Synthesis of Luffarin L and 16-*epi*-Luffarin L using a Temporary Silicon-Tethered Ring-Closing Metathesis Reaction

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SUPPORTING INFORMATION

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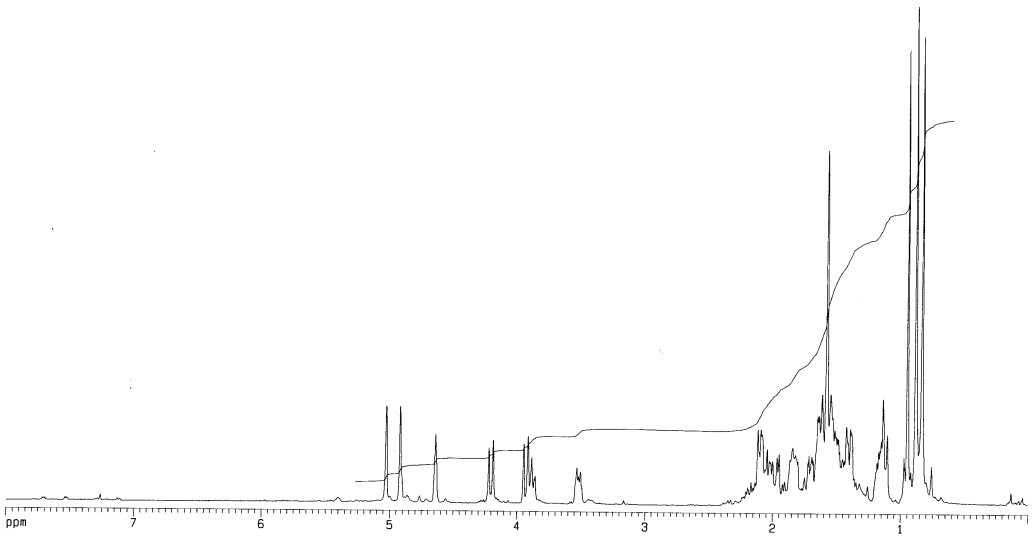
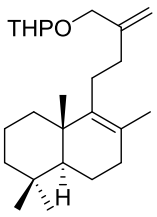
^{1}H and ^{13}C $CDCl_3$





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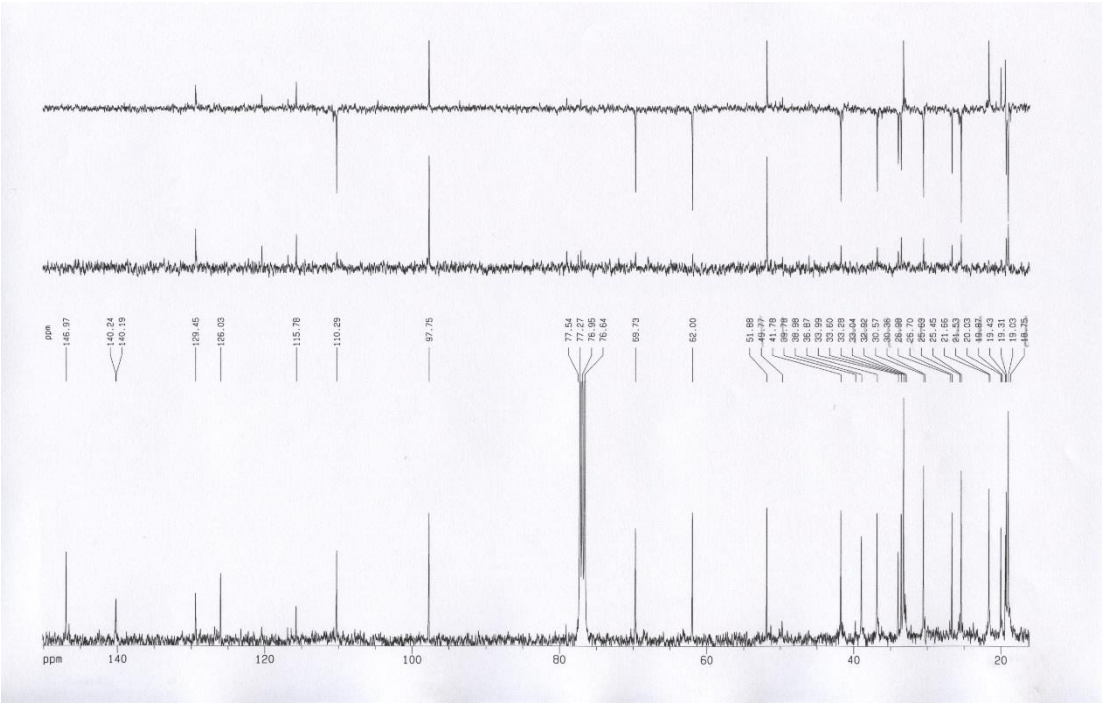

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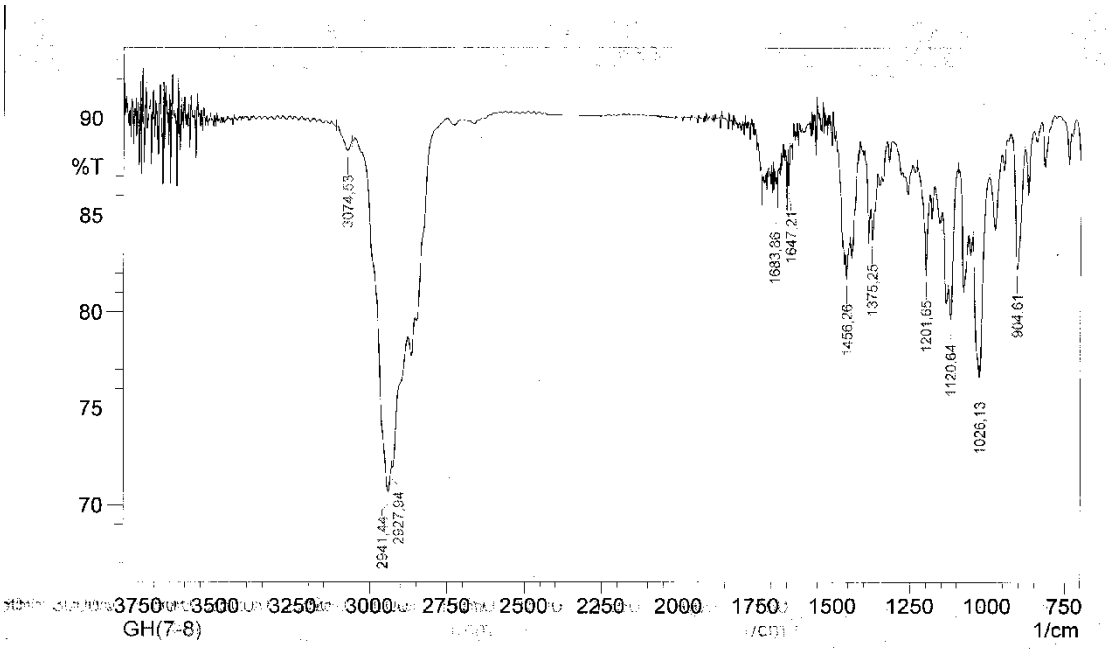
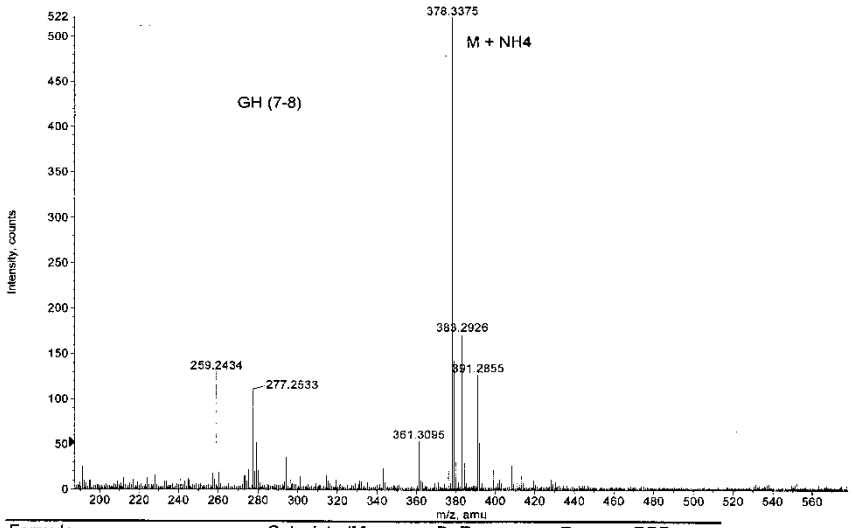
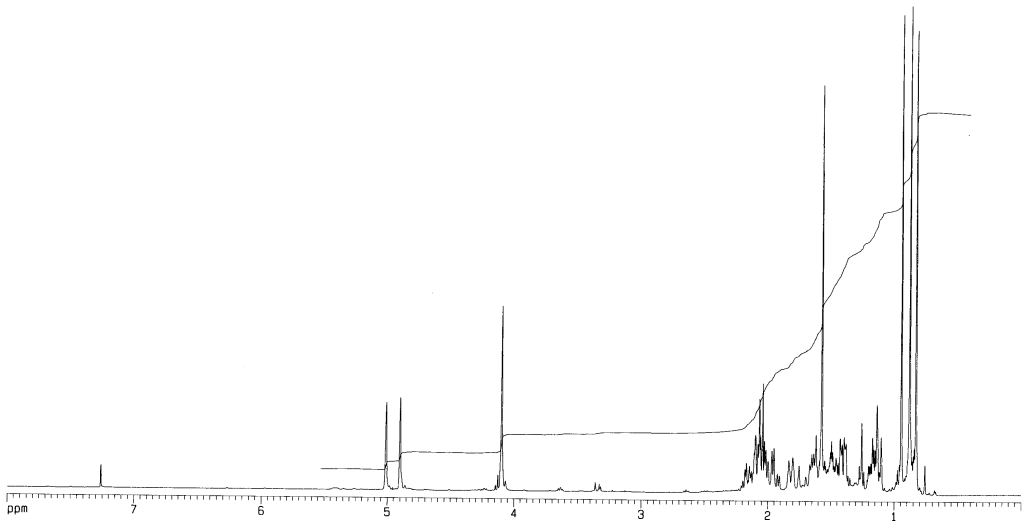
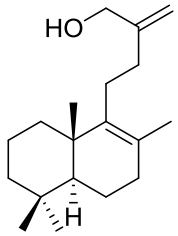




Formula	CalculatedMass	mDaError	ppmError	RDB
C24 H44 N O2	378.336656	0.843532	2.229572	3.5
C22 H45 N O2 Na	378.334251	3.248792	8.587008	0.5

^{1}H and ^{13}C $CDCl_3$





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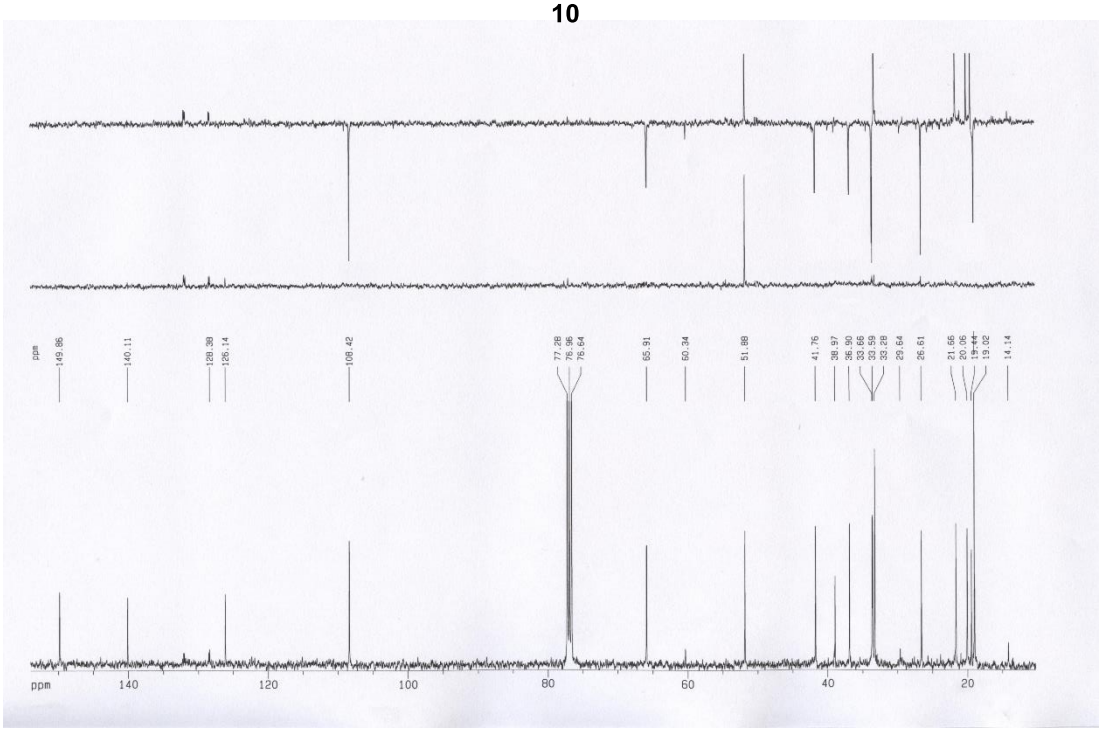

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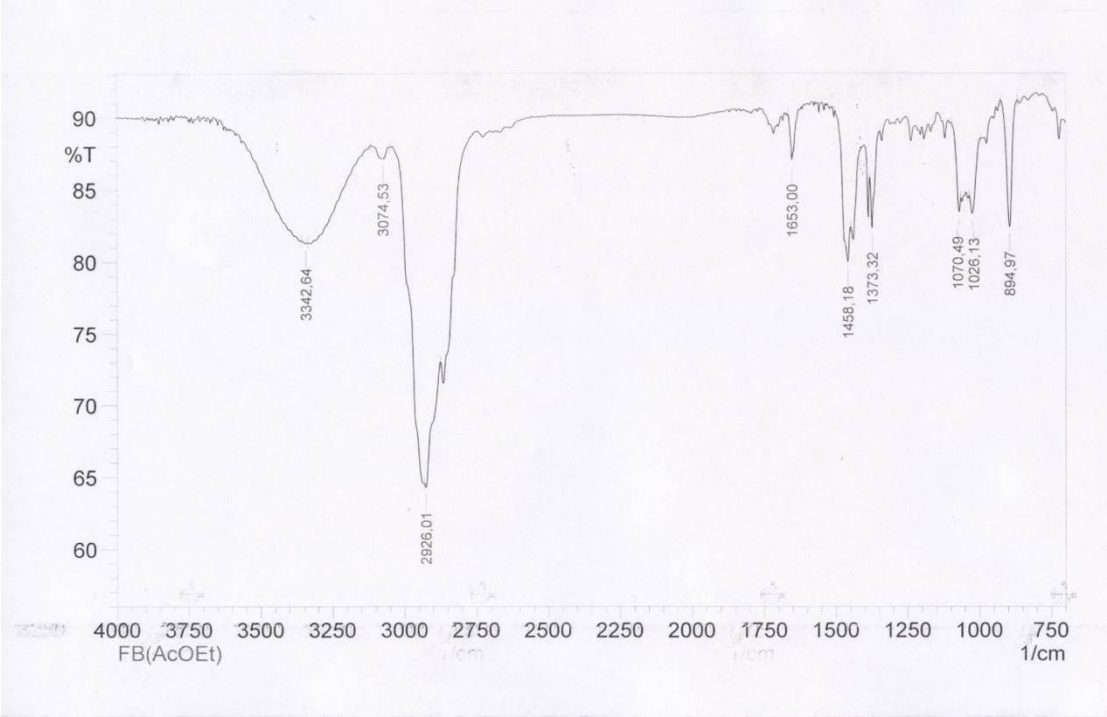
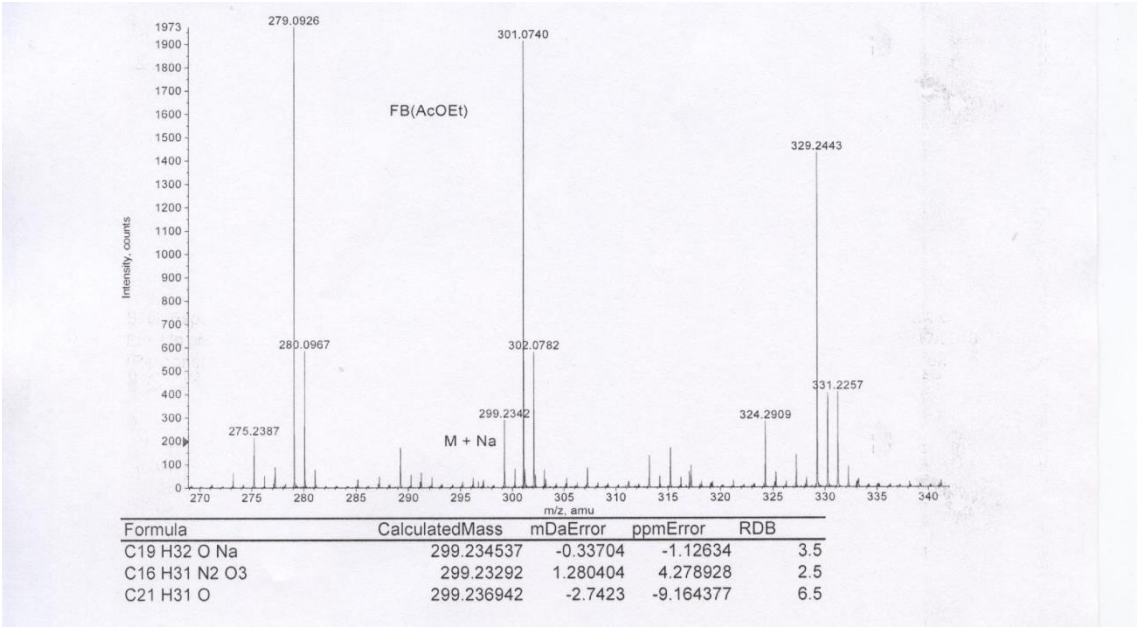
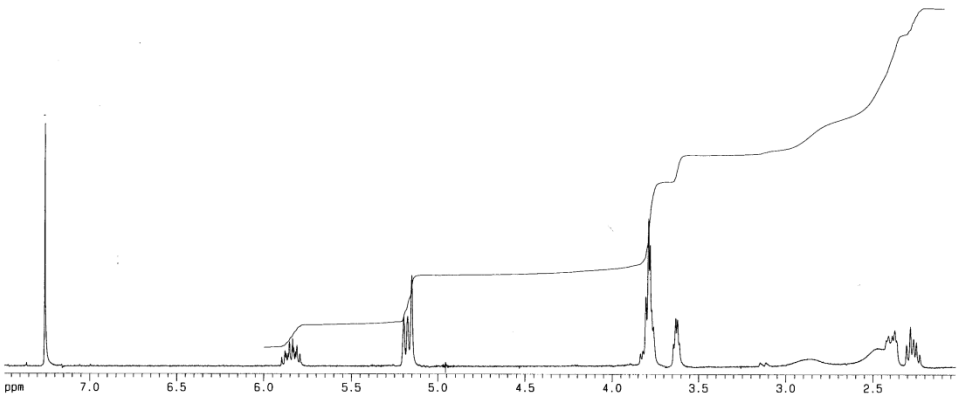
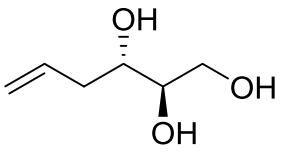




Formula	CalculatedMass	mDaError	ppmError	RDB
C19 H32 O Na	299.234537	-0.33704	-1.12634	3.5
C16 H31 N2 O3	299.23292	1.280404	4.278928	2.5
C21 H31 O	299.236942	-2.7423	-9.164377	6.5

^{1}H and ^{13}C ($CDCl_3$)





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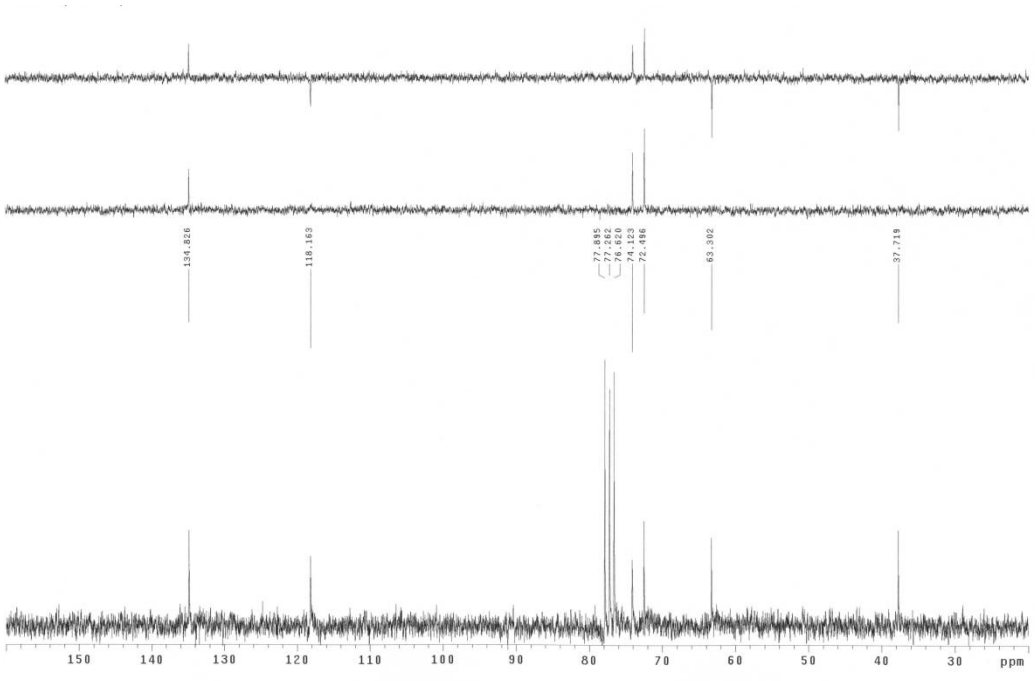

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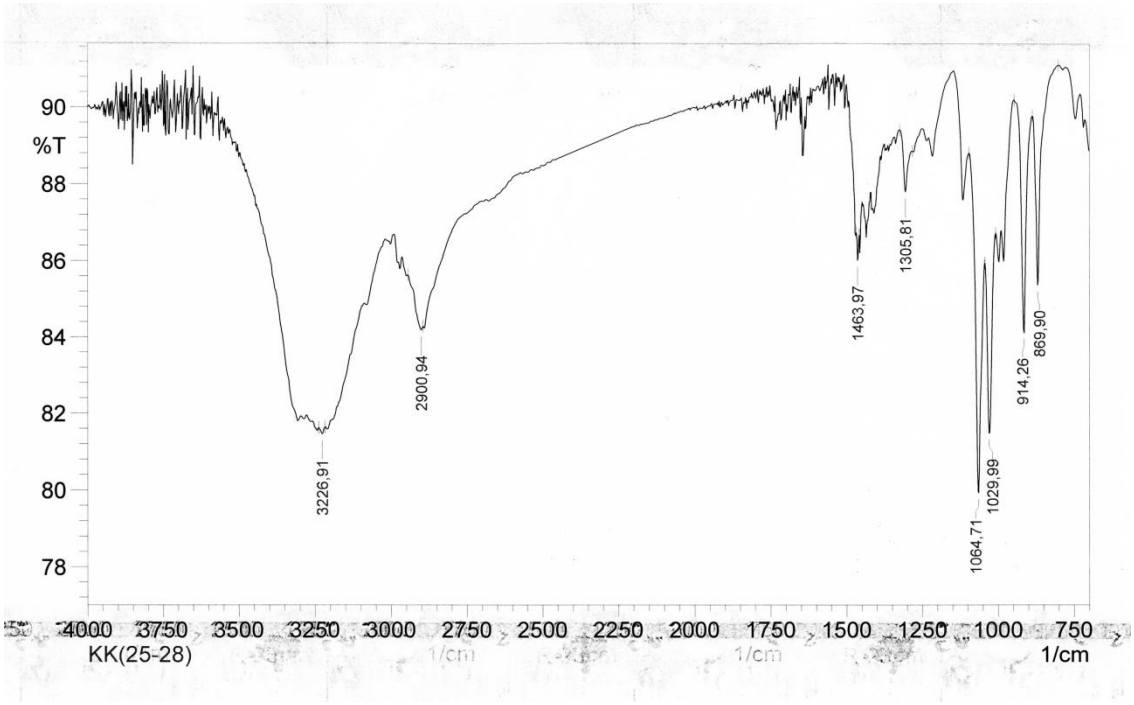
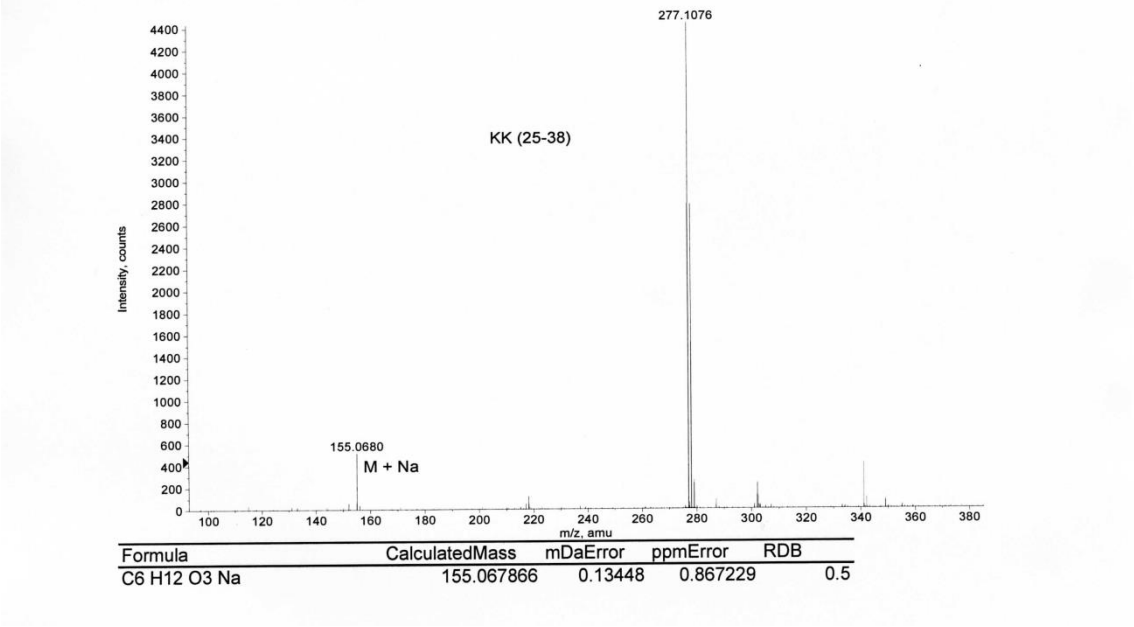
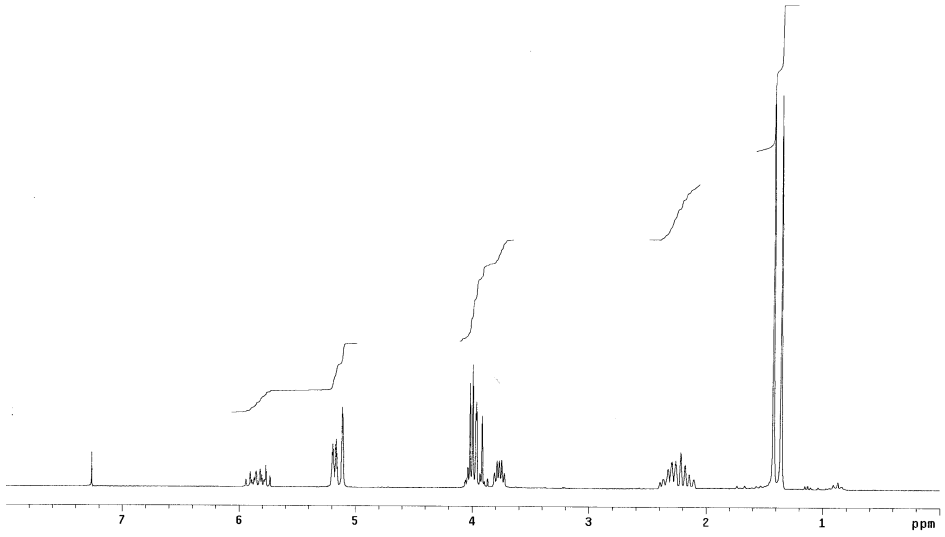
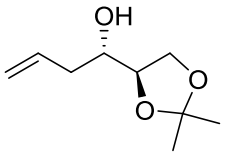




Formula	CalculatedMass	mDaError	ppmError	RDB
C6 H12 O3 Na	155.067866	0.13448	0.867229	0.5

^{1}H and ^{13}C ($CDCl_3$)





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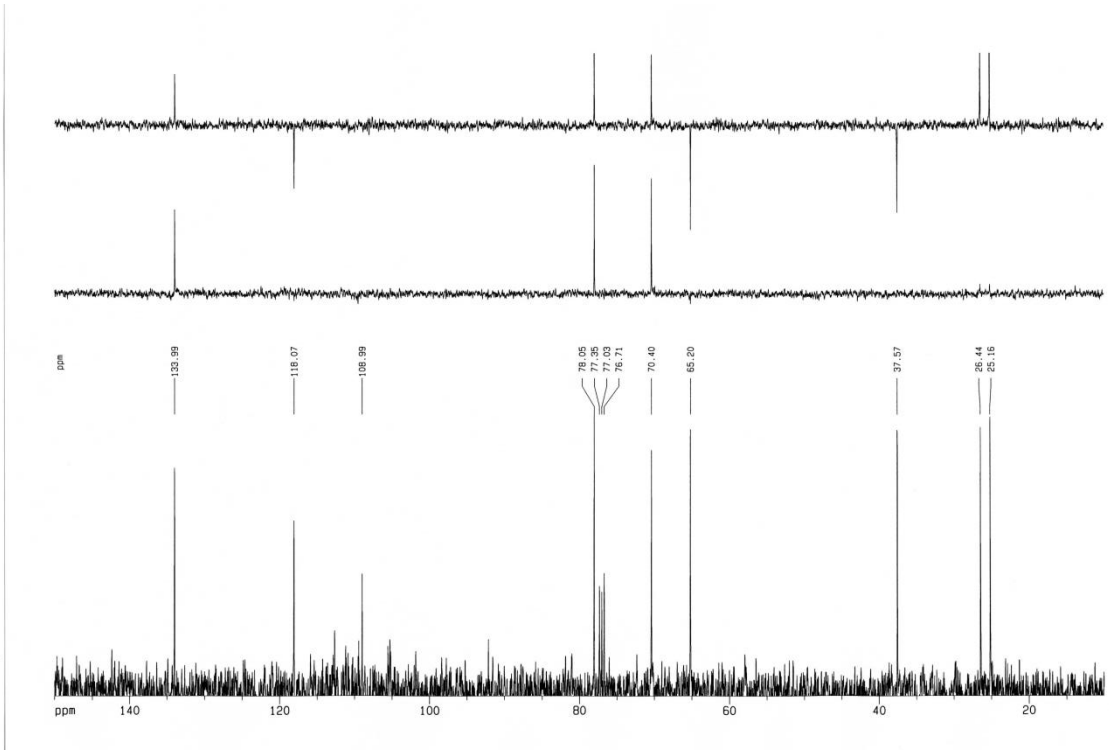

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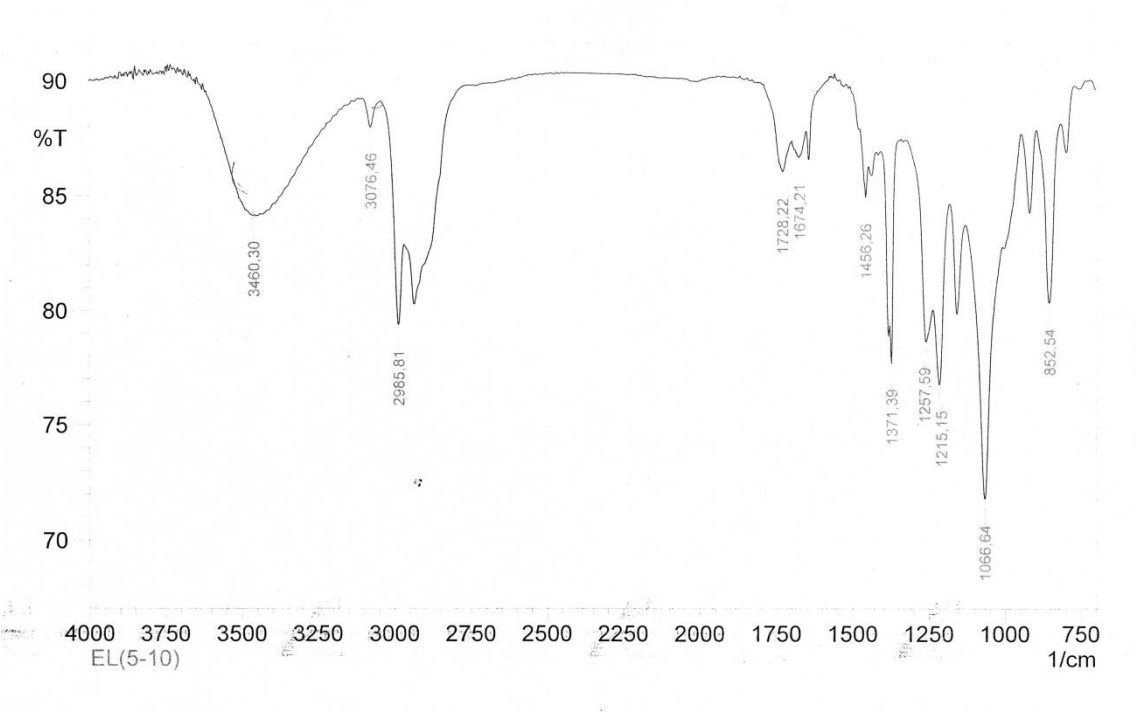
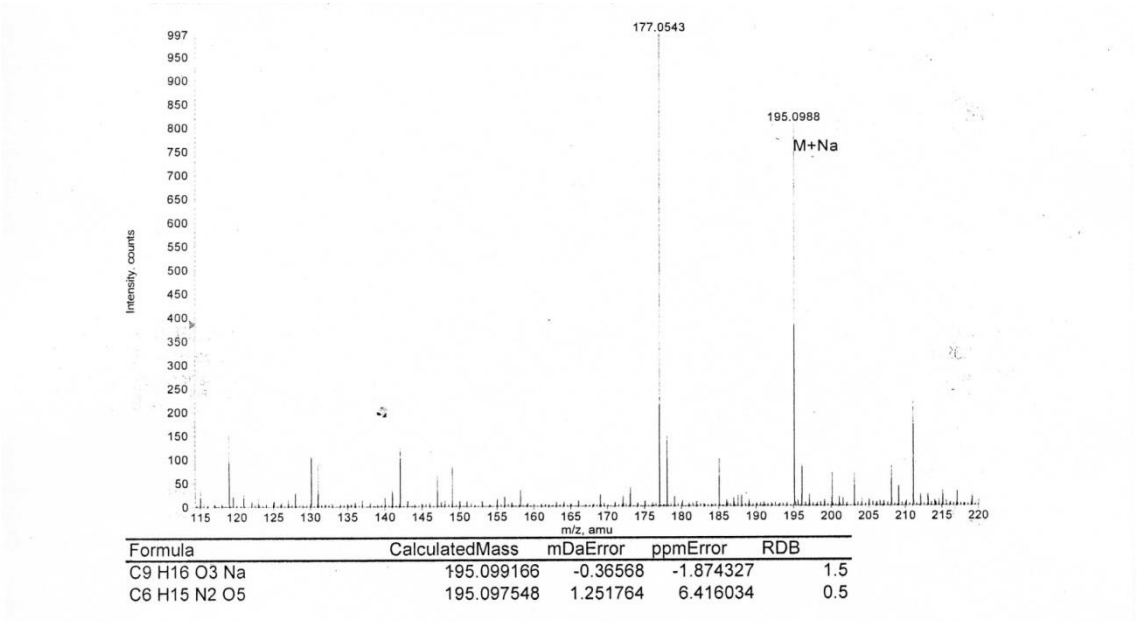
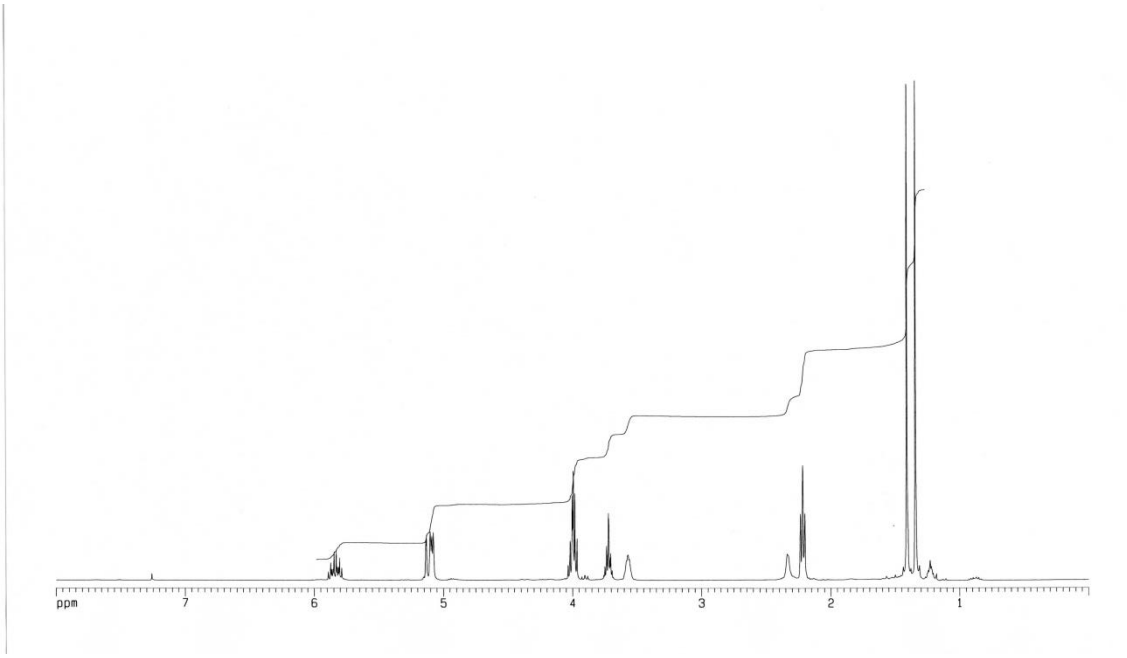
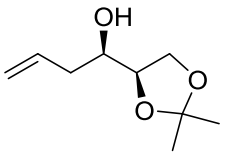




Formula	CalculatedMass	mDaError	ppmError	RDB
C9 H16 O3 Na	195.099166	-0.36568	-1.874327	1.5
C6 H15 N2 O5	195.097548	1.251764	6.416034	0.5

^{1}H and ^{13}C ($CDCl_3$)





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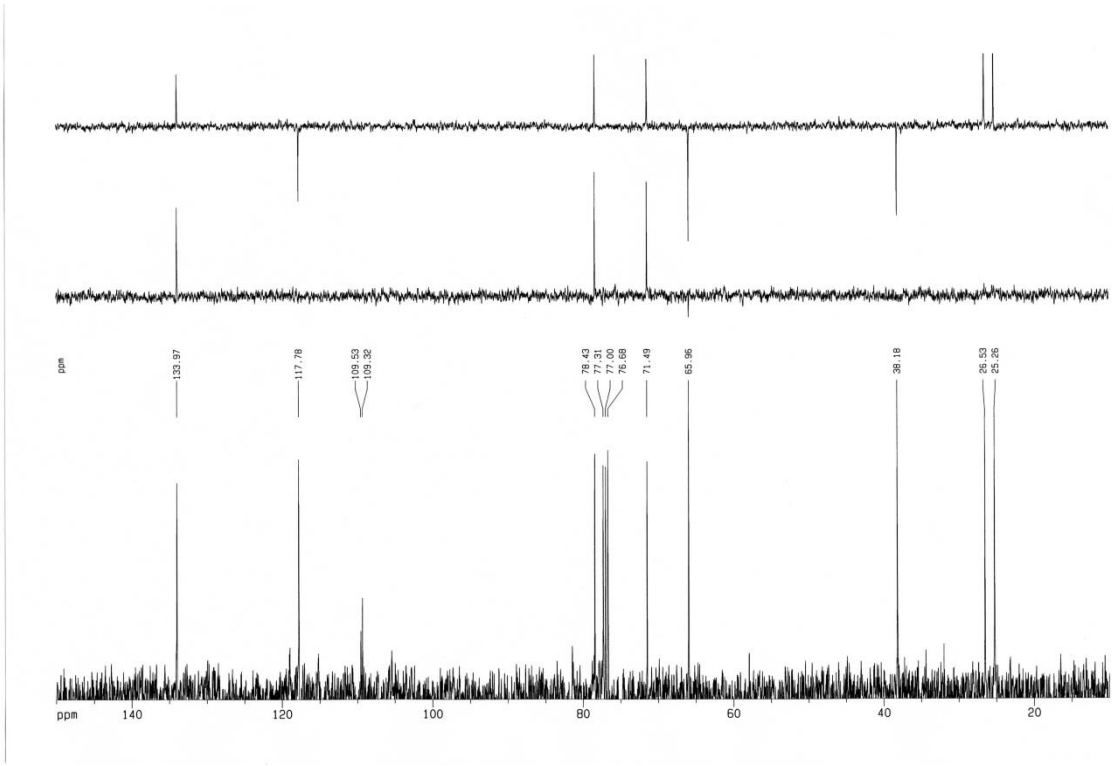

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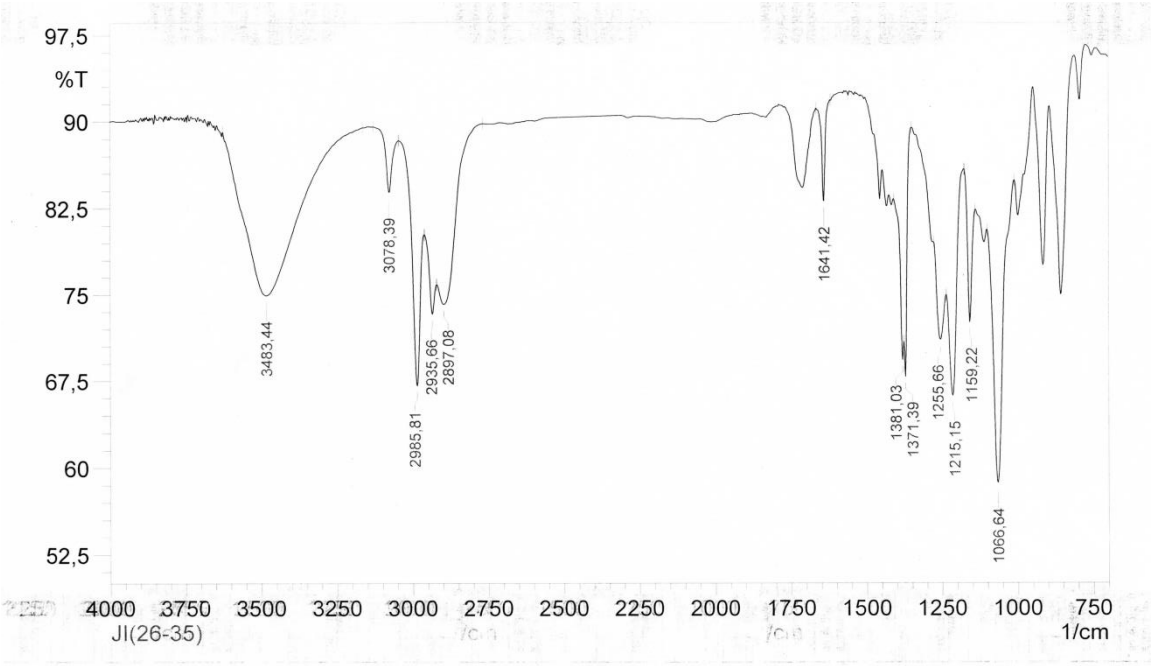
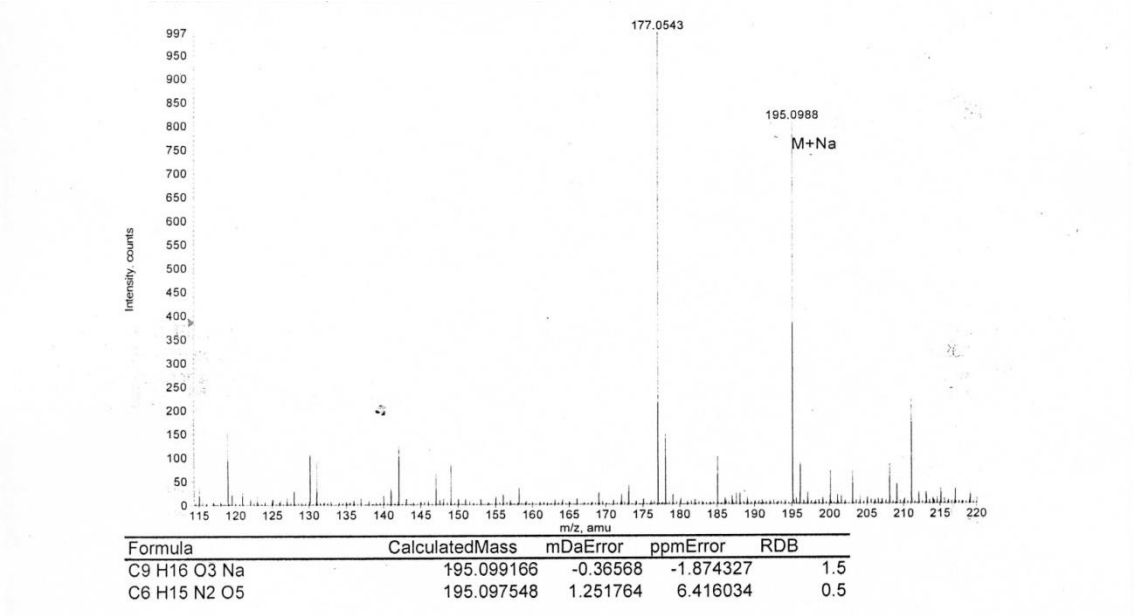
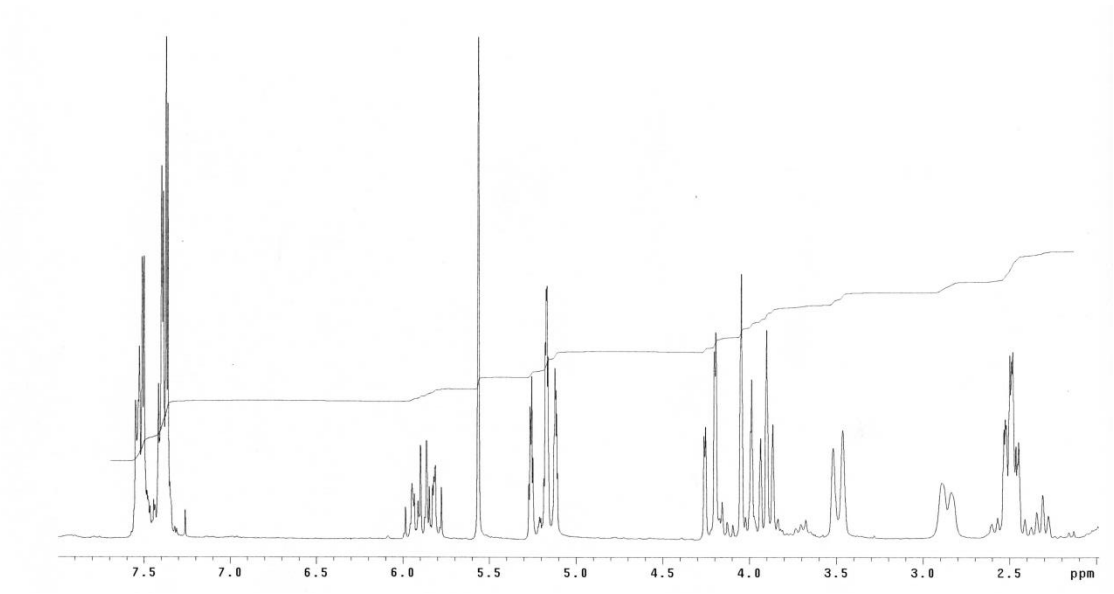
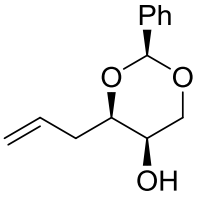




Formula	CalculatedMass	mDaError	ppmError	RDB
C9 H16 O3 Na	195.099166	-0.36568	-1.874327	1.5
C6 H15 N2 O5	195.097548	1.251764	6.416034	0.5

^{1}H and ^{13}C ($CDCl_3$)





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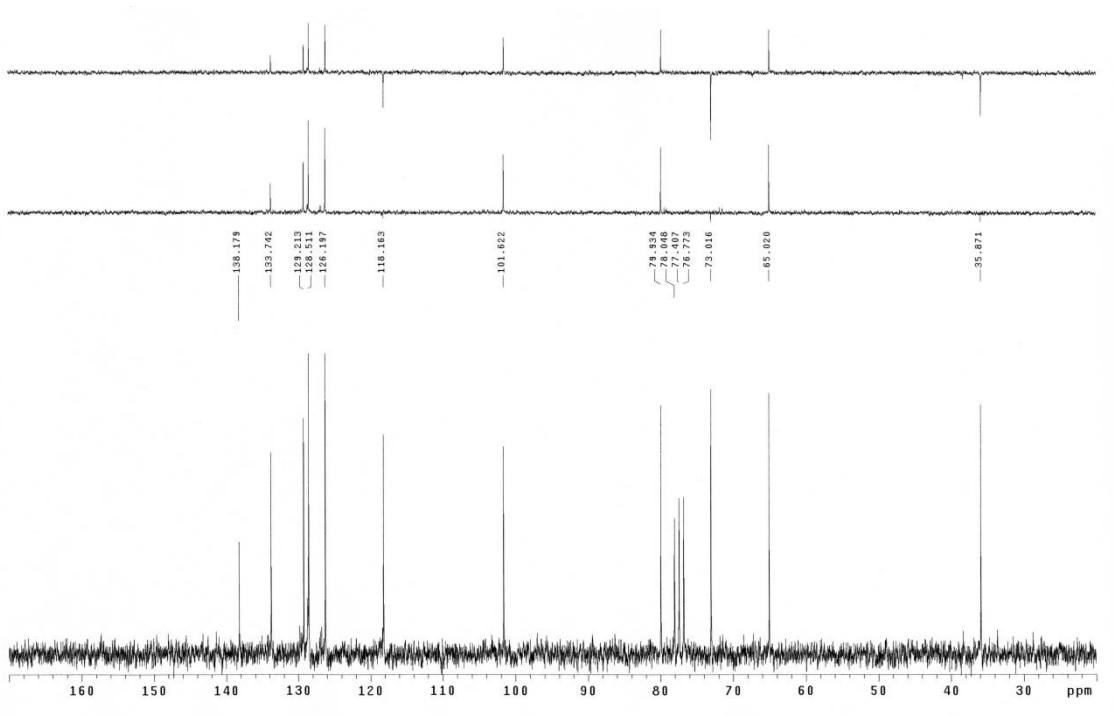

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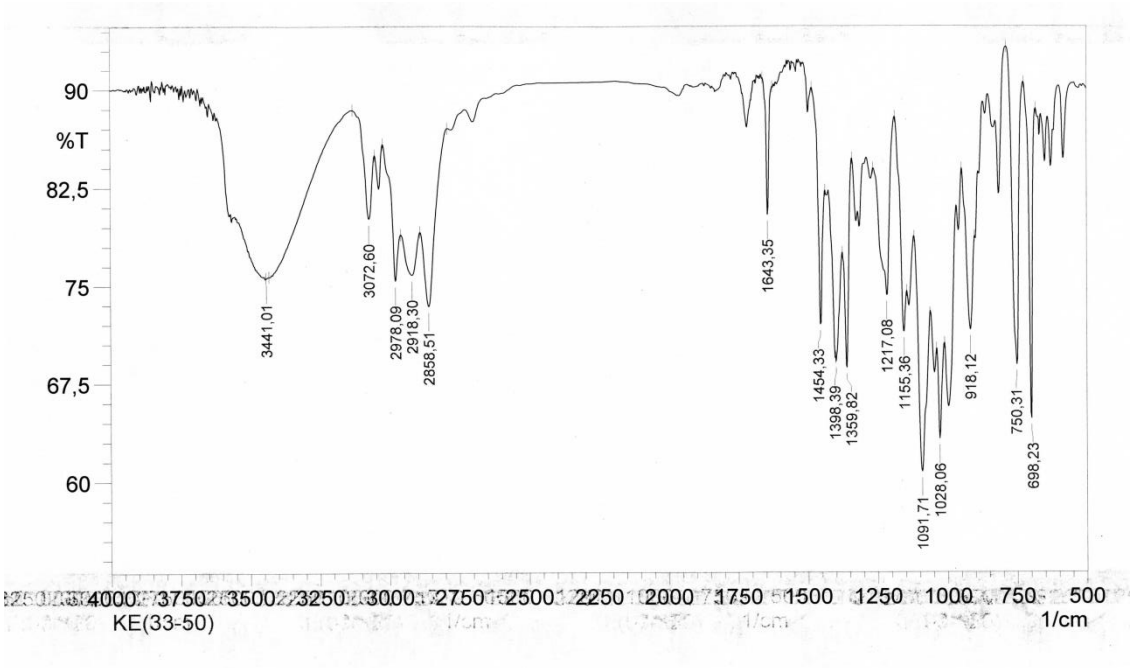
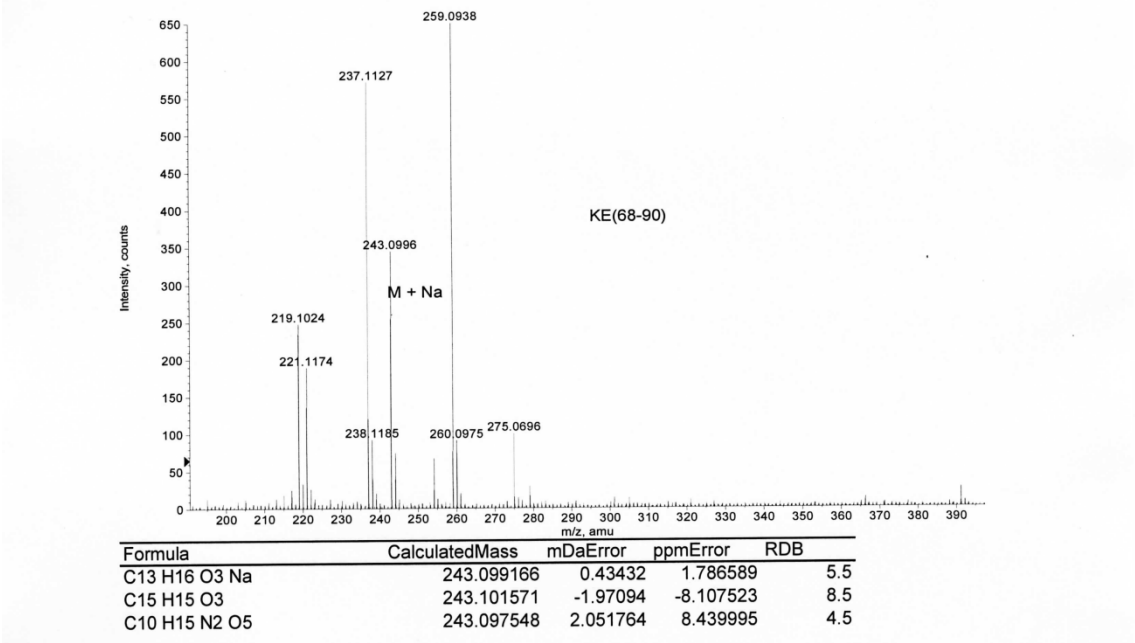
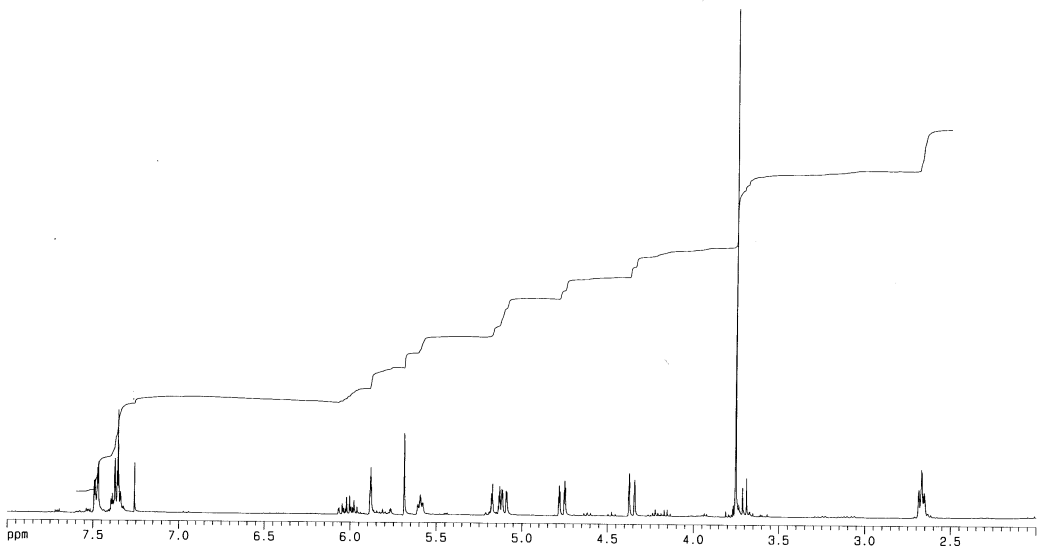
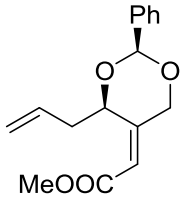




Formula	CalculatedMass	mDaError	ppmError	RDB
C13 H16 O3 Na	243.099166	0.43432	1.786589	5.5
C15 H15 O3	243.101571	-1.97094	-8.107523	8.5
C10 H15 N2 O5	243.097548	2.051764	8.439995	4.5

^{1}H and ^{13}C ($CDCl_3$)





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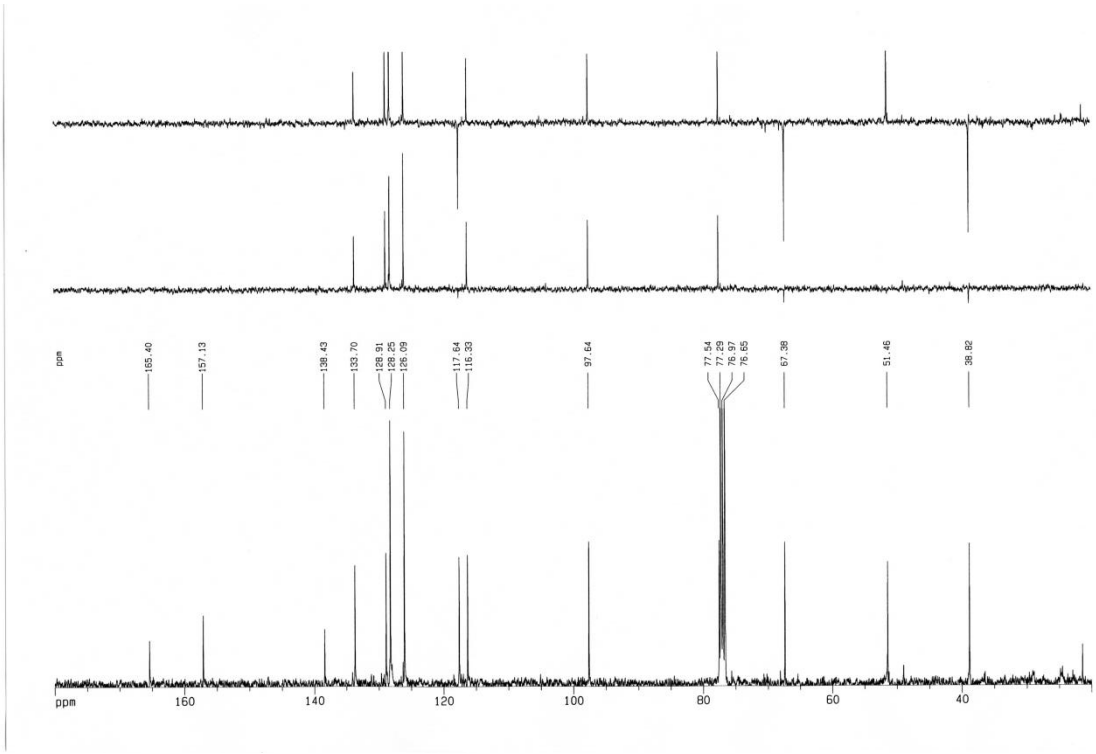

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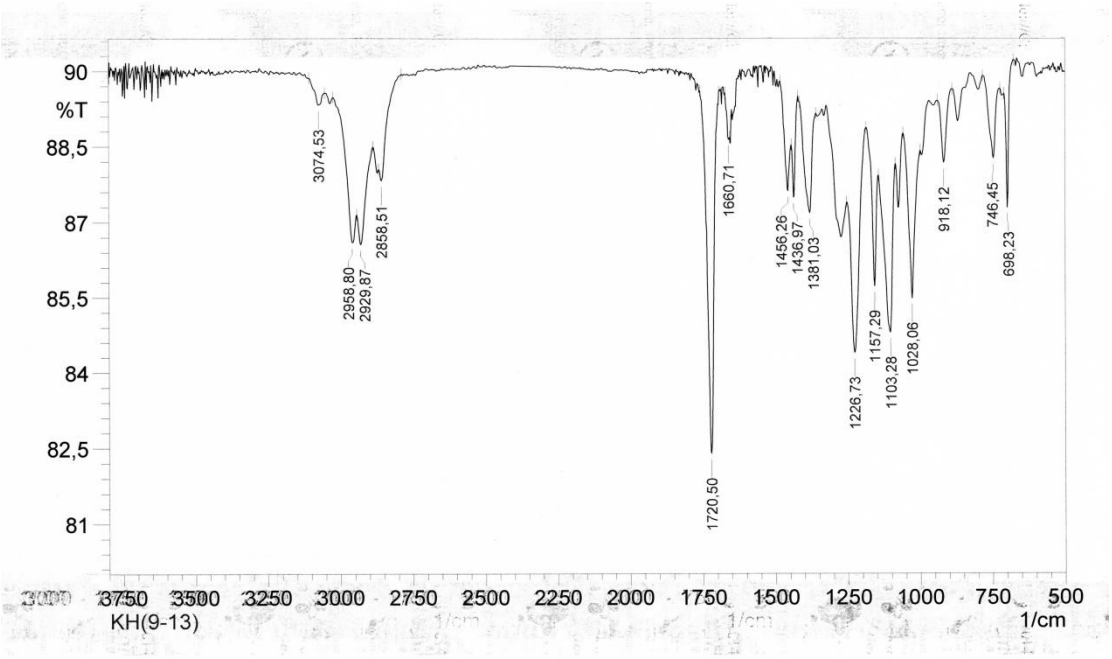
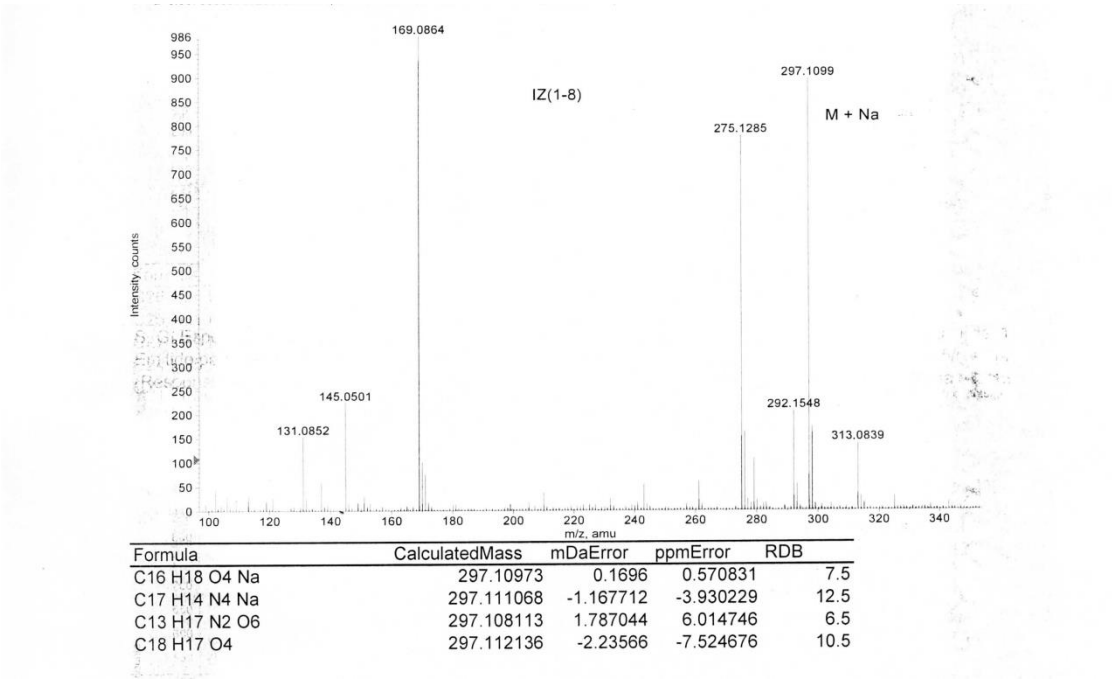
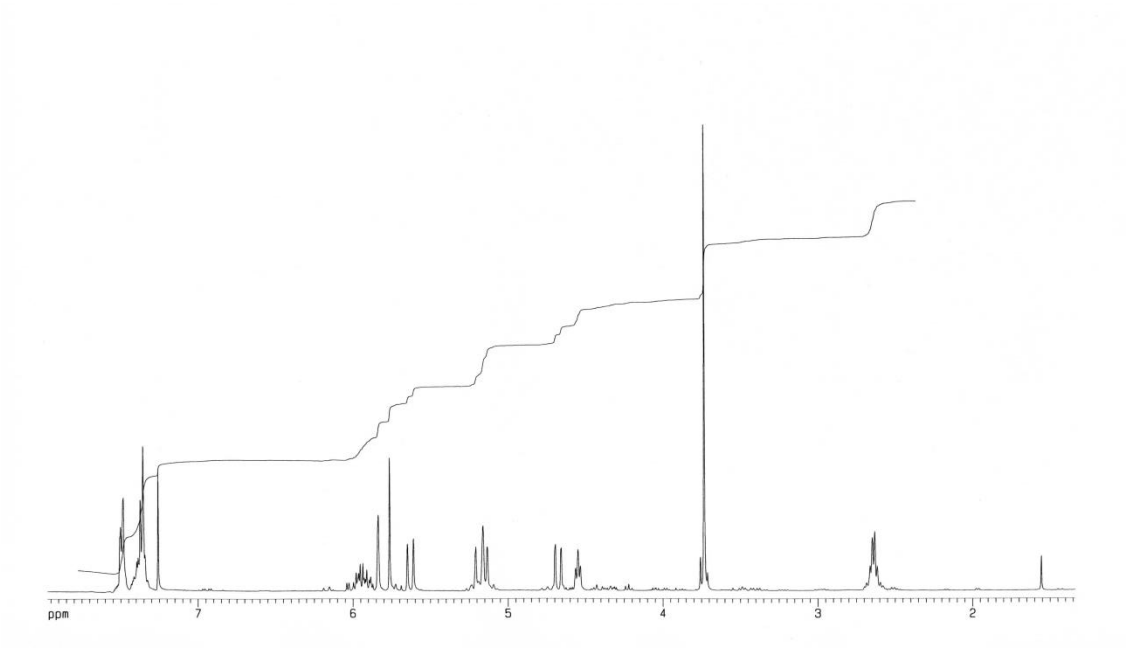
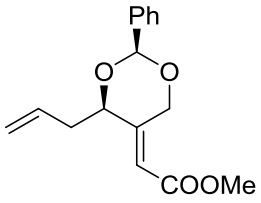




Formula	CalculatedMass	mDaError	ppmError	RDB
C16 H18 O4 Na	297.10973	0.1696	0.570831	7.5
C17 H14 N4 Na	297.111068	-1.167712	-3.930229	12.5
C13 H17 N2 O6	297.108113	1.787044	6.014746	6.5
C18 H17 O4	297.112136	-2.23566	-7.524676	10.5

^{1}H and ^{13}C ($CDCl_3$)





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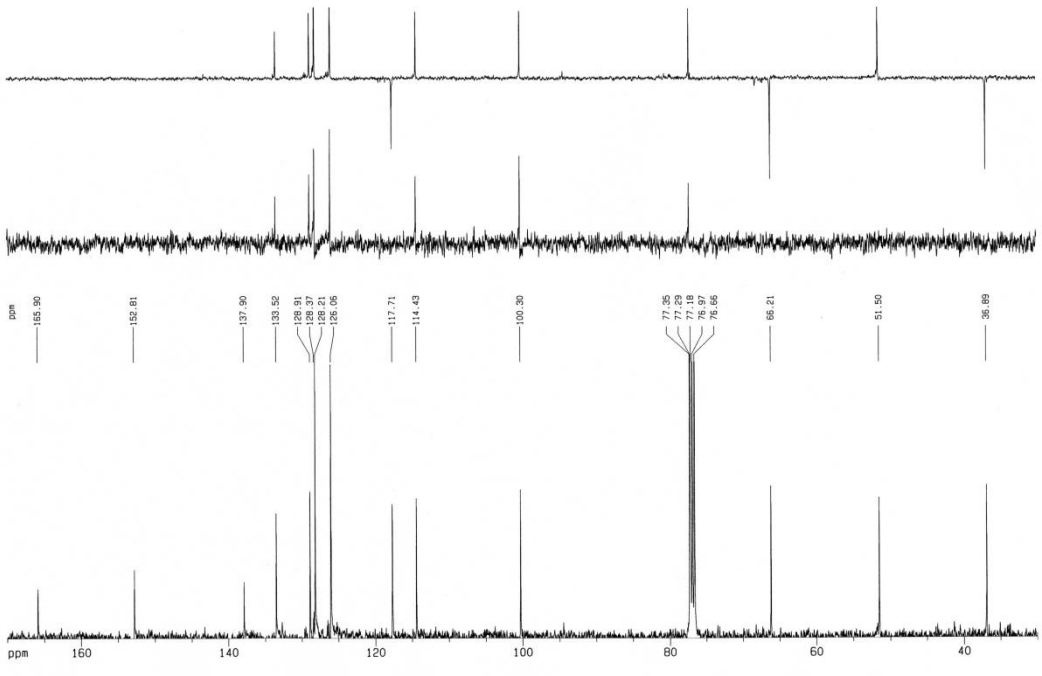

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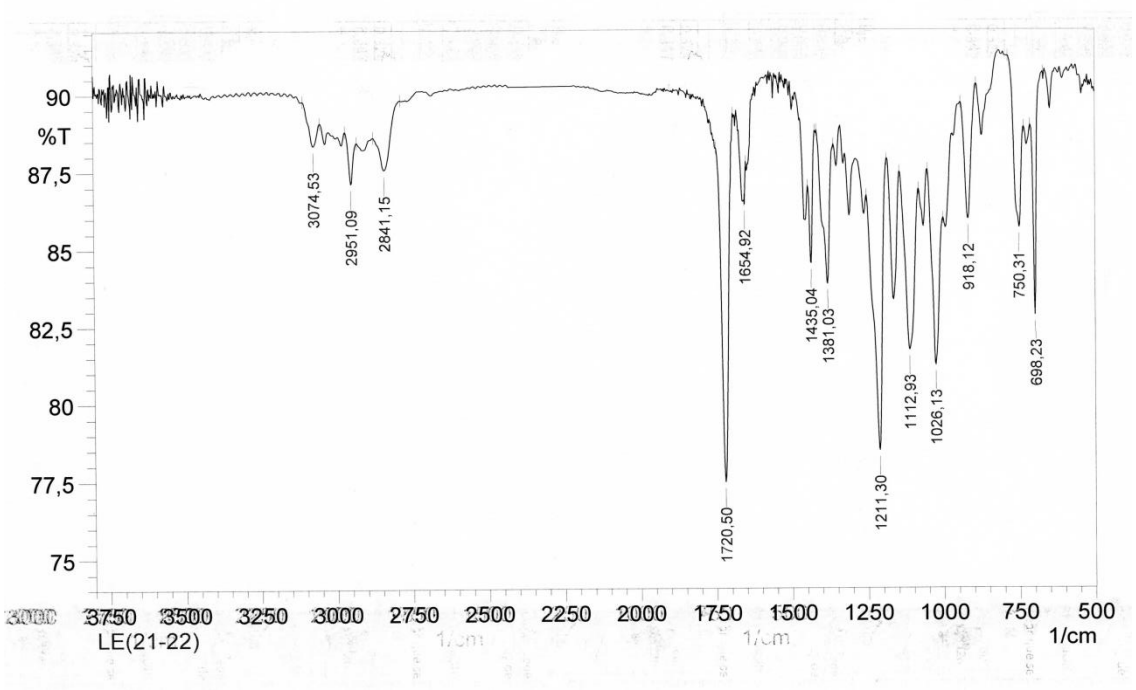
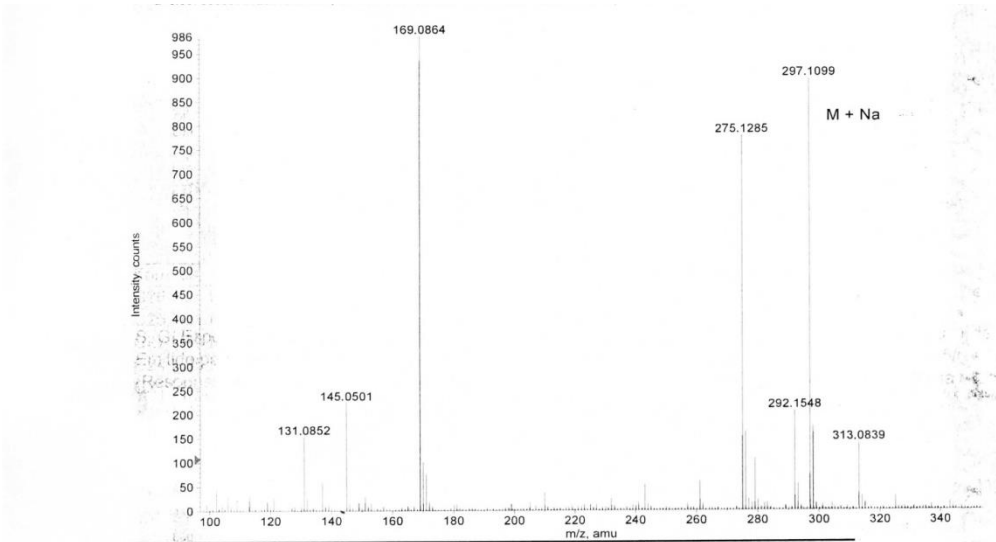
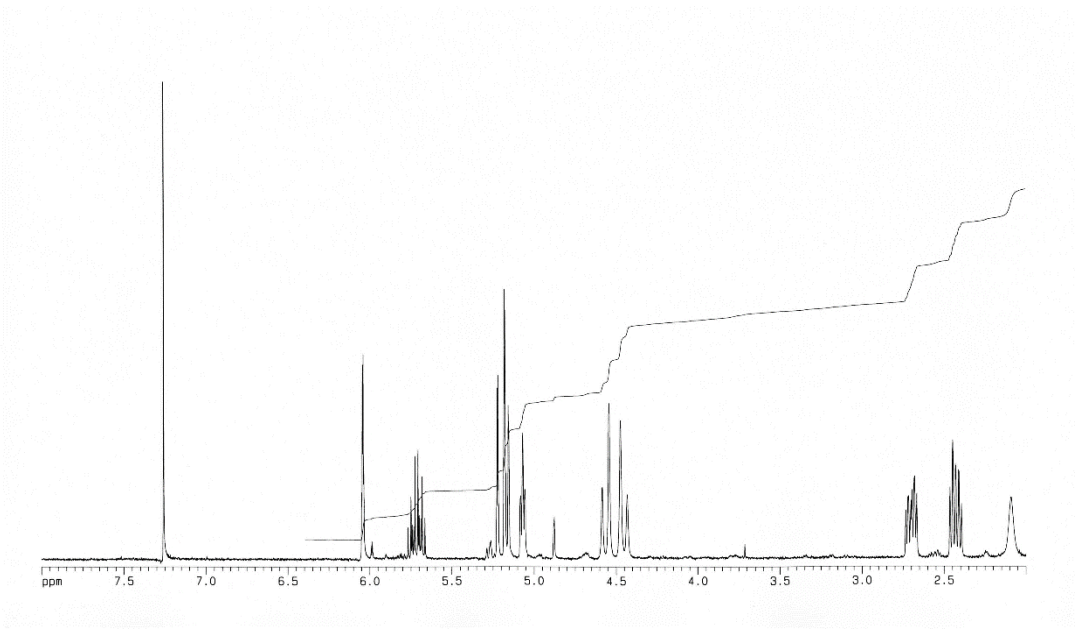
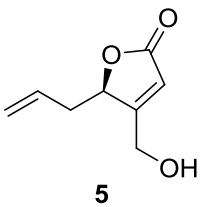




Formula	CalculatedMass	mDaError	ppmError	RDB
C16 H18 O4 Na	297.10973	0.1696	0.570831	7.5
C17 H14 N4 Na	297.111068	-1.167712	-3.930229	12.5
C13 H17 N2 O6	297.108113	1.787044	6.014746	6.5
C18 H17 O4	297.112136	-2.23566	-7.524676	10.5

^{1}H and ^{13}C ($CDCl_3$)





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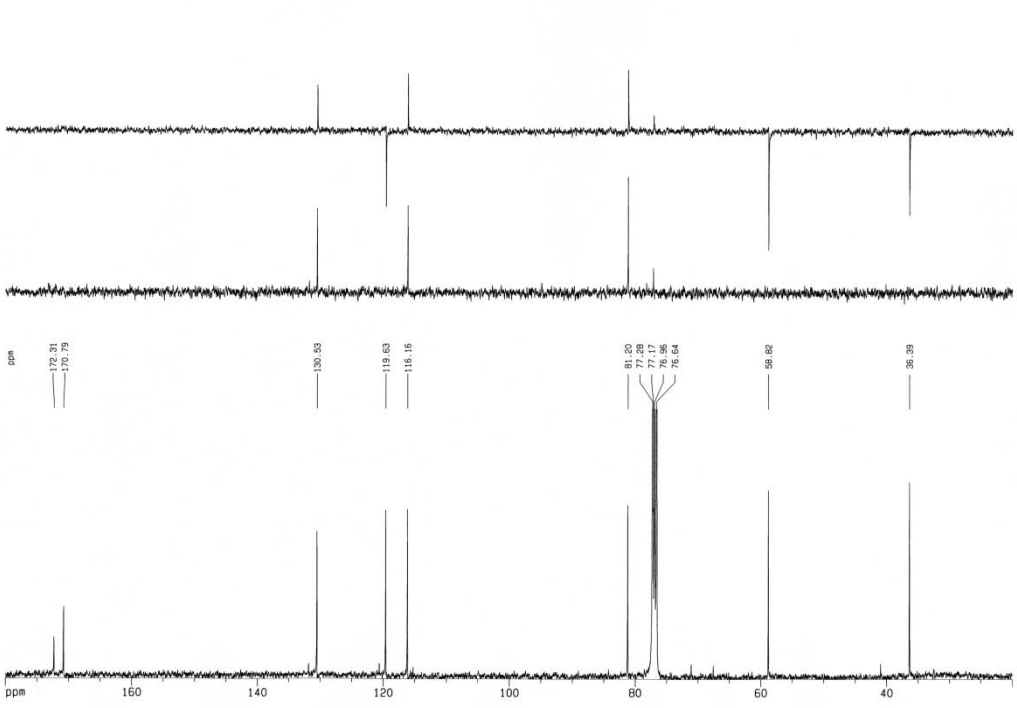

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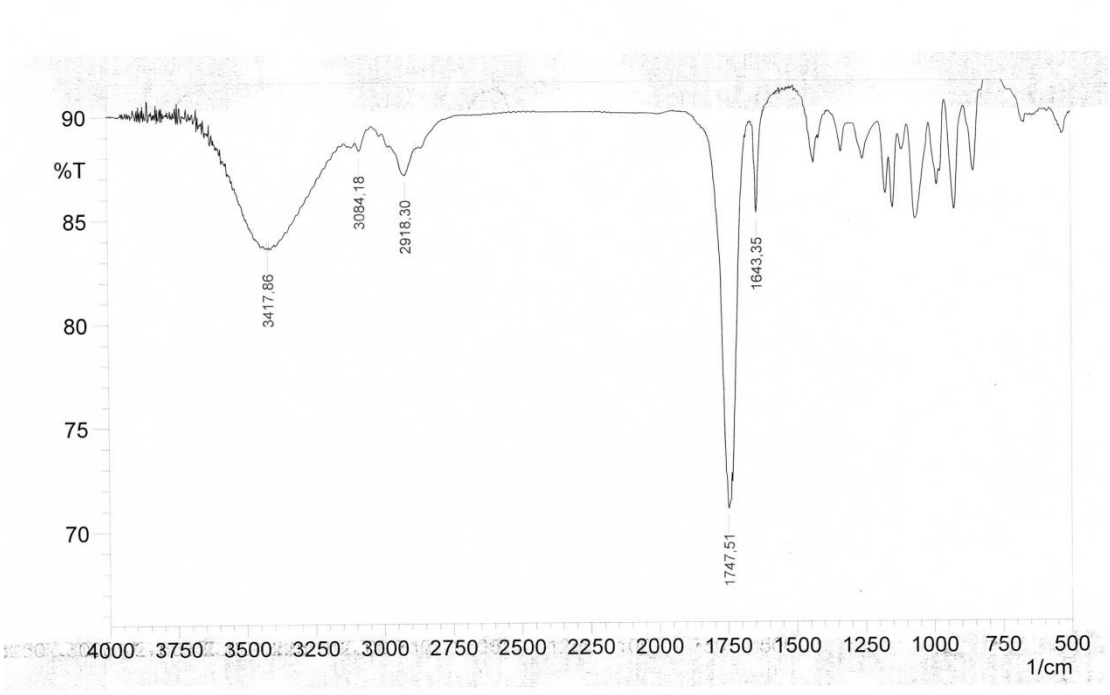
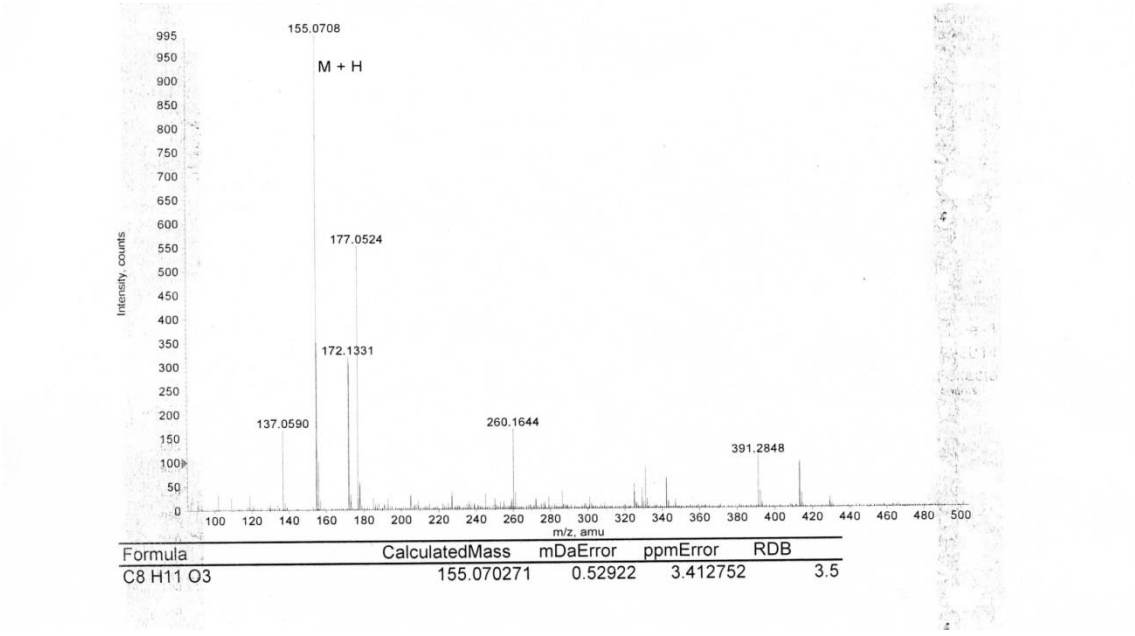
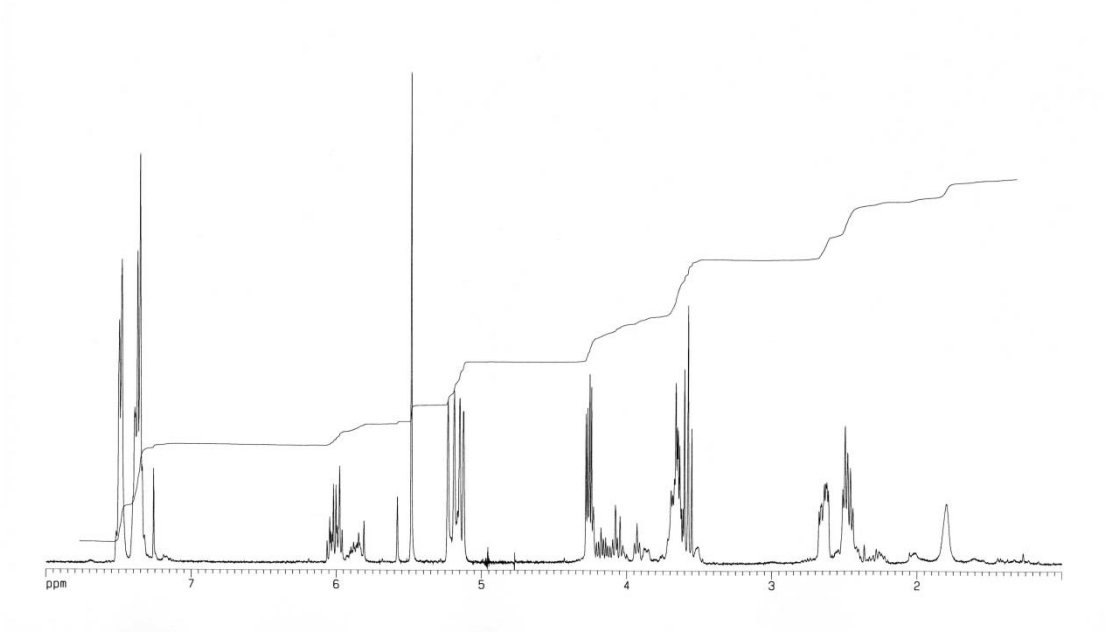
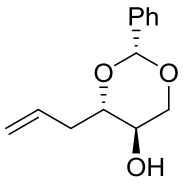




Formula	CalculatedMass	mDaError	ppmError	RDB
C8 H11 O3	155.070271	0.52922	3.412752	3.5

^{1}H and ^{13}C ($CDCl_3$)





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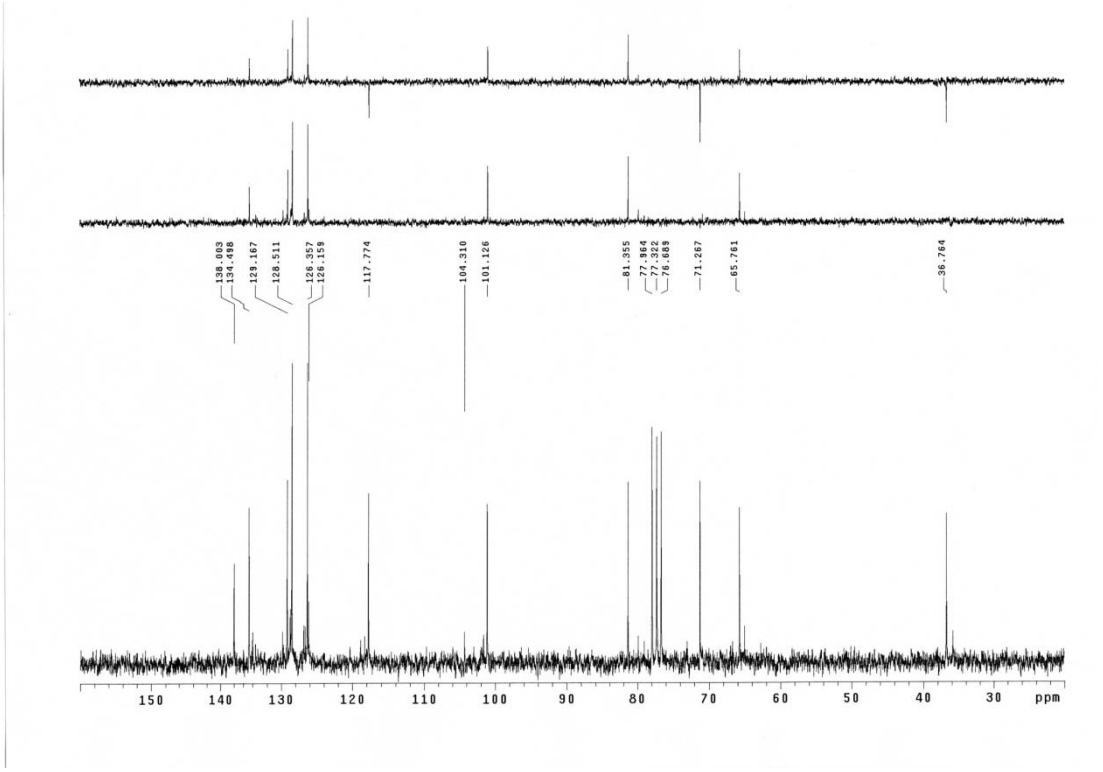
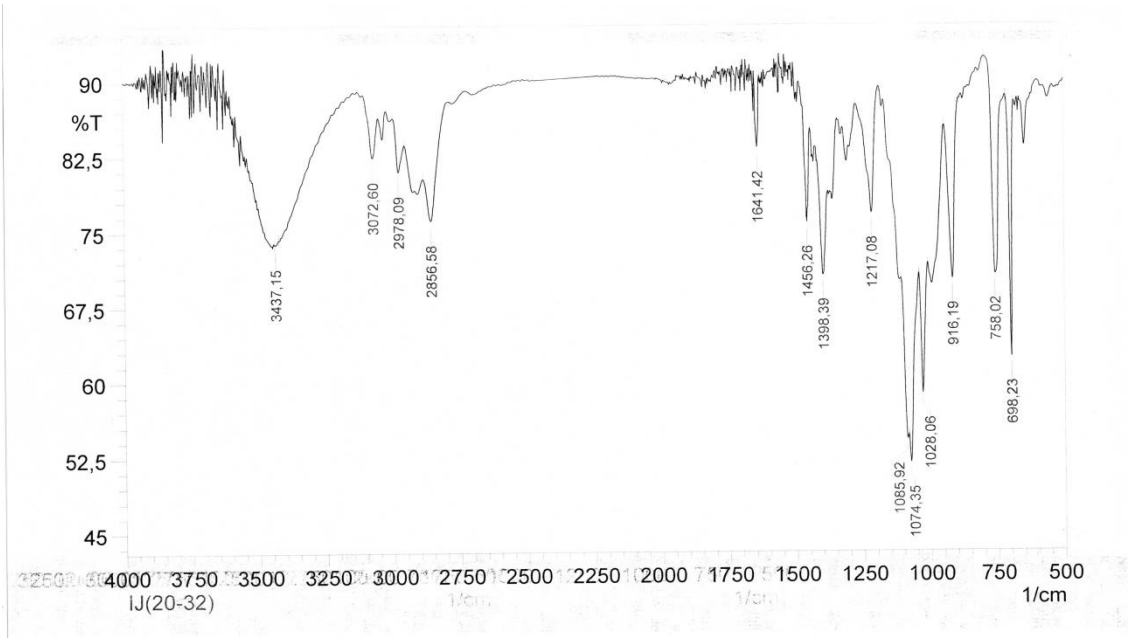


IR and HRMS


90
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82,5
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67,5
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2978,09
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1398,39
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1085,92
1074,35
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916,19
758,02
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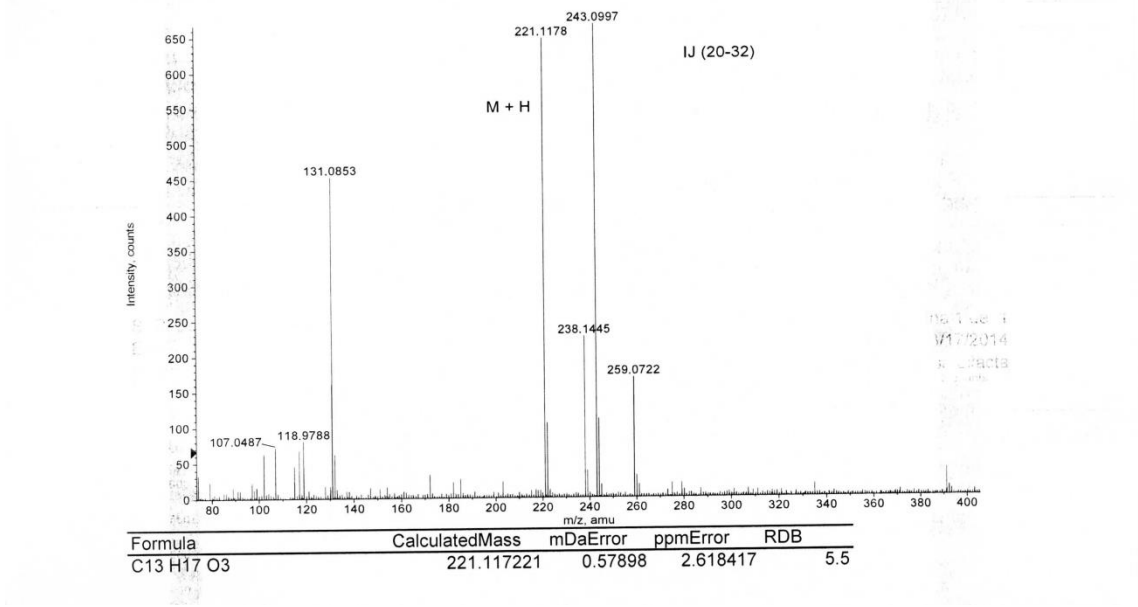
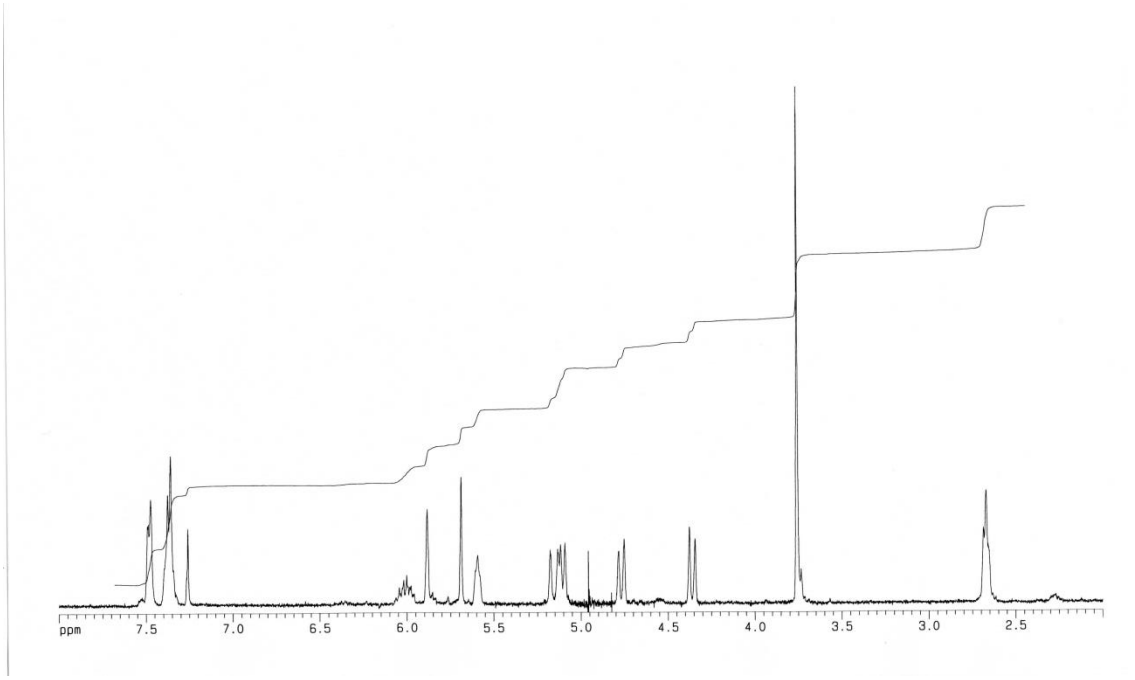
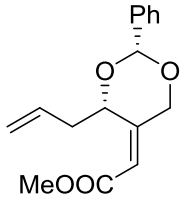
IJ(20-32)

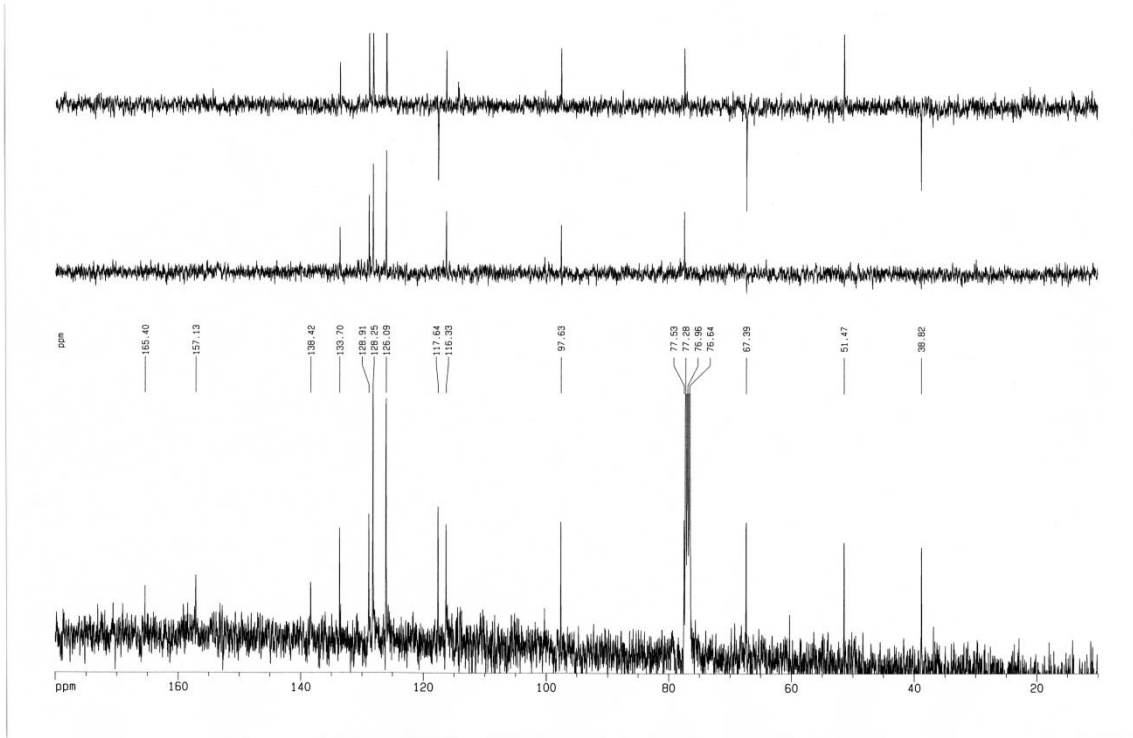

IJ (20-32)
M + H
243.0997
221.1178
131.0853
238.1445
259.0722
107.0487
118.9788
Intensity, counts
m/z, amu
Formula CalculatedMass mDaError ppmError RDB
C13 H17 O3 221.117221 0.57898 2.618417 5.5

^{1}H and ^{13}C ($CDCl_3$)

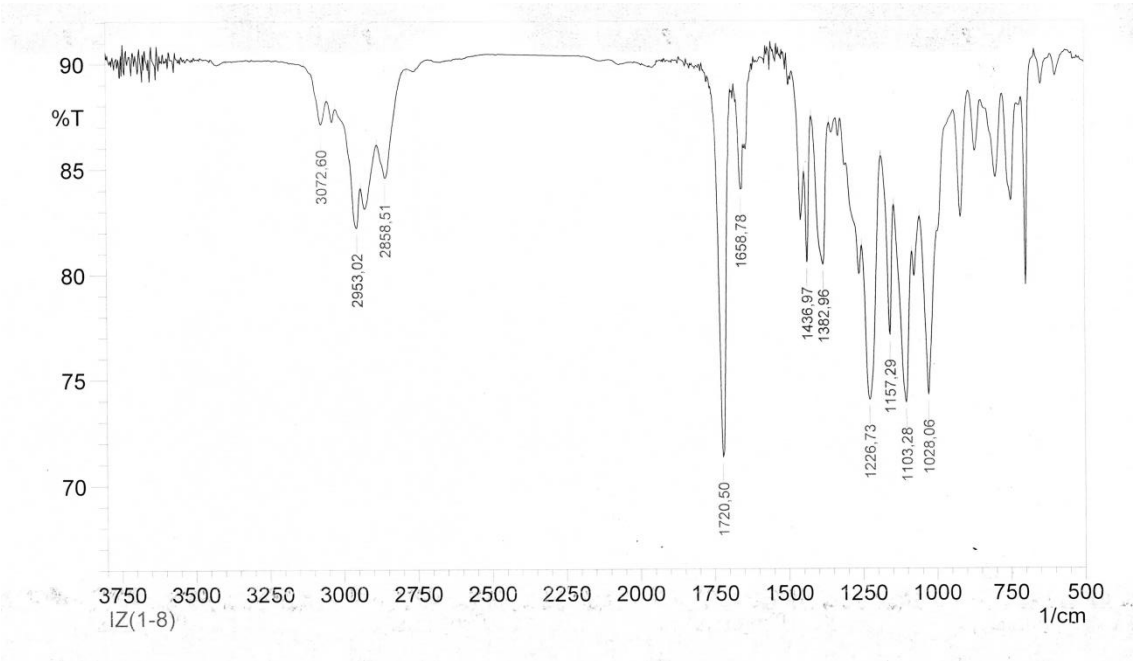


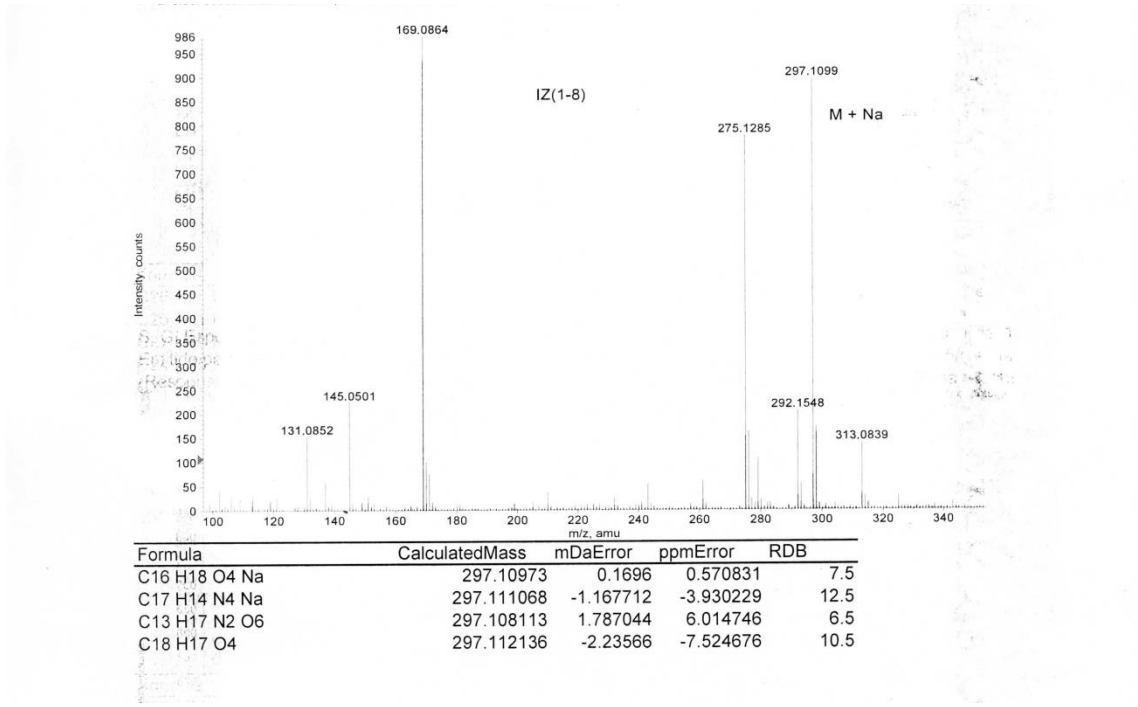


19*Z*



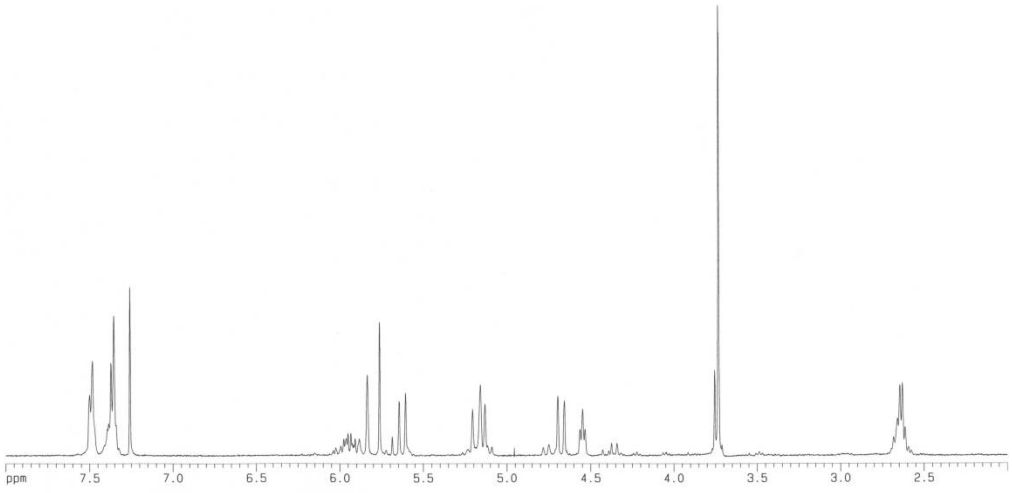
IR and HRMS

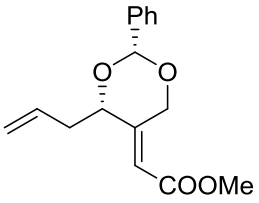




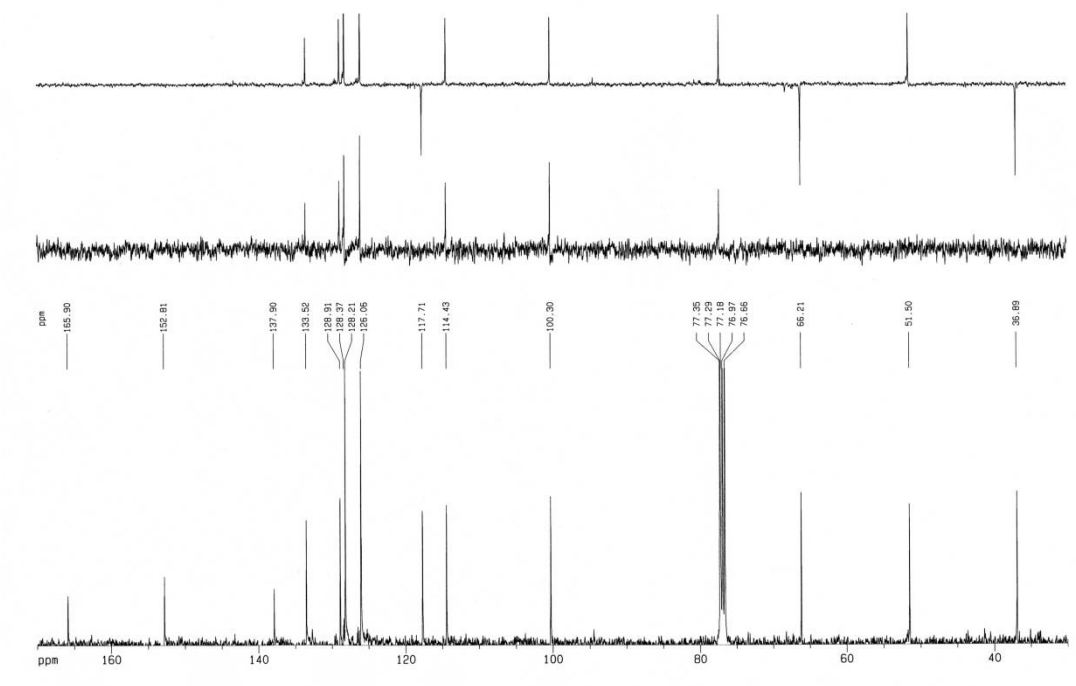
Formula	CalculatedMass	mDaError	ppmError	RDB
C16 H18 O4 Na	297.10973	0.1696	0.570831	7.5
C17 H14 N4 Na	297.111068	-1.167712	-3.930229	12.5
C13 H17 N2 O6	297.108113	1.787044	6.014746	6.5
C18 H17 O4	297.112136	-2.23566	-7.524676	10.5

^{1}H and ^{13}C ($CDCl_3$)

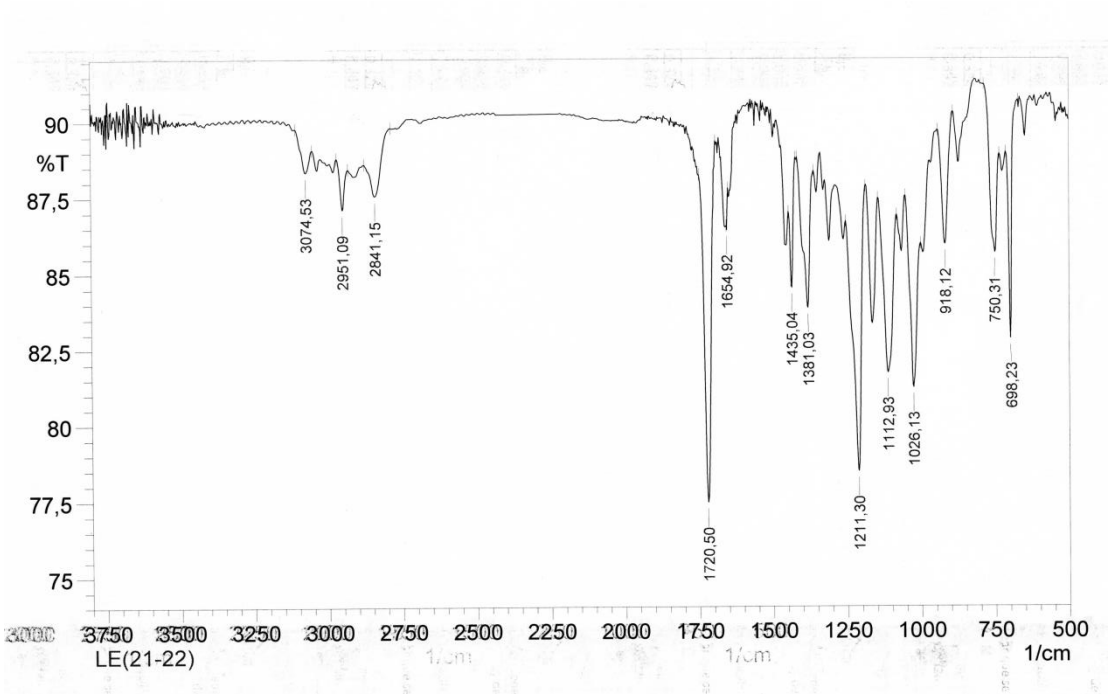


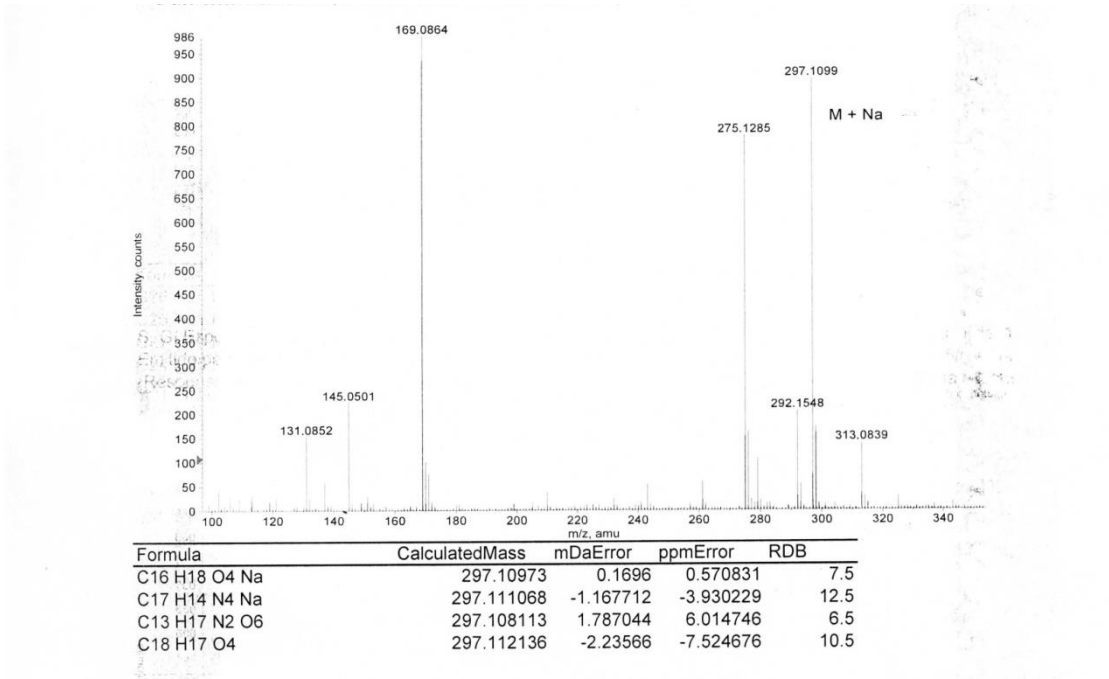


19*E*



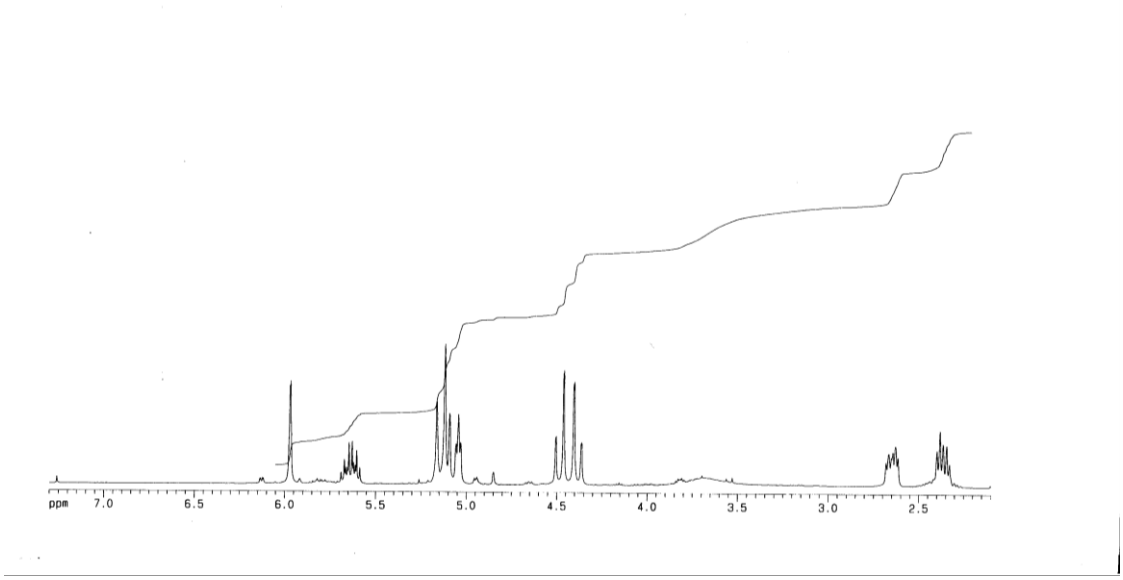
IR and HRMS

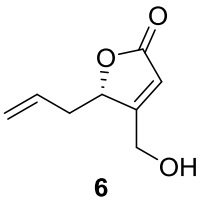




Formula	CalculatedMass	mDaError	ppmError	RDB
C16 H18 O4 Na	297.10973	0.1696	0.570831	7.5
C17 H14 N4 Na	297.111068	-1.167712	-3.930229	12.5
C13 H17 N2 O6	297.108113	1.787044	6.014746	6.5
C18 H17 O4	297.112136	-2.23566	-7.524676	10.5

^{1}H and ^{13}C ($CDCl_3$)





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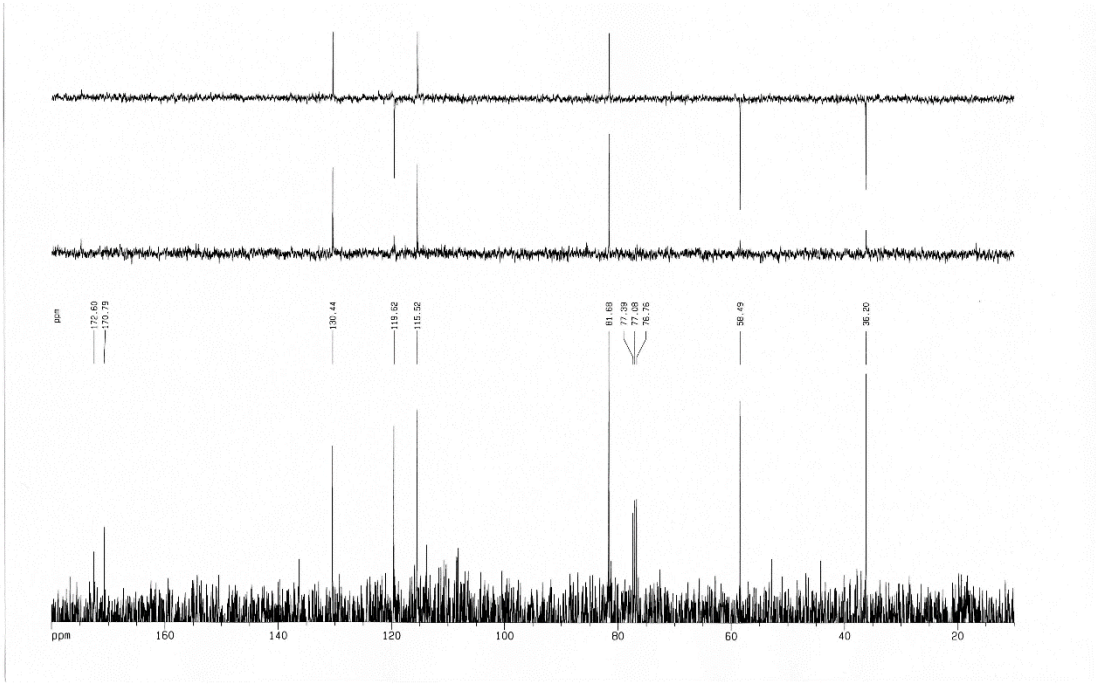
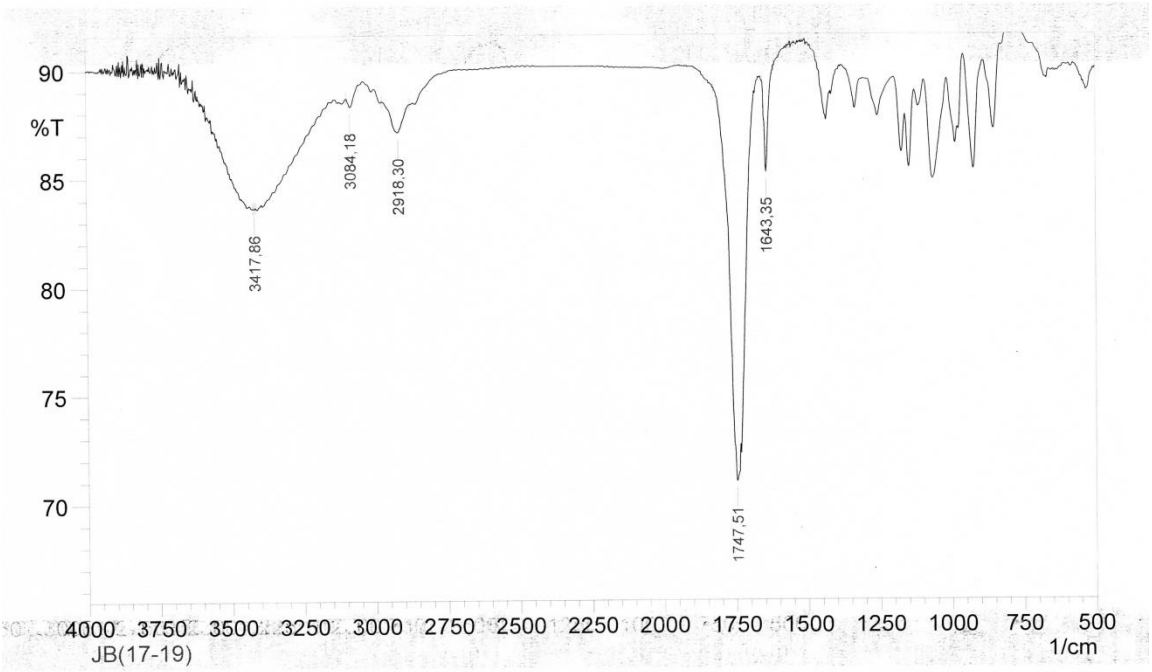
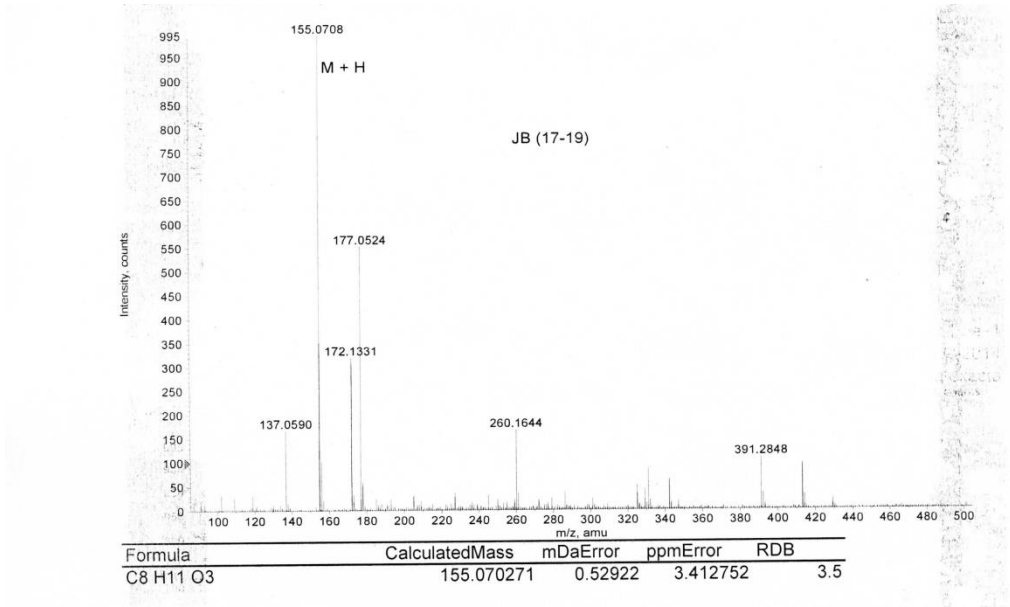


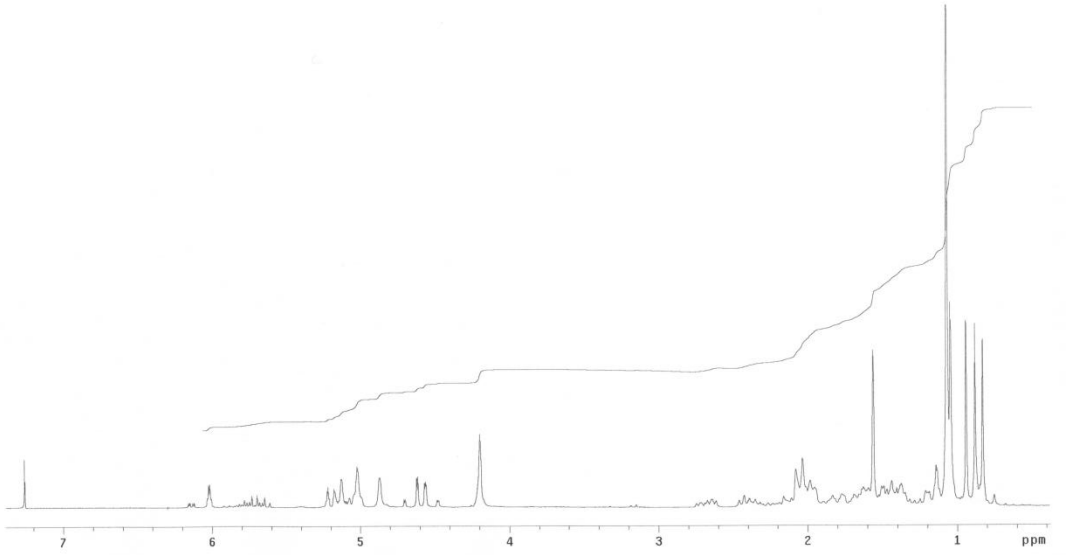
IR and HRMS

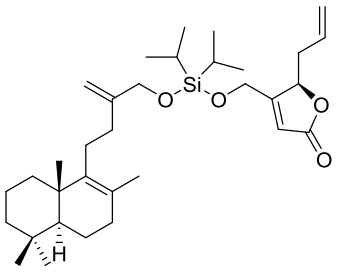




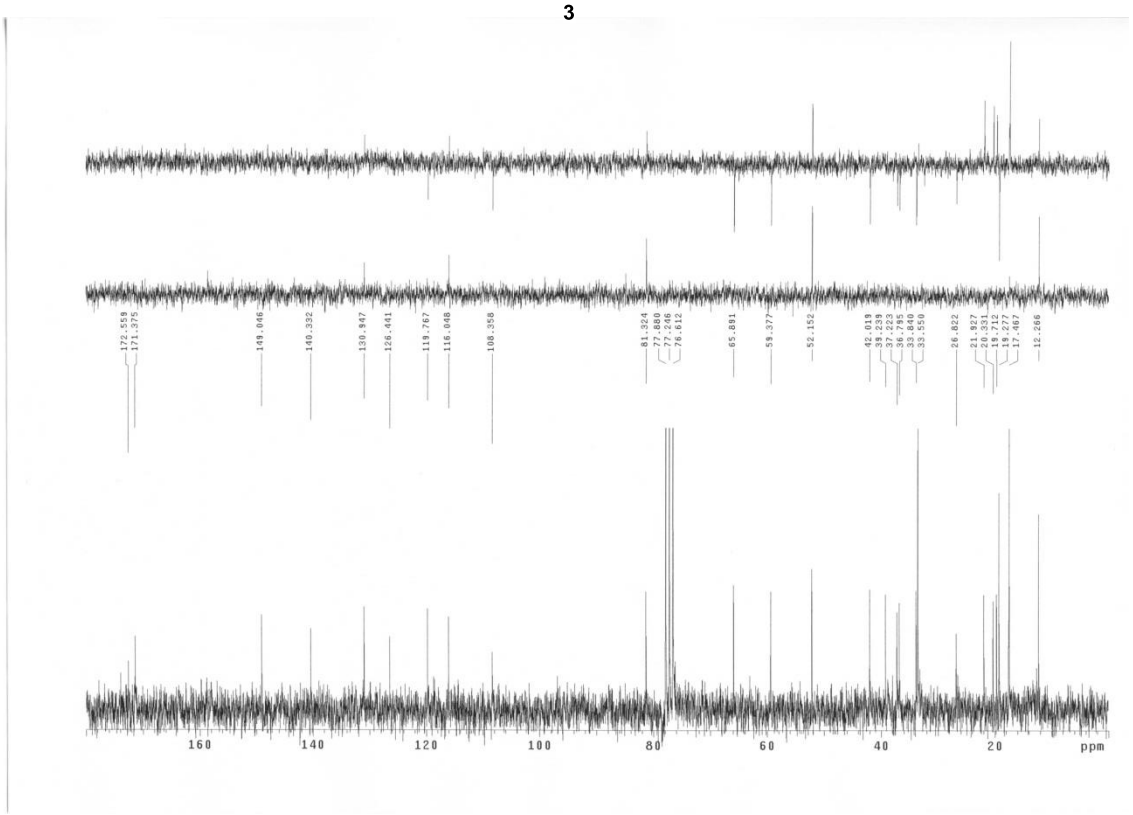
Formula	CalculatedMass	mDaError	ppmError	RDB
C8 H11 O3	155.070271	0.52922	3.412752	3.5

^{1}H and ^{13}C ($CDCl_3$)

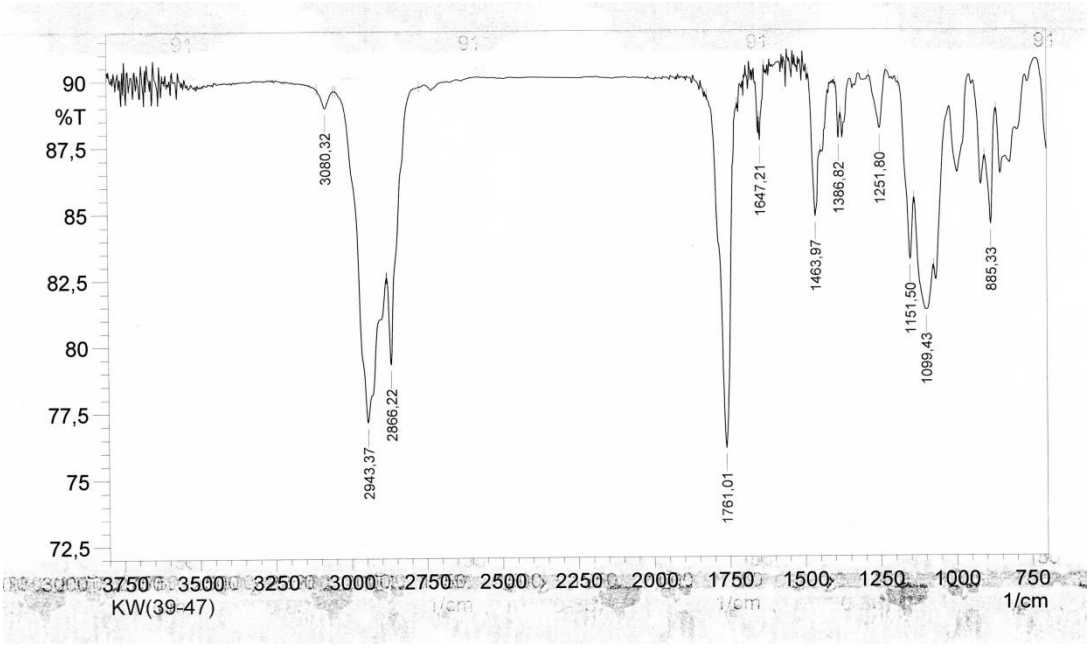


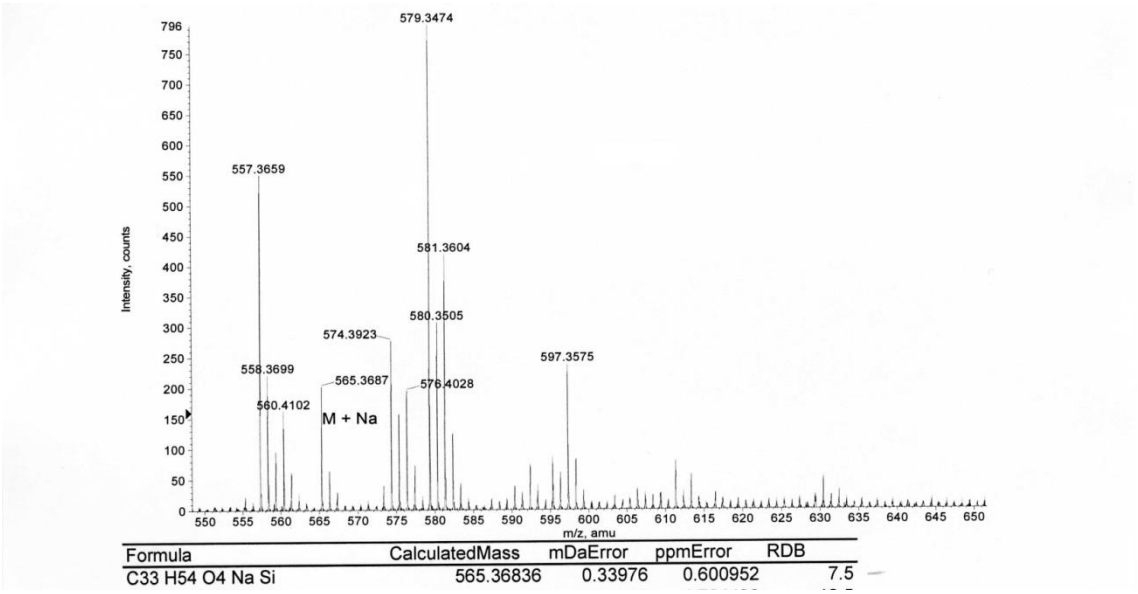


3



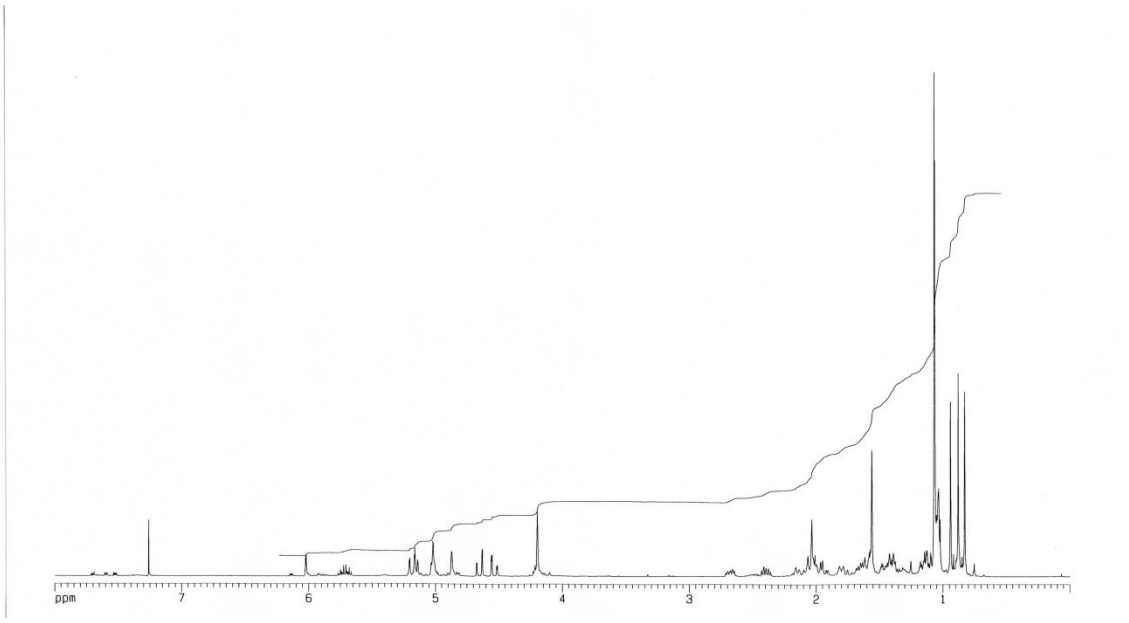
IR and HRMS

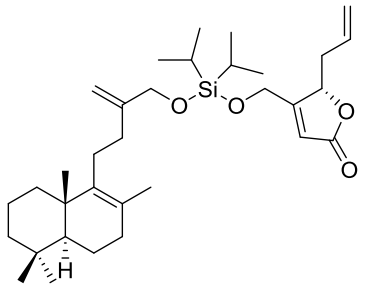




Formula	CalculatedMass	mDaError	ppmError	RDB
C33 H54 O4 Na Si	565.36836	0.33976	0.600952	7.5

^{1}H and ^{13}C ($CDCl_3$)





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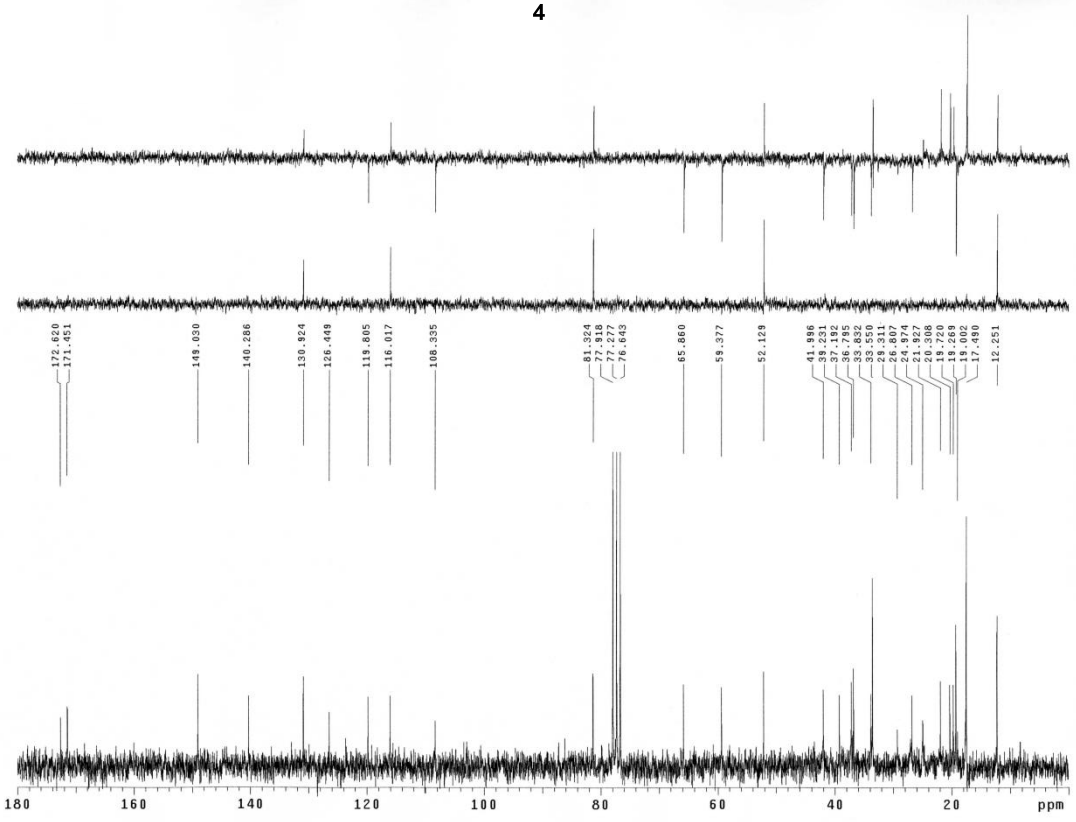
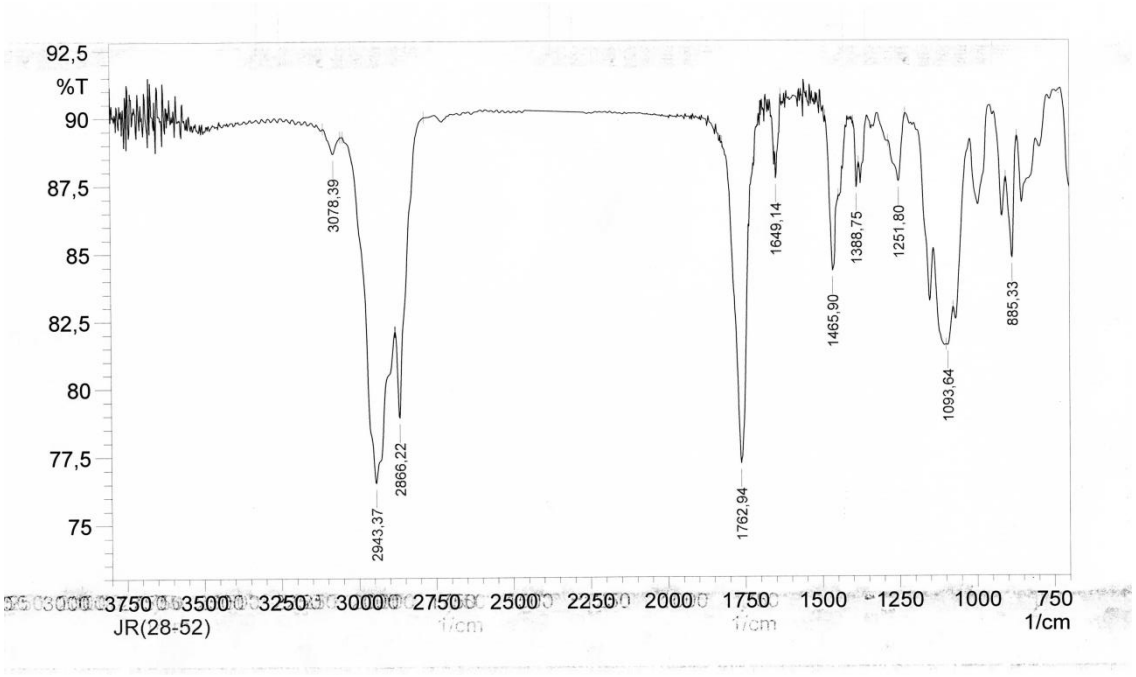
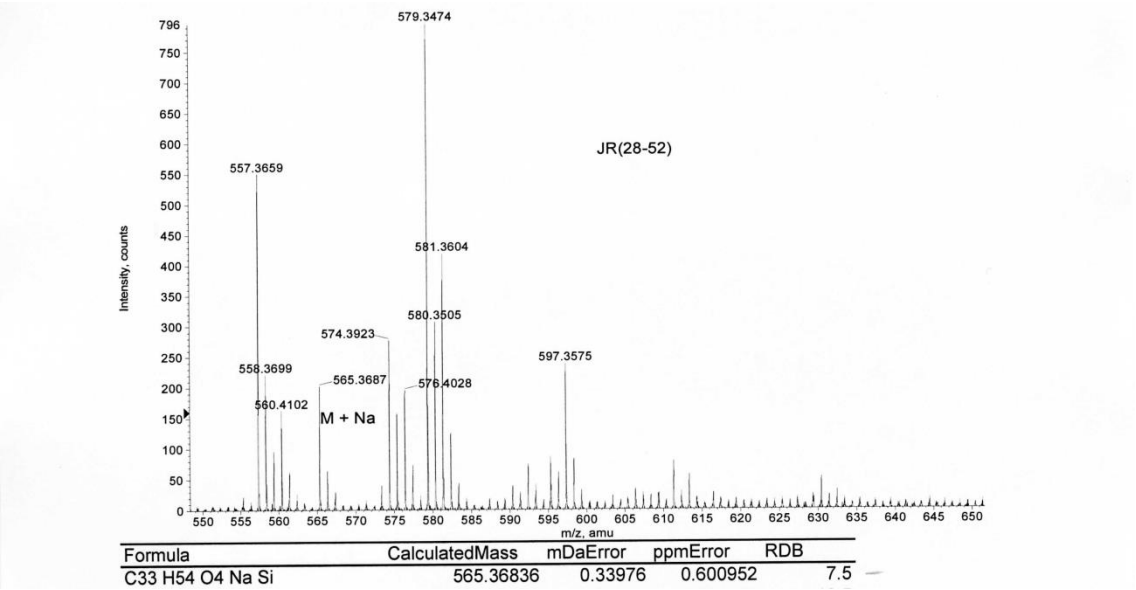
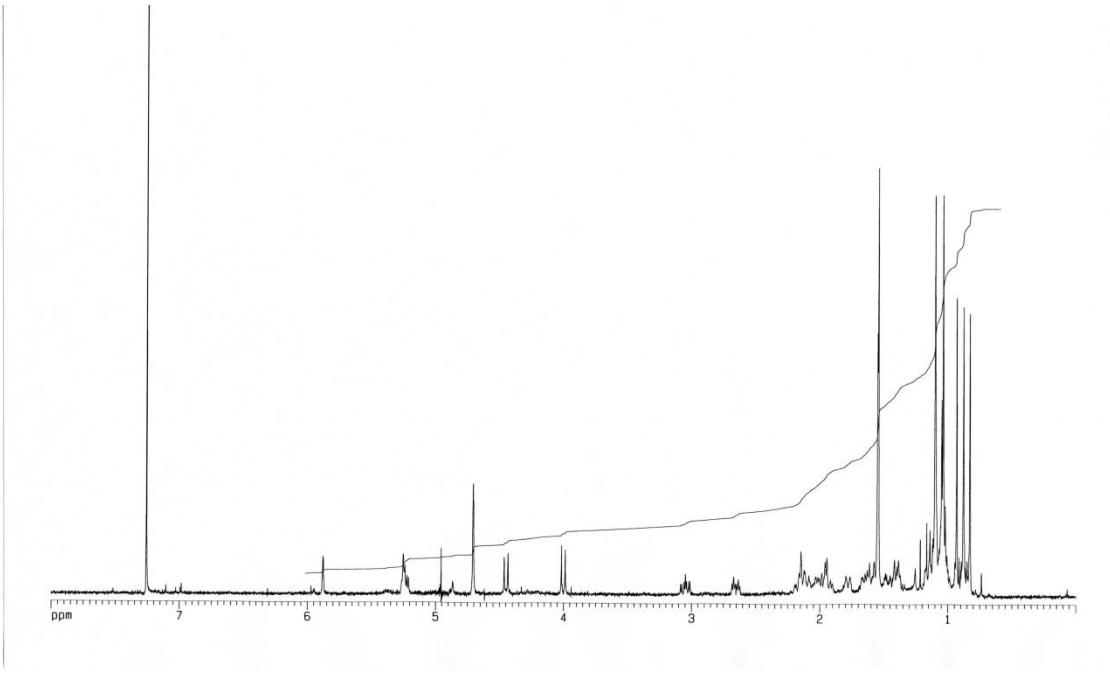


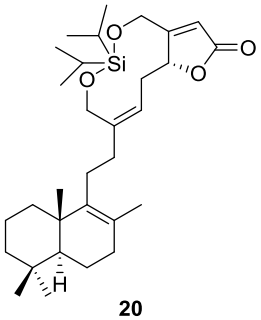
IR and HRMS


92,5
%T
90
87,5
85
82,5
80
77,5
75
3078,39
2943,37
2866,22
1762,94
1649,14
1465,90
1388,75
1251,80
1093,64
885,33
3750
3500
3250
3000
2750
2500
2250
2000
1750
1500
1250
1000
750
1/cm
JR(28-52)

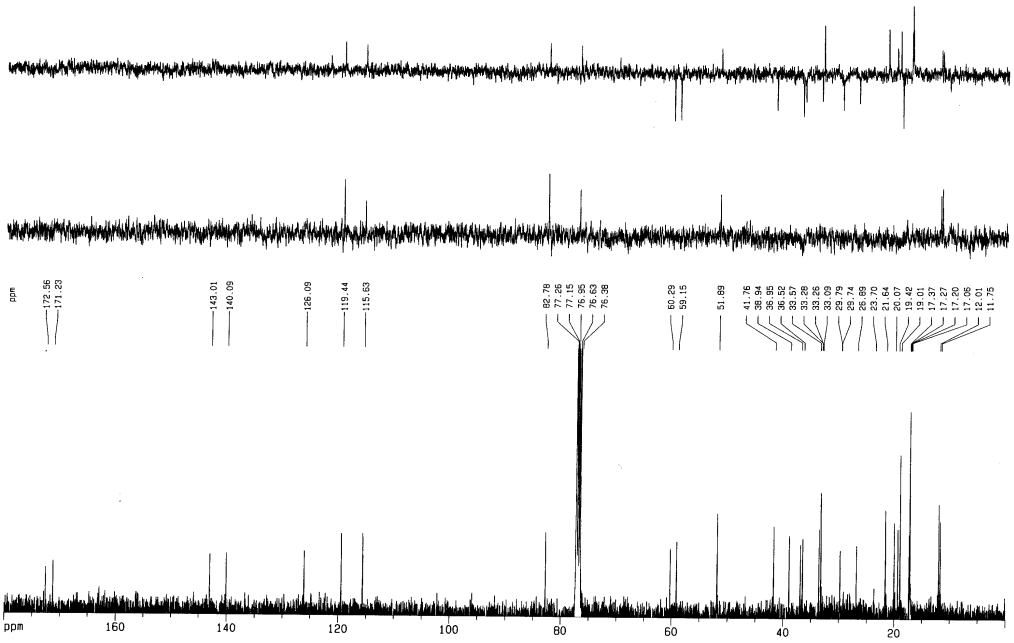

JR(28-52)
Intensity, counts
m/z, amu
579.3474
557.3659
581.3604
580.3505
574.3923
597.3575
558.3699
565.3687
576.4028
560.4102
M + Na
Formula
CalculatedMass
mDaError
ppmError
RDB
C33 H54 O4 Na Si
565.36836
0.33976
0.600952
7.5

^{1}H and ^{13}C ($CDCl_3$)

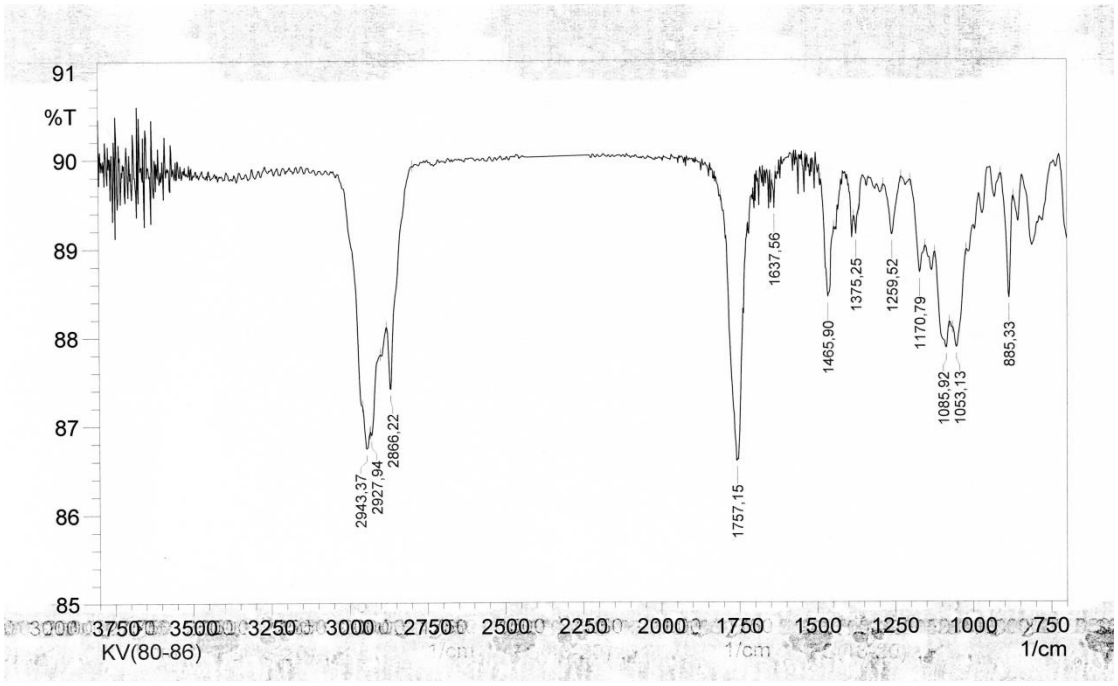


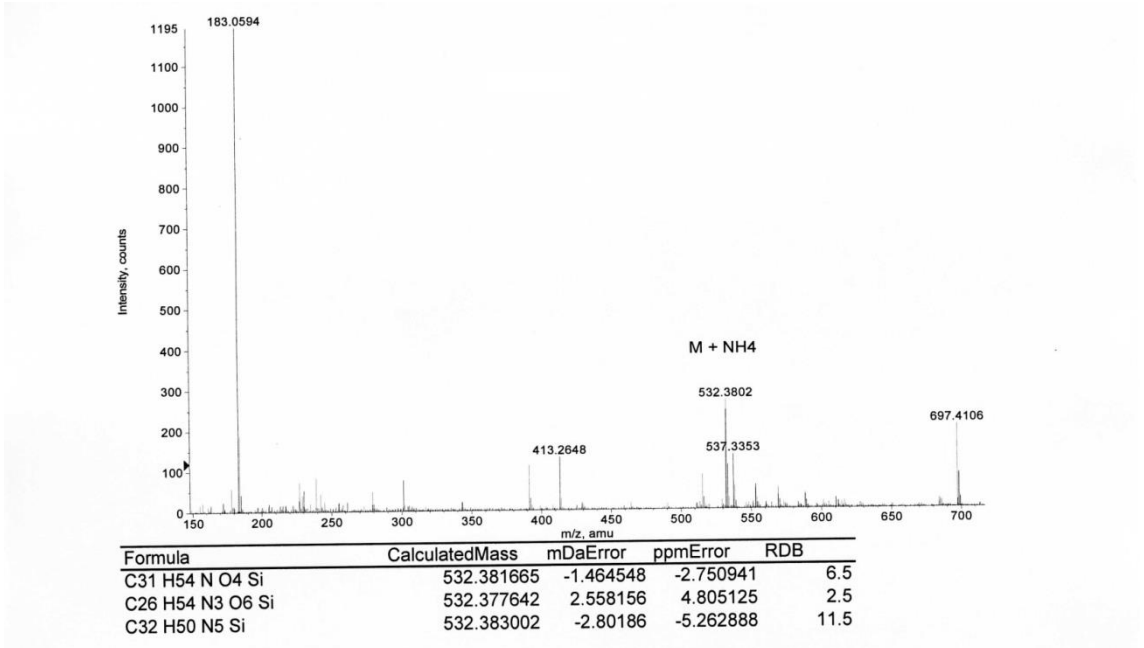


20



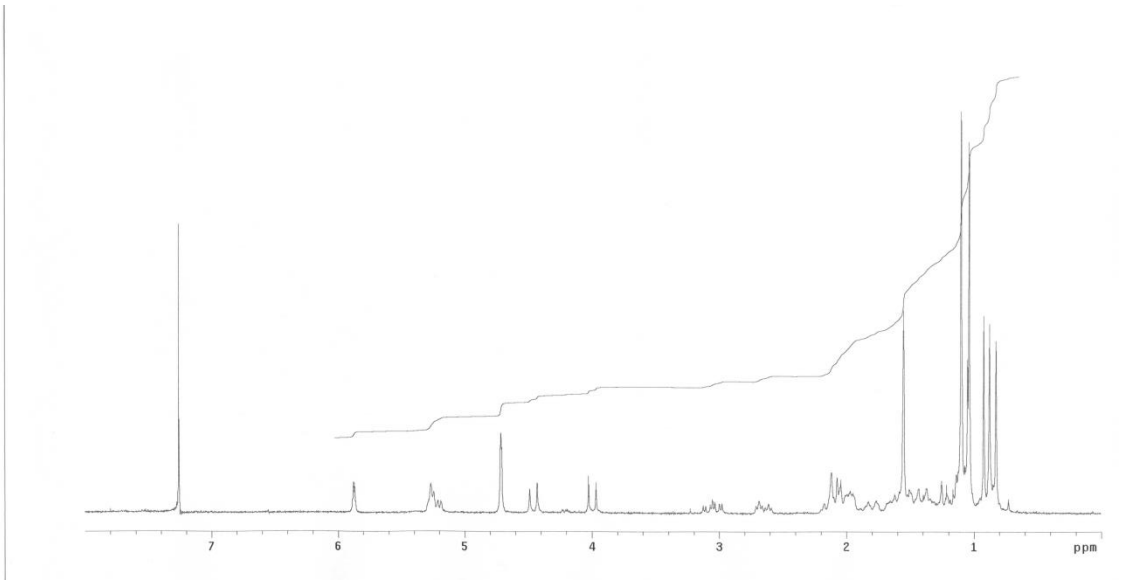
IR and HRMS

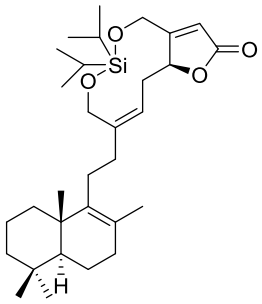




Formula	CalculatedMass	mDaError	ppmError	RDB
C31 H54 N O4 Si	532.381665	-1.464548	-2.750941	6.5
C26 H54 N3 O6 Si	532.377642	2.558156	4.805125	2.5
C32 H50 N5 Si	532.383002	-2.80186	-5.262888	11.5

^{1}H and ^{13}C ($CDCl_3$)





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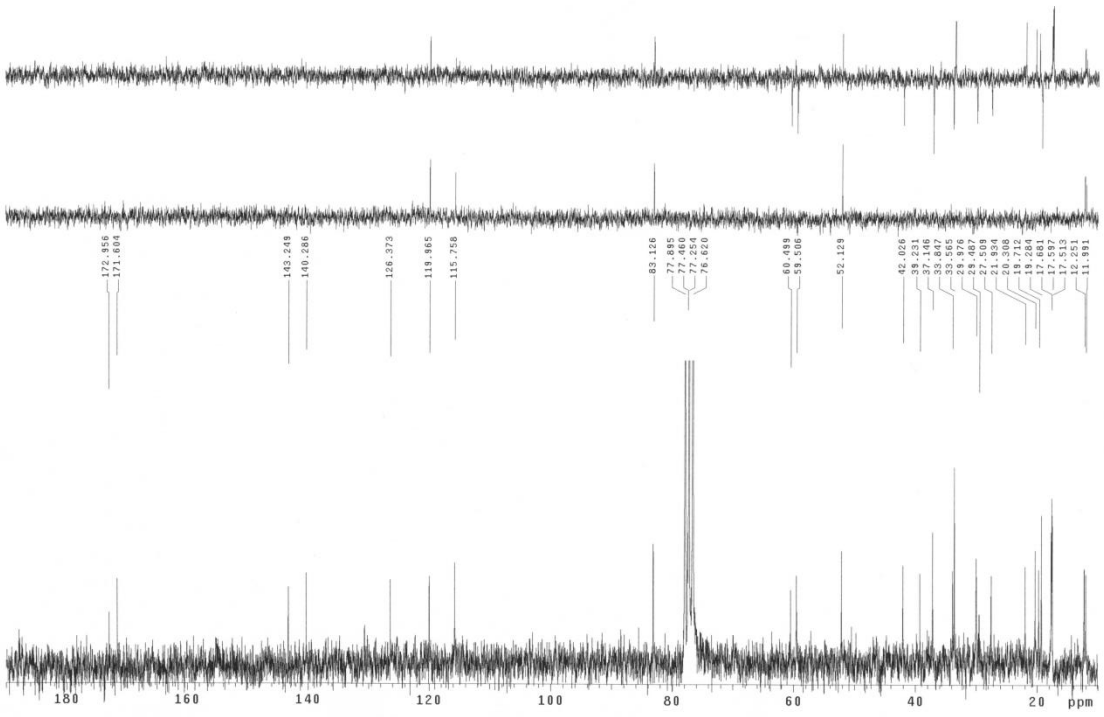
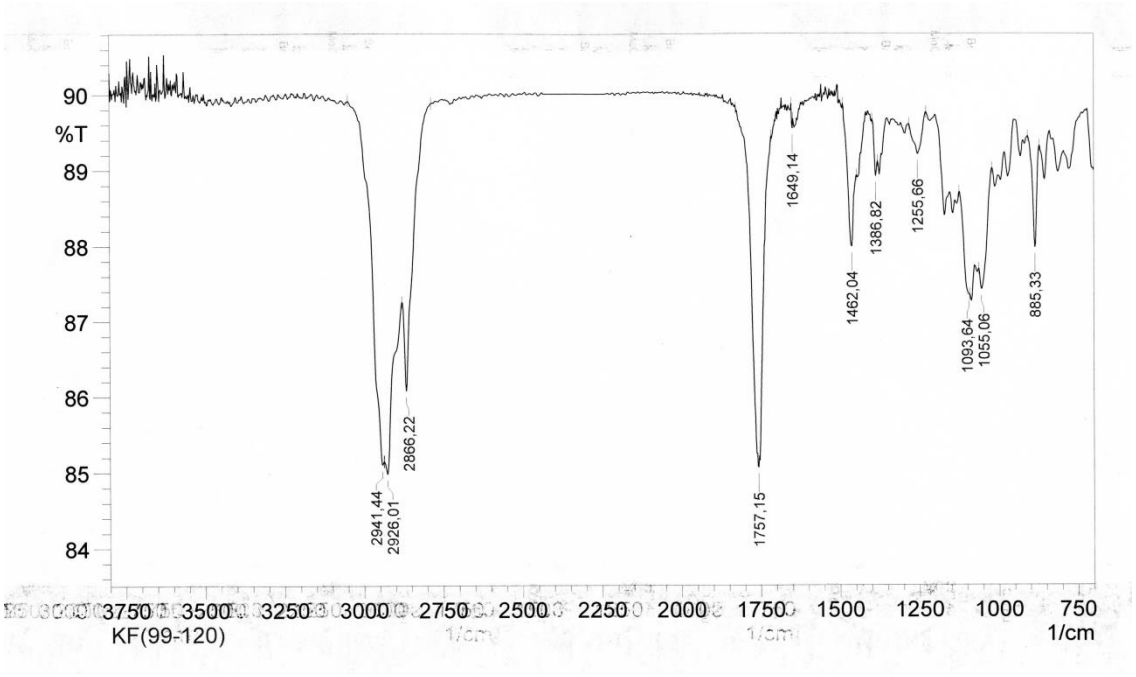
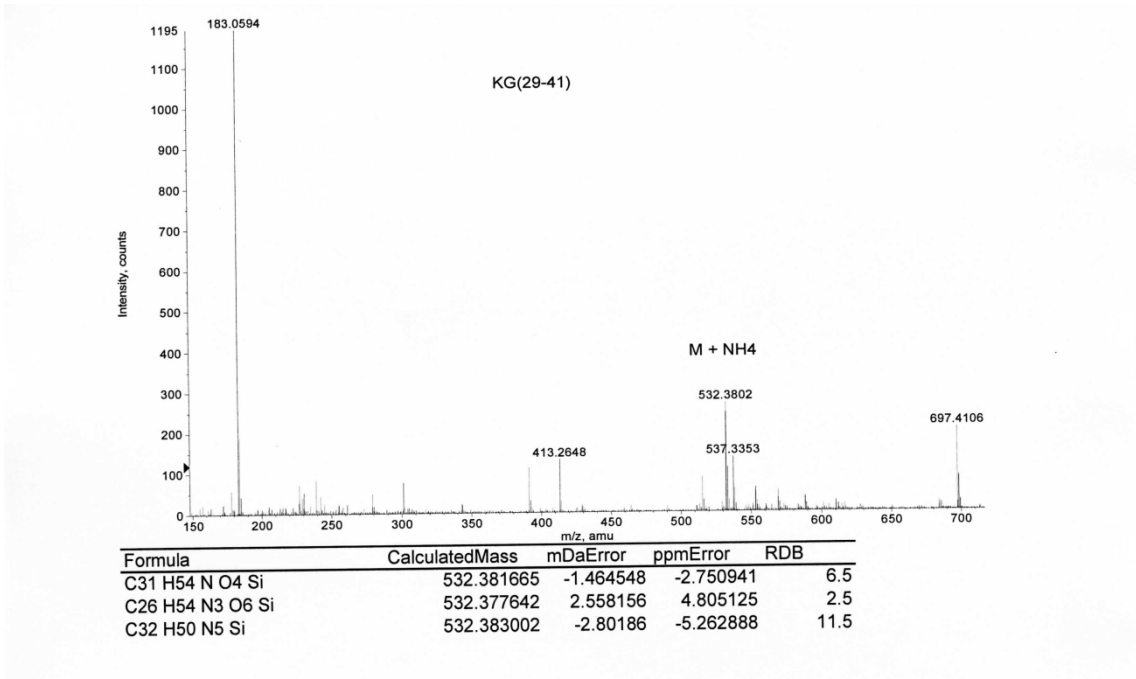


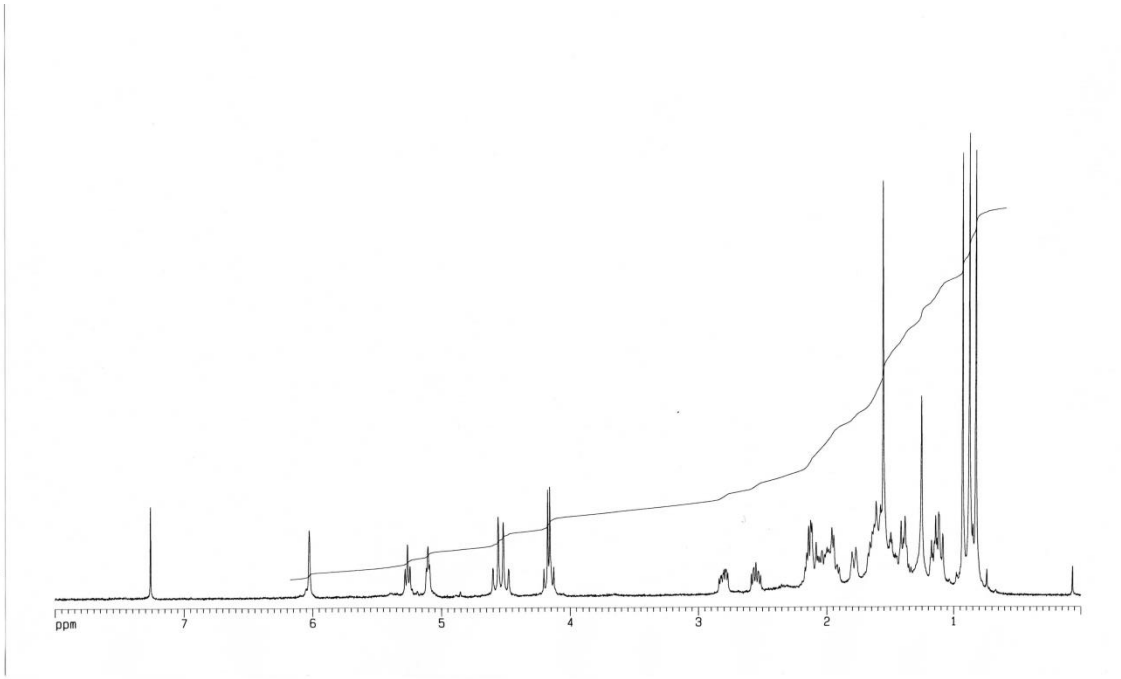
IR and HRMS

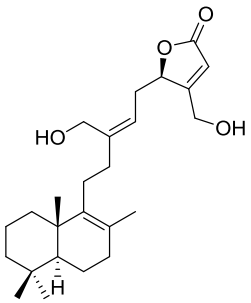




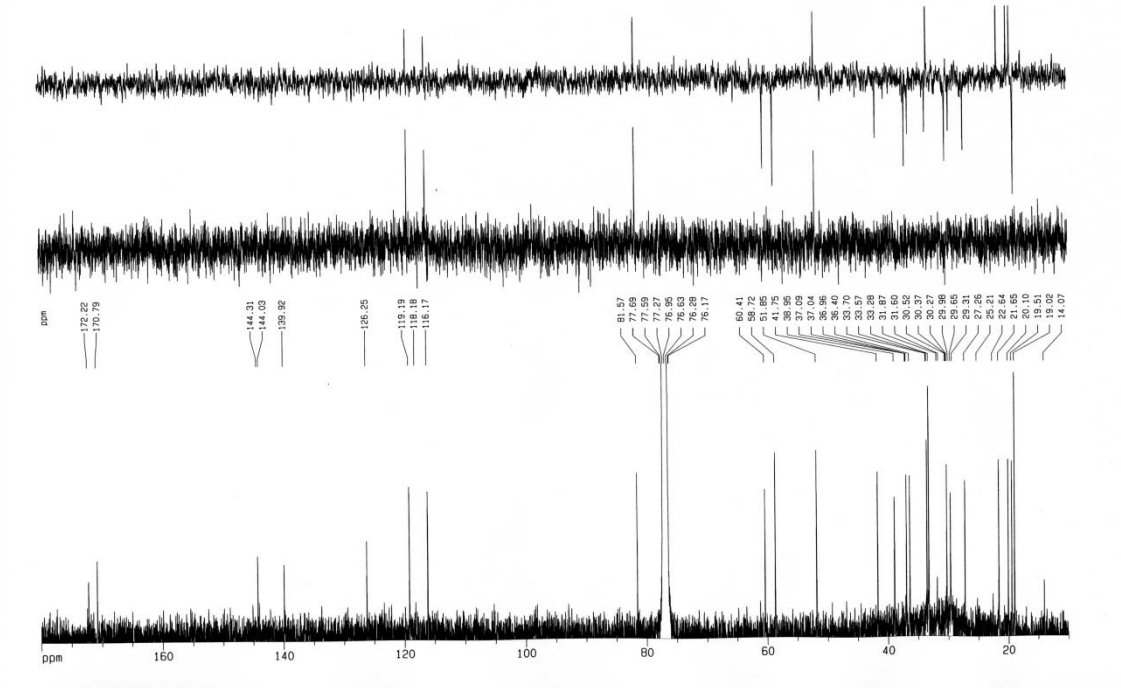
Formula	CalculatedMass	mDaError	ppmError	RDB
C31 H54 N O4 Si	532.381665	-1.464548	-2.750941	6.5
C26 H54 N3 O6 Si	532.377642	2.558156	4.805125	2.5
C32 H50 N5 Si	532.383002	-2.80186	-5.262888	11.5

^{1}H and ^{13}C ($CDCl_3$)

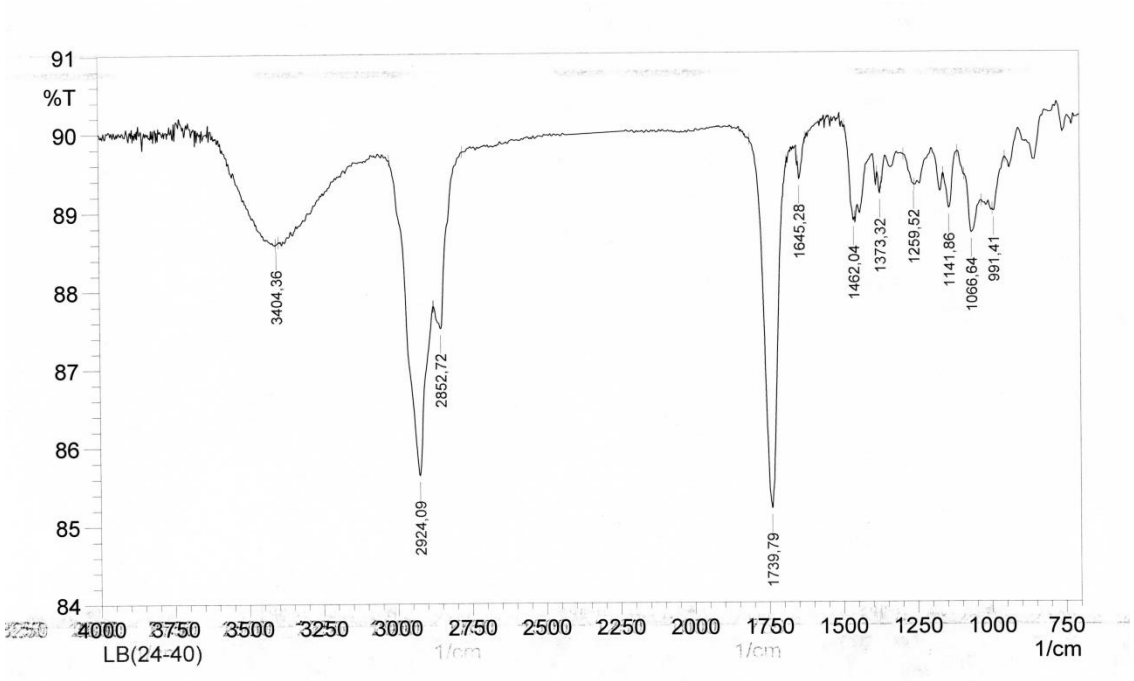


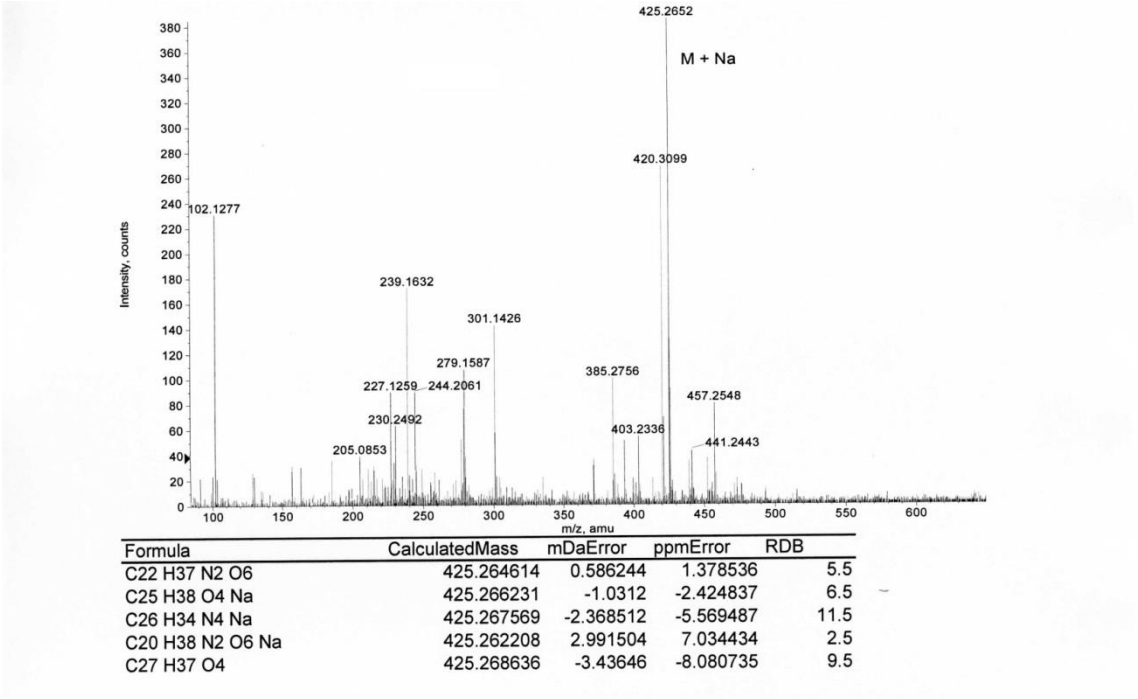


Luffarina L (**1**)



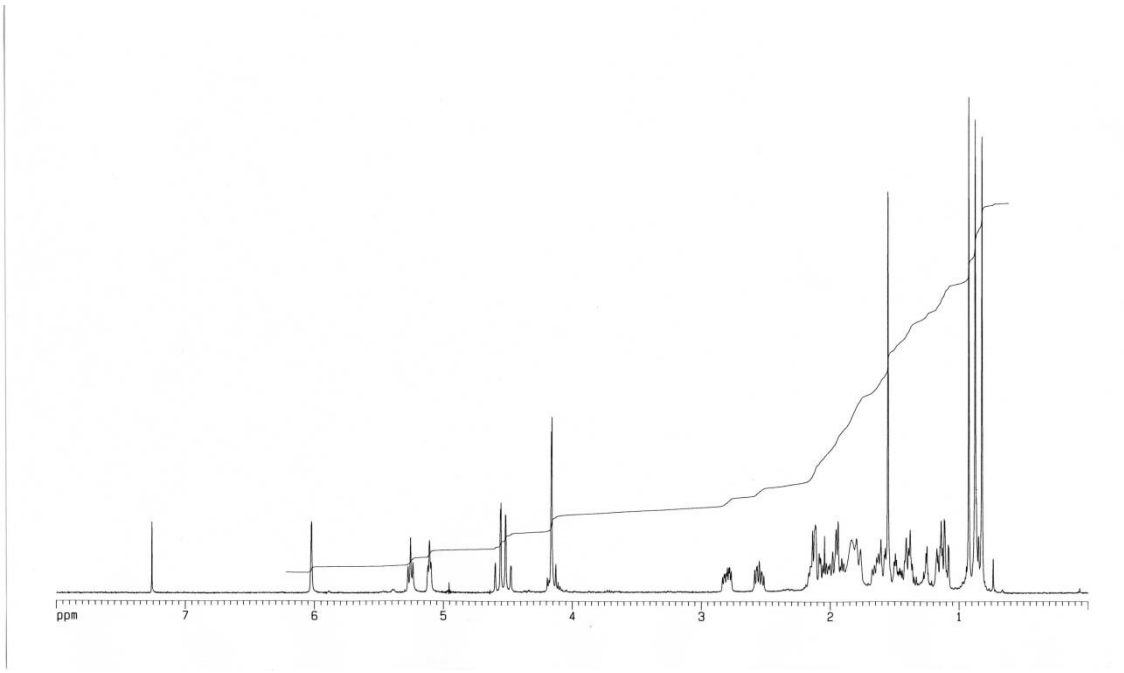
IR and HRMS

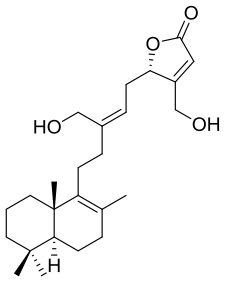




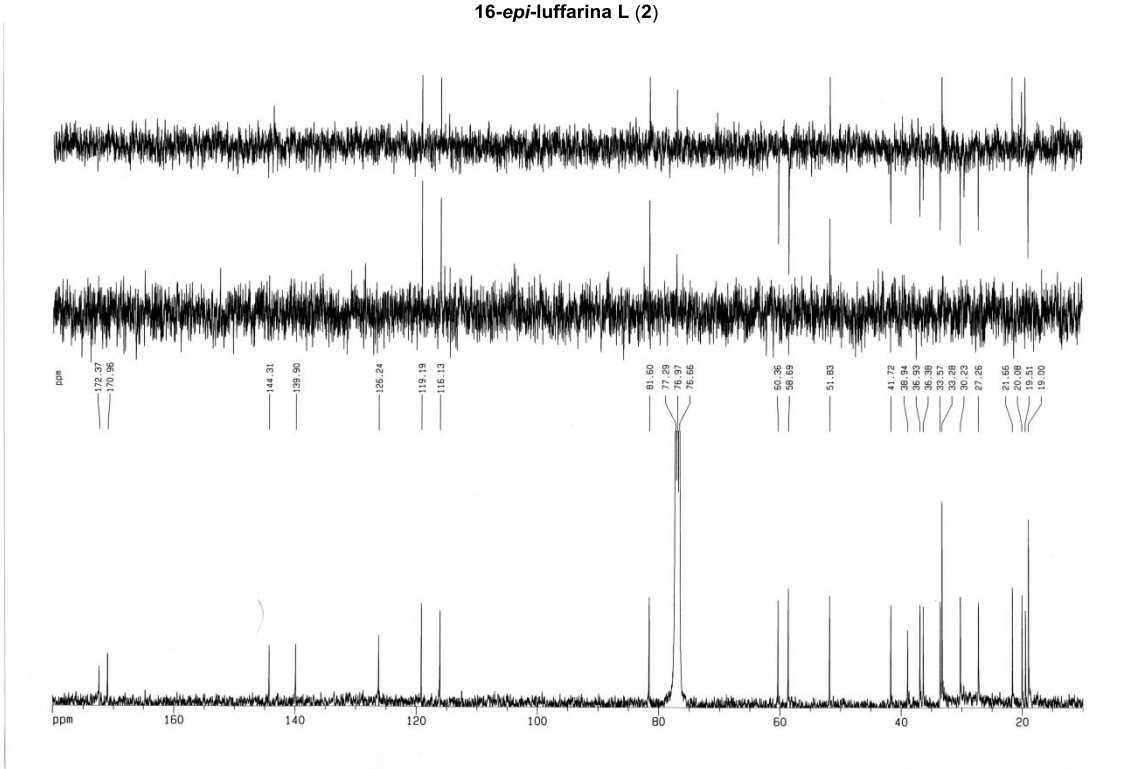
Formula	CalculatedMass	mDaError	ppmError	RDB
C22 H37 N2 O6	425.264614	0.586244	1.378536	5.5
C25 H38 O4 Na	425.266231	-1.0312	-2.424837	6.5
C26 H34 N4 Na	425.267569	-2.368512	-5.569487	11.5
C20 H38 N2 O6 Na	425.262208	2.991504	7.034434	2.5
C27 H37 O4	425.268636	-3.43646	-8.080735	9.5

^{1}H and ^{13}C ($CDCl_3$)

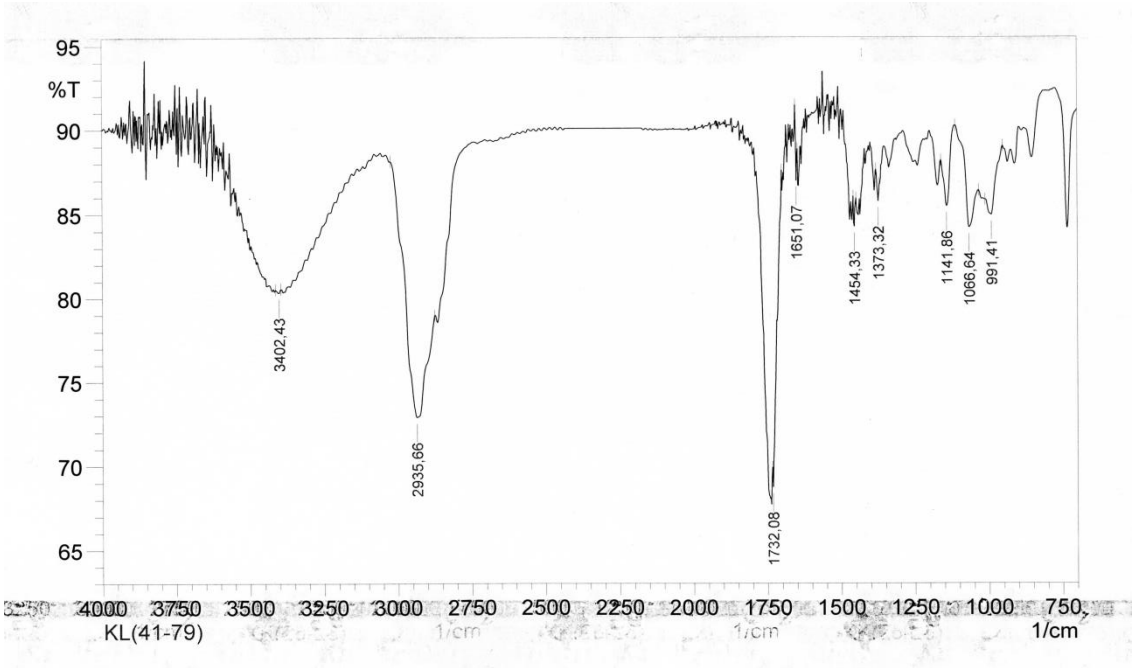


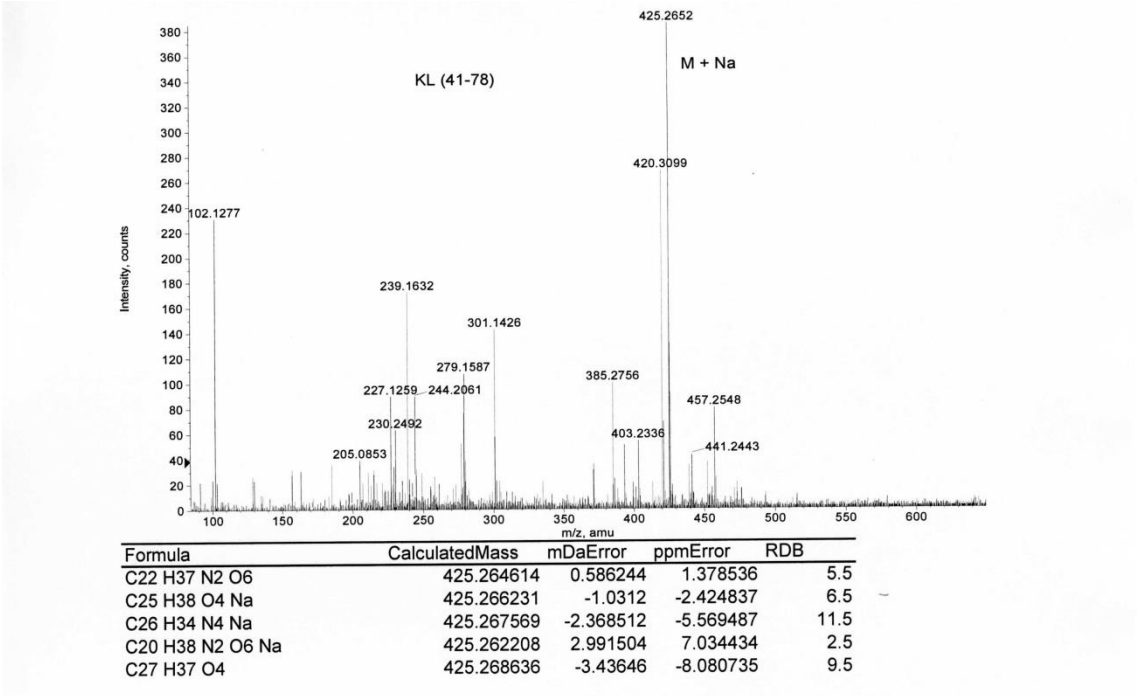


16-*epi*-luffarina L (**2**)



IR and HRMS





Formula	CalculatedMass	mDaError	ppmError	RDB
C22 H37 N2 O6	425.264614	0.586244	1.378536	5.5
C25 H38 O4 Na	425.266231	-1.0312	-2.424837	6.5
C26 H34 N4 Na	425.267569	-2.368512	-5.569487	11.5
C20 H38 N2 O6 Na	425.262208	2.991504	7.034434	2.5
C27 H37 O4	425.268636	-3.43646	-8.080735	9.5