

Synthesis of Functionalized Cinnamaldehyde Derivatives by an Oxidative Heck Reaction and Their Use as Starting Materials for Preparation of *Mycobacterium tuberculosis* 1-Deoxy-D-xylulose-5-phosphate Reductoisomerase Inhibitors

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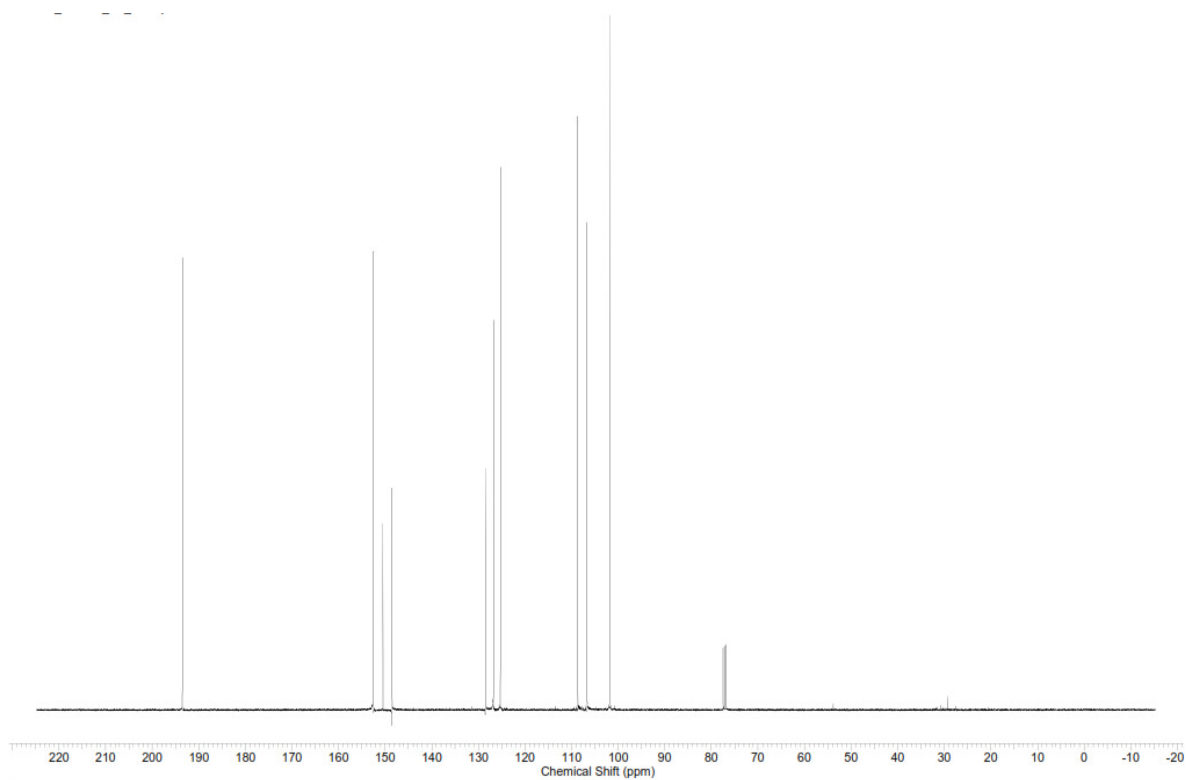
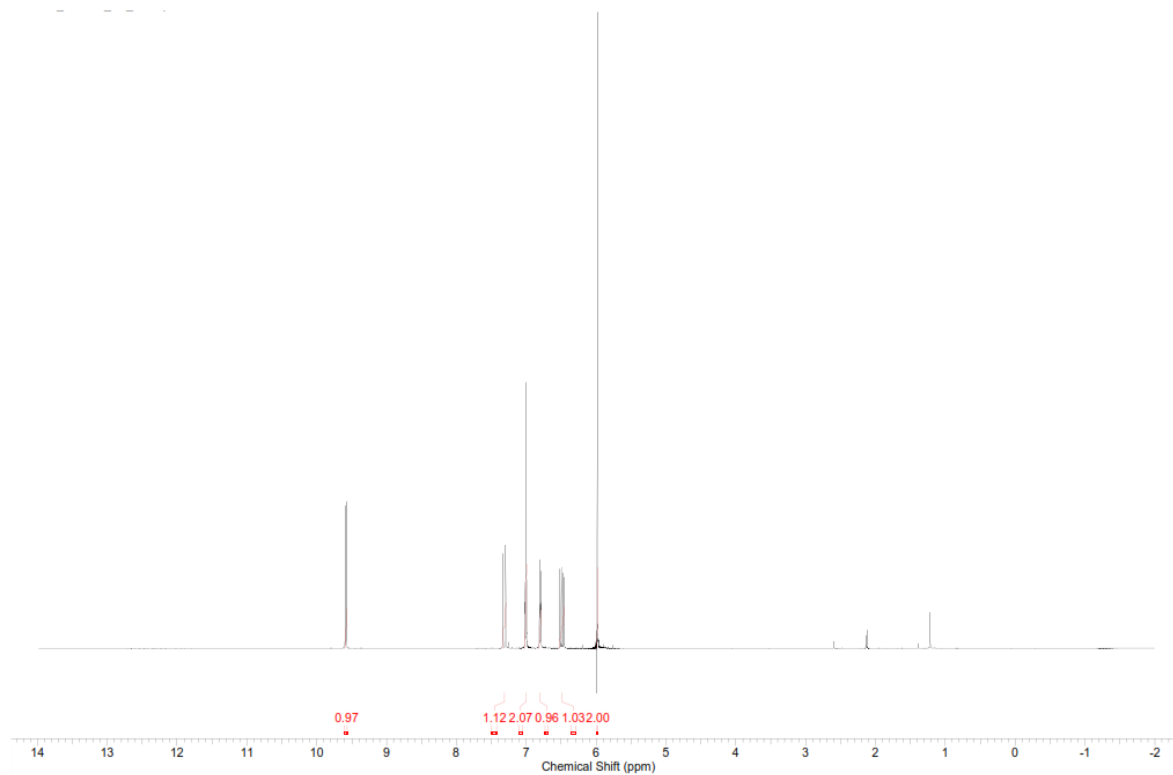
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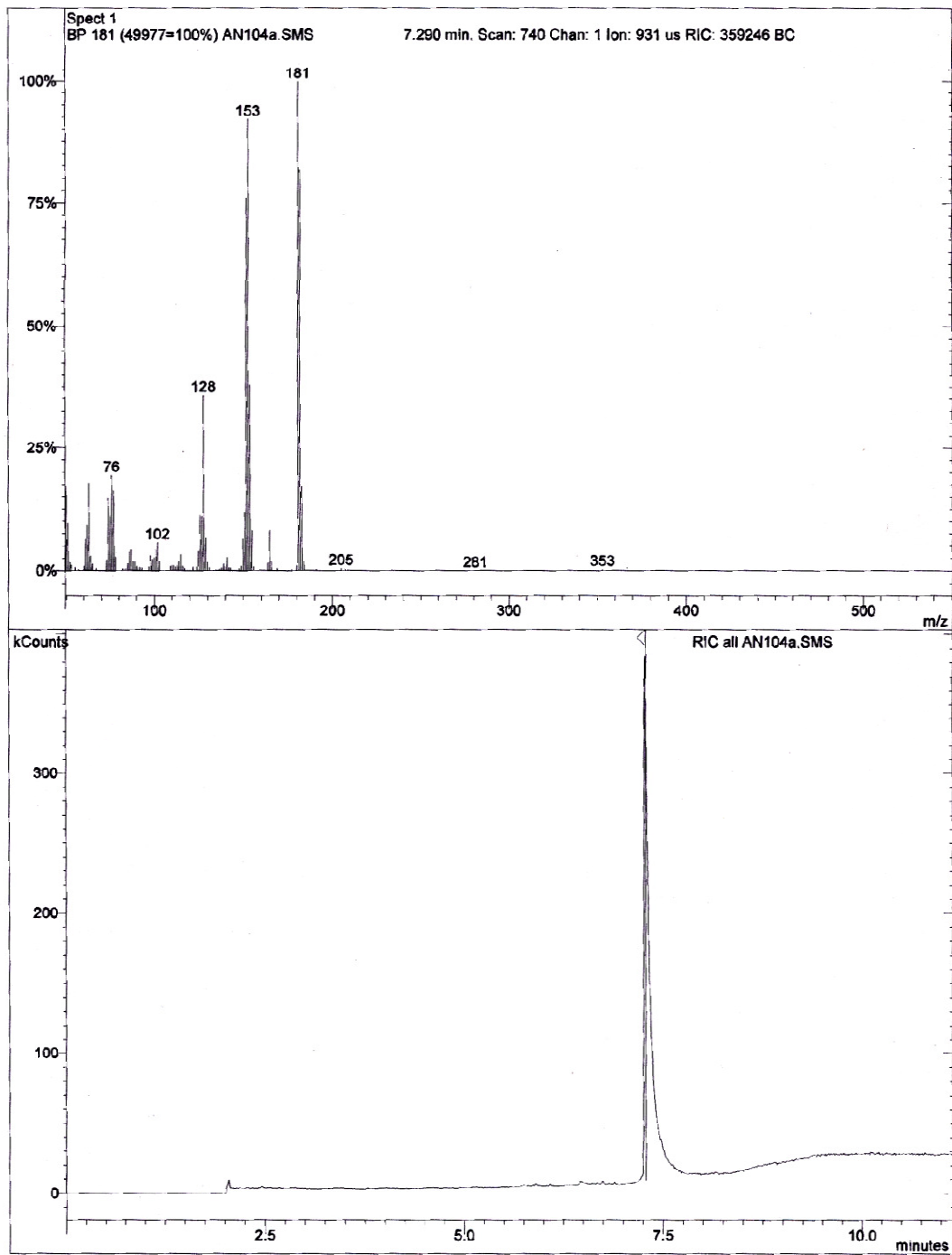
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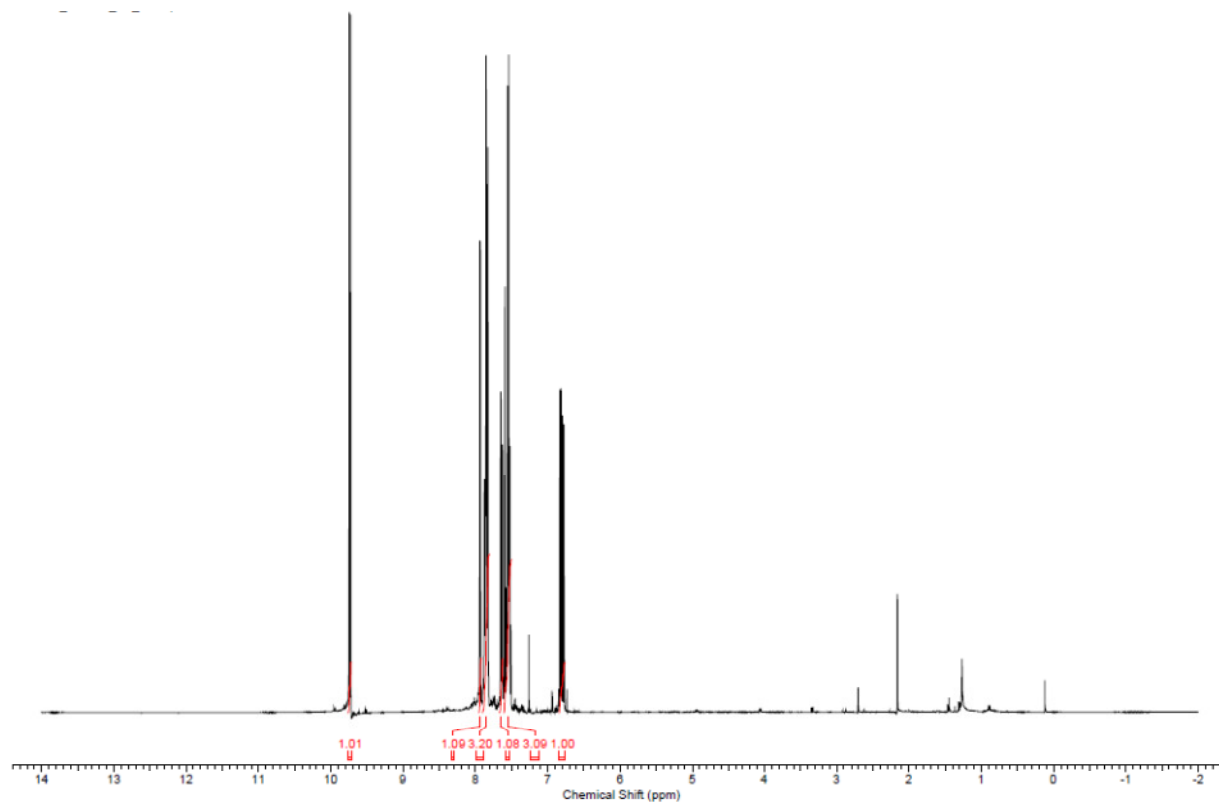
# $^1\text{H}$ NMR and $^{13}\text{C}$ NMR 6a



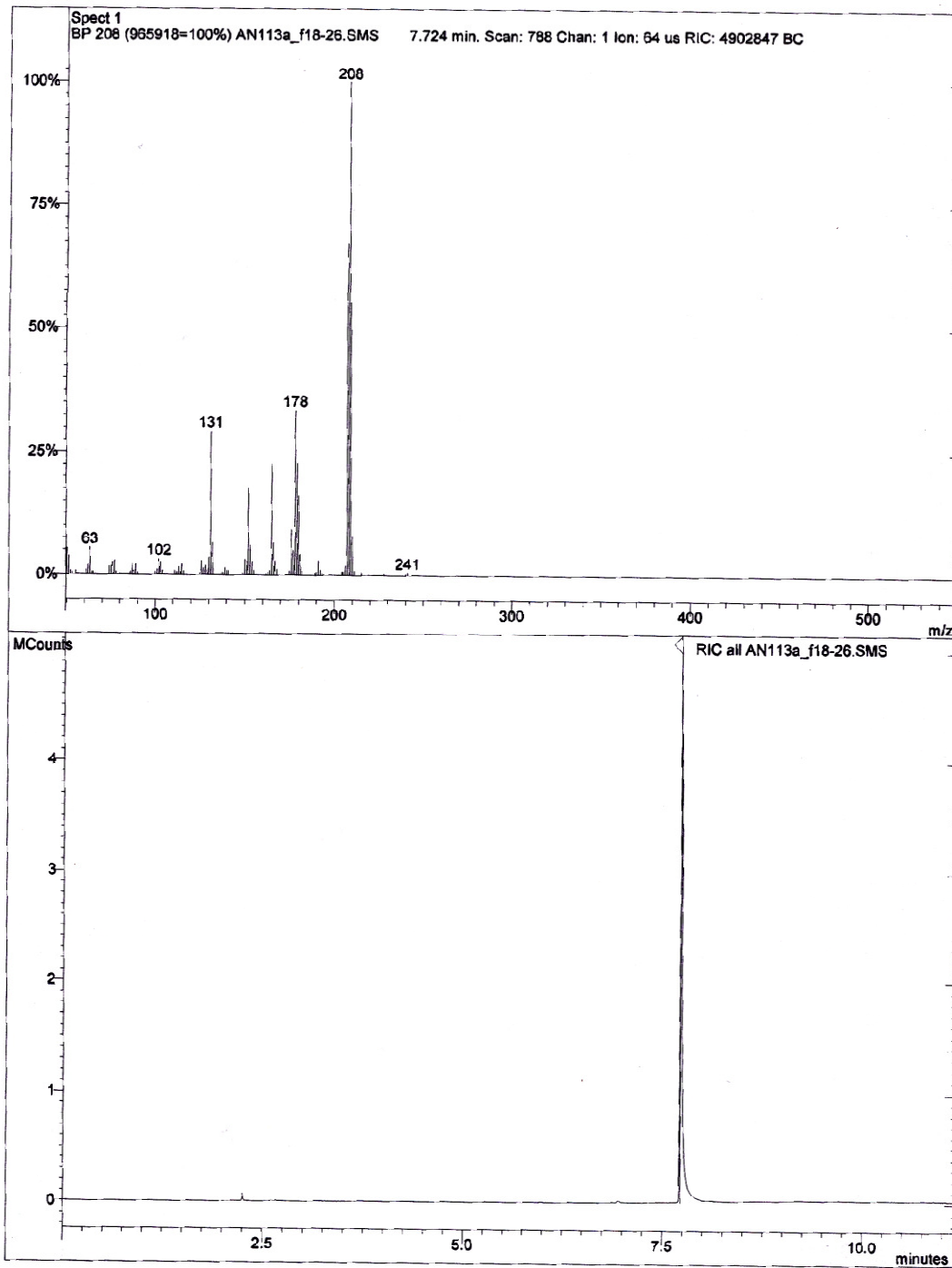
# GC-MS 6b



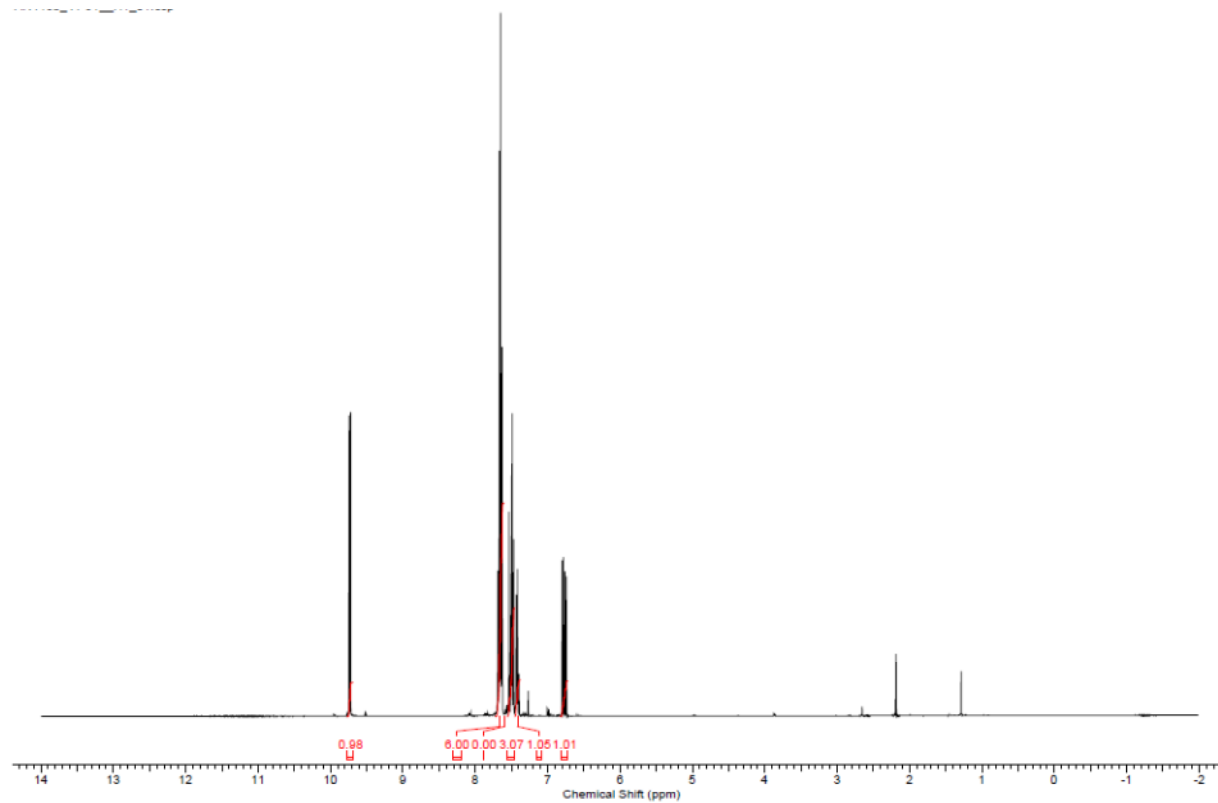
# **<sup>1</sup>H NMR 6b**



# GC-MS 6c

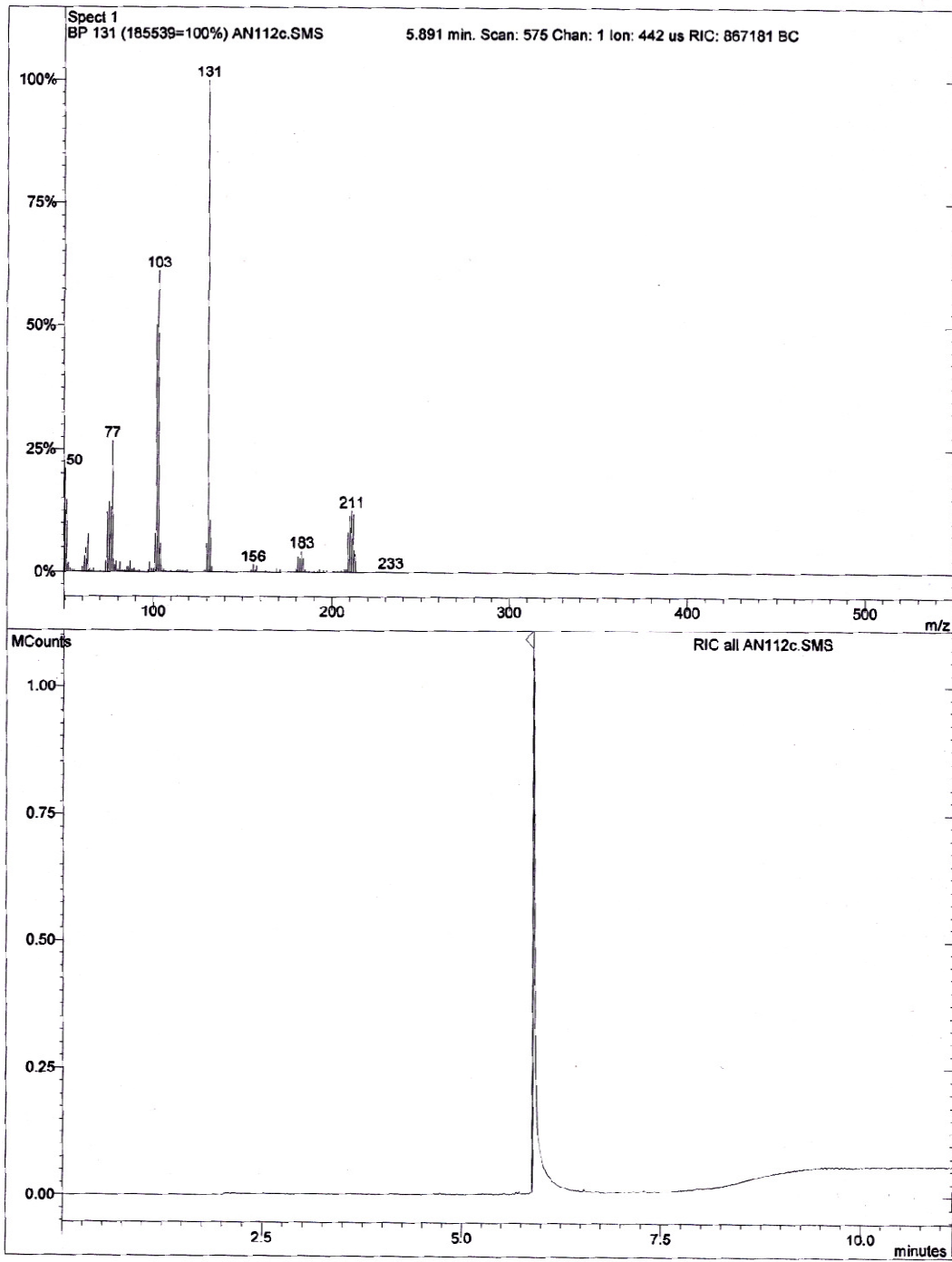


# <sup>1</sup>H NMR 6c

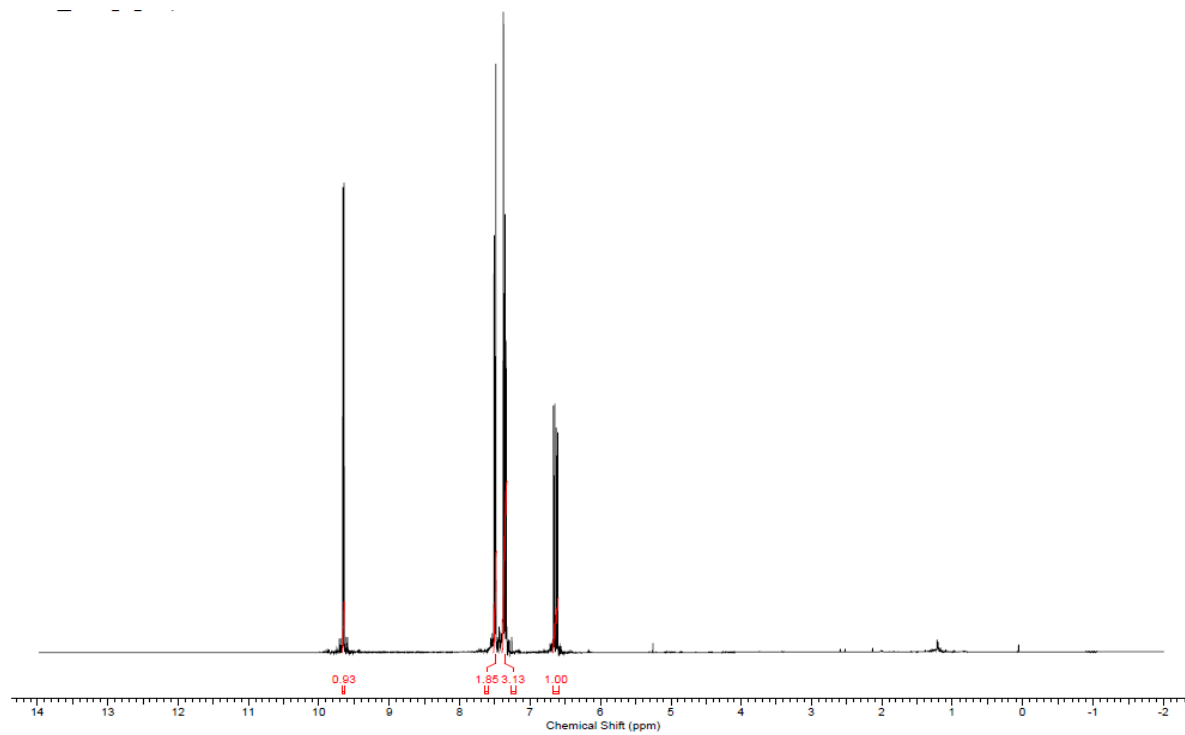




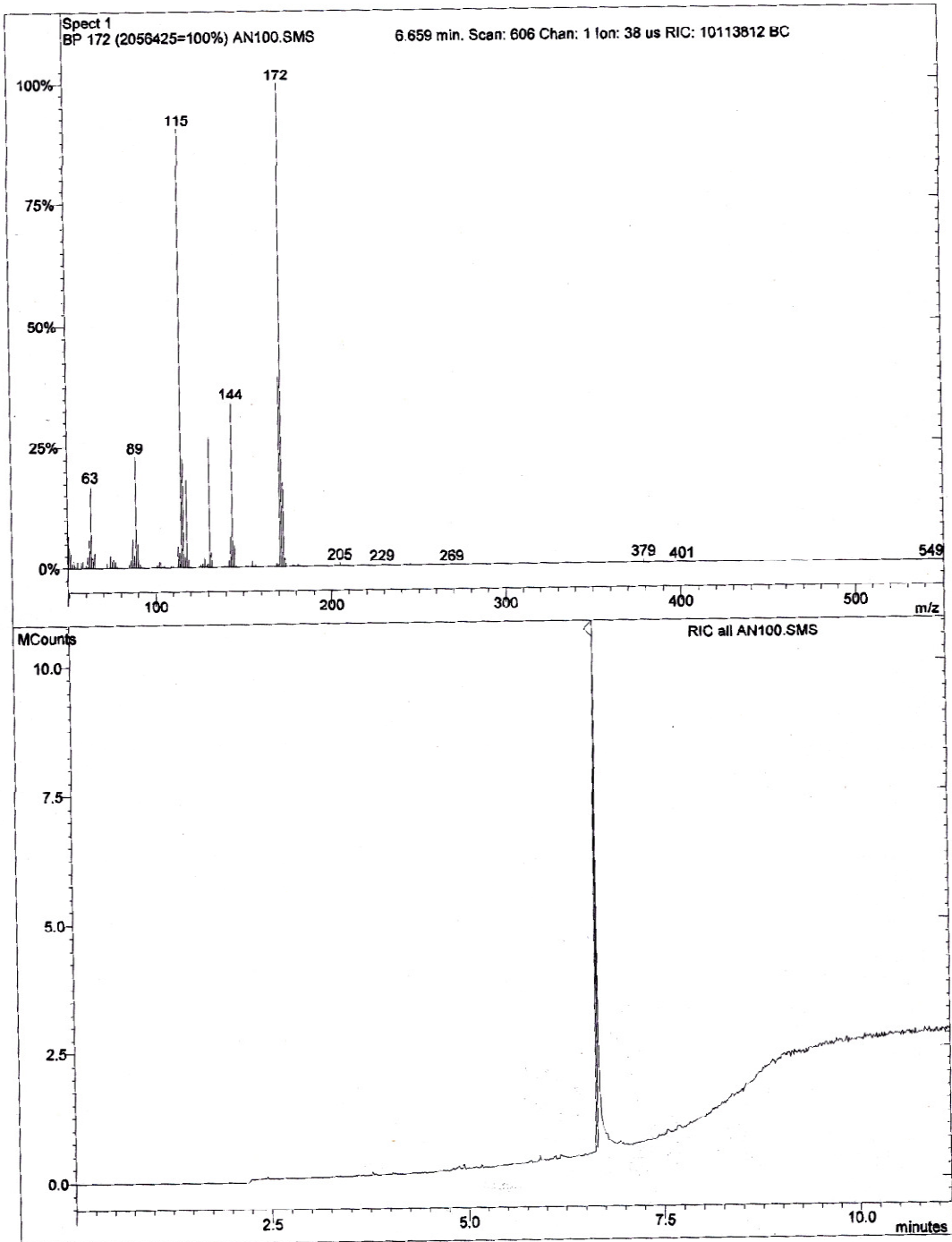
# GC-MS 6d



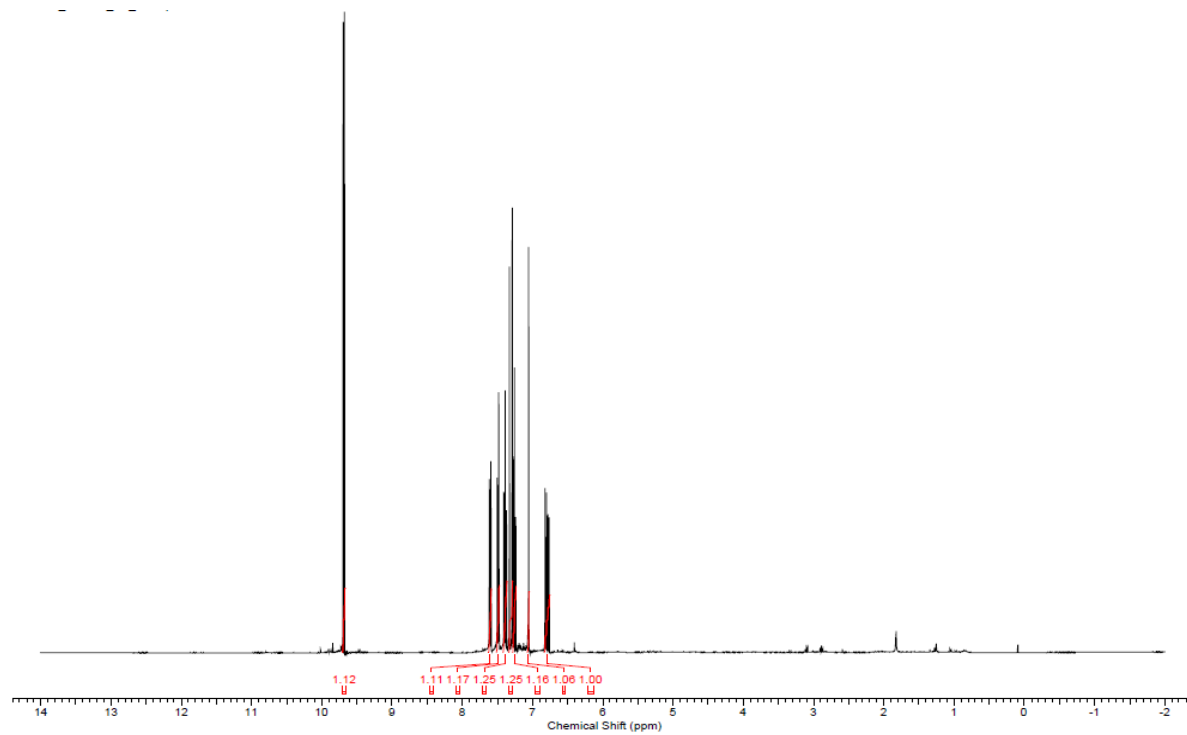
# <sup>1</sup>H NMR 6d



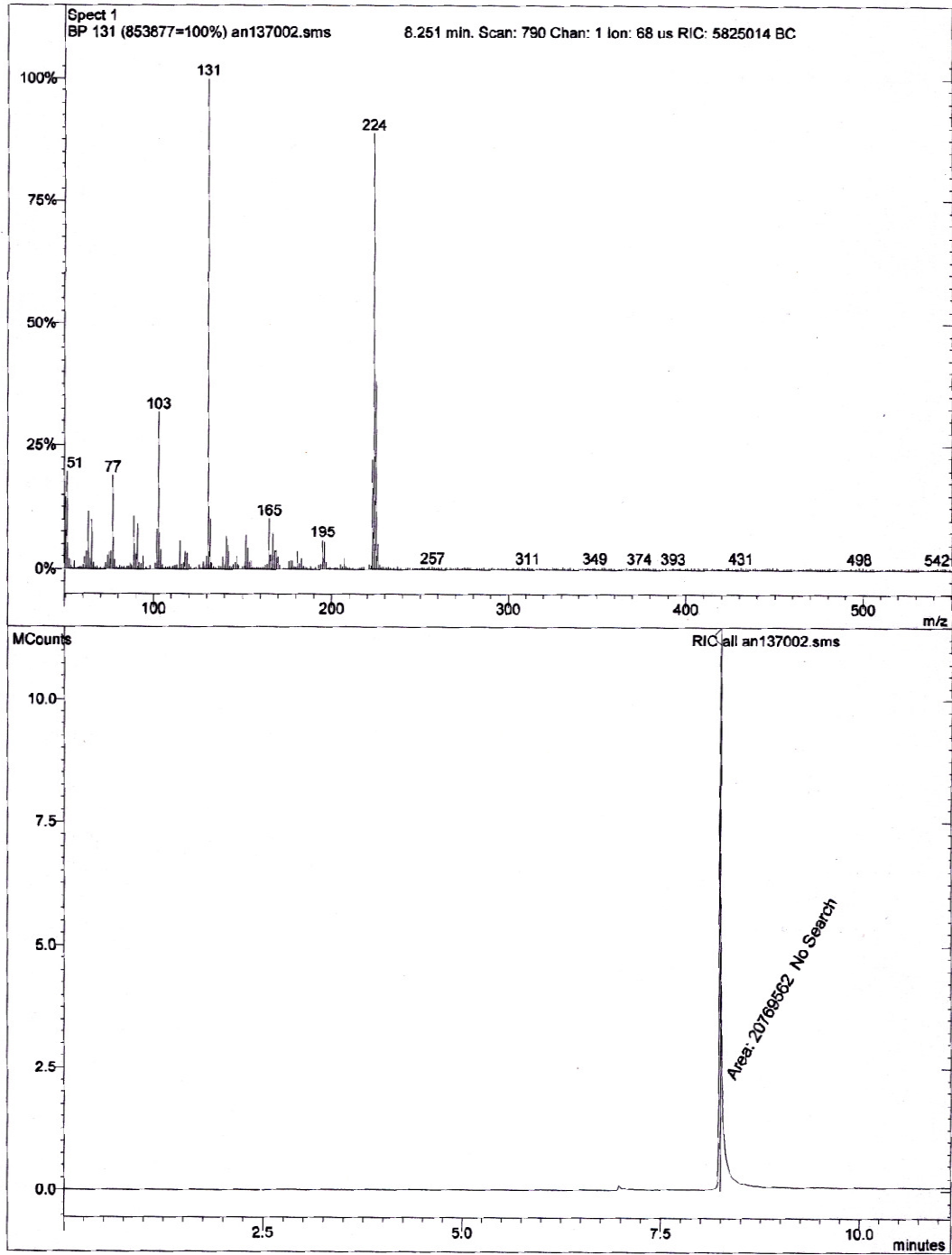
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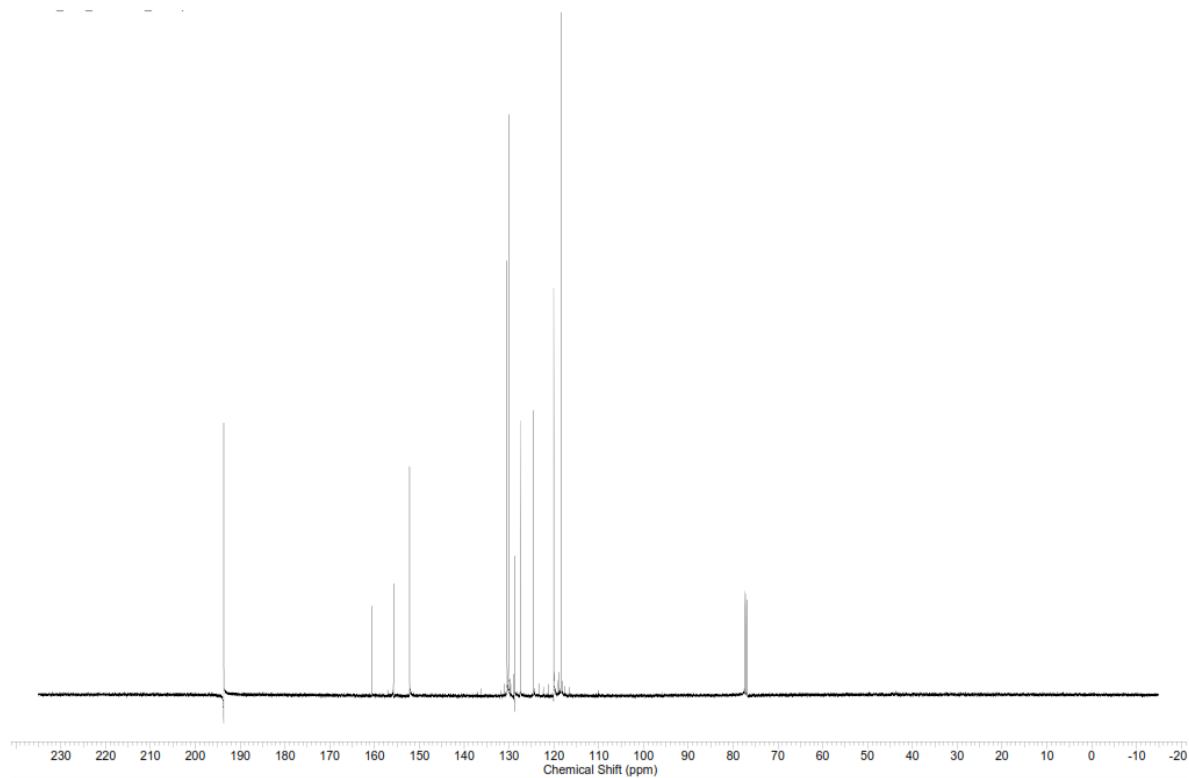
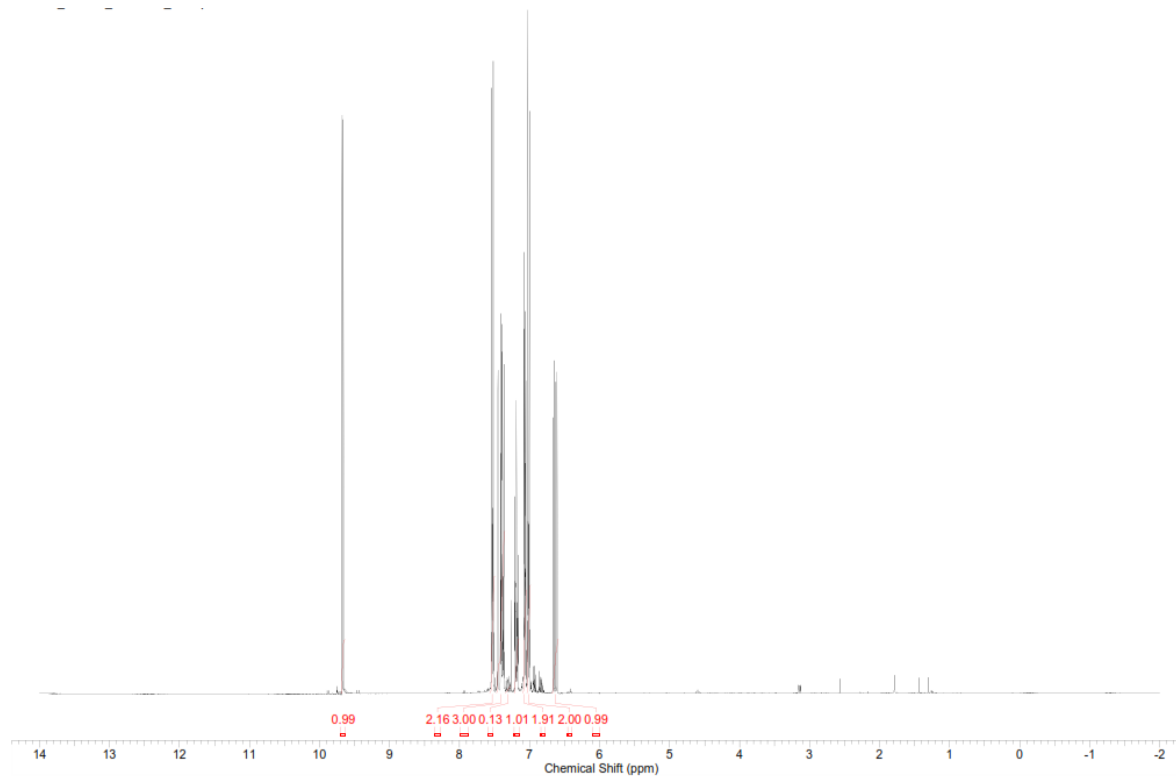
# <sup>1</sup>H NMR 6e



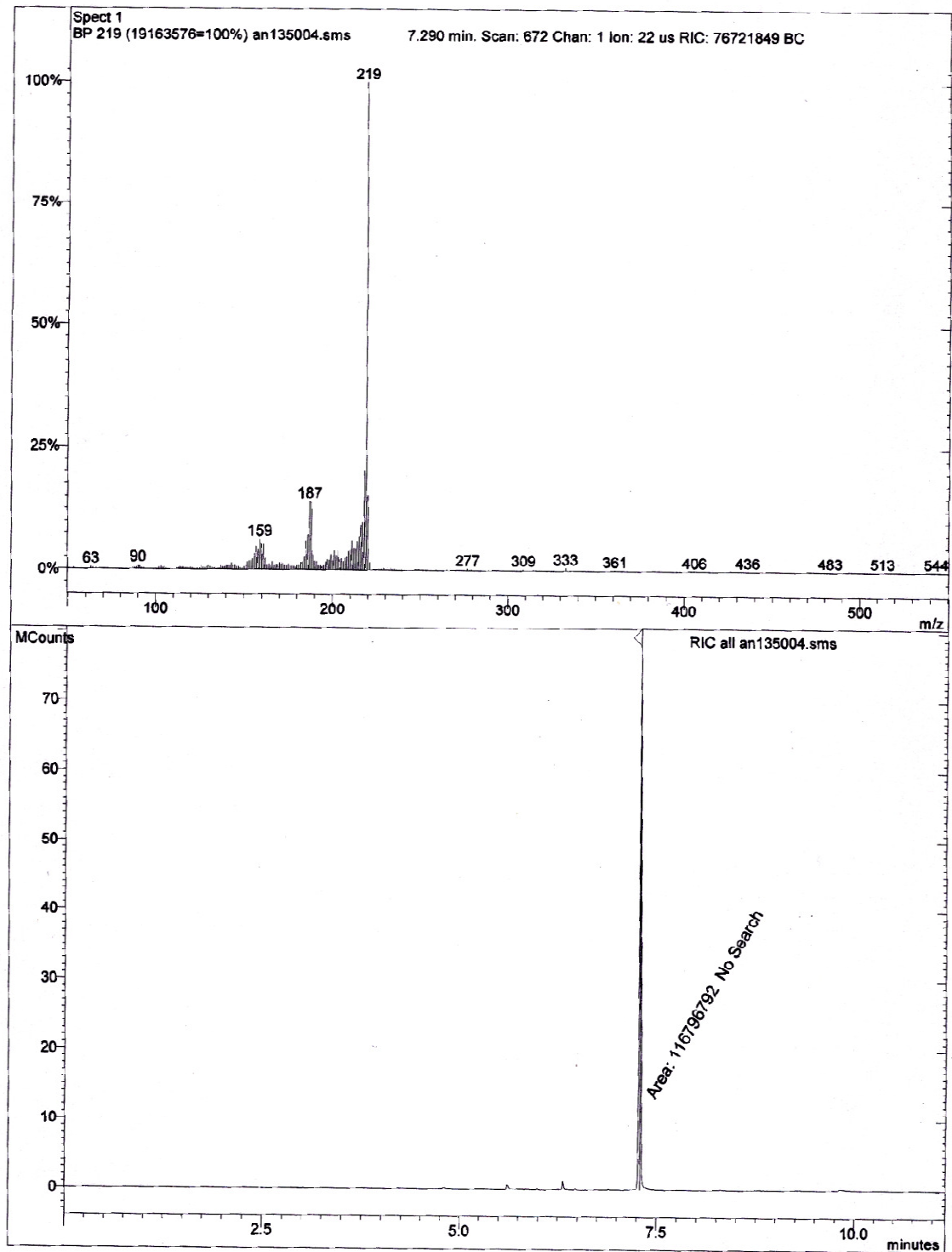
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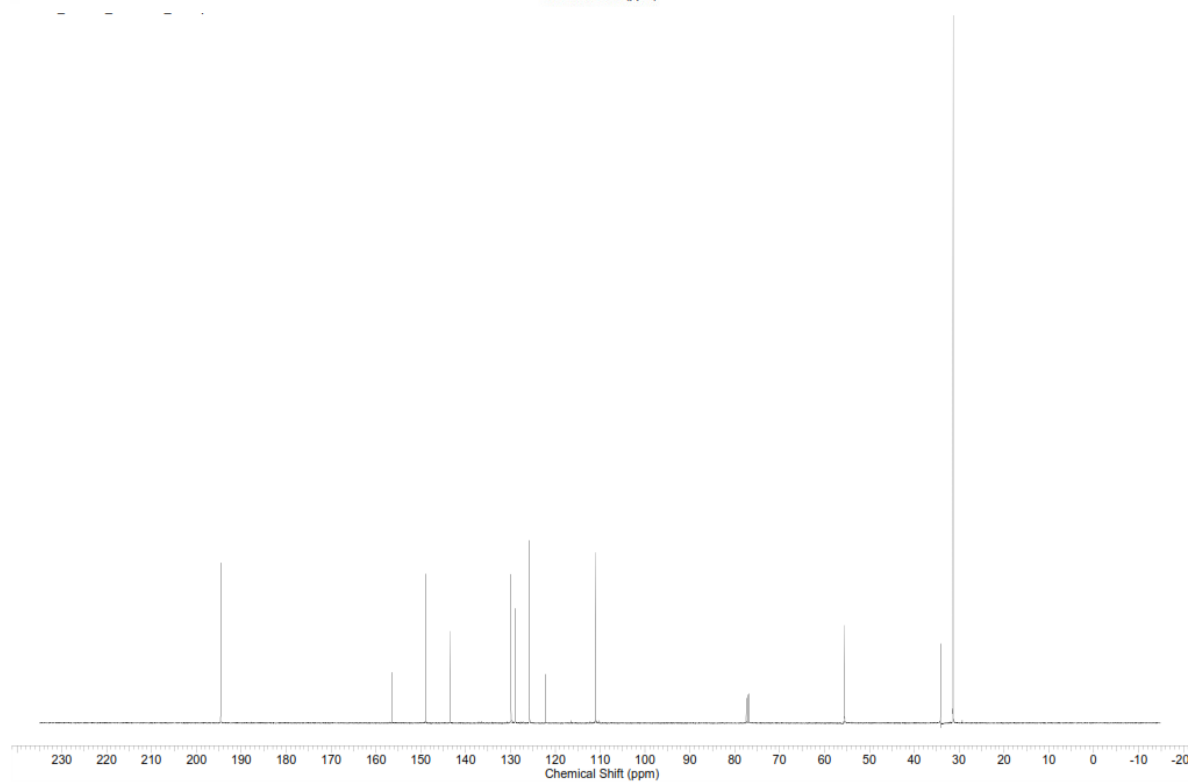
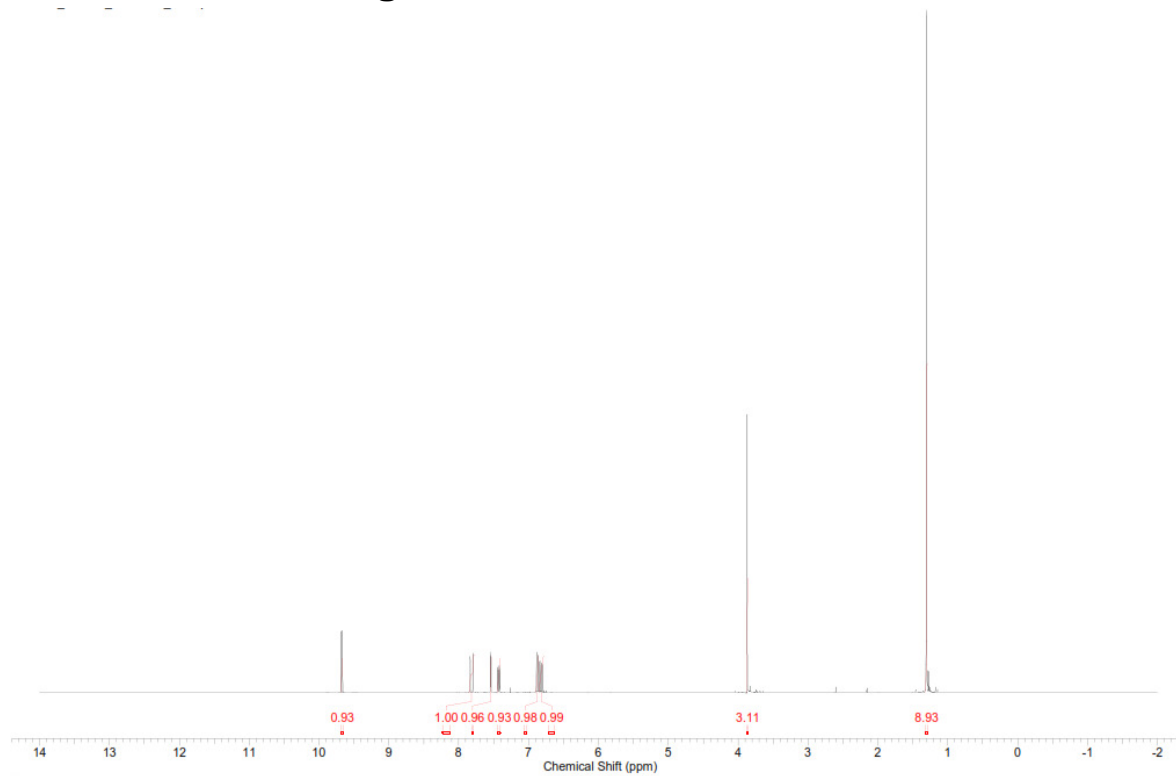
# $^1\text{H}$ NMR and $^{13}\text{C}$ NMR 6f



# GC-MS 6g

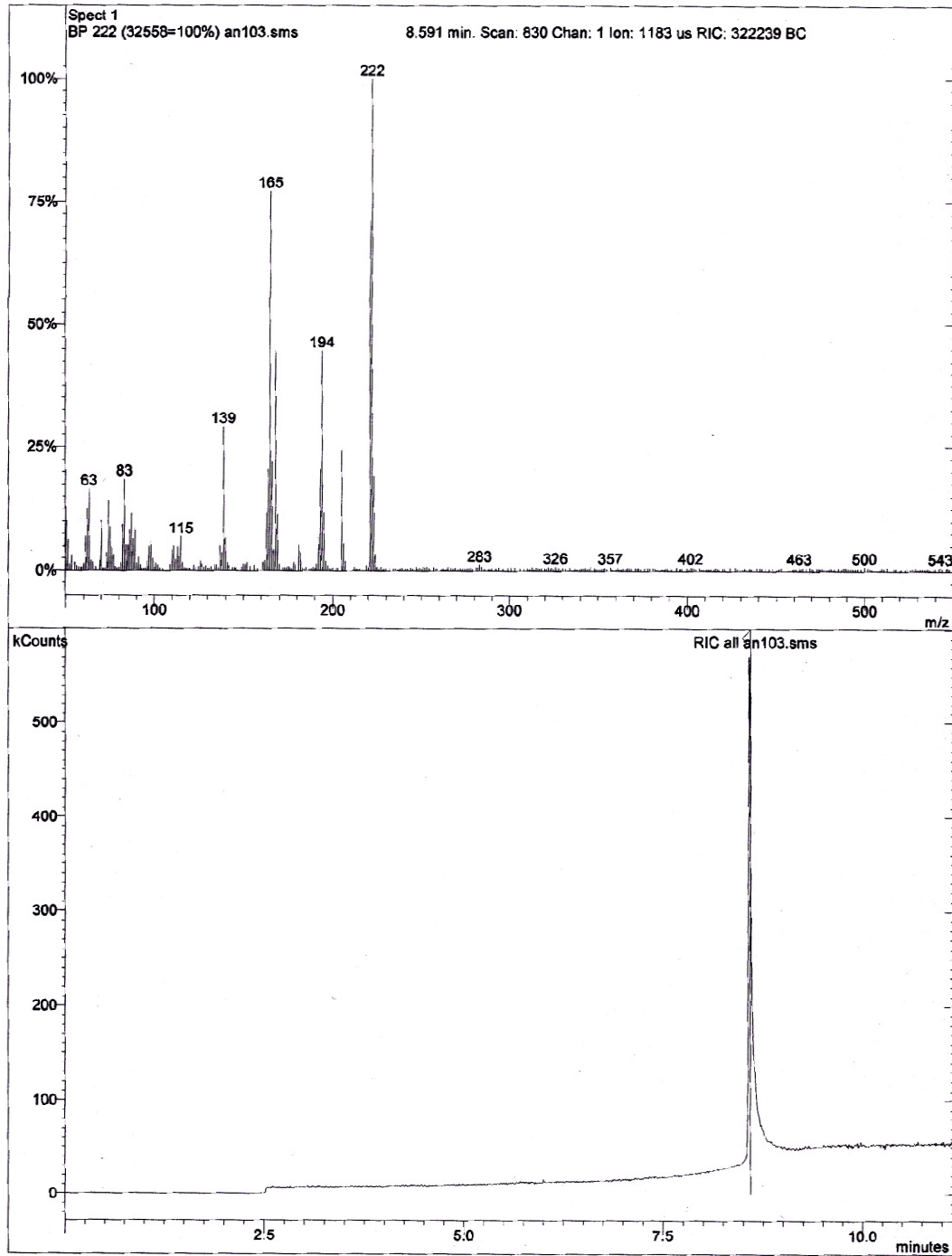


# $^1\text{H}$ NMR and $^{13}\text{C}$ NMR 6g

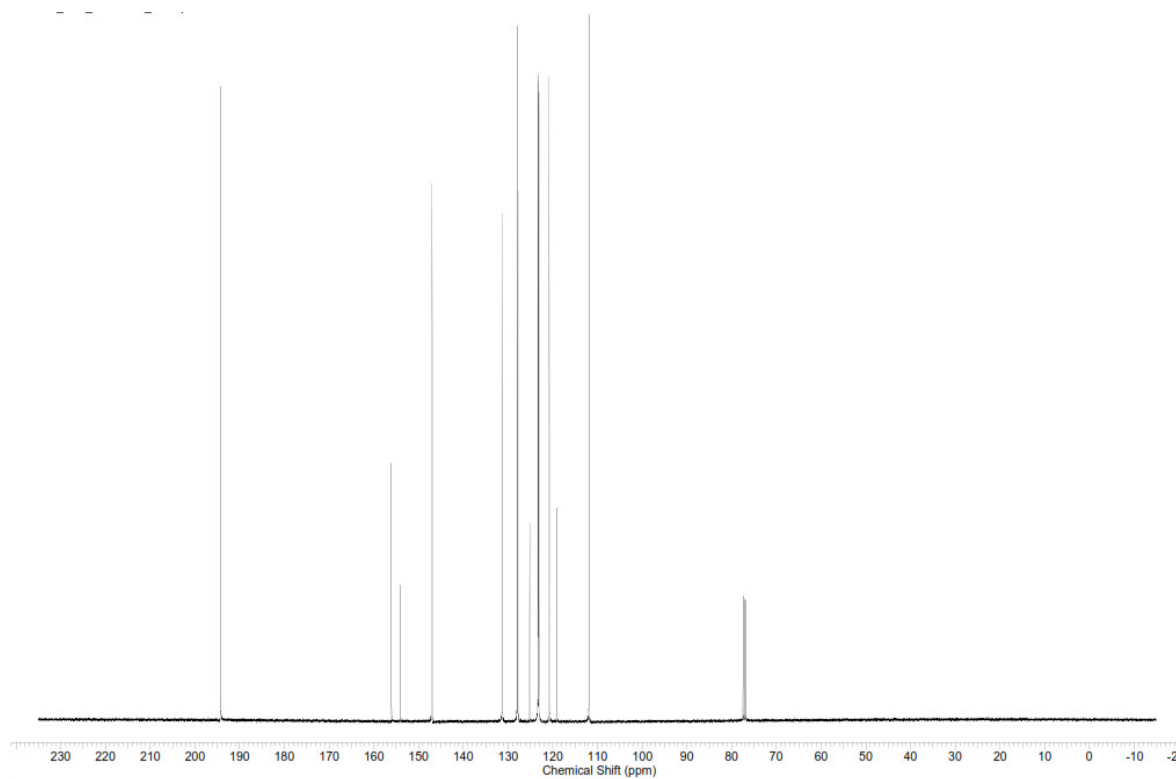
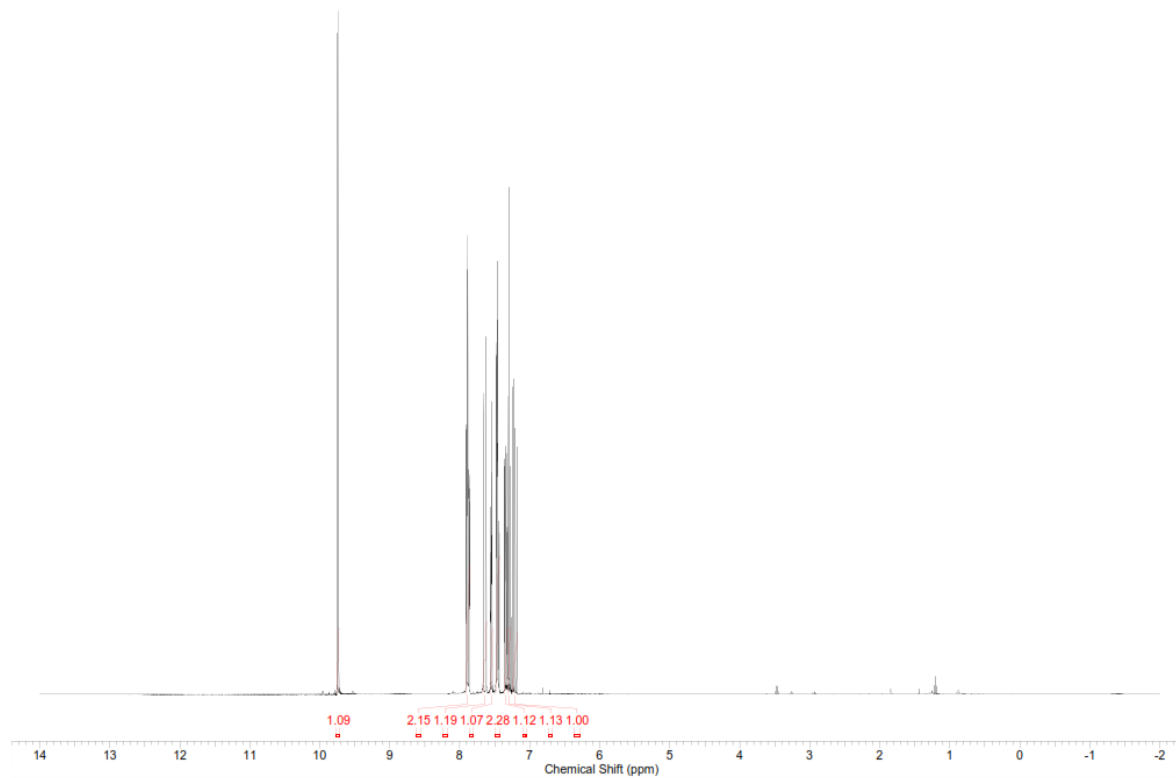




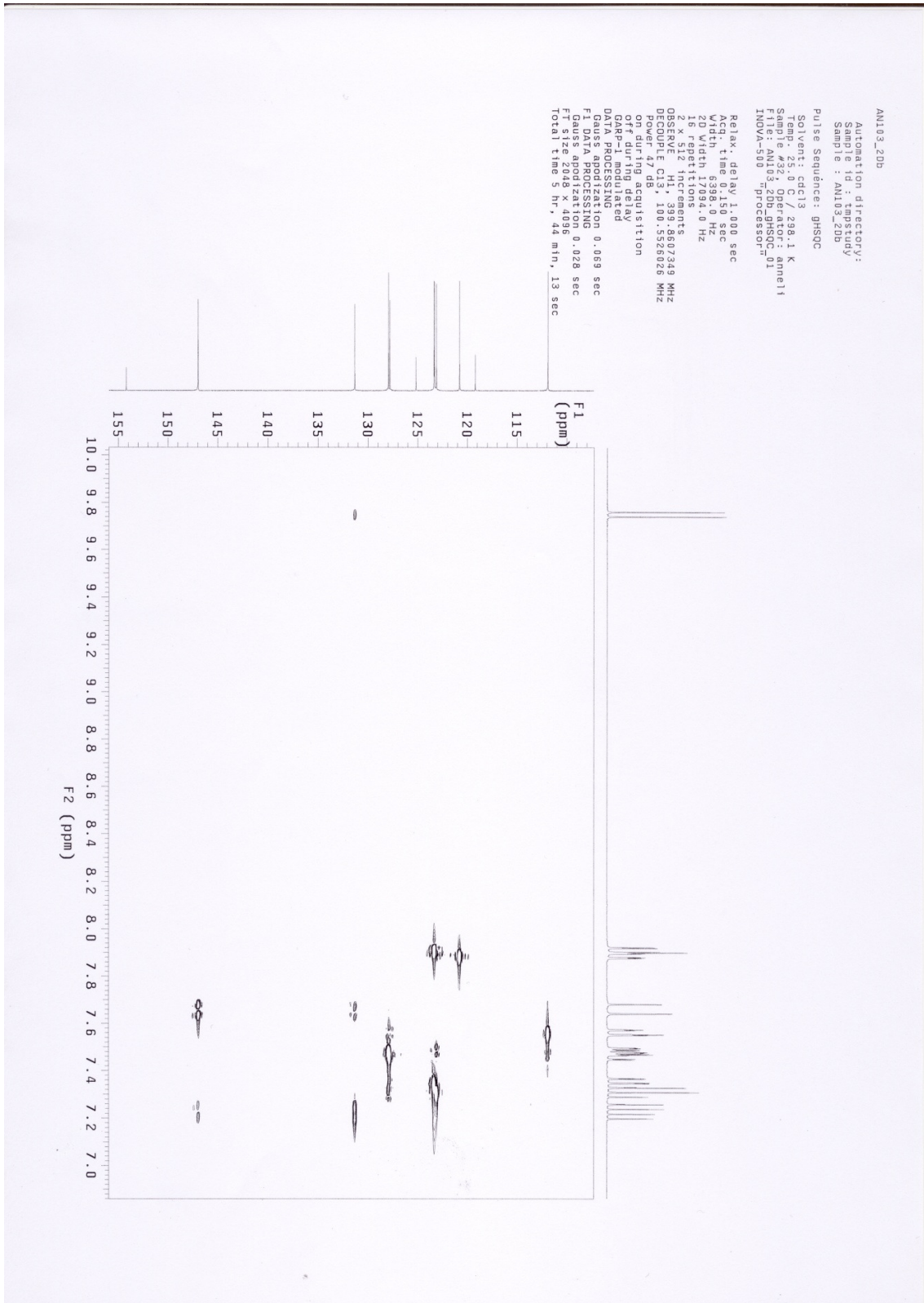
# GC-MS 6h



# $^1\text{H}$ NMR and $^{13}\text{C}$ NMR 6h



# HMBC and HSQC 6h



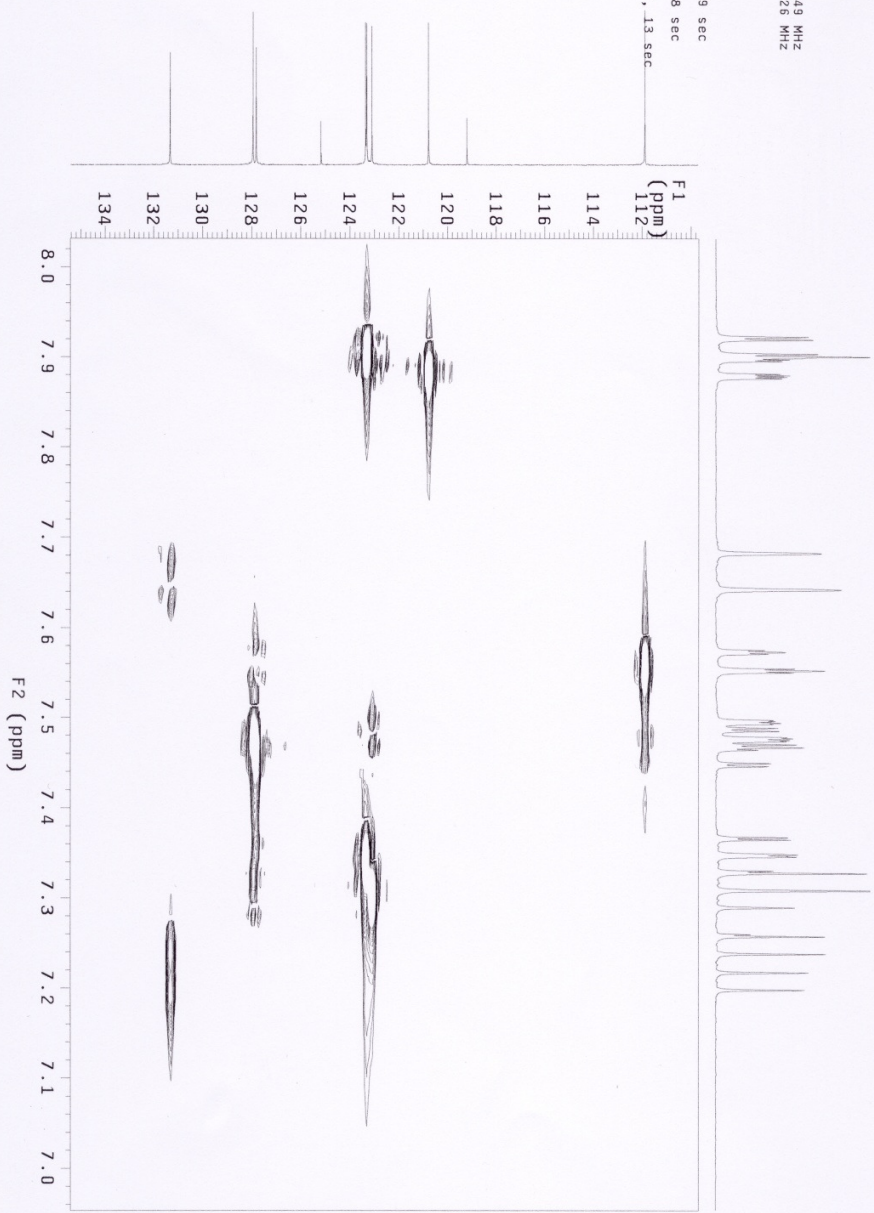
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Sample : AN103\_2DB

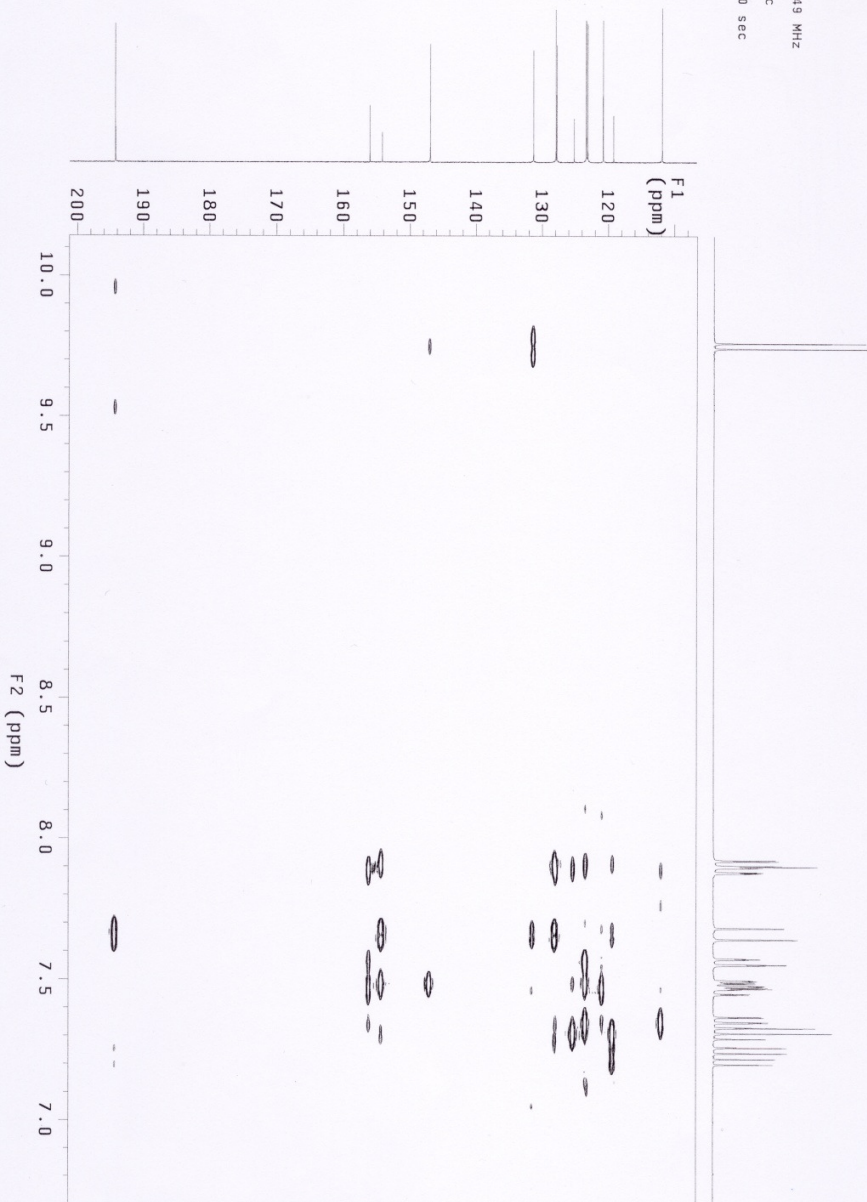
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Solvent: cdc13  
Sample #32 Operator: ampet1  
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INNOVA-500 "processor"

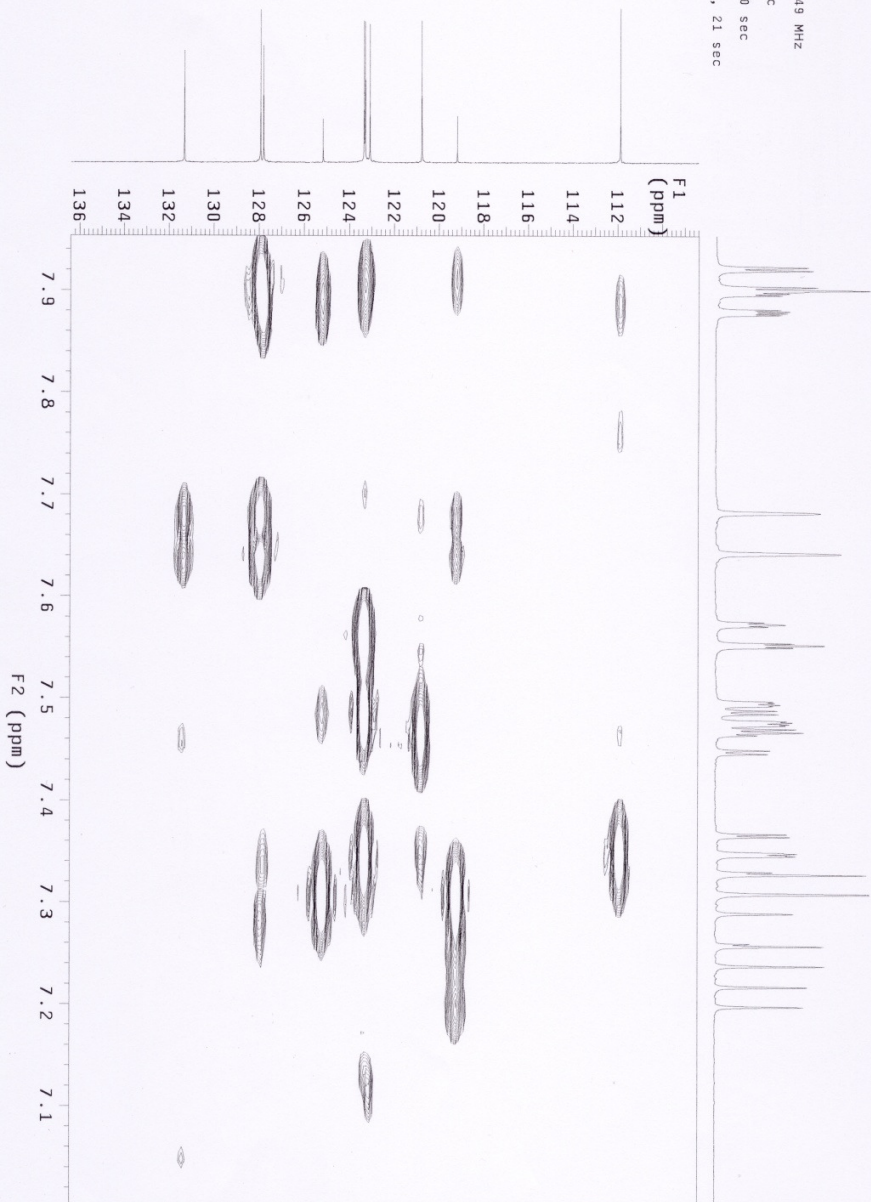
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Acq. time 0.150 sec  
Width 6398.0 Hz  
ZD Width 17094.0 Hz  
ZD Spectroscopy  
ZD Spectroscopy  
OBSERVE H1, 399.8607349 MHz  
DECUPLE C13, 100.5526026 MHz  
Power 47 dB  
on during acquisition  
off during delay  
DATA PROMPTED  
Gauss apodization 0.069 sec  
F1 DATA PROCESSING  
Gauss apodization 0.028 sec  
F1 size 2048 x 4096  
Total time 5 hr, 44 min, 13.3 sec



AN103\_2DB  
 Automation directory:  
 Sample id : tempstudy  
 Sample : AN103\_2DB  
 Pulse Sequence: ghmwbc  
 Solvent: cdcl3  
 Temp: 25.0 C / 298.1 K  
 Sample #32, Operator: annel1  
 File: AN103\_2DB\_ghmwb01  
 INOVA-500 "processor"  
 Relax. delay 1.000 sec  
 Acq. time 0.150 sec  
 Width 6398.0 Hz  
 F2 Width 4332.7 Hz  
 F2 Offset 140.0 Hz  
 2 x 512 increments  
 OBSERVE H1, 399.8607349 MHz  
 DATA PROCESSING  
 Sg, sine bell 0.075 sec  
 F1 DATA PROCESSING 0.020 sec  
 F2 gauss approximation 0.020 sec  
 F2 phase adjustment 0.020 sec  
 Total time 0 min, 9 sec



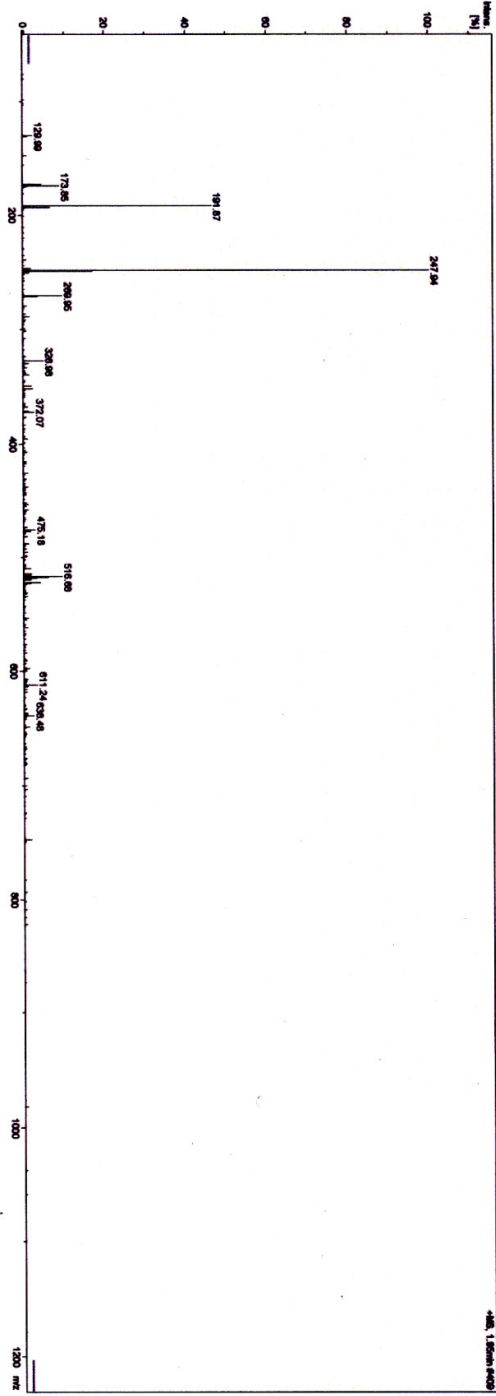
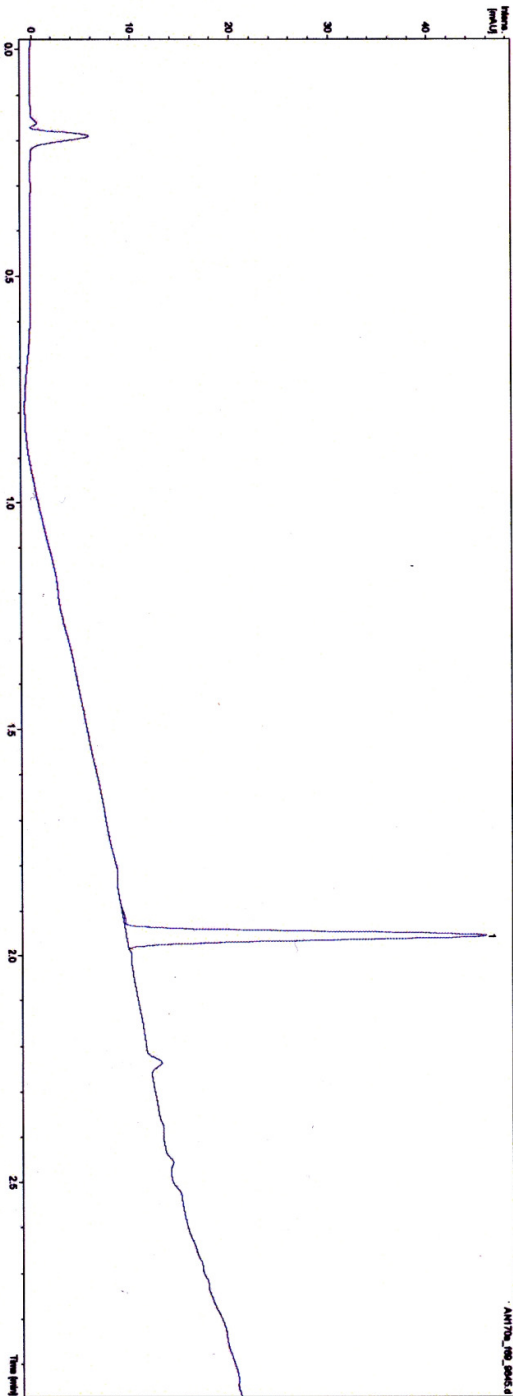
AN103\_2DB  
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 Sample ID: AN103\_2DB  
 Sample : AN103\_2DB  
 Pulse Sequence: ghm3c  
 Solvent: cdcl3  
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 Sample #32, Operator: anneti  
 File: AN103\_2DB\_ghm3c\_01  
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 Relax. delay 1.000 sec  
 Acq. time 0.150 sec  
 F2 (ppm) 6392.7  
 Width 6392.7 Hz  
 16 repetitions  
 2 x 512 increments  
 OBSERVE H1, 399.8607349 MHz  
 DATA PROCESSING  
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 F2 DATA PROCESSING 0.020 sec  
 F1 DATA PROCESSING 0.020 sec  
 FT size 2048 x 4096  
 Total time 5 hr, 53 min, 21 sec



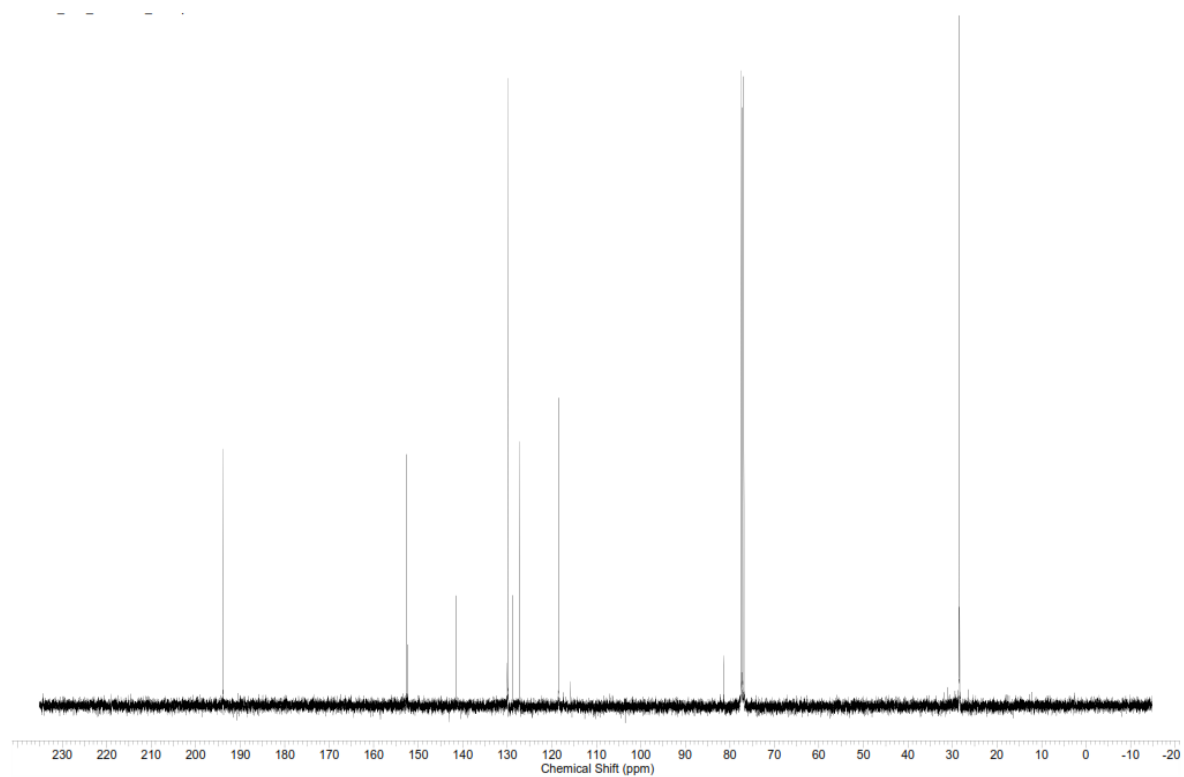
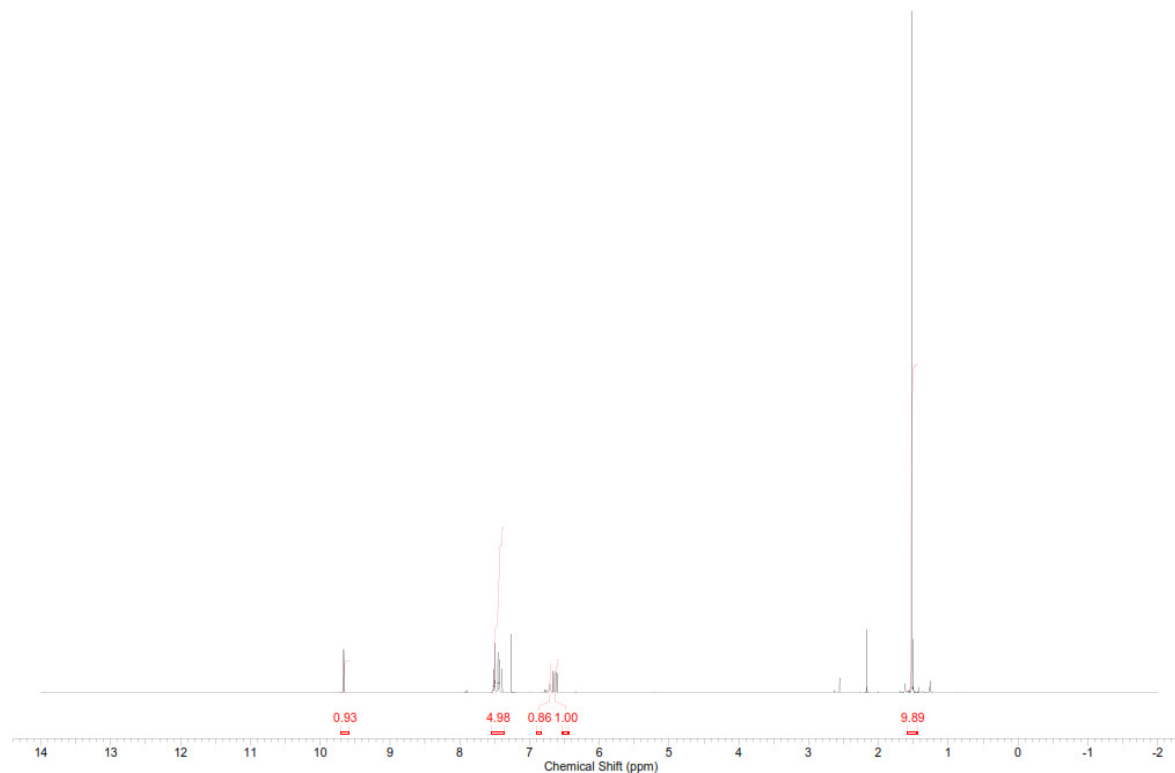
# HPLC-MS 6i

## Compound List

Cmpd. Label	Area Frac. %
Cmpd 1, 1.95 min	98.2
Cmpd 2, 2.24 min	1.8

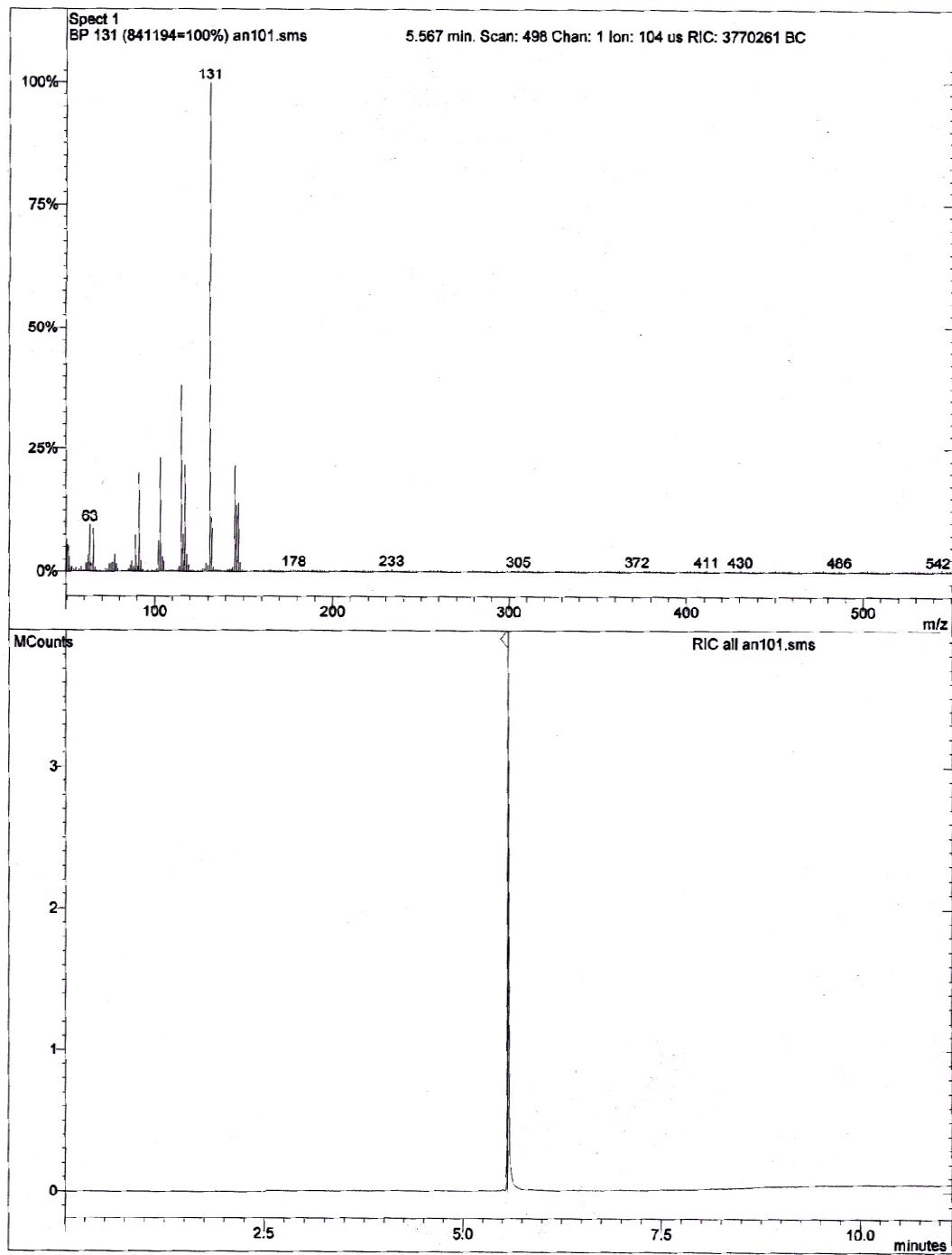


# $^1\text{H}$ NMR and $^{13}\text{C}$ NMR 6i

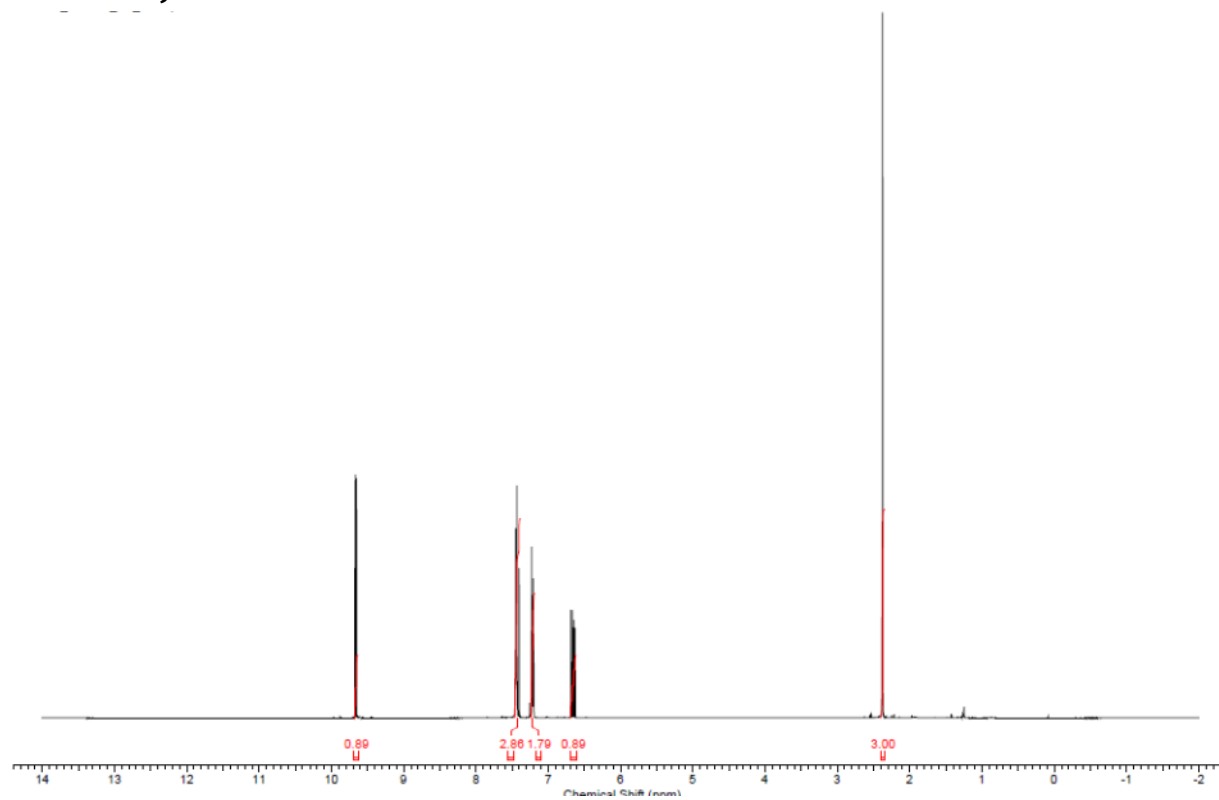




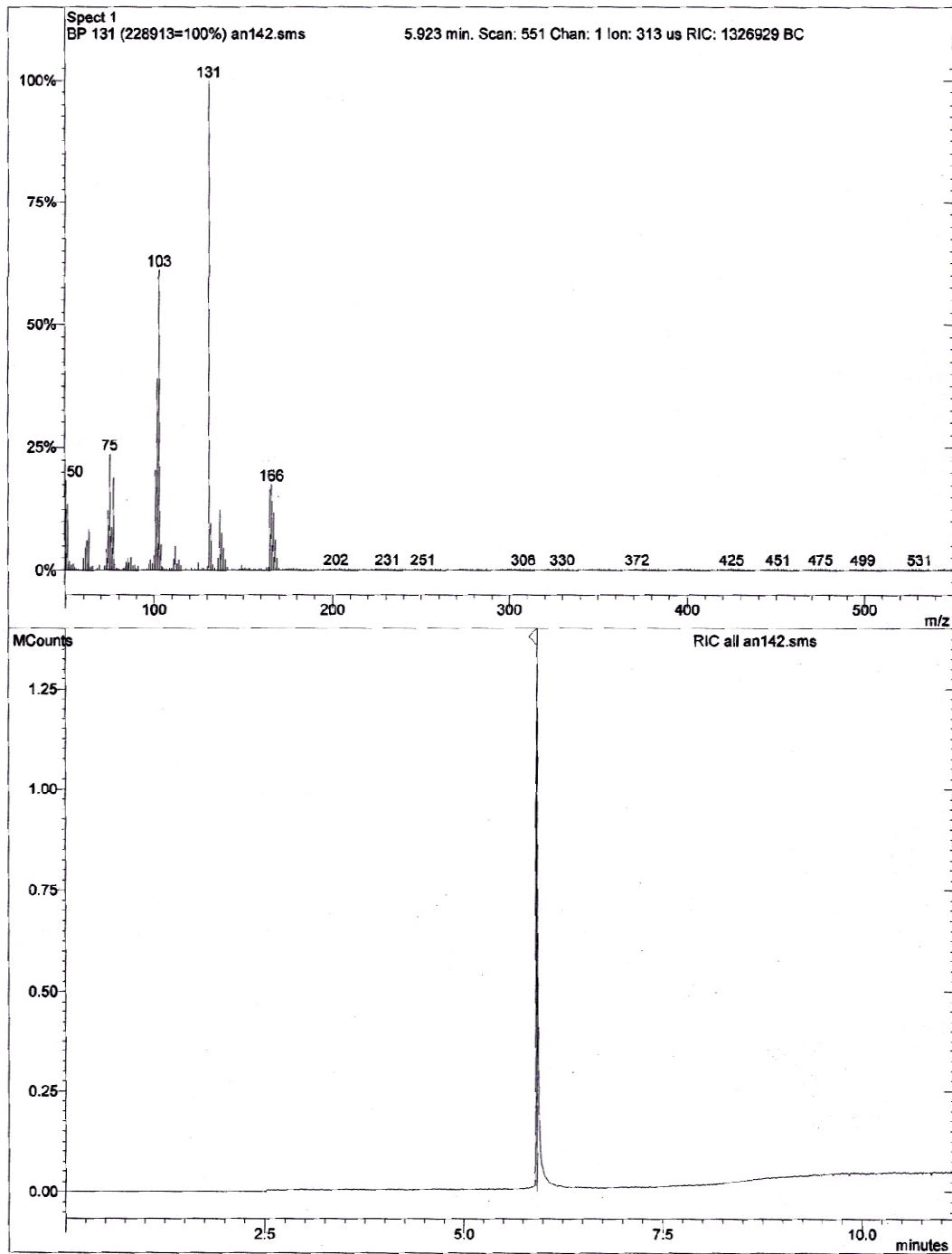
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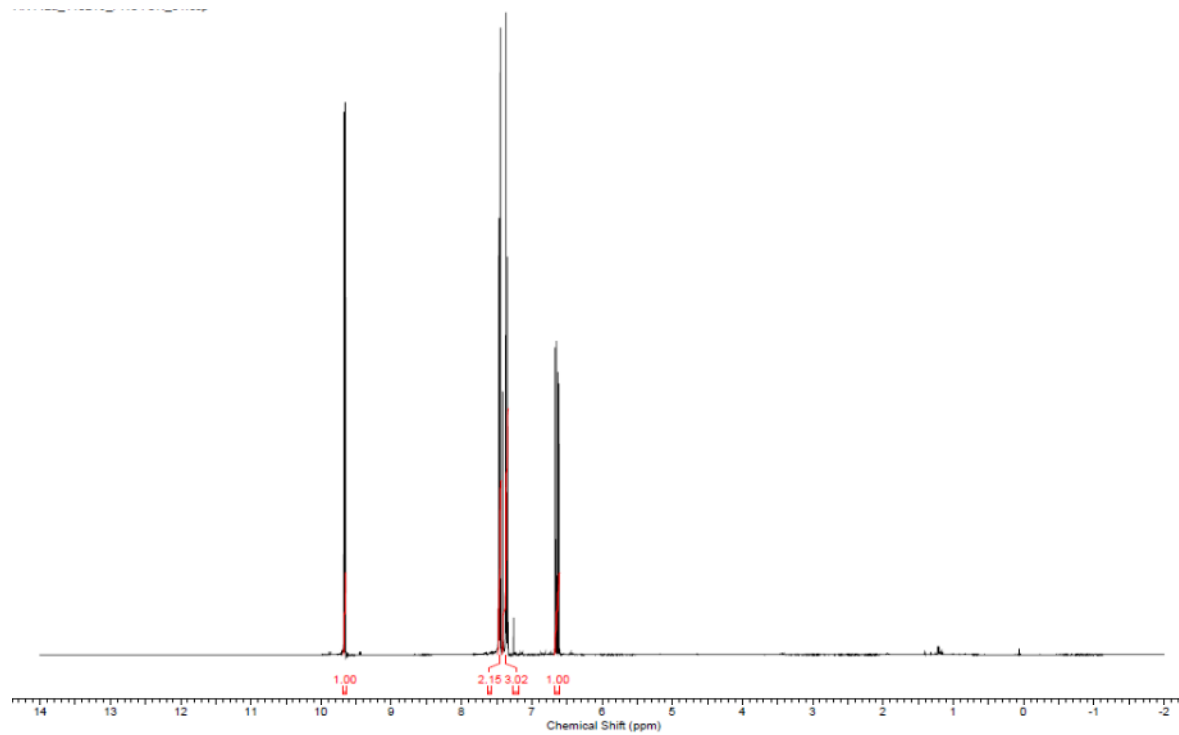
# **<sup>1</sup>H NMR 6j**



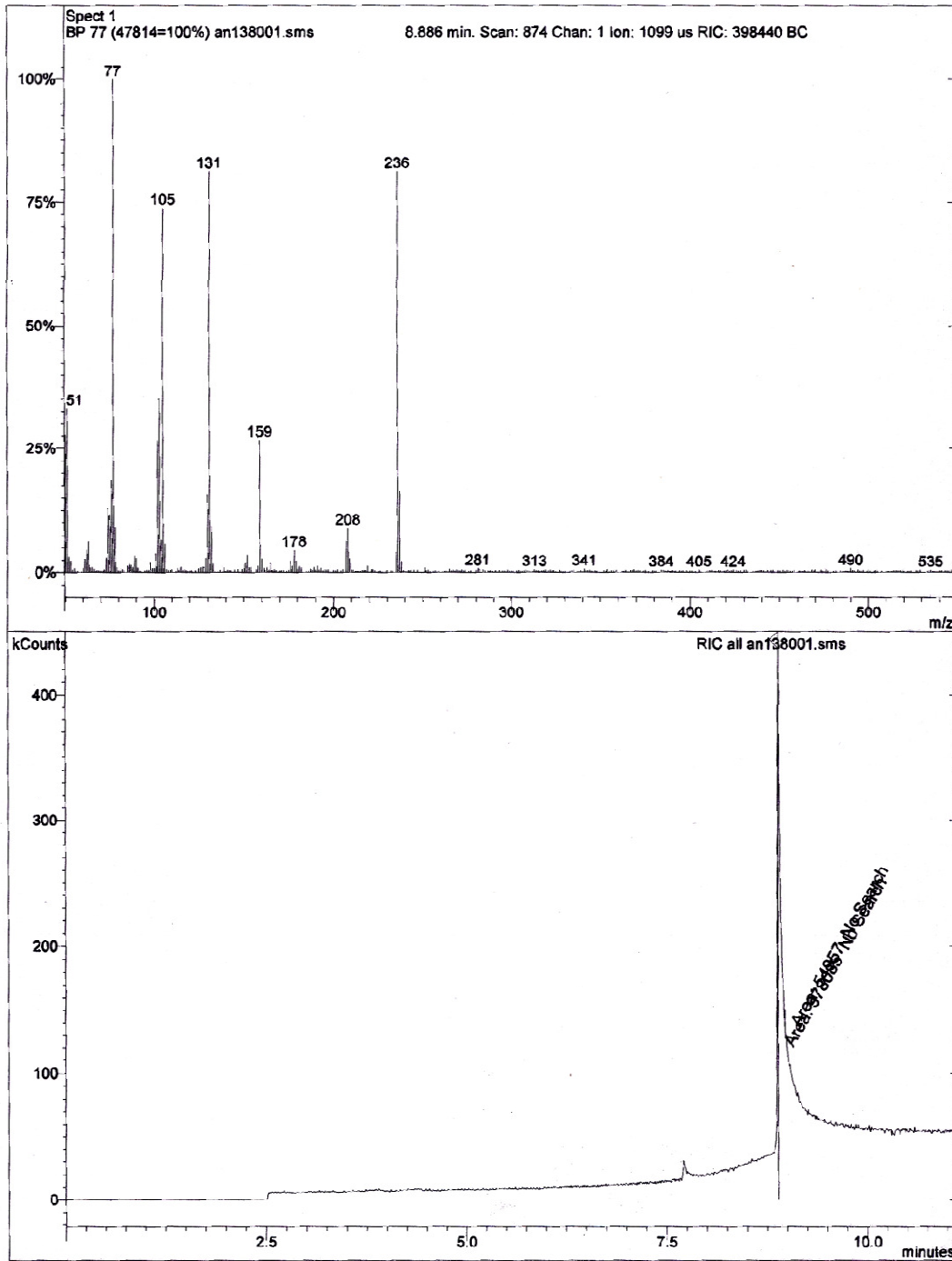
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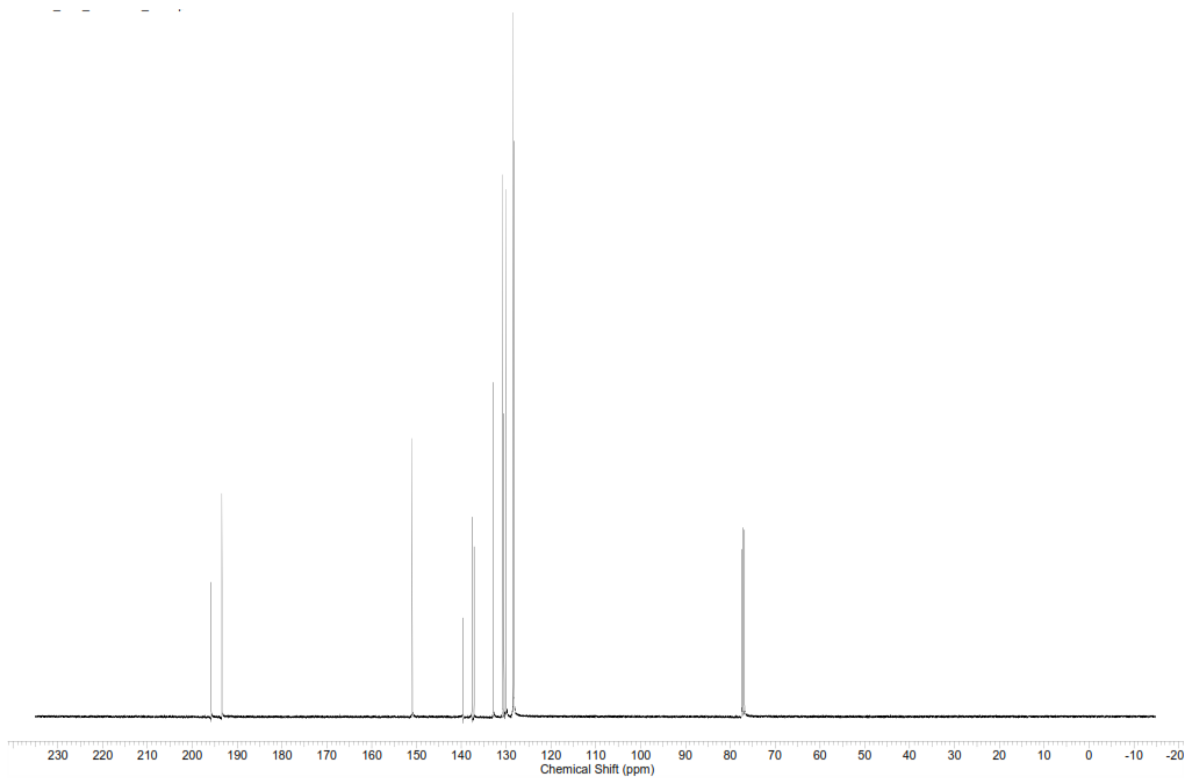
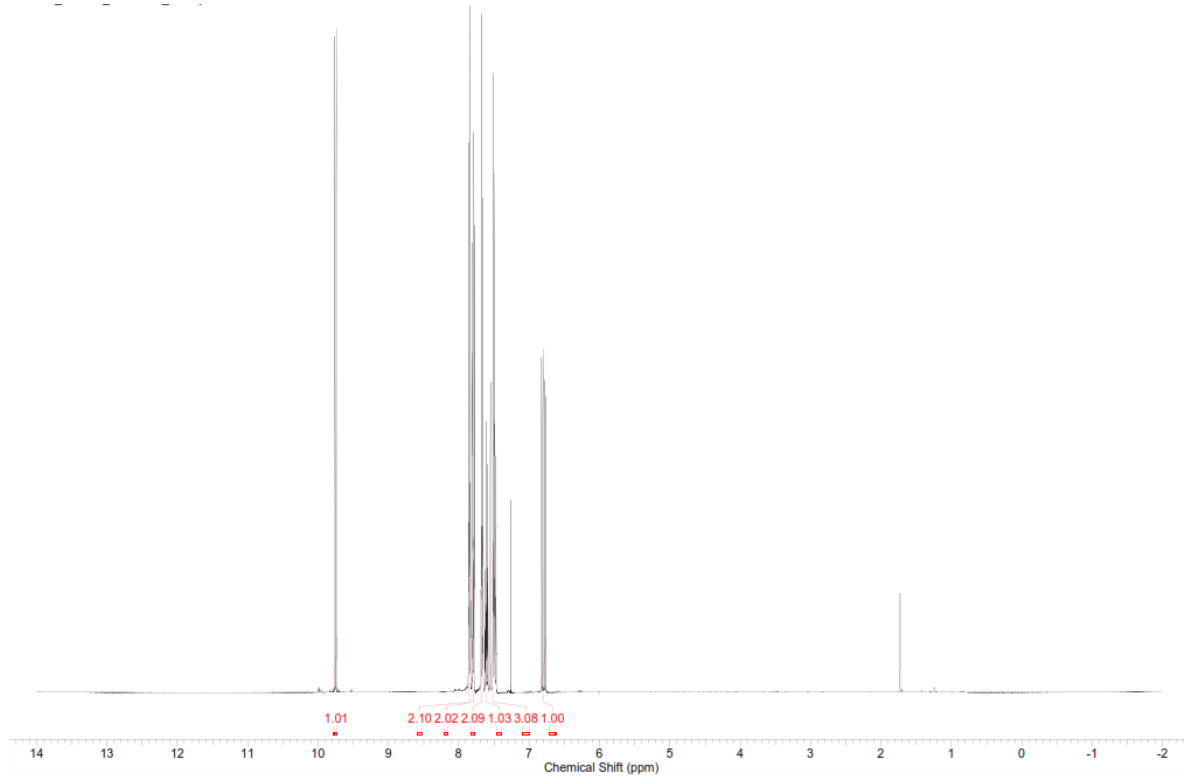
# <sup>1</sup>H NMR 6k



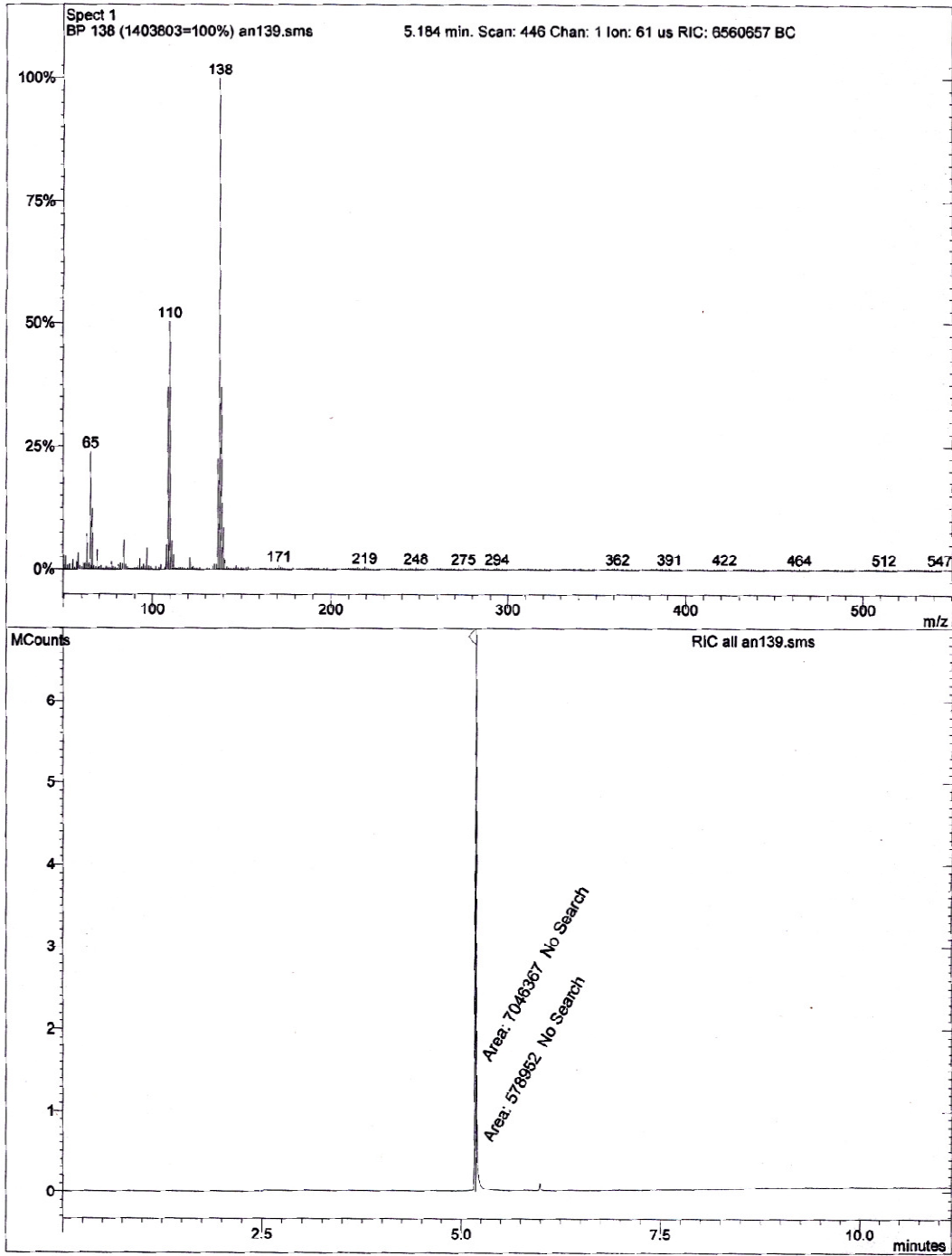
# GC-MS 6I



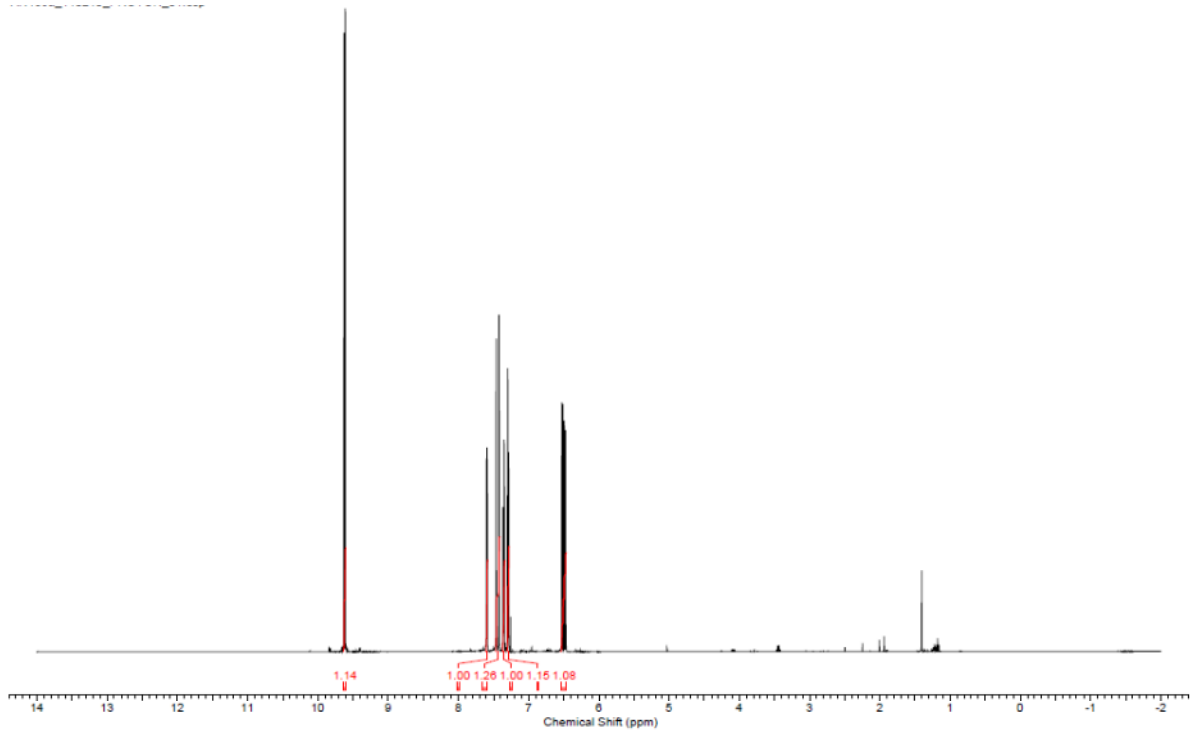
# $^1\text{H}$ NMR and $^{13}\text{C}$ NMR 6I



# GC-MS 6m

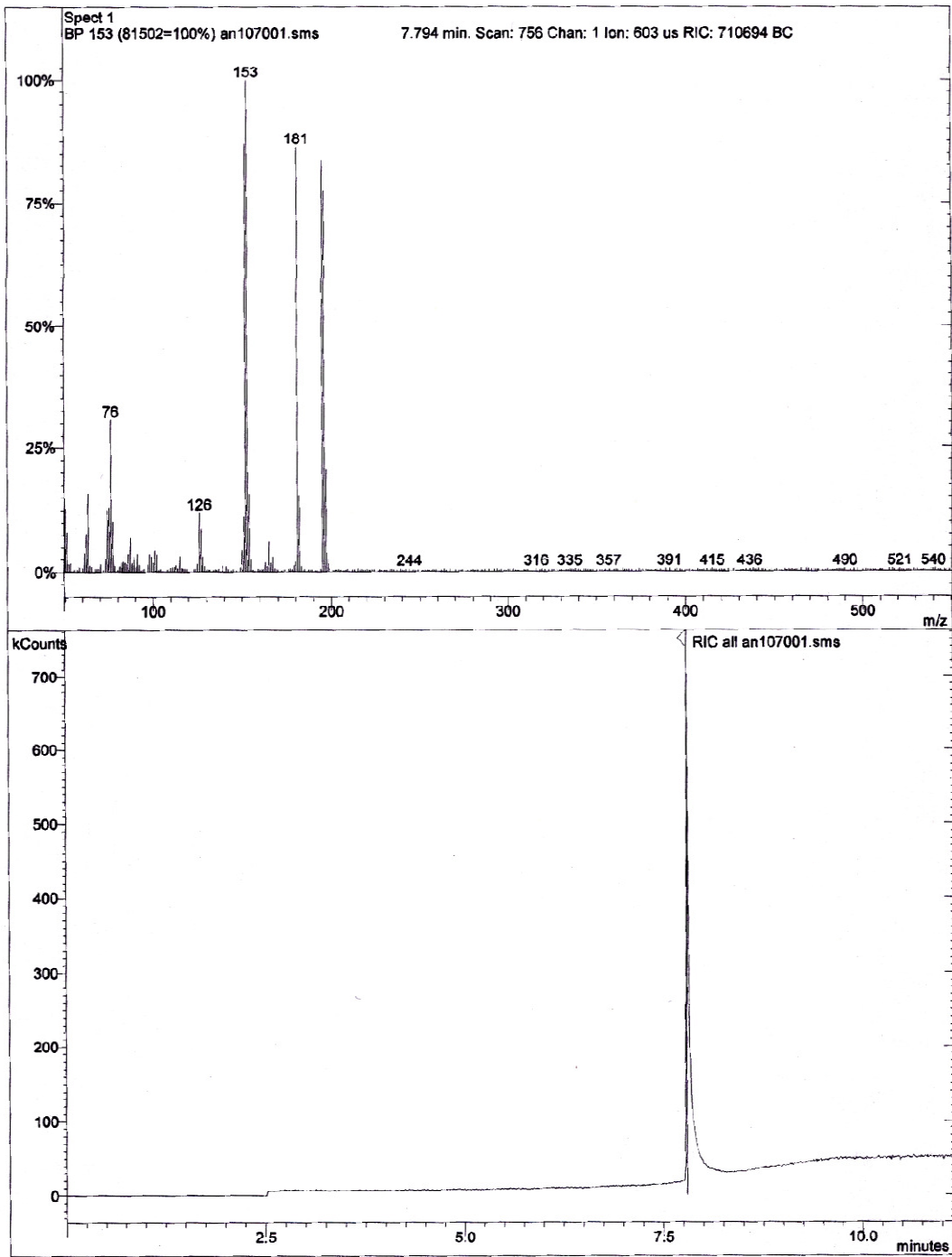


**$^1\text{H}$  NMR 6m**

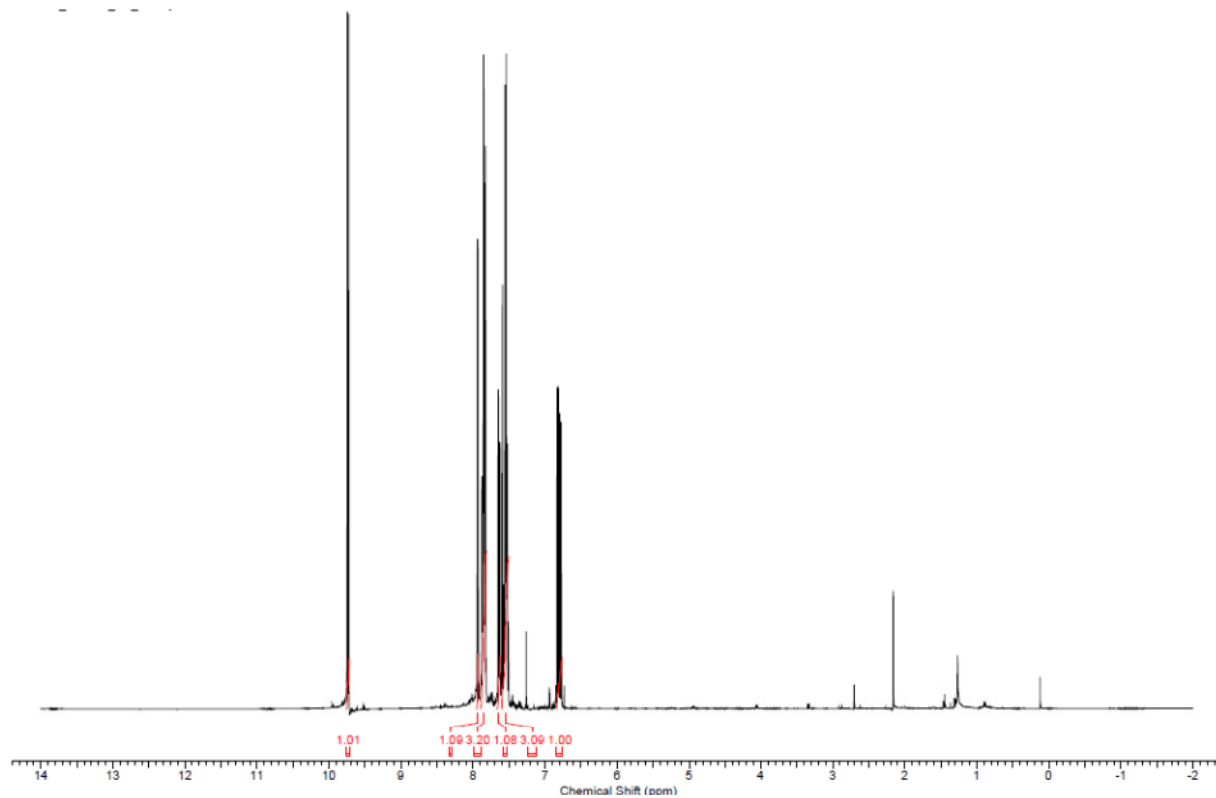




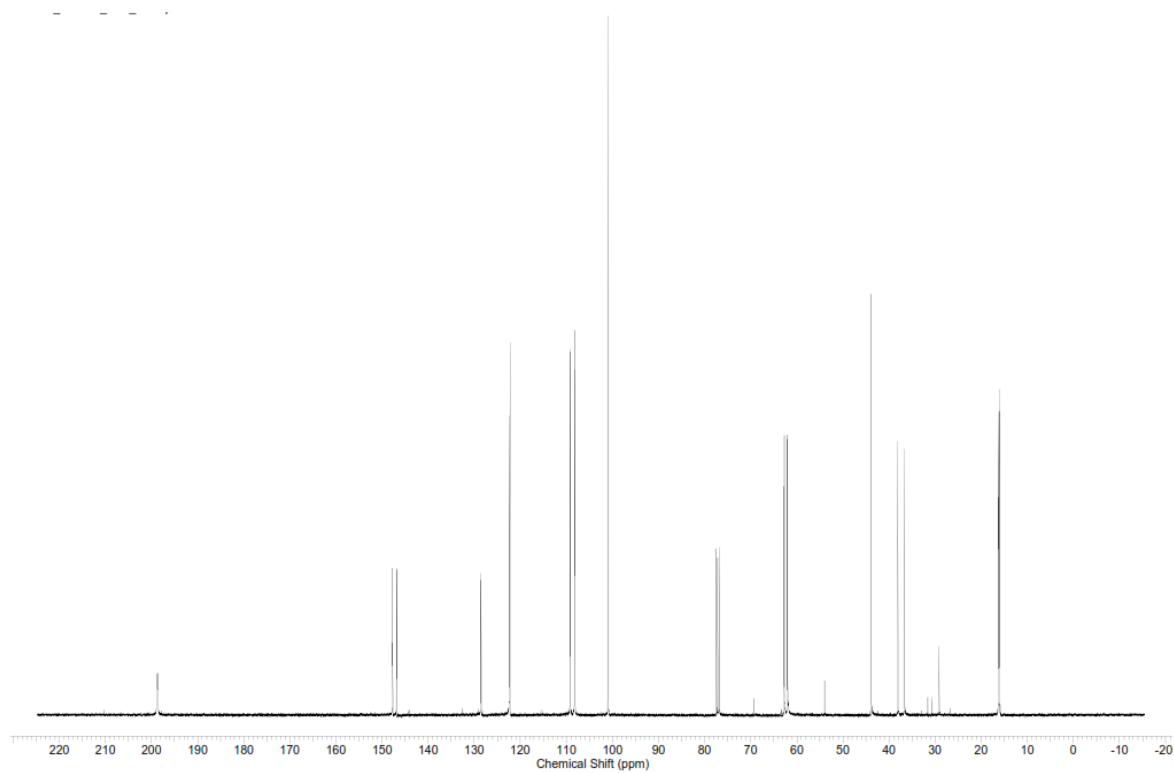
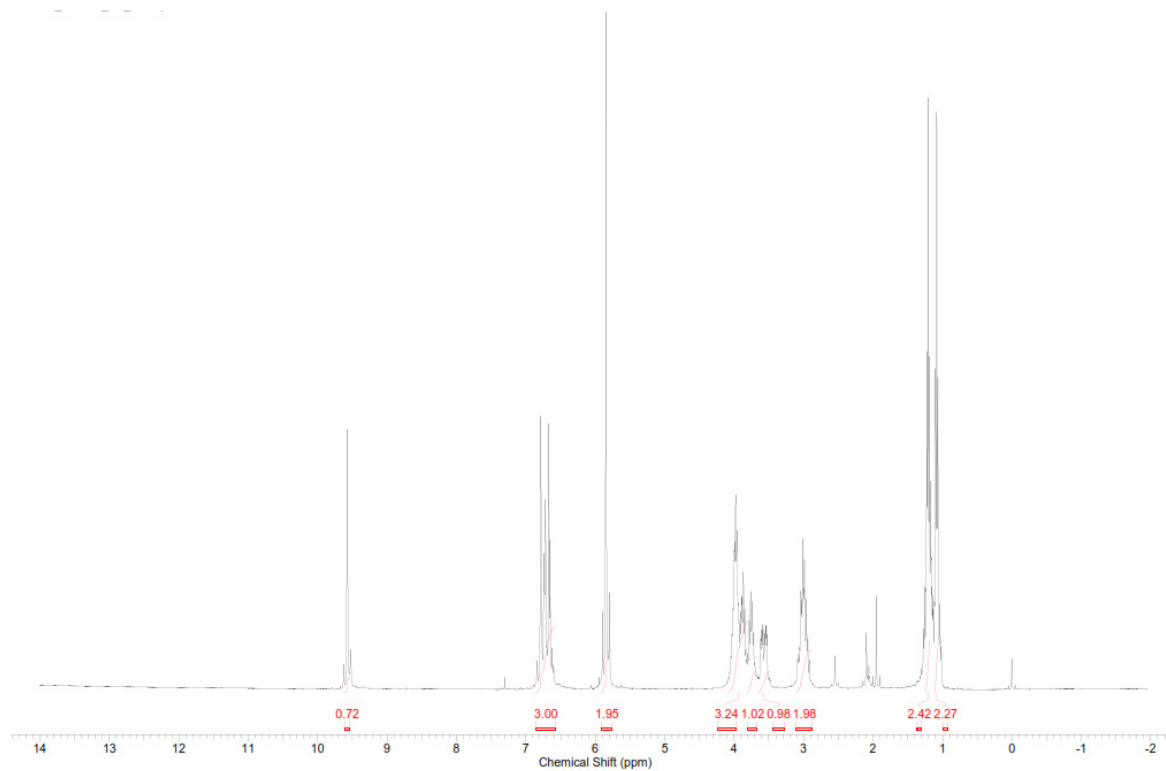
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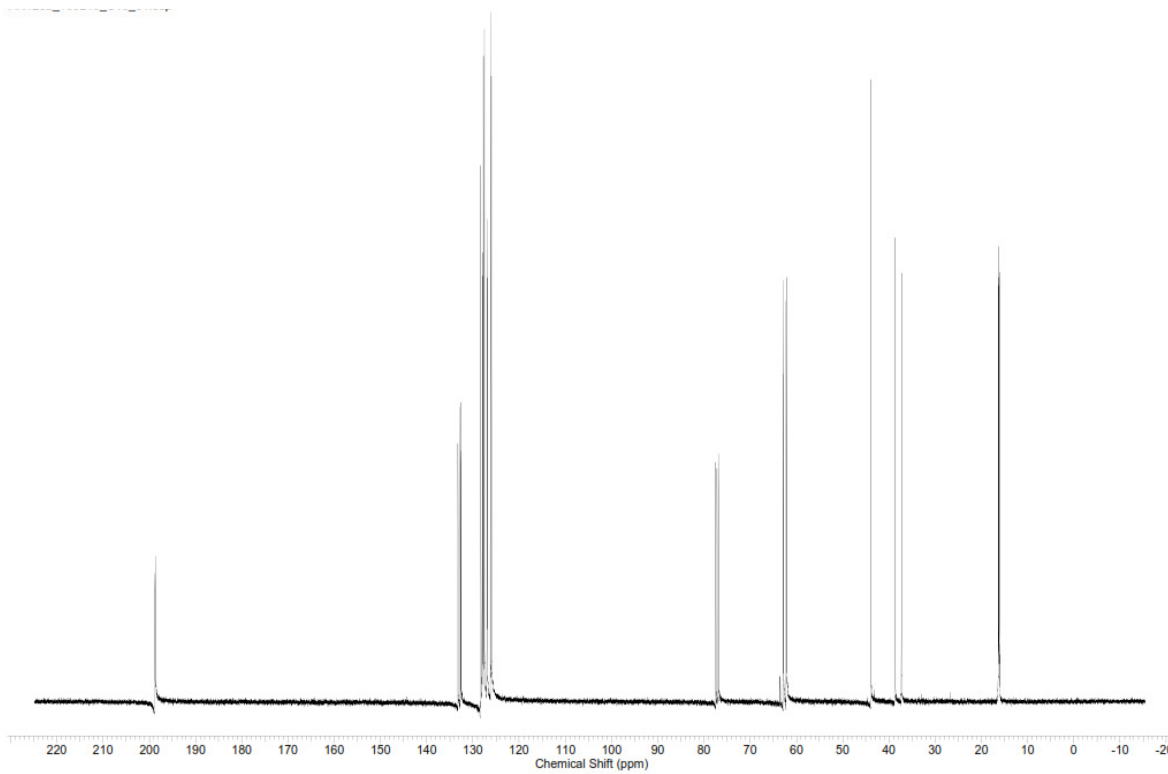
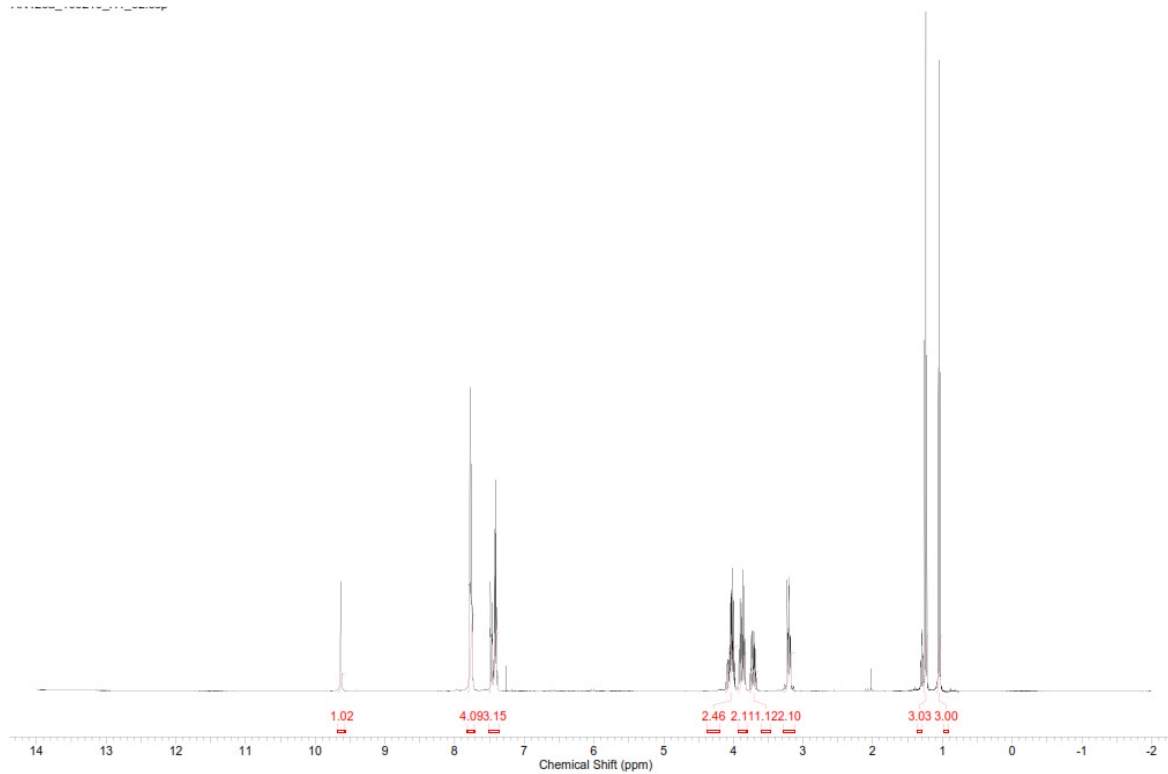
# **<sup>1</sup>H NMR 6n**



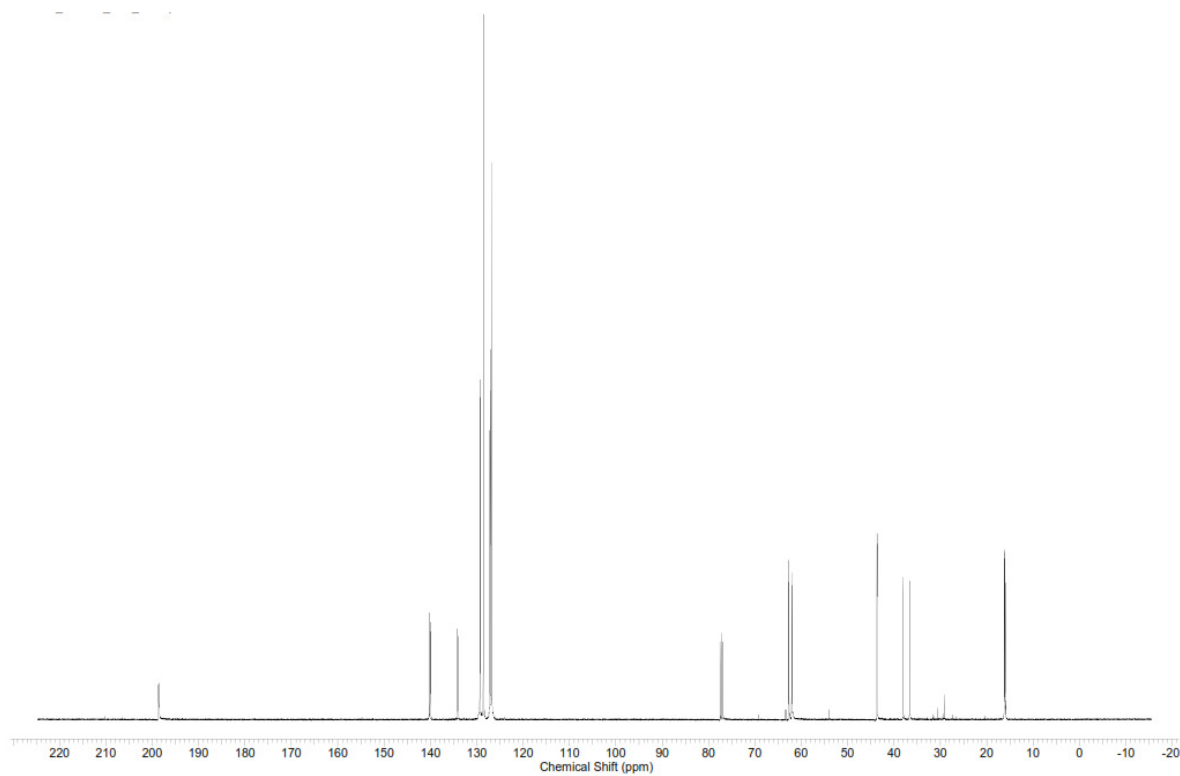
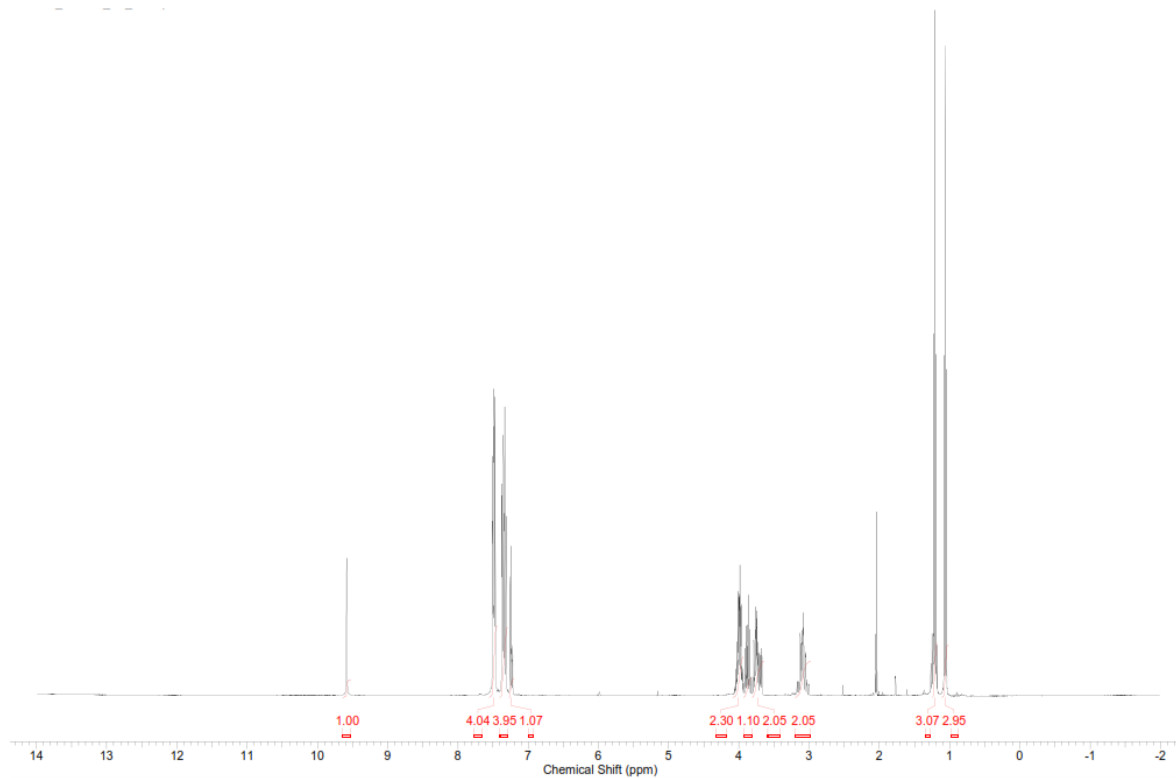
# $^1\text{H}$ NMR and $^{13}\text{C}$ NMR 10a



# <sup>1</sup>H NMR and <sup>13</sup>C NMR 10b

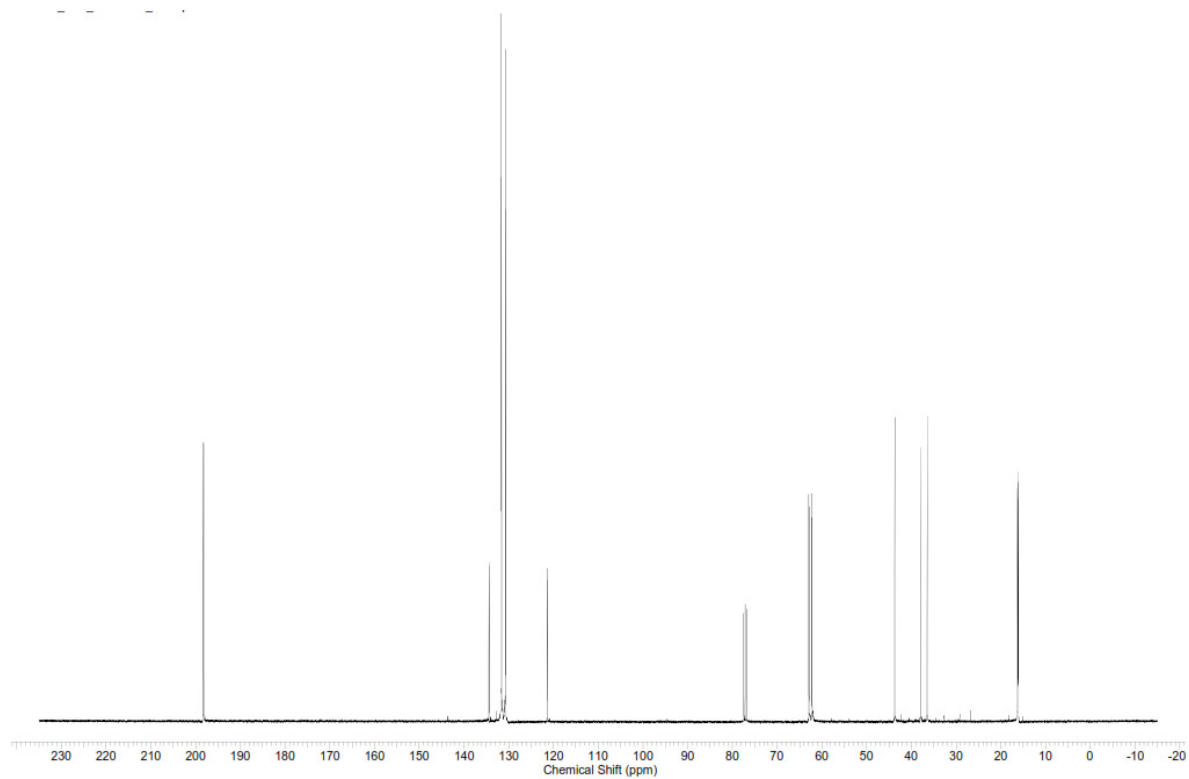
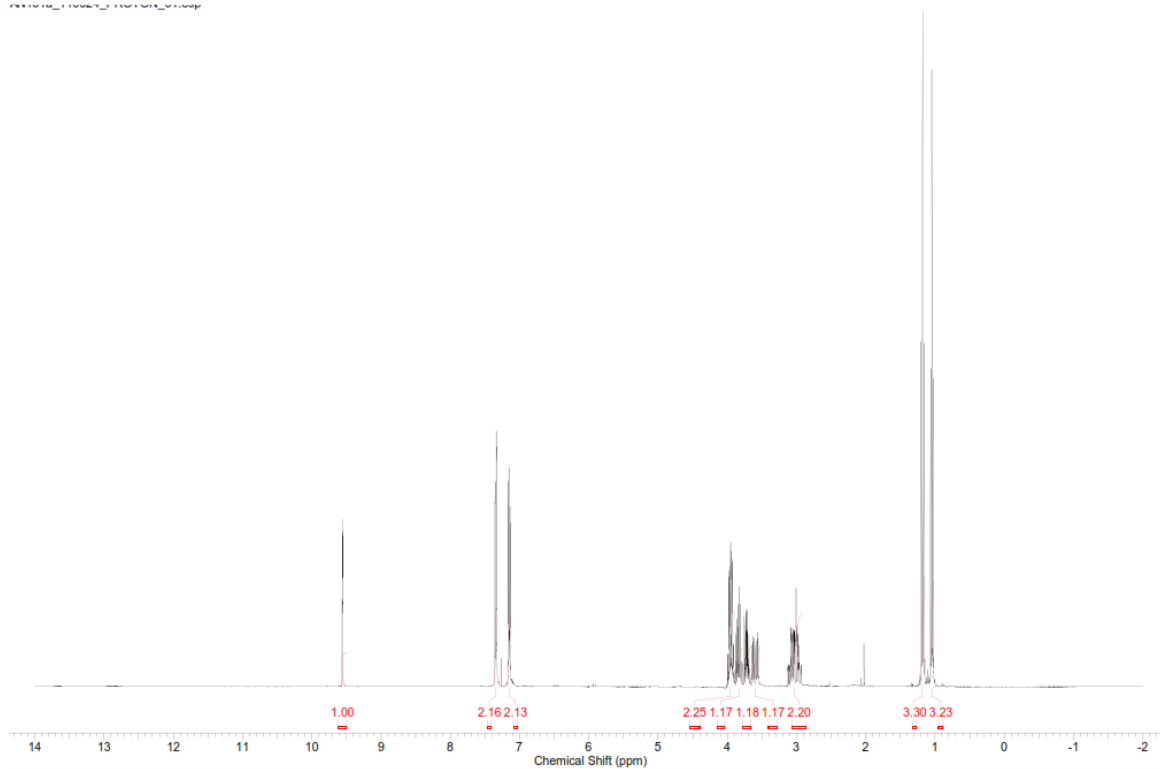


# $^1\text{H}$ NMR and $^{13}\text{C}$ NMR 10c

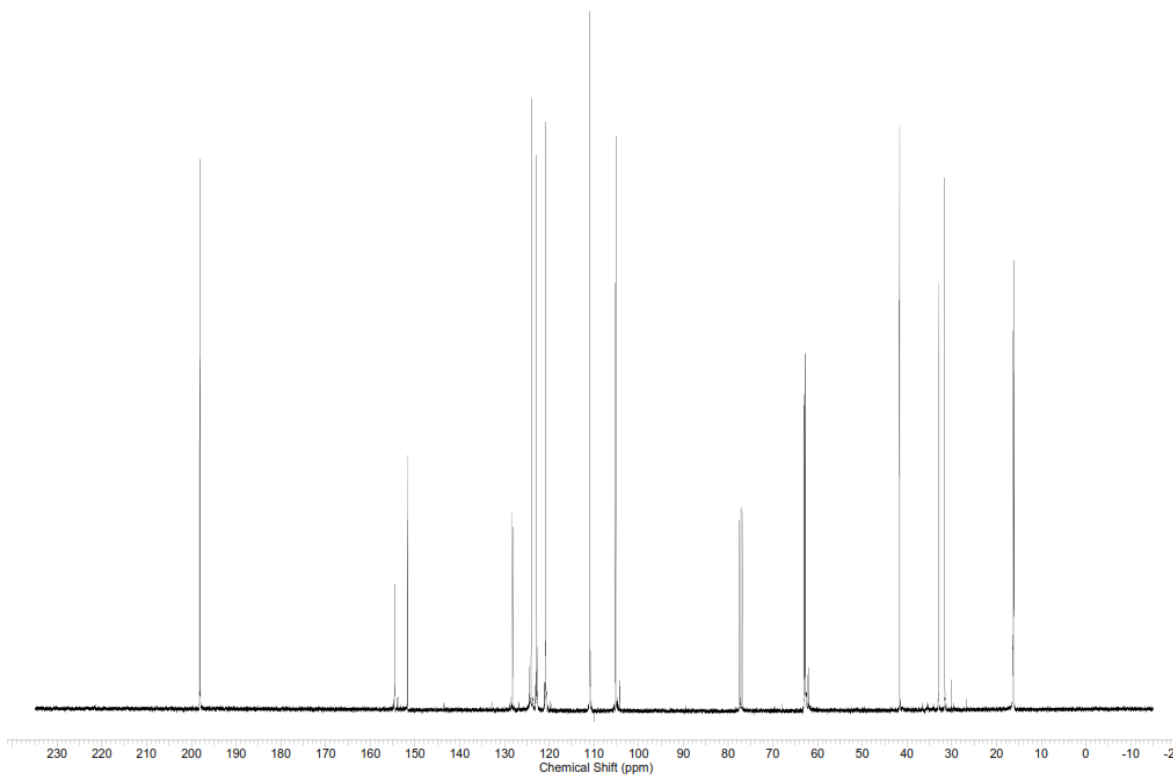
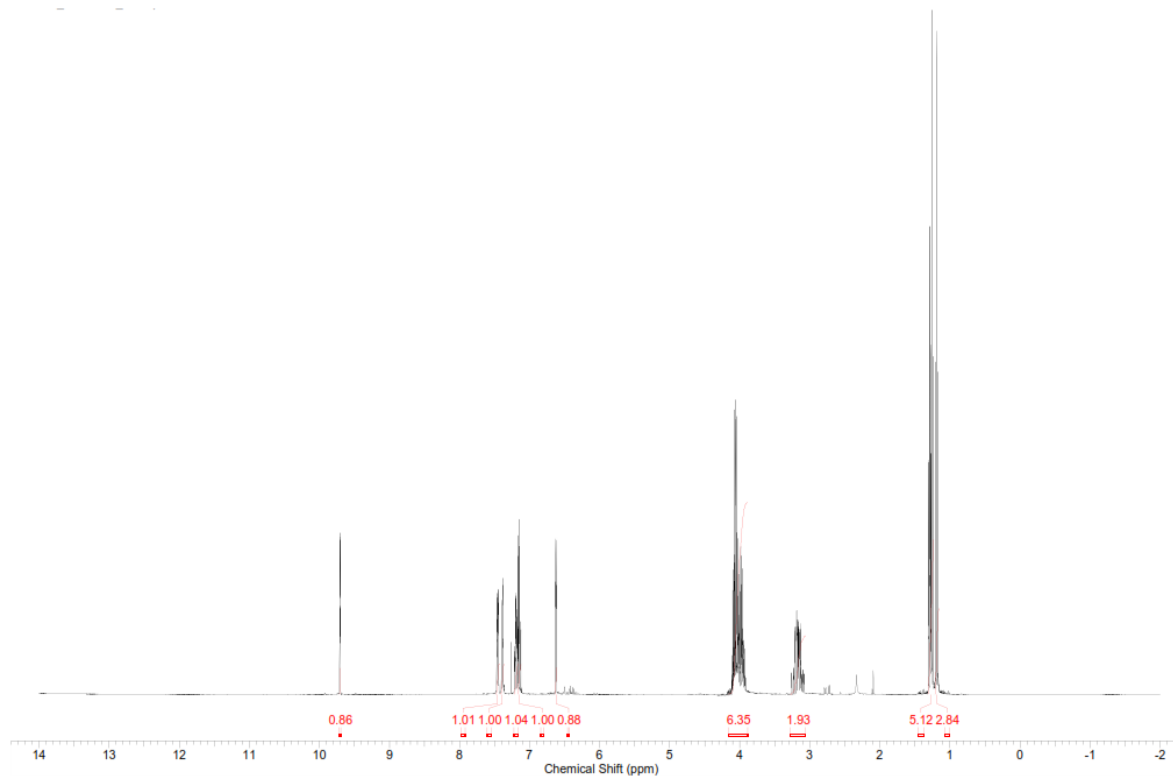


# <sup>1</sup>H NMR and <sup>13</sup>C NMR 10d

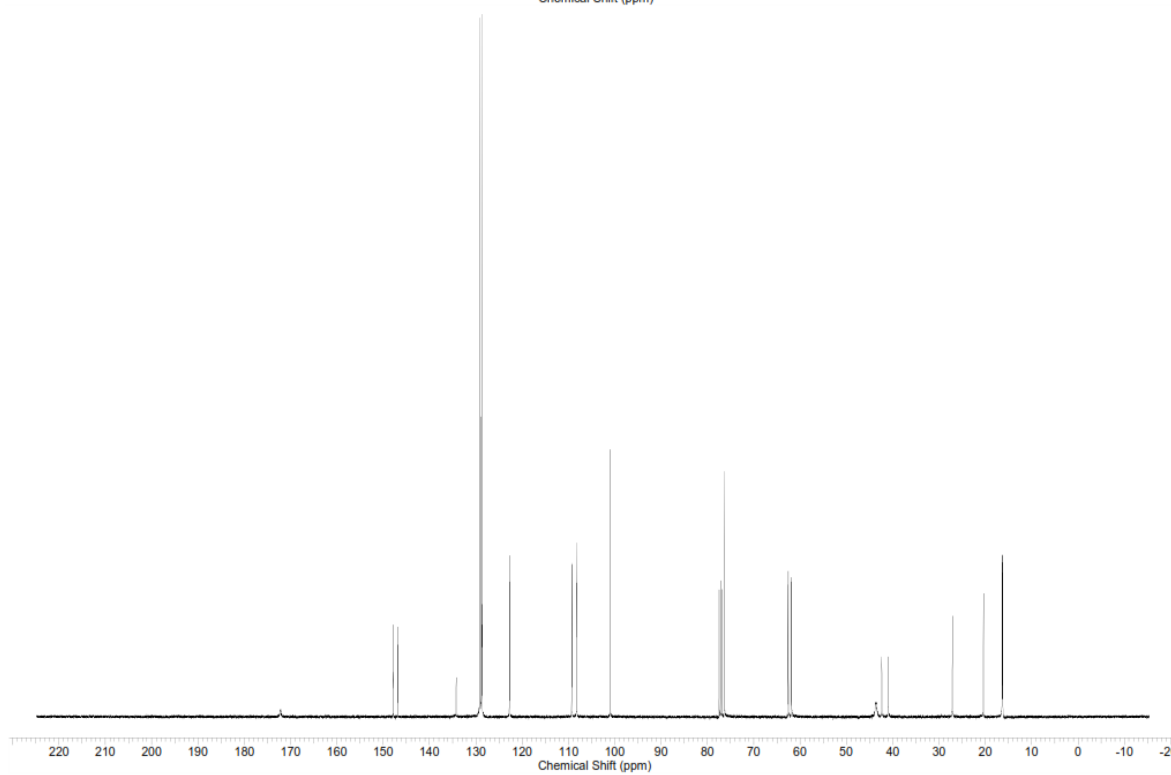
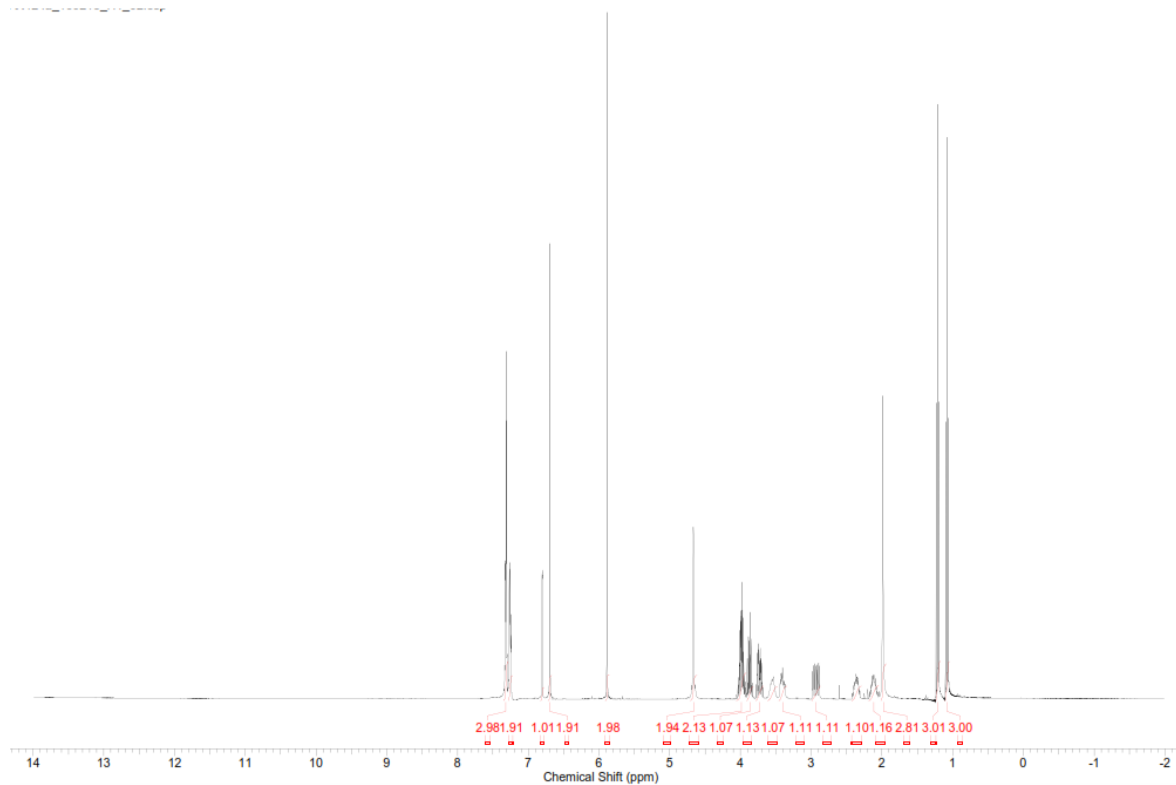
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# $^1\text{H}$ NMR and $^{13}\text{C}$ NMR 10e

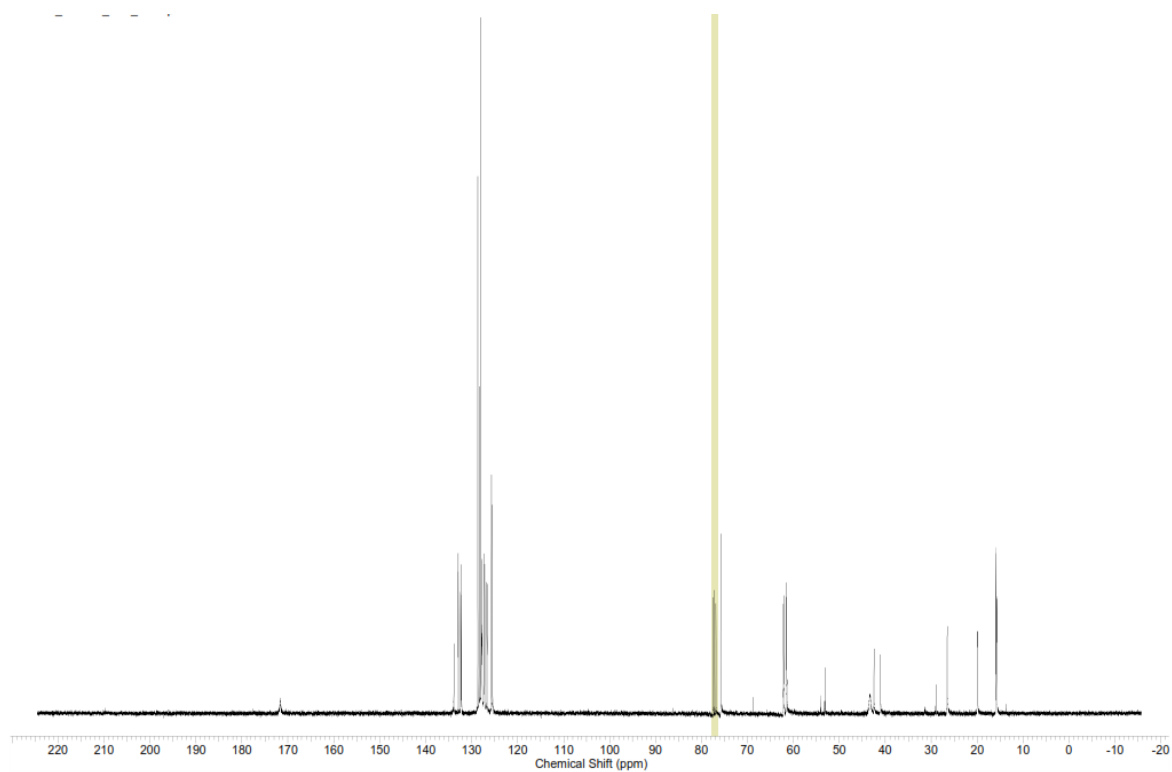
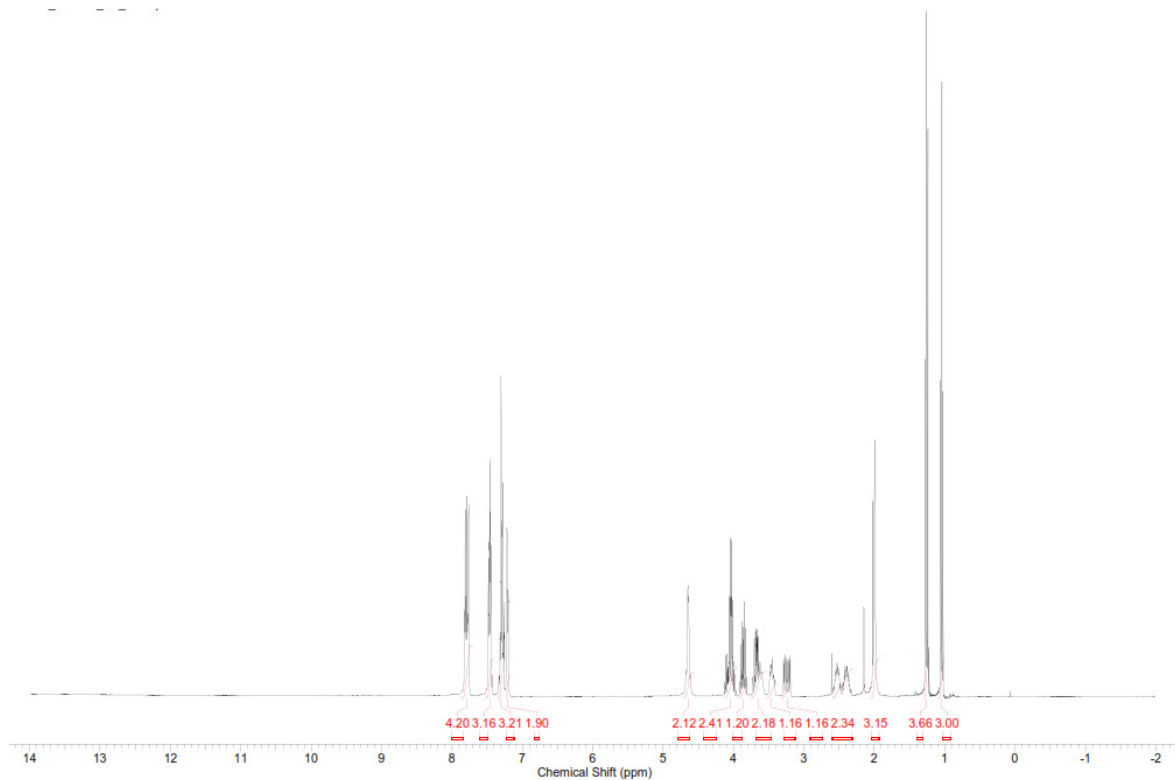


# $^1\text{H}$ NMR and $^{13}\text{C}$ NMR 11a

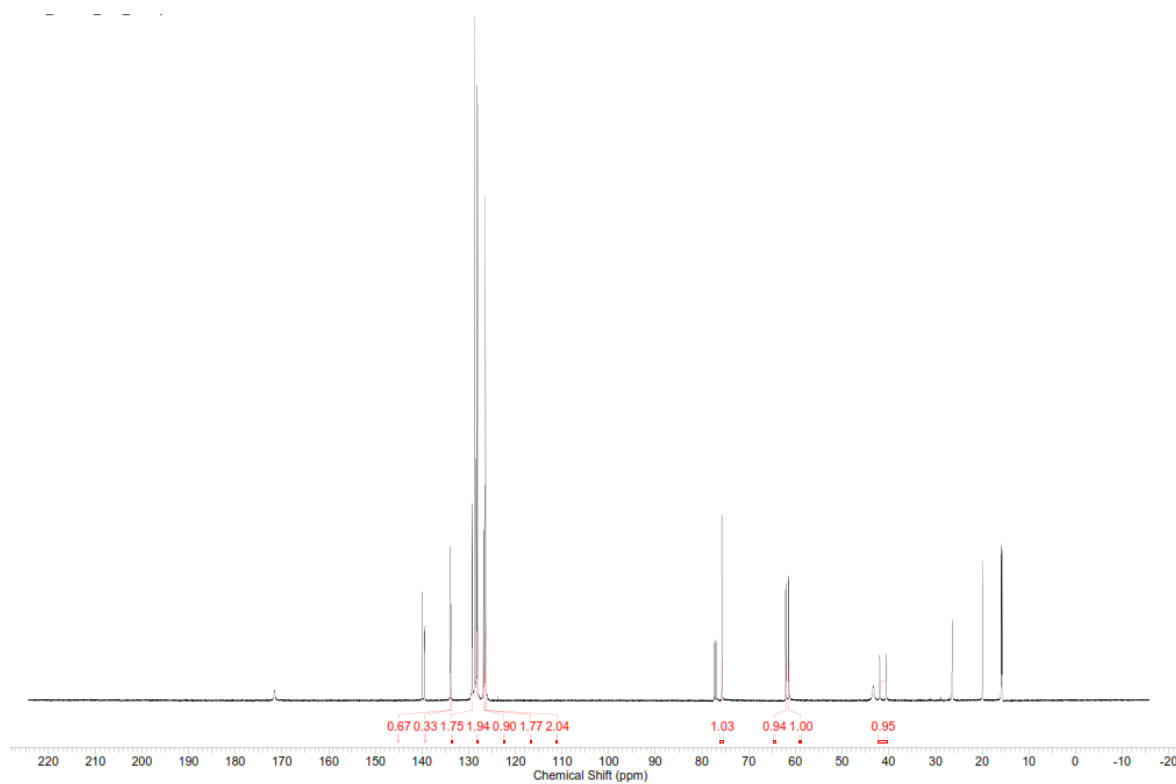
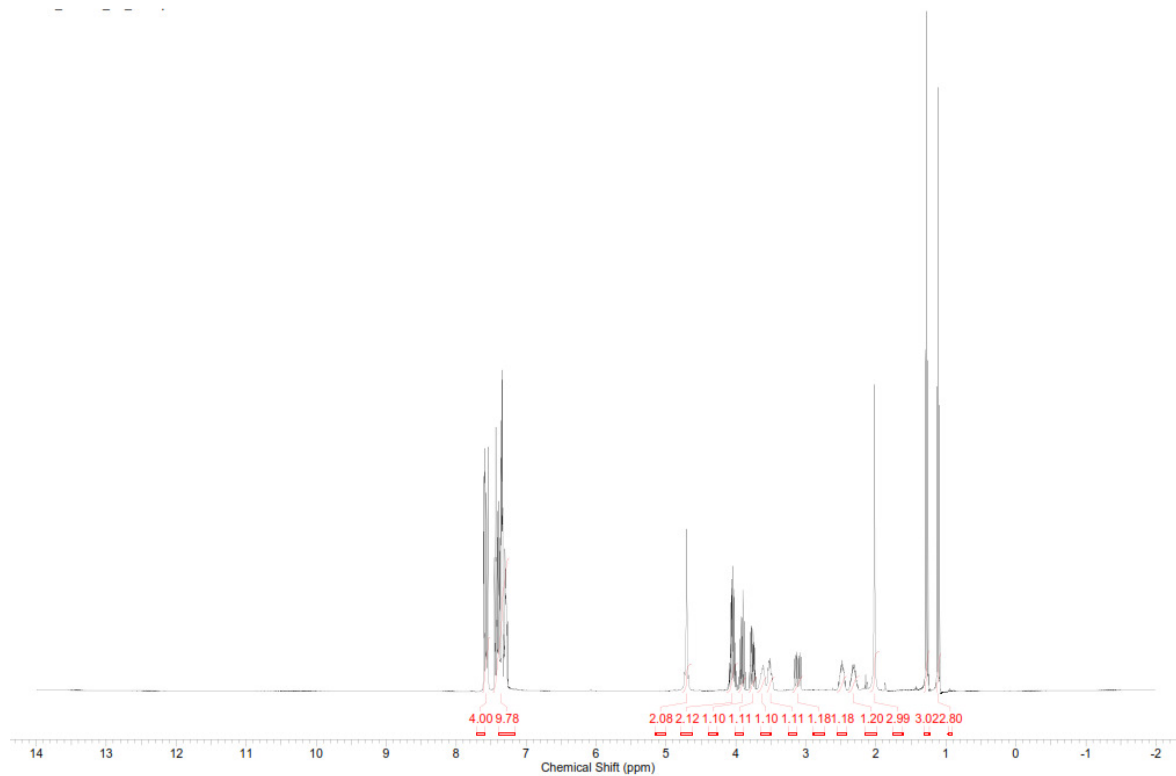




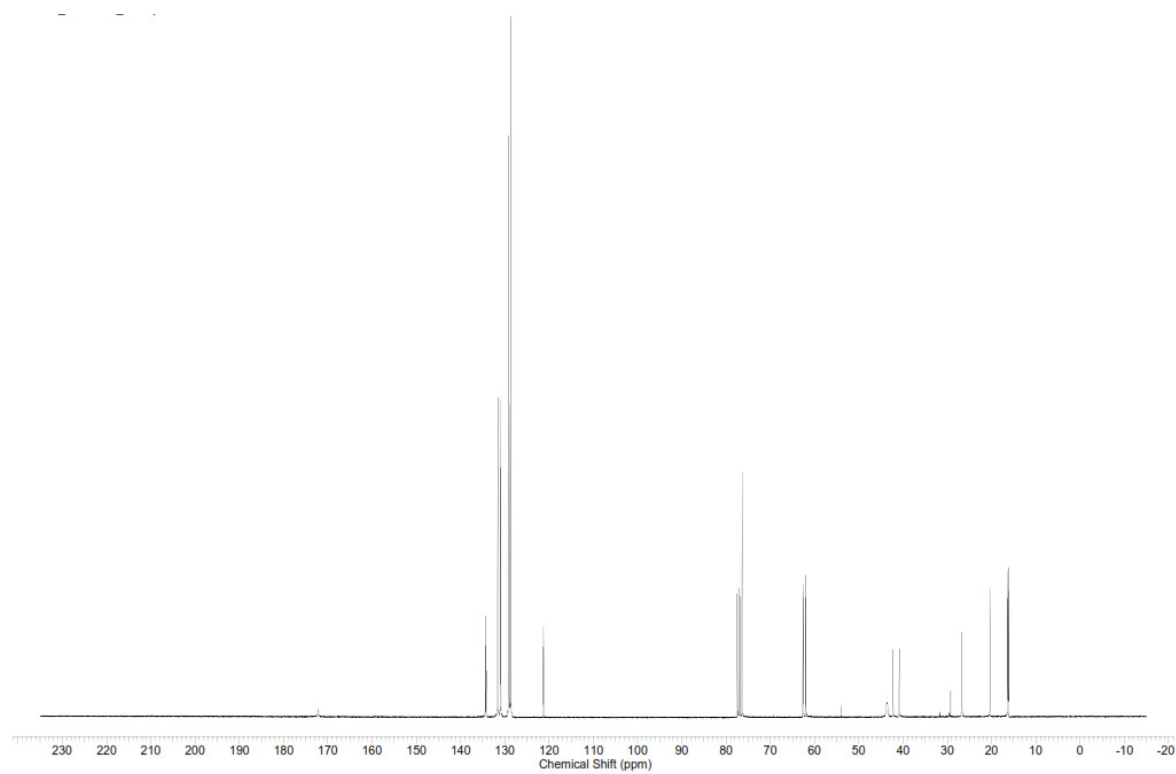
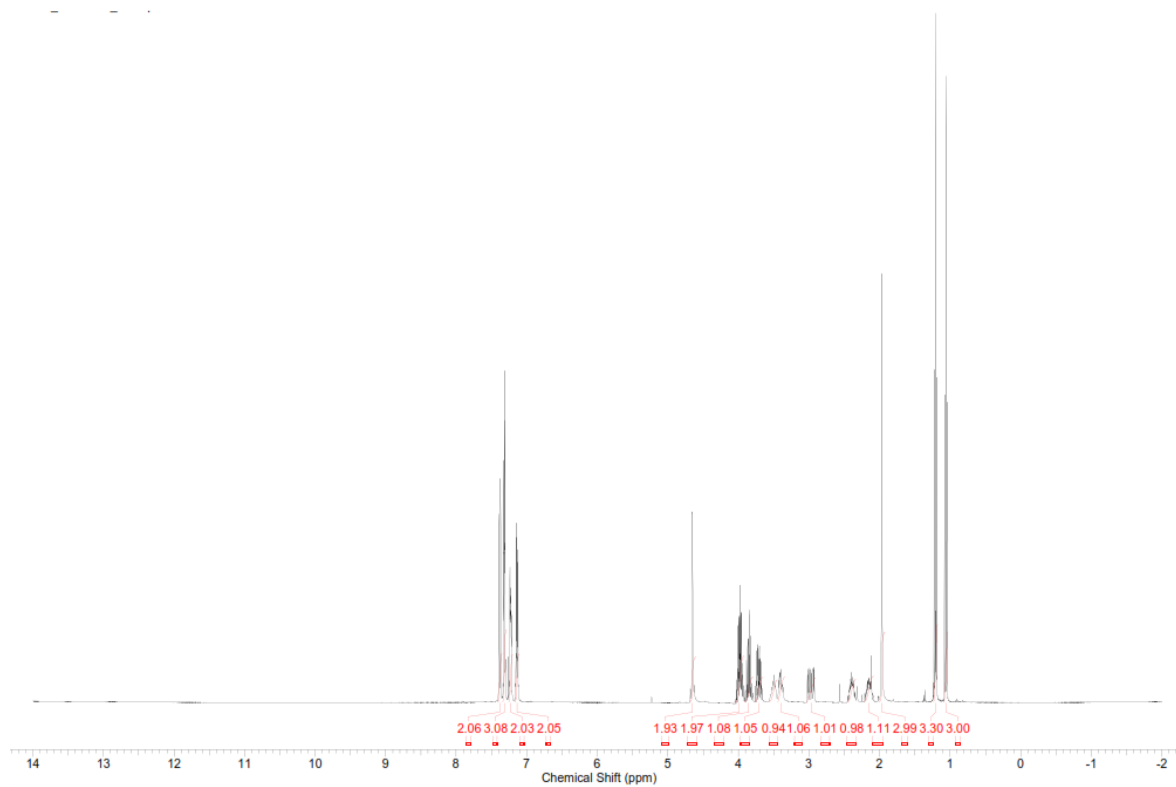
# <sup>1</sup>H NMR and <sup>13</sup>C NMR 11b



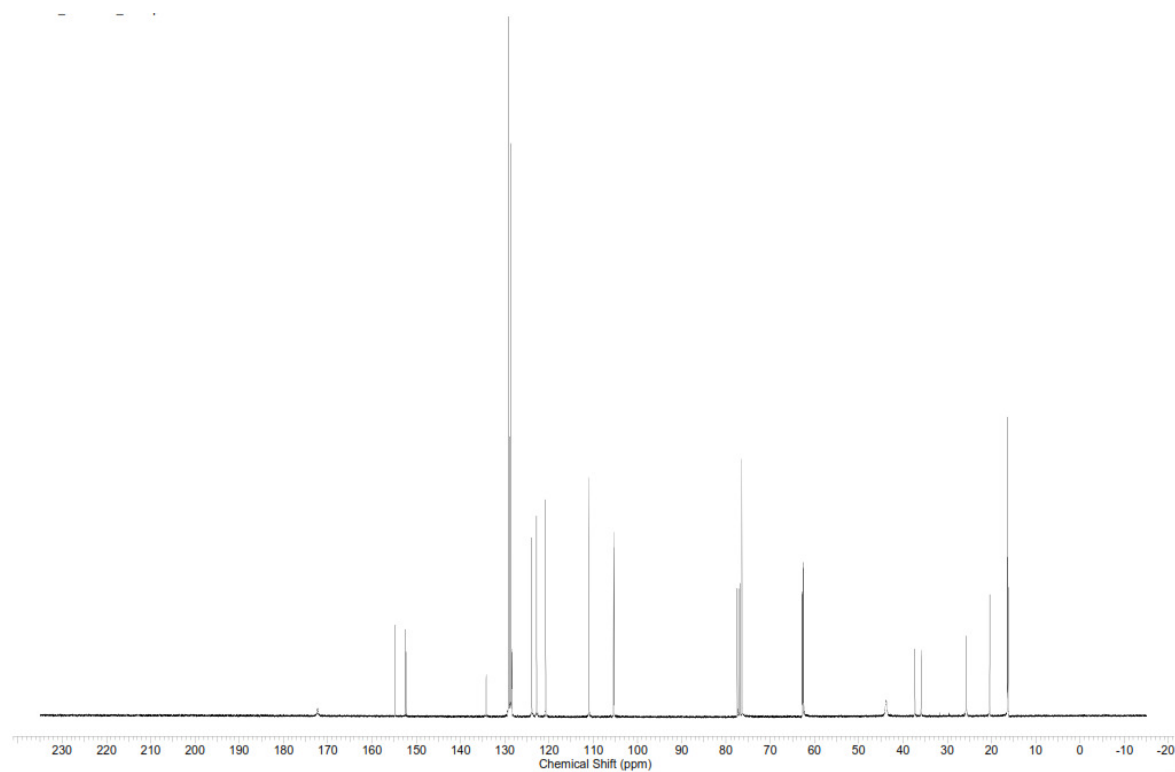
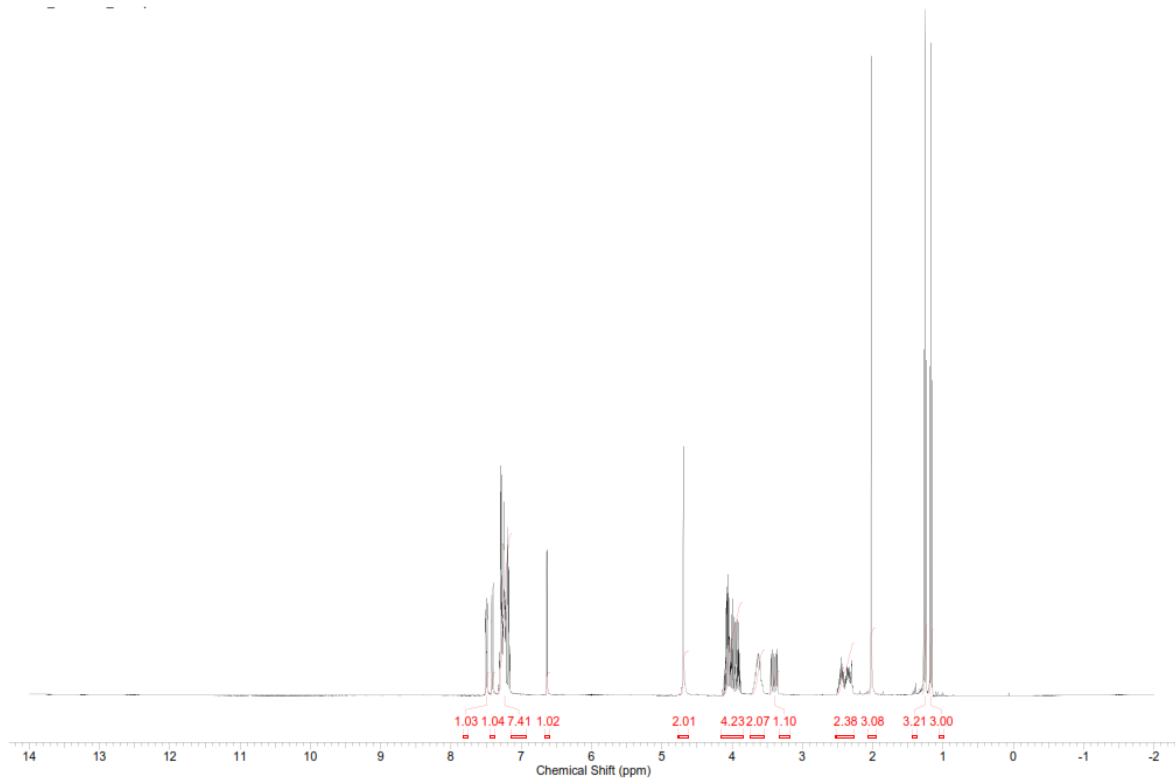
# $^1\text{H}$ NMR and $^{13}\text{C}$ NMR 11c



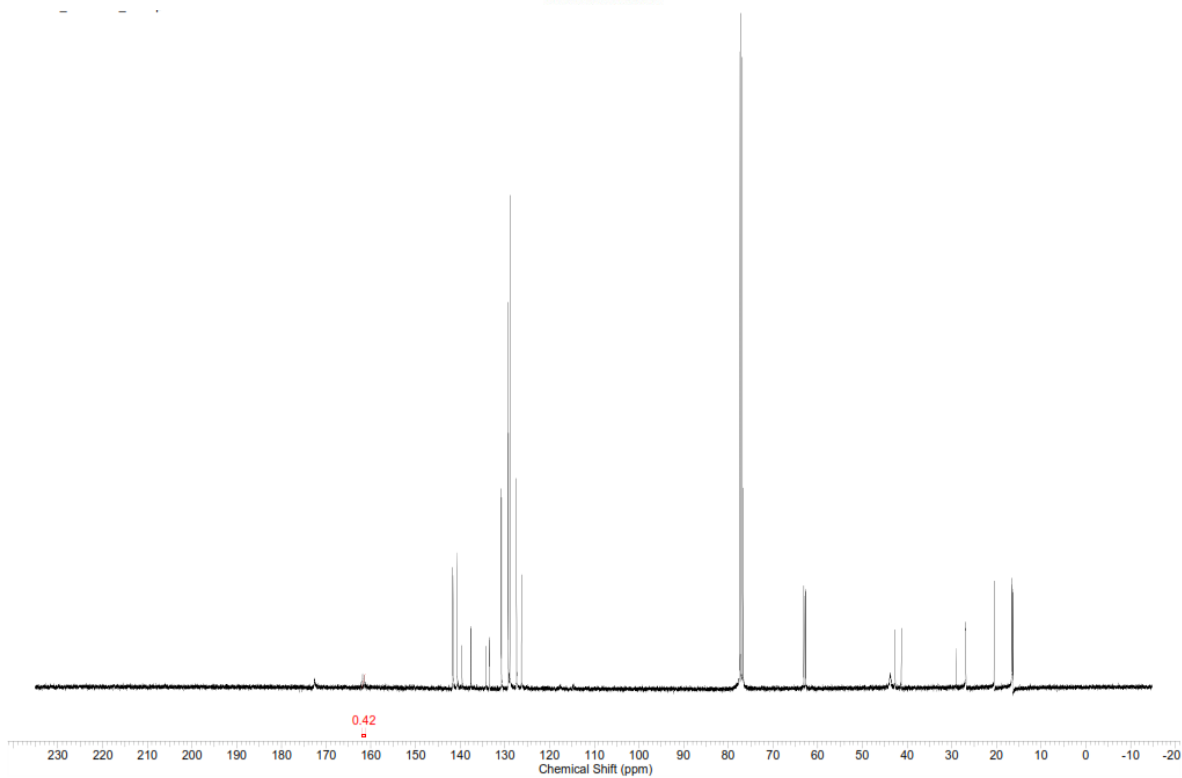
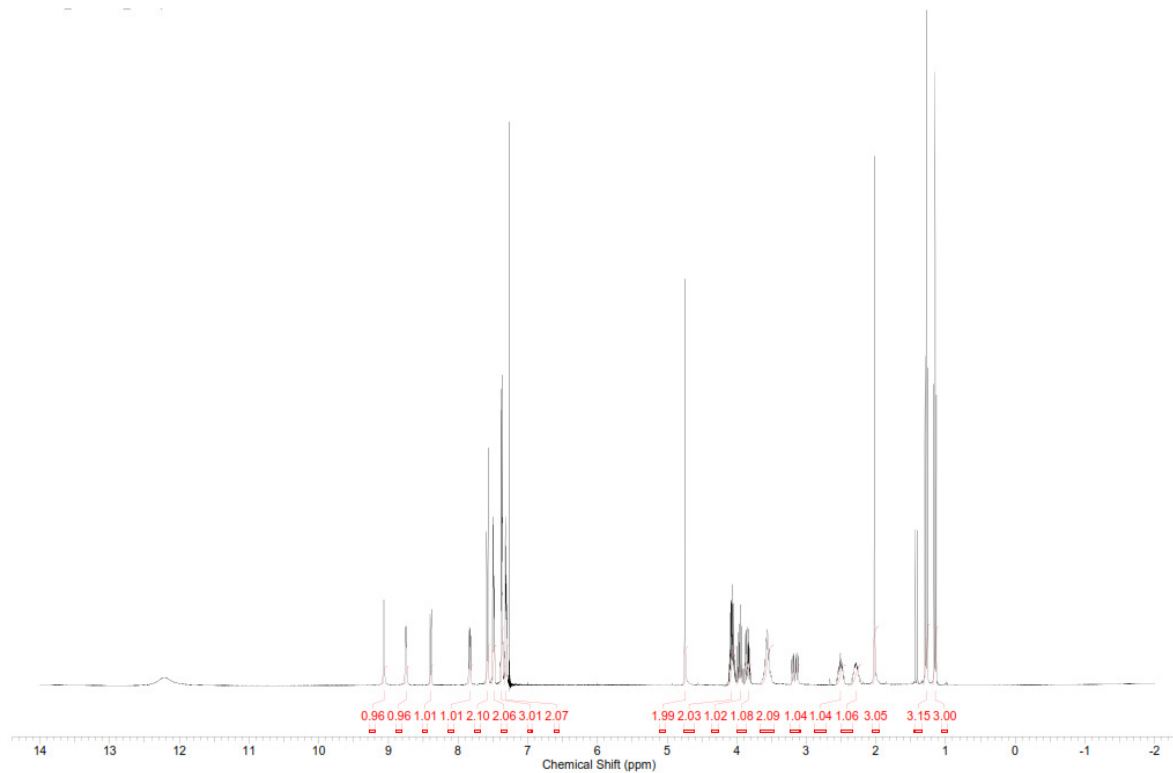
# $^1\text{H}$ NMR and $^{13}\text{C}$ NMR 11d



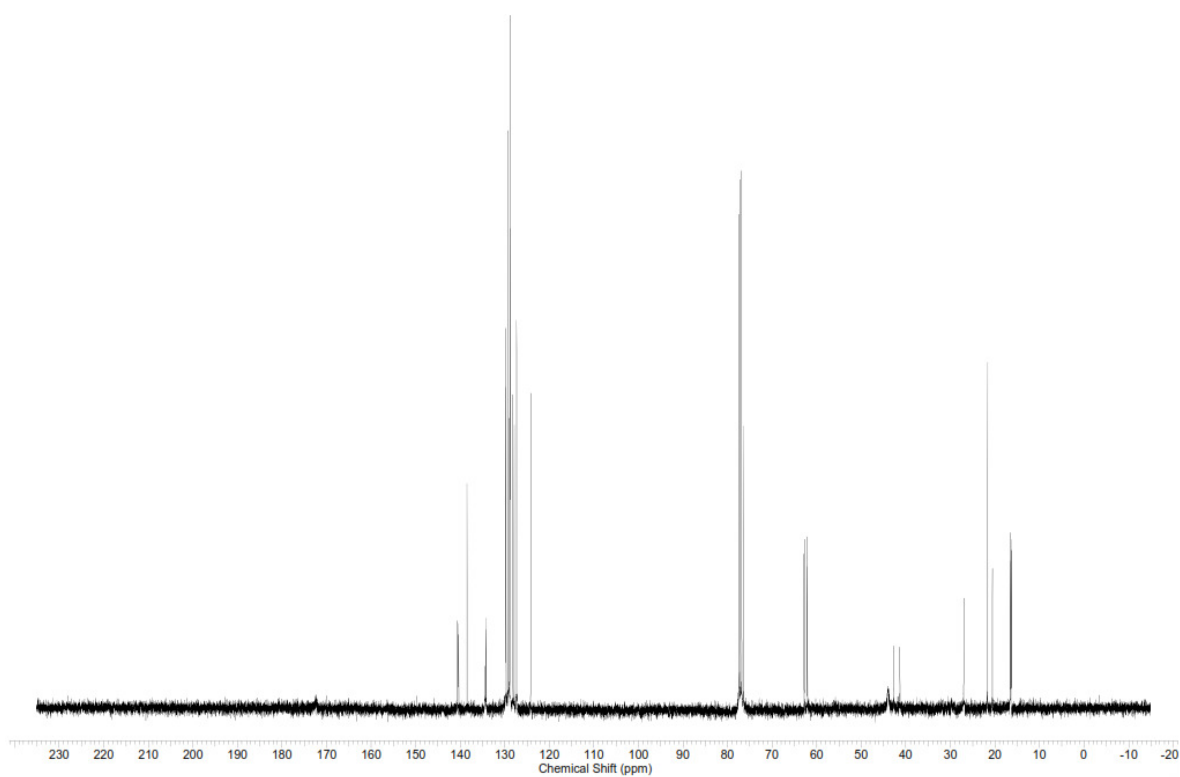
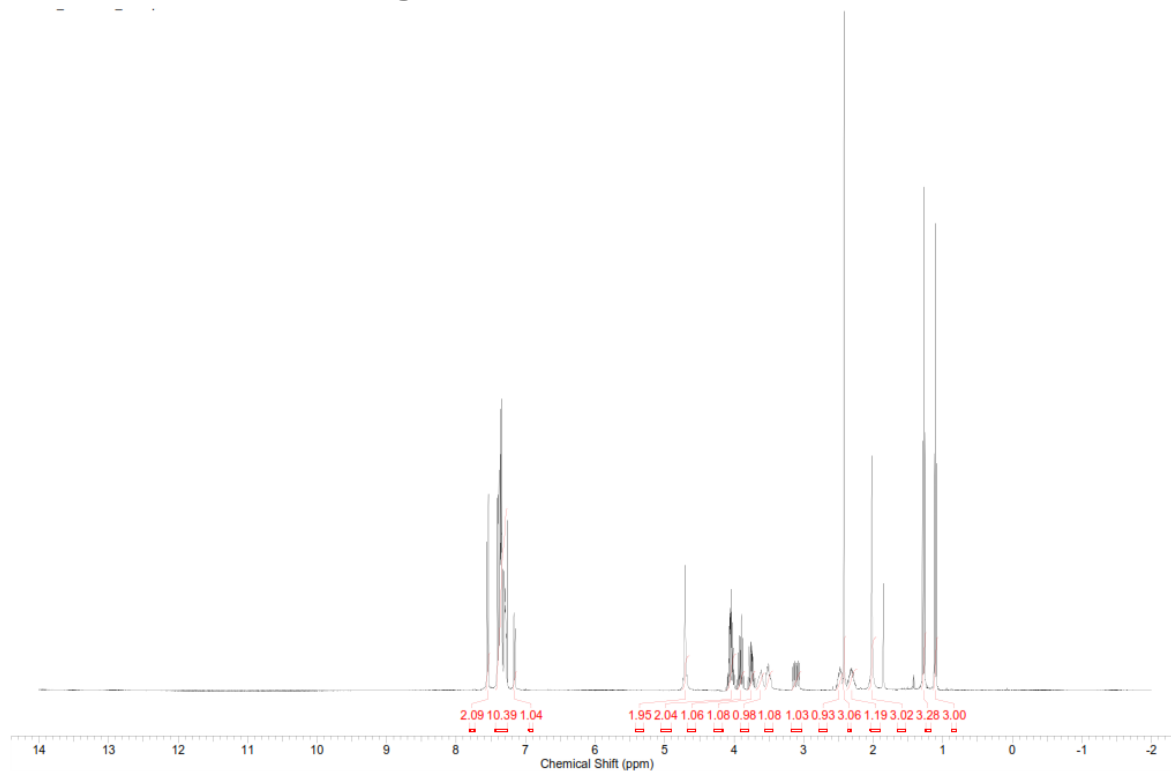
# $^1\text{H}$ NMR and $^{13}\text{C}$ NMR 11e



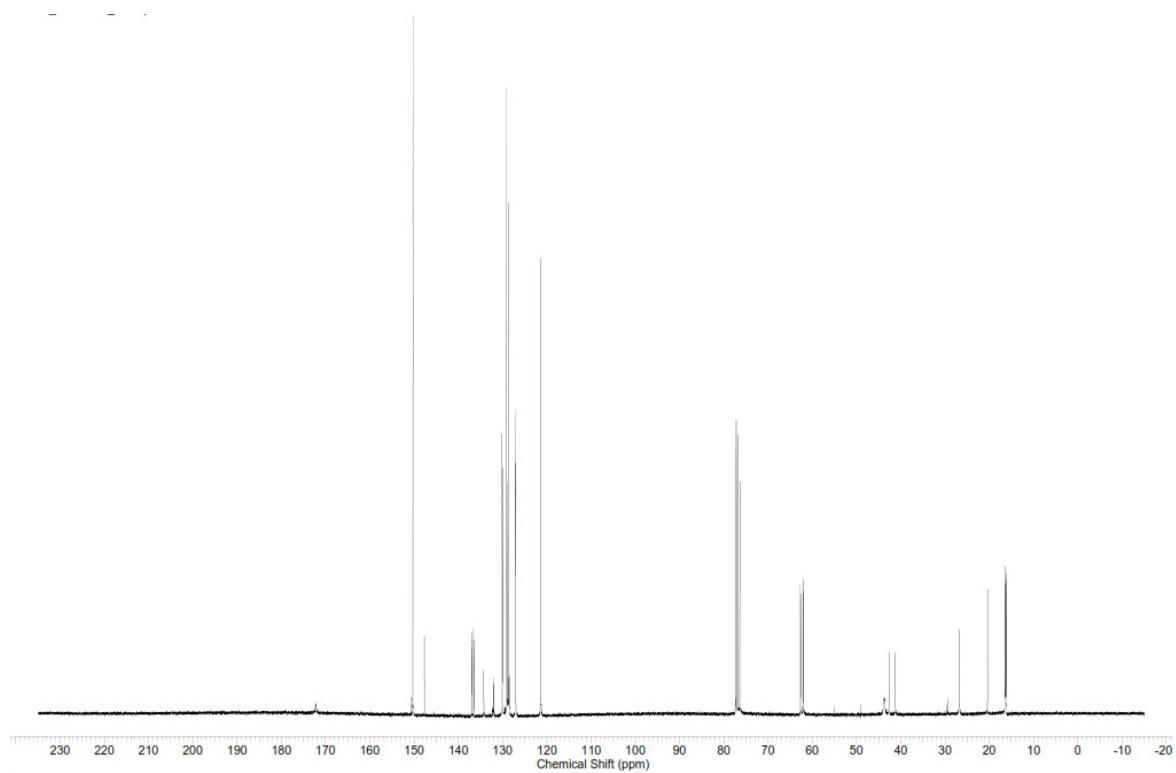
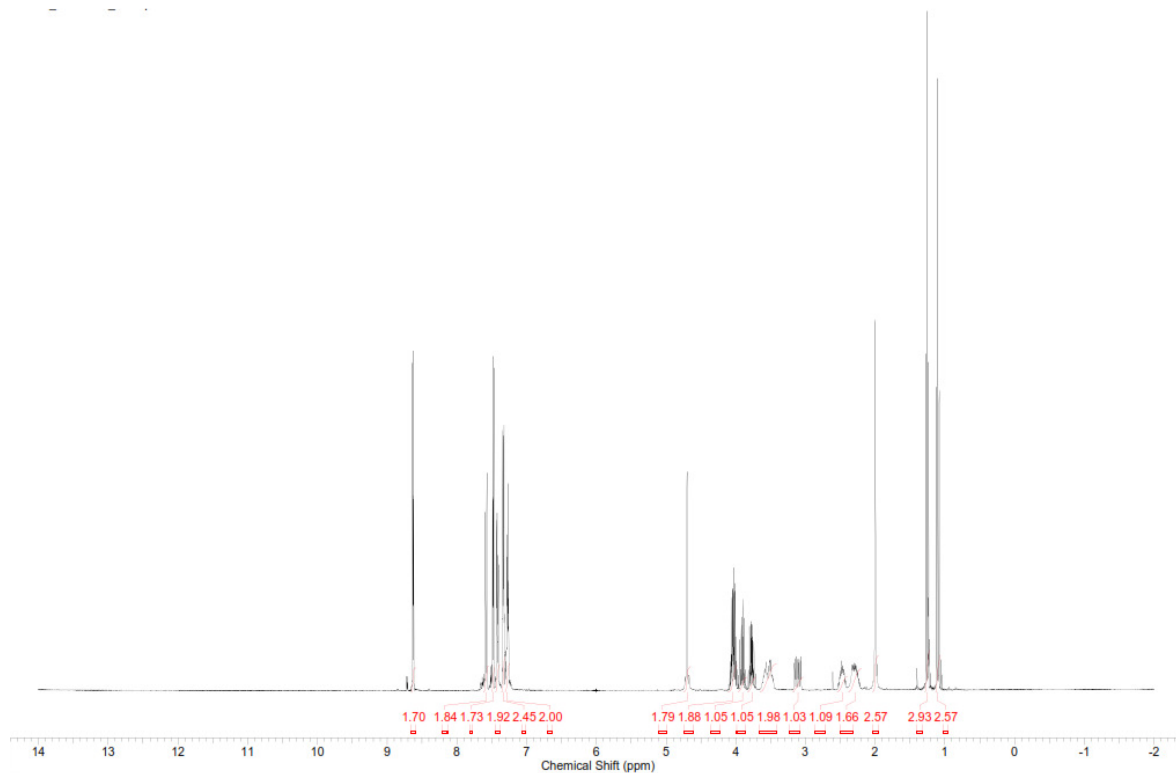
# $^1\text{H}$ NMR and $^{13}\text{C}$ NMR 11f



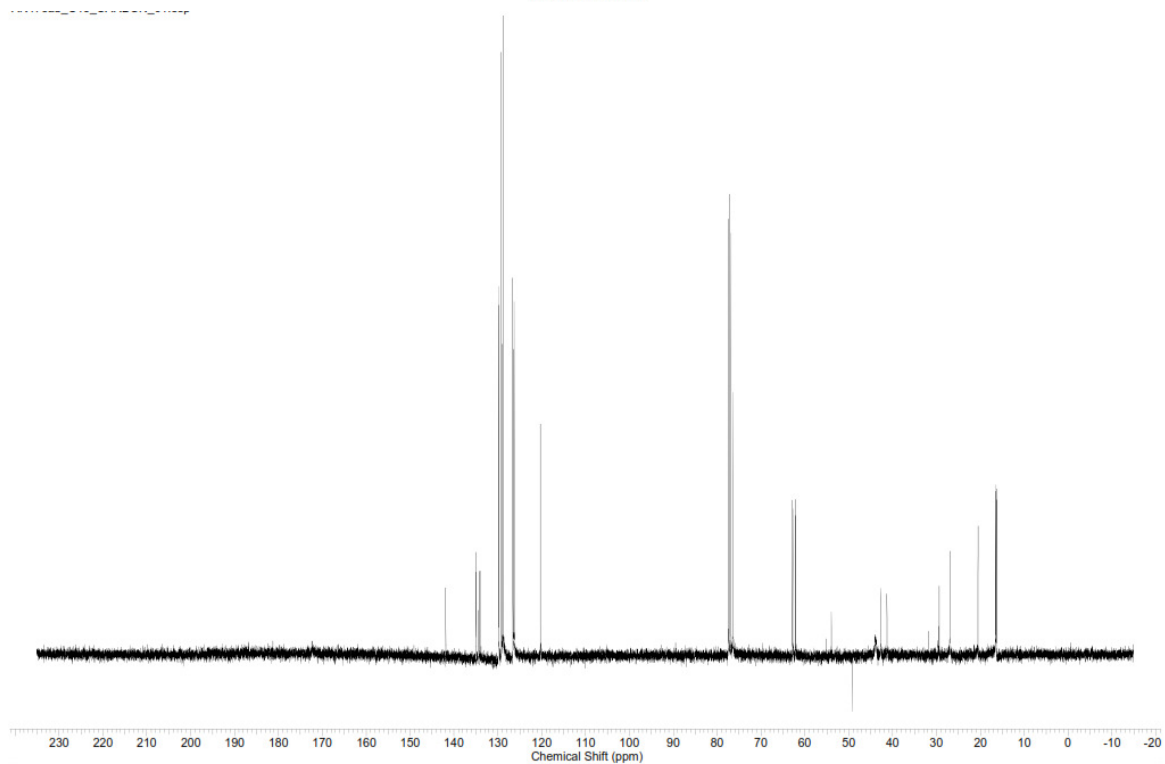
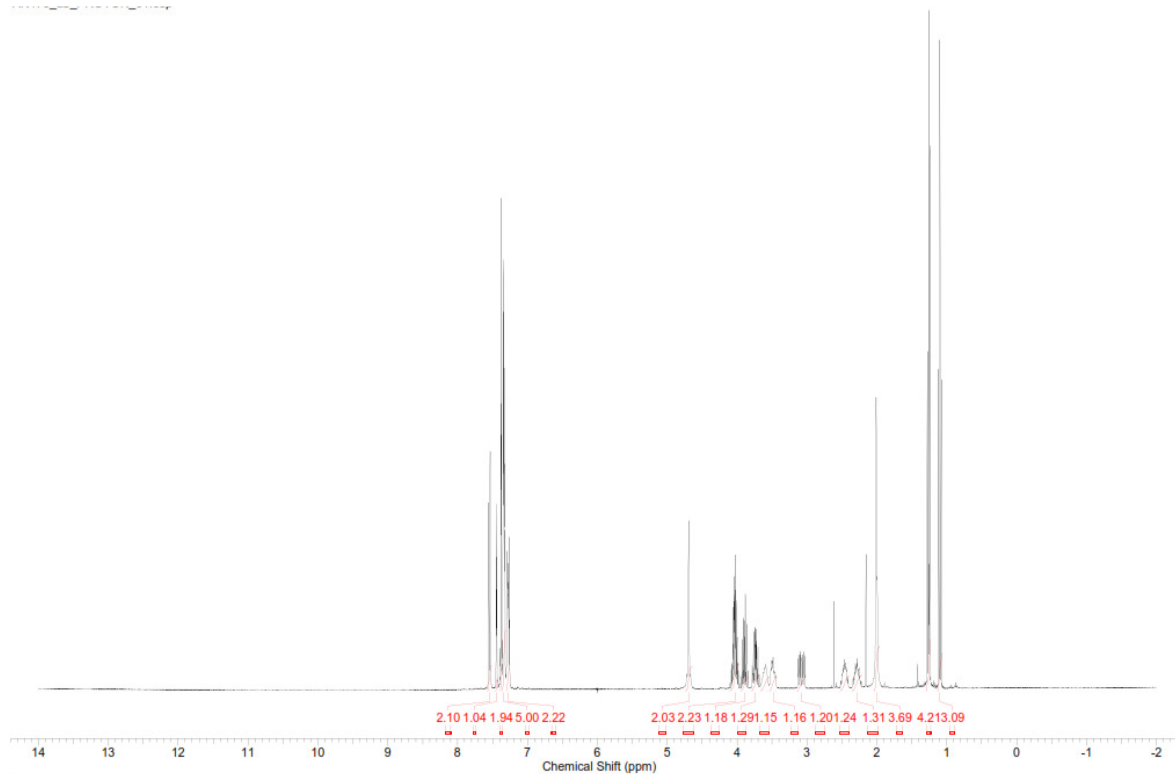
# $^1\text{H}$ NMR and $^{13}\text{C}$ NMR 11g



# <sup>1</sup>H NMR and <sup>13</sup>C NMR 11h

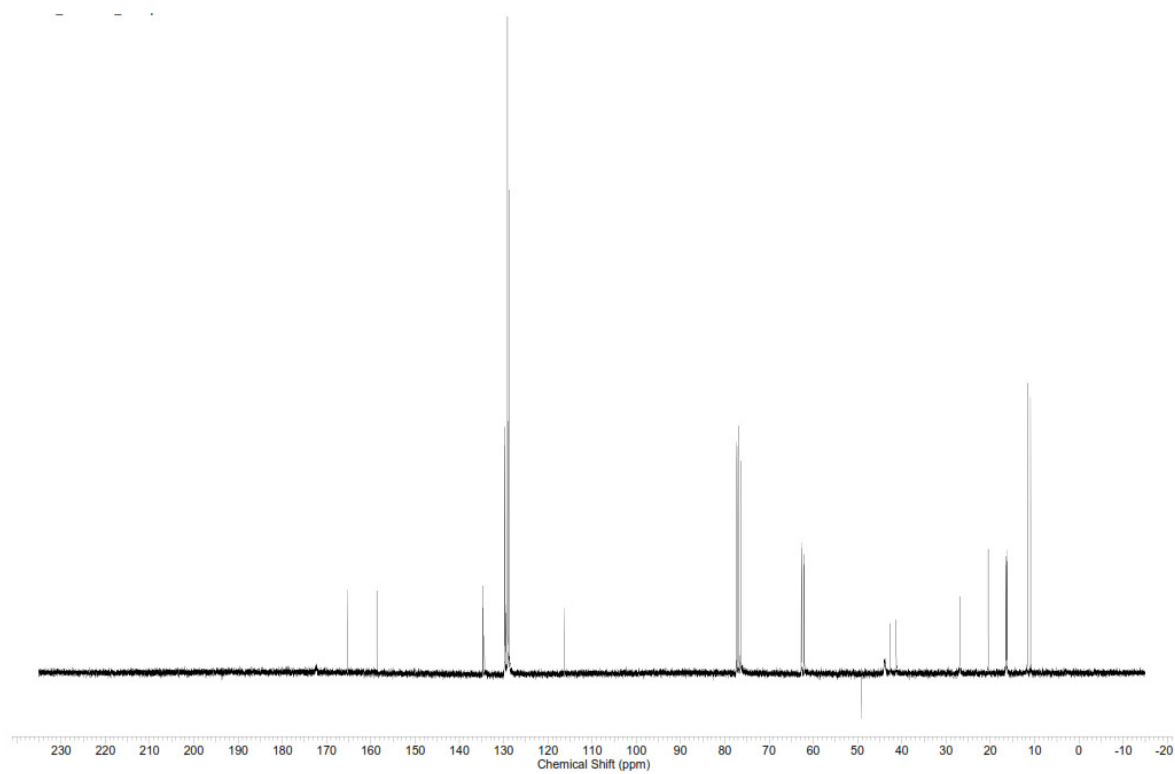
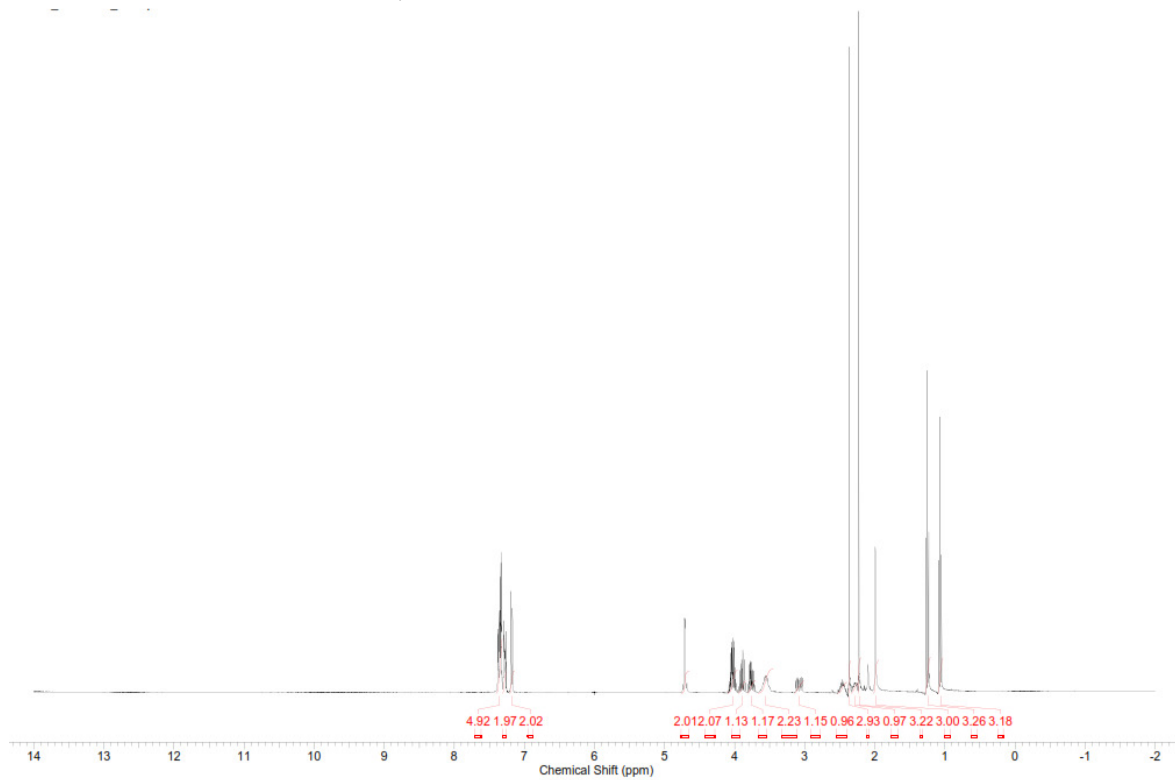


# $^1\text{H}$ NMR and $^{13}\text{C}$ NMR 11i

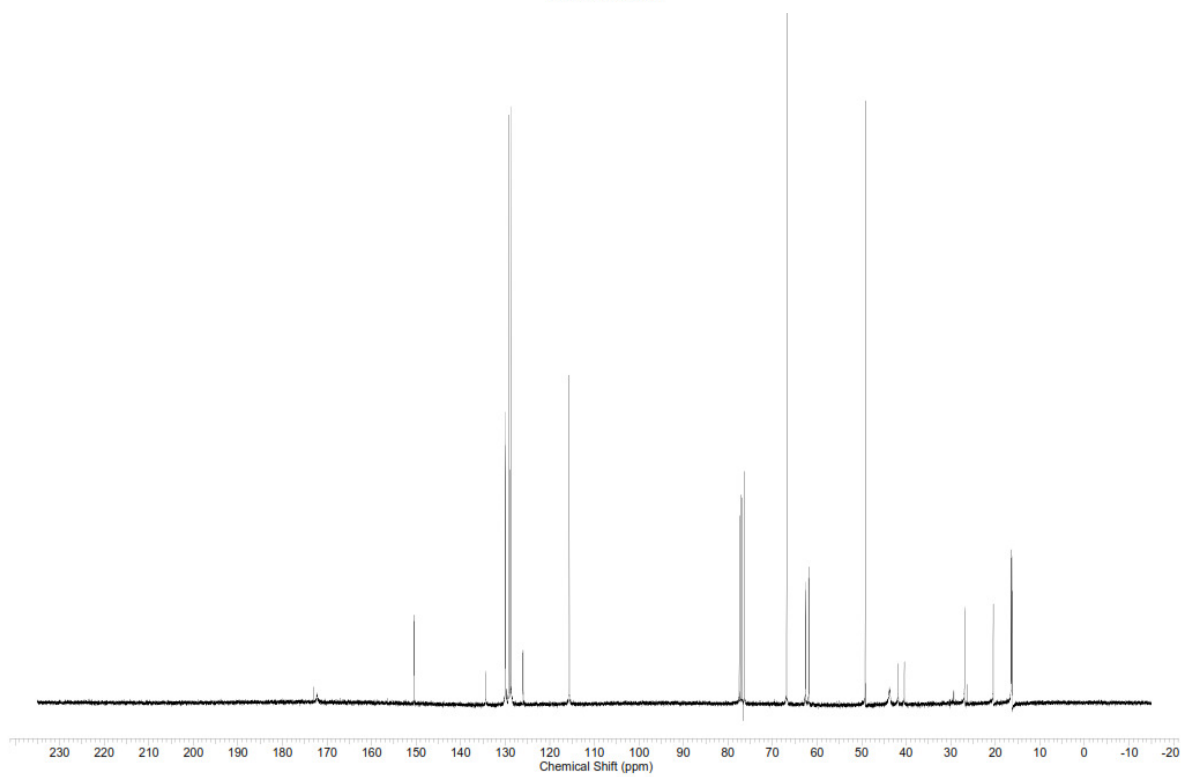
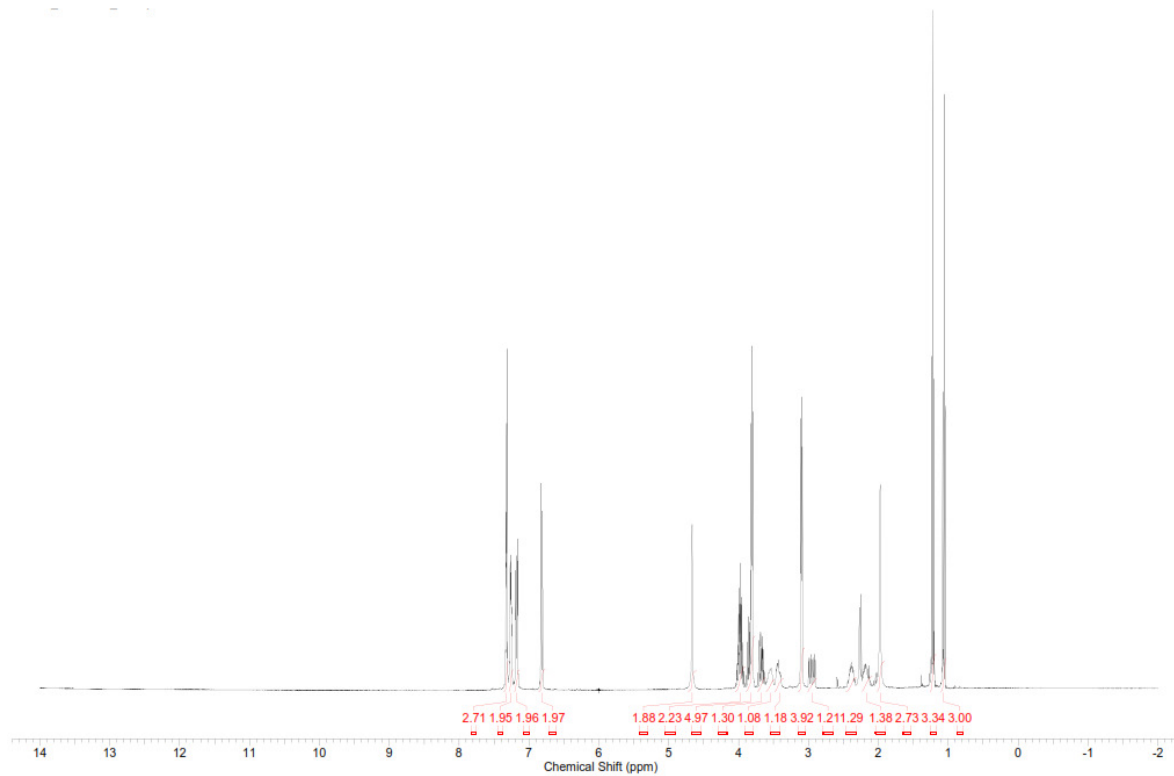




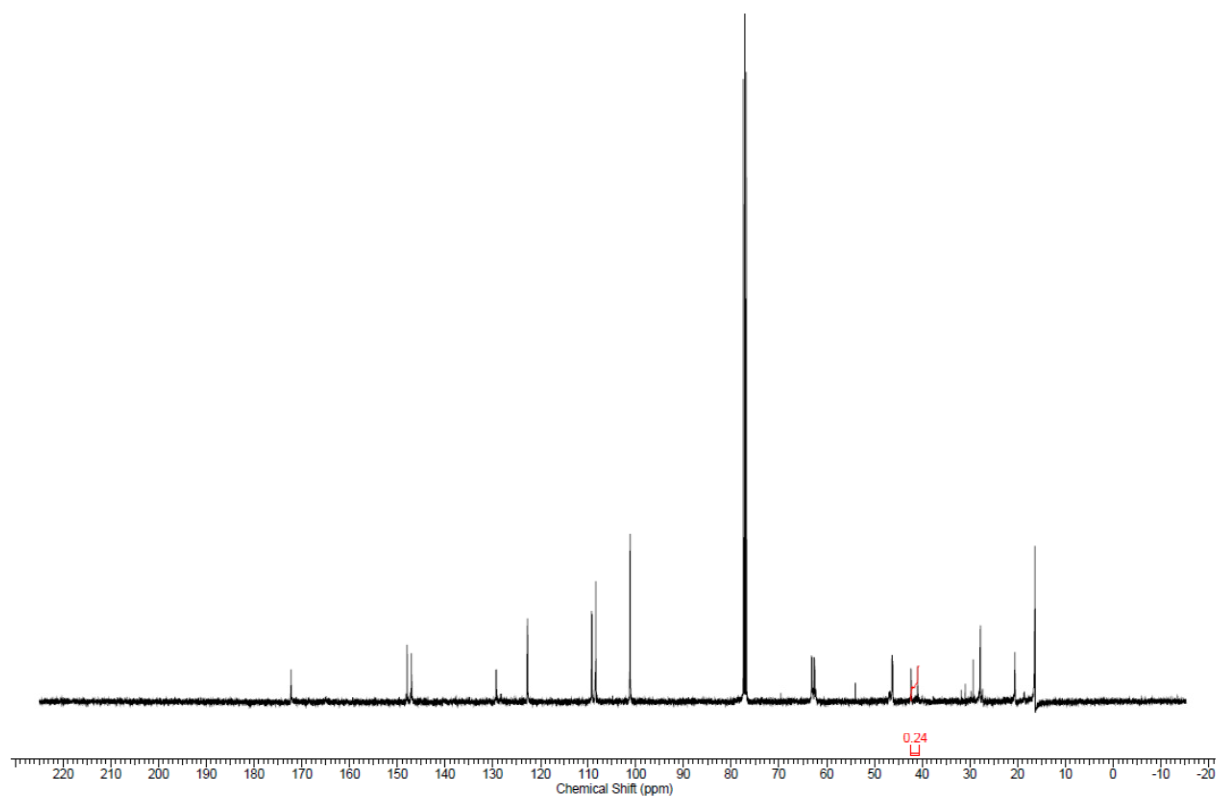
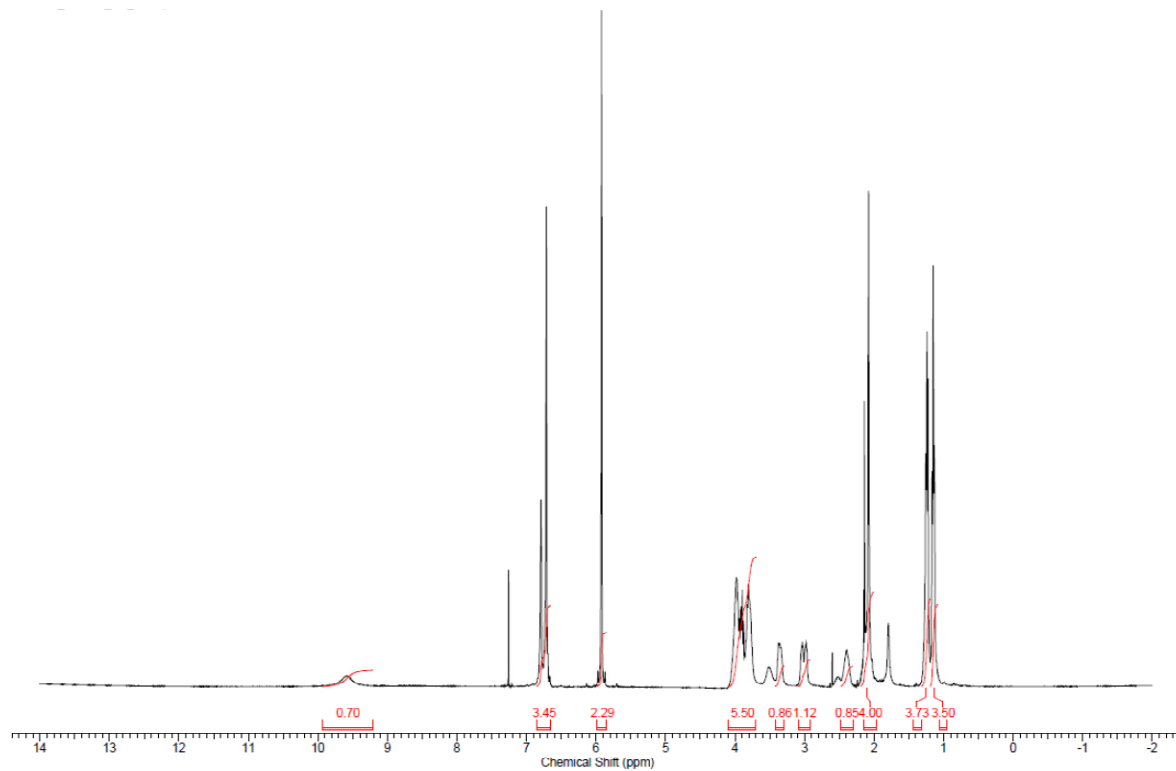
# <sup>1</sup>H NMR and <sup>13</sup>C NMR 11j



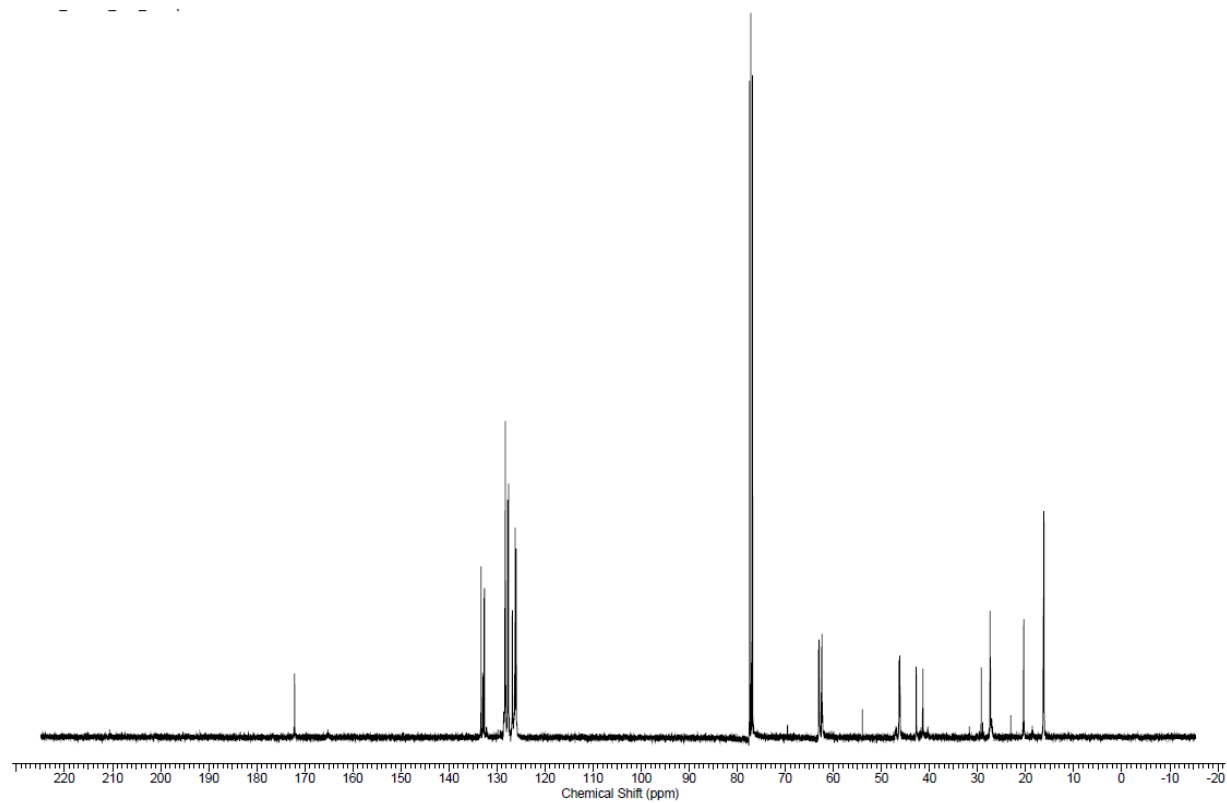
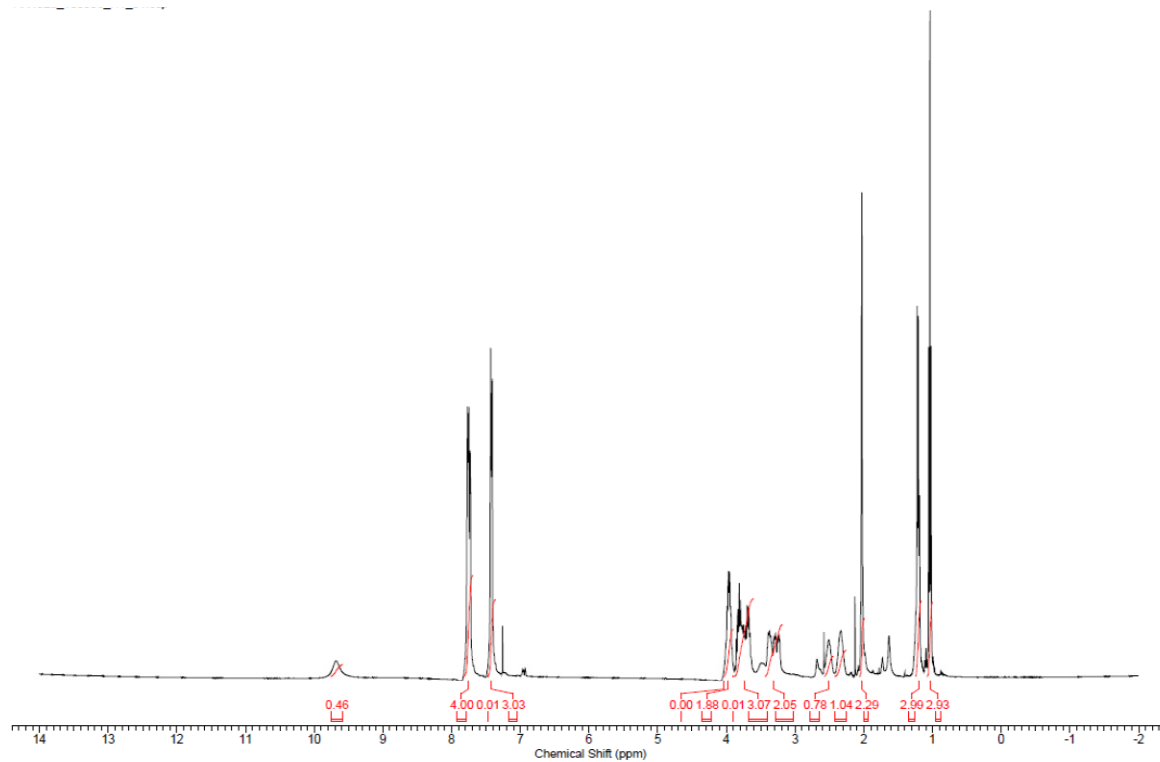
# $^1\text{H}$ NMR and $^{13}\text{C}$ NMR 11k



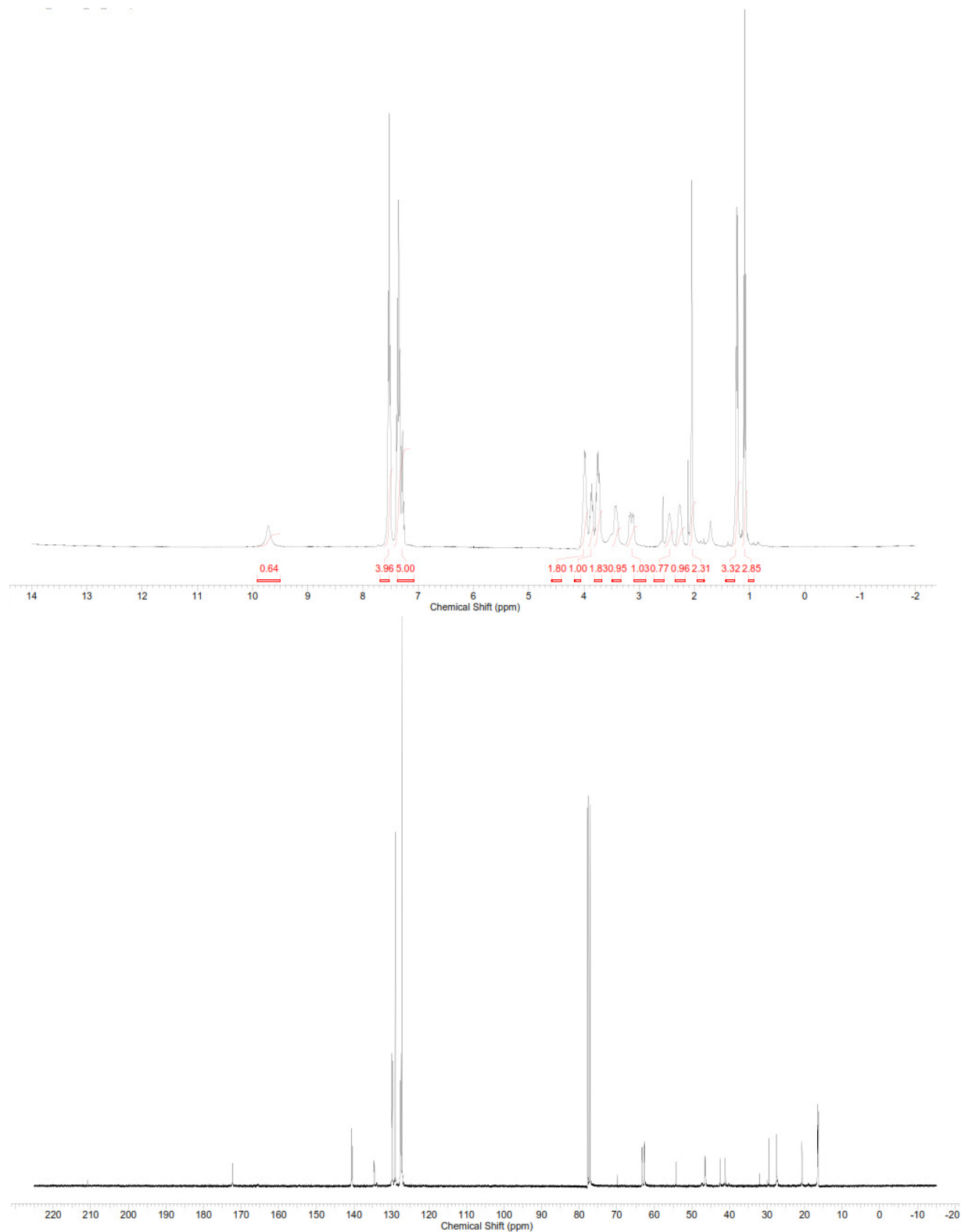
# $^1\text{H}$ NMR and $^{13}\text{C}$ NMR 12a



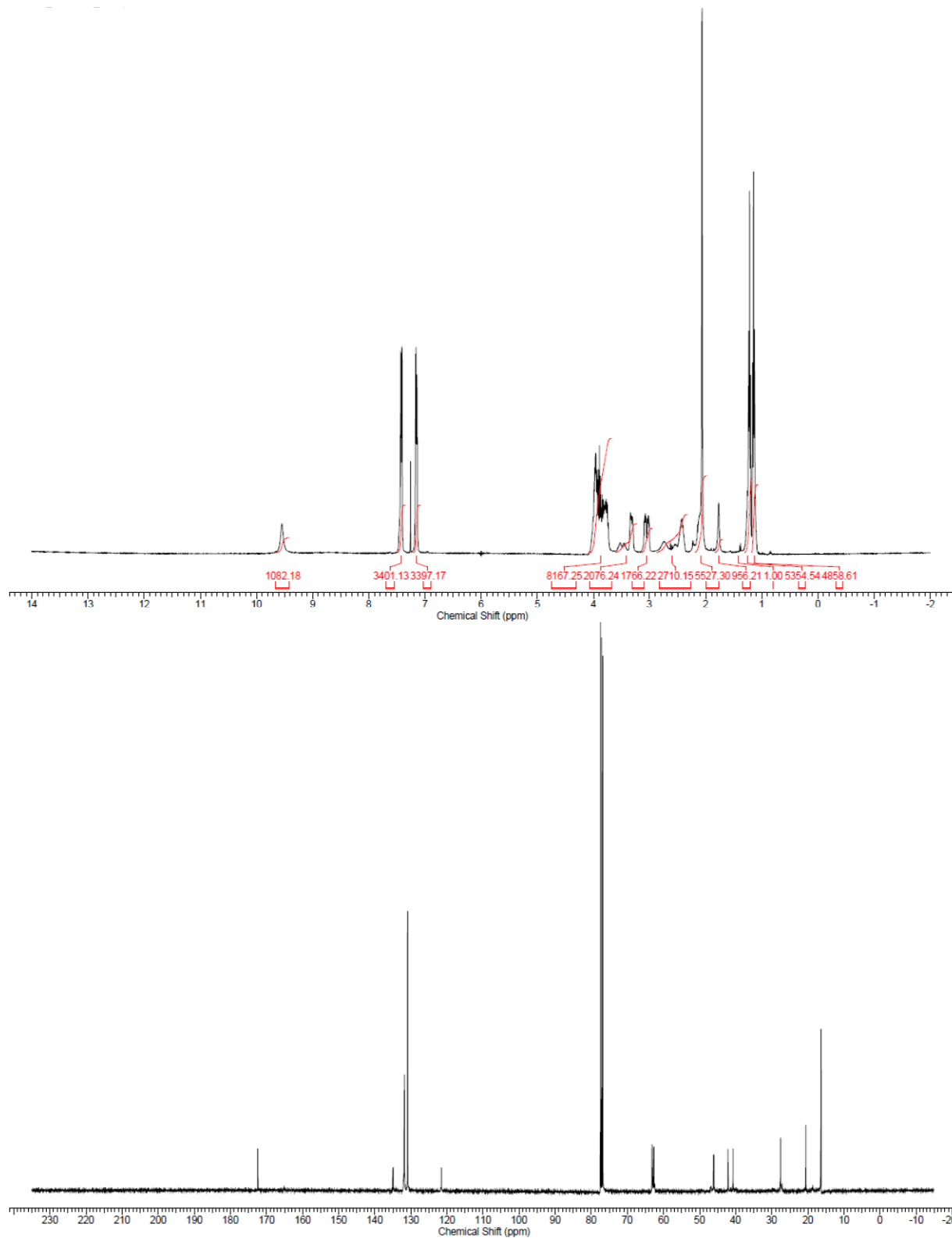
# $^1\text{H}$ NMR and $^{13}\text{C}$ NMR 12b



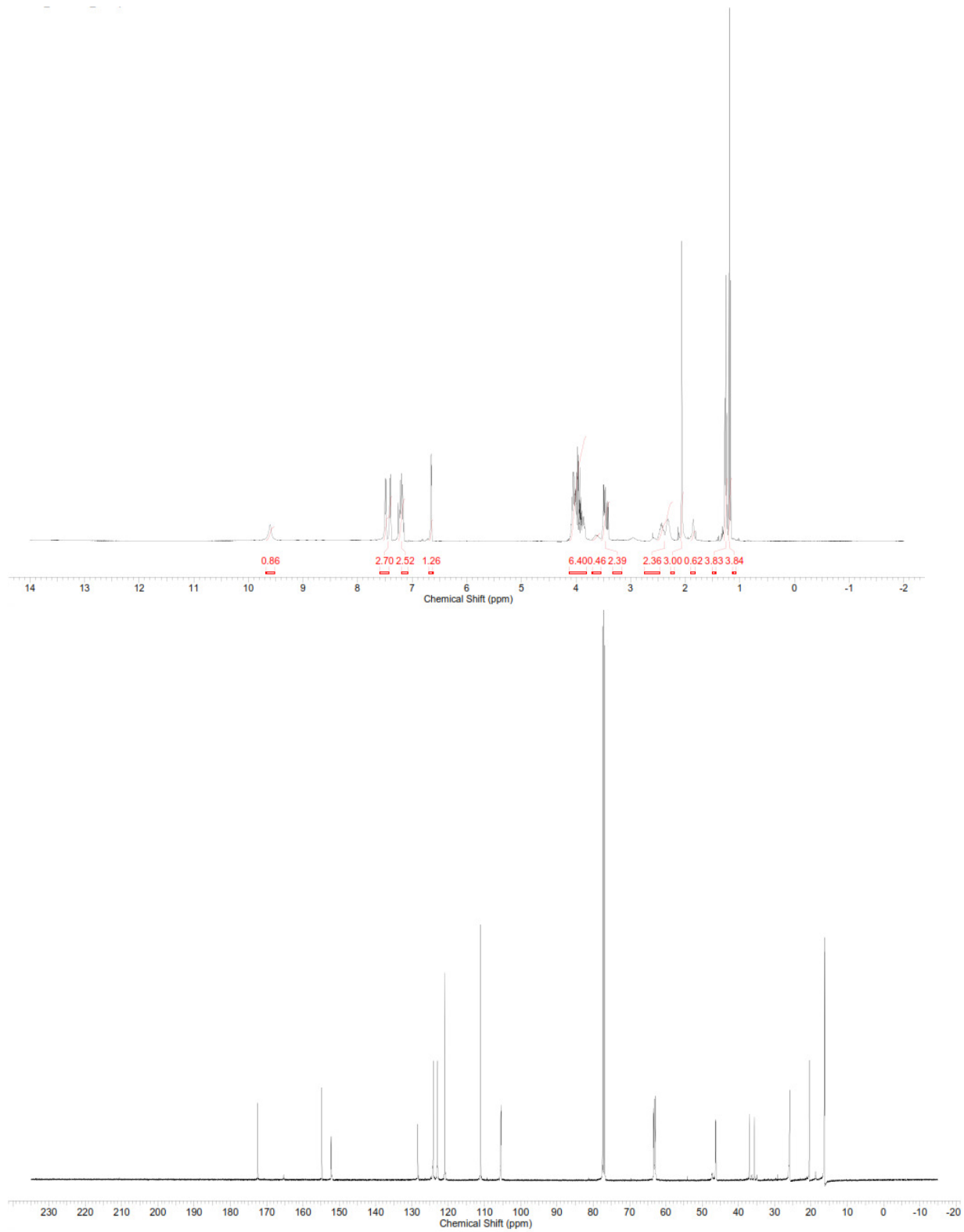
# $^1\text{H}$ NMR and $^{13}\text{C}$ NMR 12c



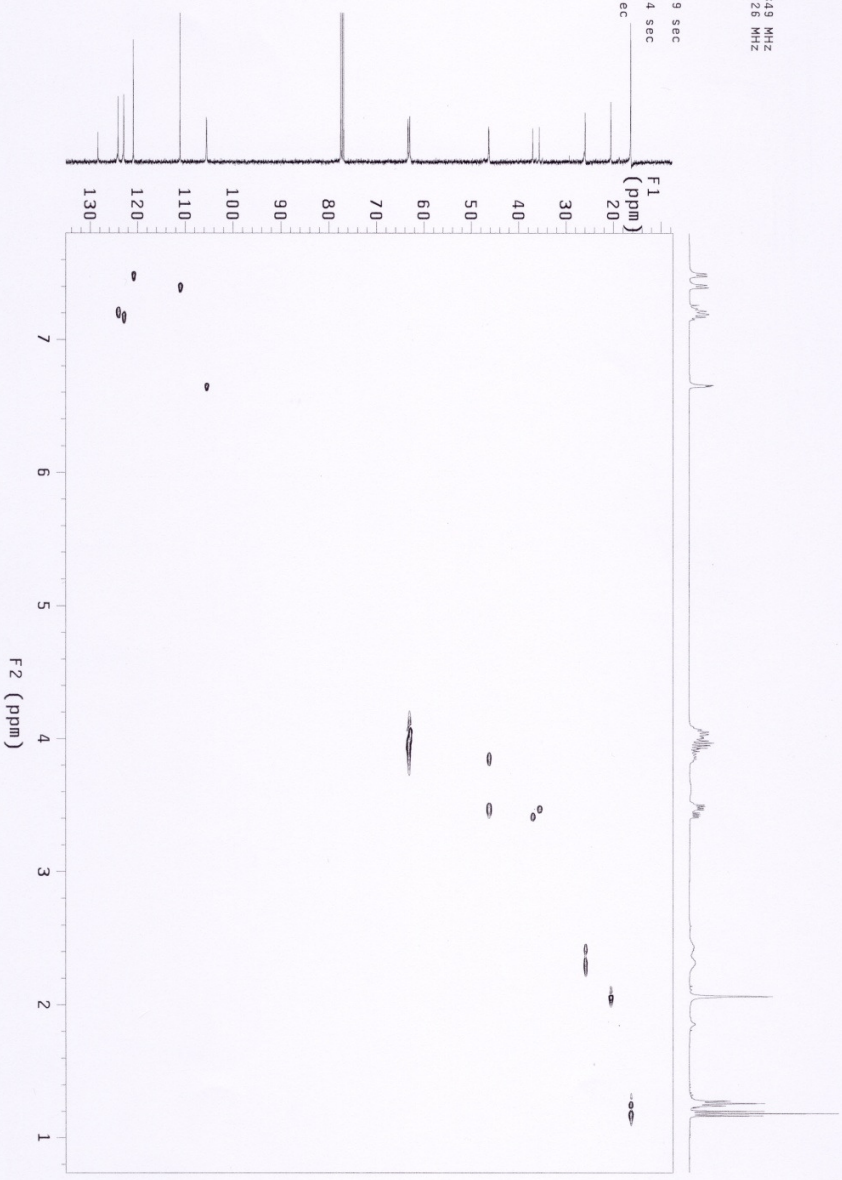
# $^1\text{H}$ NMR and $^{13}\text{C}$ NMR 12d



**<sup>1</sup>H NMR, <sup>13</sup>C NMR HSQC 12e**

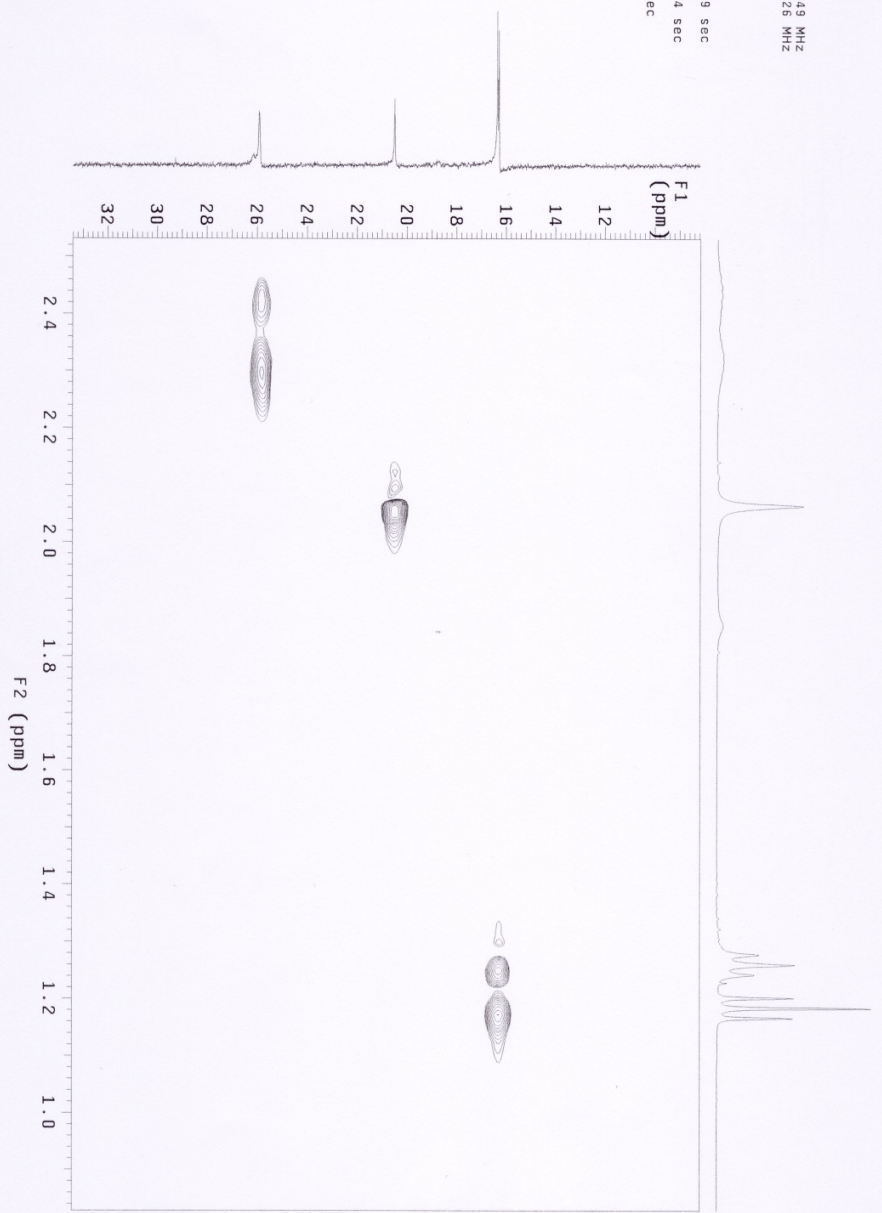


AN159\_hsqc  
 Automation directory:  
 Sample id : tempstudy  
 Sample : AN159\_hsqc  
 Pulse Sequence: ghsqc  
 Solvent: cdcl3  
 Temp: 25.0 C / 298.1 K  
 Spine 1: 100.5526026 MHz  
 File: AN159\_hsqc\_ghsqc\_01  
 INOVA-500 "processor"  
 Relax. delay: 1.000 sec  
 Acq. time: 0.150 sec  
 Width: 6398.0 Hz  
 ZD Width: 17094.0 Hz  
 ZD Offset: 100.5526026 MHz  
 2 x 128 increments  
 OBSERVE: H1, 399.8607349 MHz  
 DECOUPLE: C13, 100.5526026 MHz  
 Power: 47 db  
 on during acquisition  
 on during acquisition  
 GARP-1 modulated  
 DATA PROCESSING  
 Gauss apodization: 0.069 sec  
 F1 DATA PROCESSING  
 Gauss apodization: 0.014 sec  
 F2 apodization: 0.014 sec  
 Total time: 23 min, 15 sec

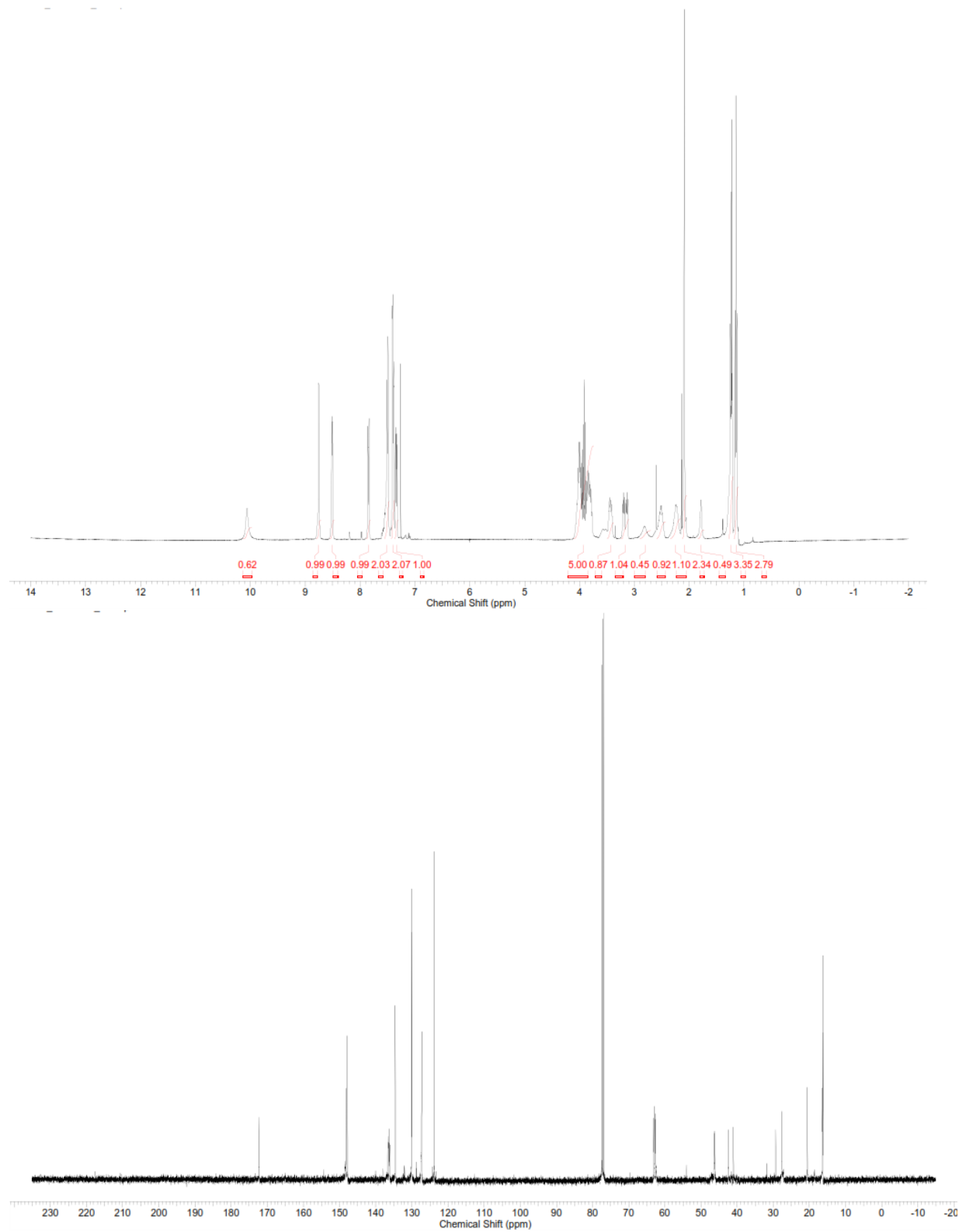




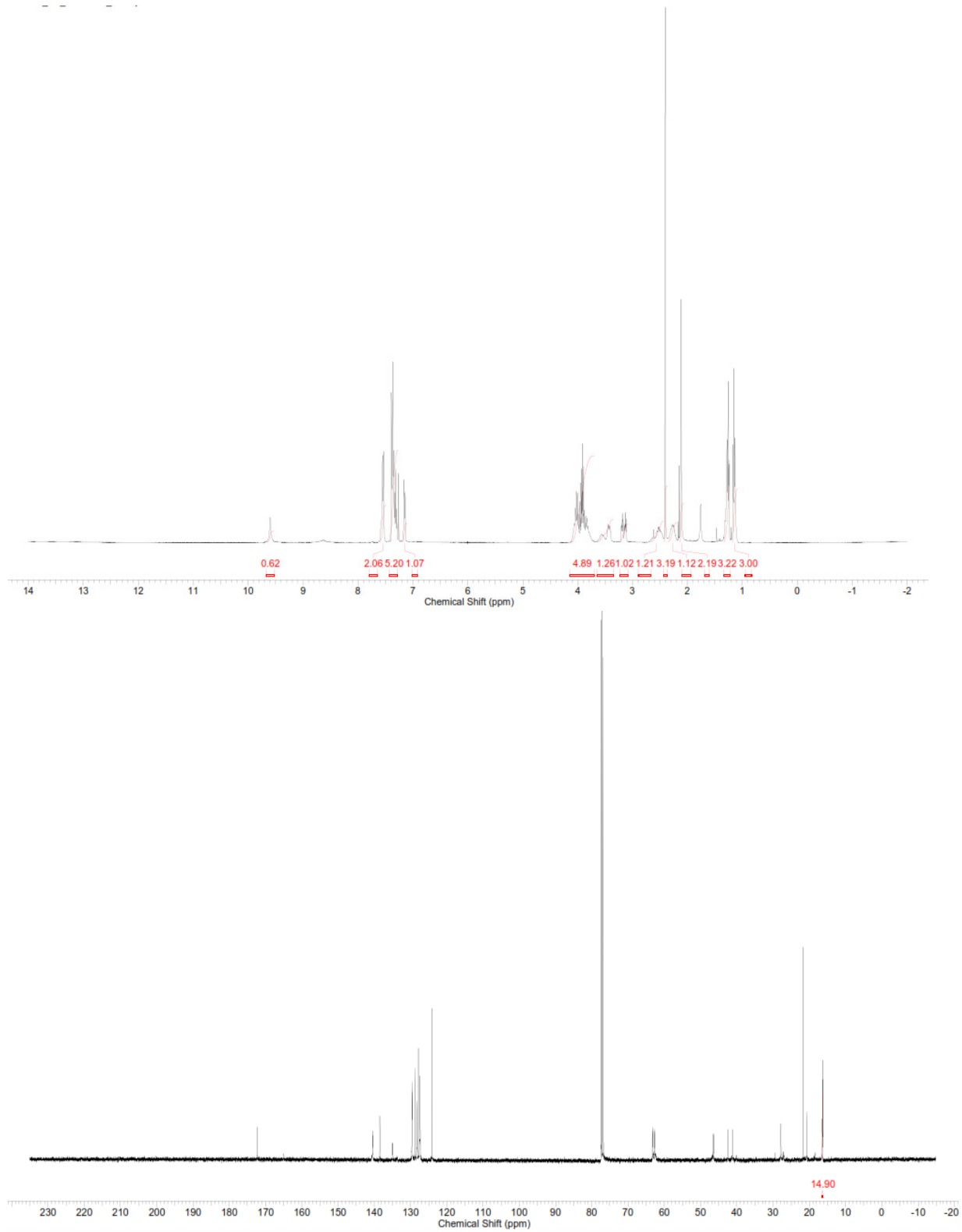
AN159\_hsqc  
Automation directory:  
Sample id: tempstudy  
Sample : AN159\_hsqc  
Pulse Sequence: ghsoc  
Solvent: cdcl3  
Temp: 25.0 C / 298.1 K  
Sample #39, Operator: annett  
File: AN159\_hsqc\_GHSQC\_01  
INOVA-500 "processor"  
Relax. delay 1.000 sec  
Acq. time 0.150 sec  
Width 6398.0 Hz  
D 20.0000000 Hz  
4 repetitions  
2 x 128 increments  
OBSERVE H1, 399.8607349 MHz  
DECUPLE C13, 100.5526026 MHz  
Power 47 db  
on during acquisition  
off during delay  
GAP-1000000  
DATA PROCESSING  
Gauss apodization 0.069 sec  
F1 DATA PROCESSING 0.014 sec  
Gauss apodization 0.014 sec  
F1 size 2048 x 4096  
Total time 29 min, 15 sec



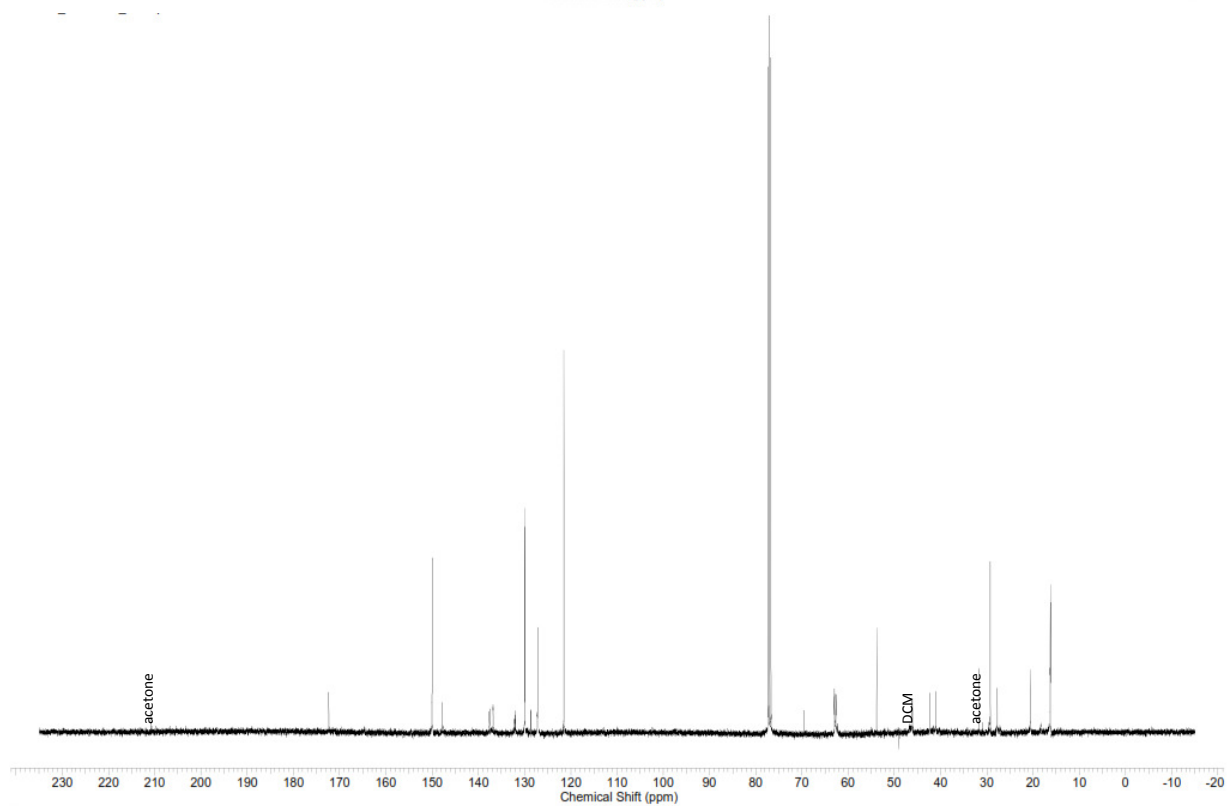
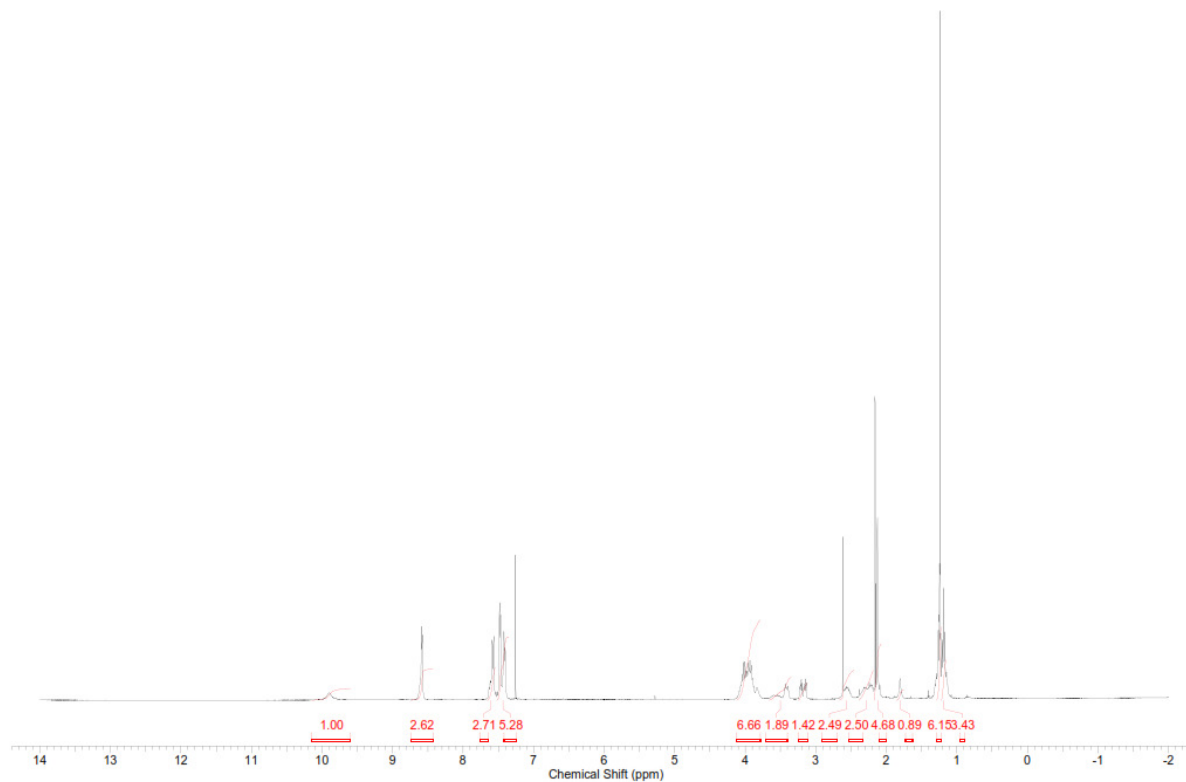
# $^1\text{H}$ NMR and $^{13}\text{C}$ NMR 12f



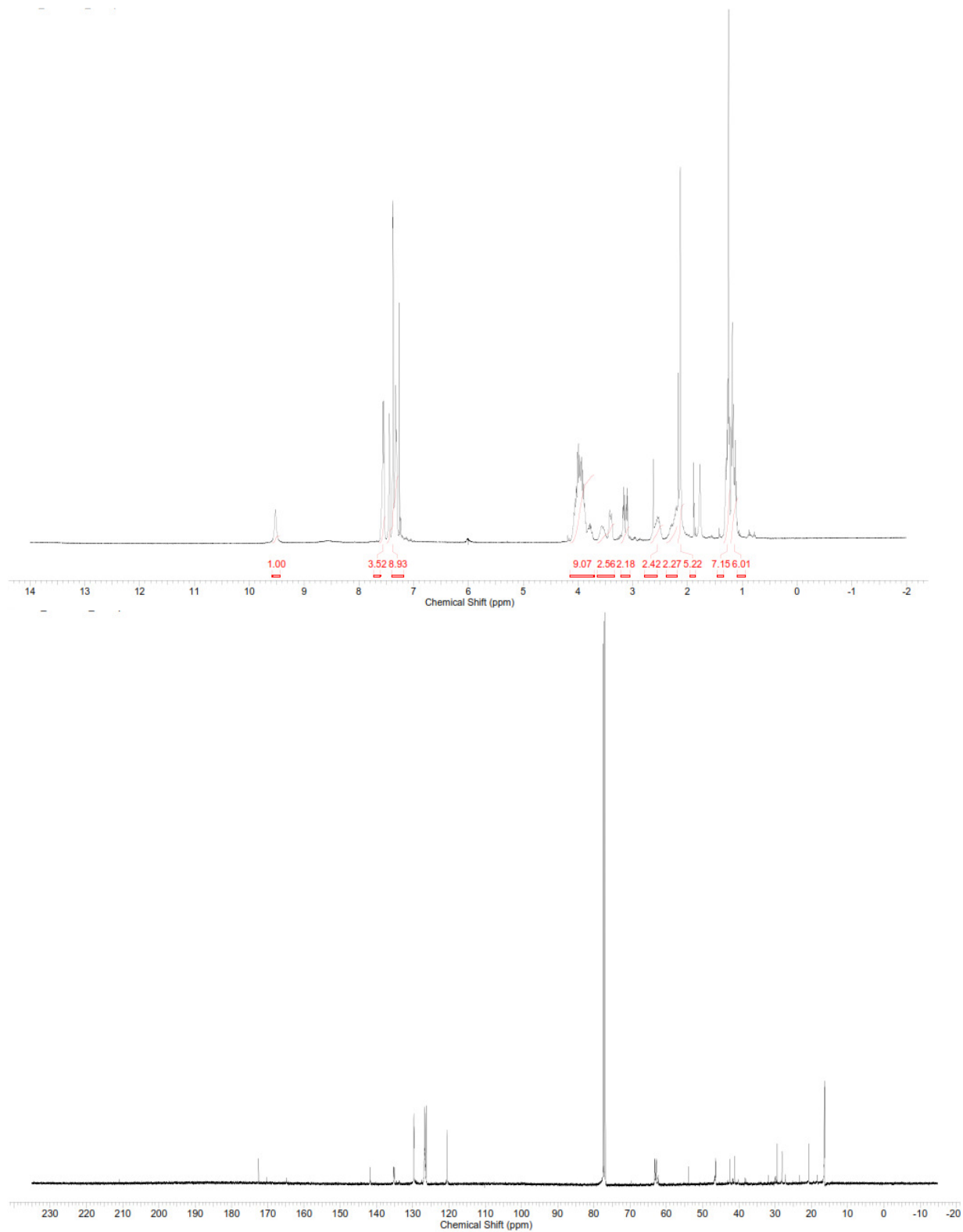
# $^1\text{H}$ NMR and $^{13}\text{C}$ NMR 12g



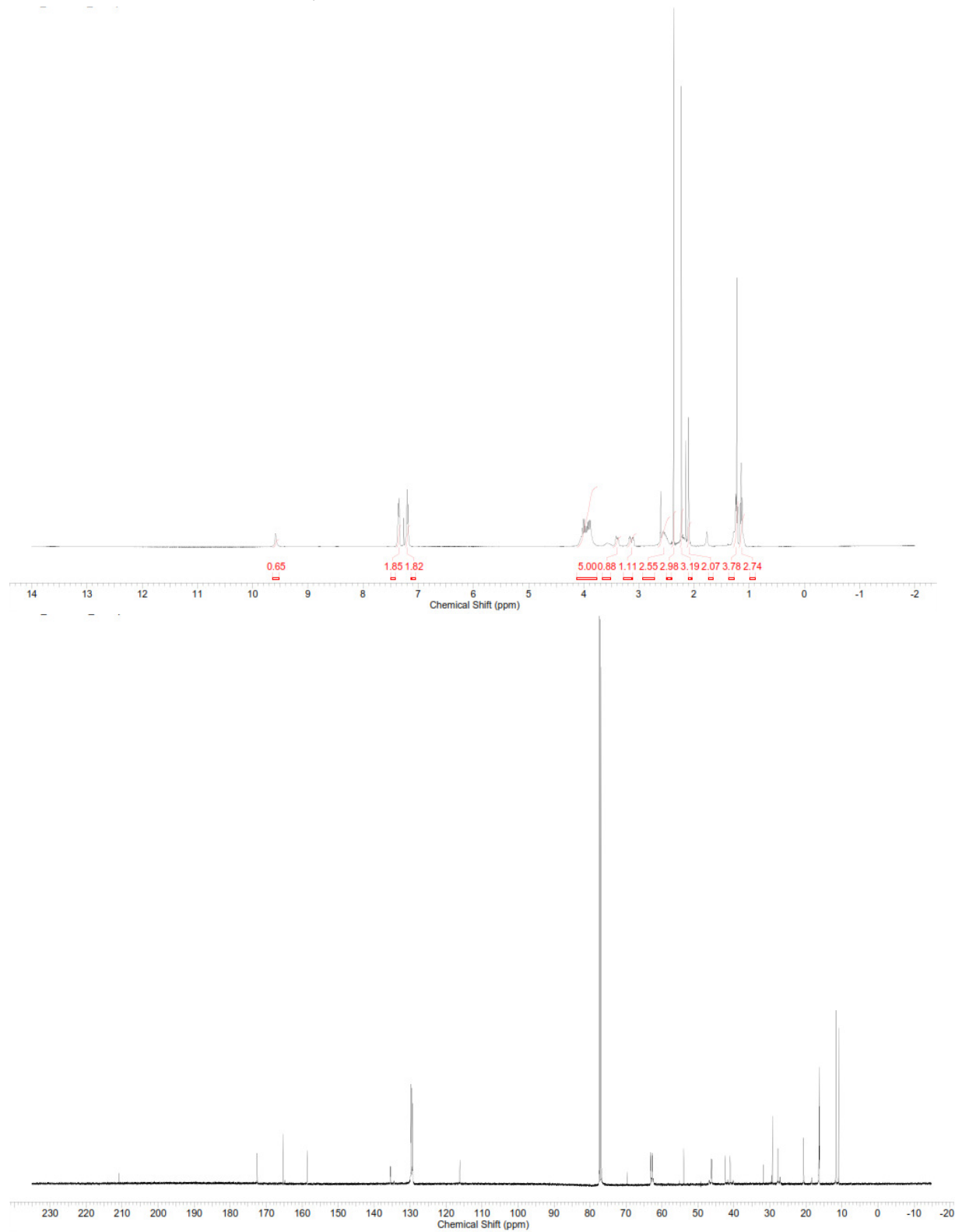
# $^1\text{H}$ NMR and $^{13}\text{C}$ NMR 12h



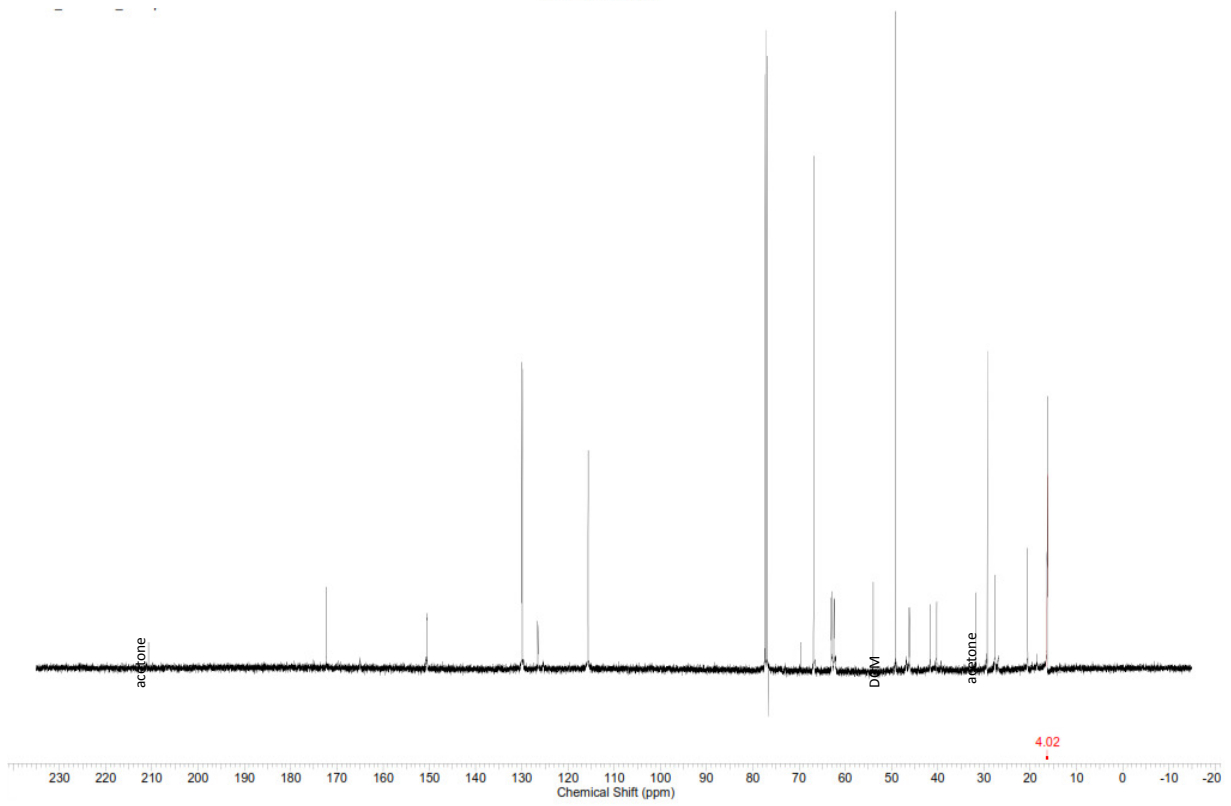
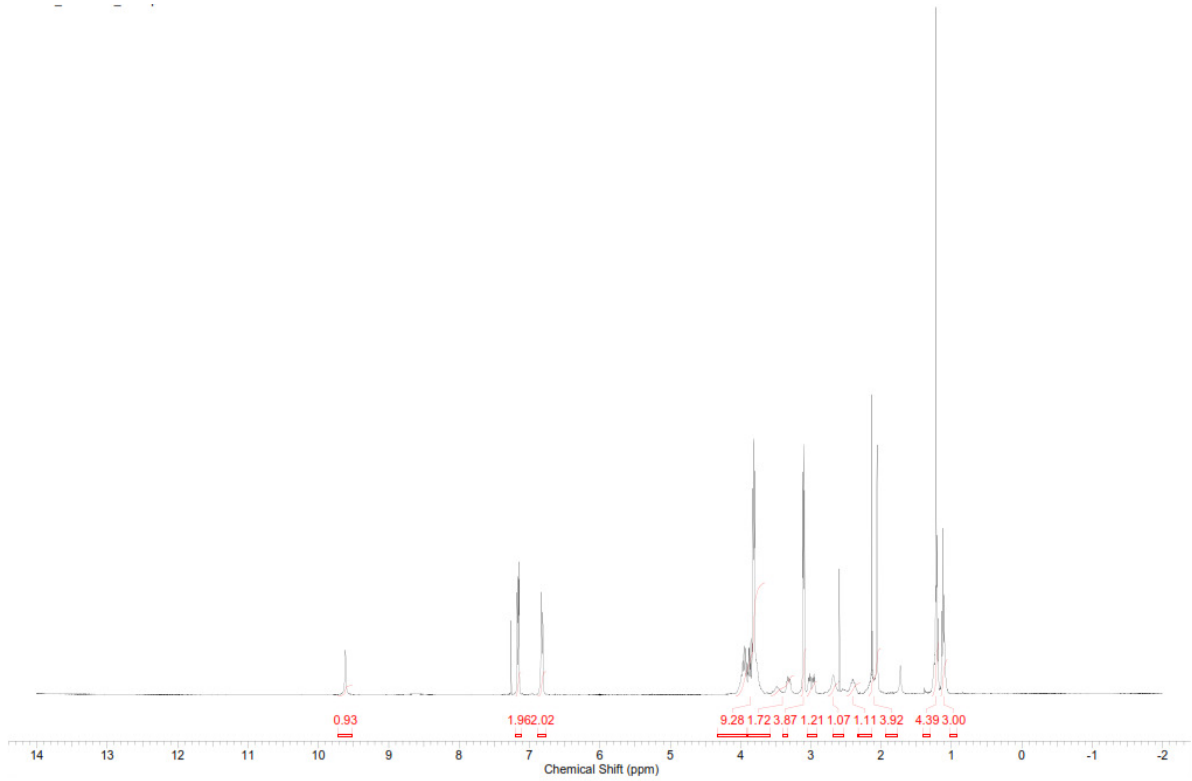
# $^1\text{H}$ NMR and $^{13}\text{C}$ NMR 12i



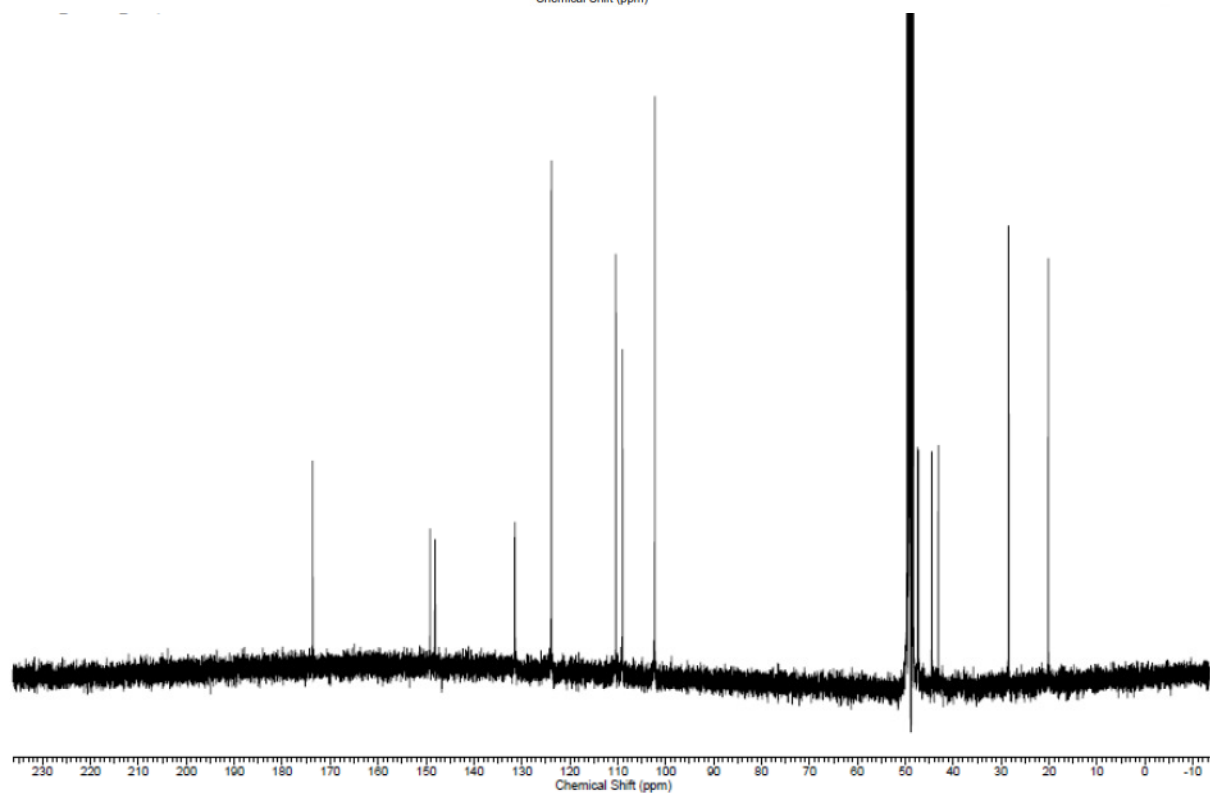
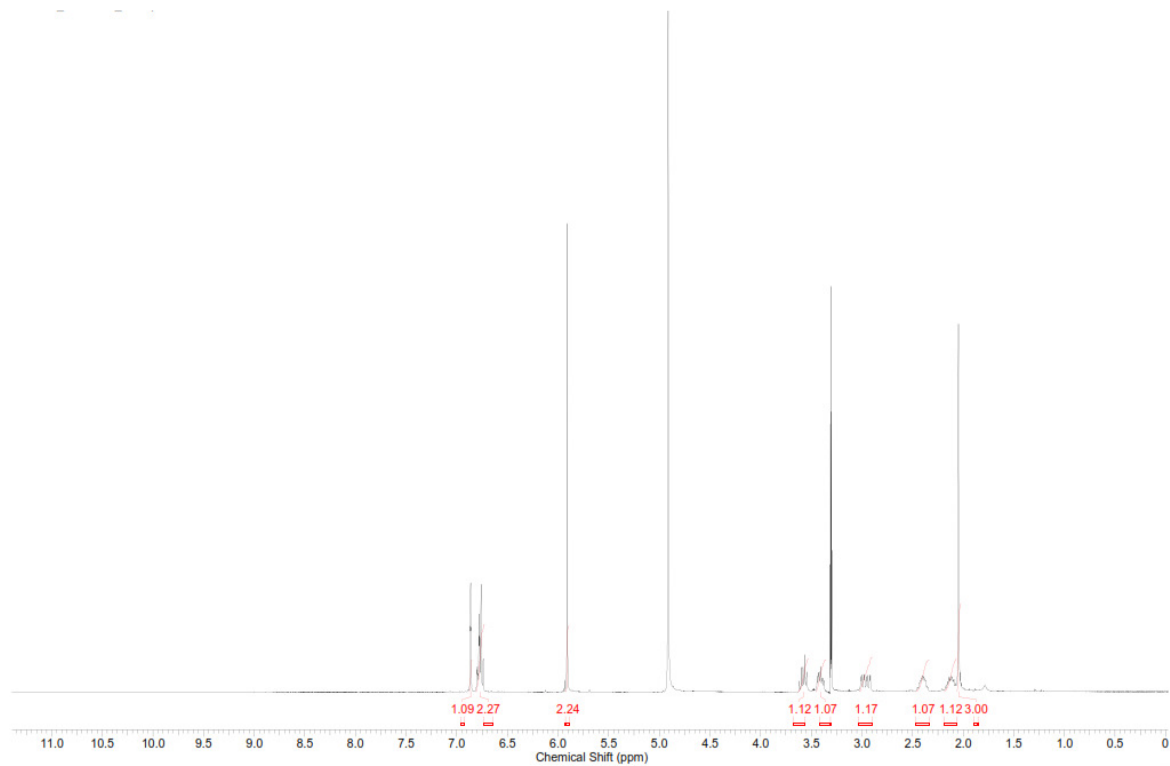
# $^1\text{H}$ NMR and $^{13}\text{C}$ NMR 12j



# $^1\text{H}$ NMR and $^{13}\text{C}$ NMR 12k

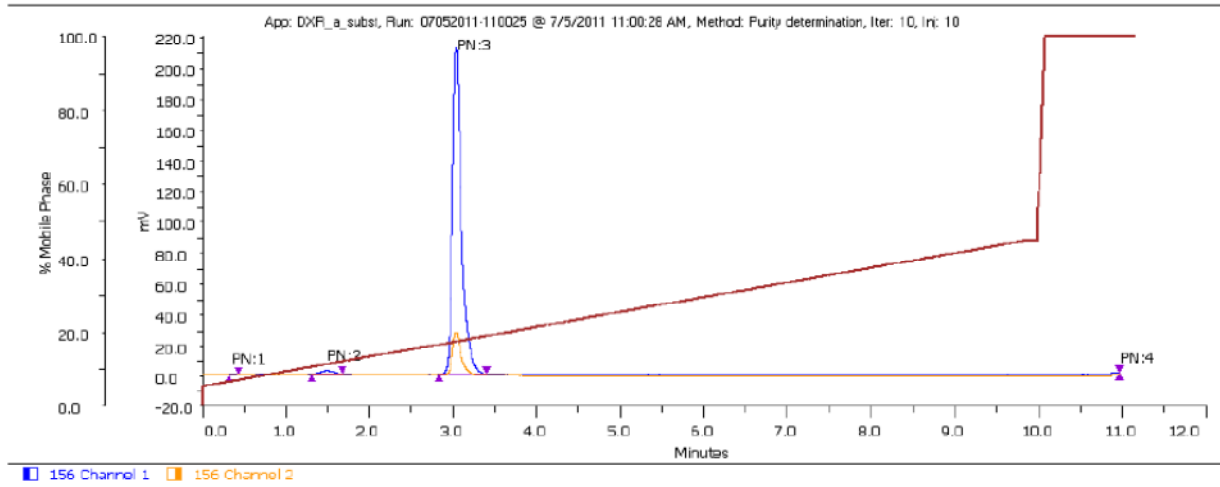


# $^1\text{H}$ NMR and $^{13}\text{C}$ NMR 13a

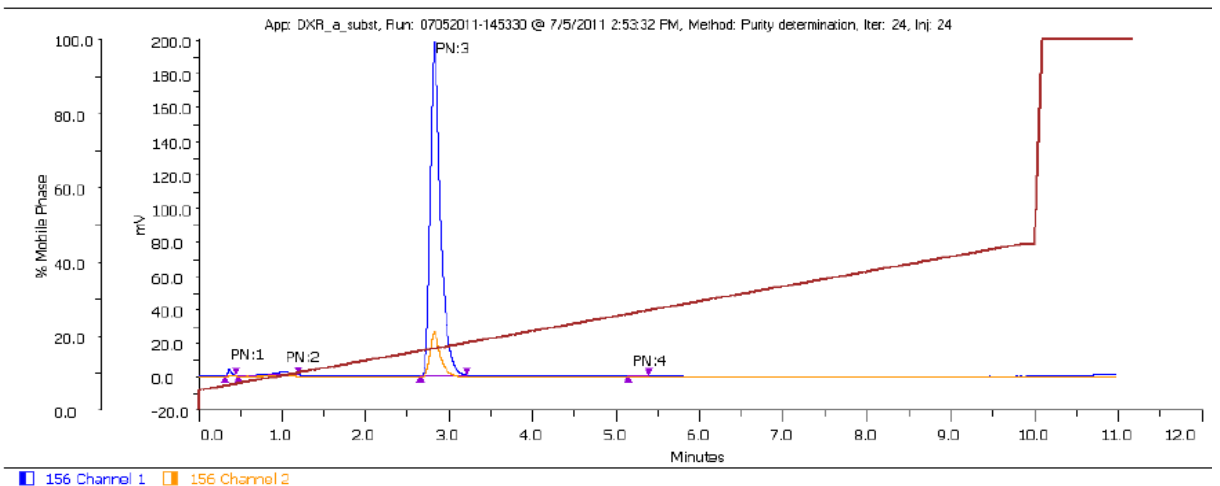




# HPLC-MS 13a

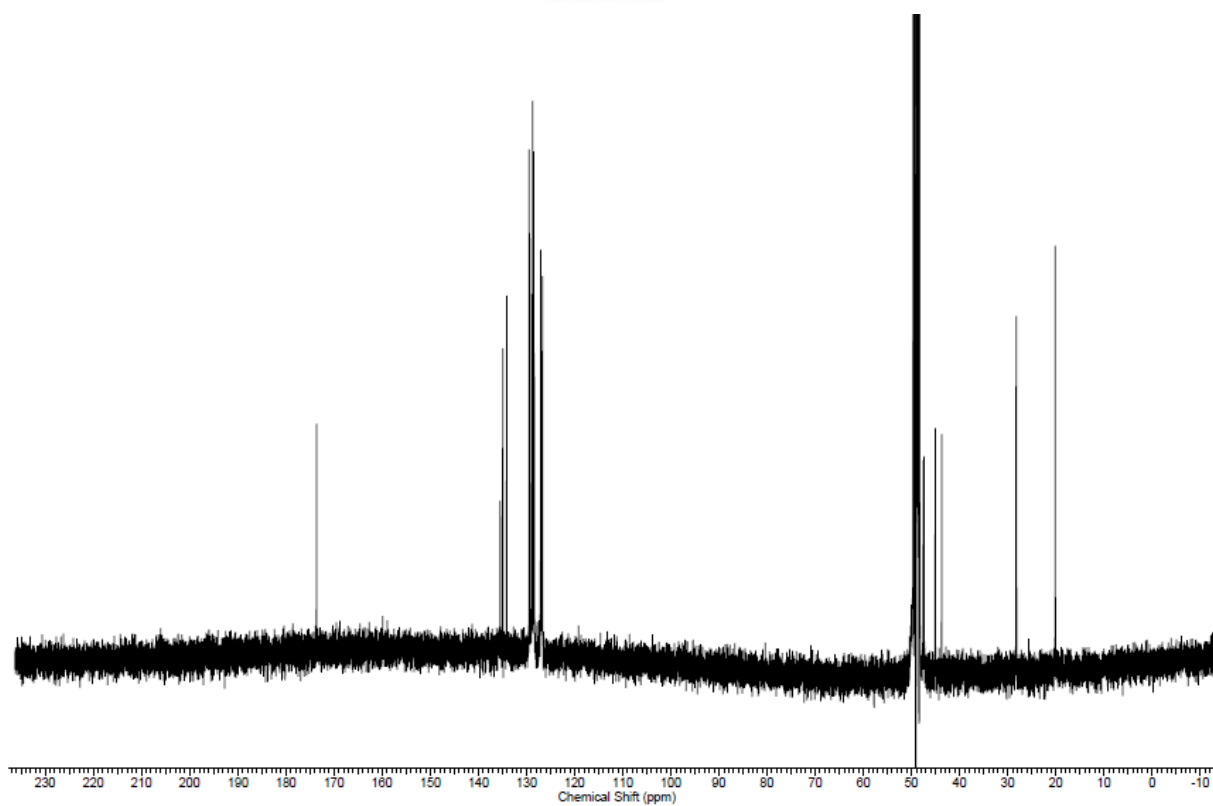
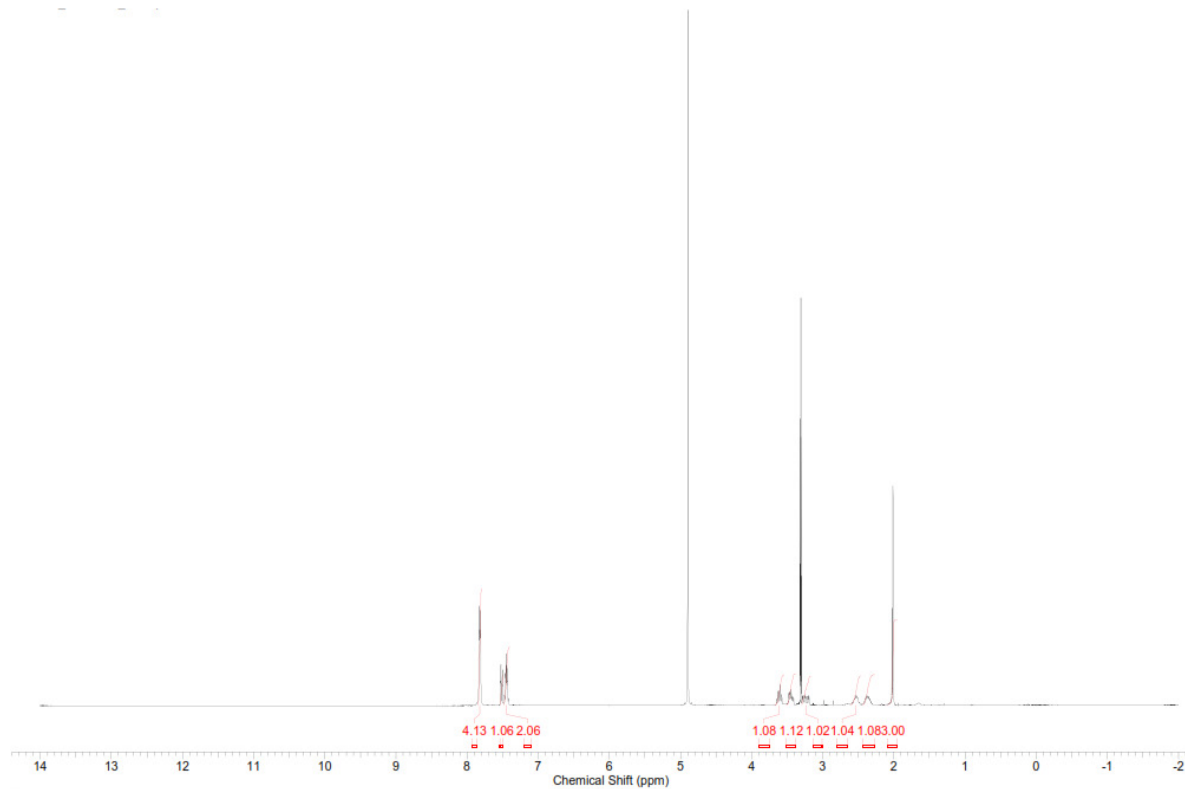


Injection Number	Peak Name	Retention Time (min)	Area (uVmin x100)	Area %	Sample Name	Sample Location	Fraction Site(s)
10	1	0.398	4510.8333	0.168	AN150_b	Sample Zone-->10	
10	2	1.483	40808.3333	1.523	AN150_b	Sample Zone-->10	
10	3	3.038	2633778.3333	98.308	AN150_b	Sample Zone-->10	
10	4	10.963	1.6667	0	AN150_b	Sample Zone-->10	

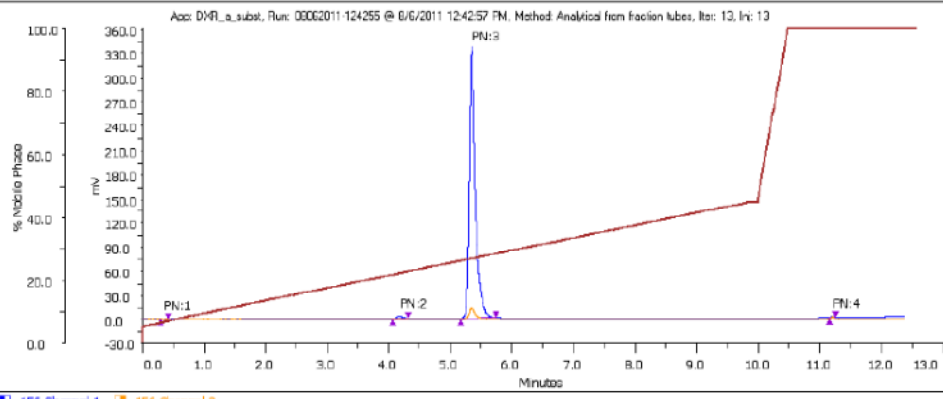


Injection Number	Peak Name	Retention Time (min)	Area (uVmin x100)	Area %	Sample Name	Sample Location	Fraction Site(s)
24	1	0.368	15516.6667	0.54	AN150_C18	Sample Zone-->11	
24	2	1.027	53141.25	1.851	AN150_C18	Sample Zone-->11	
24	3	2.828	2799876.6667	97.52	AN150_C18	Sample Zone-->11	
24	4	5.192	2535	0.088	AN150_C18	Sample Zone-->11	

# $^1\text{H}$ NMR and $^{13}\text{C}$ NMR 13b



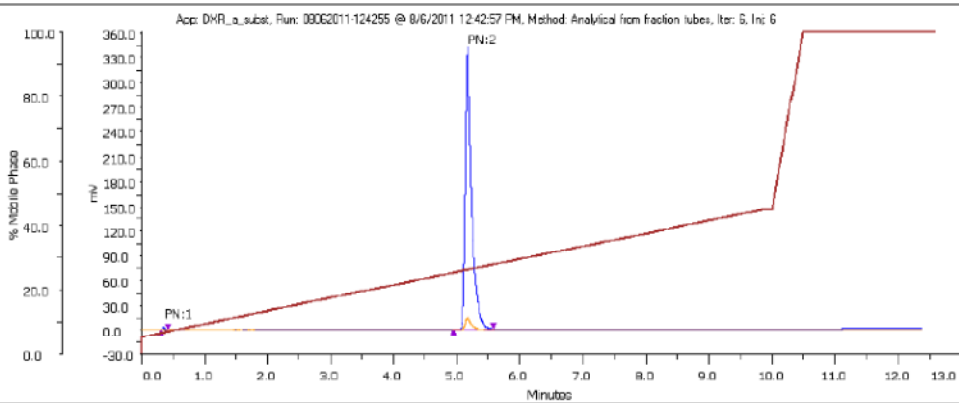
# HPLC-MS 13b



156 Channel 1 156 Channel 2

Sample Table

Injection Number	Peak Name	Retention Time (min)	Area (uVmin x100)	Area %	Sample Name	Sample Location	Fraction Site(s)
13	1	0.349	6932.5	0.181	AN149_B	Fraction Zone->2	
13	2	4.171	25193.3333	0.656	AN149_B	Fraction Zone->2	
13	3	5.352	3802125.8333	99.054	AN149_B	Fraction Zone->2	
13	4	11.214	4179.5833	0.109	AN149_B	Fraction Zone->2	

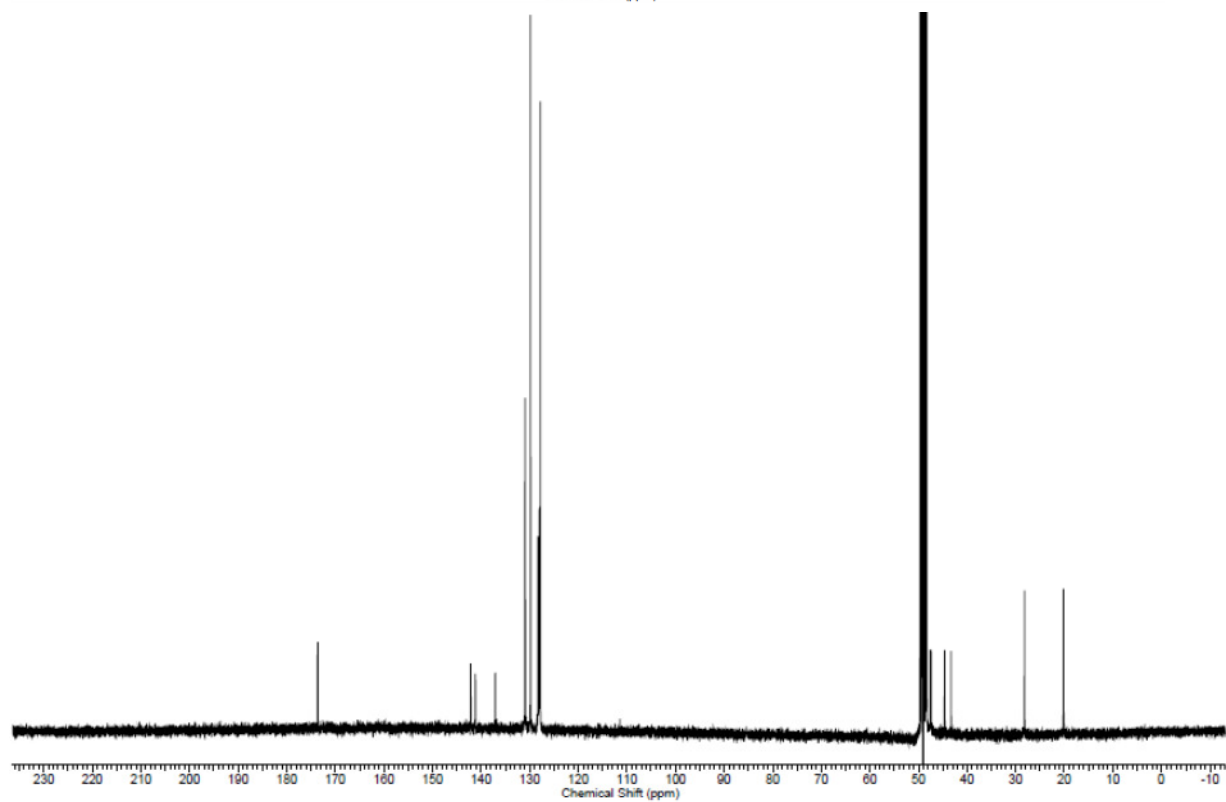
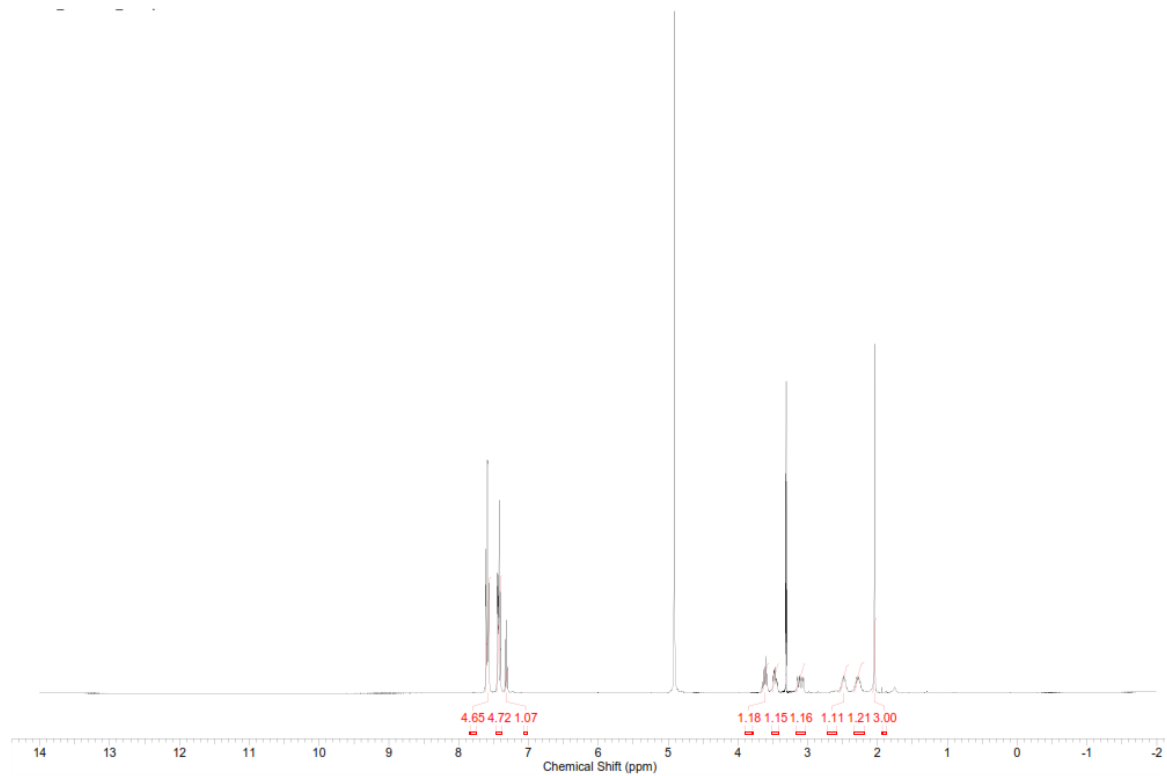


156 Channel 1 156 Channel 2

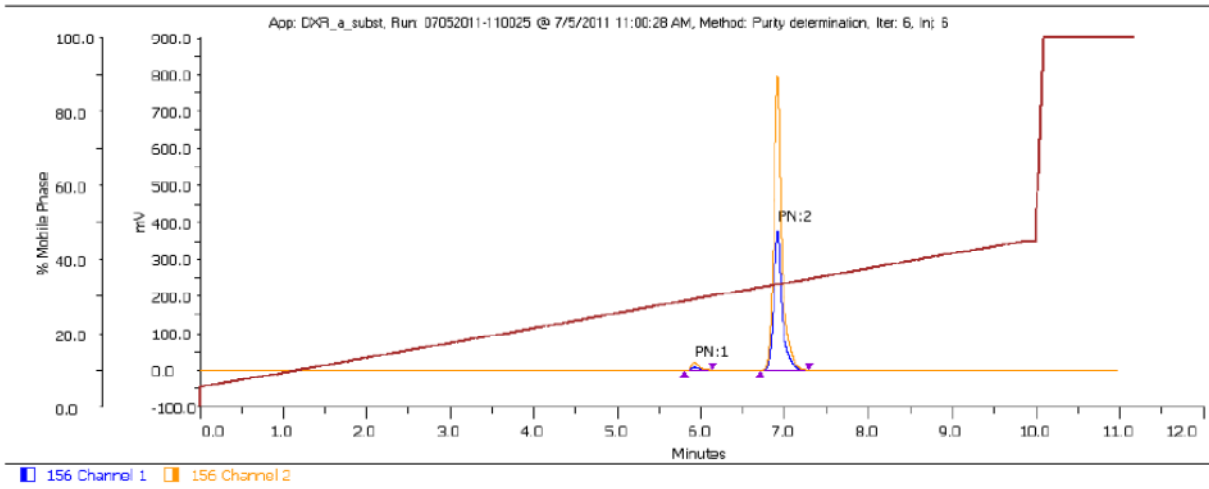
Sample Table

Injection Number	Peak Name	Retention Time (min)	Area (uVmin x100)	Area %	Sample Name	Sample Location	Fraction Site(s)
6	1	0.353	8465.8333	0.213	AN149	Fraction Zone->2	
6	2	5.177	3962759.1667	99.787	AN149	Fraction Zone->2	

# $^1\text{H}$ NMR and $^{13}\text{C}$ NMR 13c

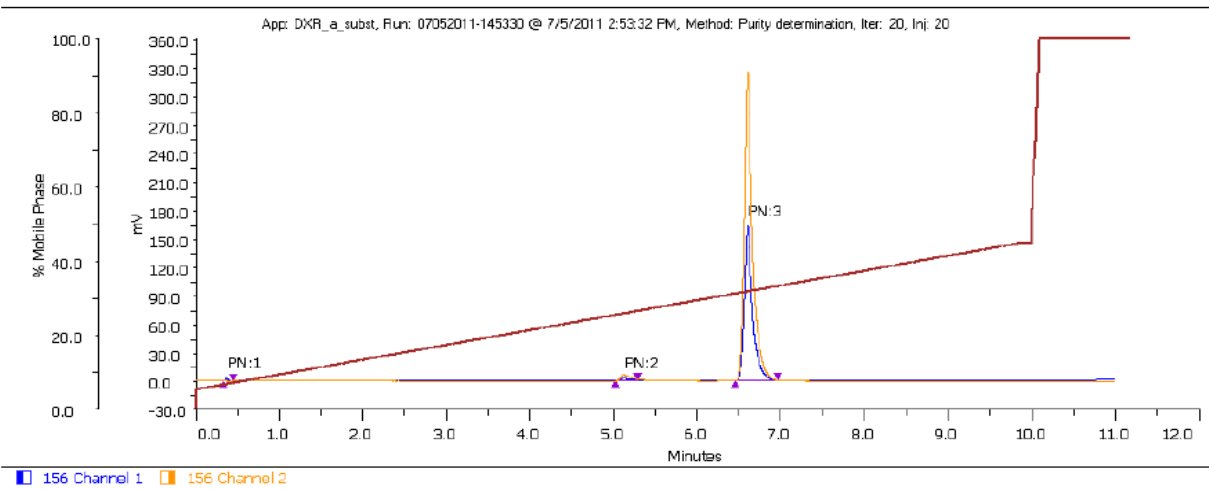


# HPLC-MS 13c



Sample Table

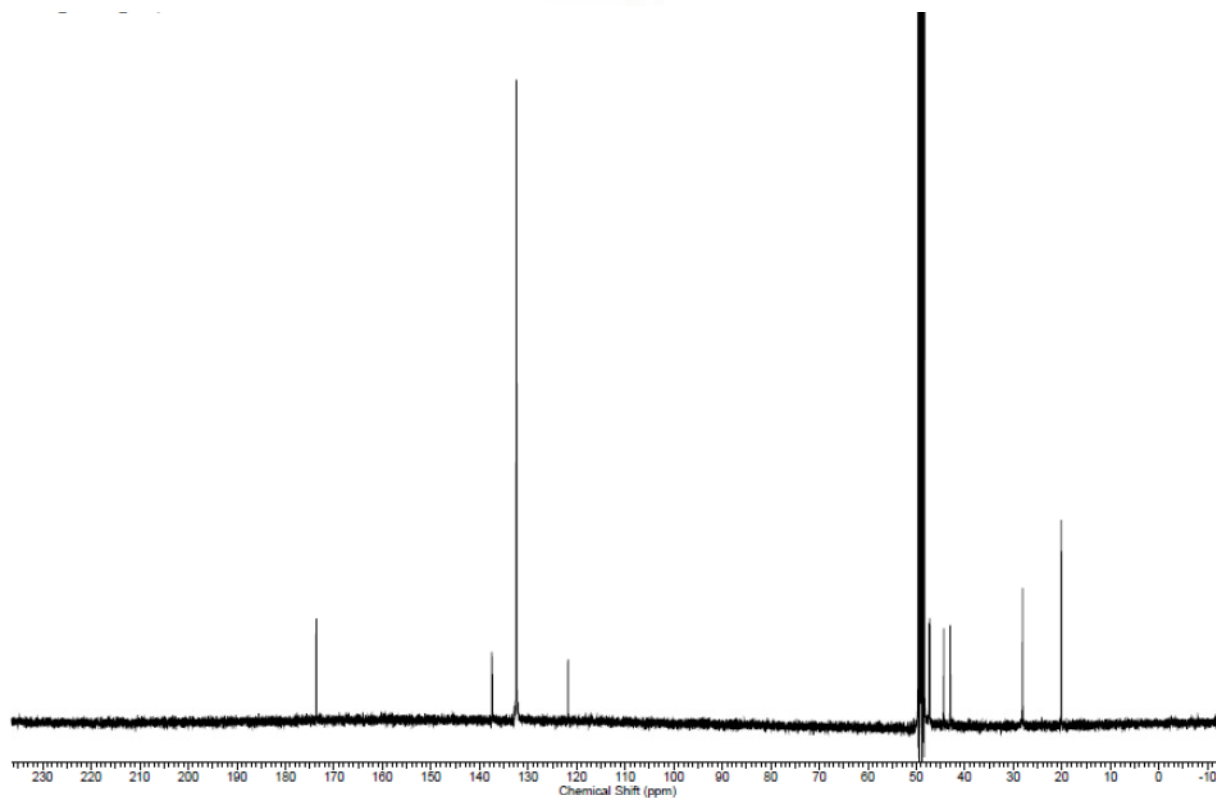
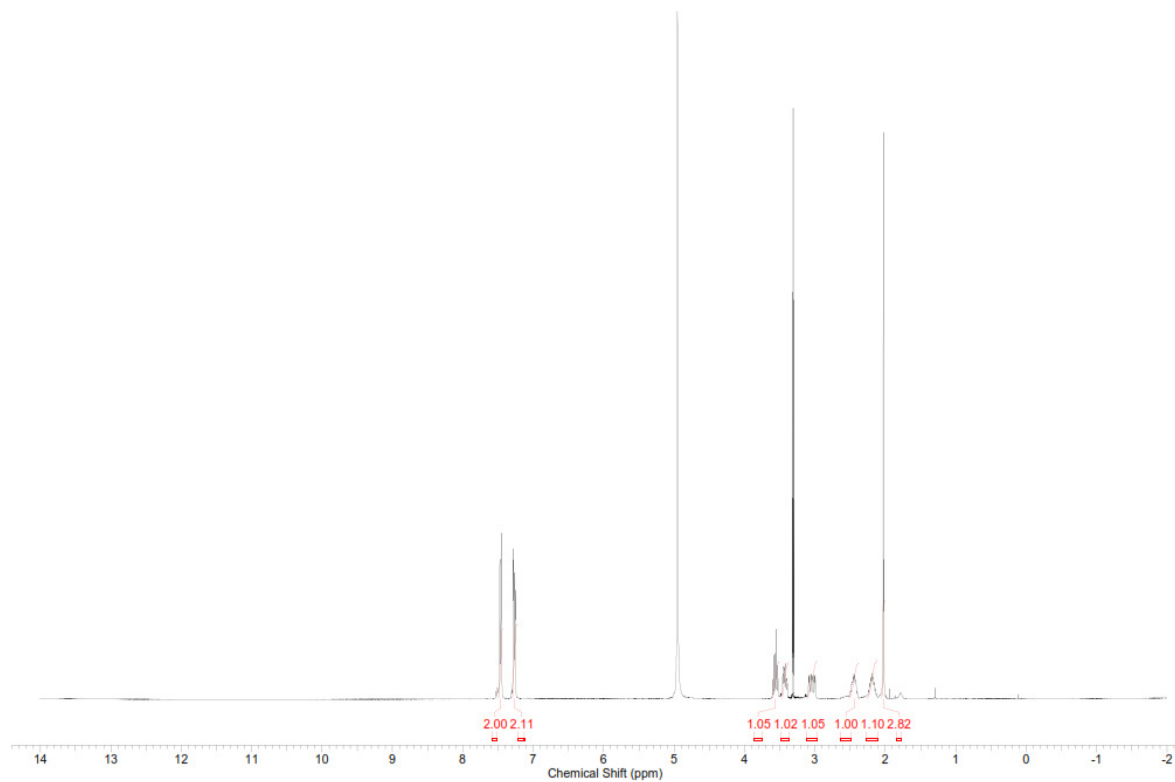
Injection Number	Peak Name	Retention Time (min)	Area (uVmin x100)	Area %	Sample Name	Sample Location	Fraction Site(s)
6	1	5.925	89540.8333	2.024	AN134_b	Sample Zone->2	
6	2	6.921	4335217.9167	97.976	AN134_b	Sample Zone->2	



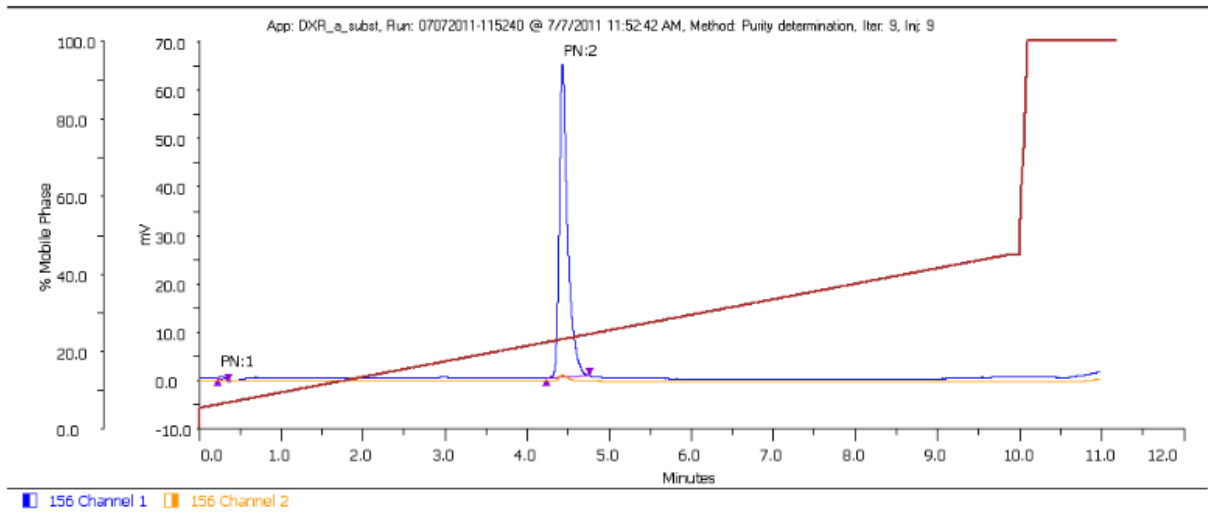
Sample Table

Injection Number	Peak Name	Retention Time (min)	Area (uVmin x100)	Area %	Sample Name	Sample Location	Fraction Site(s)
20	1	0.37	9779.1667	0.524	AN134_C18	Sample Zone->3	
20	2	5.127	25957.5	1.391	AN134_C18	Sample Zone->3	
20	3	6.611	1830722.5	98.085	AN134_C18	Sample Zone->3	

# $^1\text{H}$ NMR and $^{13}\text{C}$ NMR 13d

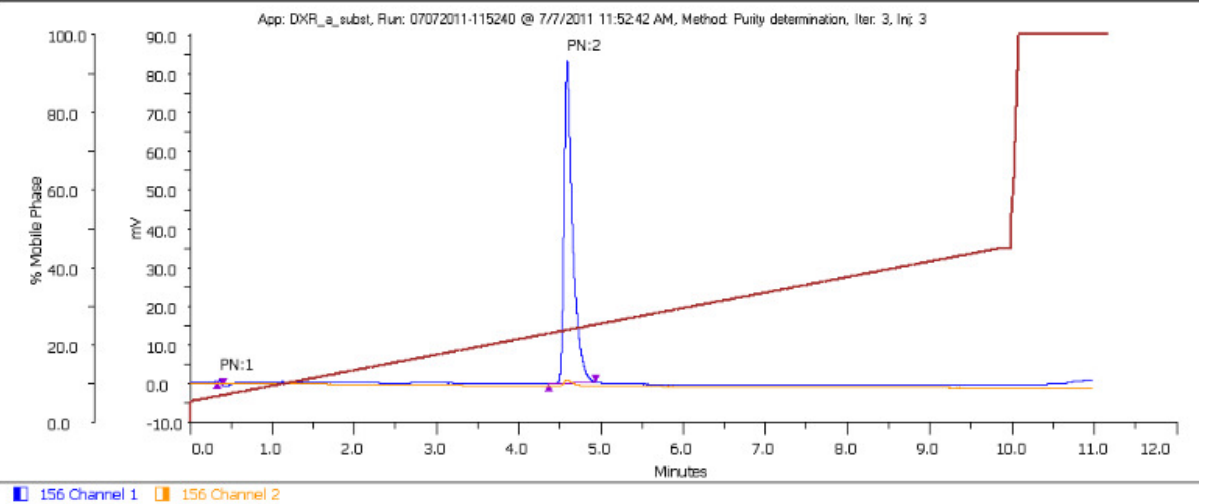


# HPLC-MS 13d



Sample Table

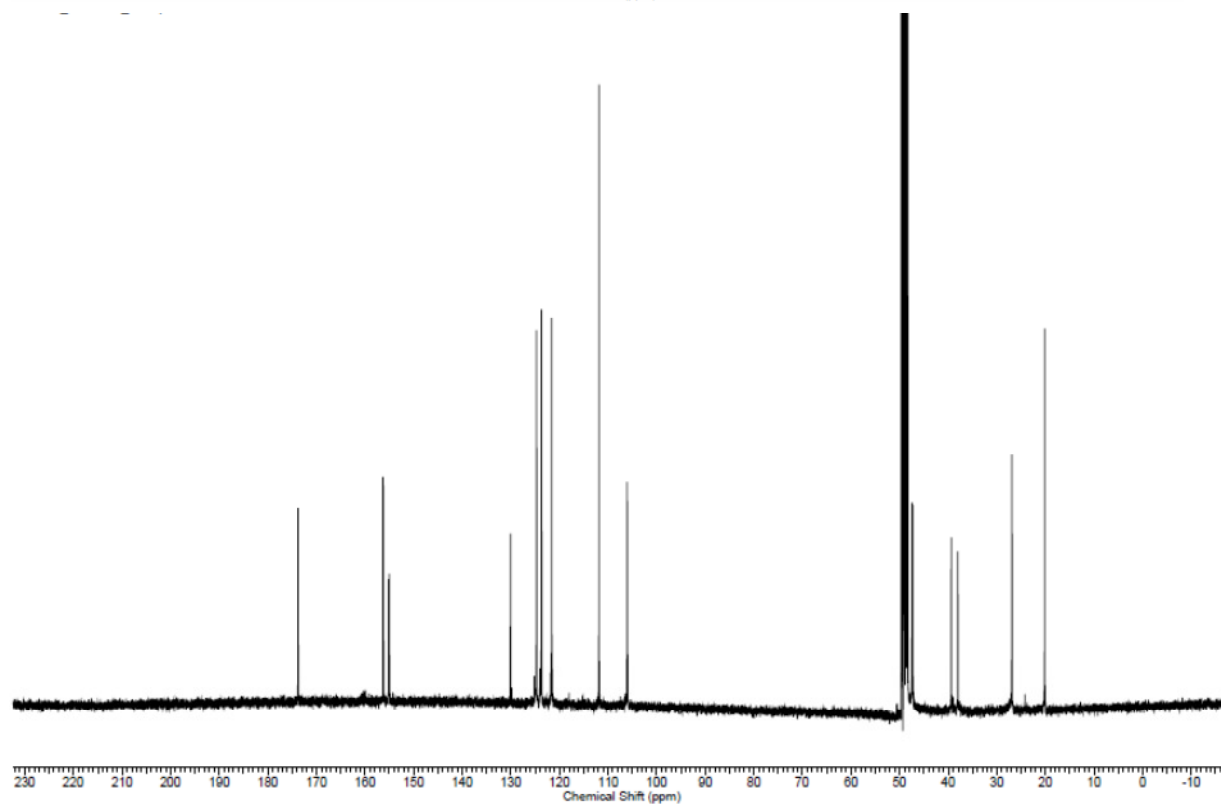
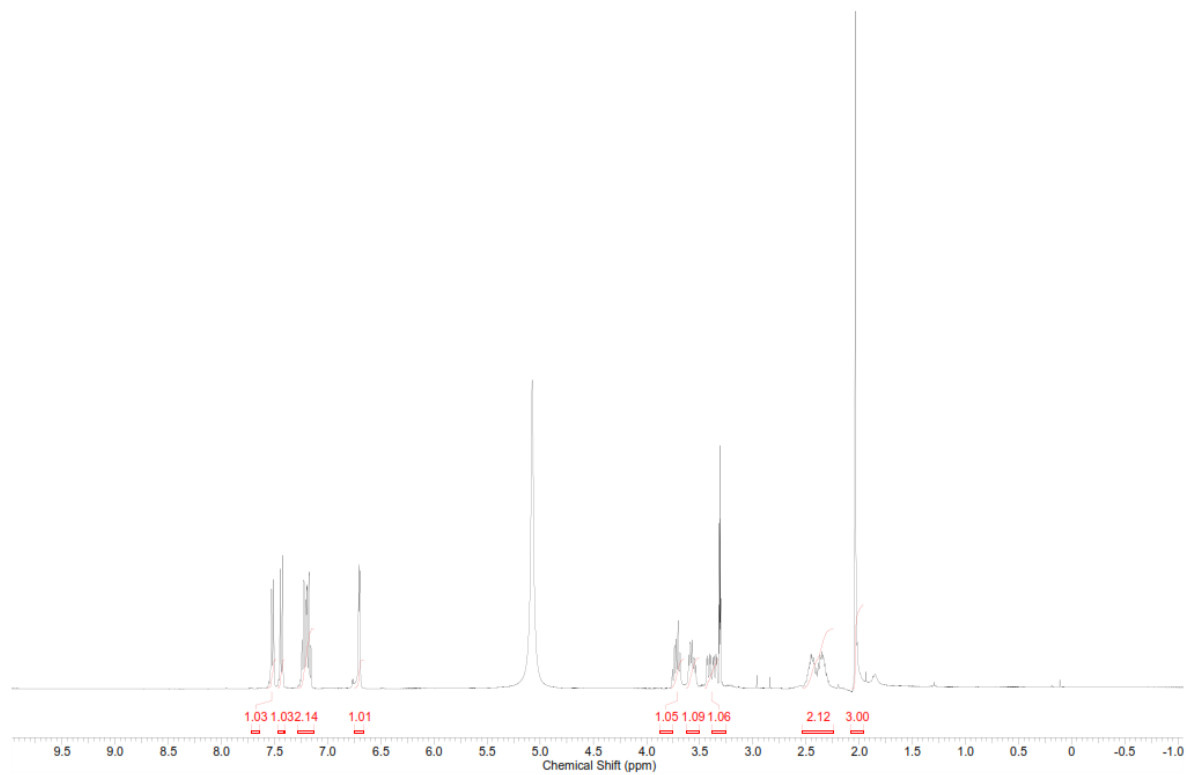
Injection Number	Peak Name	Retention Time (min)	Area (uVmin x100)	Area %	Sample Name	Sample Location	Fraction Site(s)
9	1	0.309	4522.5	0.591	AN166_b	Sample Zone->2	
9	2	4.436	760137.9167	99.409	AN166_b	Sample Zone->2	



Sample Table

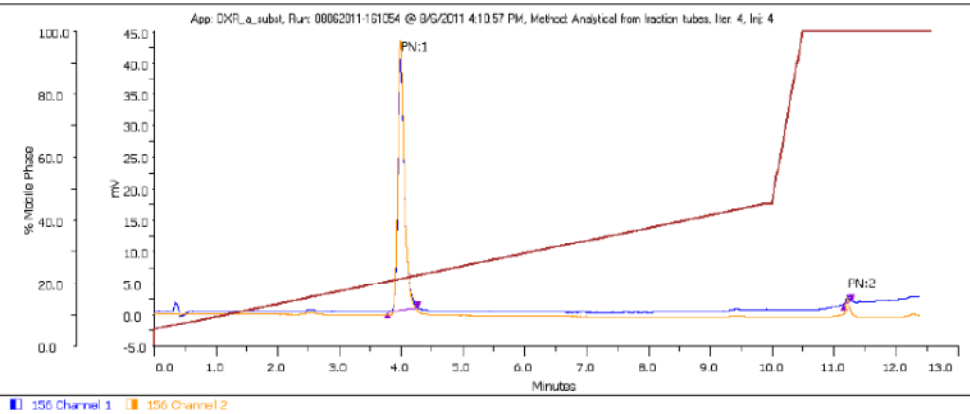
Injection Number	Peak Name	Retention Time (min)	Area (uVmin x100)	Area %	Sample Name	Sample Location	Fraction Site(s)
3	1	0.365	2530	0.253	AN166_C18	Sample Zone->2	
3	2	4.594	997845	99.747	AN166_C18	Sample Zone->2	

# $^1\text{H}$ NMR and $^{13}\text{C}$ NMR 13e



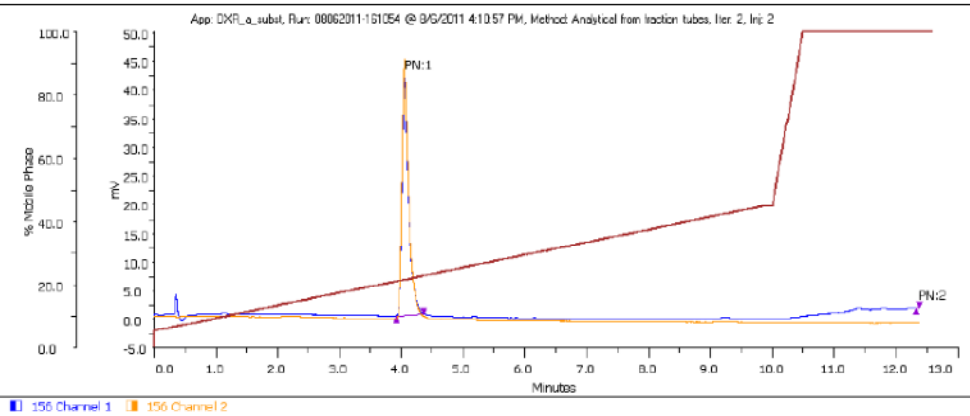


# HPLC-MS 13e



Sample Table

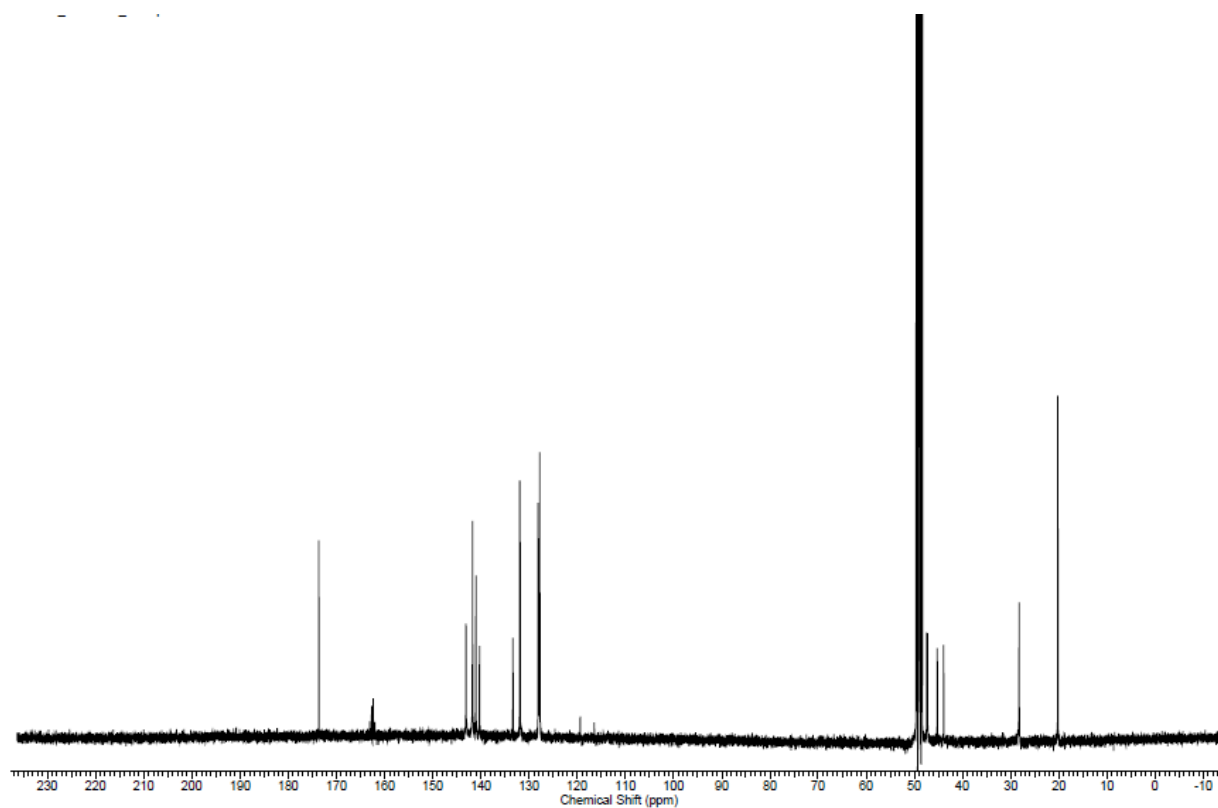
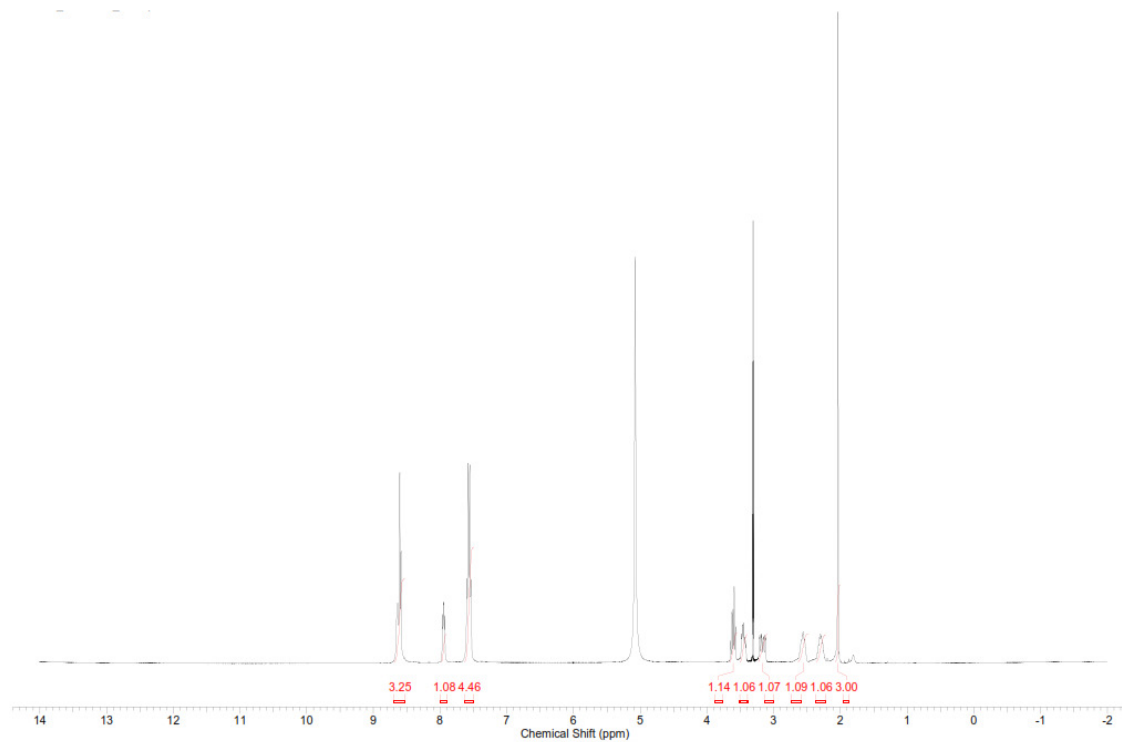
Injection Number	Peak Name	Retention Time (min)	Area (uVmin x100)	Area %	Sample Name	Sample Location	Fraction Site(s)
4	1	3.993	491699.5849	98.967	AN163-B	Fraction Zone->16	
4	2	11.226	5130.833	1.033	AN163-B	Fraction Zone->16	



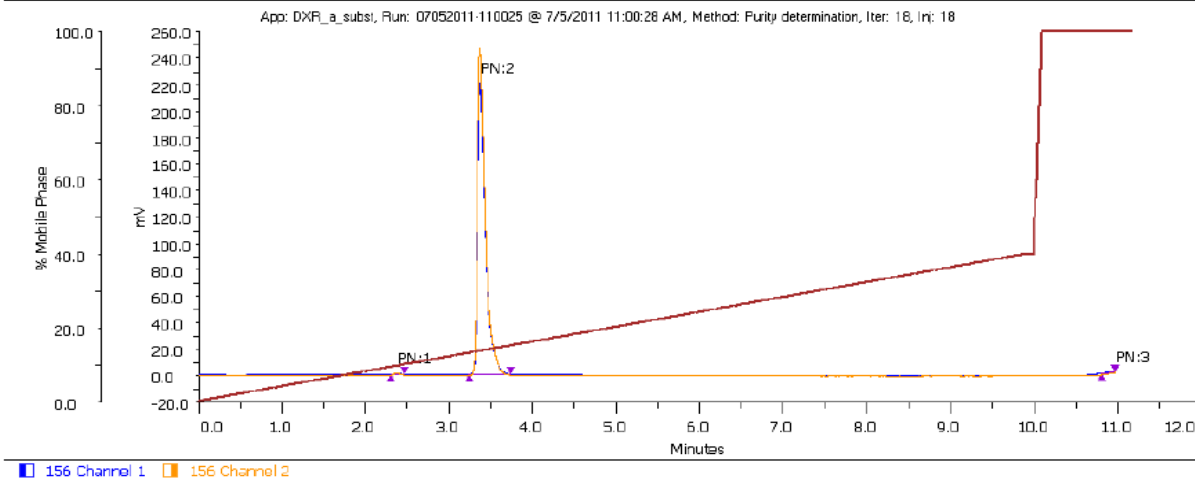
Sample Table

Injection Number	Peak Name	Retention Time (min)	Area (uVmin x100)	Area %	Sample Name	Sample Location	Fraction Site(s)
2	1	4.056	524838.3324	99.992	AN163-C18	Fraction Zone->16	
2	2	12.355	39.9998	0.008	AN163-C18	Fraction Zone->16	

# $^1\text{H}$ NMR and $^{13}\text{C}$ NMR 13f

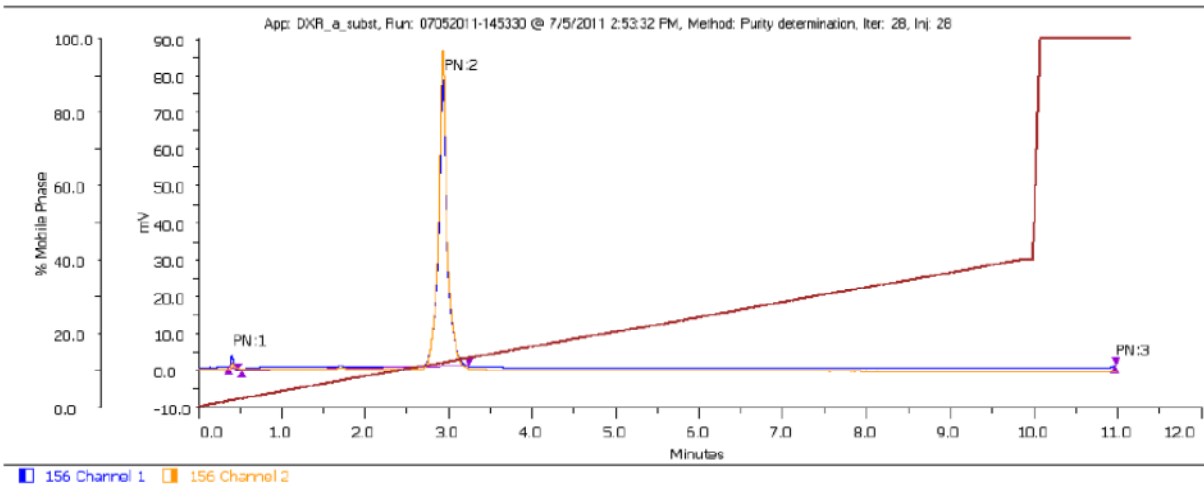


# HPLC-MS 13f



**Sample Table**

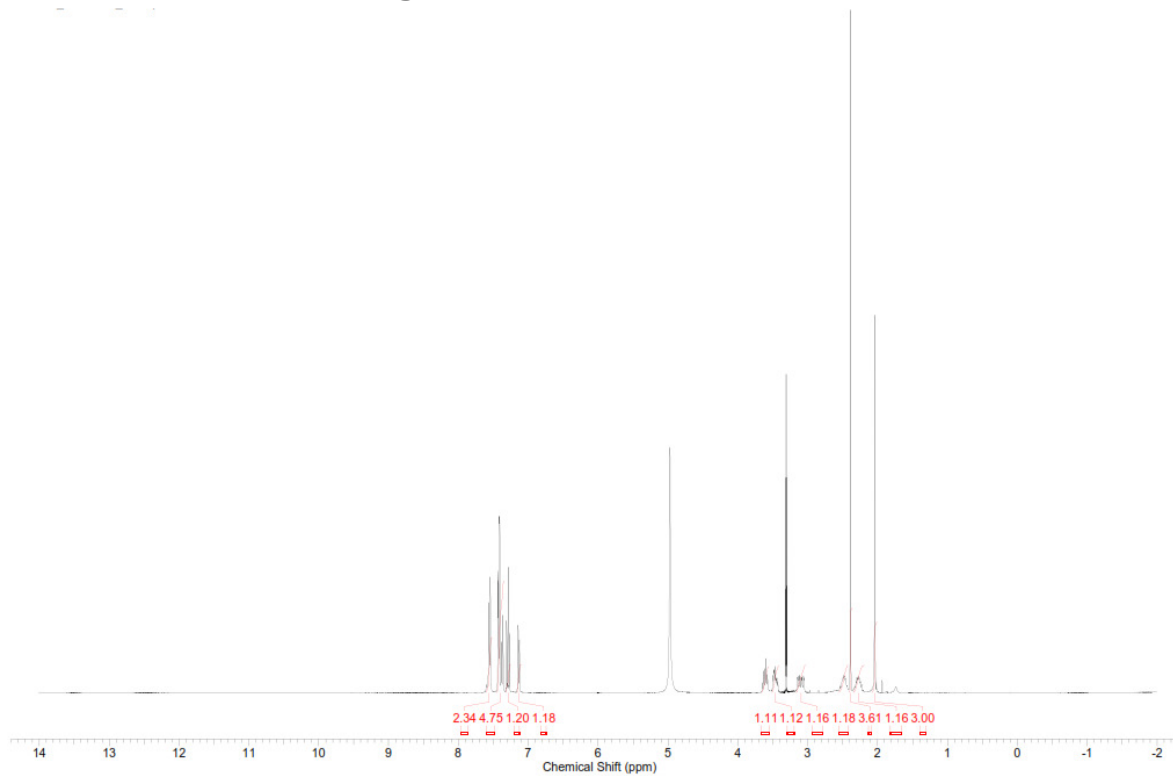
Injection Number	Peak Name	Retention Time (min)	Area (uVmin x100)	Area %	Sample Name	Sample Location	Fraction Site(s)
18	1	2.379	7543.3333	0.309	AN167_b	Sample Zone-->26	
18	2	3.378	2428355.8333	99.572	AN167_b	Sample Zone-->26	
18	3	10.904	2906.6667	0.119	AN167_b	Sample Zone-->26	



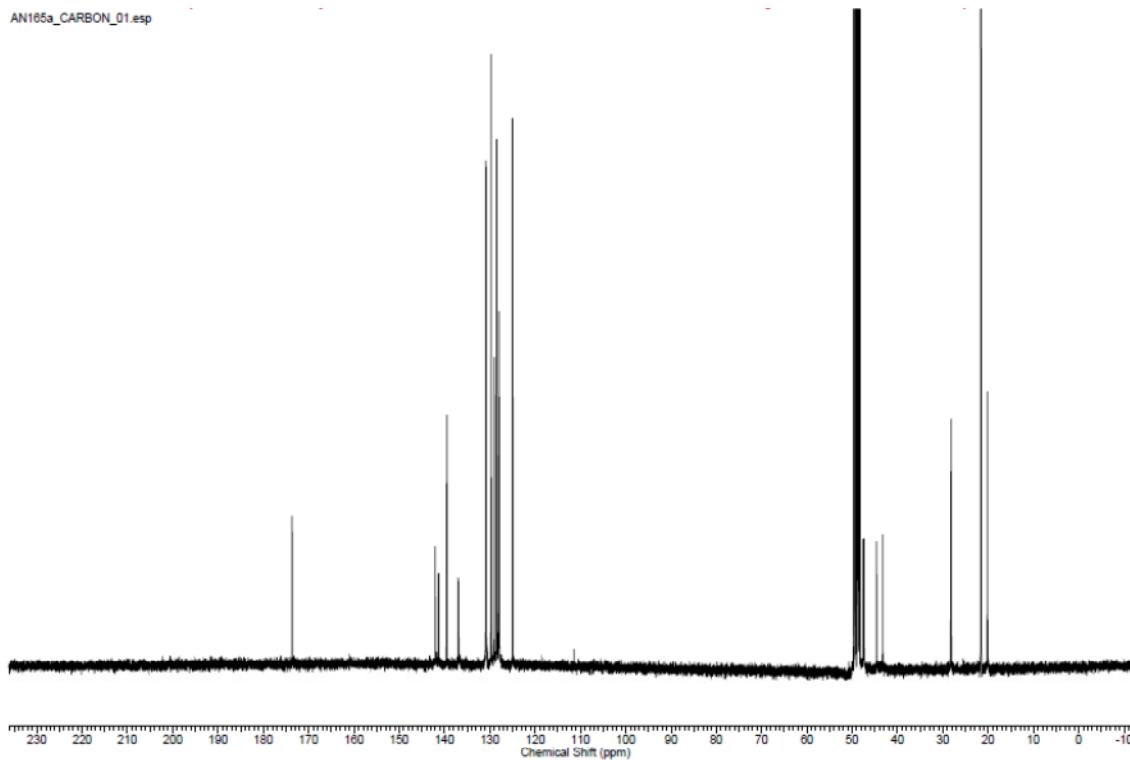
**Sample Table**

Injection Number	Peak Name	Retention Time (min)	Area (uVmin x100)	Area %	Sample Name	Sample Location	Fraction Site(s)
28	1	0.401	11255.8333	1.084	AN167_C18	Sample Zone-->19	
28	2	2.928	1027081.6667	98.916	AN167_C18	Sample Zone-->19	
28	3	10.908	1.6667	0	AN167_C18	Sample Zone-->19	

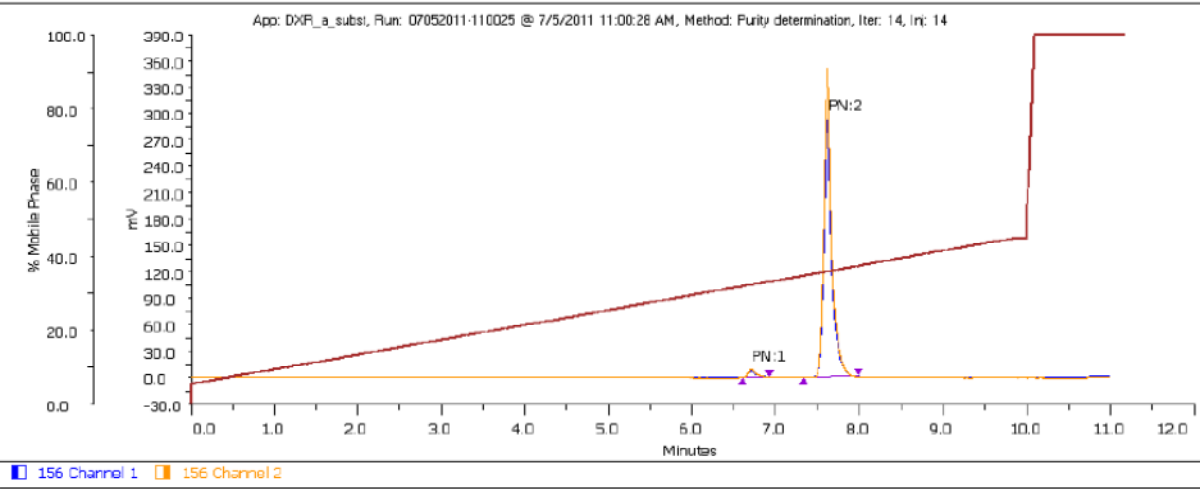
# $^1\text{H}$ NMR and $^{13}\text{C}$ NMR 13g



AN165a\_CARBON\_01.esp

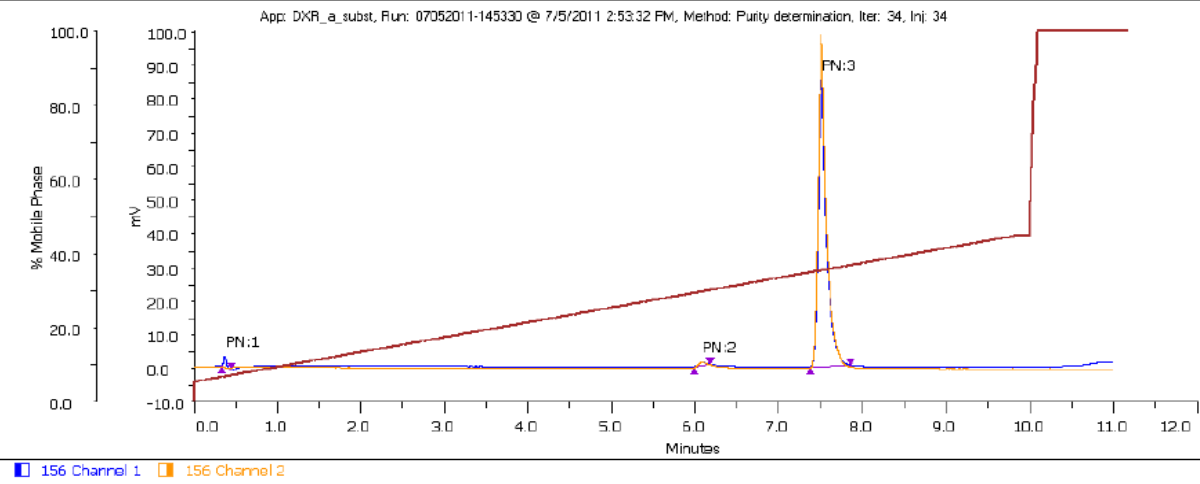


# HPLC-MS 13g



**Sample Table**

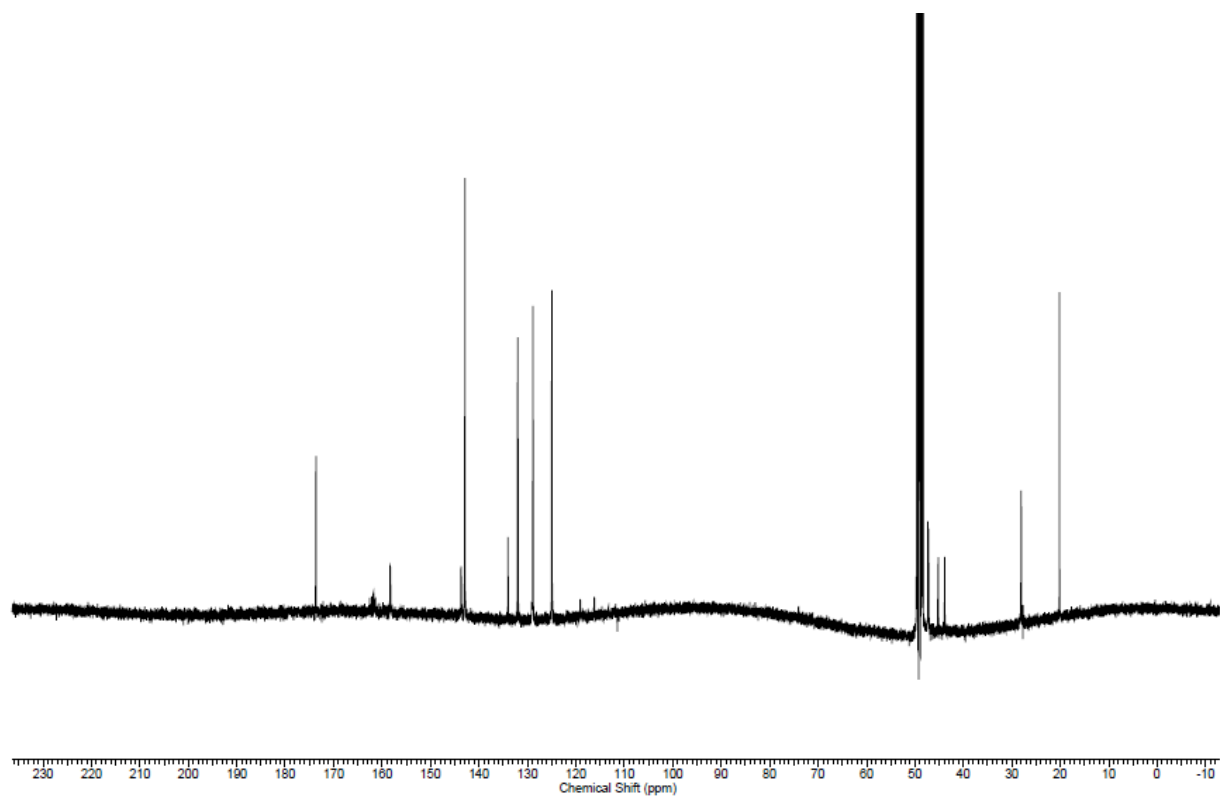
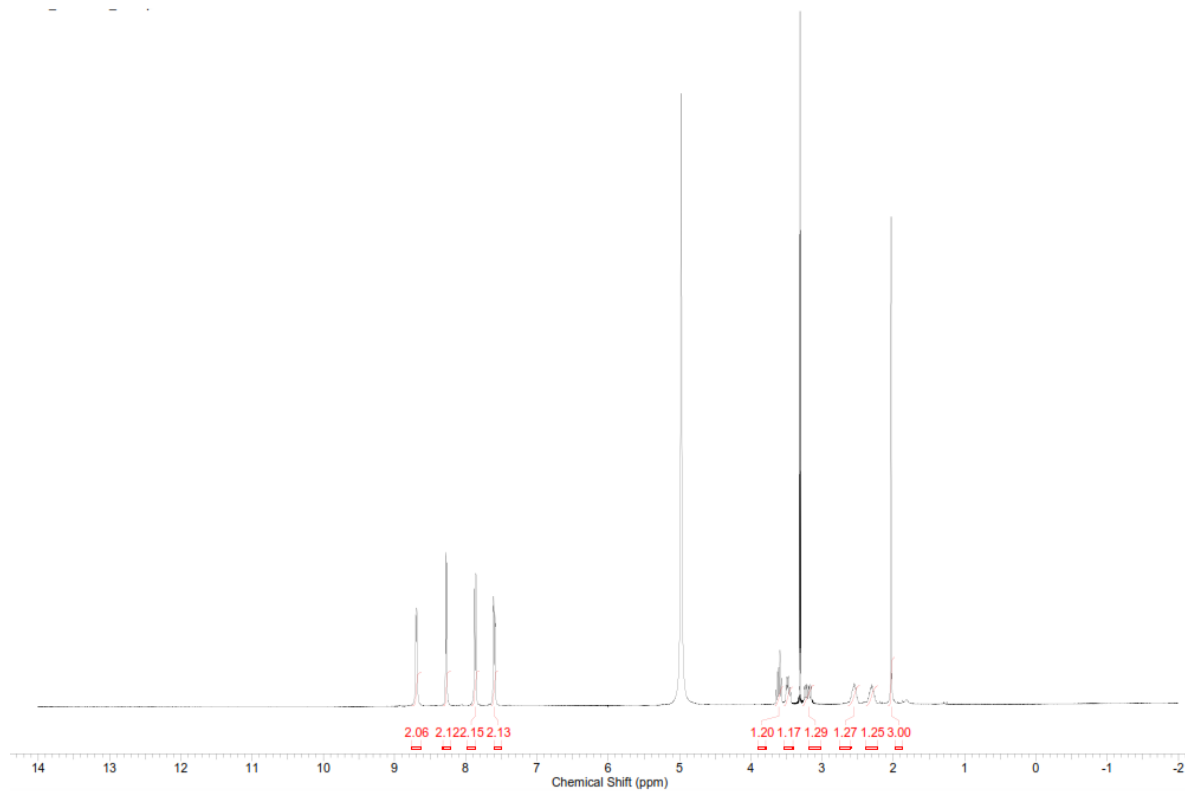
Injection Number	Peak Name	Retention Time (min)	Area (uVmin x100)	Area %	Sample Name	Sample Location	Fraction Site(s)
14	1	6.714	77273.75	2.314	AN165_b	Sample Zone->18	
14	2	7.627	3262549.1667	97.686	AN165_b	Sample Zone->18	



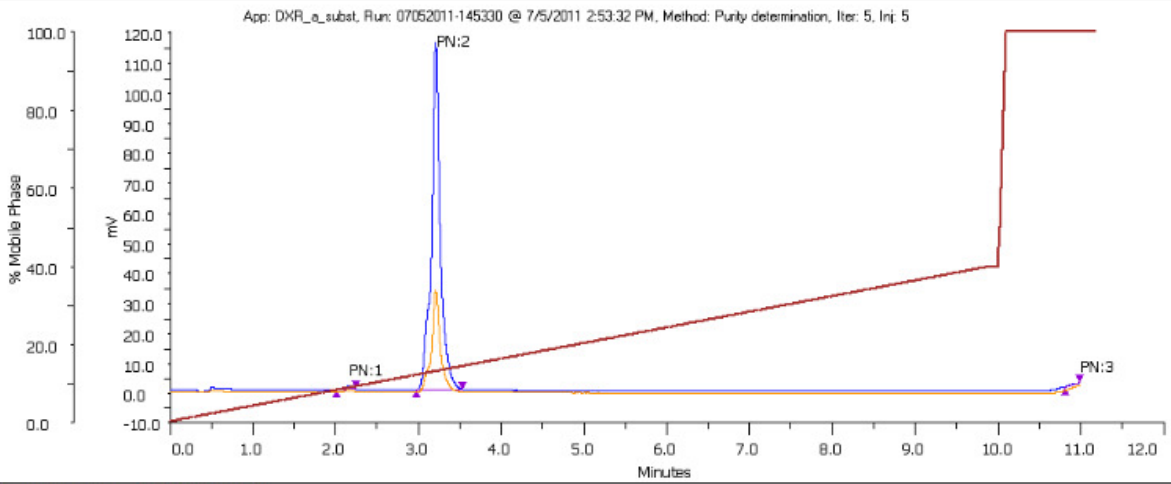
**Sample Table**

Injection Number	Peak Name	Retention Time (min)	Area (uVmin x100)	Area %	Sample Name	Sample Location	Fraction Site(s)
34	1	0.37	11995.8333	1.197	AN165_C18	Sample Zone->31	
34	2	6.061	9588.3333	0.956	AN165_C18	Sample Zone->31	
34	3	7.517	980957.5	97.847	AN165_C18	Sample Zone->31	

# $^1\text{H}$ NMR and $^{13}\text{C}$ NMR 13h

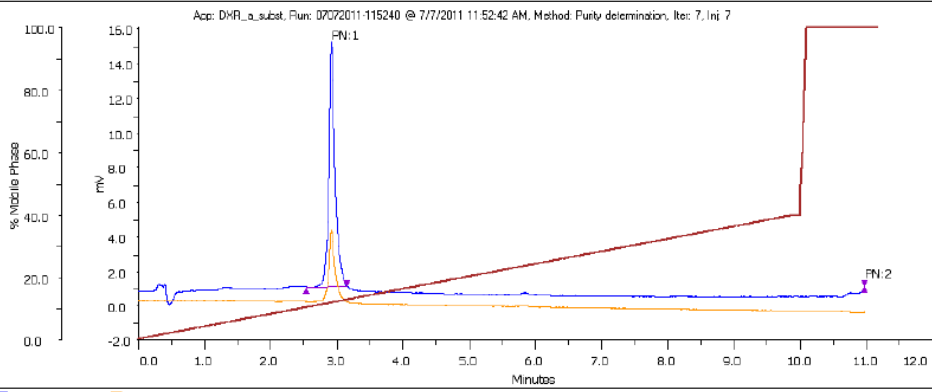


# HPLC-MS 13h



Sample Table

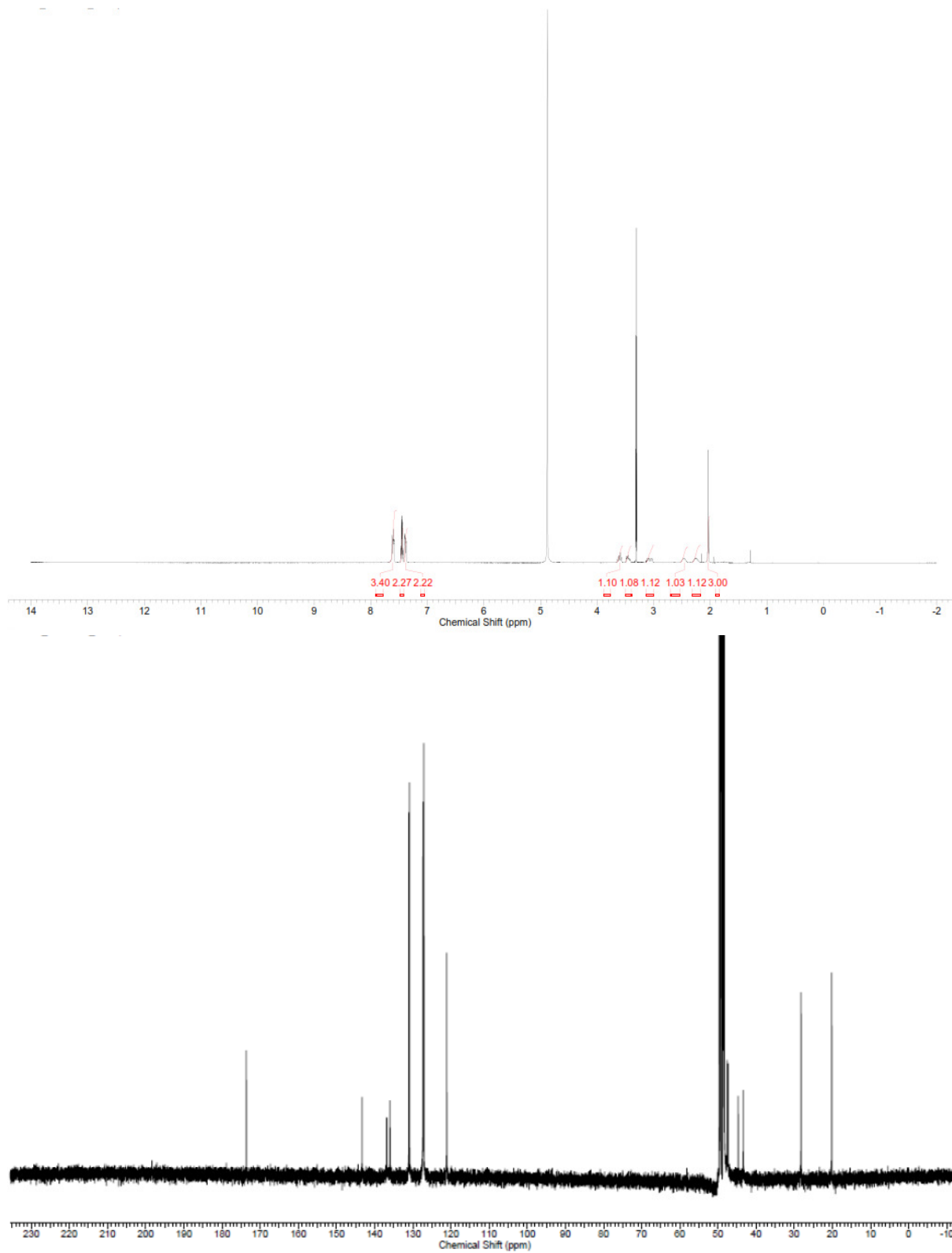
Injection Number	Peak Name	Retention Time (min)	Area (uVmin x100)	Area %	Sample Name	Sample Location	Fraction Site(s)
5	1	2.158	8697.5	0.597	AN179_B	Sample Zone->50	
5	2	3.213	1444770.4167	99.184	AN179_B	Sample Zone->50	
5	3	10.894	3182.0833	0.218	AN179_B	Sample Zone->50	



Sample Table

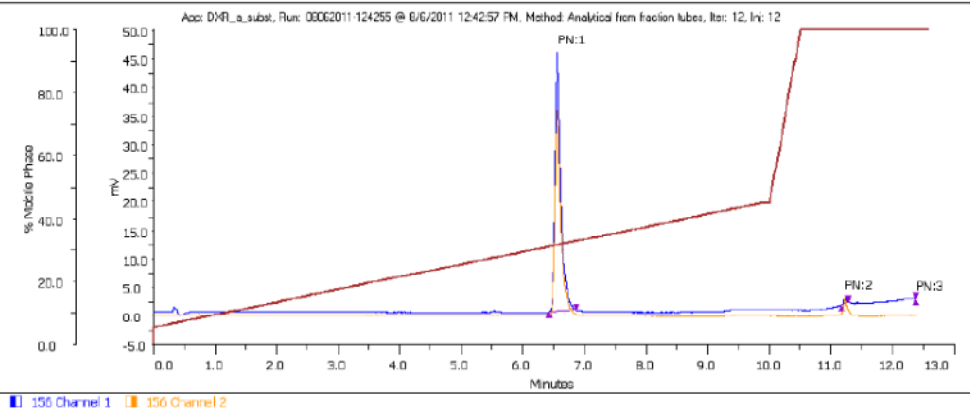
Injection Number	Peak Name	Retention Time (min)	Area (uVmin x100)	Area %	Sample Name	Sample Location	Fraction Site(s)
7	1	2.922	151603.111	99.999	AN179_C18	Sample Zone->10	
7	2	10.983	0.8333	0.001	AN179_C18	Sample Zone->10	

# <sup>1</sup>H NMR and <sup>13</sup>C NMR 13i



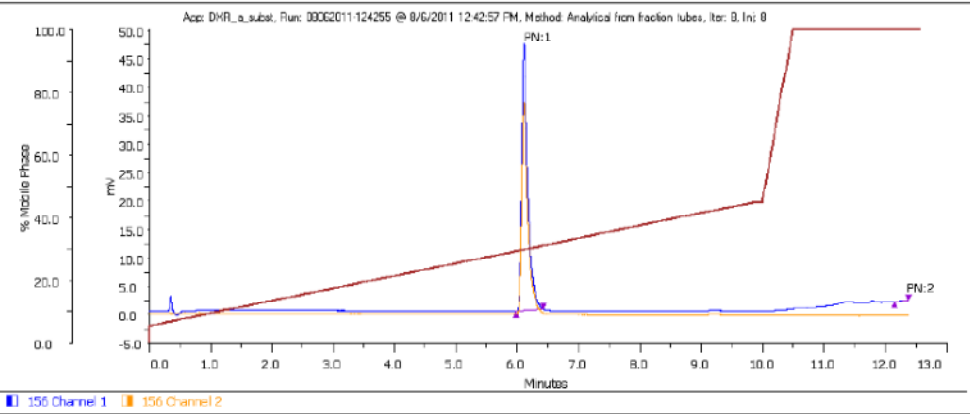


# HPLC-MS 13i



Sample Table

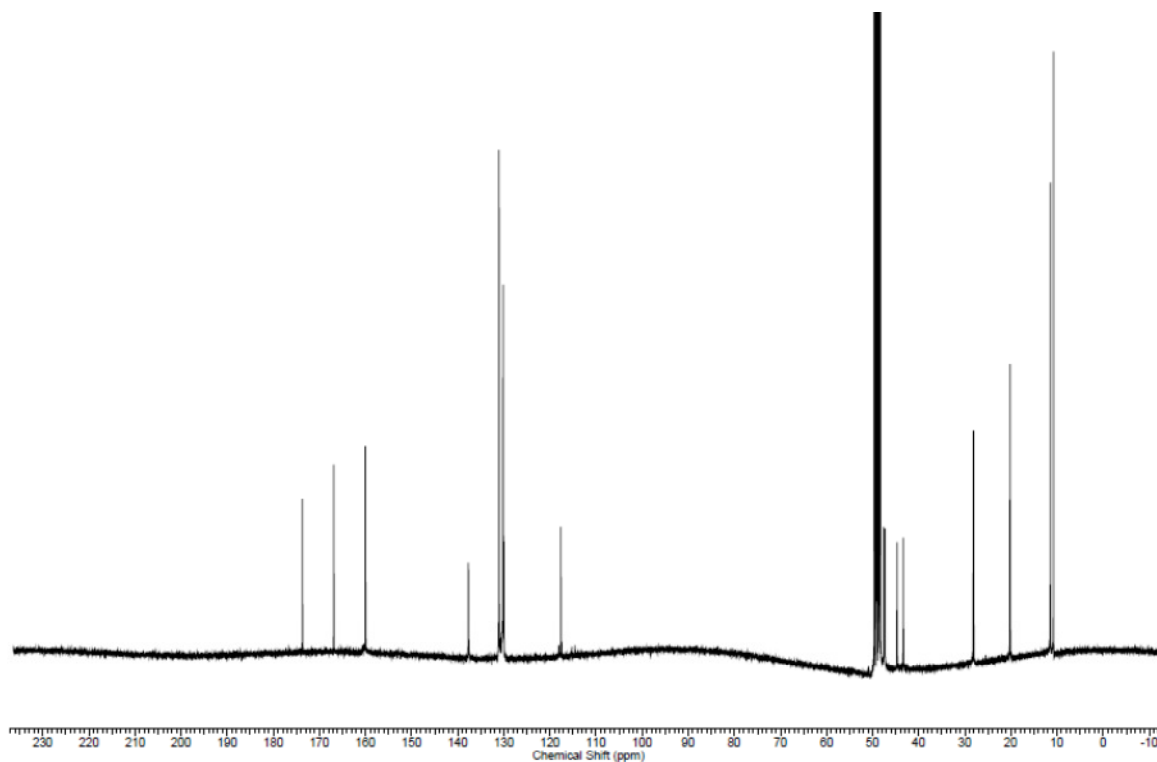
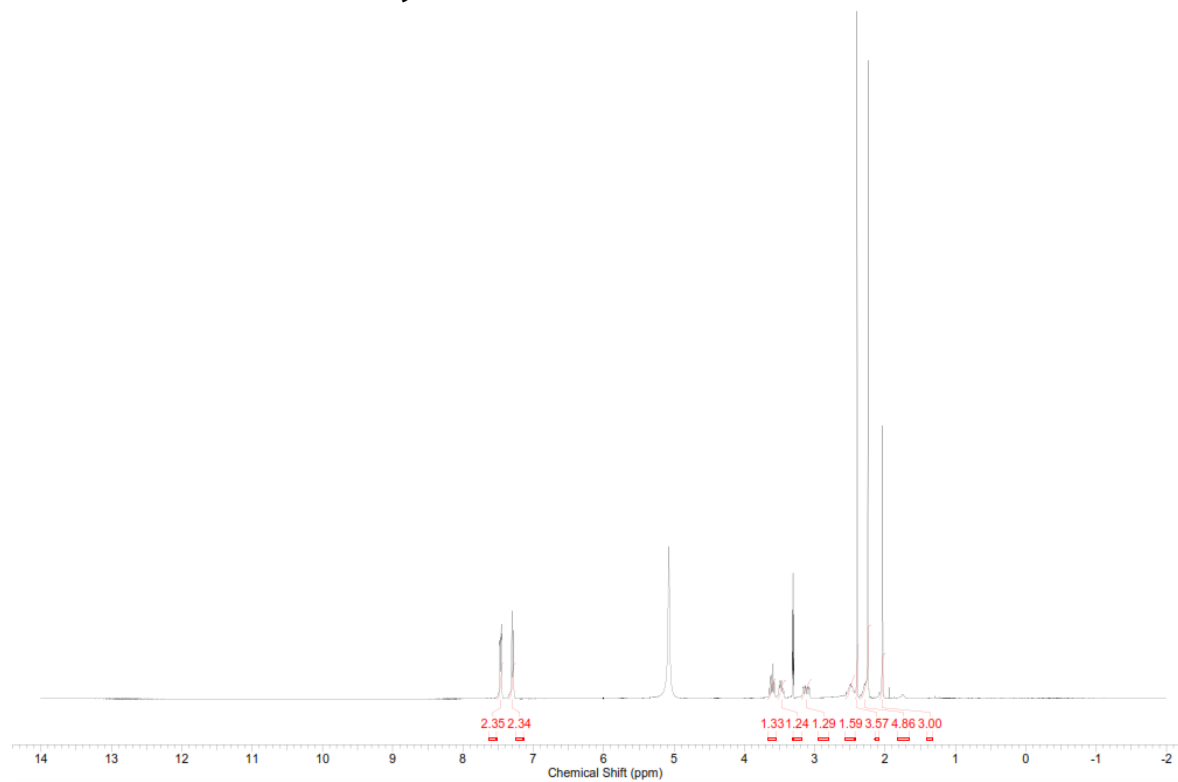
Injection Number	Peak Name	Retention Time (min)	Area (uVmin x100)	Area %	Sample Name	Sample Location	Fraction Site(s)
12	1	6.566	499062.5003	98.984	AN181-B	Fraction Zone->33	
12	2	11.241	5122.9163	1.016	AN181-B	Fraction Zone->33	
12	3	12.395	0	0	AN181-B	Fraction Zone->33	



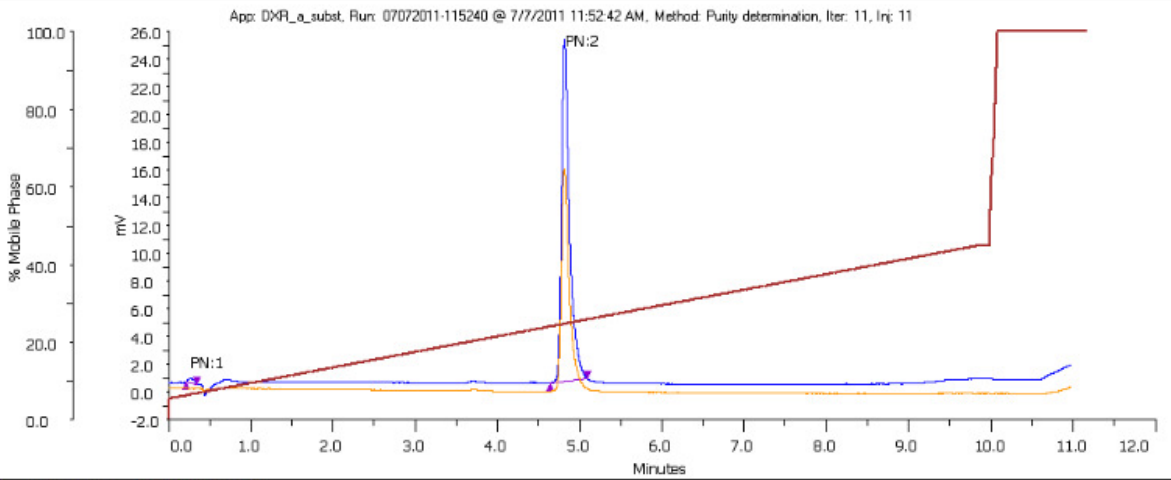
Sample Table

Injection Number	Peak Name	Retention Time (min)	Area (uVmin x100)	Area %	Sample Name	Sample Location	Fraction Site(s)
8	1	6.117	540270.0003	99.938	AN181-C18	Fraction Zone->33	
8	2	12.364	335.8331	0.062	AN181-C18	Fraction Zone->33	

# $^1\text{H}$ NMR and $^{13}\text{C}$ NMR 13j

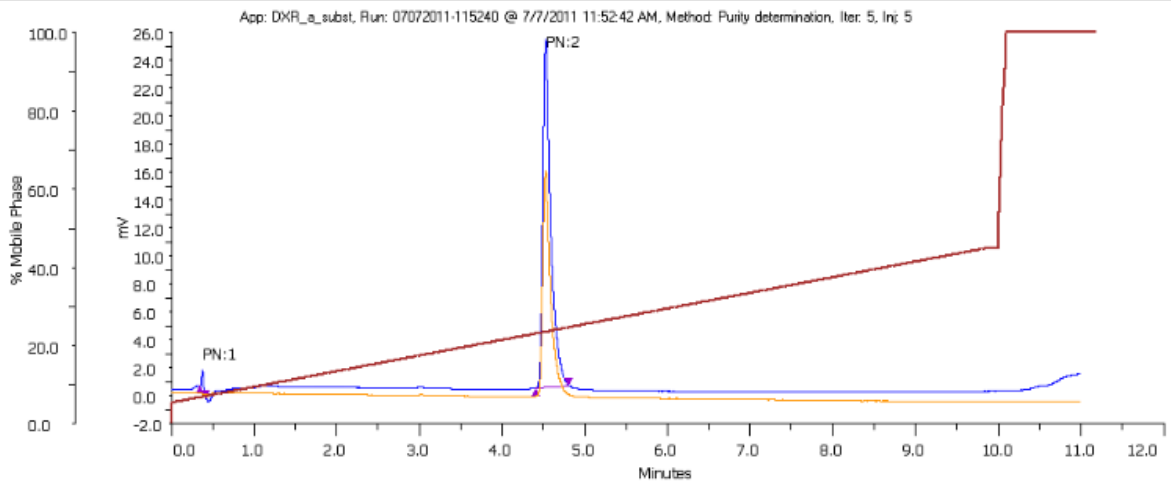


# HPLC-MS 13j



Sample Table

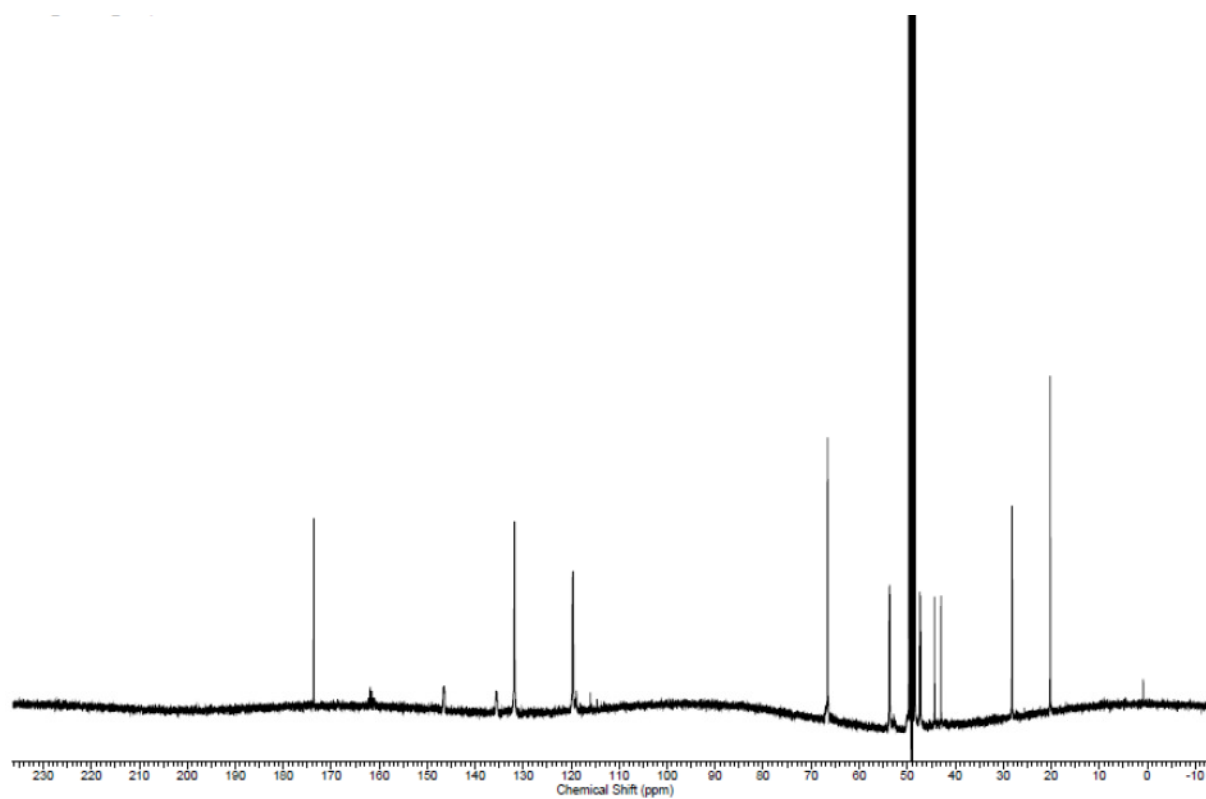
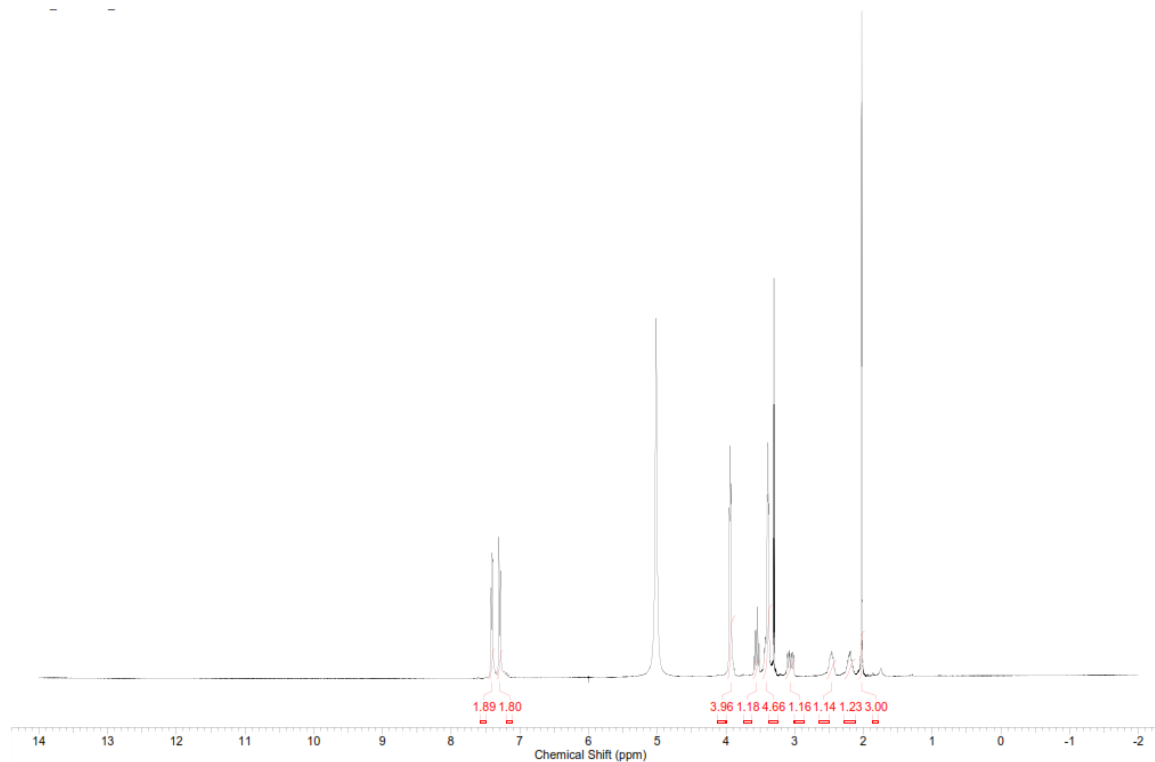
Injection Number	Peak Name	Retention Time (min)	Area (uVmin x100)	Area %	Sample Name	Sample Location	Fraction Site(s)
11	1	0.278	2915.8333	1.043	AN178_b	Sample Zone->6	
11	2	4.822	276706.6667	98.957	AN178_b	Sample Zone->6	



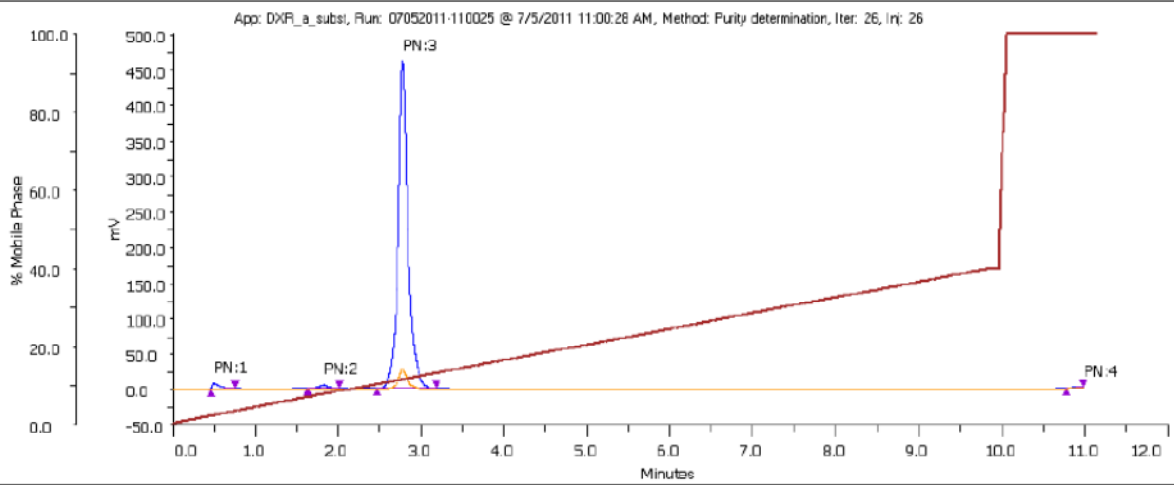
Sample Table

Injection Number	Peak Name	Retention Time (min)	Area (uVmin x100)	Area %	Sample Name	Sample Location	Fraction Site(s)
5	1	0.377	4395	1.562	AN178_C18	Sample Zone->6	
5	2	4.533	277000	98.438	AN178_C18	Sample Zone->6	

# $^1\text{H}$ NMR and $^{13}\text{C}$ NMR 13k

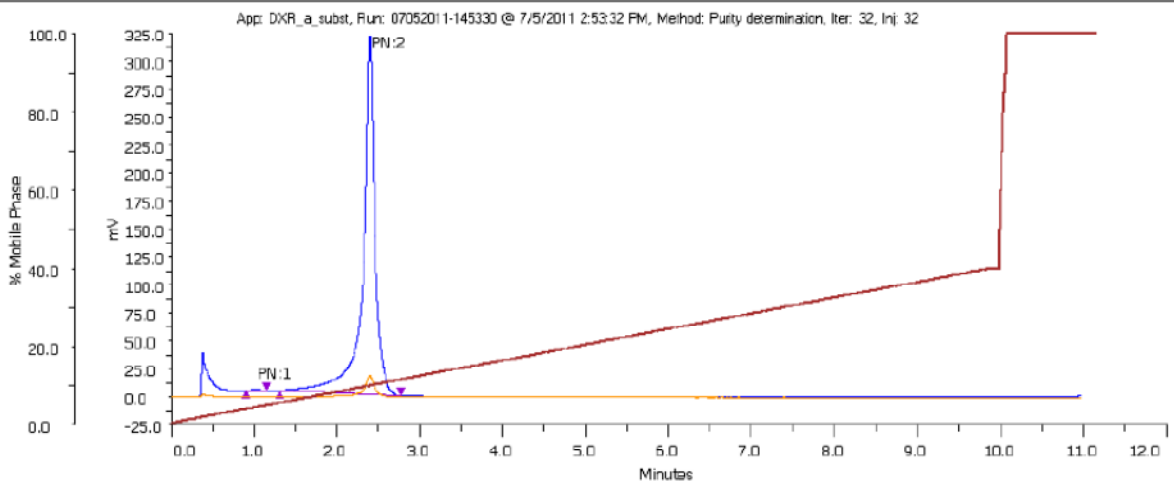


# HPLC-MS 13k



Sample Table

Injection Number	Peak Name	Retention Time (min)	Area (uVmin x100)	Area %	Sample Name	Sample Location	Fraction Site(s)
26	1	0.497	73138.3333	1.13	AN182_b	Sample Zone->42	
26	2	1.825	66913.75	1.034	AN182_b	Sample Zone->42	
26	3	2.777	6328840.8333	97.782	AN182_b	Sample Zone->42	
26	4	10.857	3514.1667	0.054	AN182_b	Sample Zone->42	



Sample Table

Injection Number	Peak Name	Retention Time (min)	Area (uVmin x100)	Area %	Sample Name	Sample Location	Fraction Site(s)
32	1	1.031	12507.4984	0.234	AN182_C18	Sample Zone->27	
32	2	2.4	5334670.8186	99.766	AN182_C18	Sample Zone->27	