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

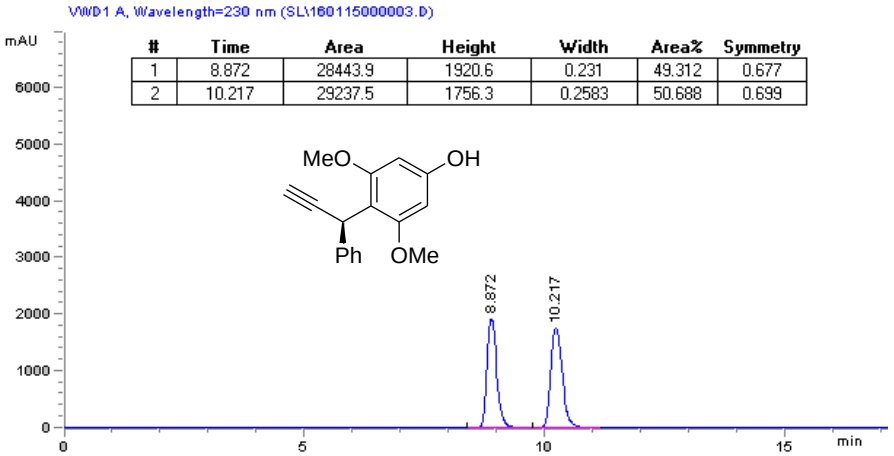
Supporting Information

Cu-catalyzed asymmetric Friedel-Crafts propargylic alkylation of phenol derivatives

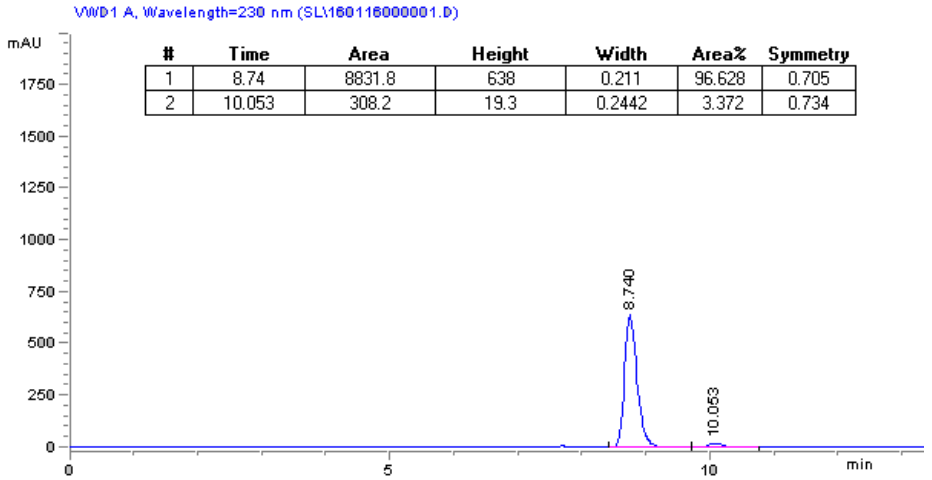
Long Shao, and Xiang-Ping Hu*

Dalian Institute of Chemical Physics, Chinese Academy of Sciences, Dalian 116023, China
xiangping@dicp.ac.cn

3,5-dimethoxy-4-((*R*)-1-phenylprop-2-ynyl)phenol (3aa).

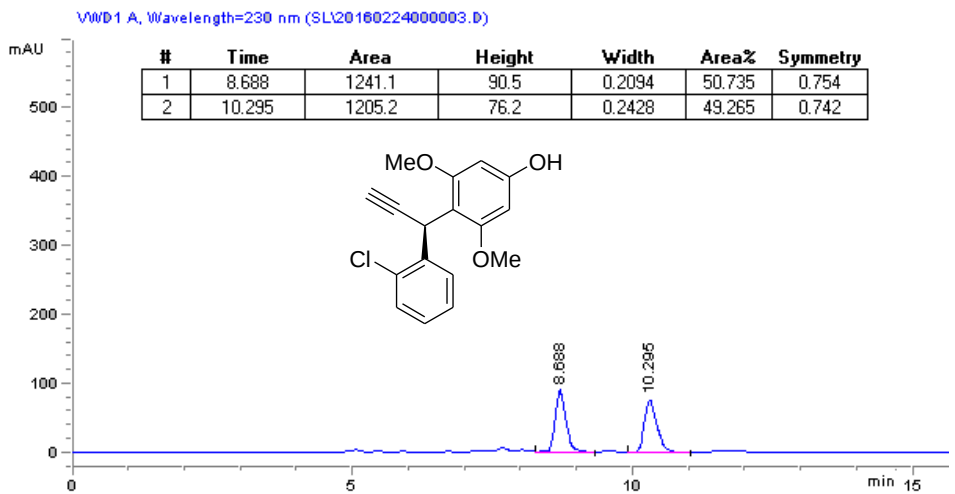


#	Time	Area	Height	Width	Area%	Symmetry
1	8.872	28443.9	1920.6	0.231	49.312	0.677
2	10.217	29237.5	1756.3	0.2583	50.688	0.699

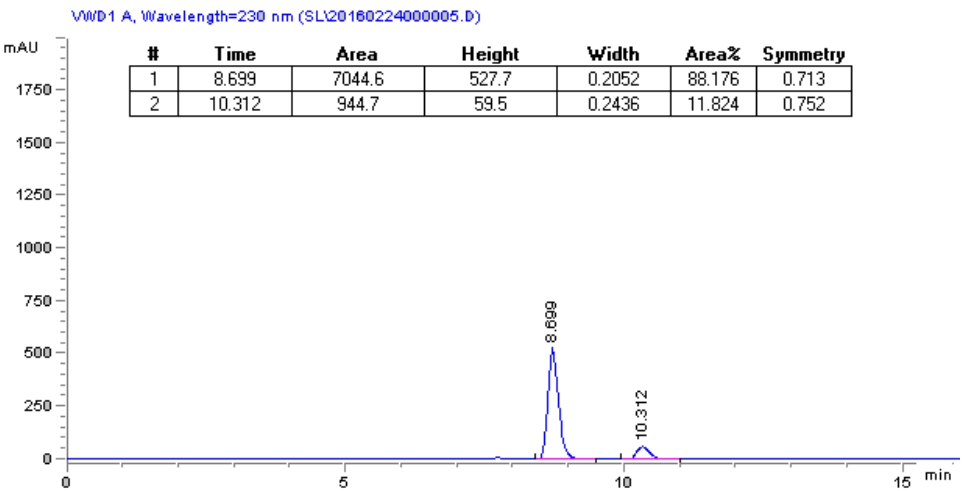


#	Time	Area	Height	Width	Area%	Symmetry
1	8.74	8831.8	638	0.211	96.628	0.705
2	10.053	308.2	19.3	0.2442	3.372	0.734

4-((*S*)-1-(2-chlorophenyl)prop-2-ynyl)-3,5-dimethoxyphenol (3ba).

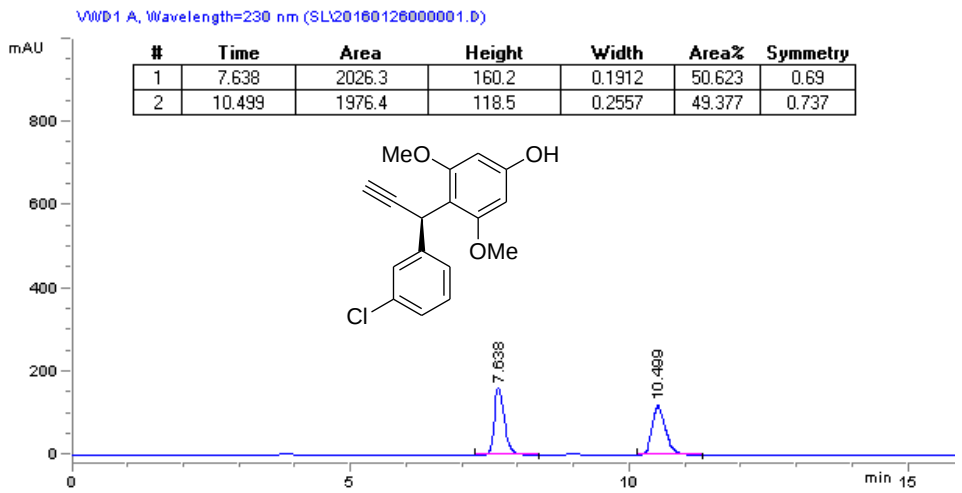


#	Time	Area	Height	Width	Area%	Symmetry
1	8.688	1241.1	90.5	0.2094	50.735	0.754
2	10.295	1205.2	76.2	0.2428	49.265	0.742

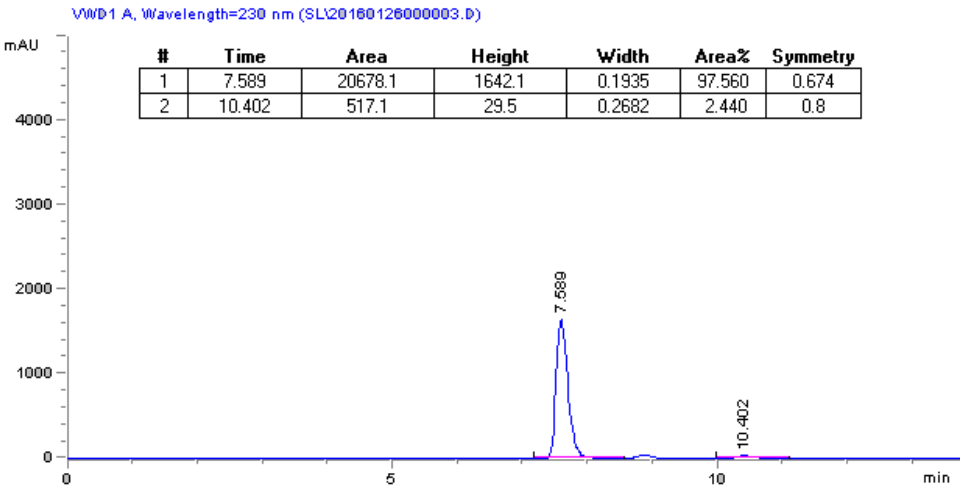


#	Time	Area	Height	Width	Area%	Symmetry
1	8.699	7044.6	527.7	0.2052	88.176	0.713
2	10.312	944.7	59.5	0.2436	11.824	0.752

4-((*R*)-1-(3-chlorophenyl)prop-2-yn-1-yl)-3,5-dimethoxyphenol (3ca).

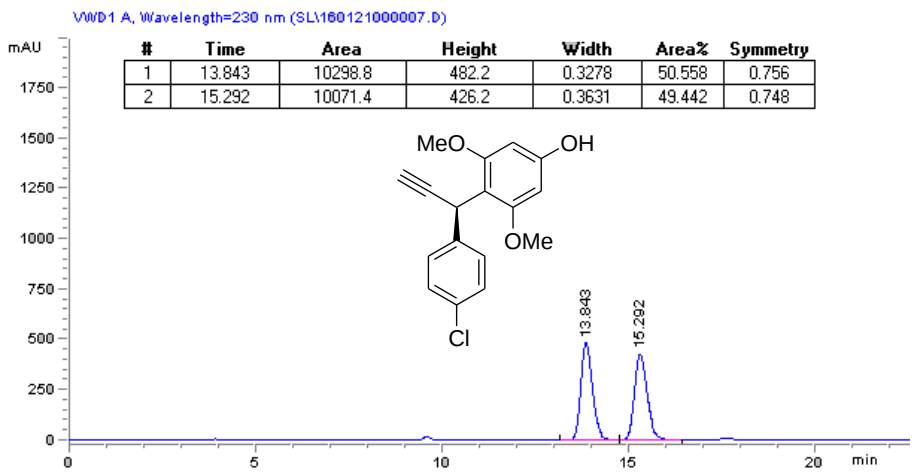


#	Time	Area	Height	Width	Area%	Symmetry
1	7.638	2026.3	160.2	0.1912	50.623	0.69
2	10.499	1976.4	118.5	0.2557	49.377	0.737

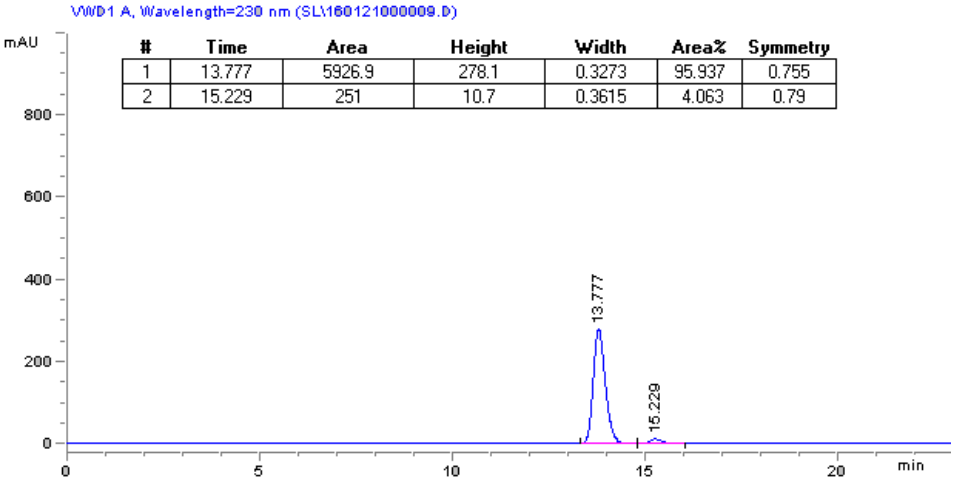


#	Time	Area	Height	Width	Area%	Symmetry
1	7.589	20678.1	1642.1	0.1935	97.560	0.674
2	10.402	517.1	29.5	0.2682	2.440	0.8

4-((*R*)-1-(4-chlorophenyl)prop-2-ynyl)-3,5-dimethoxyphenol (3da).

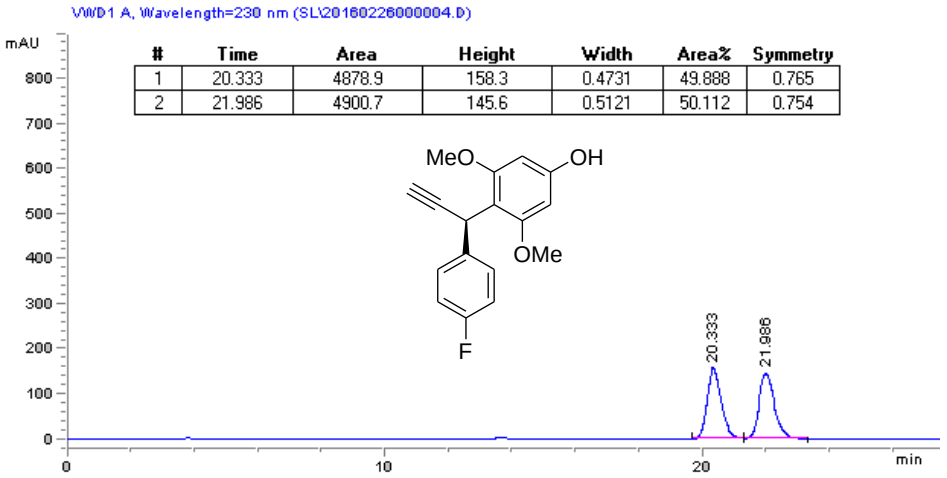


#	Time	Area	Height	Width	Area%	Symmetry
1	13.843	10298.8	482.2	0.3278	50.558	0.756
2	15.292	10071.4	426.2	0.3631	49.442	0.748

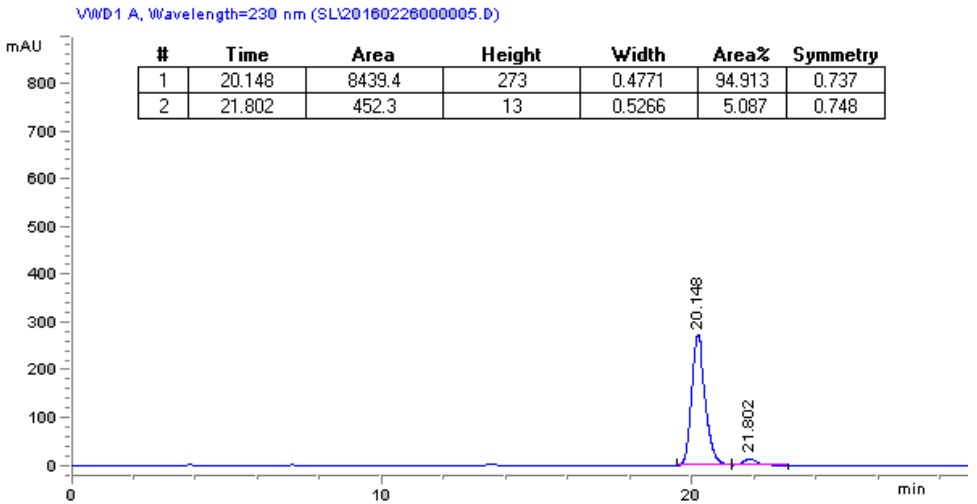


#	Time	Area	Height	Width	Area%	Symmetry
1	13.777	5926.9	278.1	0.3273	95.937	0.755
2	15.229	251	10.7	0.3615	4.063	0.79

4-((*R*)-1-(4-fluorophenyl)prop-2-ynyl)-3,5-dimethoxyphenol (3ea).

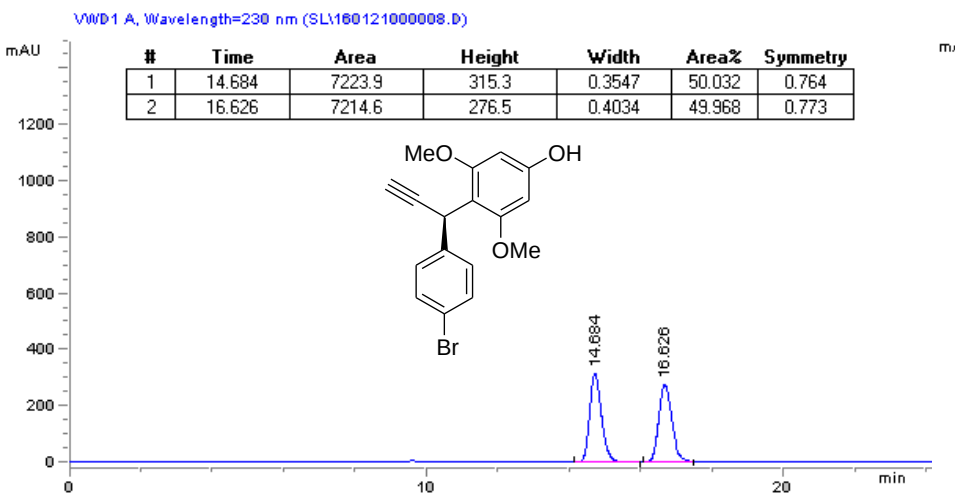


#	Time	Area	Height	Width	Area%	Symmetry
1	20.333	4878.9	158.3	0.4731	49.888	0.765
2	21.986	4900.7	145.6	0.5121	50.112	0.754

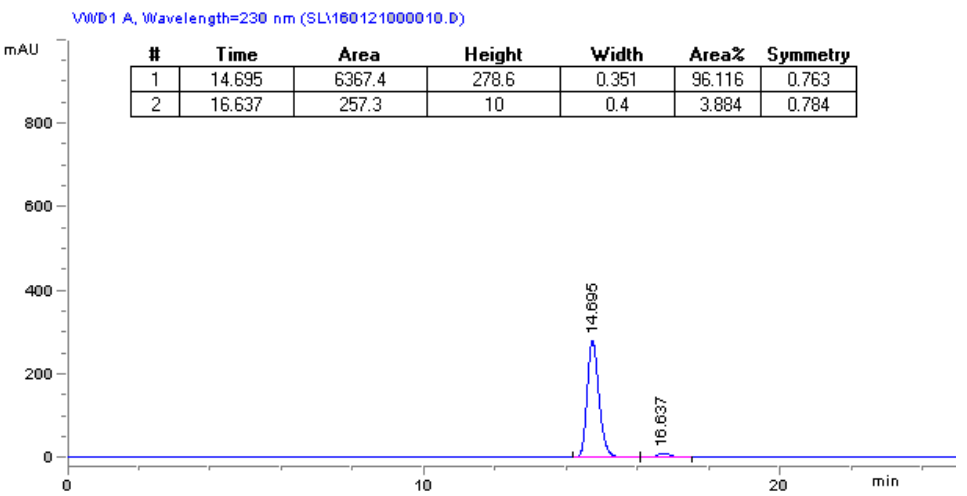


#	Time	Area	Height	Width	Area%	Symmetry
1	20.148	8439.4	273	0.4771	94.913	0.737
2	21.802	452.3	13	0.5266	5.087	0.748

4-((*R*)-1-(4-bromophenyl)prop-2-ynyl)-3,5-dimethoxyphenol (3fa).

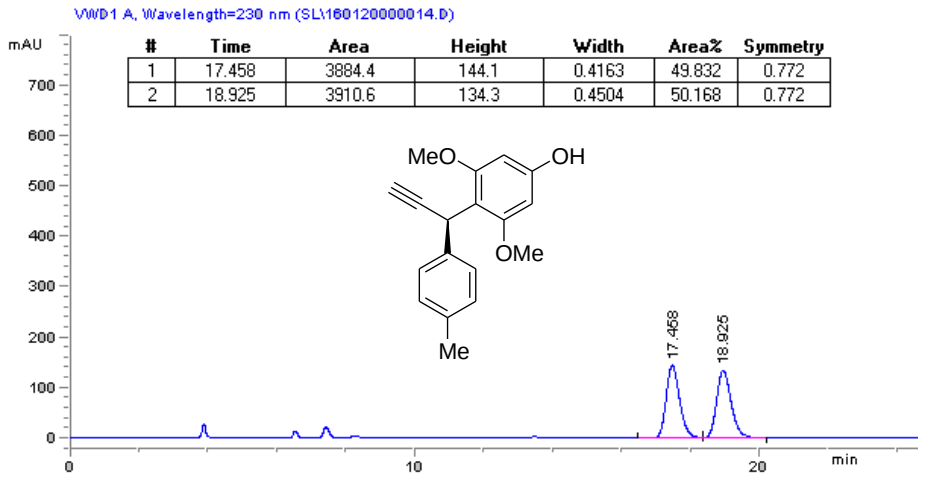


#	Time	Area	Height	Width	Area%	Symmetry
1	14.684	7223.9	315.3	0.3547	50.032	0.764
2	16.626	7214.6	276.5	0.4034	49.968	0.773

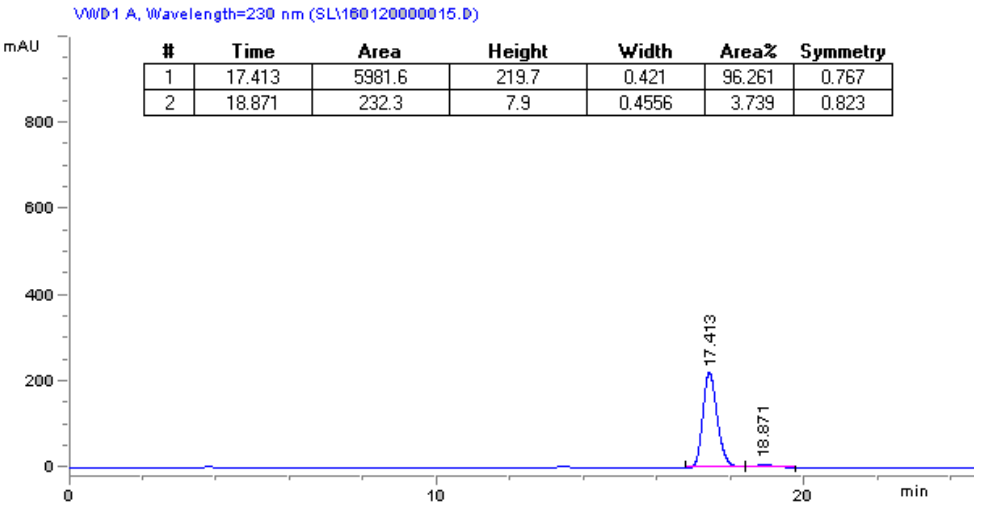


#	Time	Area	Height	Width	Area%	Symmetry
1	14.695	6367.4	278.6	0.351	96.116	0.763
2	16.637	257.3	10	0.4	3.884	0.784

3,5-dimethoxy-4-((*R*)-1-p-tolylprop-2-ynyl)phenol (3ga).

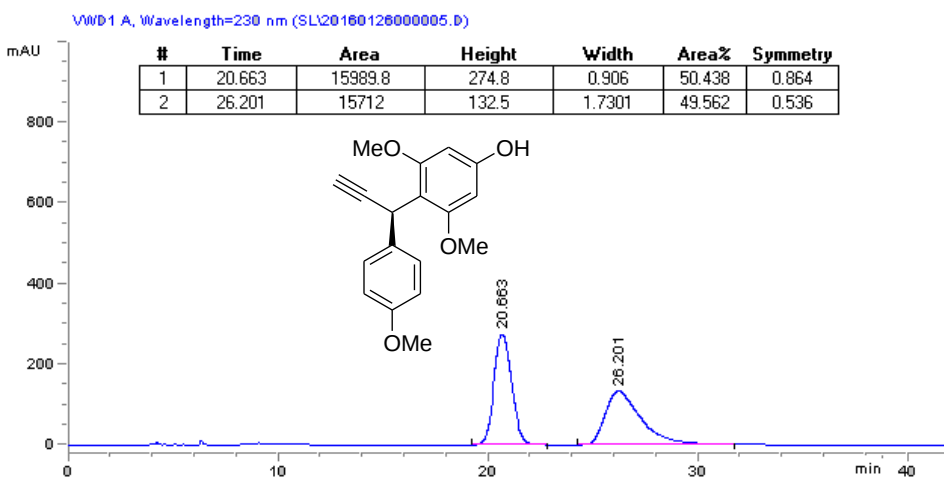


#	Time	Area	Height	Width	Area%	Symmetry
1	17.458	3884.4	144.1	0.4163	49.832	0.772
2	18.925	3910.6	134.3	0.4504	50.168	0.772

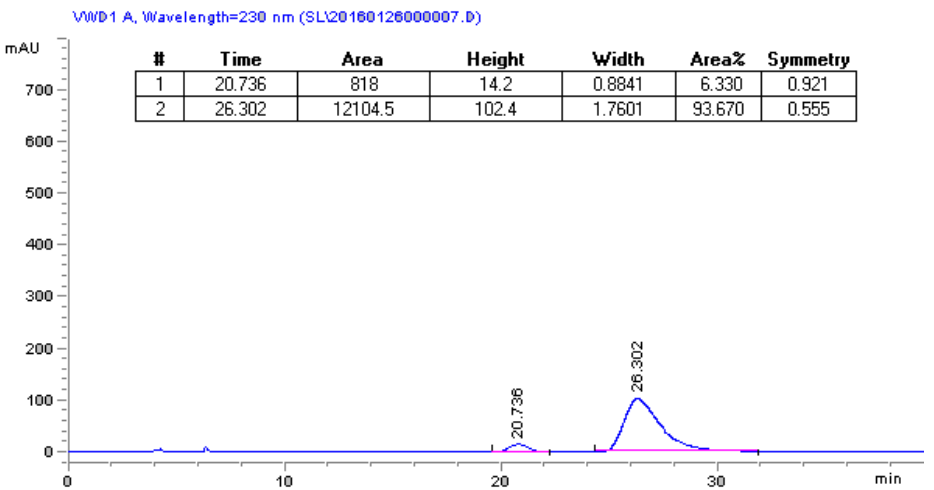


#	Time	Area	Height	Width	Area%	Symmetry
1	17.413	5981.6	219.7	0.421	96.261	0.767
2	18.871	232.3	7.9	0.4556	3.739	0.823

3,5-dimethoxy-4-((*R*)-1-(4-methoxyphenyl)prop-2-ynyl) phenol (3ha).

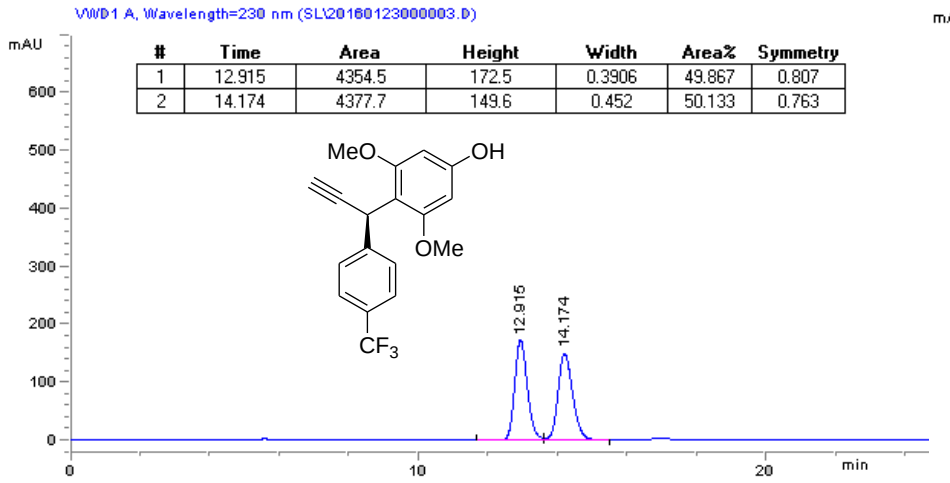


#	Time	Area	Height	Width	Area%	Symmetry
1	20.663	15989.8	274.8	0.906	50.438	0.864
2	26.201	15712	132.5	1.7301	49.562	0.536

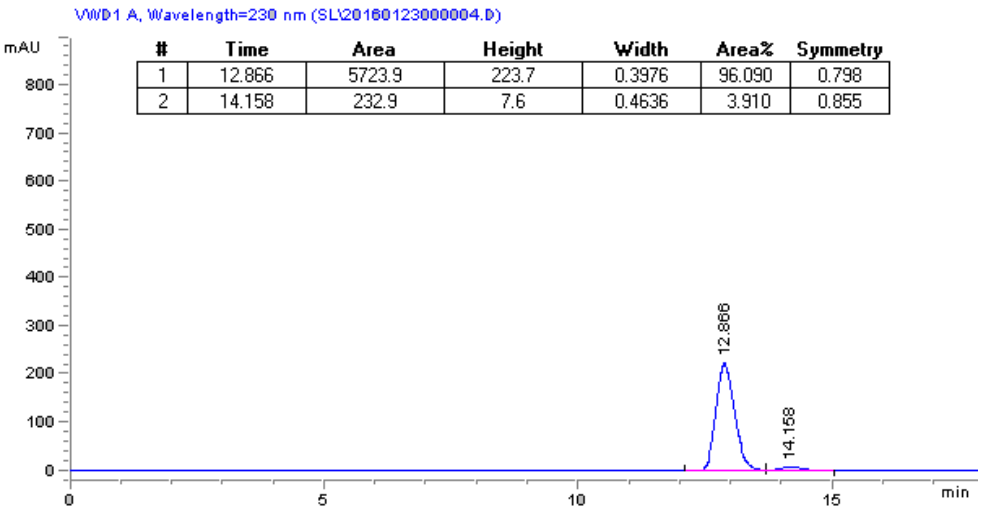


#	Time	Area	Height	Width	Area%	Symmetry
1	20.736	818	14.2	0.8841	6.330	0.921
2	26.302	12104.5	102.4	1.7601	93.670	0.555

4-((*R*)-1-(4-(trifluoromethyl)phenyl)prop-2-ynyl)-3,5-dimethoxyphenol (3ia).

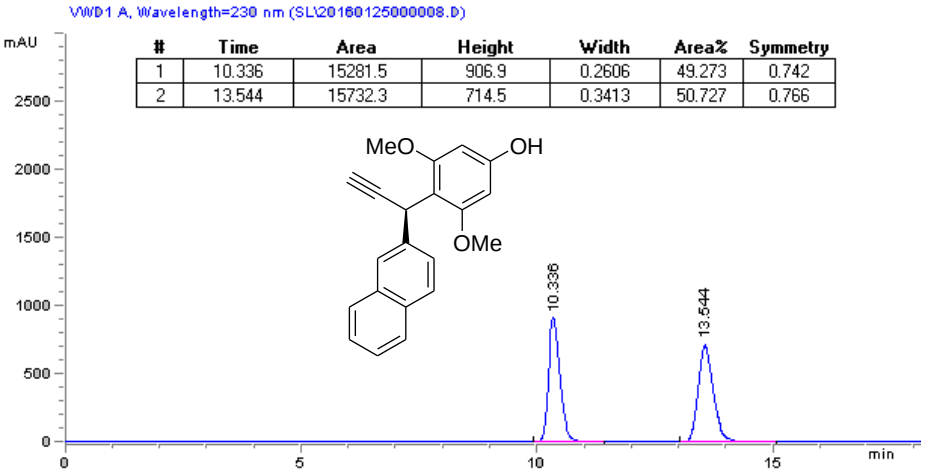


#	Time	Area	Height	Width	Area%	Symmetry
1	12.915	4354.5	172.5	0.3906	49.867	0.807
2	14.174	4377.7	149.6	0.452	50.133	0.763

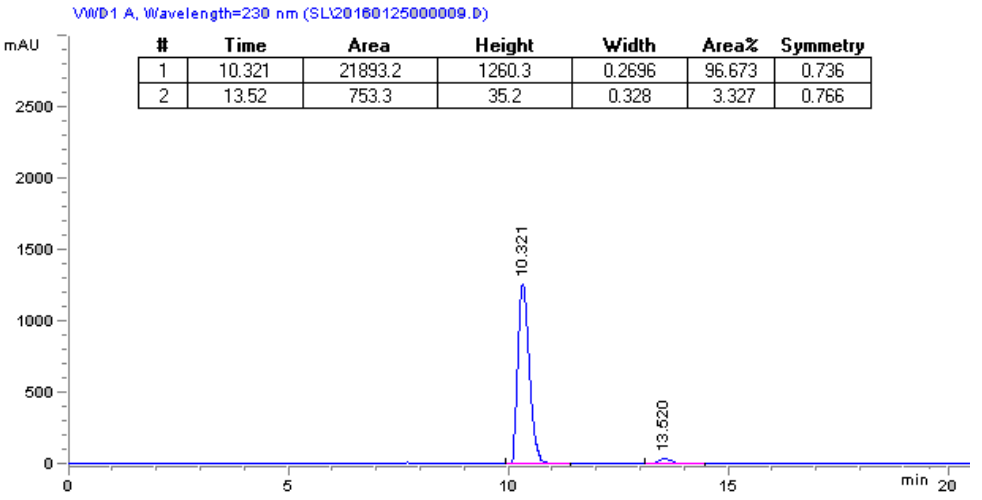


#	Time	Area	Height	Width	Area%	Symmetry
1	12.866	5723.9	223.7	0.3976	96.090	0.798
2	14.158	232.9	7.6	0.4636	3.910	0.855

3,5-dimethoxy-4-((*R*)-1-(naphthalen-3-yl)prop-2-ynyl)phenol (3ja).

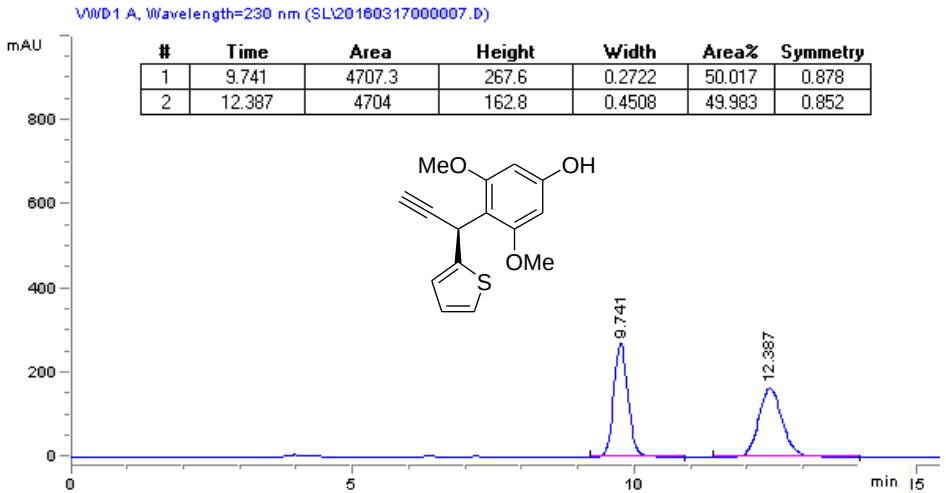


#	Time	Area	Height	Width	Area%	Symmetry
1	10.336	15281.5	906.9	0.2606	49.273	0.742
2	13.544	15732.3	714.5	0.3413	50.727	0.766

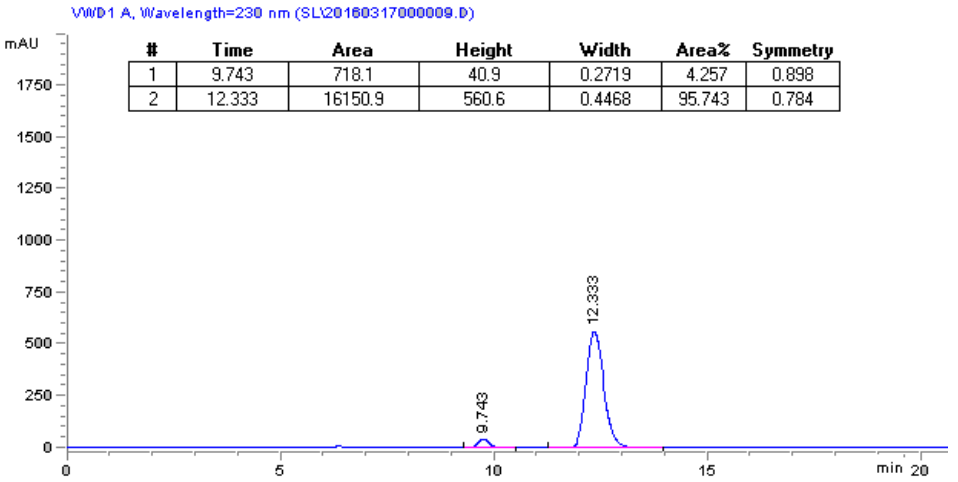


#	Time	Area	Height	Width	Area%	Symmetry
1	10.321	21893.2	1260.3	0.2696	96.673	0.736
2	13.52	753.3	35.2	0.328	3.327	0.766

3,5-dimethoxy-4-((*R*)-1-(thiophen-2-yl)prop-2-ynyl)phenol (3ka).

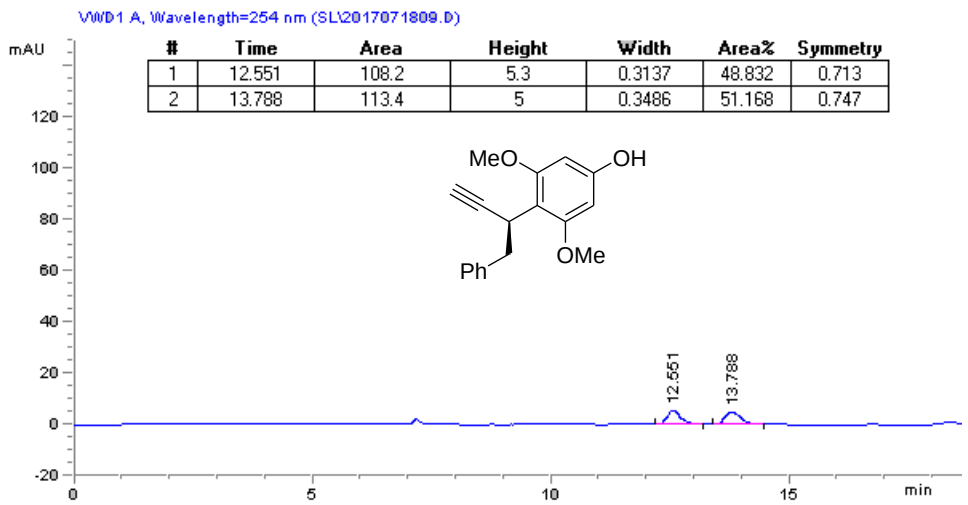


#	Time	Area	Height	Width	Area%	Symmetry
1	9.741	4707.3	267.6	0.2722	50.017	0.878
2	12.387	4704	162.8	0.4508	49.983	0.852

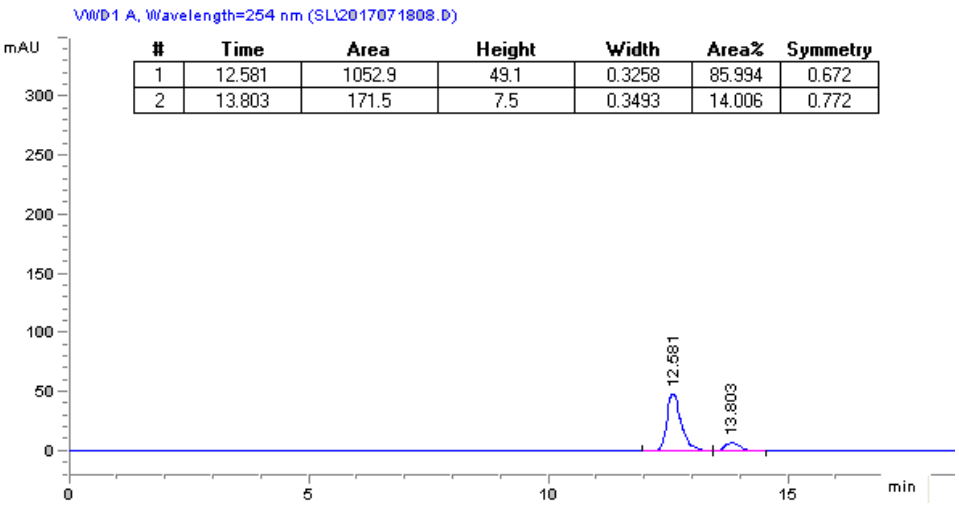


#	Time	Area	Height	Width	Area%	Symmetry
1	9.743	718.1	40.9	0.2719	4.257	0.898
2	12.333	16150.9	560.6	0.4468	95.743	0.784

3,5-dimethoxy-4-((*R*)-1-phenylbut-3-yn-2-yl)phenol (3la).

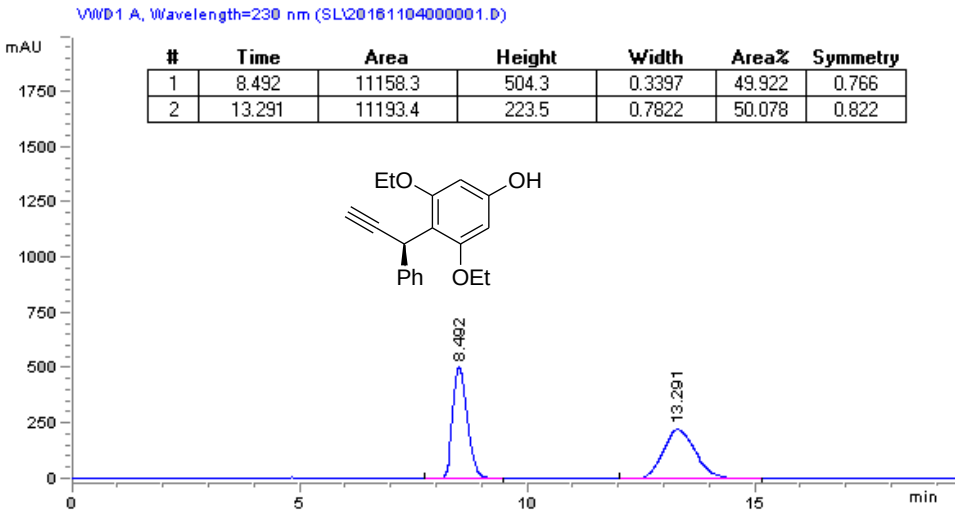


#	Time	Area	Height	Width	Area%	Symmetry
1	12.551	108.2	5.3	0.3137	48.832	0.713
2	13.788	113.4	5	0.3486	51.168	0.747

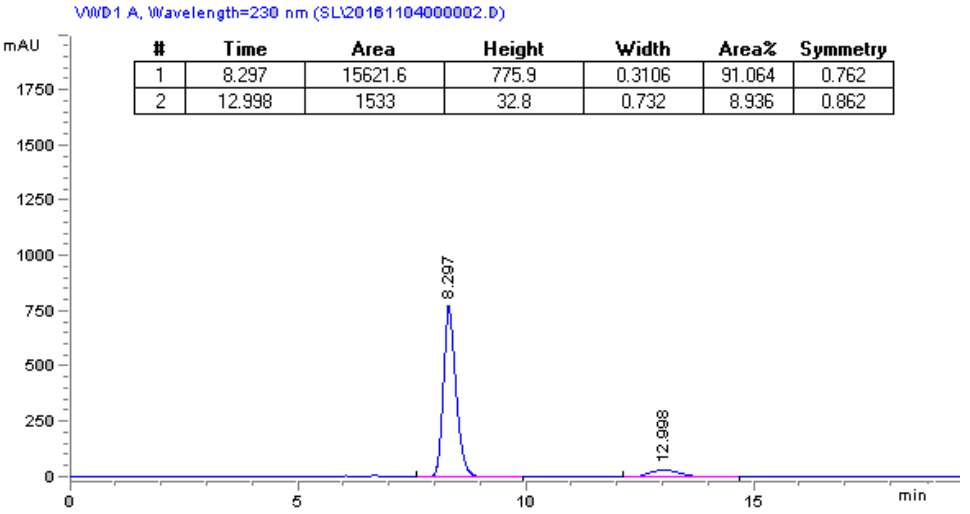


#	Time	Area	Height	Width	Area%	Symmetry
1	12.581	1052.9	49.1	0.3258	85.994	0.672
2	13.803	171.5	7.5	0.3493	14.006	0.772

3,5-diethoxy-4-((*R*)-1-phenylprop-2-ynyl)phenol (3ab).

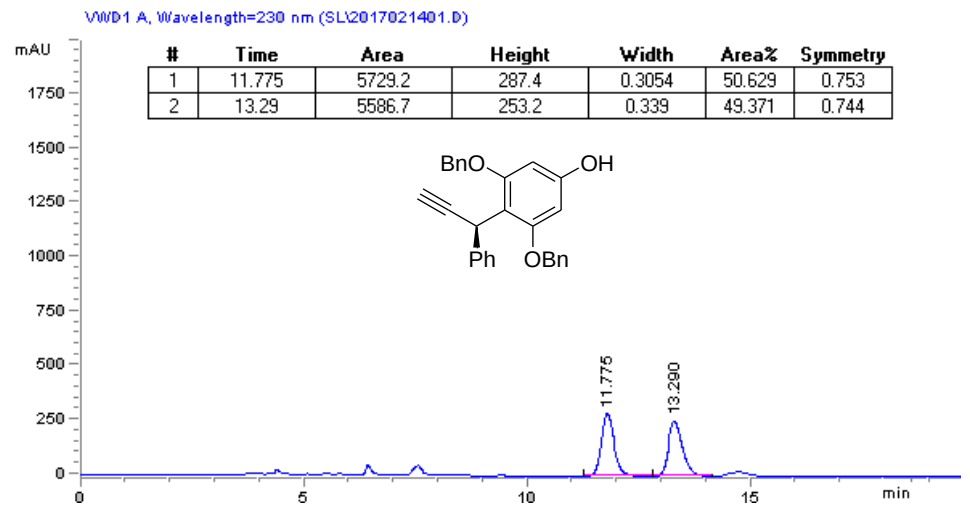


#	Time	Area	Height	Width	Area%	Symmetry
1	8.492	11158.3	504.3	0.3397	49.922	0.766
2	13.291	11193.4	223.5	0.7822	50.078	0.822

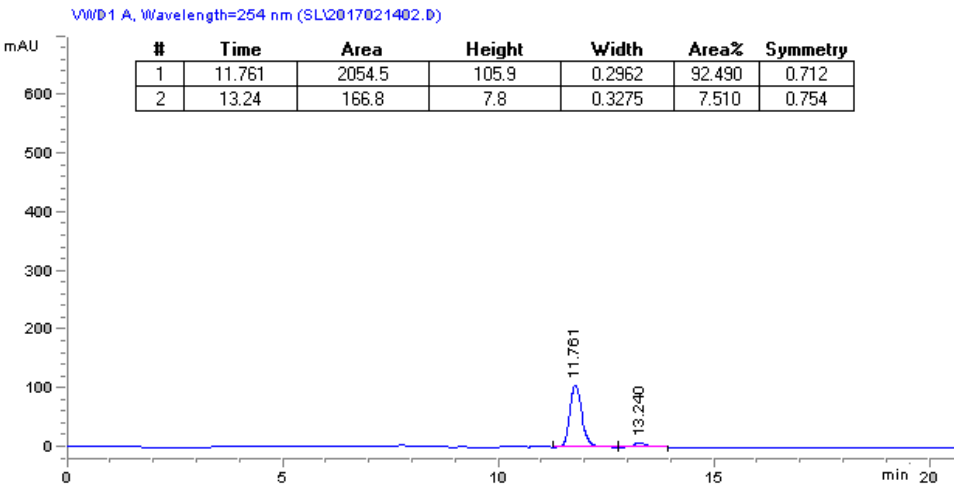


#	Time	Area	Height	Width	Area%	Symmetry
1	8.297	15621.6	775.9	0.3106	91.064	0.762
2	12.998	1533	32.8	0.732	8.936	0.862

3,5-bis(benzyloxy)-4-((*R*)-1-phenylprop-2-ynyl)phenol (3ac).

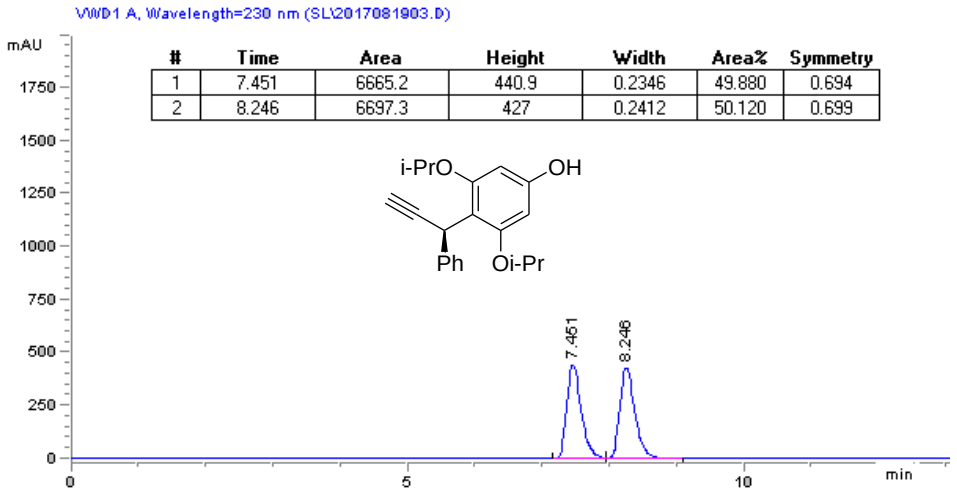


#	Time	Area	Height	Width	Area%	Symmetry
1	11.775	5729.2	287.4	0.3054	50.629	0.753
2	13.29	5586.7	253.2	0.339	49.371	0.744

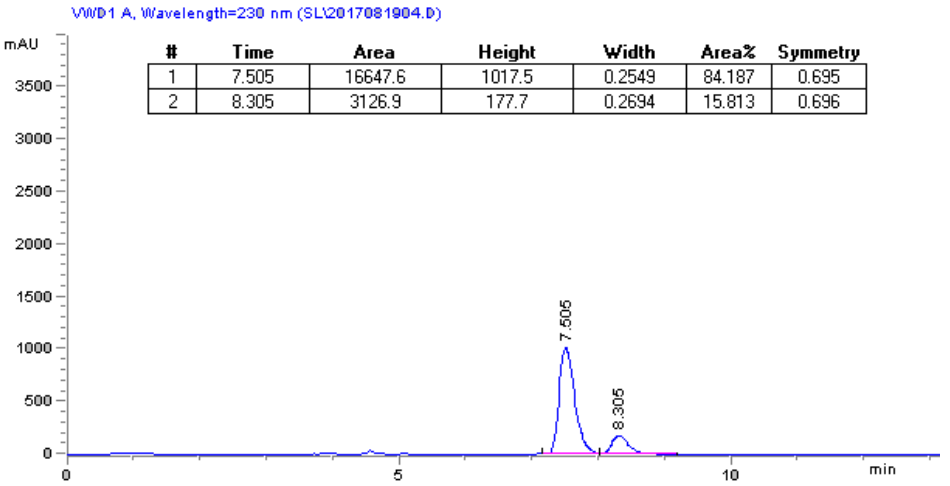


#	Time	Area	Height	Width	Area%	Symmetry
1	11.761	2054.5	105.9	0.2962	92.490	0.712
2	13.24	166.8	7.8	0.3275	7.510	0.754

3,5-diisopropoxy-4-((*R*)-1-phenylprop-2-ynyl)phenol (3ad).

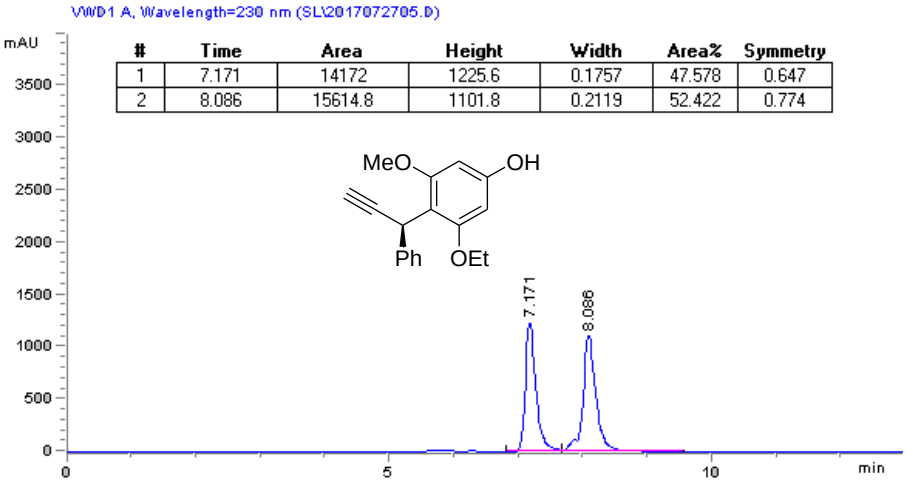


#	Time	Area	Height	Width	Area%	Symmetry
1	7.451	6665.2	440.9	0.2346	49.880	0.694
2	8.246	6697.3	427	0.2412	50.120	0.699

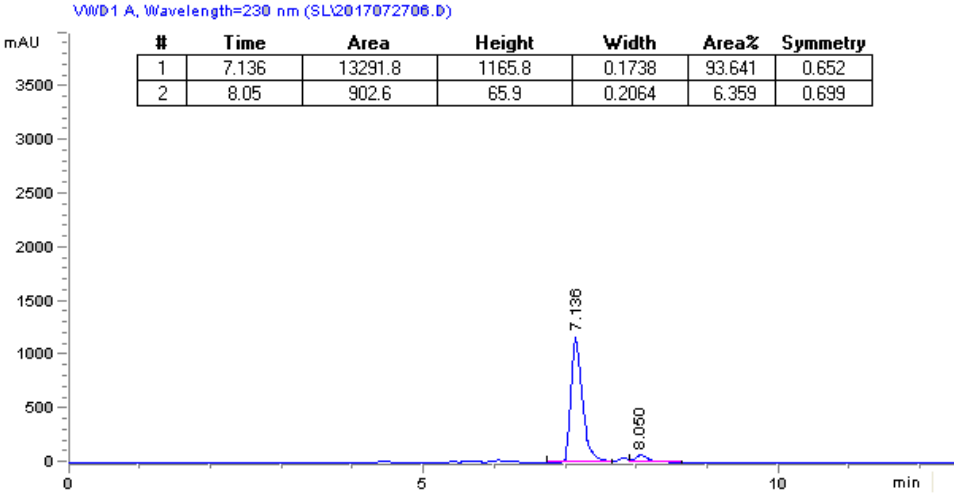


#	Time	Area	Height	Width	Area%	Symmetry
1	7.505	16647.6	1017.5	0.2549	84.187	0.695
2	8.305	3126.9	177.7	0.2694	15.813	0.696

3-ethoxy-5-methoxy-4-((*R*)-1-phenylprop-2-ynyl)phenol (3ae).

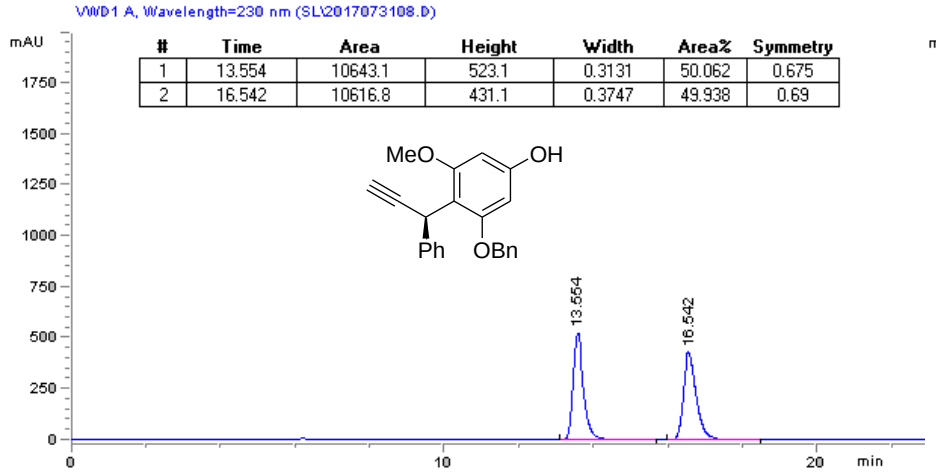


#	Time	Area	Height	Width	Area%	Symmetry
1	7.171	14172	1225.6	0.1757	47.578	0.647
2	8.086	15614.8	1101.8	0.2119	52.422	0.774

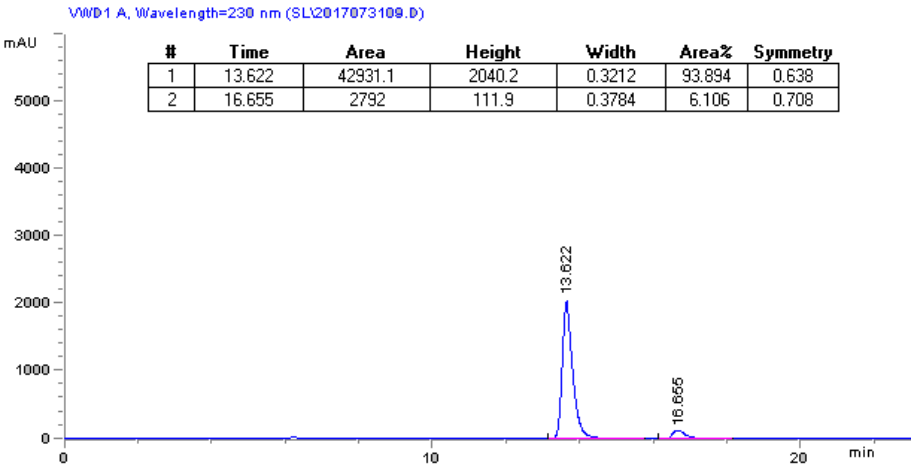


#	Time	Area	Height	Width	Area%	Symmetry
1	7.136	13291.8	1165.8	0.1738	93.641	0.652
2	8.05	902.6	65.9	0.2064	6.359	0.699

3-(benzyloxy)-5-methoxy-4-((*R*)-1-phenylprop-2-ynyl)phenol (3af).

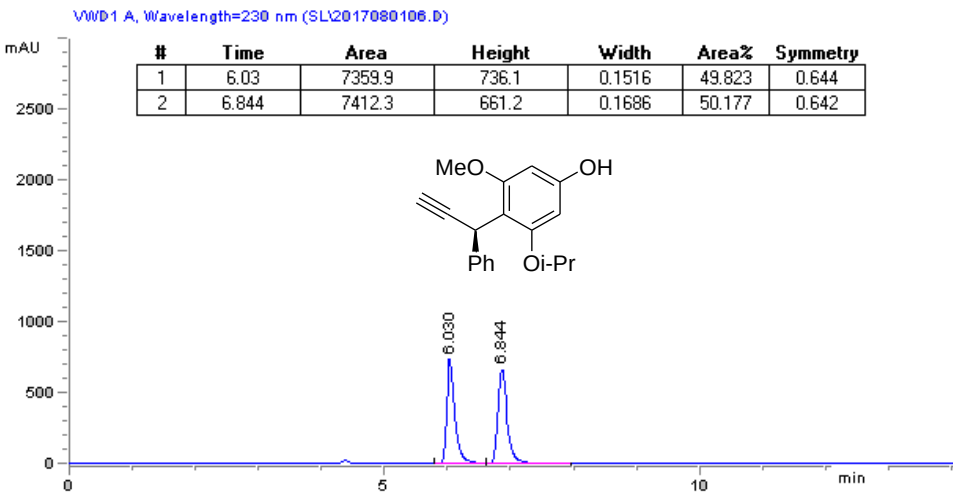


#	Time	Area	Height	Width	Area%	Symmetry
1	13.554	10643.1	523.1	0.3131	50.062	0.675
2	16.542	10616.8	431.1	0.3747	49.938	0.69

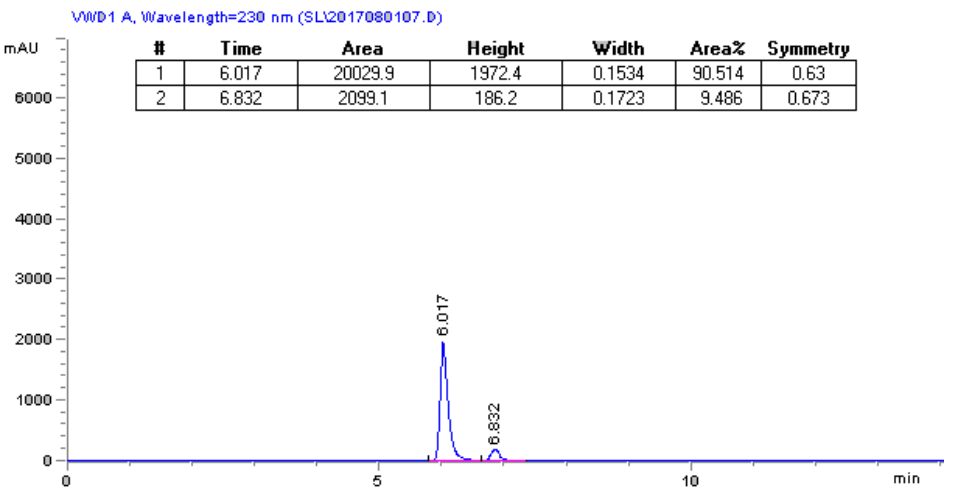


#	Time	Area	Height	Width	Area%	Symmetry
1	13.622	42931.1	2040.2	0.3212	93.894	0.638
2	16.655	2792	111.9	0.3784	6.106	0.708

3-isopropoxy-5-methoxy-4-((*R*)-1-phenylprop-2-ynyl)phenol (3ag).

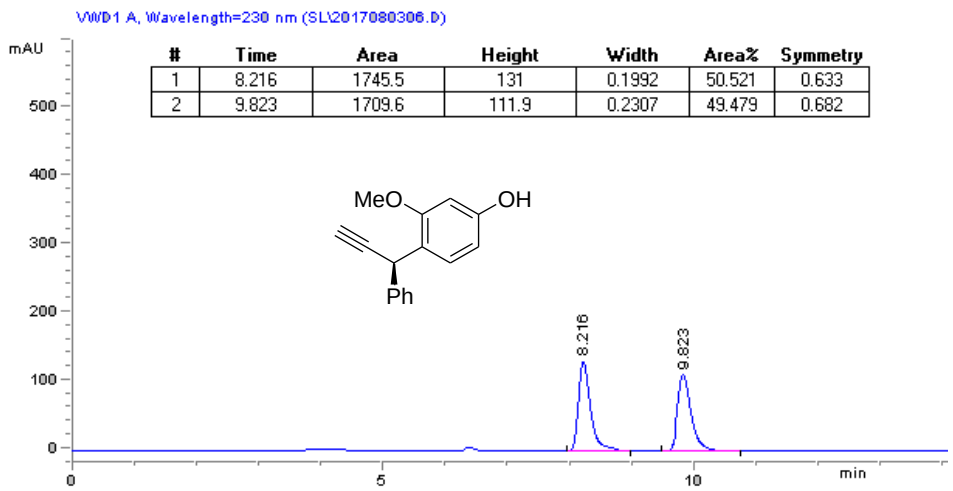


#	Time	Area	Height	Width	Area%	Symmetry
1	6.03	7359.9	736.1	0.1516	49.823	0.644
2	6.844	7412.3	661.2	0.1686	50.177	0.642

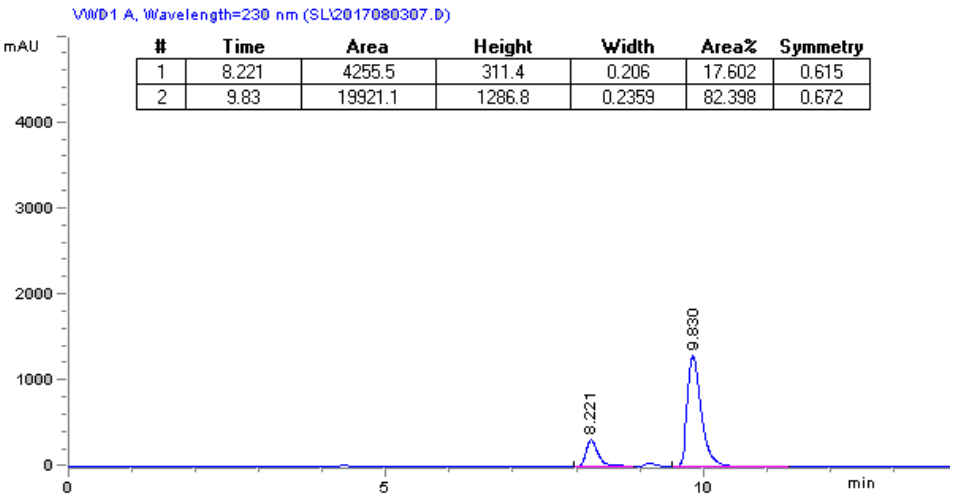


#	Time	Area	Height	Width	Area%	Symmetry
1	6.017	20029.9	1972.4	0.1534	90.514	0.63
2	6.832	2099.1	186.2	0.1723	9.486	0.673

3-methoxy-4-((*R*)-1-phenylprop-2-ynyl)phenol (3ai).

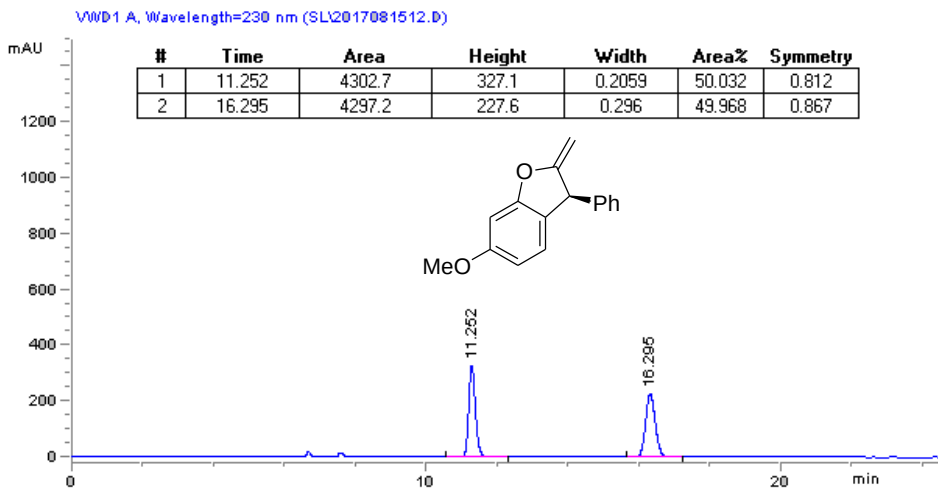


#	Time	Area	Height	Width	Area%	Symmetry
1	8.216	1745.5	131	0.1992	50.521	0.633
2	9.823	1709.6	111.9	0.2307	49.479	0.682

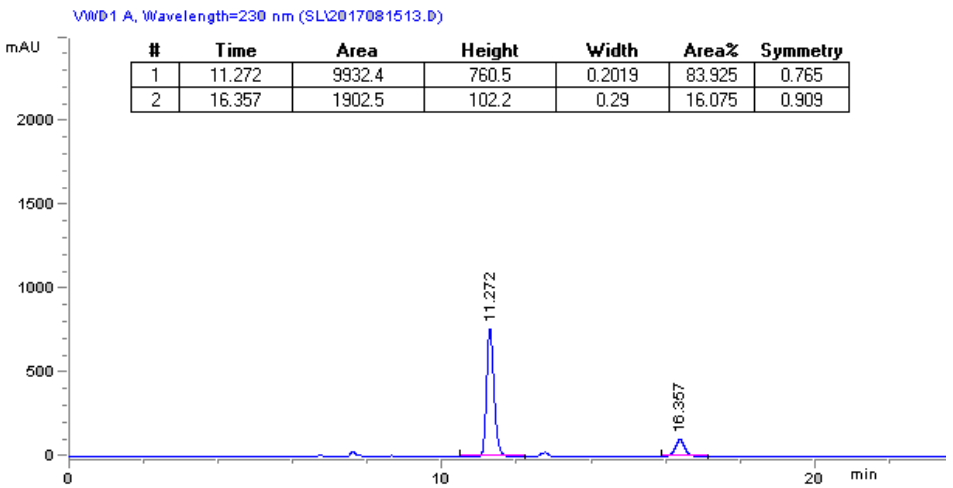


#	Time	Area	Height	Width	Area%	Symmetry
1	8.221	4255.5	311.4	0.206	17.602	0.615
2	9.83	19921.1	1286.8	0.2359	82.398	0.672

(*S*)-2,3-dihydro-5-methoxy-2-methylene-1-phenyl-1H-indene (5).

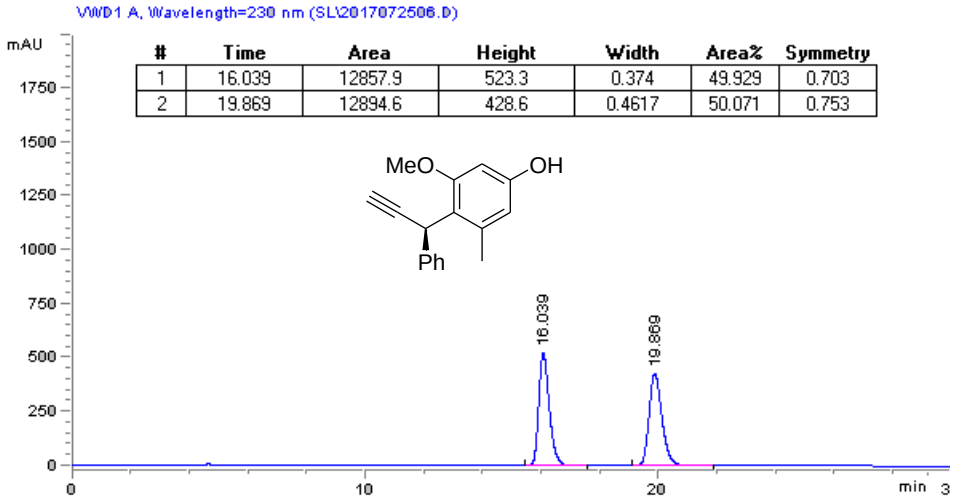


#	Time	Area	Height	Width	Area%	Symmetry
1	11.252	4302.7	327.1	0.2059	50.032	0.812
2	16.295	4297.2	227.6	0.296	49.968	0.867

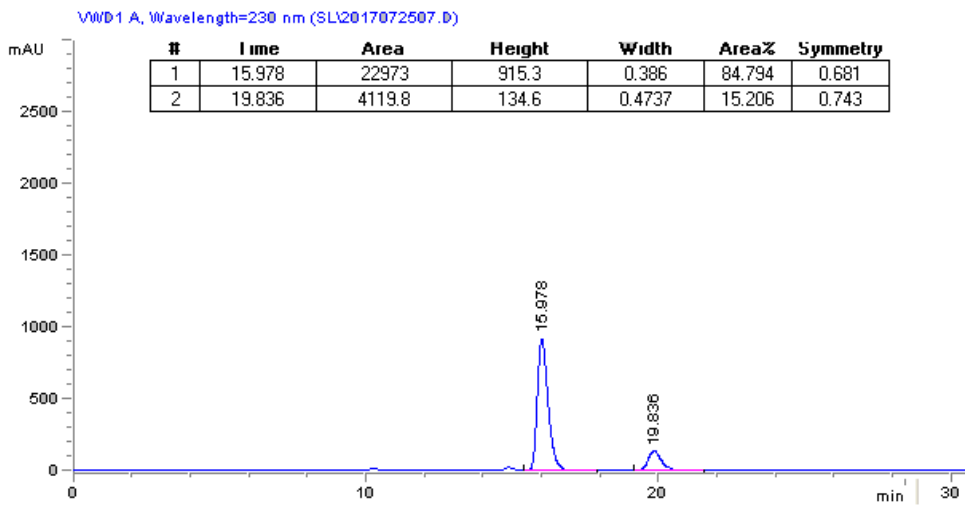


#	Time	Area	Height	Width	Area%	Symmetry
1	11.272	9932.4	760.5	0.2019	83.925	0.765
2	16.357	1902.5	102.2	0.29	16.075	0.909

3-methoxy-5-methyl-4-((*R*)-1-phenylprop-2-ynyl)phenol (3aj).

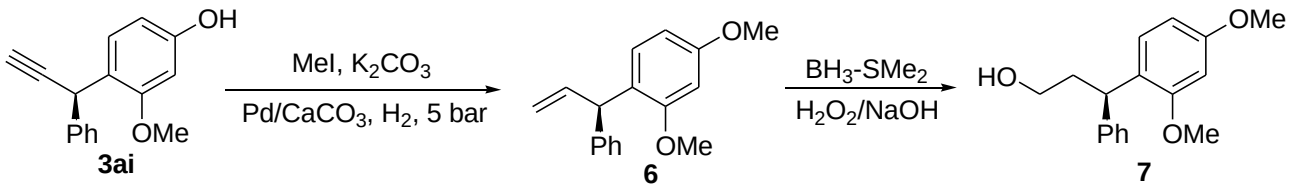


#	Time	Area	Height	Width	Area%	Symmetry
1	16.039	12857.9	523.3	0.374	49.929	0.703
2	19.869	12894.6	428.6	0.4617	50.071	0.753



#	Time	Area	Height	Width	Area%	Symmetry
1	15.978	22973	915.3	0.386	84.794	0.681
2	19.836	4119.8	134.6	0.4737	15.206	0.743

Synthesis of (*R*)-3-(2,4-dimethoxyphenyl)-3-phenylpropan-1-ol for the determination of absolute stereochemistry of propargylic alkylation products



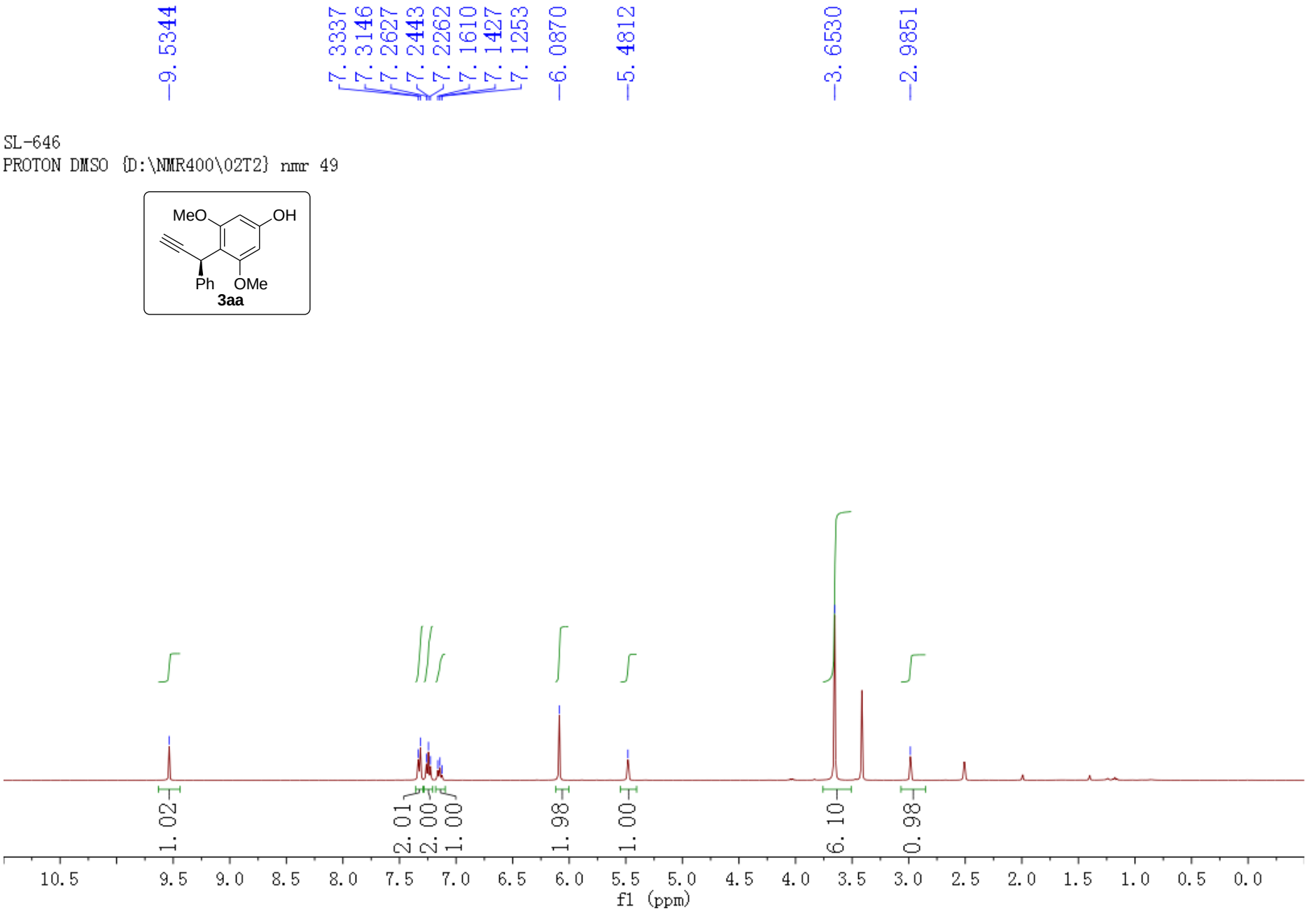
MeI (1.1 equiv) was added to a solution of **3ai** (100 mg, 0.42 mmol) and Cs_2CO_3 (1.2 equiv) in 3 mL of acetone. After being stirred at room temperature for 12 h, the solvent was removed in vacuo and purified by silica gel column chromatography (hexanes/AcOEt, 50/1). The product was added to an autoclave with $Pd/CaCO_3$ and 2 mL EtOH, then the hydrogenation was performed at room temperature under 5 bar of H_2 pressure for 2 h. After concentration of the reaction mixture under reduced pressure, the residue was purified by silica gel chromatography (hexanes/AcOEt, 50/1) to afford **6** (67% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27–7.25 (m, 2H), 7.21–7.14 (m, 3H), 7.01 (d, *J* = 9.0 Hz, 1H), 6.45–6.42 (m, 2H), 6.3–6.23 (m,

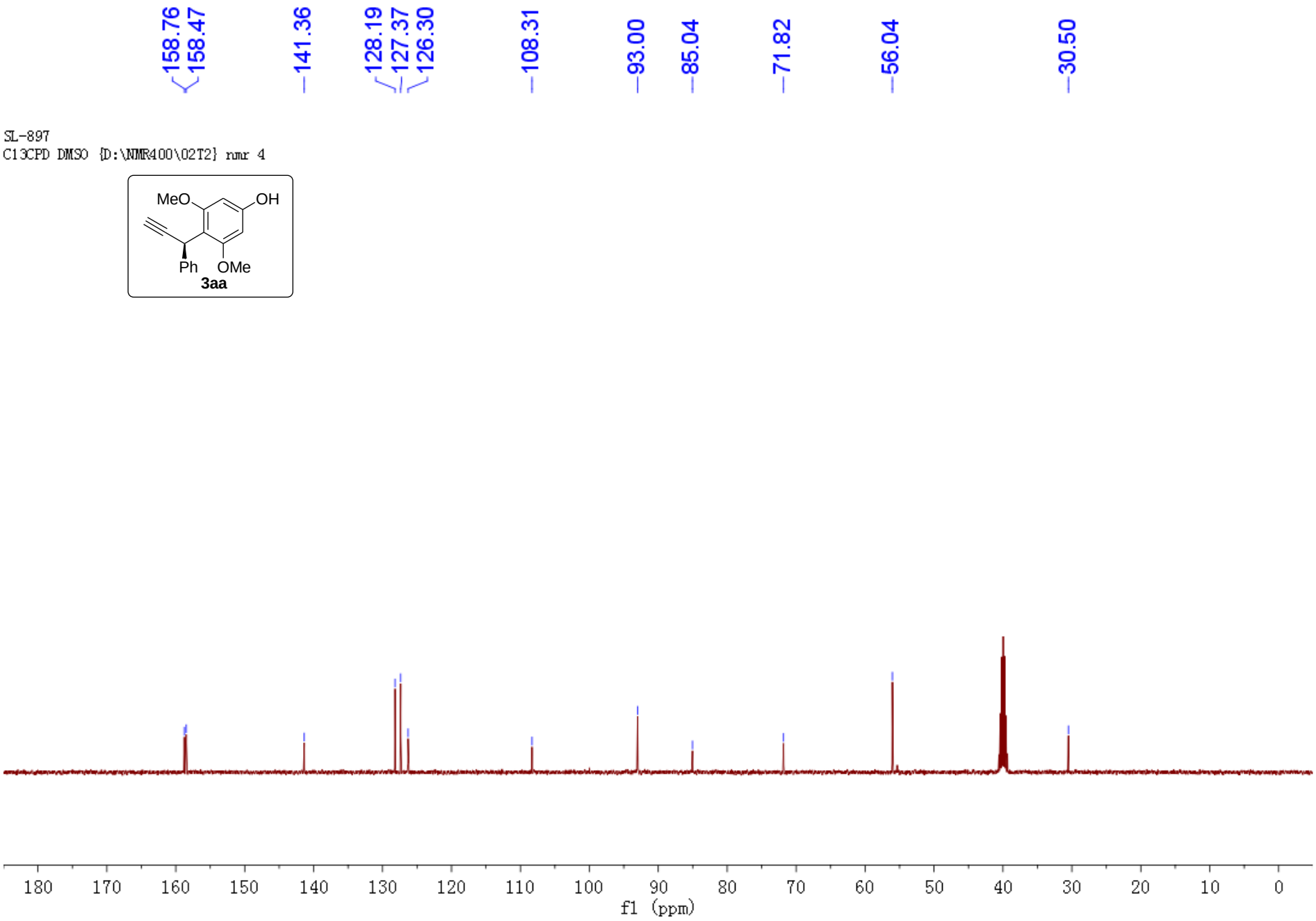
1H), 5.16 (d, *J* = 10.2 Hz, 1H), 5.06 (d, *J* = 6.6 Hz, 1H), 4.89 (d, *J* = 17.1 Hz, 1H), 3.77 (s, 3H), 3.71 (s, 3H). $[\alpha]^{21}_{D}$ = +0.6 (*c* = 1.00, $CHCl_3$, Lit. -1.0).[ref. 1]

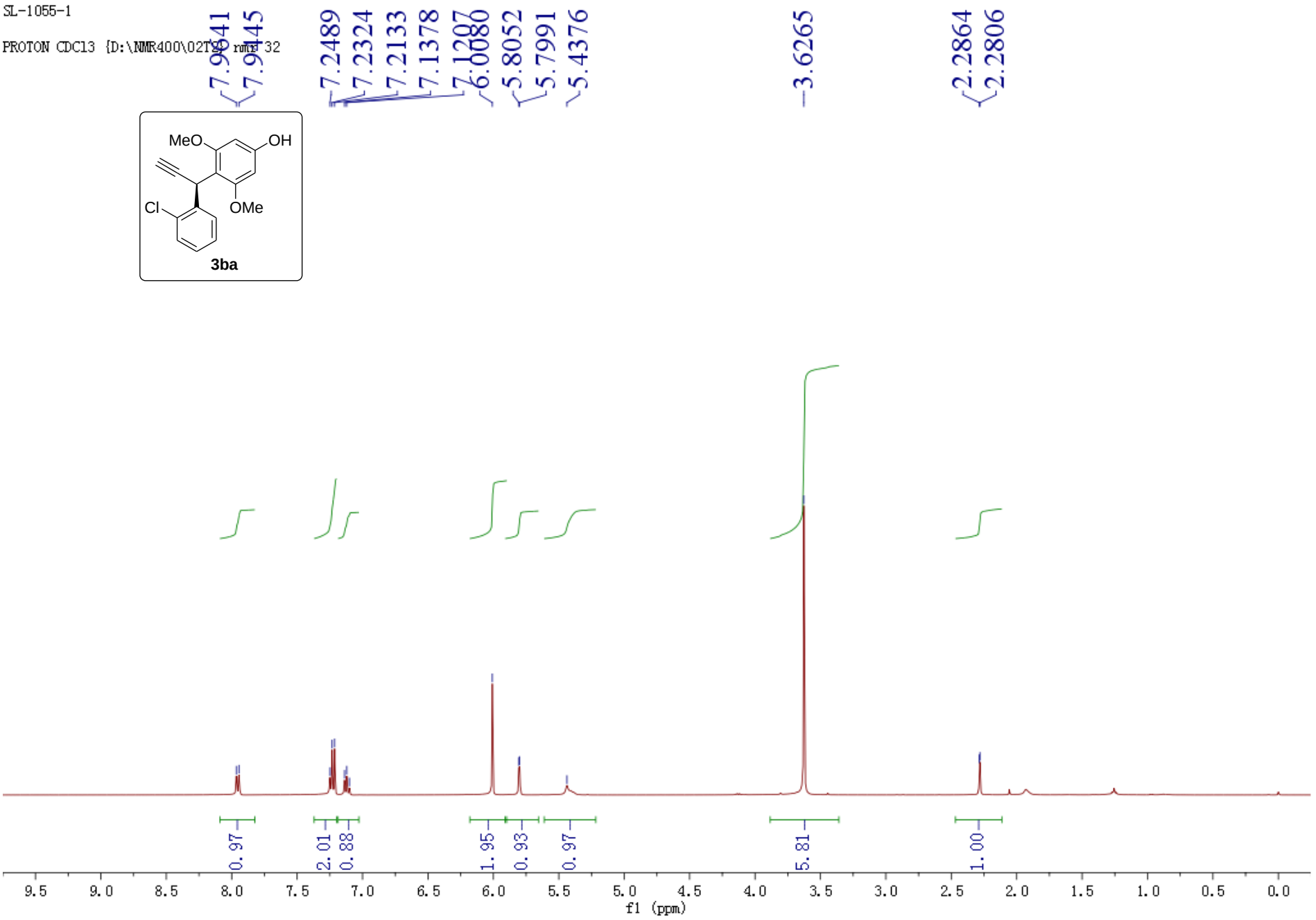
$BH_3 \cdot SMe_2$ (1.0 equiv) was added to a solution of **6** (1.0 equiv) in THF at 0 ℃. The mixture was stirred for 10 min at 0 ℃ and for 1 h at room temperature. 15% NaOH*aq* (1.5 equiv) and 30% H_2O_2*aq* (2.0 equiv) were successively added to it at 0 ℃, and the resulting mixture was stirred for 1 h at room temperature. The reaction was quenched with saturated NaCl*aq* and extracted with Et_2O. The organic layer was dried over $MgSO_4$, filtered, and concentrated under vacuum. The residue was purified by silica gel column chromatography to afford **7**(59% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 (d, *J* = 4.5 Hz, 4H), 7.16–7.15 (m, 1H), 7.04 (d, *J* = 9.1 Hz, 1H), 6.45–6.43 (m, 2H), 4.52–4.48 (m, 1H), 3.78 (s, 3H), 3.76 (s, 3H), 3.62–3.52 (m, 2H), 2.32–2.27 (m, 1H), 2.20–2.17 (m, 1H). $[\alpha]^{19}_{D}$ = +32.5 (*c* = 1.00, $CHCl_3$, Lit. +48.0).[ref. 1]

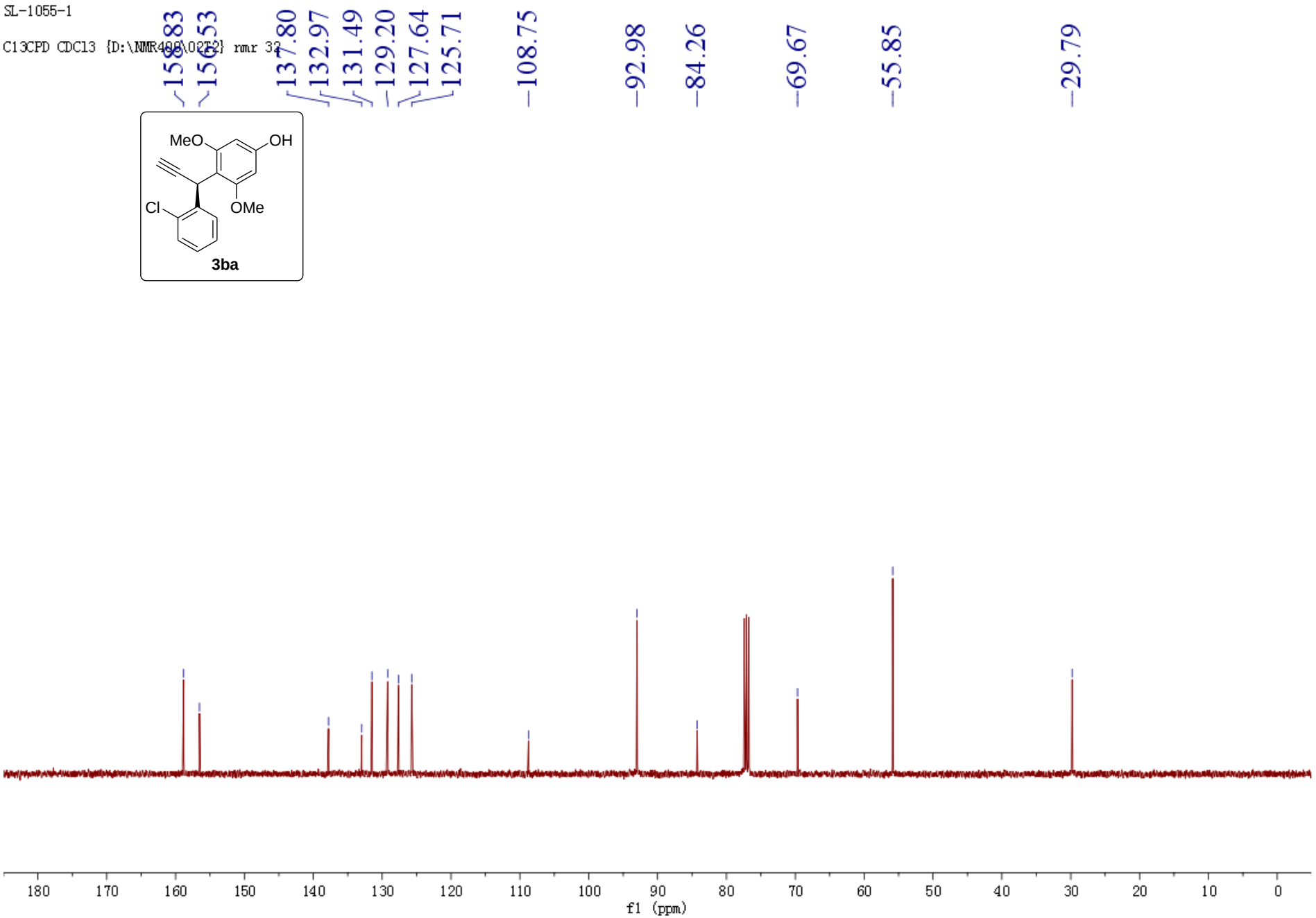
The absolute configuration of **3ai** was determined by the comparison with the known compound **7** after the derivatization, to which an *R*-absolute configuration is assigned.

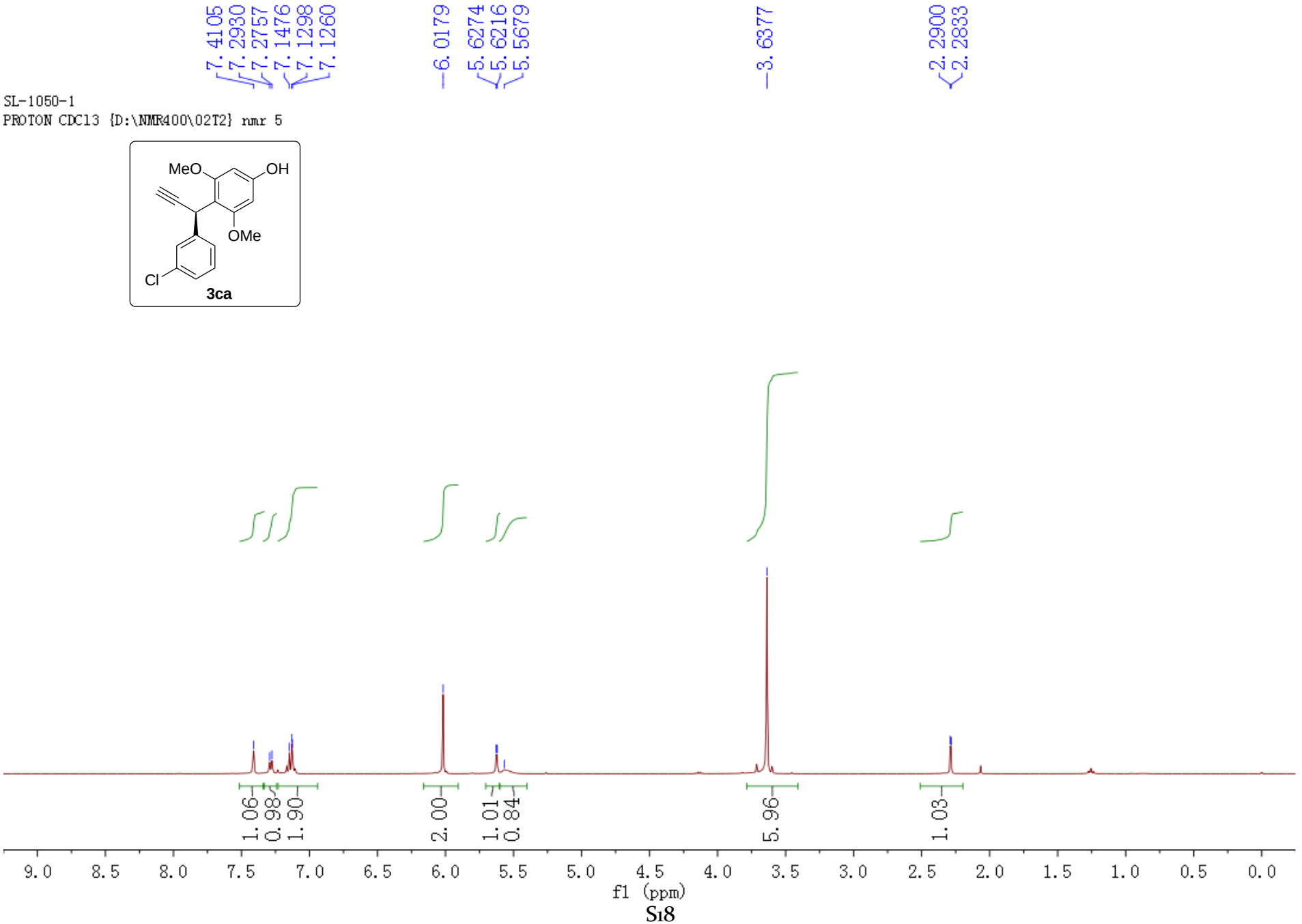
Ref. 1. Y. Luan and S. E. Schaus, *J. Am. Chem. Soc.*, 2012, **134**, 19965.


9.5344
7.3337
7.3146
7.2627
7.2443
7.2262
7.1610
7.1427
7.1253
6.0870
5.4812
3.6530
2.9851
SL-646
PROTON DMSO {D:\NMR400\02T2} nmr 49
MeO
OH
Ph
OMe
3aa
1.02
2.01
2.00
1.00
1.98
1.00
6.10
0.98
10.5
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
f1 (ppm)


158.76
158.47
141.36
128.19
127.37
126.30
108.31
93.00
85.04
71.82
56.04
30.50
SL-897
C13CPD DMSO {D:\NMR400\02T2} rumr 4
MeO
OH
Ph
OMe
3aa
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

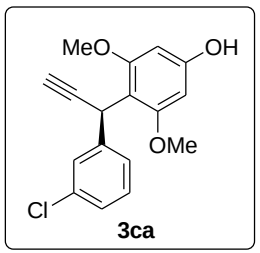

SL-1055-1
PROTON CDCl3 {D:\NMR400\02T
7.9641
7.9445
7.2489
7.2324
7.2133
7.1378
7.1207
6.0080
5.8052
5.7991
5.4376
3.6265
2.2864
2.2806
MeO
OH
Cl
OMe
3ba
0.97
2.01
0.88
1.95
0.93
0.97
5.81
1.00
f1 (ppm)


SL-1055-1
C13CPD CDCl3 {D:\NMR400\02[2]} rmr 32
158.83
156.53
137.80
132.97
131.49
129.20
127.64
125.71
108.75
92.98
84.26
69.67
55.85
29.79
MeO
OH
Cl
OMe
3ba
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

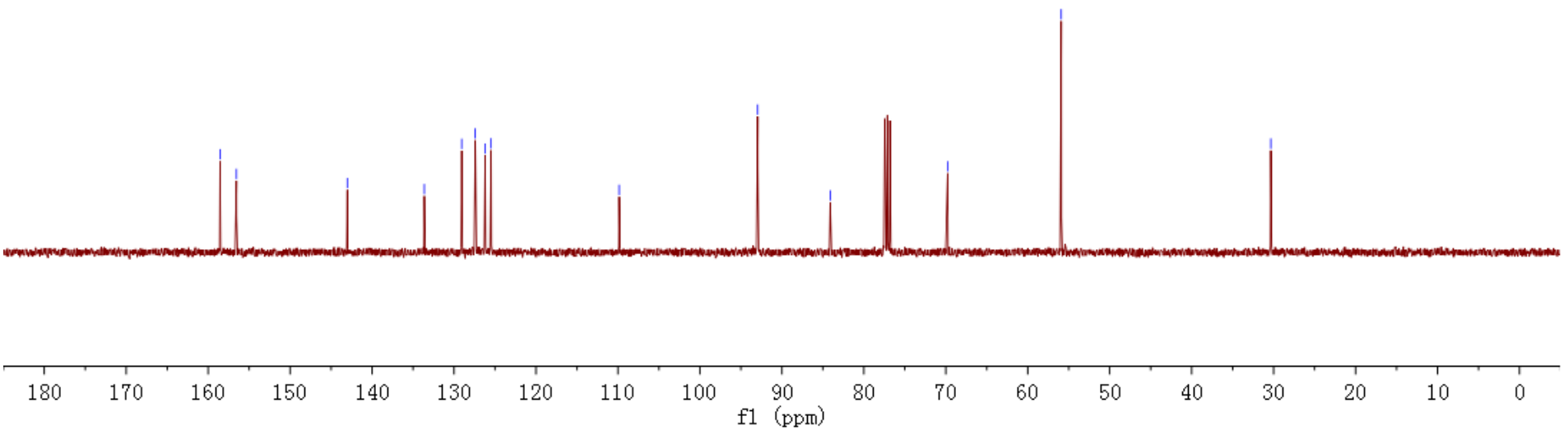

7.4105
7.2930
7.2757
7.1476
7.1298
7.1260
6.0179
5.6274
5.6216
5.5679
3.6377
2.2900
2.2833
SL-1050-1
PROTON CDC13 {D:\NMR400\02T2} nmr 5
MeO
OH
OMe
Cl
3ca
1.06
0.98
1.90
2.00
1.01
0.84
5.96
1.03
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

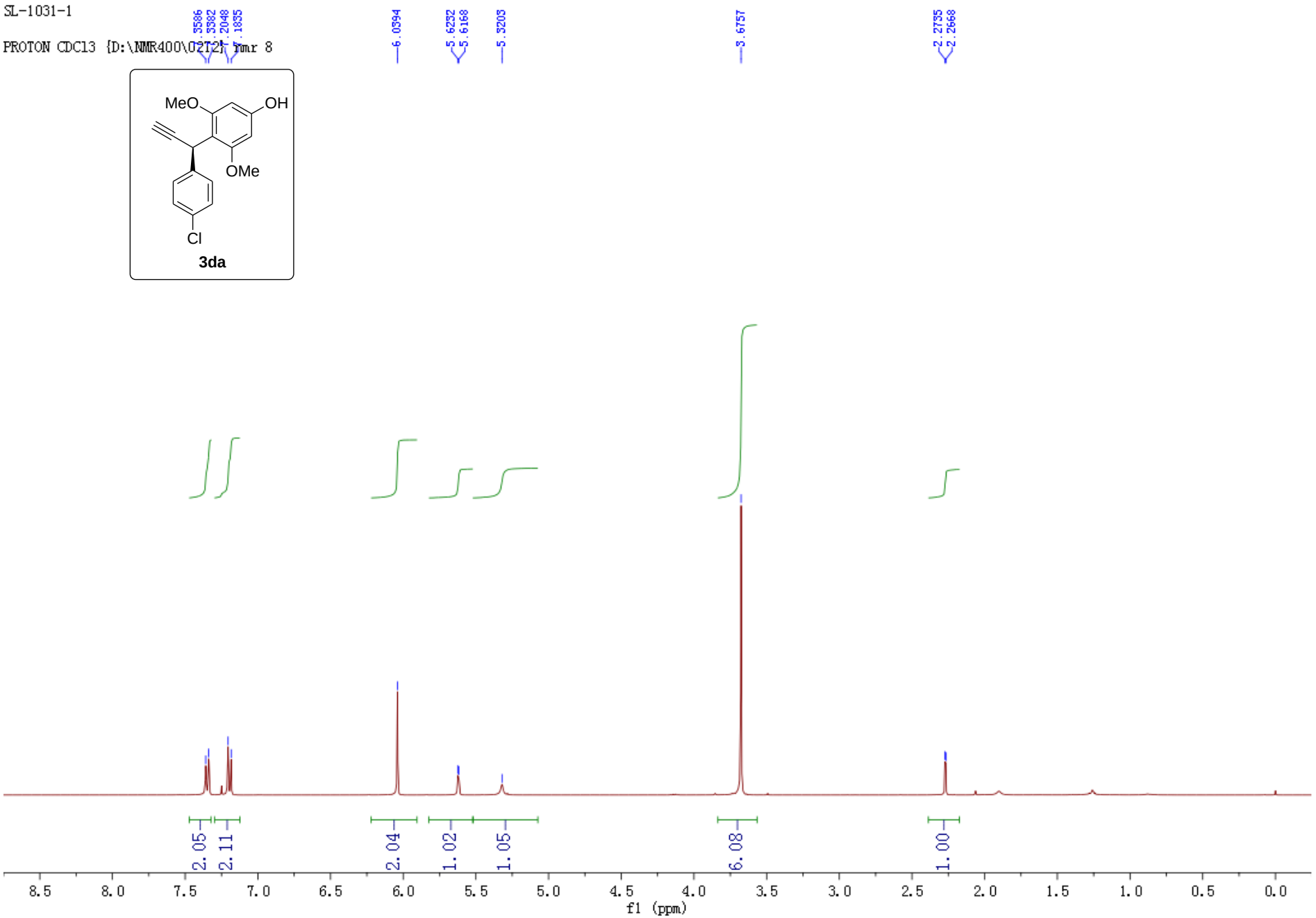
158.54
156.55
143.01
133.63
129.06
127.41
126.20
125.48
109.85
92.95
84.11
69.78
55.91
30.34

SL-1050-1
C13CPD CDCl3 {D:\NMR400\02T2} rmr 5



3ca

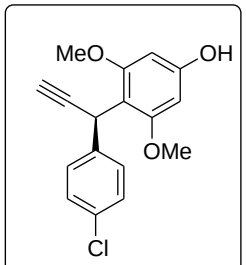



SL-1031-1
PROTON CDCl3 {D:\NMR400\DATA} nmr 8
7.3586
7.3382
7.2048
7.1835
6.0394
5.6232
5.6168
5.3203
3.6757
2.2735
2.2668
MeO
OH
OMe
Cl
3da
2.05
2.11
2.04
1.02
1.05
6.08
1.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

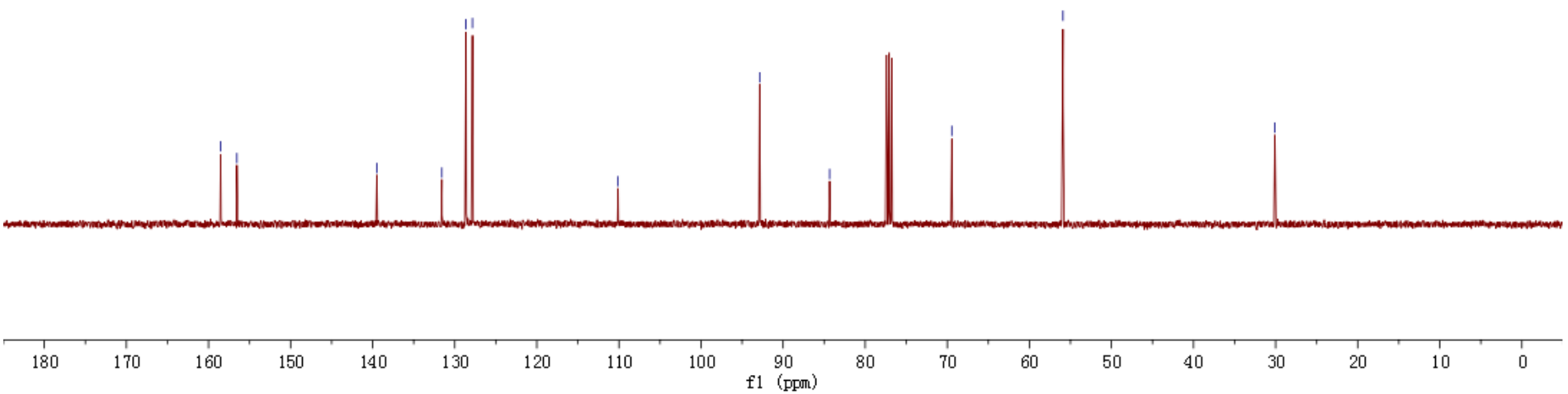
SL-1031-1

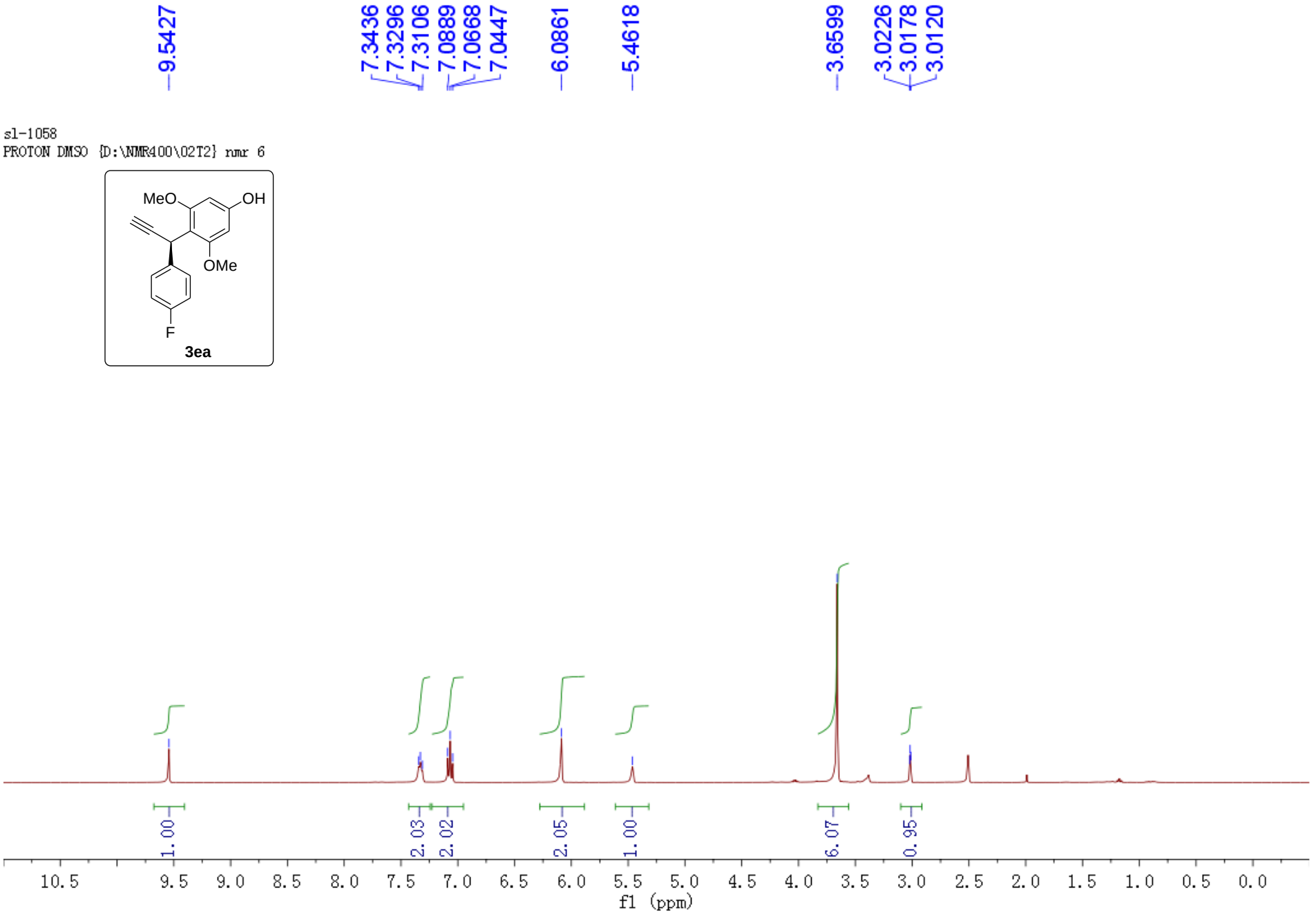
C13CPD CDCl3 {D:\NMR400\DZT2} rmr 8

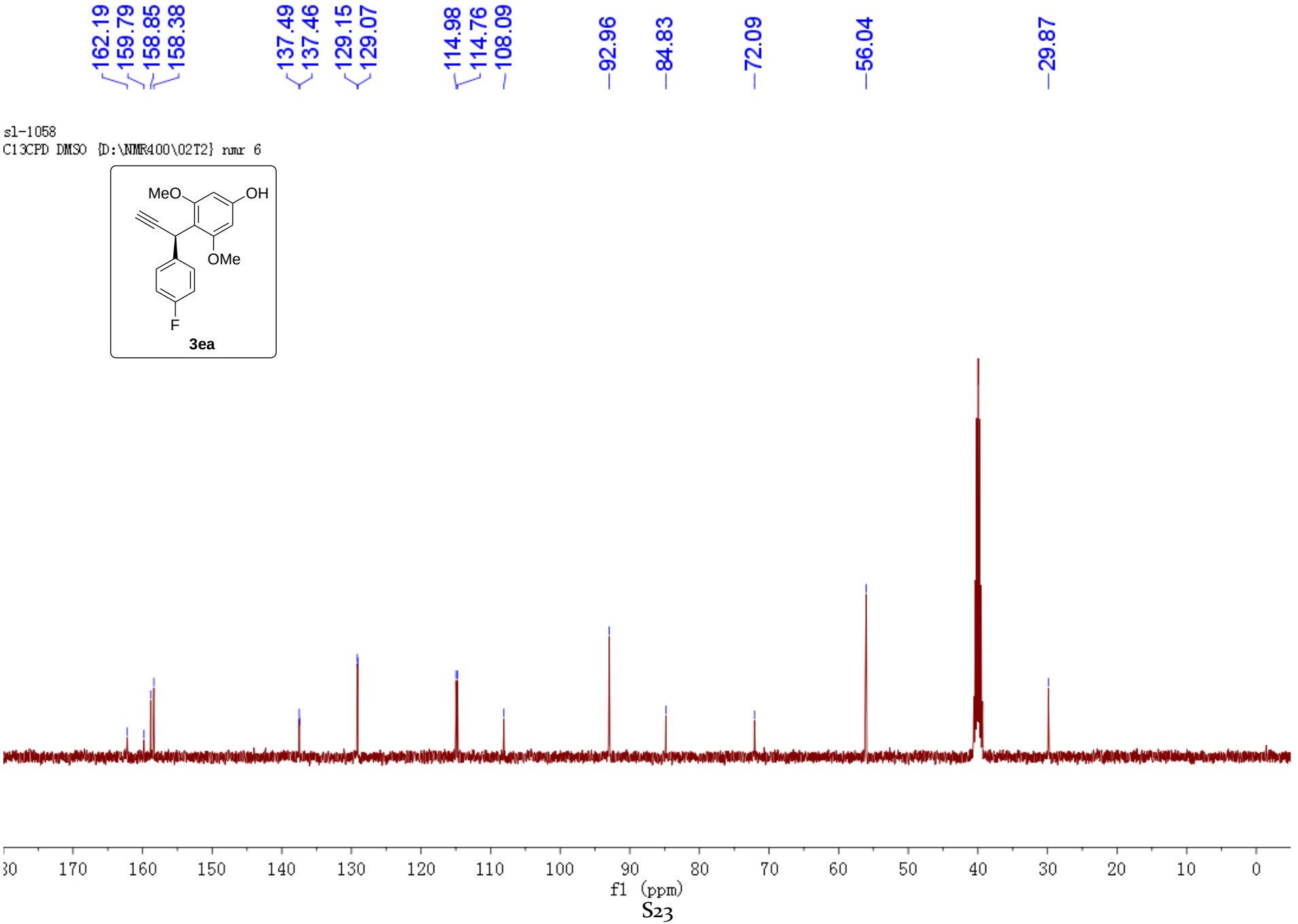
158.52
156.54
139.48
131.57
128.63
127.84
110.10
92.85
84.34
69.44
55.90
30.08

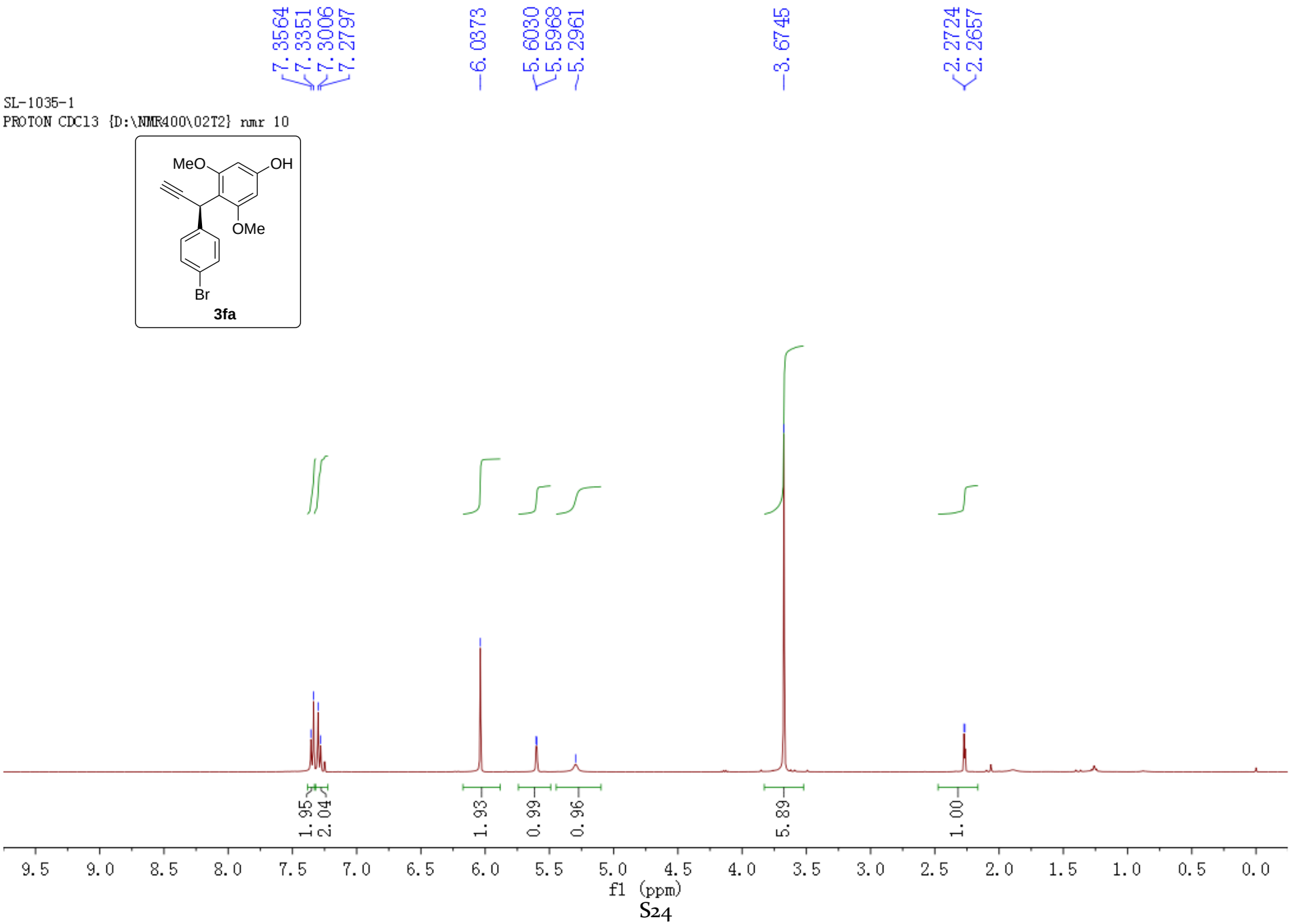


3da



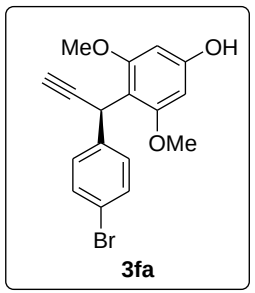

9.5427
7.3436
7.3296
7.3106
7.0889
7.0668
7.0447
6.0861
5.4618
3.6599
3.0226
3.0178
3.0120
sl-1058
PROTON DMSO {D:\NMR400\02T2} rmr 6
MeO
OH
OMe
F
3ea
1.00
2.03
2.02
2.05
1.00
6.07
0.95
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


162.19
159.79
158.85
158.38
137.49
137.46
129.15
129.07
114.98
114.76
108.09
92.96
84.83
72.09
56.04
29.87
sl-1058
C13CPD DMSO {D:\NMR400\02T2} rmr 6
MeO
OH
OMe
F
3ea
f1 (ppm)

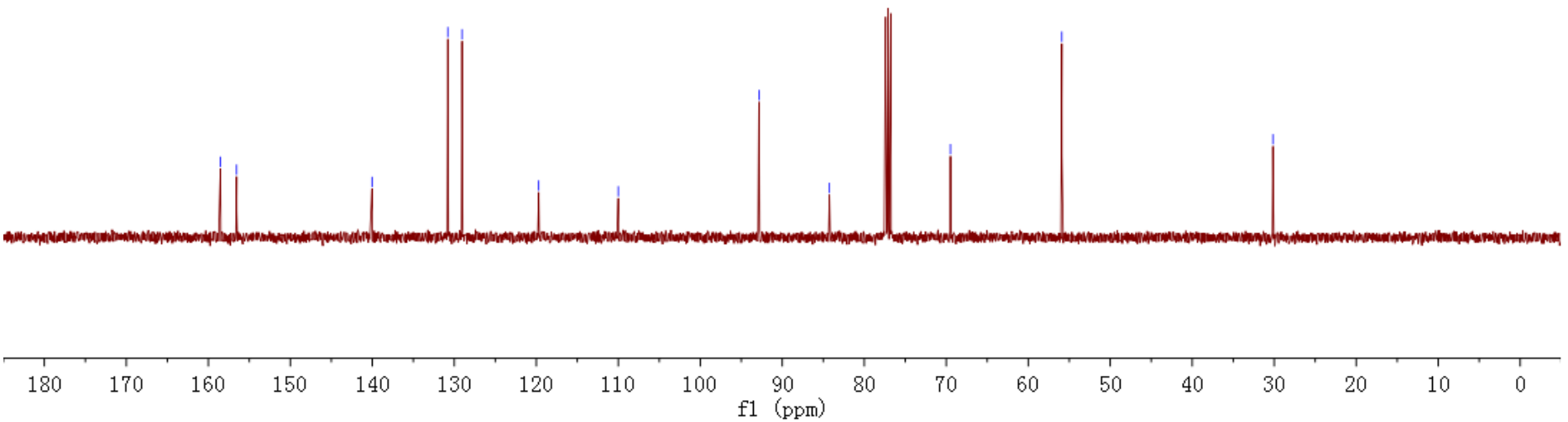

7.3564
7.3351
7.3006
7.2797
6.0373
5.6030
5.5968
5.2961
3.6745
2.2724
2.2657
SL-1035-1
PROTON CDC13 {D:\NMR400\02T2} rumr 10
MeO
OH
OMe
Br
3fa
1.95
2.04
1.93
0.99
0.96
5.89
1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

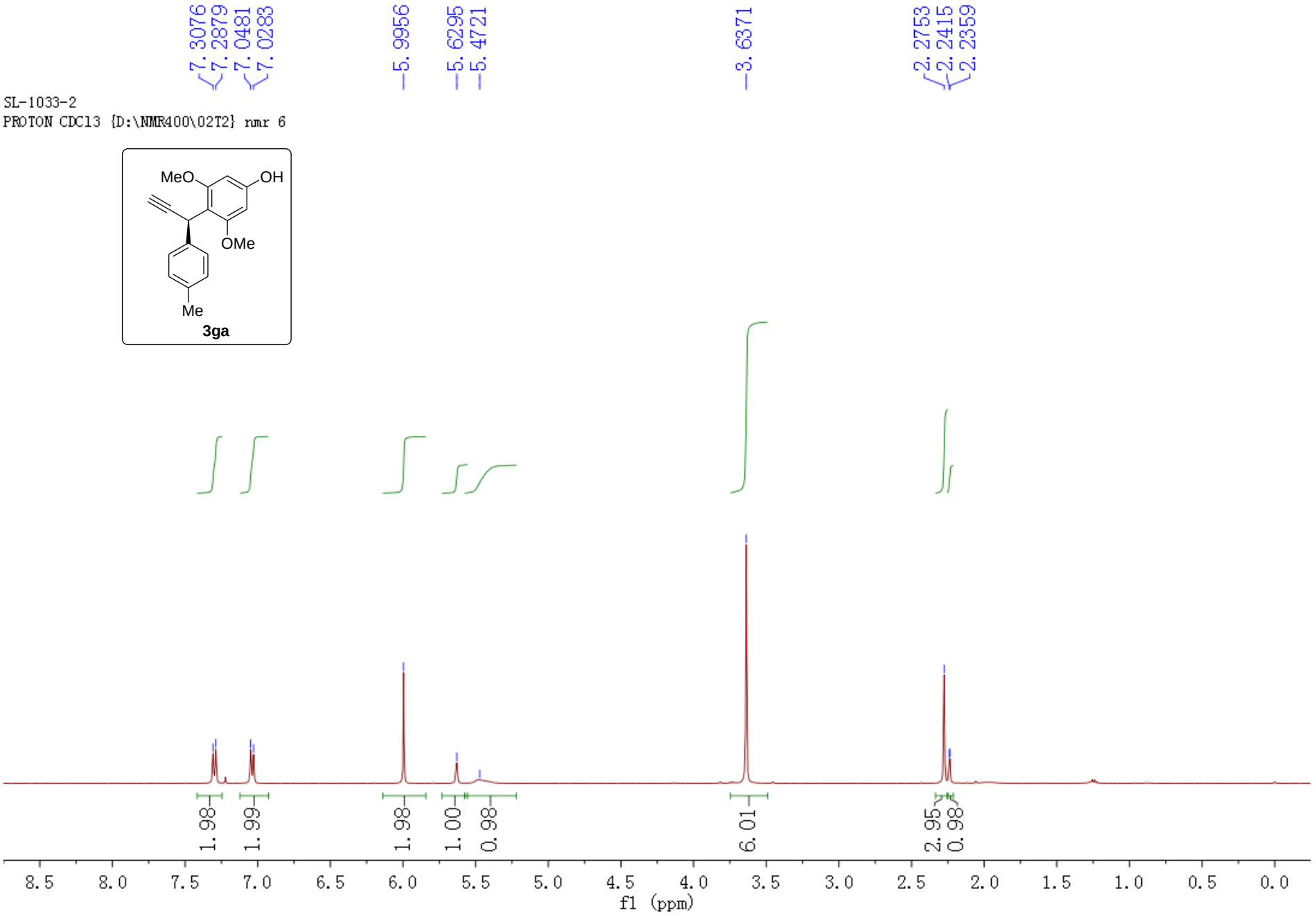
158.52
156.55
140.04
130.79
129.06
119.70
110.01
92.84
84.24
69.49
55.90
30.15

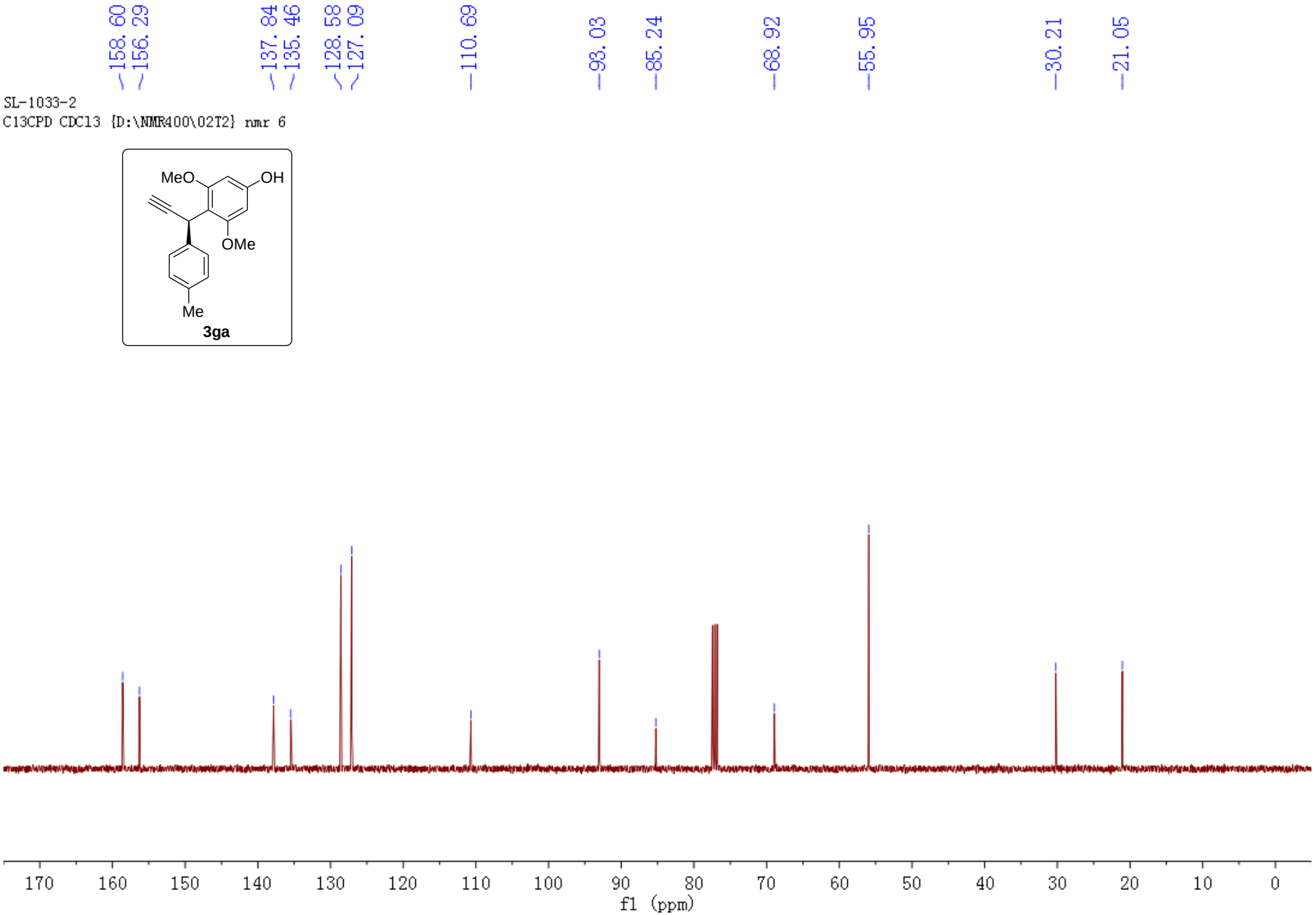
SL-1035-1
C13CPD CDCl3 {D:\NMR400\02T2} nmr 10

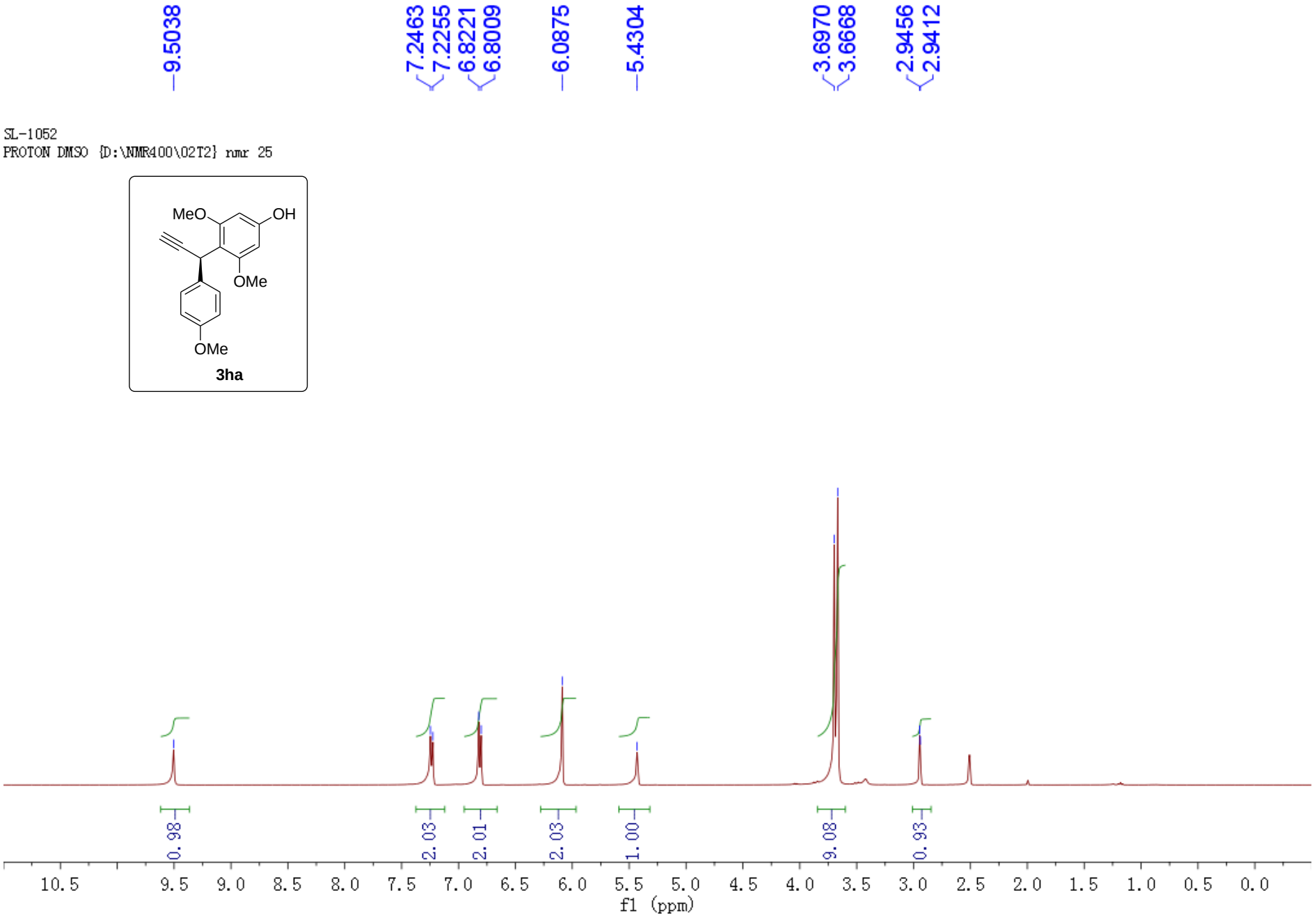


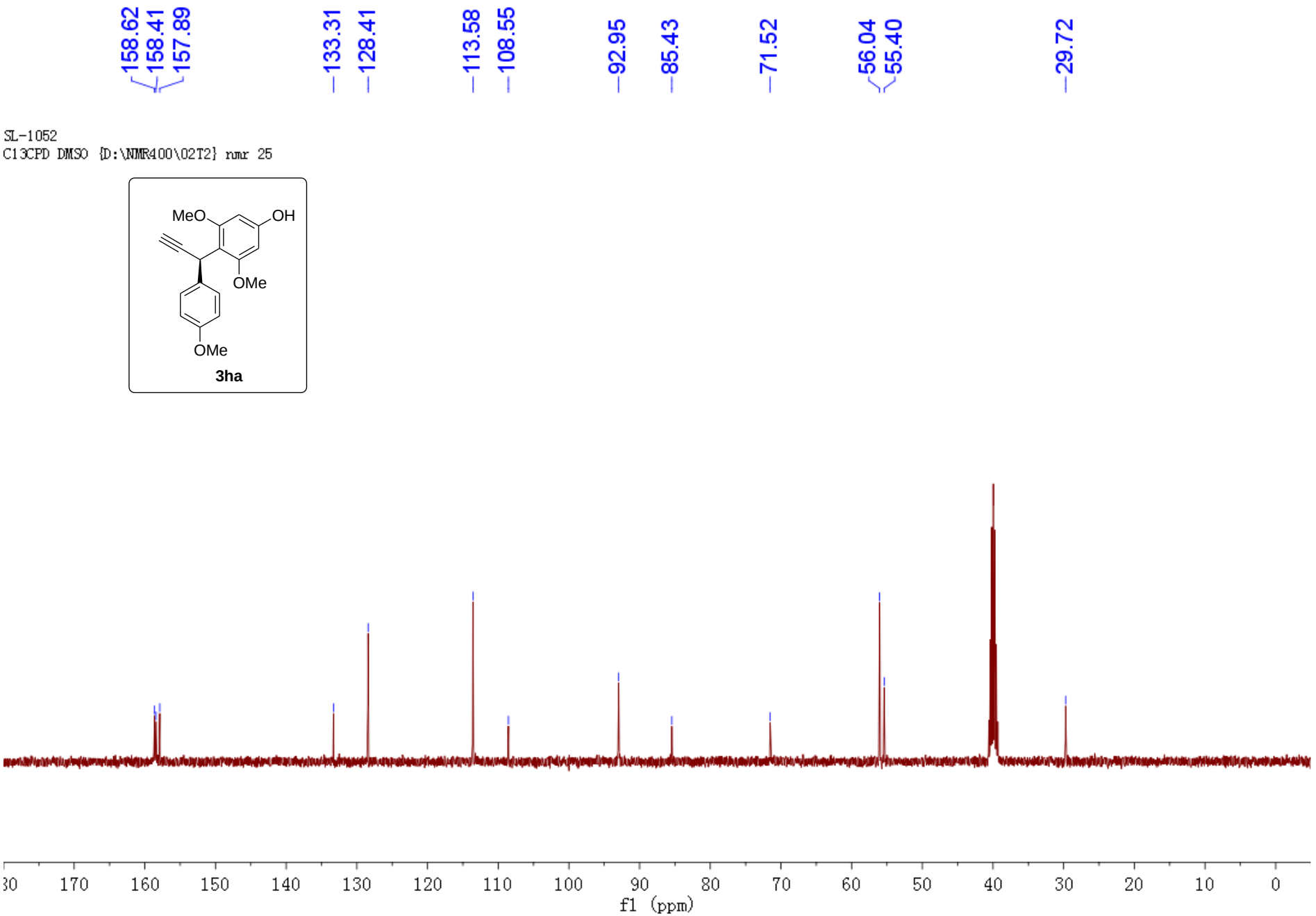
3fa

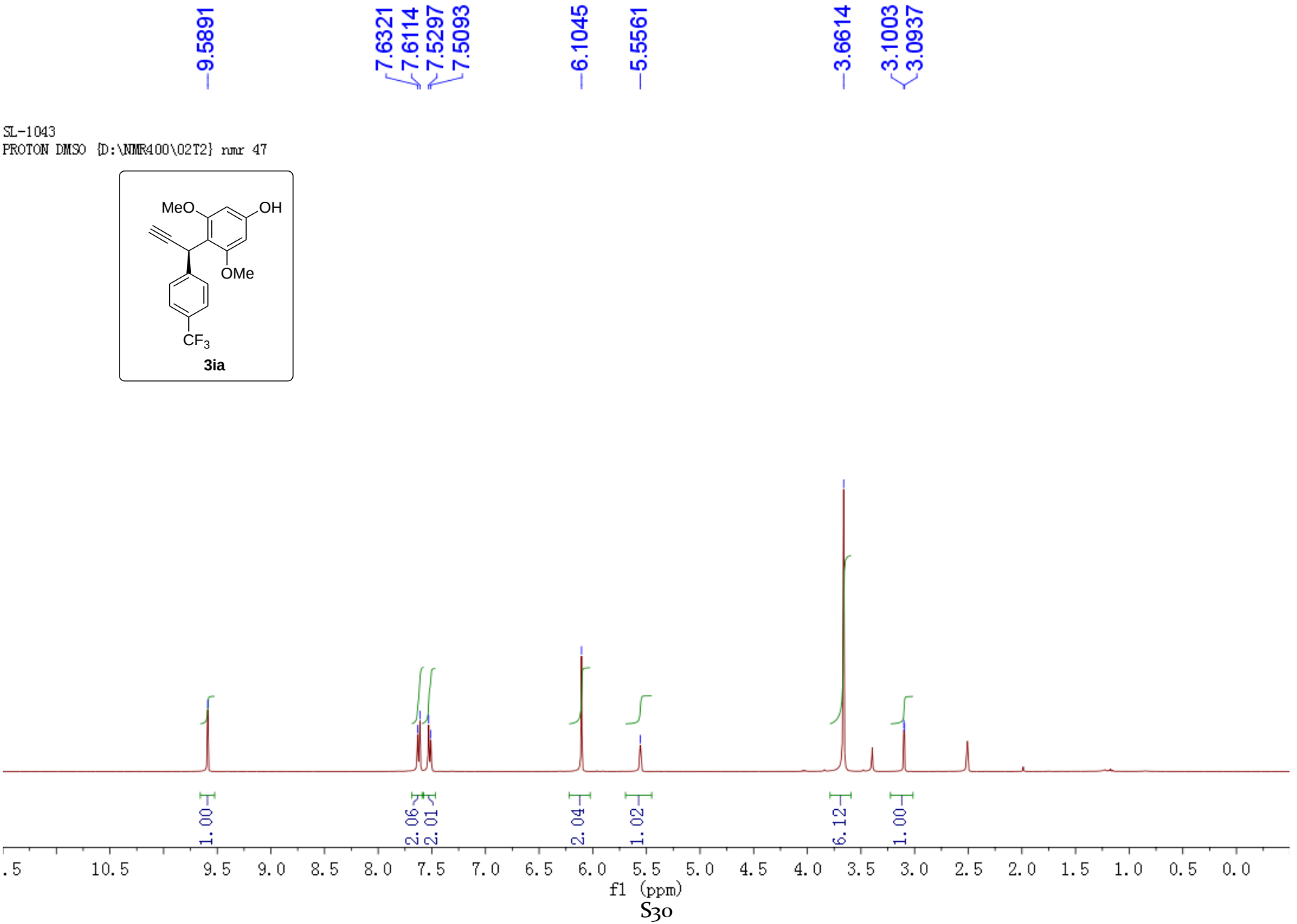


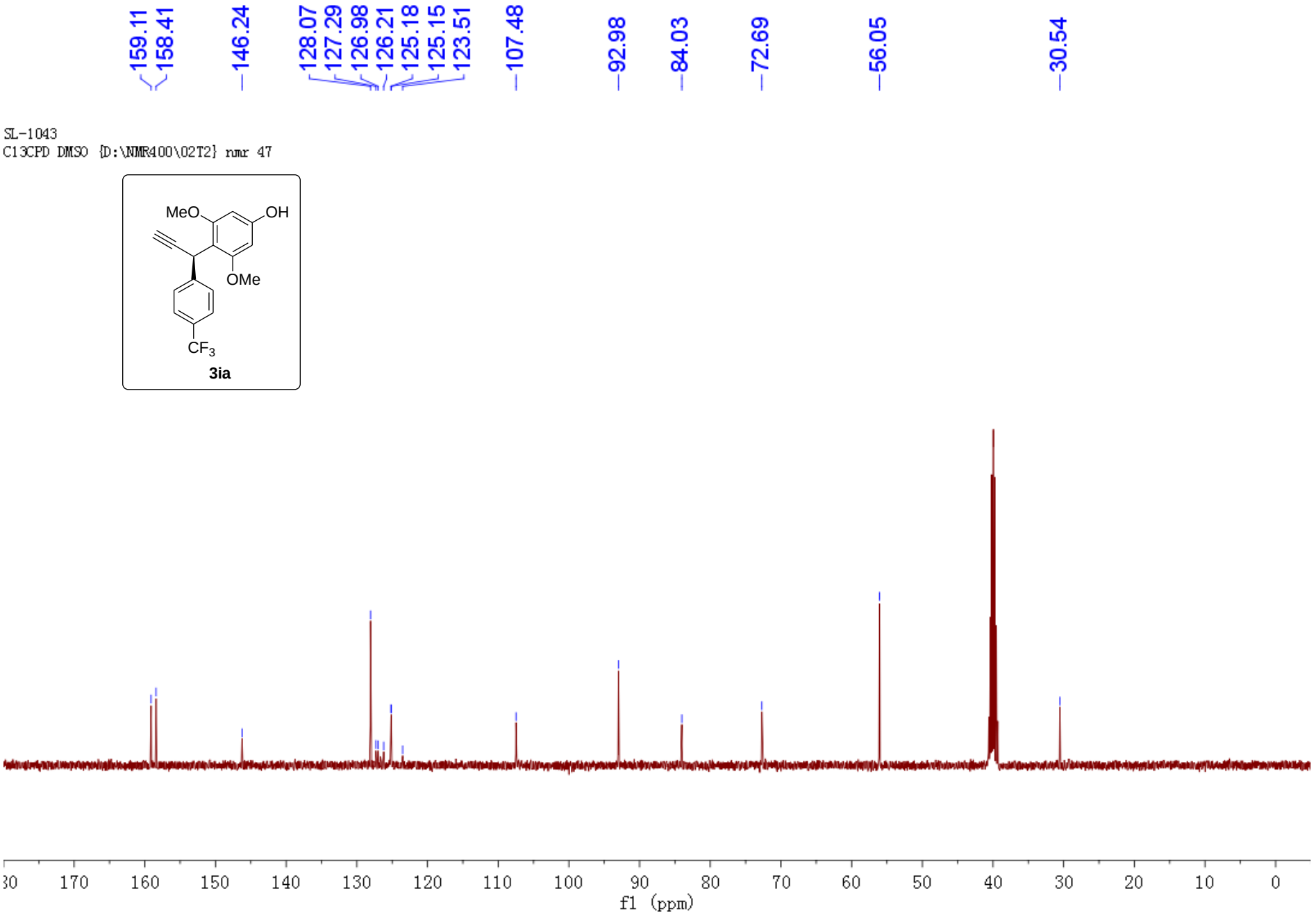

7.3076
7.2879
7.0481
7.0283
5.9956
5.6295
5.4721
3.6371
2.2753
2.2415
2.2359
SL-1033-2
PROTON CDCl3 {D:\NMR400\02T2} nmr 6
MeO
OH
OMe
Me
3ga
1.98
1.99
1.98
1.00
0.98
6.01
2.95
0.98
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

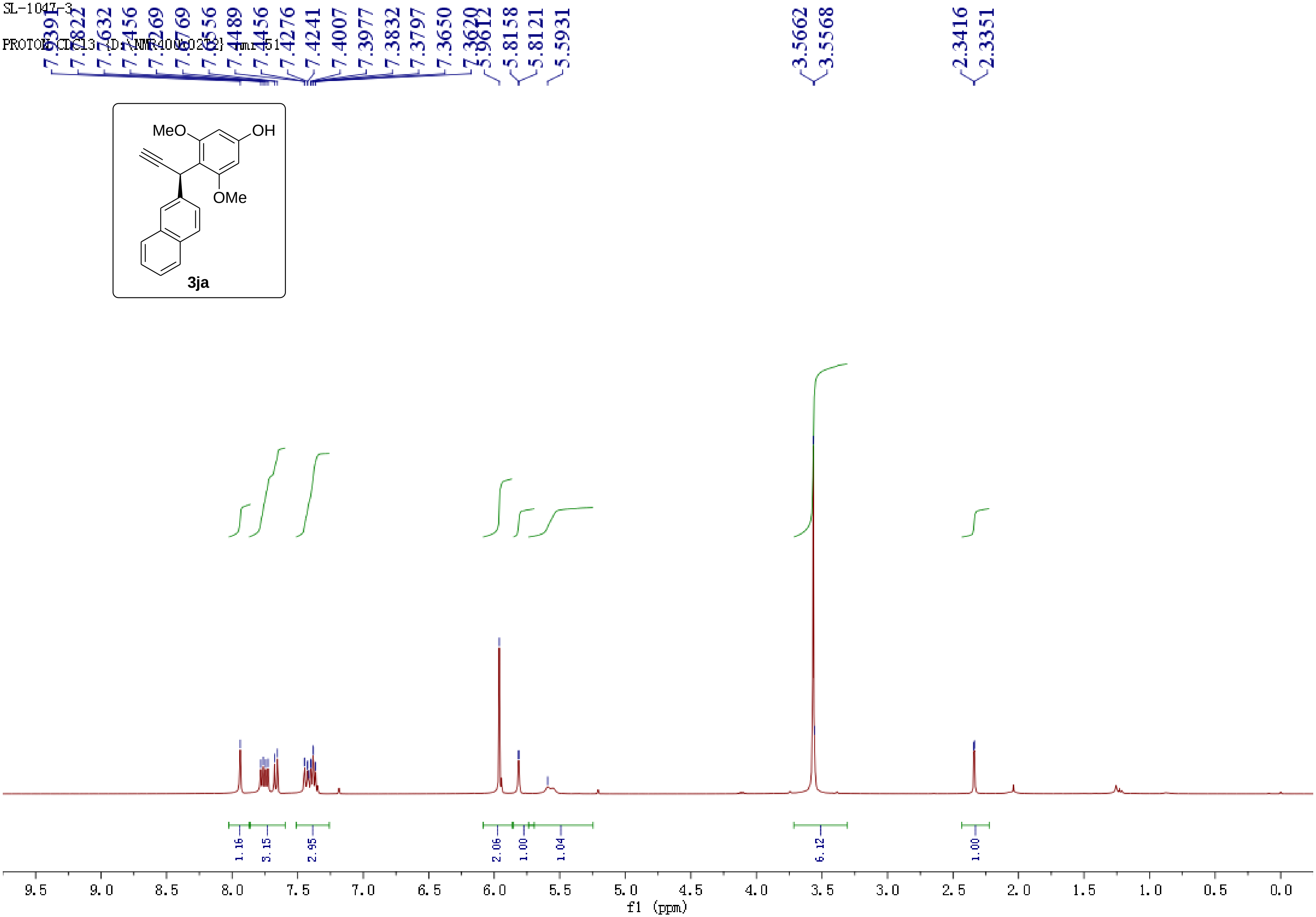

158.60
156.29
137.84
135.46
128.58
127.09
110.69
93.03
85.24
68.92
55.95
30.21
21.05
SL-1033-2
C13CPD CDCl3 {D:\NMR400\02T2} nmr 6
MeO
OH
OMe
Me
3ga
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


9.5038
7.2463
7.2255
6.8221
6.8009
6.0875
5.4304
3.6970
3.6668
2.9456
2.9412
SL-1052
PROTON DMSO {D:\NMR400\02T2} nmr 25
MeO
OH
OMe
OMe
3ha
0.98
2.03
2.01
2.03
1.00
9.08
0.93
10.5
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
f1 (ppm)


158.62
158.41
157.89
133.31
128.41
113.58
108.55
92.95
85.43
71.52
56.04
55.40
29.72
SL-1052
C13CPD DMSO {D:\NMR400\02T2} rmr 25
MeO
OH
OMe
OMe
3ha
f1 (ppm)

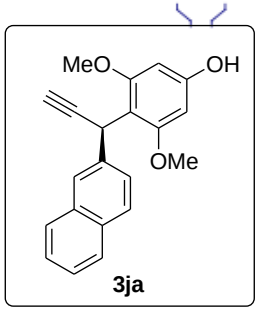

9.5891
7.6321
7.6114
7.5297
7.5093
6.1045
5.5561
3.6614
3.1003
3.0937
SL-1043
PROTON DMSO {D:\NMR400\02T2} rumr 47
MeO
OH
OMe
CF3
3ia
1.00
2.06
2.01
2.04
1.02
6.12
1.00
f1 (ppm)


159.11
158.41
146.24
128.07
127.29
126.98
126.21
125.18
125.15
123.51
107.48
92.98
84.03
72.69
56.05
30.54
SL-1043
C13CPD DMSO {D:\NMR400\02T2} rmr 47
MeO
OH
OMe
CF3
3ia
f1 (ppm)


SL-1047-3
PROTON CDCl3 {D:\NMR400\0272} nmr 51
7.9391
7.7822
7.7632
7.7456
7.7269
7.6769
7.6556
7.4489
7.4456
7.4276
7.4241
7.4007
7.3977
7.3832
7.3797
7.3650
7.3620
5.9612
5.8158
5.8121
5.5931
3.5662
3.5568
2.3416
2.3351
MeO
OH
OMe
3ja
1.16
3.15
2.95
2.06
1.00
1.04
6.12
1.00
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
f1 (ppm)

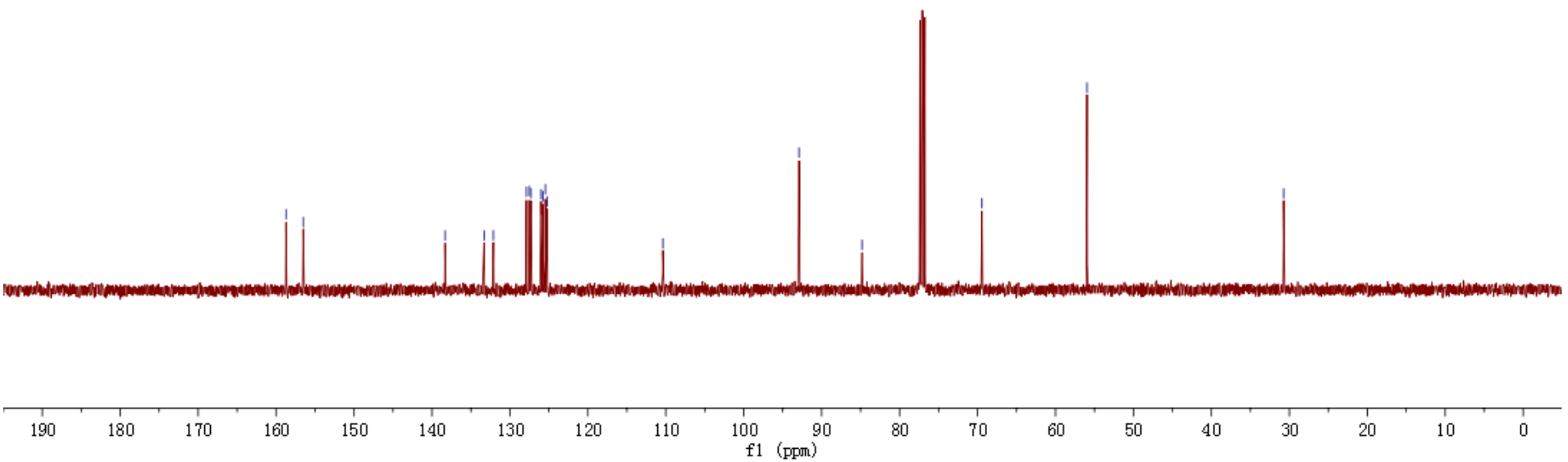
SL-1047-1

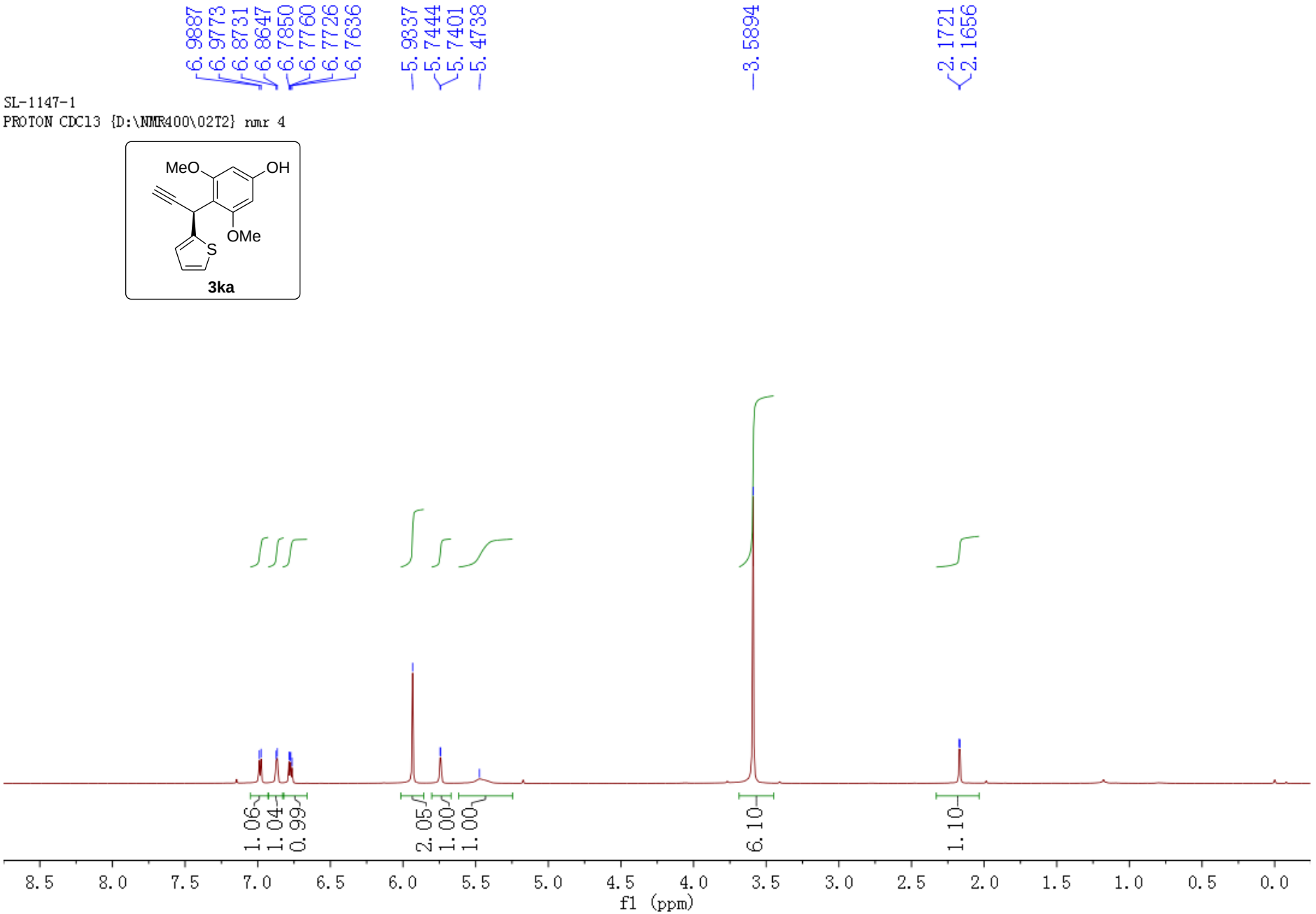
C13CPD CDCl3 {D:\NMR400\02T2} jmx 6

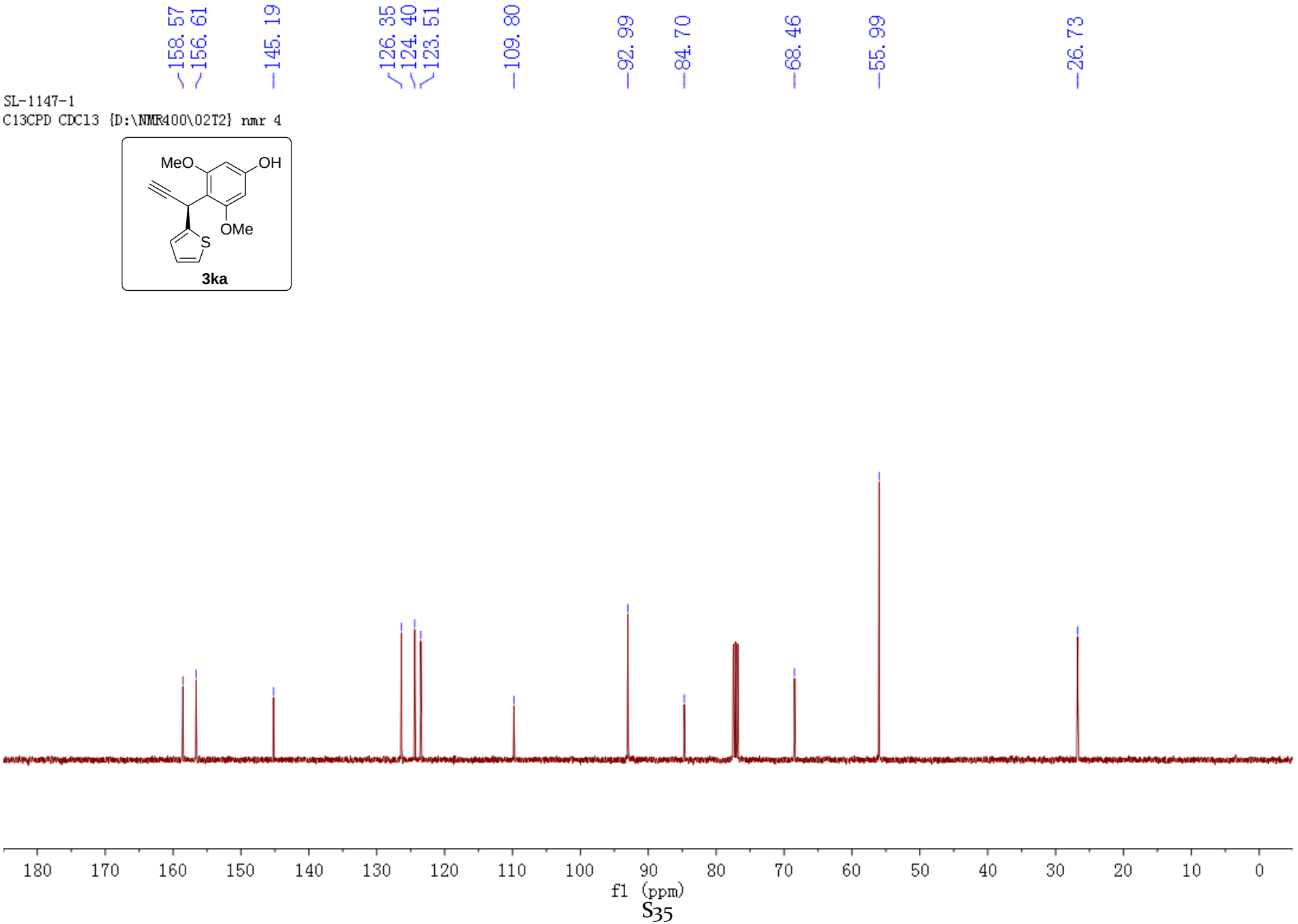


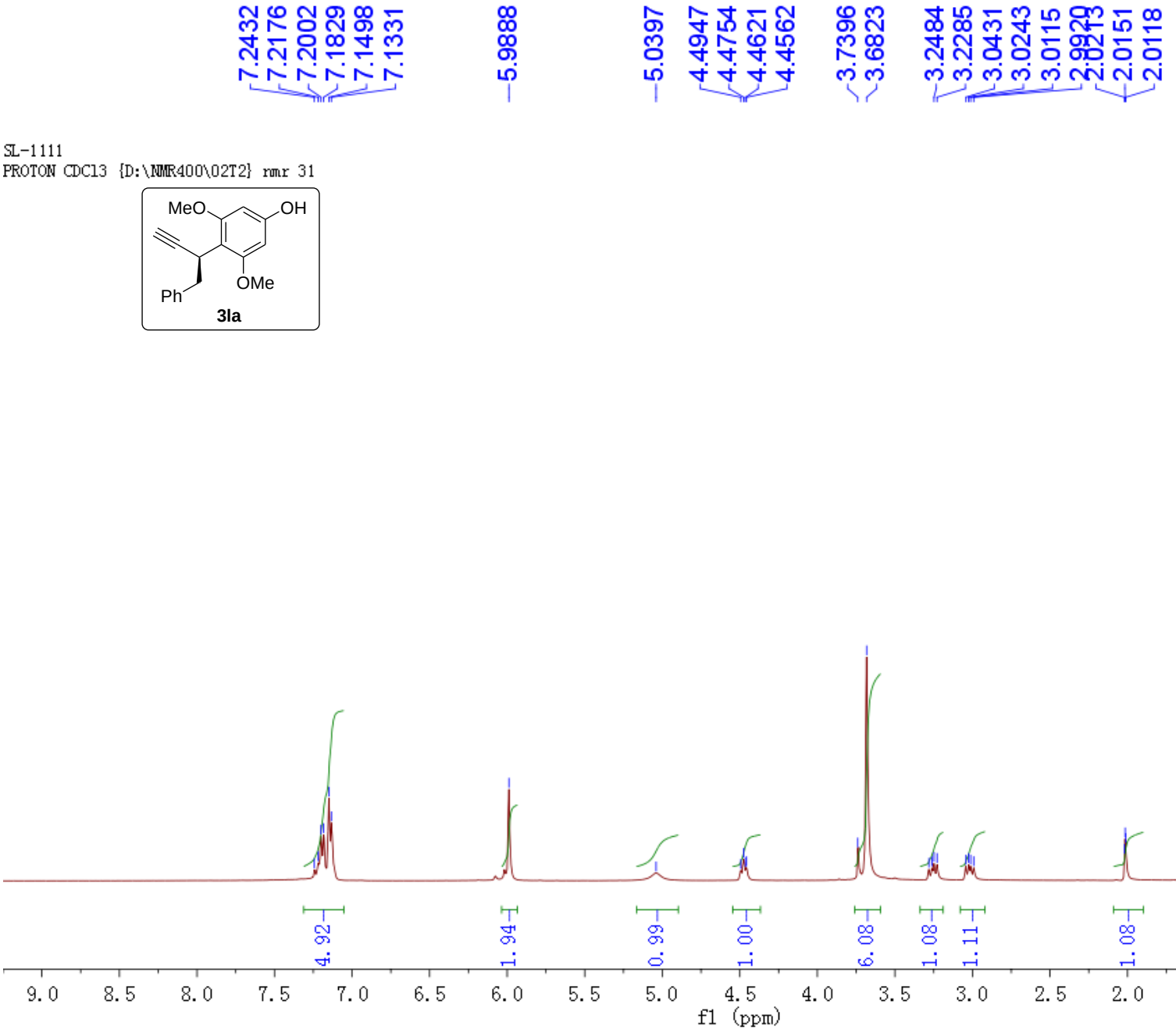
3ja

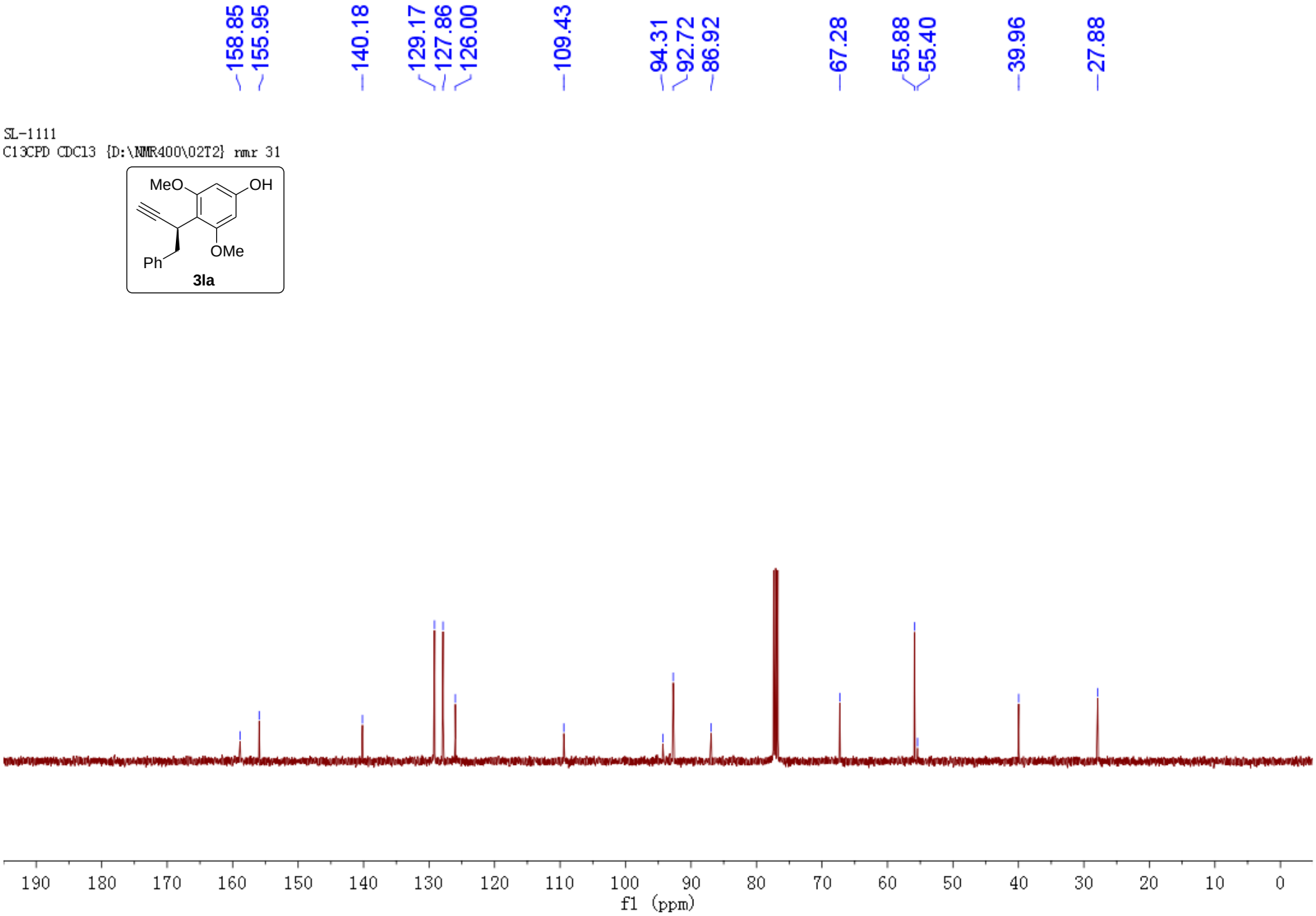
158.72
156.52
127.88
127.51
127.30
126.01
125.72
125.45
125.23
110.37
92.90
84.82
69.43
55.95
30.73

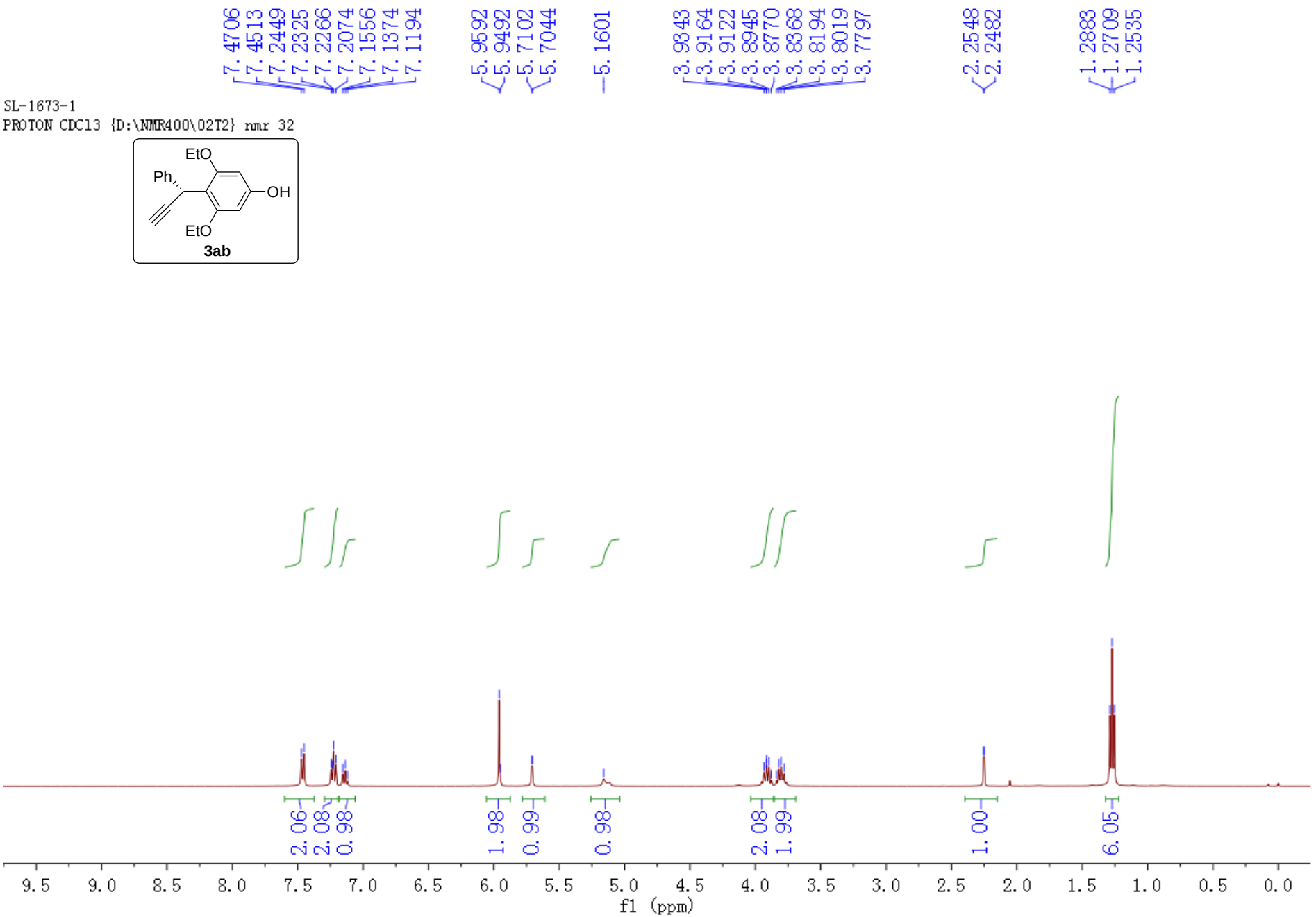


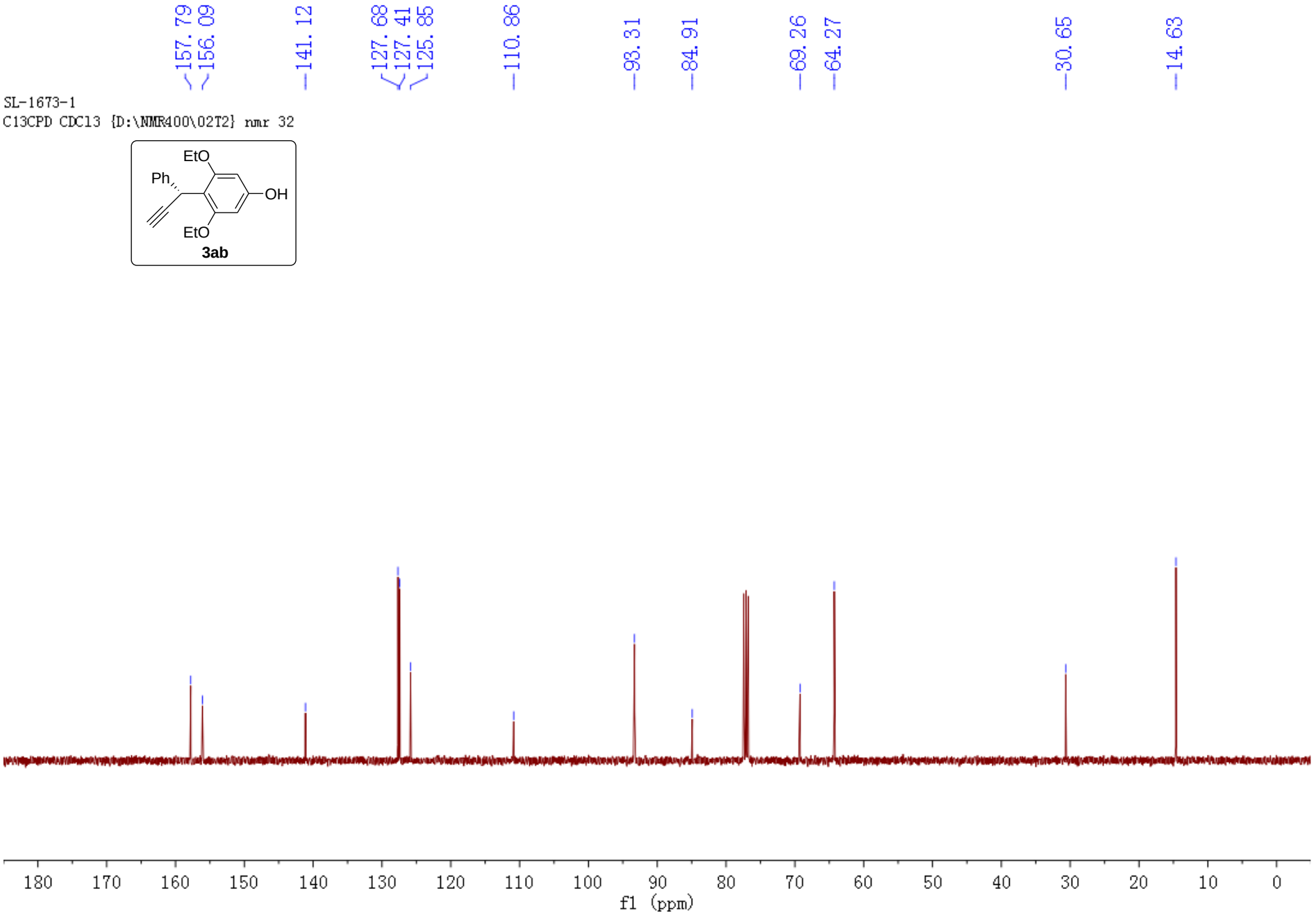

6.9887
6.9773
6.8731
6.8647
6.7850
6.7760
6.7726
6.7636
5.9337
5.7444
5.7401
5.4738
3.5894
2.1721
2.1656
SL-1147-1
PROTON CDCl3 {D:\NMR400\02T2} nmr 4
MeO
OH
OMe
S
3ka
1.06
1.04
0.99
2.05
1.00
1.00
6.10
1.10
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

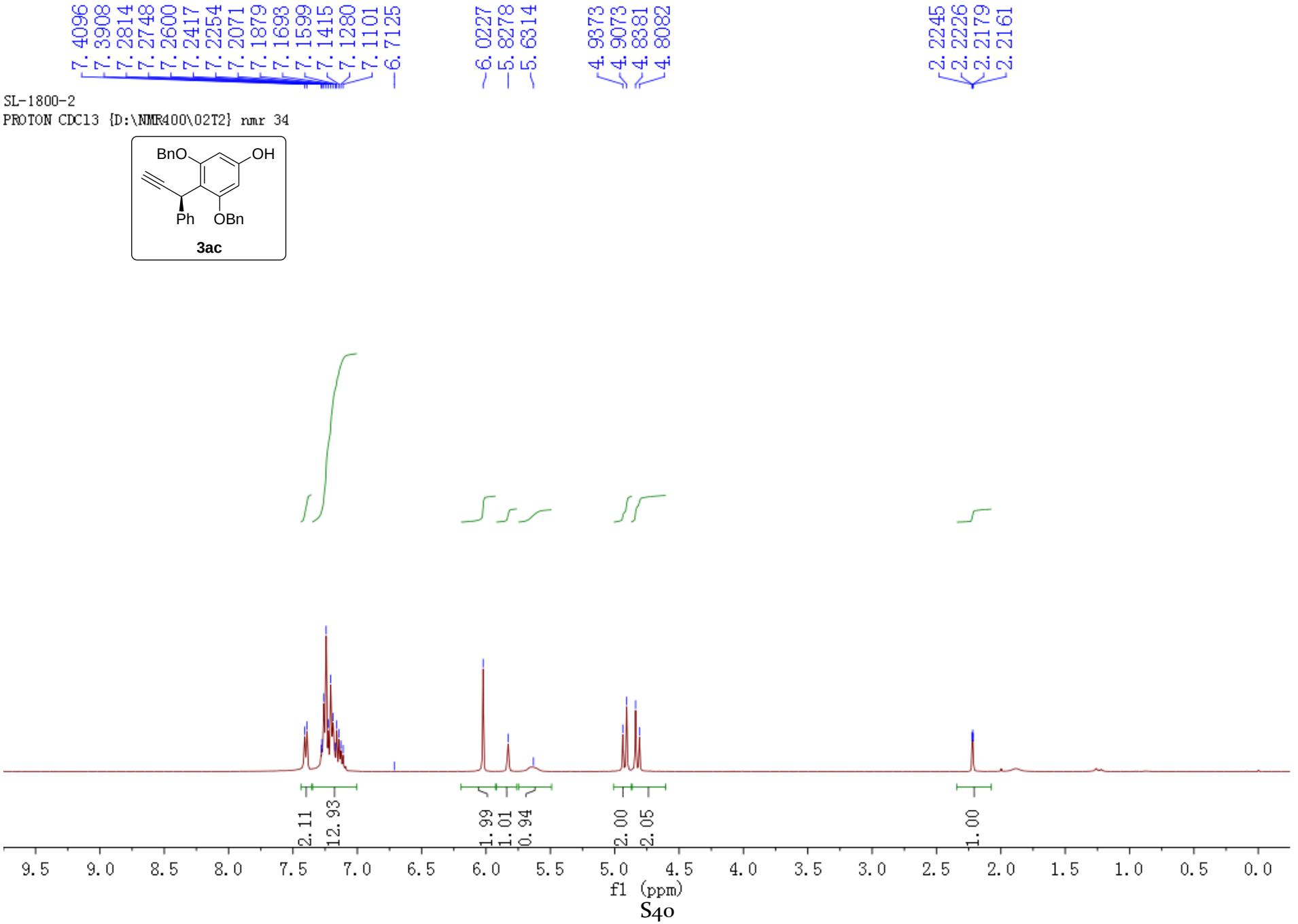

158.57
156.61
145.19
126.35
124.40
123.51
109.80
92.99
84.70
68.46
55.99
26.73
SL-1147-1
C13CPD CDCl3 {D:\NMR400\02T2} rmr 4
MeO
OH
OMe
S
3ka
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

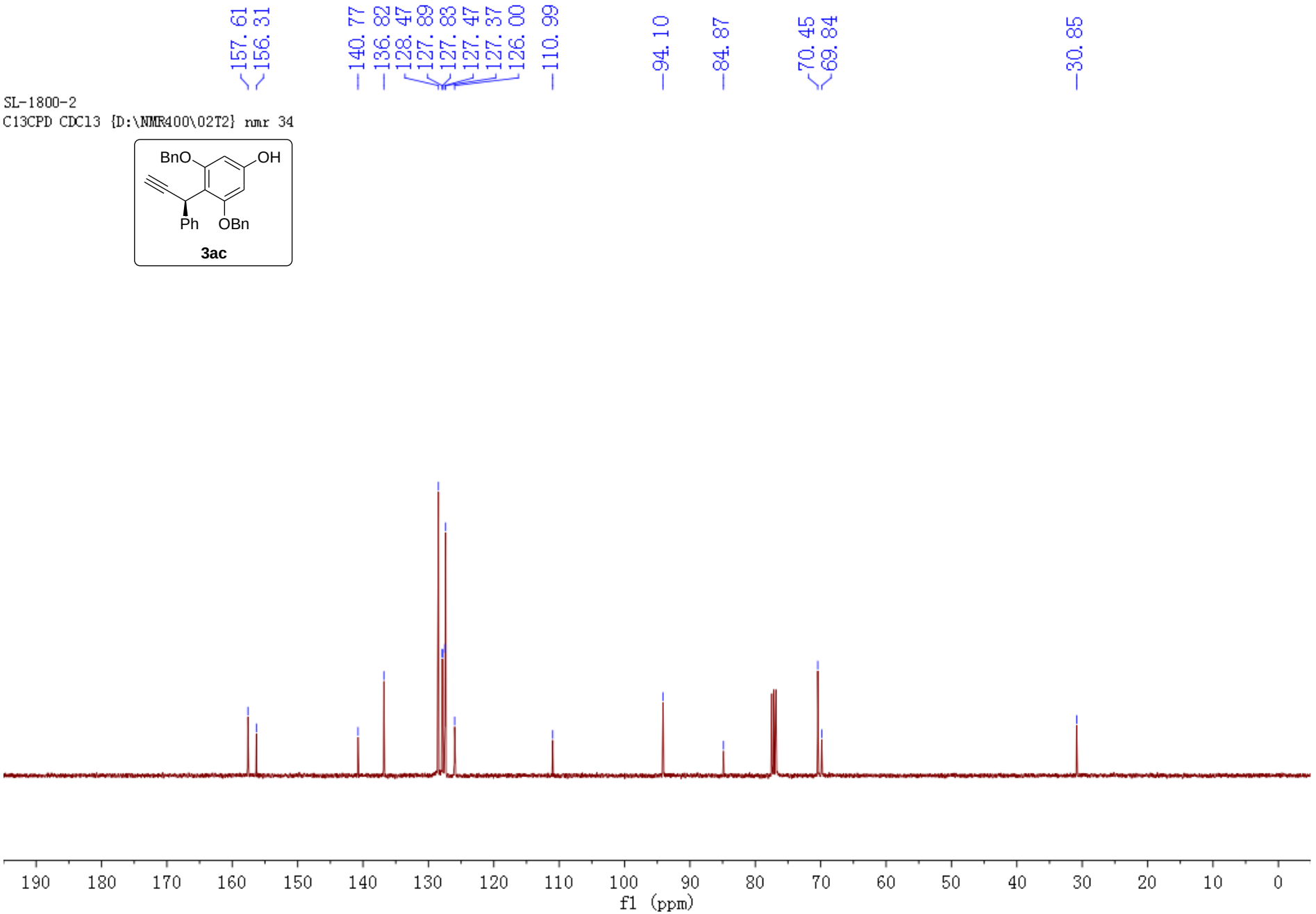

7.2432
7.2176
7.2002
7.1829
7.1498
7.1331
5.9888
5.0397
4.4947
4.4754
4.4621
4.4562
3.7396
3.6823
3.2484
3.2285
3.0431
3.0243
3.0115
2.0151
2.0118
SL-1111
PROTON CDCl3 {D:\NMR400\02T2} rmr 31
MeO
OH
OMe
Ph
3la
4.92
1.94
0.99
1.00
6.08
1.08
1.11
1.08
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

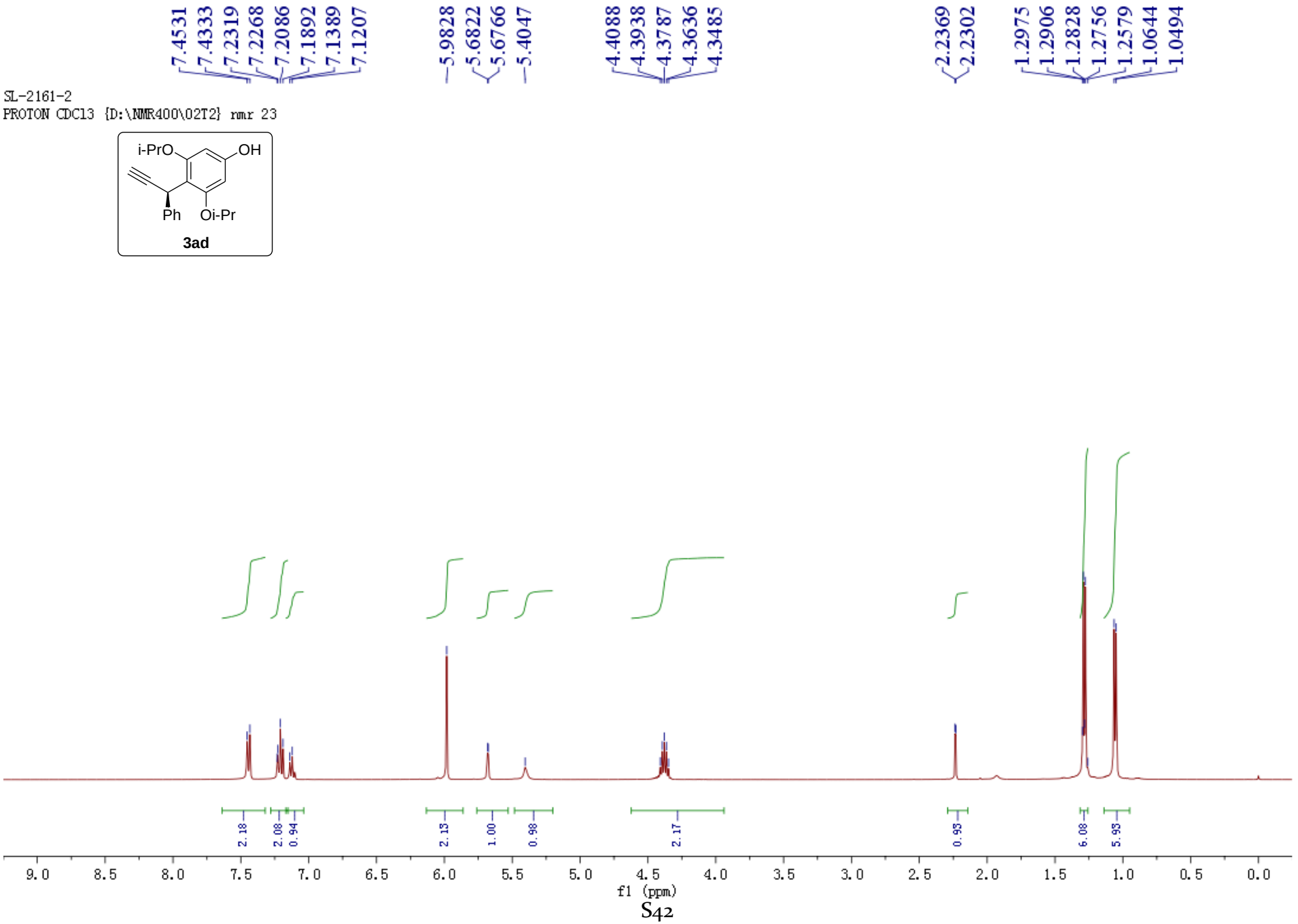

158.85
155.95
140.18
129.17
127.86
126.00
109.43
94.31
92.72
86.92
67.28
55.88
55.40
39.96
27.88
SL-1111
C13CPD CDCl3 {D:\NMR400\02T2} rmr 31
MeO
OH
OMe
Ph
3la
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

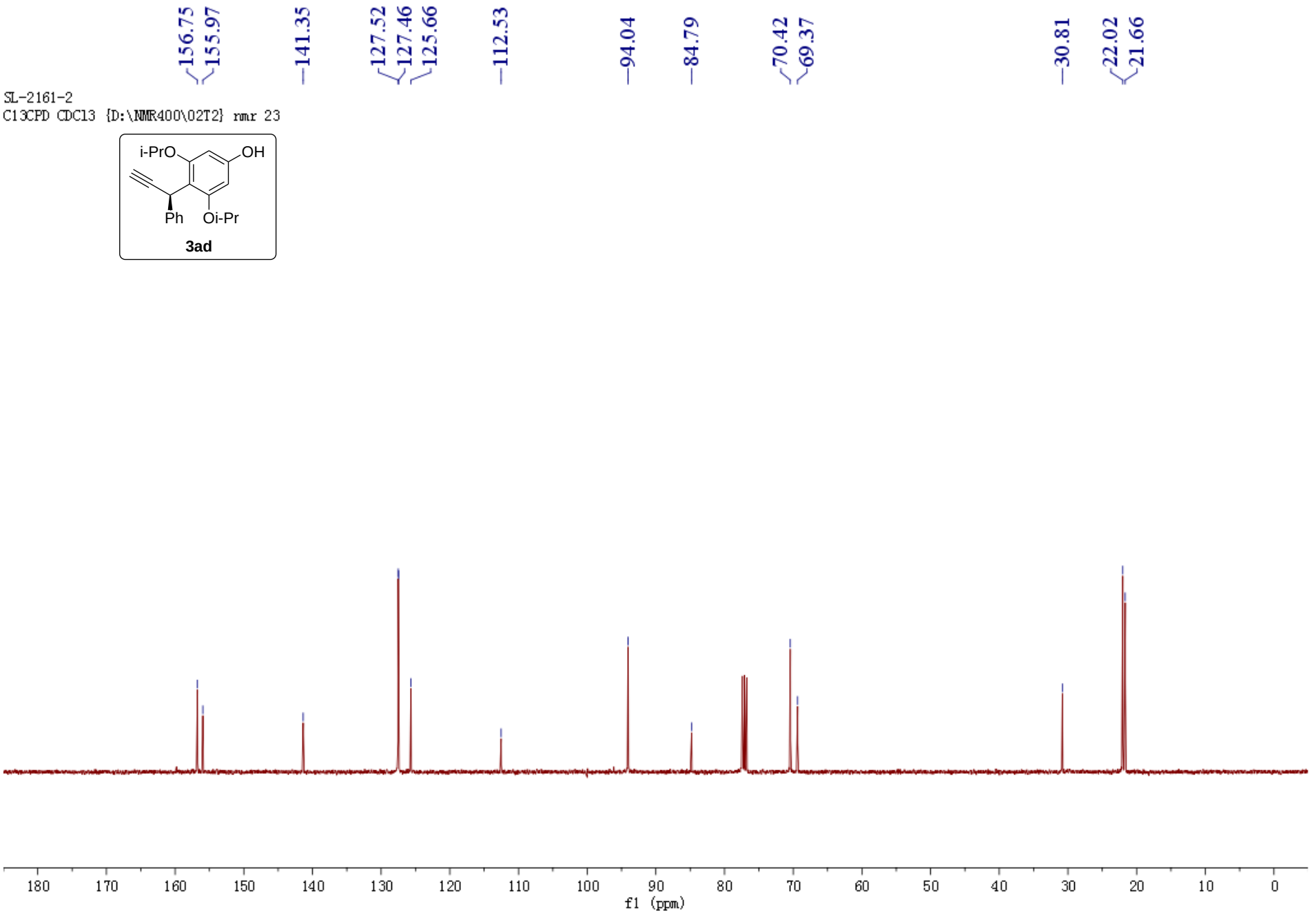

7.4706
7.4513
7.2449
7.2325
7.2266
7.2074
7.1556
7.1374
7.1194
5.9592
5.9492
5.7102
5.7044
5.1601
3.9343
3.9164
3.9122
3.8945
3.8770
3.8368
3.8194
3.8019
3.7797
2.2548
2.2482
1.2883
1.2709
1.2535
SL-1673-1
PROTON CDCl3 {D:\NMR400\02T2} nmr 32
EtO
Ph
OH
EtO
3ab
2.06
2.08
0.98
1.98
0.99
0.98
2.08
1.99
1.00
6.05
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
f1 (ppm)

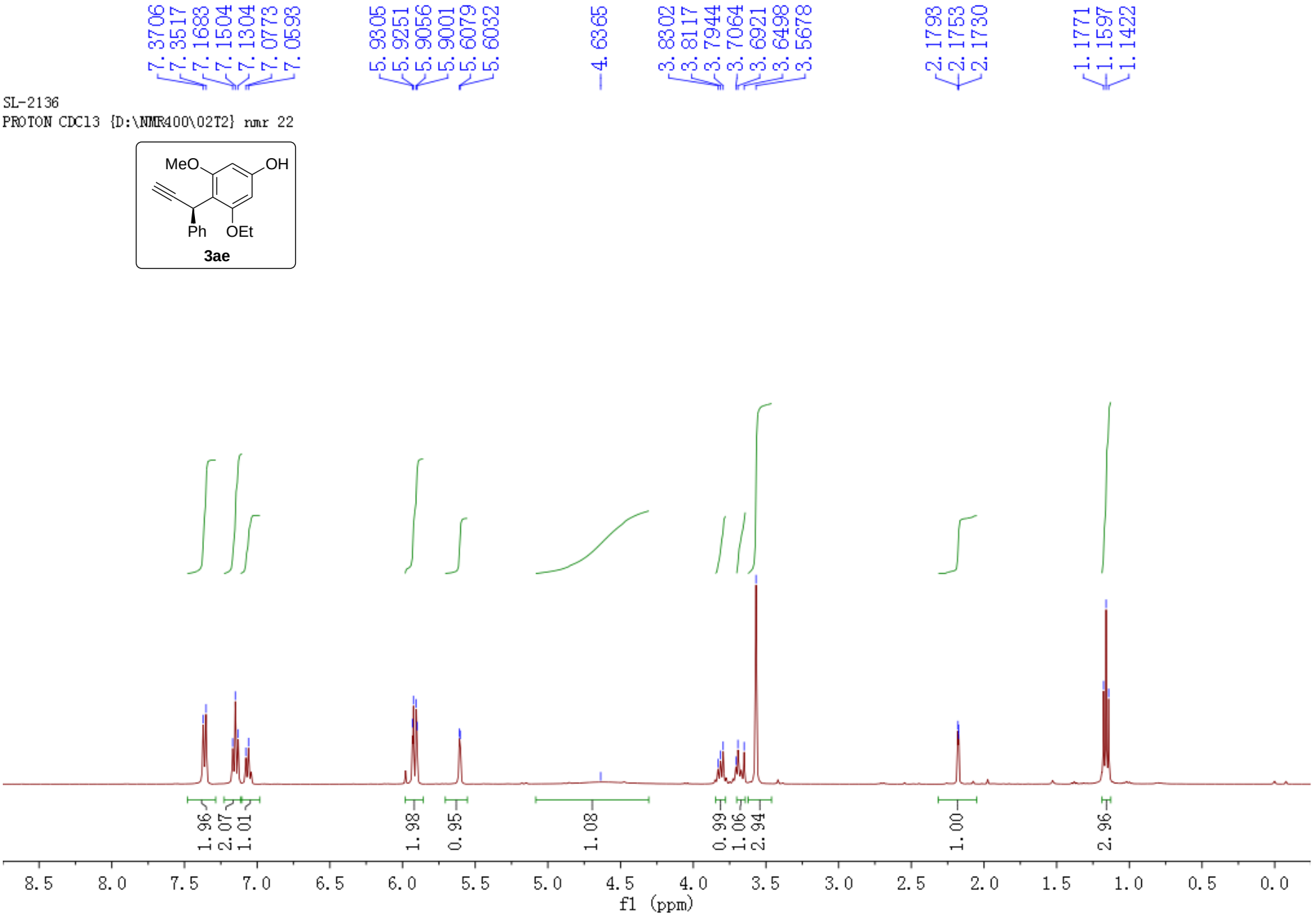

157.79
156.09
141.12
127.68
127.41
125.85
110.86
93.31
84.91
69.26
64.27
30.65
14.63
SL-1673-1
C13CPD CDCl3 {D:\NMR400\02T2} nmr 32
EtO
Ph
OH
EtO
3ab
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.4096
7.3908
7.2814
7.2748
7.2600
7.2417
7.2254
7.2071
7.1879
7.1693
7.1599
7.1415
7.1280
7.1101
6.7125
6.0227
5.8278
5.6314
4.9373
4.9073
4.8381
4.8082
2.2245
2.2226
2.2179
2.2161
SL-1800-2
PROTON CDC13 {D:\NMR400\02T2} rmr 34
BnO
OH
Ph
OBn
3ac
2.11
12.93
1.99
1.01
0.94
2.00
2.05
1.00
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
f1 (ppm)


157.61
156.31
140.77
136.82
128.47
127.89
127.83
127.47
127.37
126.00
110.99
94.10
84.87
70.45
69.84
30.85
SL-1800-2
C13CPD CDCl3 {D:\NMR400\02T2} nmr 34
BnO
OH
Ph
OBn
3ac
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

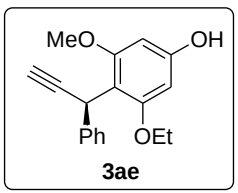

7.4531
7.4333
7.2319
7.2268
7.2086
7.1892
7.1389
7.1207
5.9828
5.6822
5.6766
5.4047
4.4088
4.3938
4.3787
4.3636
4.3485
2.2369
2.2302
1.2975
1.2906
1.2828
1.2756
1.2579
1.0644
1.0494
SL-2161-2
PROTON CDCl3 {D:\NMR400\02T2} rmr 23
i-PrO
OH
Ph
Oi-Pr
3ad
2.18
2.08
0.94
2.13
1.00
0.98
2.17
0.93
6.08
5.93
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
f1 (ppm)


156.75
155.97
141.35
127.52
127.46
125.66
112.53
94.04
84.79
70.42
69.37
30.81
22.02
21.66
SL-2161-2
C13CPD CDCl3 {D:\NMR400\02T2} rmr 23
i-PrO
OH
Ph
Oi-Pr
3ad
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

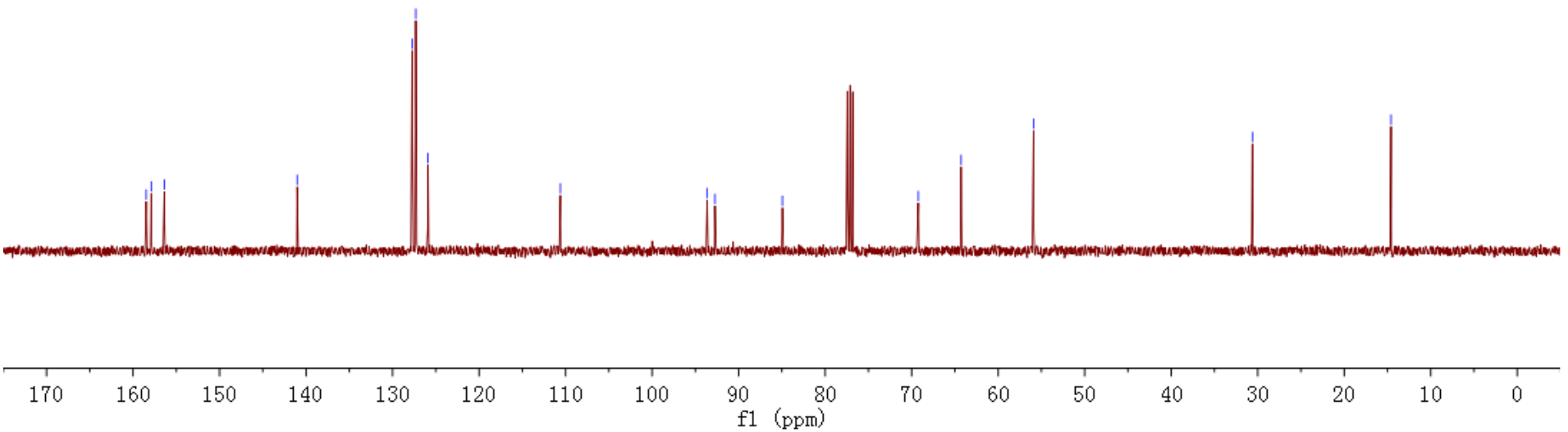

7.3706
7.3517
7.1683
7.1504
7.1304
7.0773
7.0593
5.9305
5.9251
5.9056
5.9001
5.6079
5.6032
4.6365
3.8302
3.8117
3.7944
3.7064
3.6921
3.6498
3.5678
2.1793
2.1753
2.1730
1.1771
1.1597
1.1422
SL-2136
PROTON CDCl3 {D:\NMR400\02T2} nmr 22
MeO
OH
Ph
OEt
3ae
1.96
2.07
1.01
1.98
0.95
1.08
0.99
1.06
2.94
1.00
2.96
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
f1 (ppm)

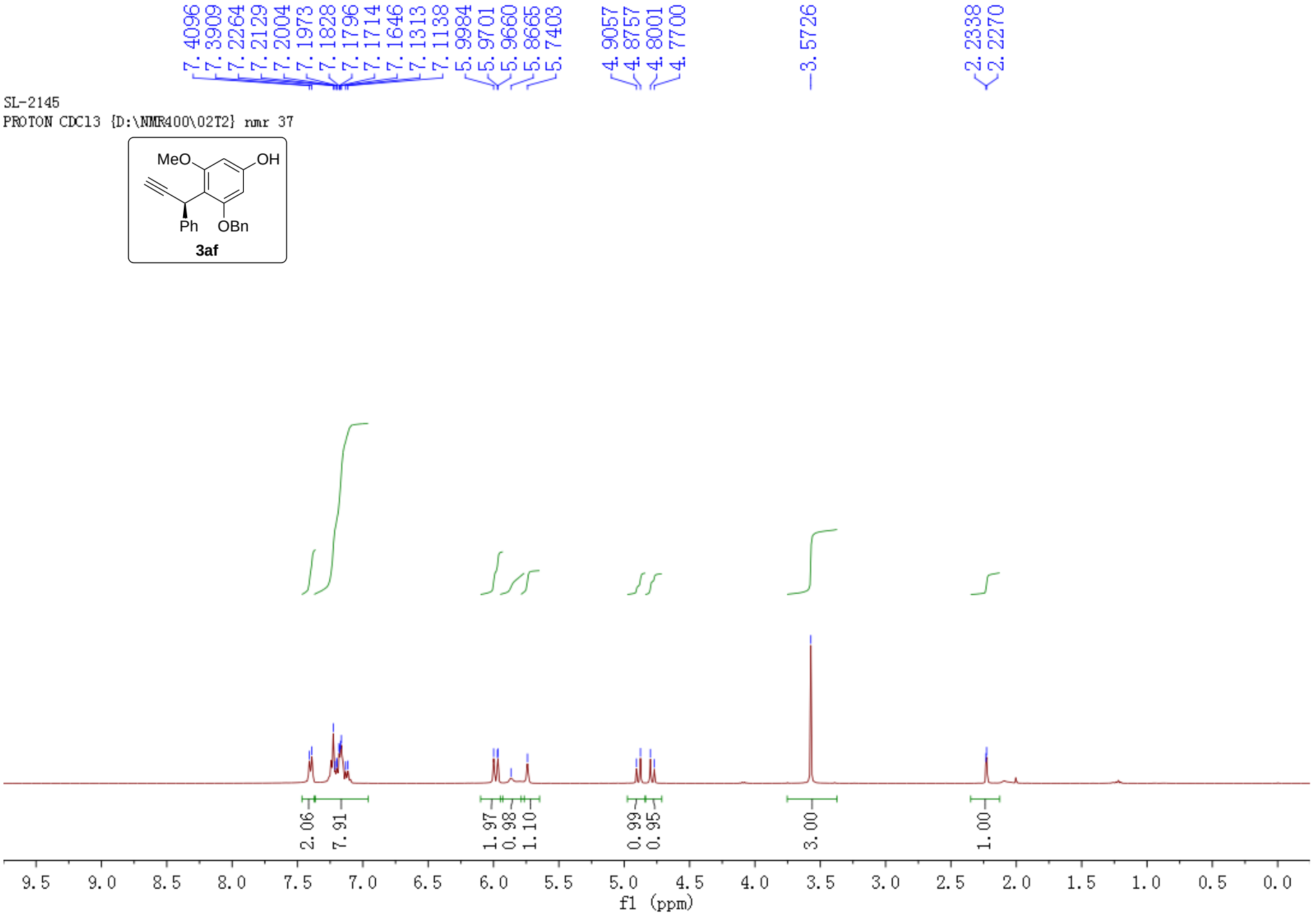
158.47
157.89
156.39
141.03
127.75
127.32
125.90
110.60
93.63
92.73
84.94
69.23
64.28
55.91
30.60
14.59

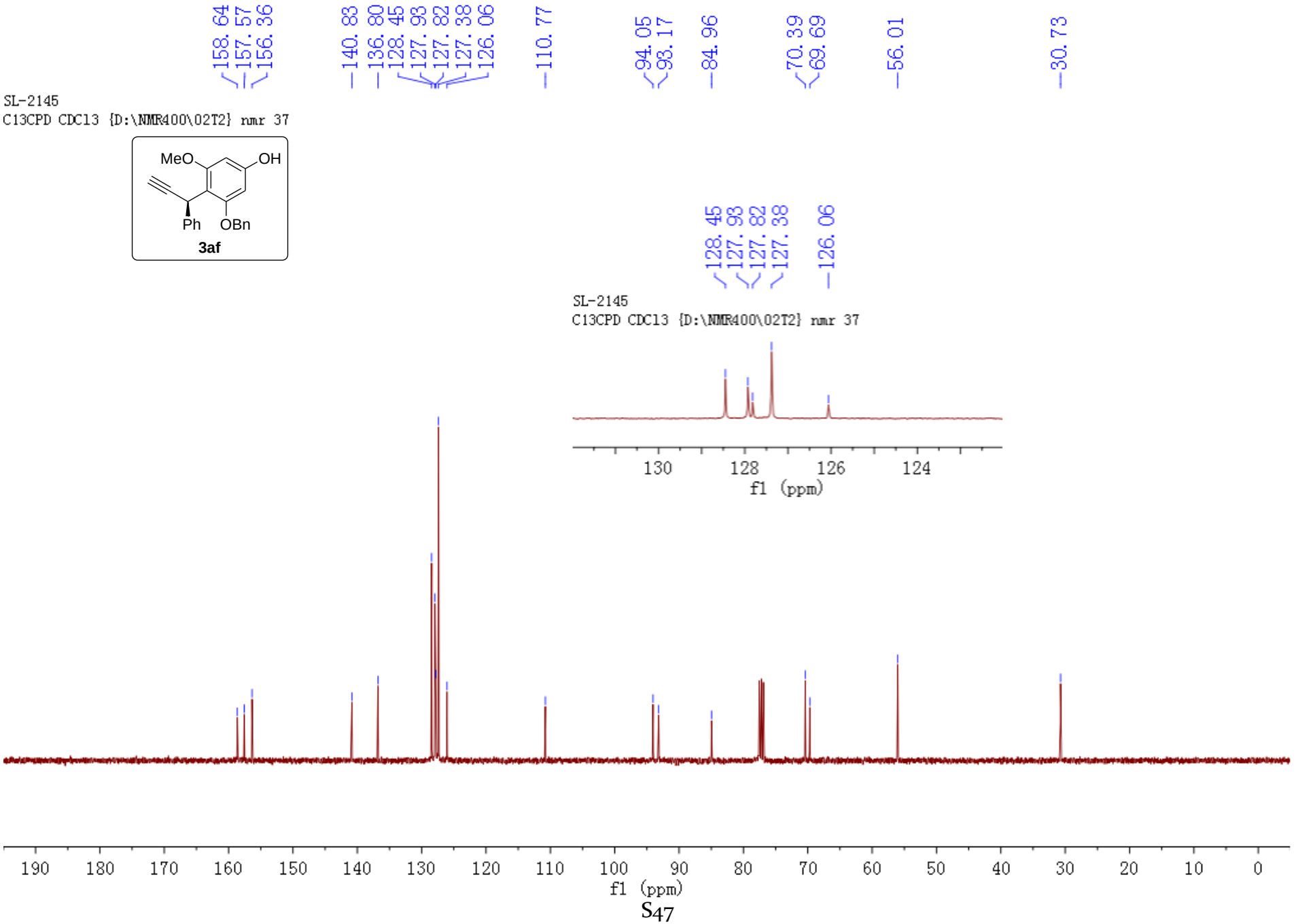
SL-2136
C13CPD CDCl3 {D:\NMR400\02T2} rmr 22

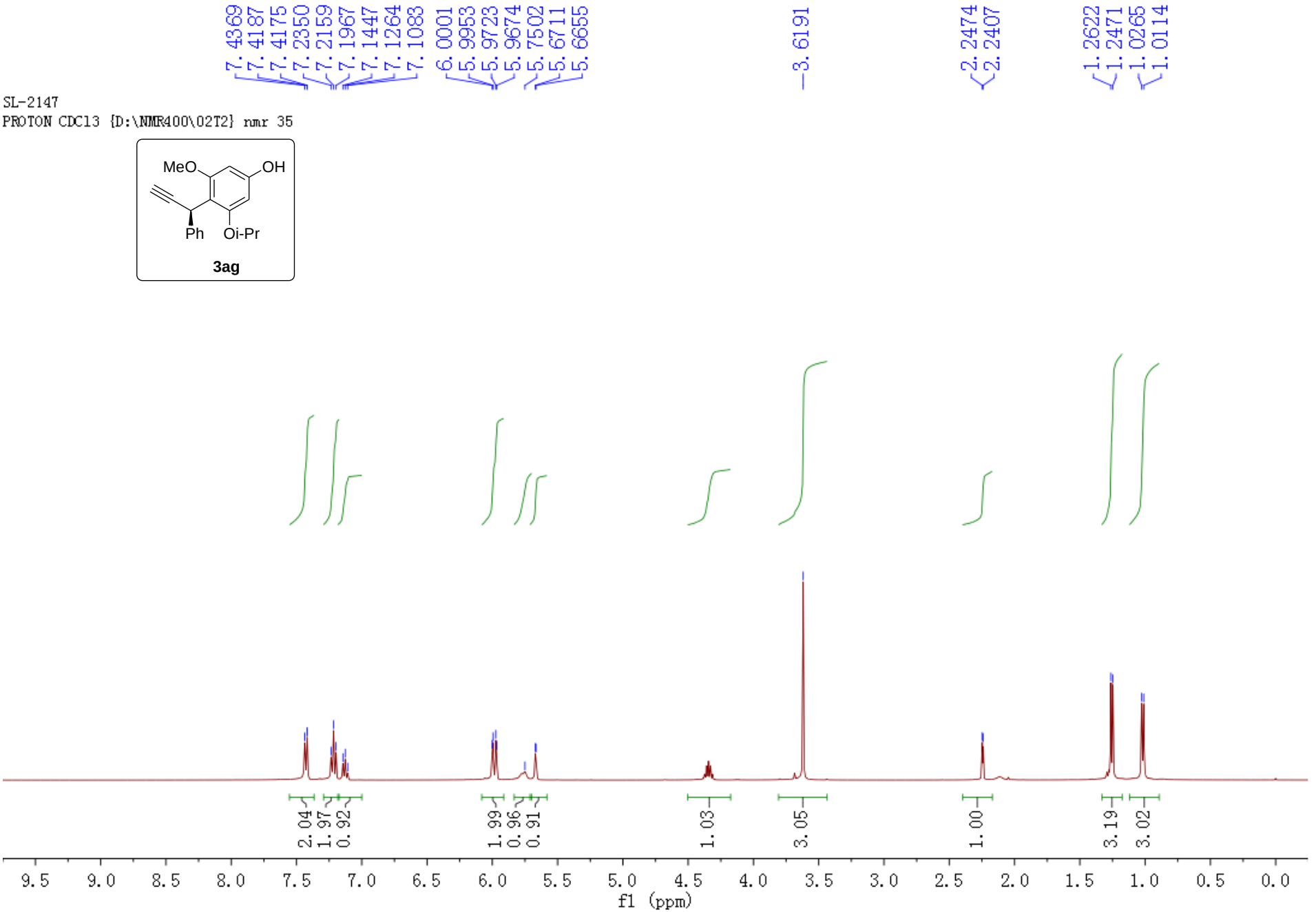


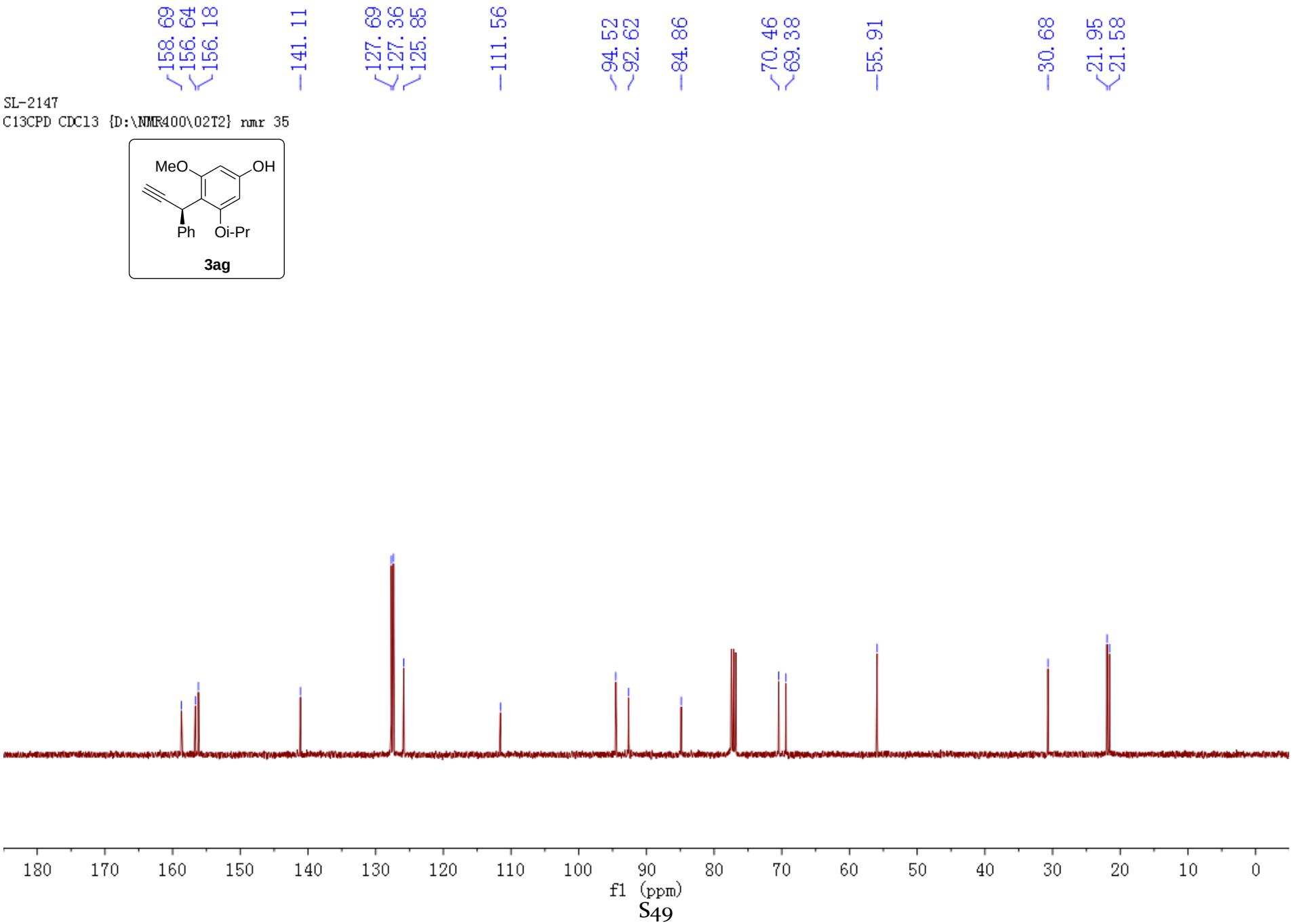
3ae

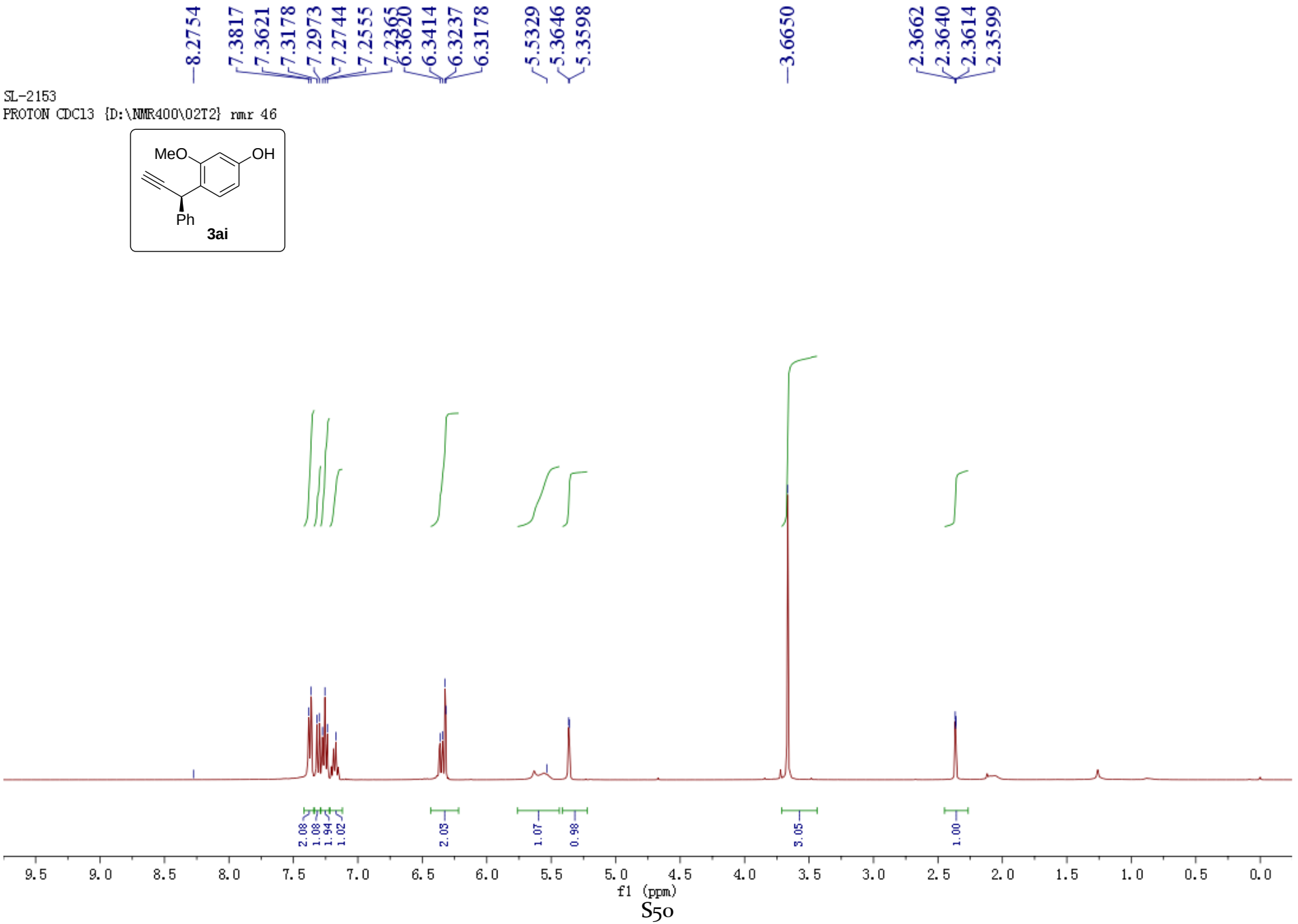



7.4096
7.3909
7.2264
7.2129
7.2004
7.1973
7.1828
7.1796
7.1714
7.1646
7.1313
7.1138
5.9984
5.9701
5.9660
5.8665
5.7403
4.9057
4.8757
4.8001
4.7700
3.5726
2.2338
2.2270
SL-2145
PROTON CDC13 {D:\NMR400\02T2} rmr 37
MeO
OH
Ph
OBn
3af
2.06
7.91
1.97
0.98
1.10
0.99
0.95
3.00
1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
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
f1 (ppm)


158.64
157.57
156.36
140.83
136.80
128.45
127.93
127.82
127.38
126.06
110.77
94.05
93.17
84.96
70.39
69.69
56.01
30.73
SL-2145
C13CPD CDCl3 {D:\NMR400\02T2} rmr 37
MeO
OH
Ph
OBn
3af
128.45
127.93
127.82
127.38
126.06
SL-2145
C13CPD CDCl3 {D:\NMR400\02T2} rmr 37
130
128
126
124
f1 (ppm)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

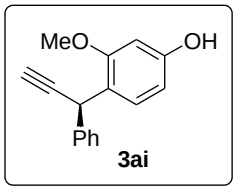

7.4369
7.4187
7.4175
7.2350
7.2159
7.1967
7.1447
7.1264
7.1083
6.0001
5.9953
5.9723
5.9674
5.7502
5.6711
5.6655
3.6191
2.2474
2.2407
1.2622
1.2471
1.0265
1.0114
SL-2147
PROTON CDC13 {D:\NMR400\02T2} rumr 35
MeO
OH
Ph
Oi-Pr
3ag
2.04
1.97
0.92
1.99
0.96
0.91
1.03
3.05
1.00
3.19
3.02
9.5
9.0
8.5
8.0
7.5
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
f1 (ppm)


158.69
156.64
156.18
141.11
127.69
127.36
125.85
111.56
94.52
92.62
84.86
70.46
69.38
55.91
30.68
21.95
21.58
SL-2147
C13CPD CDCl3 {D:\NMR400\02T2} rumr 35
MeO
OH
Ph
Oi-Pr
3ag
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

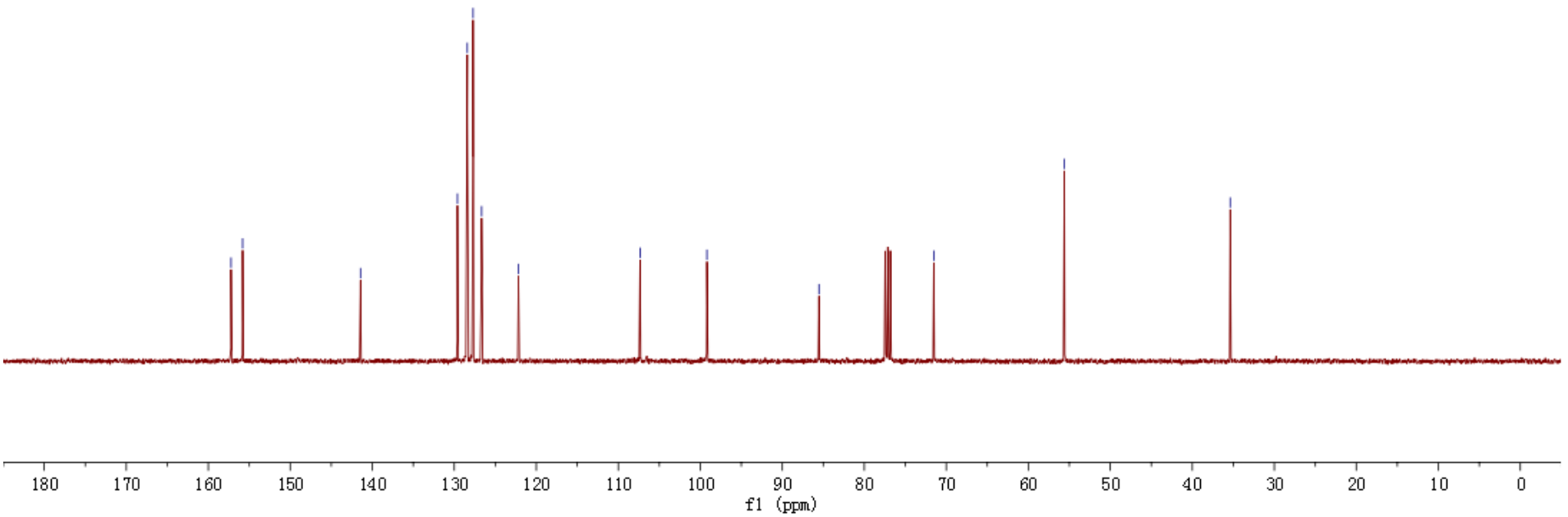

8.2754
7.3817
7.3621
7.3178
7.2973
7.2744
7.2555
7.2365
6.3620
6.3414
6.3237
6.3178
5.5329
5.3646
5.3598
3.6650
2.3662
2.3640
2.3614
2.3599
SL-2153
PROTON CDCl3 {D:\NMR400\02T2} rmr 46
MeO
OH
Ph
3ai
2.08
1.08
1.94
1.02
2.03
1.07
0.98
3.05
1.00
f1 (ppm)

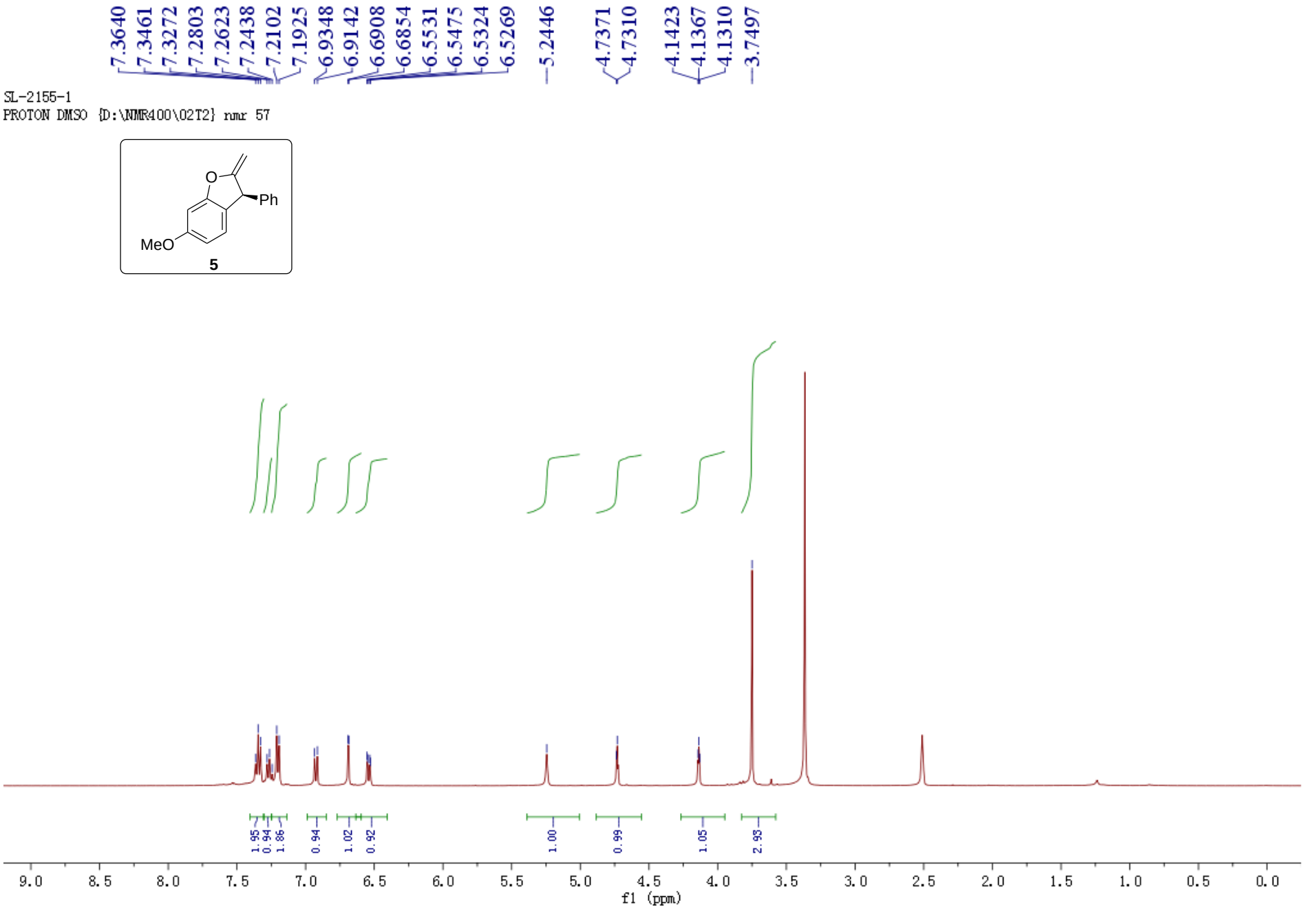
157.22
155.81
141.44
129.62
128.42
127.71
126.66
122.18
107.34
99.19
85.53
71.52
55.59
35.38

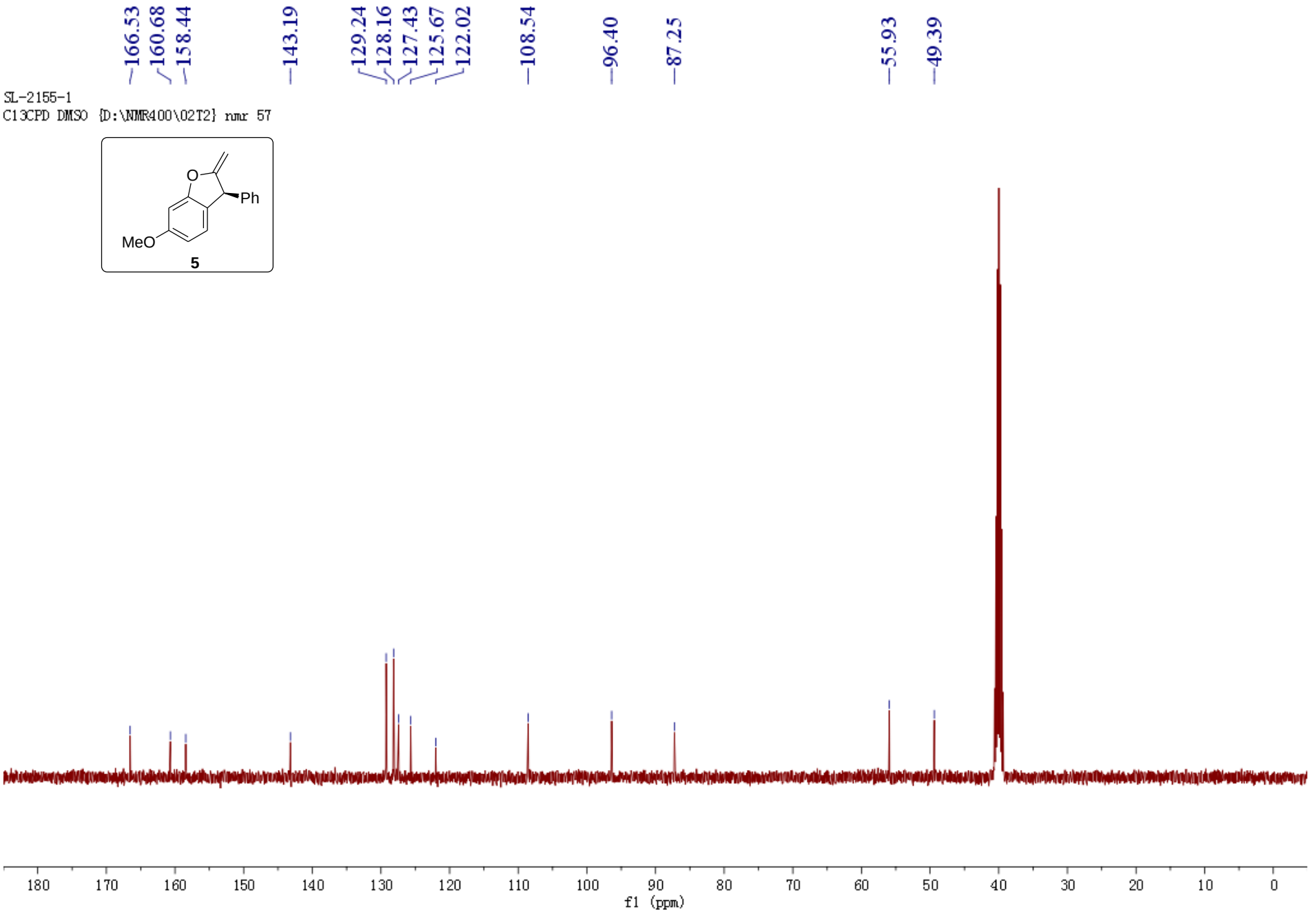
SL-2153
C13CPD CDCl3 {D:\NMR400\02T2} rmr 46

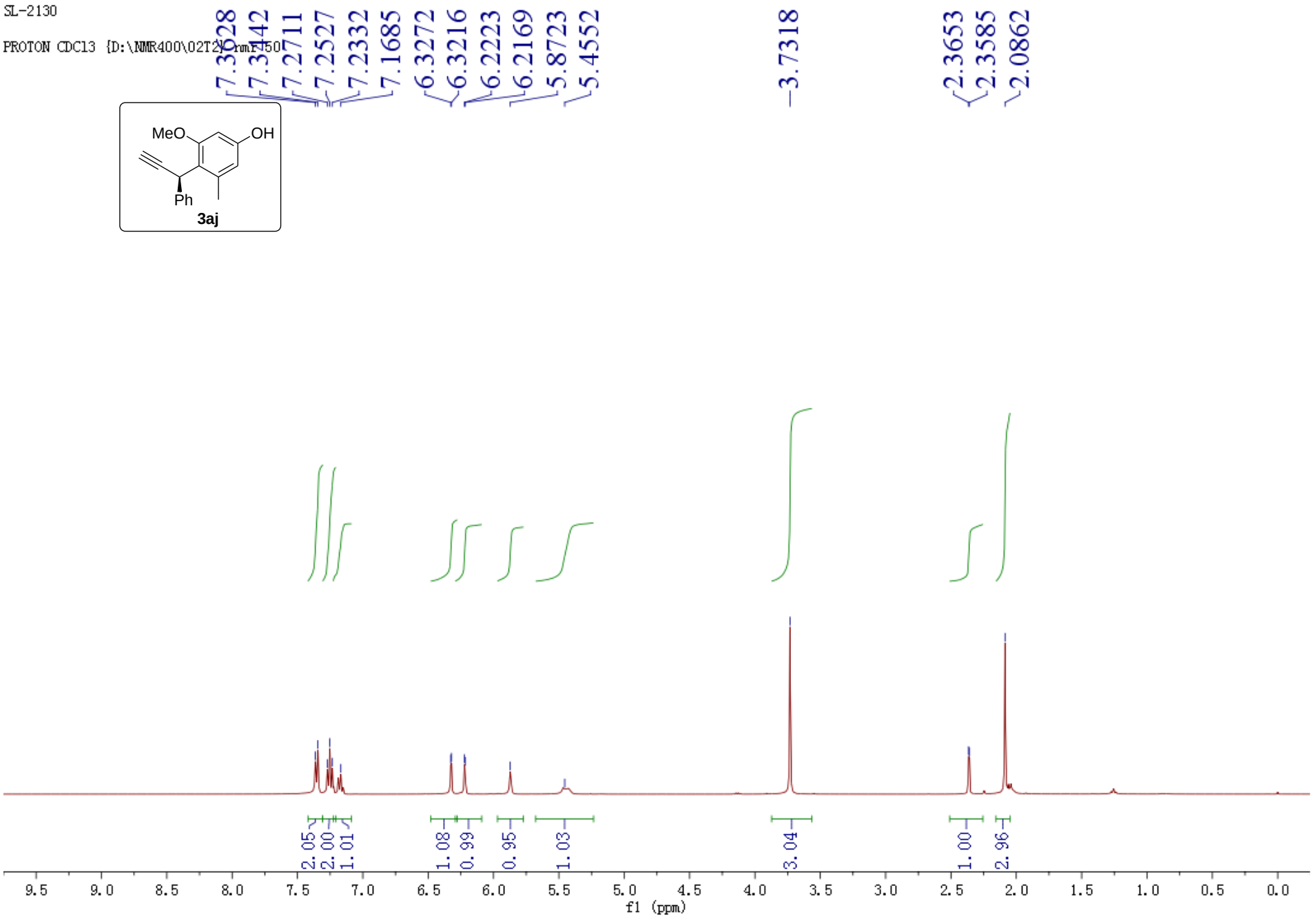


3ai



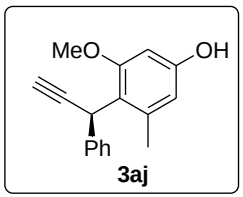

7.3640
7.3461
7.3272
7.2803
7.2623
7.2438
7.2102
7.1925
6.9348
6.9142
6.6908
6.6854
6.5531
6.5475
6.5324
6.5269
5.2446
4.7371
4.7310
4.1423
4.1367
4.1310
3.7497
SL-2155-1
PROTON DMSO {D:\NMR400\02T2} rmr 57
O
Ph
MeO
5
1.95
0.94
1.86
0.94
1.02
0.92
1.00
0.99
1.05
2.93
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


166.53
160.68
158.44
143.19
129.24
128.16
127.43
125.67
122.02
108.54
96.40
87.25
55.93
49.39
SL-2155-1
C13CPD DMSO {D:\NMR400\02T2} rmr 57
O
Ph
MeO
5
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


SL-2130
PROTON CDCl3 {D:\NMR400\02T2} rmf 50
7.3928
7.3742
7.2711
7.2527
7.2332
7.1685
6.3272
6.3216
6.2223
6.2169
5.8723
5.4552
3.7318
2.3653
2.3585
2.0862
MeO
OH
Ph
3aj
2.05
2.00
1.01
1.08
0.99
0.95
1.03
3.04
1.00
2.96
f1 (ppm)

SL-2130

C13CPD CDCl3 {D:\NMR400\D2T2} rmr 50



3aj