Electronic Supplementary Material (ESI) for RSC Advances

Supporting information:

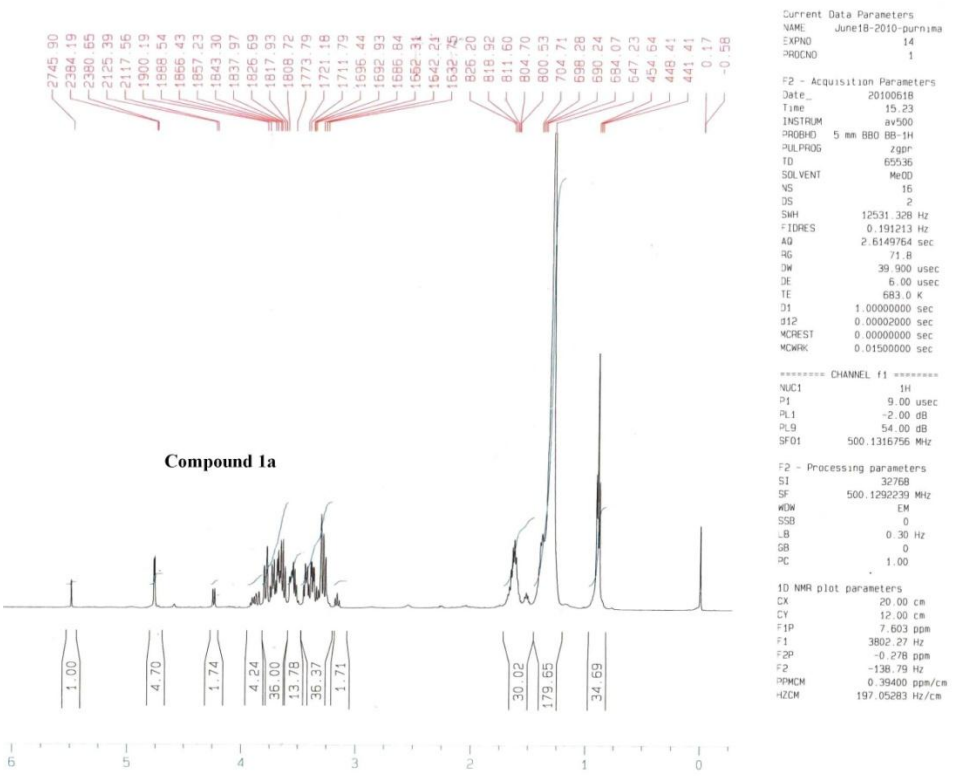
Ammonium chloride mediated synthesis of alkyl glycosides and evaluation of their immunomodulatory activity

Deepak K. Sharma,[1,2] Mallikharjuna R. Lambu,[1,2] Tabasum Sidiq,[1] Anamika Khajuria,[1]Anil K. Tripathi,[1] Syed Khalid Yousuf,[1] Debaraj Mukherjee*[1,2]

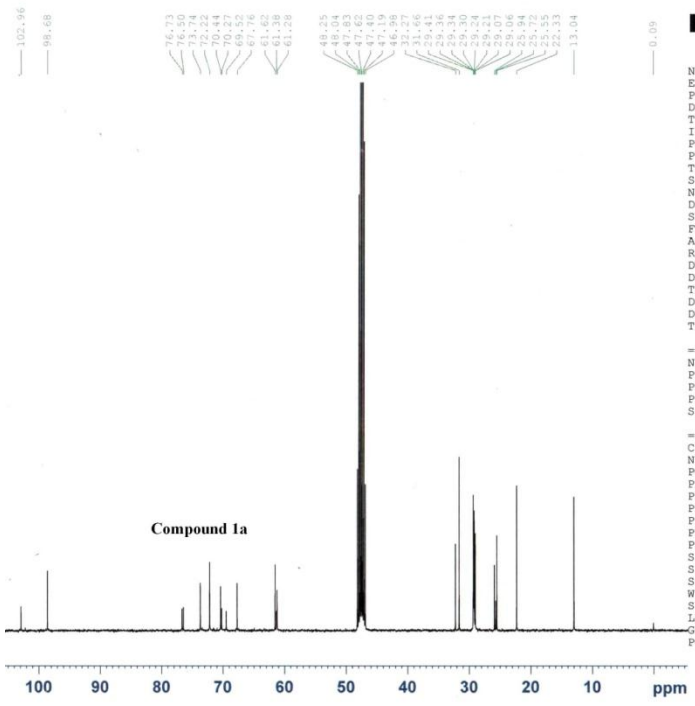
Fax: (+) 91-191-2569111-333; Tel: +91-191-2569000-006
E-mail: dmukherjee@iiim.ac.in

General information:

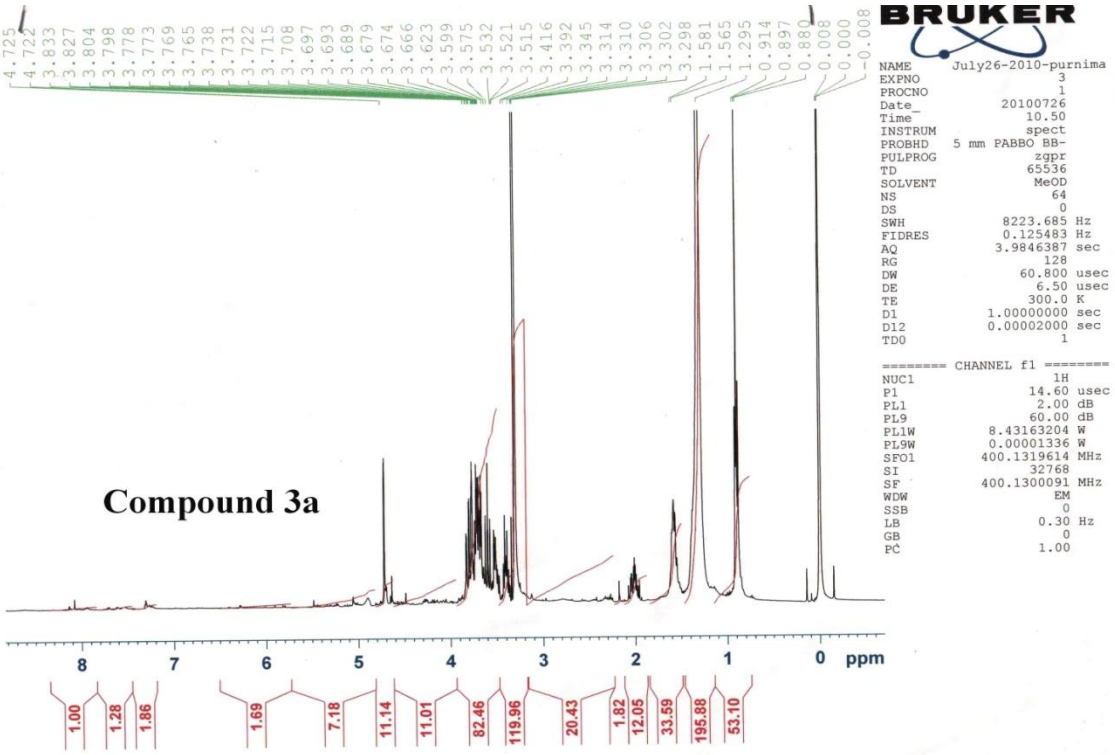
All chemicals were purchased from Sigma-Aldrich and SD Fine Chemicals, Pvt. Ltd. India, and used as received. Loba Chemie silica gel (100-200 mesh) was used for column chromatography, and thin-layer chromatography was performed on Merck-precoated silica gel 60-F254 plates. All other chemicals and solvents were obtained from commercial sources and purified using standard methods. The 1H and 13C NMR spectra were recorded on a Bruker-Avance 200,400 and 500 MHz spectrometer. Chemical shifts (δ) are reported in parts per million, using TMS (δ=0) as an internal standard in $CDCl_3$, MeOD and DMSO. Mass spectra were recorded on a Agilent-6224-TOF ESI/MS (ESI mass spectra). All products reported showed ^{1}H NMR and ^{13}C NMR spectra in agreement with the assigned structures.

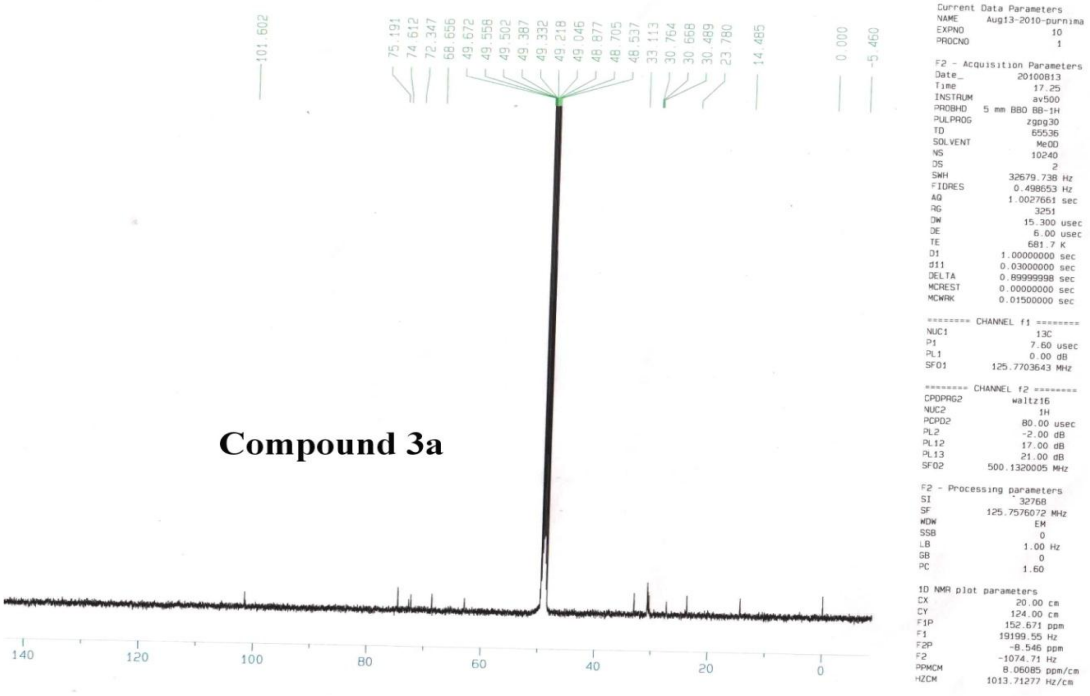


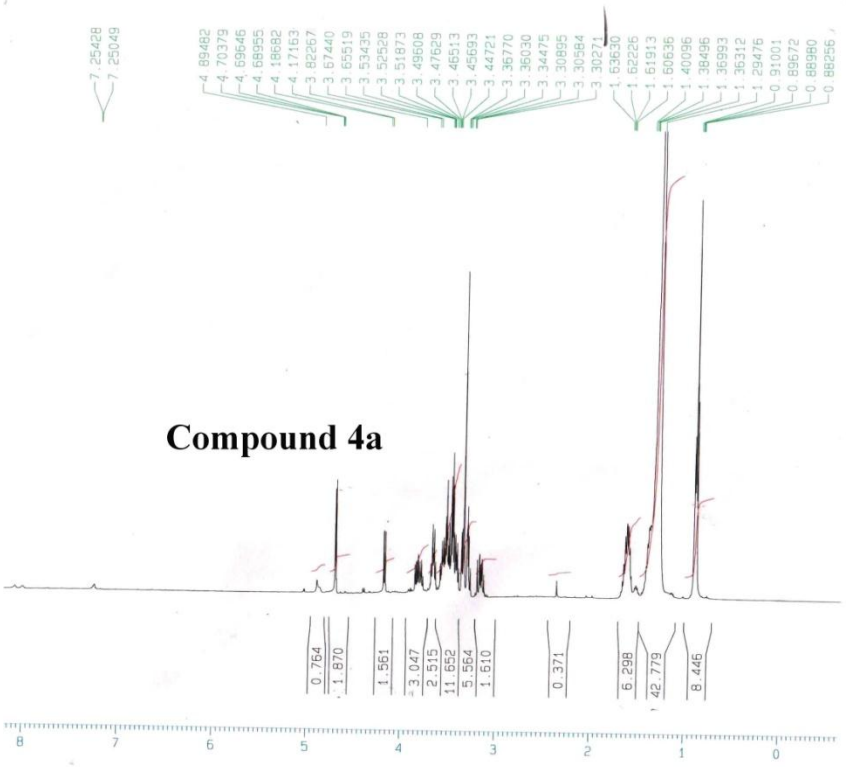

```
Current Data Parameters
NAME      June18-2010-purnima
EXPNO                      14
PROCNO                      1

F2 - Acquisition Parameters
Date_               20100618
Time                   15.23
INSTRUM                av500
PROBHD   5 mm BBO BB-1H
PULPROG                zgpr
TD                     65536
SOLVENT                MeOD
NS                        16
DS                         2
SWH               12531.328 Hz
FIDRES             0.191213 Hz
AQ                 2.6149764 sec
RG                      71.8
DW                   39.900 usec
DE                    6.00 usec
TE                   683.0 K
D1                1.00000000 sec
d12               0.00002000 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                       9.00 usec
PL1                     -2.00 dB
PL9                     54.00 dB
SFO1              500.1316756 MHz

F2 - Processing parameters
SI                        32768
SF                500.1292239 MHz
WDW                          EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.00

1D NMR plot parameters
CX                        20.00 cm
CY                        12.00 cm
F1P                        7.603 ppm
F1                      3802.27 Hz
F2P                       -0.278 ppm
F2                      -138.79 Hz
PPMCM                  0.39400 ppm/cm
HZCM                 197.05283 Hz/cm
```



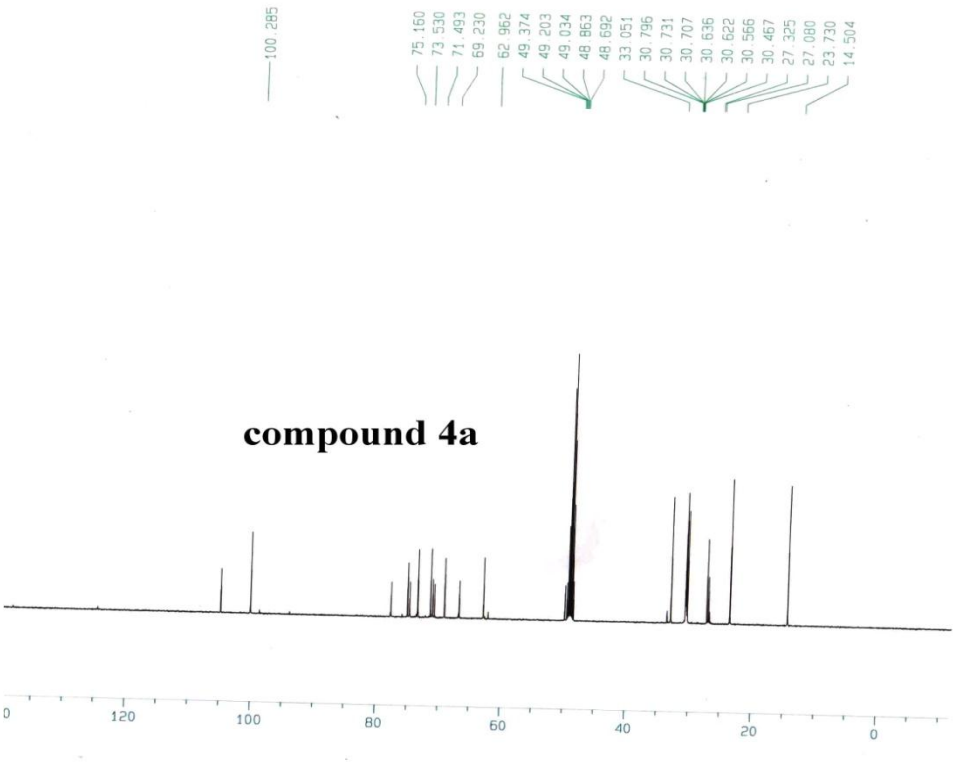

```
NAME      Aug14-2010-purnima
EXPNO                      1
PROCNO                     1
Date_               20100814
Time                   18.06
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG              zgpg30
TD                     65536
SOLVENT                MeOD
NS                     10240
DS                         4
SWH               25252.525 Hz
FIDRES             0.385323 Hz
AQ                 1.2976629 sec
RG                       128
DW                   19.800 usec
DE                    6.50 usec
TE                   300.0 K
D1                1.00000000 sec
D11               0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      13C
P1                       9.80 usec
PL1                     -1.30 dB
PL1W             49.47124481 W
SFO1             100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                       1H
PCPD2                 80.00 usec
PL2                    2.00 dB
PL12                  17.00 dB
PL13                  20.00 dB
PL2W              8.43163204 W
PL12W             0.26663160 W
PL13W             0.13363236 W
SFO2             400.1316005 MHz
SI                        32768
SF                100.6127688 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40
```








```
Current Data Parameters
NAME        Aug11-2010-purnima
EXPNO                        7
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20100811
Time                     12.22
INSTRUM                  av500
PROBHD   5 mm BBO BB-1H
PULPROG                  zgpr
TD                       65536
SOLVENT                  MeOD
NS                          16
DS                           2
SWH                  12531.328 Hz
FIDRES                0.191213 Hz
AQ                    2.6149764 sec
RG                        50.8
DW                      39.900 usec
DE                       6.00 usec
TE                      682.4 K
D1                    1.00000000 sec
d12                   0.00002000 sec
MCREST                0.00000000 sec
MCWRK                 0.01500000 sec

======== CHANNEL f1 ========
NUC1                         1H
P1                        9.00 usec
PL1                      -2.00 dB
PL9                      60.00 dB
SF01                500.1324765 MHz

F2 - Processing parameters
SI                          32768
SF                  500.1300132 MHz
WDW                            EM
SSB                             0
LB                       0.30 Hz
GB                              0
PC                           1.00

1D NMR plot parameters
CX                       20.00 cm
CY                       16.00 cm
F1P                      8.742 ppm
F1                     4372.19 Hz
F2P                     -0.646 ppm
F2                     -323.21 Hz
PPMCM                 0.46942 ppm/cm
HZCM                234.76996 Hz/cm
```




```
Current Data Parameters
NAME        Aug09-2009-purnima
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20100809
Time                     11.59
INSTRUM                  av500
PROBHD   5 mm BBO BB-1H
PULPROG                zgpg30
TD                       65536
SOLVENT                  MeOD
NS                         2971
DS                           2
SWH                  31446.541 Hz
FIDRES                0.479836 Hz
AQ                    1.0420883 sec
RG                        3251
DW                      15.900 usec
DE                       6.00 usec
TE                      683.0 K
D1                    0.50000000 sec
d11                   0.03000000 sec
DELTA                 0.40000001 sec
MCREST                0.00000000 sec
MCWRK                 0.01500000 sec

======== CHANNEL f1 ========
NUC1                        13C
P1                        7.60 usec
PL1                      0.00 dB
SF01                125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                         1H
PCPD2                    80.00 usec
PL2                      -2.00 dB
PL12                     17.00 dB
PL13                     21.00 dB
SF02                500.1320005 MHz

F2 - Processing parameters
SI                          32768
SF                  125.7576212 MHz
WDW                            EM
SSB                             0
LB                       1.00 Hz
GB                              0
PC                           1.00

1D NMR plot parameters
CX                       20.00 cm
CY                        6.00 cm
F1P                     149.253 ppm
F1                    18769.74 Hz
F2P                    -11.717 ppm
F2                    -1473.49 Hz
PPMCM                 8.04851 ppm/cm
HZCM               1012.16138 Hz/cm
```

Compound 4a

Qualitative Compound Report

Data File	XD.d	**Sample Name**	XD
Sample Type	Sample	**Position**	Vial 18
Instrument Name	Instrument 1	**User Name**	
Acq Method	vishal_12-01-13.m	**Acquired Time**	28-03-2013 PM 12:11:08
IRM Calibration Status	Success	**DA Method**	SamplePurity-Default.m
Comment			

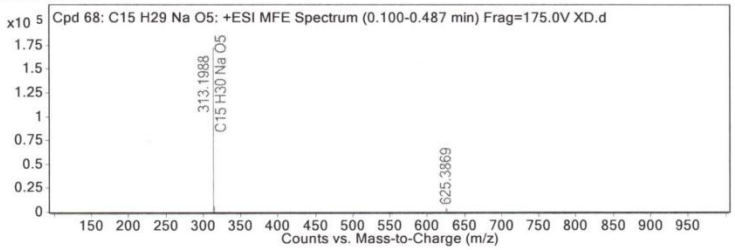
Sample Group	**Info.**
Acquisition SW Version	6200 series TOF/6500 series Q-TOF B.05.01 (B5125)

Compound Table

Compound Label	RT	Mass	Formula	MFG Formula	MFG Diff (ppm)	DB Formula
Cpd 68: C15 H29 Na O5	0.192	312.1914	C15 H29 Na O5	C15 H29 Na O5	-0.51	C15 H29 Na O5

Compound Label	*m/z*	RT	Algorithm	Mass
Cpd 68: C15 H29 Na O5	313.1988	0.192	Find by Molecular Feature	312.1914

MFE MS Spectrum



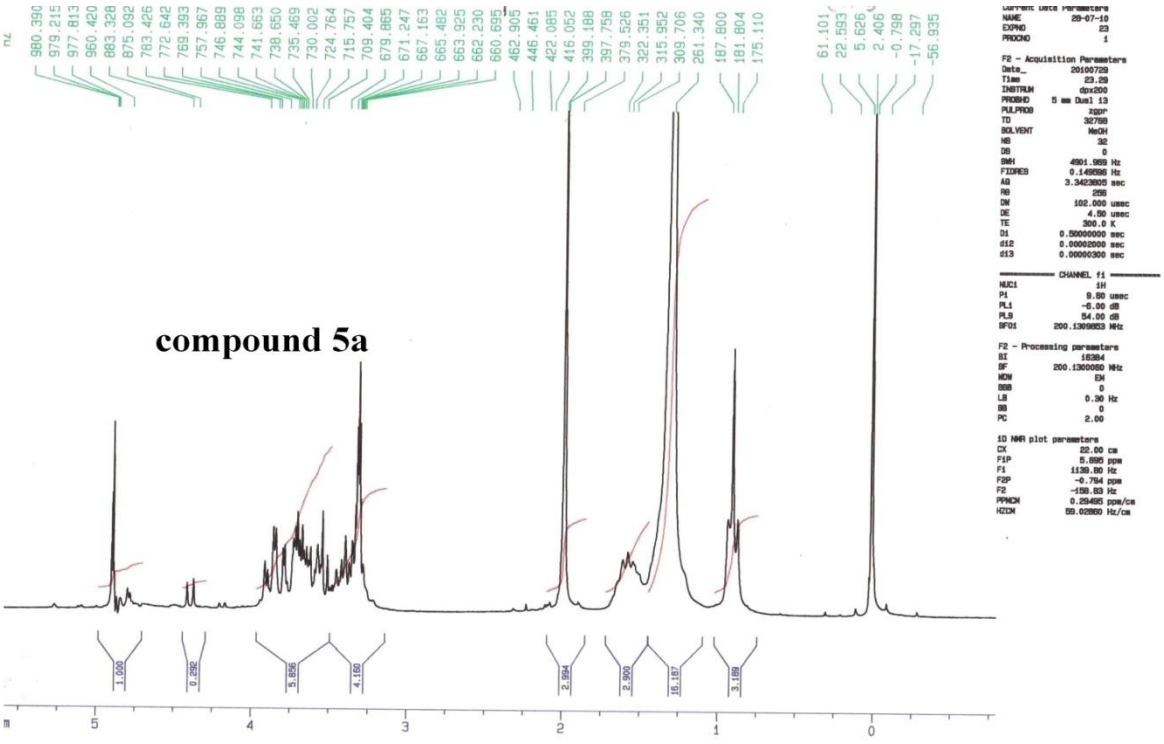
MS Spectrum Peak List

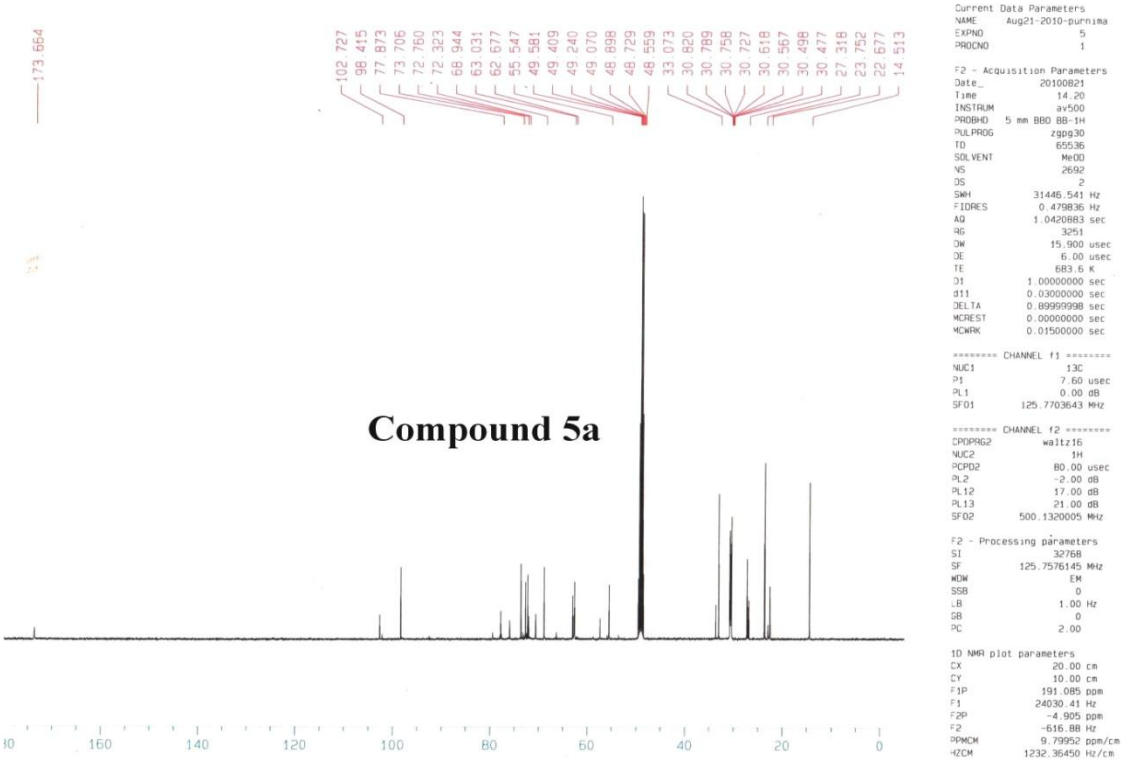
m/z	z	Abund	Formula	Ion
313.1988	1	172743.52	C15 H30 Na O5	(M+H)+
314.202	1	29838.8	C15 H30 Na O5	(M+H)+
315.2028	1	5677.93	C15 H30 Na O5	(M+H)+
625.3869	1	3661.21		(2M+H)+
626.3885	1	2633.21		(2M+H)+

Predicted Isotope Match Table

Isotope	*m/z*	Calc m/z	Diff (ppm)	Abund %	Calc Abund %	Abund Sum %	Calc Abund Sum %
1	313.1988	313.1985	-0.74	100	100	82.95	83.96
2	314.202	314.202	-0.16	17.27	16.76	14.33	14.07
3	315.2028	315.2043	4.67	3.29	2.34	2.73	1.97

--- End Of Report ---


compound 5a


Compound 5a

Compound 5a

Qualitative Compound Report

Data File	NAGRd	**Sample Name**	NAGRd
Sample Type	Sample	**Position**	Vial 20
Instrument Name	Instrument 1	**User Name**	
Acq Method	vishal_12-01-13.m	**Acquired Time**	28-03-2013 PM 12:20:13
IRM Calibration Status	Success	**DA Method**	SamplePurity-Default.m
Comment			

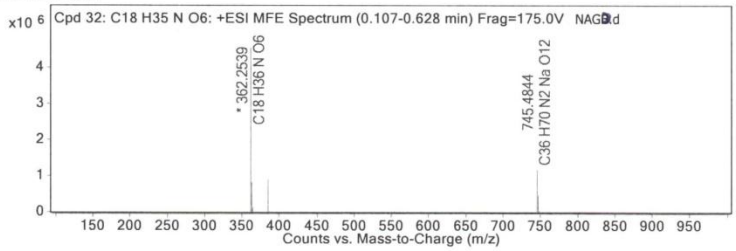
Sample Group	**Info.**
Acquisition SW Version	6200 series TOF/6500 series Q-TOF B.05.01 (B5125)

Compound Table

Compound Label	RT	Mass	Formula	MFG Formula	MFG Diff (ppm)	DB Formula
Cpd 32: C18 H35 N O6	0.188	361.2467	C18 H35 N O6	C18 H35 N O6	-0.74	C18 H35 N O6

Compound Label	*m/z*	RT	Algorithm	Mass
Cpd 32: C18 H35 N O6	362.2539	0.188	Find by Molecular Feature	361.2467

MFE MS Spectrum



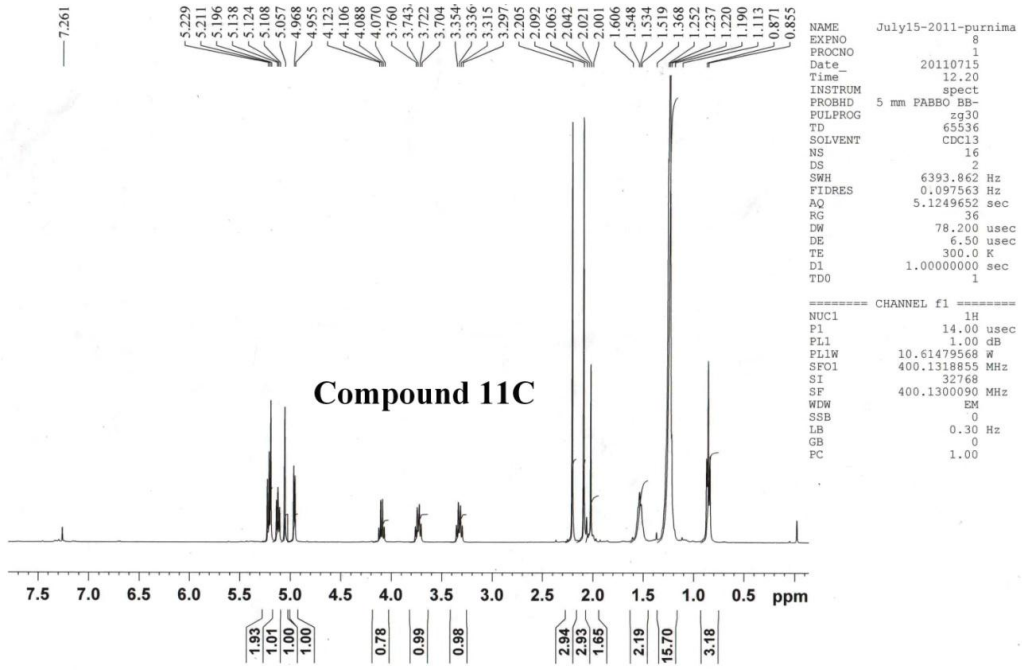
MS Spectrum Peak List

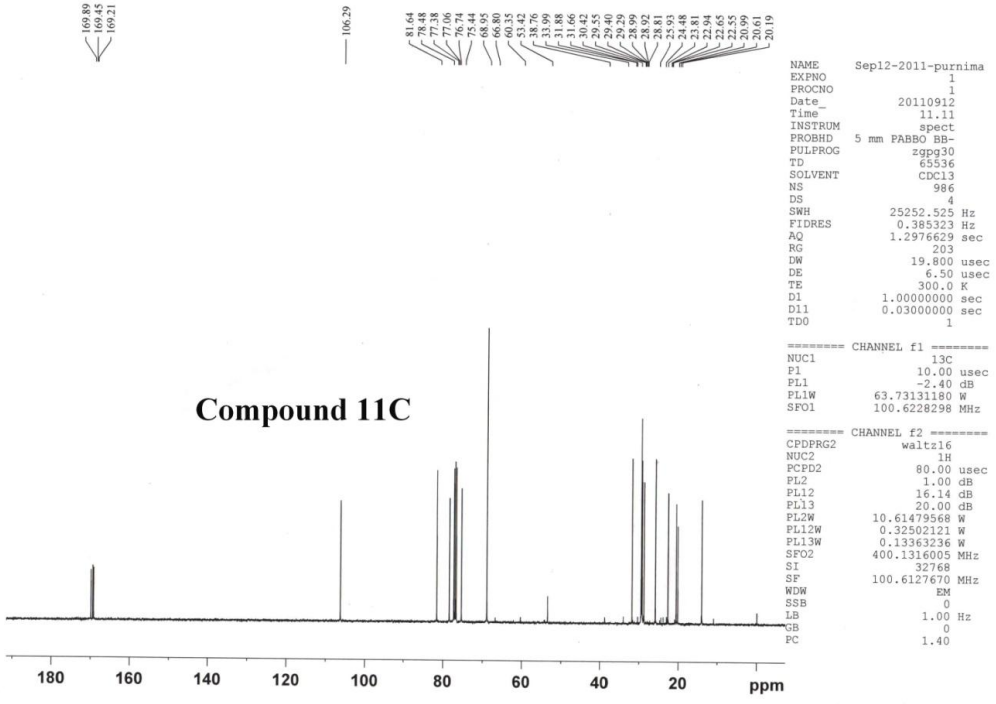
m/z	z	Abund	Formula	Ion
362.2539	1	4545140	C18 H36 N O6	(M+H)+
363.2577	1	816949.25	C18 H36 N O6	(M+H)+
364.2598	1	125287.3	C18 H36 N O6	(M+H)+
384.2363	1	907237.38	C18 H35 N Na O6	(M+Na)+
385.2392	1	172591.59	C18 H35 N Na O6	(M+Na)+
386.2417	1	25839.71	C18 H35 N Na O6	(M+Na)+
745.4844	1	1183643.88	C36 H70 N2 Na O12	(2M+Na)+
746.4876	1	465858.16	C36 H70 N2 Na O12	(2M+Na)+
747.4891	1	111986.63	C36 H70 N2 Na O12	(2M+Na)+
748.491	1	21673.11	C36 H70 N2 Na O12	(2M+Na)+

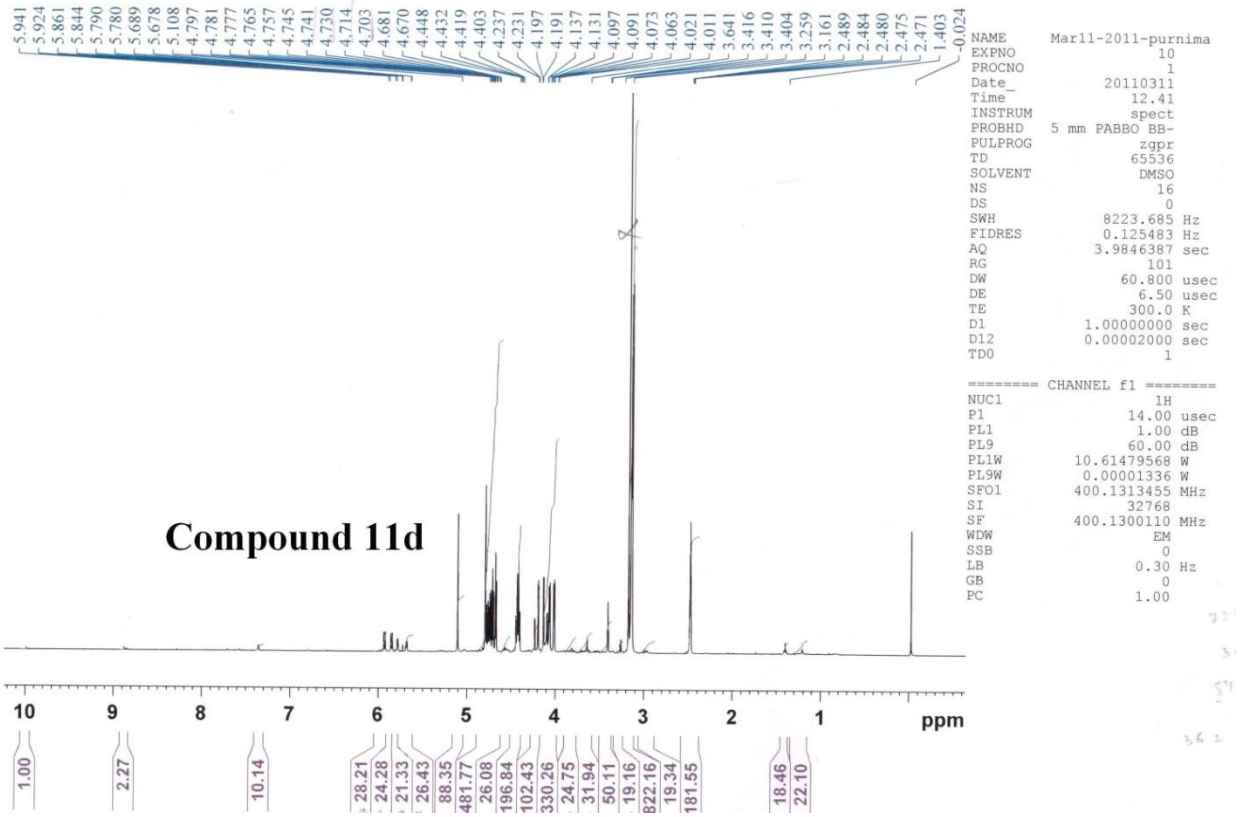
Predicted Isotope Match Table

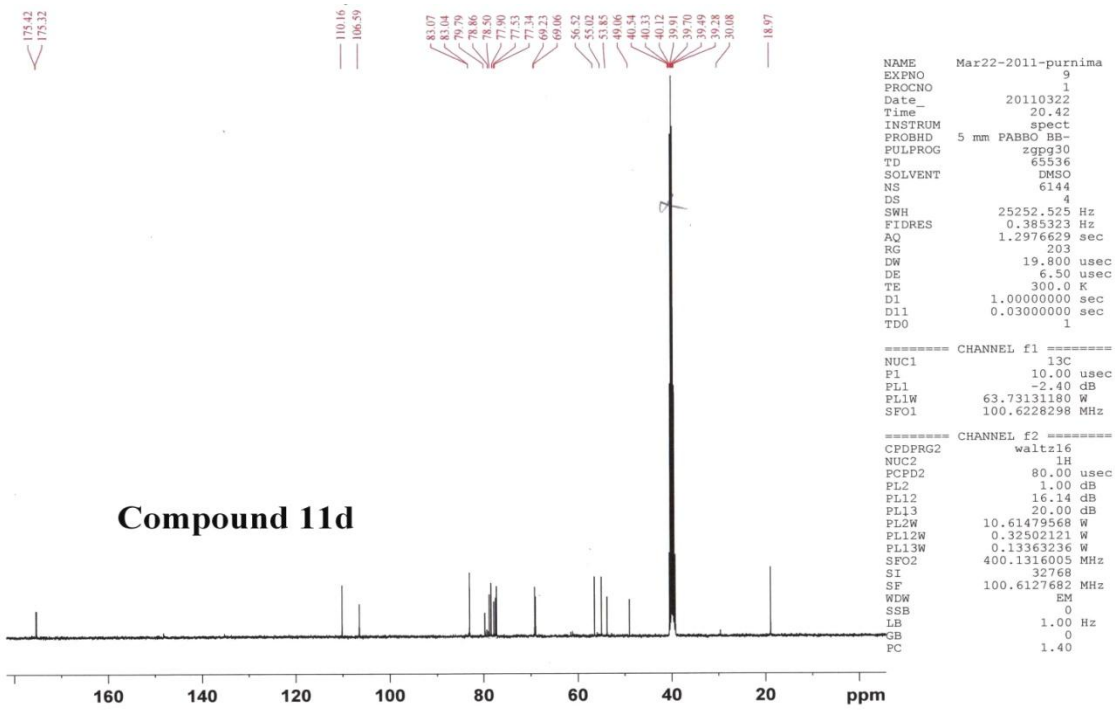
Isotope	*m/z*	Calc m/z	Diff (ppm)	Abund %	Calc Abund %	Abund Sum %	Calc Abund Sum %
1	362.2539	362.2537	-0.54	100	100	82.58	80.57
2	363.2577	363.257	-1.79	17.97	20.48	14.84	16.5
3	364.2598	364.2594	-0.99	2.76	3.22	2.28	2.6
4	365.2625	365.262	-1.18	0.33	0.37	0.28	0.3
5	366.2645	366.2645	-0.07	0.03	0.04	0.02	0.03

--- End Of Report ---


Compound 11C
7.261
5.229
5.211
5.196
5.138
5.124
5.108
5.057
4.968
4.955
4.123
4.106
4.088
4.070
3.760
3.743
3.722
3.704
3.354
3.336
3.315
3.297
2.205
2.092
2.063
2.042
2.021
2.001
1.606
1.548
1.534
1.519
1.368
1.252
1.237
1.220
1.190
1.113
0.871
0.855
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.93
1.01
1.00
1.00
0.78
0.99
0.98
2.94
2.93
1.65
2.19
15.70
3.18
NAME July15-2011-purnima
EXPNO 8
PROCNO 1
Date_ 20110715
Time_ 12.20
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6393.862 Hz
FIDRES 0.097563 Hz
AQ 5.1249652 sec
RG 36
DW 78.200 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 1.00 dB
PL1W 10.61479568 W
SFO1 400.1318855 MHz
SI 32768
SF 400.1300090 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

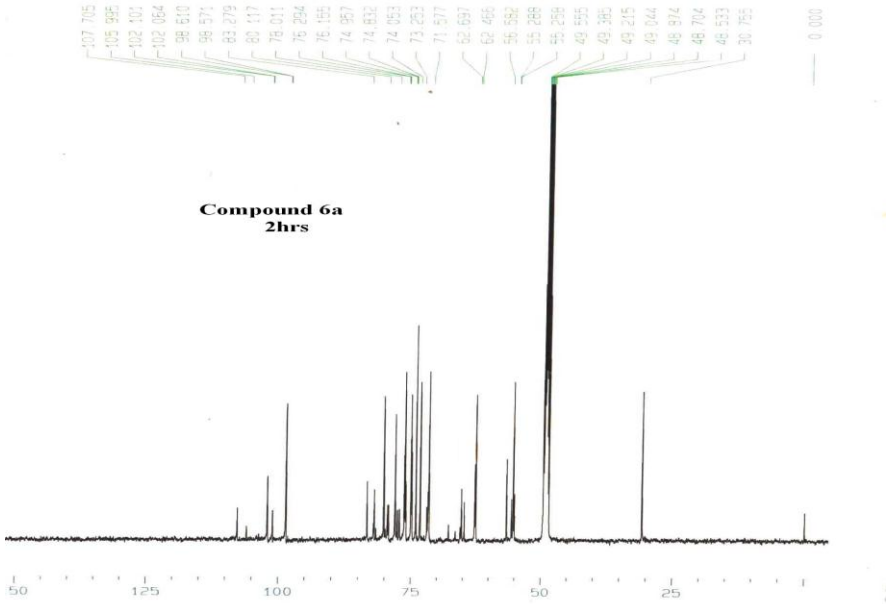

Compound 11C
169.89
169.45
169.21
106.29
81.64
78.48
77.38
77.06
76.74
75.44
68.95
66.80
60.35
53.42
38.76
33.99
31.88
31.66
30.42
29.55
29.40
29.29
28.99
28.92
28.81
25.93
24.48
23.81
22.94
22.65
22.55
20.99
20.61
20.19
180 160 140 120 100 80 60 40 20 ppm
NAME Sep12-2011-purnima
EXPNO 1
PROCNO 1
Date_ 20110912
Time_ 11.11
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 986
DS 4
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 203
DW 19.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 -2.40 dB
PL1W 63.73131180 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 16.14 dB
PL13 20.00 dB
PL2W 10.61479568 W
PL12W 0.32502121 W
PL13W 0.13363236 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127670 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



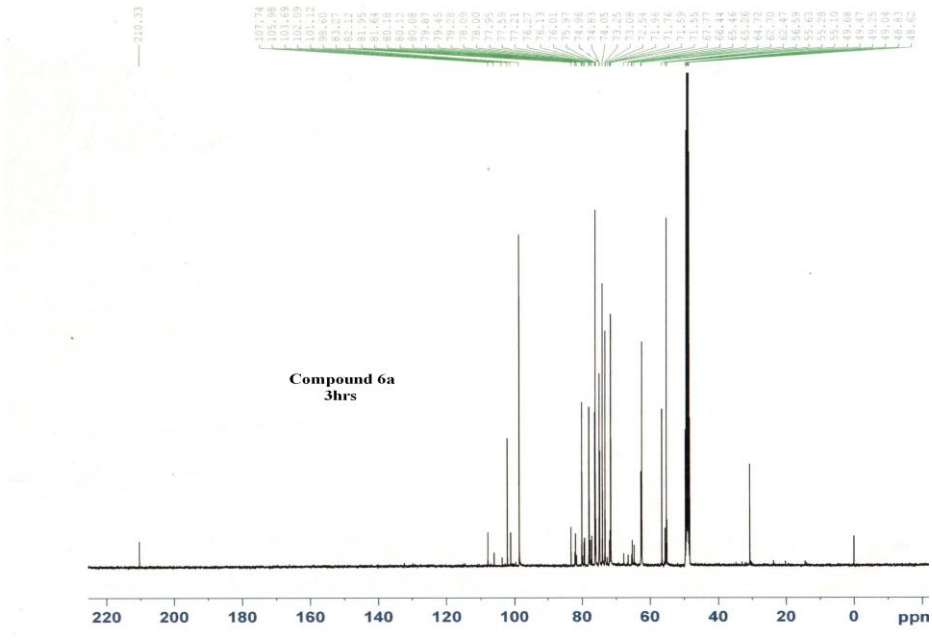


Progress of reaction between propargyl alcohol and glucose at different time interval (^{13}C NMR of reaction mixture)

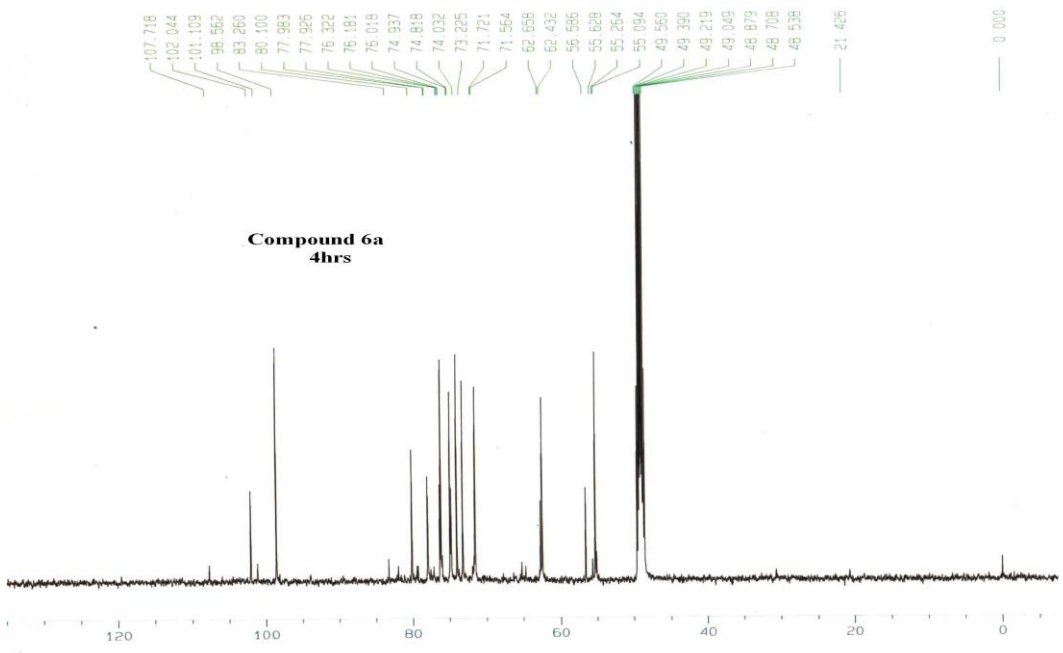
1. **After 2hrs (mixture of furanoside and pyranoside latter with preponderance)**



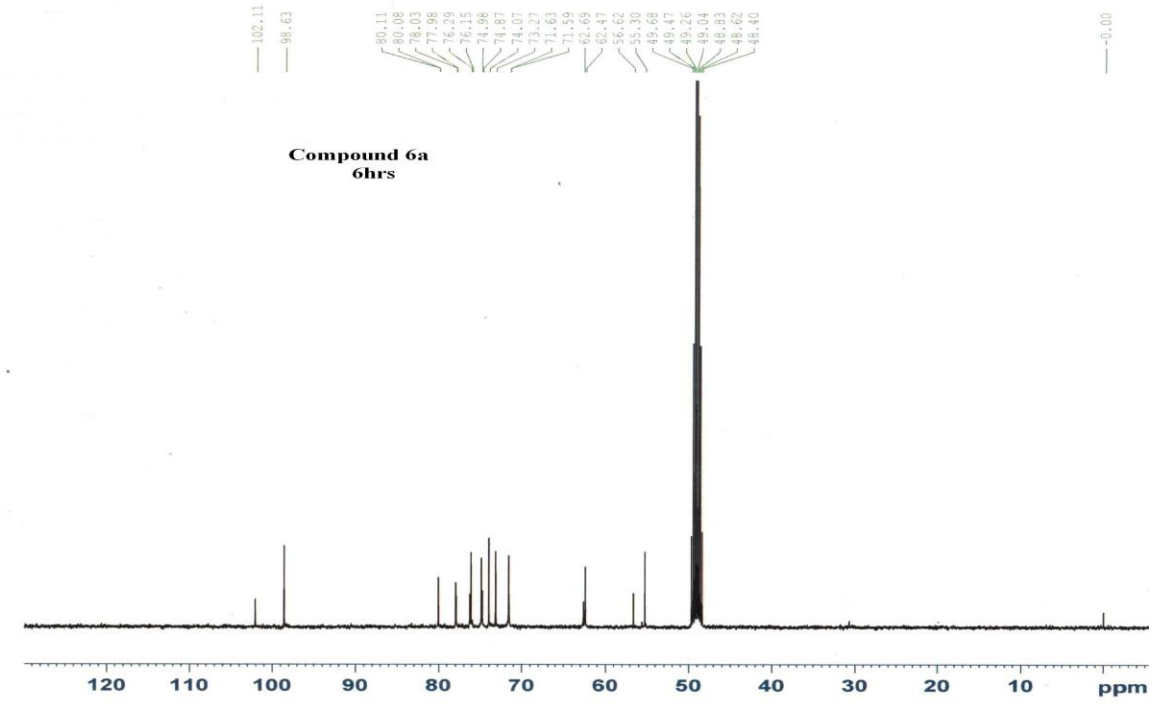
2. **After 3 hrs (mixture of furanoside and pyranoside latter with preponderance)**



3. After 4hrs (mixture of furanoside and pyranoside latter with preponderance)



4. After 6hrs (only pyranoside)



5. After 8hrs (only pyranoside)