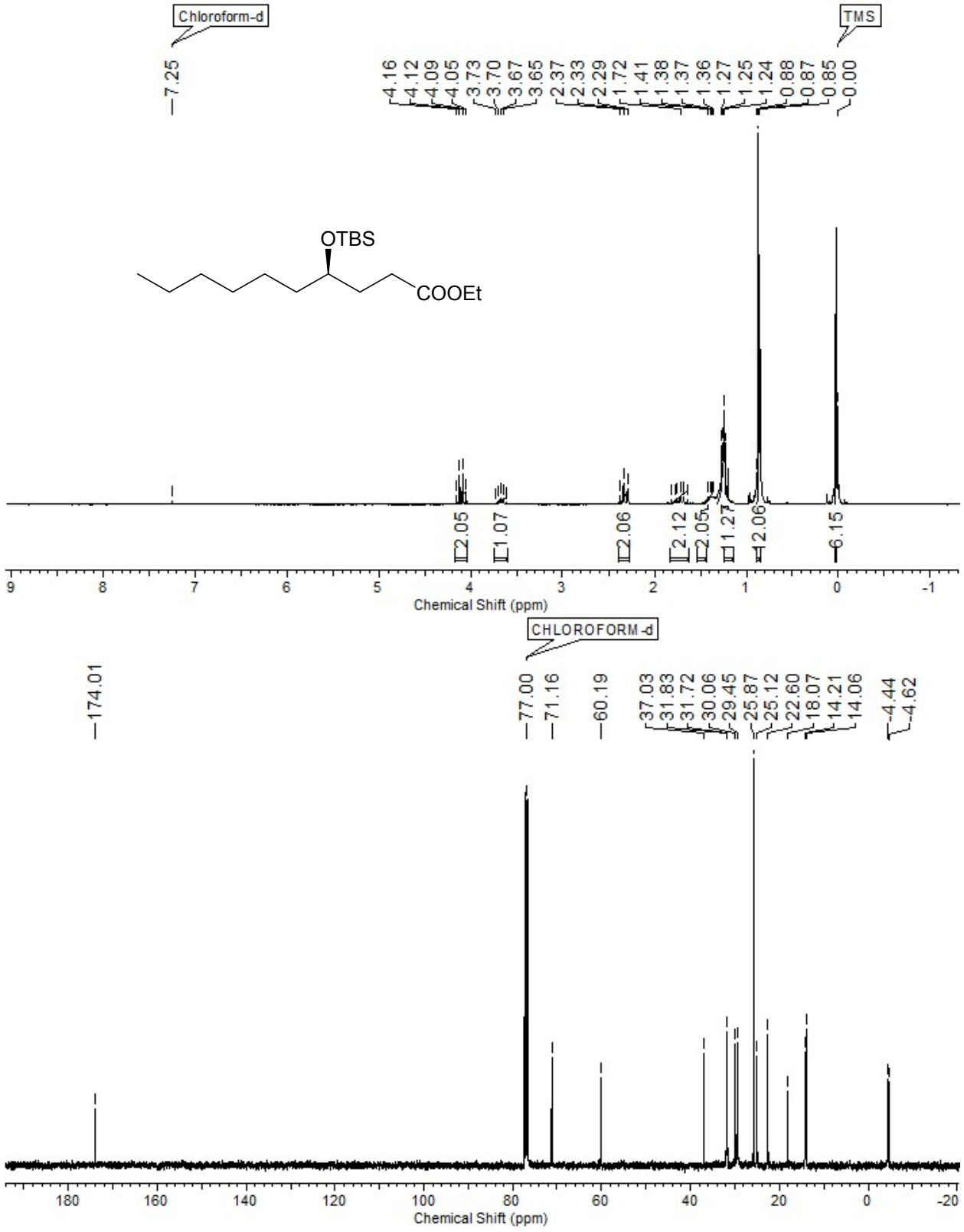
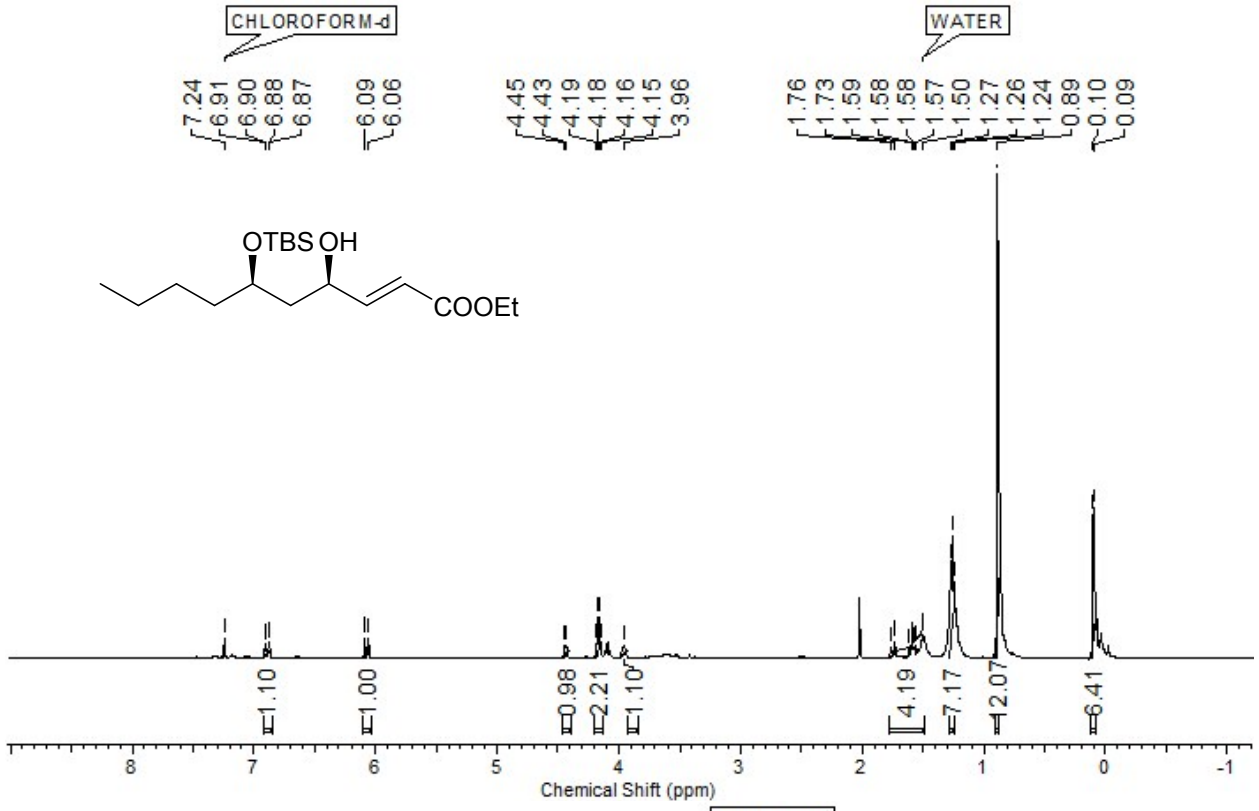
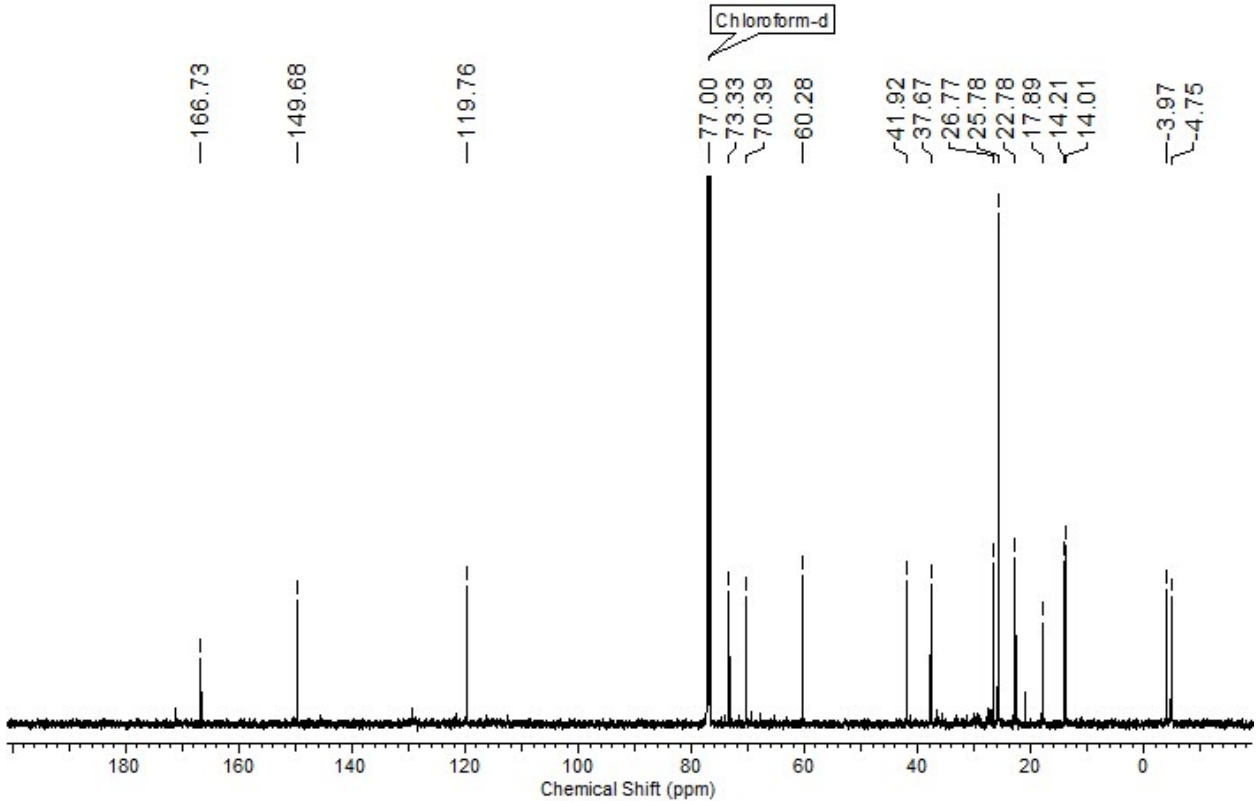
**Ethyl (*R*)-4-((tert-butyldimethylsilyl)oxy)decanoate(7b):**

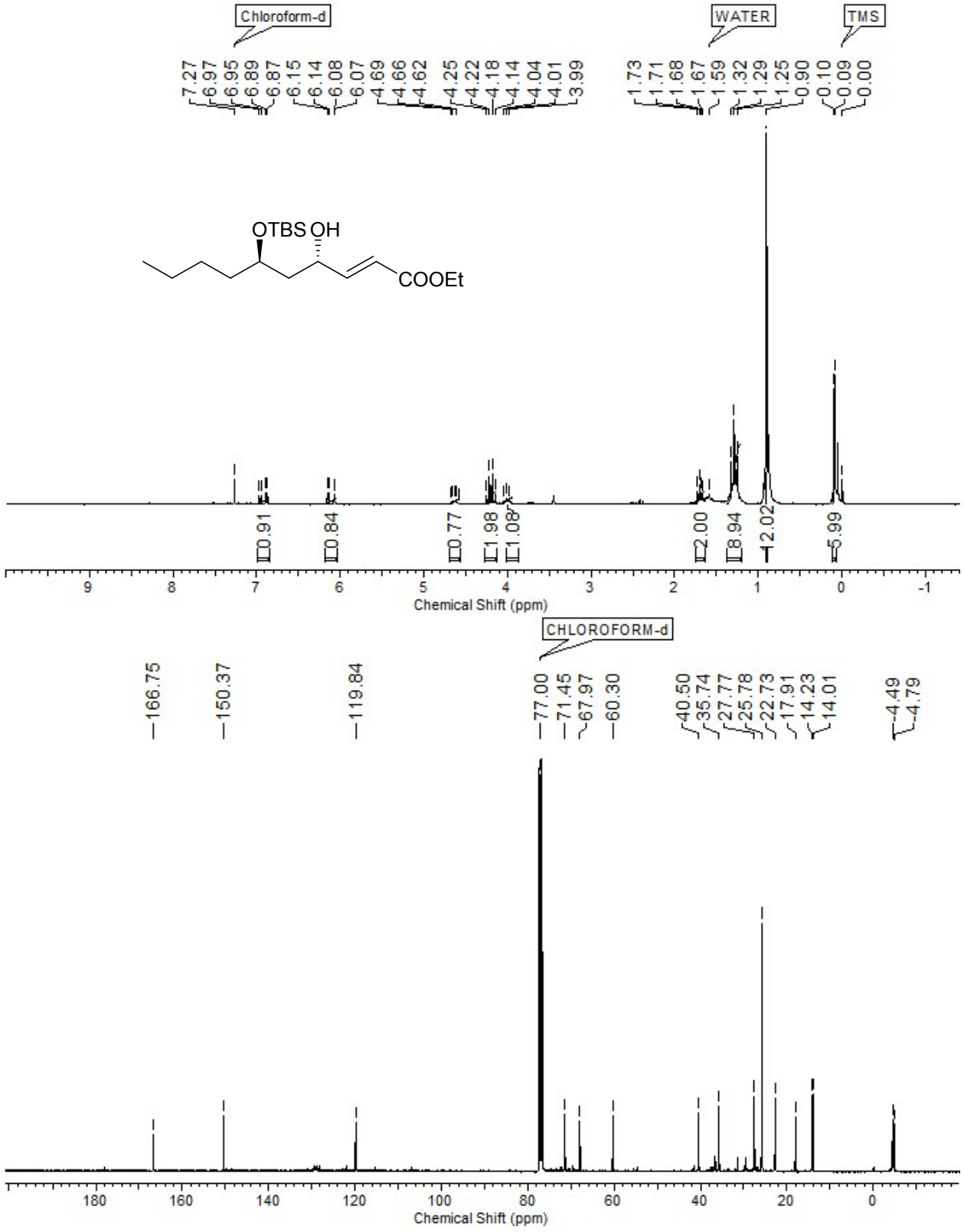


**Ethyl (4*R*,6*R*,*E*)-6-((tert-butyldimethylsilyl)oxy)-4-hydroxydec-2-enoate (8a):**

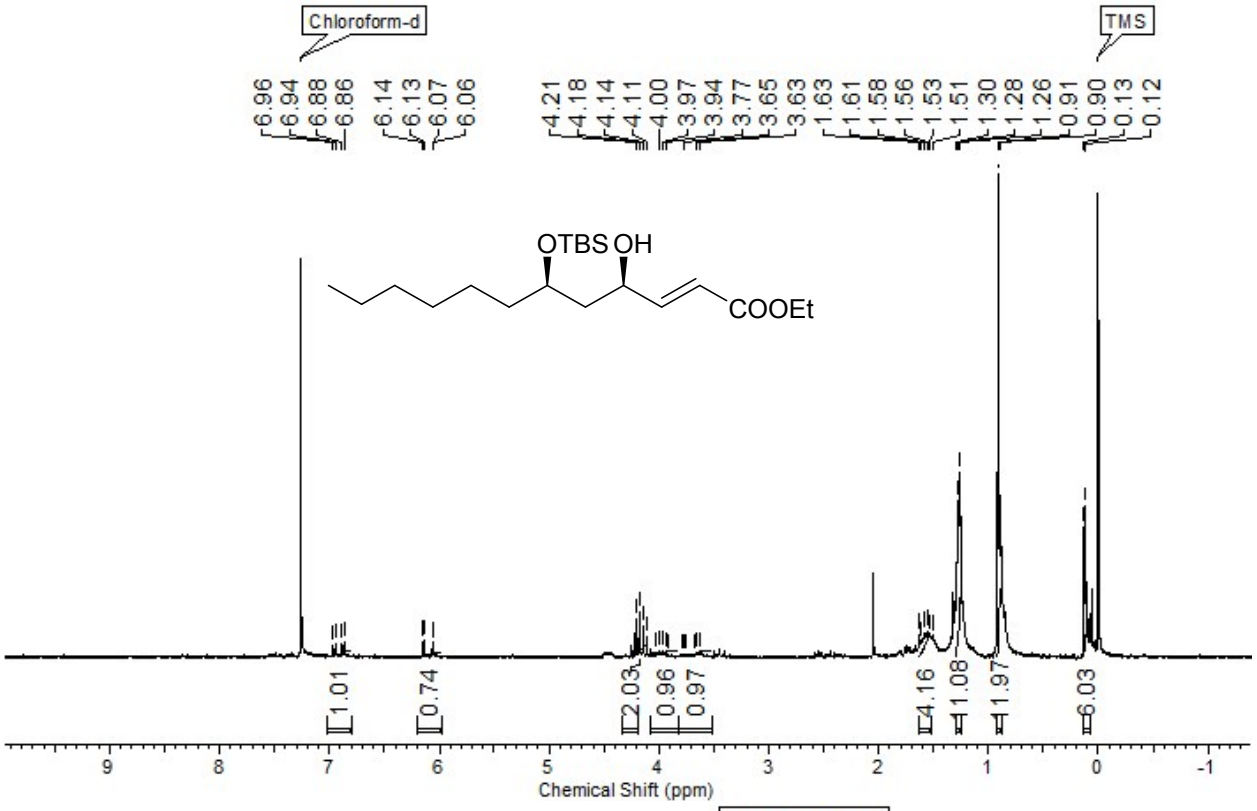


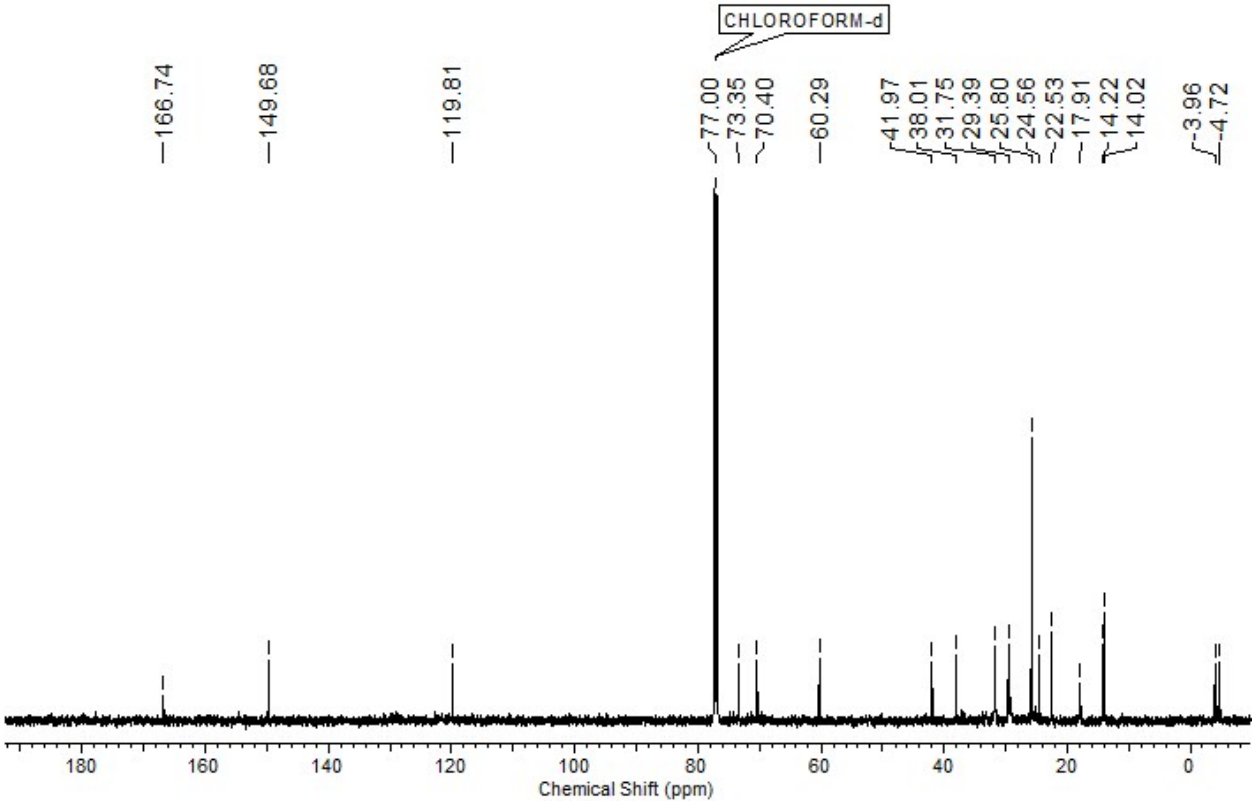



**Ethyl (4*S*,6*R*,*E*)-6-((tert-butyldimethylsilyl)oxy)-4-hydroxydec-2-enoate (8c):**

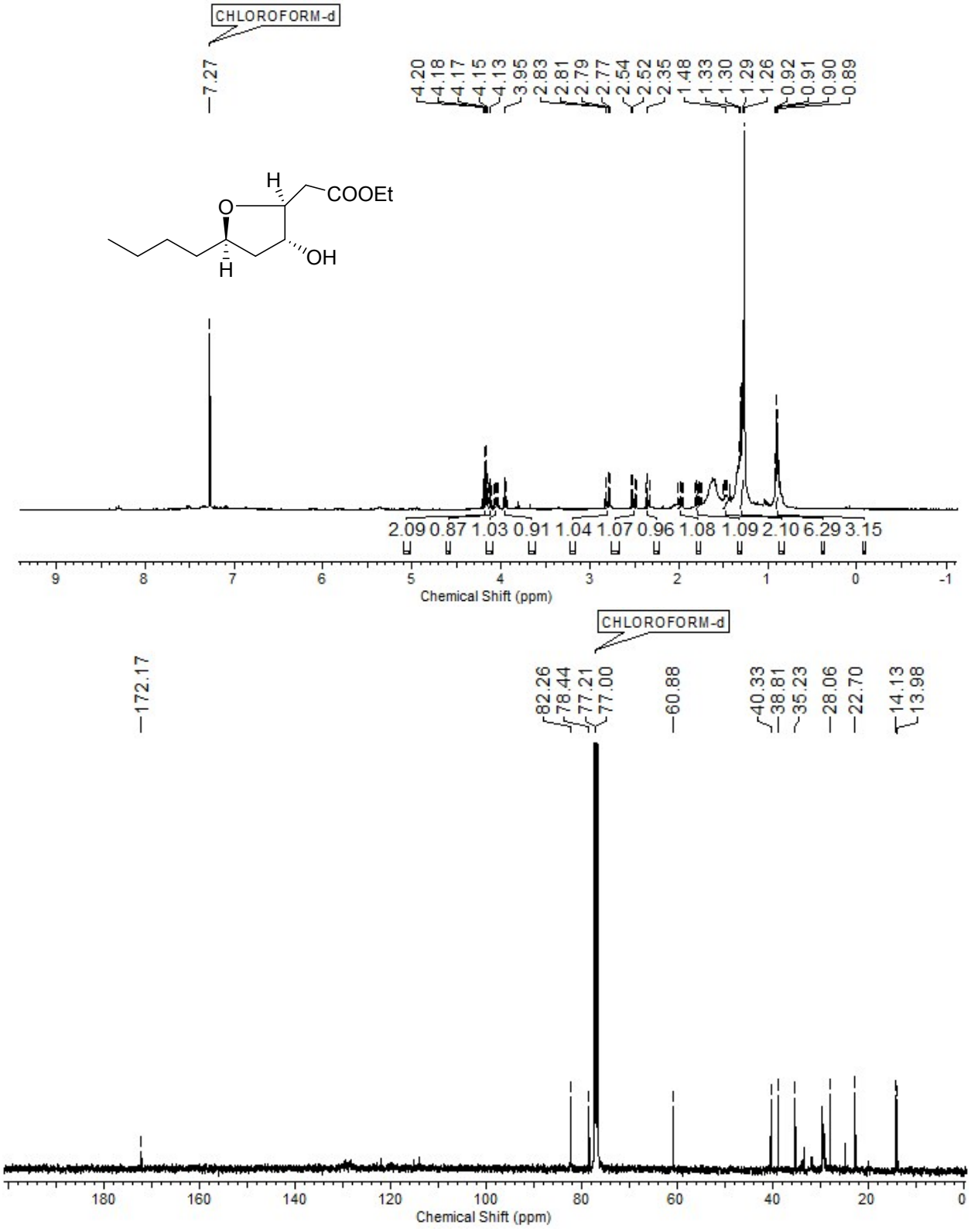


**Ethyl (4*R*,6*R*,*E*)-6-((tert-butyldimethylsilyl)oxy)-4-hydroxydodec-2-enoate: (8b)**

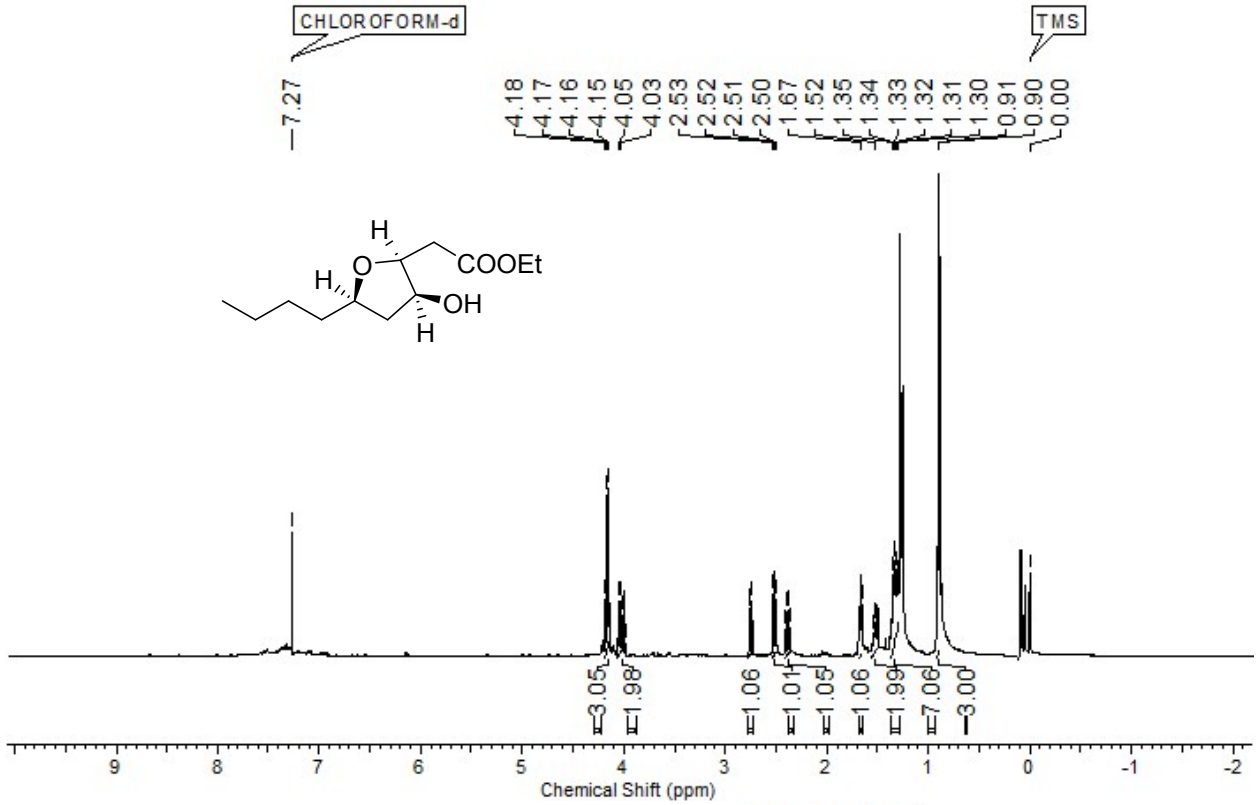


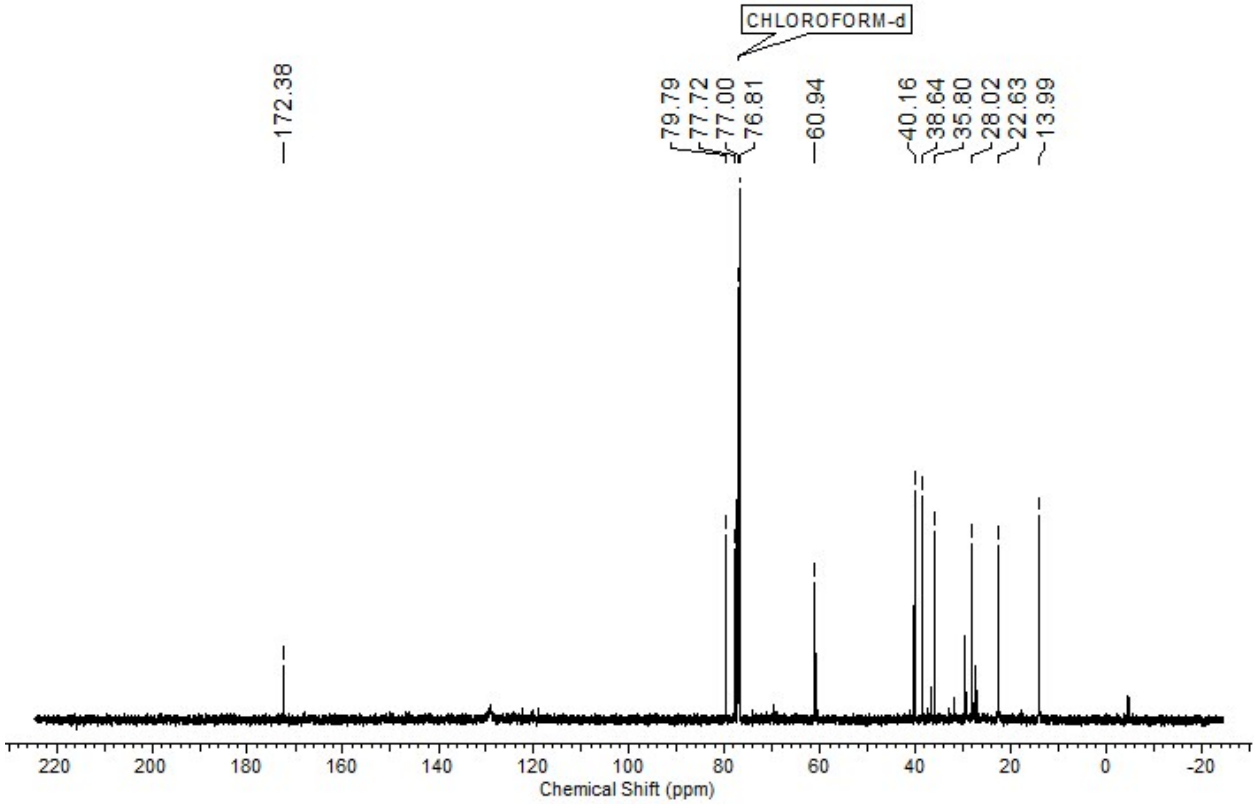



**Ethyl 2-((2*S*,3*R*,5*R*)-5-butyl-3-hydroxytetrahydrofuran-2-yl)acetate: (9)**

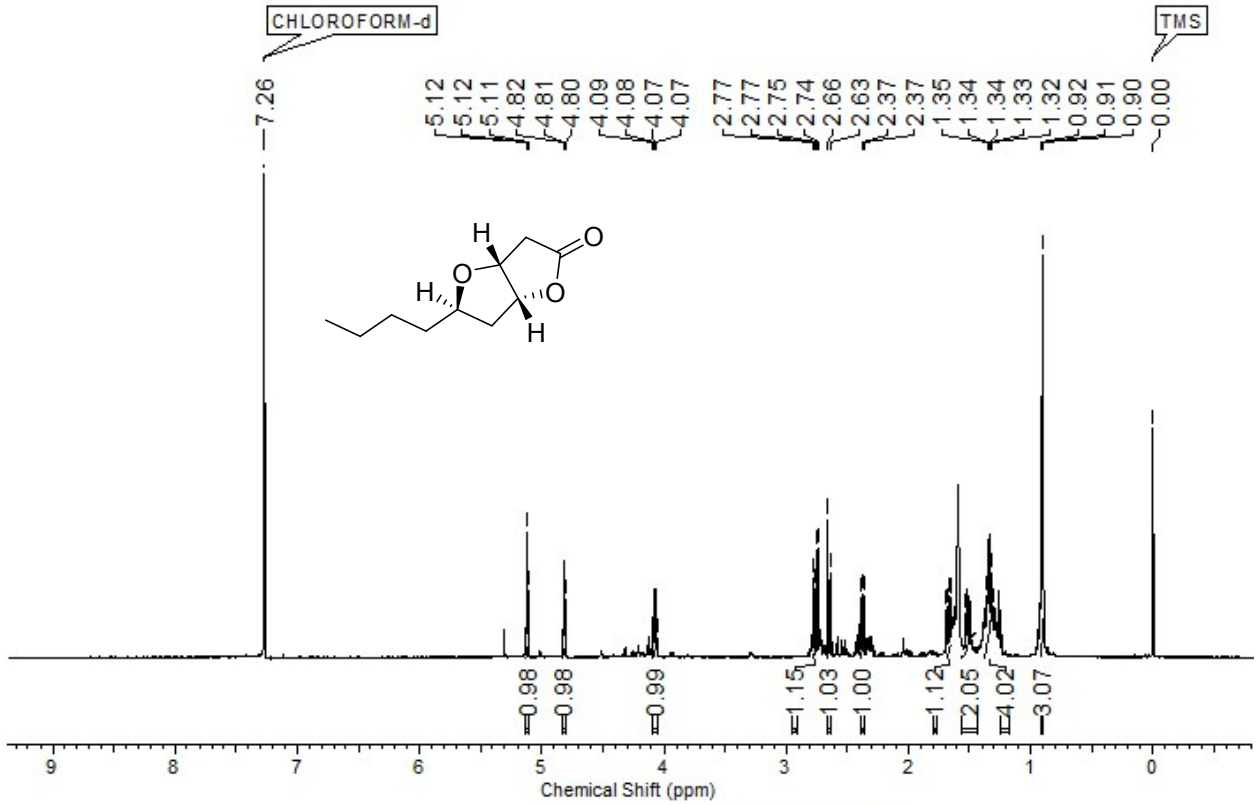


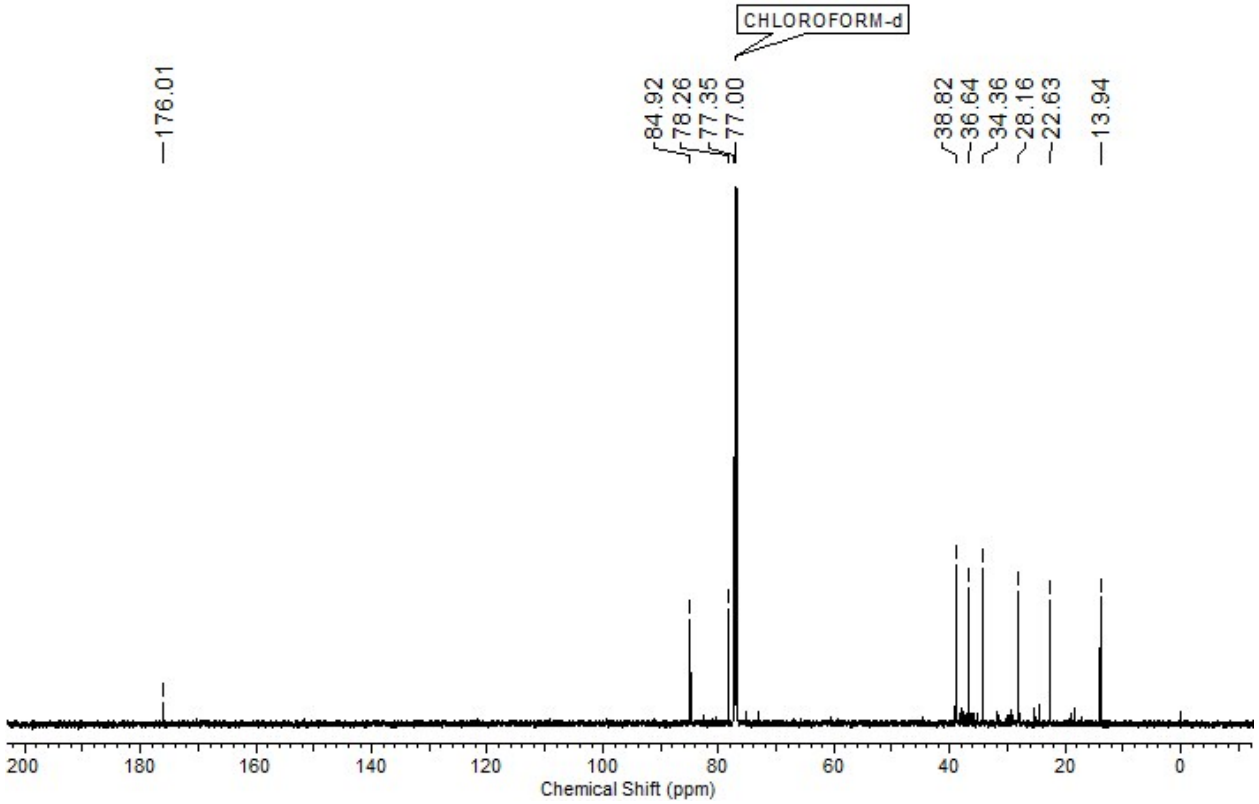
**Ethyl 2-((2*S*,3*S*,5*R*)-5-hexyl-3-hydroxytetrahydrofuran-2-yl)acetate: (10)**



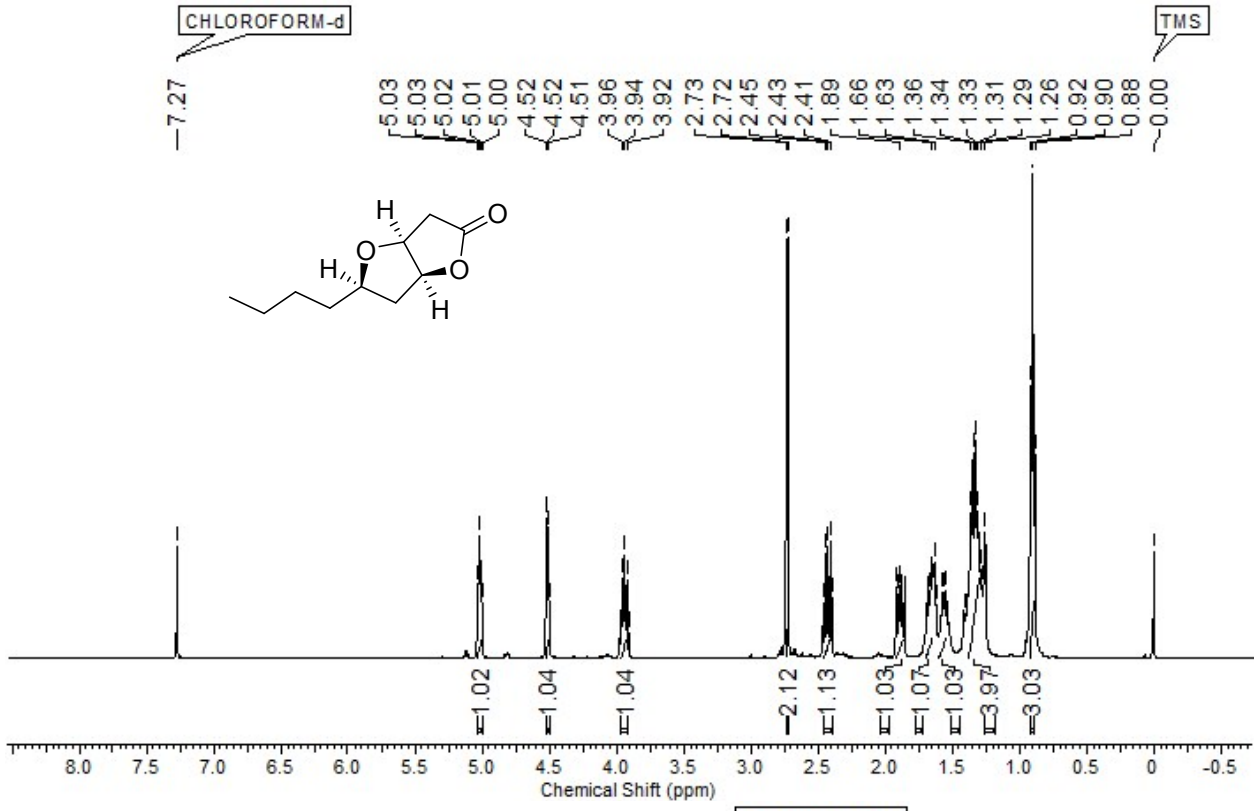



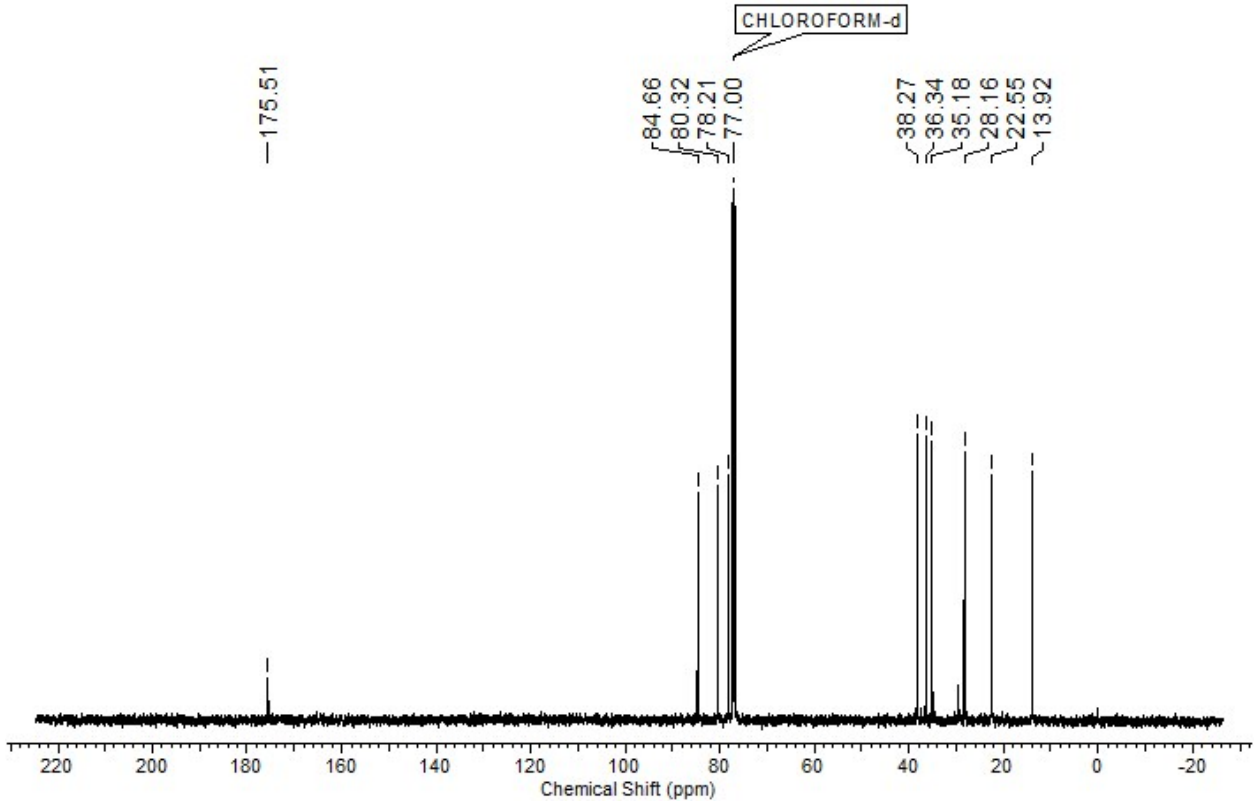
**(3a*R*,5*R*,6a*R*)-5-Butyltetrahydrofuro[3,2-b]furan-2(3H)-one: (2)**



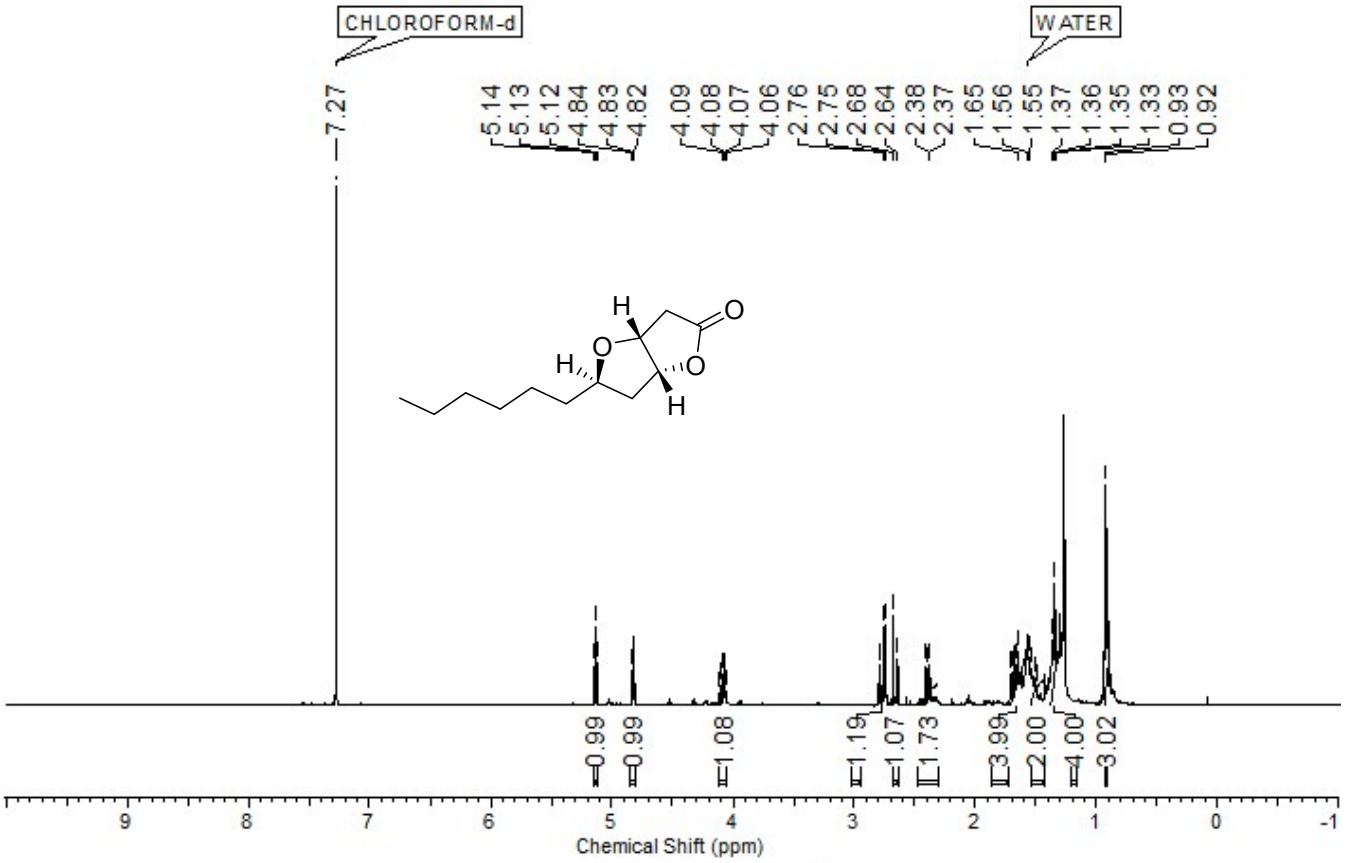



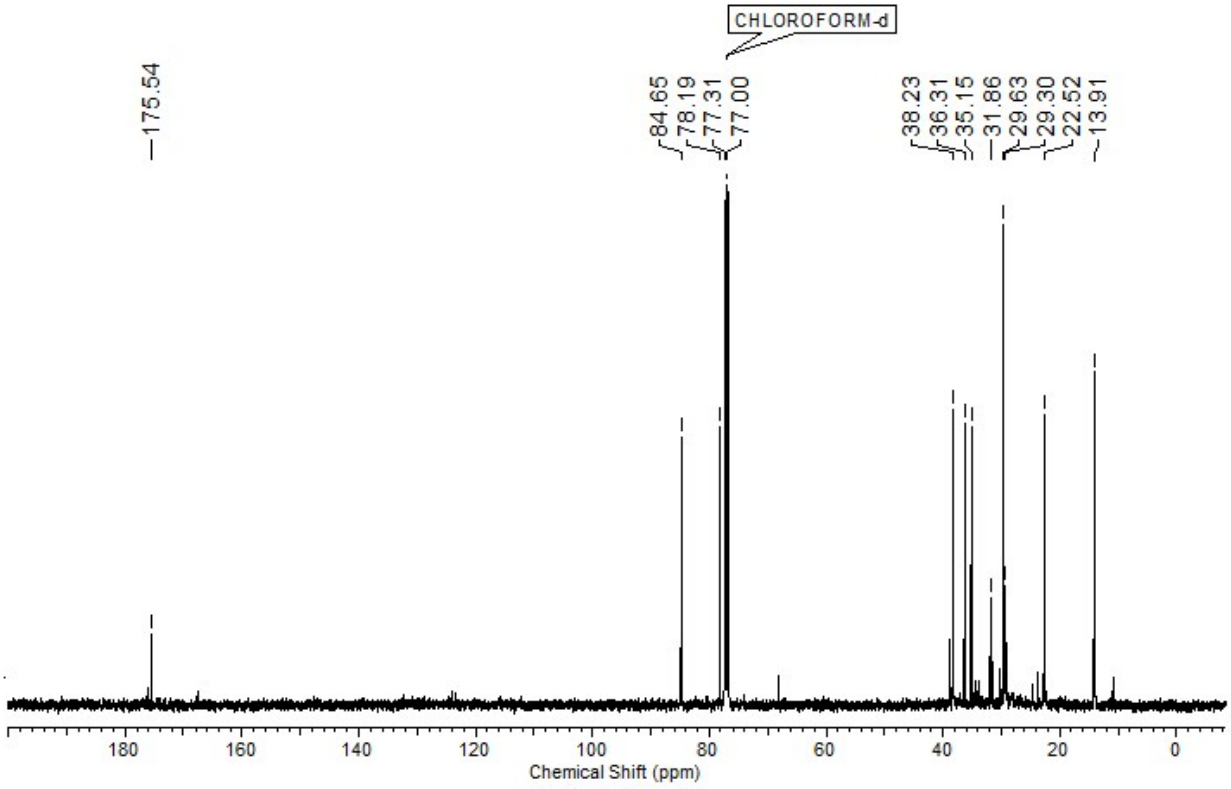
**Ethyl 2-((2*S*,3*S*,5*R*)-5-butyl-3-hydroxytetrahydrofuran-2-yl)acetate: (1)**



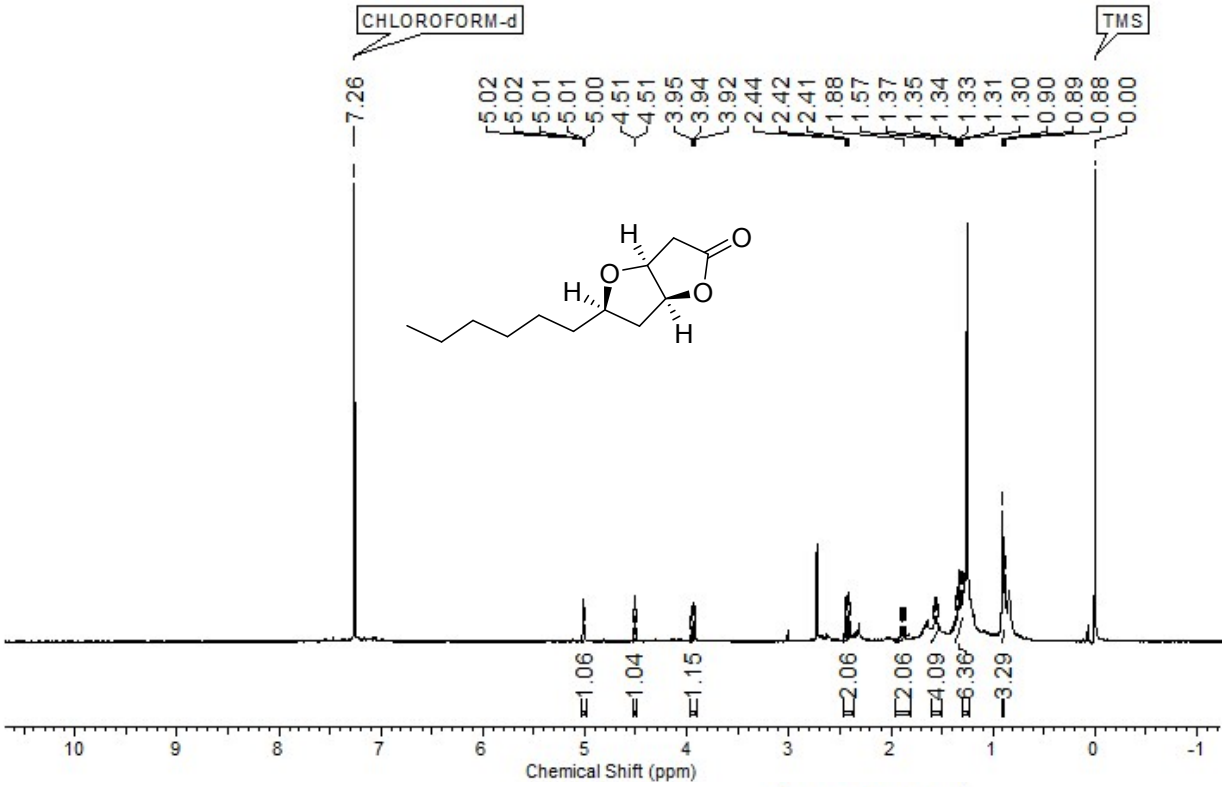



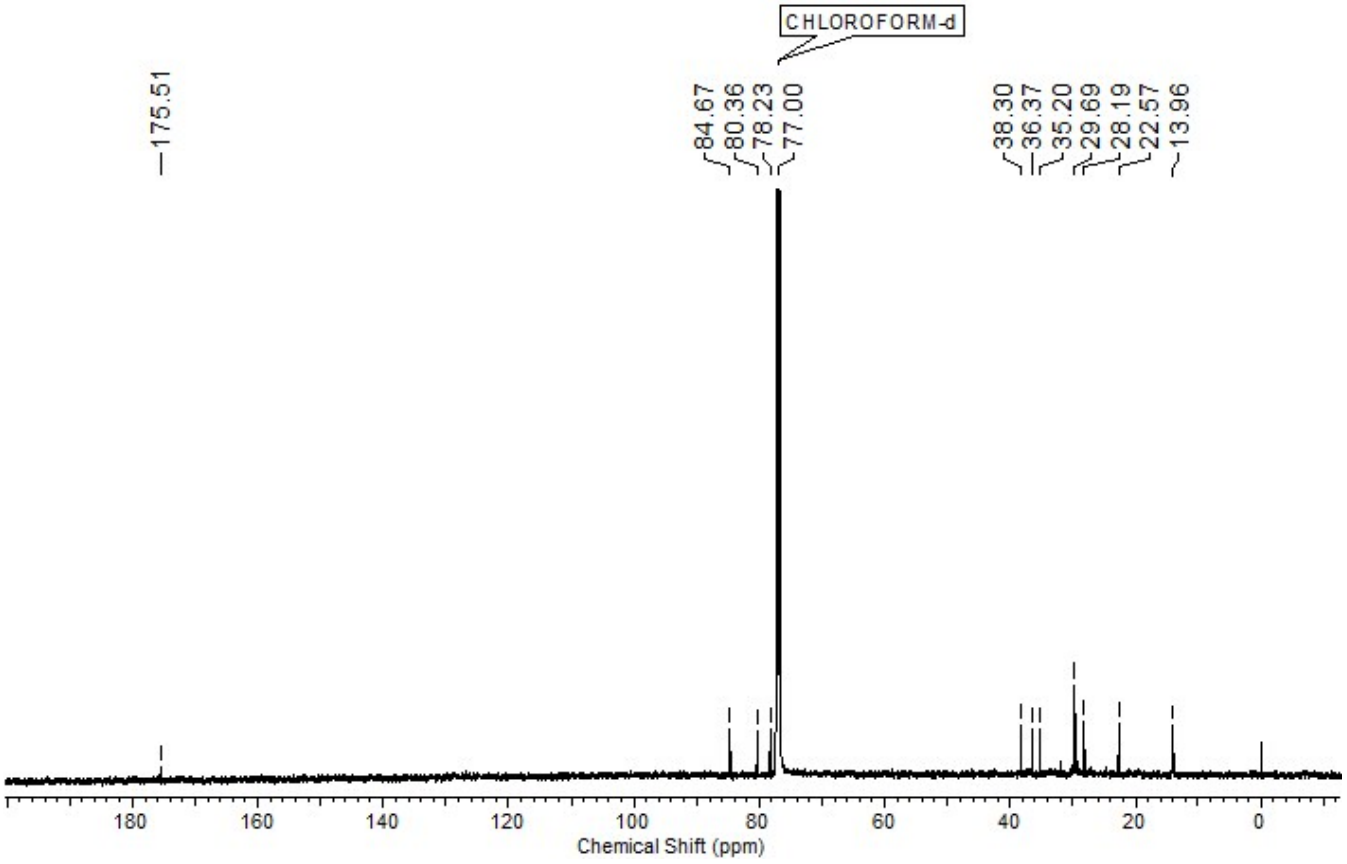
**(3a*R*,5*R*,6a*R*)-5-Hexyltetrahydrofuro[3,2-b]furan-2(3H) one:(4 )**



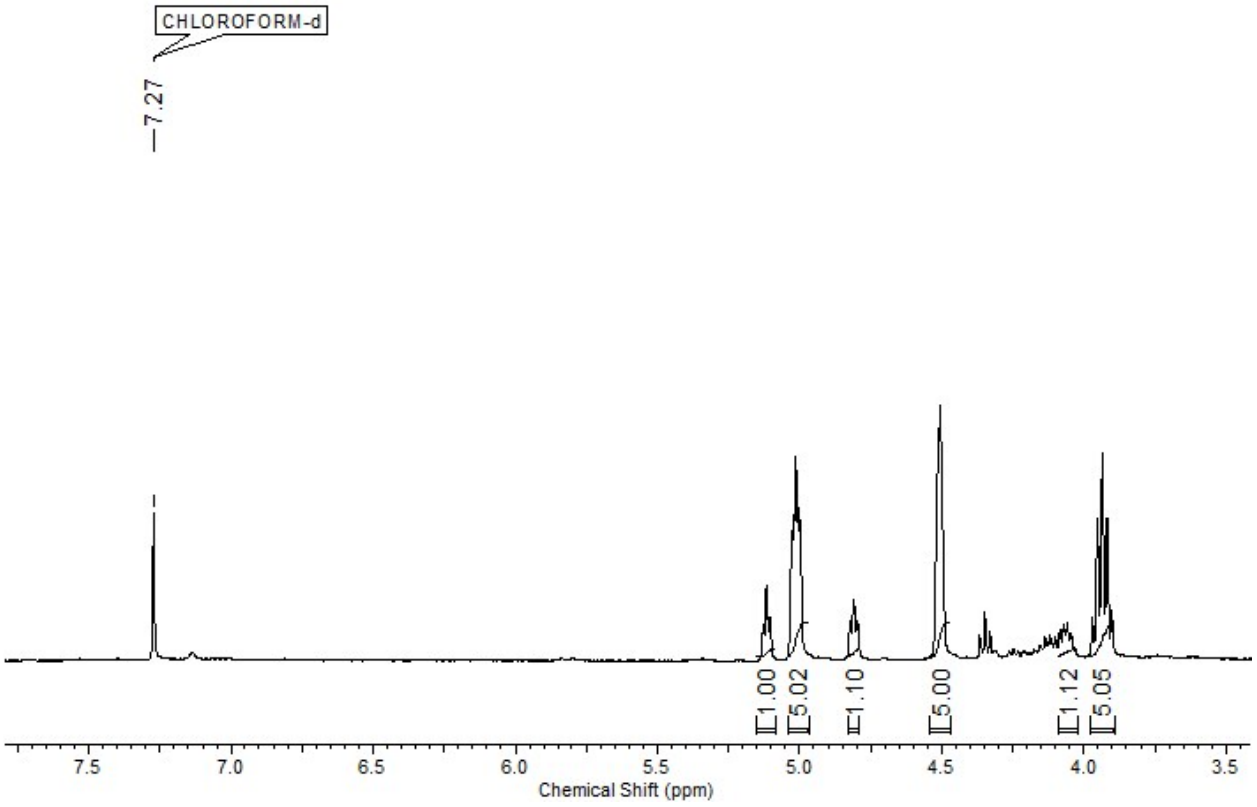



**(3a*S*,5*R*,6a*S*)-5-Hexyltetrahydrofuro[3,2-b]furan-2(3H)-one:(3)**

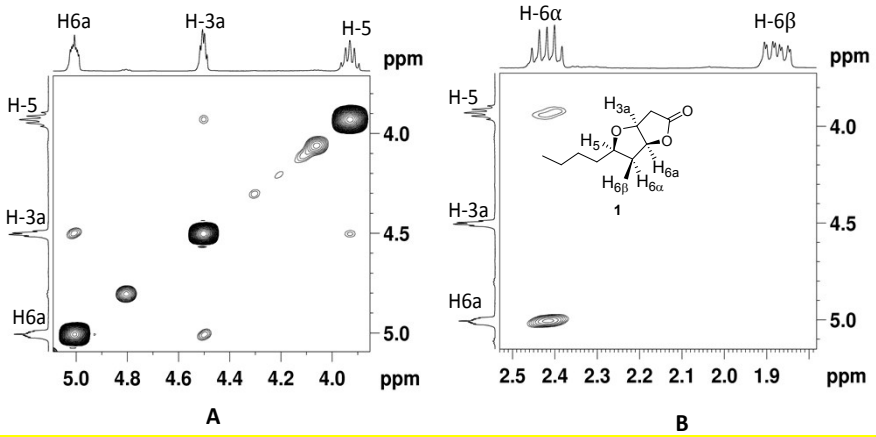


Expanded region of the spectra of crude compound **1 &2** containg mixture of diastereomers:

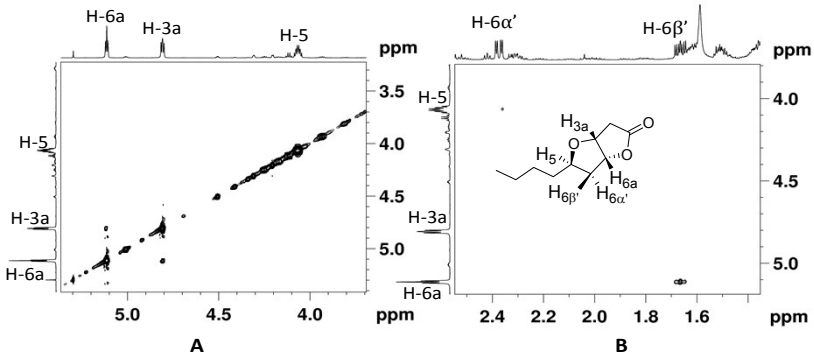


## $^1$H-$^1$H NOESY spectra of the compound 1 (400 MHz, $CDCl_3$, 298 $^o$K)



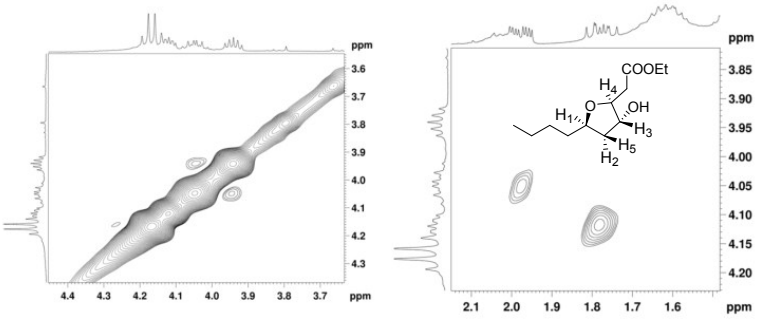

In NOESY analysis of the compound **1**, proton $H_{3a}$ shows nOe correlation with proton $H_{6a}$ indicating syn stereochemistry at the bridgehead of the substituted tetrahydrofuro[3,2-b]furan-2(3H)-one. $H_{3a}$ also shows nOe correlation with proton $H_5$, which confirms the *syn* relative stereochemistry between these three protons. Since the methylene protons $H_{6\alpha}$ and $H_{6\beta}$ resonated at different chemical shifts; the above results are also confirmed from the nOe of the $H_{6\alpha}$ and $H_{6\beta}$ with the $H_5$ and $H_{6\alpha}$ The $H_{6\alpha}$ resonating at $\delta$ 2.41 ppm showed nOe correlations with $H_5$ as well as with $H_{6\alpha}$ confirming the *syn* relative stereochemistry between these three protons. While the other methylene proton ($H_{6\beta}$) does not show nOe correlations either to $H_5$ or with $H_{6\alpha}$ confirming the *syn* relative stereochemistry between these three protons. While the other methylene proton ($H_{6\beta}$) does not show nOe correlations either to $H_5$ or ith $H_{6\alpha}$ indicating anti relative orientation with the $H_5$ and $H_{6\alpha}$.

## $^1$H-$^1$H NOESY spectra of the compound 2 (700 MHz, $CDCl_3$, 298 $^o$K)



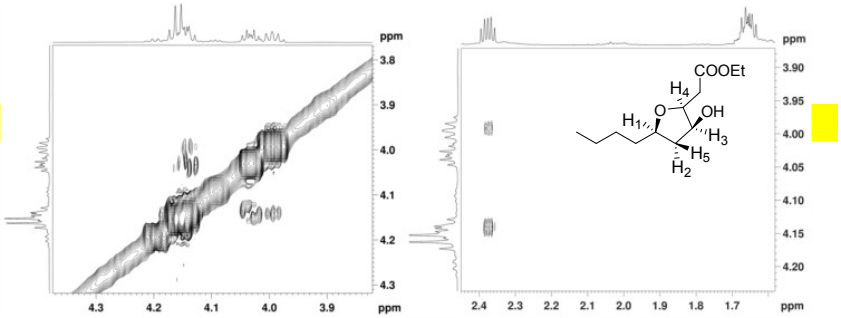

In NOESY analysis of compound **2**, the $H_5$ proton shows NOE correlation with $\delta$ 2.37 ppm ($H_{6\alpha'}$) while $H_{6a}$ shows NOE correlation with $\delta$ 1.66 ppm ($H_{6\beta'}$) indicating anti relative stereochemistry between $H_5$ and $H_{6a}$. In addition, $H_{6a}$ also shows nOe correlation with $H_{3a}$ indicating *syn* stereochemistry at the bridgehead of the substituted tetrahydrofuro[3,2-b]furan-2(3H)-one. The above results are also supported from the fact that none of the protons among $H_{3a}$ and $H_{6a}$ showed nOe correlation with $H_5$ confirming the anti-relative stereochemistry of $H_5$ with the $H_{6a}$ and $H_{3a}$ .

## $^1H$-$^1H$ NOESY spectra of the compound 9 (400 MHz, $CDCl_3$, 298 $^oK$)



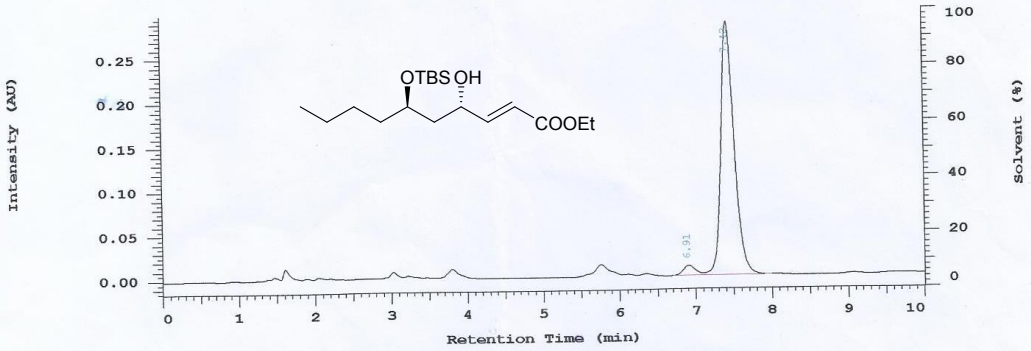

It was observed that in compound **9**, $H_4$ and $H_1$ shows nOe correlations indicating syn relative stereochemistry[14] while none of them shows nOe correlations with $H_3$ indicating anti relative stereochemistry with $H_3$ as can be seen from Figure 5. The relative stereochemistry among $H_1$ with $H_3$ was further confirmed by their nOe correlations with methylene protons ($H_2$& $H_5$). It was found that $H_1$ showed nOe correlations only with $H_2$ while $H_3$ showed nOe correlations only with $H_5$ indicating anti relative stereochemistry between $H_1$ and $H_3$.

## $^1H$-$^1H$ NOESY spectra of the compound 10 (700 MHz, $CDCl_3$, 298 $^oK$)




In NOESY analysis of compound **10**, $H_3$ proton shows nOe correlation with both methine protons $H_1$ and $H_4$ indicating syn stereochemistry among them. The relative stereochemistry was also confirmed with the help of methylene group which shows two different signals for two protons ($H_2$ and $H_5$). The $H_1$ and $H_3$ methine protons showed nOe correlations only with $H_2$ proton, but it does not show any correlation with $H_5$ proton indicating all the three methine protons ($H_1$, $H_3$ and $H_4$) being syn to each other.

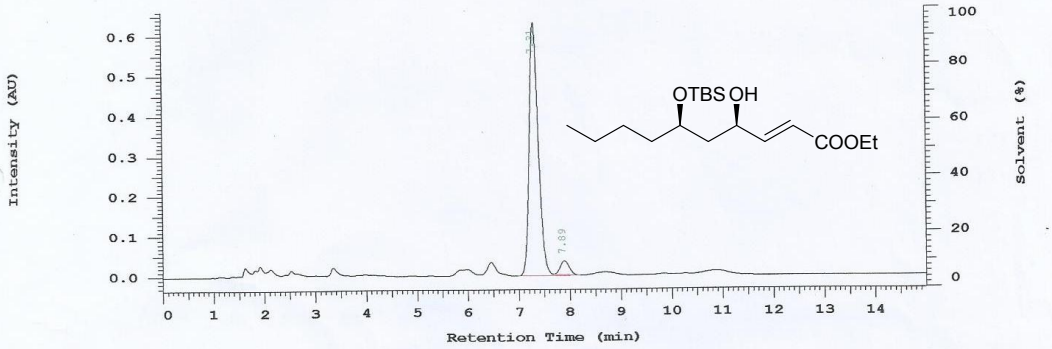
Chrom Type: HPLC Channel : 1




| No. | RT | Height | Area | Area % |
|---|---|---|---|---|
| 1 | 6.91 | 5404 | 61375 | 3.405 |
| 2 | 7.43 | 142688 | 1741276 | 96.595 |
| | | 148092 | 1802651 | 100.000 |

Peak rejection level: 0

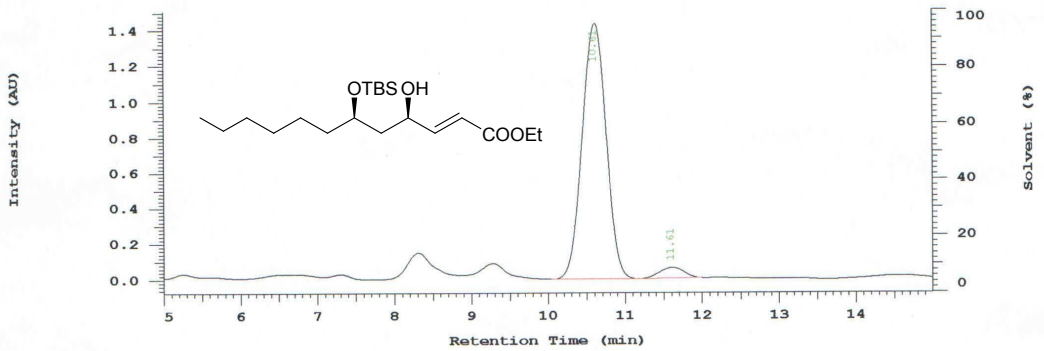
Chrom Type: HPLC Channel : 1




| No. | RT | Height | Area | Area % |
|---|---|---|---|---|
| 1 | 7.31 | 314879 | 3882718 | 94.984 |
| 2 | 7.89 | 17317 | 205022 | 5.016 |
| | | 332196 | 4087740 | 100.000 |

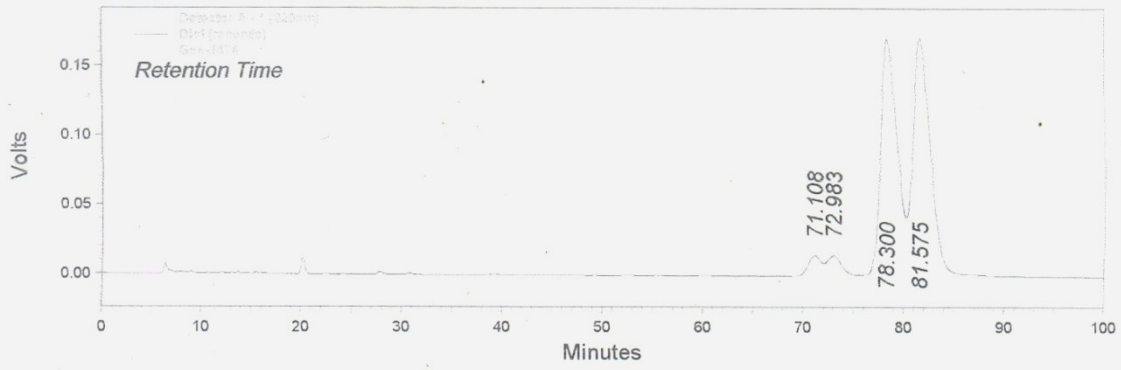
Peak rejection level: 0

Chrom Type: HPLC Channel : 1




| No. | RT | Height | Area | Area % |
|---|---|---|---|---|
| 1 | 10.61 | 715879 | 15494145 | 95.984 |
| 2 | 11.61 | 29299 | 648225 | 4.016 |
| | | 745178 | 16142370 | 100.000 |

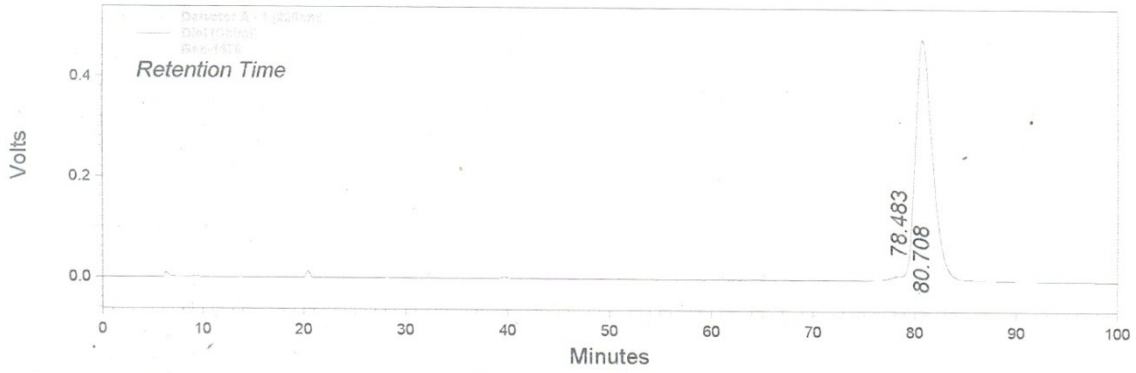
Peak rejection level: 0



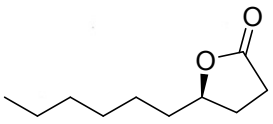

***Run Report***

Detector A - 1 (220nm)

| Retention Time | Area | Area Percent |
|---|---|---|
| 71.108 | 1256170 | 2.99 |
| 72.983 | 1371866 | 3.27 |
| 78.300 | 19033388 | 45.32 |
| 81.575 | 20338275 | 48.42 |
| Totals | 41999699 | 100.00 |

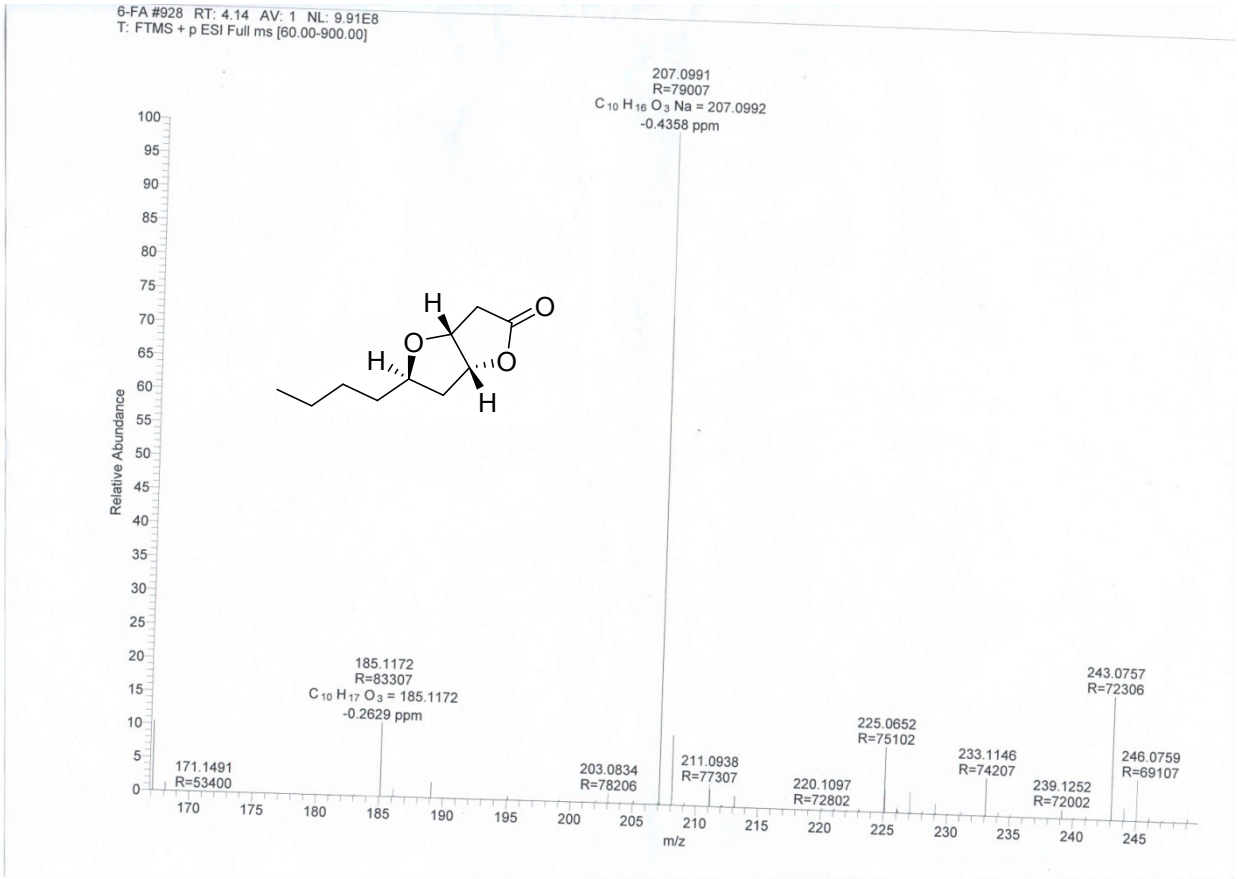


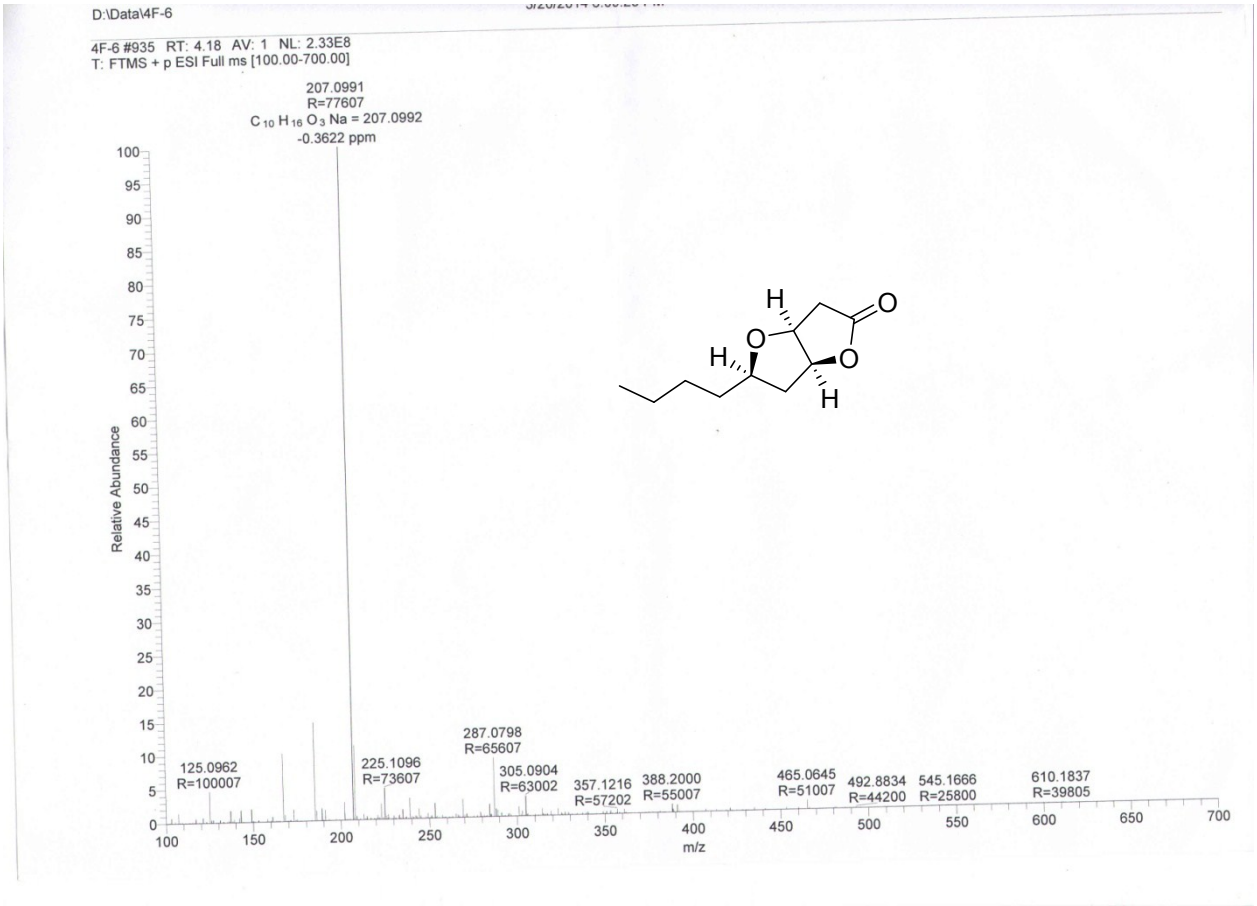

***Run Report***

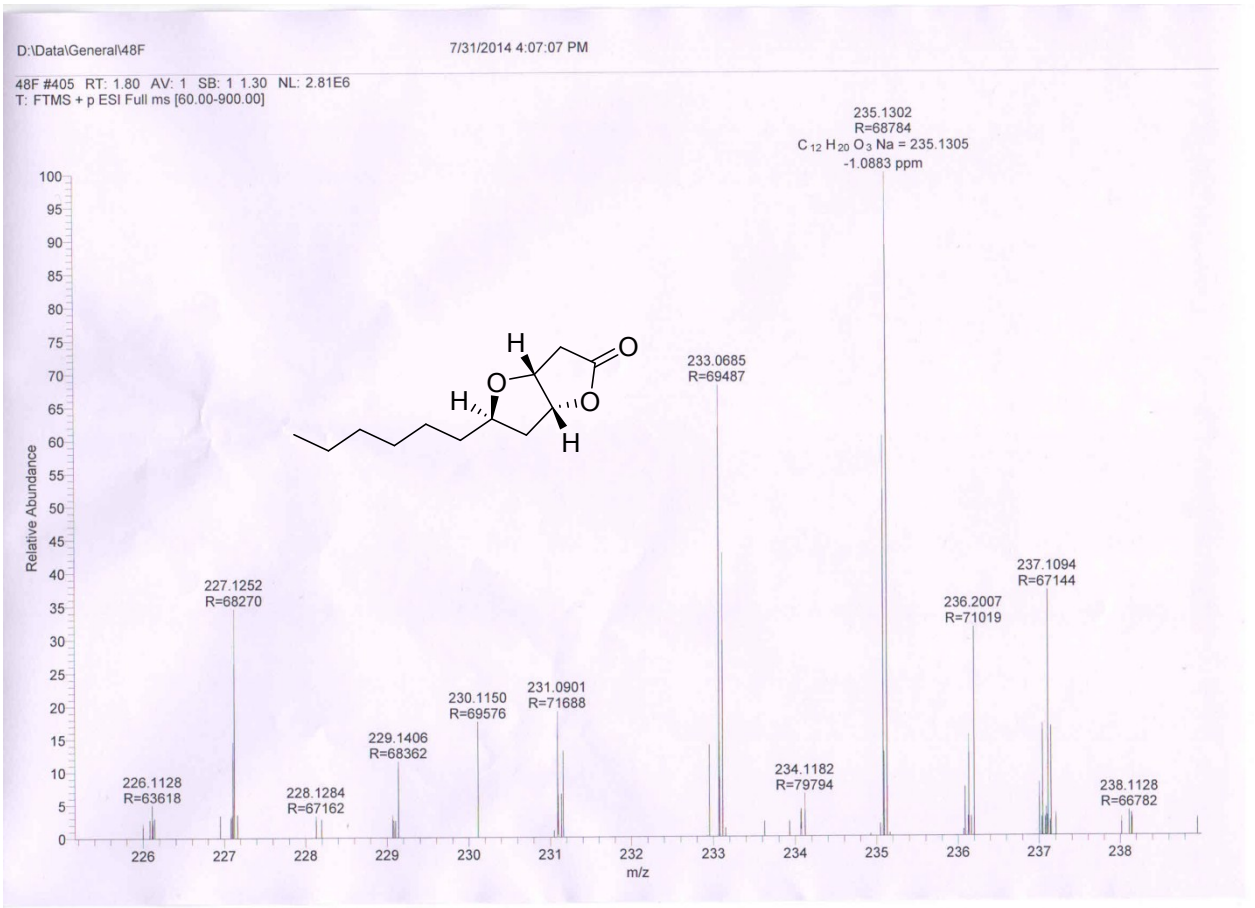


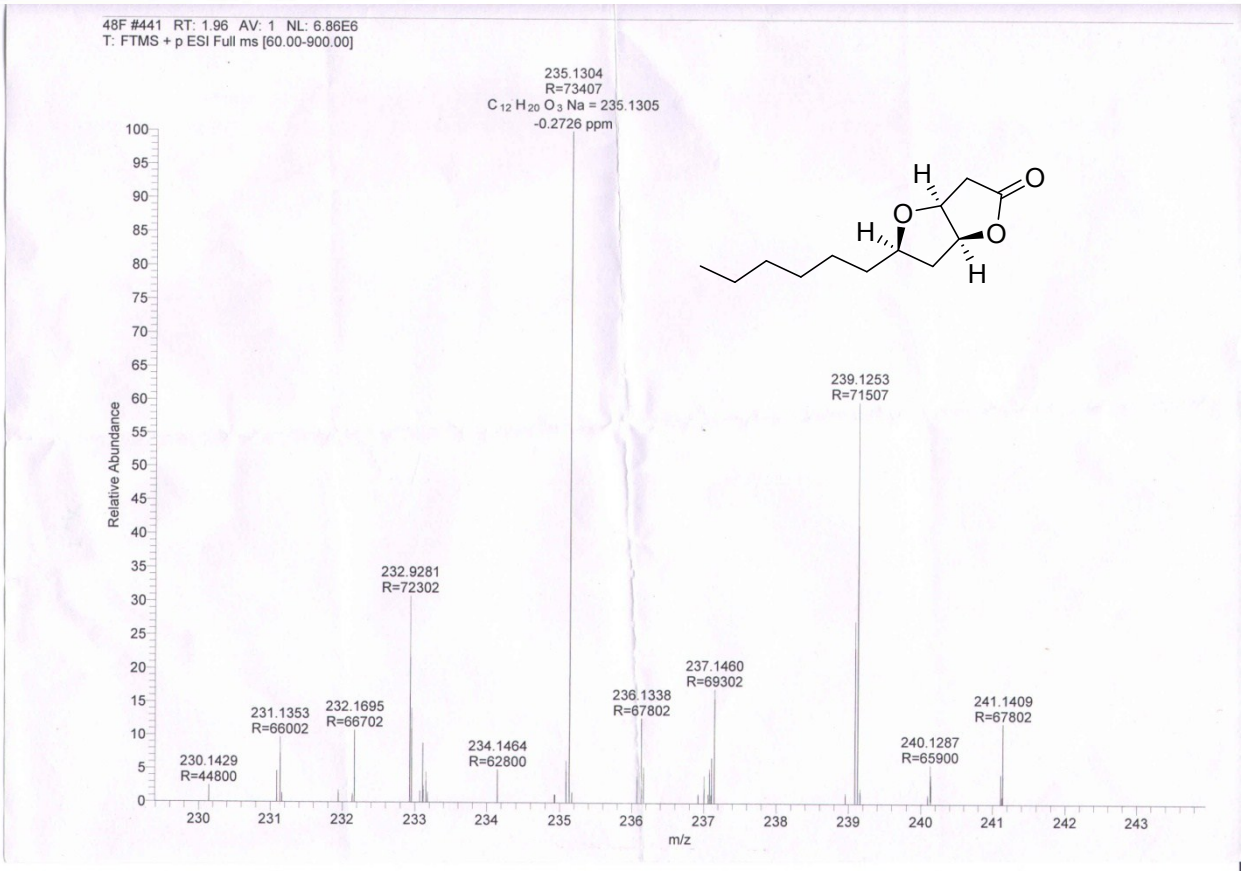

Detector A - 1 (220nm)

| Retention Time | Area | Area Percent |
|---|---|---|
| 78.483 | 492386 | 0.87 |
| 80.708 | 55985579 | 99.13 |
| Totals | 56477965 | 100.00 |


6-FA #928 RT: 4.14 AV: 1 NL: 9.91E8
T: FTMS + p ESI Full ms [60.00-900.00]
207.0991
R=79007
C 10 H 16 O 3 Na = 207.0992
-0.4358 ppm
185.1172
R=83307
C 10 H 17 O 3 = 185.1172
-0.2629 ppm
171.1491
R=53400
203.0834
R=78206
211.0938
R=77307
220.1097
R=72802
225.0652
R=75102
233.1146
R=74207
239.1252
R=72002
243.0757
R=72306
246.0759
R=69107
Relative Abundance
m/z
H
O
O
H
O
H


D:\Data\4F-6
4F-6 #935 RT: 4.18 AV: 1 NL: 2.33E8
T: FTMS + p ESI Full ms [100.00-700.00]
207.0991
R=77607
C 10 H 16 O 3 Na = 207.0992
-0.3622 ppm
Relative Abundance
m/z
125.0962
R=100007
225.1096
R=73607
287.0798
R=65607
305.0904
R=63002
357.1216
R=57202
388.2000
R=55007
465.0645
R=51007
492.8834
R=44200
545.1666
R=25800
610.1837
R=39805
H
O
H
O
O
H


D:\Data\General\48F
7/31/2014 4:07:07 PM
48F #405 RT: 1.80 AV: 1 SB: 1 1.30 NL: 2.81E6
T: FTMS + p ESI Full ms [60.00-900.00]
235.1302
R=68784
C 12 H 20 O 3 Na = 235.1305
-1.0883 ppm
233.0685
R=69487
227.1252
R=68270
237.1094
R=67144
236.2007
R=71019
231.0901
R=71688
230.1150
R=69576
229.1406
R=68362
234.1182
R=79794
226.1128
R=63618
228.1284
R=67162
238.1128
R=66782
Relative Abundance
m/z


48F #441 RT: 1.96 AV: 1 NL: 6.86E6
T: FTMS + p ESI Full ms [60.00-900.00]
235.1304
R=73407
C 12 H 20 O 3 Na = 235.1305
-0.2726 ppm
239.1253
R=71507
232.9281
R=72302
237.1460
R=69302
236.1338
R=67802
241.1409
R=67802
231.1353
R=66002
232.1695
R=66702
234.1464
R=62800
240.1287
R=65900
230.1429
R=44800
Relative Abundance
m/z