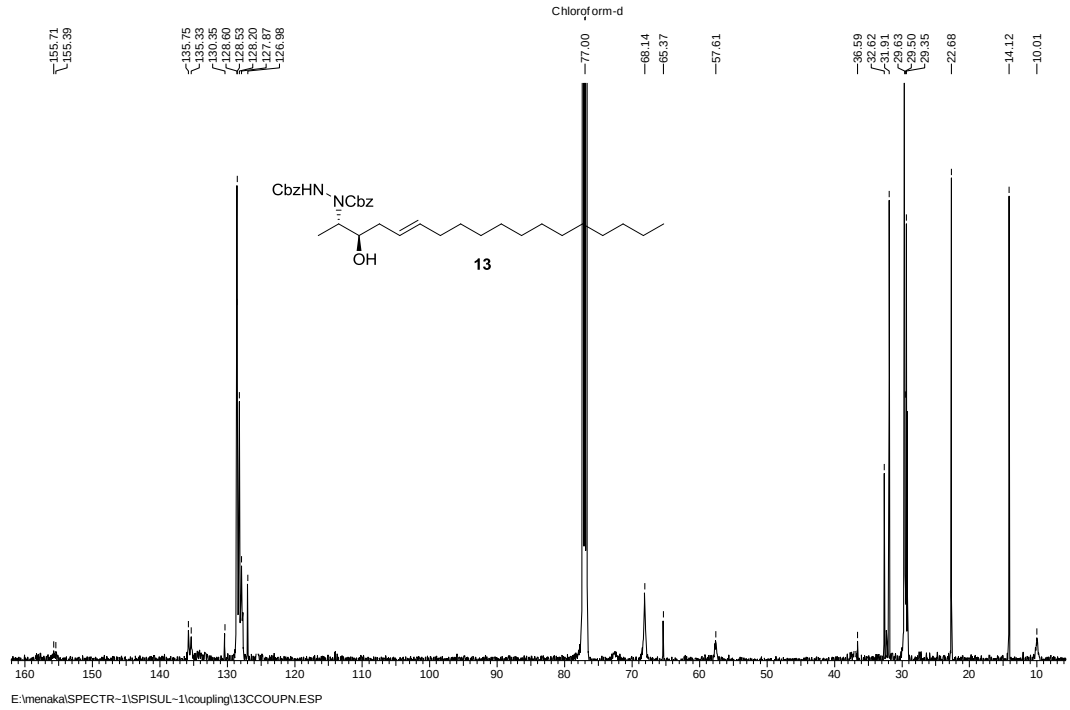

Fri4av500#004
Chloroform-d
7.30
7.28
7.27
7.25
5.90
5.76
5.68
5.60
5.55
5.08
5.02
4.17
4.09
4.02
3.93
3.86
2.16
2.13
2.09
2.06
1.10
1.06
CbzHN
NCbz
OH
11
9.53
0.98
6.37
1.92
1.94
2.96

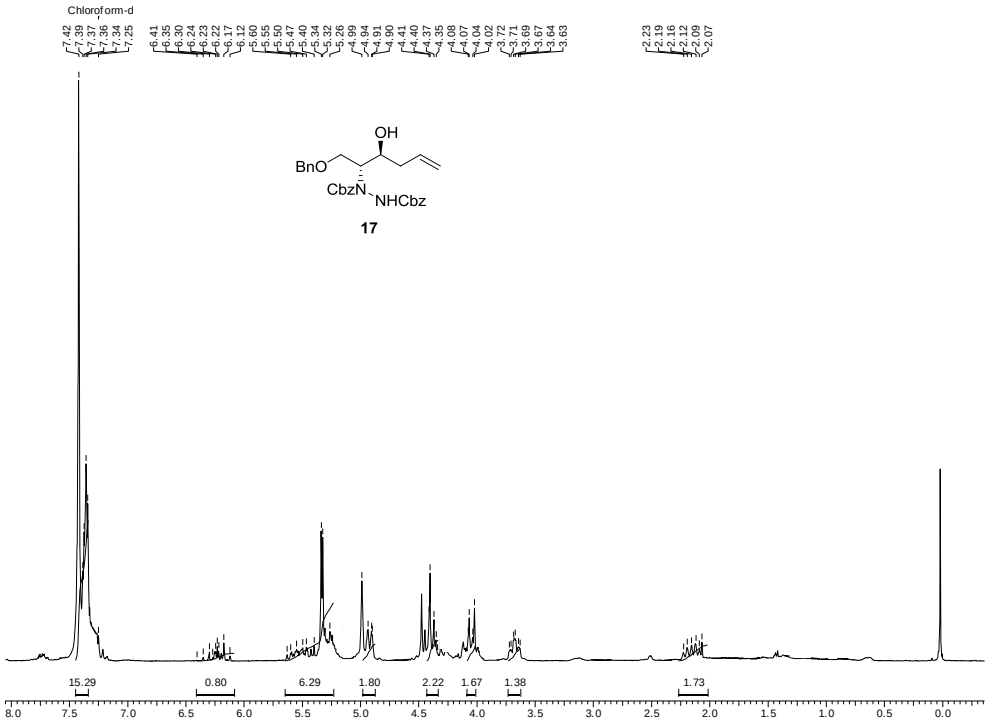

Chloroform-d
156.89
155.58
136.09
135.63
135.28
134.71
128.46
128.15
128.04
127.81
126.90
117.35
77.00
72.27
68.00
67.76
66.84
65.12
57.65
37.92
29.60
9.86
CbzHN
NCbz
OH
11
E:\menaka\SPECTR~1\CPFGCL~1\13C.ESP

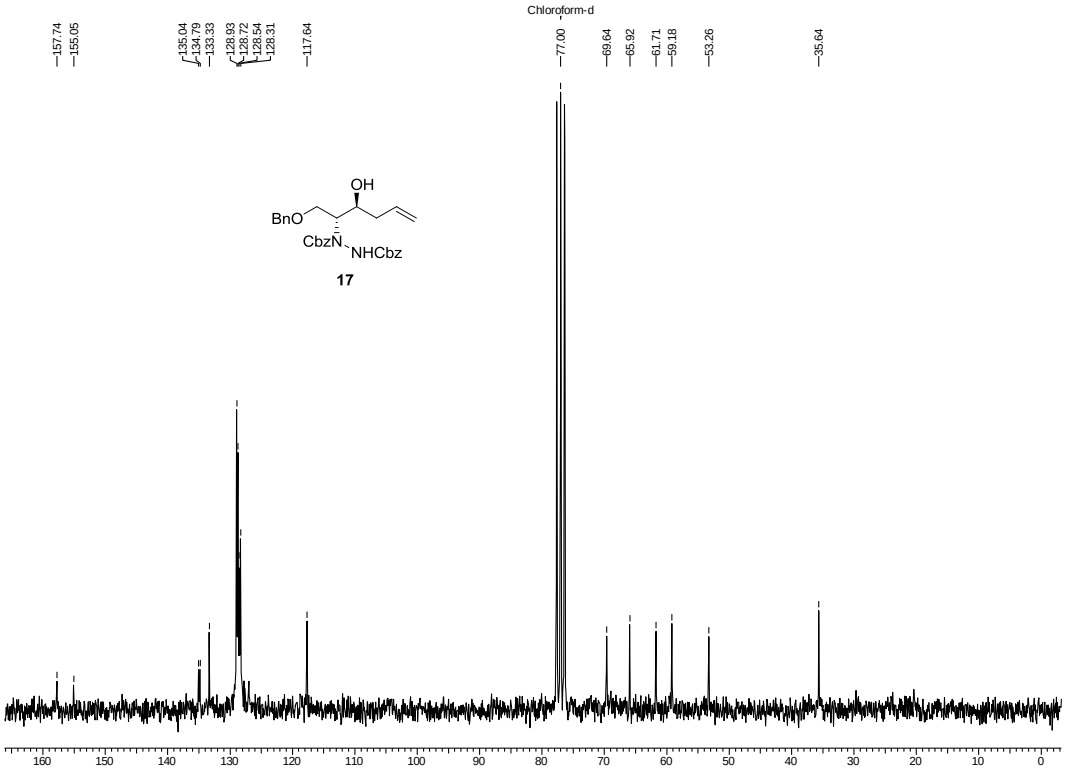


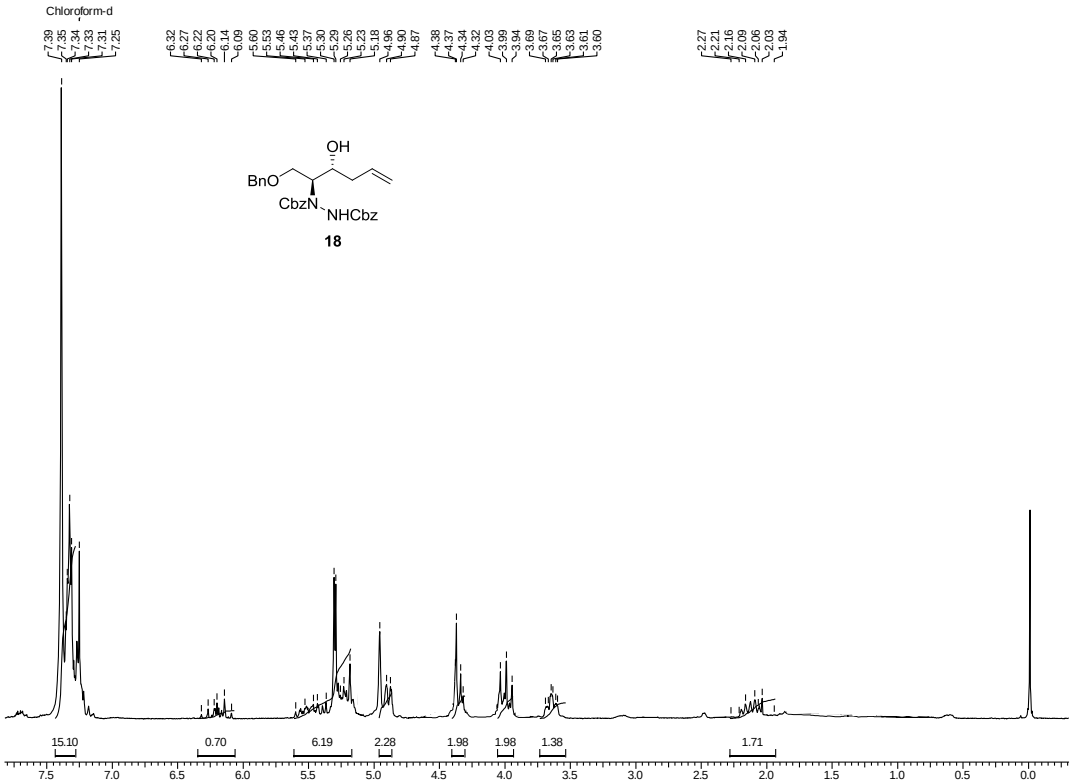


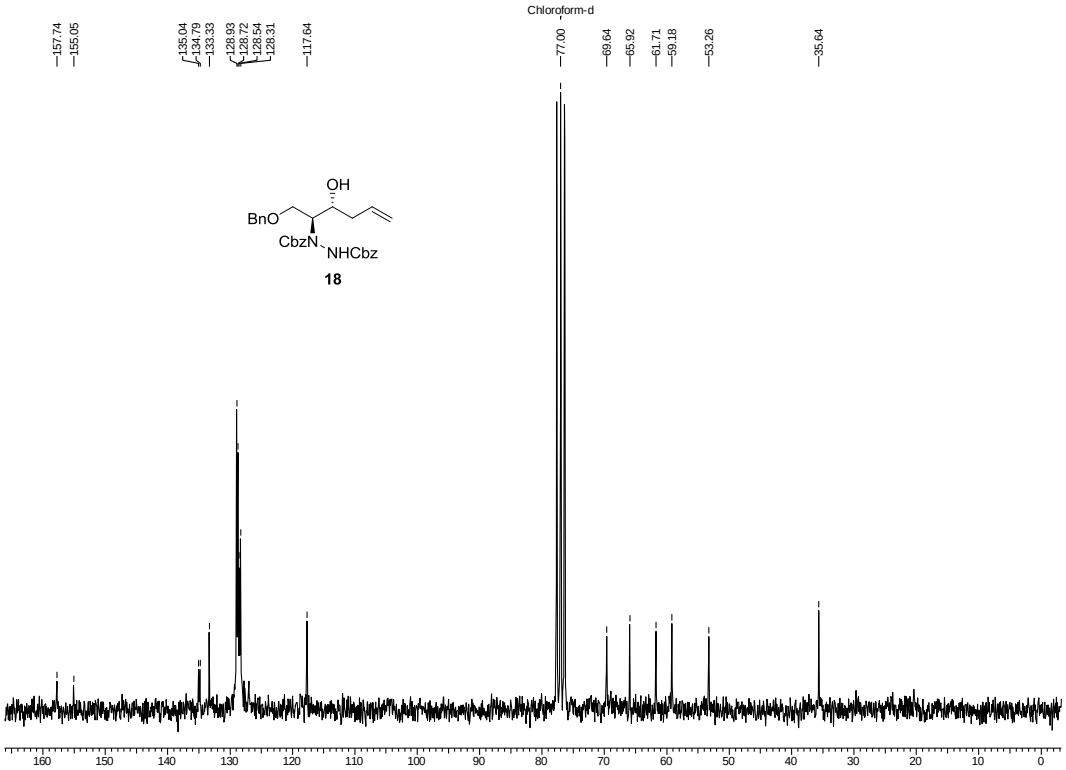

Chloroform-d
7.38
7.36
7.35
7.32
7.25
6.78
6.72
6.63
6.57
5.55
5.49
5.44
5.40
5.37
5.15
4.26
4.22
4.19
4.15
4.12
4.10
4.08
3.97
3.93
3.91
3.87
3.80
3.71
2.23
2.20
2.16
2.13
1.96
1.24
1.15
1.13
0.88
0.87
0.85
CbzHN
NCbz
OH
13
10.00
1.02
1.81
4.07
1.27
1.02
1.70
1.93
20.43
3.41

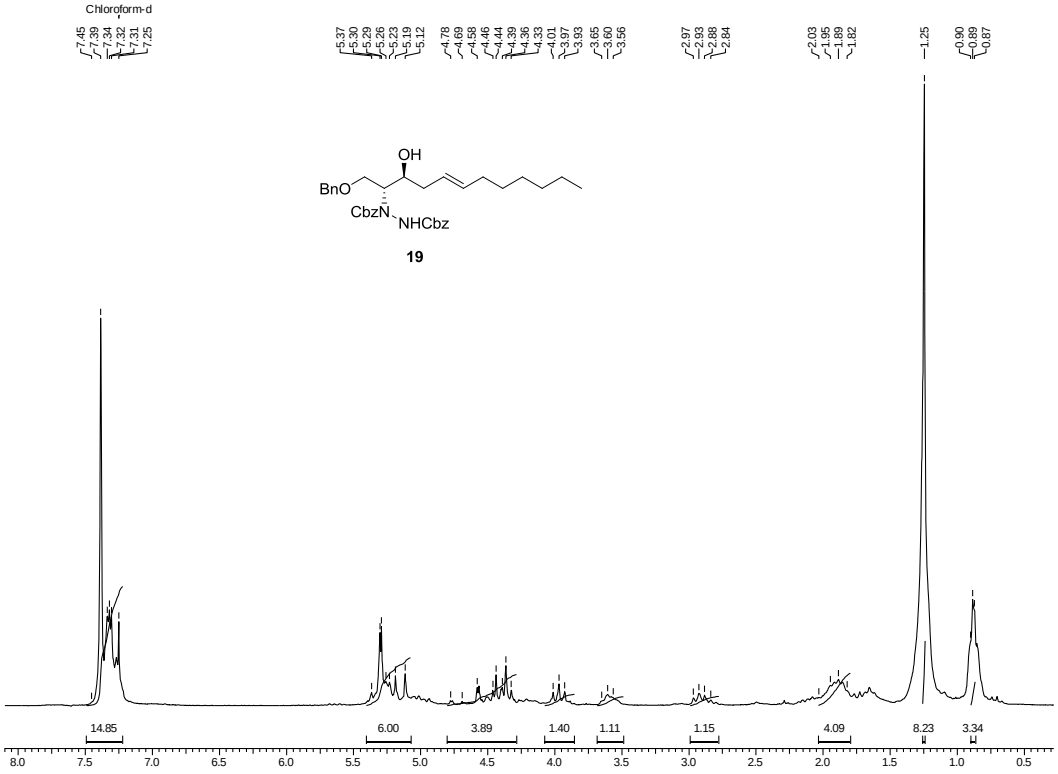

Chloroform-d
155.71
155.39
135.75
135.33
130.35
128.60
128.53
128.20
127.87
126.98
77.00
68.14
65.37
57.61
36.59
32.62
31.91
29.63
29.50
29.35
22.68
14.12
10.01
CbzHN
NCbz
OH
13
E:\menaka\SPECTR~1\SPISUL~1\coupling\13CCOUPN.ESP

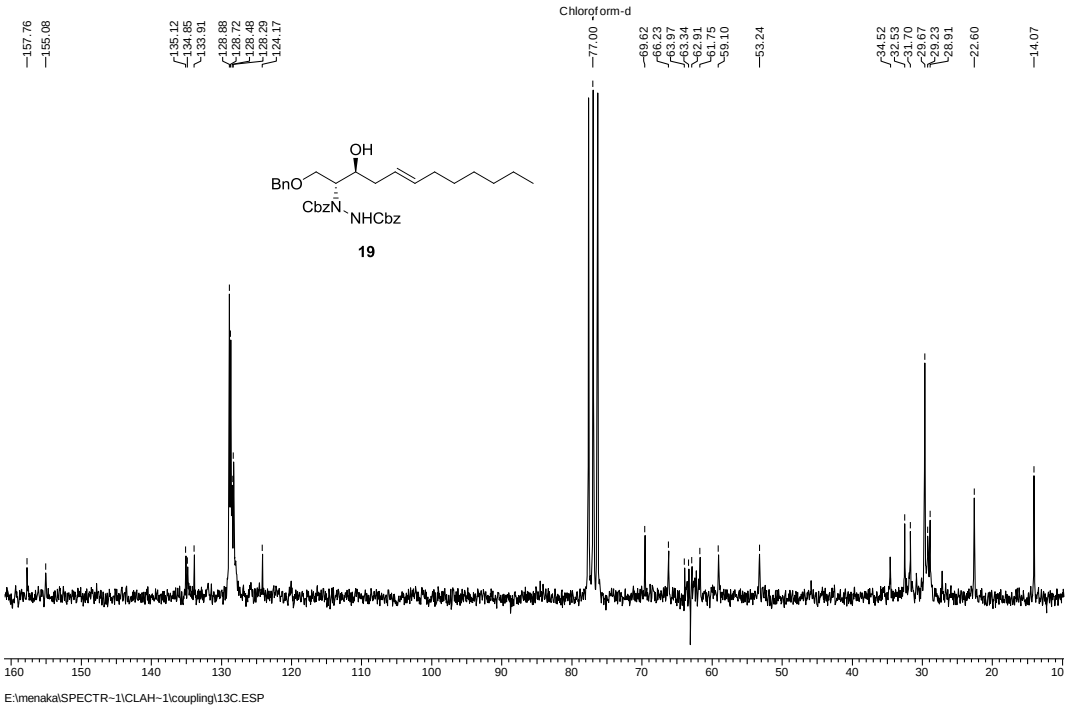

Chloroform-d
OH
BnO
CbzN
NHCbz
17

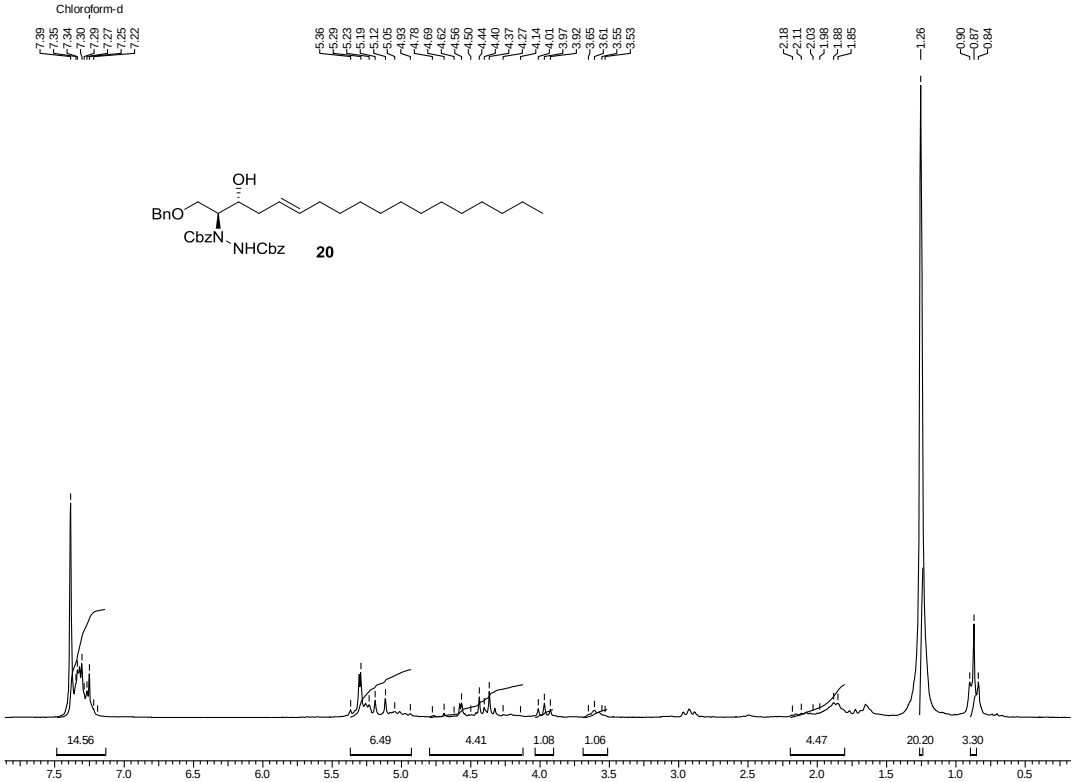

Chloroform-d
OH
BnO
CbzN
NHCbz
17

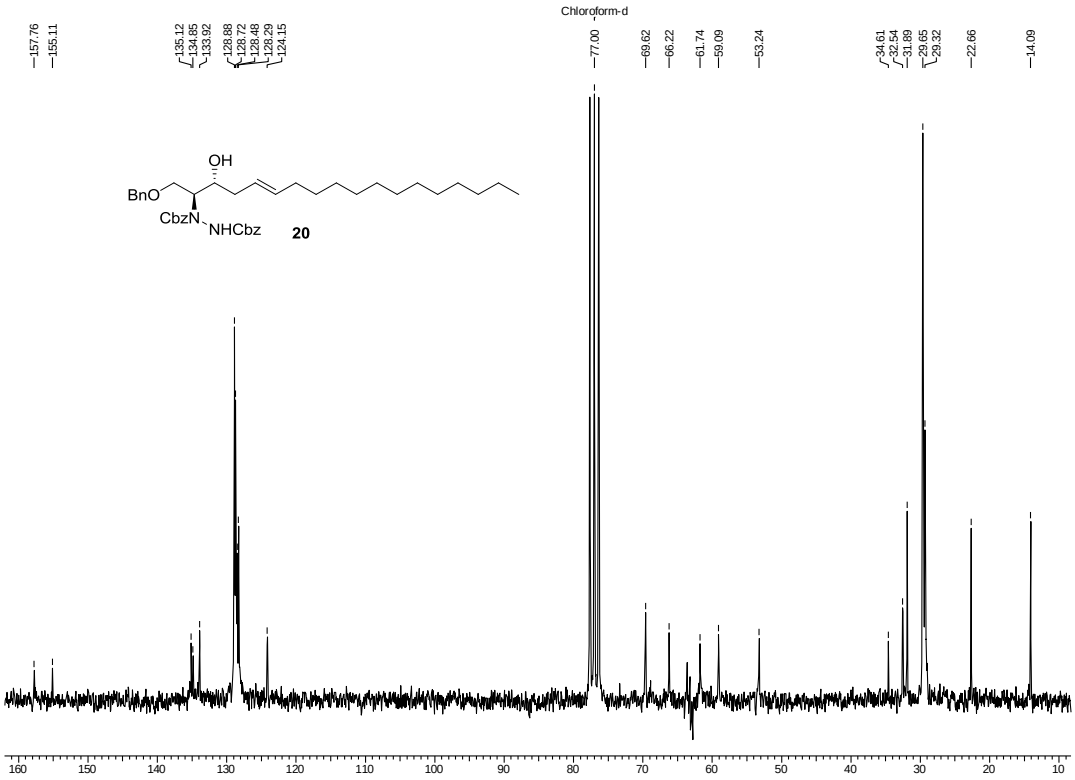


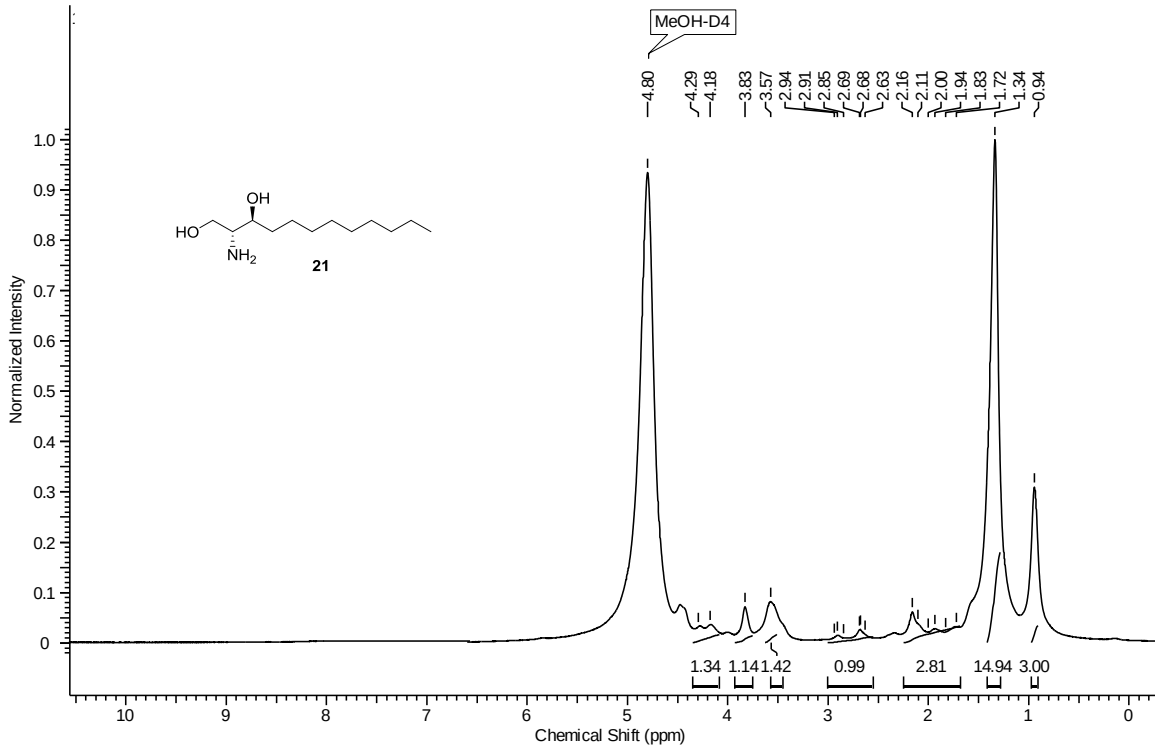


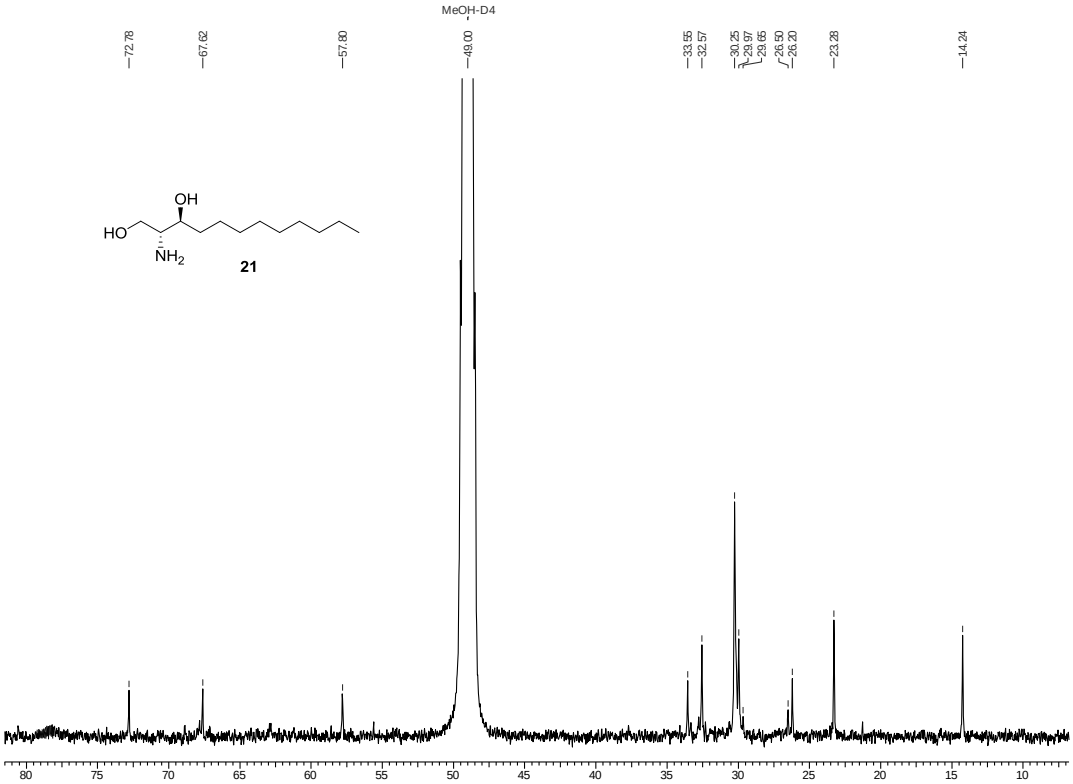

Chloroform-d
7.45
7.39
7.34
7.32
7.31
7.25
5.37
5.30
5.29
5.26
5.23
5.19
5.12
4.78
4.69
4.58
4.46
4.44
4.39
4.36
4.33
4.01
3.97
3.93
3.65
3.60
3.56
2.97
2.93
2.88
2.84
2.03
1.95
1.89
1.82
1.25
0.90
0.89
0.87
OH
BnO
CbzN
NHCbz
19
14.85
6.00
3.89
1.40
1.11
1.15
4.09
8.23
3.34

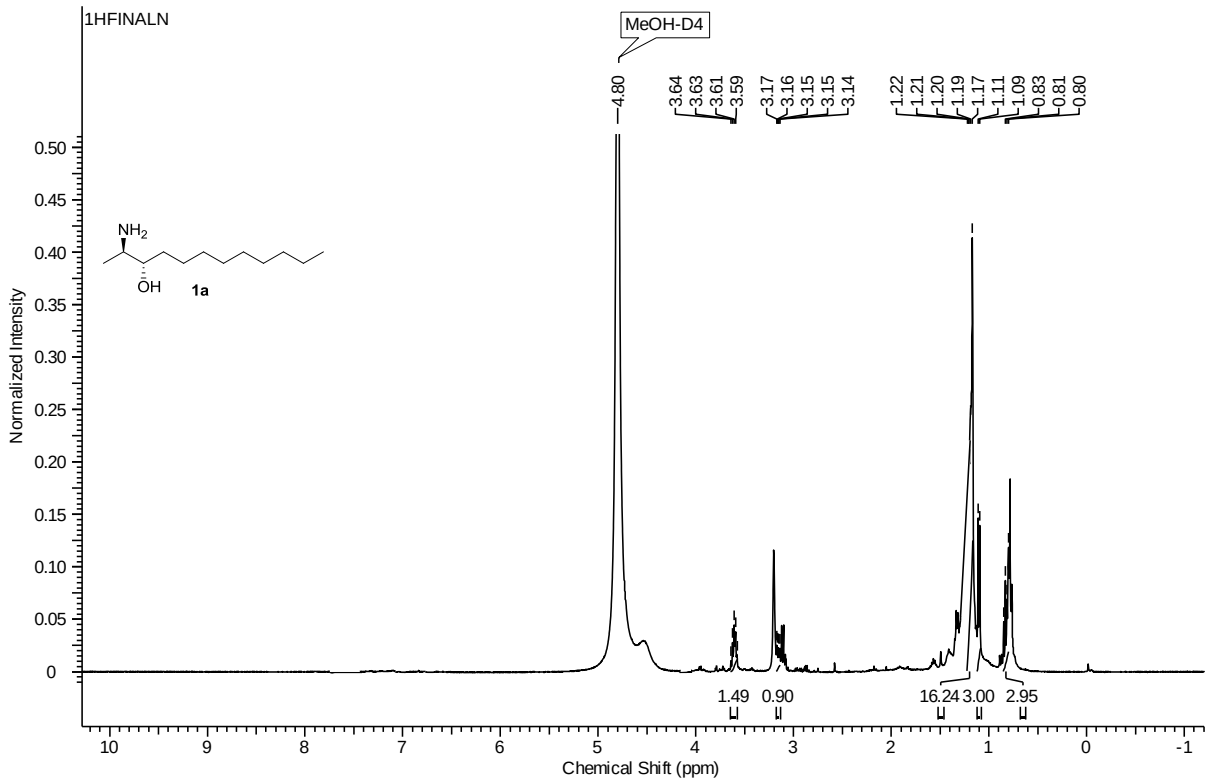

157.76
155.08
135.12
134.85
133.91
128.88
128.72
128.48
128.29
124.17
Chloroform-d
77.00
69.62
66.23
63.97
63.34
62.91
61.75
59.10
53.24
34.52
32.53
31.70
29.67
29.23
28.91
22.60
14.07
OH
BnO
CbzN
NHCbz
19
E:\menaka\SPECTR~1\CLAH~1\coupling\13C.ESP

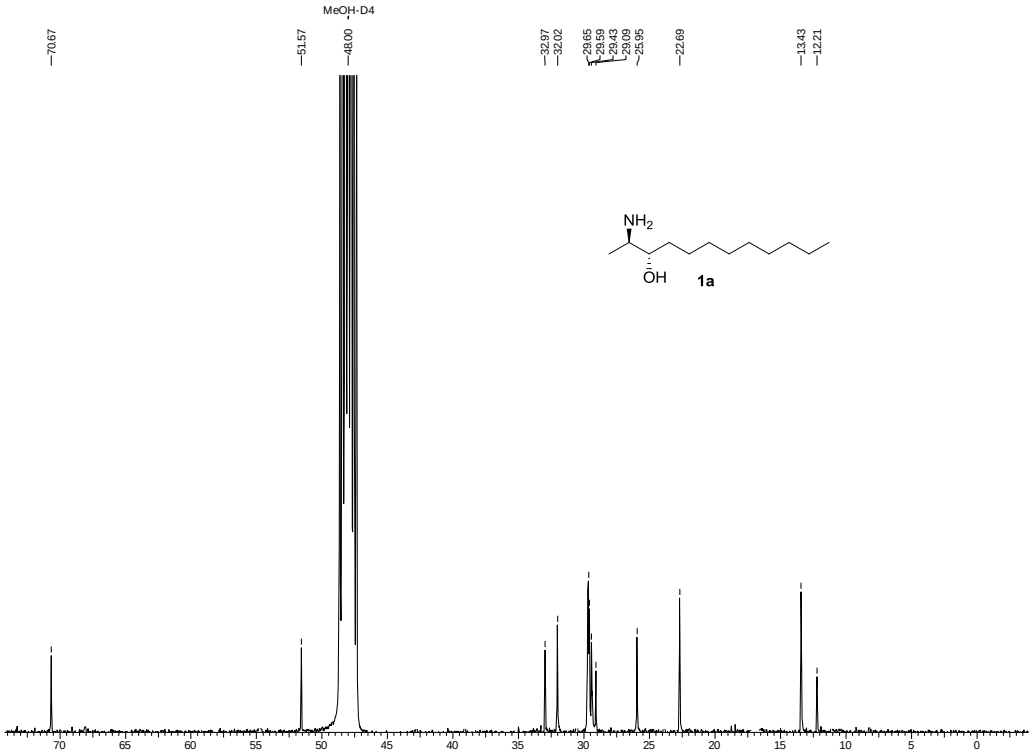


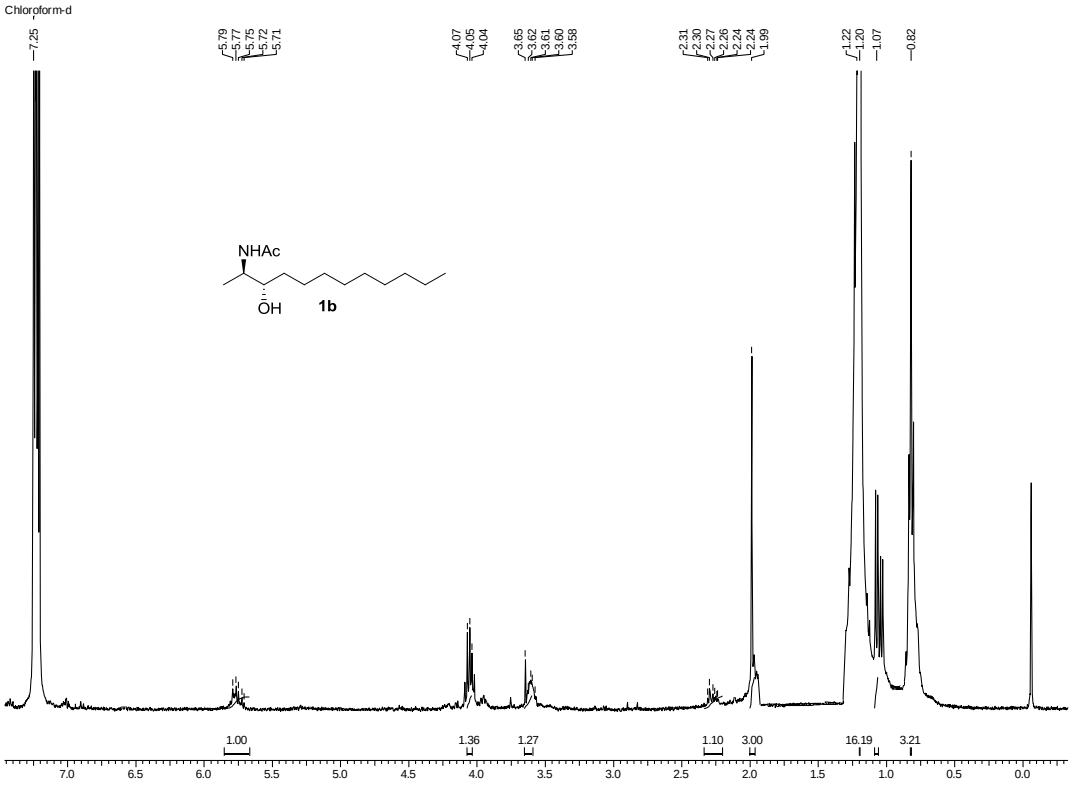


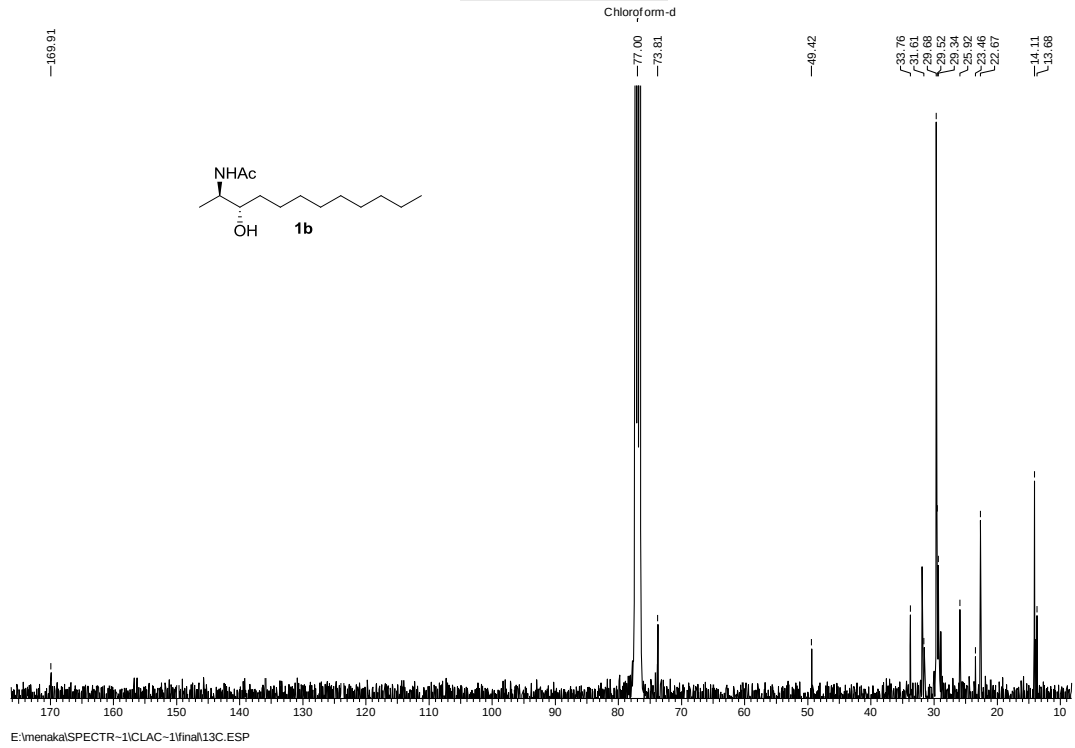

MeOH-D4
4.80
4.29
4.18
3.83
3.57
2.94
2.91
2.85
2.69
2.68
2.63
2.16
2.11
2.00
1.94
1.83
1.72
1.34
0.94
OH
HO
NH2
21
Normalized Intensity
1.34
1.14
1.42
0.99
2.81
14.94
3.00
Chemical Shift (ppm)

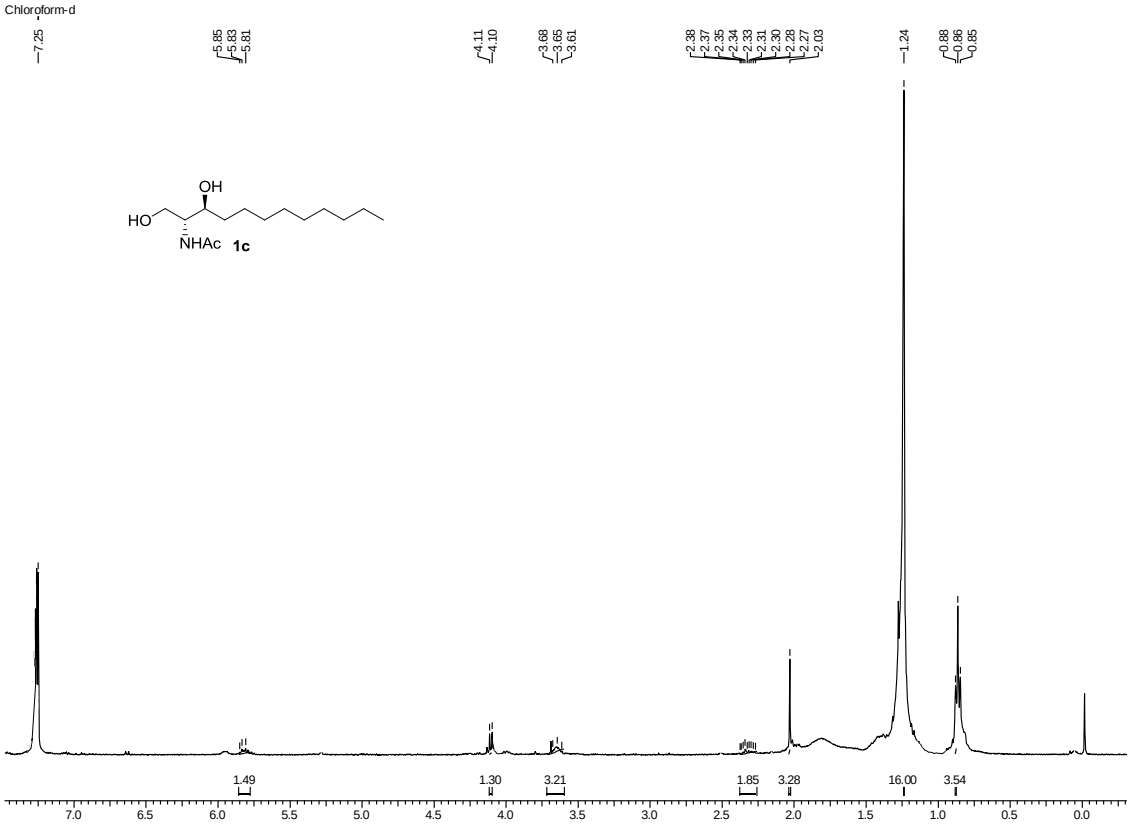

MeOH-D4
72.78
67.62
57.80
49.00
33.55
32.57
30.25
29.97
29.65
26.50
26.20
23.28
14.24
OH
HO
NH2
21

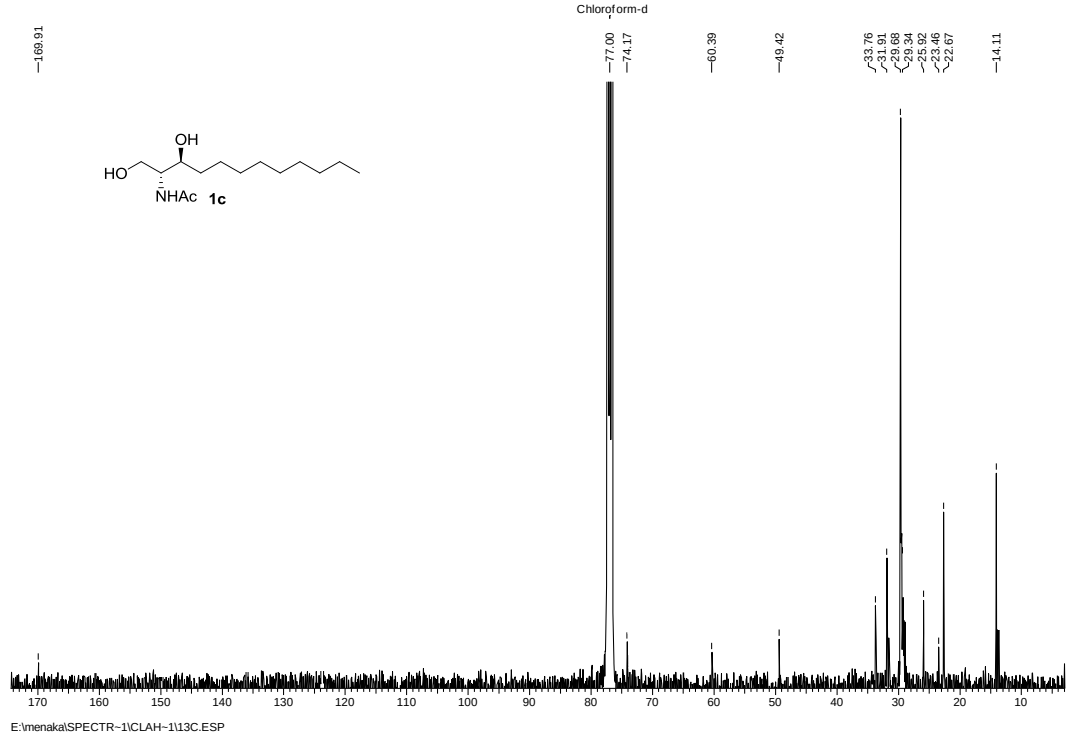

1HFINALN
MeOH-D4
4.80
3.64
3.63
3.61
3.59
3.17
3.16
3.15
3.15
3.14
1.22
1.21
1.20
1.19
1.17
1.11
1.09
0.83
0.81
0.80
NH2
OH
1a
Normalized Intensity
Chemical Shift (ppm)
1.49
0.90
16.24
3.00
2.95


MeOH-D4
70.67
51.57
48.00
32.97
32.02
29.65
29.59
29.43
29.09
25.95
22.69
13.43
12.21
NH2
OH
1a






Chloroform-d
7.25
5.85
5.83
5.81
4.11
4.10
3.68
3.65
3.61
2.38
2.37
2.35
2.34
2.33
2.31
2.30
2.28
2.27
2.03
1.24
0.88
0.86
0.85
OH
HO
NHAc
1c
1.49
1.30
3.21
1.85
3.28
16.00
3.54


Chloroform-d
169.91
77.00
74.17
60.39
49.42
33.76
31.91
29.68
29.34
25.92
23.46
22.67
14.11
OH
HO
NHAc
1c
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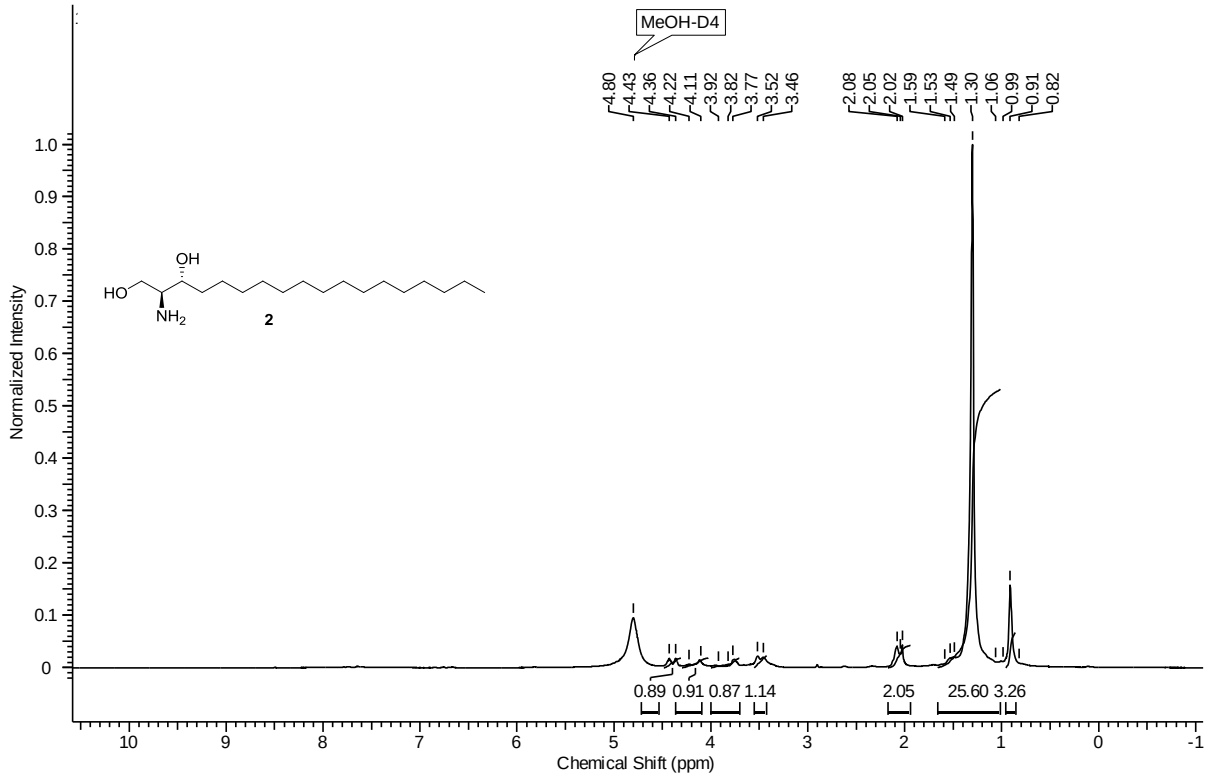
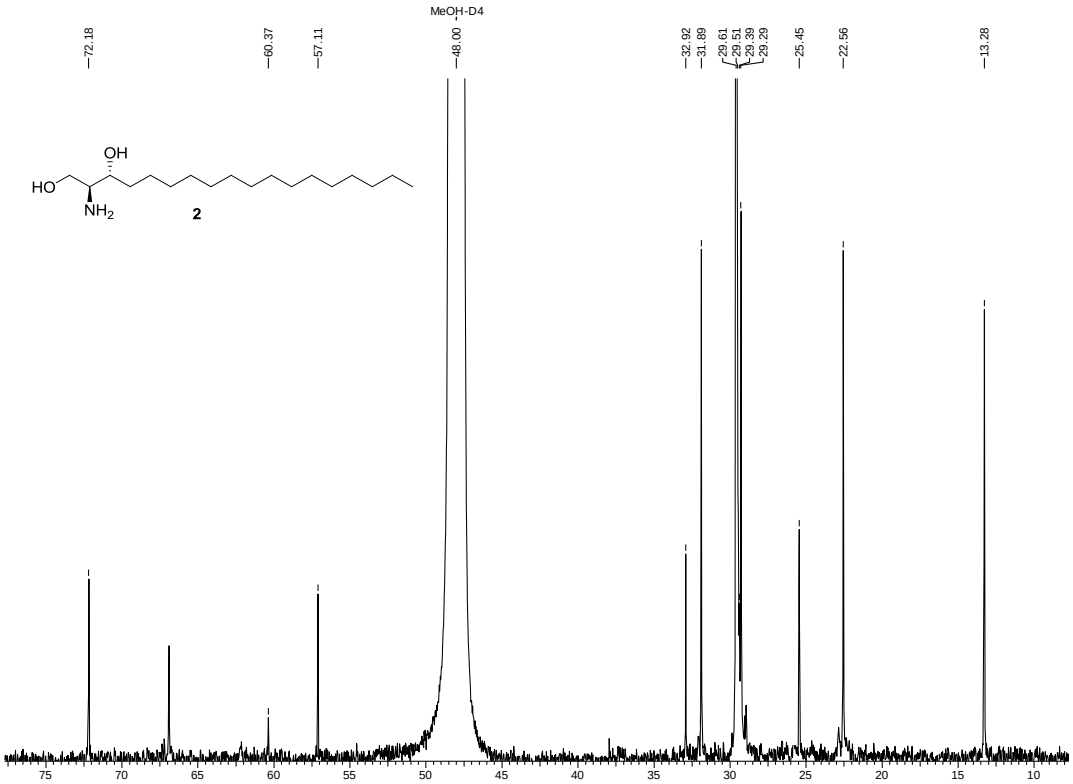
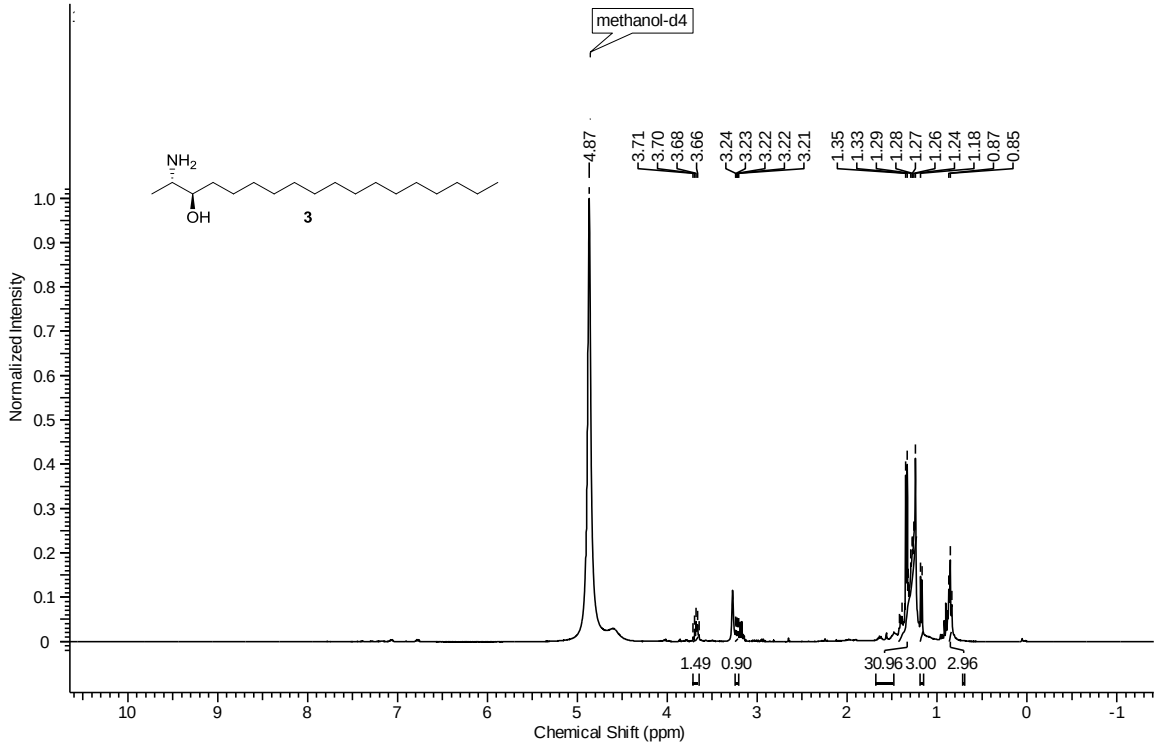
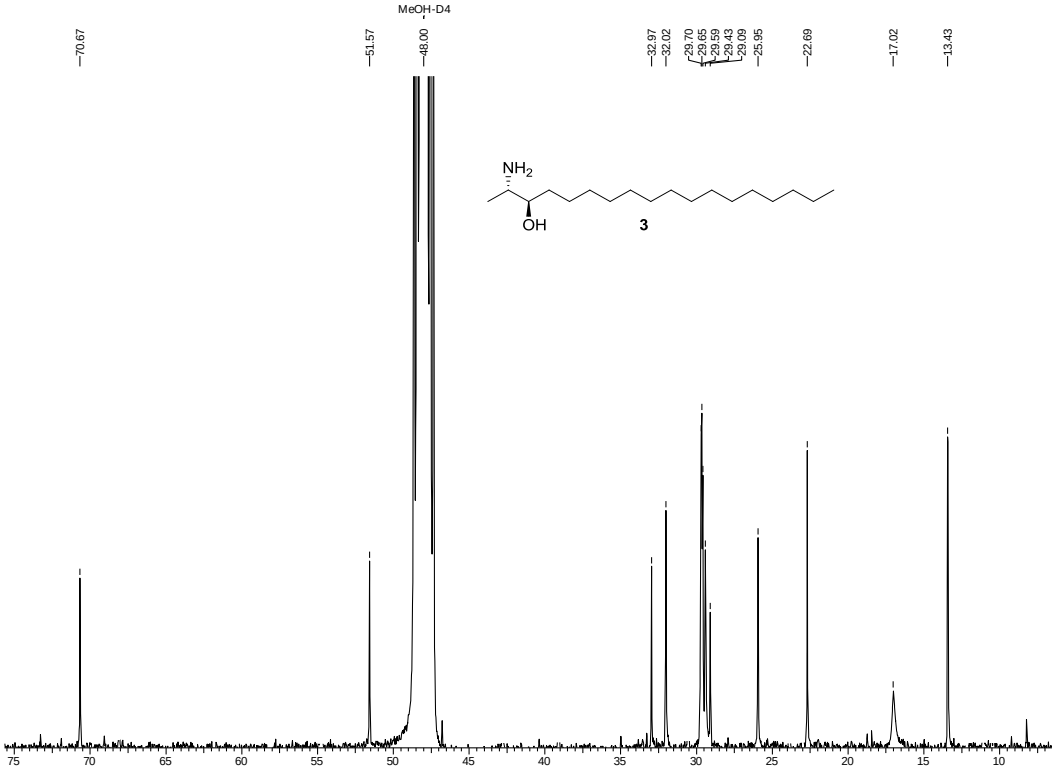








HPLC chromatogram of racemic **17**

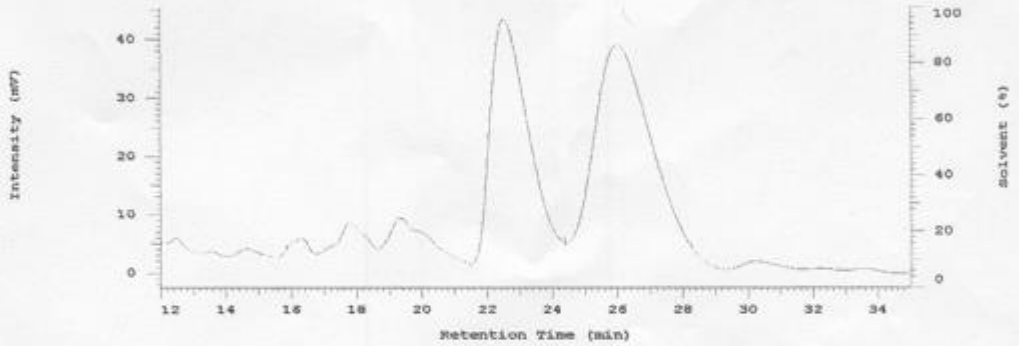
D-7000 HSM: NCLOC Series: 4799 Report: modified System: Sys 1

NATIONAL CHEMICAL LABORATORY

ORGANIC CHEMISTRY TECHNOLOGY

Analyzed: 05/09/12 02:59 PM Reported: 05/09/12 03:50 PM
Data Path: f:\win32app\hsm\NCLOC\DATA\4799\
Processing Method: Lichrosphere RP-18 (250-4) Acquisition Method: Lichrosphere RP-18 (250-4)
System(acquisition): Sys 1 Series:4799
Sample Name: D+L-Proline Vial Number: 3
Injection from vial: 1 of 1 Vial Type: UNK
Volume: 10.0 ul

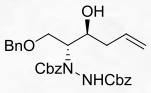
Chrom Type: HPLC Channel : 1



Peak Quantitation: AREA Calculation Method: AREA%

No.	RT	Height	Area	Area %
1	22.53	41265	3315722	44.362
2	26.01	35766	4158436	55.638
		77031	7474158	100.000

Peak rejection level: 0



17
Racemic

Group Leader :Dr. Pradeep Kumar
Column :Chiralcel OJ-RH(150 X 4.6 mm)
M.P. :MEOH:H2O (80:20)

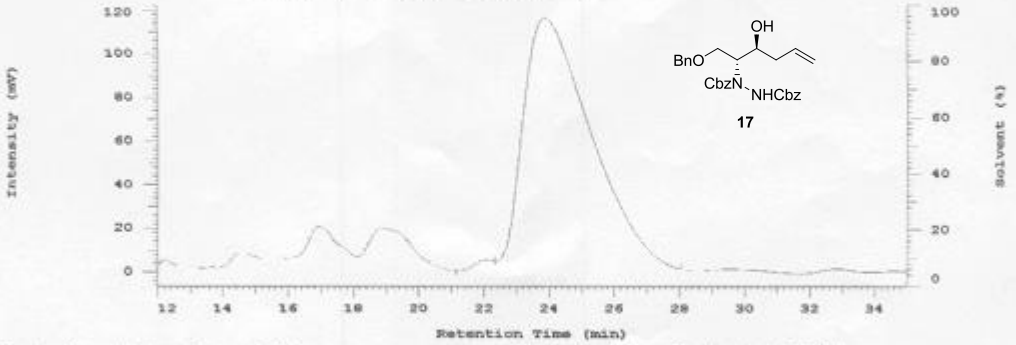
Flow Rate :0.5 ml/min (695 psi)
Sample conc: x mg/ 3ml
Inj vol: 10 ul
WAVELENGTH : 254 nm

D-7000 HSM: NCLOC Series: 4798 Report: modified System: Sys 1

NATIONAL CHEMICAL LABORATORY

ORGANIC CHEMISTRY TECHNOLOGY

Analyzed: 05/09/12 02:23 PM Reported: 05/09/12 03:06 PM
Data Path: f:\win32app\hsm\NCLOC\DATA\4798\
Processing Method: Lichrosphere RP-18 (250-4) Acquisition Method: Lichrosphere RP-18 (250-4)
System(acquisition): Sys 1 Series:4798
Sample Name: L-Proline Vial Number: 2
Injection from vial: 1 of 1 Vial Type: UNK
Volume: 10.0 ul
Chrom Type: HPLC Channel : 1



Peak Quantitation: AREA Calculation Method: AREA%

No.	RT	Height	Area	Area %
1	22.10	5033	202262	1.194
2	23.89	115323	16735961	98.806
		120356	16938223	100.000

Peak rejection level: 0

Group Leader :Dr. Pradeep Kumar
Column :Chiralcel OJ-RH(150 X 4.6 mm)
M.P. :MEOH:H2O (80:20)

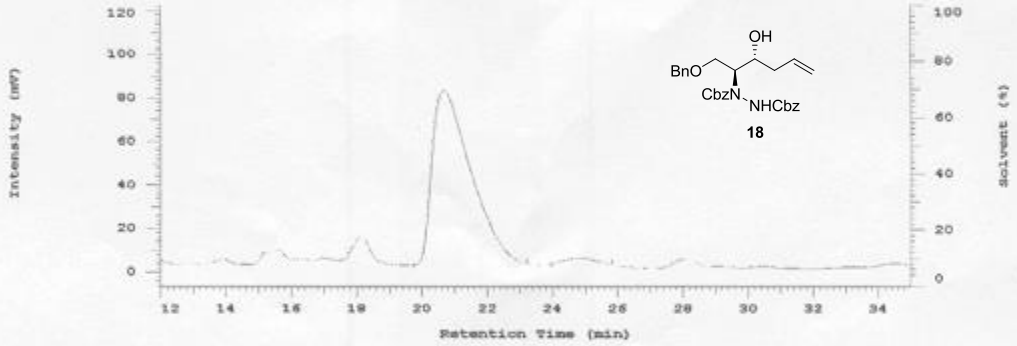
Flow Rate :0.5 ml/min (695 psi)
Sample conc: x mg/ 3ml
Inj vol: 10 ul
WAVELENGTH : 254 nm

D-7000 HSM: NCLOC Series: 4797 Report: modified System: Sys 1

NATIONAL CHEMICAL LABORATORY

ORGANIC CHEMISTRY TECHNOLOGY

Analyzed: 05/09/12 01:47 PM Reported: 05/09/12 03:07 PM
Data Path: f:\win32app\hsm\NCLOC\DATA\4797\
Processing Method: Lichrosphere RP-18 (250-4) Acquisition Method: Lichrosphere RP-18 (250-4)
System(acquisition): Sys 1 Series:4797
Sample Name: D-Proline Vial Number: 1
Injection from vial: 1 of 1 Vial Type: UNK
Volume: 10.0 ul
Chrom Type: HPLC Channel : 1



Peak Quantitation: AREA Calculation Method: AREA%

No.	RT	Height	Area	Area %
1	20.69	79744	6667622	97.416
2	24.85	2790	176845	2.584
		82534	6844467	100.000

Peak rejection level: 0

Group Leader :Dr. Pradeep Kumar
Column :Chiralcel OJ-RH(150 X 4.6 mm)
M.P. :MEOH:H2O (80:20)

Flow Rate :0.5 ml/min (695 psi)
Sample conc: x mg/ 3ml
Inj vol: 10 ul
WAVELENGTH : 254 nm