

Tetrabutylammonium fluoride (TBAF)- catalyzed addition of substituted trialkylsilylalkynes to aldehydes, ketones and trifluoromethyl ketones

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Supporting Information

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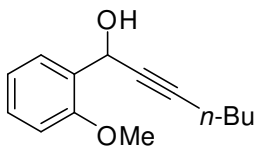
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General Information. All reactions were carried out under inert atmosphere using oven dried glassware and a magnetic stirrer. THF was distilled and dried over sodium. Trialkylsilylalkynes, tetrabutylammonium fluoride and all aldehydes/ketones were purchased from commercial sources and used without further purification. Products were purified via column chromatography using hexane/ethyl acetate. ^1H and ^{13}C NMR spectra were obtained on a 300 and 400 MHz NMR spectrometer. All NMR spectra were taken in CDCl_3 otherwise reported. Thin layer chromatography was used to monitor reaction progress.

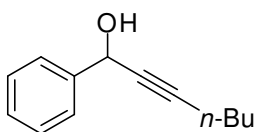
References for known compounds:

1-(2'-methoxyphenyl)hept-2-yn-1-ol (Table 1, entry 1)¹:



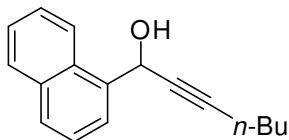
Purified via flash column chromatography with 10 % ethyl acetate/hexane, yielding 75 % of a yellow oil.

1-Phenylhept-2-yn-1-ol (Table 2, entry 2)¹:



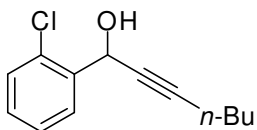
Purified via flash column chromatography with 10 % ethyl acetate/hexane, yielding 78 % of a yellow oil.

1-(naphthalen-1-yl)hept-2-yn-1-ol (Table 2, entry 3)¹:



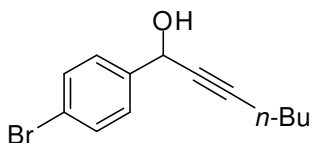
Purified via flash column chromatography with 10 % ethyl acetate/hexane, yielding 72 % of a yellow oil.

1-(4'-Bromophenyl)hept-2-yn-1-ol (Table 2, entry 4)²:



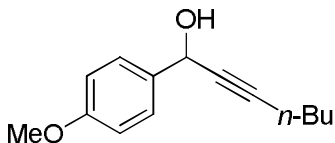
Purified via flash column chromatography with 10 % ethyl acetate/hexane, yielding 99 % of a yellow oil.

1-(4'-Bromophenyl)hept-2-yn-1-ol (Table 2, entry 5)¹:



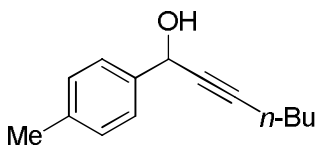
Purified via flash column chromatography with 10 % ethyl acetate/hexane, yielding 83 % of a yellow oil.

1-(4'-methoxyphenyl)hept-2-yn-1-ol (Table 2, entry 6)¹:

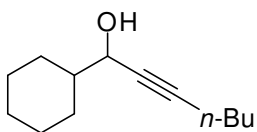


Purified via flash column chromatography with 10 % ethyl acetate/hexane, yielding 79 % of a yellow oil.

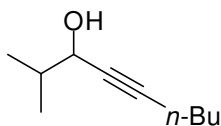
1-p-Tolylhept-2-yn-1-ol (Table 2, entry 7)¹:



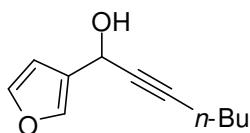
Purified via flash column chromatography with 10 % ethyl acetate/hexane, yielding 75 % of a yellow oil.

1-cyclohexylhept-2-yn-1-ol (Table 2, entry 8)¹:

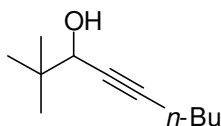
Purified via flash column chromatography with 10 % ethyl acetate/hexane, yielding 91 % of a colorless oil.

2-methylnon-4-yn-3-ol (Table 2, entry 9)¹:

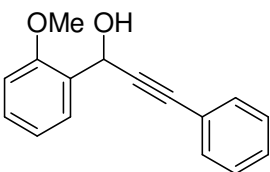
Purified via flash column chromatography with 10 % ethyl acetate/hexane, yielding 91% of a colorless oil.

1-(furan-3-yl)hept-2-yn-1-ol (Table 2, entry 10)¹:

Purified via flash column chromatography with 10 % ethyl acetate/hexane, yielding 72 % of a red oil.

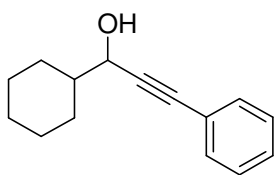
2,2-Dimethylnon-4-yn-3-ol (Table 2, entry 11)¹:

Purified via flash column chromatography with 10 % ethyl acetate/hexane, yielding 84 % of a yellow oil.

1-(2-Methoxy-phenyl)-3-phenyl-prop-2-yn-1-ol (Table 3, entry 1)³:

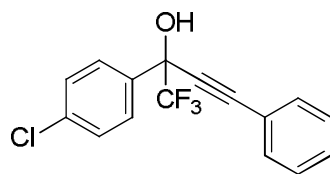
Purified via flash column chromatography with 10 % ethyl acetate/hexane, yielding 76 % of a yellow oil.

1-Cyclohexyl-3-phenyl-prop-2-yn-1-ol (Table 3, entry 2):⁴



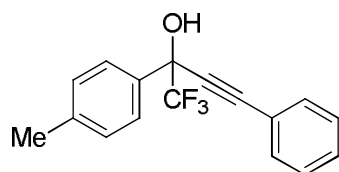
Purified via flash column chromatography with 10 % ethyl acetate/hexane, yielding 75 % of a yellow oil.

2-(4-Chlorophenyl)-1,1,1-trifluoro-4-phenylbut-3-yn-2-ol (Table 4, entry 2):⁵



Purified via flash column chromatography with 10 % ethyl acetate/hexane, yielding 98% as colorless oil.

1,1,1-Trifluoro-4-phenyl-2-(p-tolyl)but-3-yn-2-ol (Table 4, entry 3):⁵



Purified via flash column chromatography with 10 % ethyl acetate/hexane, yielding 90% as colorless oil.

References:

1. Scheidt, K. A.; Lettan, R. B. *Org. Lett.* **2005**, 7, 3227-3230.
2. Braga, A. L.; Appelt, H. R.; Silveira, C. C.; Wessjohann, L. A.; Schneider, P. H. *Tetrahedron* **2002**, 58, 10413-10416.
3. Xu, M.; Pu, L. *Org. Lett.* **2002**, 4, 4555-4557.

4. Ishikawa, T.; Mizuta, T.; Hagiwara, K.; Aikawa, T.; Kudo, T.; Saito, S. *J. Org. Chem.* **2003**, 68, 3702-3705.
5. (a) Motoki, R.; Kanai, M.; Shibasaki, M. *Org. Lett.* **2007**, 9, 2997-3000. (b) Linderman, R. J.; Lonikar, M. *J. Org. Chem.* **1988**, 53, 6013-6022.

KW273, CDCL₃
300MHz
12/06/06

7.609
7.579
7.266
6.980
6.961
6.906
6.878
5.714
3.876
2.957
2.303
2.280
2.258
1.439
1.389
0.937
0.913
0.889

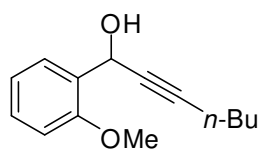
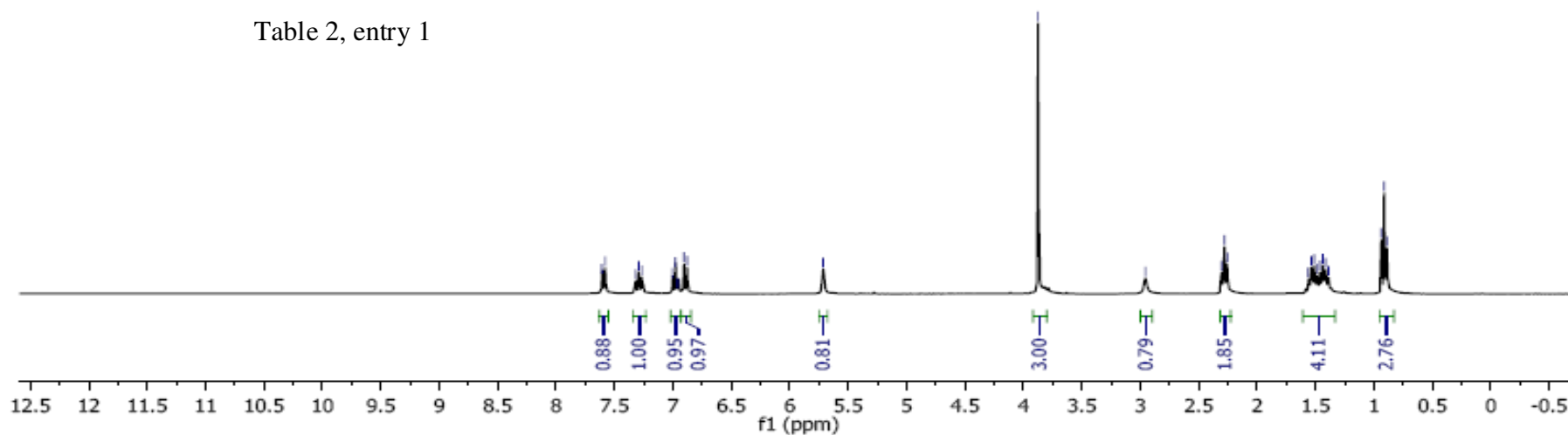


Table 2, entry 1



KW273, CDCl₃
75MHz
12/06/06

—156.992
129.714
129.538
—128.140
—120.997
—110.903
—87.418
79.334
77.730
77.306
76.882
—61.455
—55.719
—30.930
—22.204
—18.810
—13.853

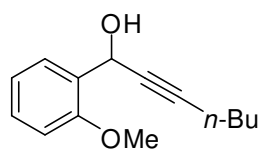
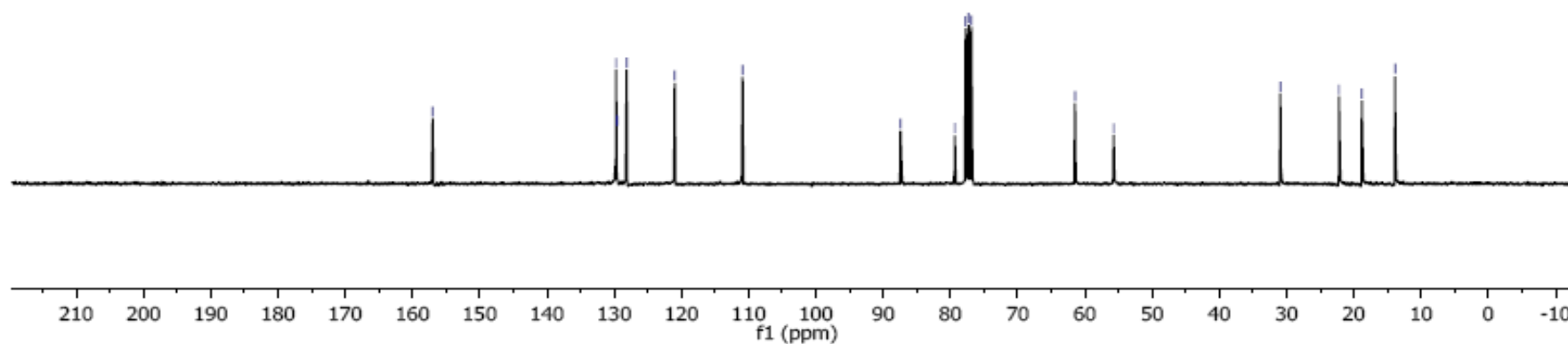


Table 2, entry 1



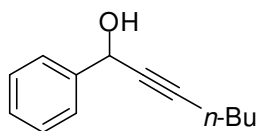
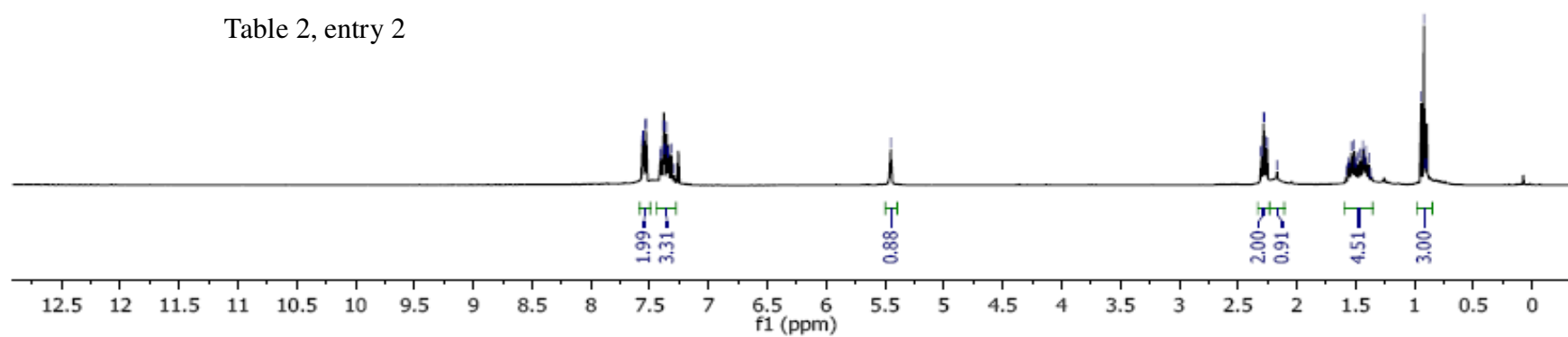
KW268, CDCl₃
300MHz7.563
7.556
7.536
7.534
7.409
7.402
7.382
7.375
7.356
7.345
7.317
7.293
— 5.4522.306
2.299
2.283
2.276
2.259
2.252
1.512
1.438
1.374
0.943
0.919
0.903

Table 2, entry 2



KW268, CDCl₃
75MHz

—141.530
—128.753
—128.408
—126.883

—87.873
—80.181
—77.740
—77.316
—76.892
—65.026

—30.893
—22.235
—18.758
—13.852

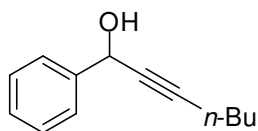
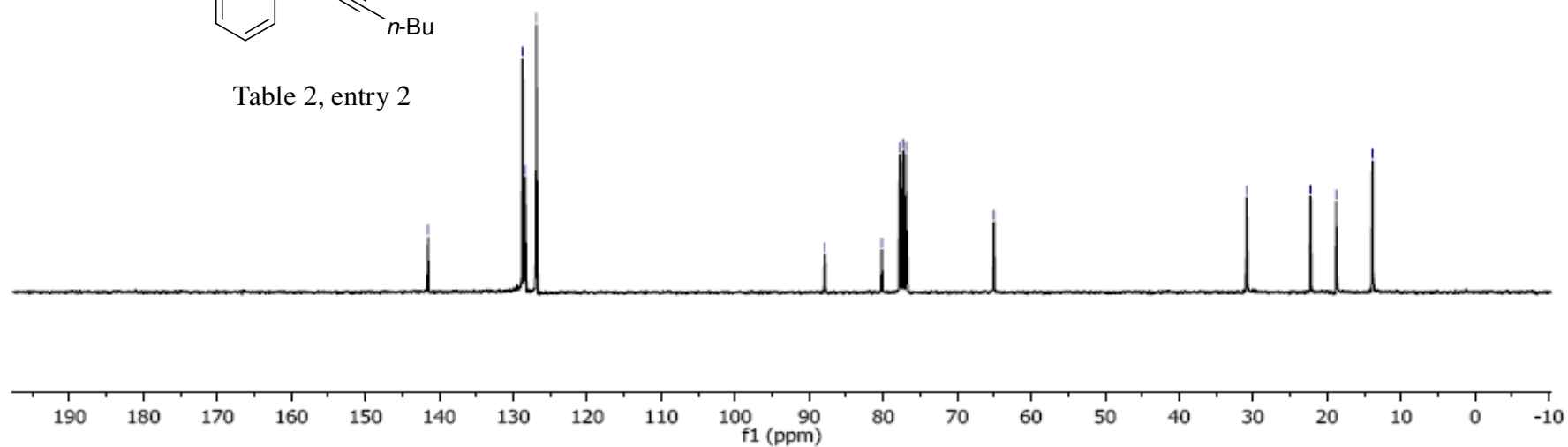


Table 2, entry 2



KW298, CDCL₃
300MHz
01/24/07

8.337
8.310
7.824
7.541
7.509
7.480
7.452
7.258
6.116

2.466
2.330
2.300
2.277
1.528
1.450
1.400
0.953
0.929
0.905

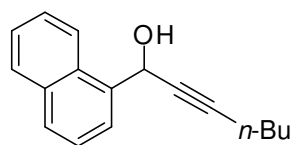
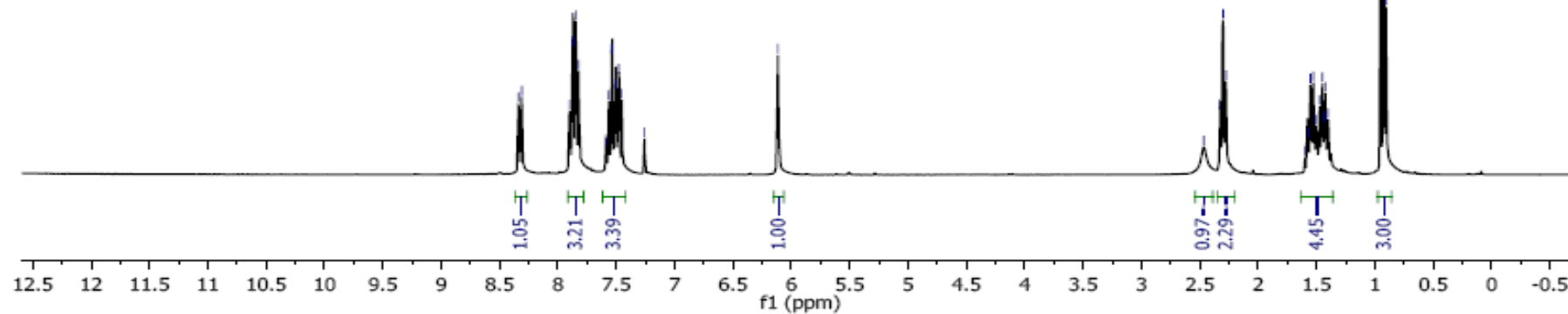


Table 2, entry 3



KW298, CDCl₃
75MHz
01/24/07

136.561
134.262

130.826
129.366

128.905
126.501

126.027
125.463

124.650
124.330

88.565

79.957
77.743

77.319
76.895

63.264

30.877

22.257

18.842

13.859

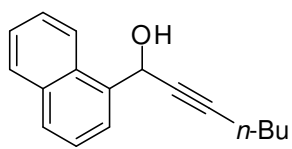
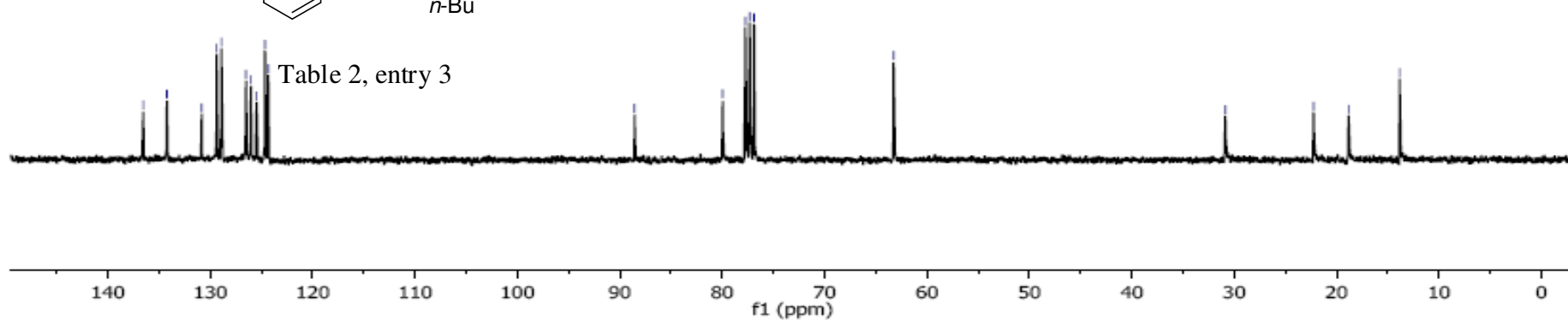


Table 2, entry 3



KW277. CDCl₃
400MHz

7.766
7.747
7.364
7.276
7.259
7.226
5.797

2.607
2.836
2.238
1.417
1.380
0.923
0.904
0.886

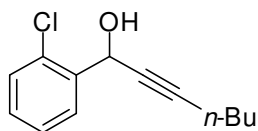
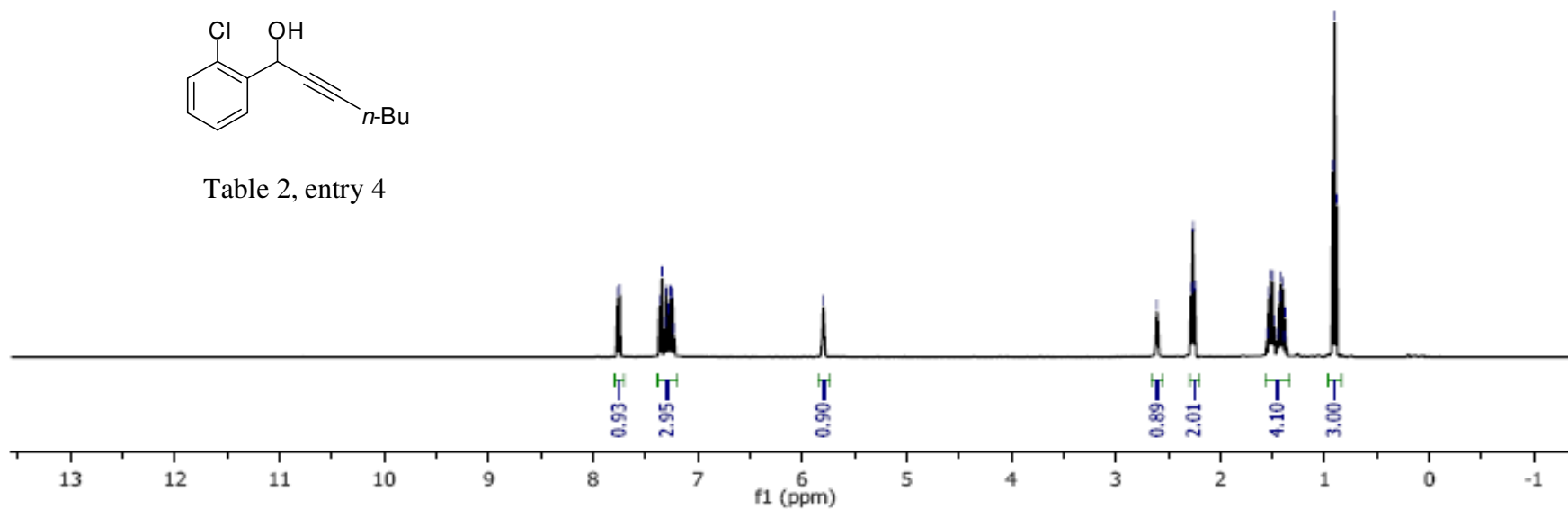


Table 2, entry 4



KW277, CDCl₃
100MHz

138.696
132.943
129.843
129.662
128.526
127.359

88.018
78.996
77.637
77.320
77.002
62.286

30.818
22.227
18.768
13.857

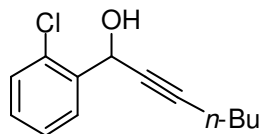
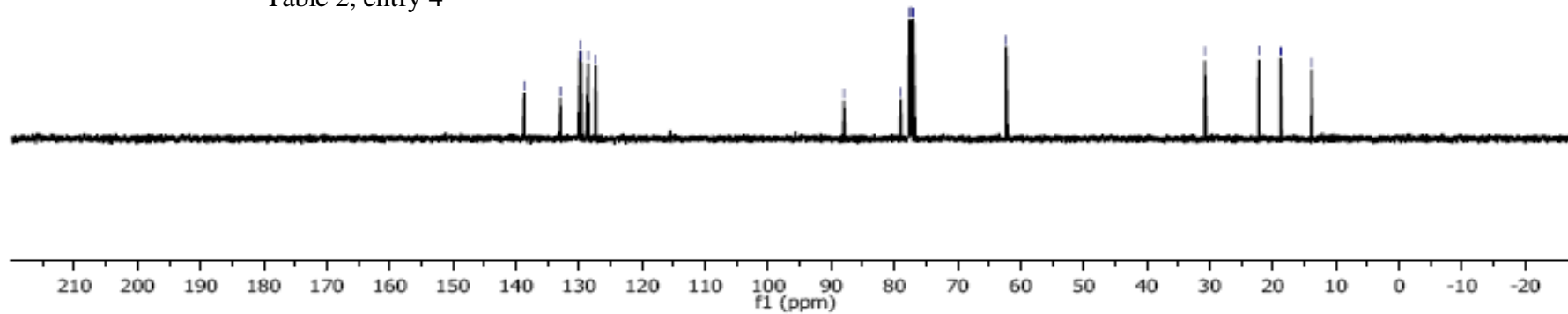


Table 2, entry 4



KW278, CDCl₃
400MHz7.485
7.464
7.395
7.373

-5.375

-2.608

2.854

2.232

1.414

1.377

0.928

0.910

0.892

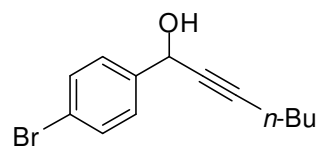
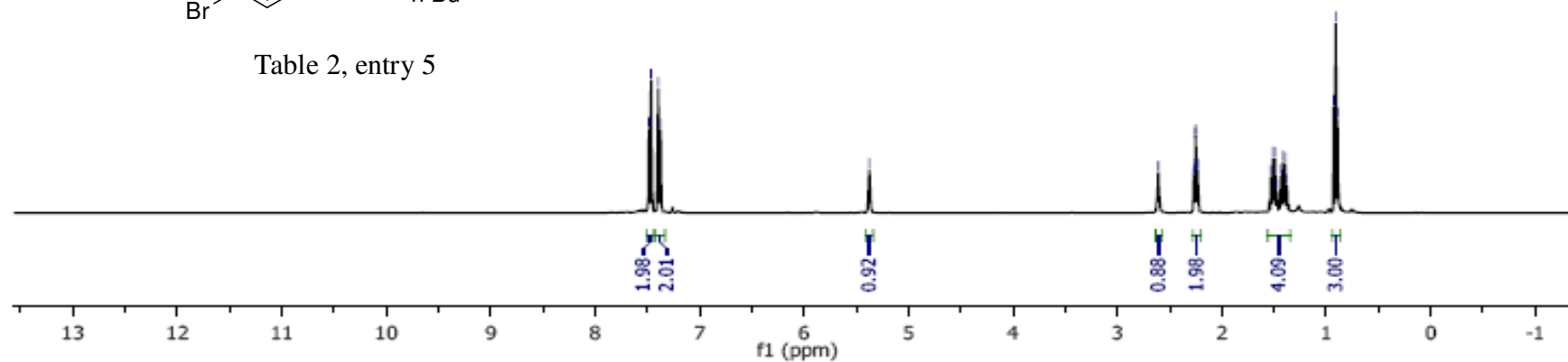


Table 2, entry 5



KW278, CDCl₃
100MHz

—140.473
—131.766
—128.581
—122.309

—88.238
—79.751
—77.655
—77.338
—77.020
—64.297

—30.841
—22.255
—18.739
—13.876

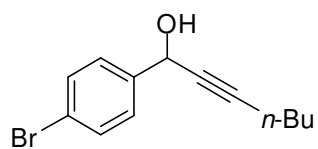
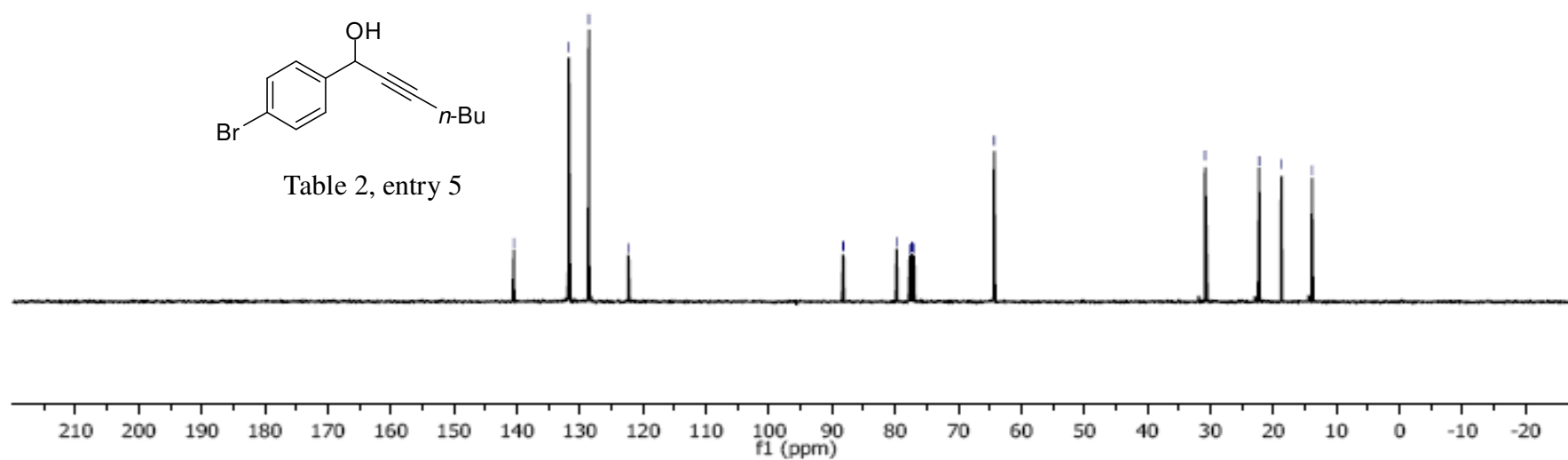


Table 2, entry 5



KW274, CDCl₃
400MHz

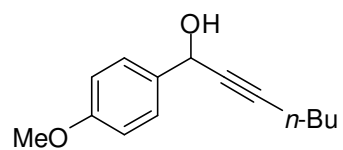
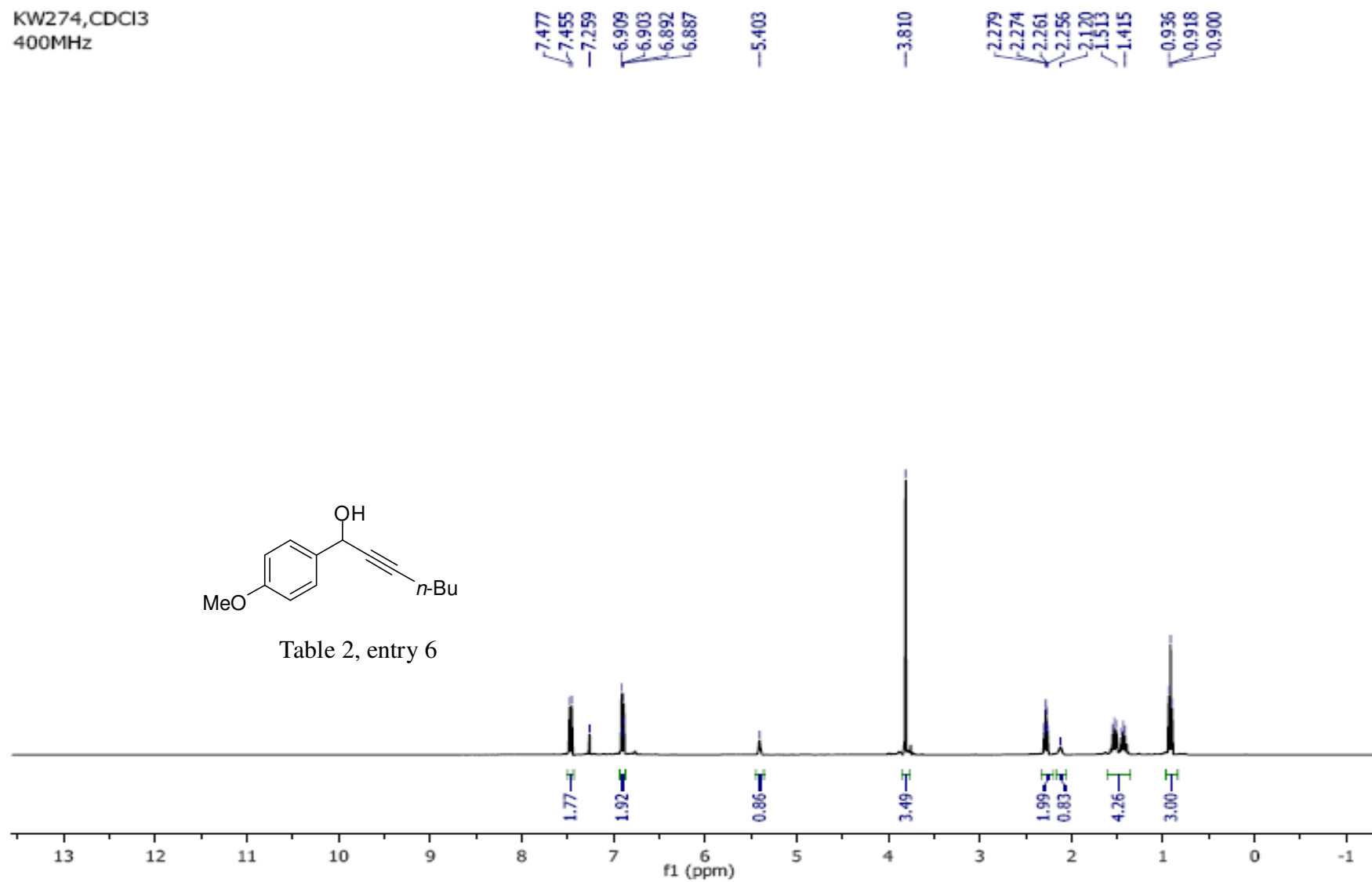


Table 2, entry 6



KW274, CDCl₃
75MHz

—159.756

—133.866

—128.291

—114.093

—87.698

77.696

76.848

64.717

64.641

55.594

55.514

—30.903

—22.227

—18.752

—13.849

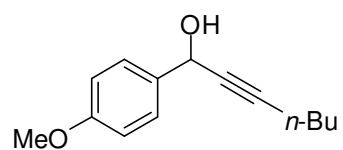
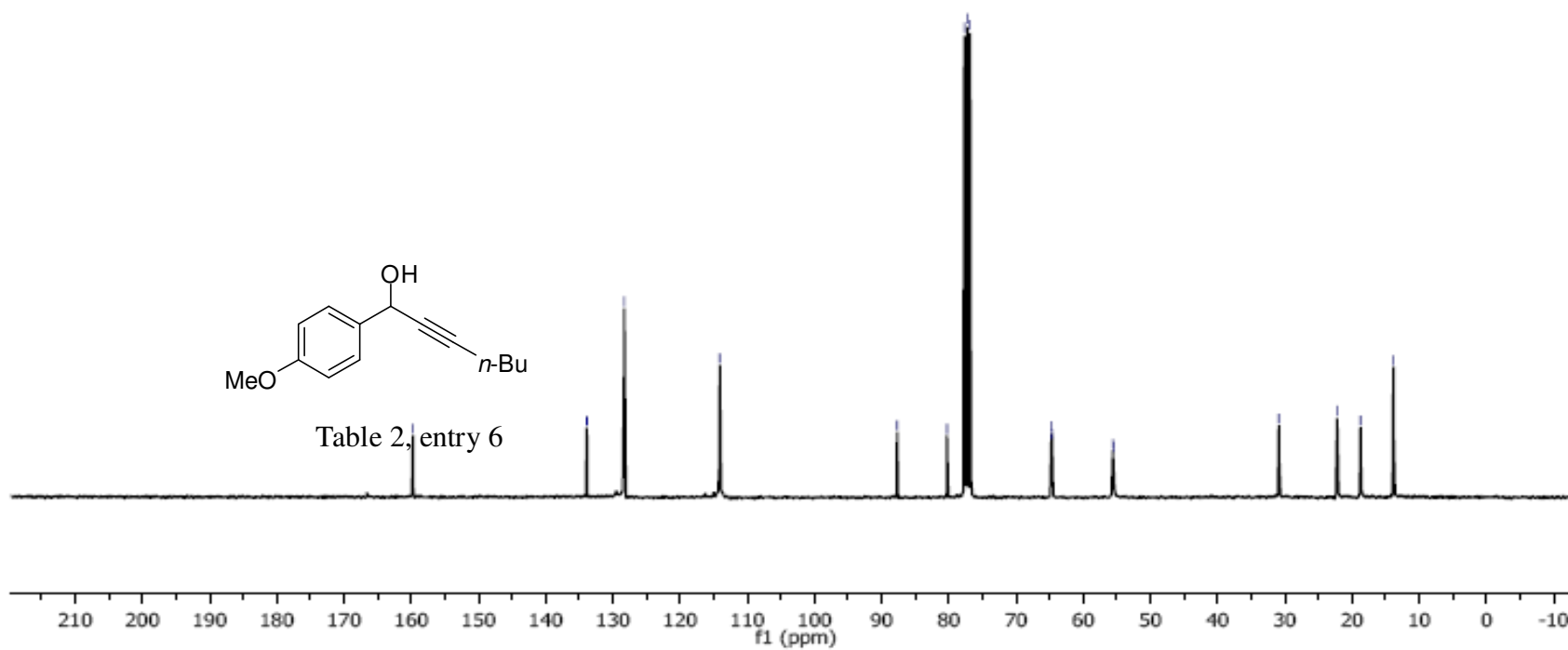


Table 2, entry 6



KW296, CDCl₃
400MHz

7.445
7.425
7.196
7.177

5.407

2.622
2.370
2.264
1.528
1.435
0.961
0.943
0.925

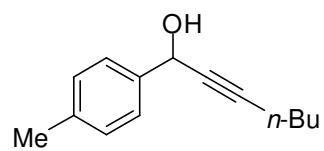
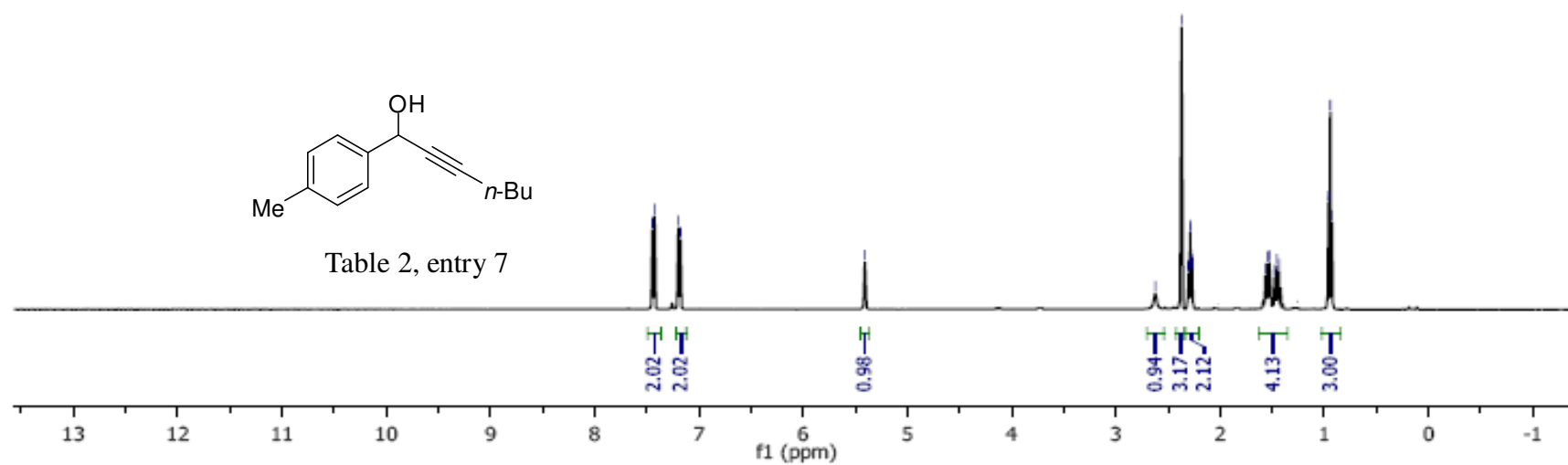


Table 2, entry 7



KW296, CDCl₃
100MHz

138.763
138.073
129.385
126.865

87.510
80.441
77.408
77.090
64.798

30.961
22.281
18.804
13.901

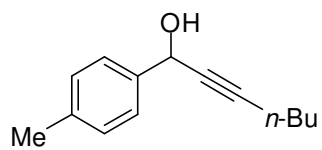
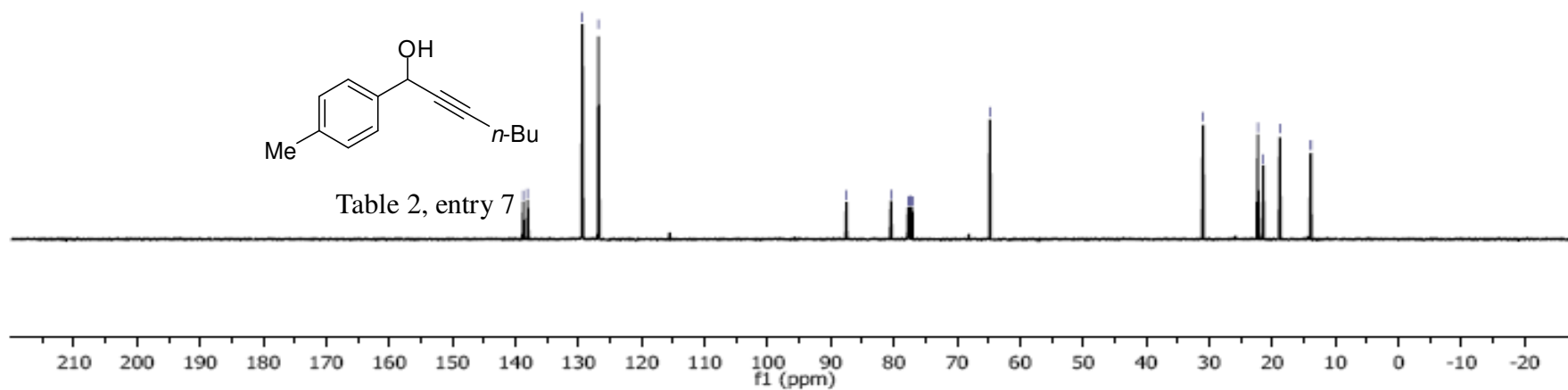
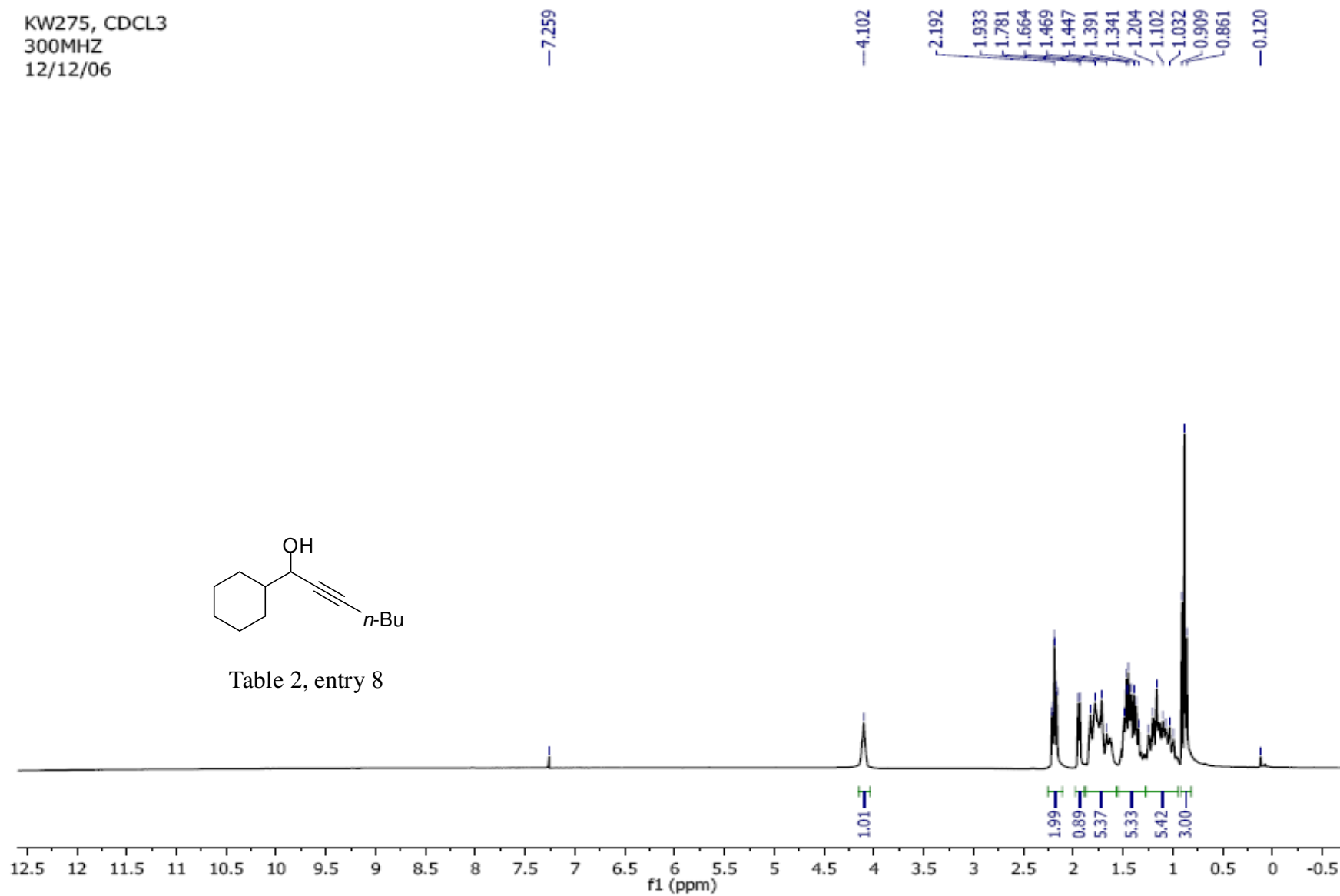


Table 2, entry 7



KW275, CDCL3
300MHZ
12/12/06



KW275C1, CDCL₃
75MHz
12/12/06

86.369
80.359
77.708
77.284
76.860
67.618
67.557
44.547
31.023
28.832
28.281
26.657
26.136
22.152
18.595
13.783

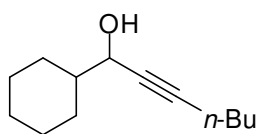
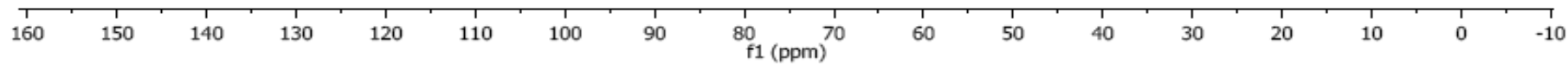


Table 2, entry 8



KW285, CDCL3
300MHz
12/20/06

4.112
2.184
2.178
2.161
2.155
2.086
1.767
1.360
0.941
0.898
0.850
-0.111

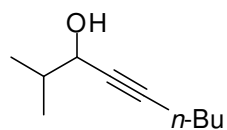
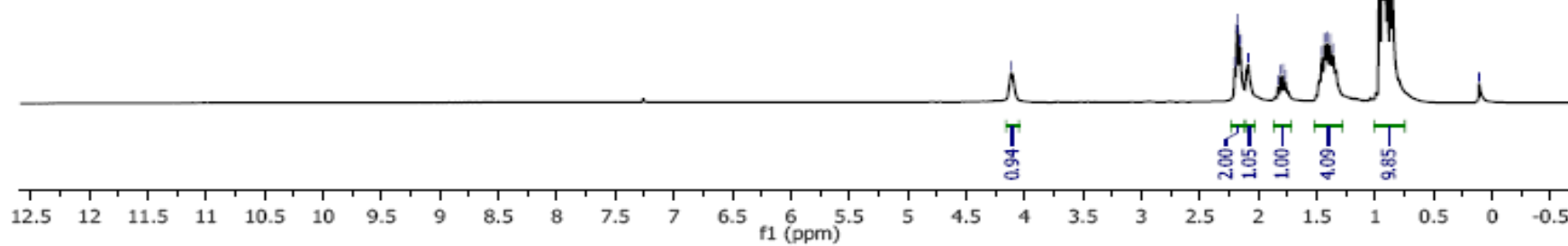


Table 2, entry 9



KW285, CDCL₃
75 MHz
12/20/06

86.264
80.021
77.702
77.279
76.854
68.216

34.827
30.990
22.101
18.347
13.754

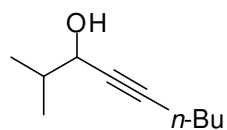
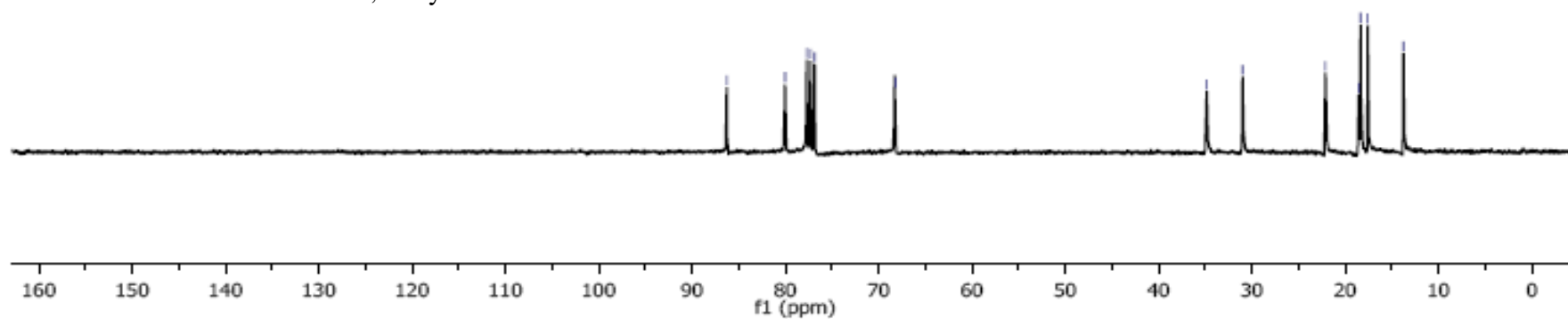
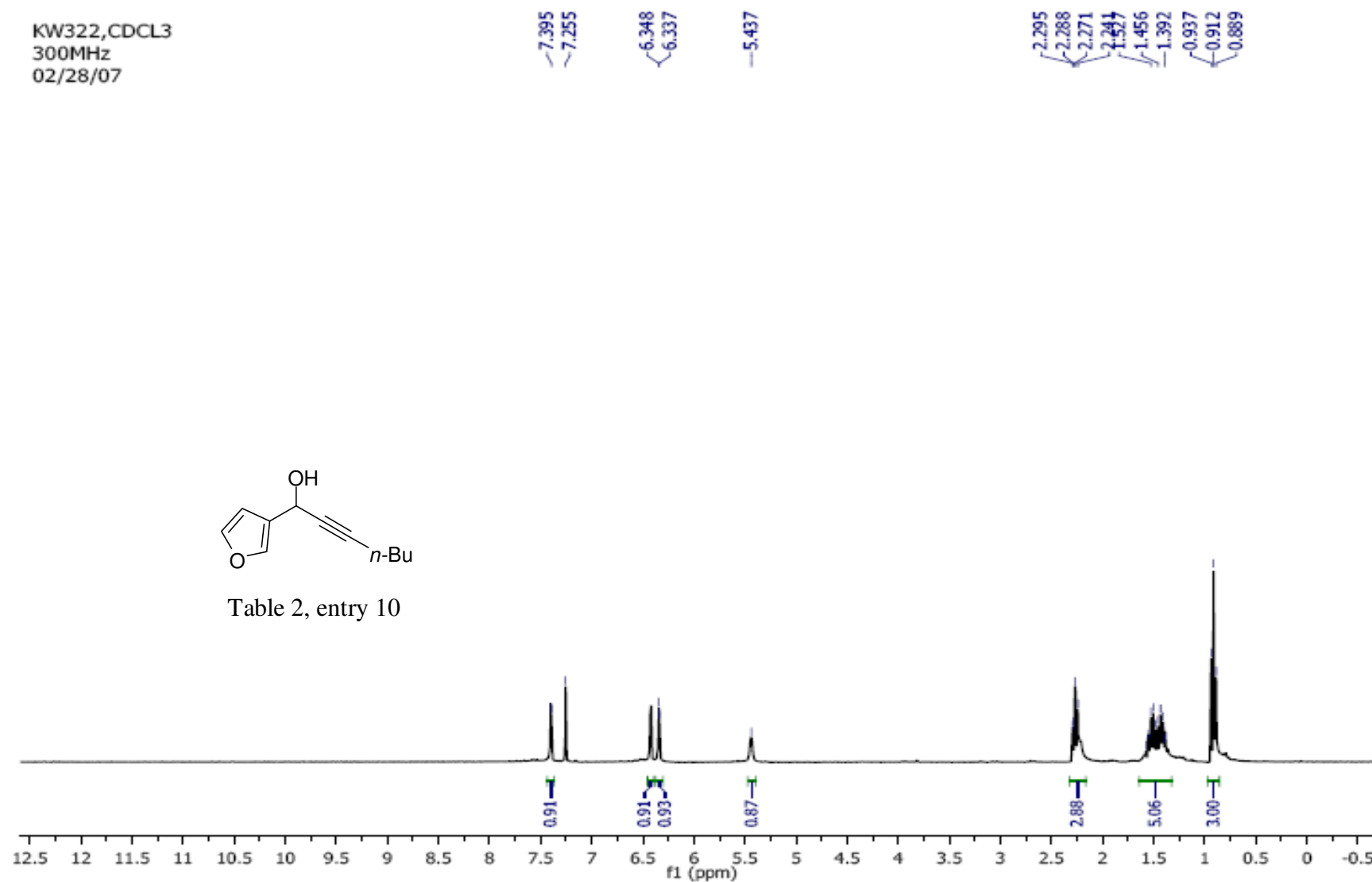


Table 2, entry 9





KW322, CDCl₃
75MHz

153.860 143.106 110.544 107.689 87.178 77.666 77.242 76.818 58.603 30.710 22.167 18.649 13.808

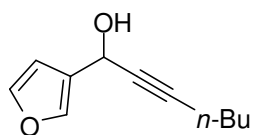
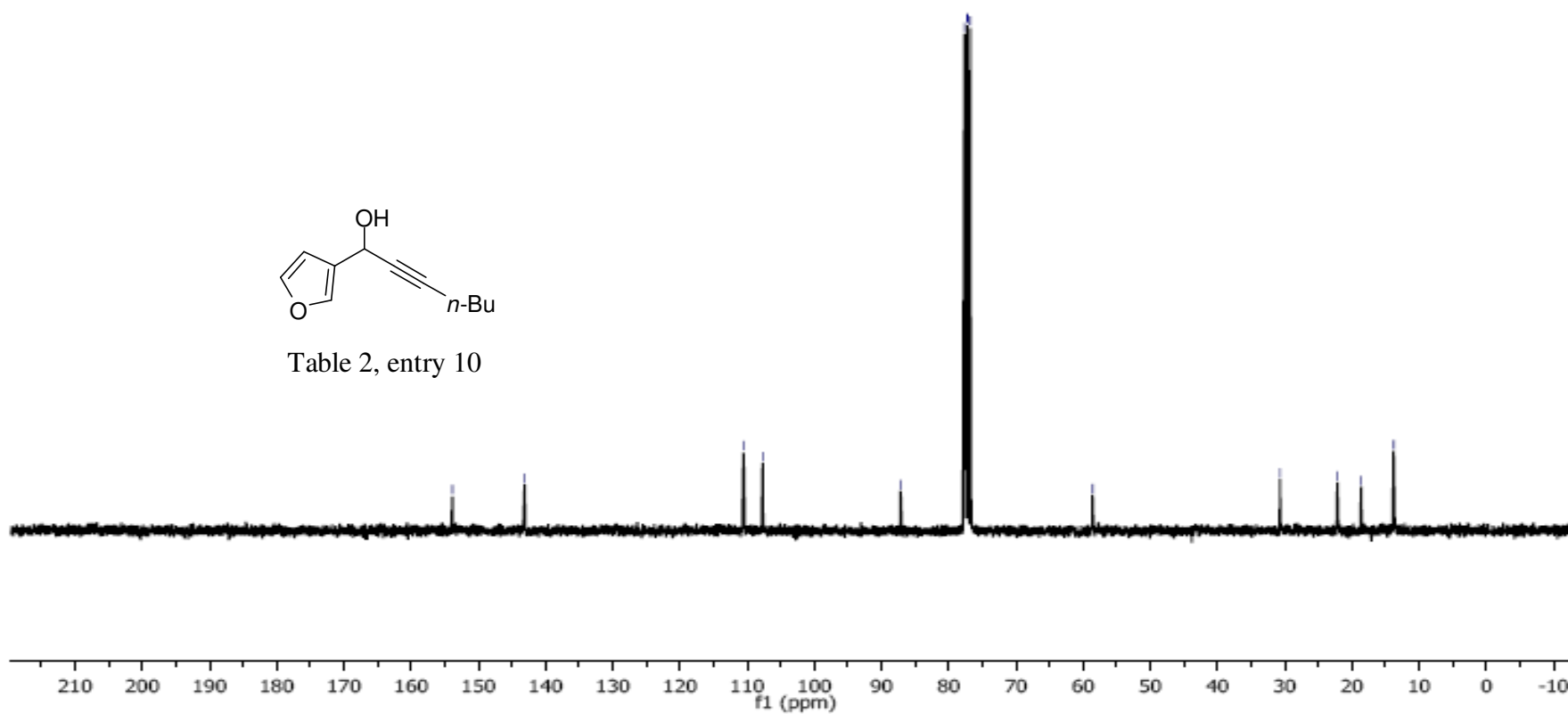
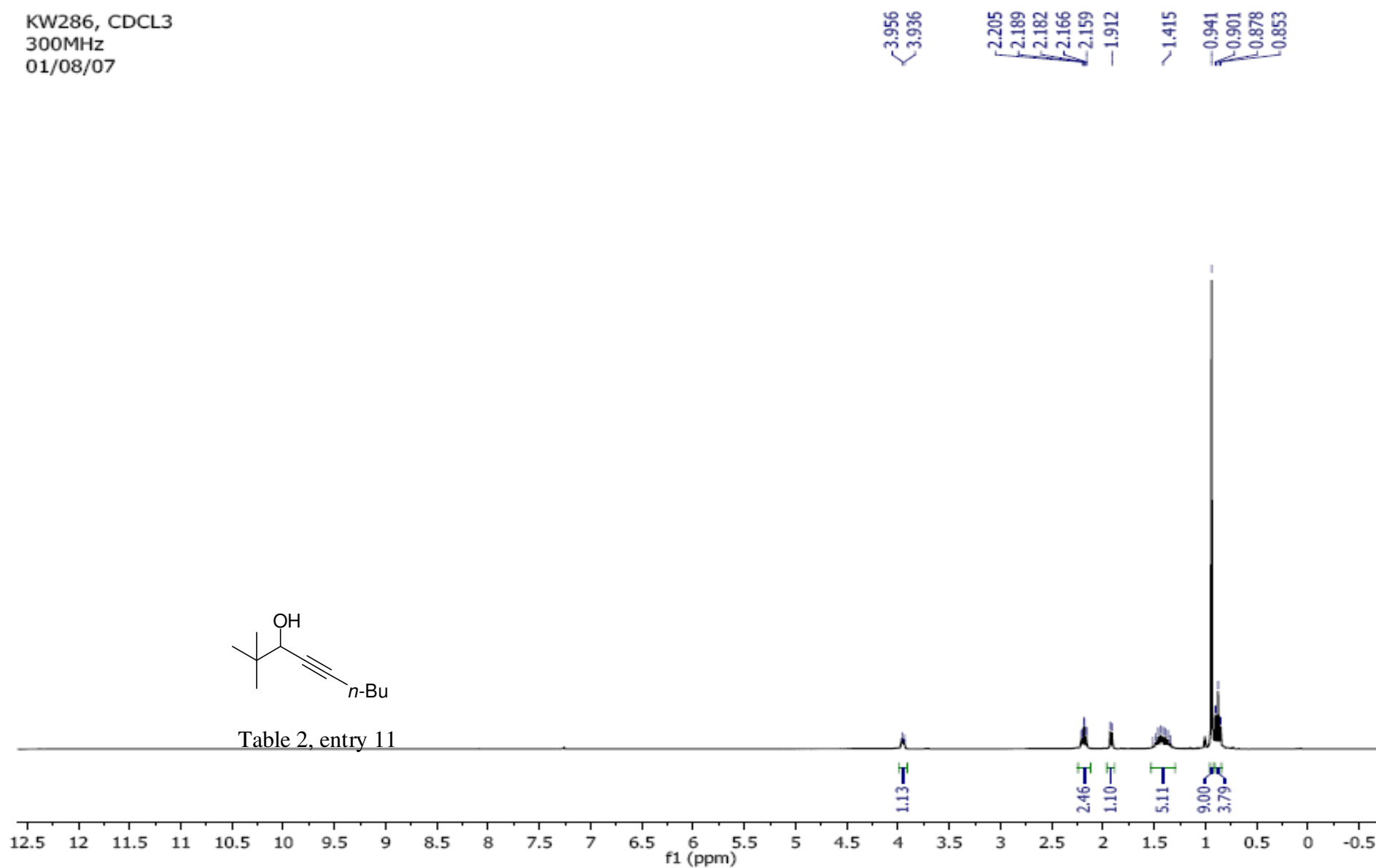


Table 2, entry 10



KW286, CDCL₃
300MHz
01/08/07



KW286, CDCL₃
75MHz
01/08/07

—86.309
—76.846
—71.763

—36.000
—31.002
—25.473
—18.538
—13.754

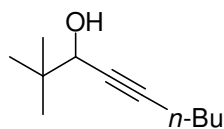
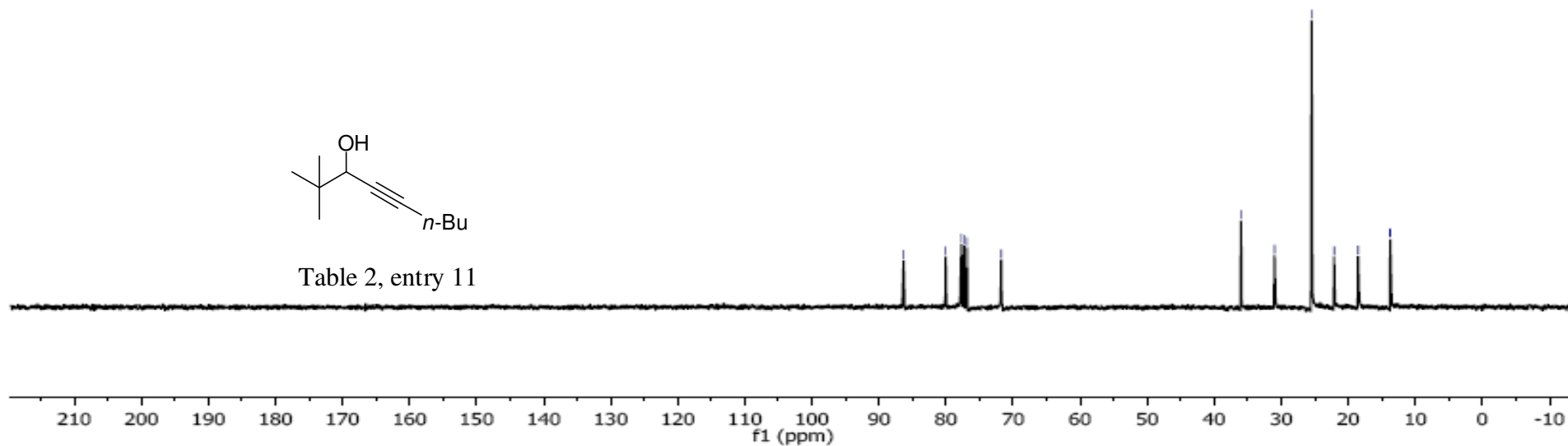


Table 2, entry 11



KW313, CDCl₃
300MHz

7.69
7.66
7.52
7.50
7.48
7.37
7.34
7.02
6.95
6.93
— 5.96
— 3.91
— 3.20

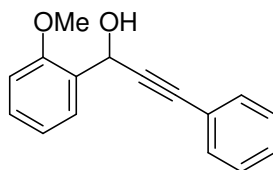
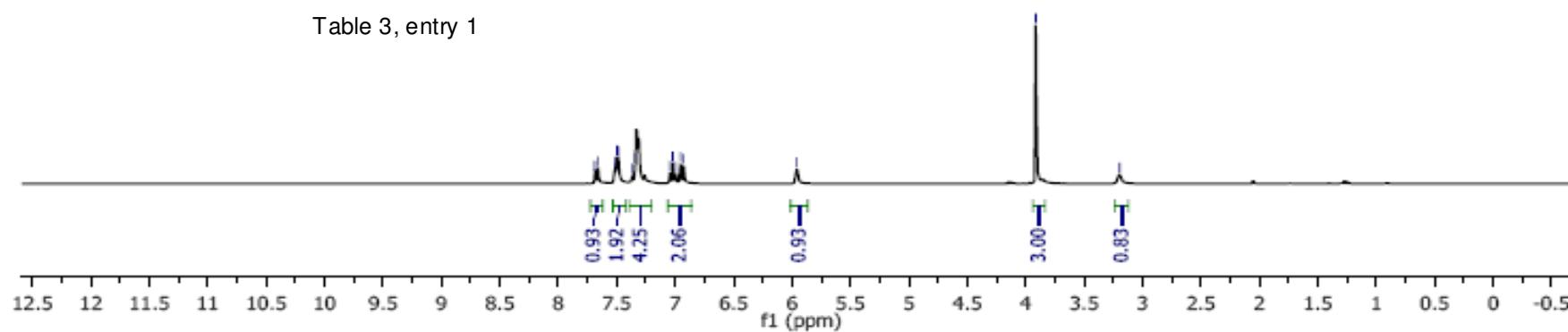


Table 3, entry 1



KW313, CDCl₃
75MHz

157.09 132.02 129.04 128.65 128.49 128.28 123.00 121.15 111.15 88.65 86.32 77.77 77.35 76.92 61.83 55.91 55.82

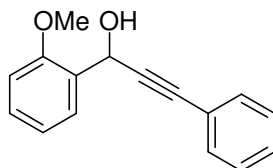
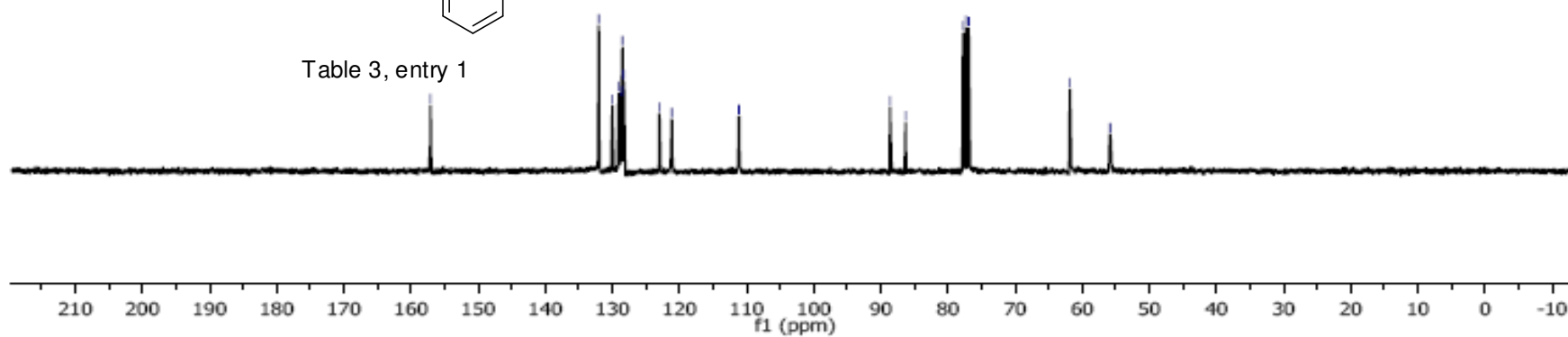
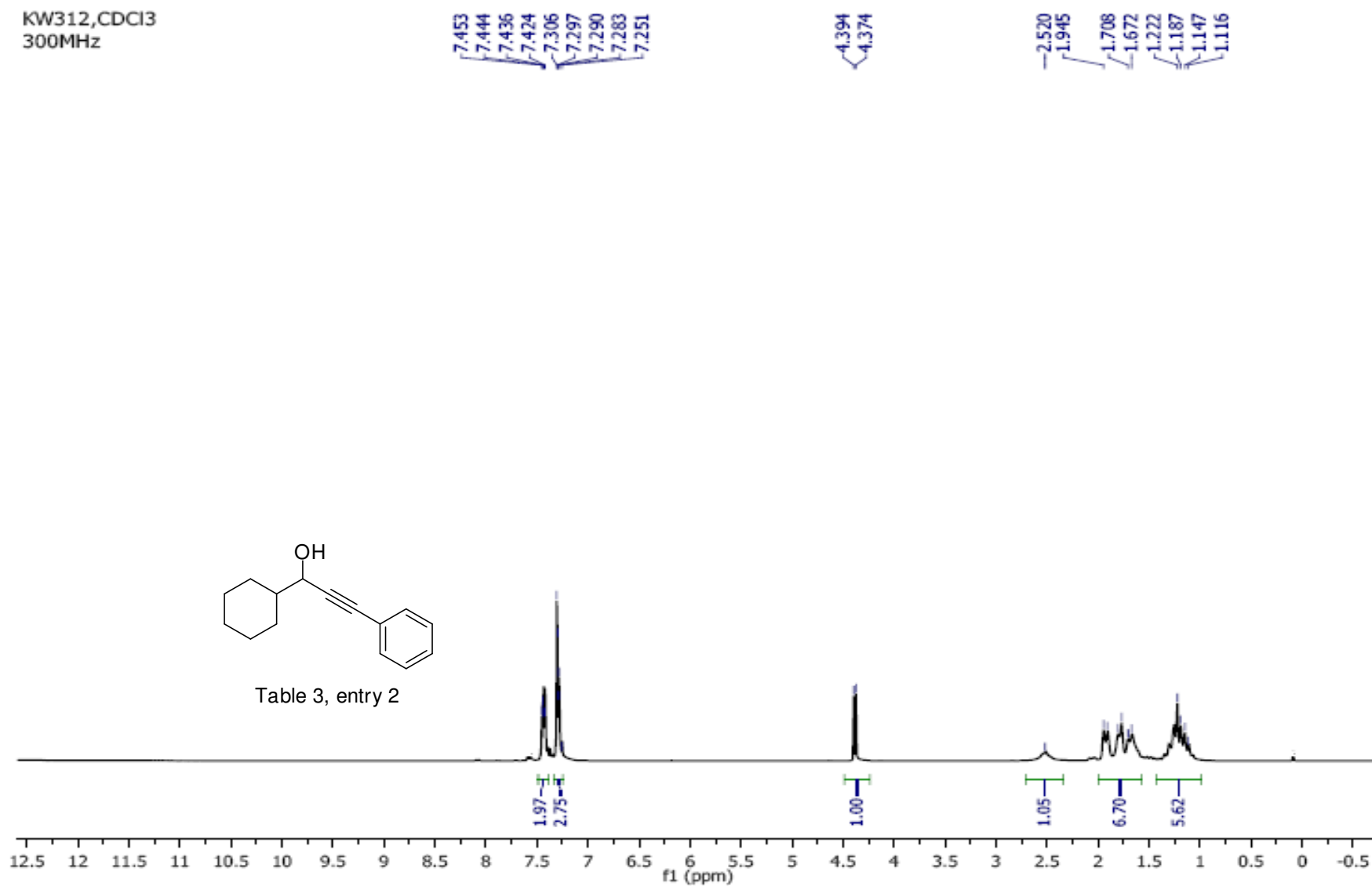


Table 3, entry 1





KW312, CDCl₃
75MHz

131.93
128.50
123.01
89.54
85.87
77.74
77.32
76.90
67.89
44.54
28.93
28.45
26.65
26.16

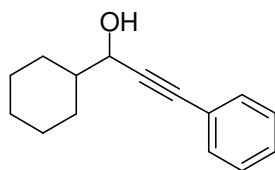
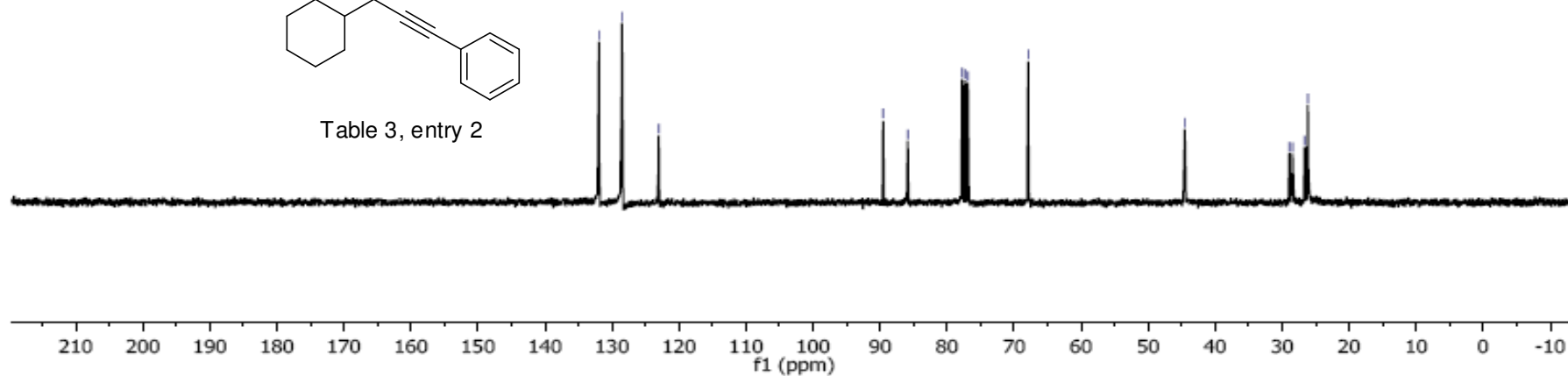


Table 3, entry 2



KW464, CDCl₃
400MHz
YELLOW OIL

7.624
7.621
7.606
7.602
7.373
7.369
7.353
7.350
7.334
7.330
7.259
7.040
7.019
7.000
6.965
6.944
5.944
5.928
— 3.931
3.169
3.153

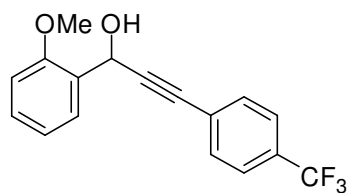
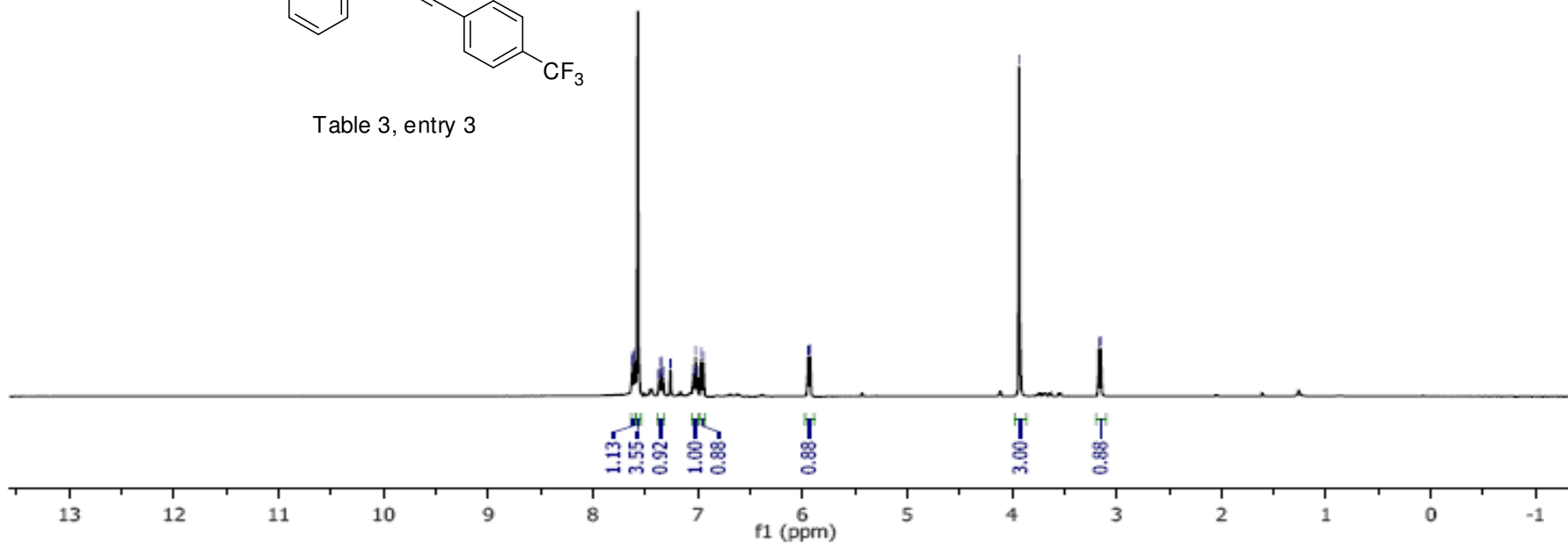


Table 3, entry 3



KW464C, CDCl₃
100MHz

—157.038
—132.220
—125.456
—125.420
—125.382
—125.344
—121.186
—111.179
—91.116
—84.839
—77.261
—76.943
—61.959
—55.861

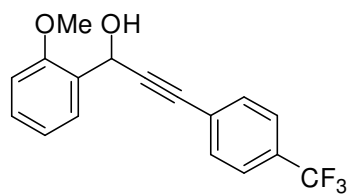
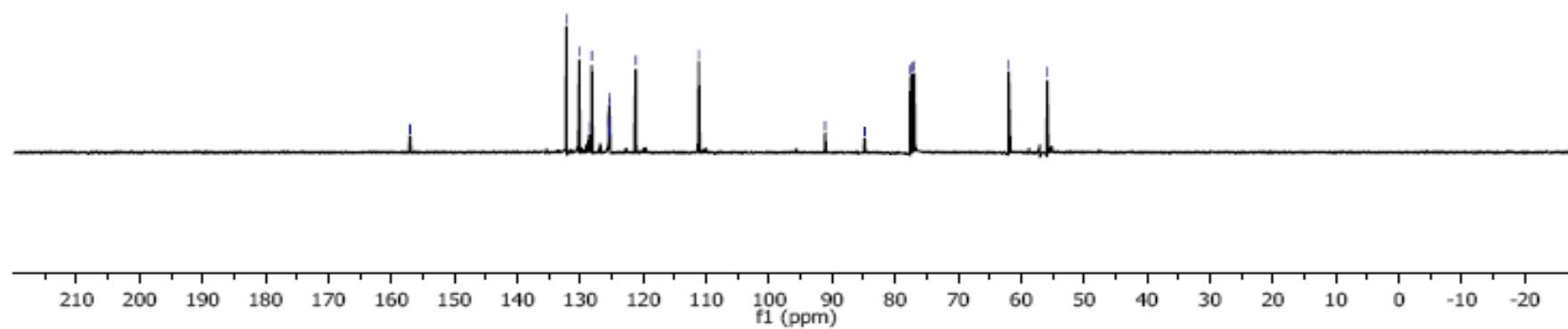


Table 3, entry 3



KW310, CDCl₃
300MHz

7.56
7.53
7.52
7.49

4.40
4.38

2.33

1.81

1.67

1.30

1.25

1.21

1.18

1.14

1.10

1.07

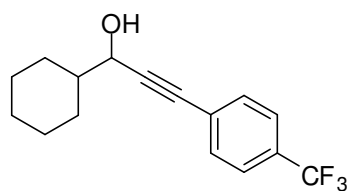
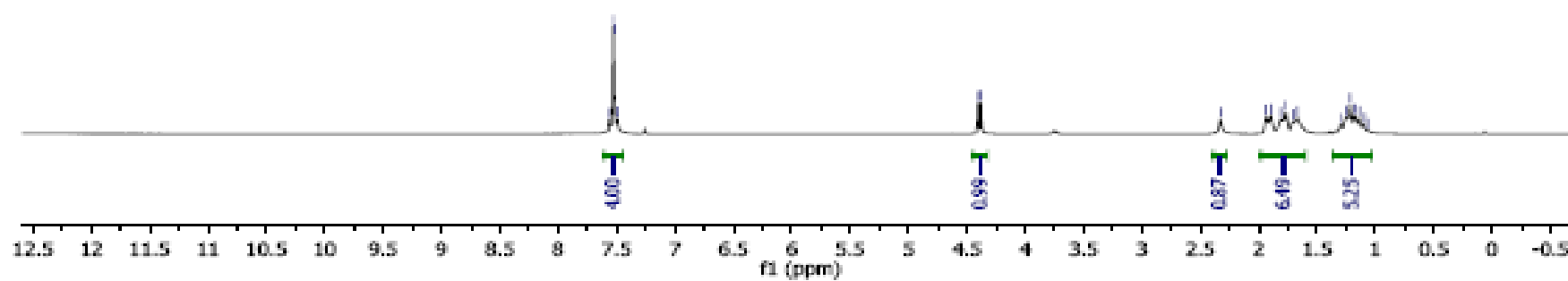


Table 3, entry 4



KW310, CDCl₃
75MHz

132.12
130.90
130.47
130.04
129.60
126.80
125.89
125.42
125.37
122.28

—92.06

—84.52

77.27

76.84

—67.80

—44.44

28.89

28.46

26.56

26.08

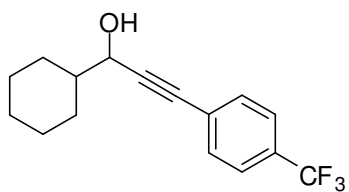
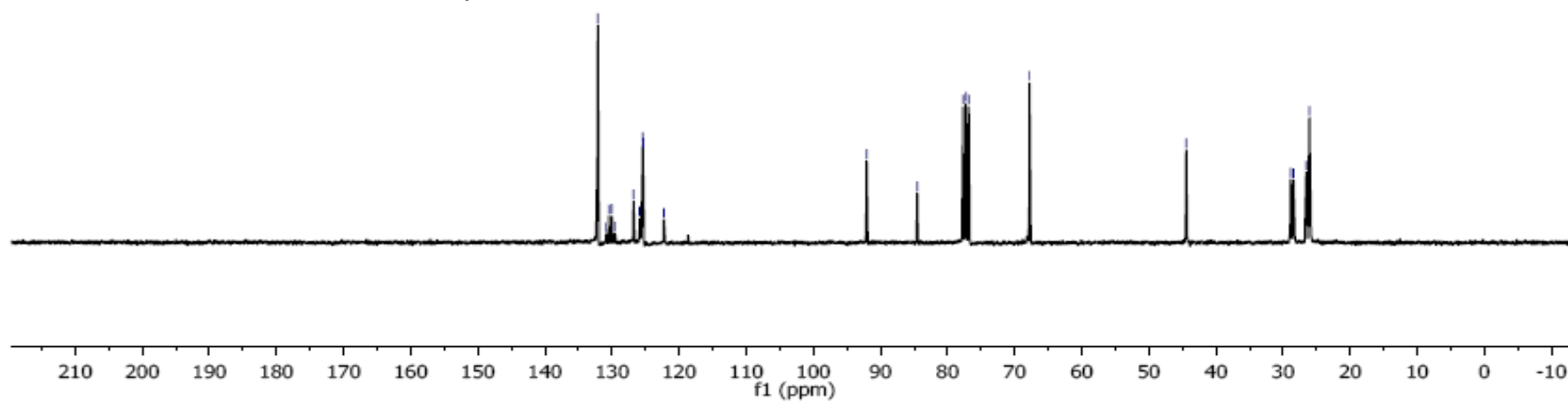
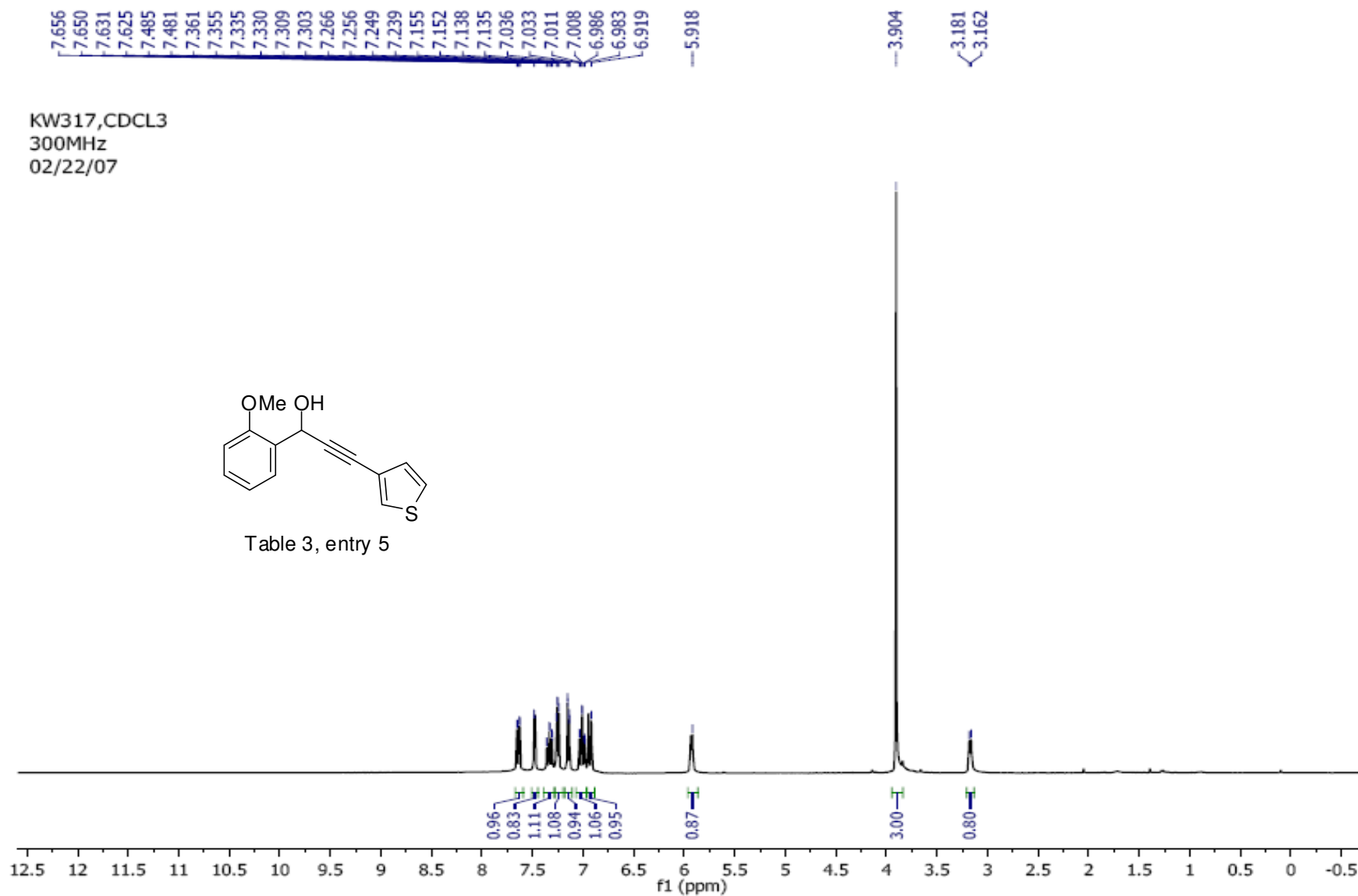


Table 3, entry 4





KW317, CDCL₃
75MHz
02/22/07

157.045
130.201
129.989
129.315
128.962
128.258
125.486
121.998
121.139
111.134
88.218
81.499
77.764
77.340
76.916
61.848
55.892
55.815

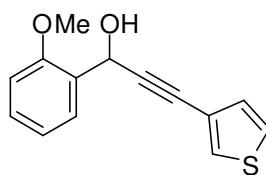
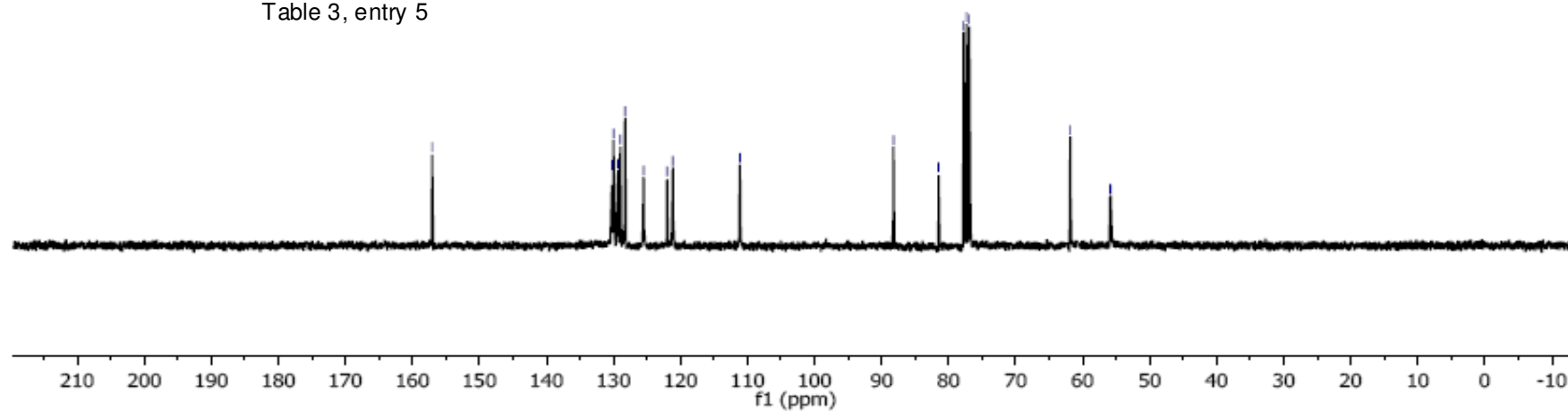


Table 3, entry 5



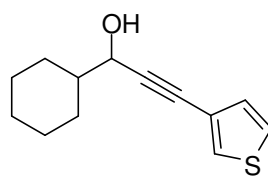
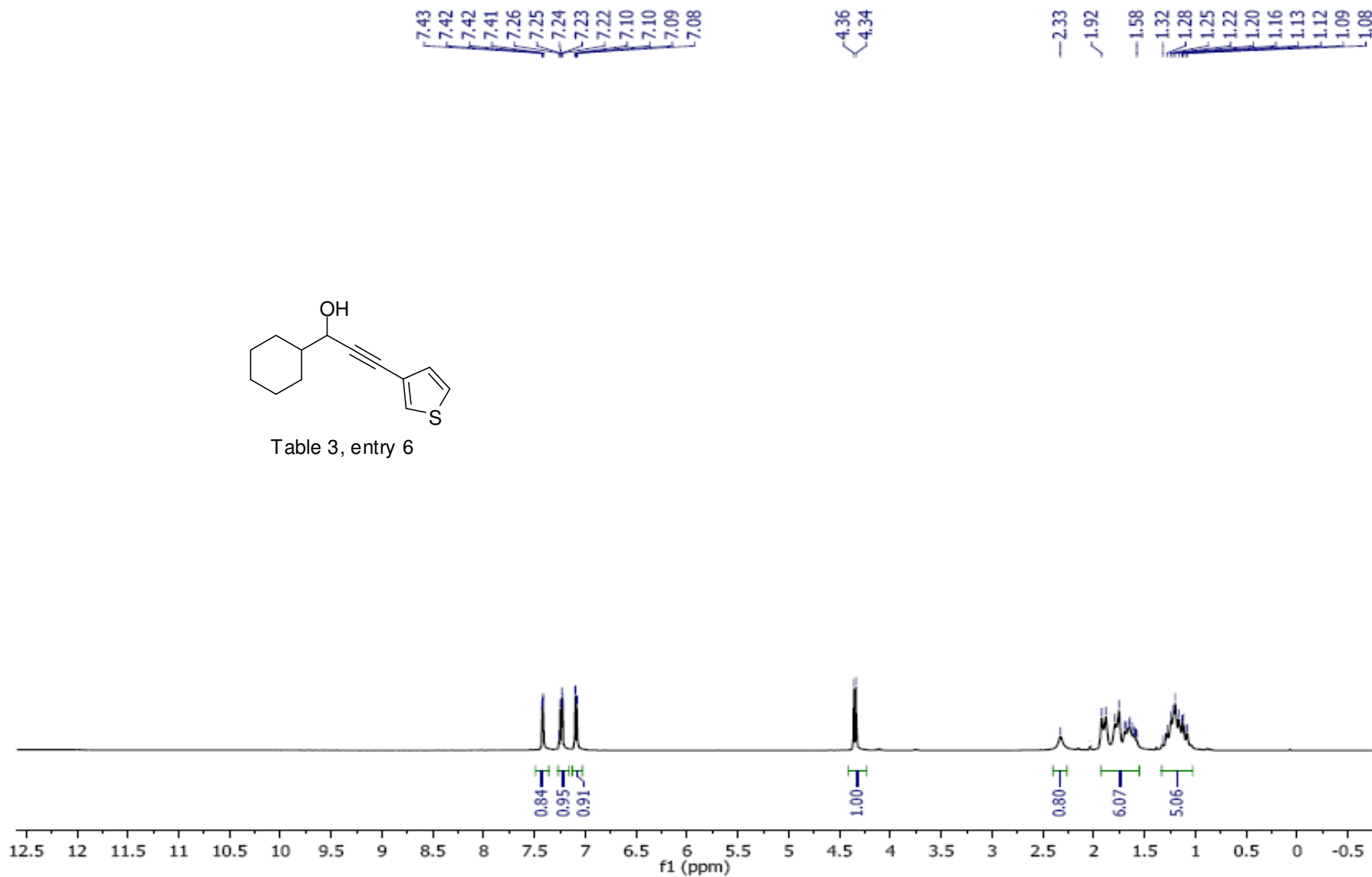


Table 3, entry 6



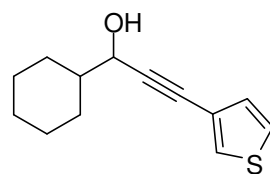
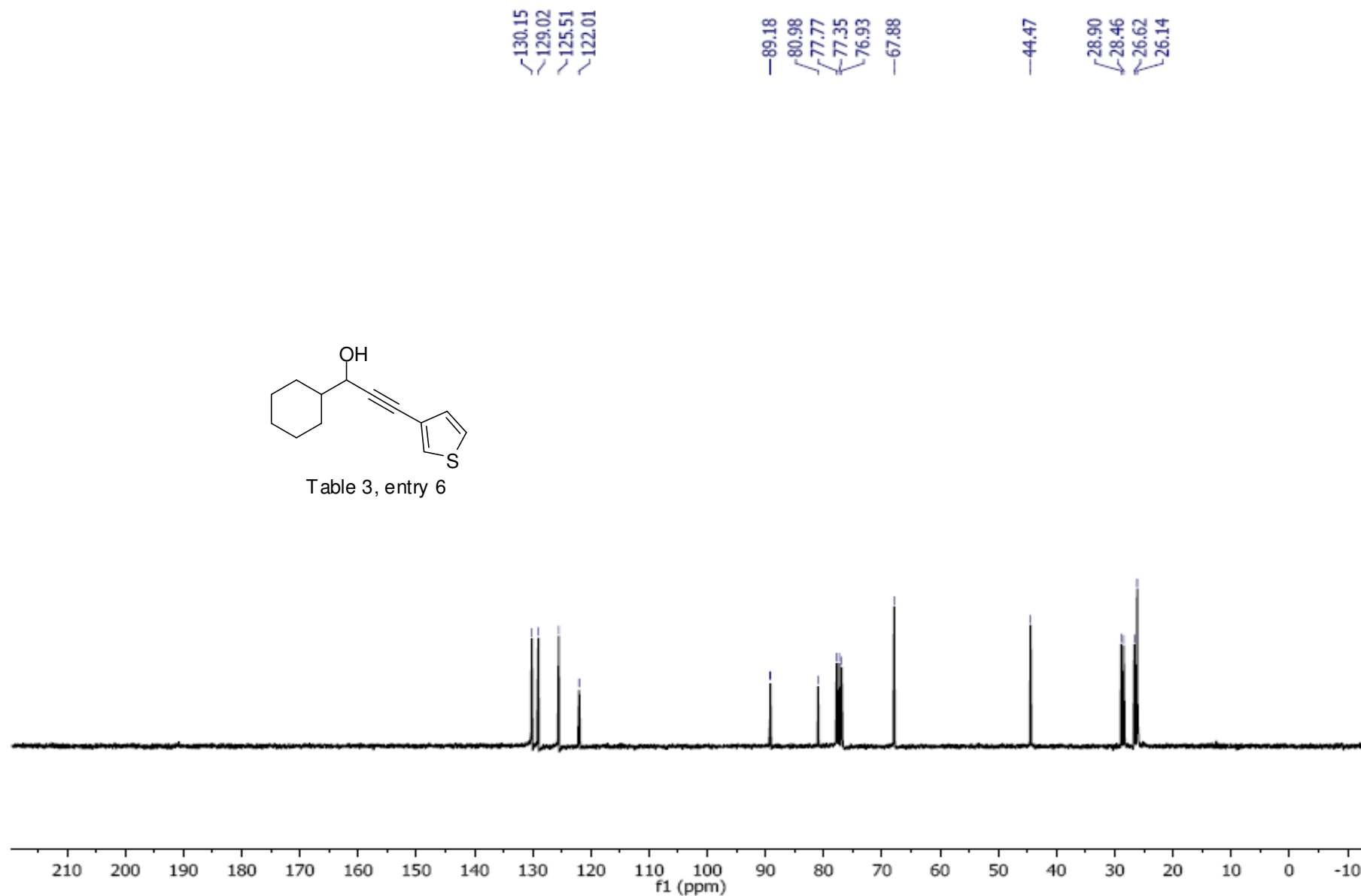


Table 3, entry 6



CVR-3661
CDCL₃, 376MHZ
19F NMR
OILY LIQUID
11 SEP 2009

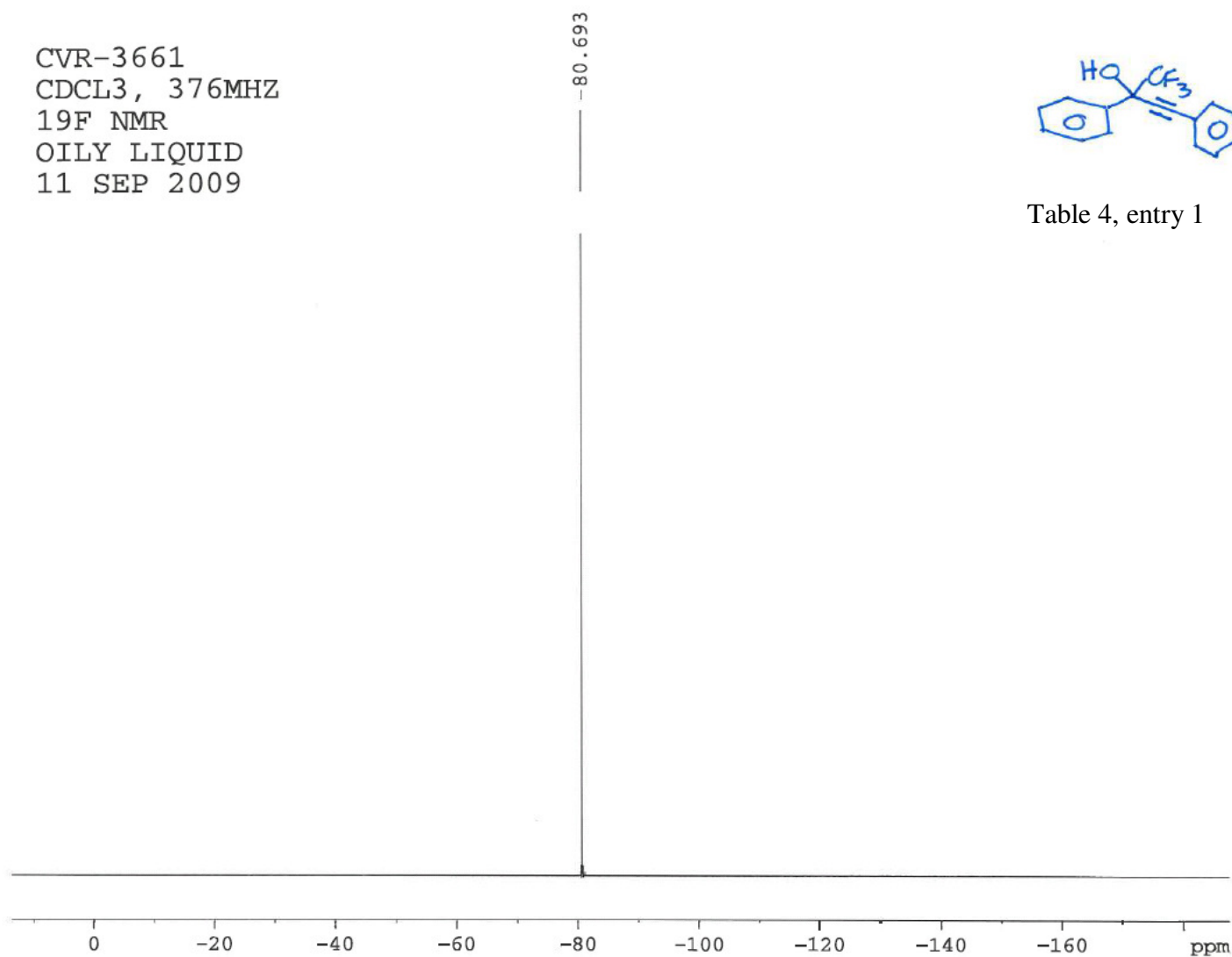


Table 4, entry 1

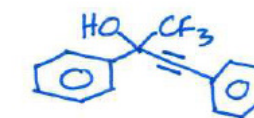


Table 4, entry 1

CVR-3661
PALE YELLOW OIL
CDCL₃, 100MHZ
COL: 5%ETOAC/HEX
10 SEP 2009

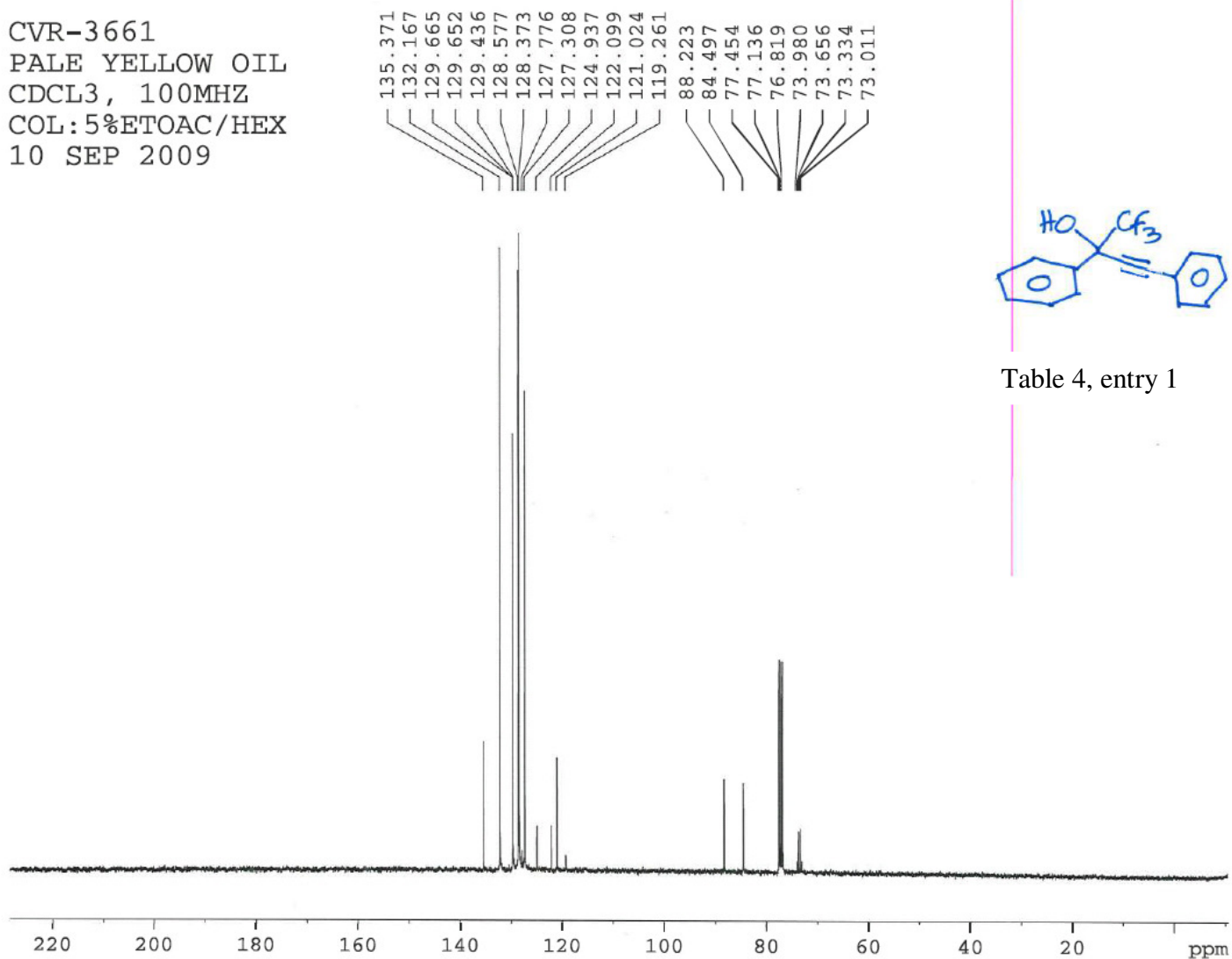


Table 4, entry 1

Manual Peak Matching Report
For Accurate Mass Determination

Theoretical mass	Experimental mass	PFK matching mass	Deviation*
276.07620	276.07667	230.98562	1.7 ppm

* The deviation is obtained from the following equation:

$$\text{deviation} = \frac{\text{experimental mass} - \text{theoretical mass}}{\text{nominal mass}}$$

Where nominal mass takes in account only ^{12}C , ^1H , ^{16}O , ^{14}N etc...

Theoretical mass correspond to the mass of the most abundant isotope peak

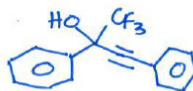


Table 4, entry 1

AK

LIST: fin084755 14-SEP-09 REG : 00:20.7 #9
Samp: CVR-3661 Start : 16:52:39 22
Comm: 70 eV EI
Mode: EI +QIMS LMR UP LR
Oper: Client: Venkat Inlet :
Base: 207.2 Inten : 47335472 Masses: 35 > 650
Norm: 207.2 RIC : 259532488 #peaks: 620
Peak: 1000.00 mmu
Data: + #9= 9>21

11360513					
No.	Mass	Intensity	%RA	%RIC	Flags
1	205.	18760960	39.63	7.23	F#
2	207.	42938368	90.71	16.54	FM#
3	207.	47335424	100.00	18.24	FM#
4	208.	13645312	28.83	5.26	FM#
5	276.	16788992	35.47	6.47	F#

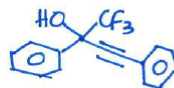


Table 4, entry 1

Date: Mon Sep 14 16:54:18 2009 ICIS: 8.3.0 SP2 for OSF1 (V4.0) build 98-238 from 26-Aug-98

SPEC: fin084755 (14-SEP-09 16:52:39)

Samp: CVR-3661

Comm: 70 eV EI

Oper:

Study:

Base: 207.16

Masses: 35.01 > 650.00

Peak: 1000.0 mmu

Intensity: 47335472

REG #9 @ 0.35 min (EI +Q1MS LMR UP LR) (+9>21)

Scans: 1 > 22

Client: Venkat

#Peaks: 620

RTC: 259532488

4.7E+07

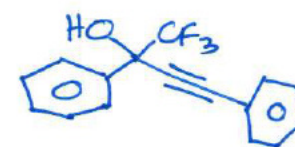
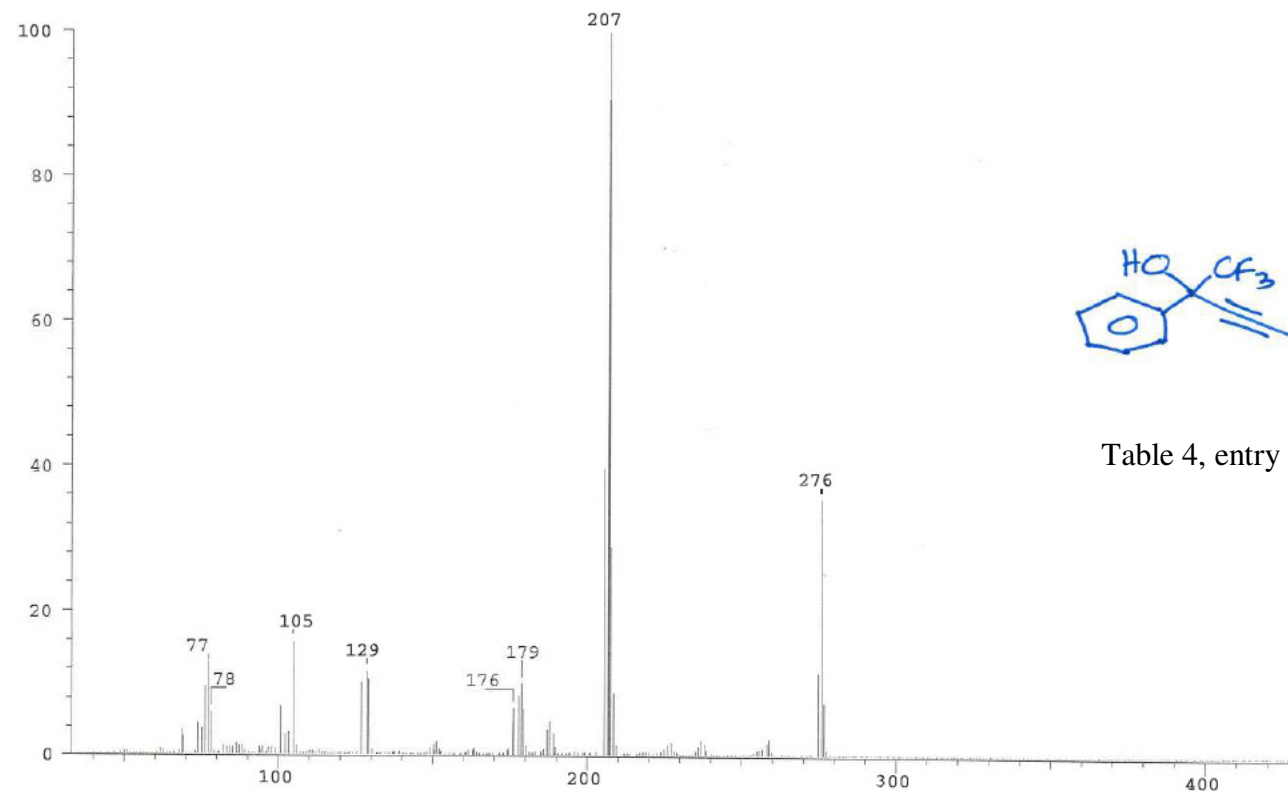


Table 4, entry 1

Date: Mon Sep 14 16:54:16 2009 ICIS: 8.3.0 SP2 for OSF1 (V4.0) build 98-238 from 26-Aug-98

CVR 3702
Colorless Oil
CDCL₃, 400MHz
Col: 10%EtOAc/hex
05 JAN 2009

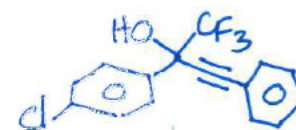
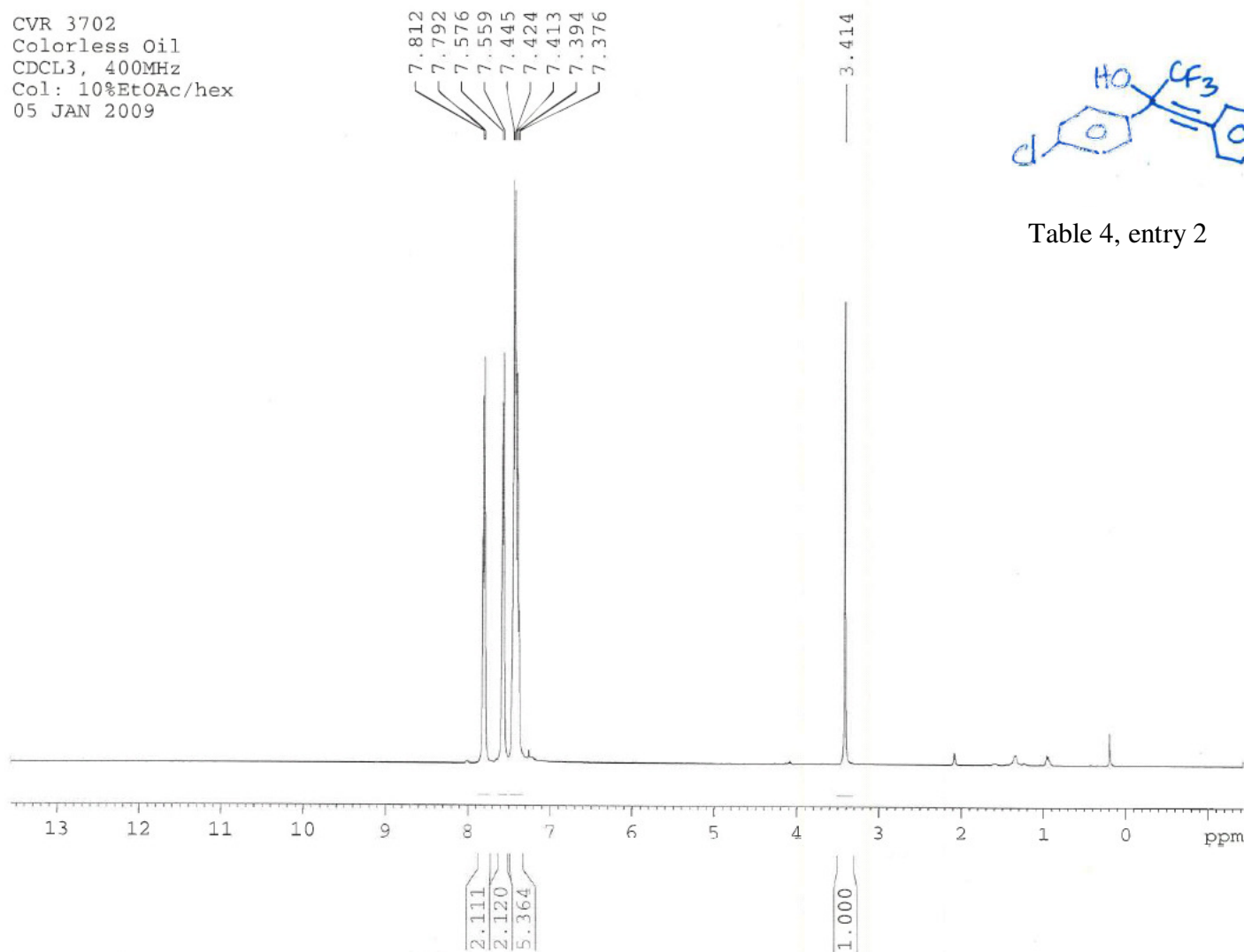
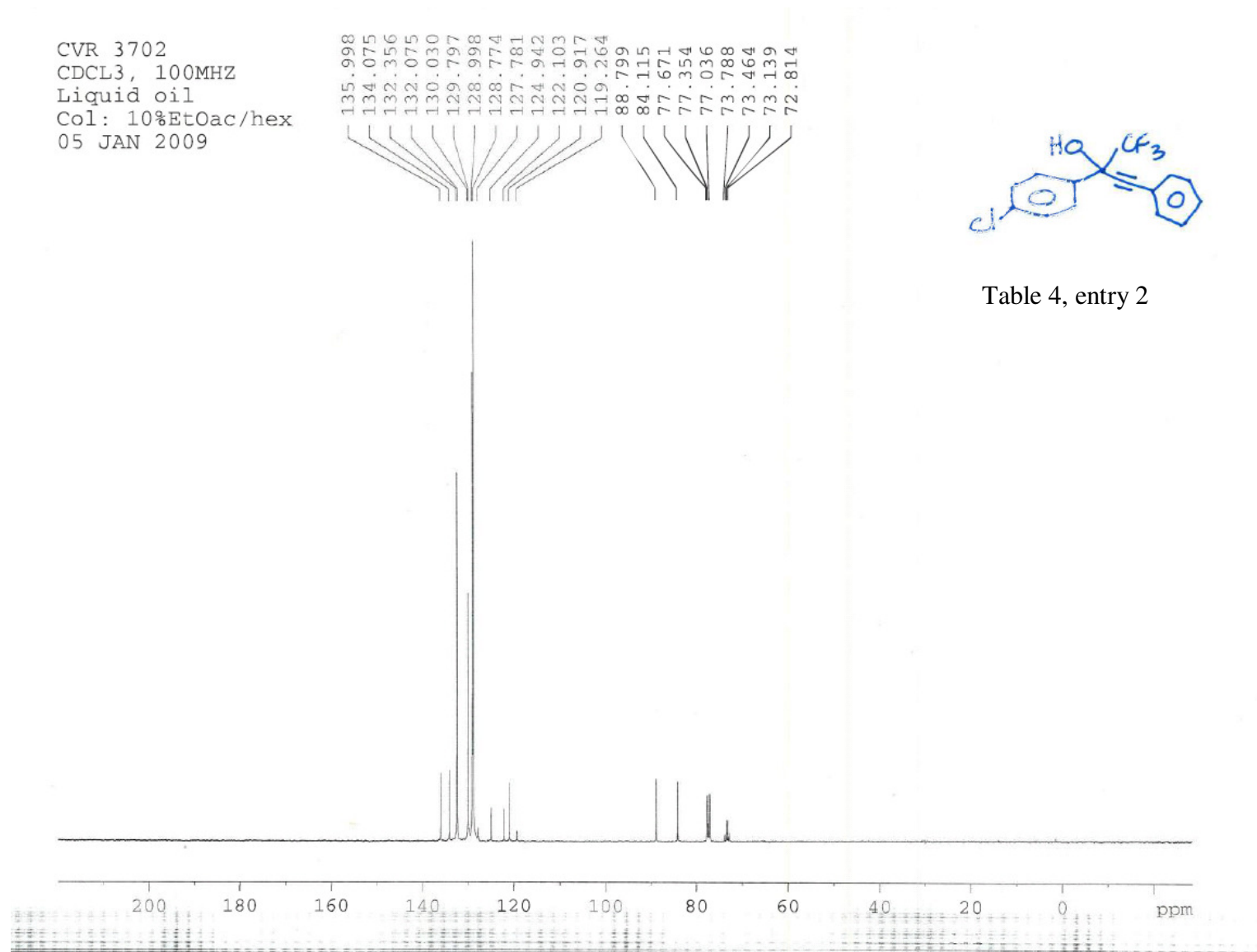


Table 4, entry 2



CVR 3702
CDCL₃
OILY LIQUID
12 JAN 2010

-80.817

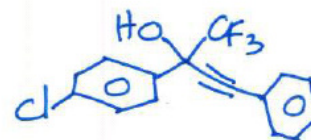
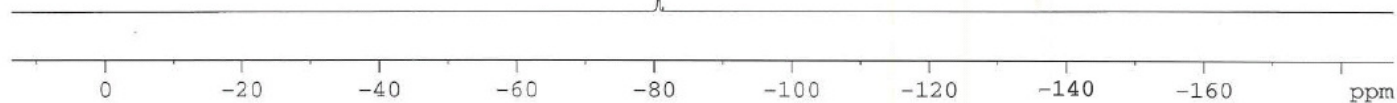


Table 4, entry 2



SPEC: fin10003 (07-JAN-10 12:01:26)
Samp: CVR-3702
Oper: Study :
Base: 239.23 Masses: 50.01 > 650.00
Peak: 1000.0 mmu Intensity: 2180869
REG #9 @ 0.40 min (EI +Q1MS LMR UP LR) (+13>21)

Scans : 1 > 42

Client: Venkat
#Peaks: 631
RIC : 34567122

2.2E+06

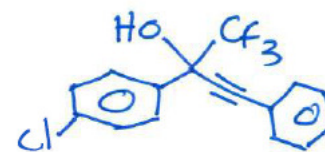
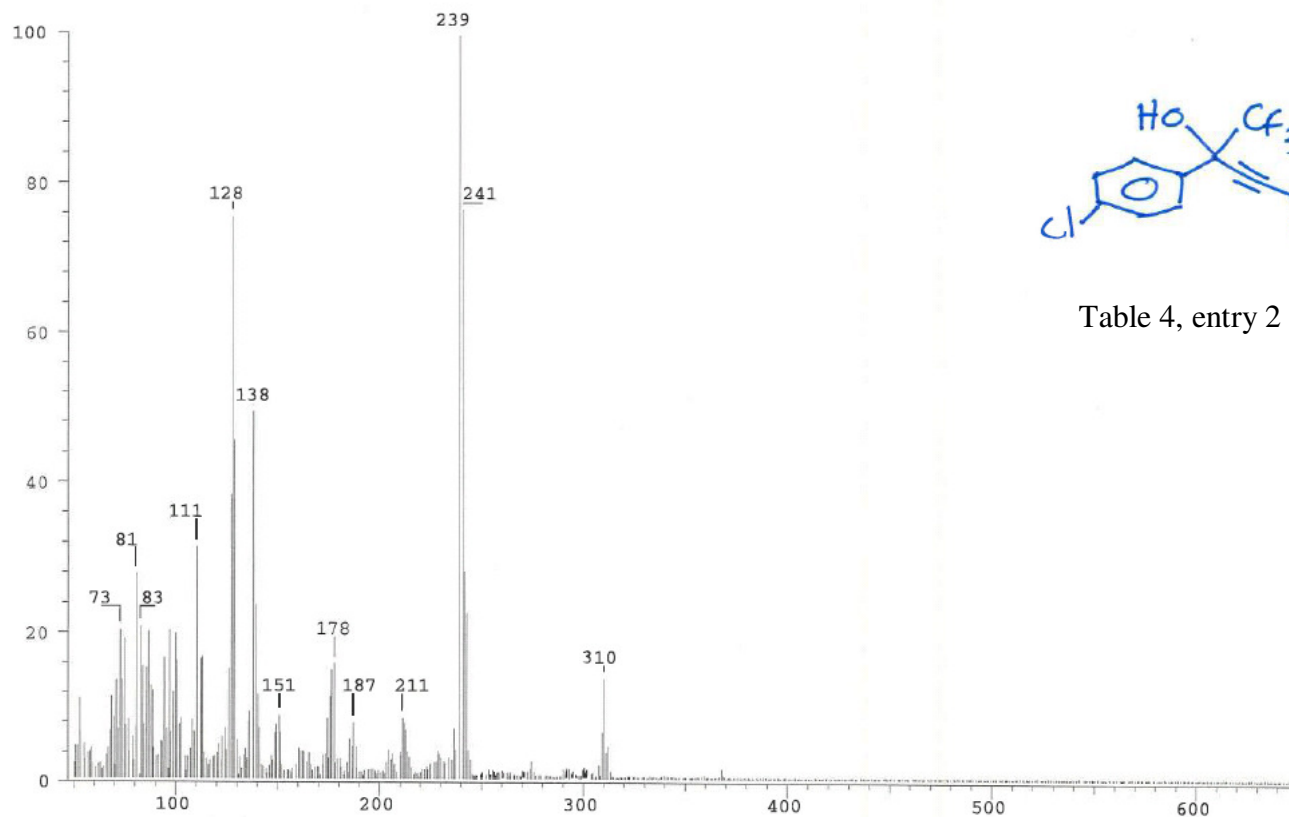


Table 4, entry 2

Date: Thu Jan 7 12:03:58 2010 ICIS: 8.3.0 SP1 for OSF1 (V4.0) build 97-324 from 20-Nov-97

SPEC: fin10003 (07-JAN-10 12:01:26)
Samp: CVR-3702
Oper:
Base: 239.23 Study :
Peak: 1000.0 mmu Masses: 50.01 > 650.00
REG #9 @ 0.40 min (EI +Q1MS LMR UP LR) (+13>21) Intensity: 2180869

Scans : 1 > 42

Client: Venkat

#Peaks: 631

RIC : 34567122

2.2E+06

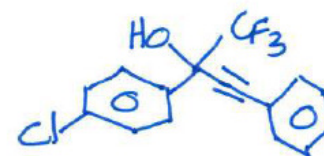
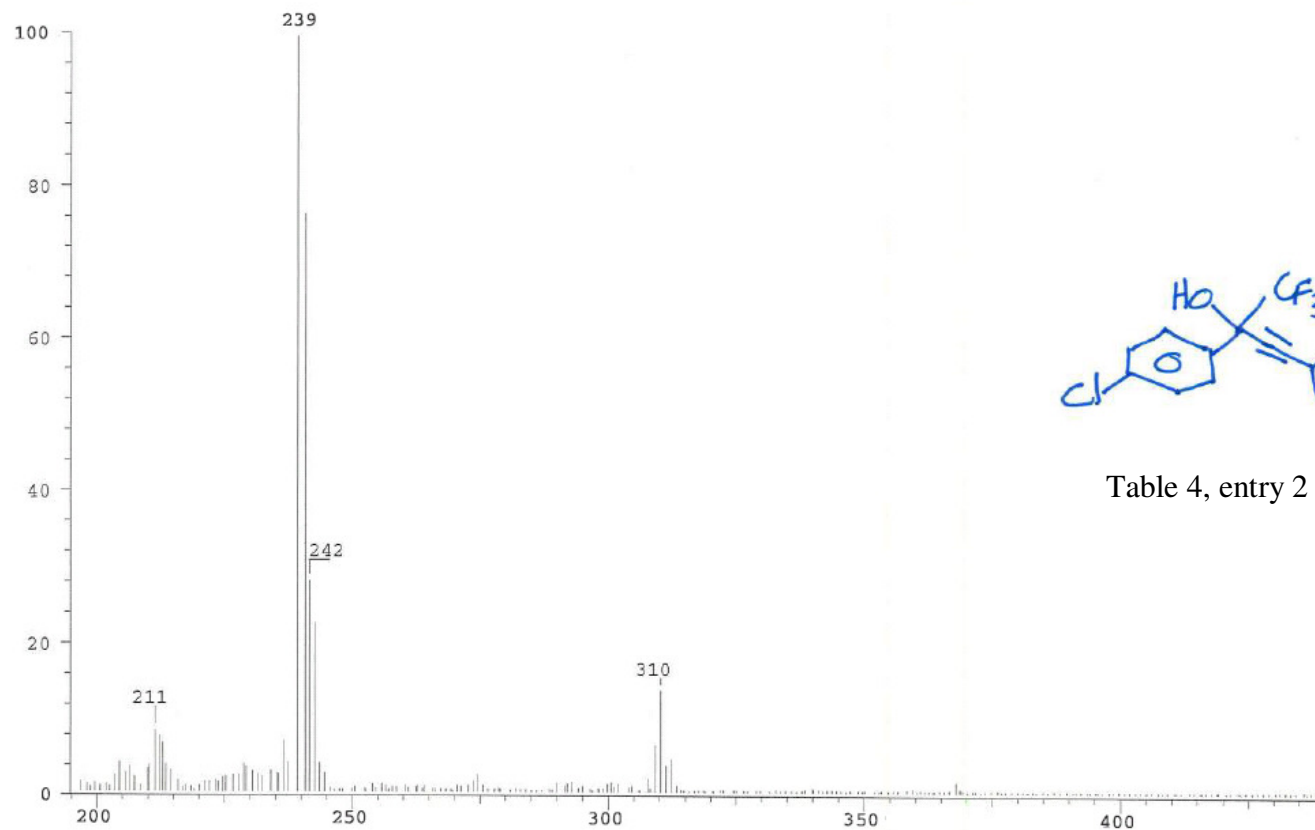


Table 4, entry 2

Date: Thu Jan 7 12:04:07 2010 ICIS: 8.3.0 SP1 for OSF1 (V4.0) build 97-324 from 20-Nov-97

CVR 3701
400MZ, CDCL3
cOL: 10%EtOAc/hex
Colorless oil
04 JAN 2010

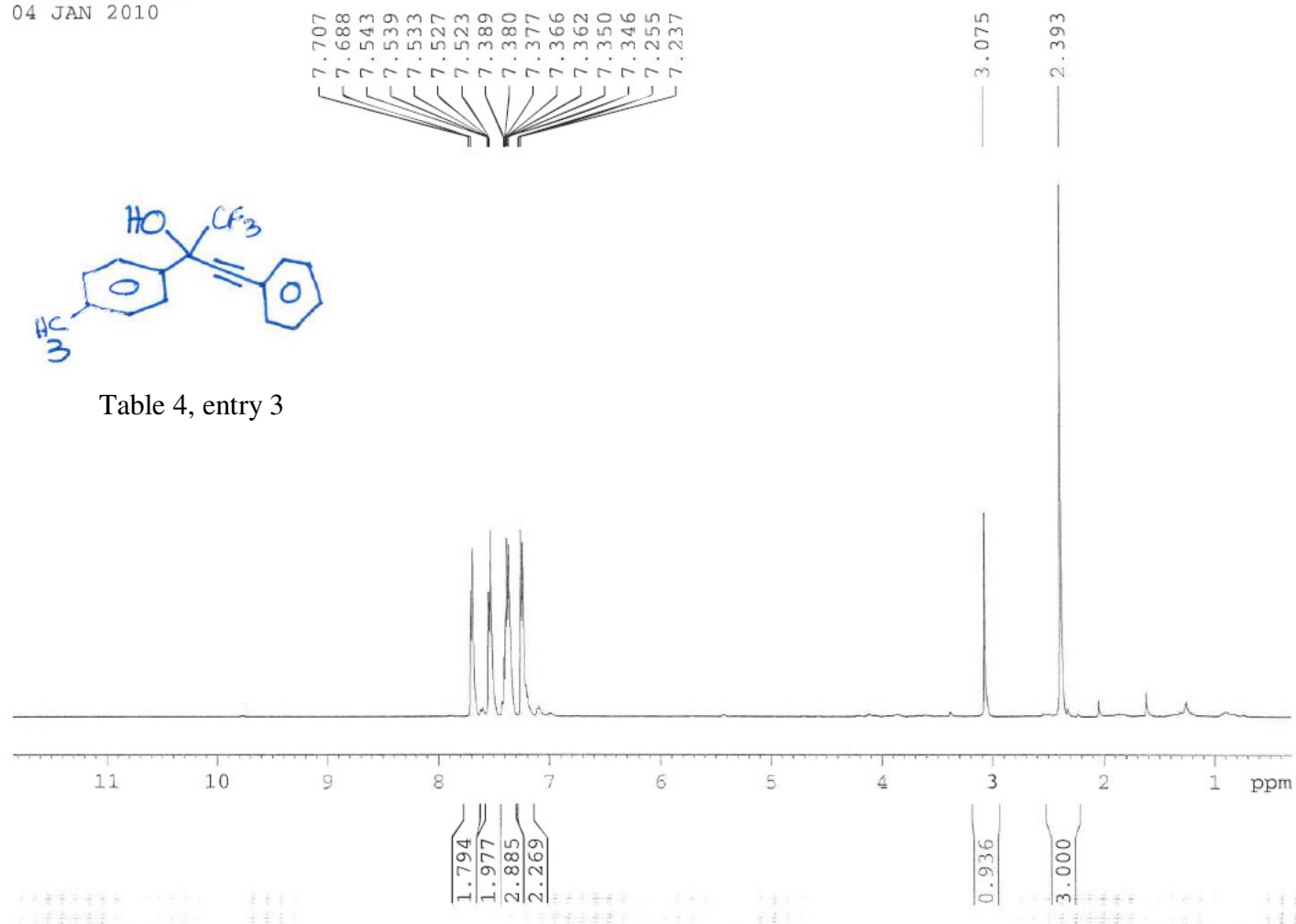


Table 4, entry 3

CVR-3701
LIQUID OIL
Col: 10%EtOAc/hex
CDCL₃, 100MHz
09 JAN 2009

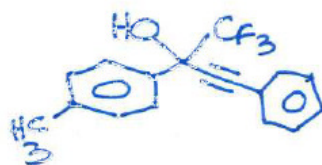
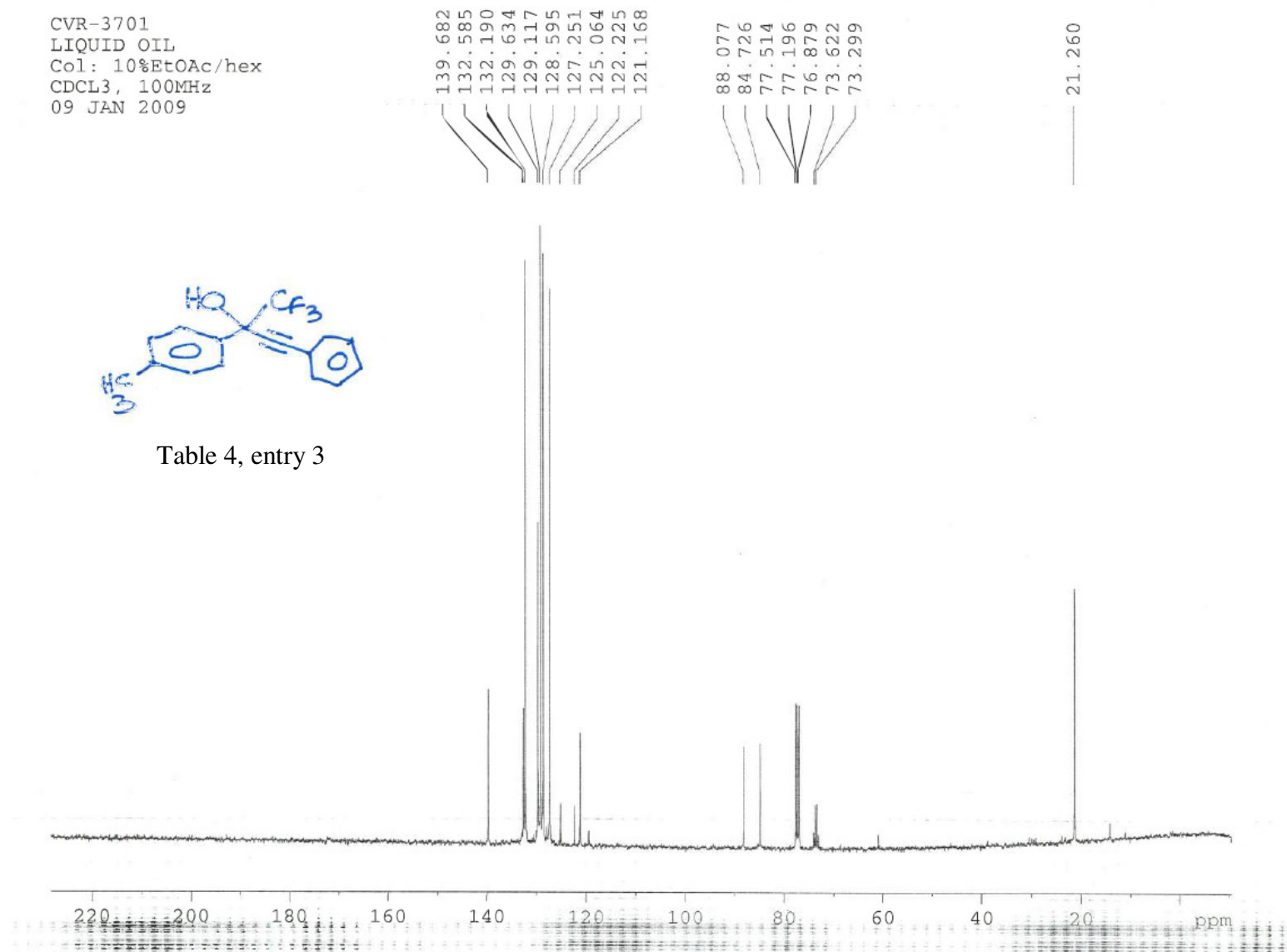


Table 4, entry 3



CVR 3701
19F NMR
CDCL₃
OILY LIQUID
12 JAN 2010

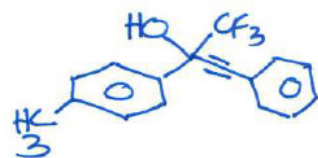
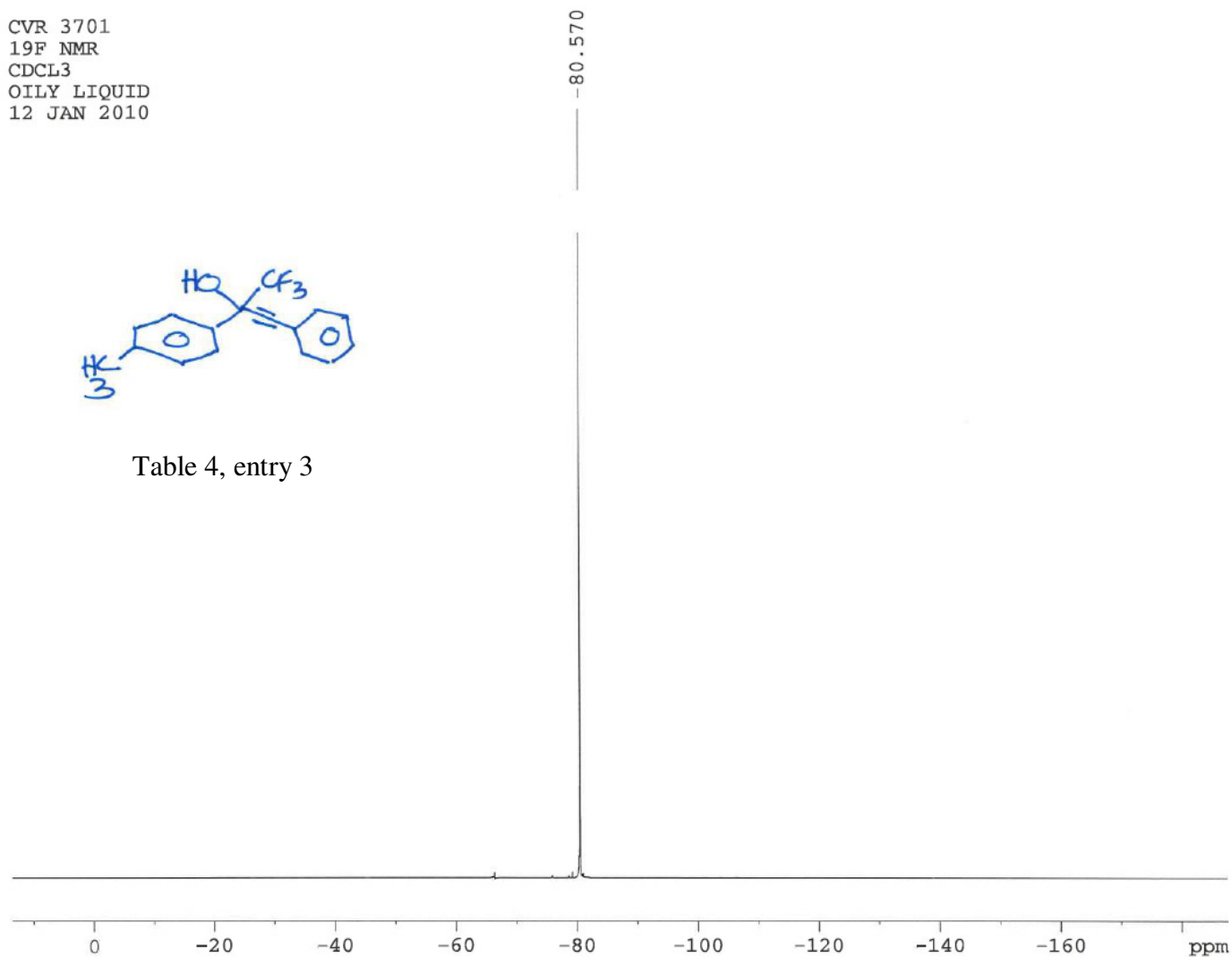


Table 4, entry 3



SPEC: fin10004.dat (08-JAN-10 09:38:08)

Samp: CVR-3701

Oper:

Base: 128.90

Peak: 1000.0 mmu

Study :

Masses: 50.01 > 650.00

Intensity: 16494850

Scans : 1 > 12

Client: Venkat

#Peaks: 615

RIC : 181576523

Scan 3 @ 0.21 min (EI +Q1MS LMR UP LR)

1.6E+07

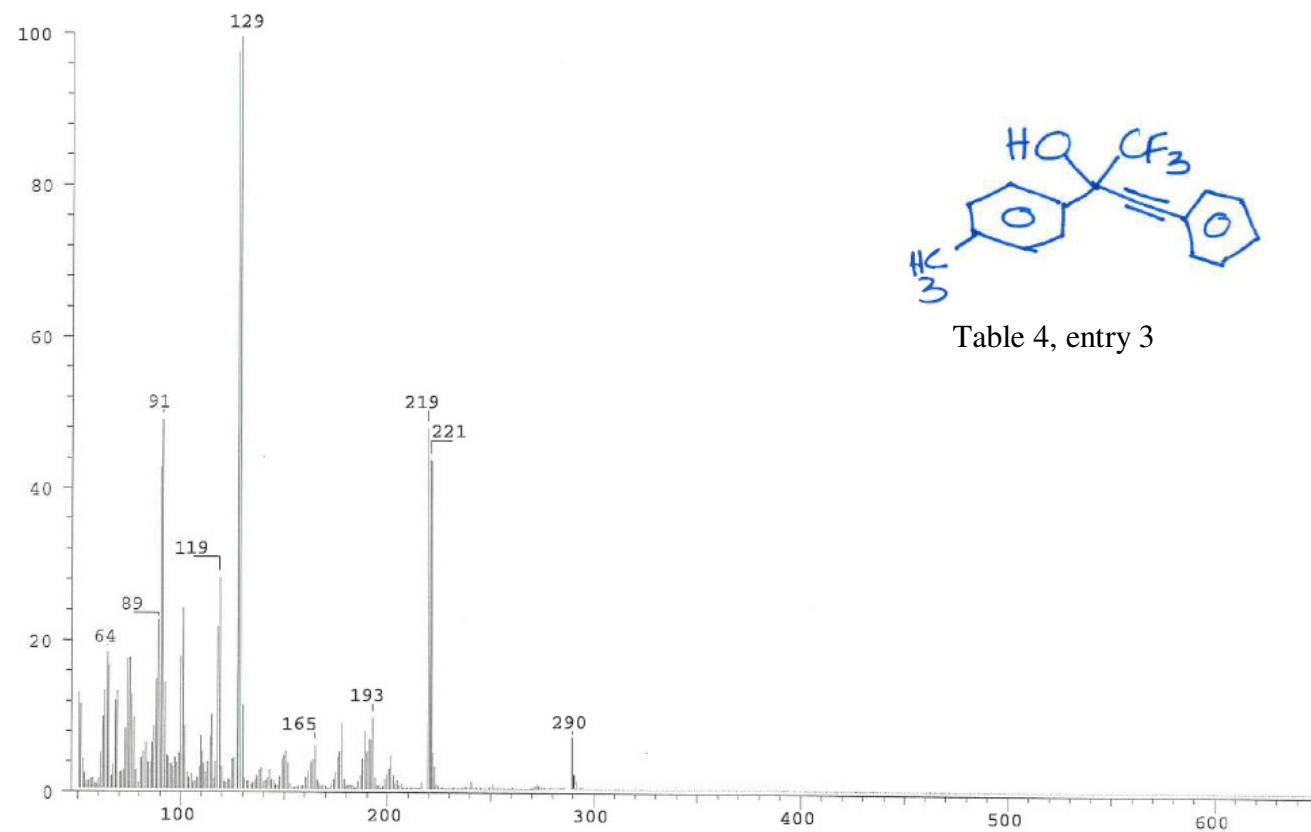


Table 4, entry 3

Date: Fri Jan 8 09:40:02 2010 ICIS: 8.3.0 SP1 for OSF1 (V4.0) build 97-324 from 20-Nov-97

LIST: fin10004 08-JAN-10 Elapse: 00:12.9 3
 Samp: CVR-3701 Start : 09:38:08 12
 Mode: EI +Q1MS LMR UP LR
 Oper: Client: Venkat Inlet :
 Base: 128.9 Inten : 16494850 Masses: 50 > 650
 Norm: 128.9 RIC : 181576523 #peaks: 615
 Peak: 1000.00 mmu

659794					
No.	Mass	Intensity	%RA	%RIC	Flags
1	50.	2105104	12.76	1.16	FM
2	51.	1856859	11.26	1.02	F
3	61.	770019	4.67	0.42	F
4	62.	1574076	9.54	0.87	F
5	63.	2162760	13.11	1.19	F
6	64.	2983203	18.09	1.64	F
7	65.	2702122	16.38	1.49	F
8	68.	1932277	11.71	1.06	F
9	69.	2138042	12.96	1.18	F
10	73.	1309887	7.94	0.72	F
11	74.	2833079	17.18	1.56	F
12	75.	2864436	17.37	1.58	F
13	76.	2065642	12.52	1.14	F
14	77.	1566815	9.50	0.86	F
15	82.	804434	4.88	0.44	FM
16	83.	1007563	6.11	0.55	FM
17	86.	1004412	6.09	0.55	F
18	87.	1369969	8.31	0.75	F
19	88.	2411677	14.62	1.33	F
20	89.	3705893	22.47	2.04	F
21	90.	7033073	42.64	3.87	F
22	91.	8079235	48.98	4.45	F
23	92.	2338223	14.18	1.29	F
24	93.	736684	4.47	0.41	FM
25	94.	690355	4.29	0.38	F
26	97.	682300	4.14	0.38	F
27	99.	777398	4.71	0.43	F
28	100.	2900467	17.58	1.60	F
29	101.	3955192	23.98	2.18	F
30	102.	1368895	8.30	0.75	F
31	110.	1164337	7.06	0.64	FM
32	110.	810583	4.91	0.45	F
33	114.	1158855	7.03	0.64	F
34	115.	1628733	9.87	0.90	F
35	118.	3553300	21.55	1.96	F
36	119.	4636006	28.11	2.55	FM
37	126.	687916	4.17	0.38	F
38	128. S	16159944	97.97	8.90	F
39	129. S	16494850	100.00	9.08	F
40	130. S	1844466	11.18	1.02	F
41	150.	742827	4.50	0.41	F
42	151.	828491	5.02	0.46	F
43	165.	947398	5.74	0.52	F
44	176.	695228	4.21	0.38	F
45	177.	815183	4.94	0.45	F
46	178.	1442261	8.74	0.79	F
47	189.	1273792	7.72	0.70	F
48	190.	820220	4.97	0.45	F
49	191.	1083400	6.57	0.60	F
50	192.	1083838	6.57	0.60	F
51	193.	1556378	9.44	0.86	FM
52	202.	734804	4.45	0.40	FM
53	219.	7915911	47.99	4.36	F
54	221.	7218822	43.76	3.98	F
55	221.	7191125	43.60	3.96	F
56	222.	804339	4.88	0.44	F
57	290.	1135846	6.89	0.63	F

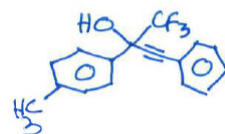
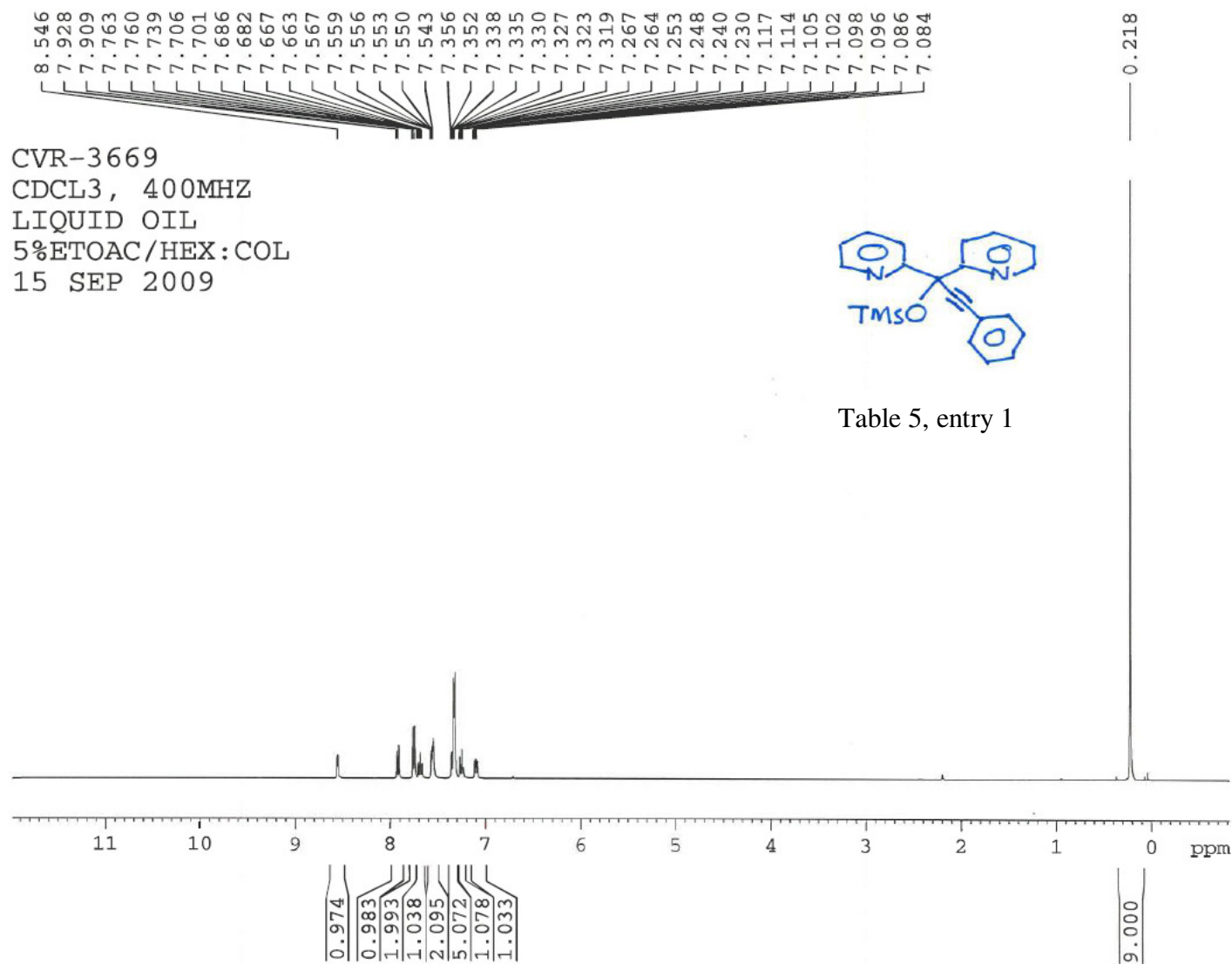


Table 4, entry 3

Date: Fri Jan 8 09:46:51 2010 ICIS: 8.3.0 SP1 for OSF1 (V4.0) build 97-324 from 20-Nov-97



CVR-3669
LIQUID OIL
COL: 5%ETOAC/HEX
100MHZ, CDCL3
15 SEP 2009

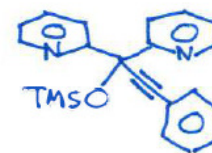
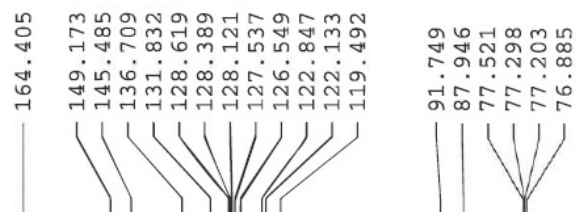
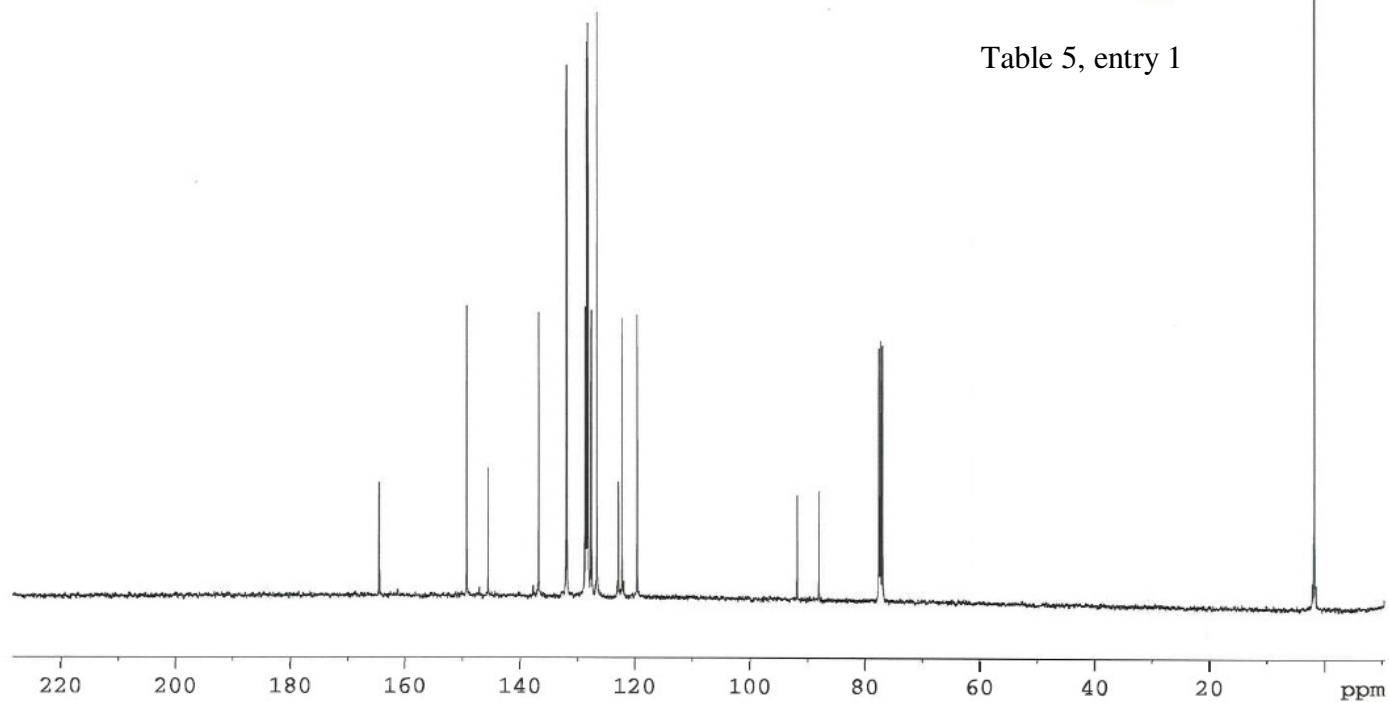


Table 5, entry 1



Manual Peak Matching Report
For Accurate Mass Determination

Theoretical mass	Experimental mass	PFK matching mass	Deviation*
358.15013	358.15108	330.99923	2.6 ppm

* The deviation is obtained from the following equation:

$$\text{deviation} = \frac{\text{experimental mass} - \text{theoretical mass}}{\text{nominal mass}}$$

Where nominal mass takes in account only ^{12}C , ^1H , ^{16}O , ^{14}N etc...

Theoretical mass correspond to the mass of the most abundant isotope peak

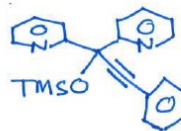


Table 5, entry 1

SPEC: fin084757.dat (15-SEP-09 11:43:17)

Samp: CVR-3669

Comm: 70 eV EI

Oper:

Base: 279.36

Peak: 1000.0 mmu

Scan 5 @ 0.26 min (EI +Q1MS LMR UP LR)

Study:

Masses: 35.01 > 650.00

Intensity: 8204084

Scans: 1 > 12

Client: Venkat

#Peaks: 621

RIC: 114061917

8.2E+06

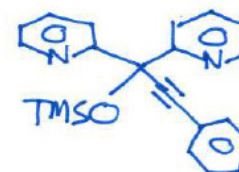
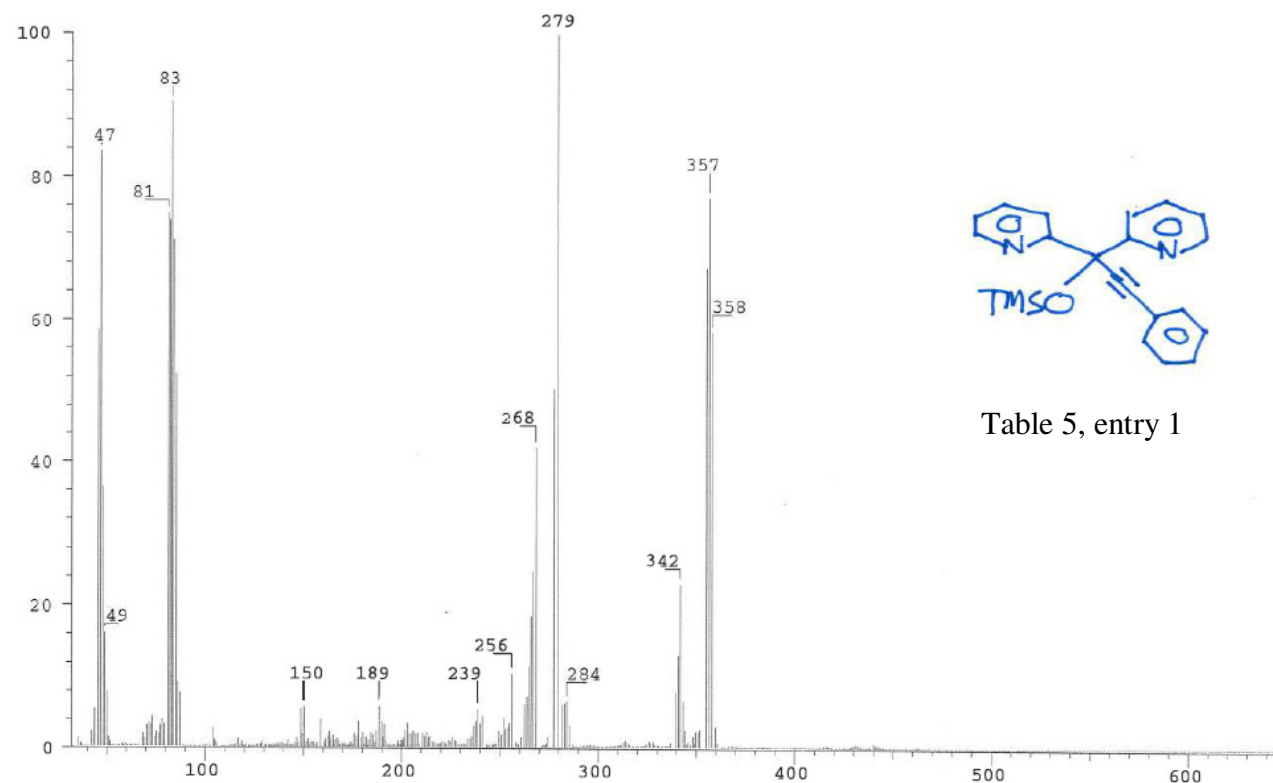


Table 5, entry 1

Date: Tue Sep 15 11:46:01 2009 ICIS: 8.3.0 SP2 for OSF1 (V4.0) build 98-238 from 26-Aug-98

SPEC: fin084757.dat (15-SEP-09 11:43:17)

Samp: CVR-3669

Comm: 70 eV EI

Oper:

Base: 279.36

Peak: 1000.0 mmu

Scan 5 @ 0.26 min (EI +Q1MS LMR UP LR)

Study:

Masses: 35.01 > 650.00

Intensity: 8204084

Scans: 1 > 12

Client: Venkat

#Peaks: 621

RIC: 114061917

8.2E+06

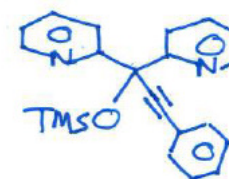
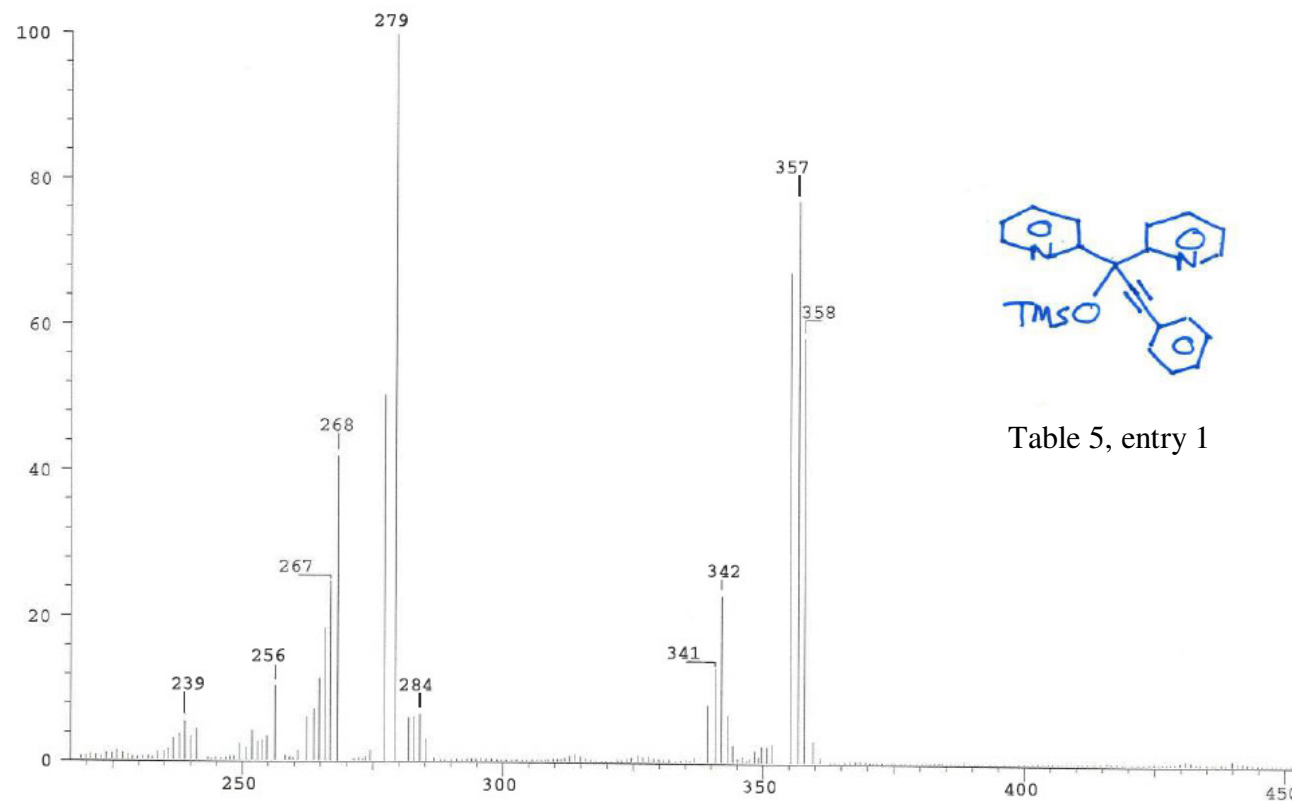
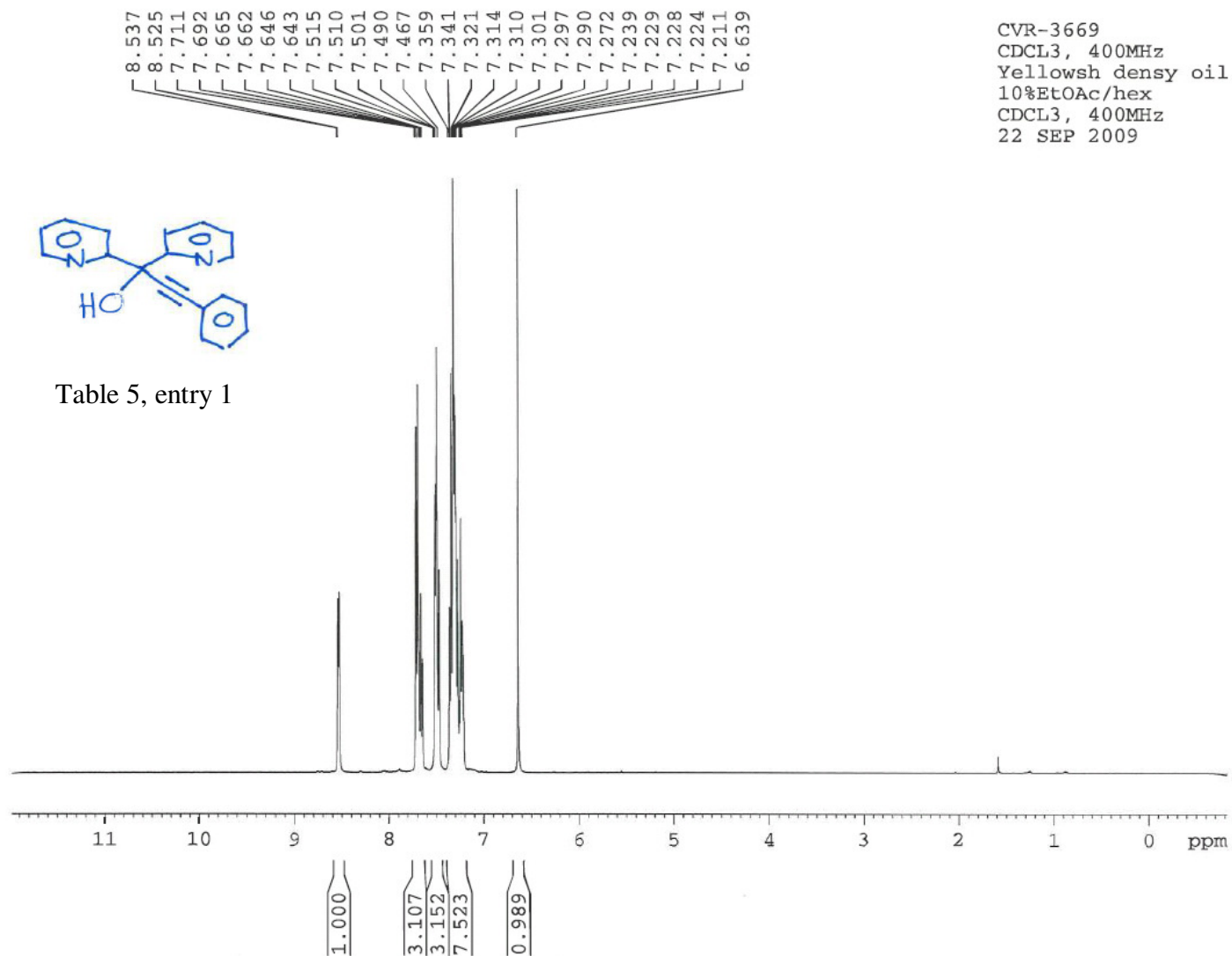


Table 5, entry 1

Date: Tue Sep 15 11:46:04 2009 ICIS: 8.3.0 SP2 for OSF1 (V4.0) build 98-238 from 26-Aug-98



CVR-3669
after H3O+
Oily liquid
10%EtOAc/hex
100MHz, CDCl3
22 SEP 2009

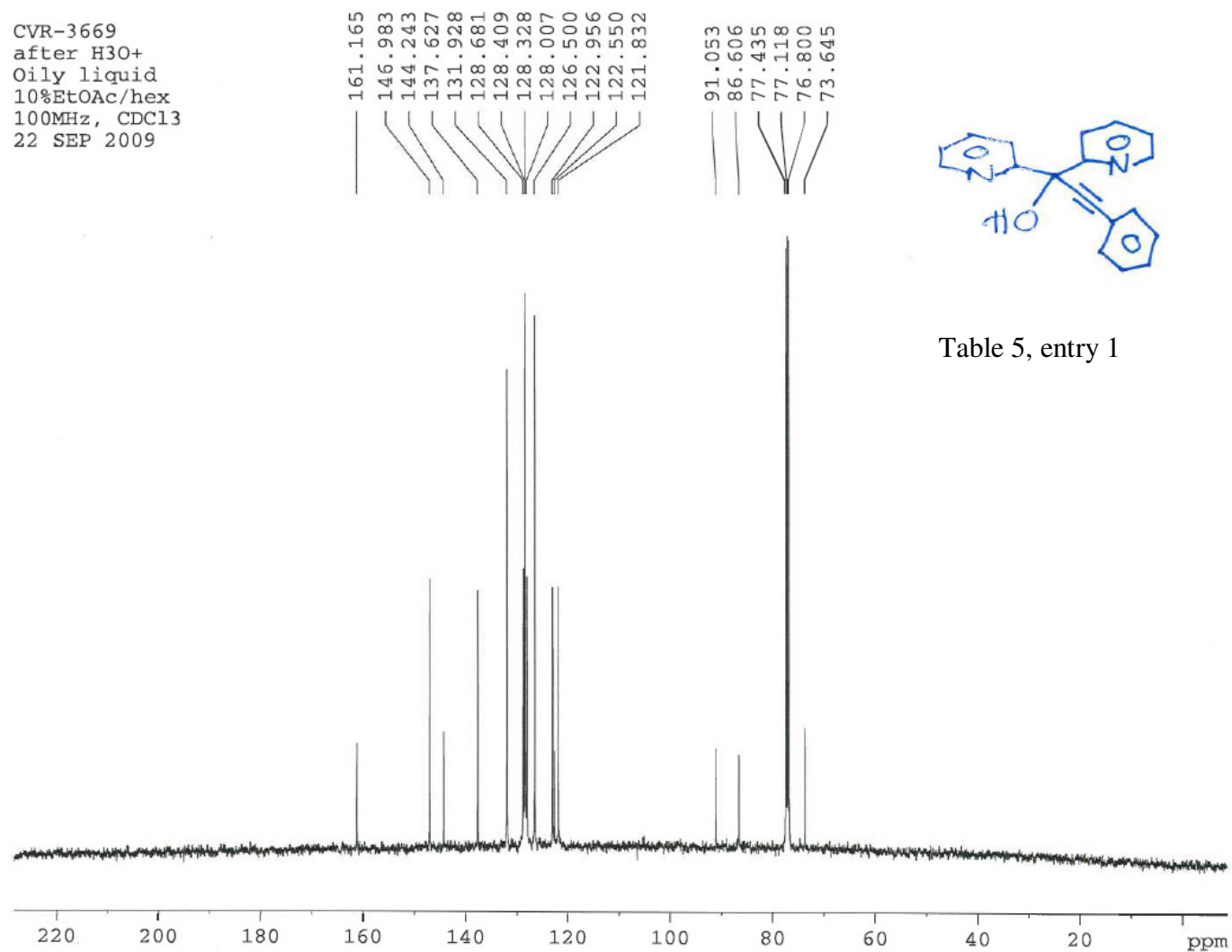


Table 5, entry 1

CVR-3690
CDCL₃, 400MHZ
LIQUID OIL
COL: HEXANES
07 OCT 2009

7.685
7.665
7.582
7.577
7.566
7.494
7.491
7.473
7.400
7.395
7.389
7.369
7.367
7.350
7.305
7.289

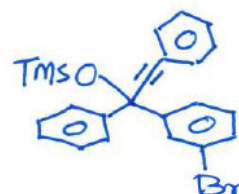
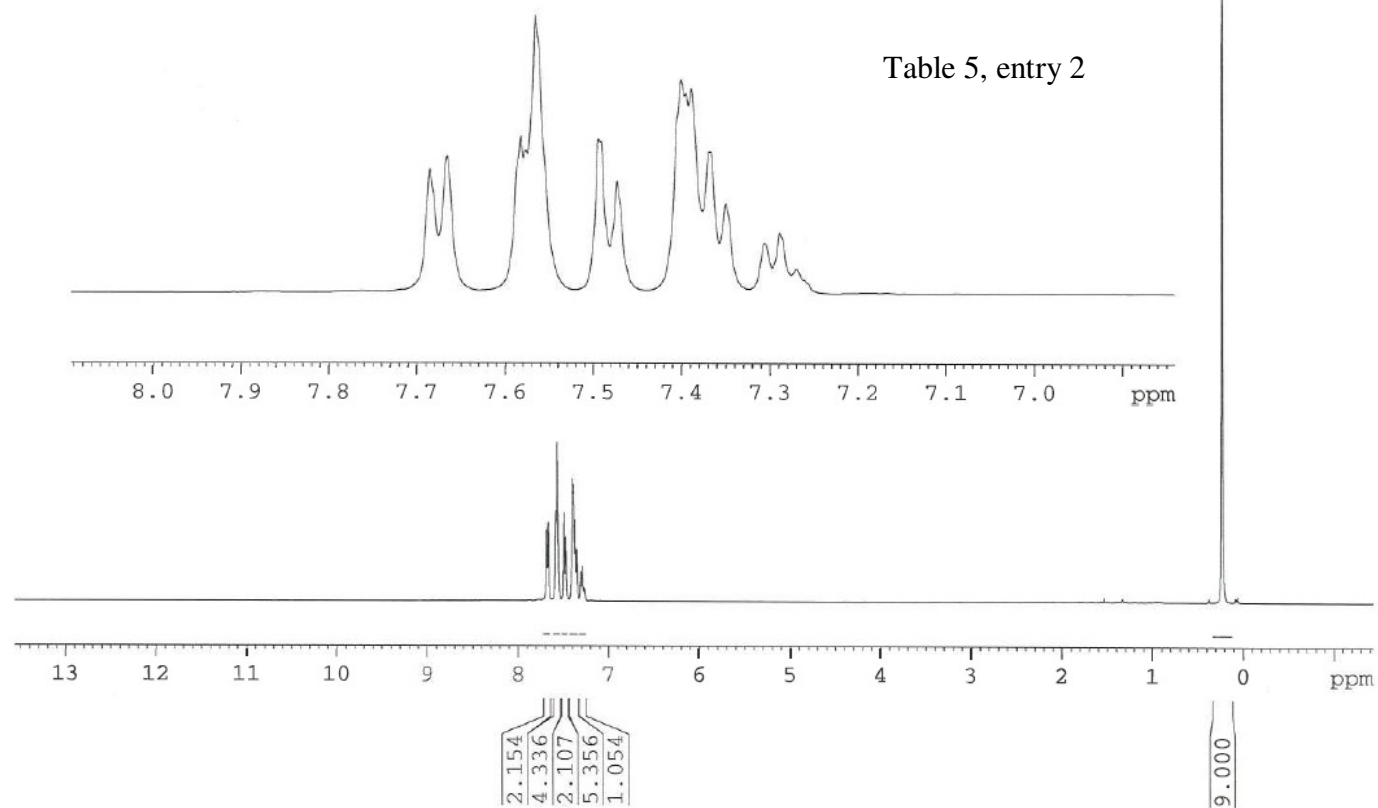


Table 5, entry 2



CVR-3690
CDCL₃, 100MHZ
LIQUID OIL
COL:HEXANES
07 OCT 2009

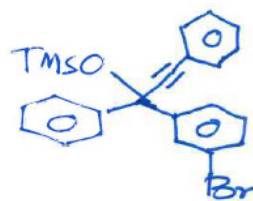
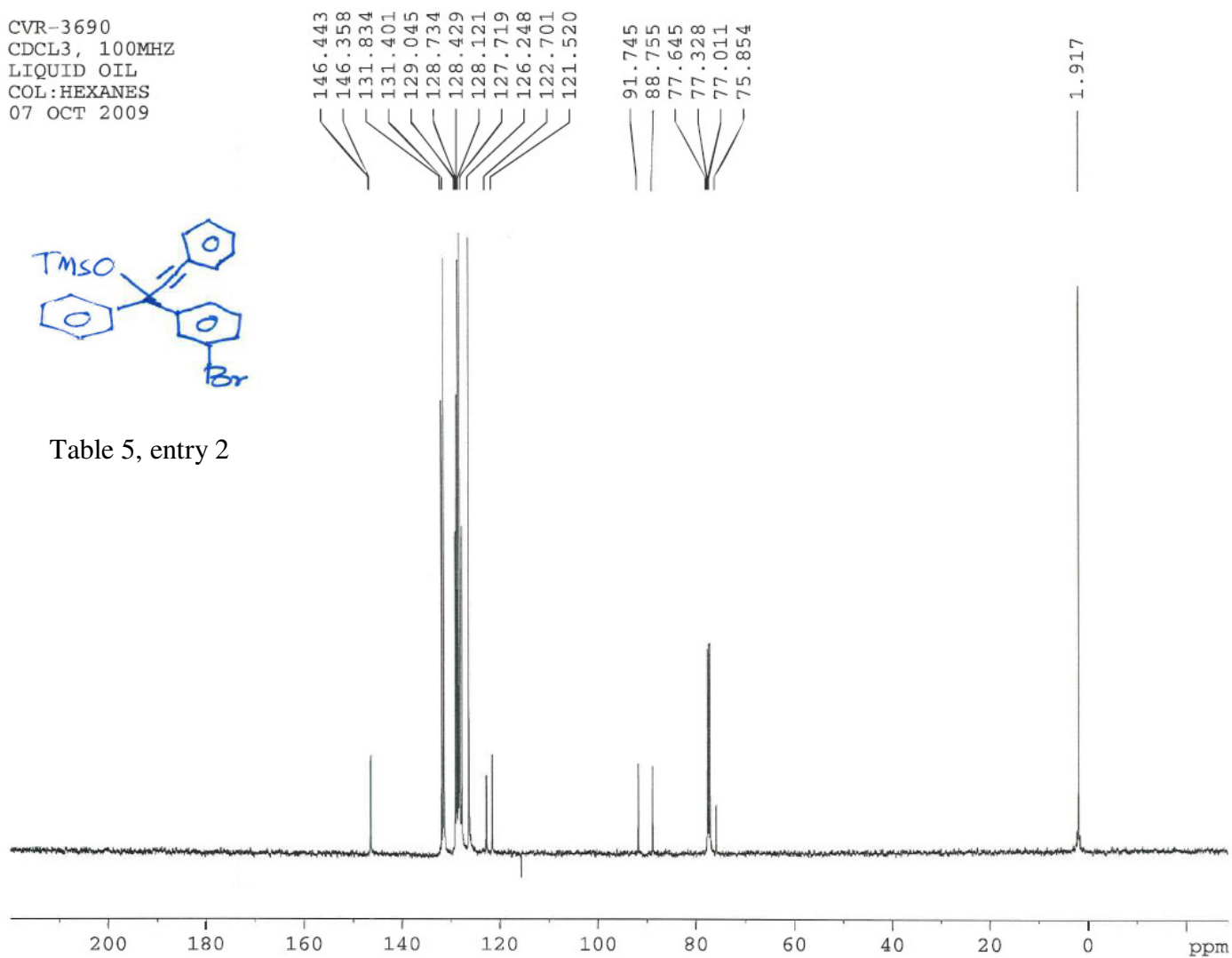


Table 5, entry 2



CVR-3690
CDCL₃, 400MHZ
LIQUID OIL
COL: HEXANES
07 OCT 2009

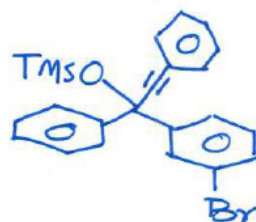
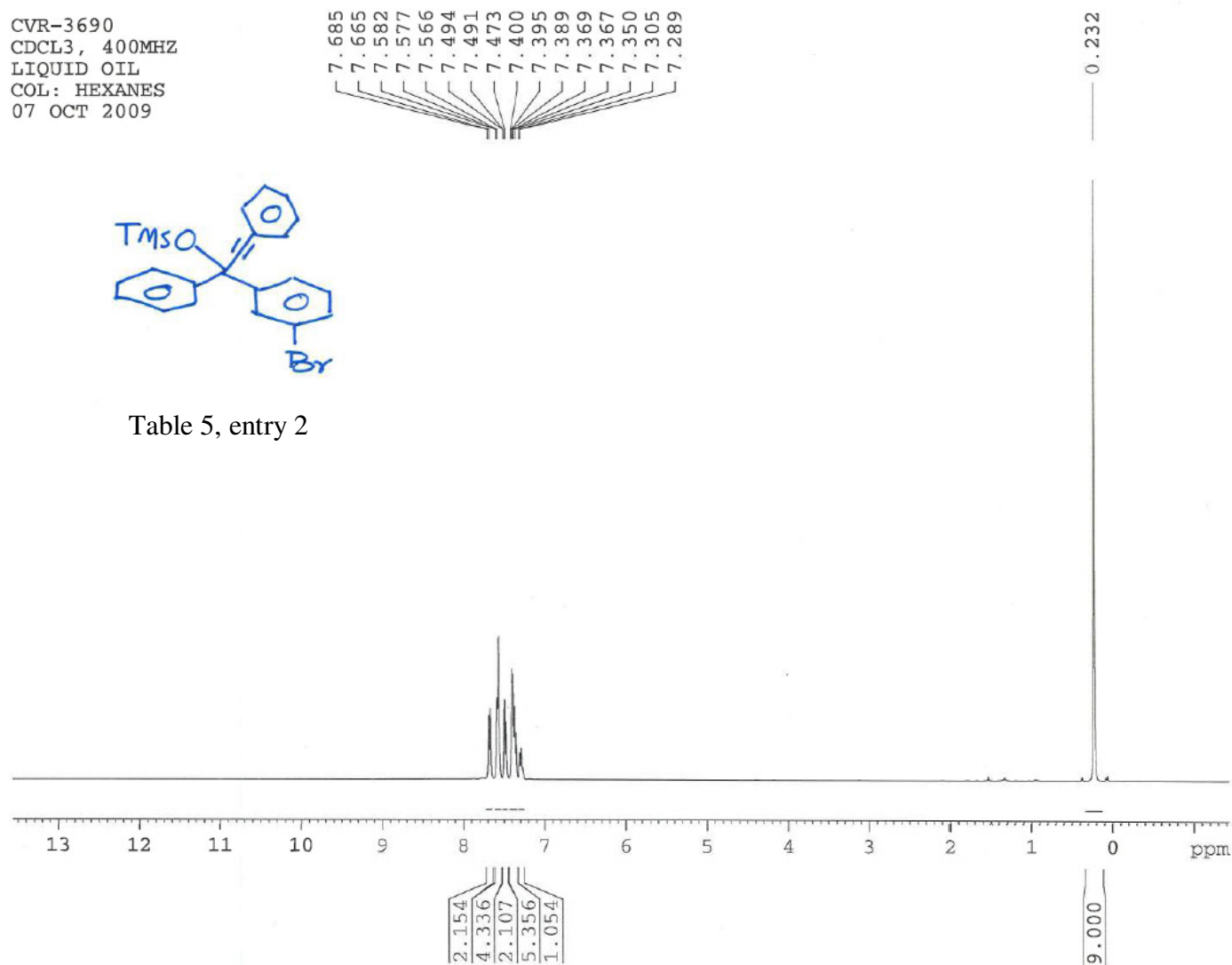


Table 5, entry 2



Manual Peak Matching Report
For Accurate Mass Determination

Theoretical mass	Experimental mass	PFK matching mass	Deviation*
434.07014	434.07124	430.99284	2.5 ppm

* The deviation is obtained from the following equation:

$$\text{deviation} = \frac{\text{experimental mass} - \text{theoretical mass}}{\text{nominal mass}}$$

Where nominal mass takes in account only ^{12}C , ^1H , ^{16}O , ^{14}N etc...

Theoretical mass correspond to the mass of the most abundant isotope peak

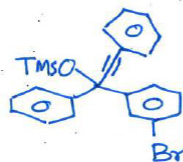


Table 5, entry 2

SPEC: fin084809 (12-OCT-09 16:21:43)

Samp: CVR-3690

Comm: 70 eV EI

Oper:

Study :

Base: 433.90

Masses: 50.01 > 650.00

Peak: 1000.0 mmu

Intensity: 3964275

REG #9 @ 0.68 min (EI +Q1MS LMR UP LR) (+25>32)

Scans : 1 > 35

Client: Venkat

#Peaks: 605

RIC : 29917173

4.0E+06

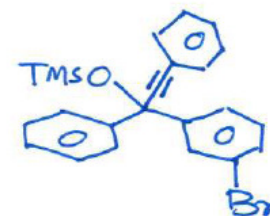
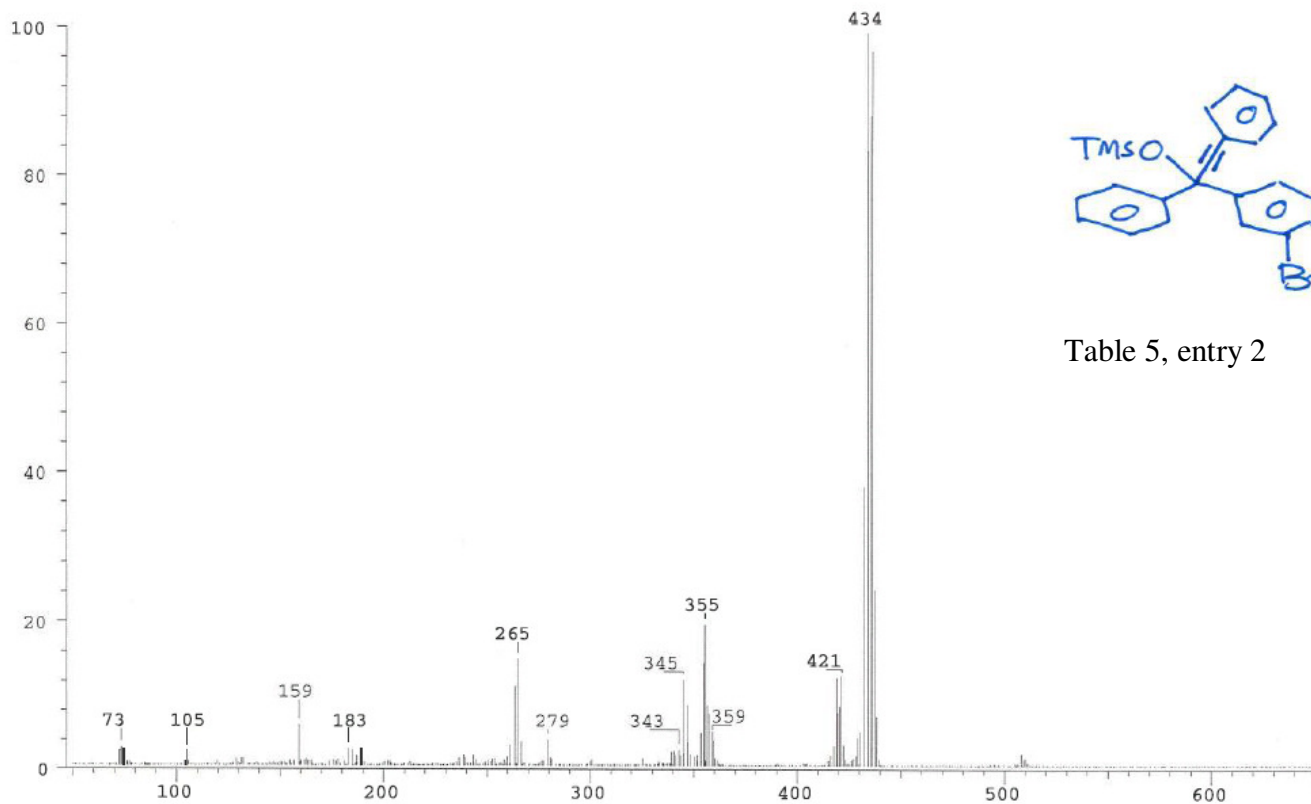


Table 5, entry 2

Date: Mon Oct 12 16:23:15 2009 ICIS: 8.3.0 SP1 for OSF1 (V4.0) build 97-324 from 20-Nov-97

LIST: fin084809 12-OCT-09 Elapse: 00:45.5 29
Samp: CVR-3690 Start : 16:21:43 35
Comm: 70 eV EI
Mode: EI +Q1MS LMR UP LR
Oper: Client: Venkat
Base: 434.2 Inten : 186980 Inlet :
Norm: 434.2 RIC : 2863564 Masses: 50 > 650
Peak: 1000.00 mmu #peaks: 622

35526					
No.	Mass	Intensity	%RA	%RIC	Flags
1	159.	71936	38.47	2.51	F
2	263.	39630	21.19	1.38	F
3	263.	64005	34.23	2.24	F
4	265.	71952	38.48	2.51	F
5	265.	54336	29.06	1.90	F
6	344.	45827	24.51	1.60	F
7	345.	39557	21.15	1.38	F
8	346.	38918	20.81	1.36	F
9	354.	98594	52.73	3.44	F
10	355.	113532	60.72	3.96	FM
11	356.	52487	28.07	1.83	F
12	431.	38575	20.63	1.35	FM
13	432.	65920	35.26	2.30	F
14	433.	131132	70.13	4.58	F
15	434.	186980	100.00	6.53	F
16	435.	174904	93.54	6.11	F
17	436.	145600	77.87	5.08	F
18	437.	37572	20.09	1.31	FM

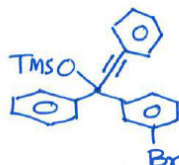


Table 5, entry 2

CVR-3719
LIQUID OIL
CDCL₃, 400MHZ
10%ETOAC/HEX
02 MAR 2010

7.449
7.441
7.435
7.323
7.315
7.309
6.785
6.776
6.752
6.700
6.693
6.678
6.671

3.775
3.756
2.862
2.844
2.827
2.821
2.010
1.992
1.975
1.968
1.601

0.260
0.132

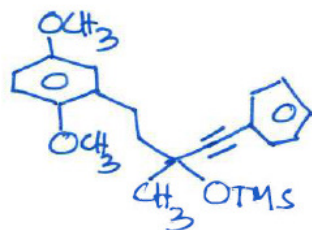
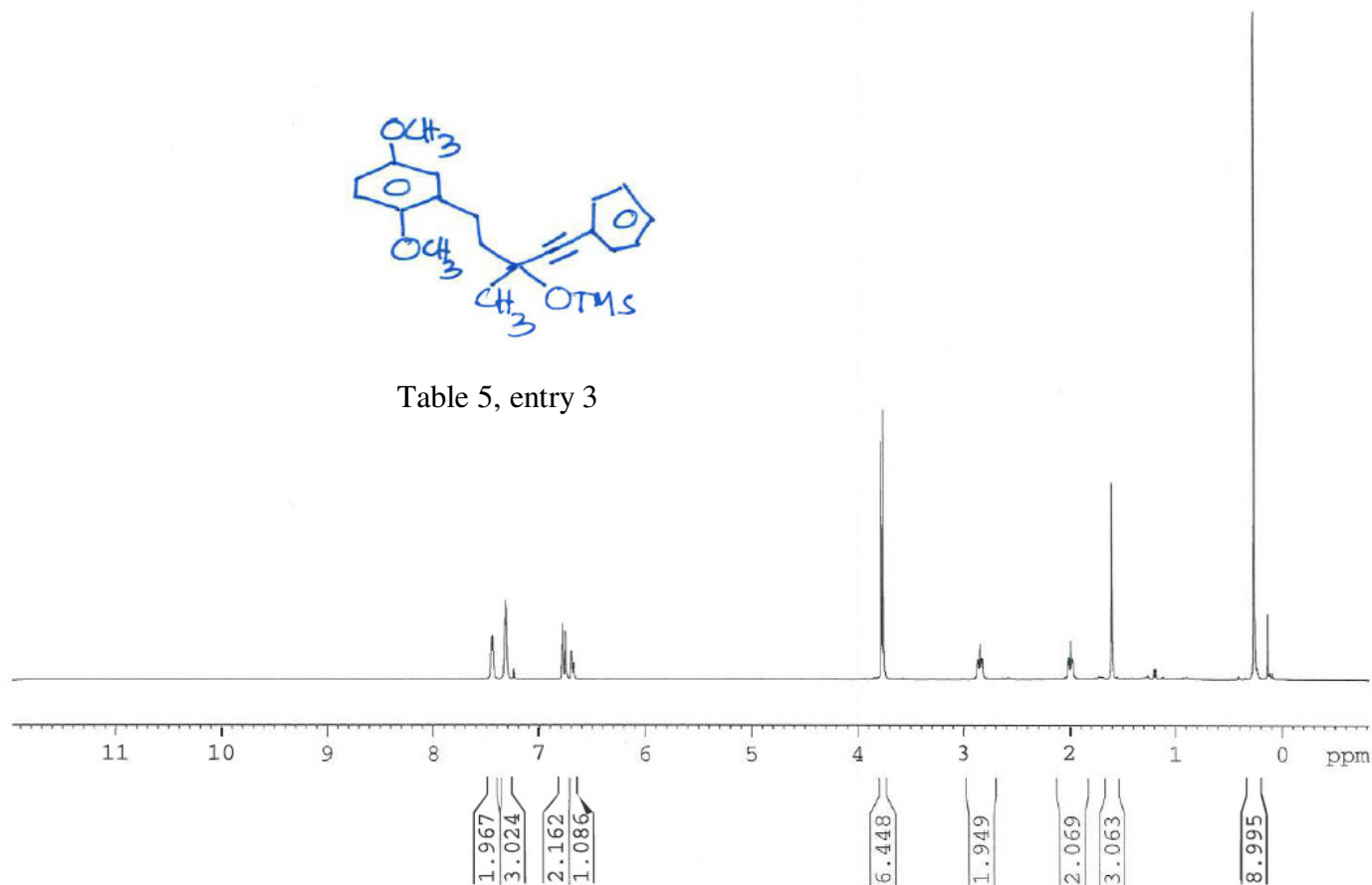


Table 5, entry 3



CVR-3719
LIQUID OIL
CDCL₃, 100MHZ
COL: 10%ETOAC/HEX
02 MAR 2010

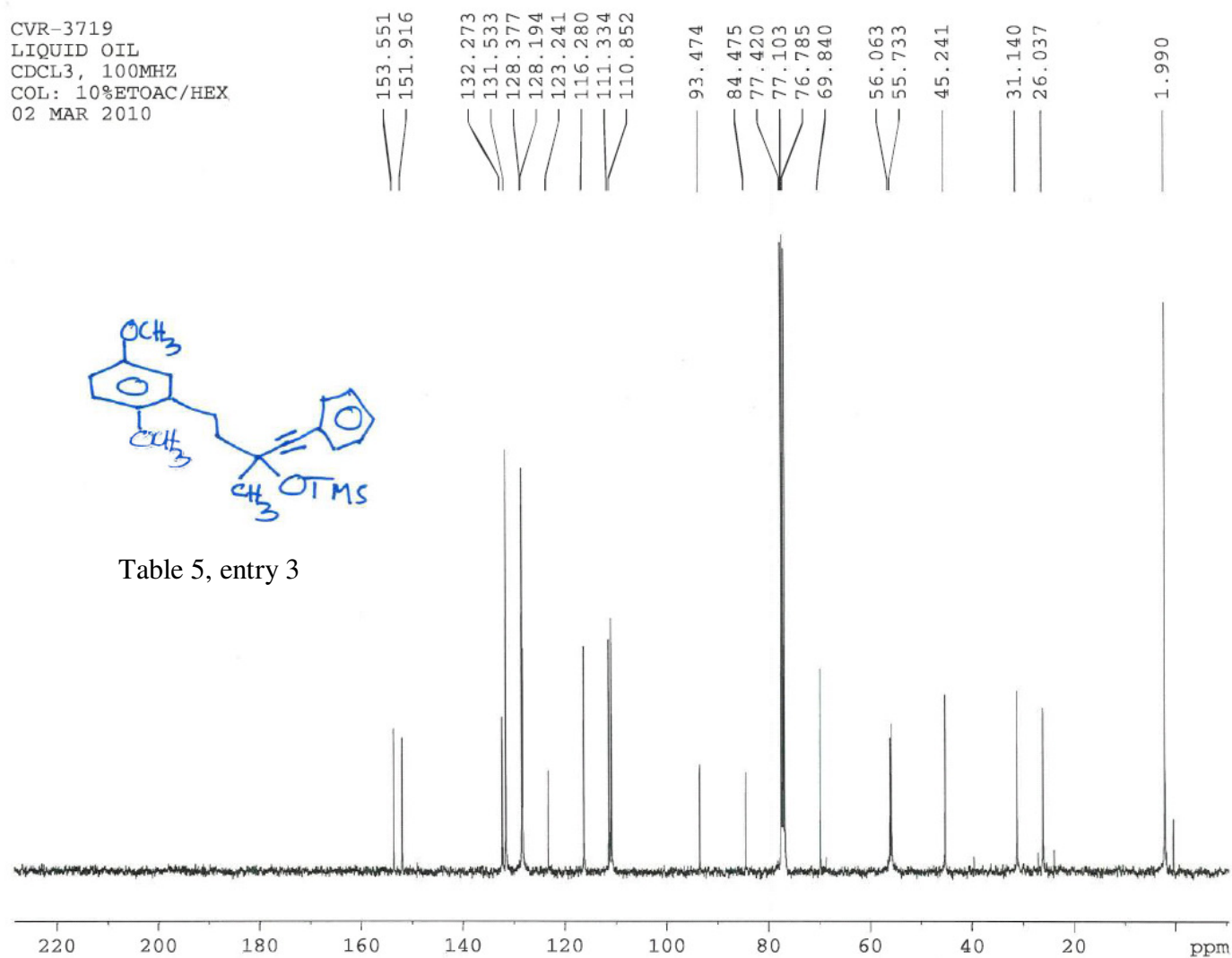
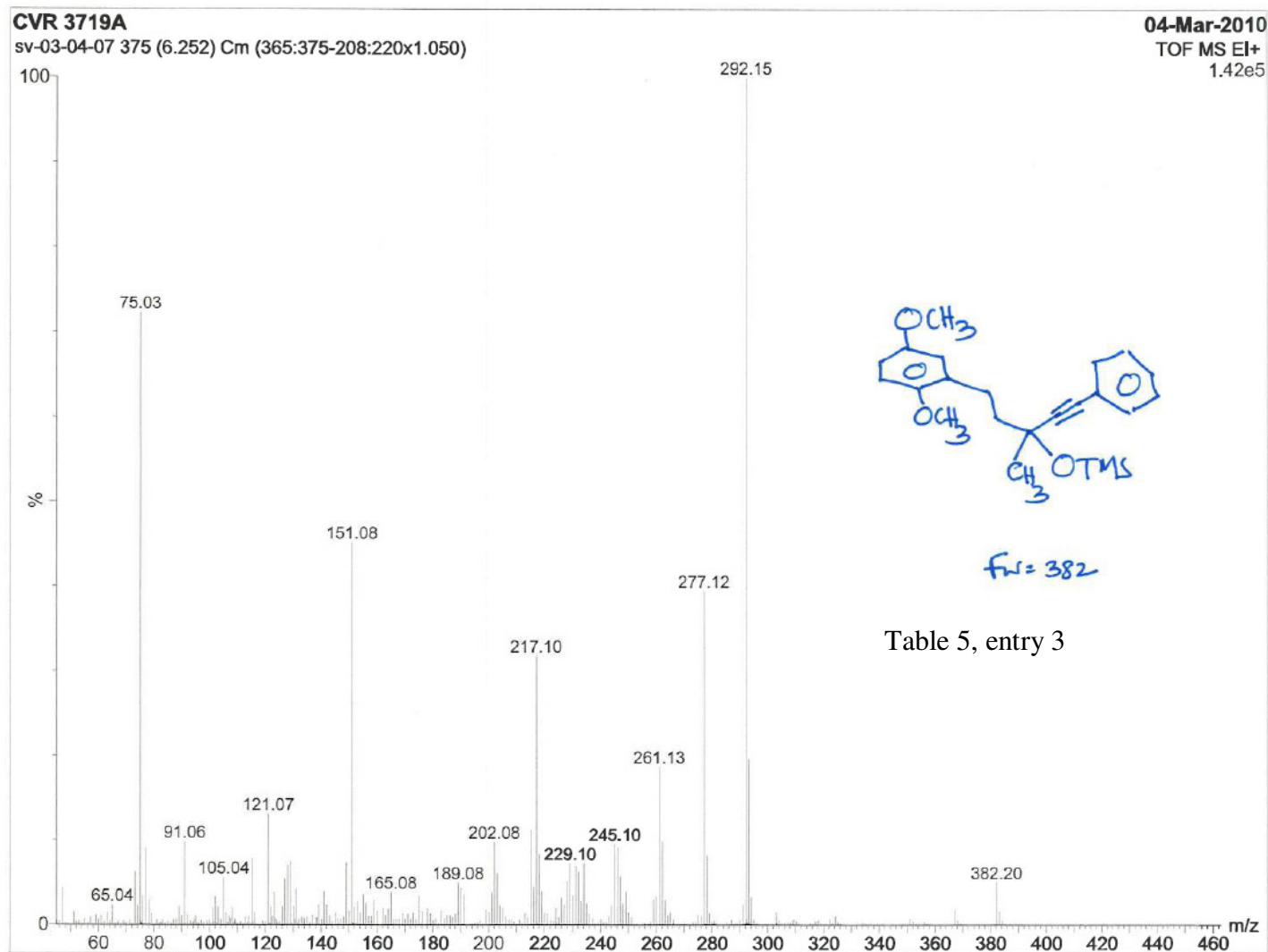


Table 5, entry 3



Elemental Composition Report

Page 1

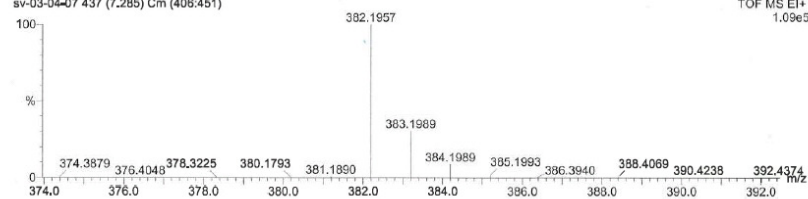
Single Mass Analysis

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0

Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions

33 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

CVR 3719A
sv-03-04-07 437 (7.285) Cm (406:451)04-Mar-2010
TOF MS EI+
1.09e5Minimum: -1.5
Maximum: 5.0 10.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
382.1957	382.1964	-0.7	-1.9	10.0	1	C23 H30 O3 Si

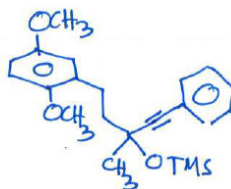


Table 5, entry 3

CVR-3718
CDCL₃, 400MHz
Clear oil
Col: Hexanes
26 FEB 2010

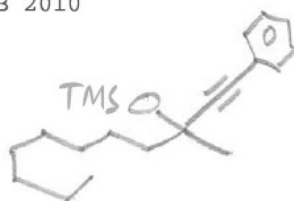
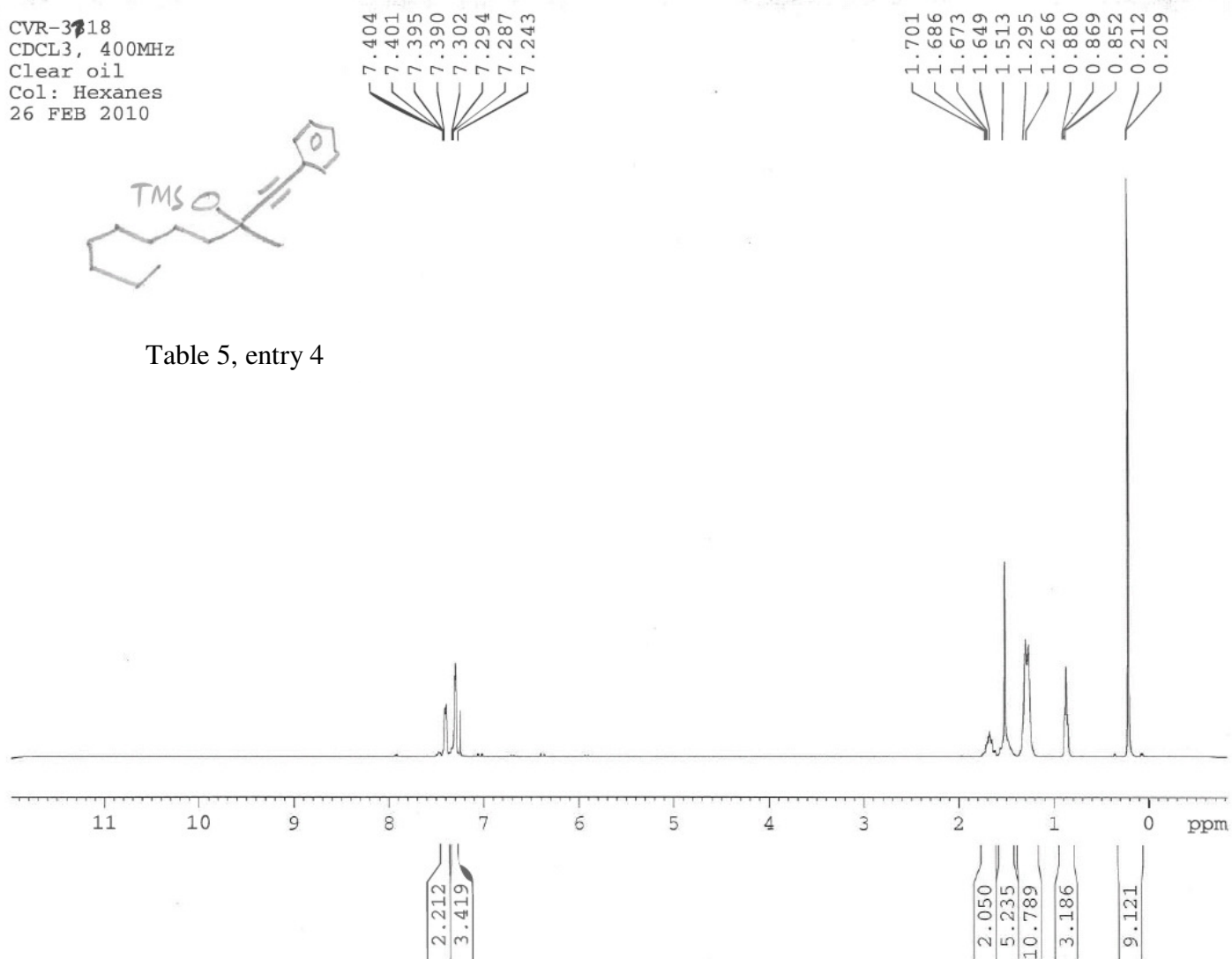


Table 5, entry 4



CVR-3718
CLEAR OIL
CDCL₃, 100MHZ
COL: HEXANES
26 FEB 2010

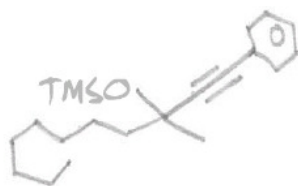
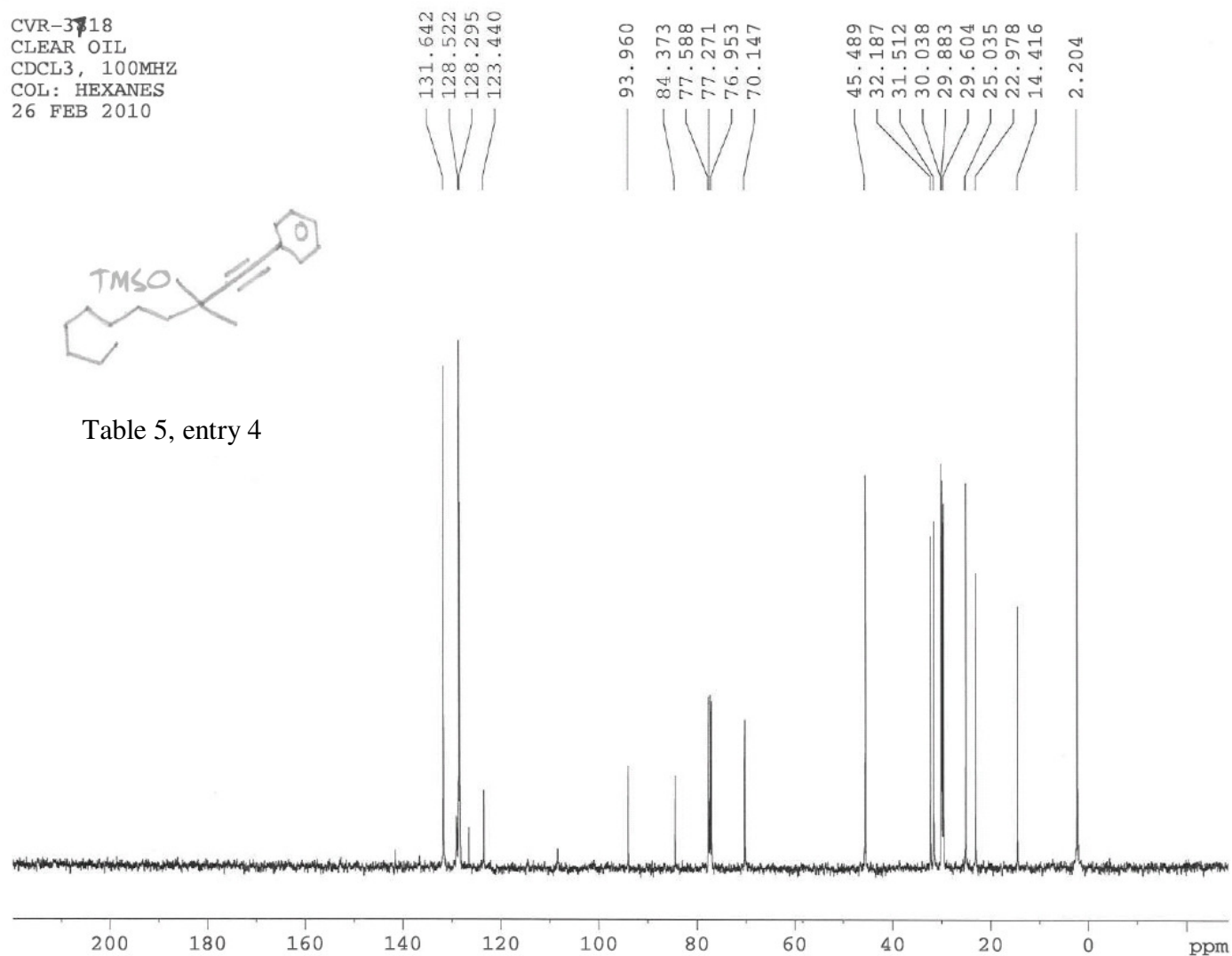
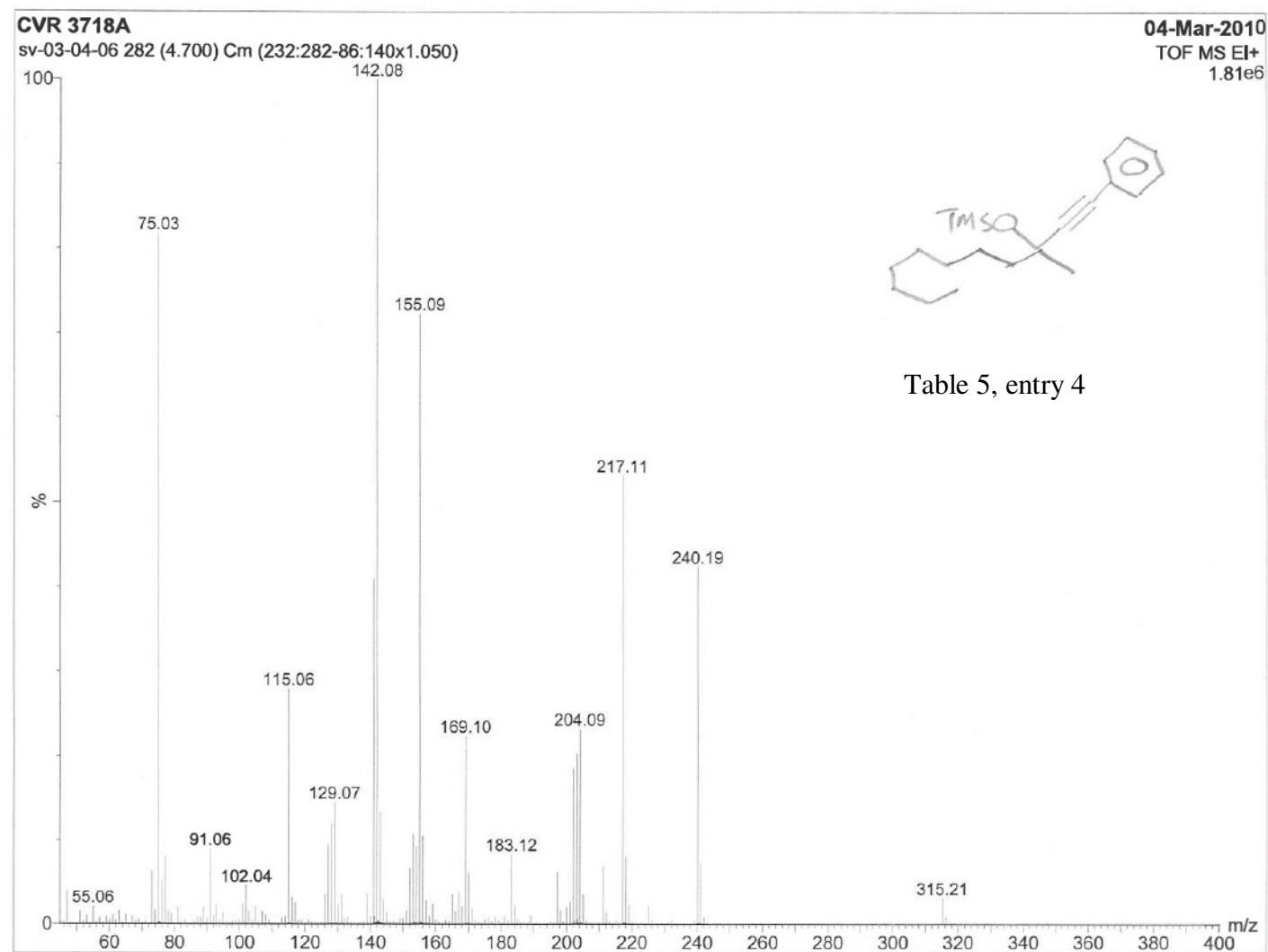
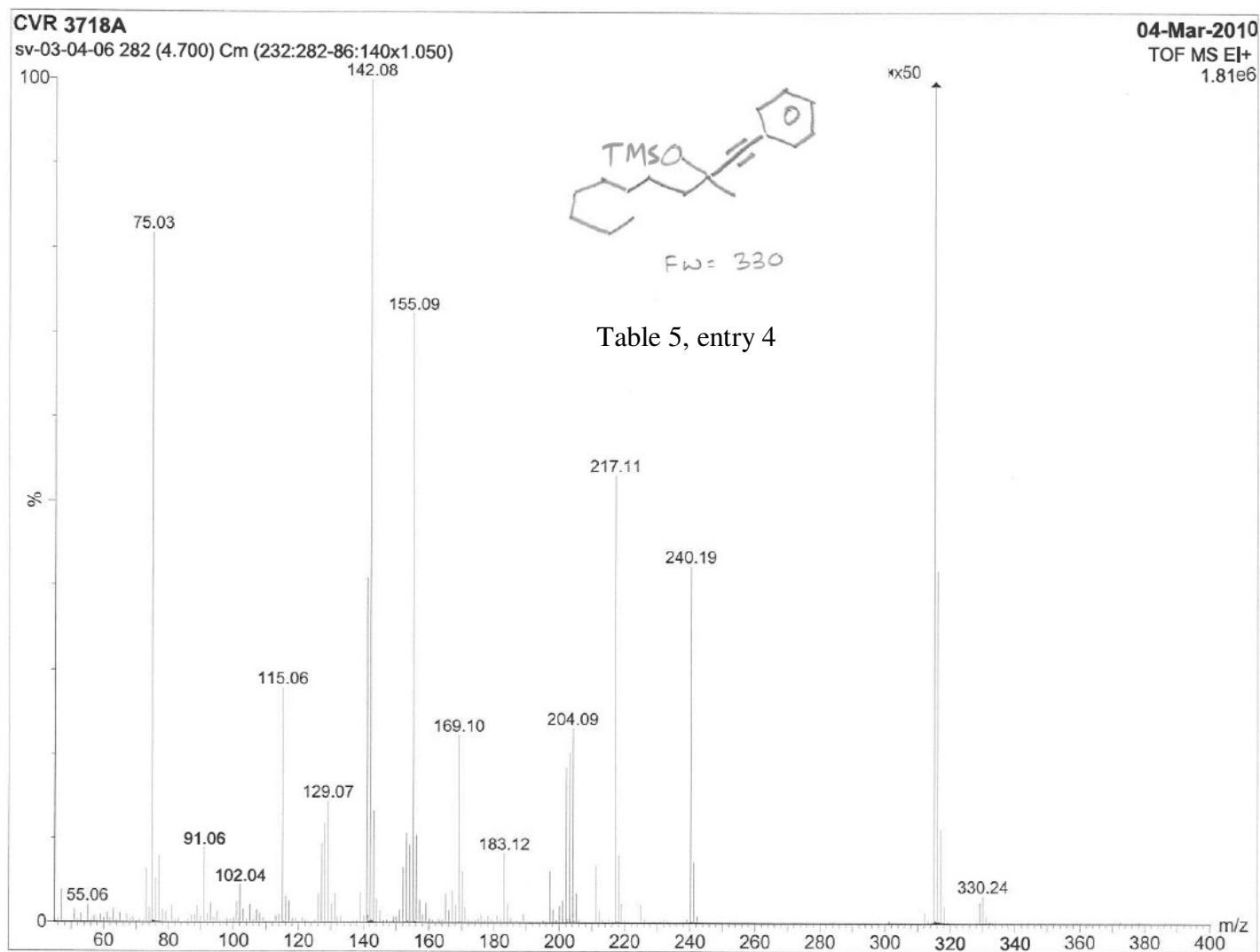


Table 5, entry 4







Elemental Composition Report

Page 1

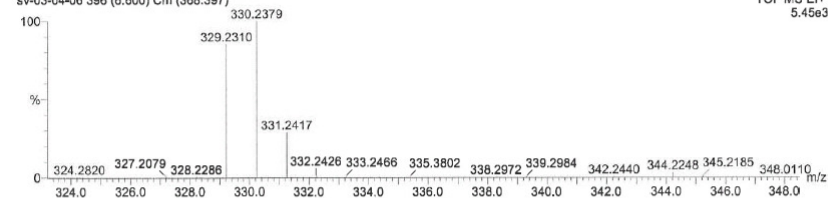
Single Mass Analysis

Tolerance = 3.0 PPM / DBE: min = -1.5, max = 50.0

Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions

9 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

CVR 3718A
sv-03-04-06 396 (6.600) Cm (368:397)04-Mar-2010
TOF MS EI+
5.45e3

Minimum:				-1.5		
Maximum:		5.0	3.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
330.2379	330.2379	0.0	0.0	6.0	1	C21 H34 O Si

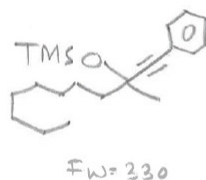


Table 5, entry 4

CVR-3718
LIQUID OIL
CDCL₃, 400MHZ
10%EtOAc/Hex
01 MAR 2010

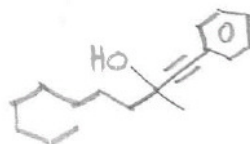
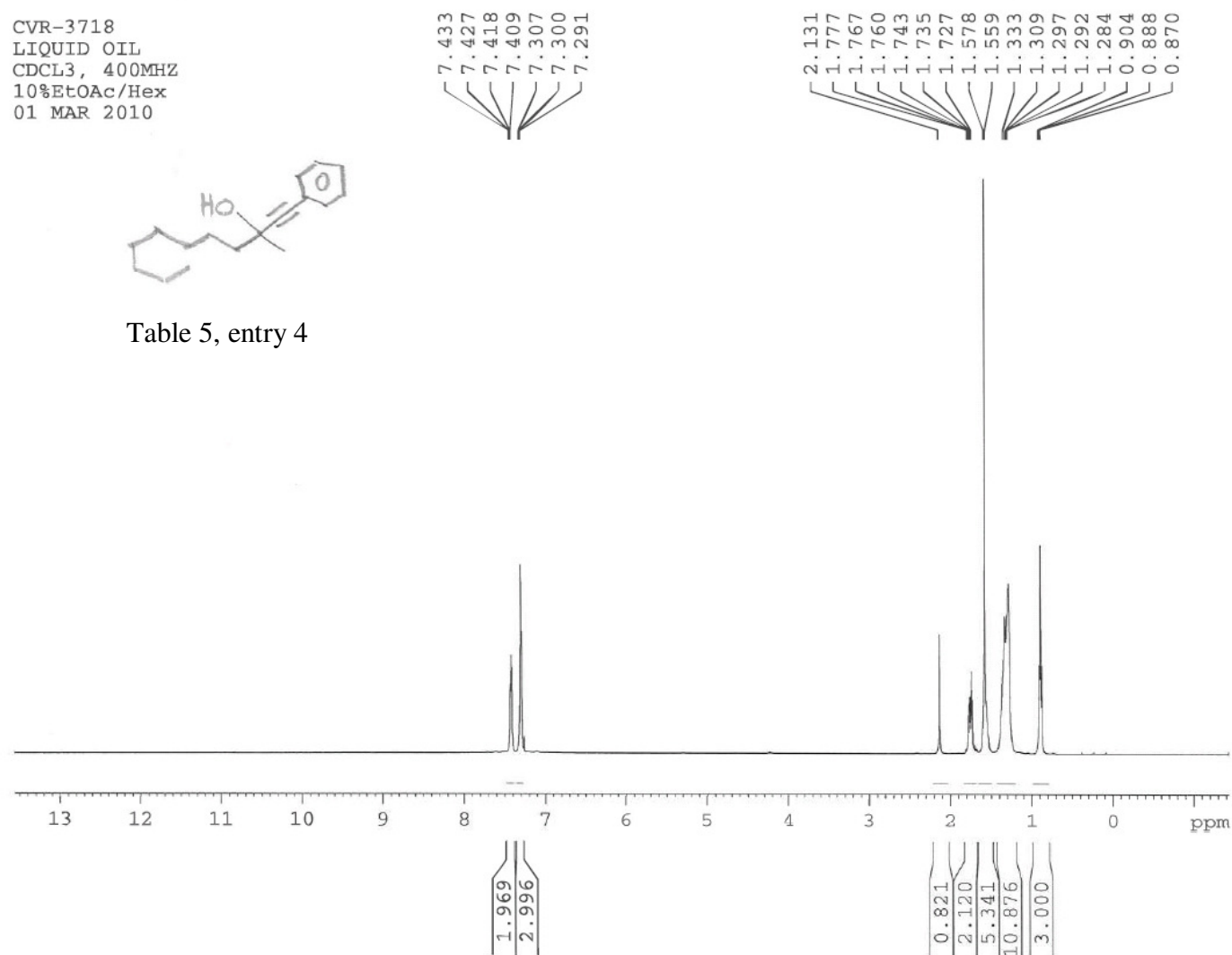


Table 5, entry 4



CVR-3718
CDCL₃, 100MHz
Liquid Oil
10%EtOAc/Hex
01 MA R 2010

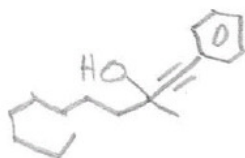
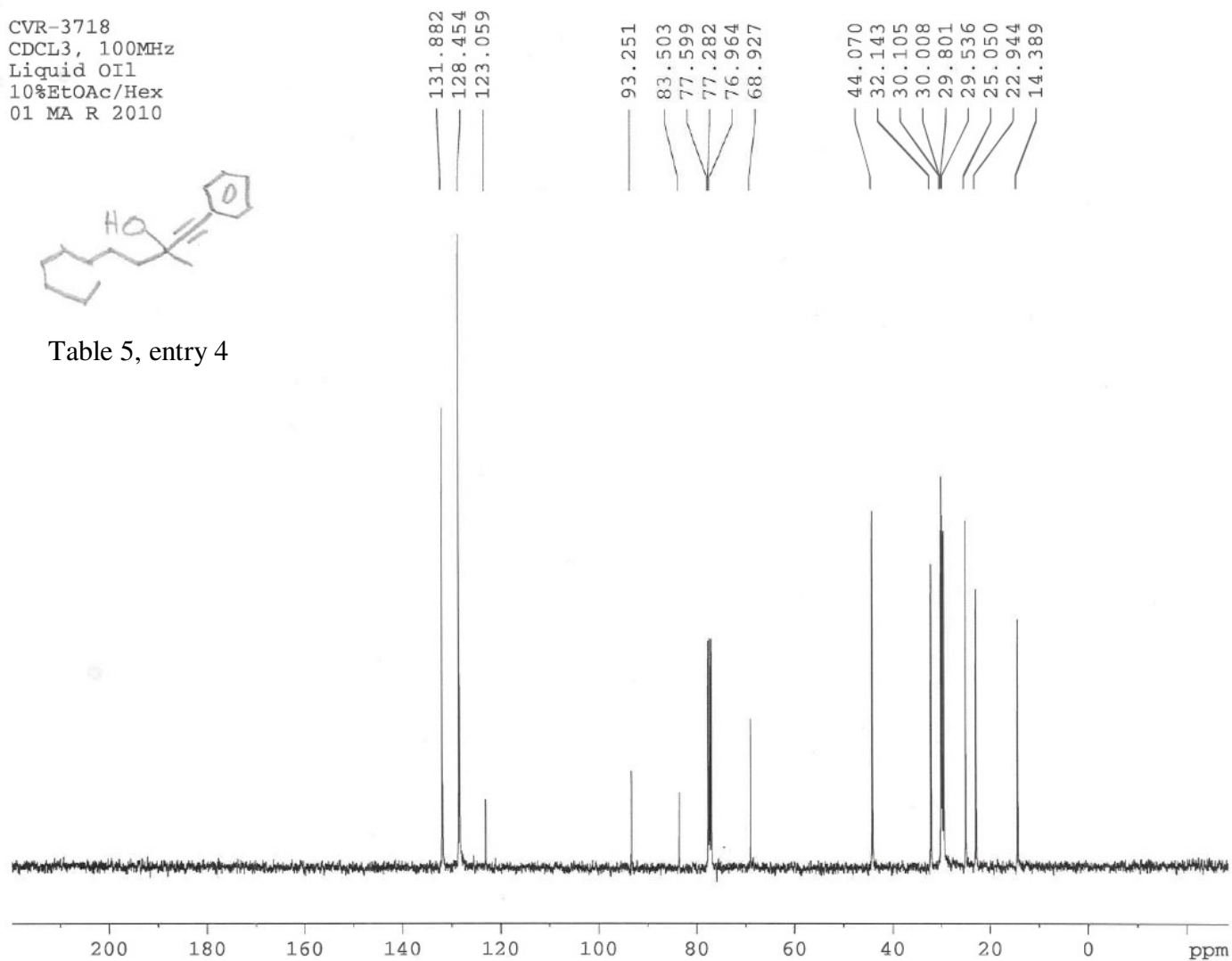
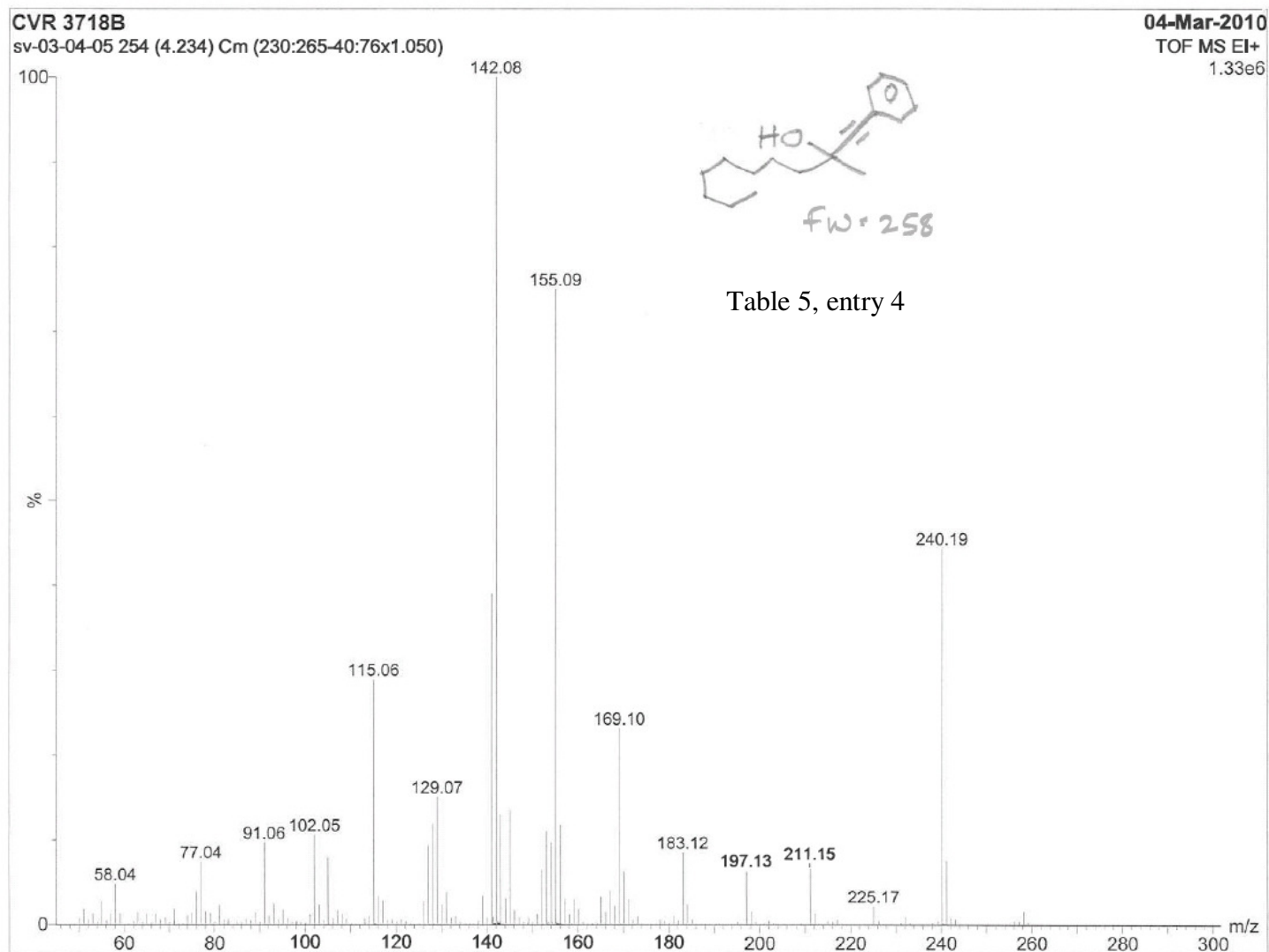
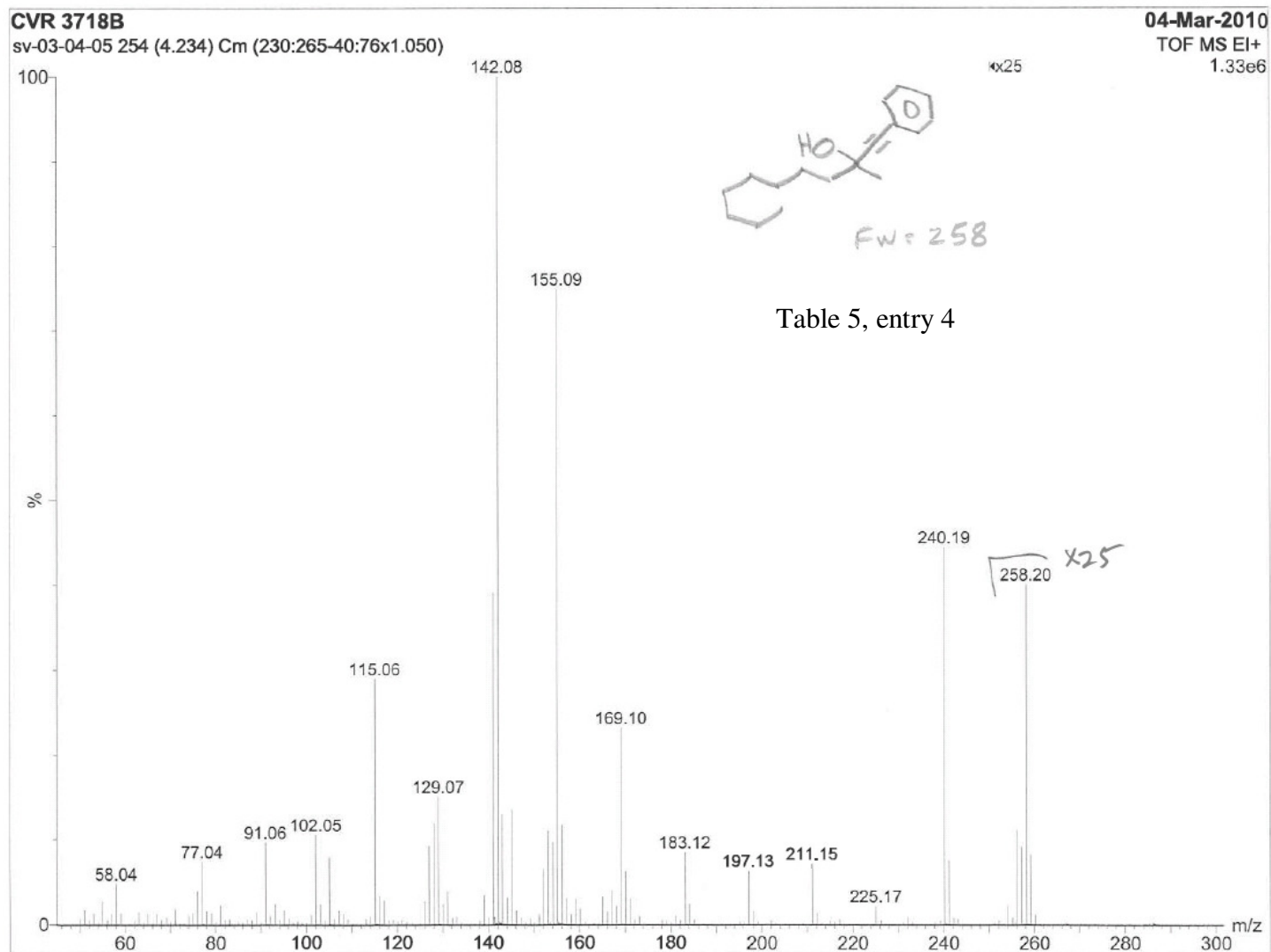


Table 5, entry 4







Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 3.0 PPM / DBE: min = -1.5, max = 50.0

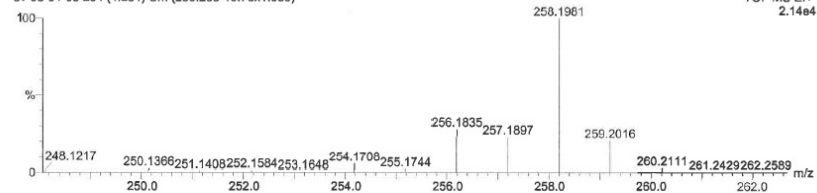
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions

41 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

CVR 3718B

sv-03-04-05 254 (4.234) Cm (230:265-40:76x1.050)

04-Mar-2010
TOF MS EI+
2.14e4

Minimum:				-1.5		
Maximum:	5.0	3.0		50.0		
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
258.1981	258.1984	-0.3	-1.0	6.0	1	C18 H26 O

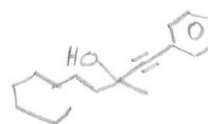


Table 5, entry 4