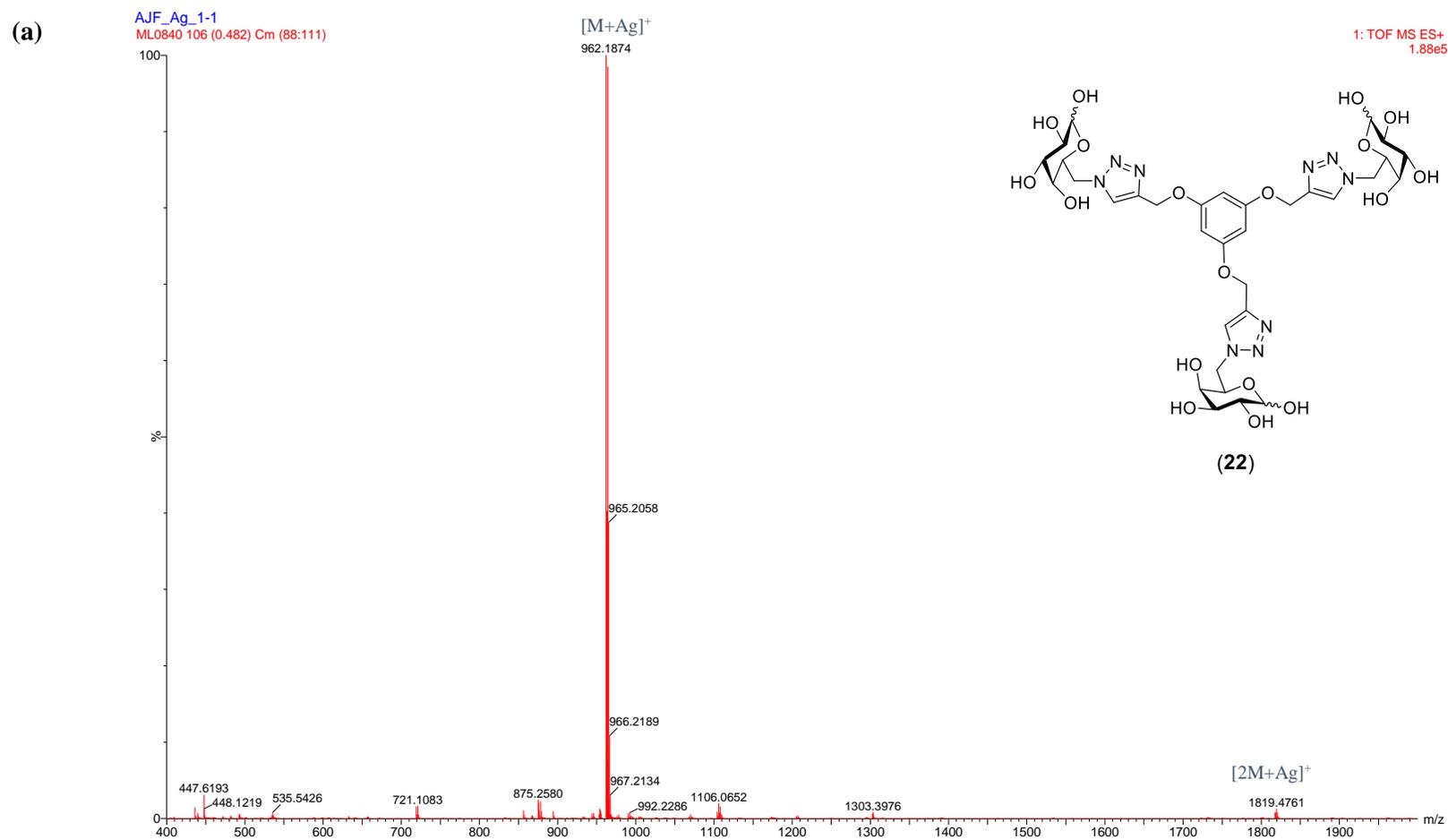


APPENDIX

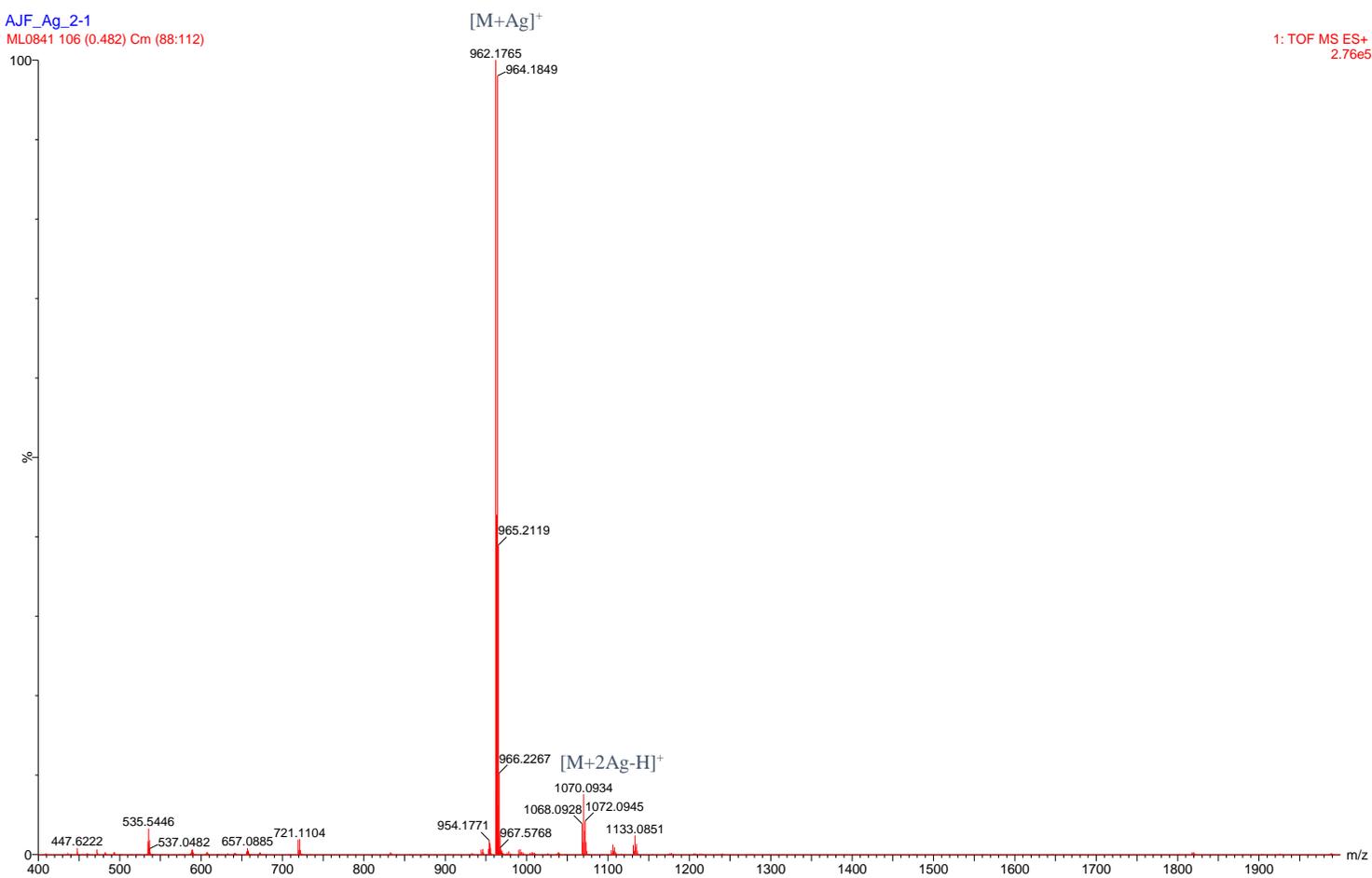
APPENDIX A:

ES-MS analysis of Ag(I)-binding using ligands (22, 23 and 39)



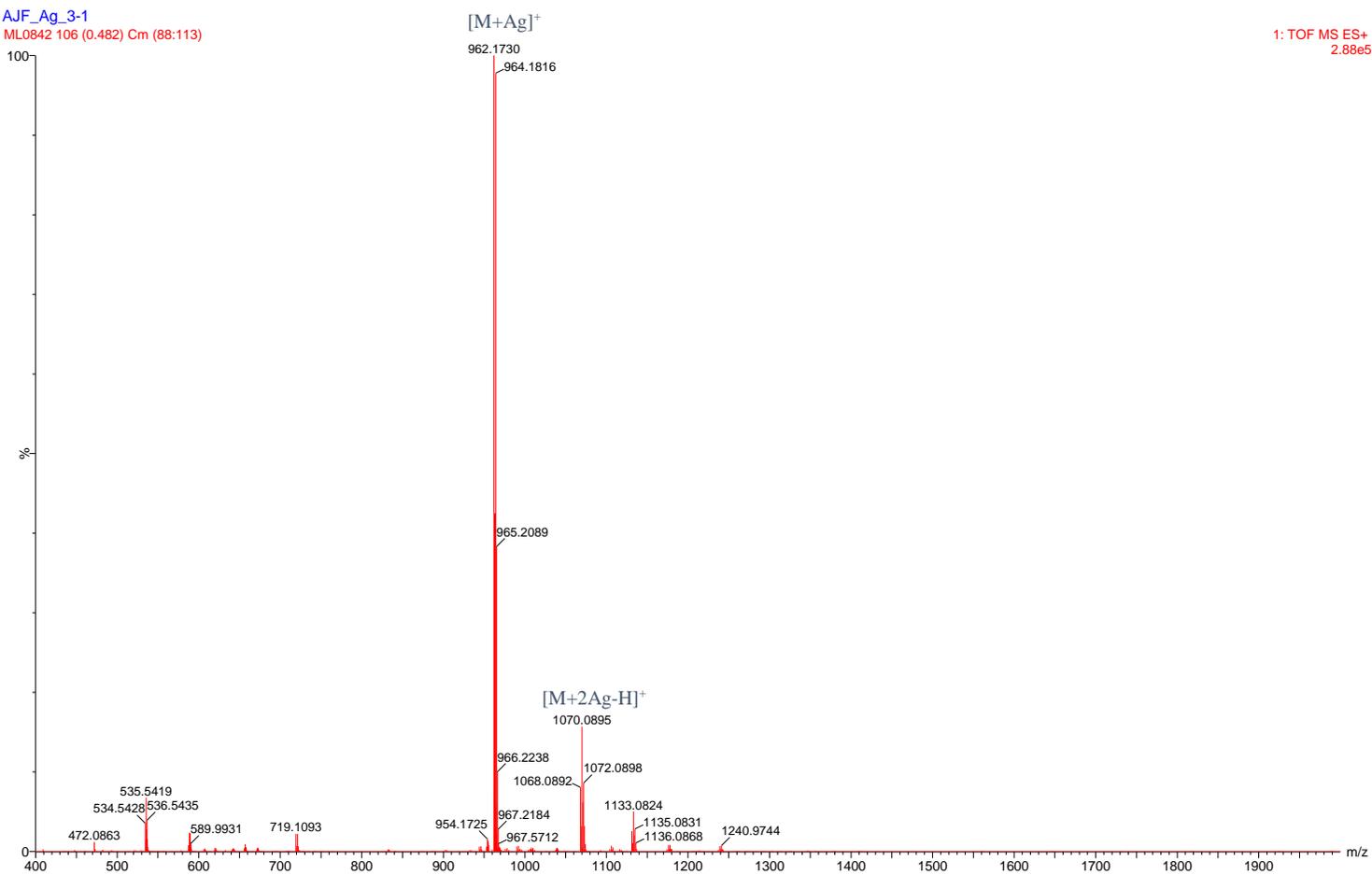
(b)

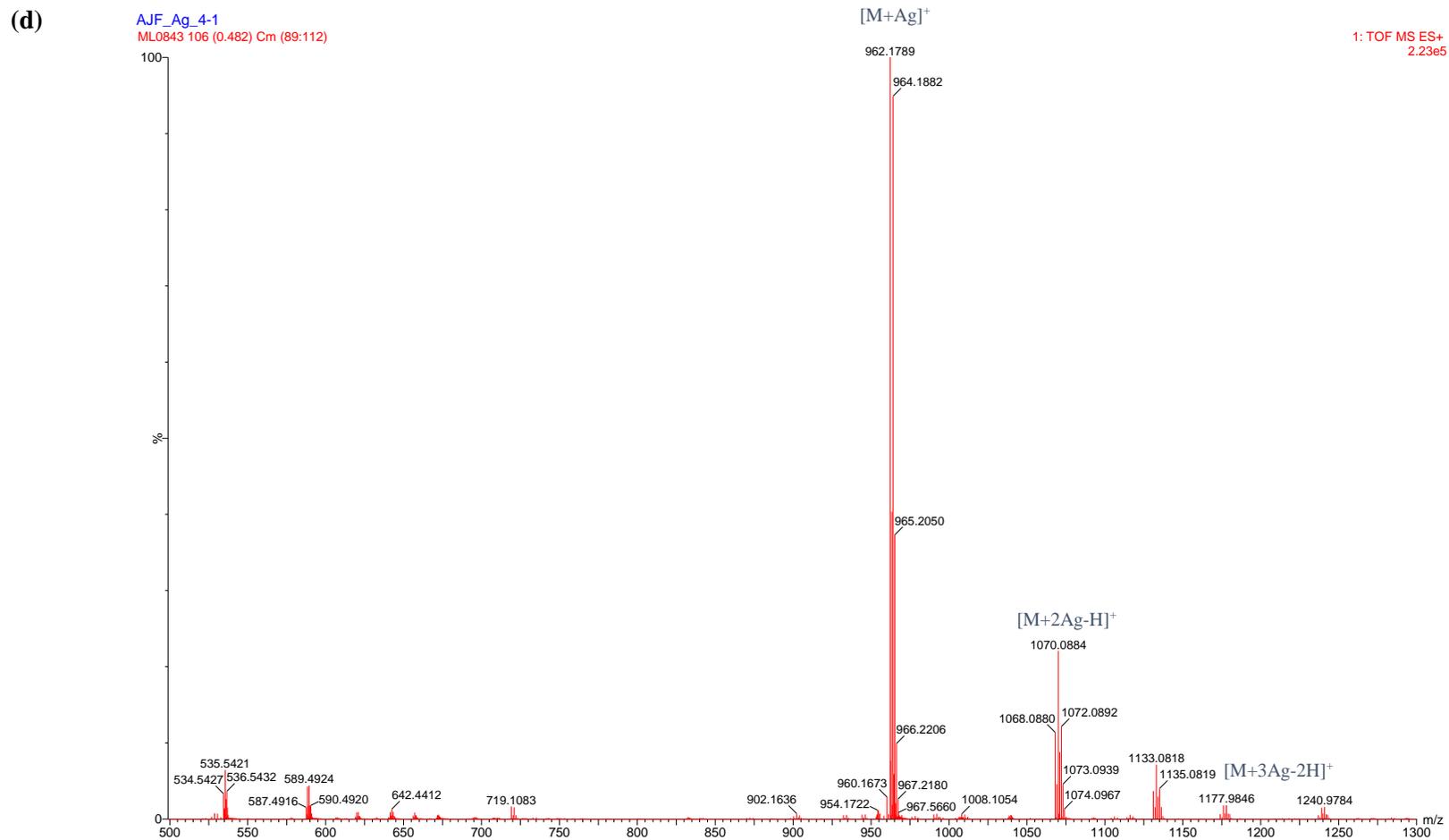
AJF_Ag_2-1
ML0841 106 (0.482) Cm (88:112)



(c)

AJF_Ag_3-1
ML0842 106 (0.482) Cm (88:113)

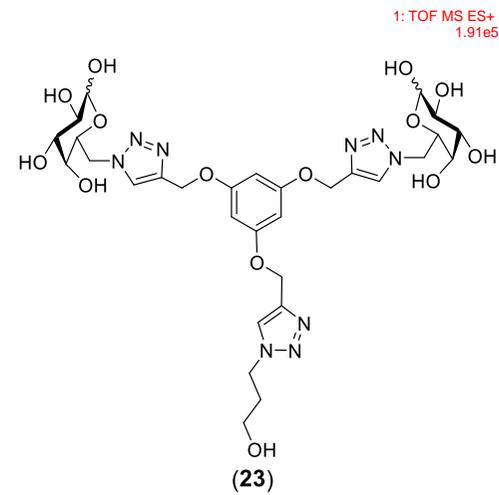
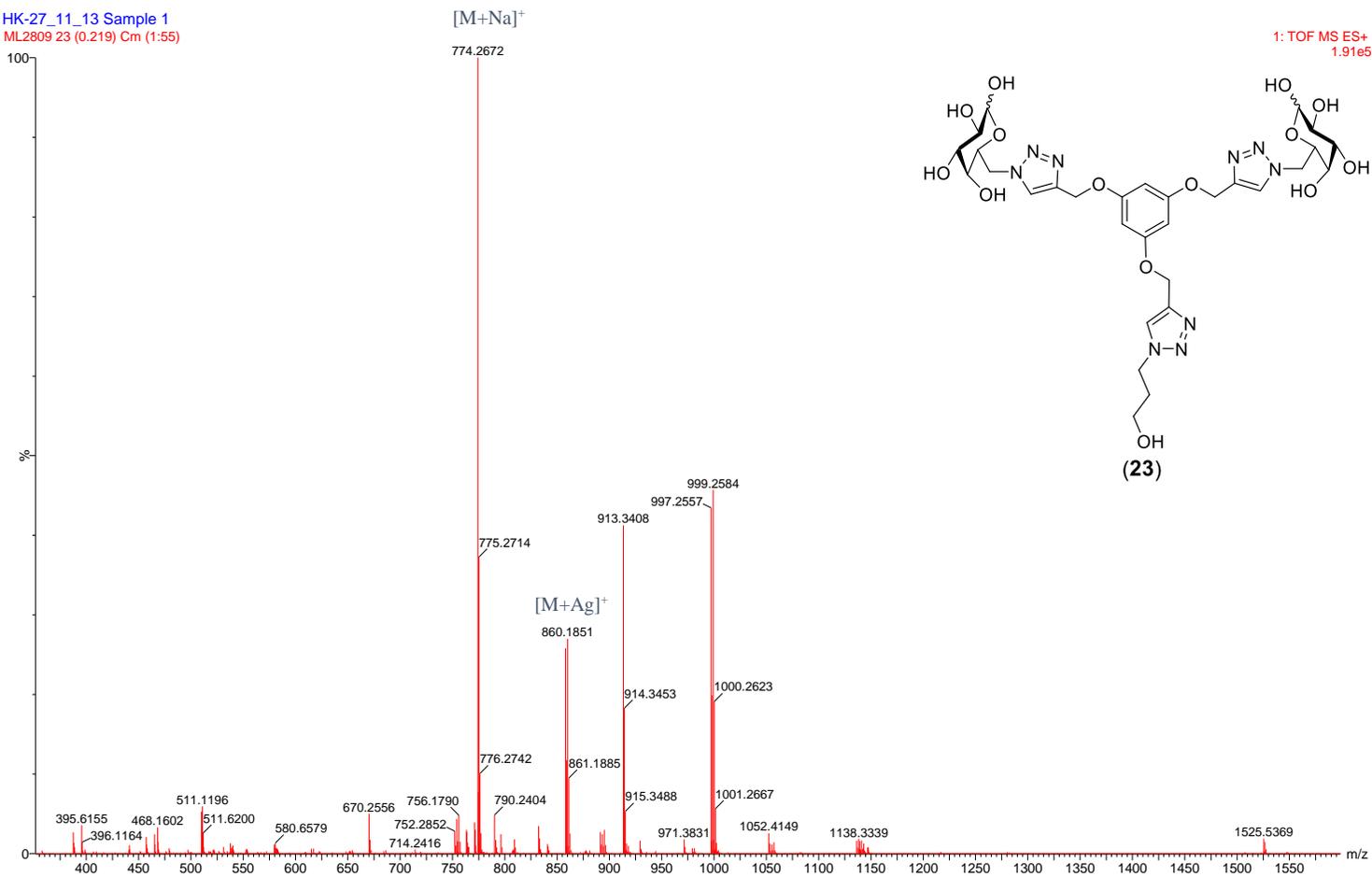




Appendix A1: ESI spectra of a solution of (22) with (a) 1 equiv., (b) 2 equiv., (c) 3 equiv., (d) 4 equiv. of AgNO_3 in H_2O .

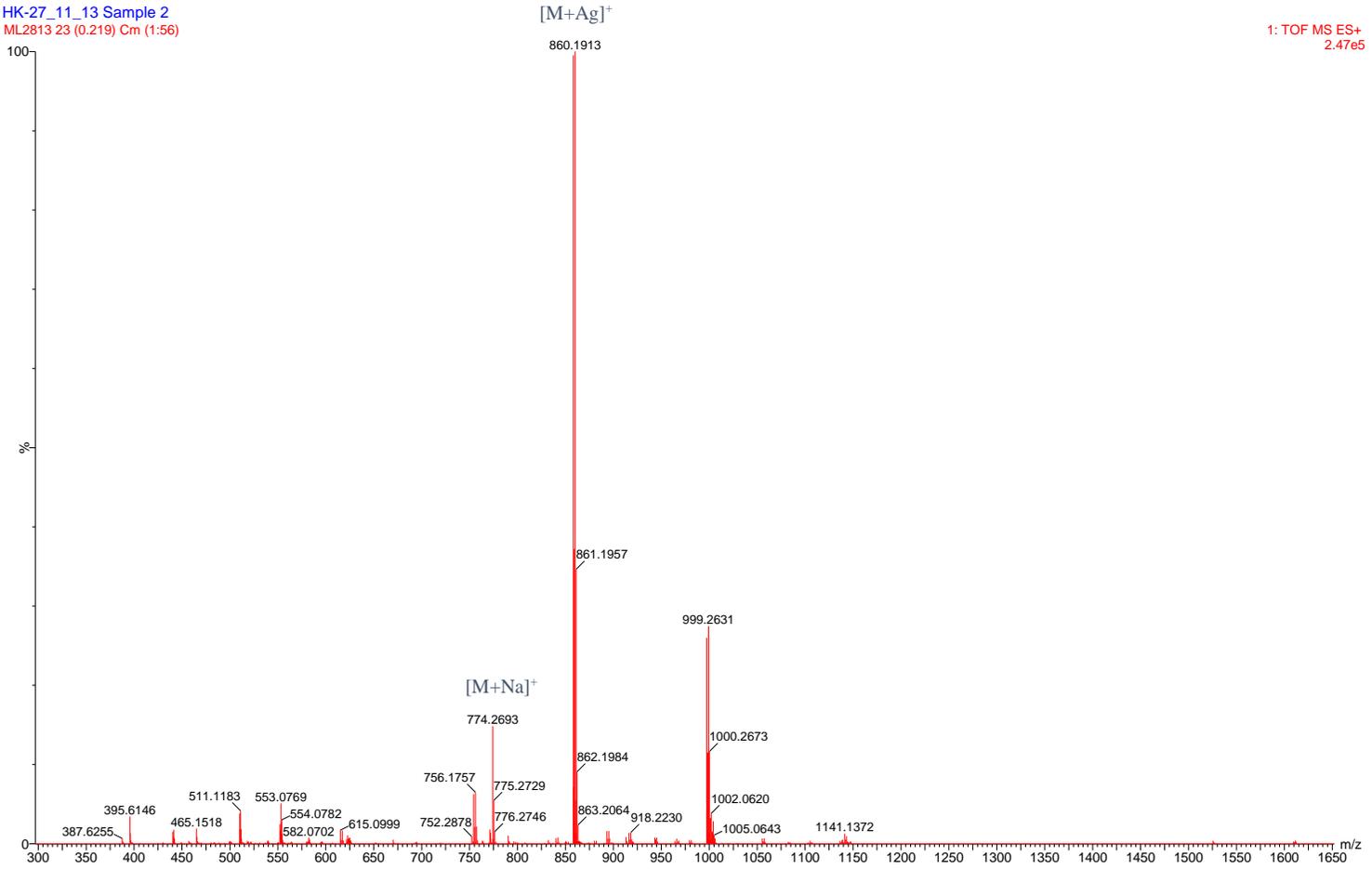
(a)

HK-27_11_13 Sample 1
ML2809 23 (0.219) Cm (1:55)



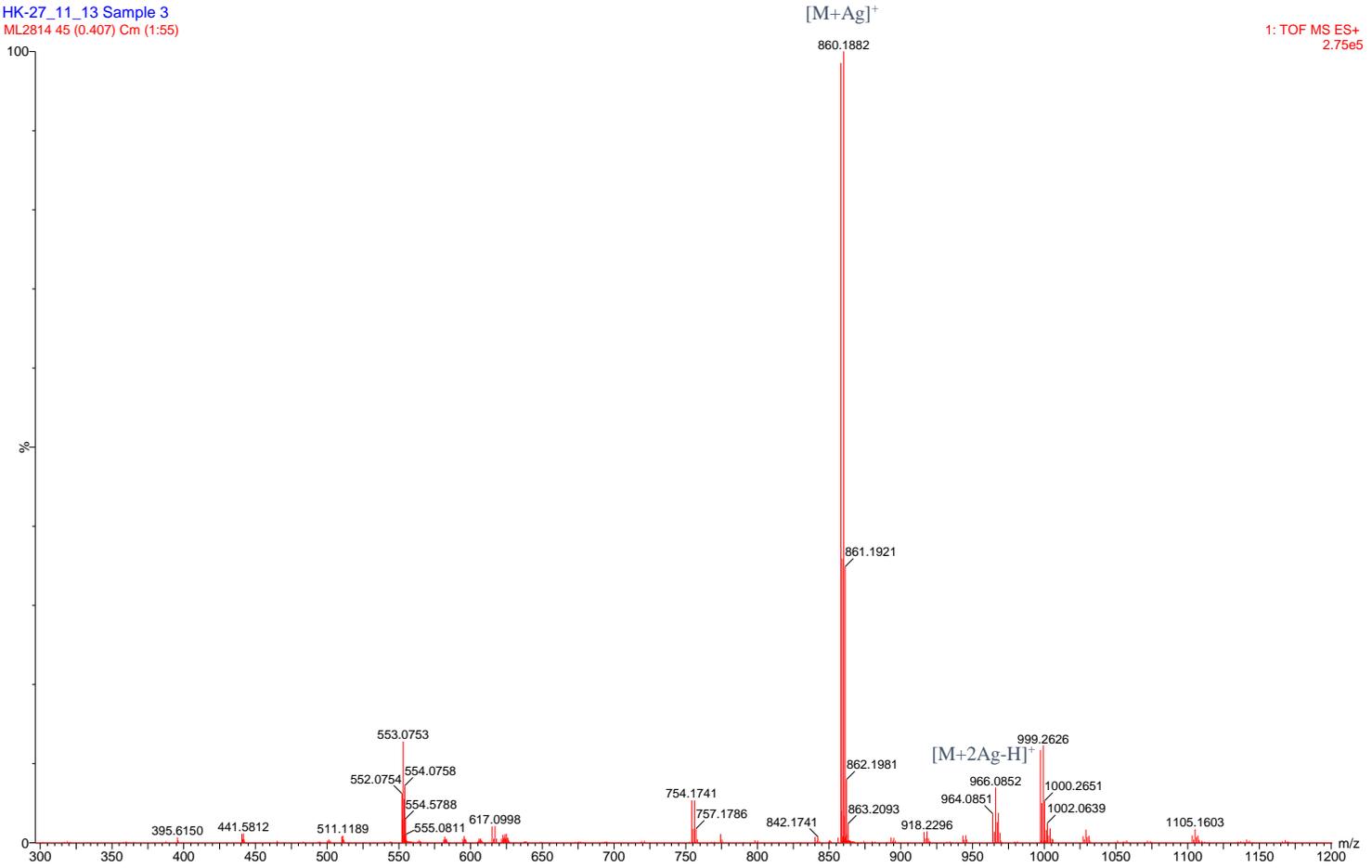
(b)

HK-27_11_13 Sample 2
ML2813 23 (0.219) Cm (1:56)



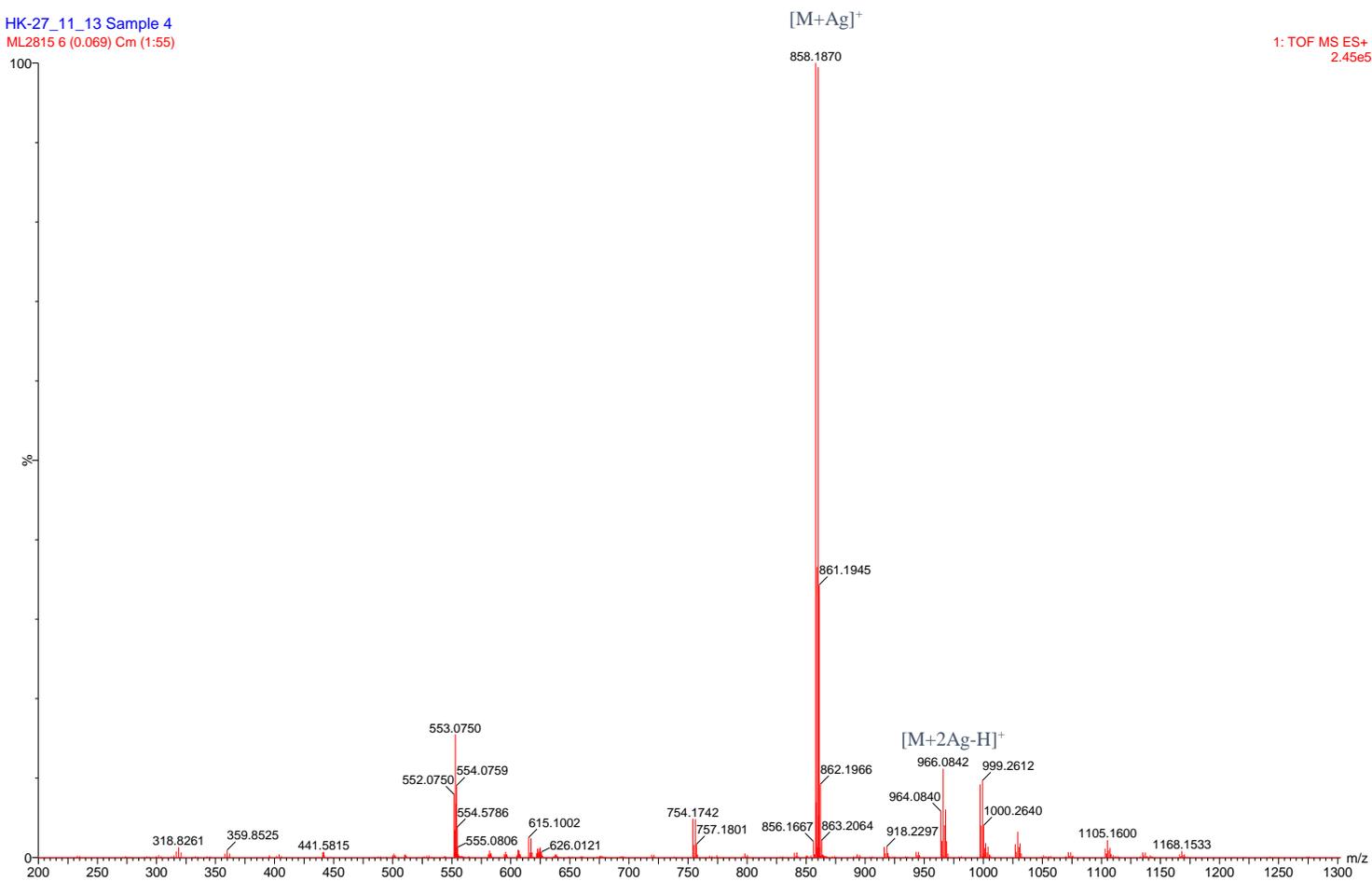
(c)

HK-27_11_13 Sample 3
ML2814 45 (0.407) Cm (1:55)



(d)

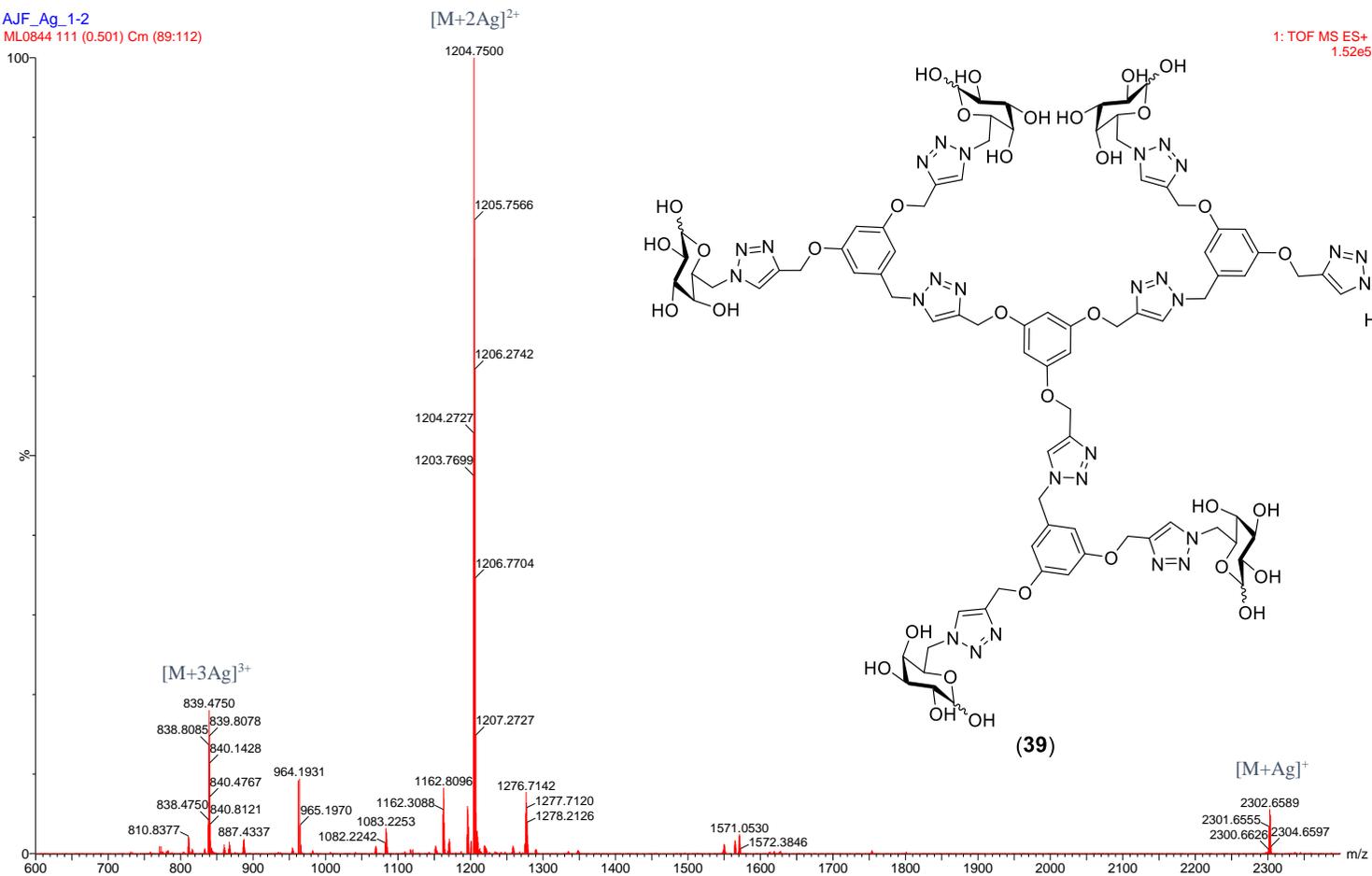
HK-27_11_13 Sample 4
ML2815 6 (0.069) Cm (1:55)



Appendix A2: ESI spectra of a solution of (23) with (a) 1 equiv., (b) 2 equiv., (c) 3 equiv., (d) 4 equiv. of AgNO_3 in H_2O .

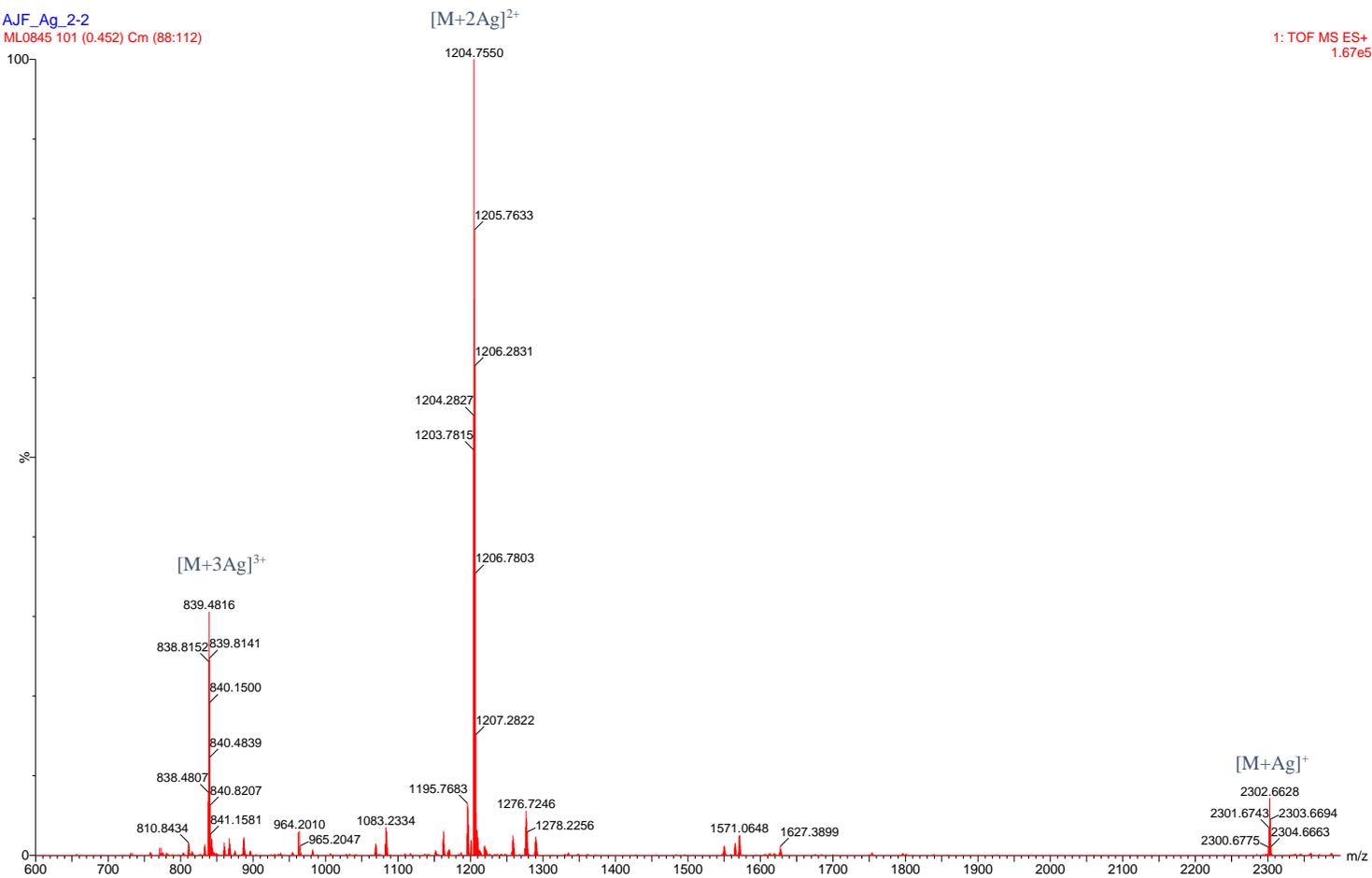
(a)

AJF_Ag_1-2
ML0844 111 (0.501) Cm (89:112)



(b)

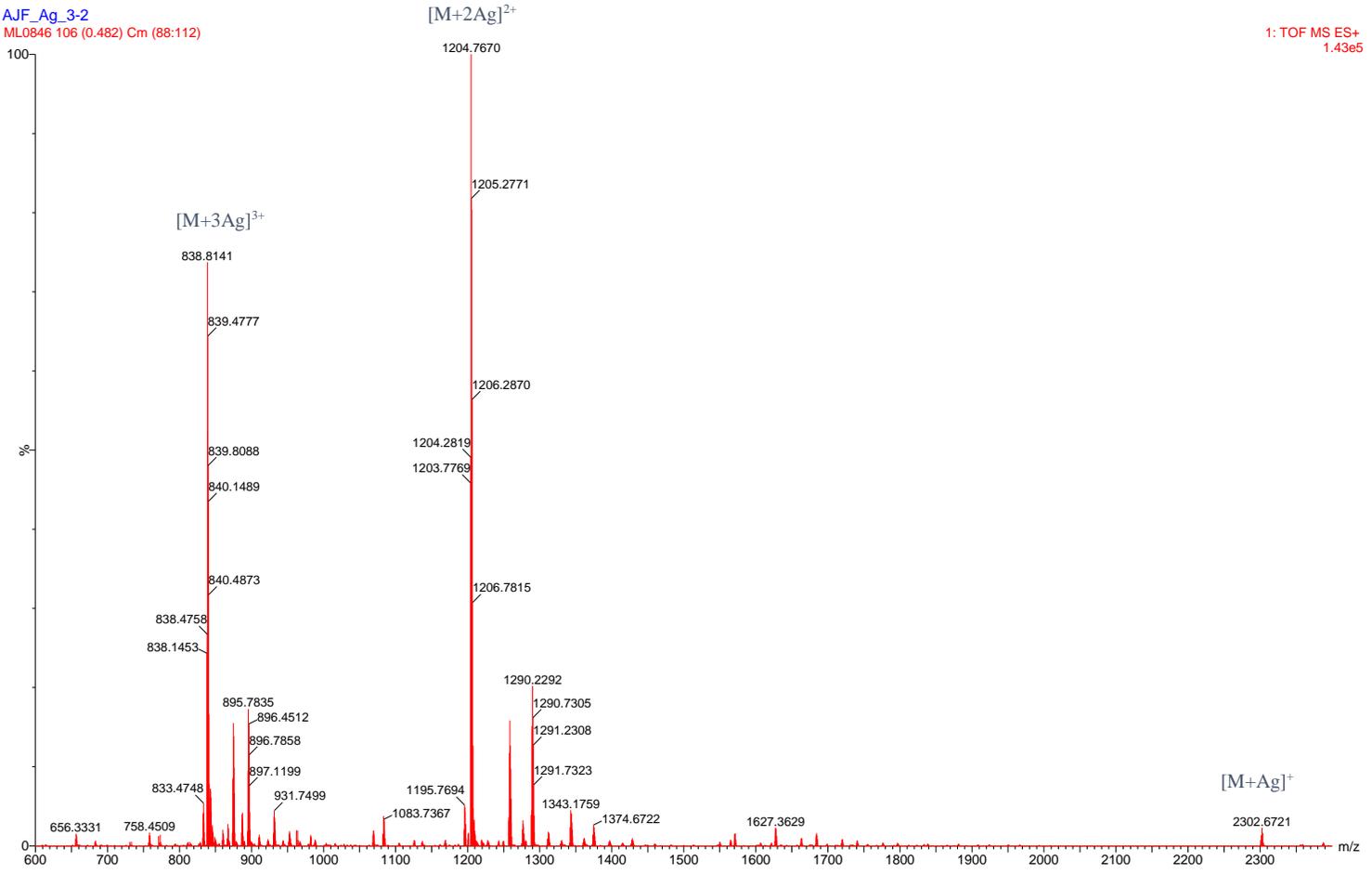
AJF_Ag_2-2
ML0845 101 (0.452) Cm (88:112)



(c)

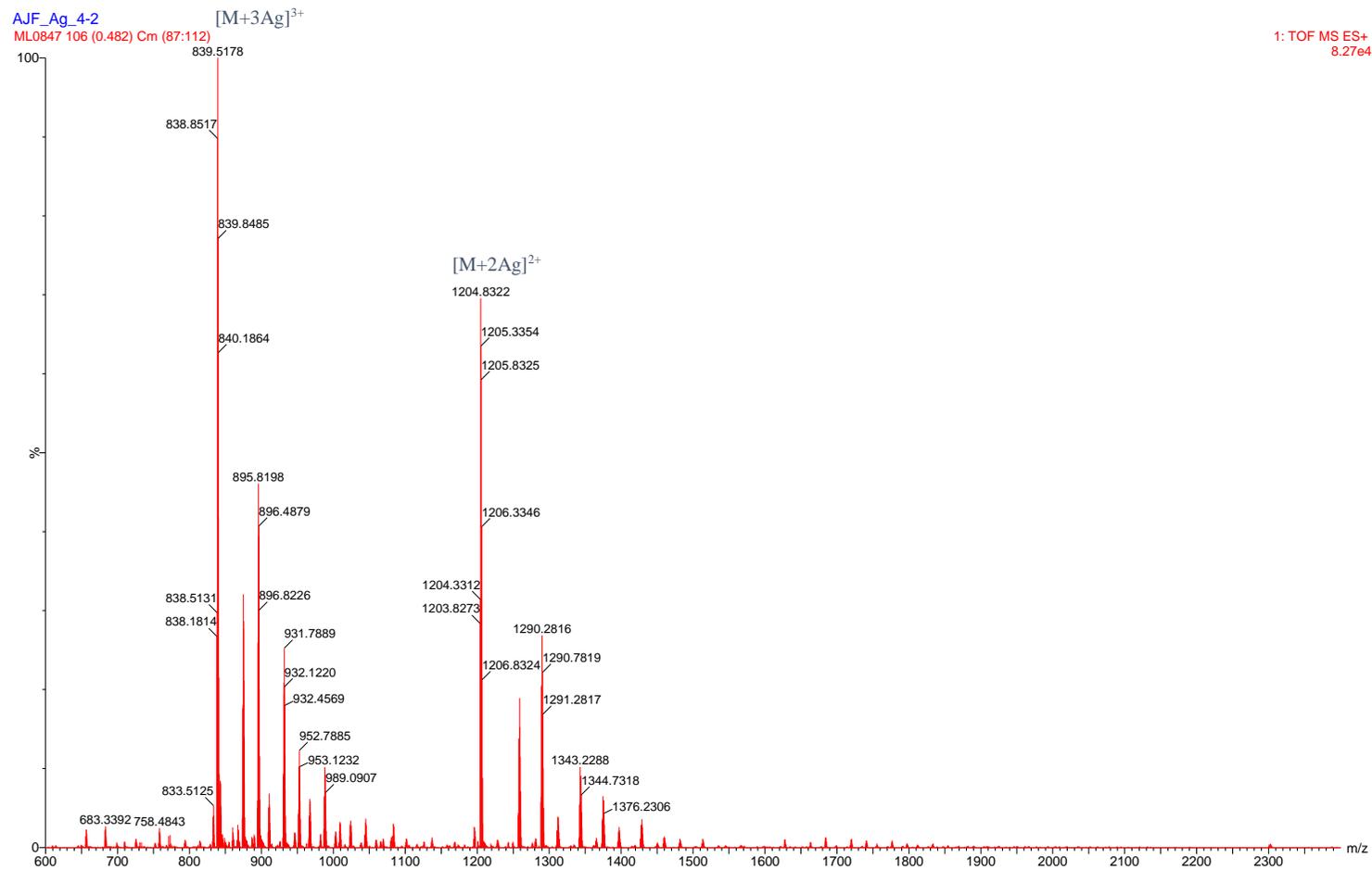
AJF_Ag_3-2
ML0846 106 (0.482) Cm (88:112)

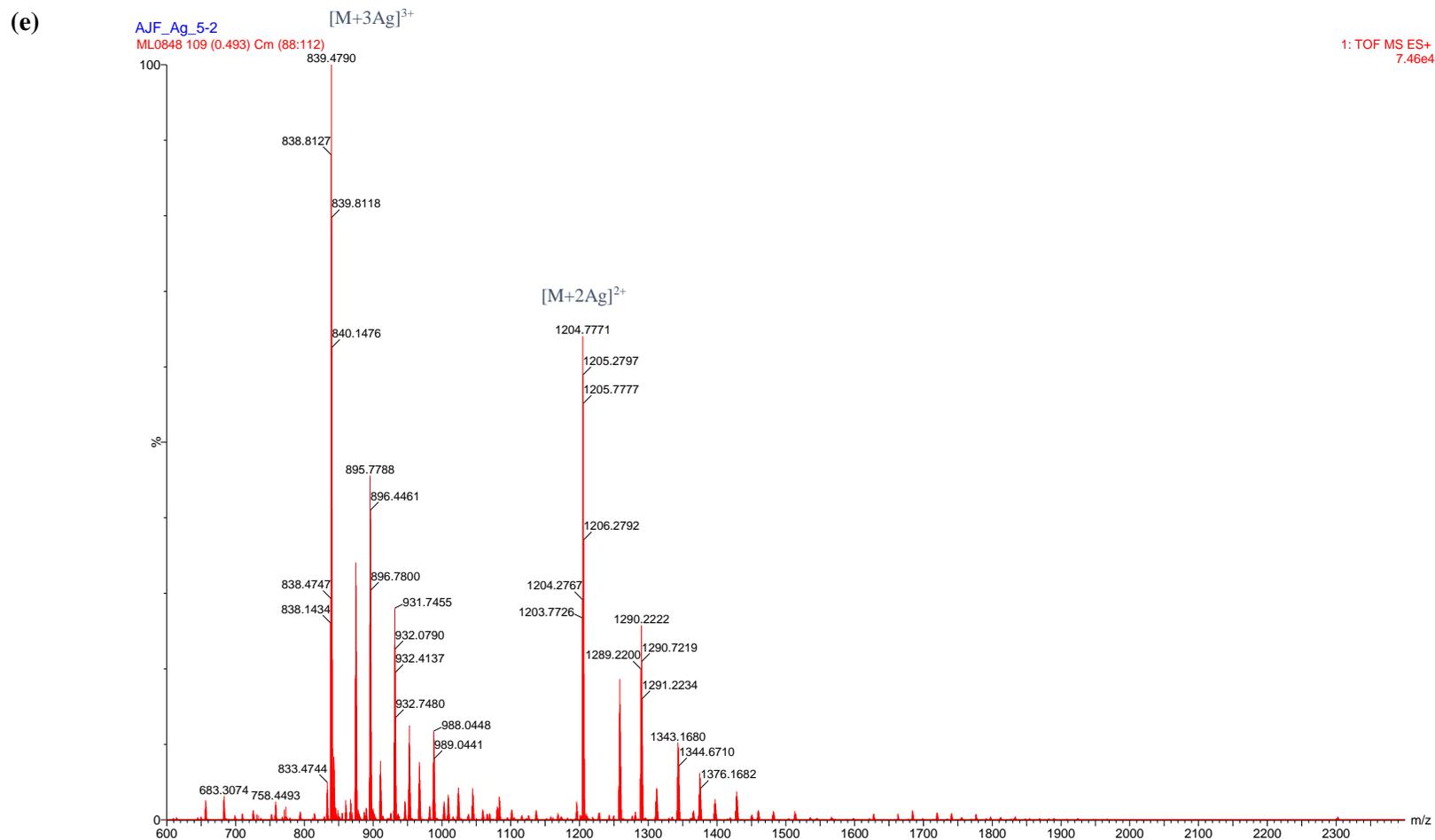
1: TOF MS ES+
1.43e5



(d)

AJF_Ag_4-2
ML0847 106 (0.482) Cm (87:112)



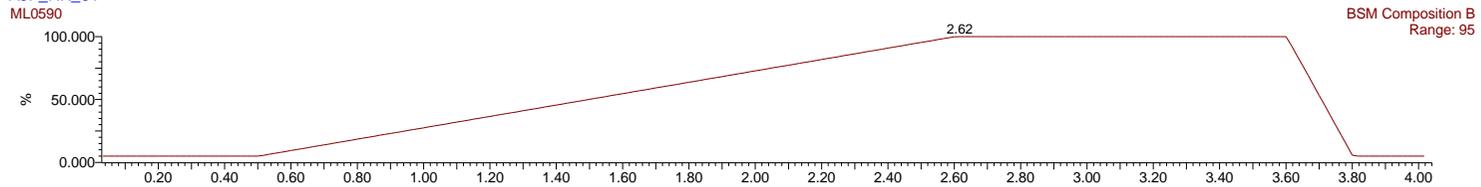


Appendix A3: ESI spectra of a solution of (39) with (a) 1 equiv., (b) 2 equiv., (c) 4 equiv., (d) 6 equiv., (e) 7.5 equiv. of $AgNO_3$ in H_2O .

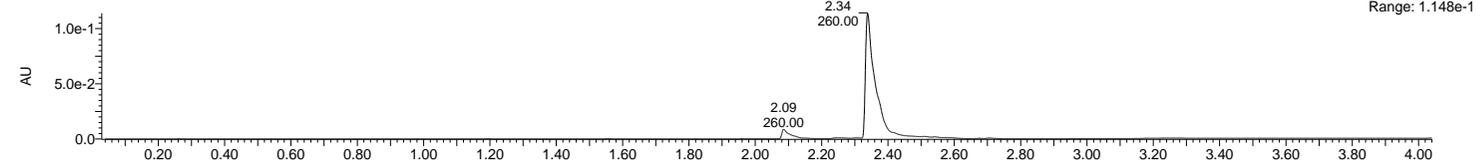
APPENDIX B:

HPLC, Mass and NMR spectra

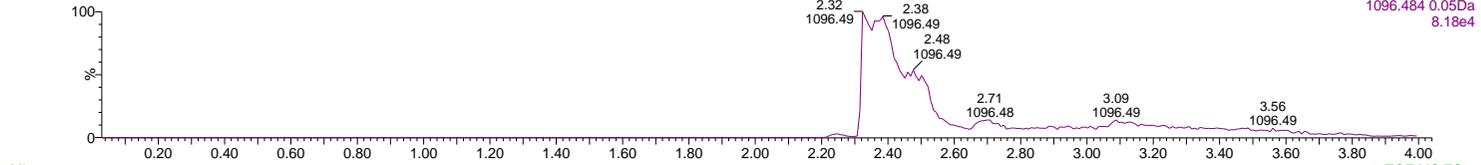
AJF_HK_01
ML0590



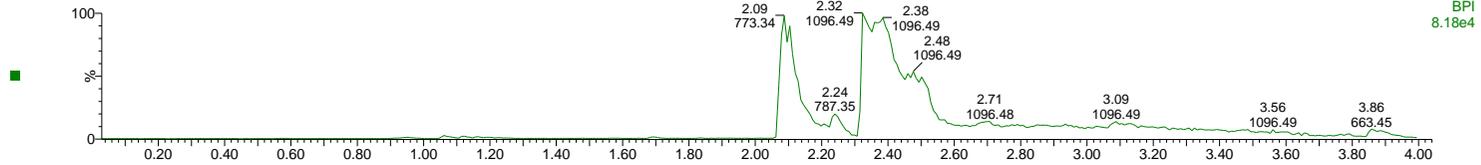
ML0590



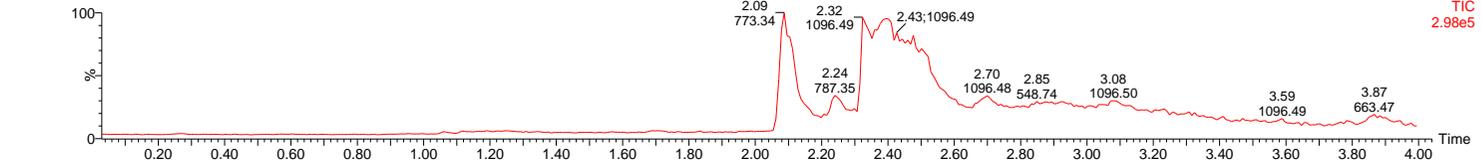
ML0590



ML0590



ML0590



AJF_HK_01

ML0590 260 (2.324) Cm (259:261)



Elemental Composition Report: **AJF-HK-01**

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

671 formula(e) evaluated with 1 results within limits (up to 20 closest results for each mass)

Elements Used:

C: 51-51 H: 0-150 N: 0-25 O: 0-25

Minimum:

-1.5

Maximum:

10.0

5.0

50.0

Mass

Calc. Mass

mDa

PPM

DBE

i-FIT

i-FIT (Norm)

Formula

1096.4884

1096.4839

4.5

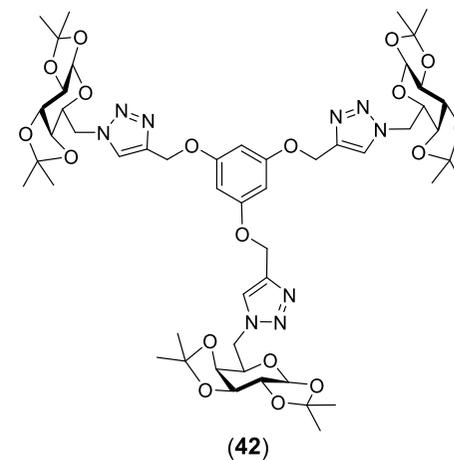
4.1

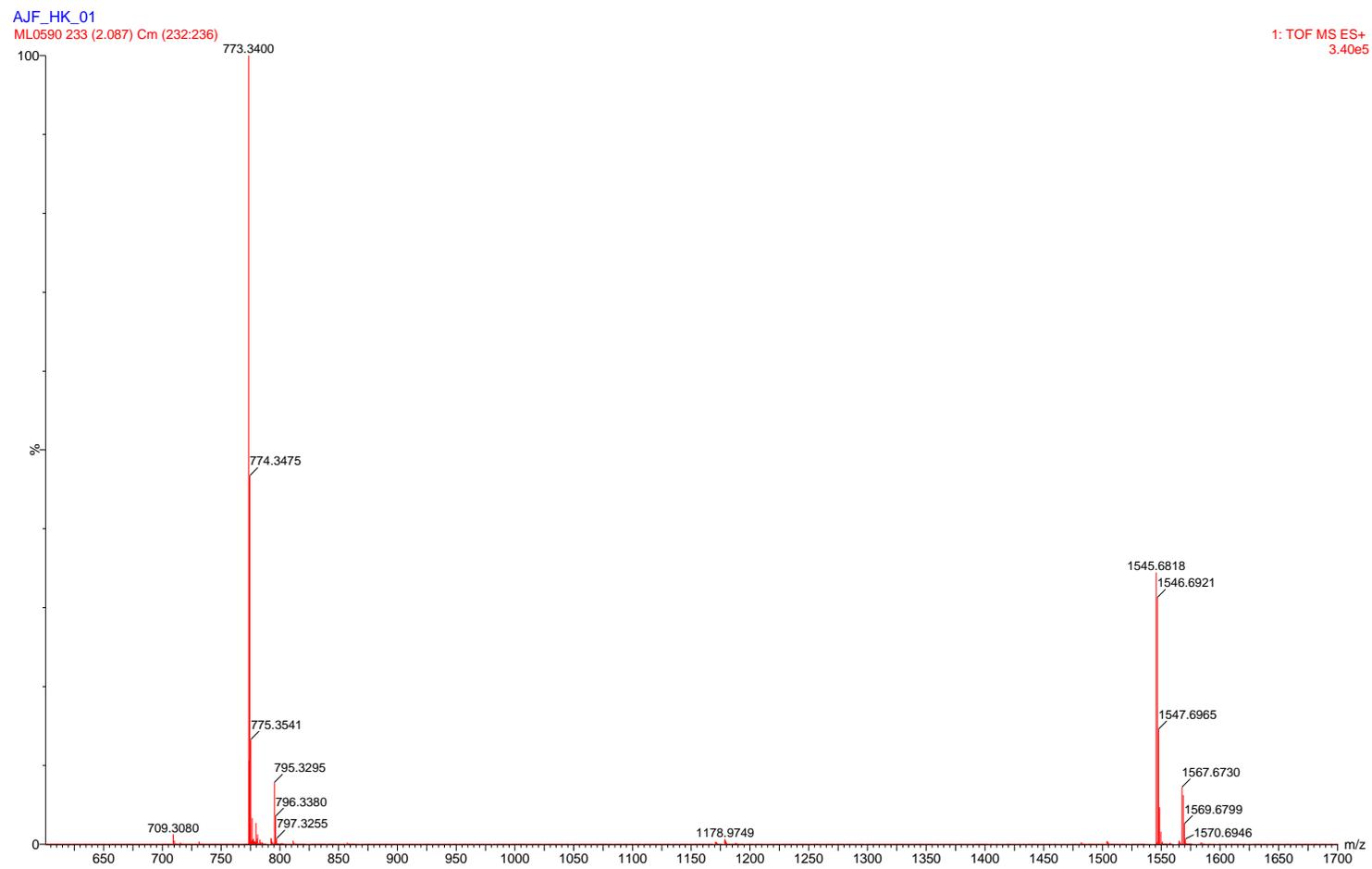
21.5

113.1

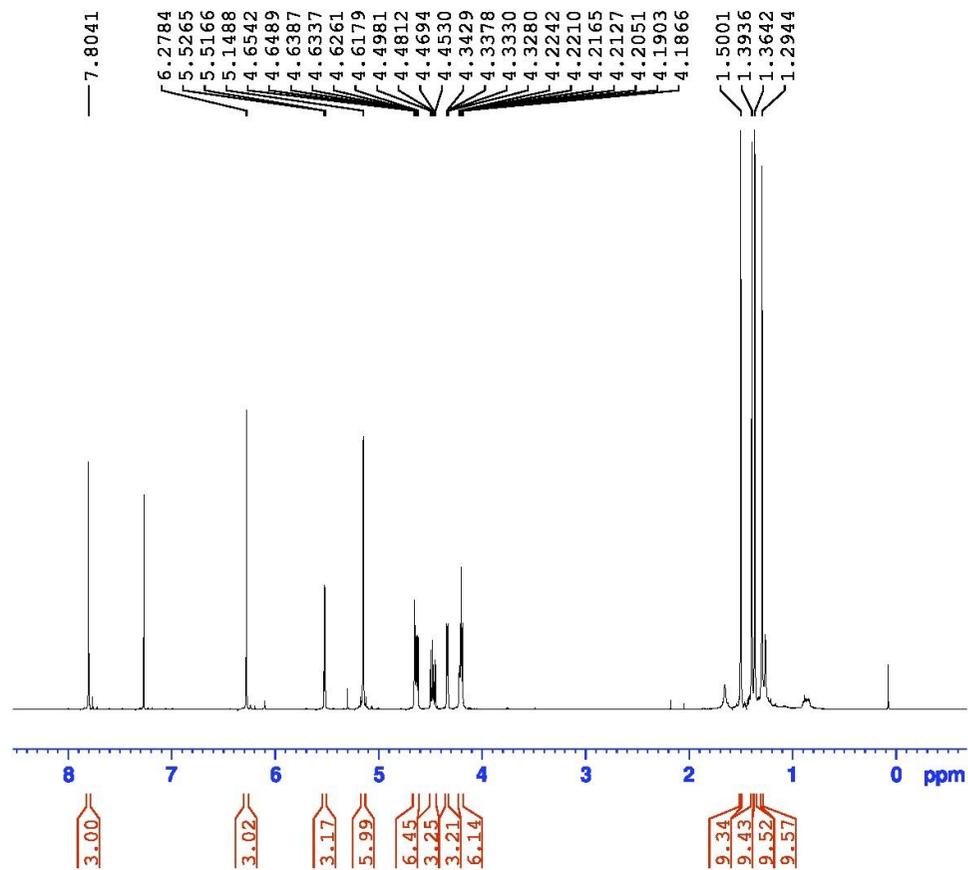
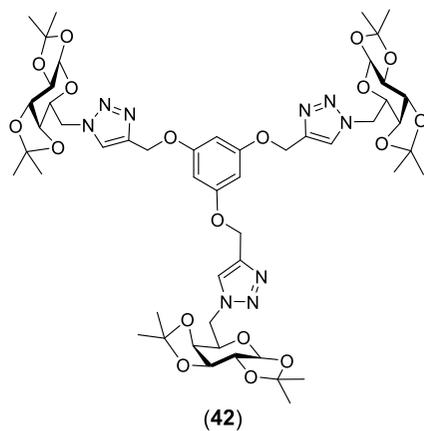
0.0

C₅₁ H₇₀ N₉ O₁₈





Appendix B1: HRMS (ESI) spectra of compound (42).



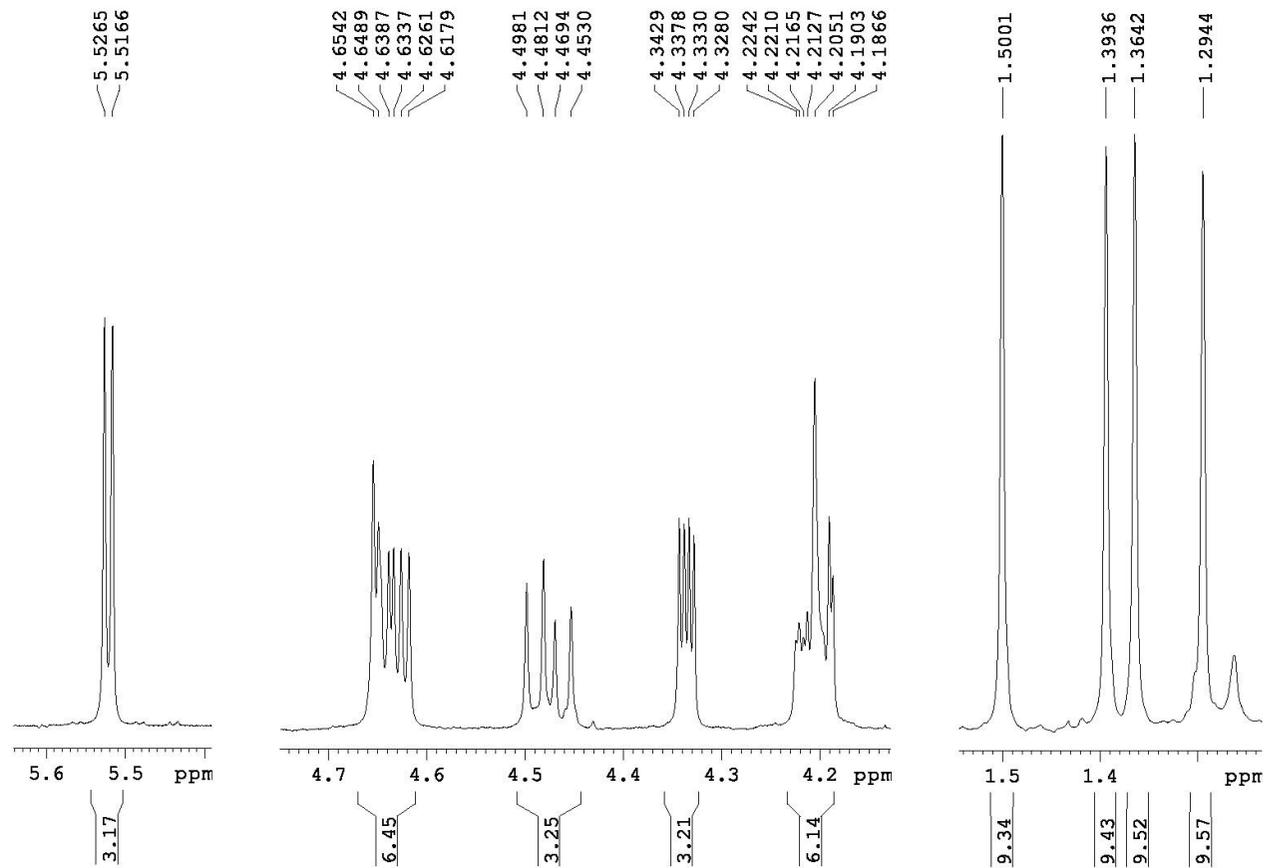
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 PROCNO 1

F2 - Acquisition Parameter
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 PROBHD 5 mm DUL 13C-1
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 10288.065 Hz
 FIDRES 0.156983 Hz
 AQ 3.1850495 sec
 RG 322.5
 DW 48.600 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 TDO 1

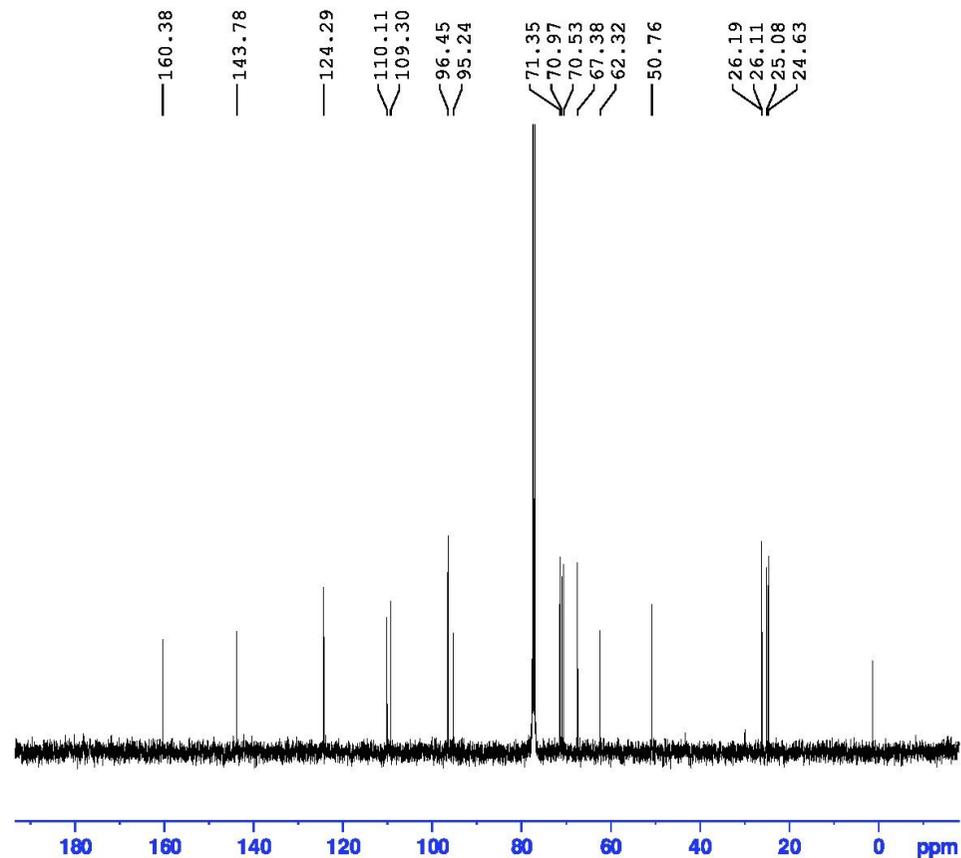
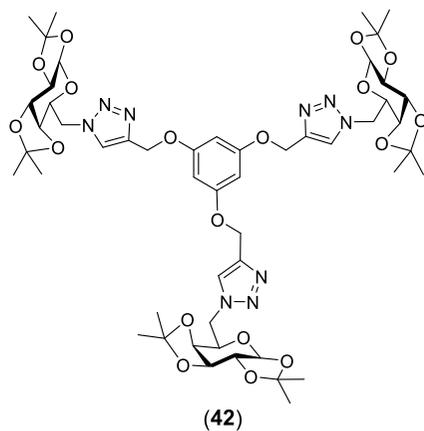
CHANNEL f1
 NUC1 1H
 P1 9.25 usec
 PL1 -2.70 dB
 SFO1 500.1330885 MHz

F2 - Processing parameters
 SI 32768
 SF 500.1300083 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Appendix B2: ^1H NMR spectrum of compound (42).



Appendix B3: Selected areas ^1H NMR of compound (42).



Current Data Parameters
NAME A25553
EXPNO 1
PROCNO 1

F2 - Acquisition Parameter
Date_ 20120207
Time 14.41
INSTRUM DRX500
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 16384
SOLVENT CDCl3
NS 1024
DS 4
SWH 30120.482 Hz
FIDRES 1.838408 Hz
AQ 0.2719744 sec
RG 10321.3
DW 16.600 usec
DE 6.00 usec
TE 300.0 K
D1 0.69999999 sec
d11 0.03000000 sec
DELTA 0.59999996 sec
TD0 1

CHANNEL f1
NUC1 13C
P1 8.00 usec
PL1 -2.30 dB
SFO1 125.7703643 MHz

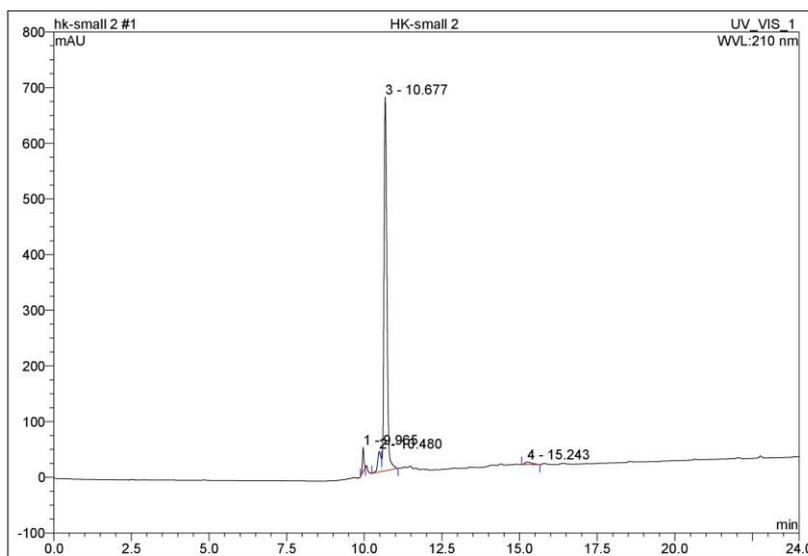
CHANNEL f2
CPDPRG[2] waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.30 dB
PL12 16.21 dB
PL13 120.00 dB
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577626 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

Appendix B4: ^{13}C NMR spectrum of compound (42).

1 HK-small 2

Sample Name:	HK-small 2	Injection Volume:	20.0
Vial Number:	RA1	Channel:	UV_VIS_1
Sample Type:	unknown	Wavelength:	210
Control Program:	poly-p4 28min +230nm	Bandwidth:	10
Quantif. Method:	dna method	Dilution Factor:	1.0000
Recording Time:	17/9/2013 13:32	Sample Weight:	1.0000
Run Time (min):	24.00	Sample Amount:	1.0000

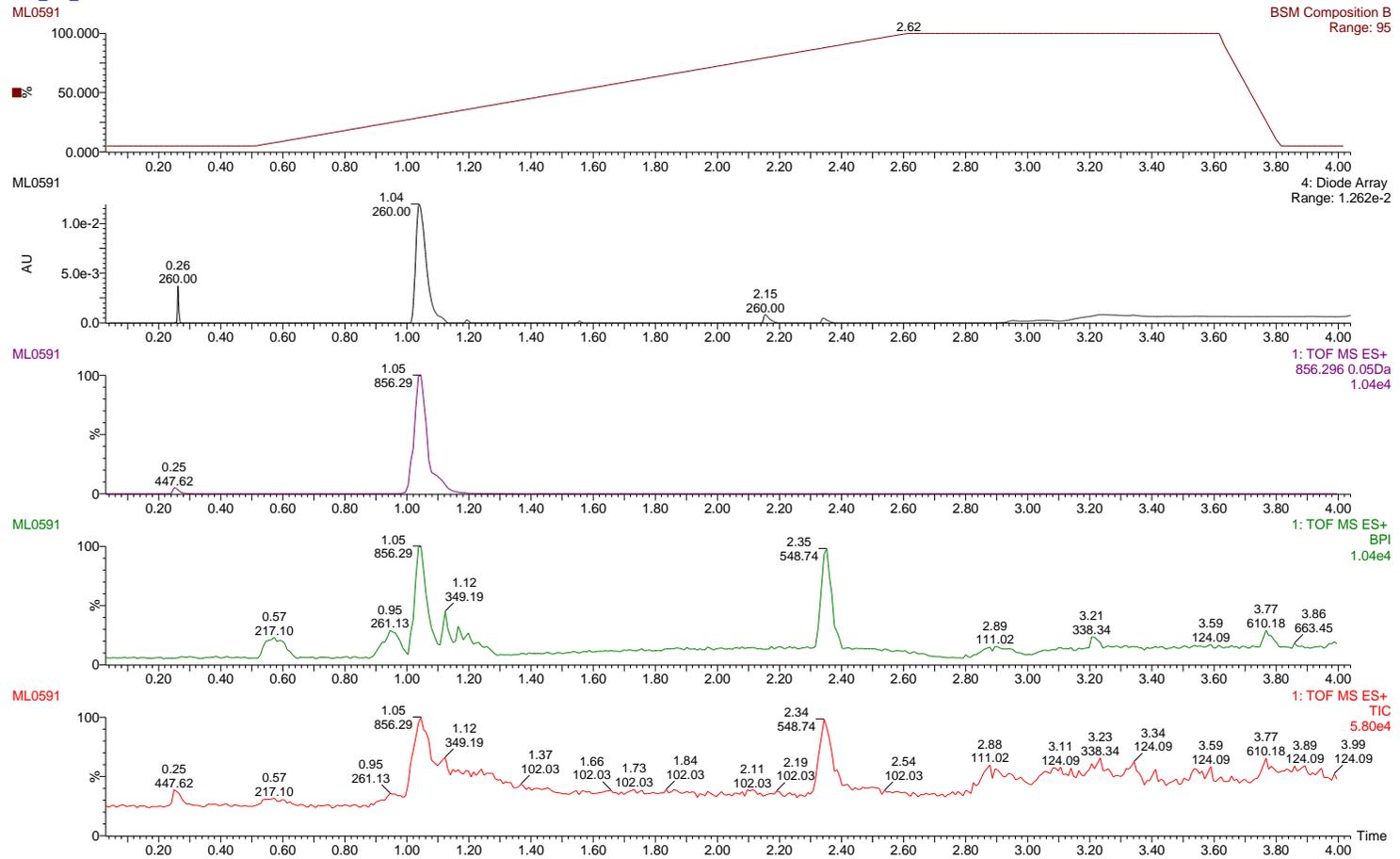


No.	Ret. Time min	Peak Name	Height mAU	Area mAU*min	Rel. Area %	Amount	Type
1	9.97	n.a.	43.782	2.431	3.00	n.a.	BMB
2	10.48	n.a.	36.670	4.476	5.52	n.a.	BM
3	10.68	n.a.	671.359	73.108	90.08	n.a.	MB
4	15.24	n.a.	4.300	1.145	1.41	n.a.	BMB
Total:			756.111	81.160	100.00	0.000	

default/integration

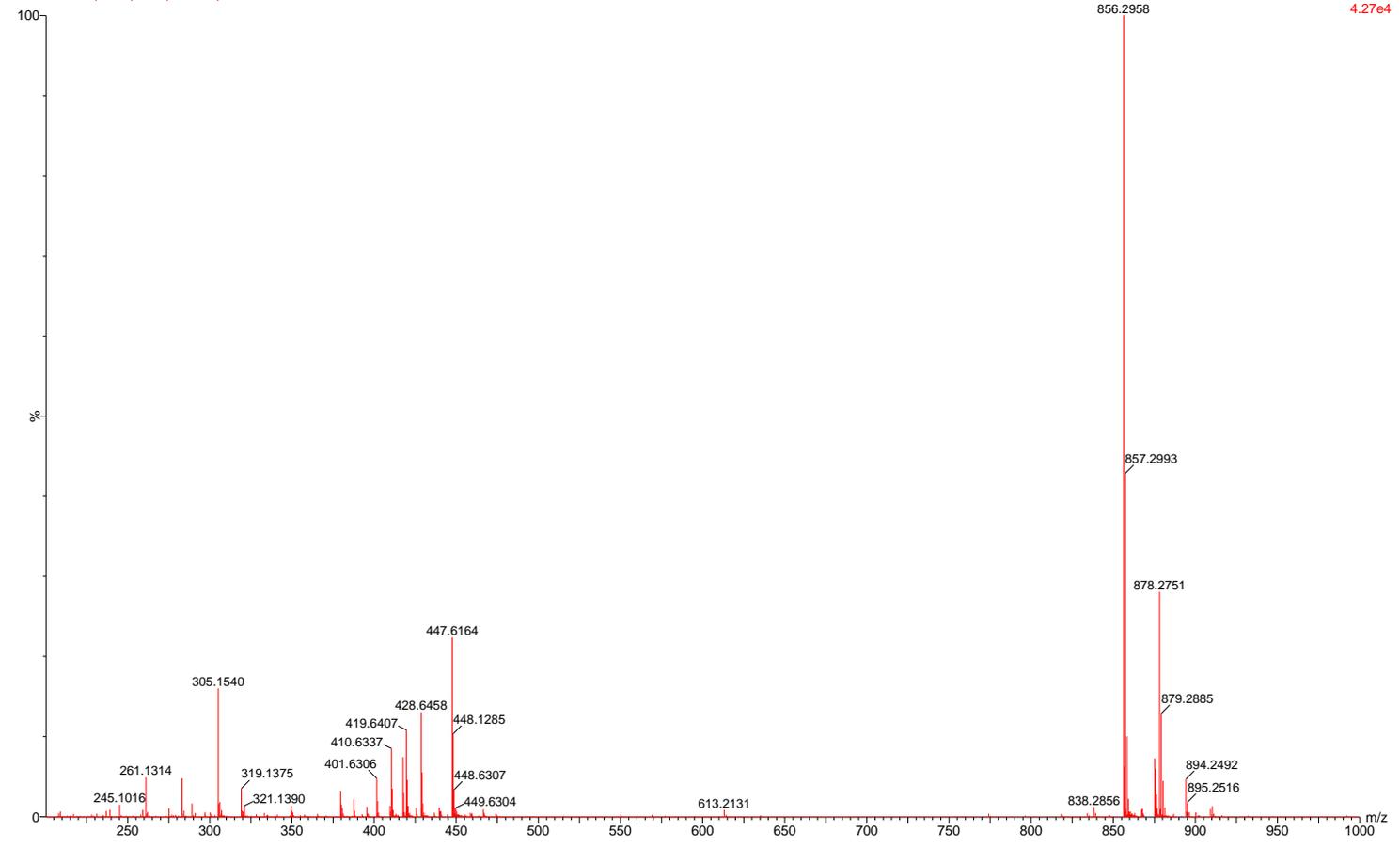
Chromeleon (c) Dionex 1996-2006
Version 6.80 SP4 Build 2361 (130805)*Appendix B5: HPL chromatogram of compound (22).*

AJF_HK_02
ML0591



AJF_HK_02

ML0591 116 (1.045) Cm (114:118)



Elemental Composition Report: **AJF-HK-02**

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

667 formula(e) evaluated with 1 results within limits (up to 20 closest results for each mass)

Elements Used:

C: 33-33 H: 0-150 N: 0-25 O: 0-25

Minimum:

-1.5

Maximum:

10.0

5.0

50.0

Mass

Calc. Mass

mDa

PPM

DBE

i-FIT

i-FIT (Norm)

Formula

856.2958

856.2961

-0.3

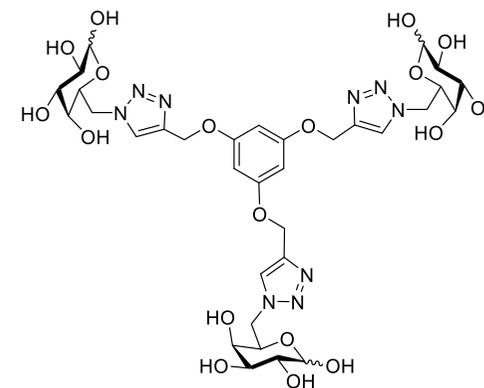
-0.4

15.5

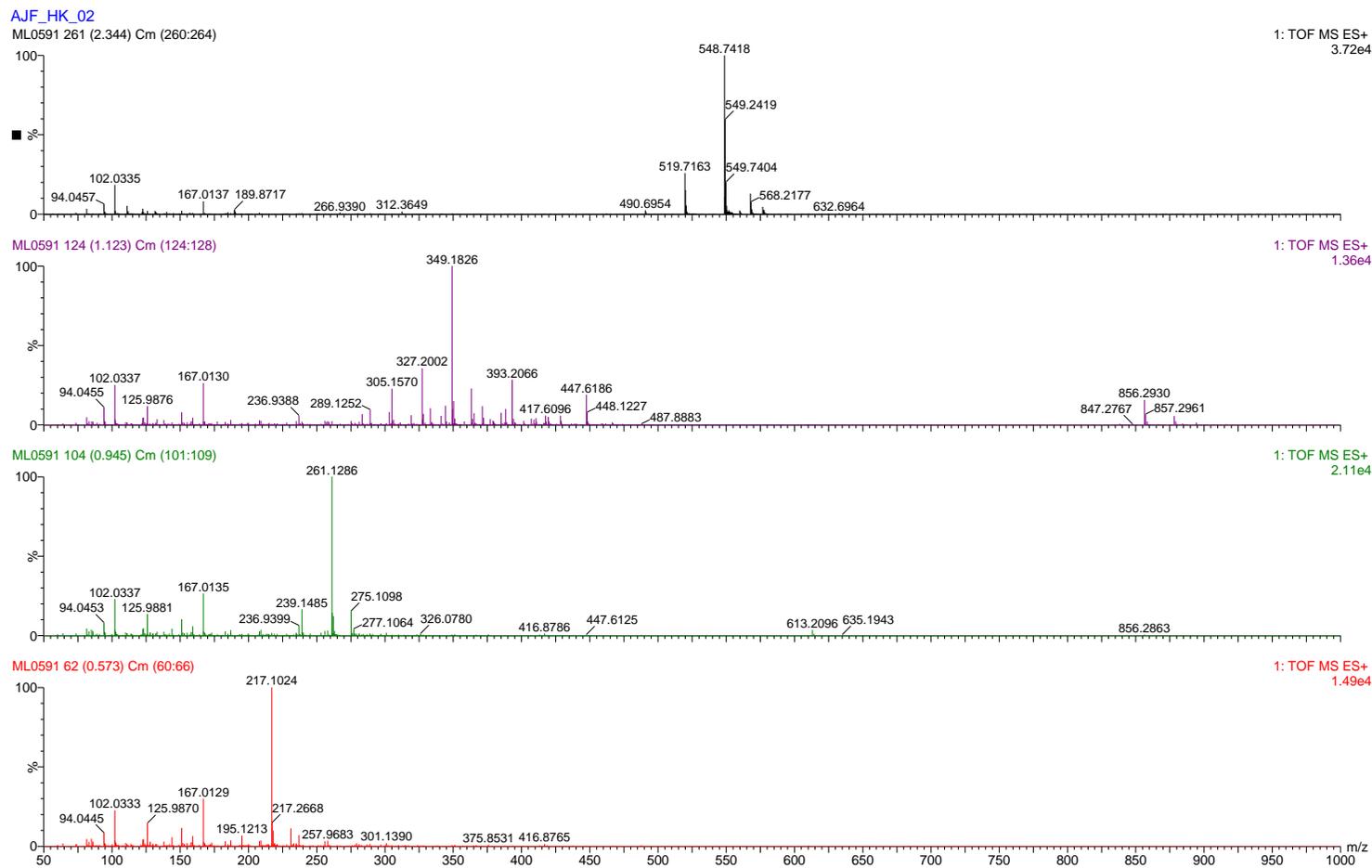
137.7

0.0

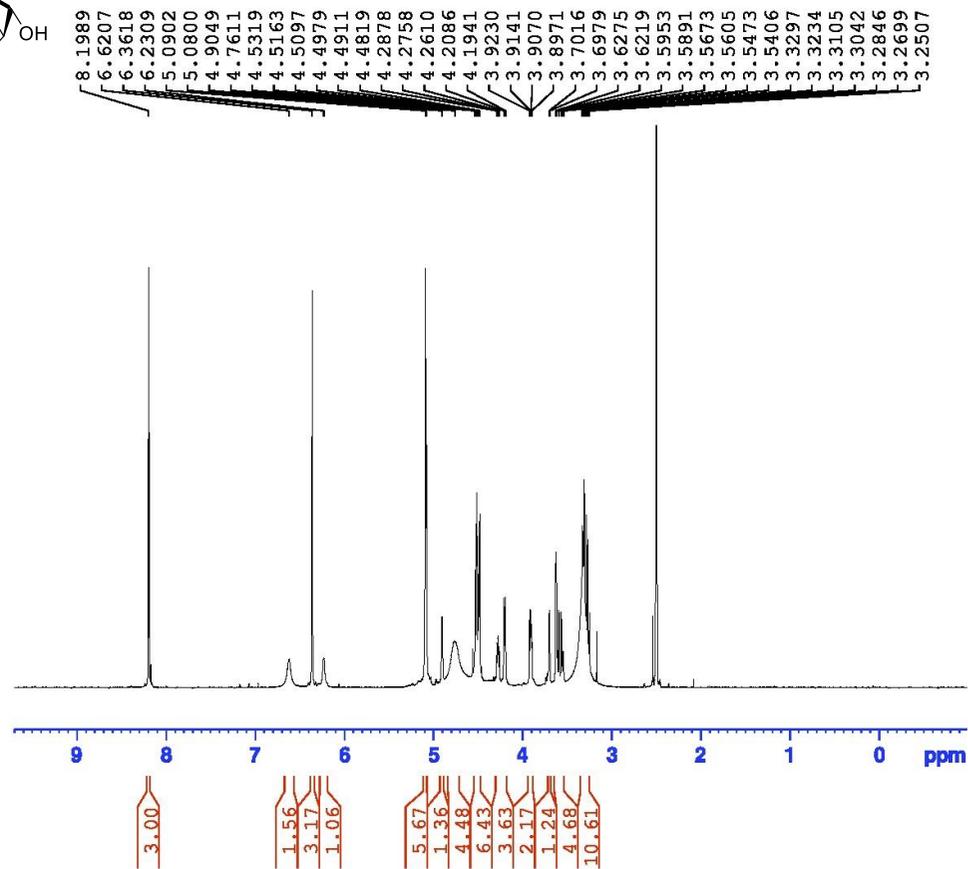
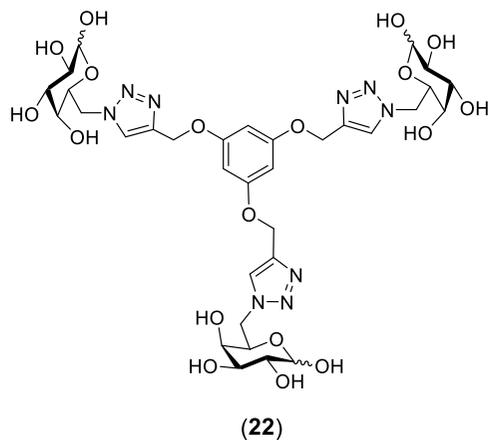
C₃₃ H₄₆ N₉ O₁₈



(22)



Appendix B6: HRMS (ESI) spectra of compound (22).



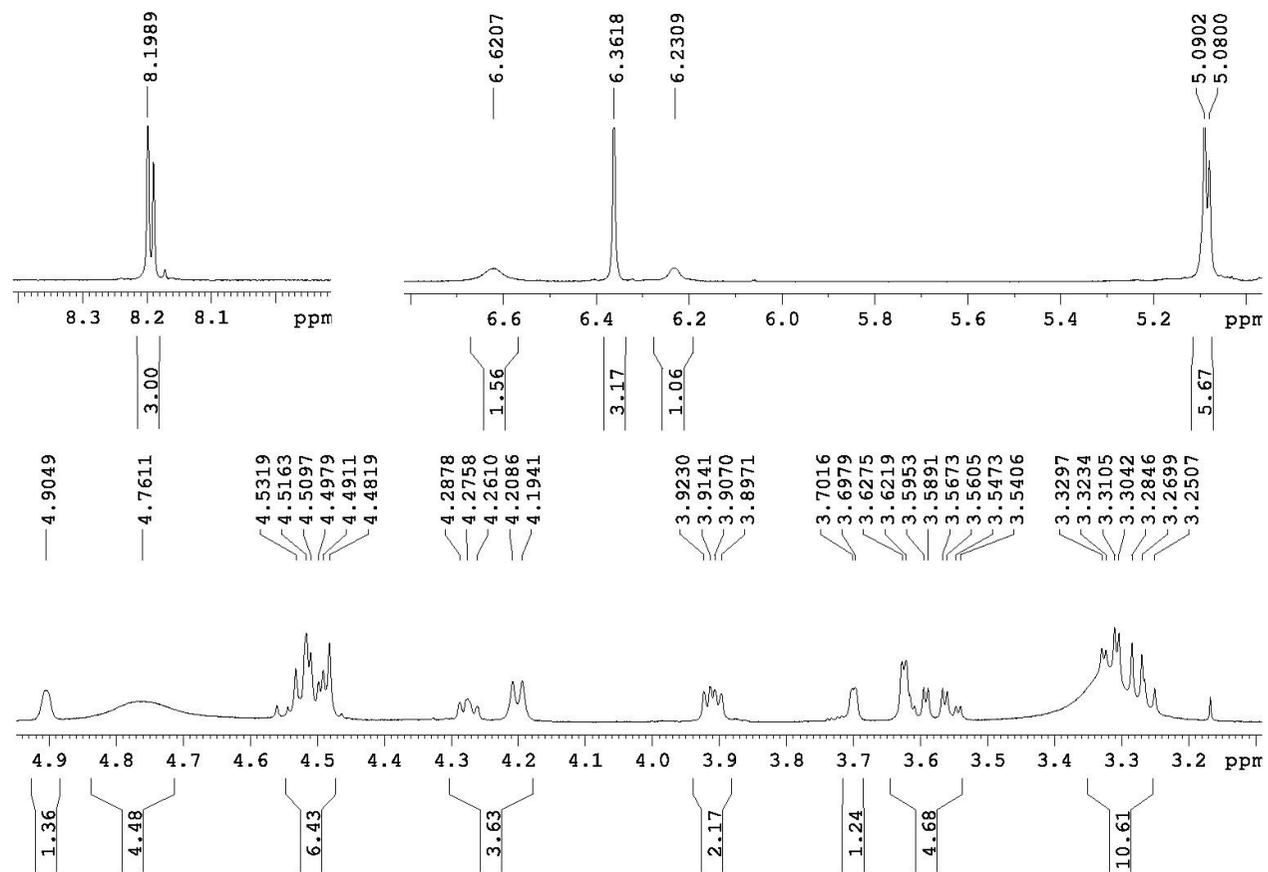
Current Data Parameters
NAME A26823
EXPNO 1
PROCNO 1

F2 - Acquisition Parameter
Date_ 20120330
Time 13.50
INSTRUM DRX500
PROBED 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 10288.065 Hz
FIDRES 0.156983 Hz
AQ 3.1850495 sec
RG 322.5
DW 48.600 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

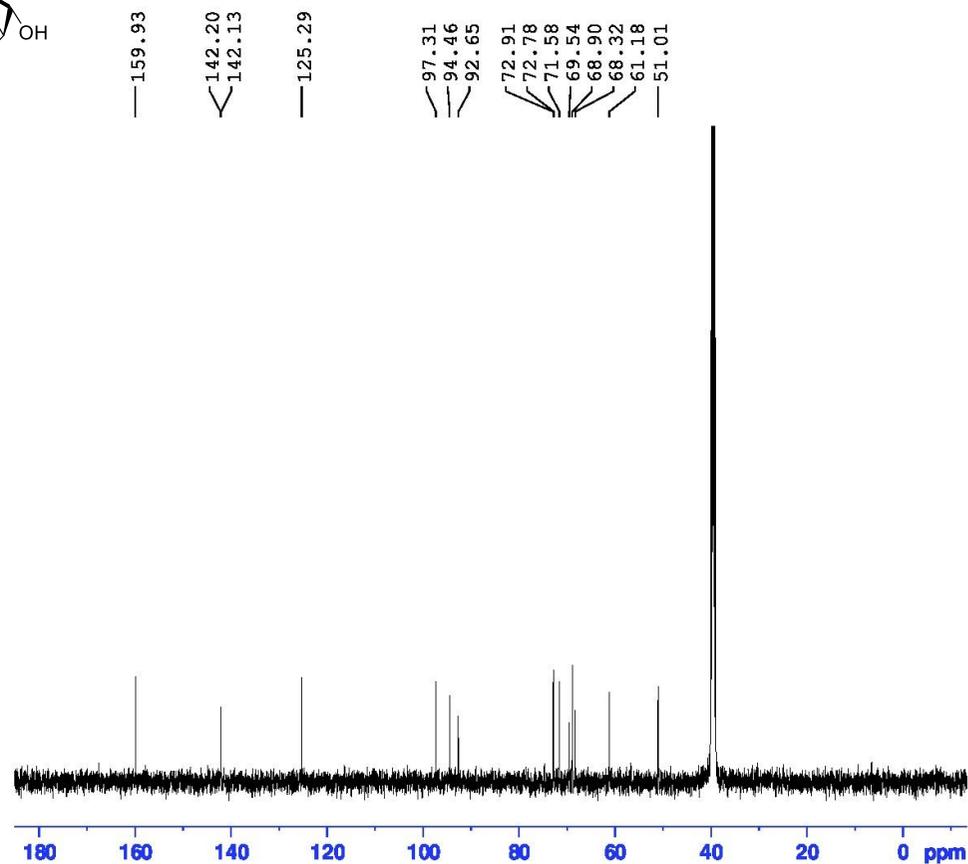
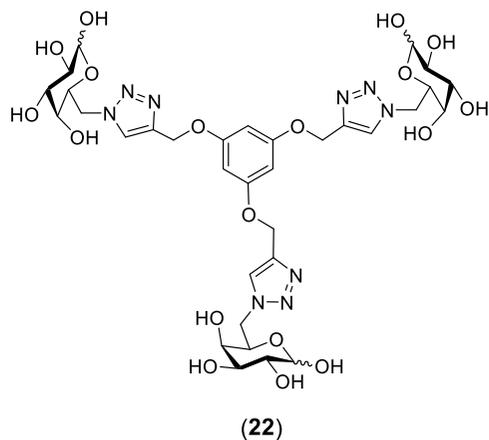
CHANNEL f1
NUC1 1H
P1 9.25 usec
PL1 -2.70 dB
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Appendix B7: ¹H NMR spectrum of compound (22).



Appendix B8: Selected areas ^1H NMR of compound (22).



Current Data Parameters
 NAME A26823
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameter
 Date_ 20120330
 Time 14.09
 INSTRUM DRX500
 PROBRD 5 mm DUL 13C-1
 PULPROG zgpg30
 TD 16384
 SOLVENT DMSO
 NS 1024
 DS 4
 SWH 30120.482 Hz
 FIDRES 1.838408 Hz
 AQ 0.2719744 sec
 RG 10321.3
 DW 16.600 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.69999999 sec
 d11 0.03000000 sec
 DELTA 0.59999996 sec
 TD0 1

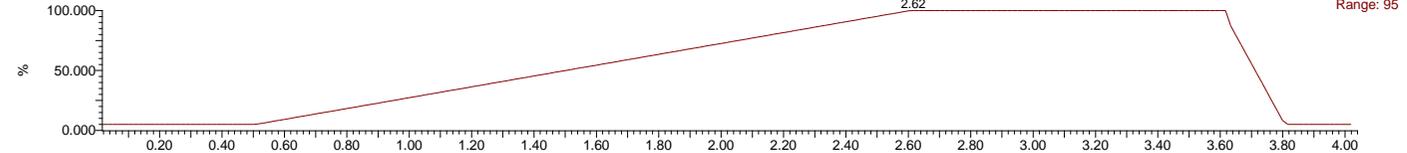
CHANNEL f1
 NUC1 13C
 P1 8.00 usec
 PL1 -2.30 dB
 SFO1 125.7703643 MHz

CHANNEL f2
 CPDPRG[2] waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -2.30 dB
 PL12 16.21 dB
 PL13 120.00 dB
 SFO2 500.1320005 MHz

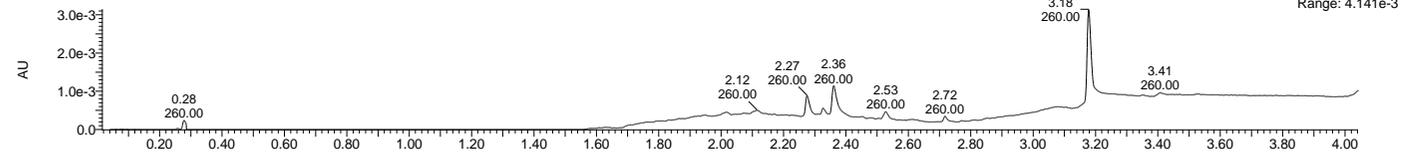
F2 - Processing parameters
 SI 32768
 SF 125.7578507 MHz
 WDW EM
 SSB 0
 LB 2.00 Hz
 GB 0
 PC 1.40

Appendix B9: ^{13}C NMR spectrum of compound (22).

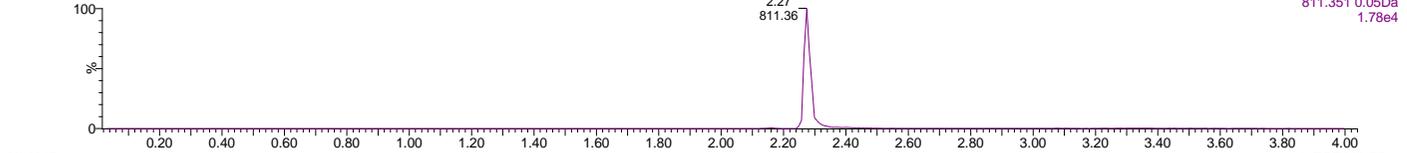
HK-18_11_13
ML2817



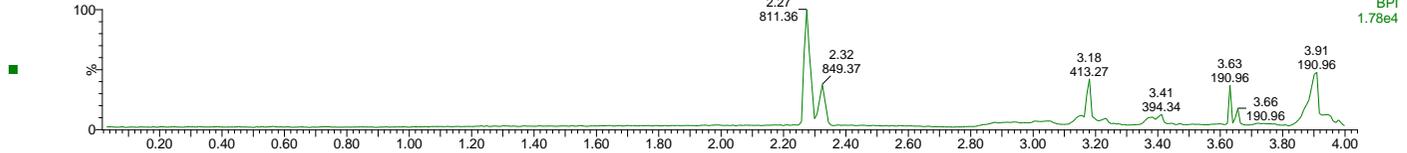
ML2817



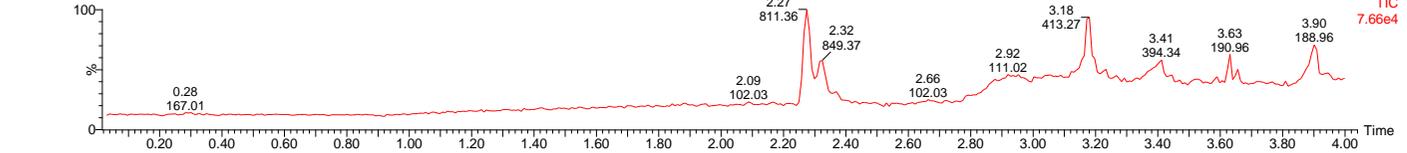
ML2817

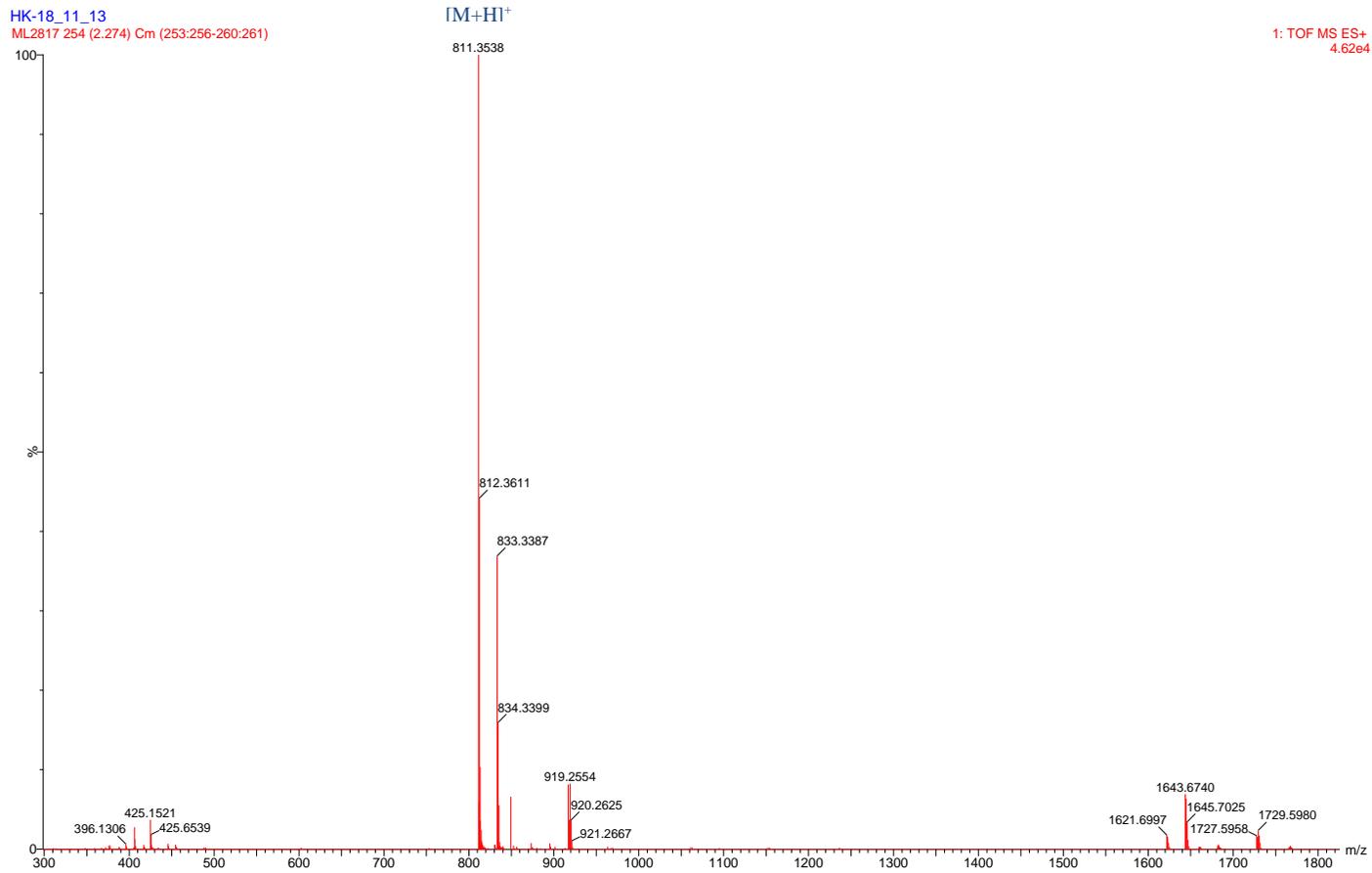


ML2817



ML2817



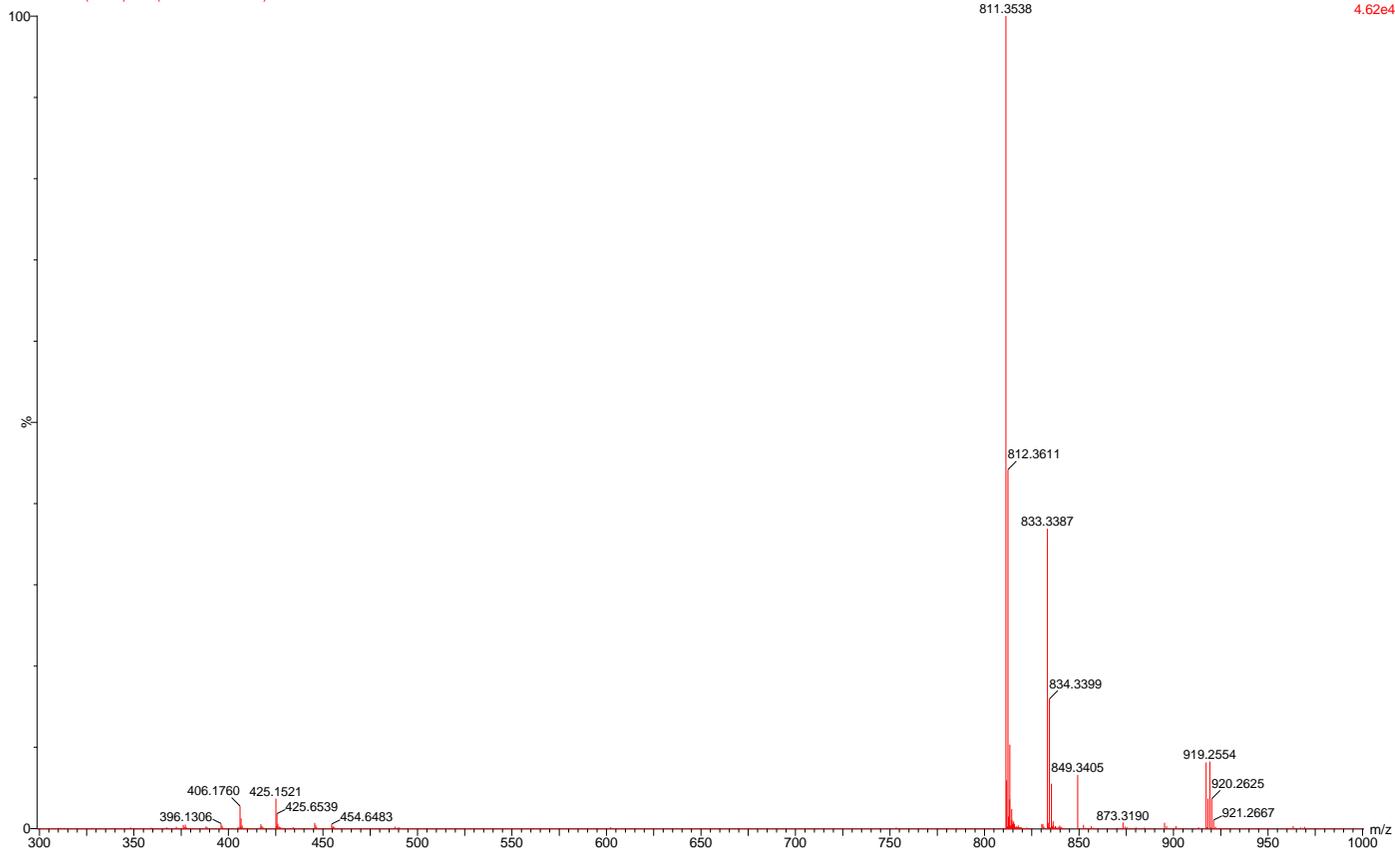


HK-18_11_13

ML2817 254 (2.274) Cm (253:256-260:261)

[M+H]⁺

1: TOF MS ES+
4.62e4



Elemental Composition Report: **HK-18/11/13**

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 80.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

1110 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 39-39 H: 0-180 N: 0-30 O: 0-40

Minimum:

-1.5

Maximum:

10.0

5.0

80.0

Mass

Calc. Mass

mDa

PPM

DBE

i-FIT

i-FIT (Norm)

Formula

811.3538

811.3514

2.4

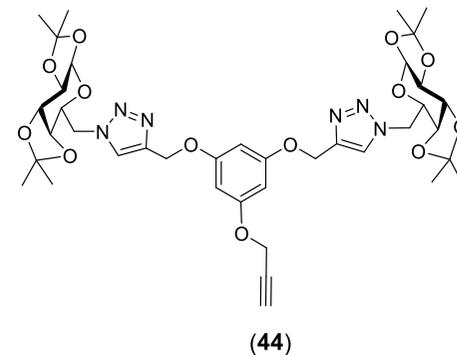
3.0

17.5

146.9

0.0

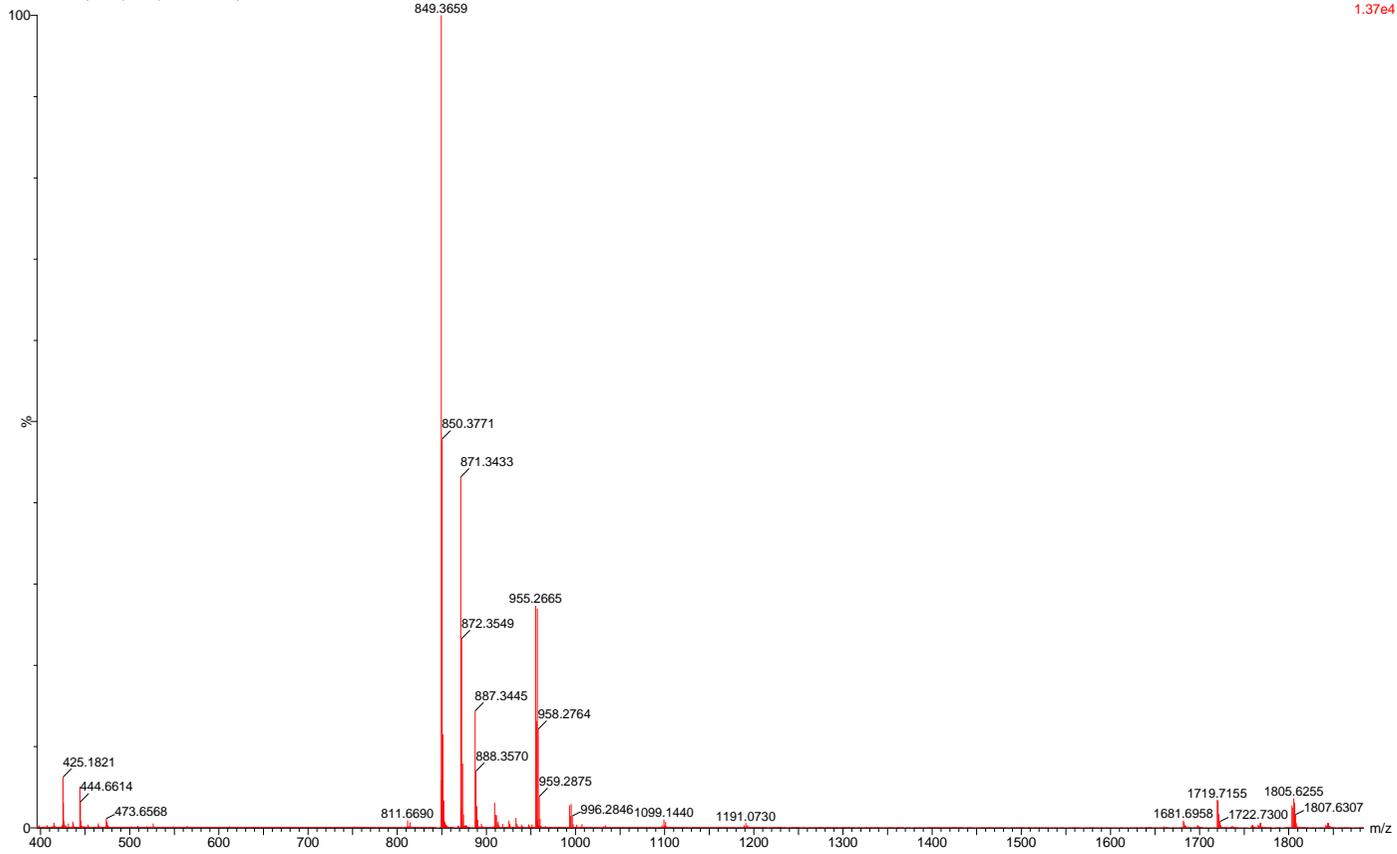
C₃₉ H₅₁ N₆ O₁₃



HK-18_11_13

ML2817 260 (2.324) Cm (258:261-256)

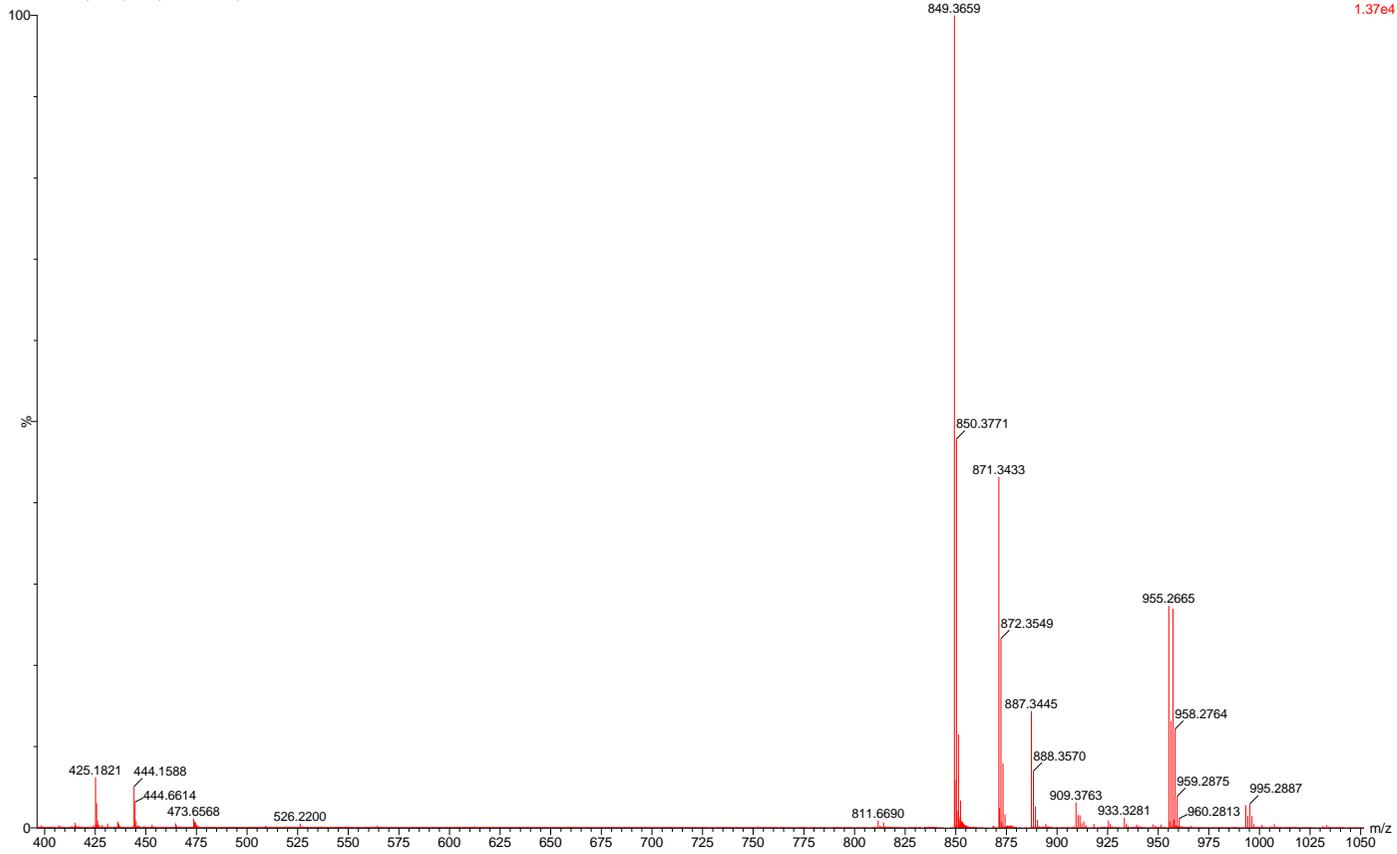
1: TOF MS ES+
1.37e4



HK-18_11_13

ML2817 260 (2.324) Cm (258:261-256)

1: TOF MS ES+
1.37e4



Elemental Composition Report: **HK-18/11/13**

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 80.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

1926 formula(e) evaluated with 6 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 39-45 H: 0-180 N: 0-30 O: 0-40

Minimum:

-1.5

Maximum:

10.0

5.0

80.0

Mass

Calc. Mass

mDa

PPM

DBE

i-FIT

i-FIT (Norm)

Formula

849.3659

849.3657

0.2

0.2

14.5

85.7

1.8

C41 H57 N2 O17

849.3657

0.2

0.2

25.5

84.9

0.9

C39 H45 N16 O7

849.3671

-1.2

-1.4

19.5

86.0

2.0

C42 H53 N6 O13

849.3671

-1.2

-1.4

30.5

85.8

1.8

C40 H41 N20 O3

849.3684

-2.5

-2.9

24.5

86.5

2.6

C43 H49 N10 O9

849.3697

-3.8

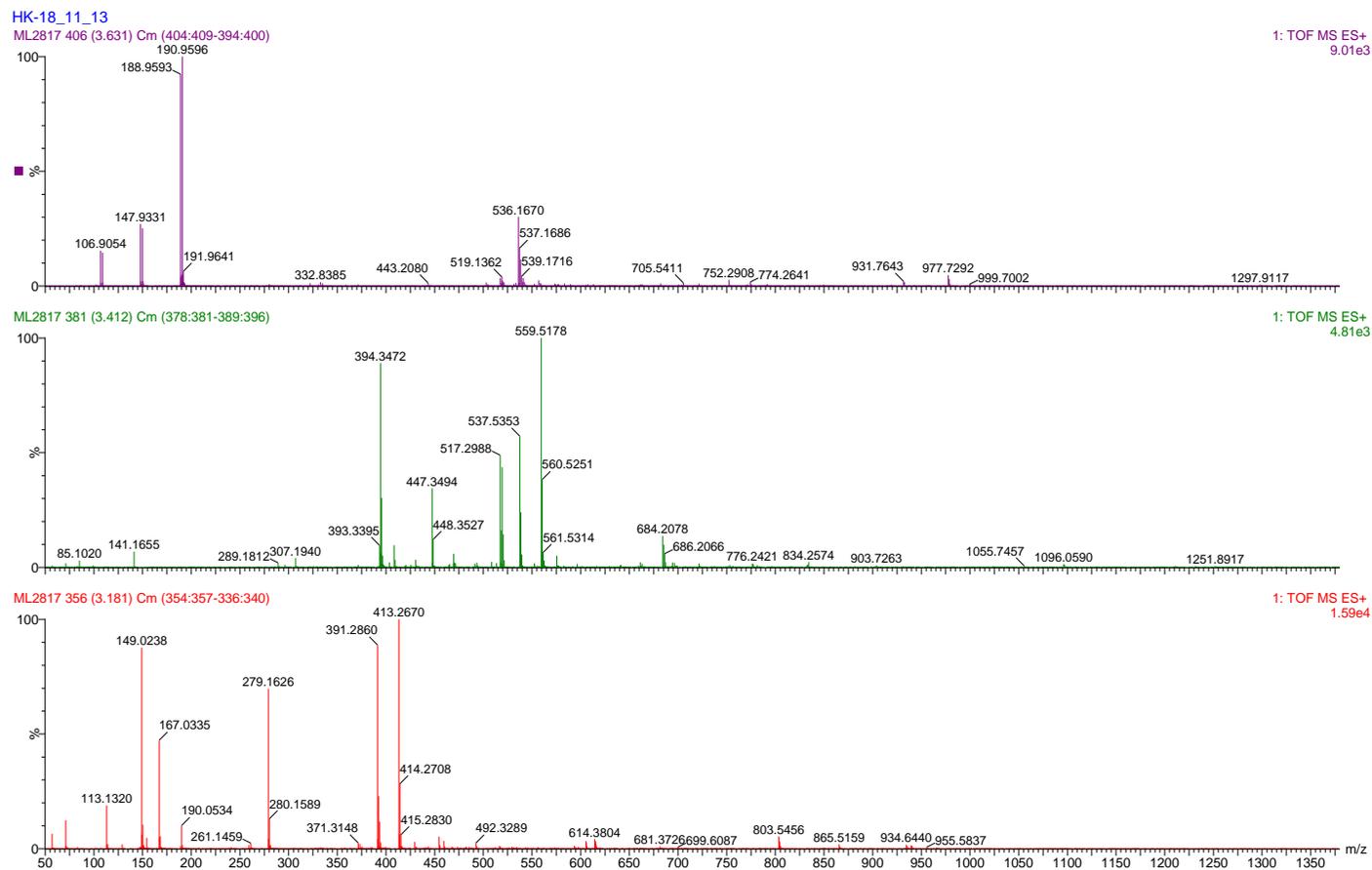
-4.5

29.5

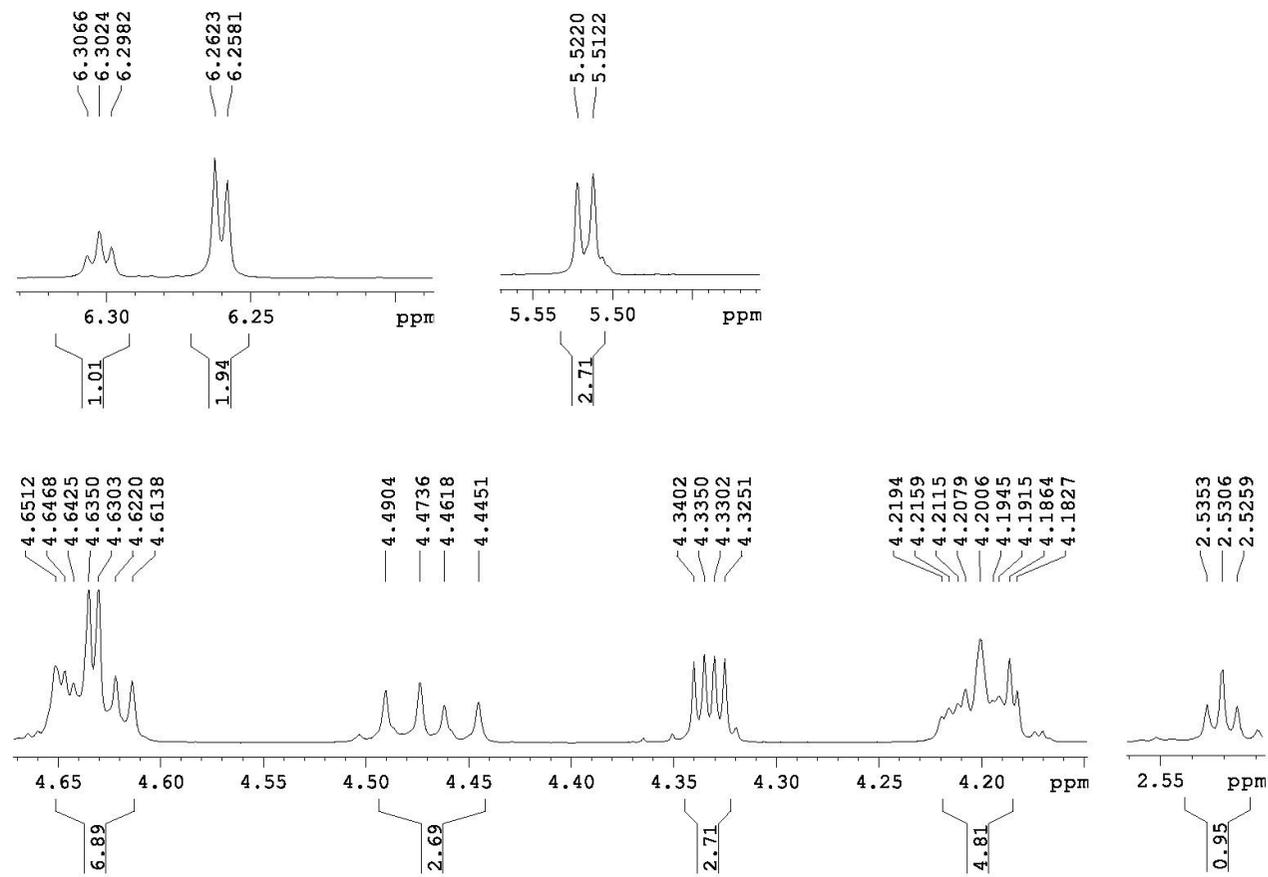
87.2

3.2

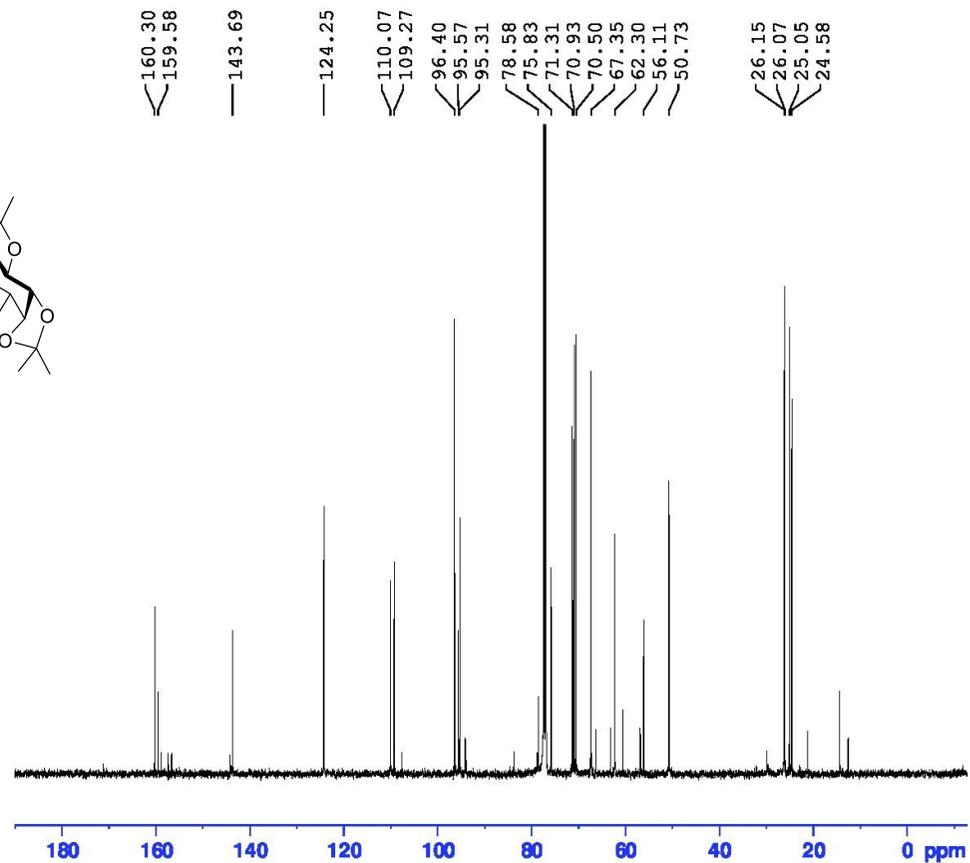
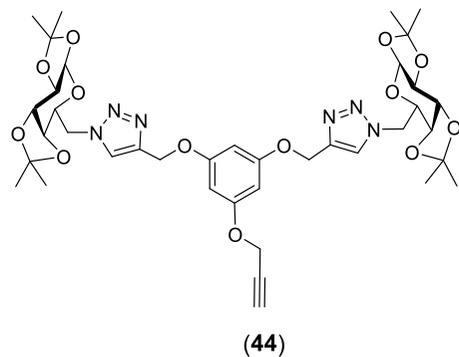
C44 H45 N14 O5



Appendix B10: HRMS (ESI) spectra of compound (44).



Appendix B12: Selected areas ¹H NMR of compound (44).



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NAME A36216
EXPNO 2
PROCNO 1

F2 - Acquisition Parameter
Date_ 20131208
Time 19.12
INSTRUM DRX500
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 16384
SOLVENT CDCl3
NS 8192
DS 4
SWH 30120.482 Hz
FIDRES 1.838408 Hz
AQ 0.2719744 sec
RG 11585.2
DW 16.600 usec
DE 6.00 usec
TE 300.0 K
D1 0.69999999 sec
d11 0.03000000 sec
DELTA 0.59999996 sec
TD0 1

CHANNEL f1
NUC1 13C
P1 20.00 usec
PL1 4.20 dB
SFO1 125.7703643 MHz

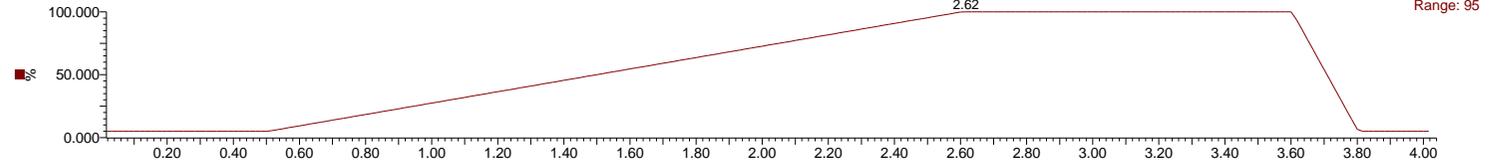
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CPDPRG[2] waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.30 dB
PL12 12.84 dB
PL13 120.00 dB
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577671 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

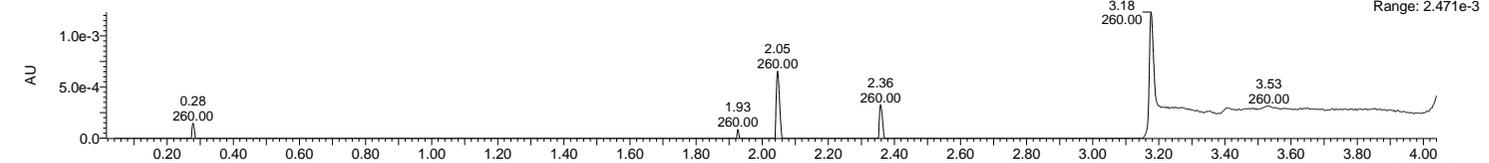
Appendix B13: ^{13}C NMR spectrum of compound (44).

HK-22_11_13

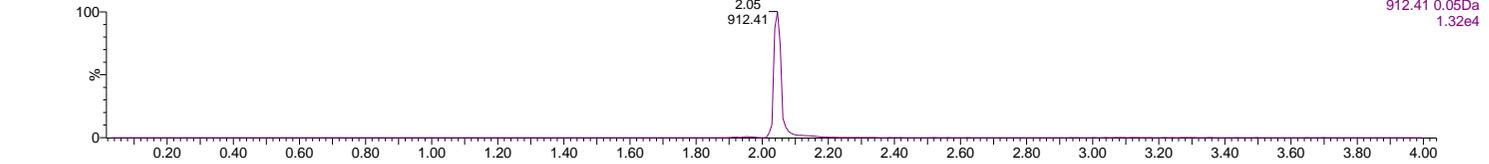
ML2818



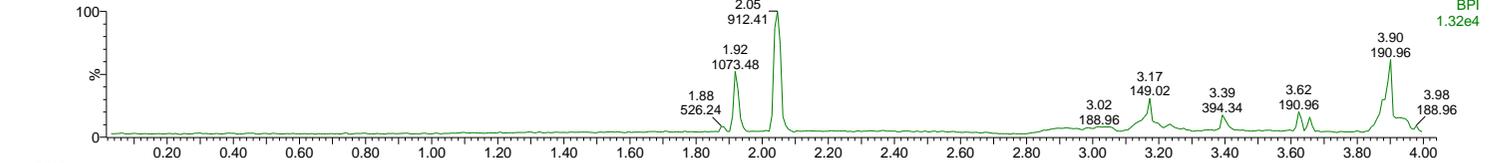
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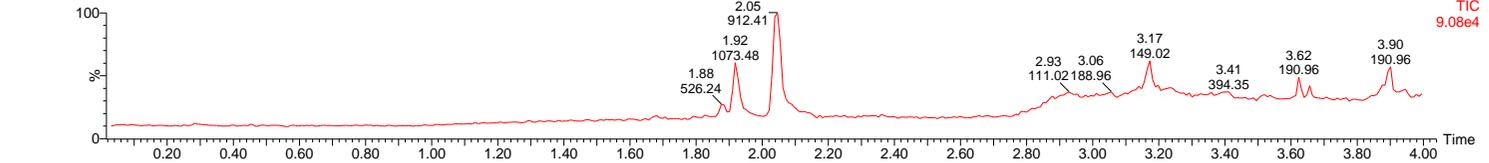
ML2818



ML2818



ML2818

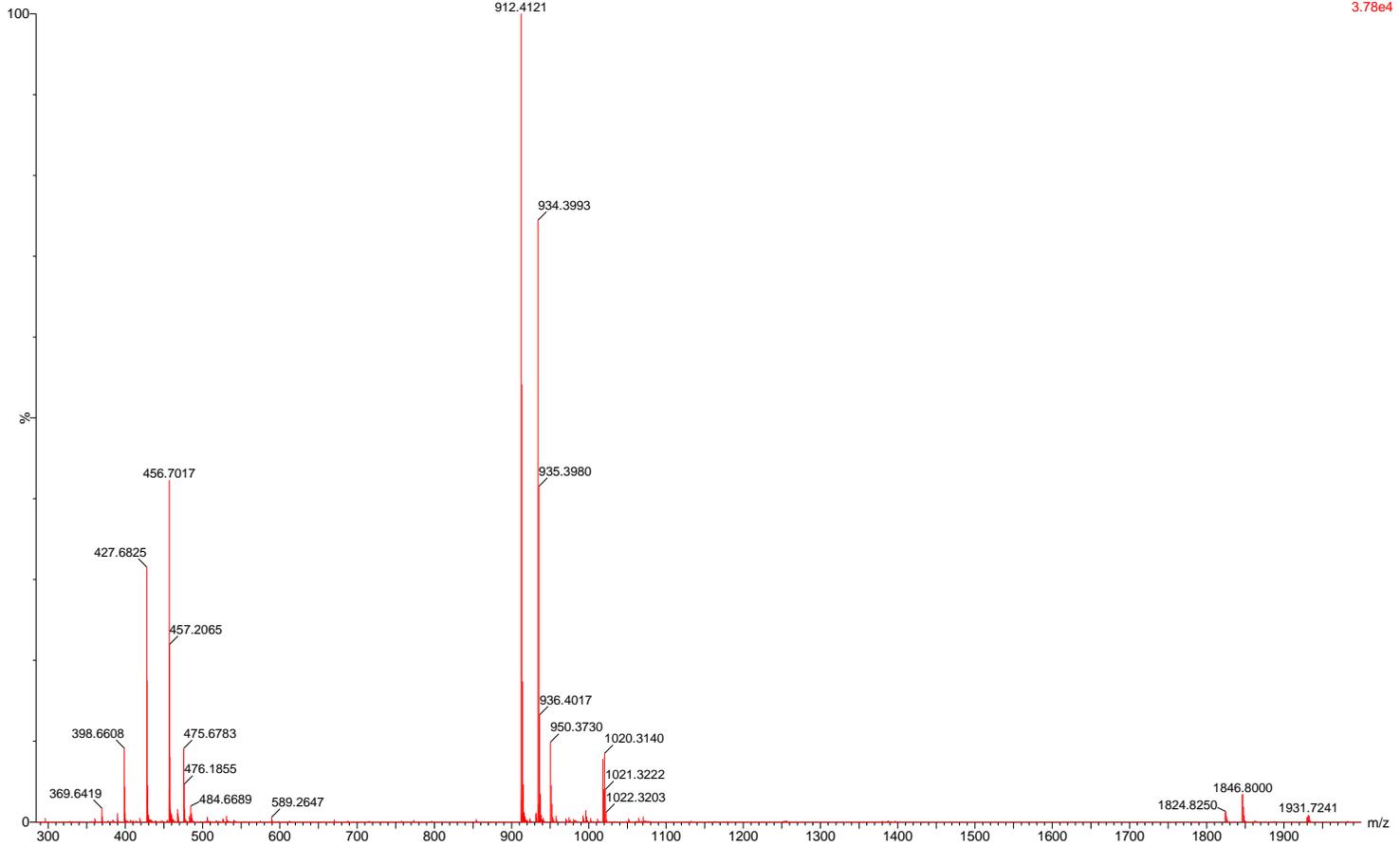


HK-22_11_13

ML2818 228 (2.046) Cm (226:230-261:269)

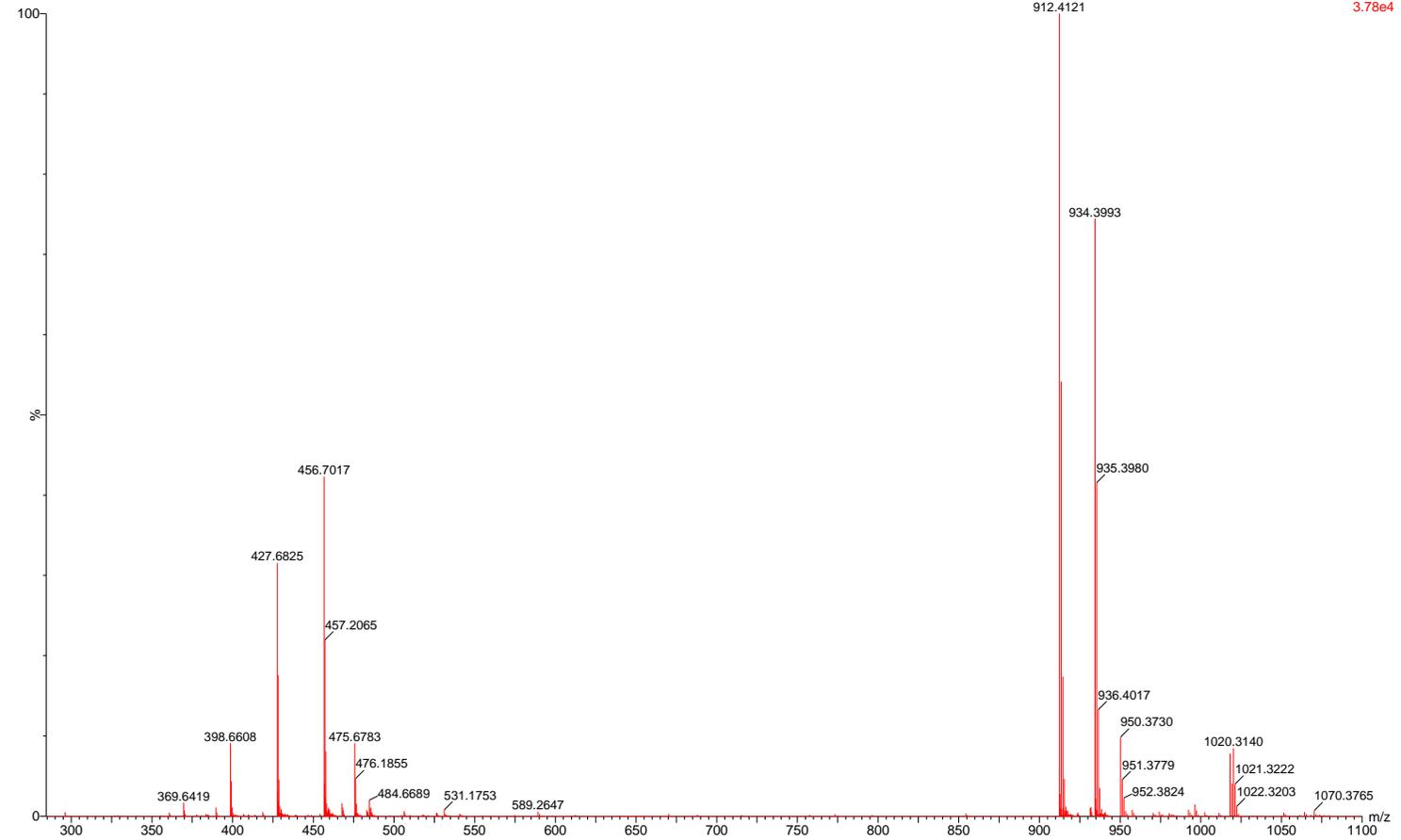
$[M+H]^+$

1: TOF MS ES+
3.78e4



HK-22_11_13

ML2818 228 (2.046) Cm (226:230-261:269)



Elemental Composition Report: **HK-22/11/13**

Single Mass Analysis

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 80.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

1201 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 42-42 H: 0-180 N: 0-30 O: 0-40

Minimum:

-1.5

Maximum:

10.0

10.0

80.0

Mass

Calc. Mass

mDa

PPM

DBE

i-FIT

i-FIT (Norm)

Formula

912.4121

912.4103

1.8

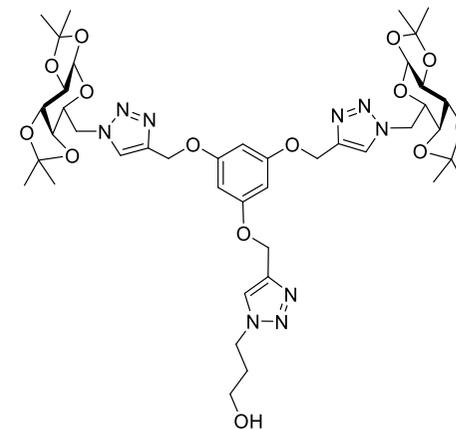
2.0

18.5

121.2

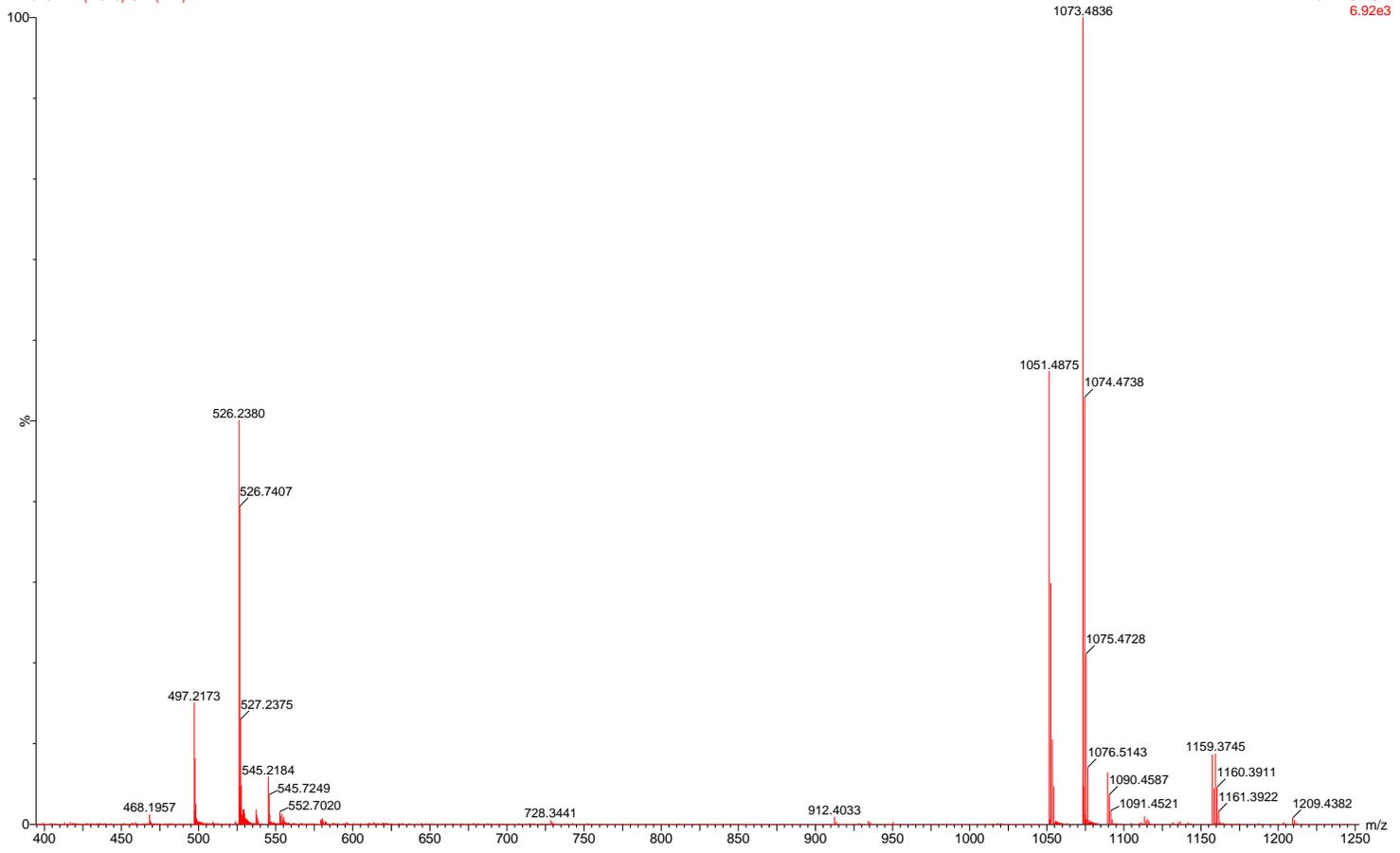
0.0

C₄₂ H₅₈ N₉ O₁₄



(47)

HK-22_11_13
ML2818 214 (1.918) Cm (214)



Elemental Composition Report: **HK-22/11/13**

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 80.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

2245 formula(e) evaluated with 6 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 40-45 H: 0-180 N: 0-30 O: 0-40

Minimum:

-1.5

Maximum:

10.0

5.0

80.0

Mass

Calc. Mass

mDa

PPM

DBE

i-FIT

i-FIT (Norm)

Formula

1051.4875

1051.4894

-1.9

-1.8

7.5

23.4

2.6

C40 H75 N8 O24

1051.4908

-3.3

-3.1

12.5

22.2

1.5

C41 H71 N12 O20

1051.4835

4.0

3.8

27.5

23.7

3.0

C45 H59 N22 O9

1051.4921

-4.6

-4.4

28.5

24.3

3.6

C40 H55 N30 O6

1051.4921

-4.6

-4.4

6.5

21.6

0.9

C44 H79 N2 O26

1051.4921

-4.6

-4.4

17.5

22.2

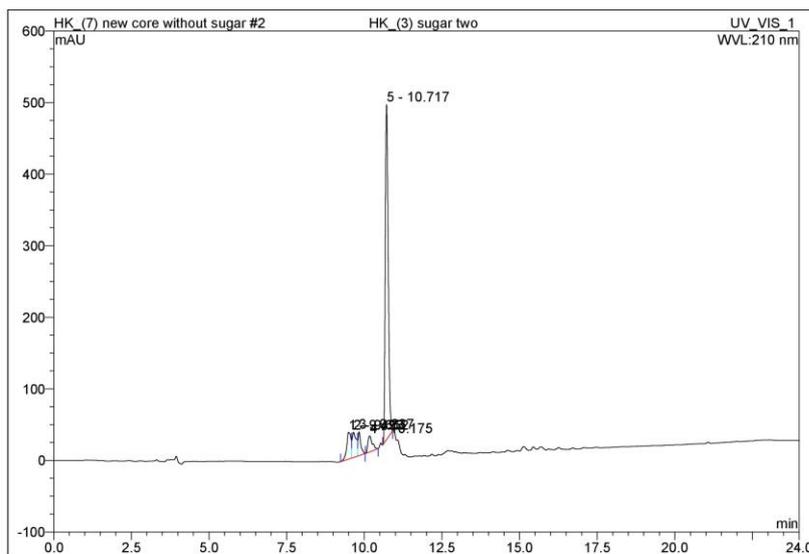
1.5

C42 H67 N16 O16

Appendix B14: HRMS (ESI) spectra of compound (47).

2 HK_(3) sugar two

Sample Name:	HK_(3) sugar two	Injection Volume:	20.0
Vial Number:	RA2	Channel:	UV_VIS_1
Sample Type:	unknown	Wavelength:	210
Control Program:	poly-p4 28min +230nm	Bandwidth:	10
Quantif. Method:	dna method	Dilution Factor:	1.0000
Recording Time:	11/12/2013 15:56	Sample Weight:	1.0000
Run Time (min):	24.00	Sample Amount:	1.0000



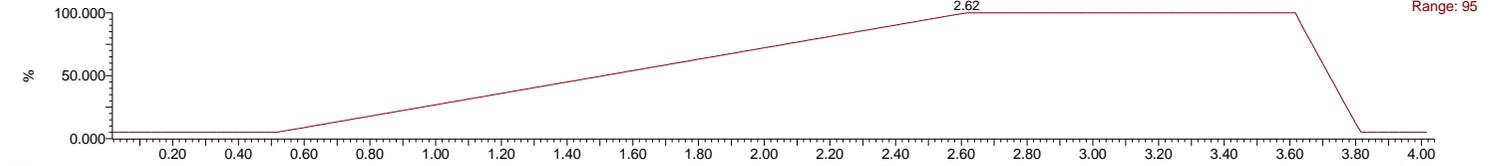
No.	Ret. Time min	Peak Name	Height mAU	Area mAU*min	Rel. Area %	Amount	Type
1	9.49	n.a.	37.374	5.825	8.29	n.a.	BM
2	9.65	n.a.	35.186	5.464	7.78	n.a.	M
3	9.84	n.a.	33.381	3.317	4.72	n.a.	MB
4	10.18	n.a.	22.278	3.643	5.19	n.a.	BMB
5	10.72	n.a.	468.329	51.989	74.02	n.a.	BMB
Total:			596.548	70.239	100.00	0.000	

default/Integration

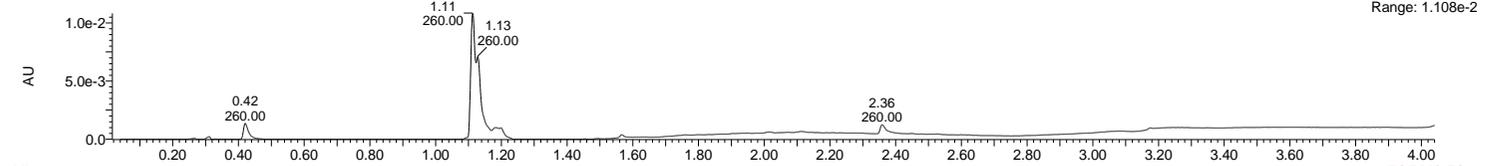
Chromeleon (c) Dionex 1996-2006
Version 6.80 SP4 Build 2361 (130805)*Appendix B15: HPL chromatogram of compound (23).*

HK-27_11_13

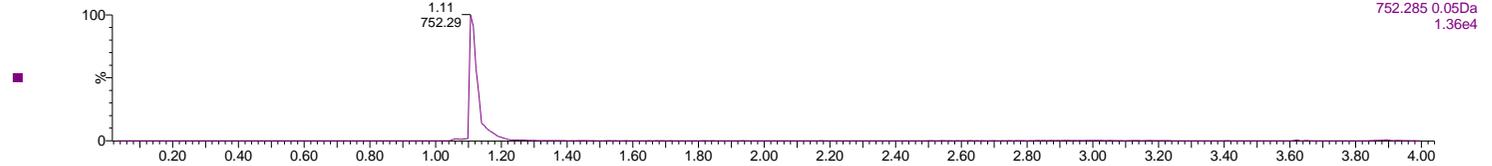
ML2812



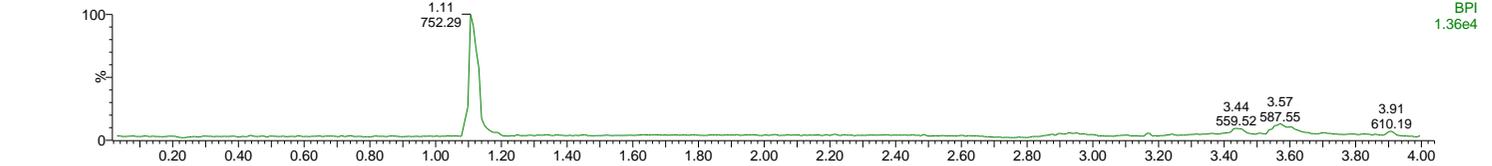
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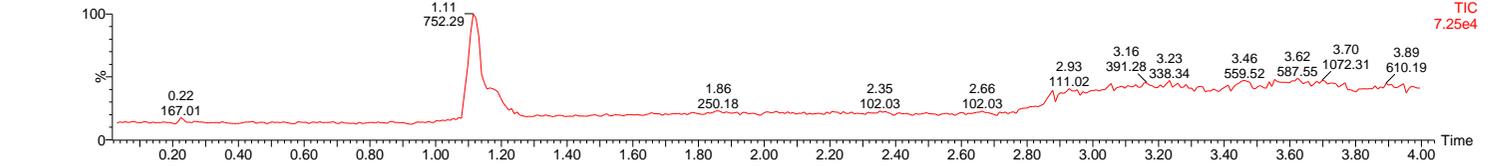
ML2812



ML2812



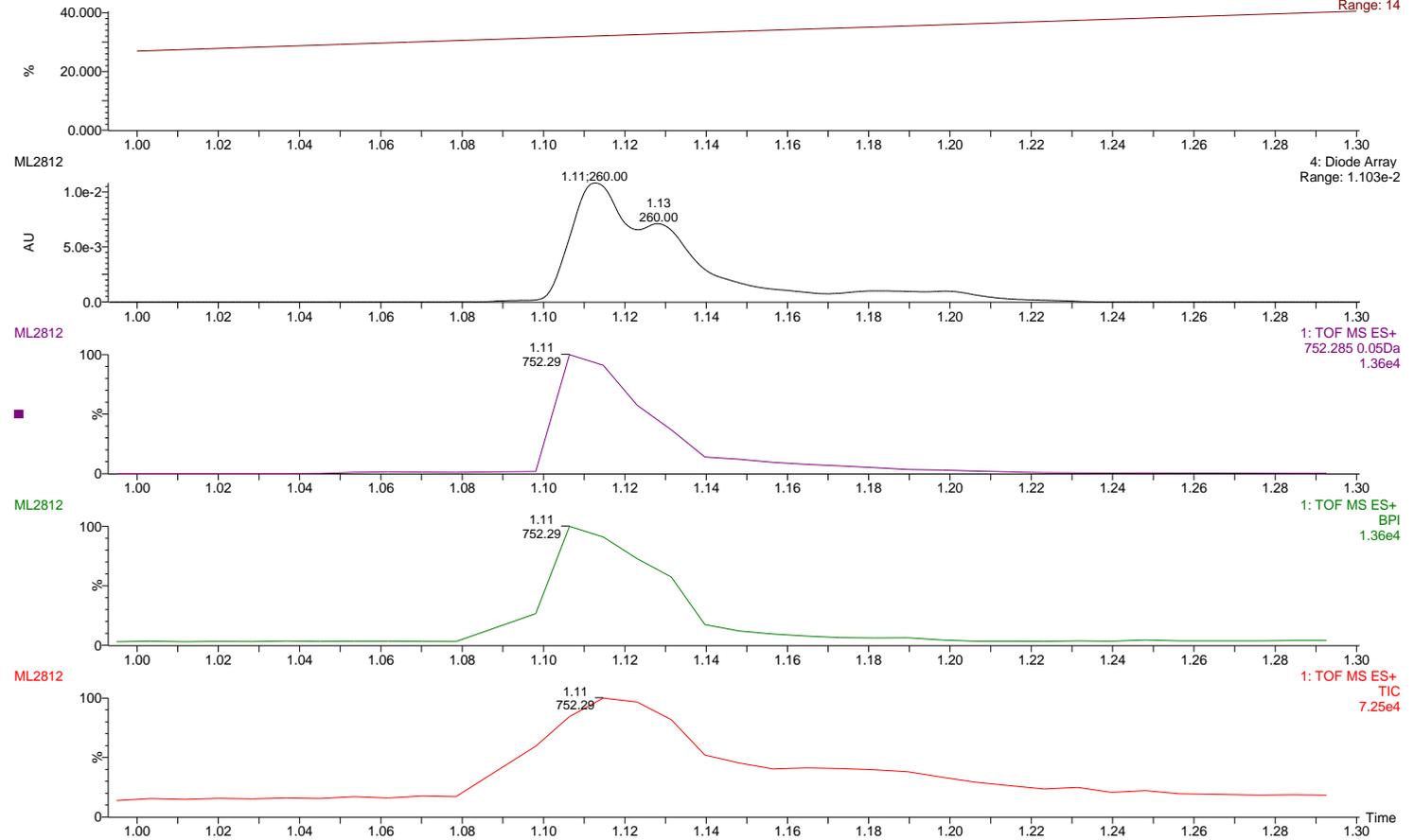
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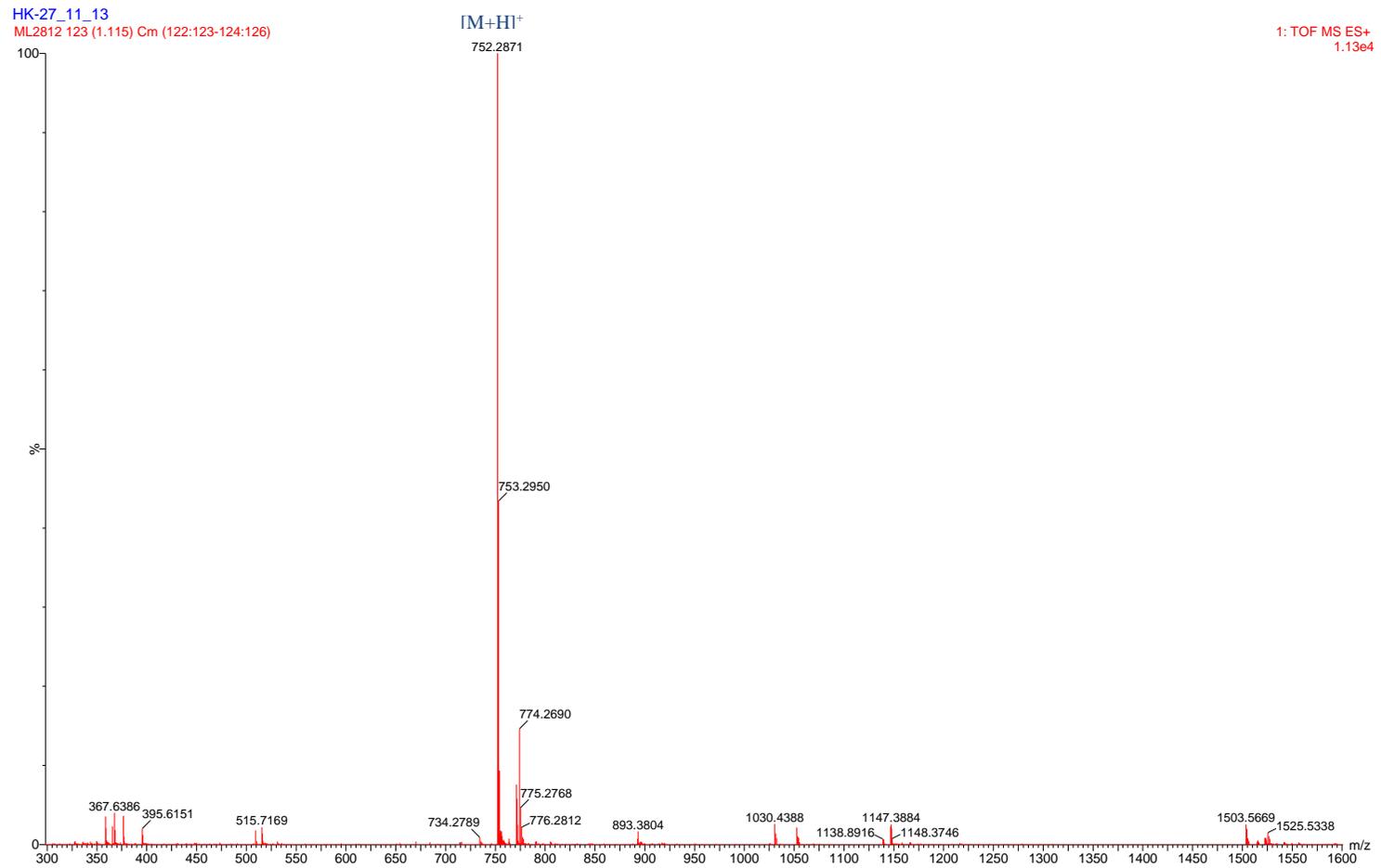


HK-27_11_13

ML2812

BSM Composition B
Range: 14





Elemental Composition Report: **HK-27/11/13**

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 80.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

1028 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 30-30 H: 0-180 N: 0-30 O: 0-40

Minimum:

-1.5

Maximum:

10.0

5.0

80.0

Mass

Calc. Mass

mDa

PPM

DBE

i-FIT

i-FIT (Norm)

Formula

752.2871

752.2851

2.0

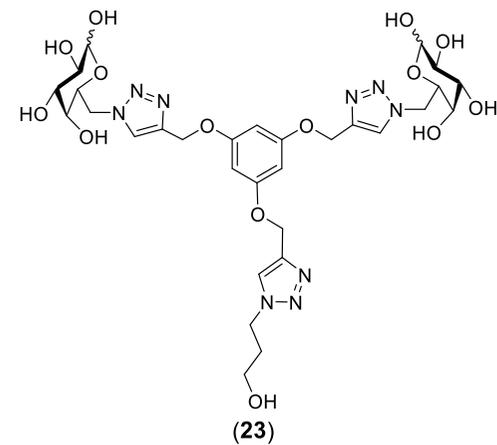
2.7

14.5

87.6

0.0

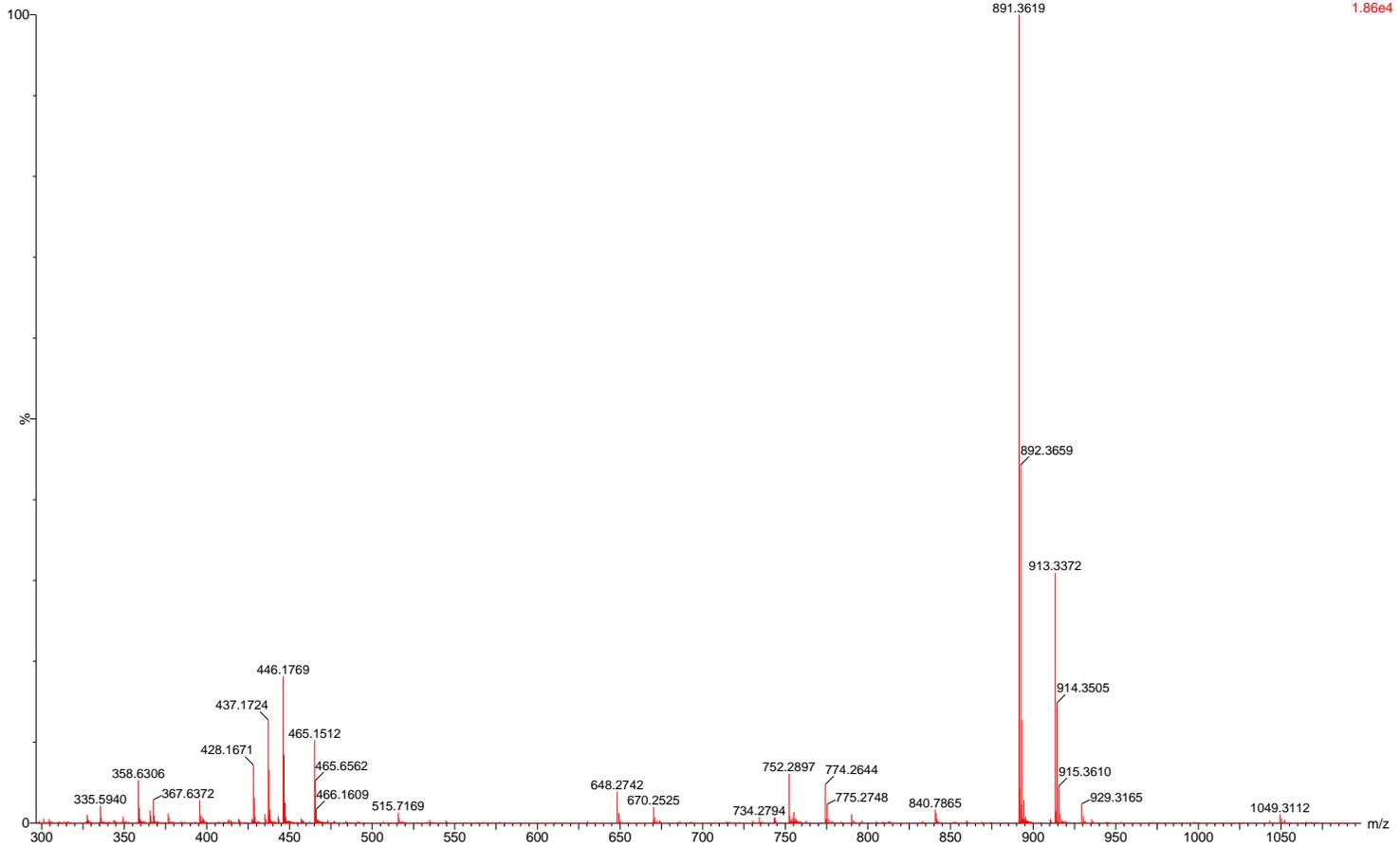
C₃₀ H₄₂ N₉ O₁₄



HK-27_11_13

ML2812.124 (1.123) Cm (124:126-122)

1: TOF MS ES+
1.86e4



Elemental Composition Report: **HK-27/11/13**

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 80.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

2729 formula(e) evaluated with 11 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 35-45 H: 0-180 N: 0-30 O: 0-40

Minimum:

-1.5

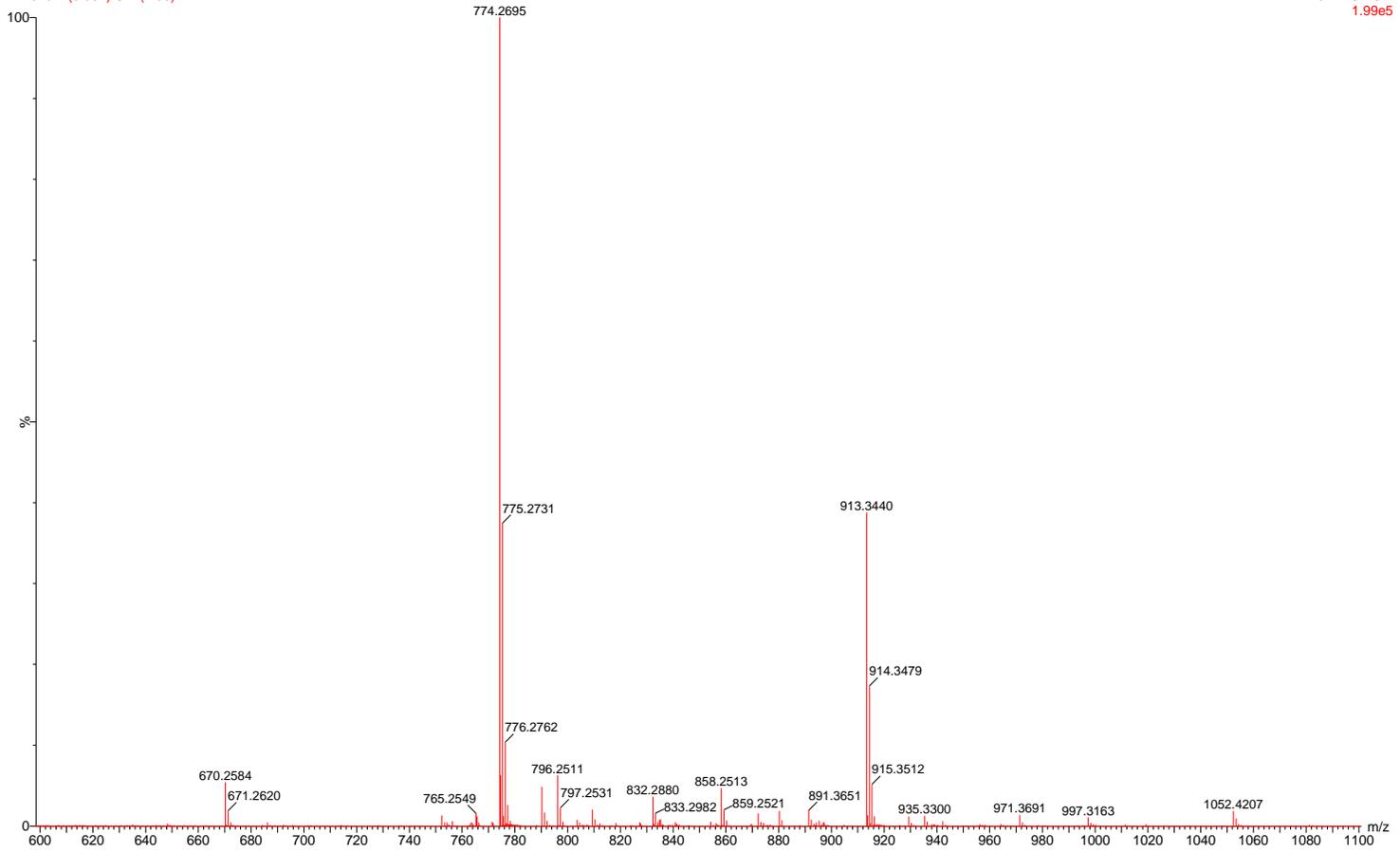
Maximum:

10.0 5.0 80.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
891.3619	891.3624	-0.5	-0.6	27.5	93.4	5.2	C38 H43 N20 O7
	891.3624	-0.5	-0.6	16.5	92.8	4.7	C40 H55 N6 O17
	891.3610	0.9	1.0	22.5	91.7	3.6	C37 H47 N16 O11
	891.3610	0.9	1.0	33.5	93.6	5.5	C35 H35 N30 O
	891.3610	0.9	1.0	11.5	91.5	3.3	C39 H59 N2 O21
	891.3637	-1.8	-2.0	21.5	94.5	6.4	C41 H51 N10 O13
	891.3637	-1.8	-2.0	32.5	94.9	6.7	C39 H39 N24 O3
	891.3597	2.2	2.5	17.5	89.1	1.0	C36 H51 N12 O15
	891.3650	-3.1	-3.5	26.5	96.2	8.1	C42 H47 N14 O9
	891.3651	-3.2	-3.6	15.5	96.4	8.2	C44 H59 O19
	891.3583	3.6	4.0	12.5	88.8	0.6	C35 H55 N8 O19

HK-27_11_13
ML2810 1 (0.032) Cm (1:56)

1: TOF MS ES+
1.99e5



Elemental Composition Report: **HK-27/11/13**

Single Mass Analysis

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 80.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

1029 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 30-30 H: 0-180 N: 0-30 O: 0-40 Na: 1-1

Minimum:

-1.5

Maximum:

10.0

10.0

80.0

Mass

Calc. Mass

mDa

PPM

DBE

i-FIT

i-FIT (Norm)

Formula

774.2695

774.2671

2.4

3.1

14.5

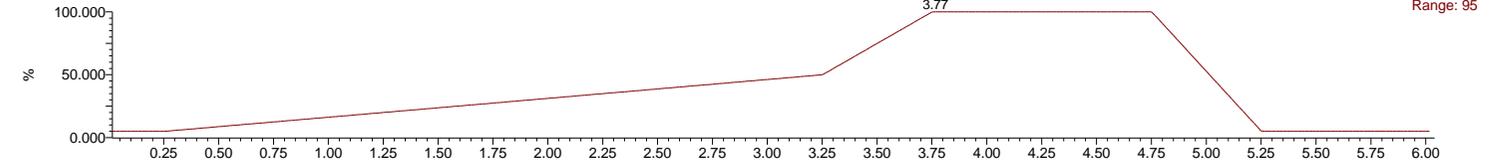
260.1

0.0

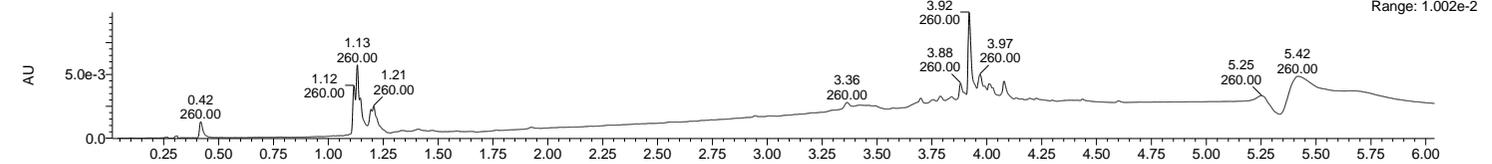
C30 H41 N9 O14 Na

HK-27_11_13

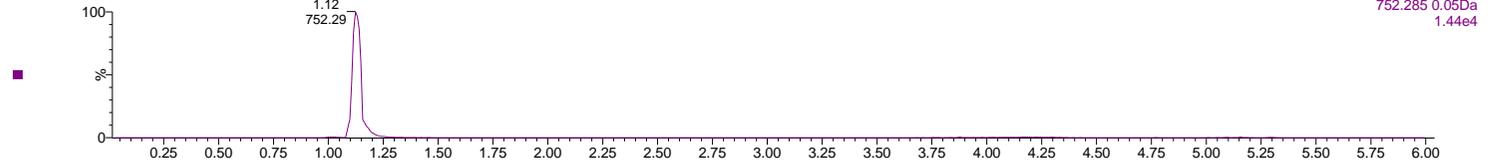
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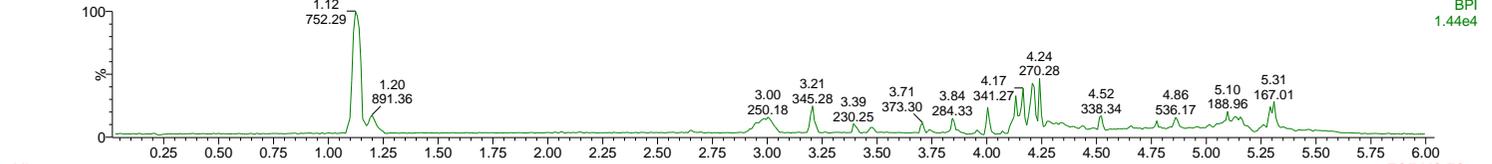
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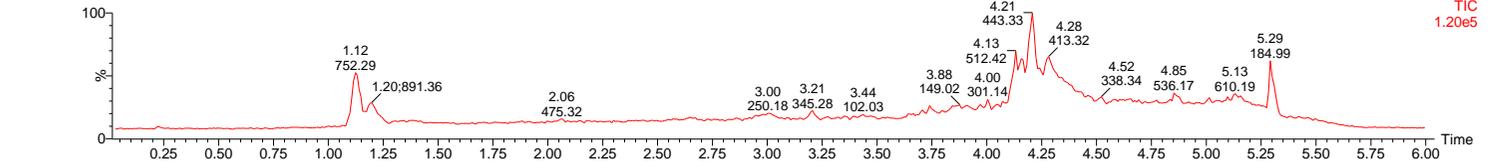
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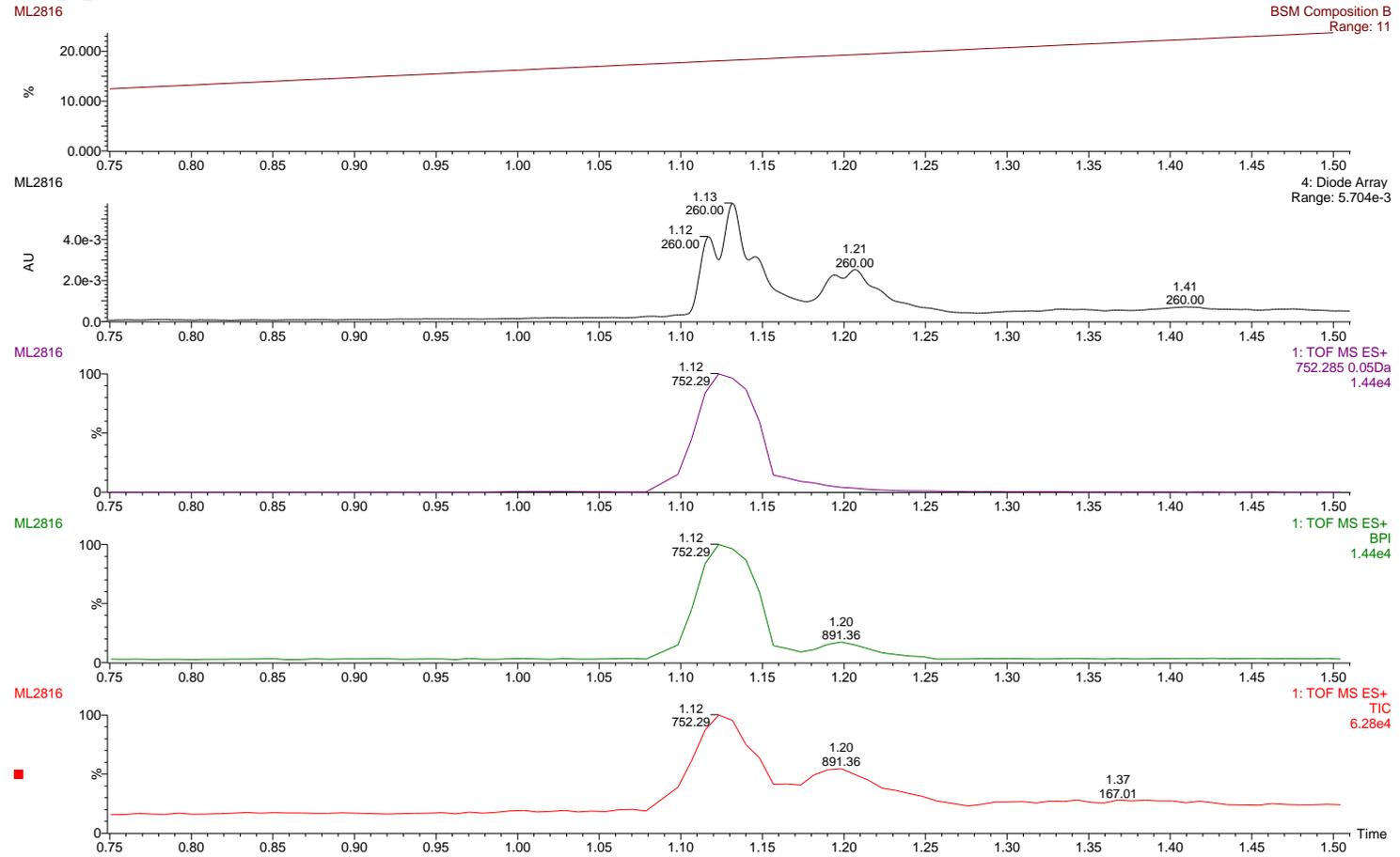
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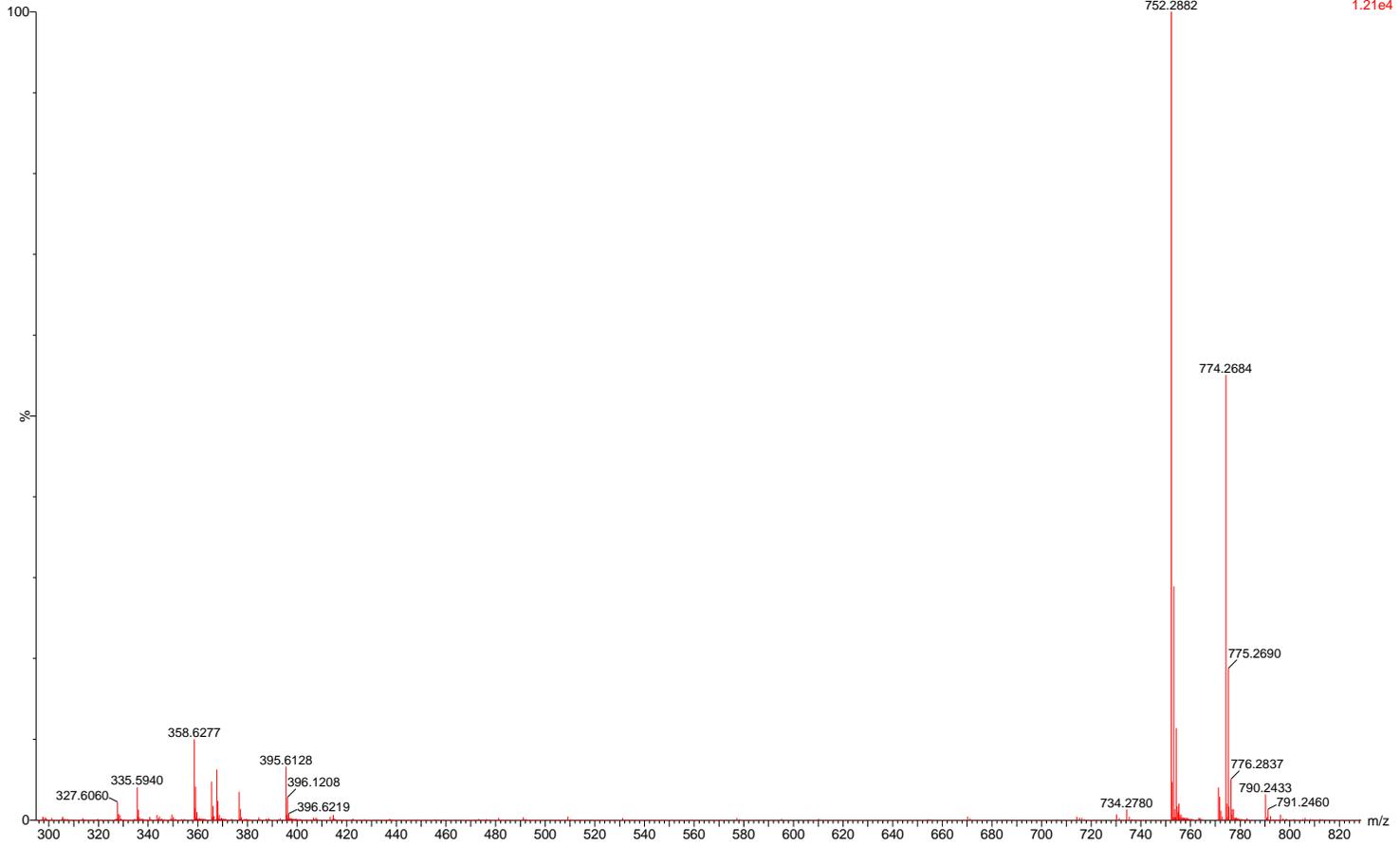
ML2816



HK-27_11_13
ML2816



HK-27_11_13
ML2816 123 (1.115)



Elemental Composition Report: **HK-27/11/13**

Single Mass Analysis

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 80.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

1028 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 30-30 H: 0-180 N: 0-30 O: 0-40

Minimum:

-1.5

Maximum:

10.0

10.0

80.0

Mass

Calc. Mass

mDa

PPM

DBE

i-FIT

i-FIT (Norm)

Formula

752.2882

752.2851

3.1

4.1

14.5

73.7

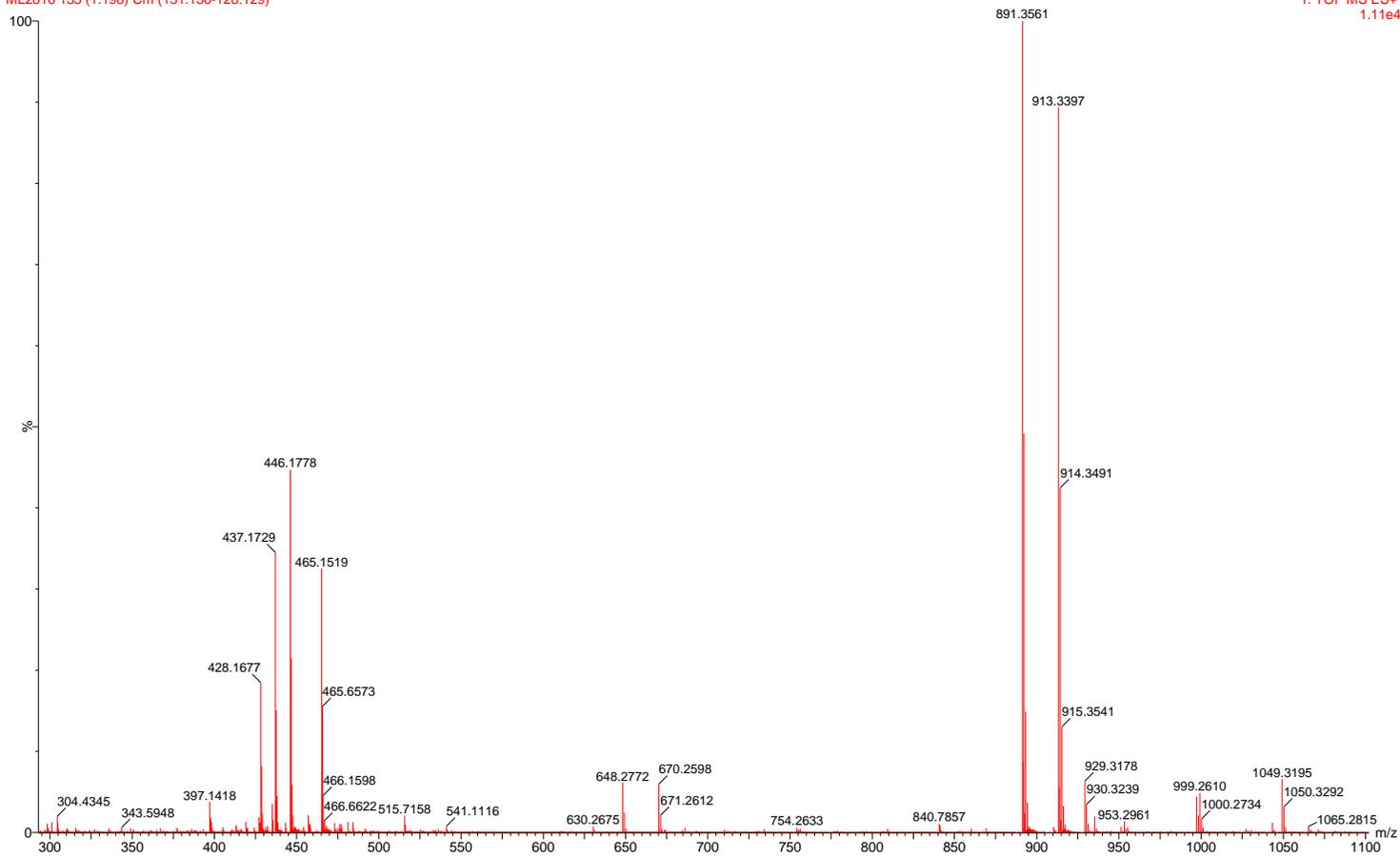
0.0

C30 H42 N9 O14

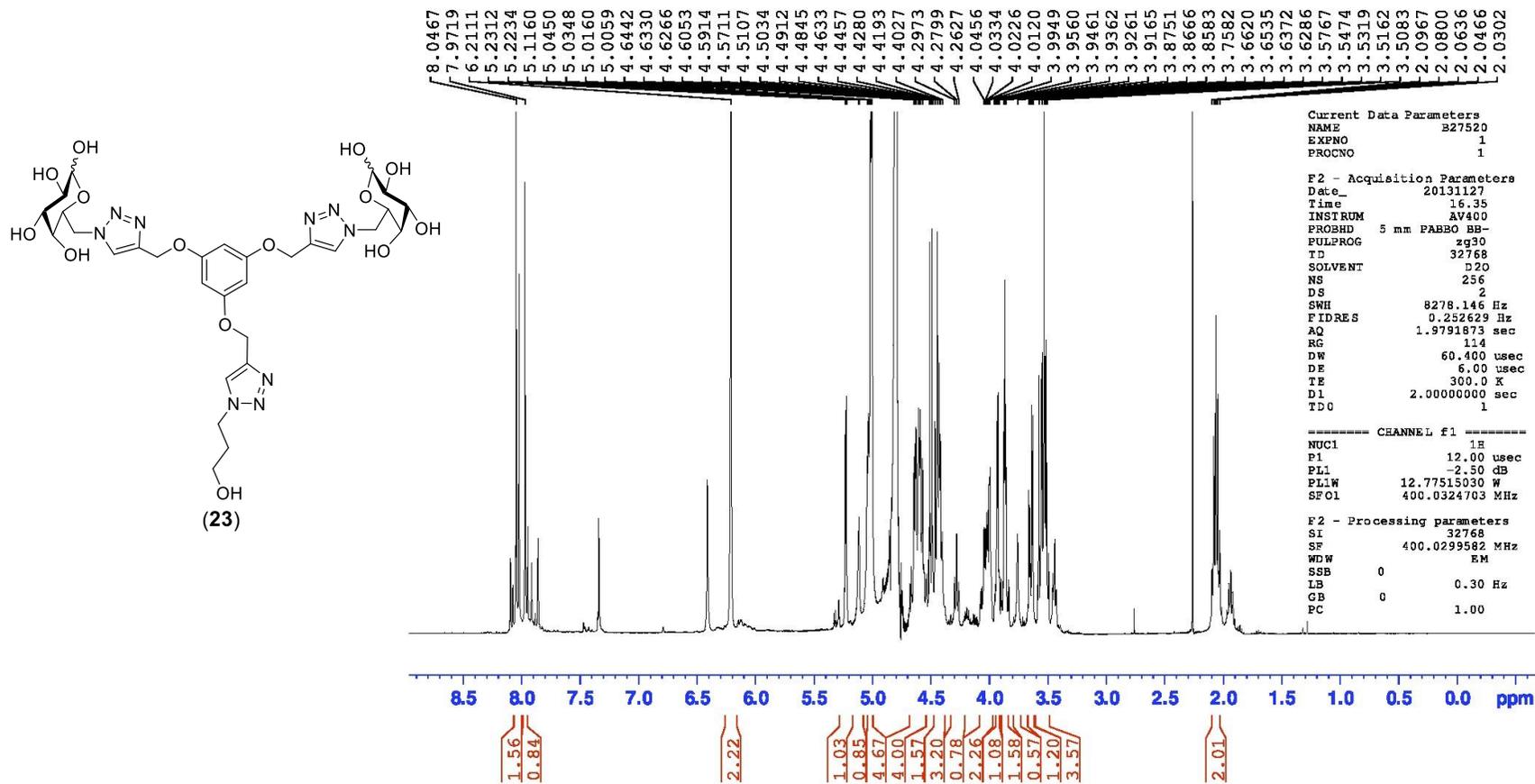
HK-27_11_13

ML2816 133 (1.198) Cm (131:136-128:129)

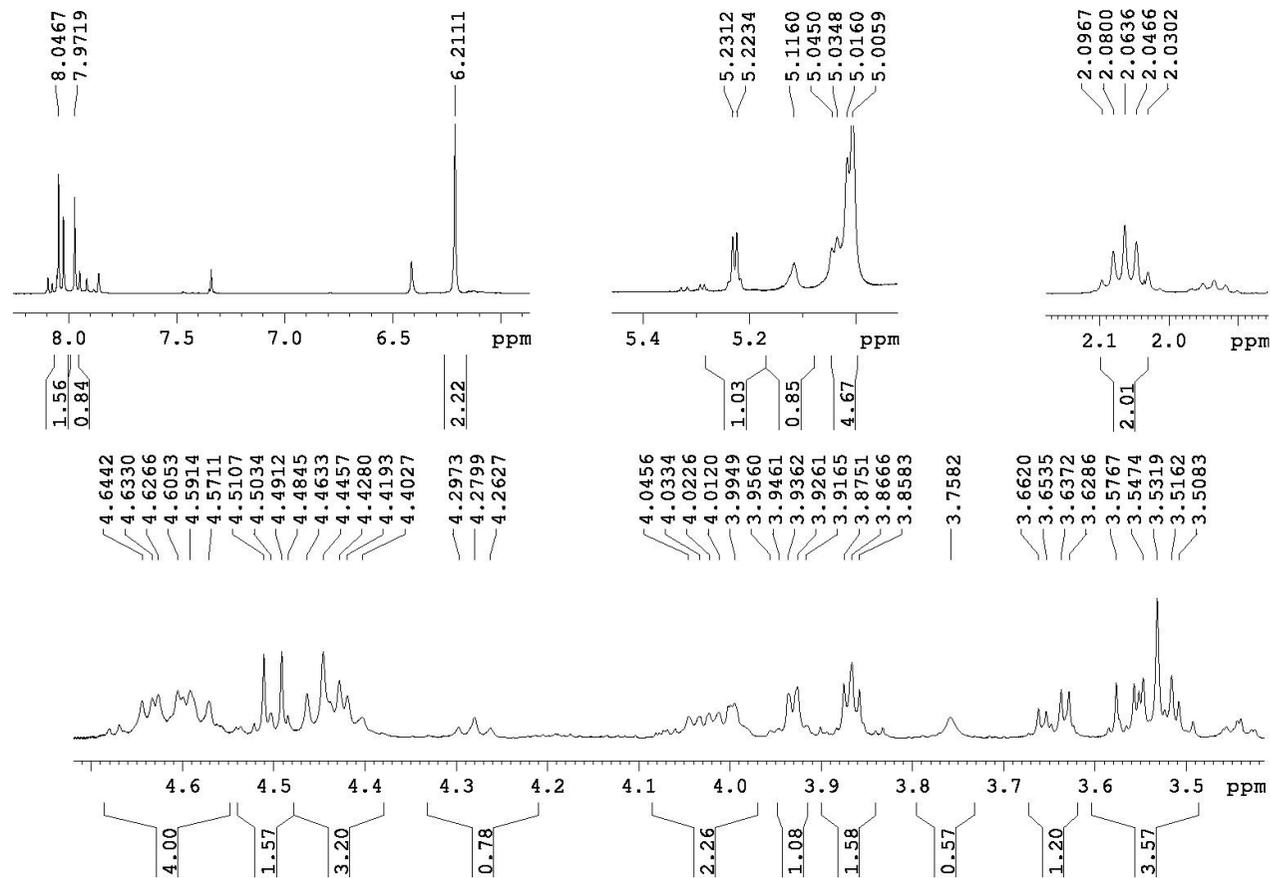
1: TOF MS ES+
1.11e4



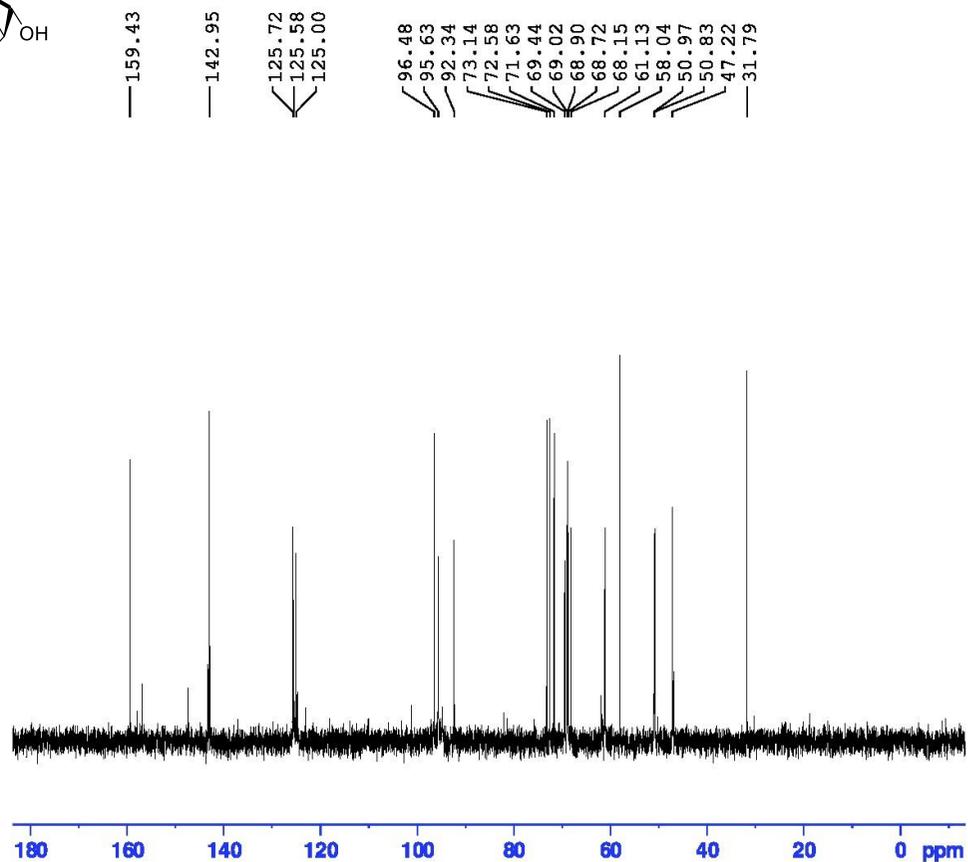
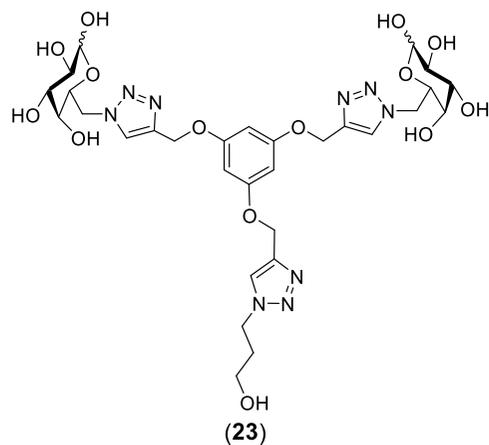
Appendix B16: HRMS (ESI) spectra of compound (23).



Appendix B17: ¹H NMR spectrum of compound (23).



Appendix B18: Selected areas ¹H NMR of compound (23).



```

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PROCNO    1

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NS        8192
DS        2
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FIDRES    1.463673 Hz
AQ        0.3416064 sec
RG        18390.4
DW        20.850 usec
DE        20.00 usec
TE        300.0 K
D1        0.69999999 sec
D11       0.03000000 sec
TD0       1

===== CHANNEL f1 =====
NUC1      13C
P1        8.00 usec
PL1       -2.80 dB
PL1W      57.91424179 W
SFO1      100.5976818 MHz

===== CHANNEL f2 =====
CPDPRG [2] waltz16
NUC2      1H
PCPD2     90.00 usec
PL2       -3.20 dB
PL12      14.30 dB
PL13      15.30 dB
PL2W      15.00949383 W
PL12W     0.26691073 W
PL13W     0.21201472 W
SFO2      400.0316001 MHz

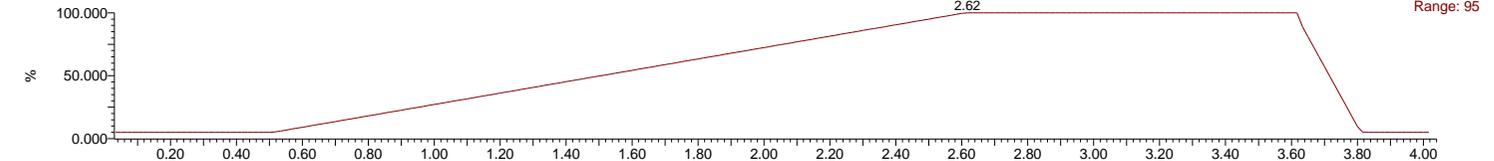
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SSB       0
LB        1.00 Hz
GB        0
PC        1.40

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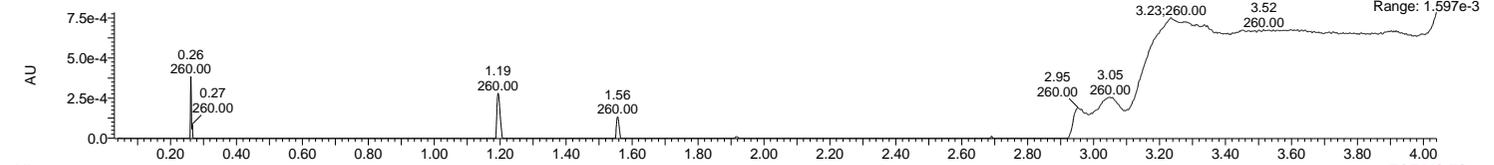
Appendix B19: ^{13}C NMR spectrum of compound (23).

AJF_HK_03

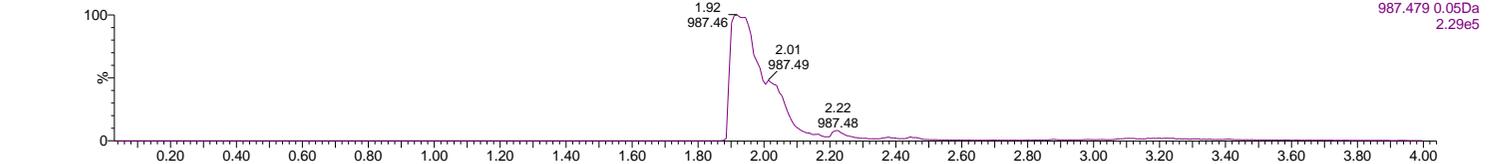
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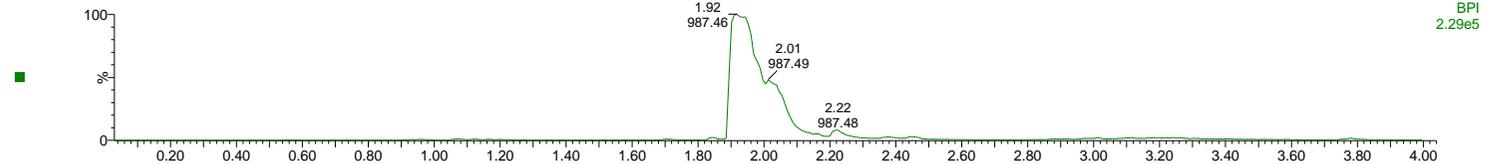
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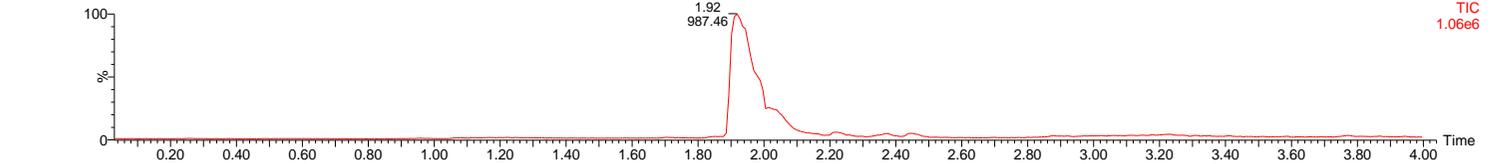
ML0592



ML0592



ML0592





Elemental Composition Report: **AJF-HK-03**

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

670 formula(e) evaluated with 1 results within limits (up to 20 closest results for each mass)

Elements Used:

C: 45-45 H: 0-150 N: 0-25 O: 0-25

Minimum:

-1.5

Maximum:

10.0

5.0

50.0

Mass

Calc. Mass

mDa

PPM

DBE

i-FIT

i-FIT (Norm)

Formula

987.4778

987.4787

-0.9

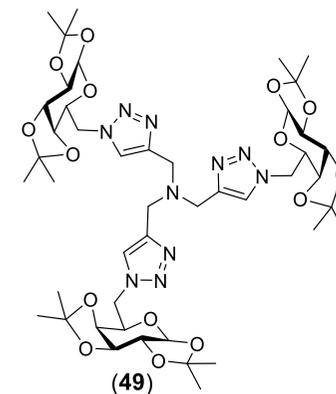
-0.9

17.5

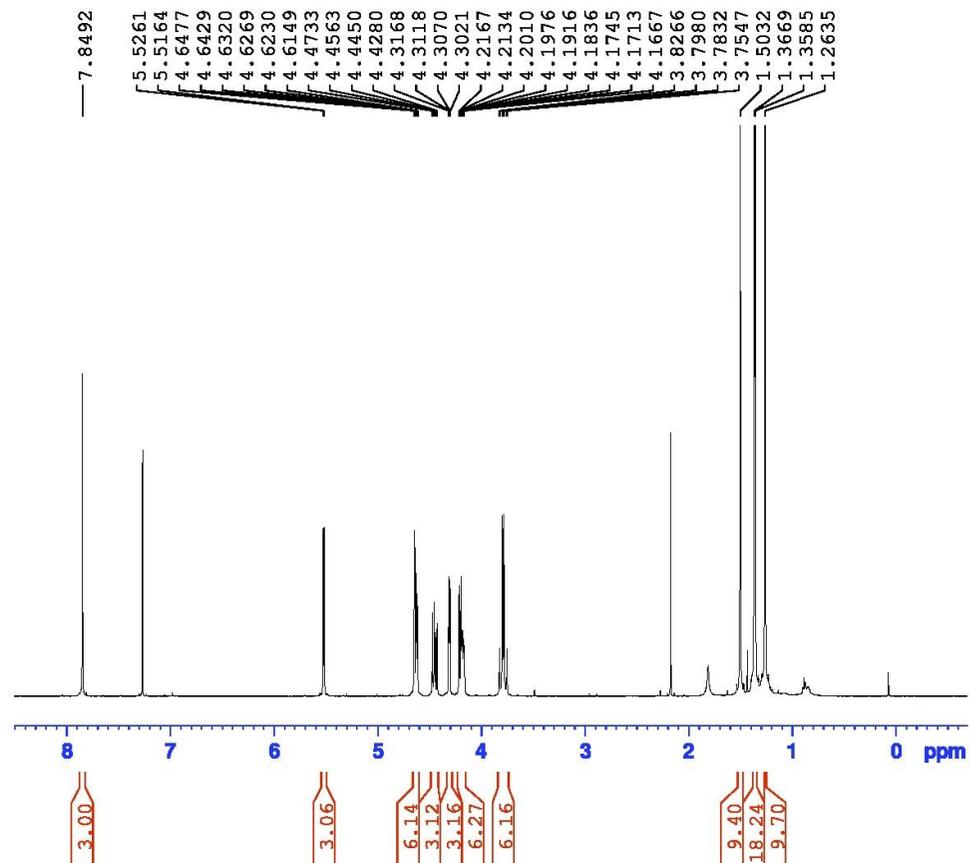
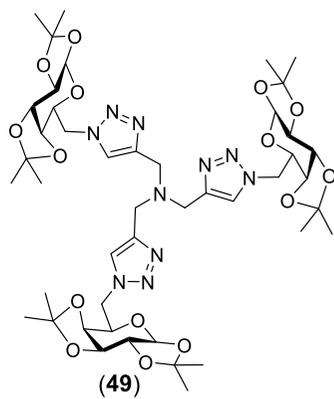
114.9

0.0

C₄₅ H₆₇ N₁₀ O₁₅



Appendix B20: HRMS (ESI) spectra of compound (49).



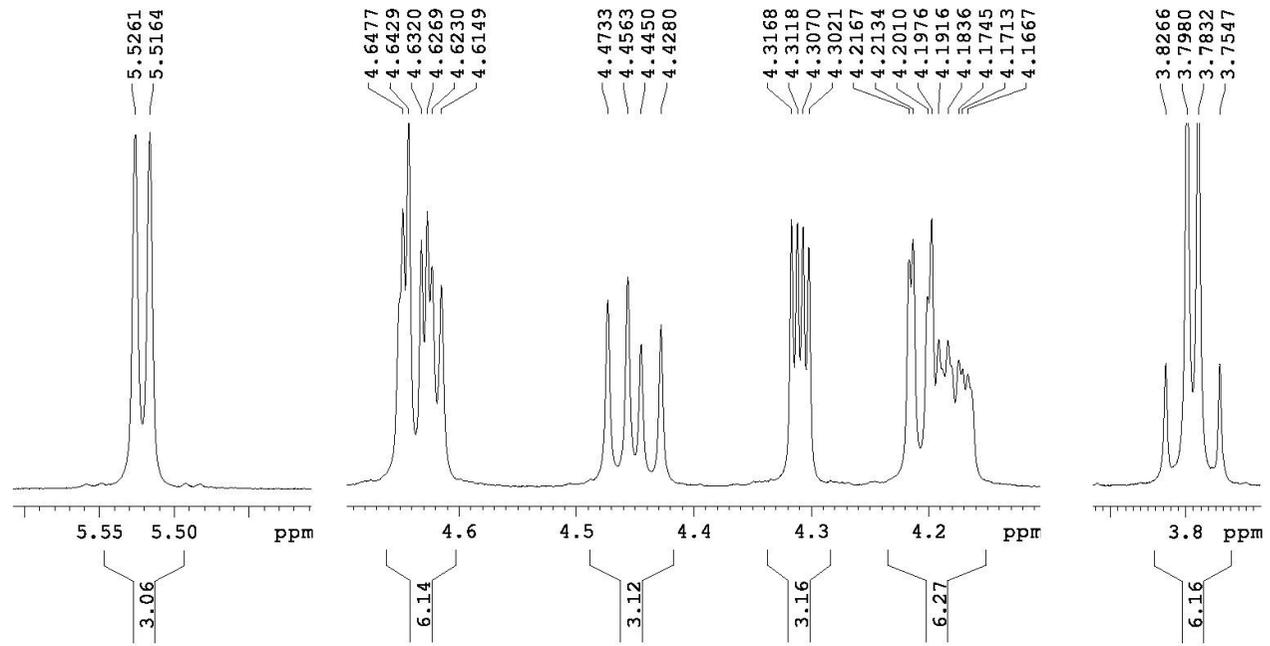
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PROCNO 1

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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10288.065 Hz
FIDRES 0.156983 Hz
AQ 3.1850495 sec
RG 322.5
DW 48.600 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

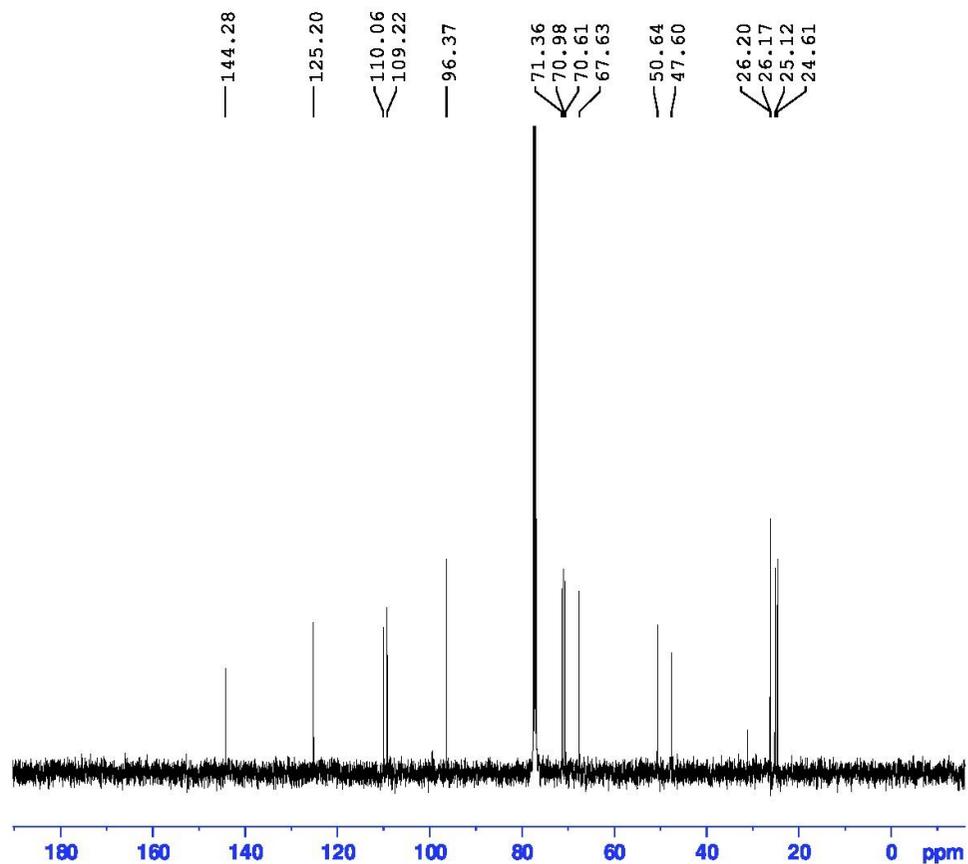
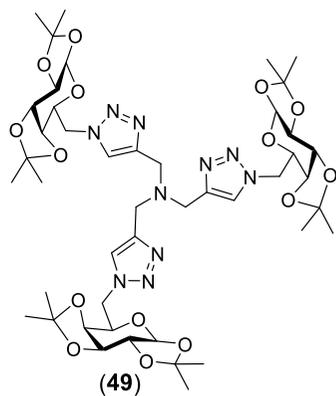
CHANNEL f1
NUC1 1H
P1 9.25 usec
PL1 -2.70 dB
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1300082 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Appendix B21: ^1H NMR spectrum of compound (49).



Appendix B22: Selected areas ¹H NMR of compound (49).



Current Data Parameters
 NAME A25175
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameter
 Date_ 20120120
 Time 15.01
 INSTRUM DRX500
 PROBHD 5 mm DUL 13C-1
 PULPROG zgpg30
 TD 16384
 SOLVENT CDCl3
 NS 1024
 DS 4
 SWH 30120.482 Hz
 FIDRES 1.838408 Hz
 AQ 0.2719744 sec
 RG 9195.2
 DW 16.600 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.69999999 sec
 d11 0.03000000 sec
 DELTA 0.59999996 sec
 TD0 1

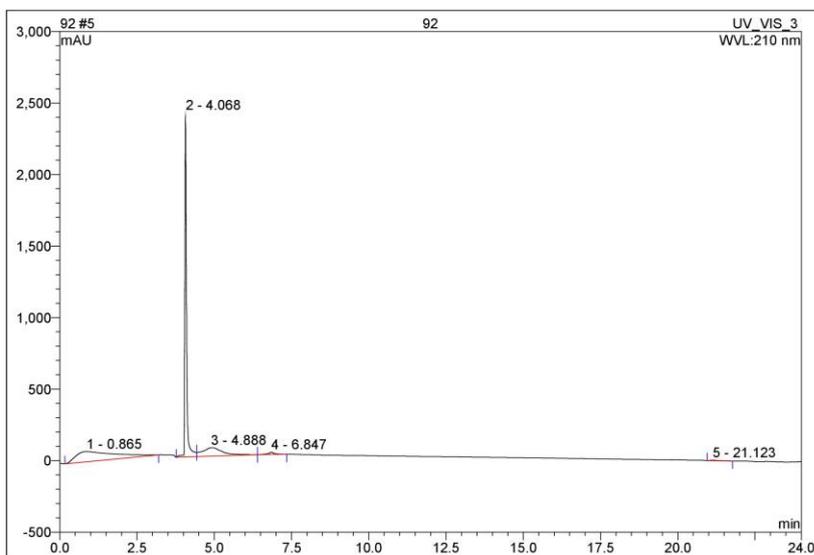
CHANNEL f1
 NUC1 13C
 P1 8.00 usec
 PL1 -2.30 dB
 SFO1 125.7703643 MHz

CHANNEL f2
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -2.30 dB
 PL12 16.21 dB
 PL13 120.00 dB
 SFO2 500.1320005 MHz

F2 - Processing parameters
 SI 32768
 SF 125.7577623 MHz
 WDW EM
 SSB 0
 LB 2.00 Hz
 GB 0
 PC 1.40

Appendix B23: ^{13}C NMR spectrum of compound (49).

5 92			
Sample Name:	92	Injection Volume:	20.0
Vial Number:	RA5	Channel:	UV_VIS_3
Sample Type:	unknown	Wavelength:	210
Control Program:	poly-p4 NO TFA lines A-D 28min +210nm	Bandwidth:	10
Quantif. Method:	dna method	Dilution Factor:	1.0000
Recording Time:	2/4/2012 16:21	Sample Weight:	1.0000
Run Time (min):	24.00	Sample Amount:	1.0000

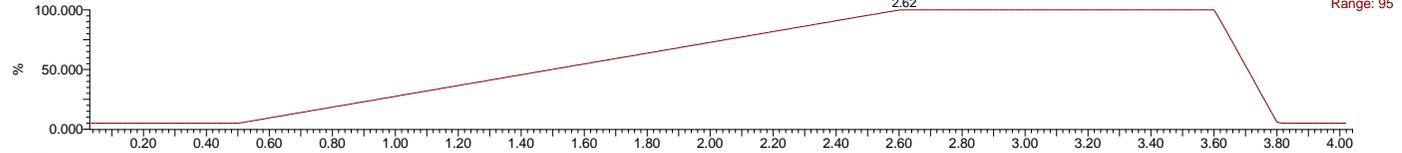


No.	Ret. Time min	Peak Name	Height mAU	Area mAU*min	Rel. Area %	Amount	Type
1	0.87	n.a.	71.232	100.633	32.78	n.a.	BMB
2	4.07	n.a.	2408.572	156.190	50.87	n.a.	BM
3	4.89	n.a.	58.018	45.003	14.66	n.a.	MB
4	6.85	n.a.	15.443	4.126	1.34	n.a.	bMB
5	21.12	n.a.	3.771	1.063	0.35	n.a.	BMB
Total:			2557.036	307.016	100.00	0.000	

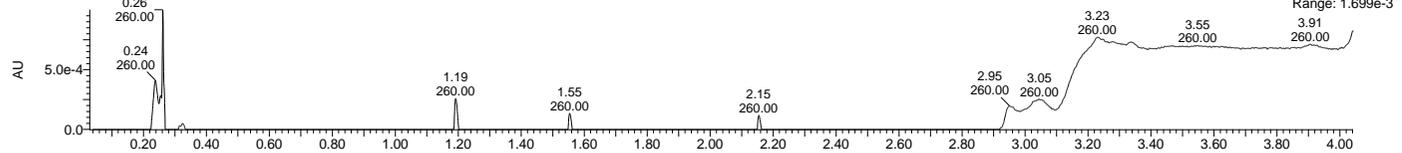
default/integration

Chromeleon (c) Dionex 1996-2006
Version 6.80 SP4 Build 2361 (130805)*Appendix B24: HPL chromatogram of compound (38).*

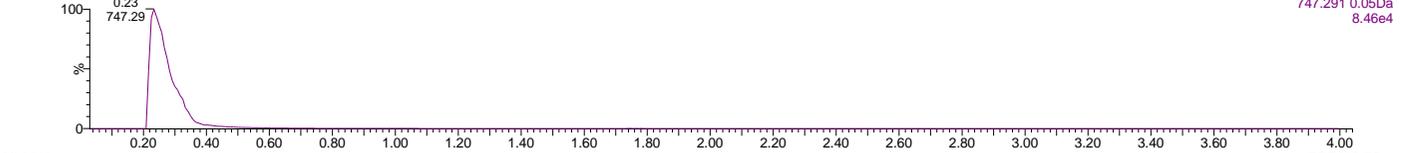
AJF_HK_04
ML0593



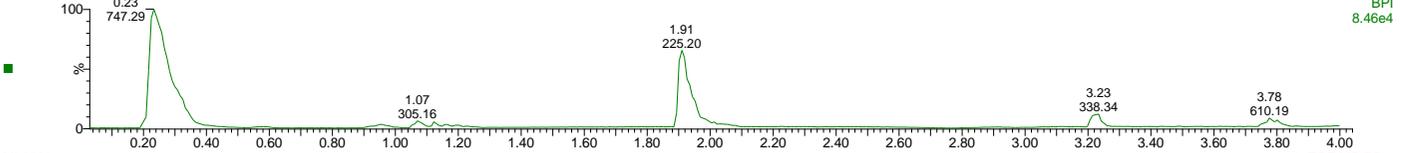
ML0593



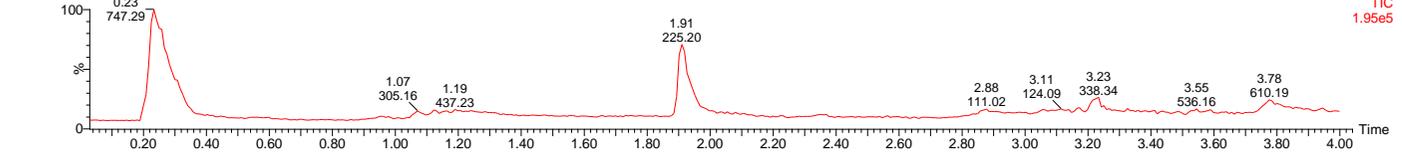
ML0593



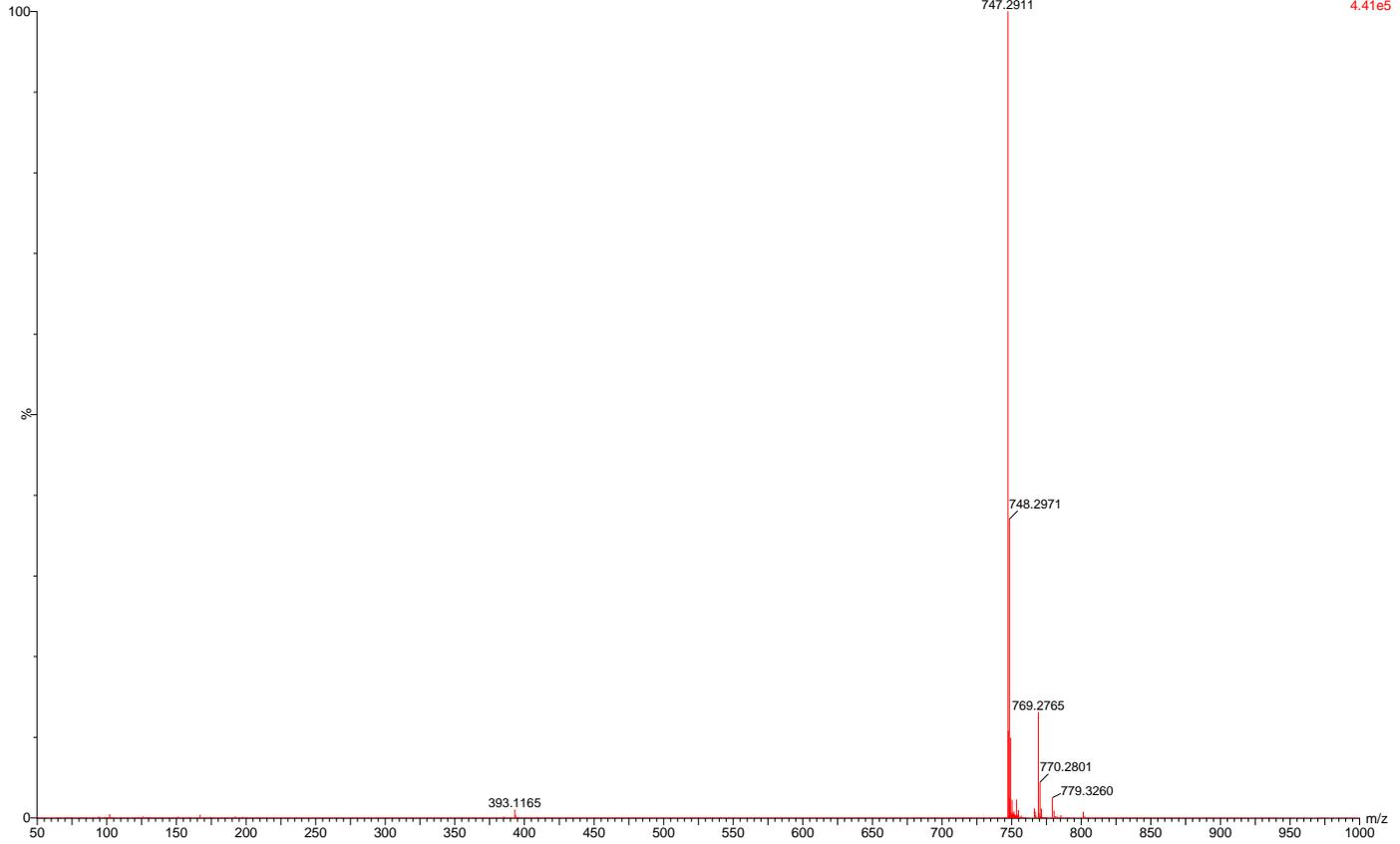
ML0593



ML0593



AJF_HK_04
ML0593 24 (0.233) Cm (23:28)



Elemental Composition Report: **AJF-HK-04**

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

669 formula(e) evaluated with 1 results within limits (up to 20 closest results for each mass)

Elements Used:

C: 27-27 H: 0-150 N: 0-25 O: 0-25

Minimum:

-1.5

Maximum:

10.0

5.0

50.0

Mass

Calc. Mass

mDa

PPM

DBE

i-FIT

i-FIT (Norm)

Formula

747.2911

747.2909

0.2

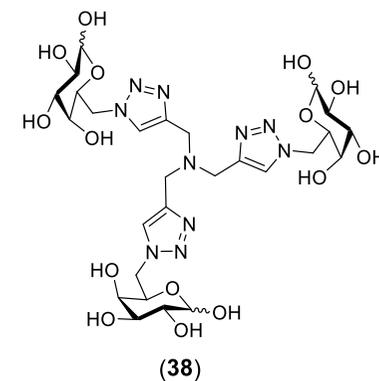
0.3

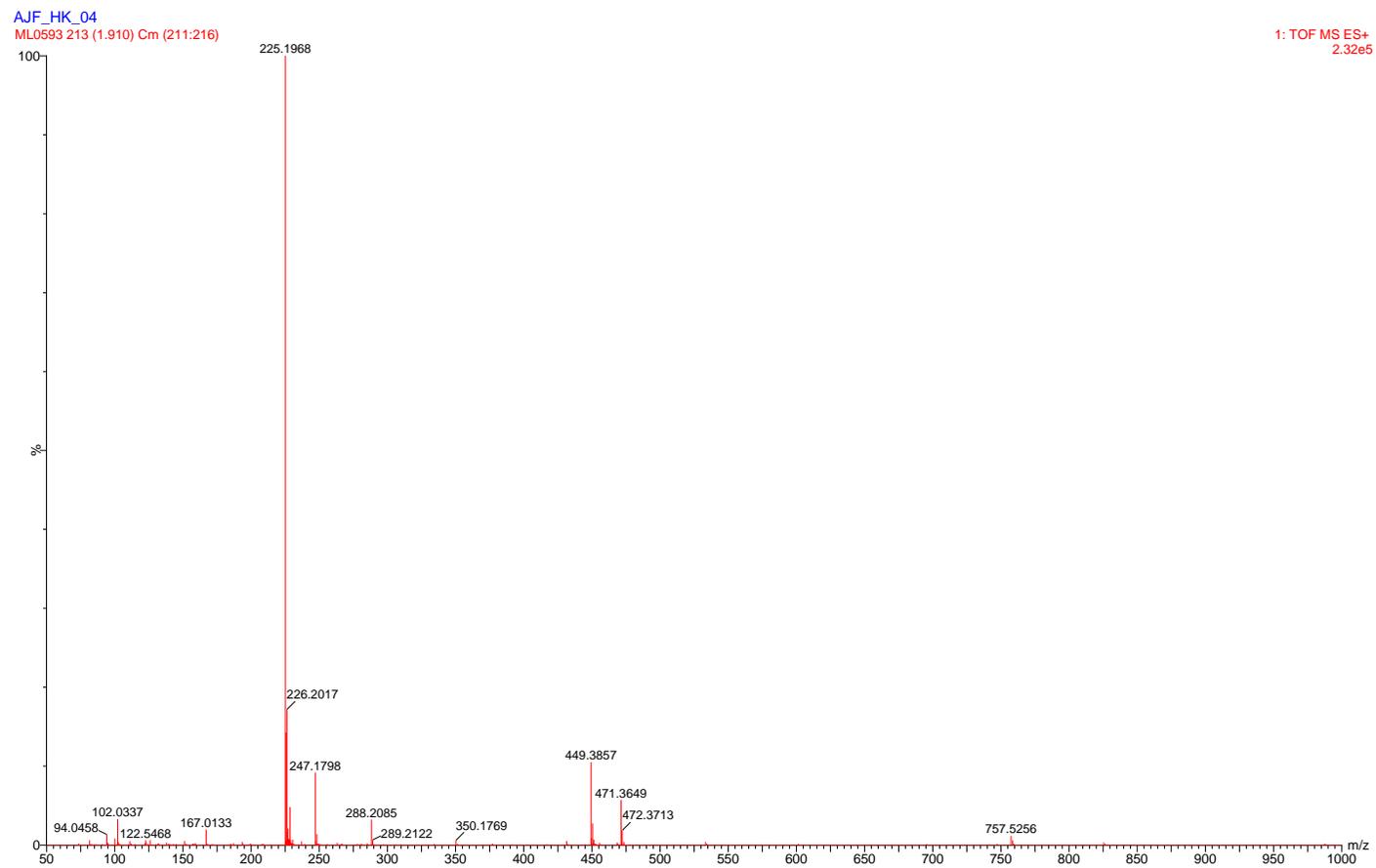
11.5

221.2

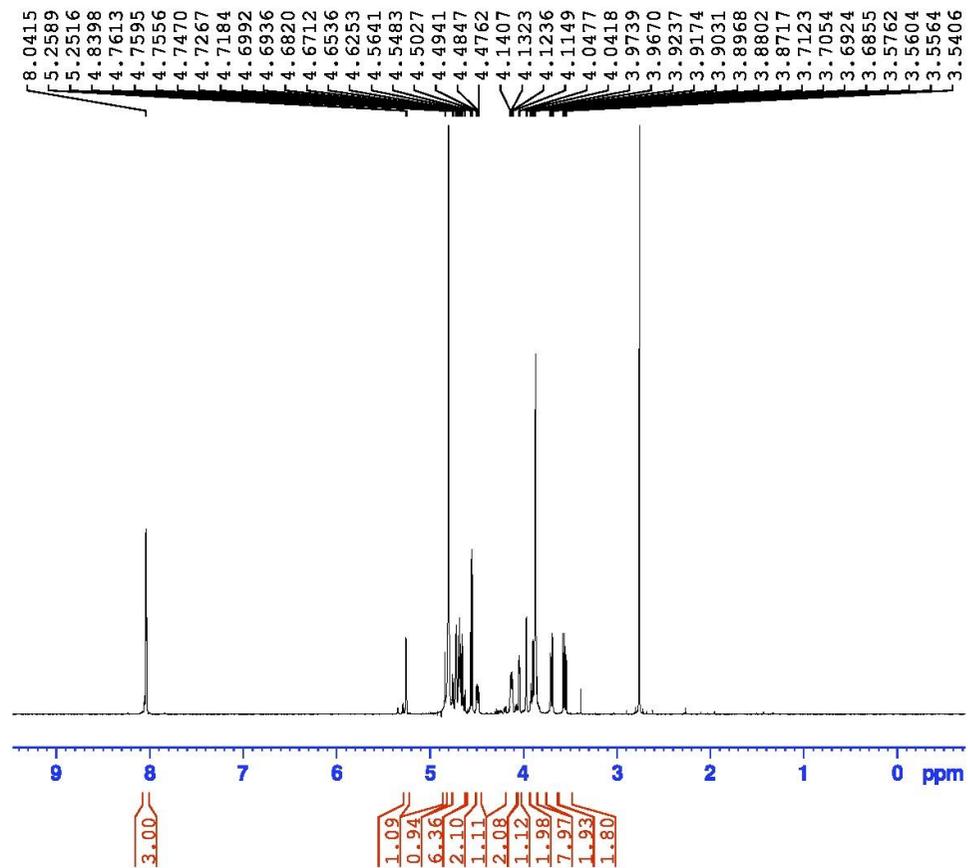
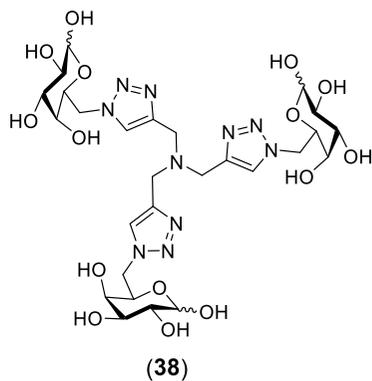
0.0

C₂₇ H₄₃ N₁₀ O₁₅





Appendix B25: HRMS (ESI) spectra of compound (38).



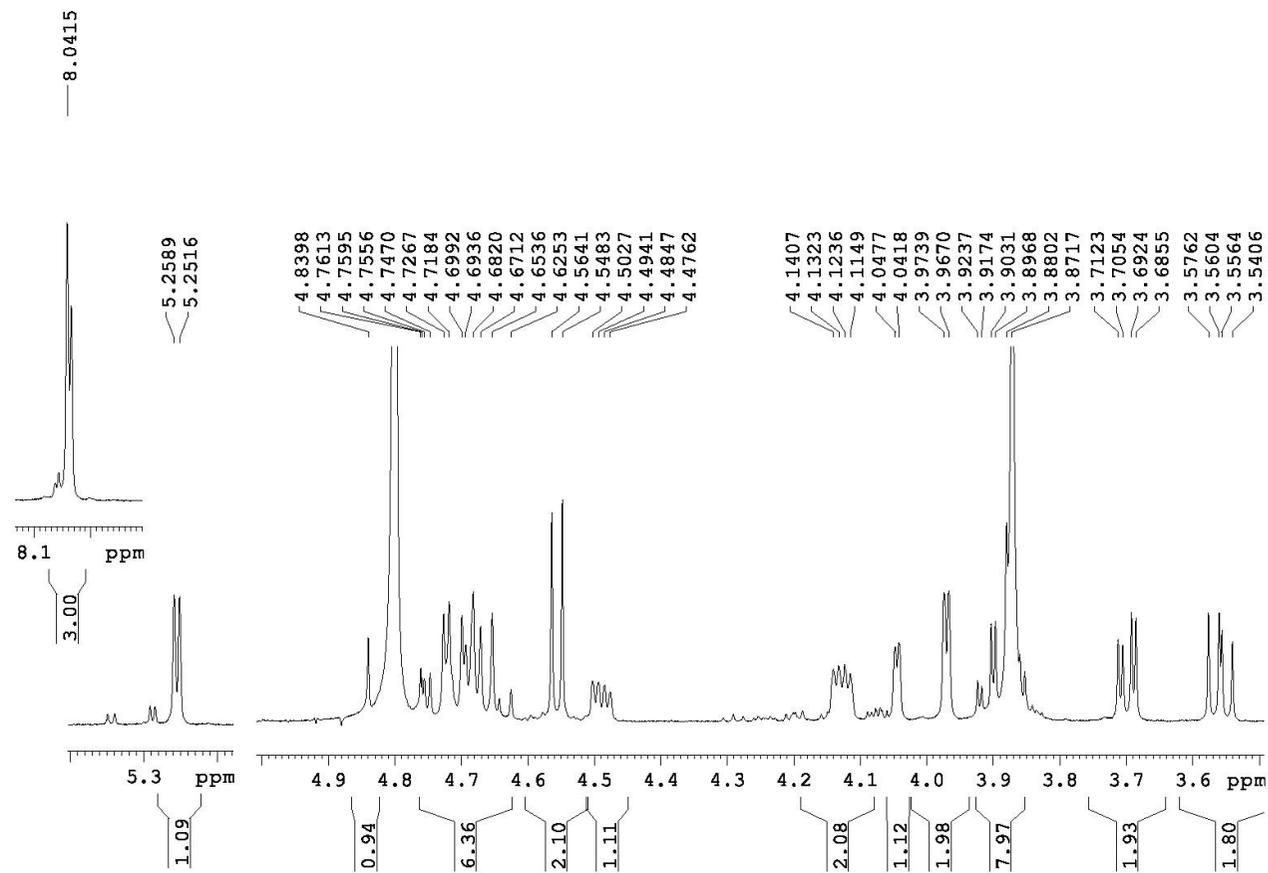
Current Data Parameters
NAME A26822
EXPNO 1
PROCNO 1

F2 - Acquisition Parameter
Date_ 20120330
Time 13.23
INSTRUM DRX500
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT D2O
NS 16
DS 2
SWH 10288.065 Hz
FIDRES 0.156983 Hz
AQ 3.1850495 sec
RG 322.5
DW 48.600 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

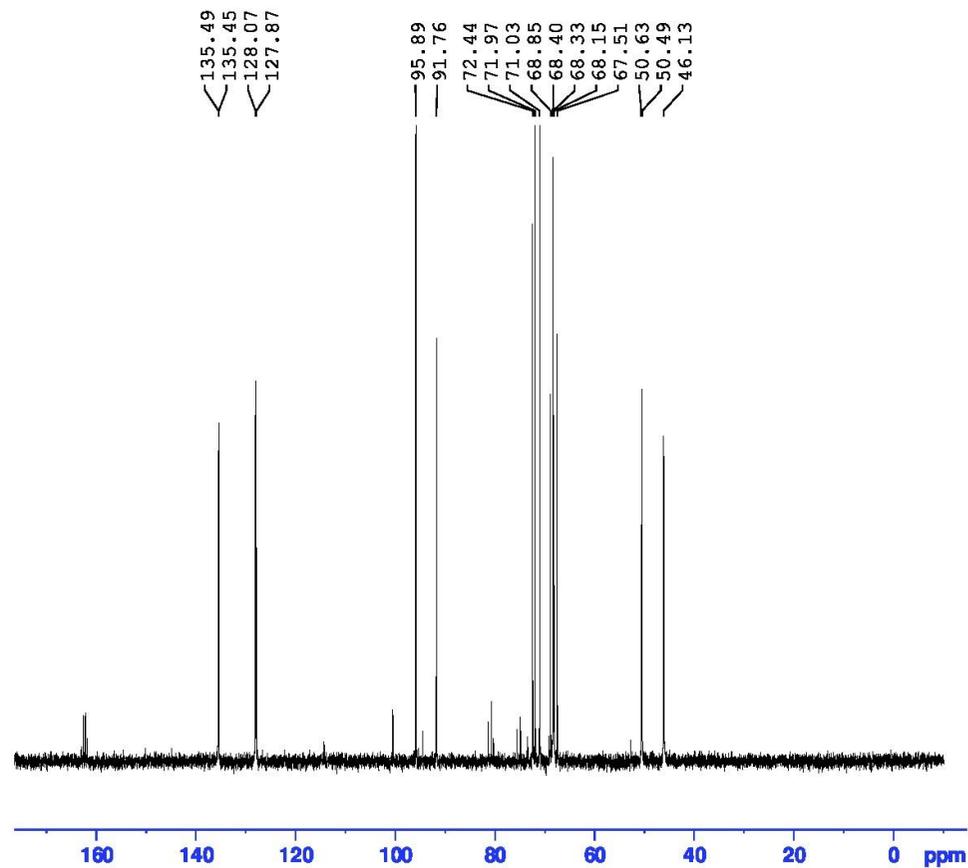
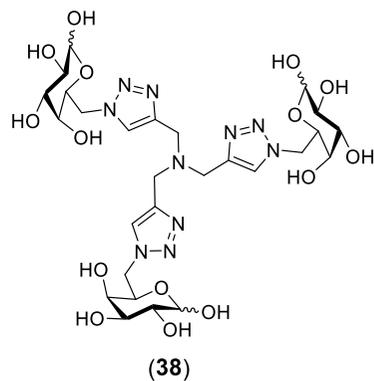
==== CHANNEL f1 =====
NUC1 1H
P1 9.25 usec
PL1 -2.70 dB
SFO1 500.1330885 MHz

F2 - Processing parameters
SI 32768
SF 500.1299518 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Appendix B26: ¹H NMR spectrum of compound (38).



Appendix B27: Selected areas ^1H NMR of compound (38).



```

Current Data Parameters
NAME          D175901
EXPNO         1
PROCNO        1

F2 - Acquisition Parameters
Date_         20130223
Time          9.40
INSTRUM       spect
PROBHD        5 mm QNP 1H/13
PULPROG       zgpg30
TD            16384
SOLVENT       D2O
NS            40960
DS            4
SWH           22058.824 Hz
FIDRES        1.346364 Hz
AQ            0.3713707 sec
RG            2050
DW            22.667 usec
DE            6.50 usec
TE            293.3 K
D1            0.69999999 sec
D11           0.03000000 sec
TD0           1

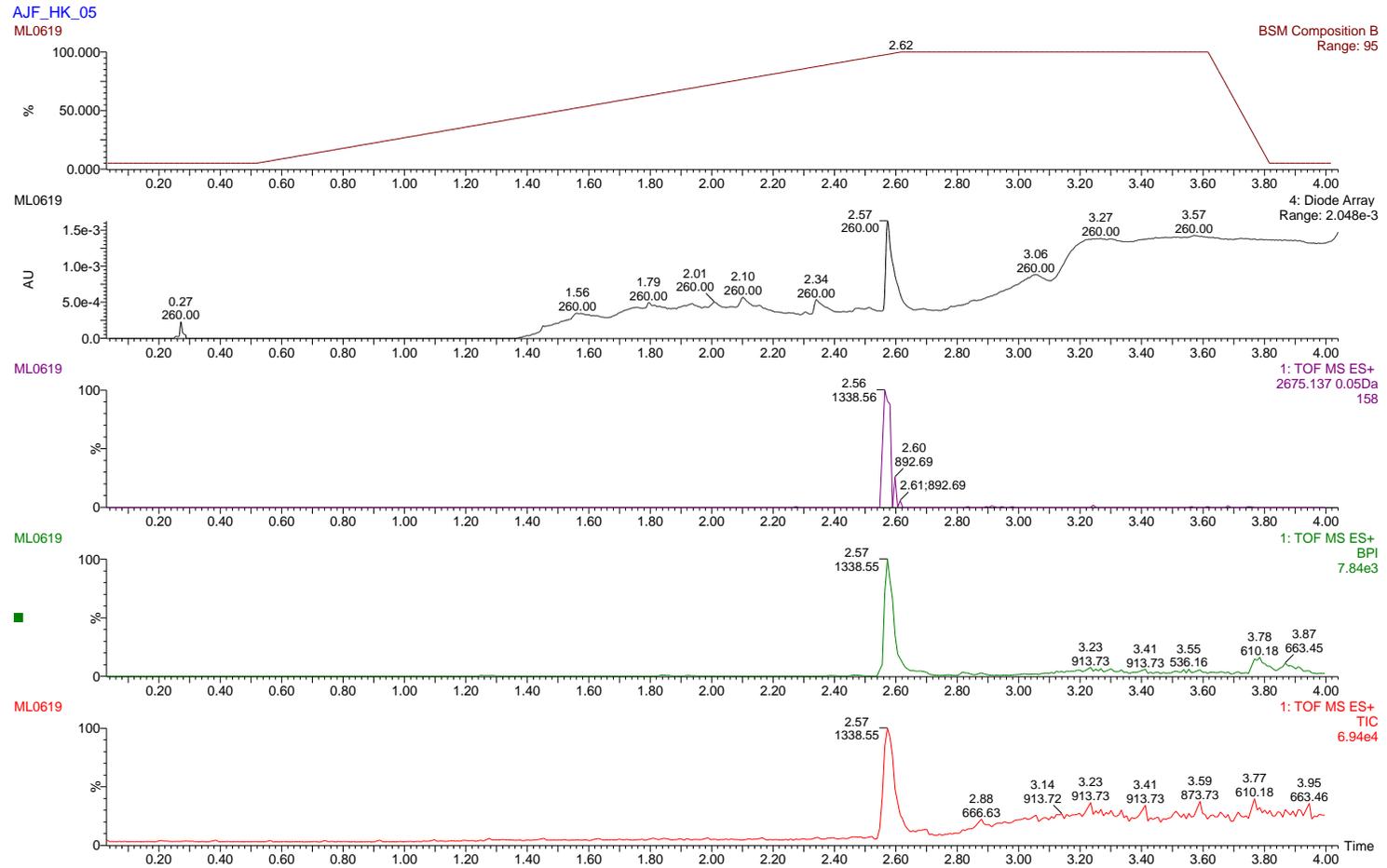
----- CHANNEL f1 -----
NUC1          13C
P1            7.00 usec
PL1           -2.60 dB
PL1W          61.34123230 W
SFO1          100.6228298 MHz

----- CHANNEL f2 -----
CPDPRG[2]    waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           -4.00 dB
PL12          14.06 dB
PL13          120.00 dB
PL2W          19.93825150 W
PL12W         0.31166428 W
PL13W         0 W
SFO2          400.1316005 MHz

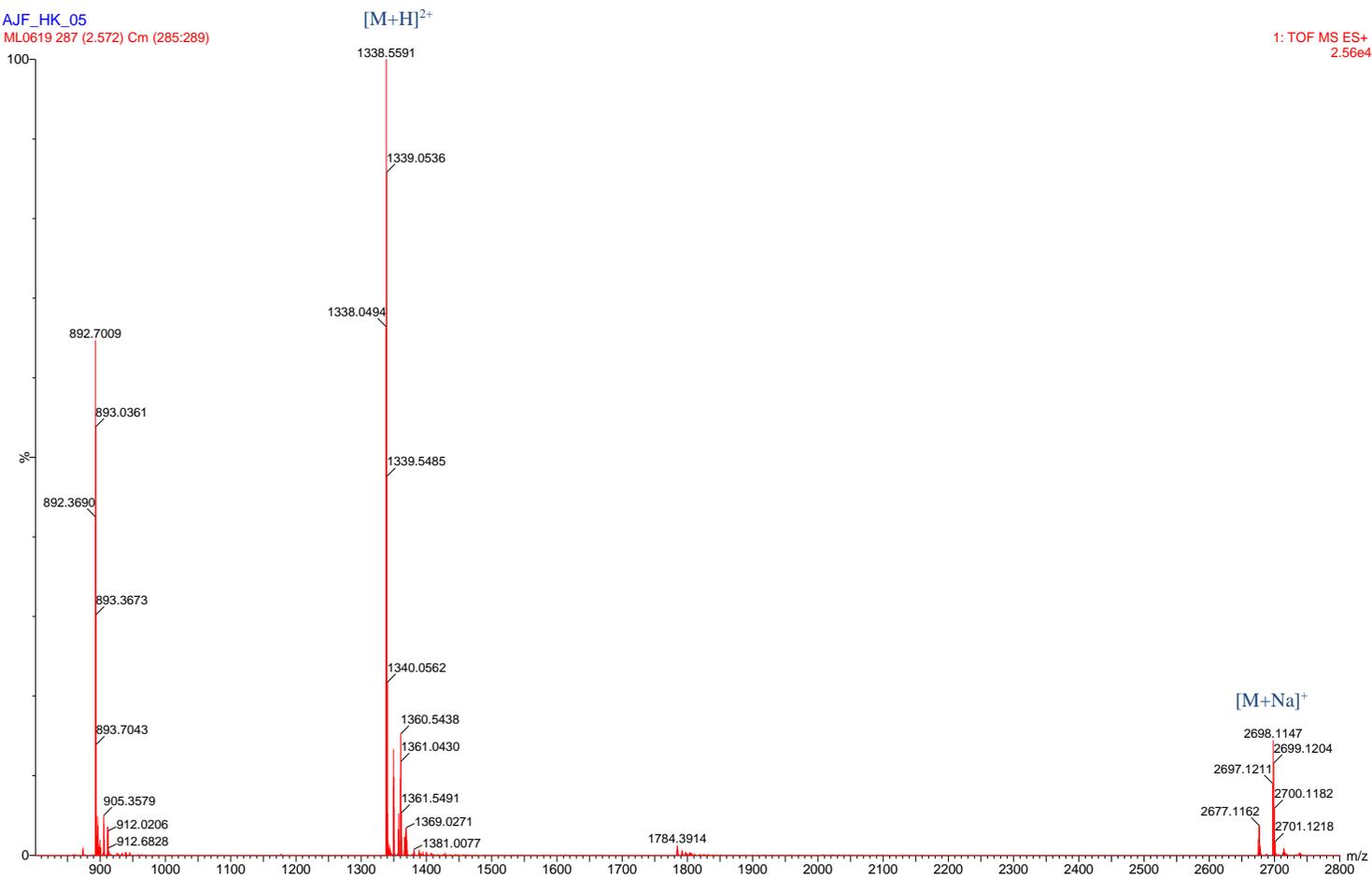
F2 - Processing parameters
SI            32768
SF            100.6128193 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40

```

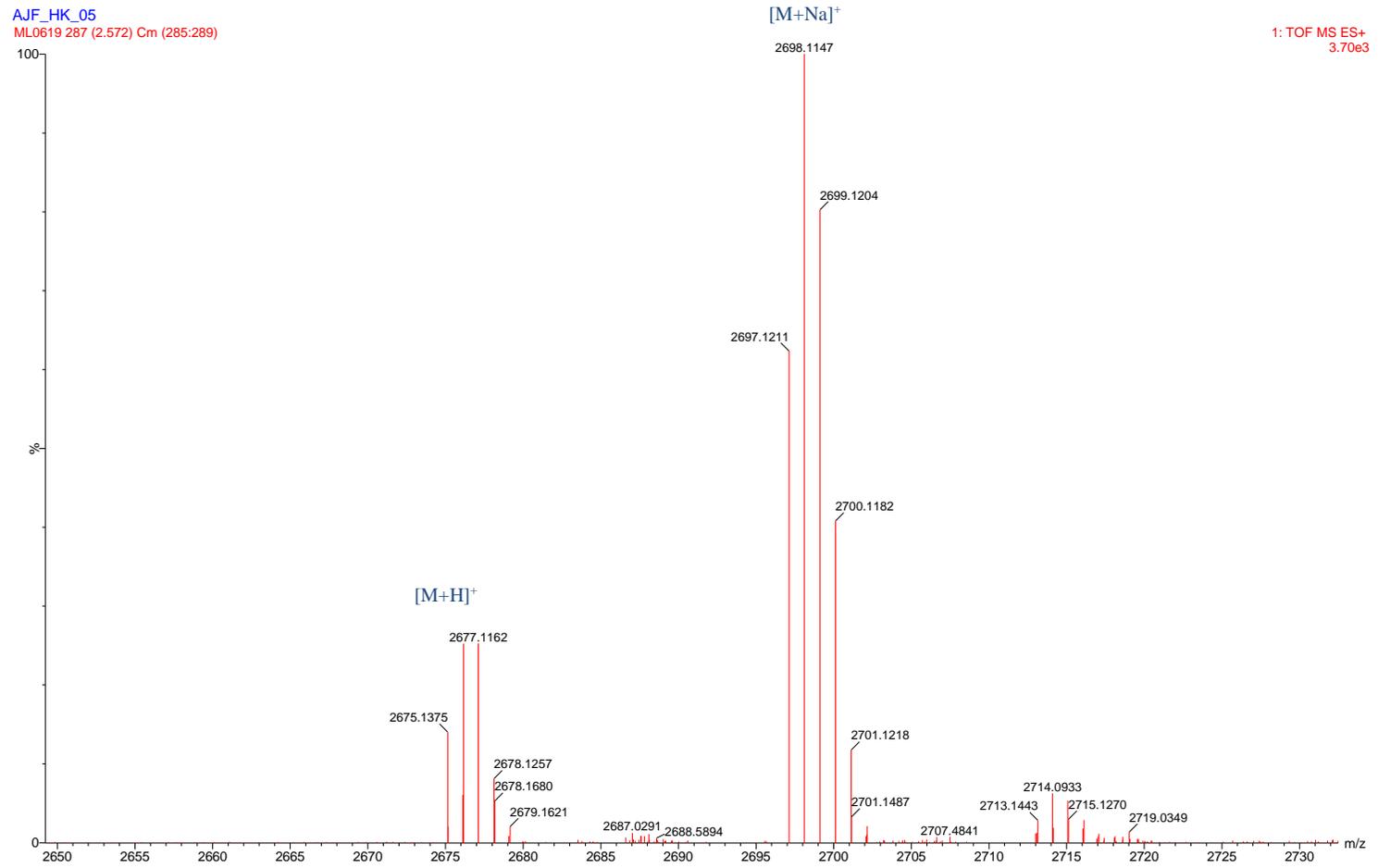
Appendix B28: ^{13}C NMR spectrum of compound (38).

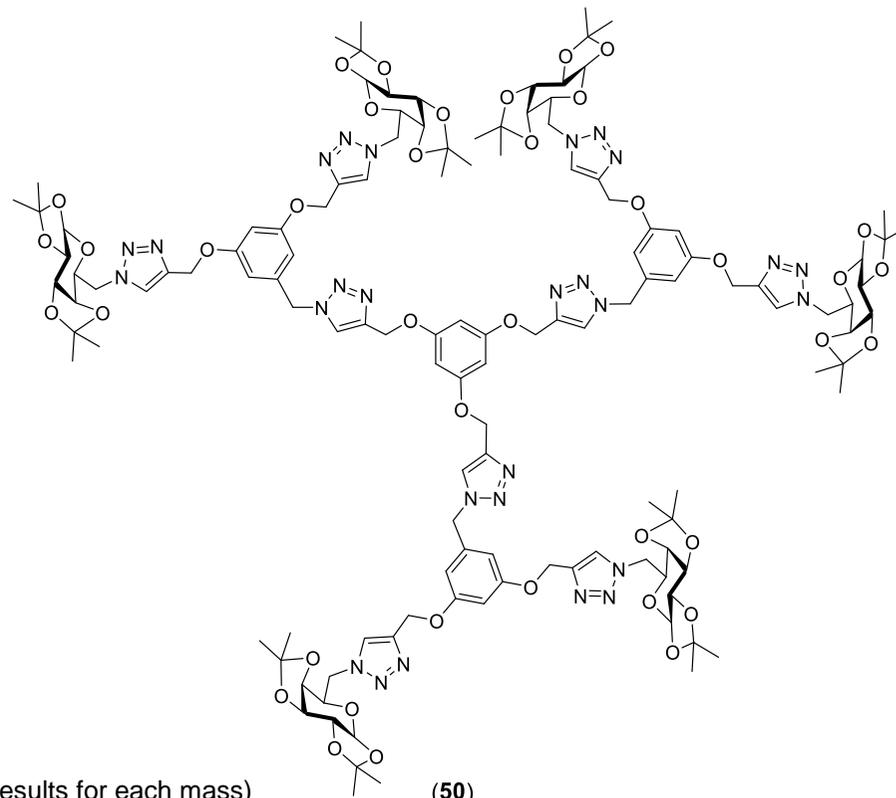


AJF_HK_05
ML0619 287 (2.572) Cm (285:289)



AJF_HK_05
ML0619 287 (2.572) Cm (285:289)





Elemental Composition Report: **AJF-HK-05**

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 70.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

2299 formula(e) evaluated with 2 results within limits (up to 20 closest results for each mass)

Elements Used:

C: 126-126 H: 0-180 N: 0-50 O: 0-50

Minimum:

-1.5

Maximum:

10.0 5.0 70.0

Mass

Calc. Mass

mDa

PPM

DBE

i-FIT

i-FIT (Norm)

Formula

2675.1375

2675.1367

0.8

0.3

60.5

42.0

0.6

C126 H160 N27 O39

2675.1492

-11.7

-4.4

65.5

42.2

0.8

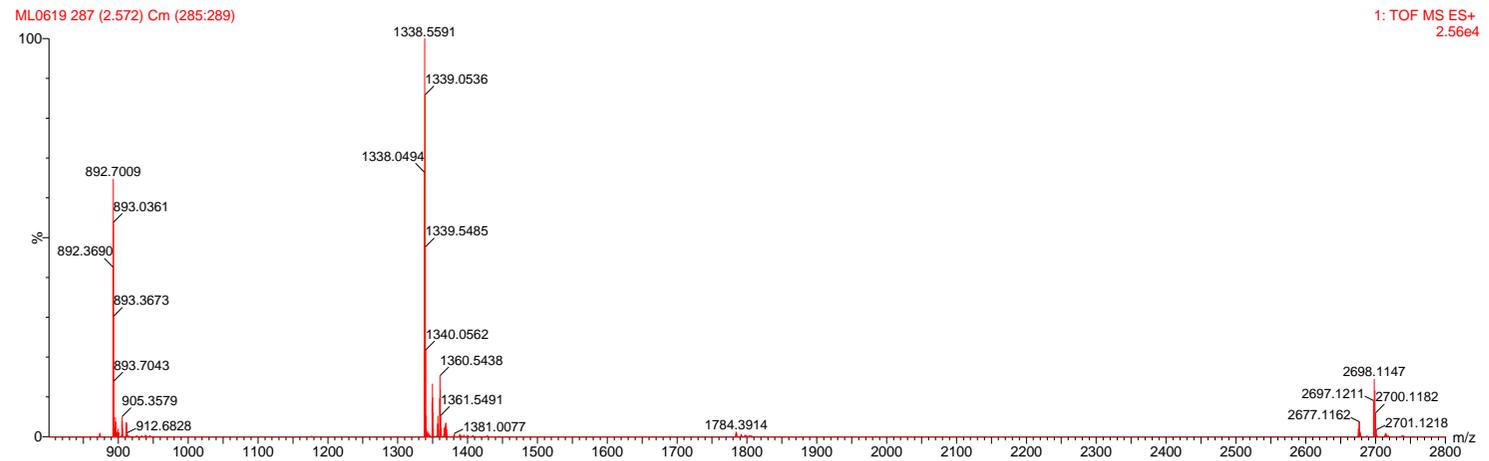
C126 H156 N33 O34

AJF_HK_05

ML0619 (0.030) Is (1.00,1.00) C126H159N27O39



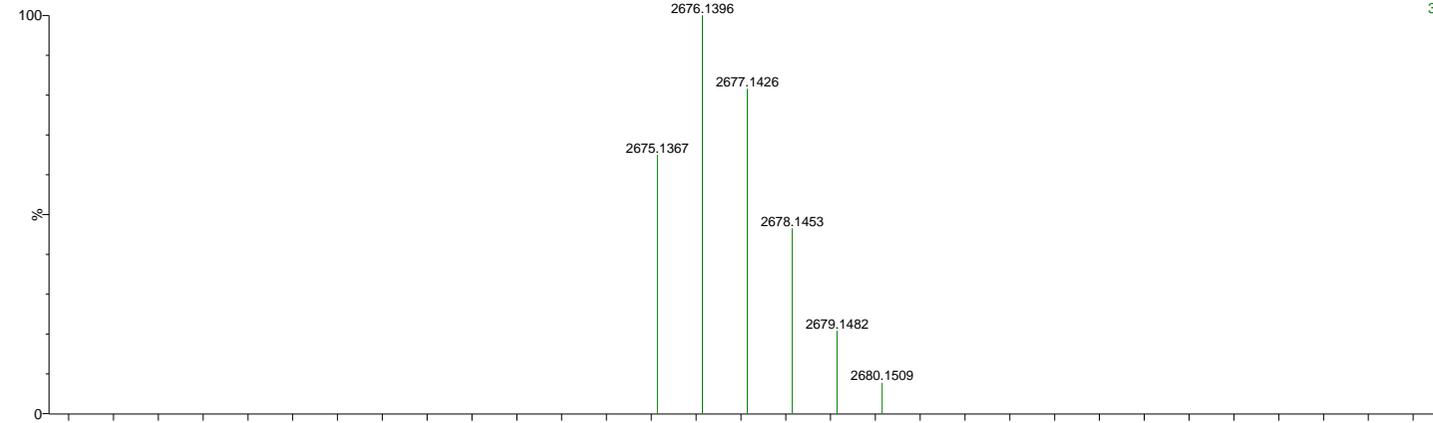
ML0619 287 (2.572) Cm (285:289)



AJF_HK_05

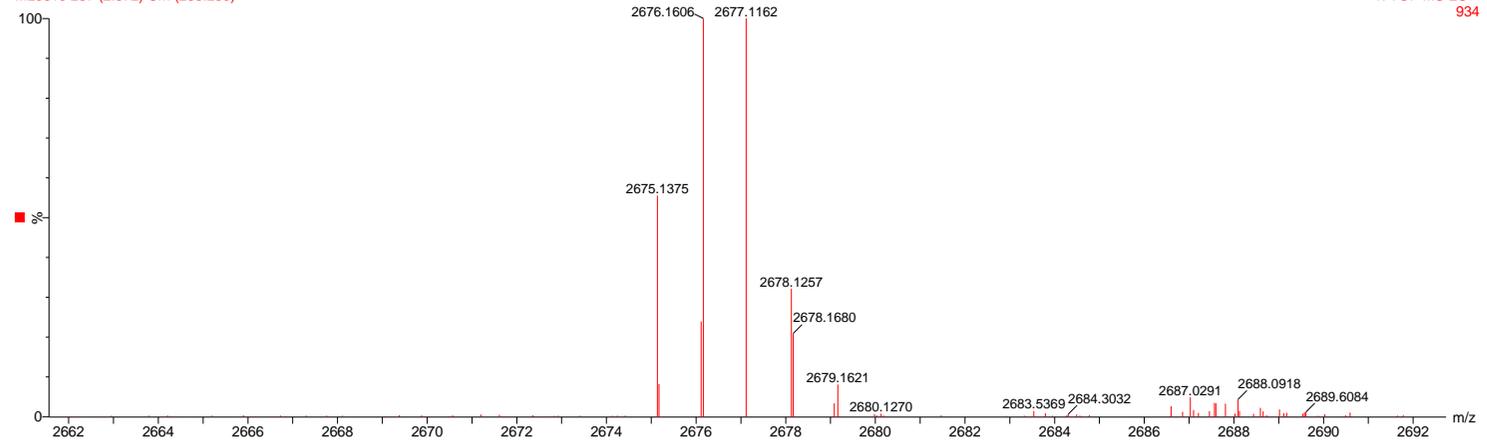
ML0619 (0.030) Is (1.00,1.00) C126H159N27O39

1: TOF MS ES+
3.08e12



ML0619 287 (2.572) Cm (285:289)

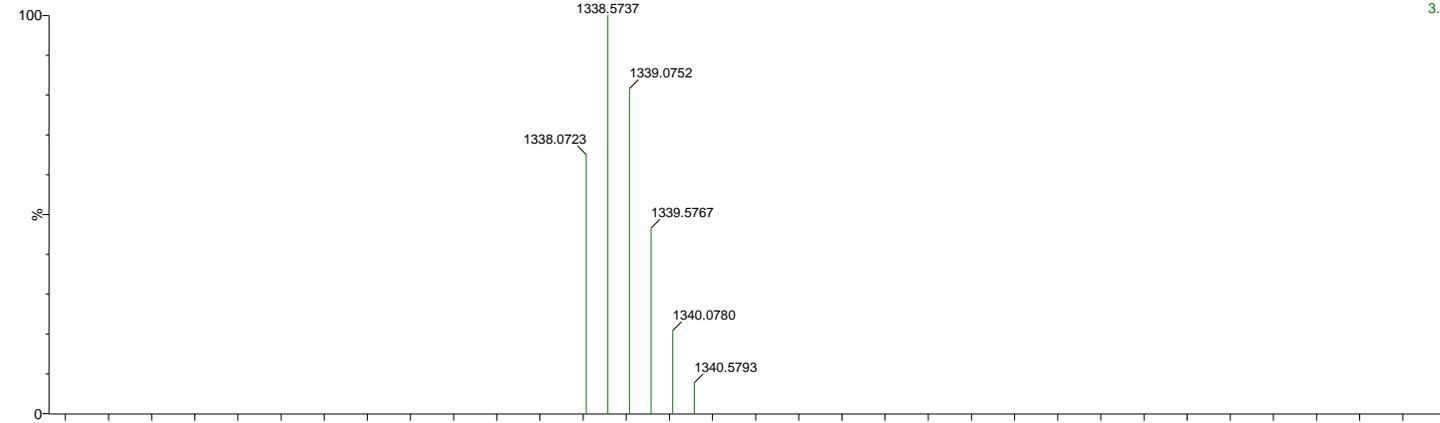
1: TOF MS ES+
934



AJF_HK_05

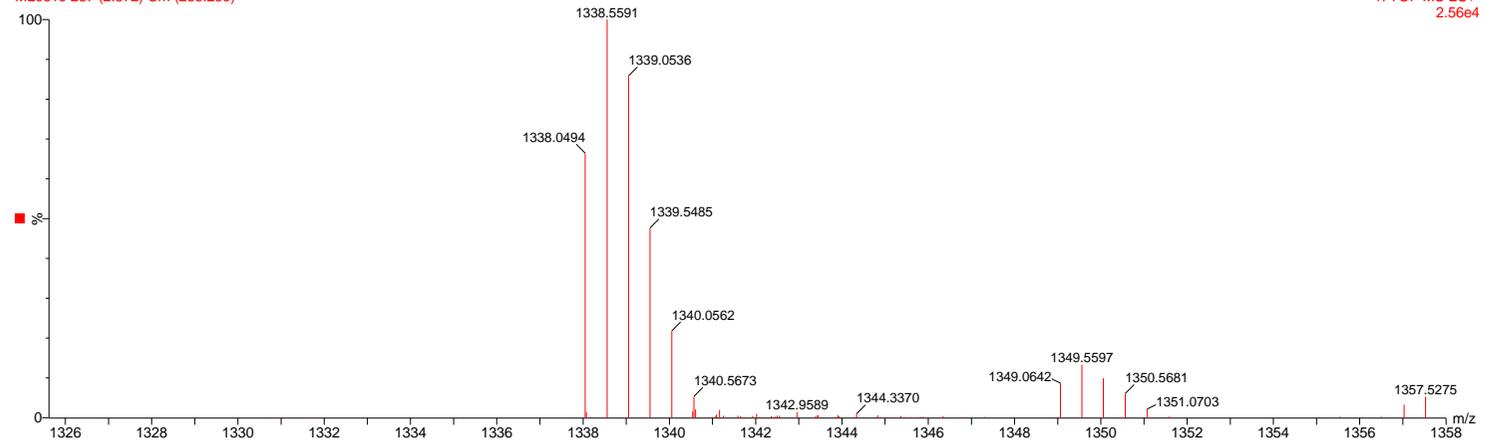
ML0619 (0.030) Is (1.00,1.00) C126H159N27O39

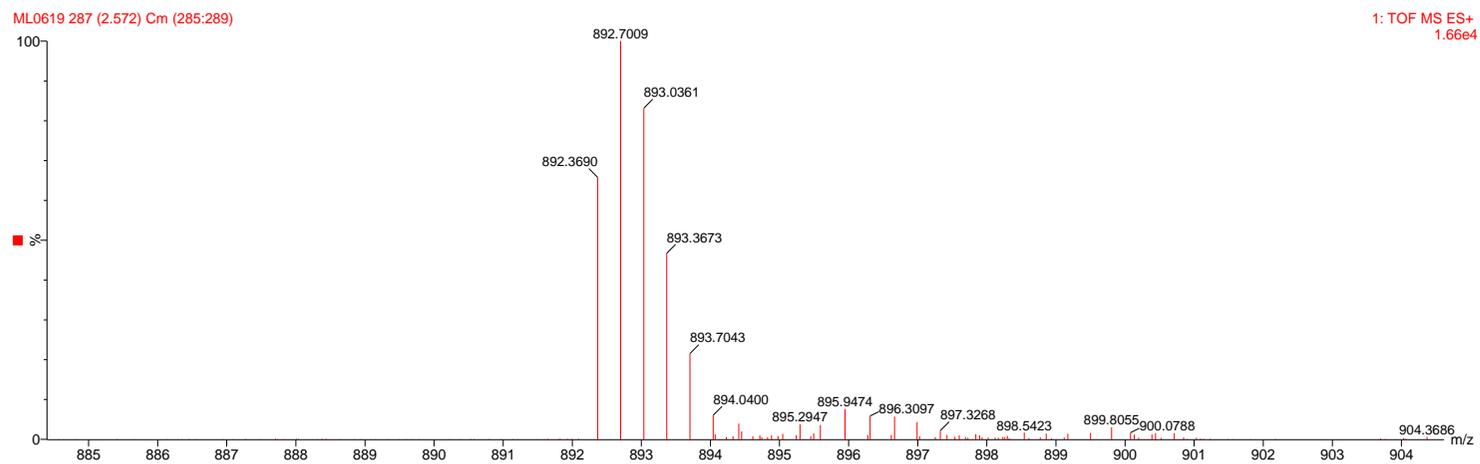
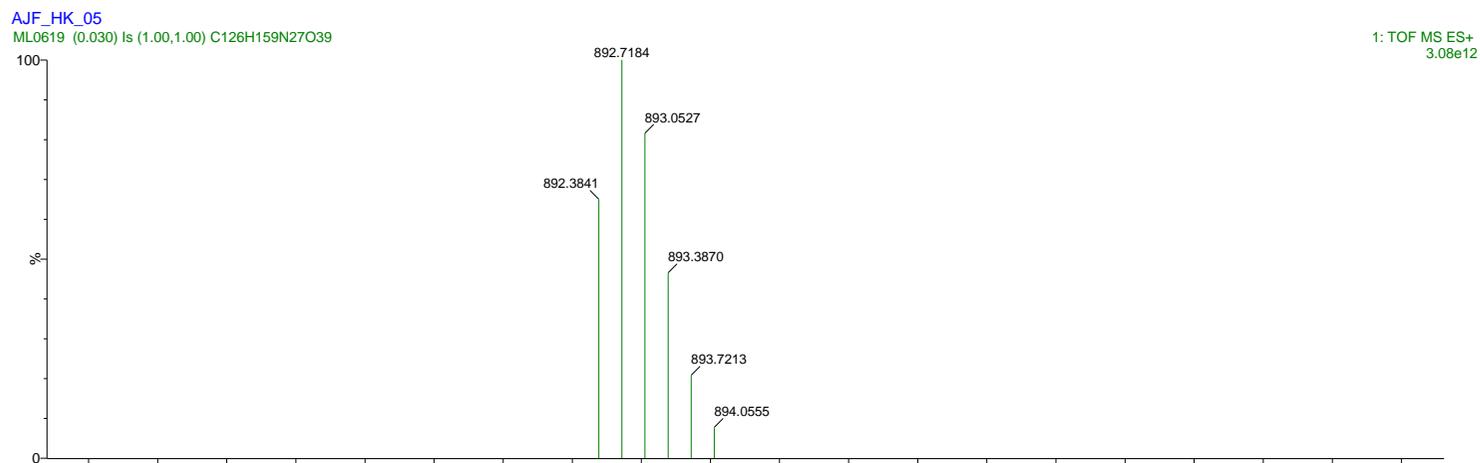
1: TOF MS ES+
3.08e12



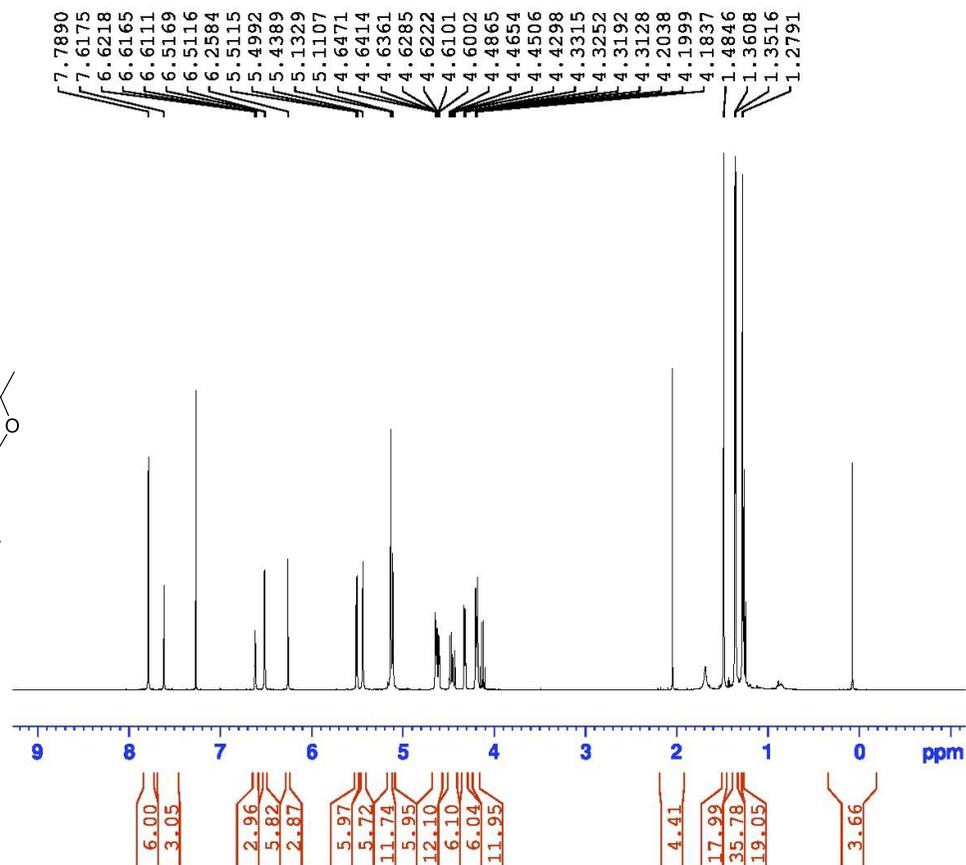
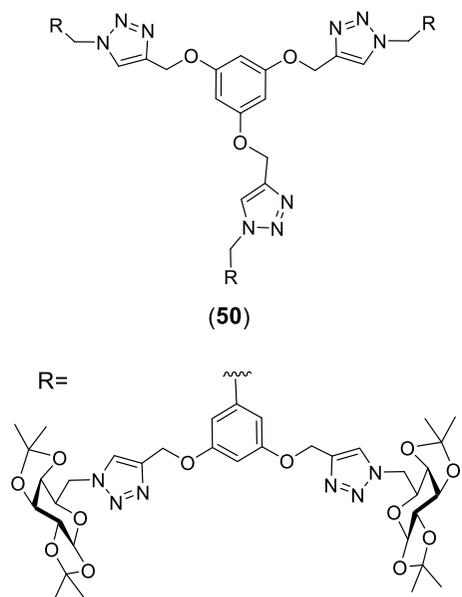
ML0619 287 (2.572) Cm (285:289)

1: TOF MS ES+
2.56e4





Appendix B29: HRMS (ESI) spectra of compound (50).



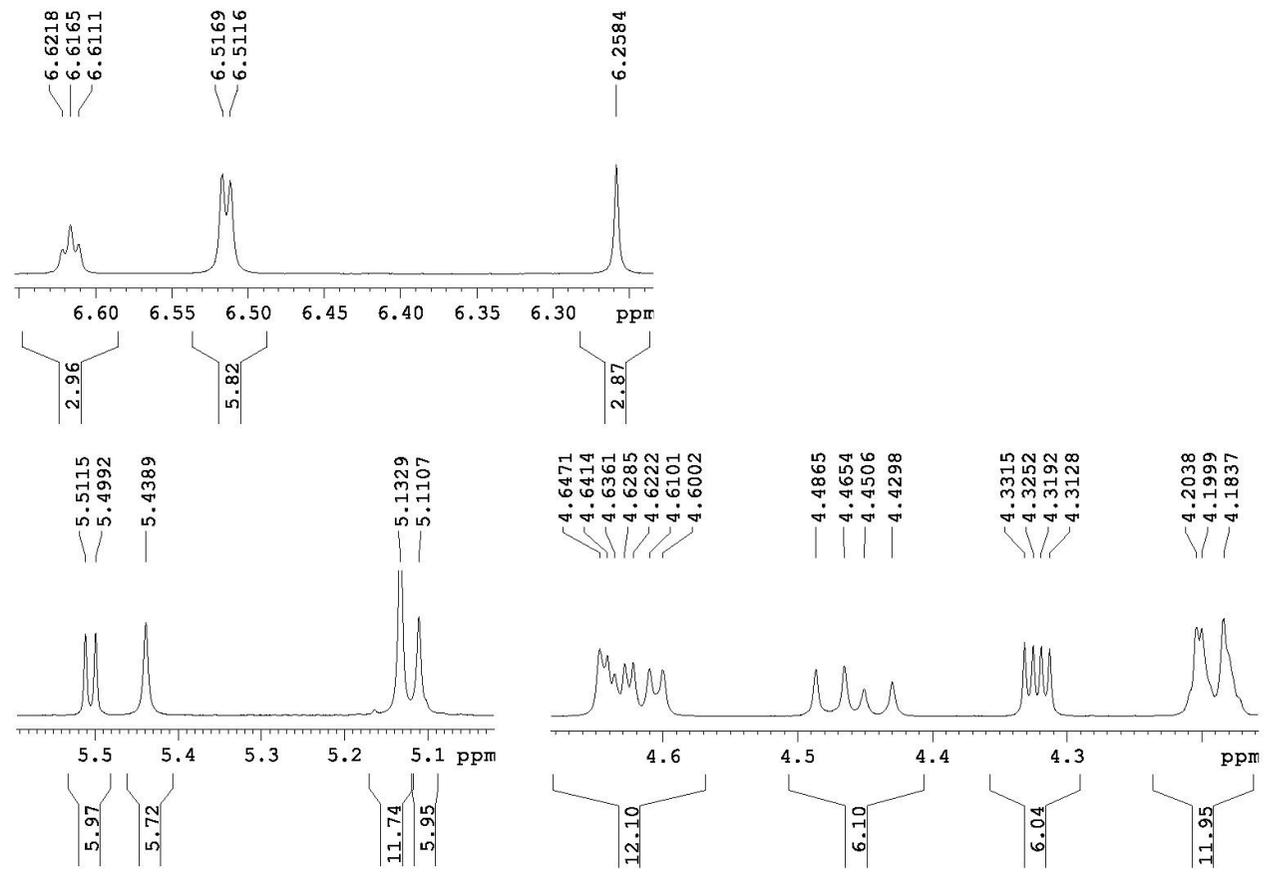
Current Data Parameters
 NAME d155590
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameter
 Date_ 20120203
 Time 6.50
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 32564
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.252539 Hz
 AQ 1.9798912 sec
 RG 161
 DW 60.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 2.00000000 sec
 TD0 1

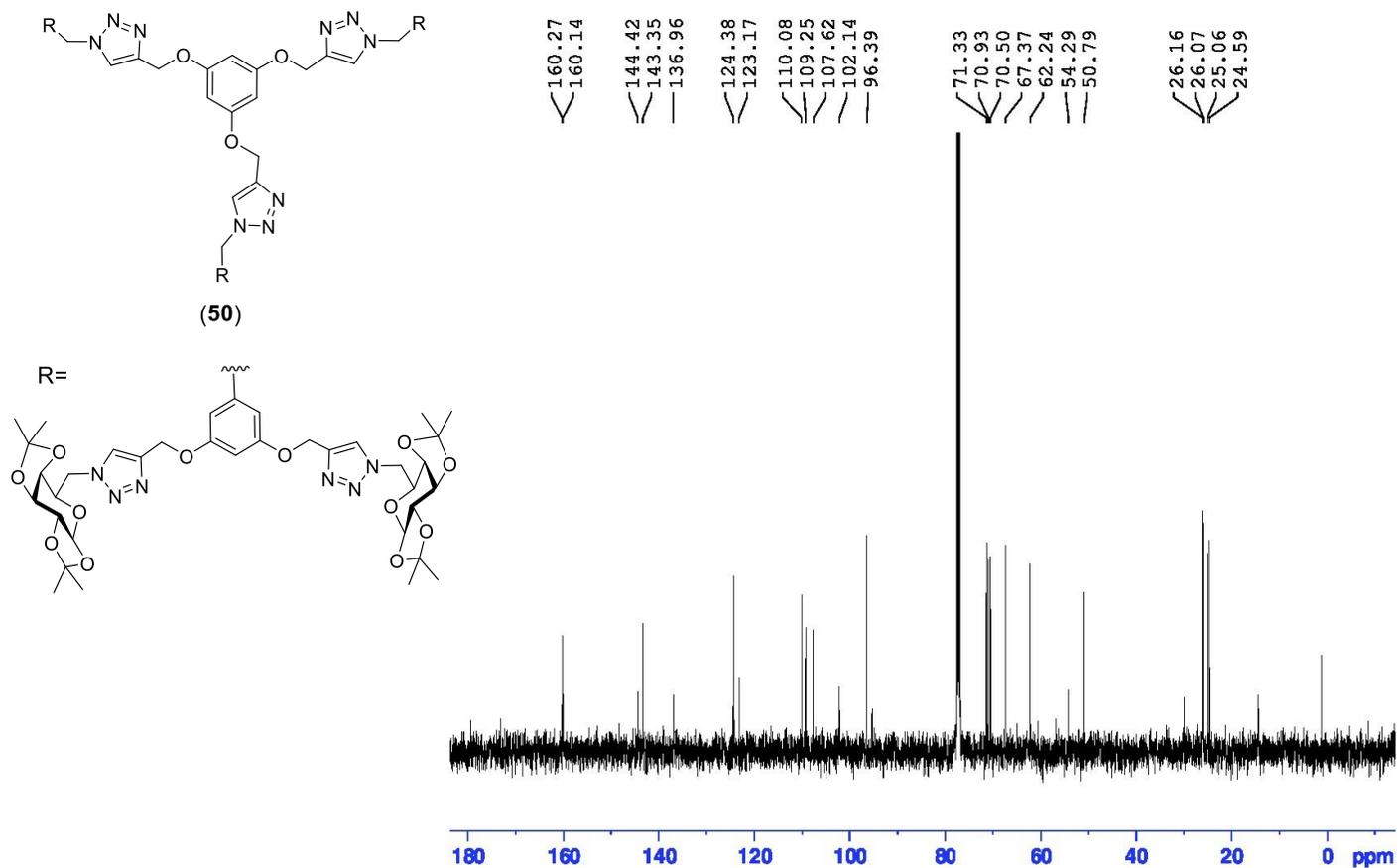
==== CHANNEL f1 =====
 NUC1 1H
 P1 9.40 usec
 PL1 -4.00 dB
 PL1W 19.93825150 W
 SF01 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300061 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Appendix B30: ¹H NMR spectrum of compound (50).



Appendix B31: Selected areas ¹H NMR of compound (50).



Current Data Parameters
 NAME A25555
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameter
 Date_ 20120207
 Time 15.16
 INSTRUM DRX500
 PROBHD 5 mm DUL 13C-1
 PULPROG zgpg30
 TD 16384
 SOLVENT CDC13
 NS 1024
 DS 4
 SWH 30120.482 Hz
 FIDRES 1.838408 Hz
 AQ 0.2719744 sec
 RG 11585.2
 DW 16.600 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.69999999 sec
 d11 0.03000000 sec
 DELTA 0.59999996 sec
 TDC 1

CHANNEL f1
 NUC1 13C
 P1 8.00 usec
 PL1 -2.30 dB
 SFO1 125.7703643 MHz

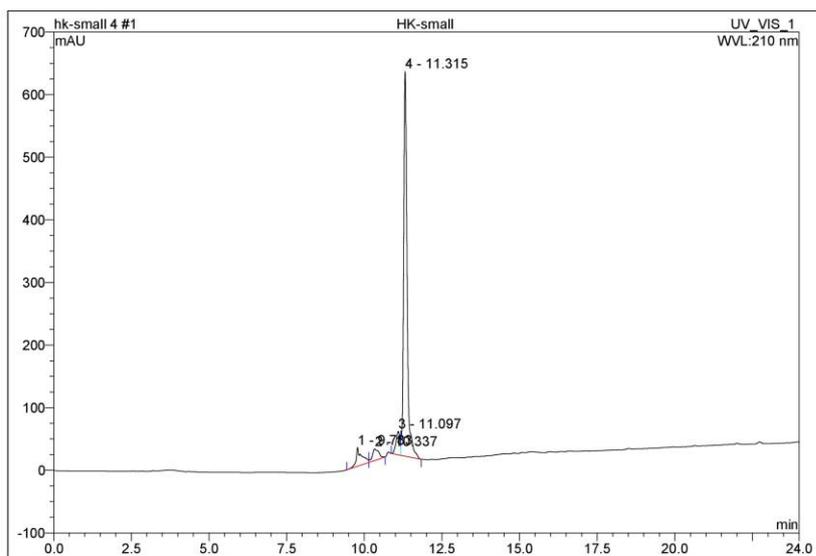
CHANNEL f2
 CPDPRG[2] waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -2.30 dB
 PL12 16.21 dB
 PL13 120.00 dB
 SFO2 500.1320005 MHz

F2 - Processing parameters
 SI 32768
 SF 125.7577665 MHz
 WDW EM
 SSB 0
 LB 2.00 Hz
 GB 0
 PC 1.40

Appendix B32: ¹³C NMR spectrum of compound (50).

1 HK-small

Sample Name:	HK-small	Injection Volume:	20.0
Vial Number:	RA1	Channel:	UV_VIS_1
Sample Type:	unknown	Wavelength:	210
Control Program:	poly-p4 28min +230nm	Bandwidth:	10
Quantif. Method:	dna method	Dilution Factor:	1.0000
Recording Time:	17/9/2013 14:11	Sample Weight:	1.0000
Run Time (min):	24.00	Sample Amount:	1.0000

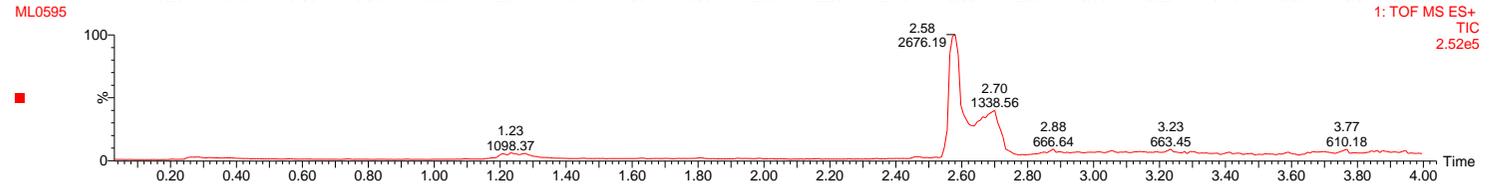
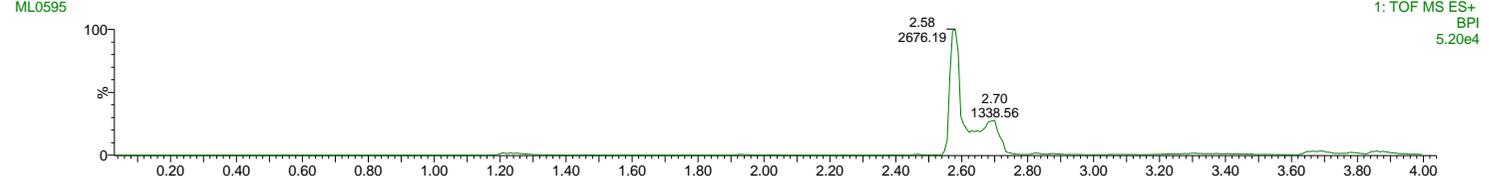
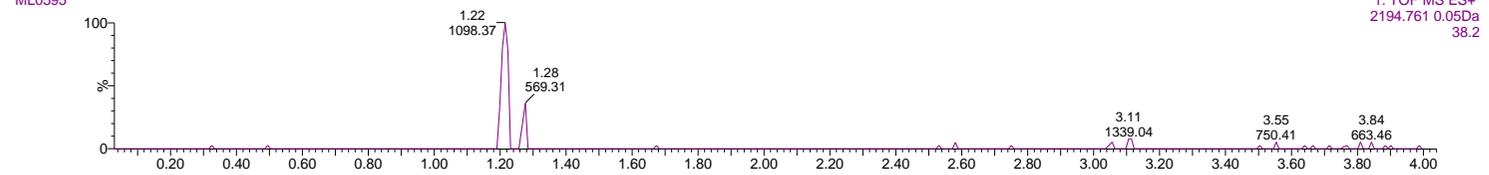
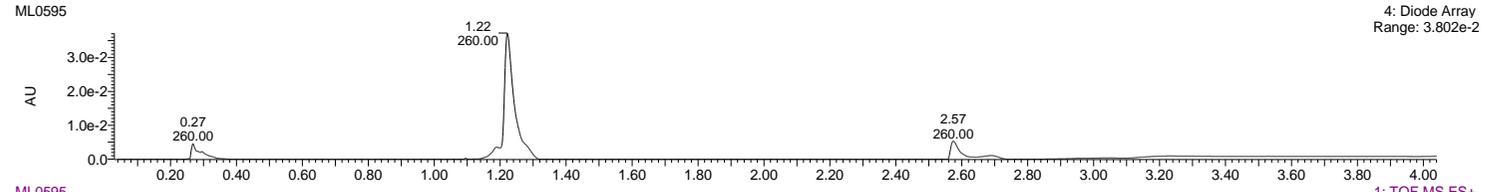
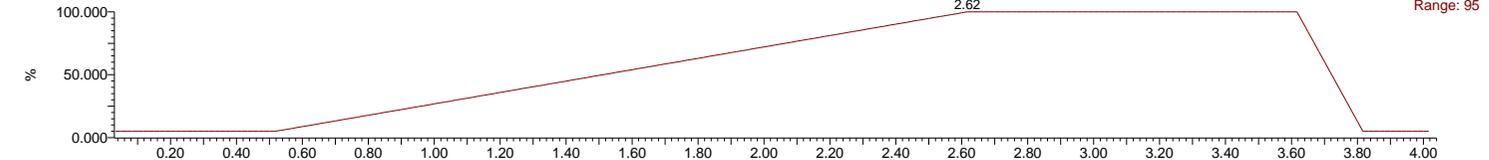


No.	Ret. Time min	Peak Name	Height mAU	Area mAU*min	Rel. Area %	Amount	Type
1	9.78	n.a.	30.062	6.175	6.45	n.a.	BM
2	10.34	n.a.	18.106	4.265	4.45	n.a.	MB
3	11.10	n.a.	37.740	5.644	5.89	n.a.	BM
4	11.32	n.a.	614.847	79.708	83.21	n.a.	MB
Total:			700.755	95.793	100.00	0.000	

default/integration

Chromeleon (c) Dionex 1996-2006
Version 6.80 SP4 Build 2361 (130805)*Appendix B33: HPL chromatogram of compound (39).*

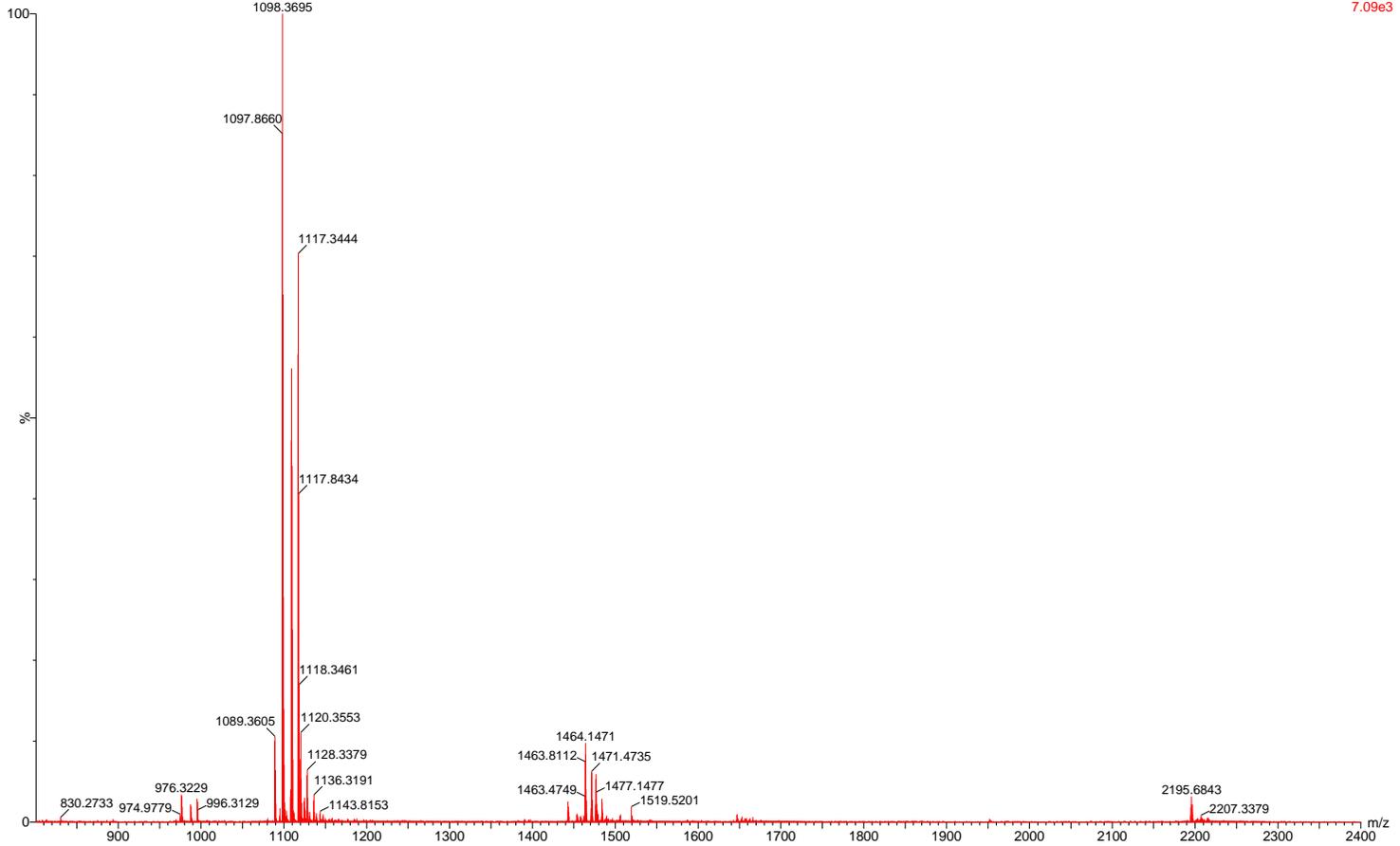
AJF_HK_06
ML0595



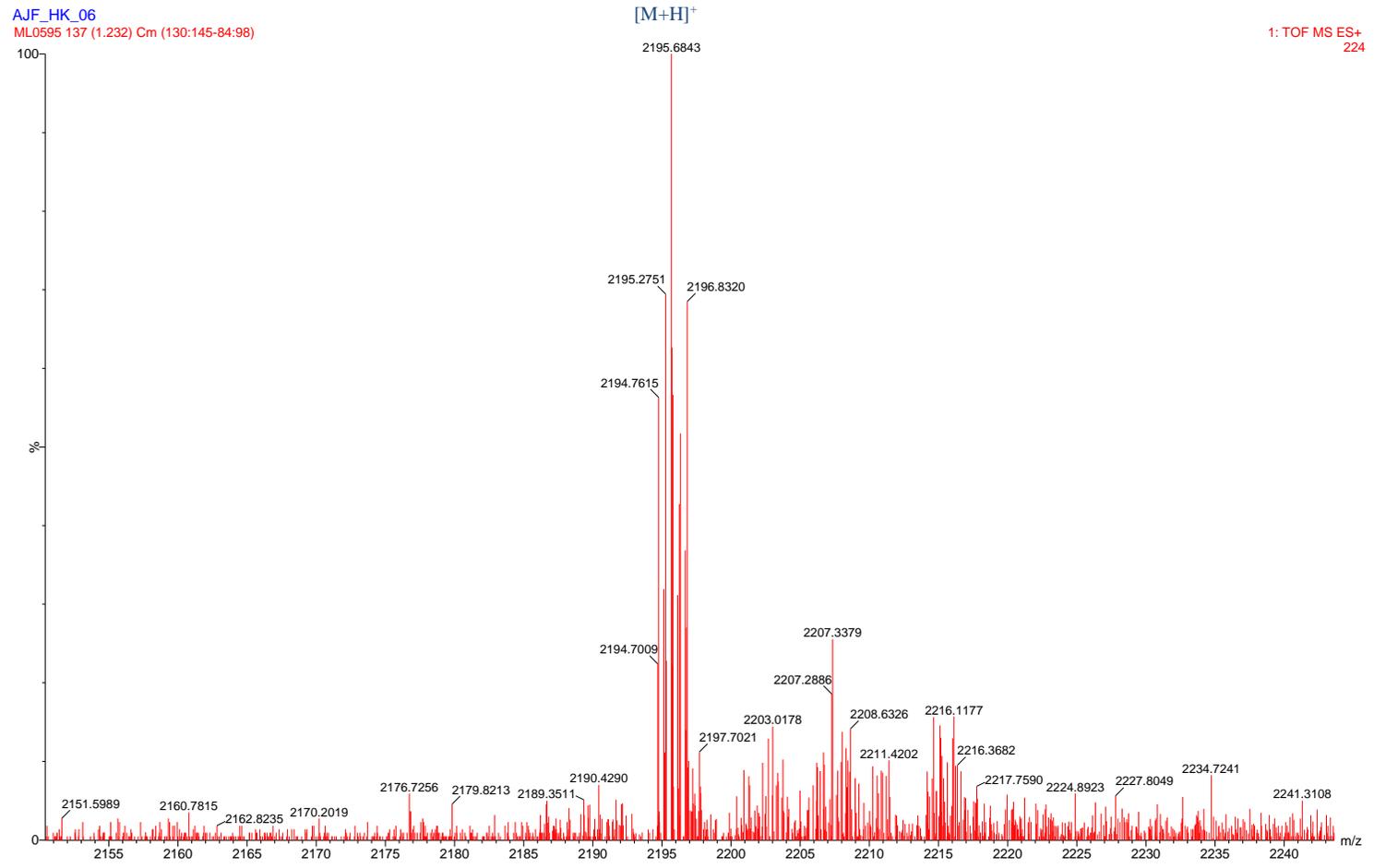
AJF_HK_06

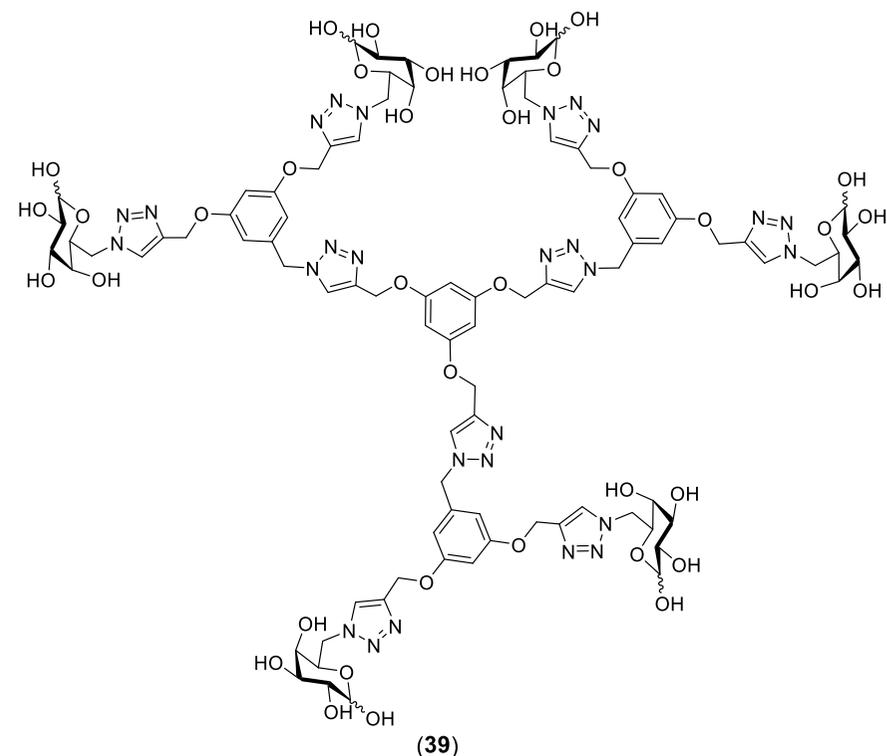
ML0595 137 (1.232) Cm (130:145-84:98)

1: TOF MS ES+
7.09e3



AJF_HK_06
ML0595 137 (1.232) Cm (130:145-84:98)





Elemental Composition Report: **AJF-HK-06**

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

2485 formula(e) evaluated with 1 results within limits (up to 20 closest results for each mass)

Elements Used:

C: 90-90 H: 0-180 N: 0-50 O: 0-50

Minimum:

-1.5

Maximum:

10.0 5.0 50.0

Mass

Calc. Mass

mDa

PPM

DBE

i-FIT

i-FIT (Norm)

Formula

2194.7615

2194.7611

0.4

0.2

48.5

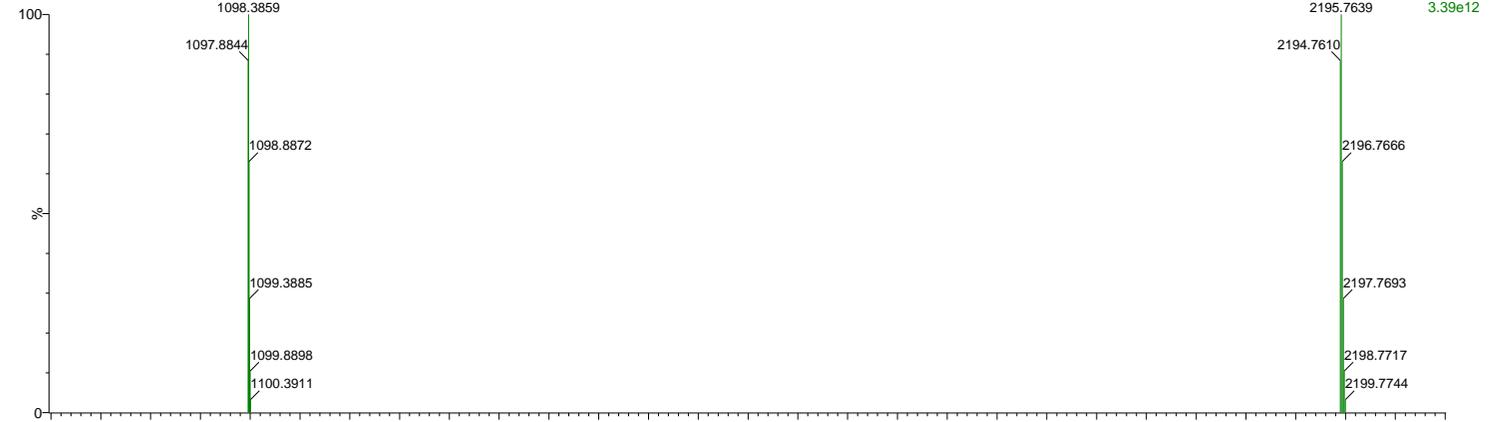
193.1

0.0

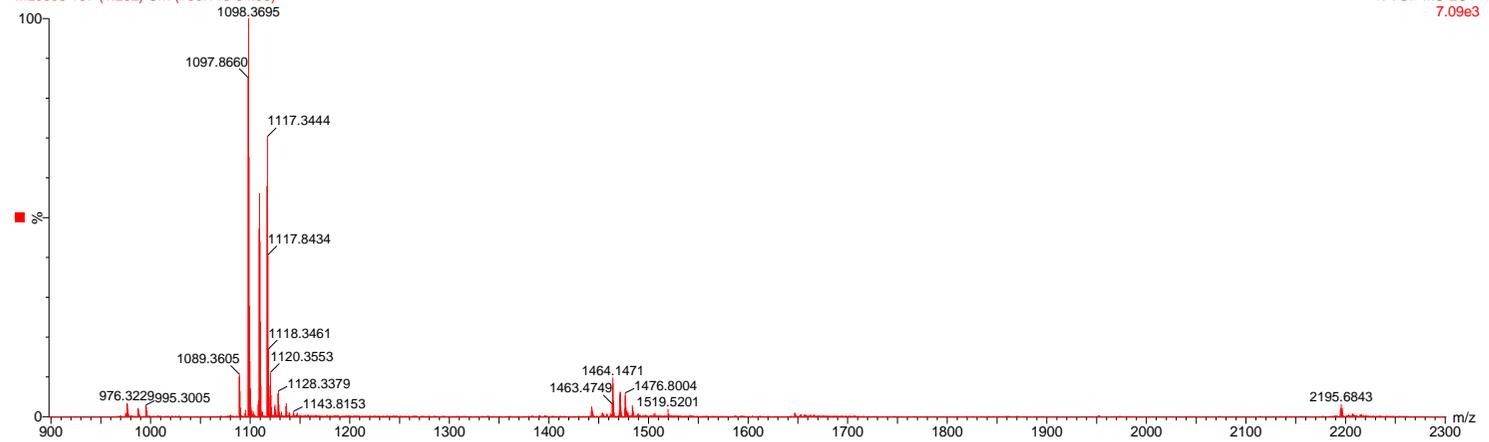
C₉₀ H₁₁₂ N₂₇ O₃₉

AJF_HK_06

ML0595 (0.030) Is (1.00,1.00) C90H111N27O39



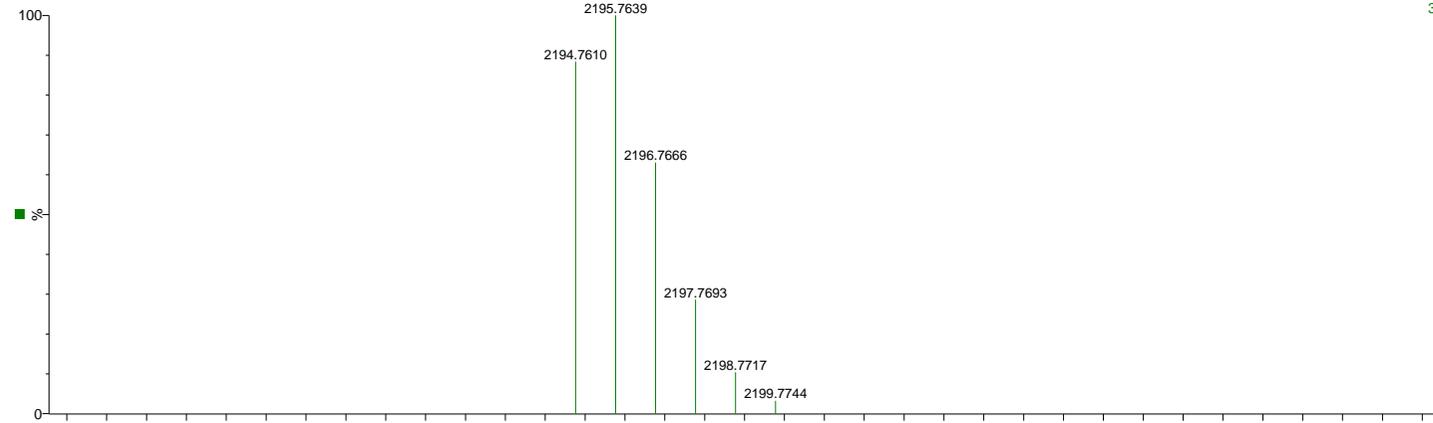
ML0595 137 (1.232) Cm (130:145-84:98)



AJF_HK_06

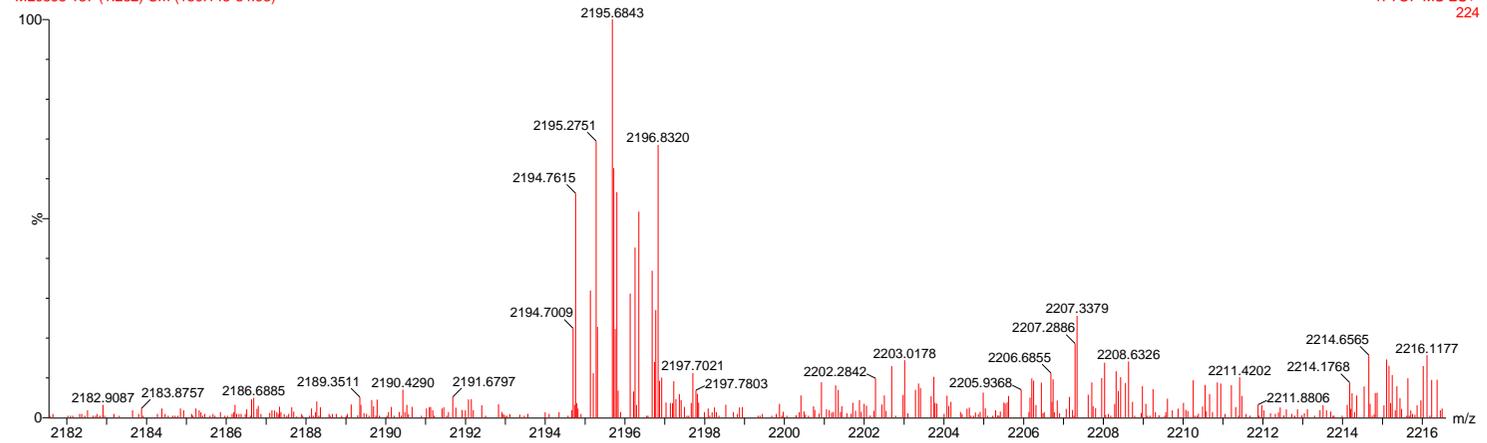
ML0595 (0.030) Is (1.00,1.00) C90H111N27O39

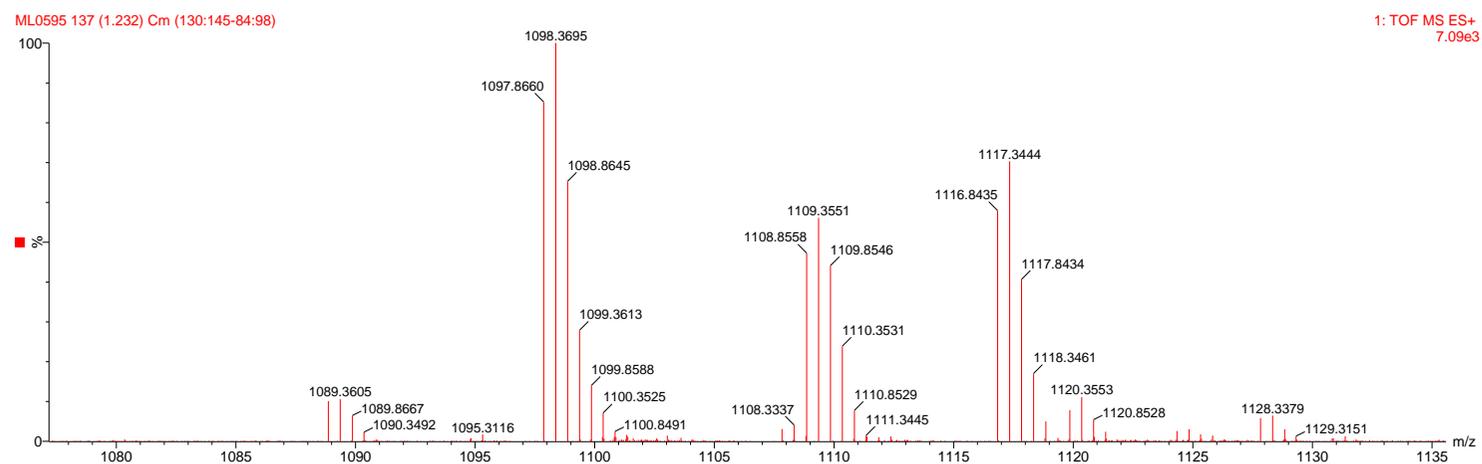
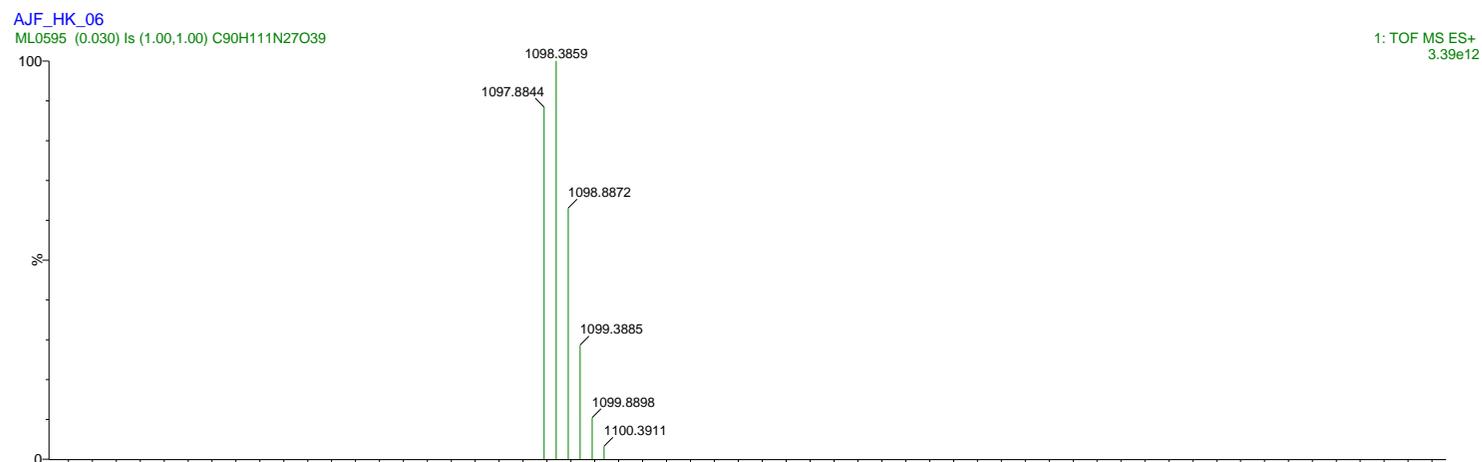
1: TOF MS ES+
3.39e12



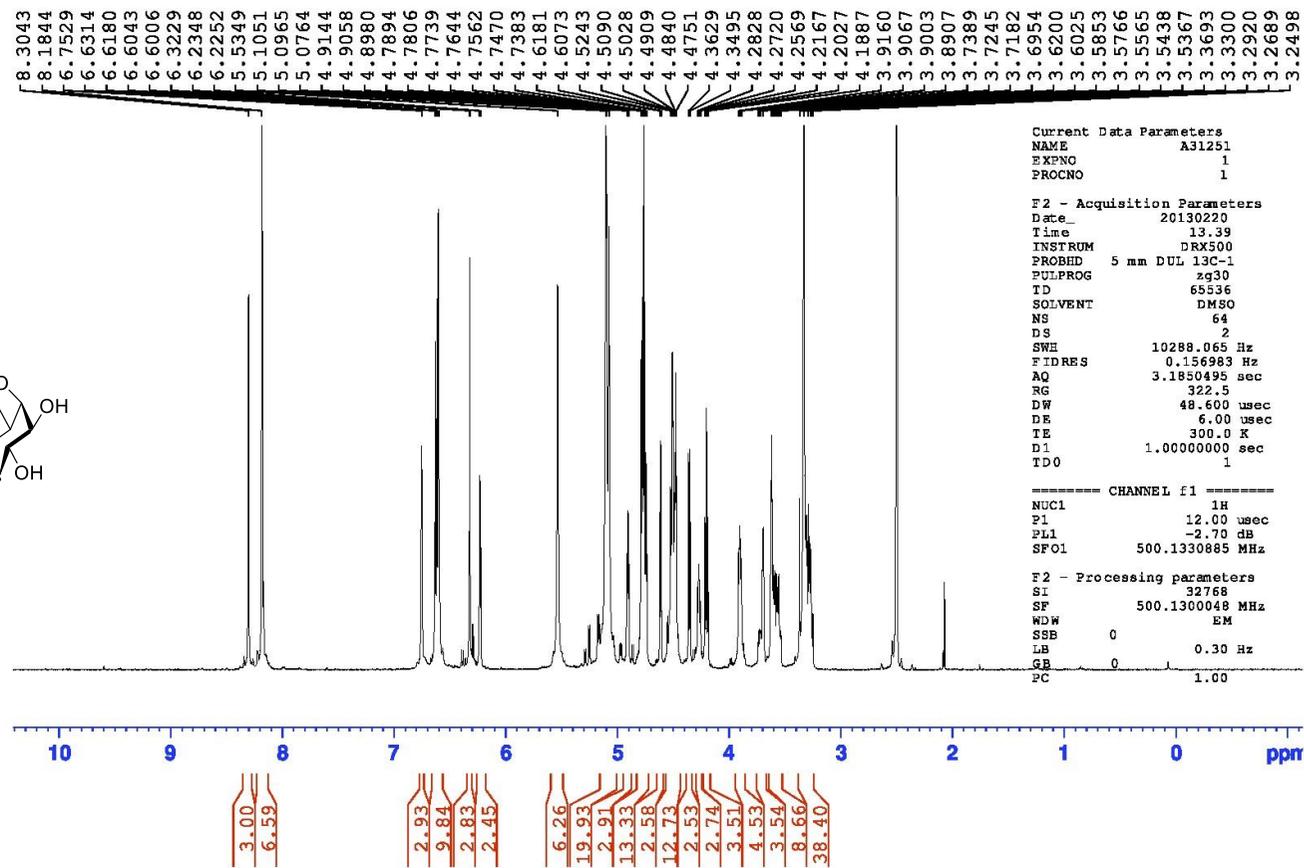
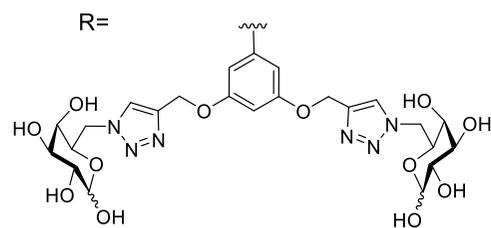
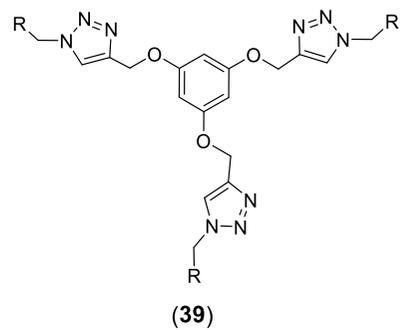
ML0595 137 (1.232) Cm (130:145-84:98)

1: TOF MS ES+
224

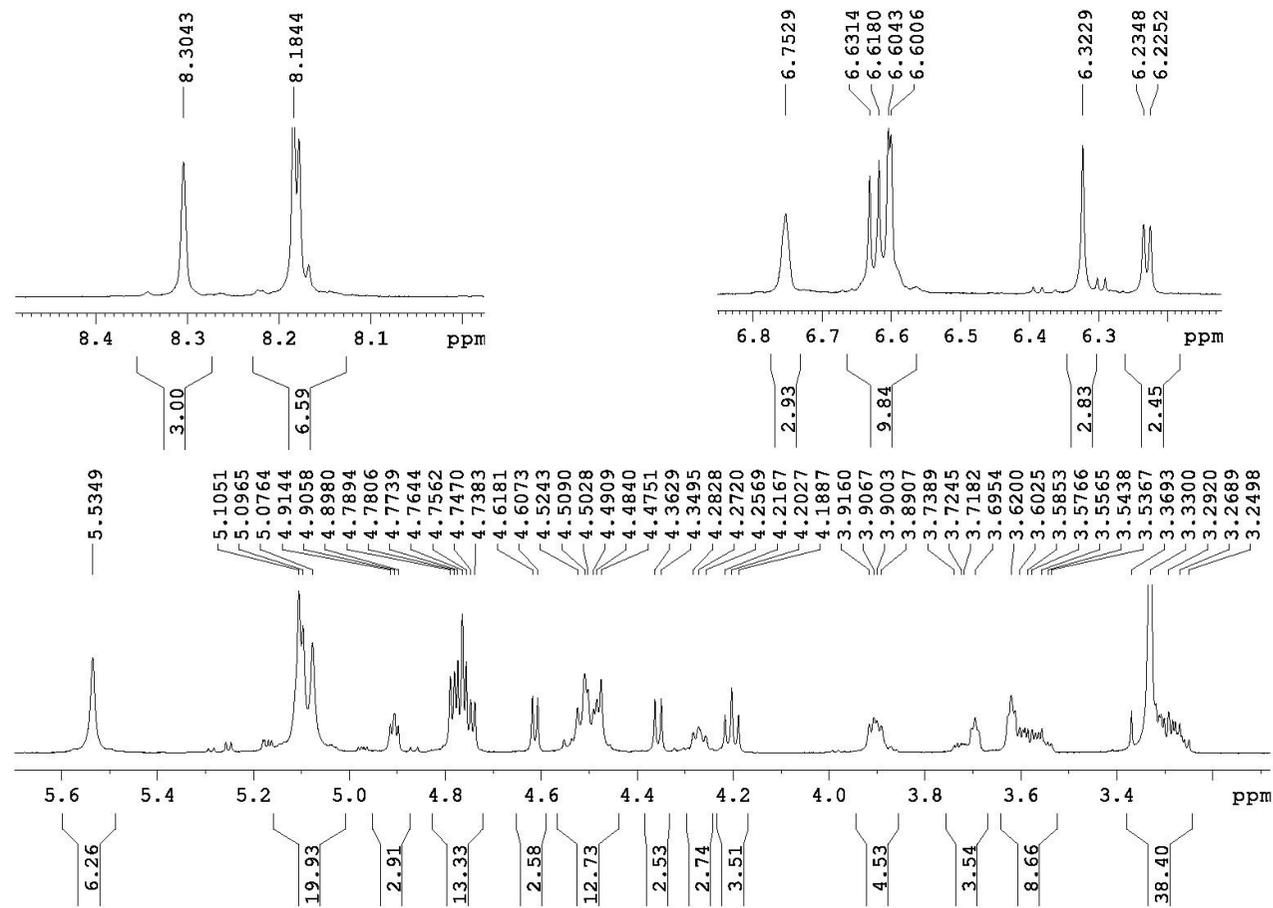




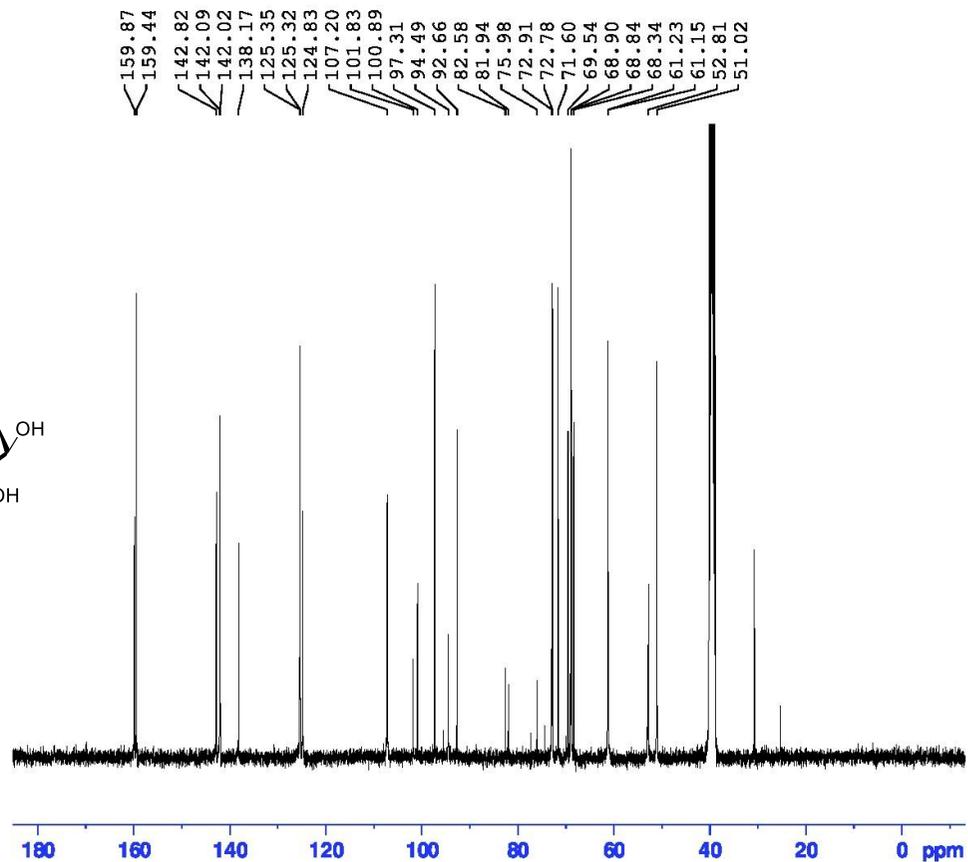
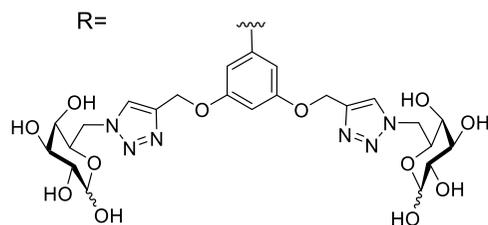
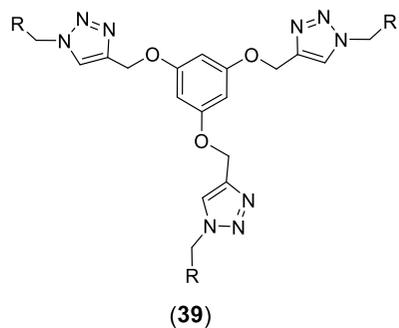
Appendix B34: HRMS (ESI) spectra of compound (39).



Appendix B35: ¹H NMR spectrum of compound (39).



Appendix B36: Selected areas ^1H NMR of compound (39).



Current Data Parameters
 NAME B24056
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20130223
 Time 8.41
 INSTRUM AV400
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 16384
 SOLVENT DMSO
 NS 40960
 DS 2
 SWE 23980.814 Hz
 FIDRES 1.463673 Hz
 AQ 0.3416064 sec
 RG 18390.4
 DW 20.850 usec
 DE 20.00 usec
 TE 300.0 K
 D1 0.69999999 sec
 D11 0.03000000 sec
 TDO 1

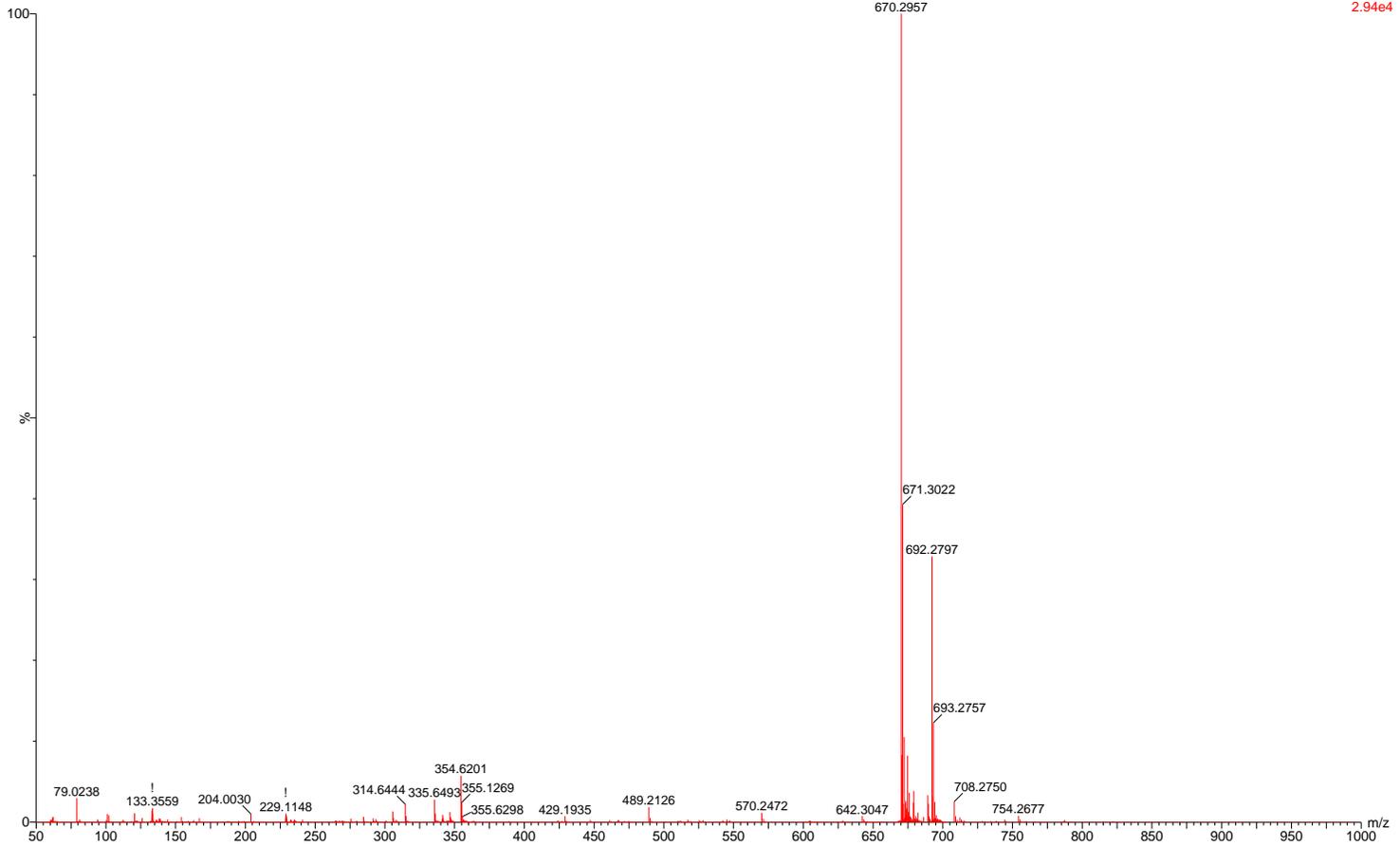
===== CHANNEL f1 =====
 NUC1 13C
 P1 8.00 usec
 PL1 -2.80 dB
 PL1W 57.91424179 W
 SFO1 100.5976818 MHz

===== CHANNEL f2 =====
 CPDPRG [2] waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 -3.20 dB
 PL12 14.30 dB
 PL13 15.30 dB
 PL2W 15.00949383 W
 PL12W 0.26691073 W
 PL13W 0.21201472 W
 SFO2 400.0316001 MHz

F2 - Processing parameters
 SI 32768
 SF 100.5876711 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Appendix B37: ¹³C NMR spectrum of compound (39).

AJF_HK_07
ML0596 186 (1.674)



Elemental Composition Report: **AJF-HK-07**

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

1041 formula(e) evaluated with 1 results within limits (up to 20 closest results for each mass)

Elements Used:

C: 30-30 H: 0-180 N: 0-50 O: 0-50

Minimum:

-1.5

Maximum:

10.0

5.0

50.0

Mass

Calc. Mass

mDa

PPM

DBE

i-FIT

i-FIT (Norm)

Formula

670.2957

670.2949

0.8

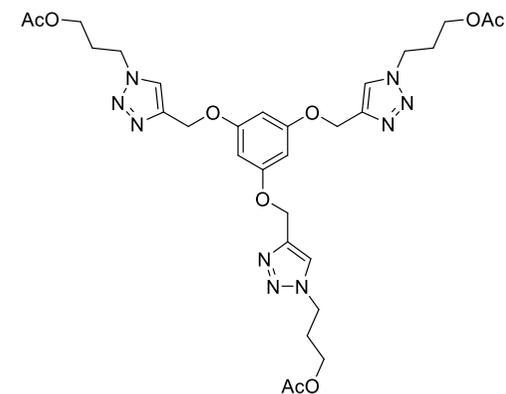
1.2

15.5

68.5

0.0

C30 H40 N9 O9

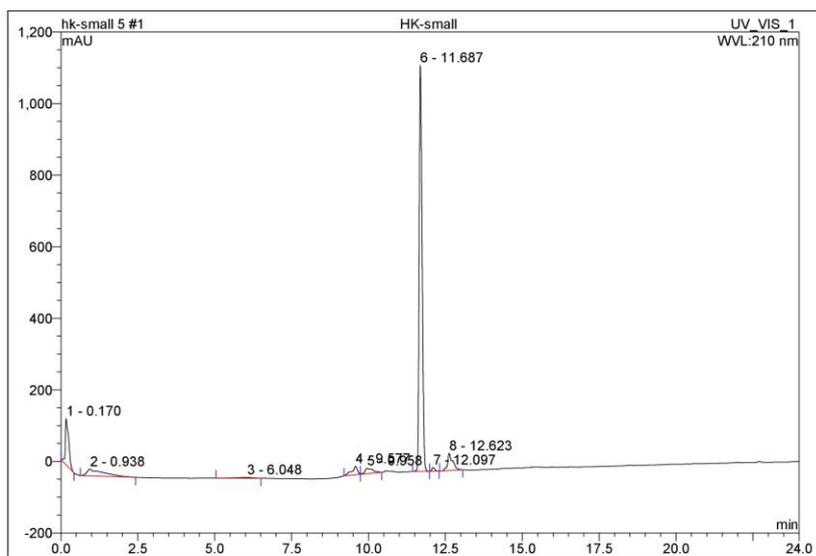


(54)

Appendix B38: HRMS (ESI) spectra of compound (54).

1 HK-small

Sample Name:	HK-small	Injection Volume:	20.0
Vial Number:	RA1	Channel:	UV_VIS_1
Sample Type:	unknown	Wavelength:	210
Control Program:	poly-p4 28min +230nm	Bandwidth:	10
Quantif. Method:	dna method	Dilution Factor:	1.0000
Recording Time:	17/9/2013 14:47	Sample Weight:	1.0000
Run Time (min):	24.00	Sample Amount:	1.0000

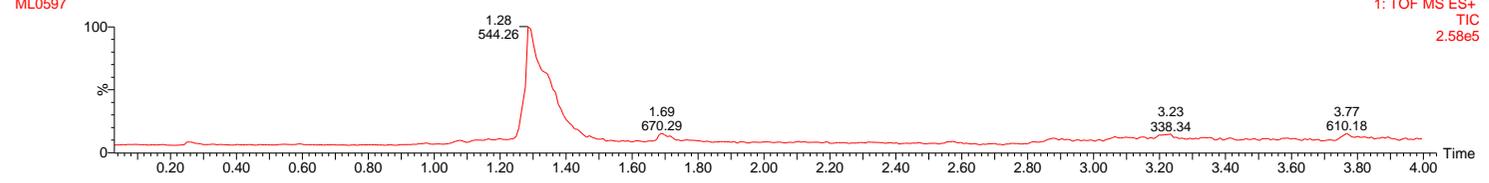
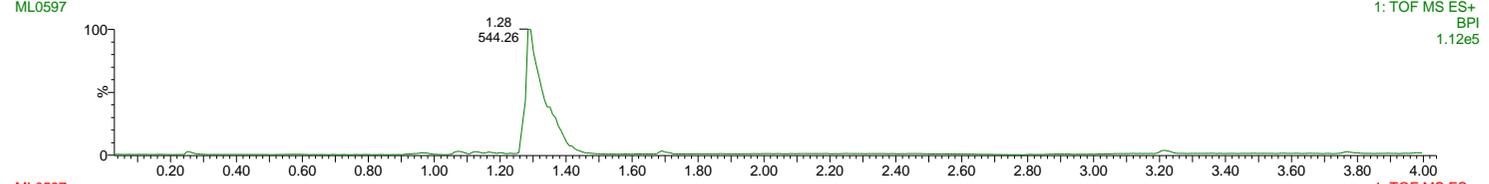
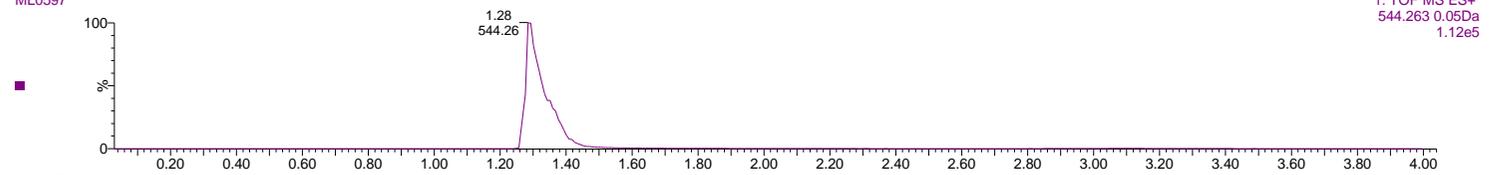
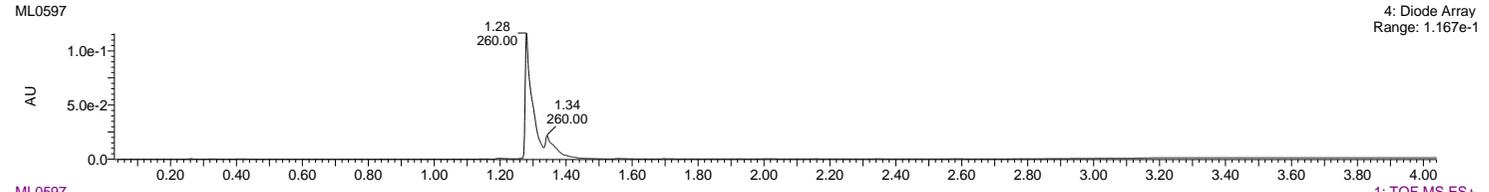
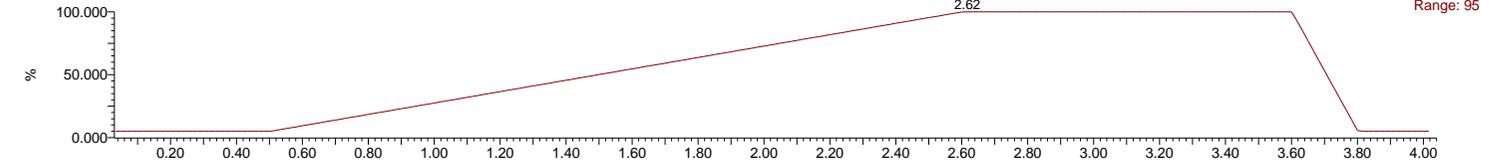


No.	Ret. Time min	Peak Name	Height mAU	Area mAU*min	Rel. Area %	Amount	Type
1	0.17	n.a.	129.971	16.784	9.52	n.a.	BMB
2	0.94	n.a.	19.025	11.923	6.76	n.a.	BMB
3	6.05	n.a.	2.234	1.380	0.78	n.a.	BMB
4	9.58	n.a.	23.598	5.128	2.91	n.a.	BM
5	9.96	n.a.	14.881	4.435	2.52	n.a.	MB
6	11.69	n.a.	1136.193	126.265	71.60	n.a.	BMB
7	12.10	n.a.	10.306	1.132	0.64	n.a.	BMB
8	12.62	n.a.	48.214	9.292	5.27	n.a.	BMB
Total:			1384.423	176.339	100.00	0.000	

default/Integration

Chromeleon (c) Dionex 1996-2006
Version 6.80 SP4 Build 2361 (130805)*Appendix B39: HPL chromatogram of compound (51).*

AJF_HK_08
ML0597

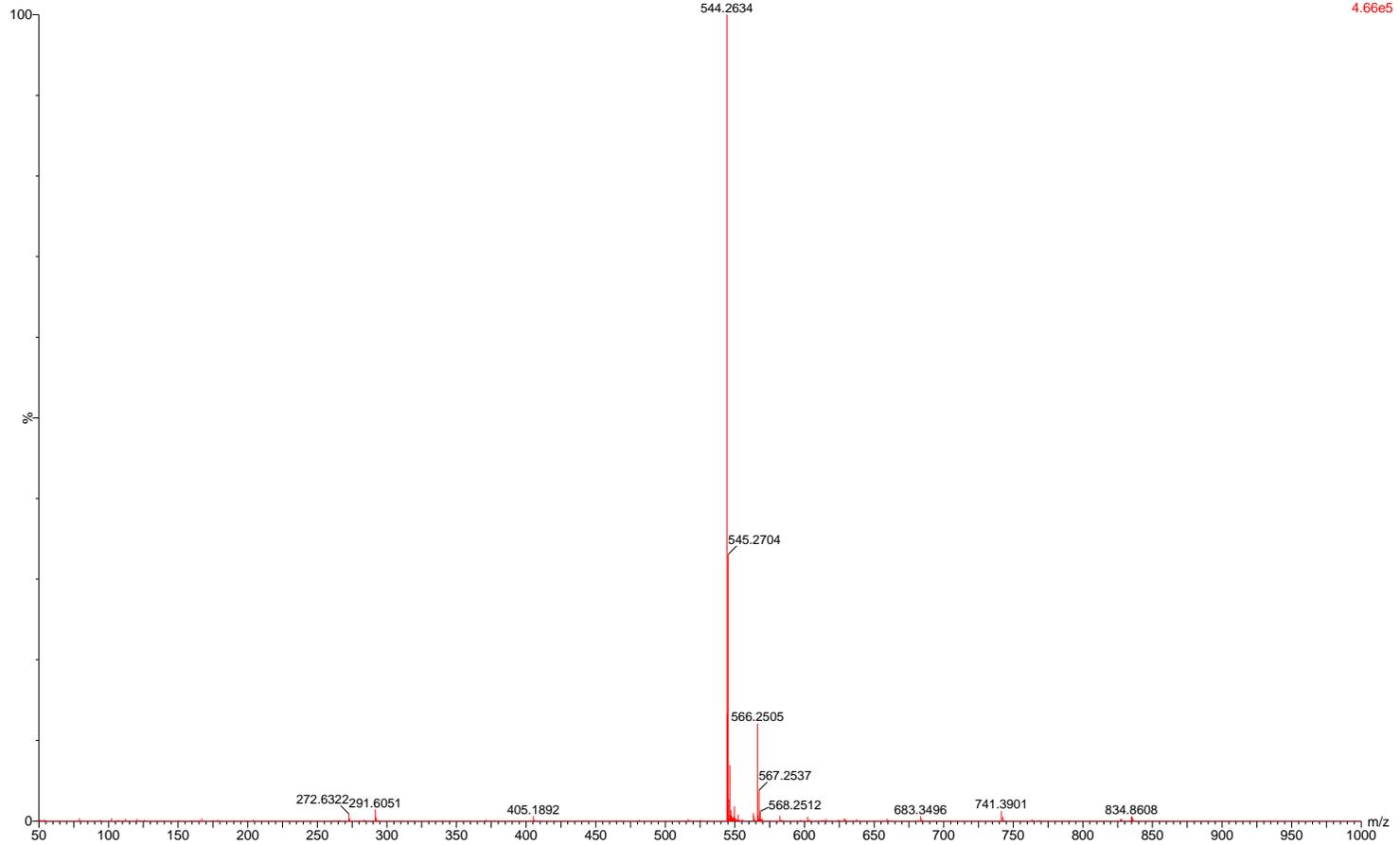


AJF_HK_08

ML0597 142 (1.284) Cm (142:146)

[M+H]⁺

1: TOF MS ES+
4.66e5



Elemental Composition Report: **AJF-HK-08**

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

694 formula(e) evaluated with 1 results within limits (up to 20 closest results for each mass)

Elements Used:

C: 24-24 H: 0-180 N: 0-50 O: 0-50

Minimum:

-1.5

Maximum:

10.0

5.0

50.0

Mass

Calc. Mass

mDa

PPM

DBE

i-FIT

i-FIT (Norm)

Formula

544.2634

544.2632

0.2

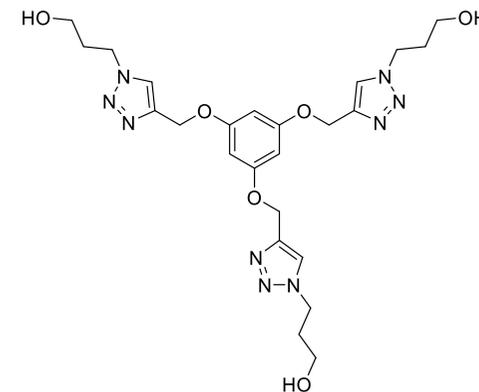
0.4

12.5

325.5

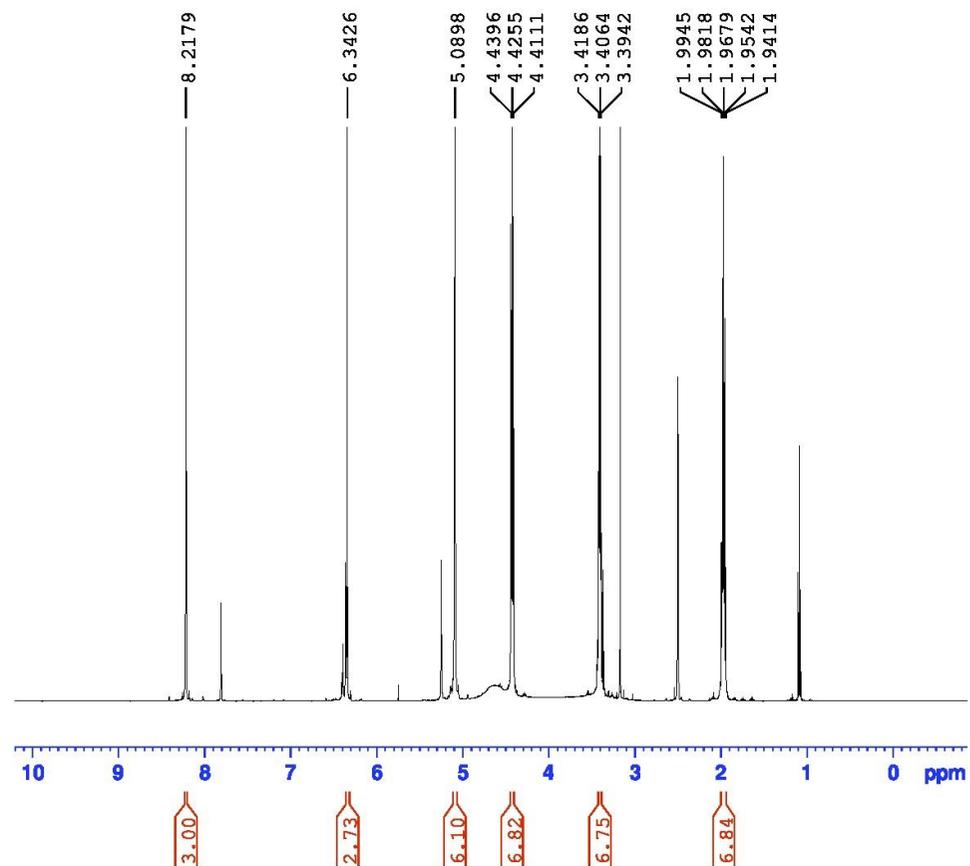
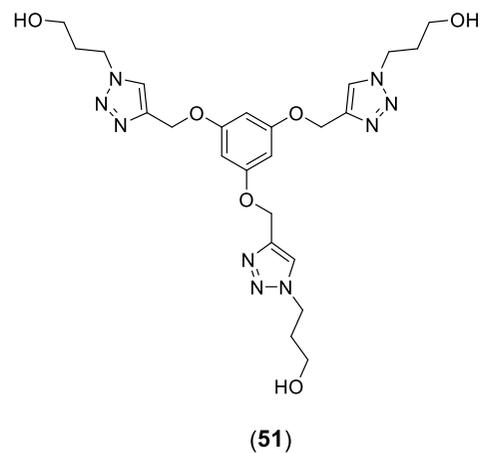
0.0

C₂₄ H₃₄ N₉ O₆



(51)

Appendix B40: HRMS (ESI) spectra of compound (51).



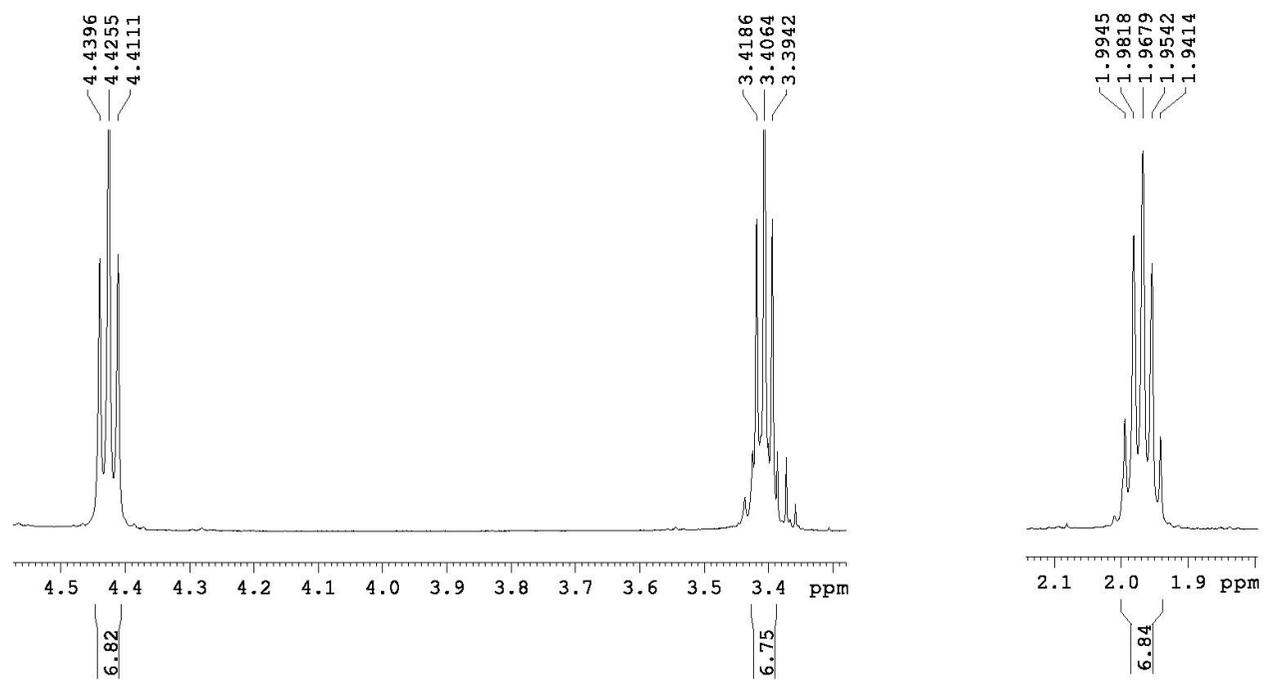
Current Data Parameters
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 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameter
 Date_ 20120302
 Time 15.13
 INSTRUM DRX500
 PROBED 5 mm DUL 13C-1
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 16
 DS 2
 SWH 10288.065 Hz
 FIDRES 0.156983 Hz
 AQ 3.1850495 sec
 RG 143.7
 DW 48.600 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

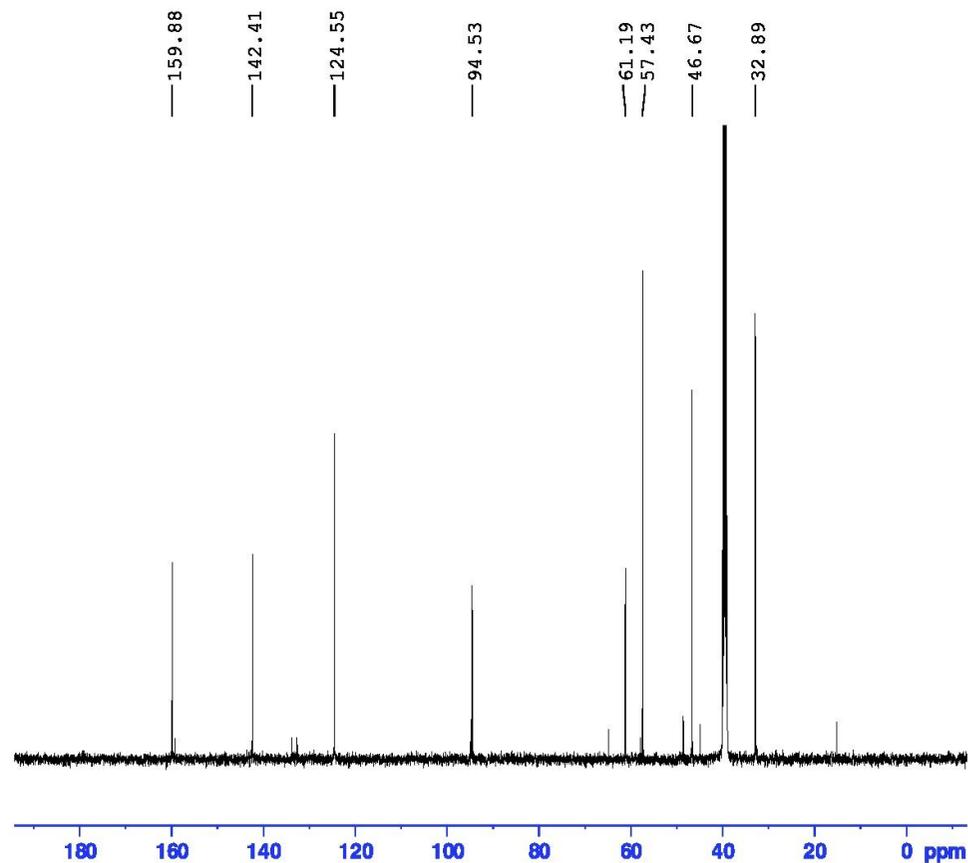
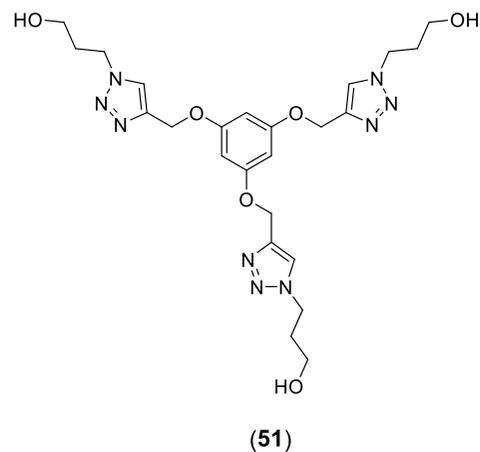
CHANNEL f1
 NUC1 1H
 P1 9.25 usec
 PL1 -2.70 dB
 SFO1 500.1330885 MHz

F2 - Processing parameters
 SI 32768
 SF 500.1300056 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Appendix B41: ¹H NMR spectrum of compound (51).



Appendix B42: Selected areas ¹H NMR of compound (51).



Current Data Parameters
 NAME A26278
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameter
 Date_ 20120302
 Time 17.12
 INSTRUM DRX500
 PROBHD 5 mm DUL 13C-1
 PULPROG zgpg30
 TD 16384
 SOLVENT DMSO
 NS 1024
 DS 4
 SWH 30120.482 Hz
 FIDRES 1.838408 Hz
 AQ 0.2719744 sec
 RG 8192
 DW 16.600 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.69999999 sec
 d11 0.03000000 sec
 DELTA 0.59999996 sec
 TDC 1

CHANNEL f1
 NUC1 13C
 P1 8.00 usec
 PL1 -2.30 dB
 SFO1 125.7703643 MHz

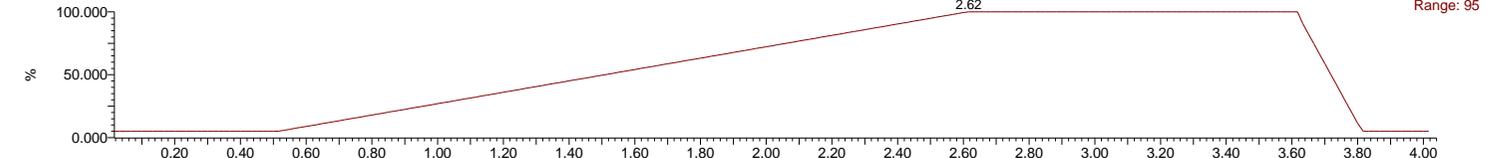
CHANNEL f2
 CPDPRG[2] waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -2.30 dB
 PL12 16.21 dB
 PL13 120.00 dB
 SFO2 500.1320005 MHz

F2 - Processing parameters
 SI 32768
 SF 125.7578517 MHz
 WDW EM
 SSB 0
 LB 2.00 Hz
 GB 0
 PC 1.40

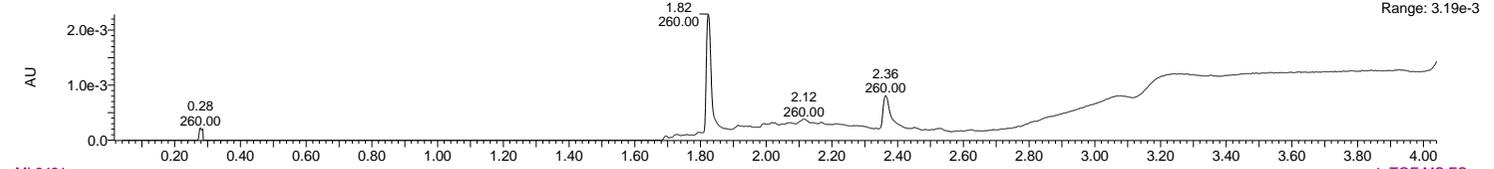
Appendix B43: ^{13}C NMR spectrum of compound (51).

AJF_HK-12-13

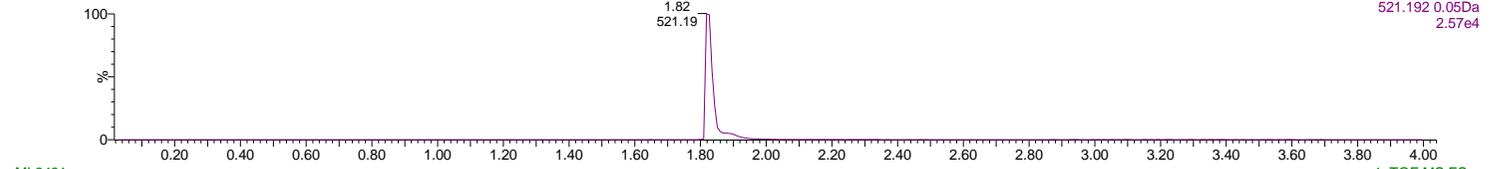
ML2421



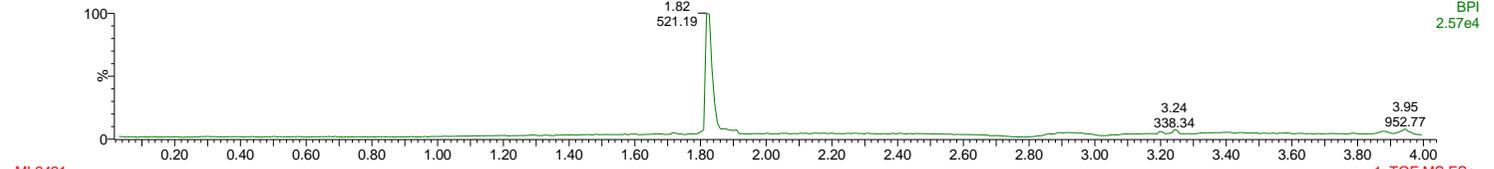
ML2421



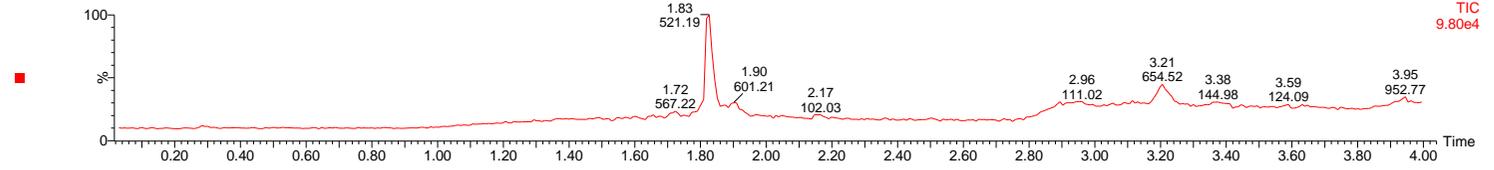
ML2421



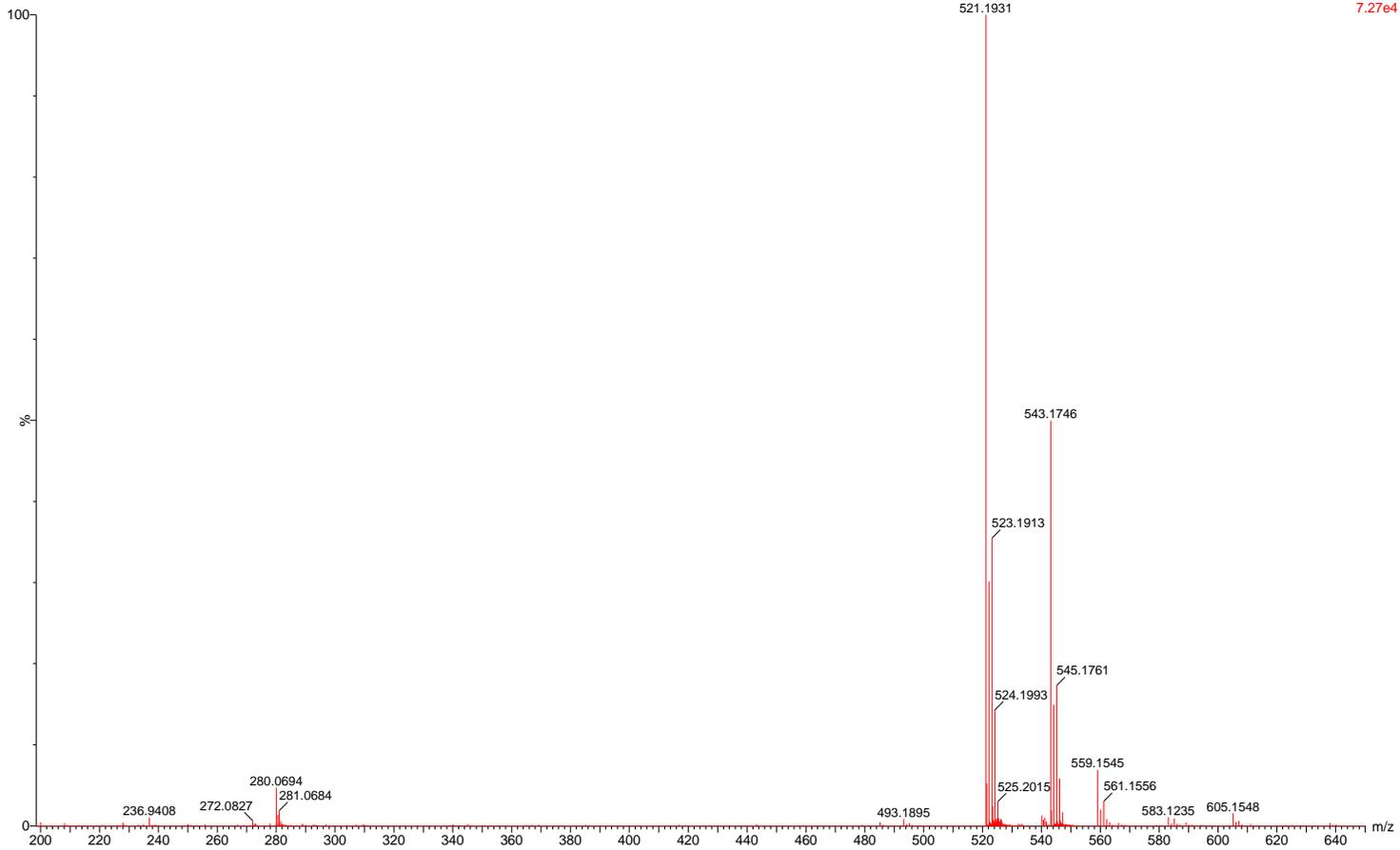
ML2421



ML2421



AJF_HK-12-13
ML2421 203 (1.826) Cm (201:205)



Elemental Composition Report: **AJF-HK-12-13**

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 80.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

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

Elements Used:

C: 23-23 H: 0-150 N: 0-30 O: 0-30 35Cl: 1-1

Minimum: -1.5

Maximum: 10.0 5.0 80.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
521.1931	521.1915	1.6	3.1	11.5	250.8	0.0	C23 H30 N6 O6 35Cl

Elemental Composition Report: **AJF-HK-12-13**

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 80.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

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

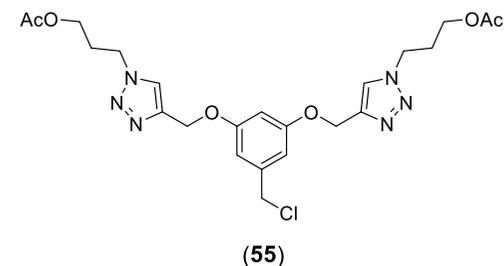
Elements Used:

C: 23-23 H: 0-150 N: 0-30 O: 0-30 35Cl: 1-1 Na: 1-1

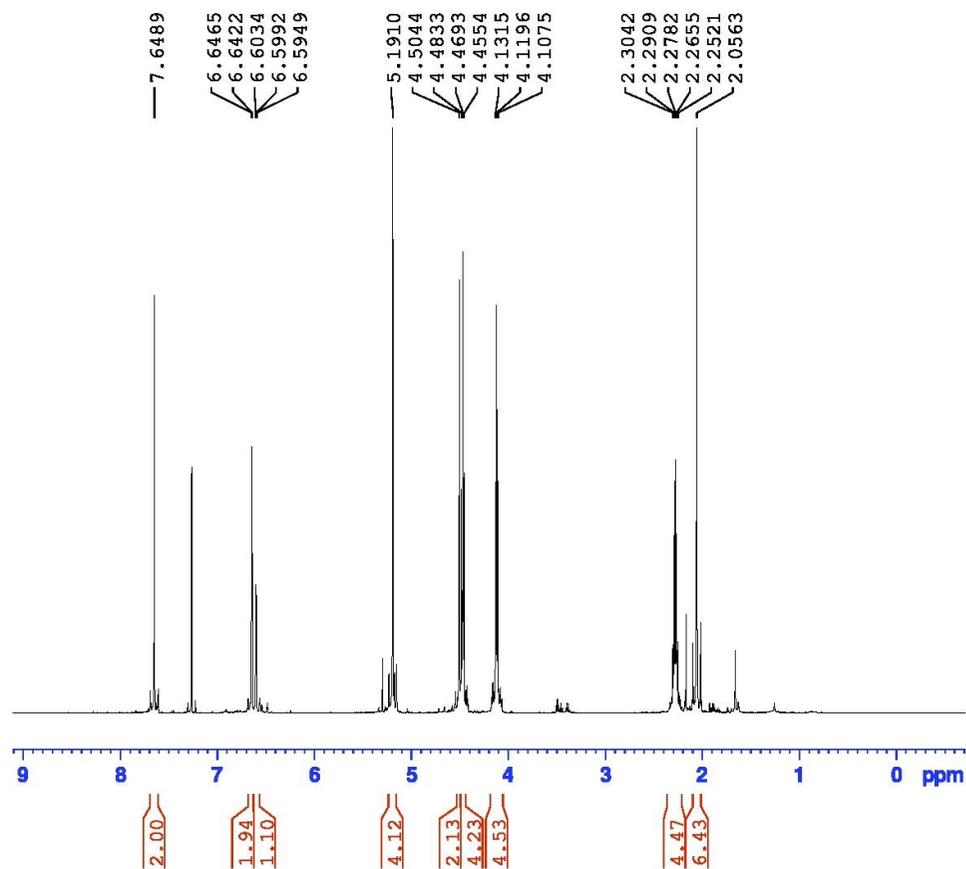
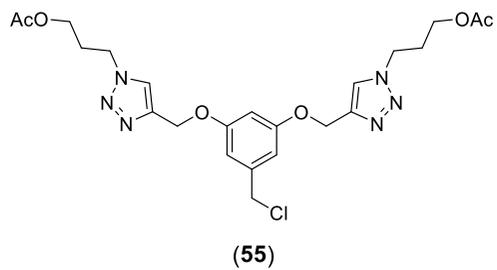
Minimum: -1.5

Maximum: 10.0 5.0 80.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
543.1746	543.1735	1.1	2.0	11.5	199.3	0.0	C23 H29 N6 O6 35Cl Na



Appendix B44: HRMS (ESI) spectra of compound (55).



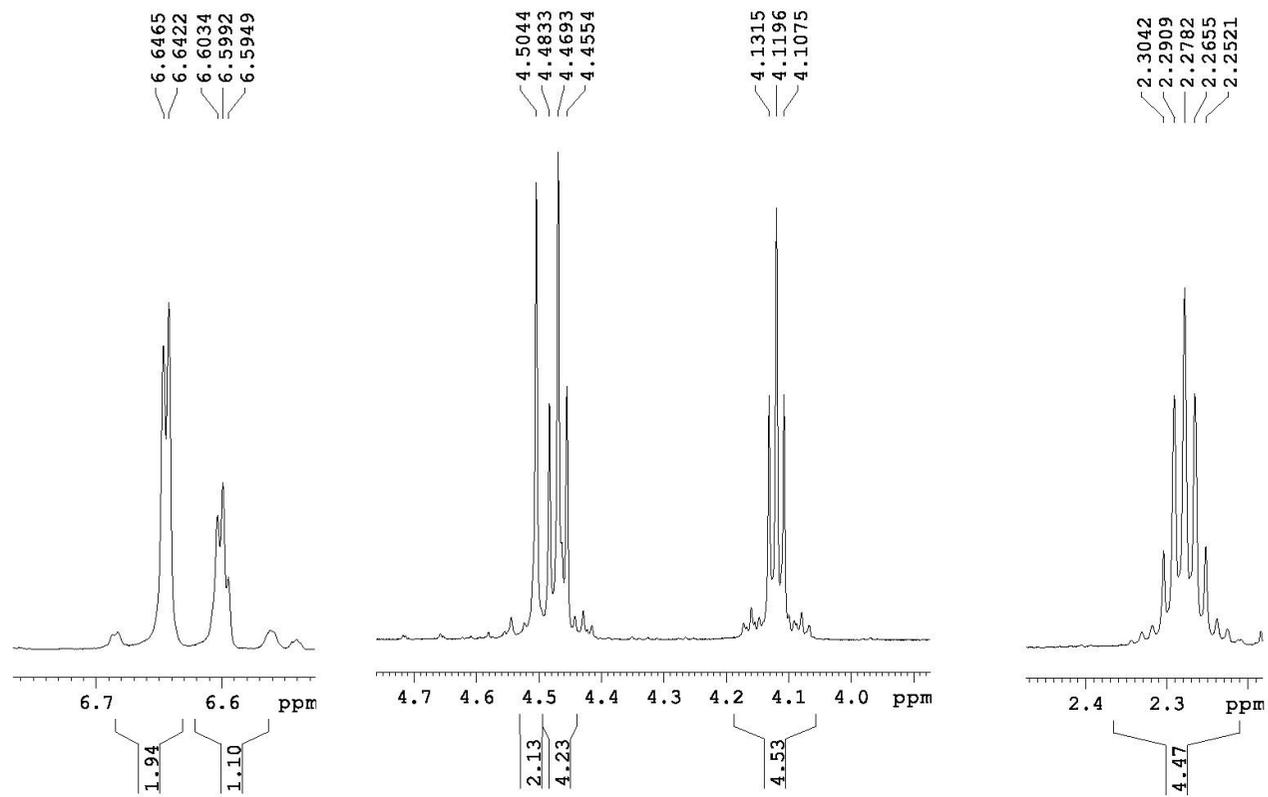
Current Data Parameters
 NAME A32201
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameter
 Date_ 20130405
 Time 19.04
 INSTRUM DRX500
 PROBED 5 mm DUL 13C-1
 PULPROG zg30
 ID 65536
 SOLVENT CDCl3
 NS 32
 DS 2
 SWH 10288.065 Hz
 FIDRES 0.156983 Hz
 AQ 3.1850495 sec
 RG 574.7
 DW 48.600 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

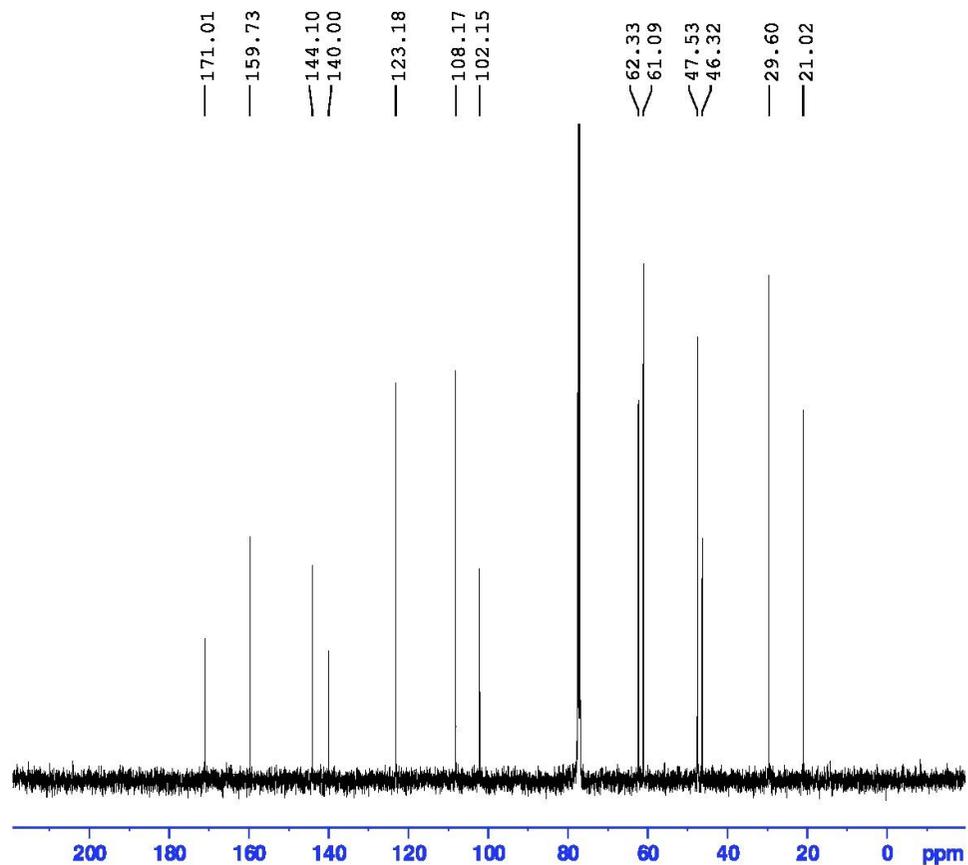
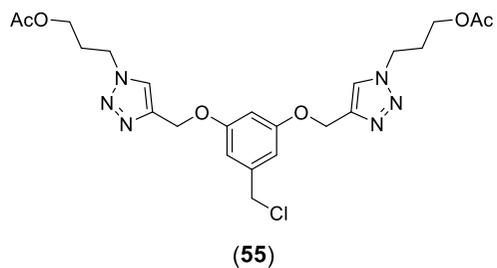
CHANNEL f1
 NUC1 1H
 P1 12.00 usec
 PL1 -2.70 dB
 SFO1 500.1330885 MHz

F2 - Processing parameters
 SI 32768
 SF 500.1300108 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Appendix B45: ¹H NMR spectrum of compound (55).



Appendix B46: Selected areas ^1H NMR of compound (55).



Current Data Parameters
 NAME A32201
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameter
 Date_ 20130405
 Time 19.40
 INSTRUM DRX500
 PROBED 5 mm DUL 13C-1
 PULPROG zgpg30
 TD 16384
 SOLVENT CDCl3
 NS 2048
 DS 4
 SWH 30120.482 Hz
 FIDRES 1.838408 Hz
 AQ 0.2719744 sec
 RG 11585.2
 DW 16.600 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.69999999 sec
 d11 0.03000000 sec
 DELTA 0.59999996 sec
 TD0 1

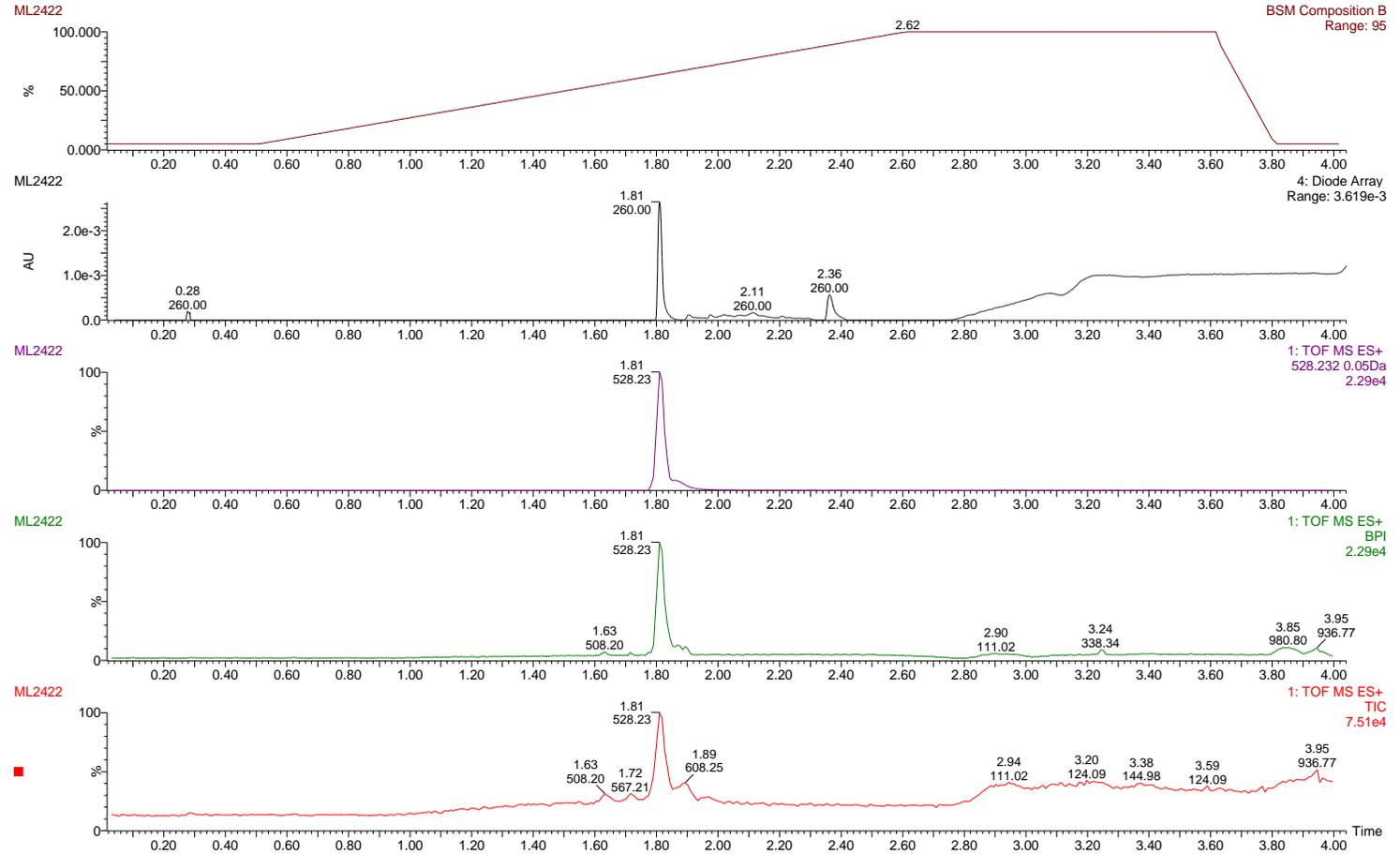
CHANNEL f1
 NUC1 13C
 P1 8.00 usec
 PL1 -2.30 dB
 SFO1 125.7703643 MHz

CHANNEL f2
 CPDPRG[2] waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -2.30 dB
 PL12 12.84 dB
 PL13 120.00 dB
 SFO2 500.1320005 MHz

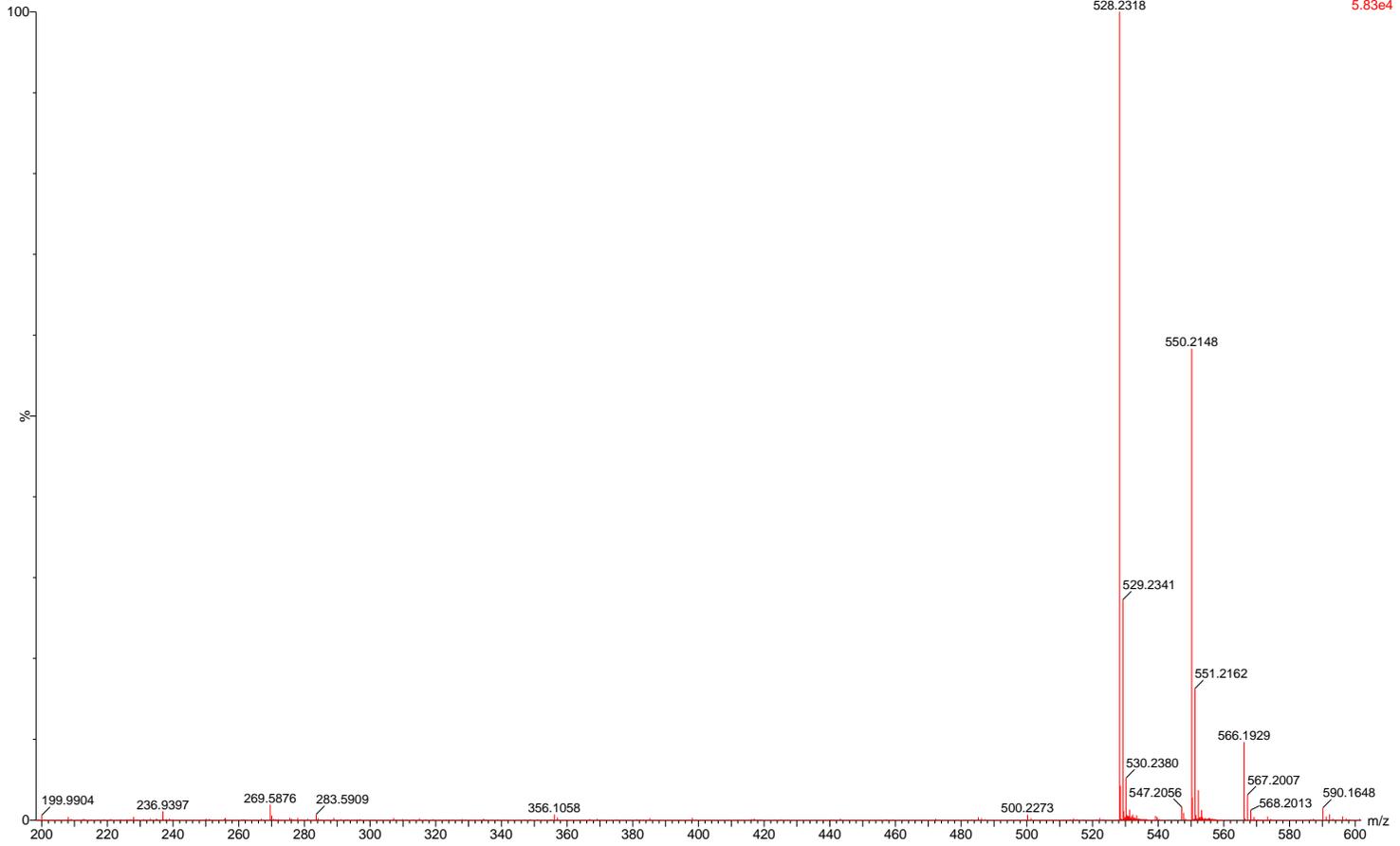
F2 - Processing parameters
 SI 32768
 SF 125.7577639 MHz
 WDW EM
 SSB 0
 LB 2.00 Hz
 GB 0
 PC 1.40

Appendix B47: ¹³C NMR spectrum of compound (55).

AJF_HK-13-13



AJF_HK-13-13
ML2422 201 (1.810) Cm (200:203)



Elemental Composition Report: **AJF-HK-13-13**

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 80.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

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

Elements Used:

C: 23-23 H: 0-150 N: 0-30 O: 0-30

Minimum: -1.5

Maximum: 10.0 5.0 80.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
528.2318	528.2319	-0.1	-0.2	13.5	187.4	0.0	C23 H30 N9 O6

Elemental Composition Report: **AJF-HK-13-13**

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 80.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

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

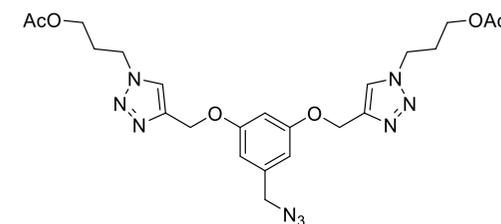
Elements Used:

C: 23-23 H: 0-150 N: 0-30 O: 0-30 Na: 1-1

Minimum: -1.5

Maximum: 10.0 5.0 80.0

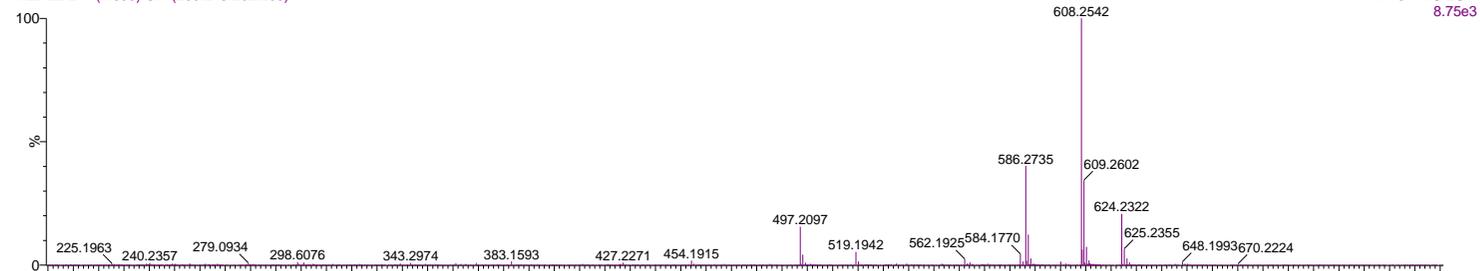
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
550.2148	550.2138	1.0	1.8	13.5	159.0	0.0	C23 H29 N9 O6 Na



(56)

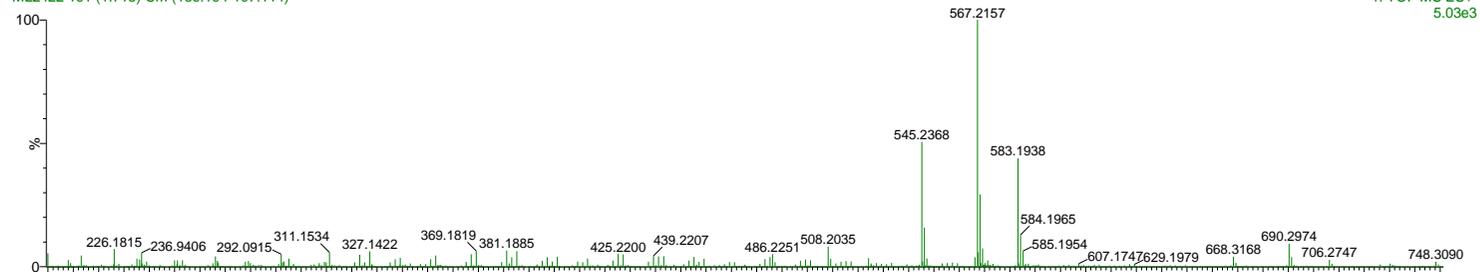
AJF_HK-13-13

ML2422 211 (1.893) Cm (209:213-202:206)



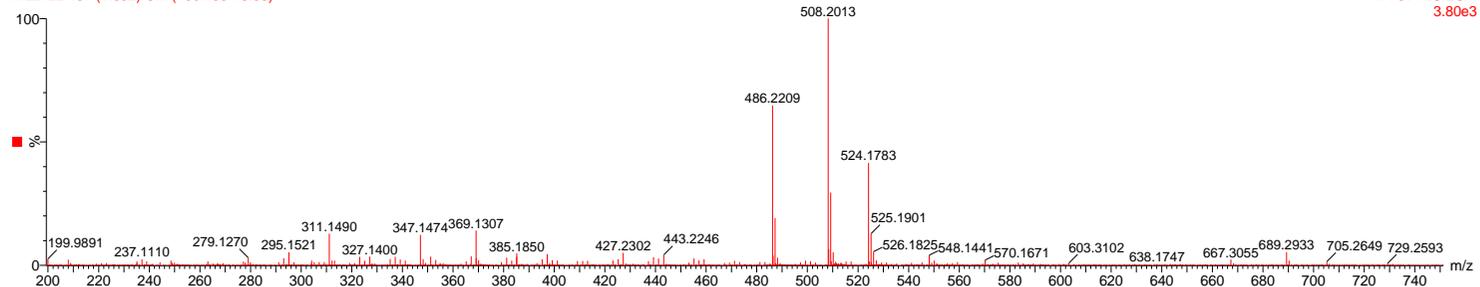
1: TOF MS ES+
8.75e3

ML2422 191 (1.715) Cm (189:194-107:114)



1: TOF MS ES+
5.03e3

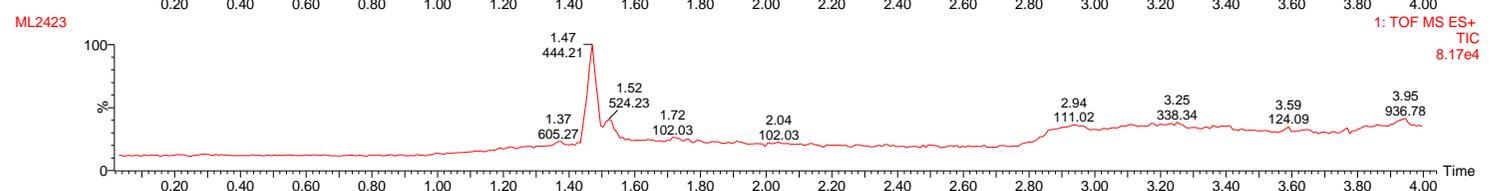
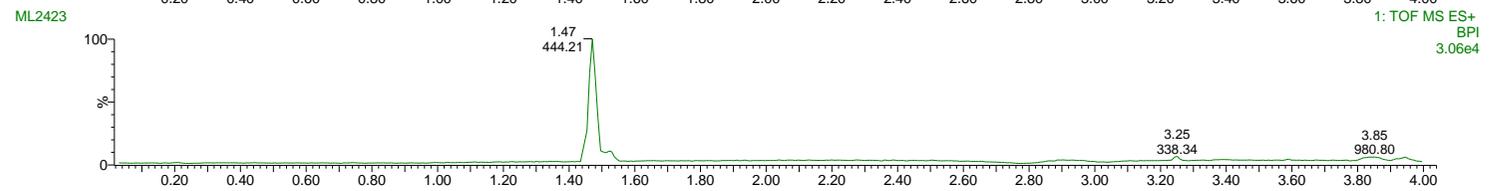
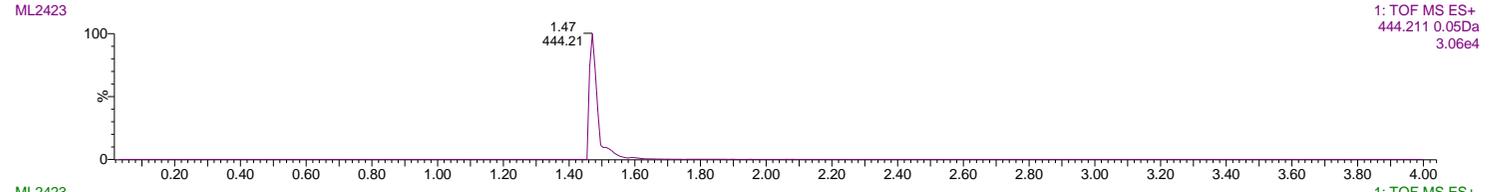
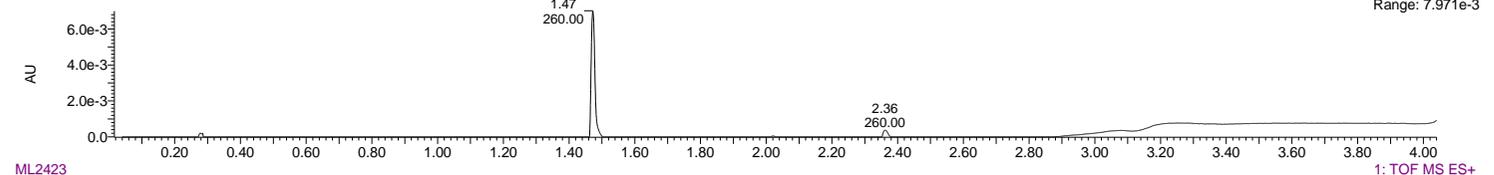
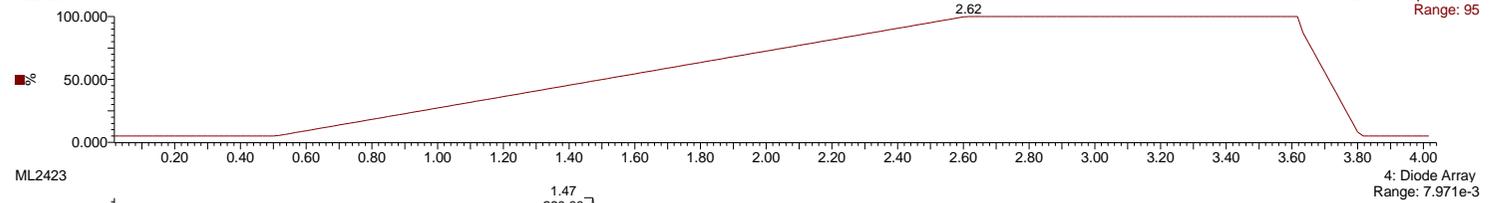
ML2422 181 (1.632) Cm (180:183-78:85)



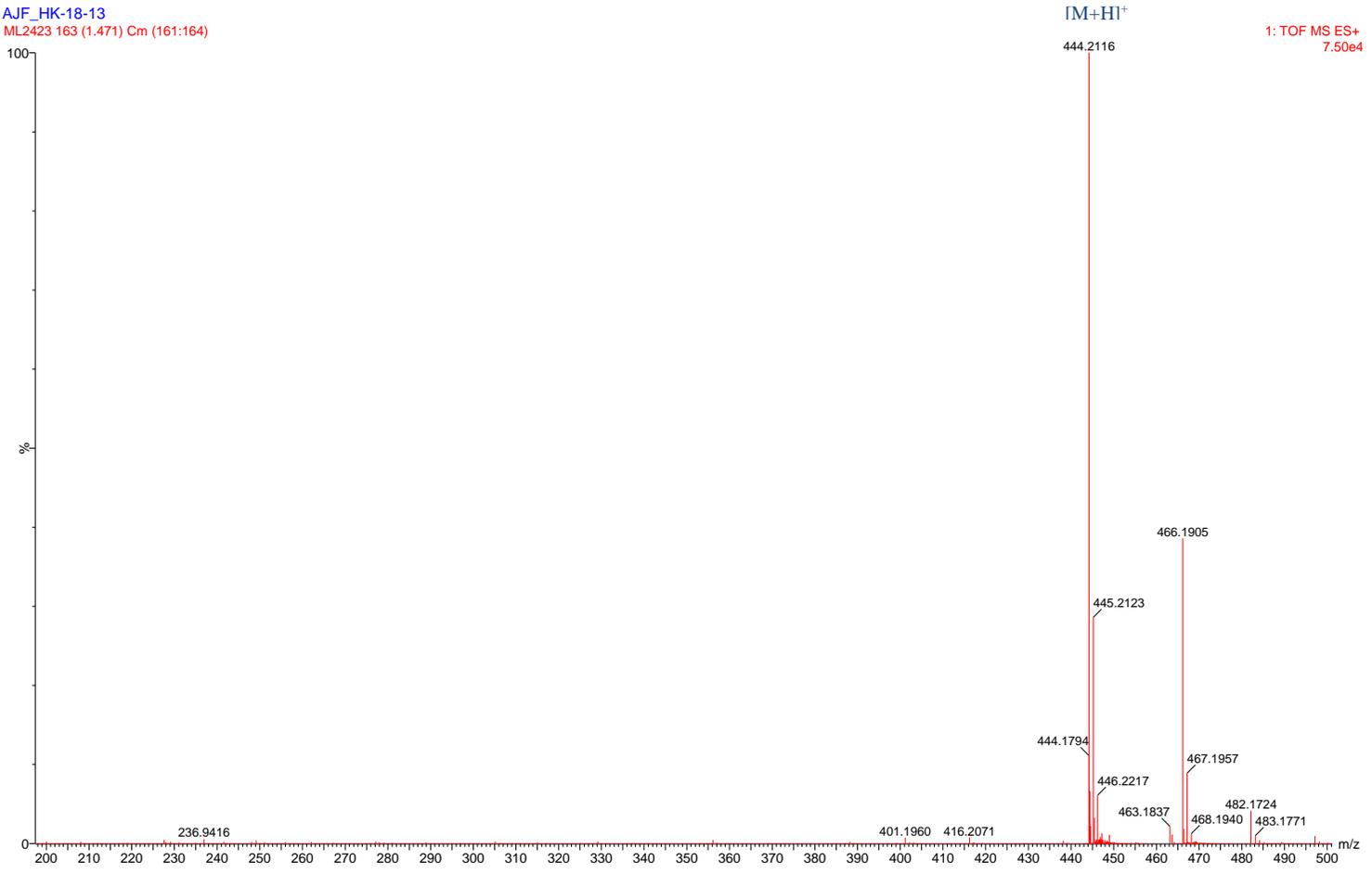
1: TOF MS ES+
3.80e3

Appendix B48: HRMS (ESI) spectra of compound (56).

AJF_HK-18-13
ML2423



AJF_HK-18-13
ML2423 163 (1.471) Cm (161:164)



Elemental Composition Report: **AJF-HK-18-13**

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 80.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

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

Elements Used:

C: 19-19 H: 0-150 N: 0-30 O: 0-30

Minimum:

-1.5

Maximum:

10.0

5.0

80.0

Mass

Calc. Mass

mDa

PPM

DBE

i-FIT

i-FIT (Norm)

Formula

444.2116

444.2108

0.8

1.8

11.5

279.5

0.0

C₁₉ H₂₆ N₉ O₄

Elemental Composition Report: **AJF-HK-18-13**

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 80.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

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

Elements Used:

C: 19-19 H: 0-150 N: 0-30 O: 0-30 Na: 1-1

Minimum:

-1.5

Maximum:

10.0

5.0

80.0

Mass

Calc. Mass

mDa

PPM

DBE

i-FIT

i-FIT (Norm)

Formula

466.1905

466.1927

-2.2

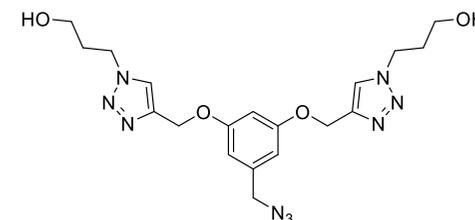
-4.7

11.5

187.7

0.0

C₁₉ H₂₅ N₉ O₄ Na

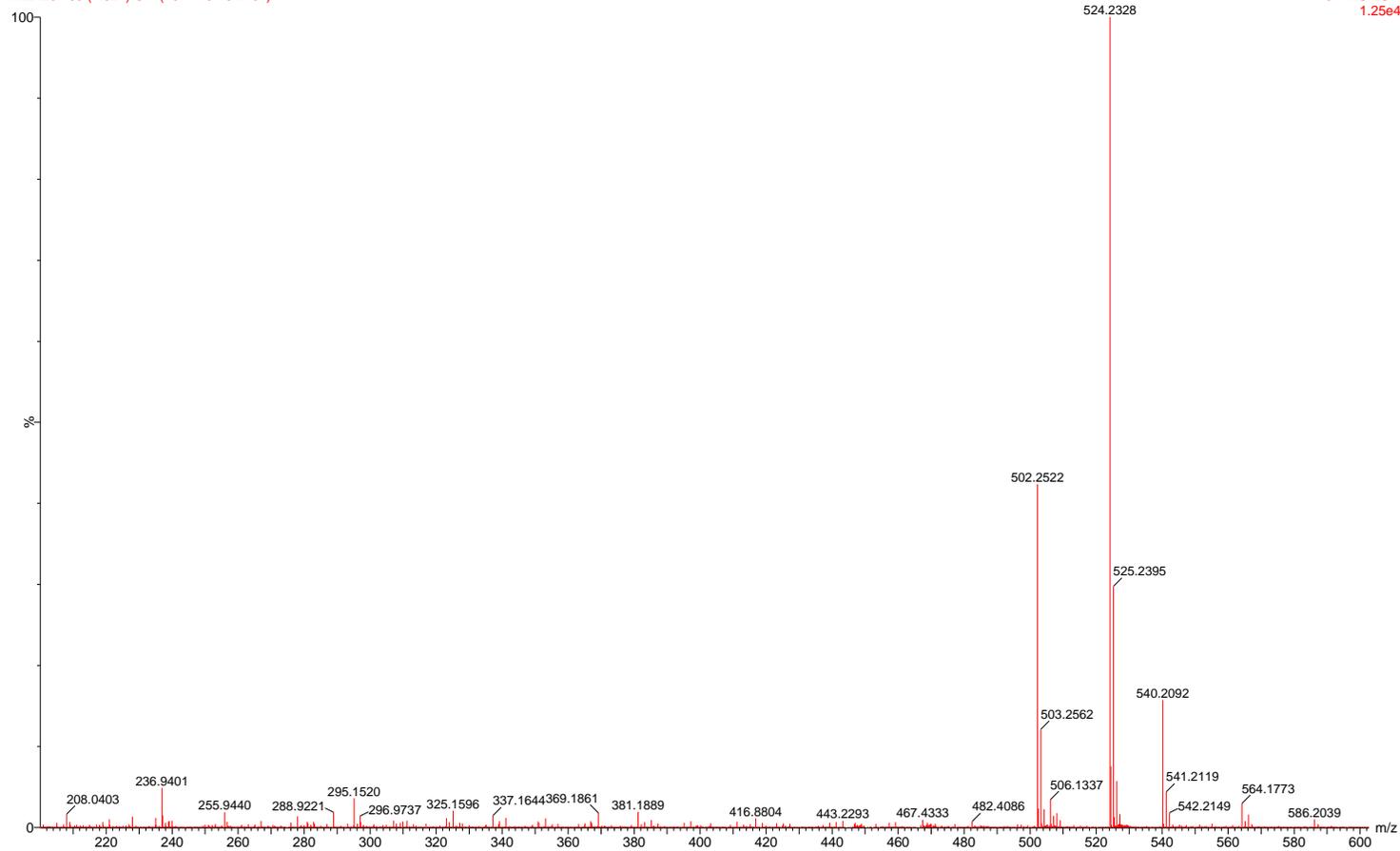


(57)

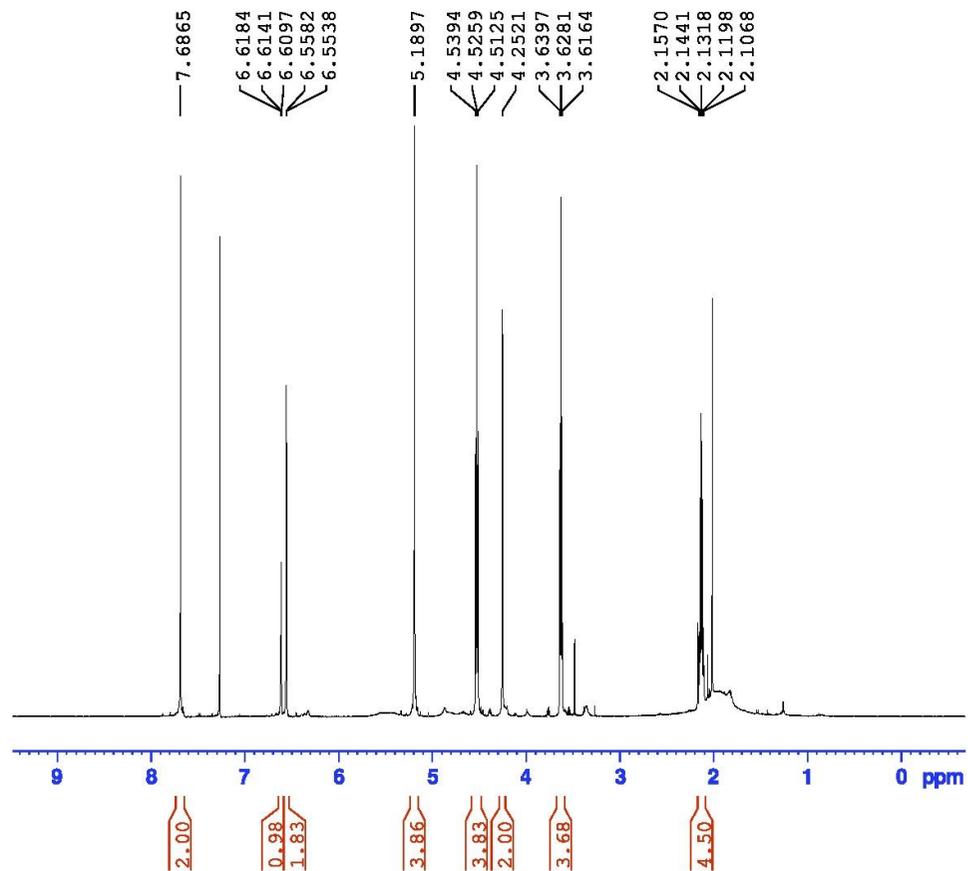
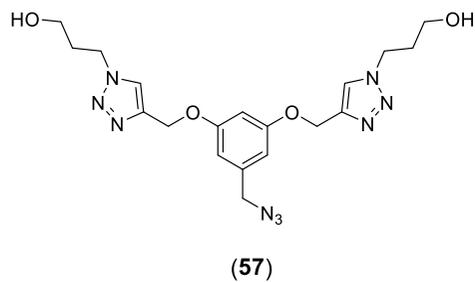
AJF_HK-18-13

ML2423 169 (1.521) Cm (167:173-161:164)

1: TOF MS ES+
1.25e4



Appendix B49: HRMS (ESI) spectra of compound (57).



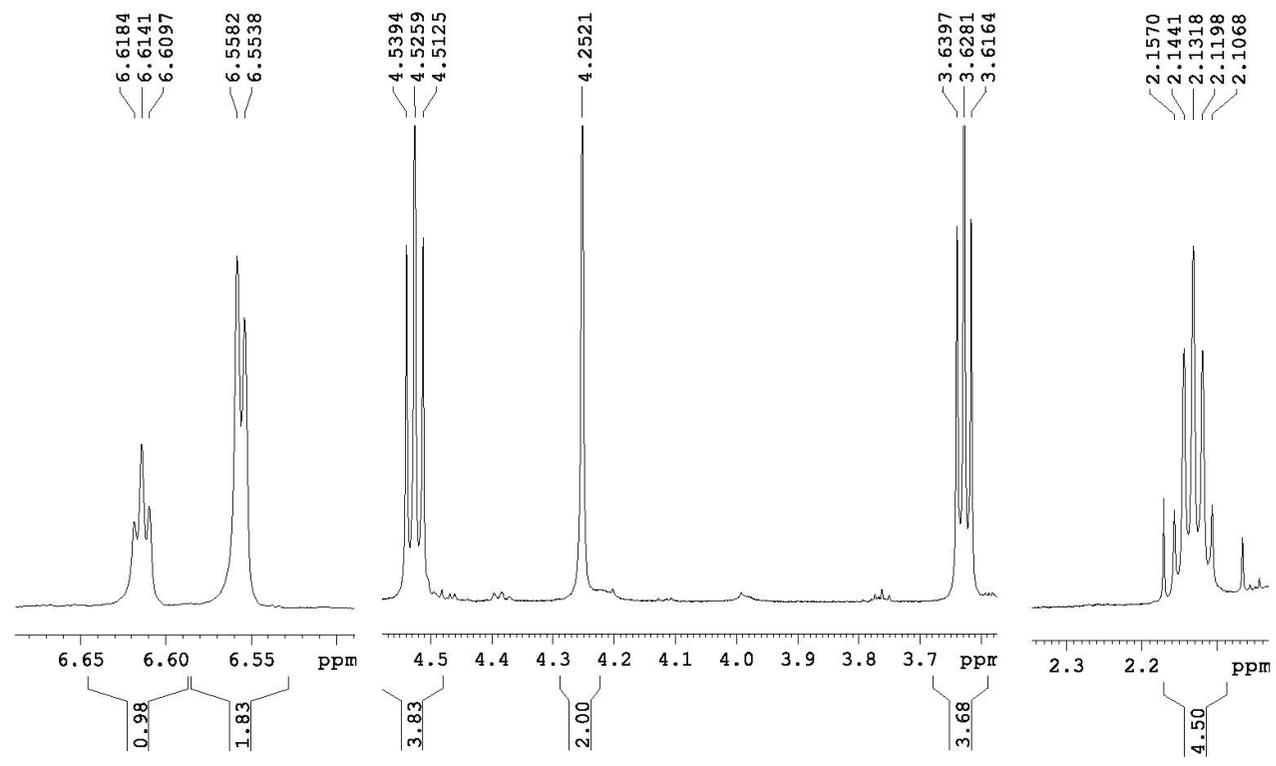
Current Data Parameters
 NAME A32612
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameter
 Date_ 20130429
 Time 16.59
 INSTRUM DRX500
 PROBHD 5 mm DUL 13C-1
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 10288.065 Hz
 FIDRES 0.156983 Hz
 AQ 3.1850495 sec
 RG 574.7
 DW 48.600 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 TDC 1

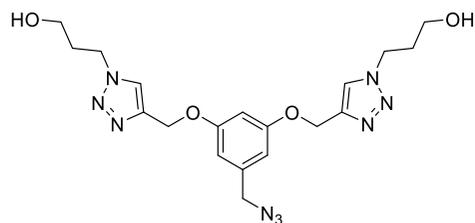
CHANNEL f1
 NUC1 1H
 P1 12.00 usec
 PL1 -2.70 dB
 SFO1 500.1330885 MHz

F2 - Processing parameters
 SI 32768
 SF 500.1300095 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

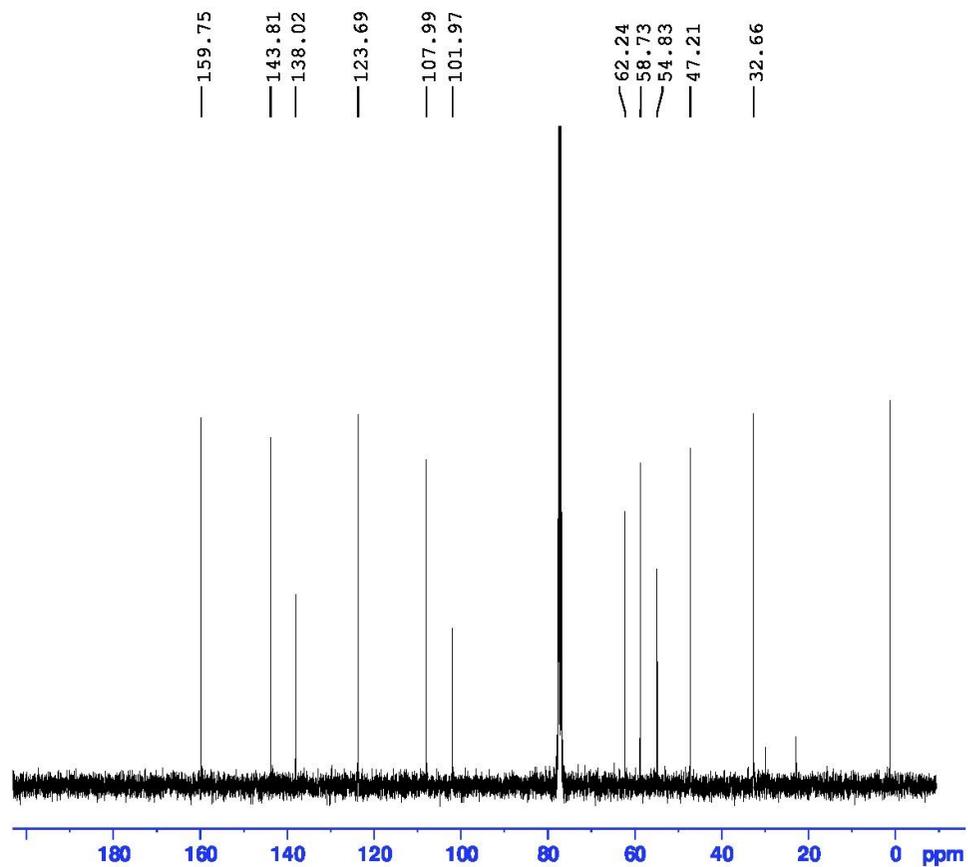
Appendix B50: ^1H NMR spectrum of compound (57).



Appendix B51: Selected areas ¹H NMR of compound (57).



(57)



```

Current Data Parameters
NAME          D189480
EXPNO         1
PROCNO        1

F2 - Acquisition Parameters
Date_         20131017
Time          19.49
INSTRUM       spect
PROBHD        5 mm QNP 1H/13
PULPROG       zgpg30
TD            16384
SOLVENT       CDCl3
NS            8192
DS            4
SWH           22058.824 Hz
FIDRES        1.346364 Hz
AQ            0.3713707 sec
RG            2050
DW            22.667 usec
DE            6.50 usec
TE            322.2 K
D1            2.0000000 sec
D11           0.0300000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            7.00 usec
PL1           -2.60 dB
PL1W          61.34123230 W
SFO1          100.6228298 MHz

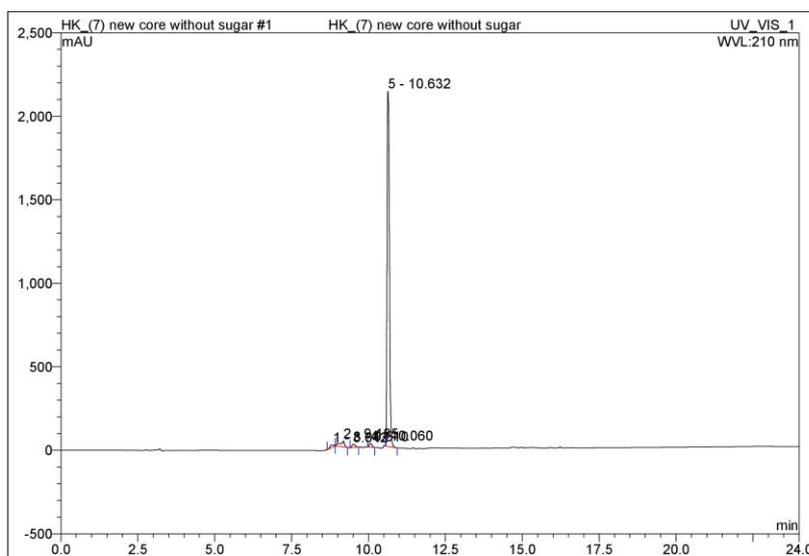
===== CHANNEL f2 =====
CPDPRG [2]    waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           -4.00 dB
PL12          14.06 dB
PL13          120.00 dB
PL2W          19.93825150 W
PL12W         0.31166428 W
PL13W         0 W
SFO2          400.1316005 MHz

F2 - Processing parameters
SI            32768
SF            100.6127512 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

Appendix B52: ^{13}C NMR spectrum of compound (57).

1 HK_(7) new core without sugar

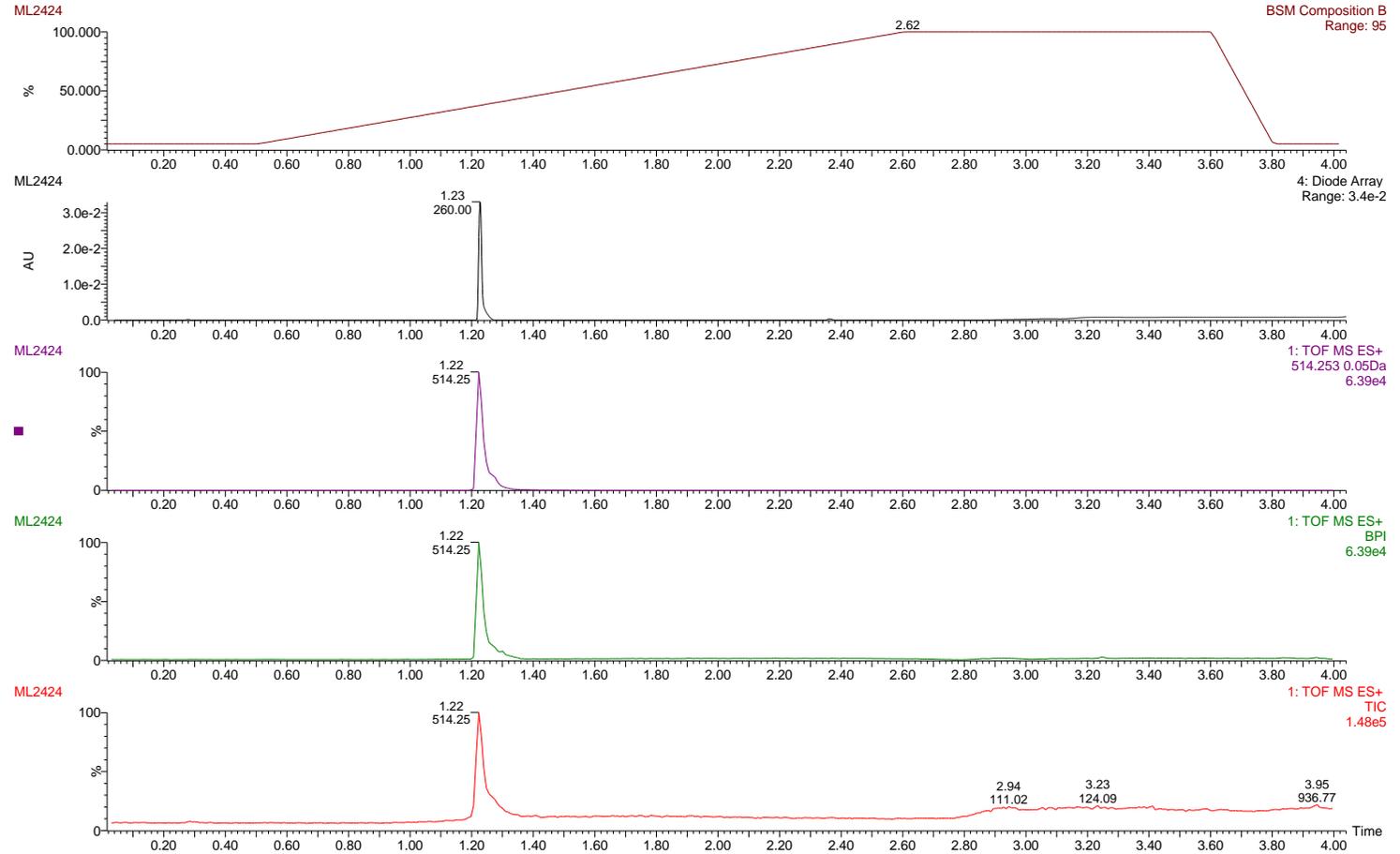
Sample Name:	HK_(7) new core without sugar	Injection Volume:	20.0
Vial Number:	RA1	Channel:	UV_VIS_1
Sample Type:	unknown	Wavelength:	210
Control Program:	poly-p4 28min +230nm	Bandwidth:	10
Quantif. Method:	dna method	Dilution Factor:	1.0000
Recording Time:	11/12/2013 15:21	Sample Weight:	1.0000
Run Time (min):	24.00	Sample Amount:	1.0000



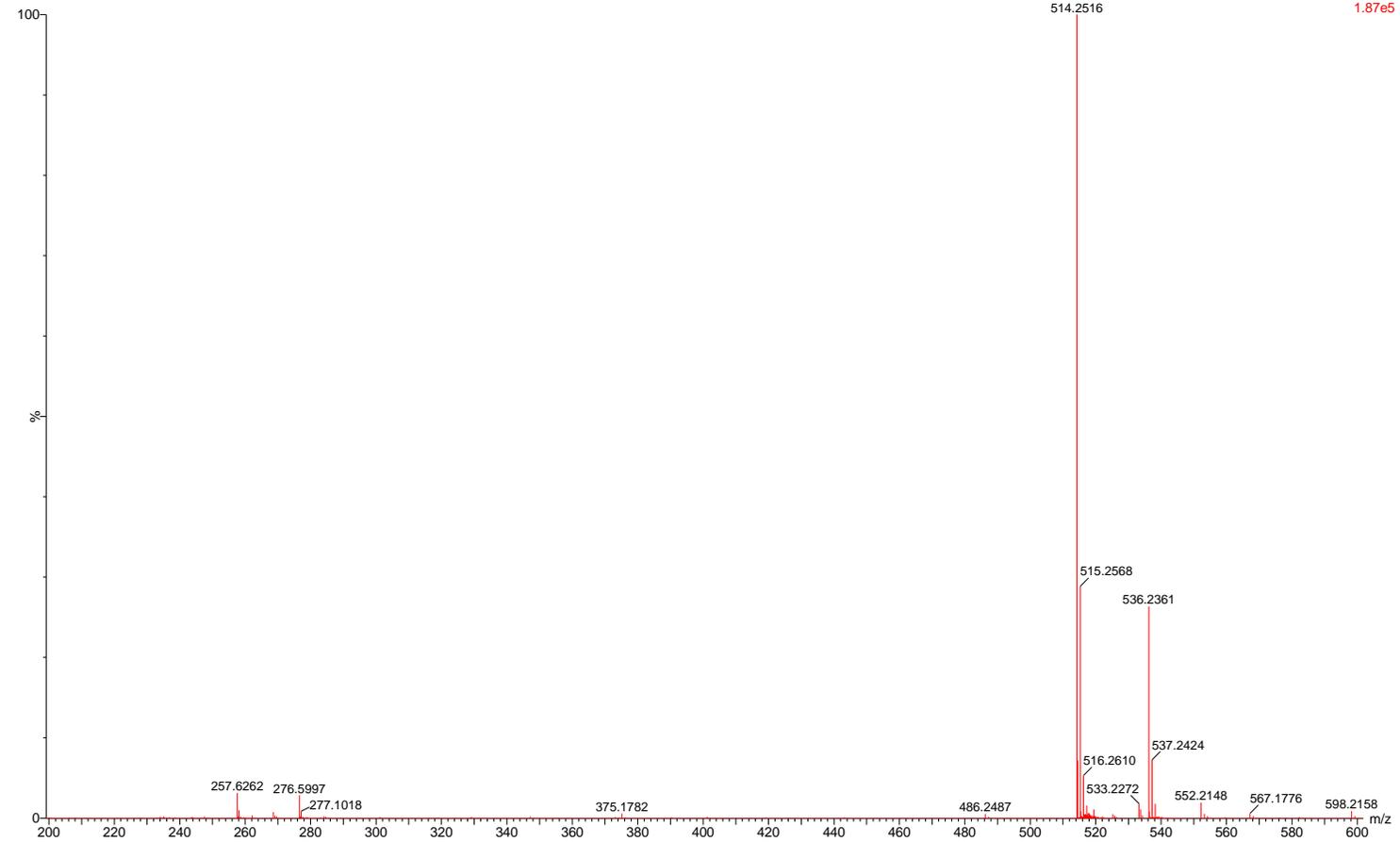
No.	Ret. Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	8.84	n.a.	12.075	2.110	1.02	n.a.	BMB
2	9.19	n.a.	35.818	6.044	2.92	n.a.	bMB
3	9.51	n.a.	19.575	2.452	1.18	n.a.	BMB
4	10.06	n.a.	23.528	2.470	1.19	n.a.	BMB
5	10.63	n.a.	2128.743	194.215	93.69	n.a.	BMB
Total:			2219.739	207.291	100.00	0.000	

Appendix B53: HPL chromatogram of compound (52).

AJF_HK-19-13



AJF_HK-19-13
ML2424 136 (1.223) Cm (135:139)



Elemental Composition Report: **AJF-HK-19-13**

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 80.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

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

Elements Used:

C: 23-23 H: 0-150 N: 0-30 O: 0-30

Minimum:

-1.5

Maximum:

10.0

5.0

80.0

Mass

Calc. Mass

mDa

PPM

DBE

i-FIT

i-FIT (Norm)

Formula

514.2516

514.2526

-1.0

-1.9

12.5

314.2

0.0

C₂₃ H₃₂ N₉ O₅

Elemental Composition Report: **AJF-HK-19-13**

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 80.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

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

Elements Used:

C: 23-23 H: 0-150 N: 0-30 O: 0-30 Na: 1-1

Minimum:

-1.5

Maximum:

10.0

5.0

80.0

Mass

Calc. Mass

mDa

PPM

DBE

i-FIT

i-FIT (Norm)

Formula

536.2361

536.2346

1.5

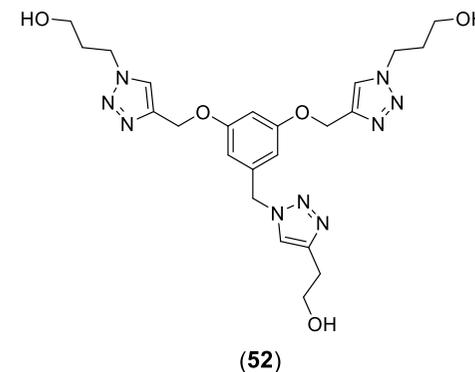
2.8

12.5

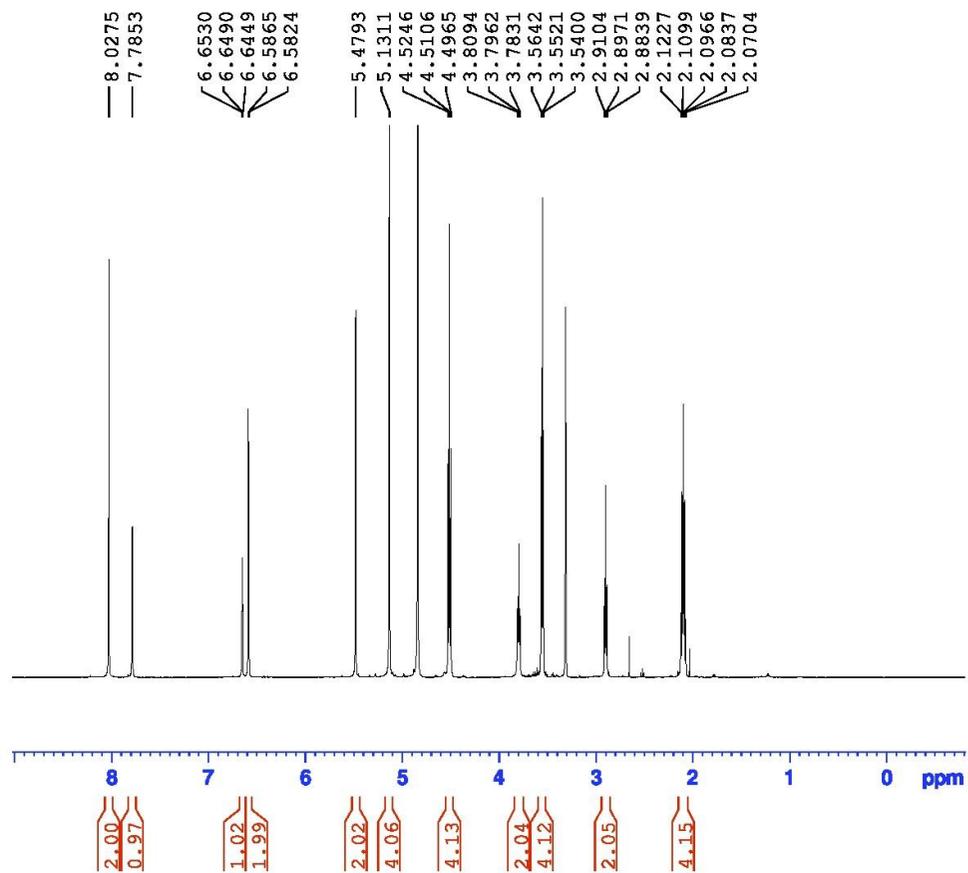
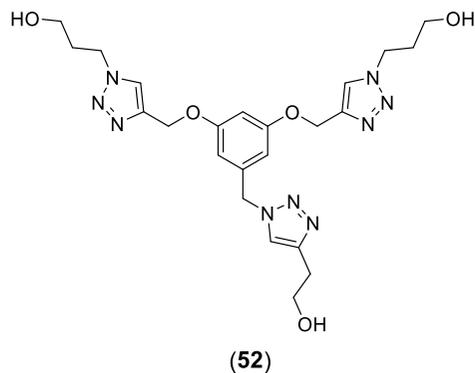
242.2

0.0

C₂₃ H₃₁ N₉ O₅ Na



Appendix B54: HRMS (ESI) spectra of compound (52).



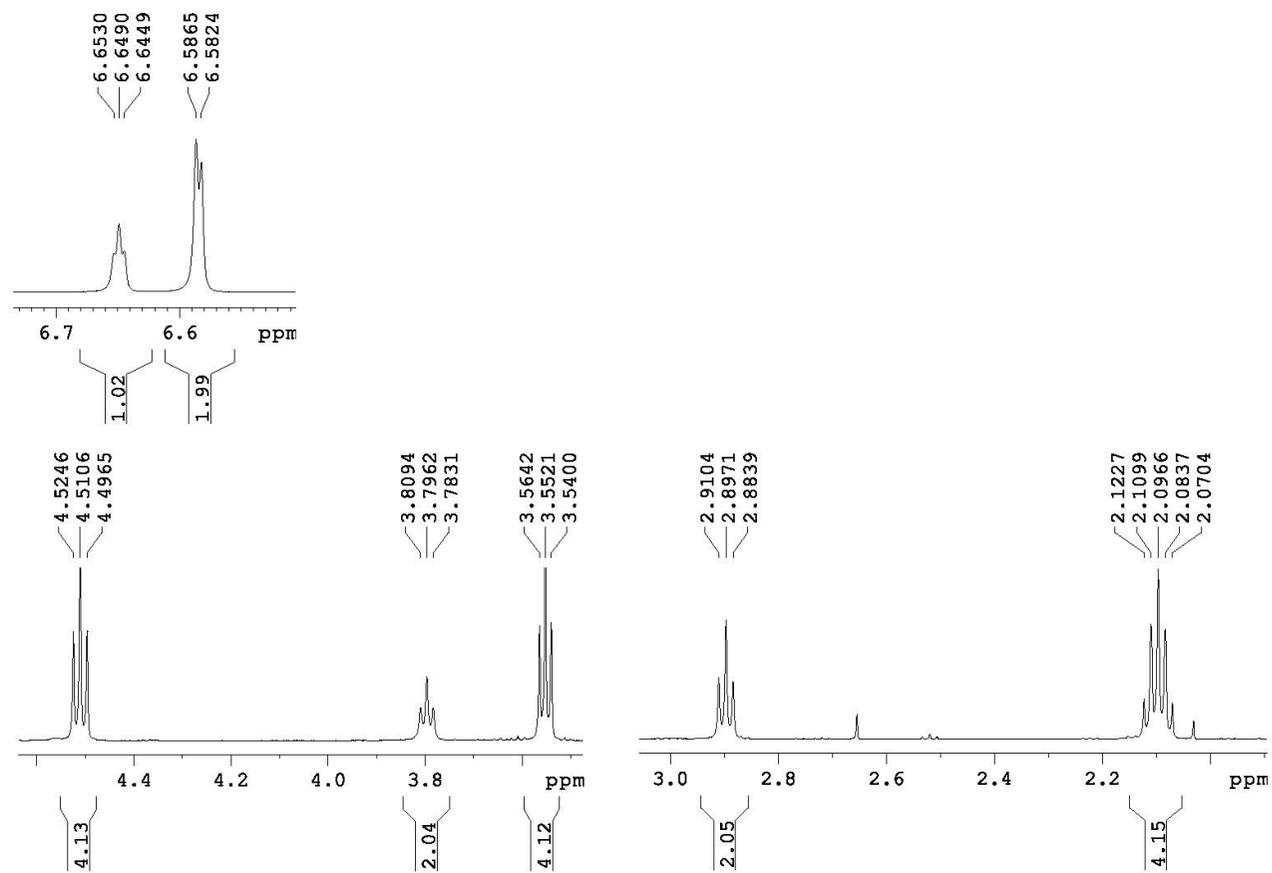
Current Data Parameters
 NAME A32459
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameter
 Date_ 20130423
 Time 12.54
 INSTRUM DRX500
 PROBHD 5 mm DUL 13C-1
 PULPROG zg30
 TD 65536
 SOLVENT MeOD
 NS 128
 DS 2
 SWH 10288.065 Hz
 FIDRES 0.156983 Hz
 AQ 3.1850495 sec
 RG 456.1
 DW 48.600 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

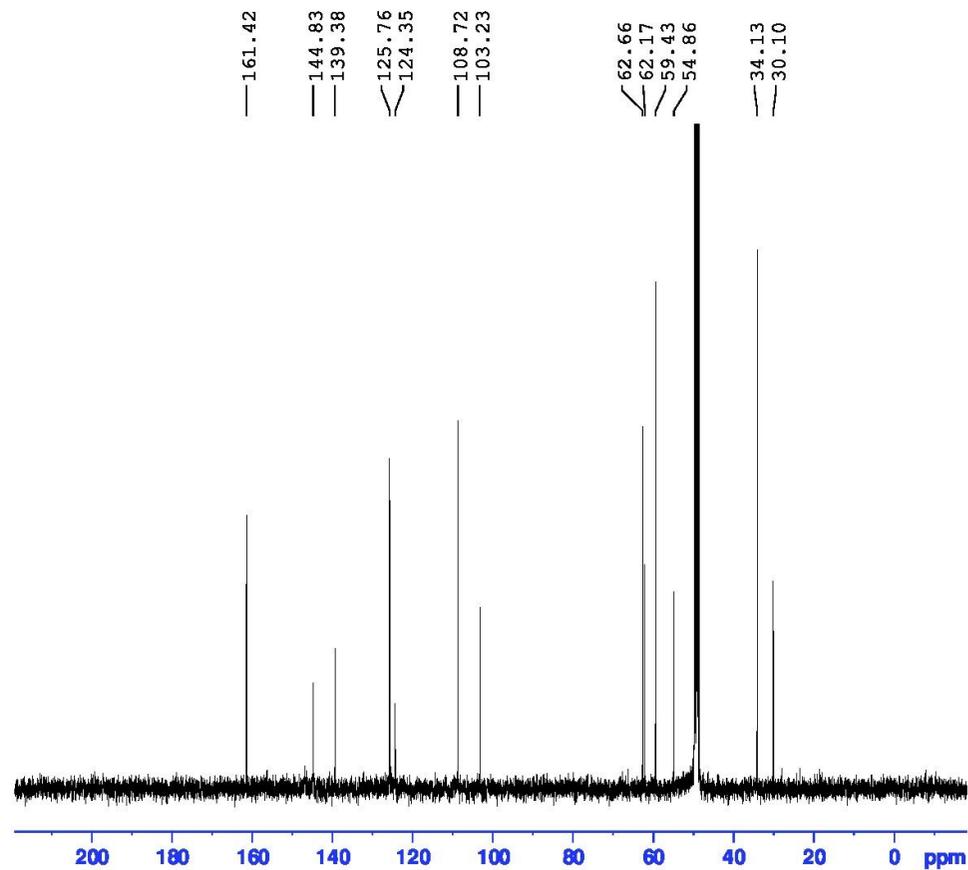
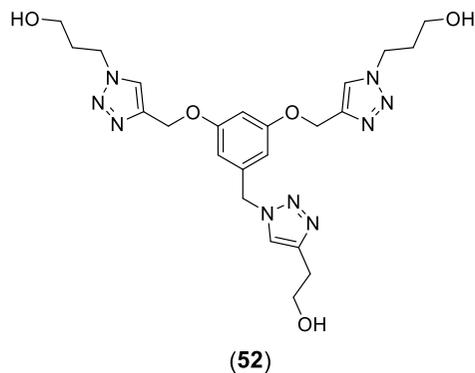
CHANNEL f1
 NUC1 1H
 P1 12.00 usec
 PL1 -2.70 dB
 SFO1 500.1330885 MHz

F2 - Processing parameters
 SI 32768
 SF 500.1300108 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Appendix B55: ¹H NMR spectrum of compound (52).



Appendix B56: Selected areas ^1H NMR of compound (52).



Current Data Parameters
NAME A32459
EXPNO 2
PROCNO 1

F2 - Acquisition Parameter
Date_ 20130423
Time 14.05
INSTRUM DRX500
PROBED 5 mm DUL 13C-1
PULPROG zgpg30
TD 16384
SOLVENT MeOD
NS 4096
DS 4
SWH 30120.482 Hz
FIDRES 1.838408 Hz
AQ 0.2719744 sec
RG 6502
DW 16.600 usec
DE 6.00 usec
TE 300.0 K
D1 0.69999999 sec
d11 0.03000000 sec
DELTA 0.59999996 sec
TD0 1

CHANNEL f1
NUC1 13C
P1 8.00 usec
PL1 -2.30 dB
SFO1 125.7703643 MHz

CHANNEL f2
CPDPRG[2] waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.30 dB
PL12 12.84 dB
PL13 120.00 dB
SFO2 500.1320005 MHz

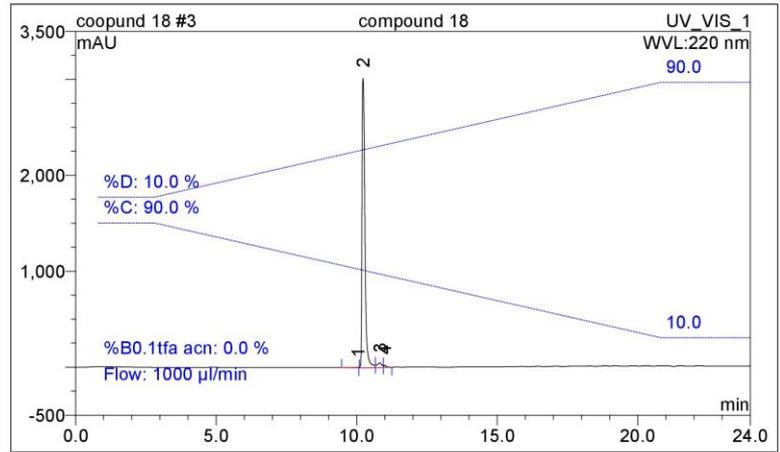
F2 - Processing parameters
SI 32768
SF 125.7575917 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

Appendix B57: ¹³C NMR spectrum of compound (52).

Sample Analysis Report

Sample Name:	compound 18	Sample No.:	3
Sequence Name:	coopund 18	Injection vol.:	20.0
Program Method:	poly-p4 28min +230nm	Dilution Factor:	1.0000
Quantitation Method:	dna method	Sample Wt.:	1.0000
Date Time Collected:	21/2/2015 5:07 PM	Sample Amt.:	1.0000
System Operator:	Administrator		

Peak No.	Component Name	Retention Time	Area mAU*min	Relative Area %	Height mAU	Relative Height %
----------	----------------	----------------	--------------	-----------------	------------	-------------------

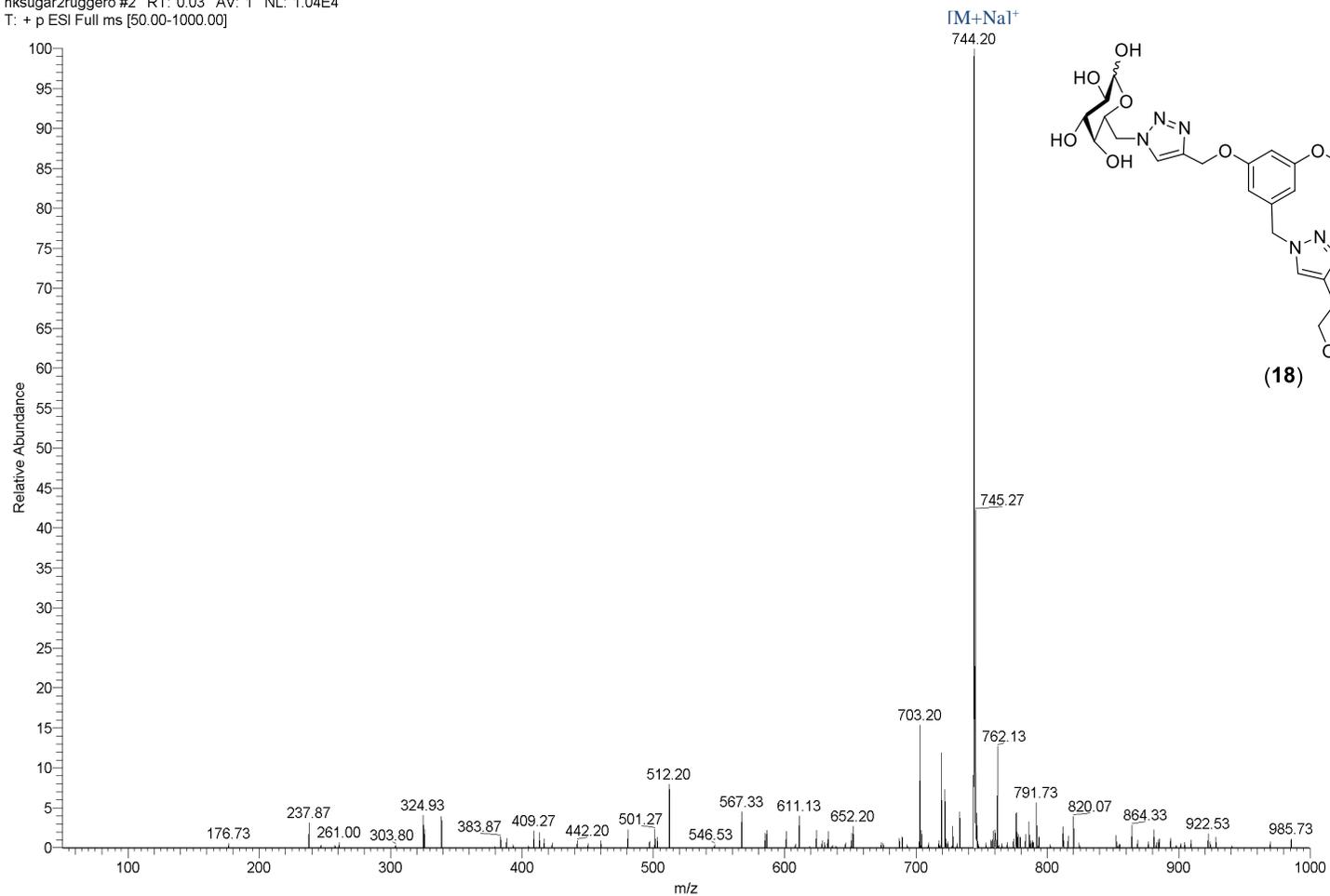


Appendix B58: HPL chromatogram of compound (18).

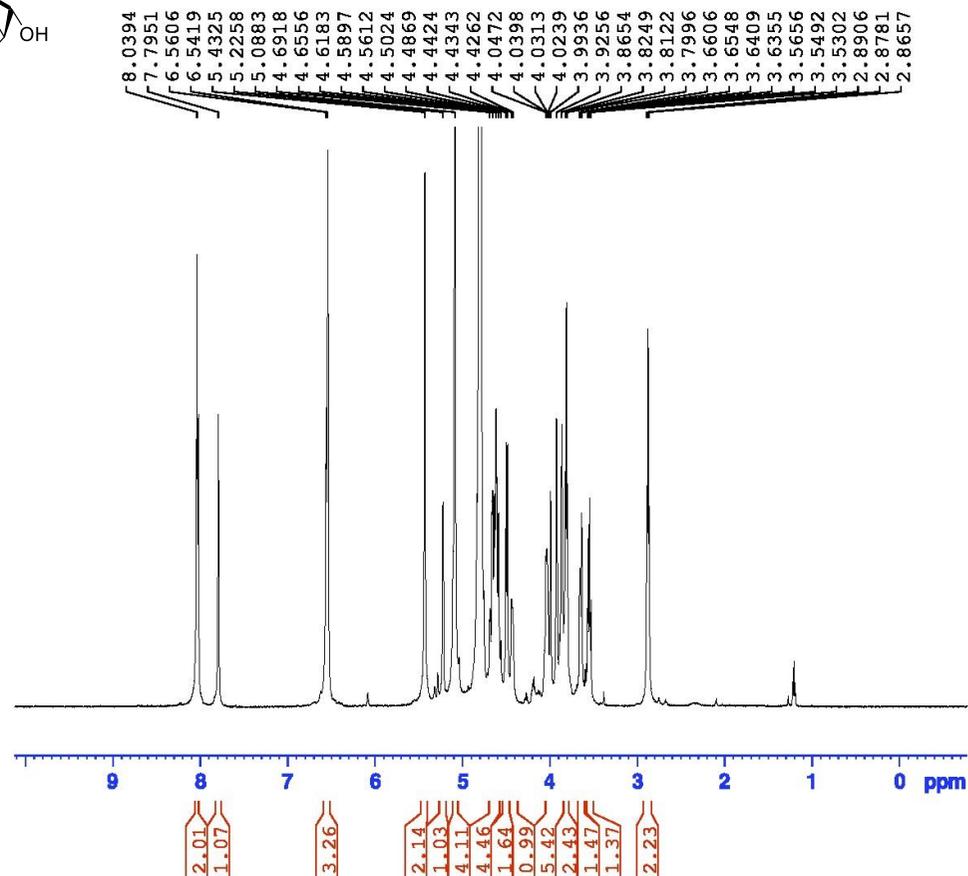
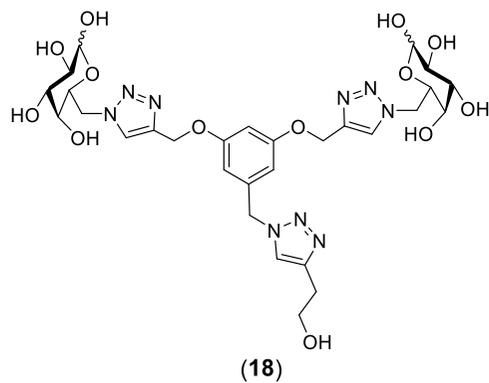
G:\data\...\Burley\hksugar2ruggero
MeOH

25/06/2014 17:31:54

hksugar2ruggero #2 RT: 0.03 AV: 1 NL: 1.04E4
T: + p ESI Full ms [50.00-1000.00]



Appendix B59: LRMS (ESI) spectra of compound (18).



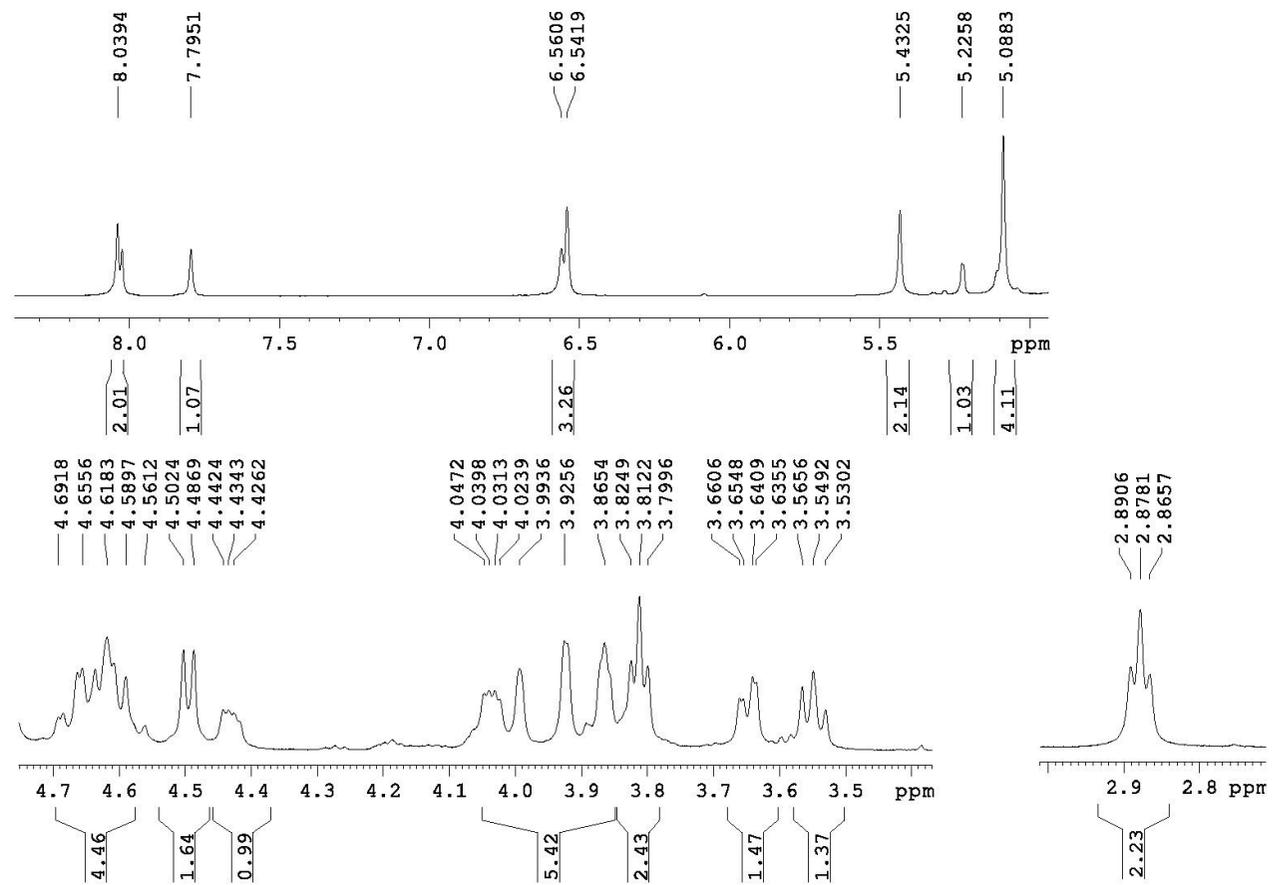
Current Data Parameters
 NAME A38769
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameter
 Date_ 20140401
 Time 16.15
 INSTRUM DRX500
 PROBED 5 mm DUL 13C-1
 PULPROG zg30
 TD 65536
 SOLVENT D2O
 NS 128
 DS 2
 SWH 10288.065 Hz
 FIDRES 0.156983 Hz
 AQ 3.1850495 sec
 RG 322.5
 DW 48.600 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

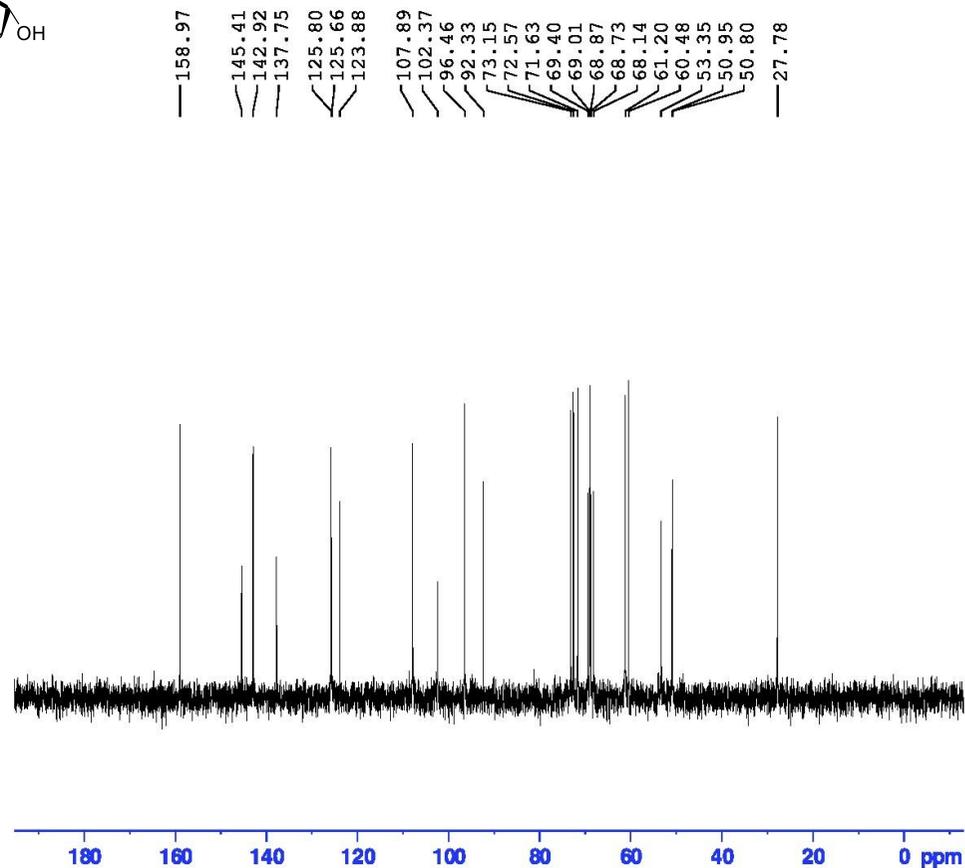
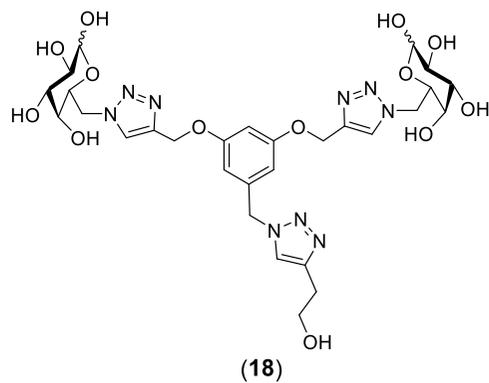
CHANNEL f1
 NUC1 1H
 P1 18.50 usec
 PL1 0.30 dB
 SFO1 500.1330885 MHz

F2 - Processing parameters
 SI 32768
 SF 500.1299529 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Appendix B60: ¹H NMR spectrum of compound (18).



Appendix B61: Selected areas ^1H NMR of compound (18).



Current Data Parameters
NAME A38769
EXPNO 1
PROCNO 1

F2 - Acquisition Parameter
Date_ 20140401
Time 16.04
INSTRUM DRX500
PROBED 5 mm DUL 13C-1
PULPROG zgpg30
TD 16384
SOLVENT D2O
NS 4096
DS 4
SWH 30120.482 Hz
FIDRES 1.838408 Hz
AQ 0.2719744 sec
RG 11585.2
DW 16.600 usec
DE 6.00 usec
TE 300.0 K
D1 0.69999999 sec
d11 0.03000000 sec
DELTA 0.59999996 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 20.00 usec
PL1 4.20 dB
SFO1 125.7703643 MHz

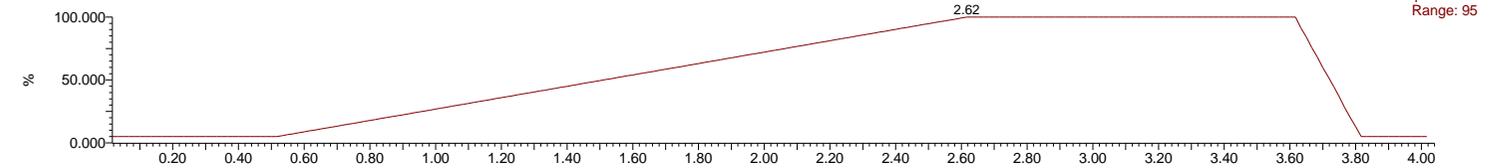
==== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.30 dB
PL12 12.84 dB
PL13 120.00 dB
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

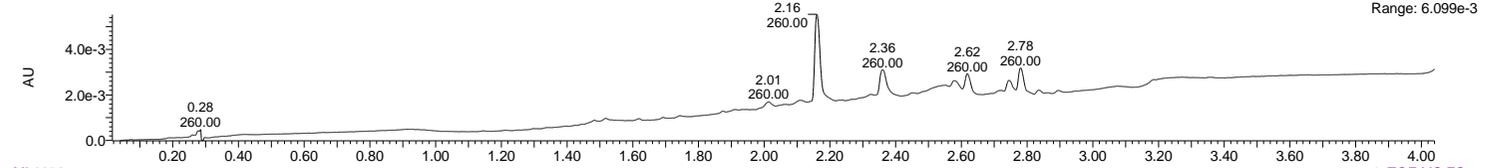
Appendix B62: ¹³C NMR spectrum of compound (18).

AJF_HK_24_13P1

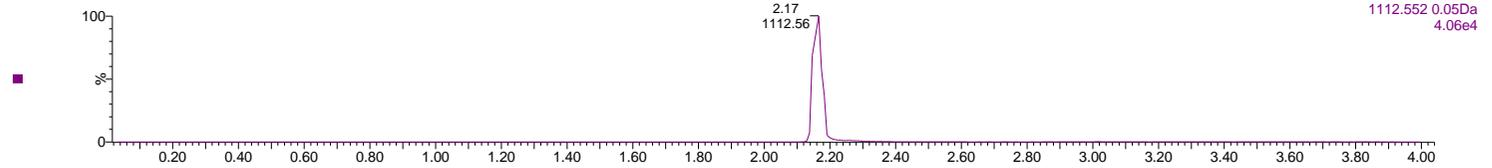
ML2332



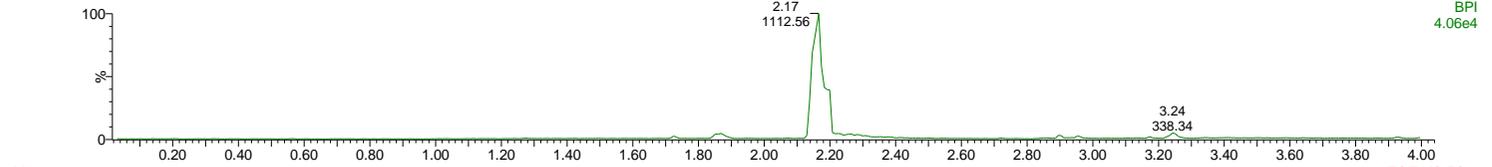
ML2332



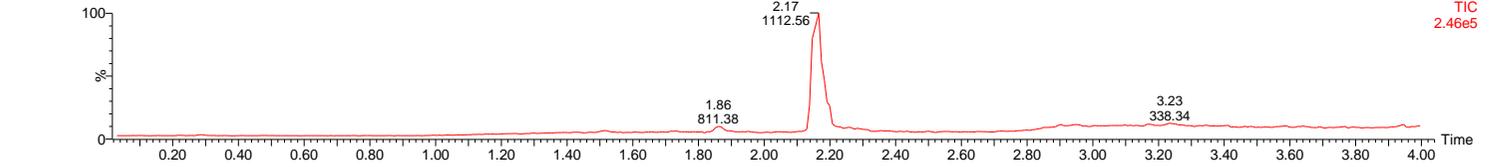
ML2332



ML2332

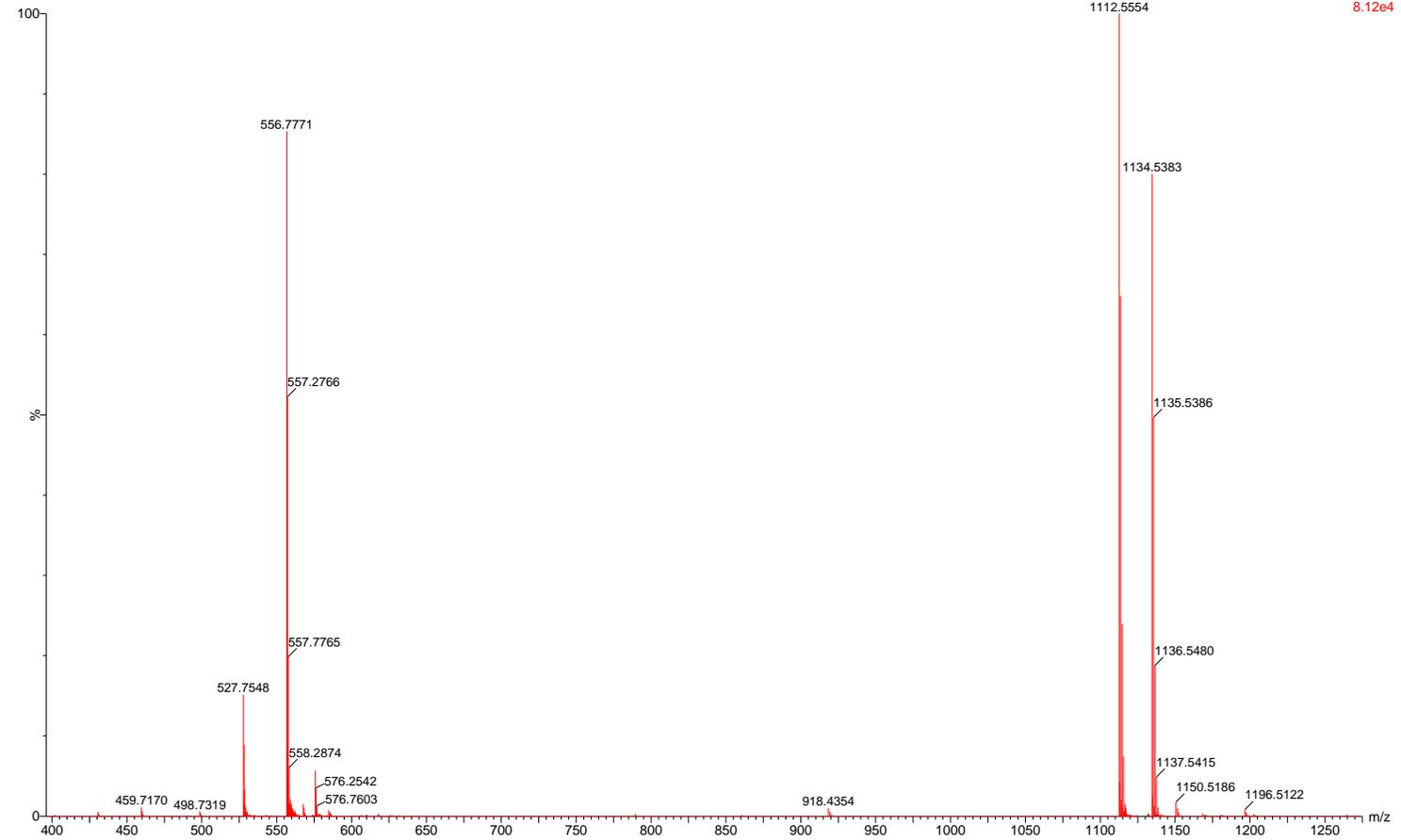


ML2332



AJF_HK_24_13P1

ML2332 241 (2.166) Cm (241:244)



Elemental Composition Report: **AJF-HK-24-13P1**

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

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

Elements Used:

C: 53-53 H: 0-150 N: 0-30 O: 0-30

Minimum:

-1.5

Maximum:

10.0

5.0

50.0

Mass

Calc. Mass

mDa

PPM

DBE

i-FIT

i-FIT (Norm)

Formula

1112.5554

1112.5516

3.8

3.4

19.5

93.9

0.0

C₅₃ H₇₈ N₉ O₁₇

Elemental Composition Report: **AJF-HK-24-13P1**

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

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

Elements Used:

C: 53-53 H: 0-150 N: 0-30 O: 0-30 Na: 1-1

Minimum:

-1.5

Maximum:

10.0

5.0

50.0

Mass

Calc. Mass

mDa

PPM

DBE

i-FIT

i-FIT (Norm)

Formula

1134.5383

1134.5335

4.8

4.2

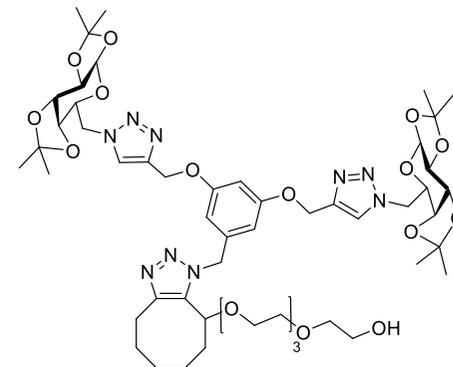
19.5

89.8

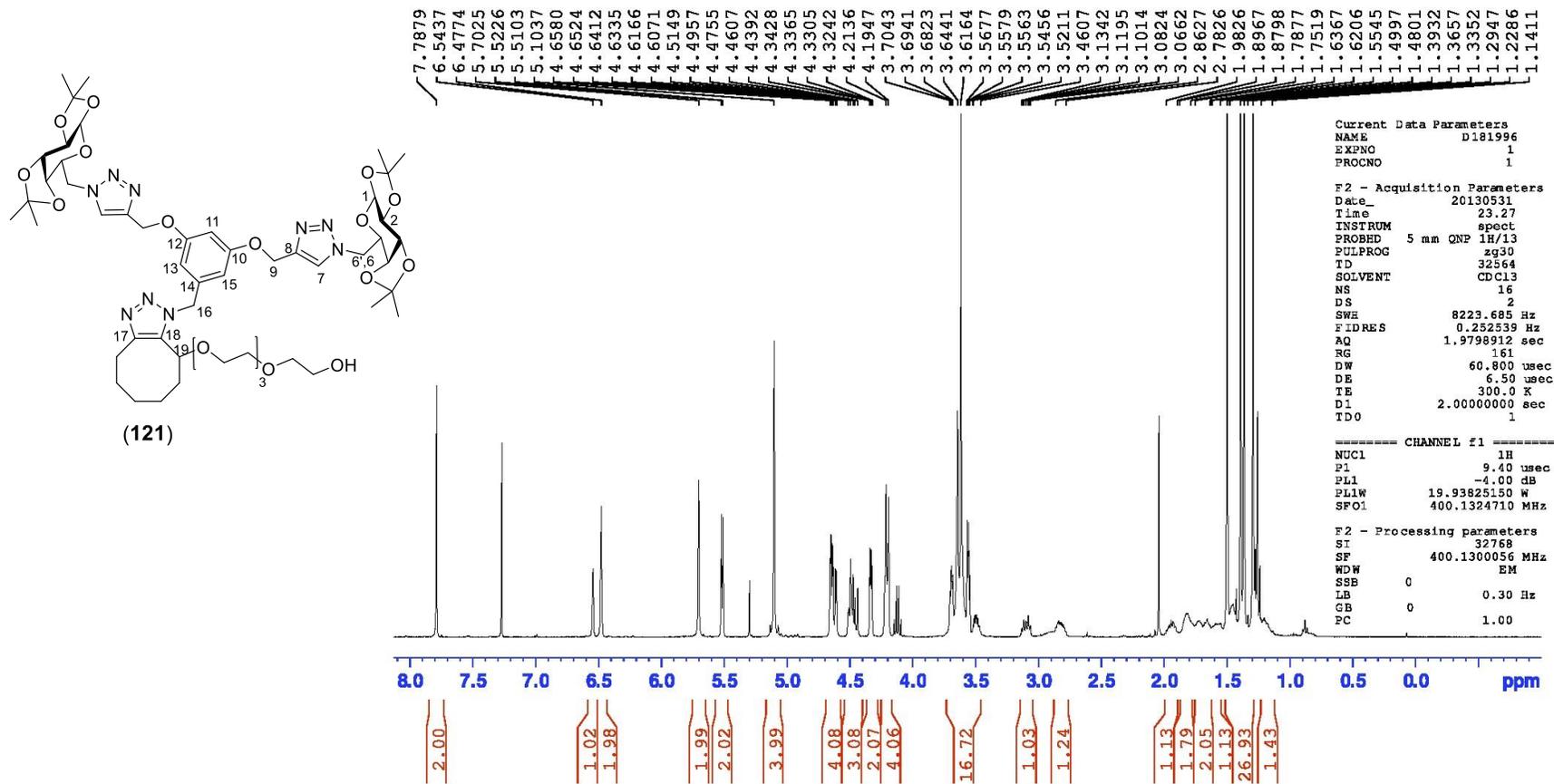
0.0

C₅₃ H₇₇ N₉ O₁₇ Na

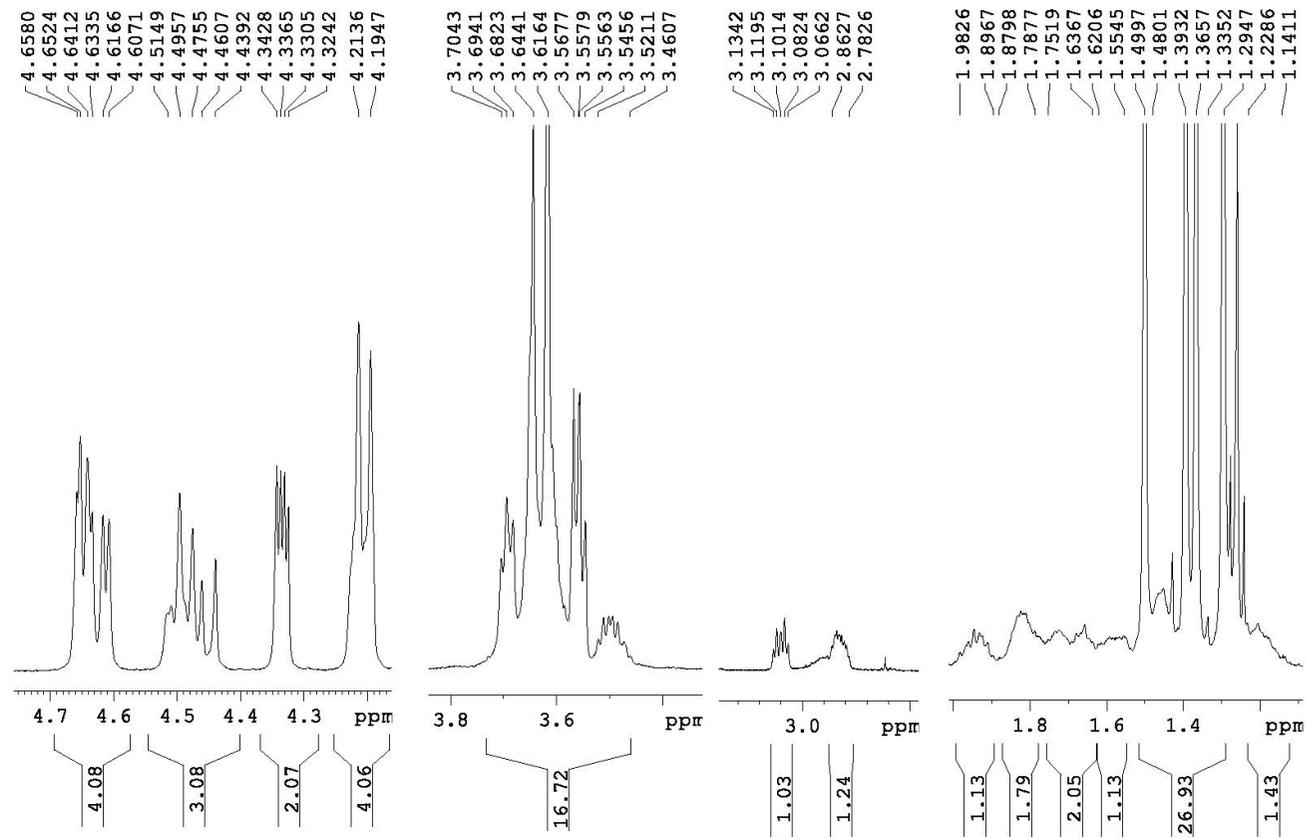
Appendix B63: HRMS (ESI) spectra of compound (121).



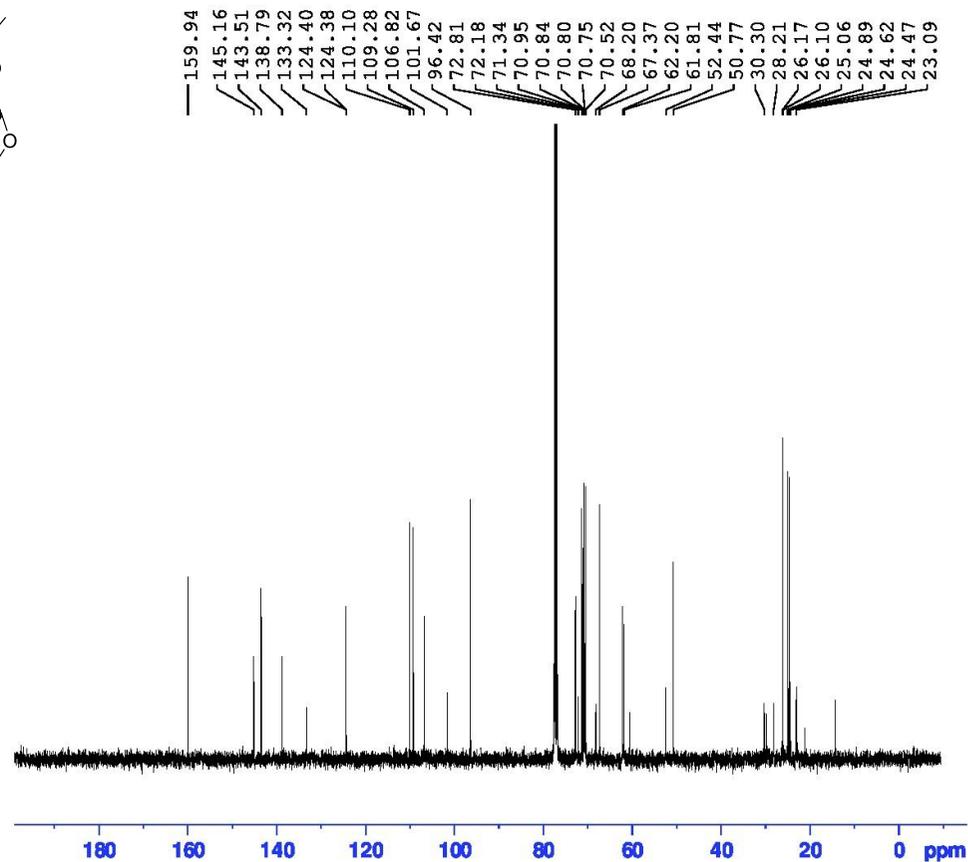
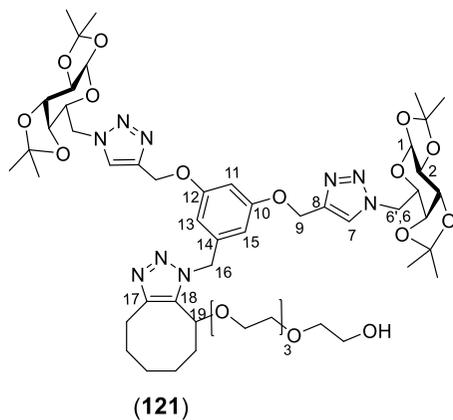
(121)



Appendix B64: ¹H NMR spectrum of compound (121).



Appendix B65: Selected areas ¹H NMR of compound (121).



Current Data Parameters
NAME D183144
EXPNO 1
PROCNO 1

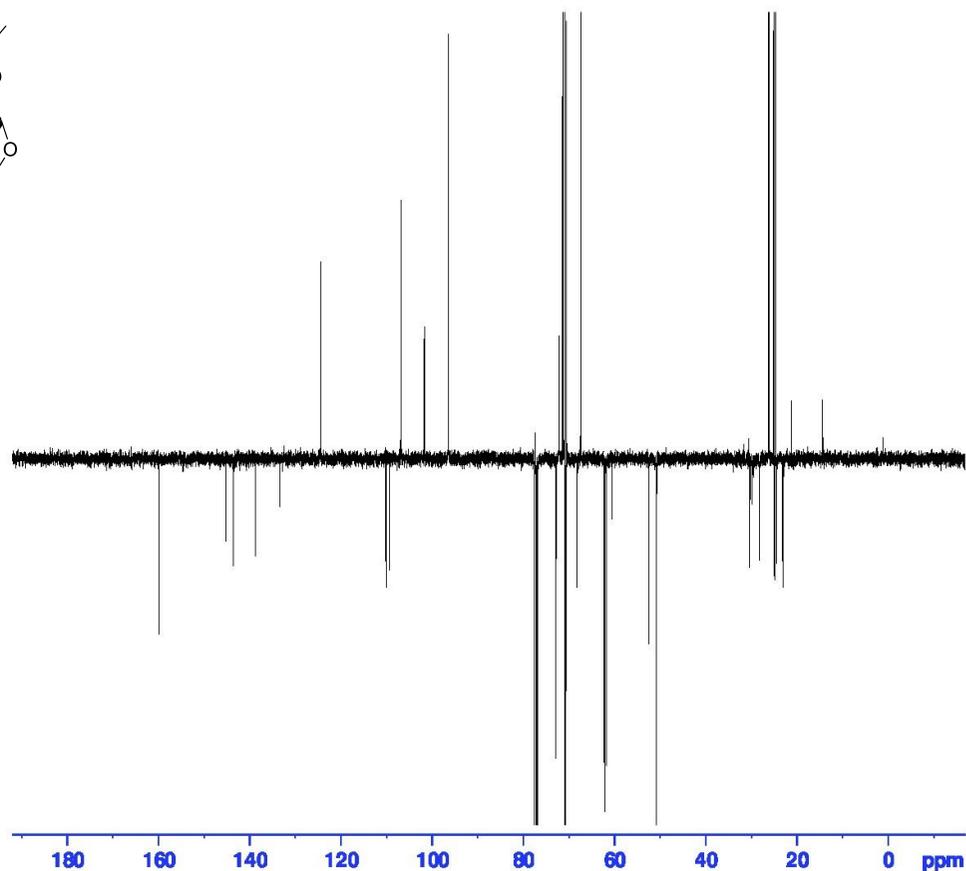
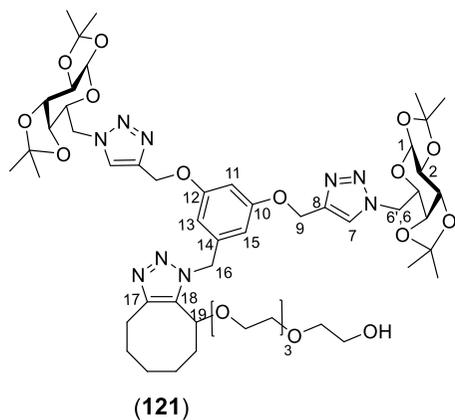
F2 - Acquisition Parameters
Date_ 20130618
Time 22.30
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 16384
SOLVENT CDCl3
NS 4096
DS 4
SWH 22058.824 Hz
FIDRES 1.346364 Hz
AQ 0.3713707 sec
RG 2050
DW 22.667 usec
DE 6.50 usec
TE 300.0 K
D1 0.69999999 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 7.00 usec
PL1 -2.60 dB
PL1W 61.34123230 W
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG [2] waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 14.06 dB
PL13 120.00 dB
PL2W 19.93825150 W
PL12W 0.31166428 W
PL13W 0 W
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127492 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Appendix B66: ^{13}C NMR spectrum of compound (121).



```

Current Data Parameters
NAME      D183874
EXPNO     1
PROCNO    1

F2 - Acquisition Parameters
Date_     20130703
Time      6.07
INSTRUM   spect
PROBHD    5 mm QNP 1H/13
PULPROG   zgpg30
ID        32768
SOLVENT   CDCl3
NS         2048
DS         4
SWH        24038.461 Hz
FIDRES     0.733896 Hz
AQ         0.6815744 sec
RG         256
DW         20.800 usec
DE         6.50 usec
TE         300.0 K
CST2      145.000000
CPC12     1.000000
D1        2.0000000 sec
D2        0.0034428 sec
D12       0.0002000 sec
D16       0.0002000 sec
D28       0.5000000 sec
ID0       1

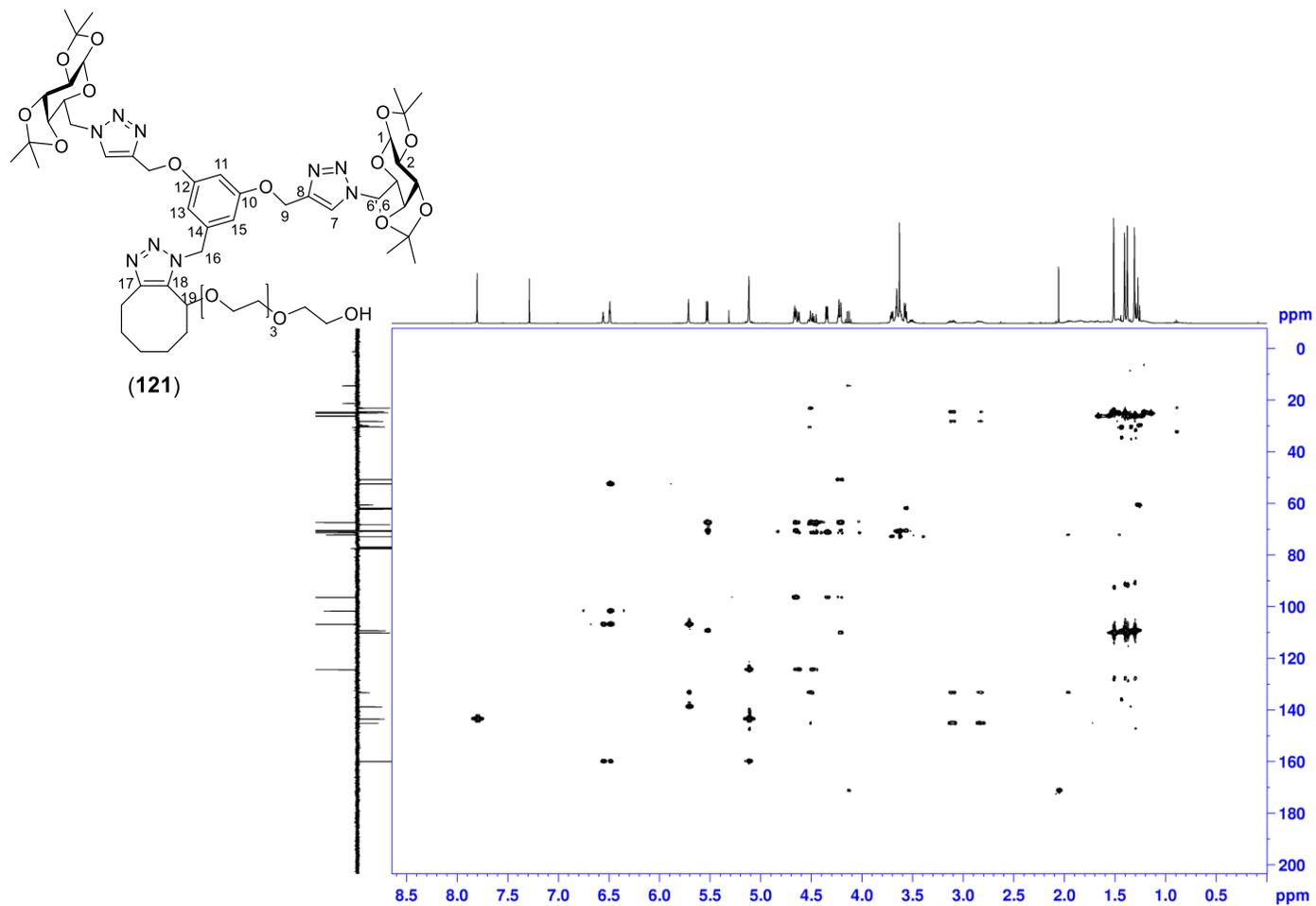
----- CHANNEL f1 -----
NUC1      13C
P1        7.00 usec
PL1       2000.00 usec
PL0       120.00 dB
PL2       -2.60 dB
PL12      0 W
PL1W      61.34123230 W
SFO1      100.6282298 MHz
SF2       8.65 dB
SFNAM[2]  Ccp60comp.4
SFOALZ    0.300
SFOFFS2   0 Hz

----- CHANNEL f2 -----
CPDPRG [2] waltz16
NUC2      1H
P2        15.00 usec
P3        10.00 usec
P4        20.00 usec
PCPD2     60.00 usec
PL2       -4.00 dB
PL12      14.05 dB
PL13      120.00 dB
PL1W      13.9385150 W
PL12W     0.31166428 W
PL13W     0 W
SFO2      400.1316005 MHz

----- GRADIENT CHANNEL -----
GFNAM[1]  SINE.100
GFNAM[2]  SINE.100
GFNAM[3]  SINE.100
GS21     31.00 %
GS22     31.00 %
GS23     31.00 %
PL6      1000.00 usec

F2 - Processing parameters
SI        32768
SF        100.6127492 MHz
WDW       EM
SSB       0
GB        1.00 Hz
PC        1.40
  
```

Appendix B67: DEPT C- NMR spectrum of compound (121).



```

Current Data Parameters
NAME      D182559
EXPNO    2
PROCNO   1

F2 - Acquisition Parameters
Date_    20190111
Time     22.04
INSTRUM  spect
PROBHD   5 mm QNP 1H/13
PULPROG  zgpg30
SOLVENT  CDCl3
NS       16
DS       2
SWH      8012.820 Hz
FIDRES   0.2555504 sec
AQ       20.00
RG       652
AQ       52.400 usec
DE       6.50 usec
TE       300.2
CROSSDC  180.0000000
CROSSDC  170.0000000
CROSSDC  10.0000000
CROSSDC  0.0000000
CROSSDC  1.5000000 usec
DI       0.0000000 usec
DE       0.0500000 usec
D15      0.0000000 usec
CROSSDC  0.0000000 usec
CROSSDC  0.0000000 usec

----- CHANNEL f1 -----
NUC1     13C
P1       9.40 usec
P2       18.80 usec
P3       -4.00 dB
P4       19.9382510 Hz
P5       400.1324000 MHz

----- CHANNEL f2 -----
P3       7.00 usec
P4       20.00 dB
P5       -3.00 dB
P6       67.25992312 MHz
P7       100.628193 MHz
P8       8.26 dB
P9       Cnp60comp4
P10      0 Hz
P11      0 Hz

----- CHANNELS -----
CPDPRG1  SMO10_100
CPDPRG2  SMO10_100
CPDPRG3  SMO10_100
CPDPRG4  SMO10_100
CPDPRG5  SMO10_100
CPDPRG6  SMO10_100
CPDPRG7  SMO10_100
CPDPRG8  SMO10_100
CPDPRG9  SMO10_100
CPDPRG10 SMO10_100
CPDPRG11 SMO10_100
CPDPRG12 SMO10_100
CPDPRG13 SMO10_100
CPDPRG14 SMO10_100
CPDPRG15 SMO10_100
CPDPRG16 SMO10_100

F1 - Acquisition parameters
ID       256
PROCNO  10.6278 MHz
PULPROG  zgpg30
SOLVENT  H2O
FIDRES   0.2555504 sec
AQ       20.00
RG       652
AQ       52.400 usec
DE       6.50 usec
TE       300.2

F2 - Processing parameters
SI       32768
SF       400.1300000 MHz
WDW      EM
SSB      0 Hz
LB       0 Hz
GB       0
PC       1.40

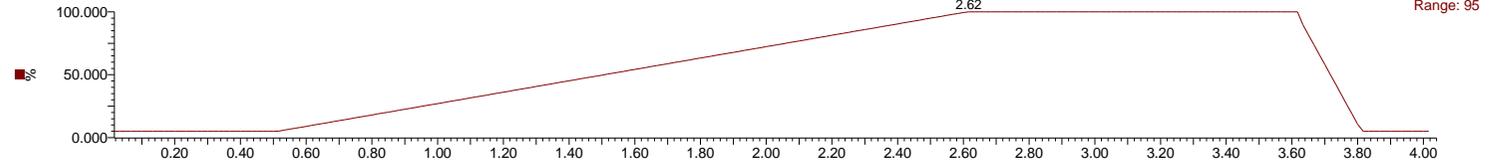
F1 - Processing parameters
SI       131072
SF       600.1300000 MHz
WDW      EM
SSB      0 Hz
LB       0
GB       0
PC       1.40

```

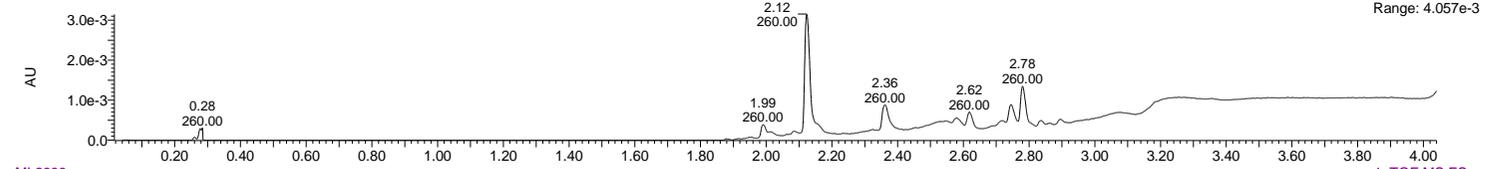
Appendix B69: HMBC NMR spectrum of compound (121).

AJF_HK_24_13P2

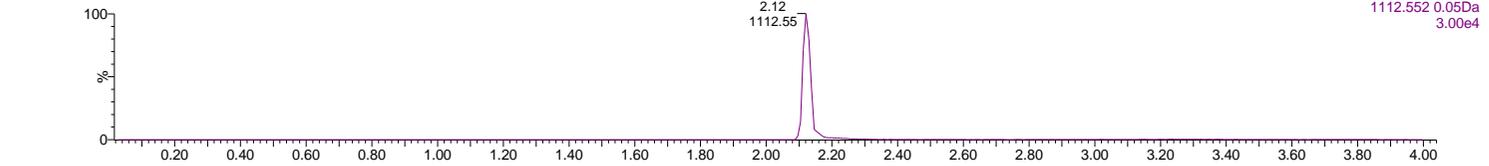
ML2333



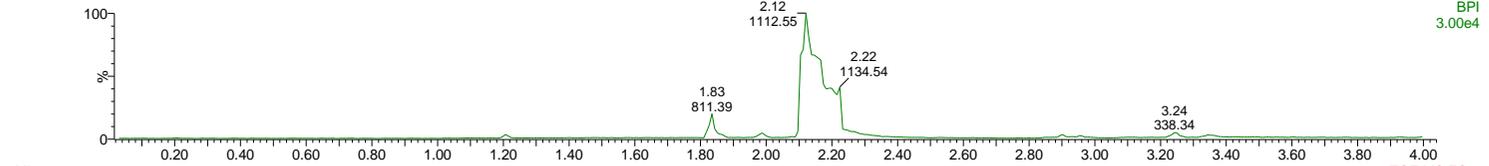
ML2333



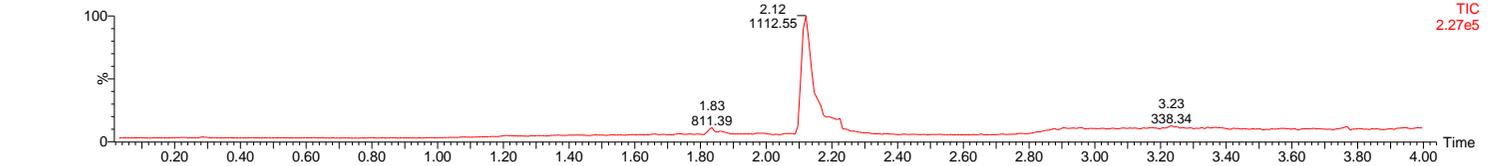
ML2333



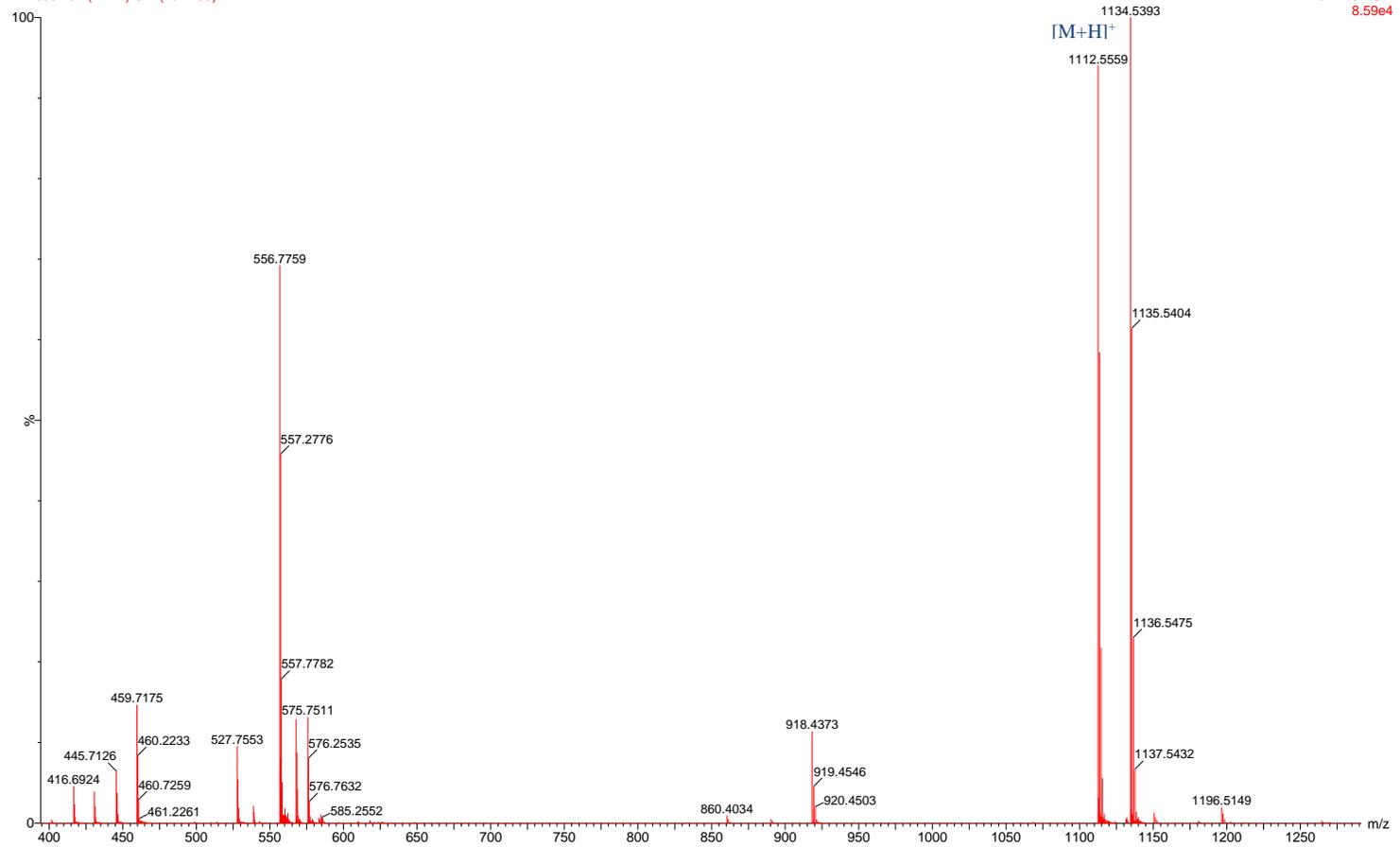
ML2333



ML2333



AJF_HK_24_13P2
ML2333 237 (2.121) Cm (234:238)



Elemental Composition Report: **AJF-HK-24-13P2**

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

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

Elements Used:

C: 53-53 H: 0-150 N: 0-30 O: 0-30

Minimum:

-1.5

Maximum:

10.0

5.0

50.0

Mass

Calc. Mass

mDa

PPM

DBE

i-FIT

i-FIT (Norm)

Formula

1112.5559

1112.5516

4.3

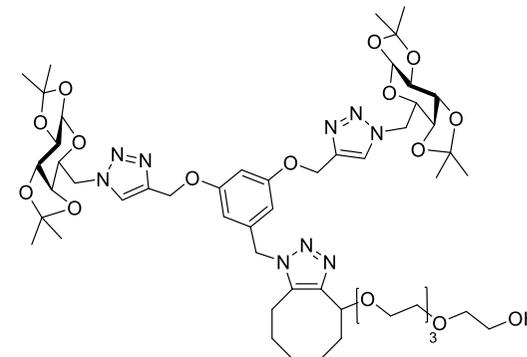
3.9

19.5

114.4

0.0

C53 H78 N9 O17



(122)

Elemental Composition Report: **AJF-HK-24-13P2**

Single Mass Analysis

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

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

953 formula(e) evaluated with 2 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 53-53 H: 0-150 N: 0-30 O: 0-30 Na: 1-1

Minimum:

-1.5

Maximum:

10.0

10.0

50.0

Mass

Calc. Mass

mDa

PPM

DBE

i-FIT

i-FIT (Norm)

Formula

1134.5393

1134.5335

5.8

5.1

19.5

133.4

0.1

C53 H77 N9 O17 Na

1134.5461

-6.8

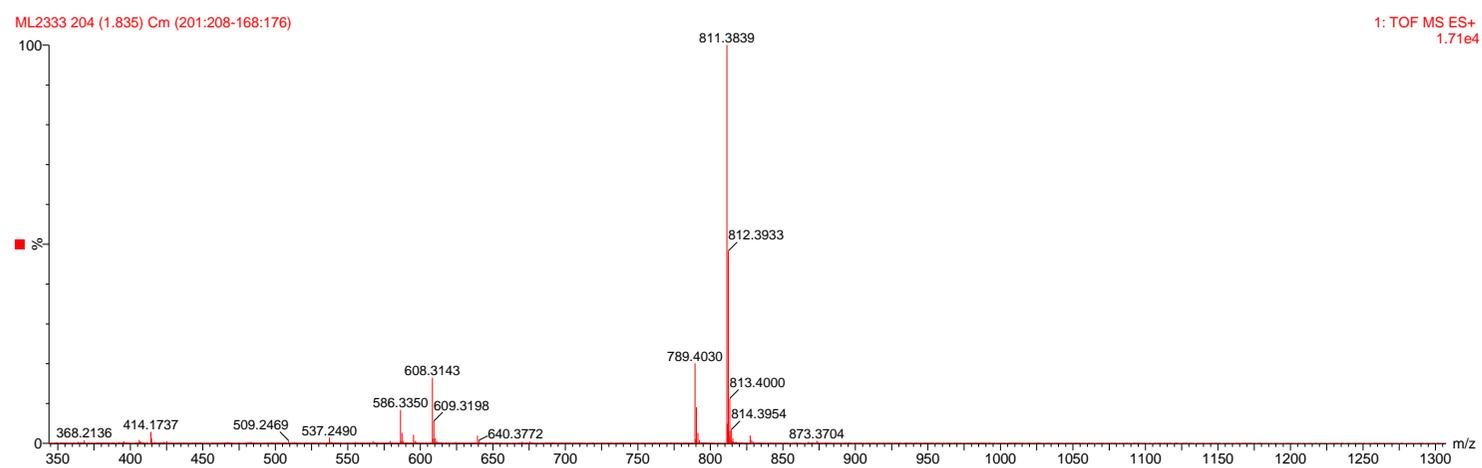
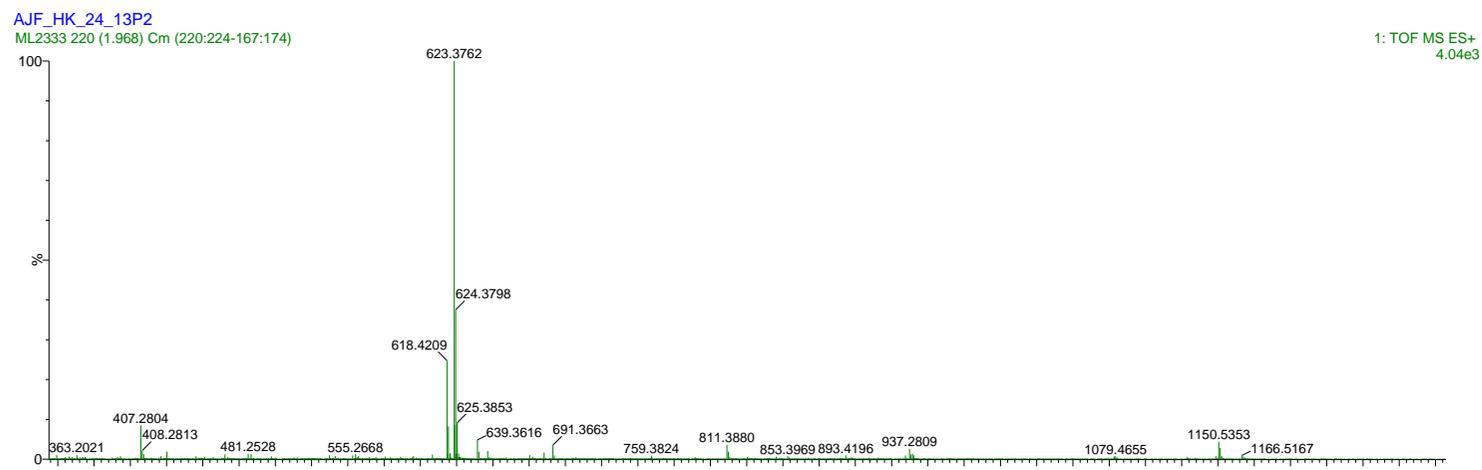
-6.0

24.5

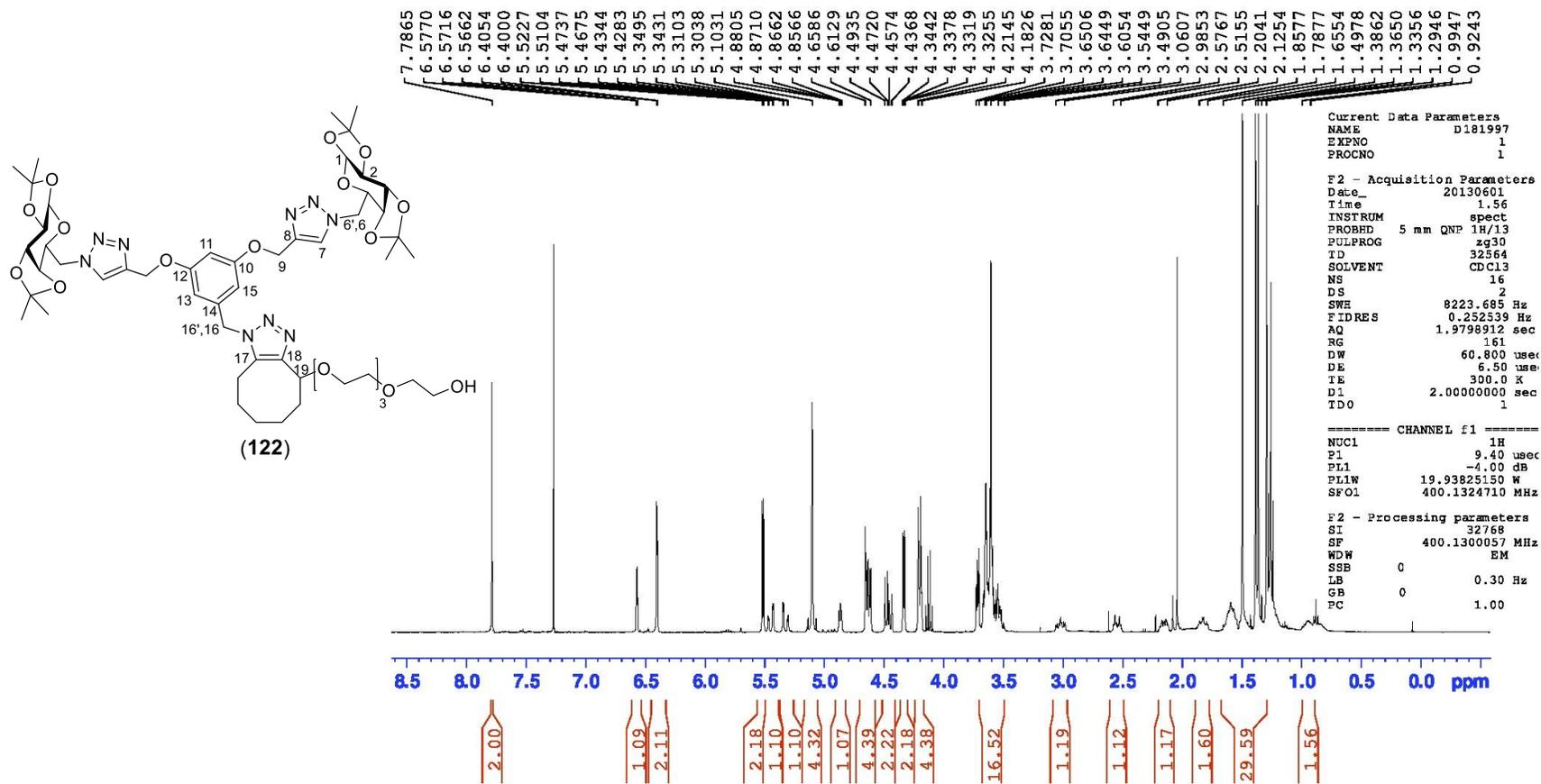
135.8

2.5

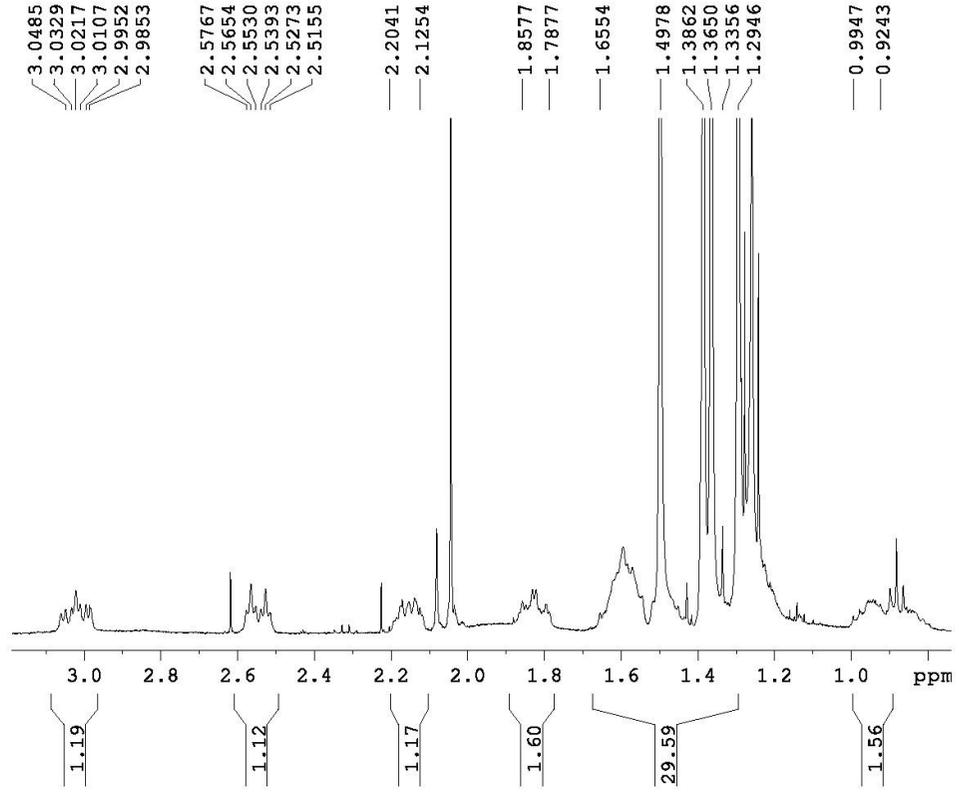
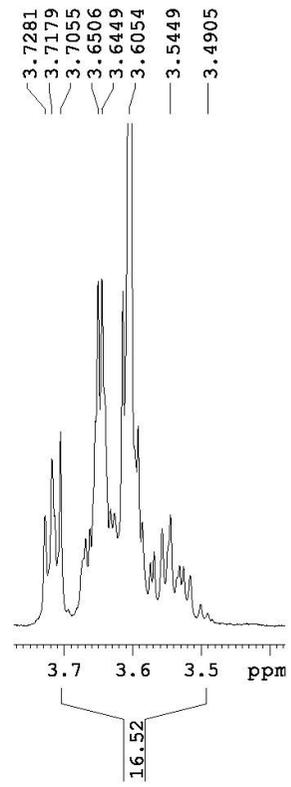
C53 H73 N15 O12 Na

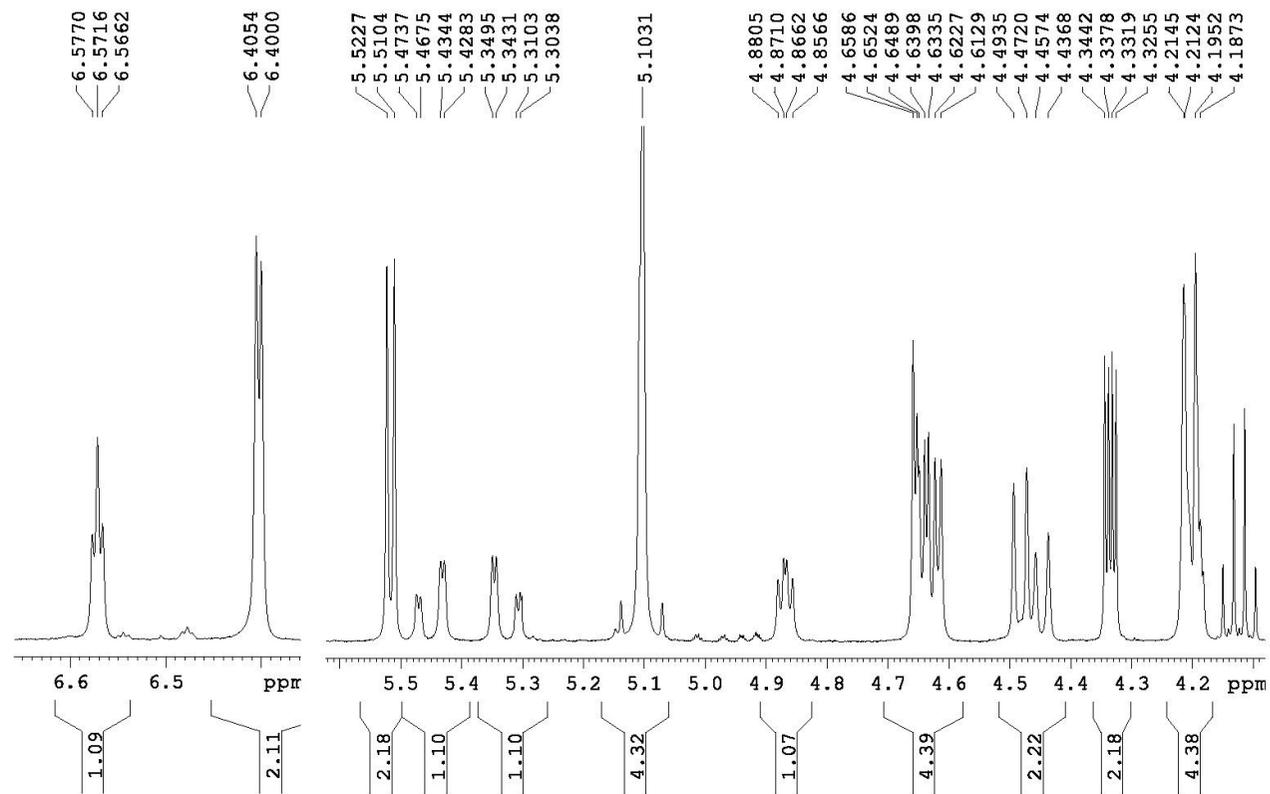


Appendix B71: HRMS (ESI) spectra of compound (122).

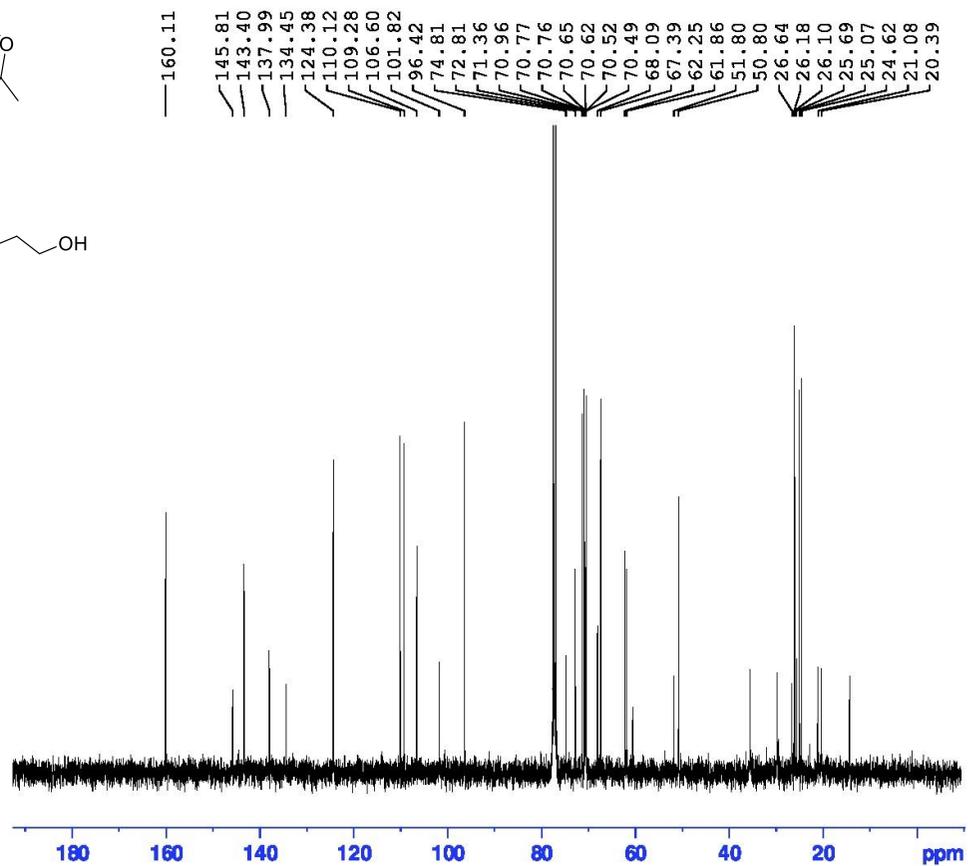
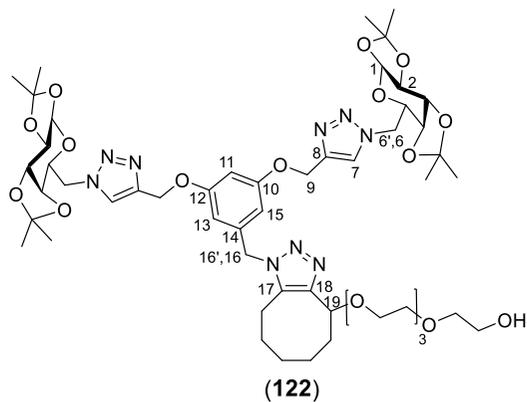


Appendix B72: ¹H NMR spectrum of compound (122).





Appendix B73: Selected areas ¹H NMR of compound (122).



```

Current Data Parameters
NAME      D183145
EXPNO     1
PROCNO    1

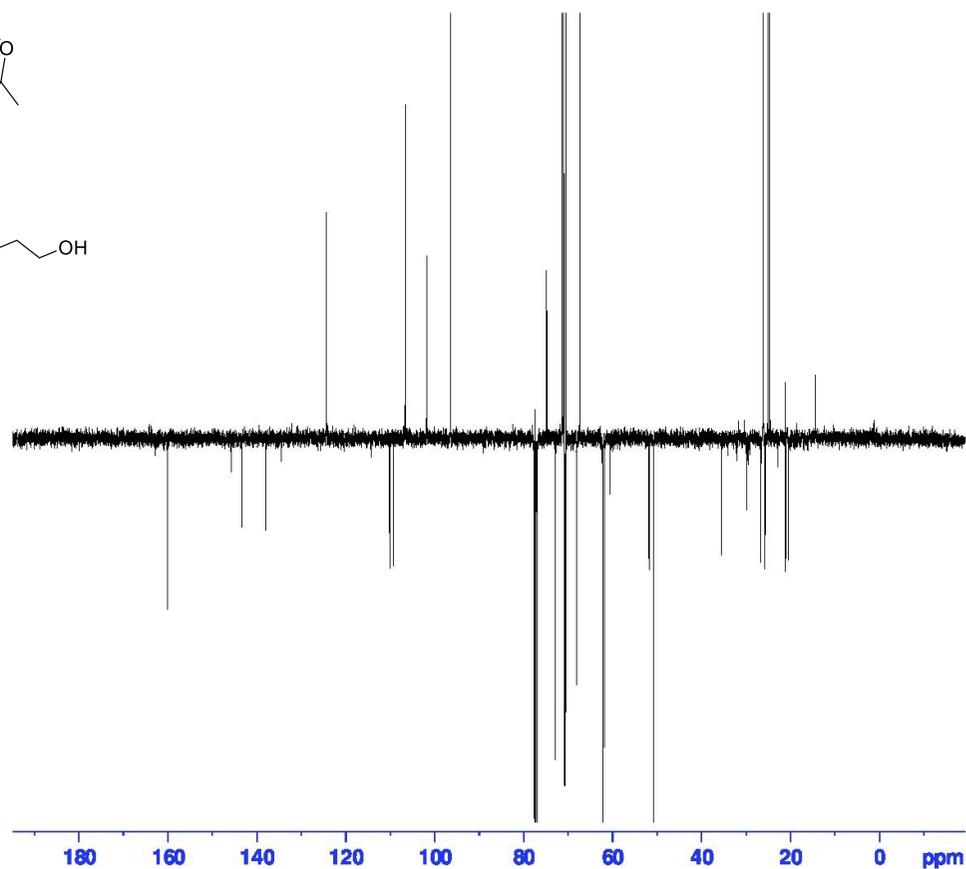
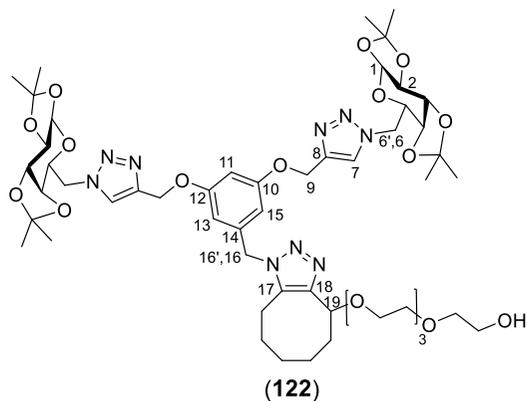
F2 - Acquisition Parameters
Date_     20130518
Time      23.52
INSTRUM   spect
PROBHD    5 mm QNP 1H/13
PULPROG   zgpg30
TD         16384
SOLVENT   CDCl3
NS         4096
DS         4
SWH        22058.824 Hz
FIDRES     1.346364 Hz
AQ         0.3713707 sec
RG         2050
DW         22.667 usec
DE         6.50 usec
TE         300.0 K
D1         0.69999999 sec
D11        0.03000000 sec
TDC        1

----- CHANNEL f1 -----
NUC1       13C
P1         7.00 usec
PL1        -2.60 dB
PL1W       61.34123230 W
SFO1       100.6228298 MHz

----- CHANNEL f2 -----
CPDPRG12   waltz16
NUC2        1H
PCPD2       80.00 usec
PL2         -4.00 dB
PL12        14.06 dB
PL13        120.00 dB
PL2W       19.93825150 W
PL12W       0.31166428 W
PL13W       0 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI          32768
SF          100.6127485 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40
  
```

Appendix B74: ^{13}C NMR spectrum of compound (122).



```

Current Data Parameters
NAME          D183875
EXPNO        1
PROCNO       1

F2 - Acquisition Parameters
Date_        20130704
Time         22.44
INSTRUM      spect
PROBHD       5 mm QNP 1H/13
PULPROG      zgpg30
TD           32768
SOLVENT      CDCl3
NS           2048
DS           4
SWH          24038.461 Hz
FIDRES       0.733596 Hz
AQ           0.6815744 sec
RG           2050
DN           20.800 usec
DE           6.50 usec
TE           300.0 K
CHET2        145.000000
CPC12        1.500000
DI           2.0000000 sec
DE           0.00344828 sec
D12          0.0002000 sec
D16          0.0002000 sec
D28          0.5000000 sec
TD0          1

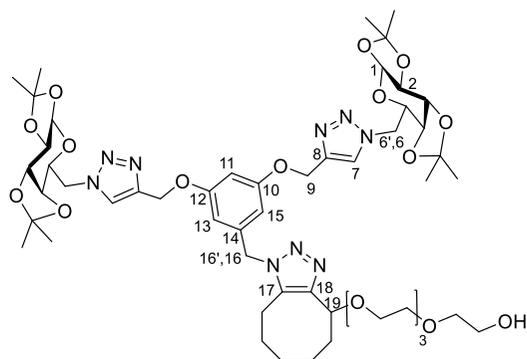
----- CHANNEL f1 -----
NUC1          13C
P1            7.00 usec
PL1          2000.00 usec
PL0          120.00 dB
PL2          -2.60 dB
FLOW         0 W
PL1W         61.34123230 W
SFO1         100.628298 MHz
SP2          8.66 dB
SFOAM [2]    Crp60comp.f
SFOAM2       0 Hz
SPOFFS2      0 Hz

----- CHANNEL f2 -----
CPDPRG [2]   waltz16
NUC2          1H
P2            15.00 usec
P3            10.00 usec
P4            20.00 usec
PCPD2        80.00 usec
PL2          -4.00 dB
PL12         14.06 dB
PL13         120.00 dB
PL1W         19.93823150 W
PL12W        0.31166428 W
PL13W        0 W
SFO2         400.1316005 MHz

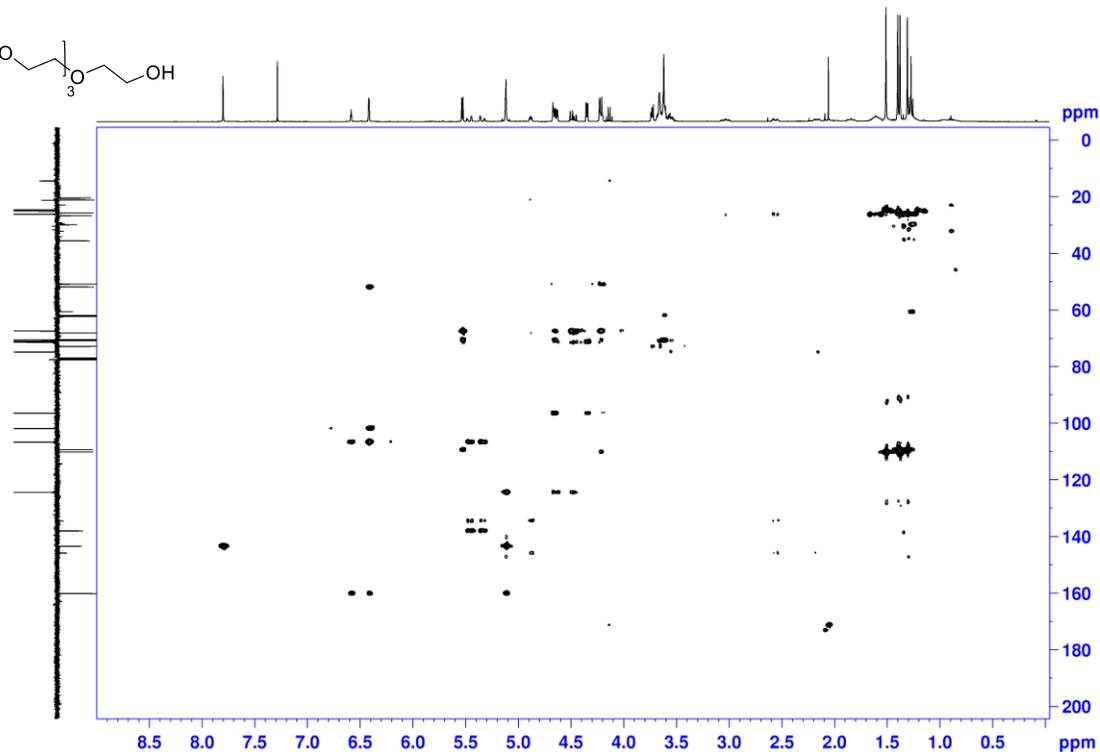
----- GRADIENT CHANNEL -----
GPNAM [1]    SINE.100
GPNAM [2]    SINE.100
GPNAM [3]    SINE.100
GPZ1         31.00 %
GPZ2         31.00 %
GPZ3         31.00 %
P16          1000.00 usec

F2 - Processing parameters
SI           32768
SF           100.6127493 MHz
WDW          EM
SSB          0
GB           1.00 Hz
PC           1.40
  
```

Appendix B75: DEPT C- NMR spectrum of compound (122).



(122)



```

Current Data Parameters
=====
EXPNO      2
PROCNO     1

F2 - Acquisition Parameter
=====
Date_      20130611
Time       19.54
INSTRUM    spect
PROBHD     5 mm QNP 1H/13
PULPROG    zgpg30
TD         65536
SOLVENT    CDCl3
NS         16
DS         16
SWH        8012.820 H
FIDRES     1.954255 Hz
AQ         0.2555904 s
RG         2050
DF         62.40 usec
DE         6.50 usec
TE         300.2 K
=====
CHNMR2  120.0000000
CHNMR3  130.0000000
CHNMR4  10.0000000
CHNMR5  0.0000000
DO      0.0961357
DI      1.500000000 #
DE      0.0000000 #
DIF     0.0000000 #
IM0     0.0000000 #

===== CHANNEL f1 =====
NUC1     13C
P1       9.40 usec
P2       18.00 usec
PT1      -4.00 dB
PL1W     19.93820150 W
SFO1     400.13240100 MHz

===== CHANNEL f2 =====
NUC2     13C
P3       9.00 usec
P4       2000.00 usec
P12      -3.00 dB
PL12W    87.25922122 W
RFU2     100.6220138 MHz
SP7      2.26 dB
CPDPRG7  Crp40comp.4
RF CAL7  0 Hz
RF GEN7  0 Hz

===== GRADIENT CHANNEL =====
GRNAM(1)  znsq10.100
GRNAM(3)  znsq10.100
GRNAM(4)  znsq10.100
GRNAM(5)  znsq10.100
GRNAM(6)  znsq10.100
CP1       80.00 %
CP2       14.00 %
CP3       -8.00 %
CP4       -4.00 %
CP5       -2.00 %
P15       1000.00 usec

F1 - Acquisition parameter:
=====
TD         256
SFO1     100.6220138 MHz
FIDRES     87.25922122 Hz
SF         222.095 MHz
PROCNO     Echo-AntiEcho

F2 - Processing parameter
=====
SF         400.1300000 MHz
WDW        gq1w2
SSB        2
GB         0 Hz
GB2        0
PC         1.40

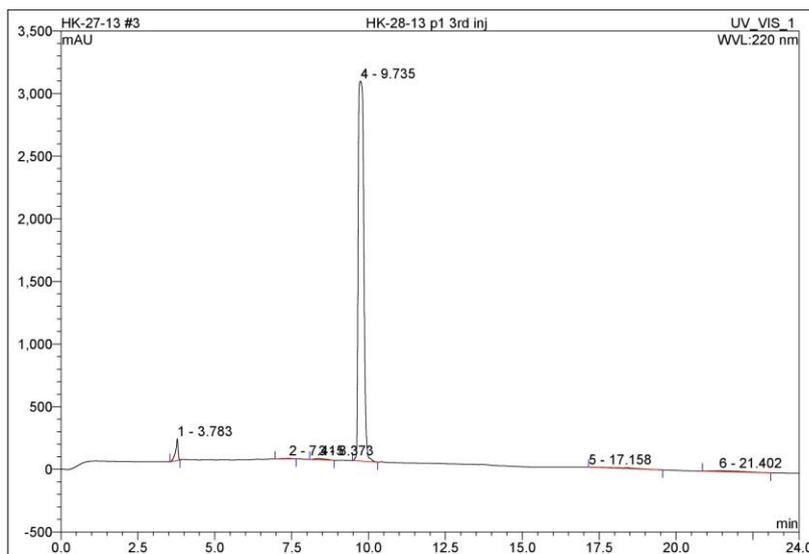
F1 - Processing parameter:
=====
SI         32768
MC1       echo-antiecho
SF         100.6127690 MHz
WDW        gq1w2
SSB        2
GB         0 Hz
GB2        0

```

Appendix B77: HMBC NMR spectrum of compound (122).

3 HK-28-13 p1 3rd inj

Sample Name:	HK-28-13 p1 3rd inj	Injection Volume:	20.0
Vial Number:	RA3	Channel:	UV_VIS_1
Sample Type:	unknown	Wavelength:	220
Control Program:	poly-p4 28min +230nm	Bandwidth:	10
Quantif. Method:	dna method	Dilution Factor:	1.0000
Recording Time:	24/6/2013 17:49	Sample Weight:	1.0000
Run Time (min):	24.00	Sample Amount:	1.0000



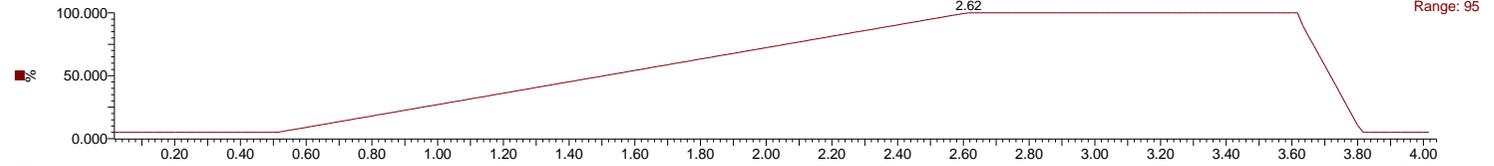
No.	Ret. Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	3.78	n.a.	173.268	16.223	2.42	n.a.	BMB
2	7.42	n.a.	3.950	1.622	0.24	n.a.	BMB
3	8.37	n.a.	10.140	4.226	0.63	n.a.	BMB
4	9.74	n.a.	3033.827	633.805	94.59	n.a.	BMB
5	17.16	n.a.	0.000	5.923	0.88	n.a.	BMB
6	21.40	n.a.	6.434	8.236	1.23	n.a.	BMB
Total:			3227.620	670.035	100.00	0.000	

default/Integration

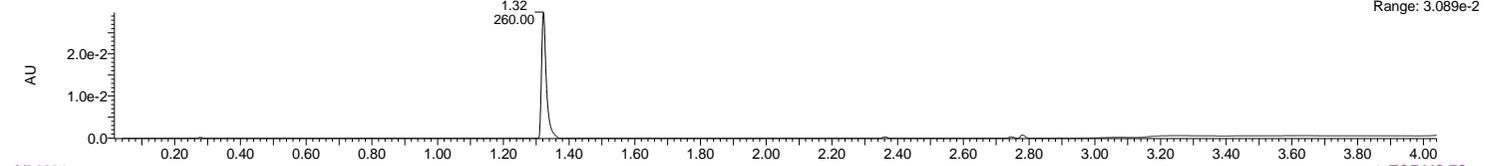
Chromeleon (c) Dionex 1996-2006
Version 6.80 SP4 Build 2361 (130805)*Appendix B79: HPL chromatogram of compound (24).*

AJF_HK_28_13P1

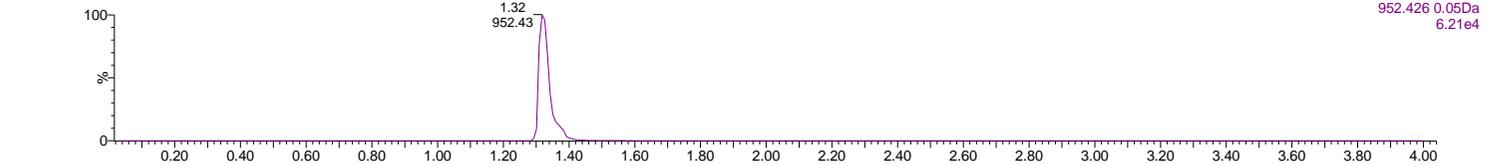
ML2334



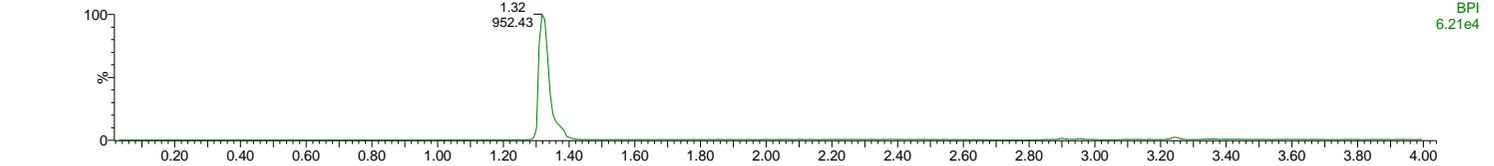
ML2334



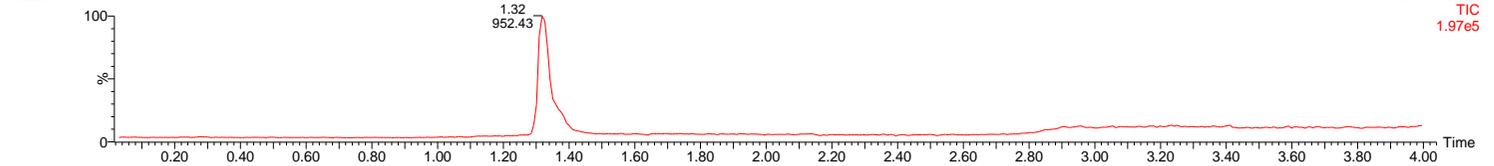
ML2334



ML2334

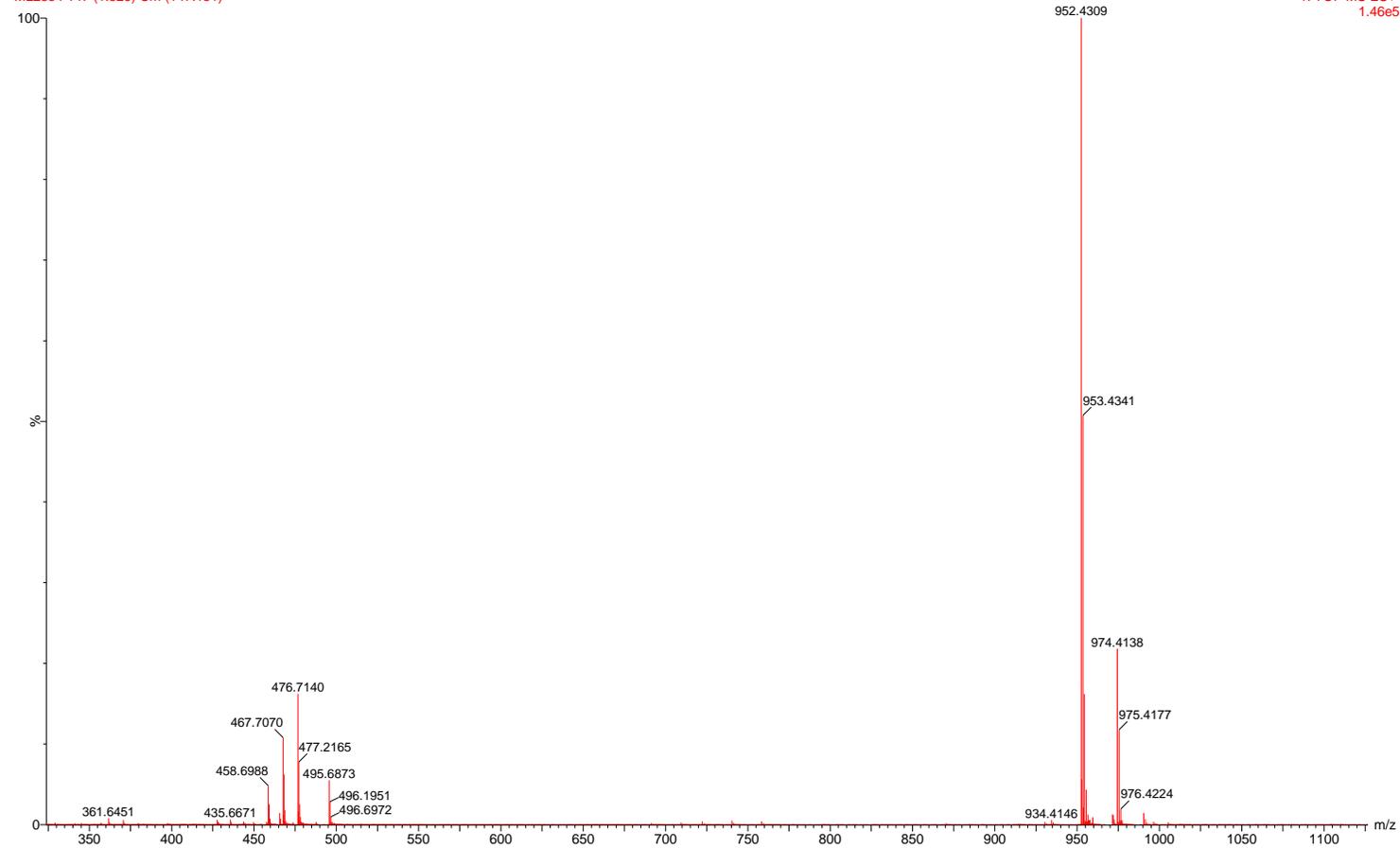


ML2334



AJF_HK_28_13P1

ML2334 147 (1.326) Cm (147:151)



Elemental Composition Report: **AJF-HK-28-13P1**

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

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

Elements Used:

C: 41-41 H: 0-150 N: 0-30 O: 0-30

Minimum:

-1.5

Maximum:

10.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
952.4309	952.4264	4.5	4.7	15.5	156.3	0.0	C41 H62 N9 O17

Elemental Composition Report: **AJF-HK-28-13P1**

Single Mass Analysis

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

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

953 formula(e) evaluated with 2 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 41-41 H: 0-150 N: 0-30 O: 0-30 Na: 1-1

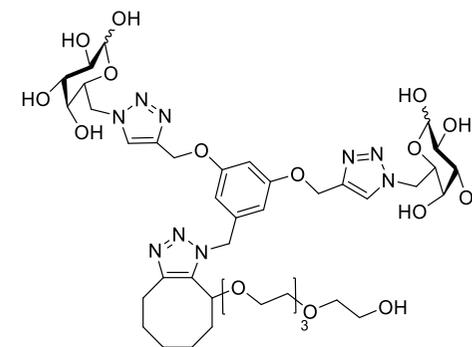
Minimum:

-1.5

Maximum:

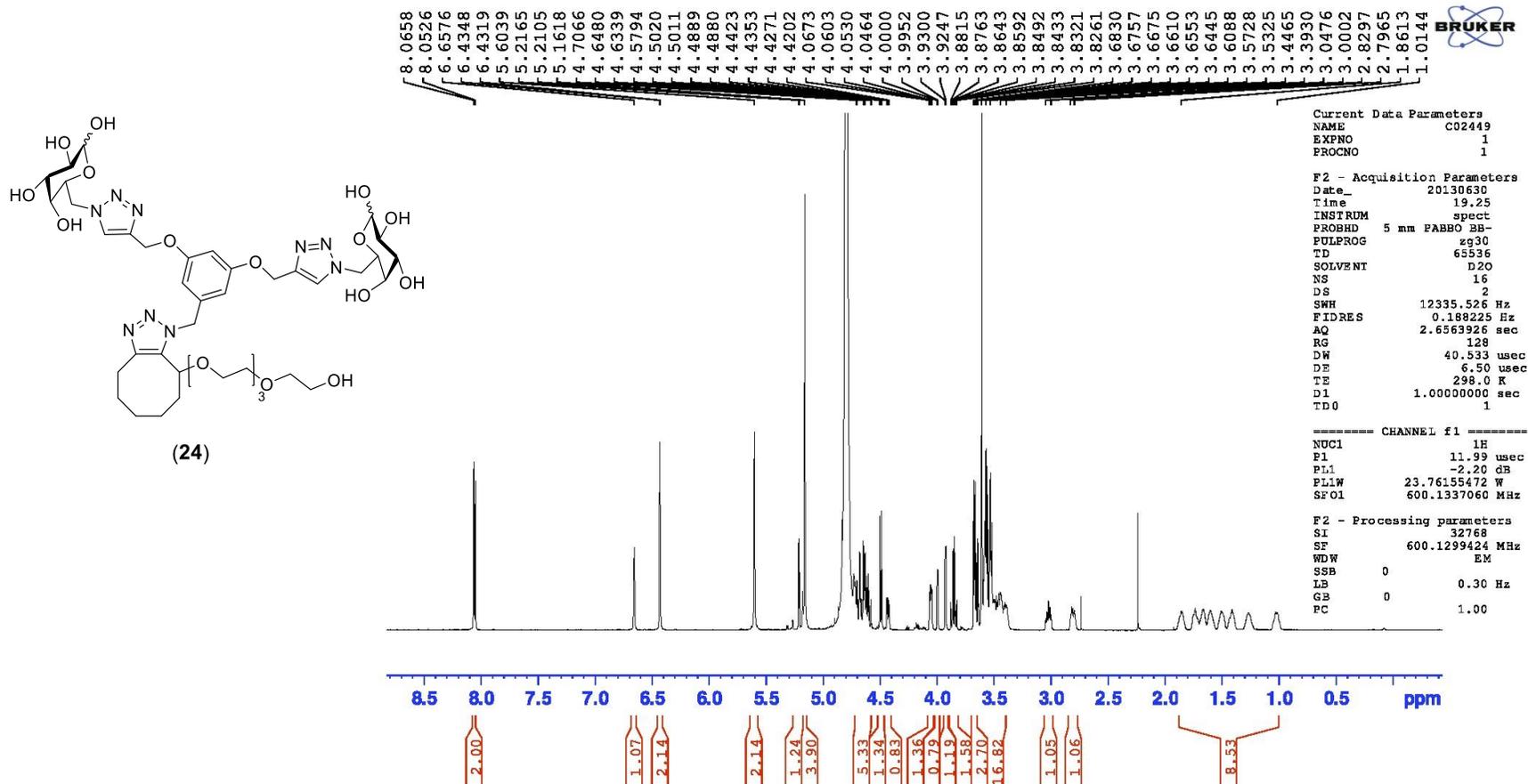
10.0 10.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
974.4138	974.4083	5.5	5.6	15.5	188.8	0.7	C41 H61 N9 O17 Na
	974.4209	-7.1	-7.3	20.5	188.8	0.7	C41 H57 N15 O12 Na

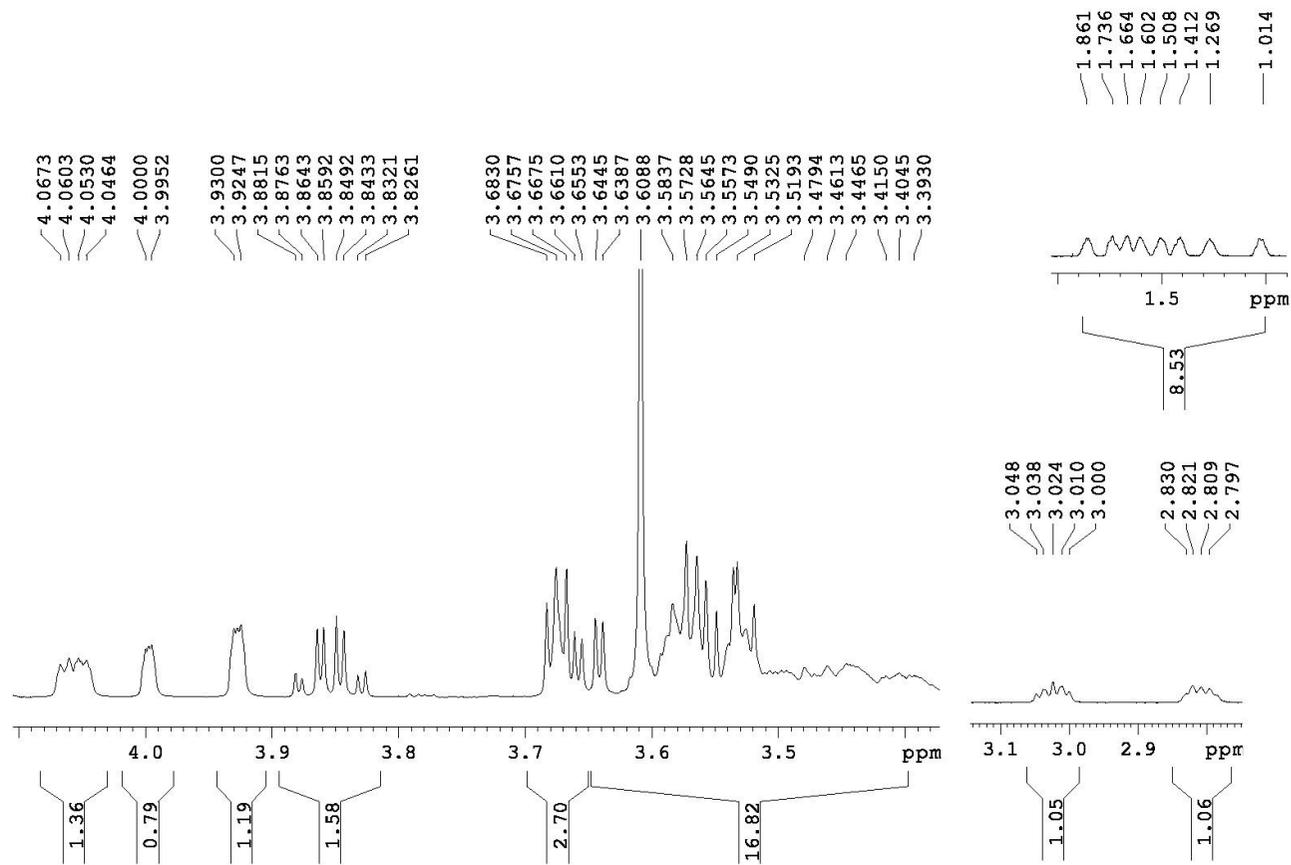


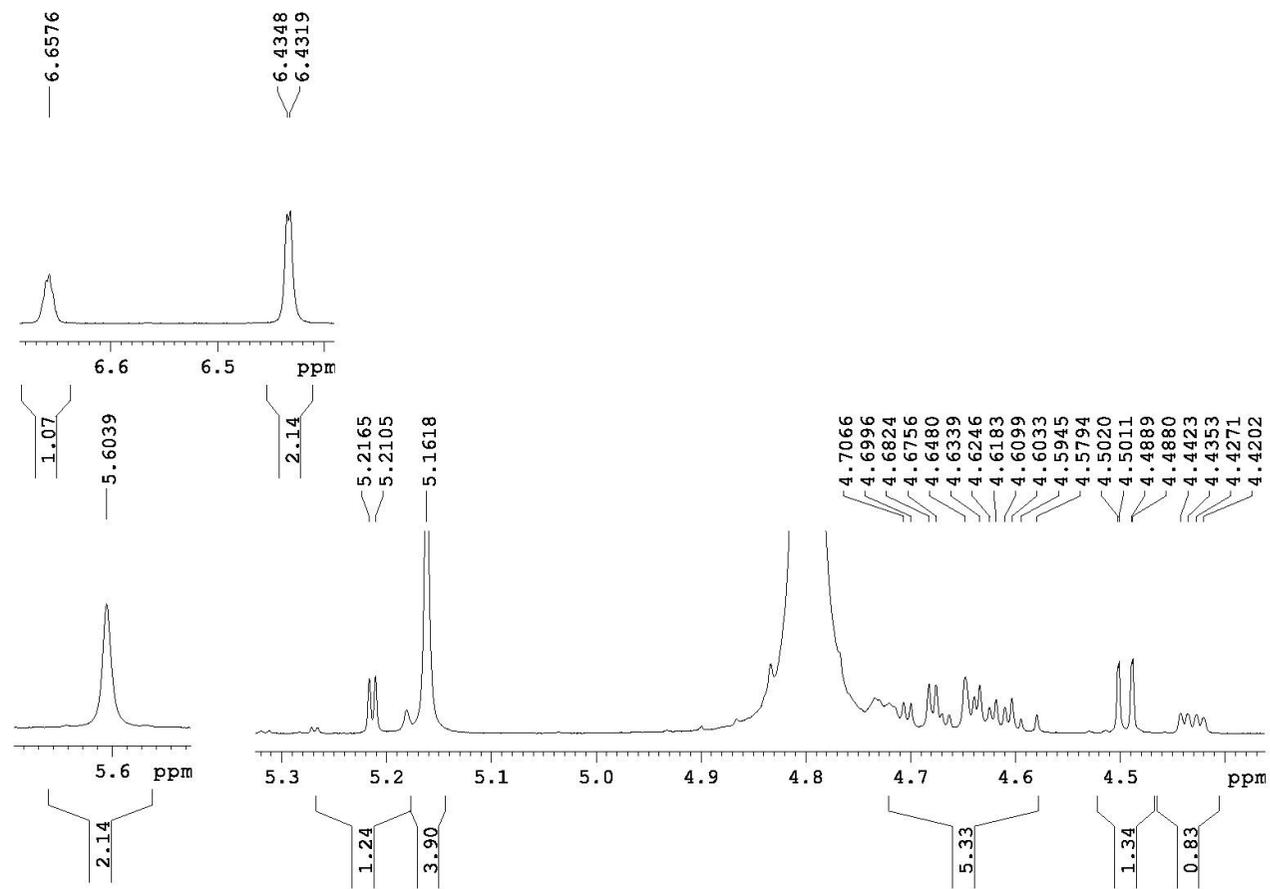
(24)

Appendix B80: HRMS (ESI) spectra of compound (24).

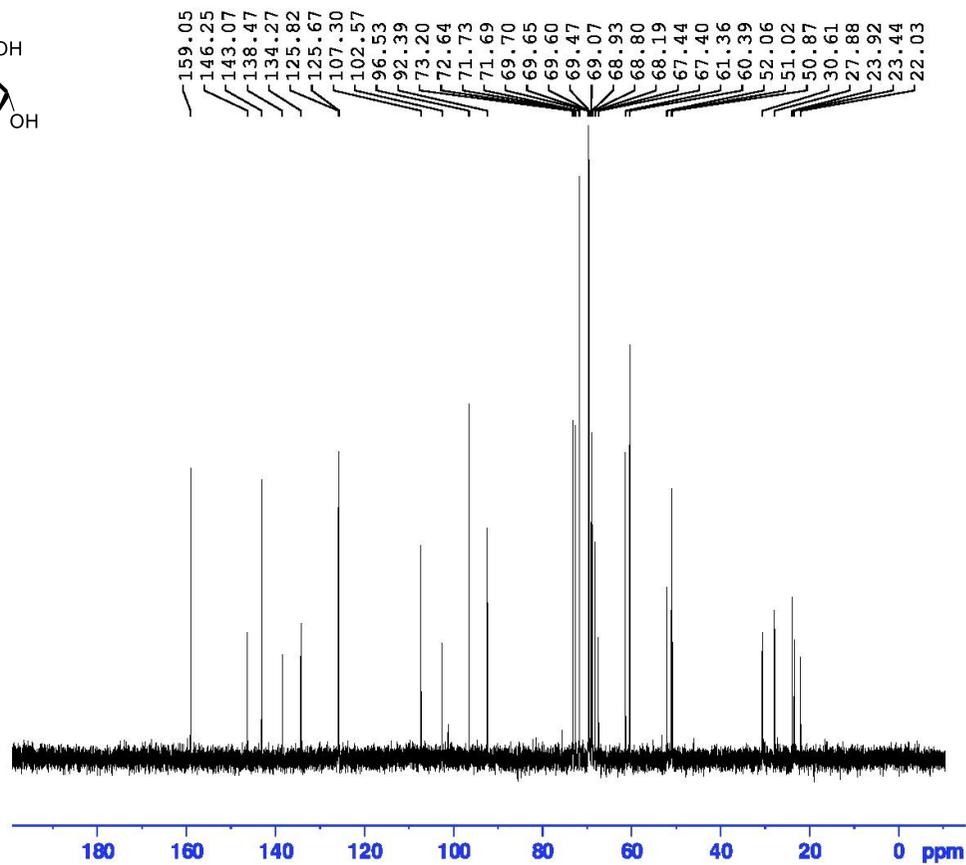
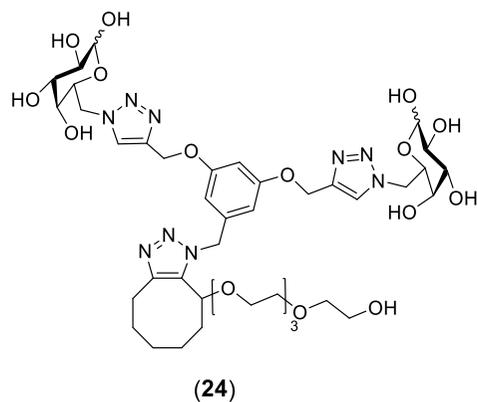


Appendix B81: ¹H NMR spectrum of compound (24).





Appendix B82: Selected areas ^1H NMR of compound (24).



Current Data Parameters
NAME C02449
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130701
Time 1.15
INSTRUM spect
PROBHD 5 mm F4BBO BBO
PULPROG zgpg30
TD 32768
SOLVENT D2O
DS 4
SWH 33333.332 Hz
FIDRES 1.017253 Hz
AQ 0.4915200 sec
RG 2050
DW 15.000 usec
DE 8.25 usec
TE 300.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 0 dB
PL1W 90.17671967 W
SF01 150.9178993 MHz

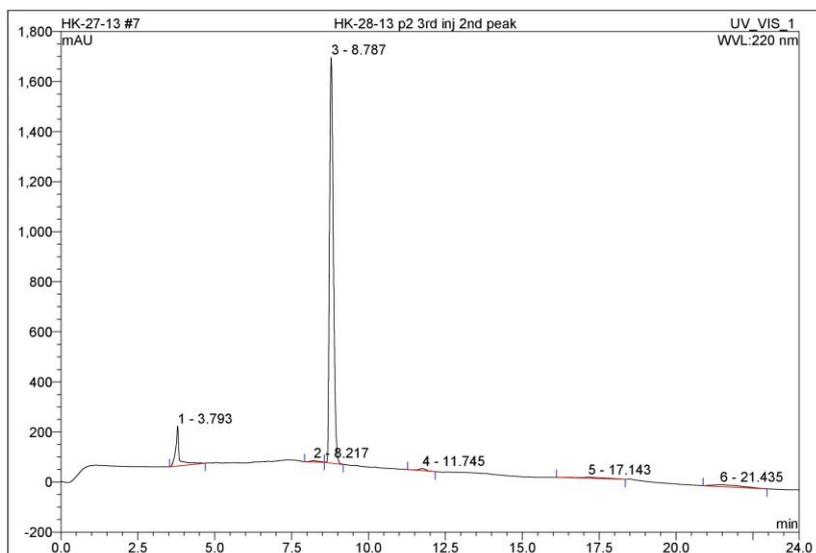
===== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 1H
PCPD2 69.05 usec
PL2 -2.20 dB
PL12 13.00 dB
PL13 14.00 dB
PL2W 23.76155472 W
PL12W 0.71758747 W
PL13W 0.56999999 W
SF02 600.1324005 MHz

F2 - Processing parameters
SI 32768
SF 150.9028090 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Appendix B83: ^{13}C NMR spectrum of compound (24).

7 HK-28-13 p2 3rd inj 2nd peak

Sample Name:	HK-28-13 p2 3rd inj 2nd peak	Injection Volume:	20.0
Vial Number:	RA7	Channel:	UV_VIS_1
Sample Type:	unknown	Wavelength:	220
Control Program:	poly-p4 28min +230nm	Bandwidth:	10
Quantif. Method:	dna method	Dilution Factor:	1.0000
Recording Time:	24/6/2013 19:44	Sample Weight:	1.0000
Run Time (min):	24.00	Sample Amount:	1.0000



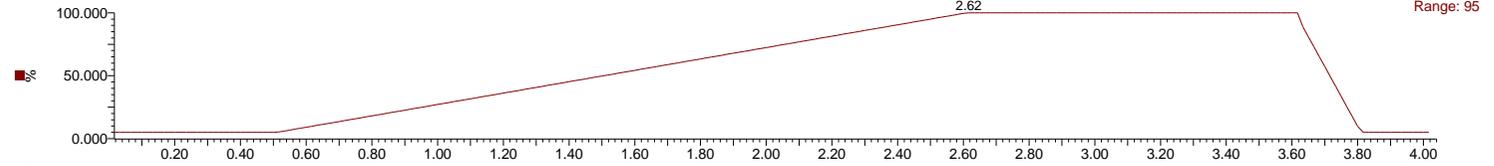
No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	3.79	n.a.	159.996	23.094	8.37	n.a.	BMB
2	8.22	n.a.	5.056	1.612	0.58	n.a.	BMb
3	8.79	n.a.	1620.621	235.657	85.42	n.a.	bMB
4	11.75	n.a.	8.153	2.139	0.78	n.a.	BMB
5	17.14	n.a.	4.423	4.775	1.73	n.a.	BMB
6	21.44	n.a.	7.267	8.617	3.12	n.a.	BMB
Total:			1805.516	275.894	100.00	0.000	

default/integration

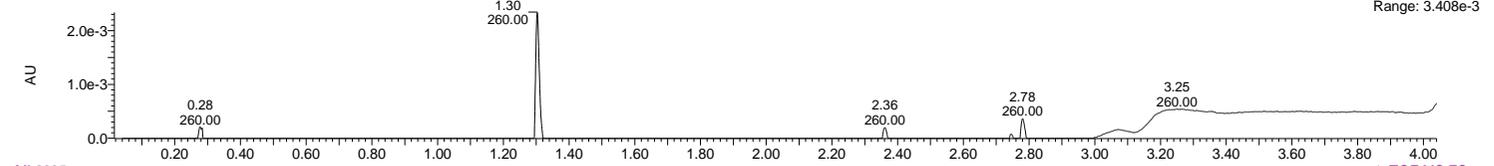
Chromeleon (c) Dionex 1996-2006
Version 6.80 SP4 Build 2361 (130805)*Appendix B84: HPL chromatogram of compound (25).*

AJF_HK_28_13P2

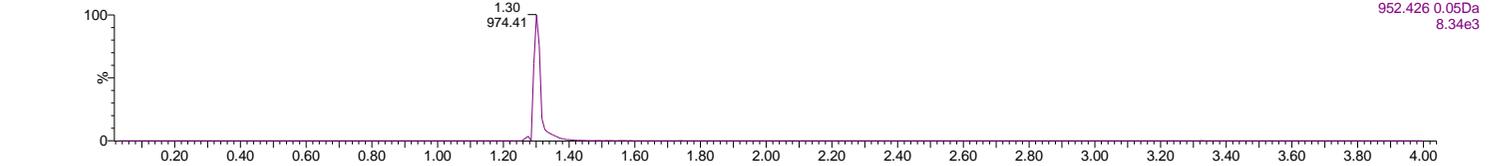
ML2335



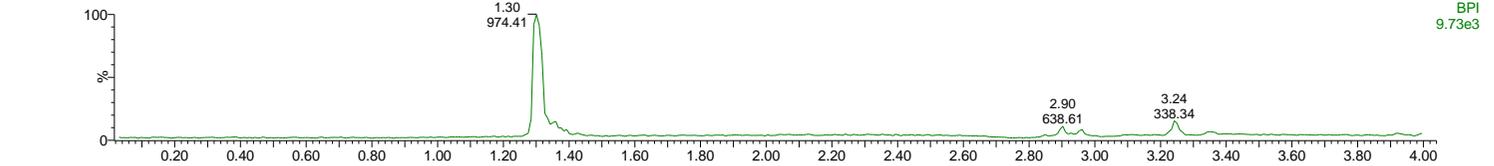
ML2335



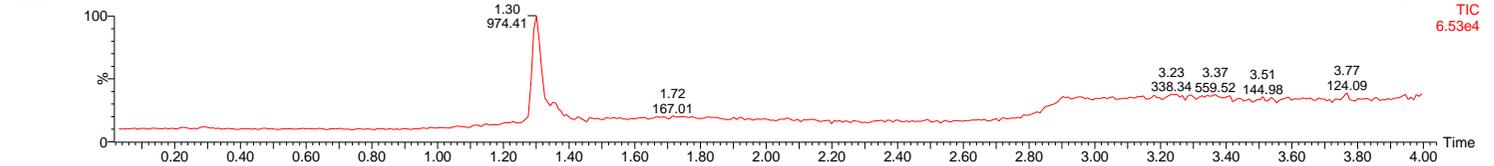
ML2335



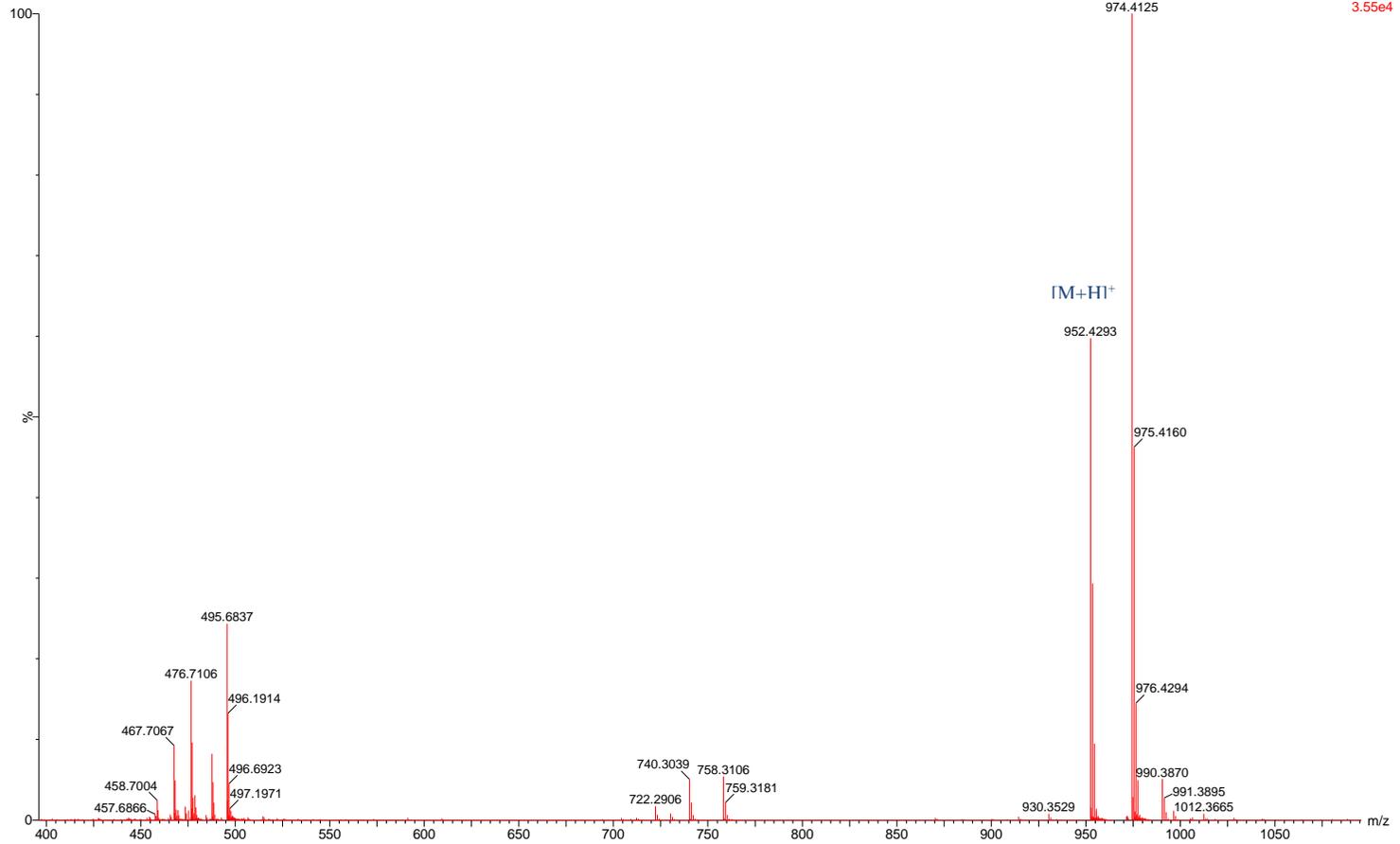
ML2335



ML2335



AJF_HK_28_13P2
ML2335 144 (1.301) Cm (142:146)



Elemental Composition Report: **AJF-HK-28-13P2**

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

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

Elements Used:

C: 41-41 H: 0-150 N: 0-30 O: 0-30

Minimum:

-1.5

Maximum:

10.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
952.4293	952.4264	2.9	3.0	15.5	138.0	0.0	C41 H62 N9 O17

Elemental Composition Report: **AJF-HK-28-13P2**

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

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

Elements Used:

C: 41-41 H: 0-150 N: 0-30 O: 0-30 Na: 1-1

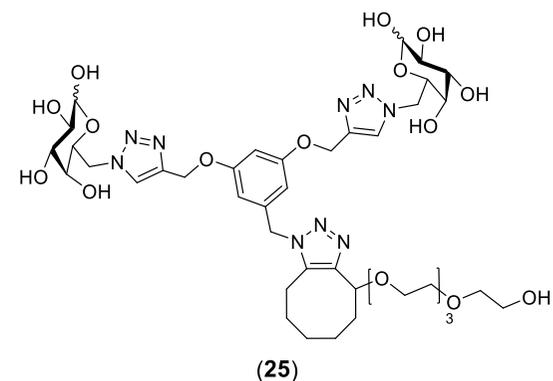
Minimum:

-1.5

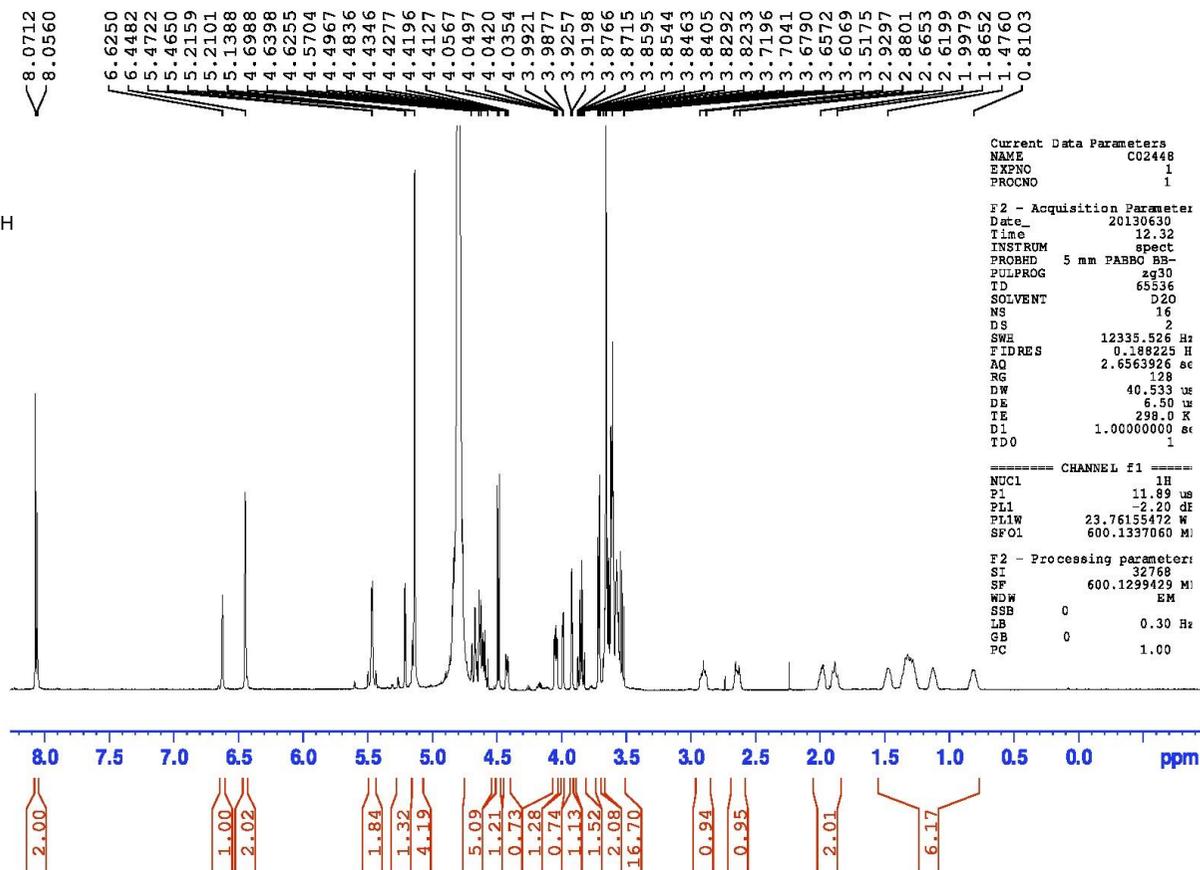
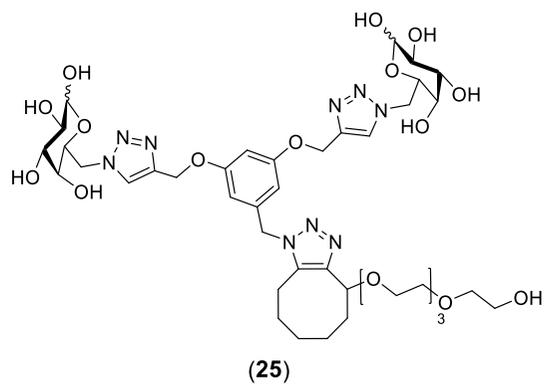
Maximum:

10.0 5.0 50.0

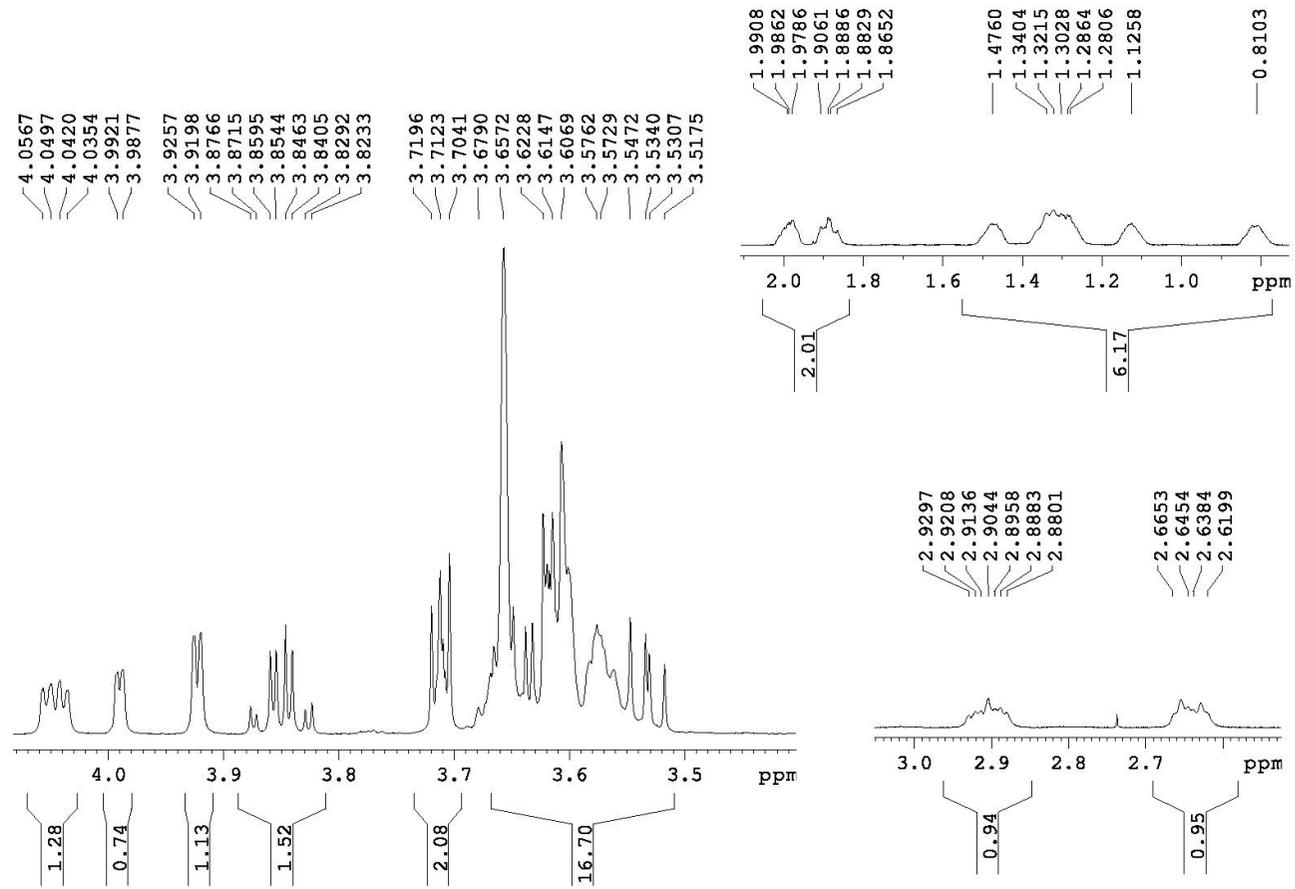
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
974.4125	974.4083	4.2	4.3	15.5	121.0	0.0	C41 H61 N9 O17 Na

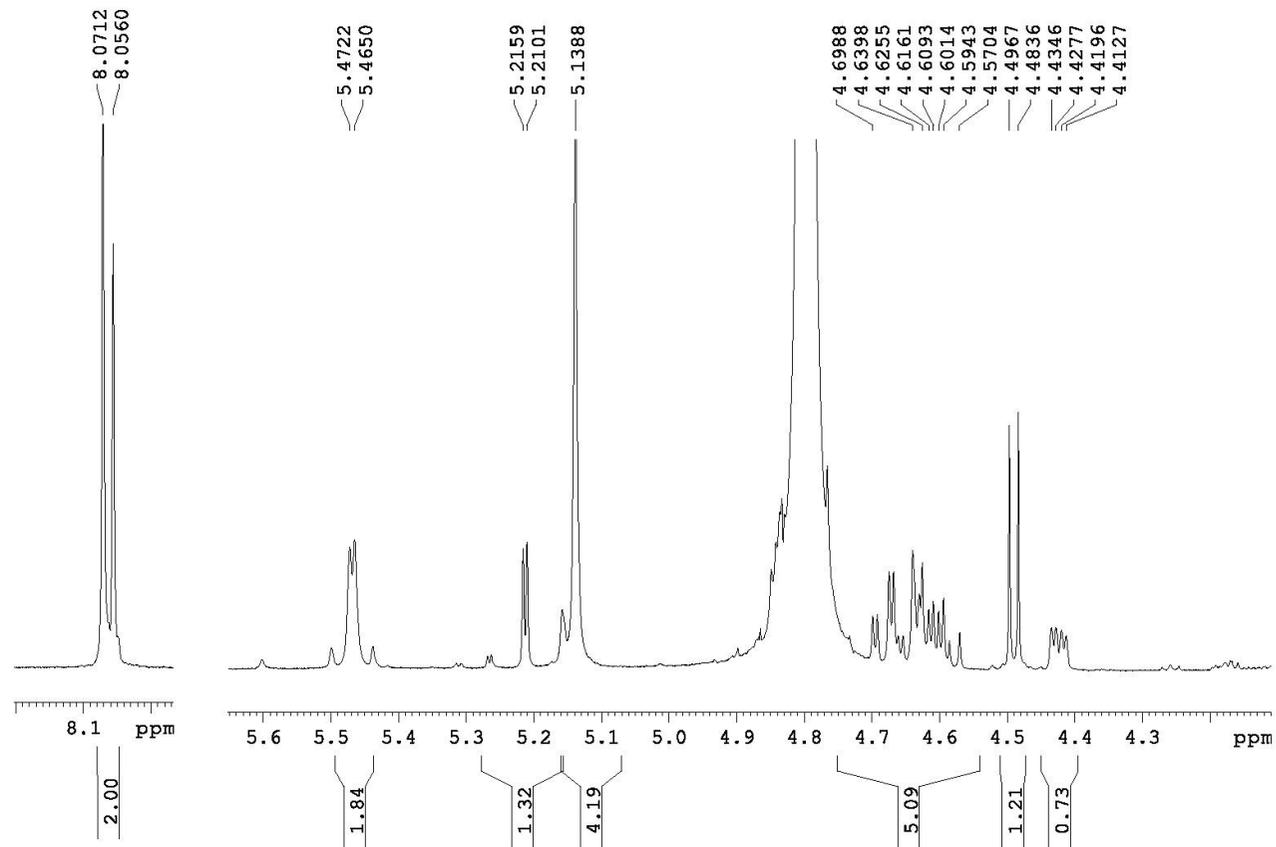


Appendix B85: HRMS (ESI) spectra of compound (25).

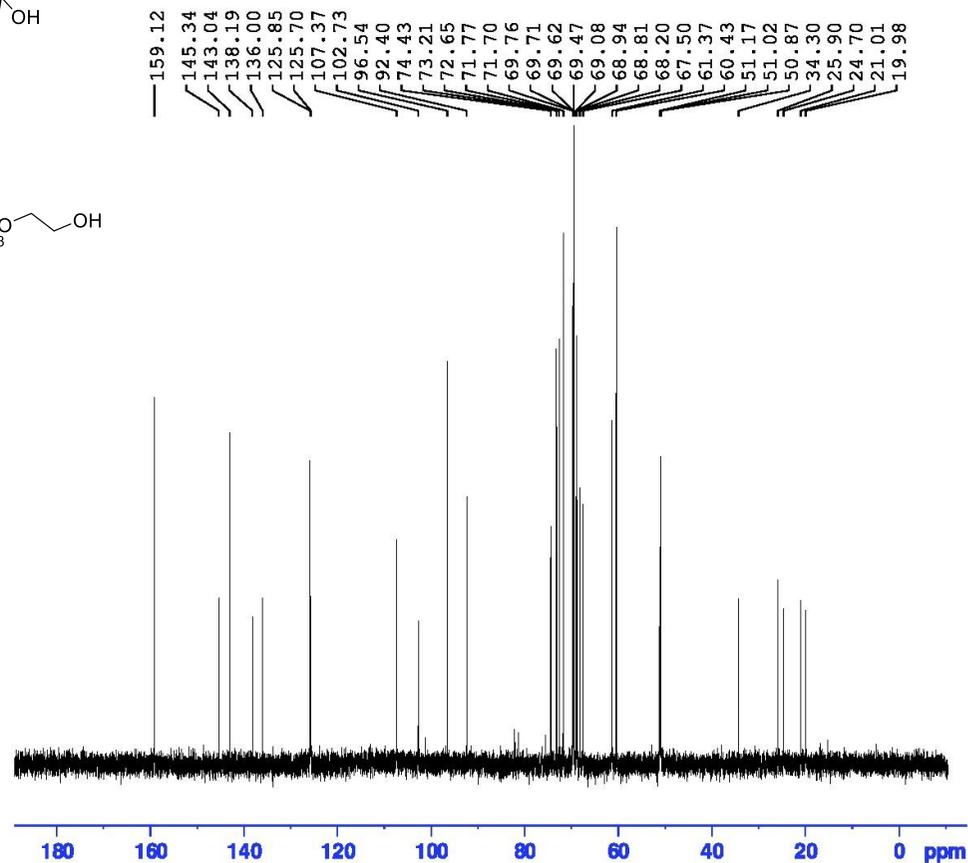
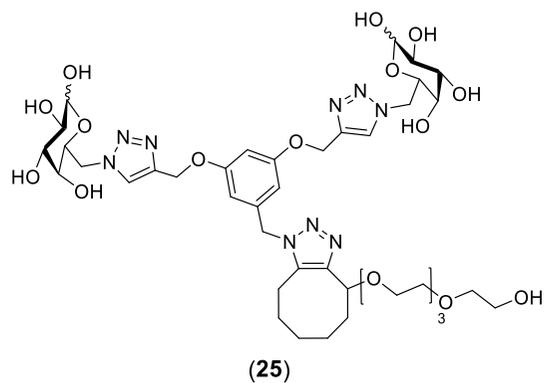


Appendix B86: ¹H NMR spectrum of compound (25).





Appendix B87: Selected areas ¹H NMR of compound (25).



```

Current Data Parameters
NAME          C02448
EXPNO         2
PROCNO        1

F2 - Acquisition Parameters
Date_         20130630
Time          18.22
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            32768
SOLVENT       D2O
NS            8192
DS            4
SWH           33333.332 Hz
FIDRES        1.017253 Hz
AQ            0.4915200 sec
RG            2050
DW            15.000 usec
DE            8.25 usec
TE            301.9 K
D1            2.00000000 sec
D11           0.03000000 sec
TDC           1

----- CHANNEL f1 -----
NUC1          13C
P1            8.00 usec
PL1           0 dB
PL1W          90.17671967 W
SFO1          150.9178993 MHz

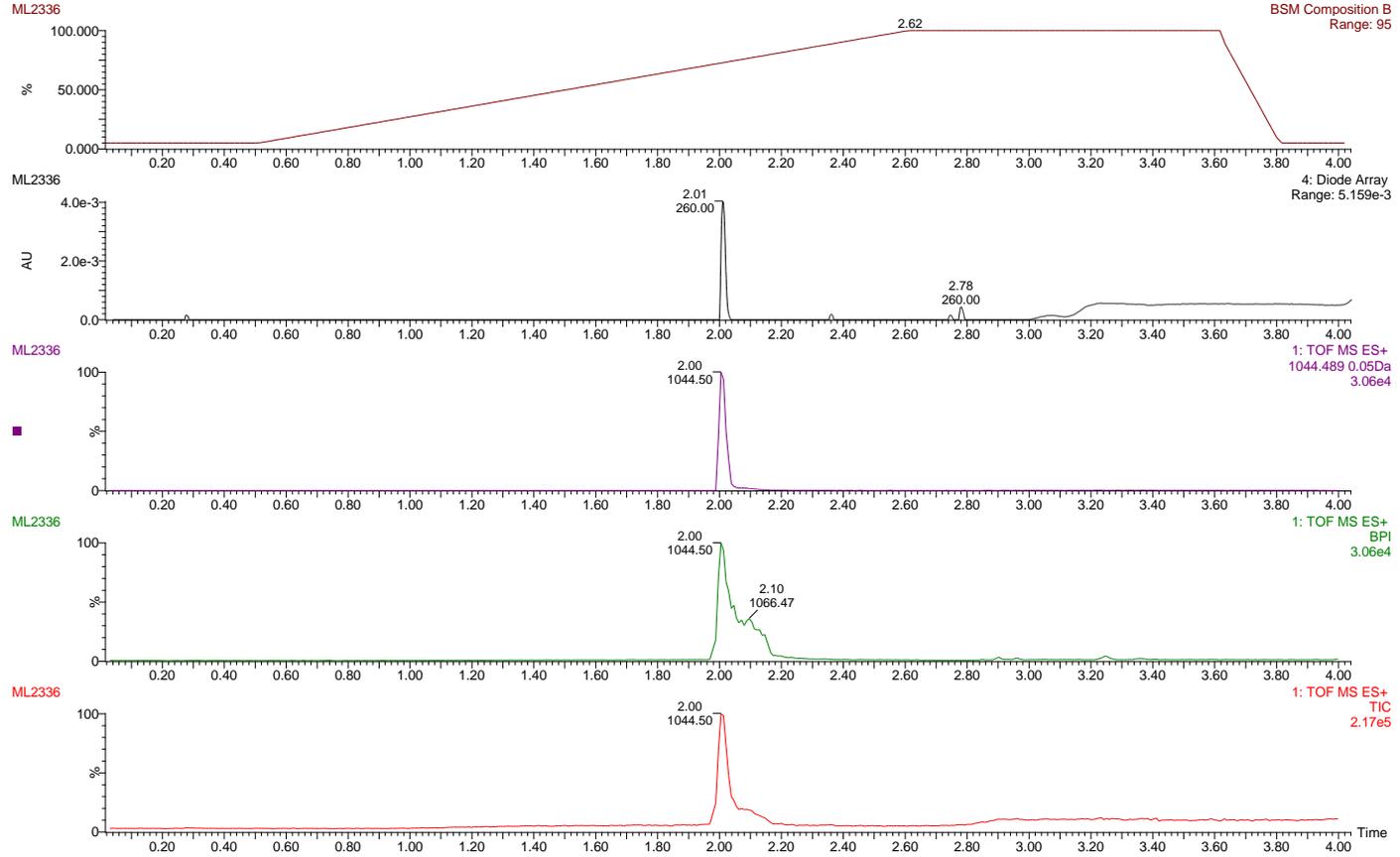
----- CHANNEL f2 -----
CPDPRG [2]   waltz16
NUC2          1H
PCPD2         69.05 usec
PL2           -2.20 dB
PL12          13.00 dB
PL13          14.00 dB
PL2W          23.76155472 W
PL12W         0.71758747 W
PL13W         0.569999999 W
SFO2          600.1324005 MHz

F2 - Processing parameters
SI            32768
SF            150.9028090 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40

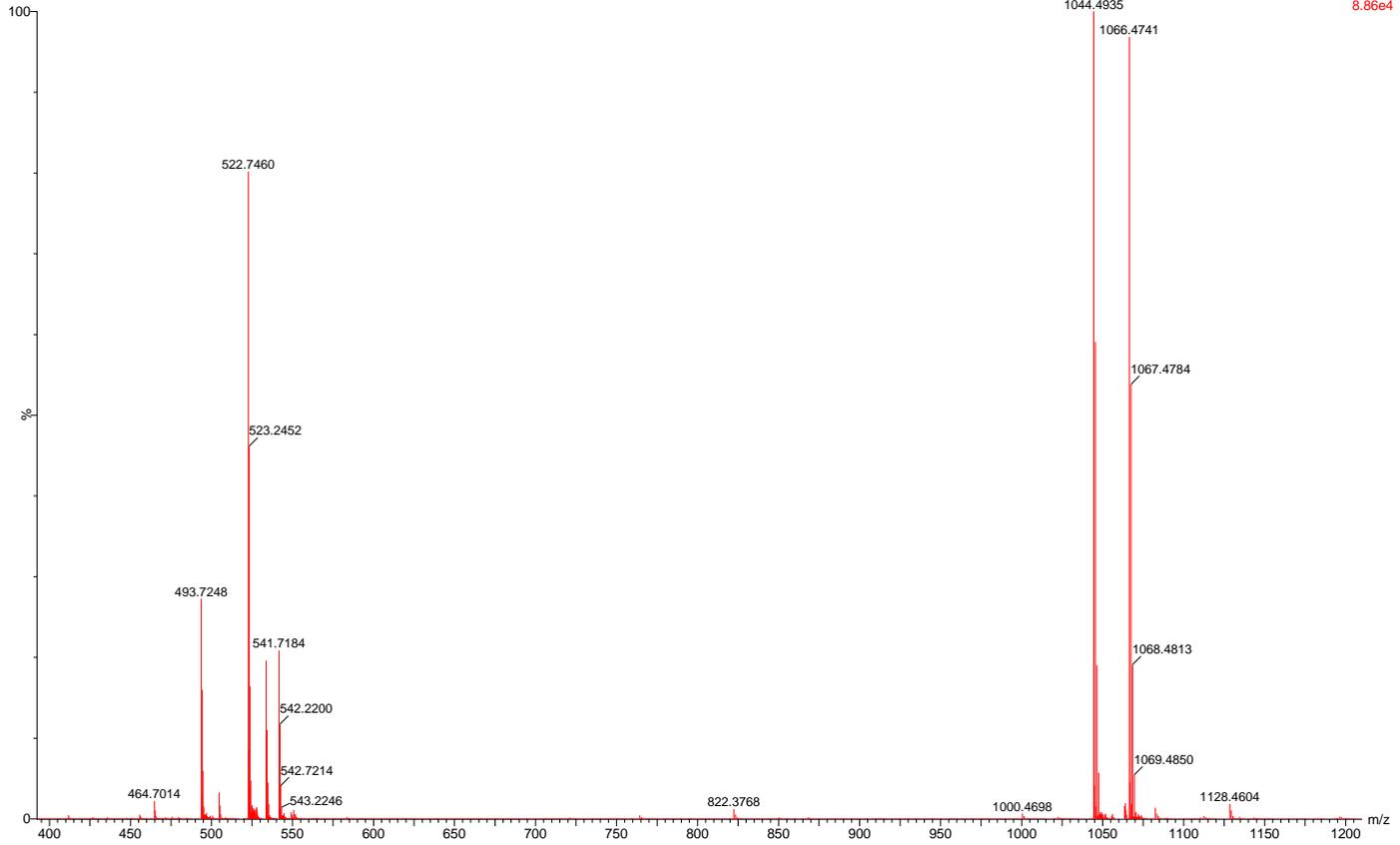
```

Appendix B88: ^{13}C NMR spectrum of compound (25).

AJF_HK_29_13 protected sugars



AJF_HK_29_13 protected sugars
ML2336 223 (2.005) Cm (222:225)



Elemental Composition Report: **AJF-HK-29-13 protected sugars**

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

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

Elements Used:

C: 48-48 H: 0-150 N: 0-30 O: 0-30

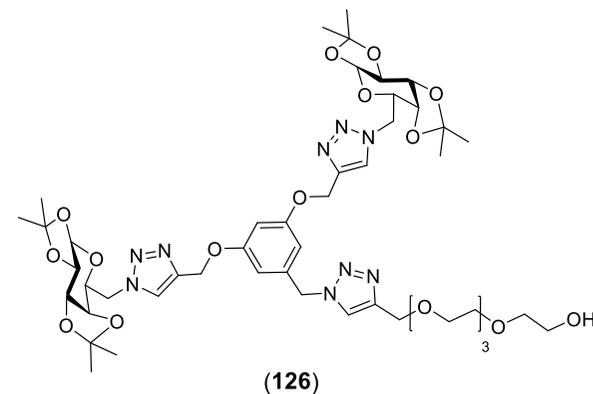
Minimum:

-1.5

Maximum:

10.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
1044.4935	1044.4890	4.5	4.3	18.5	103.4	0.0	C ₄₈ H ₇₀ N ₉ O ₁₇



Elemental Composition Report: **AJF-HK-29-13 protected sugars**

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

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

Elements Used:

C: 48-48 H: 0-150 N: 0-30 O: 0-30 Na: 1-1

Minimum:

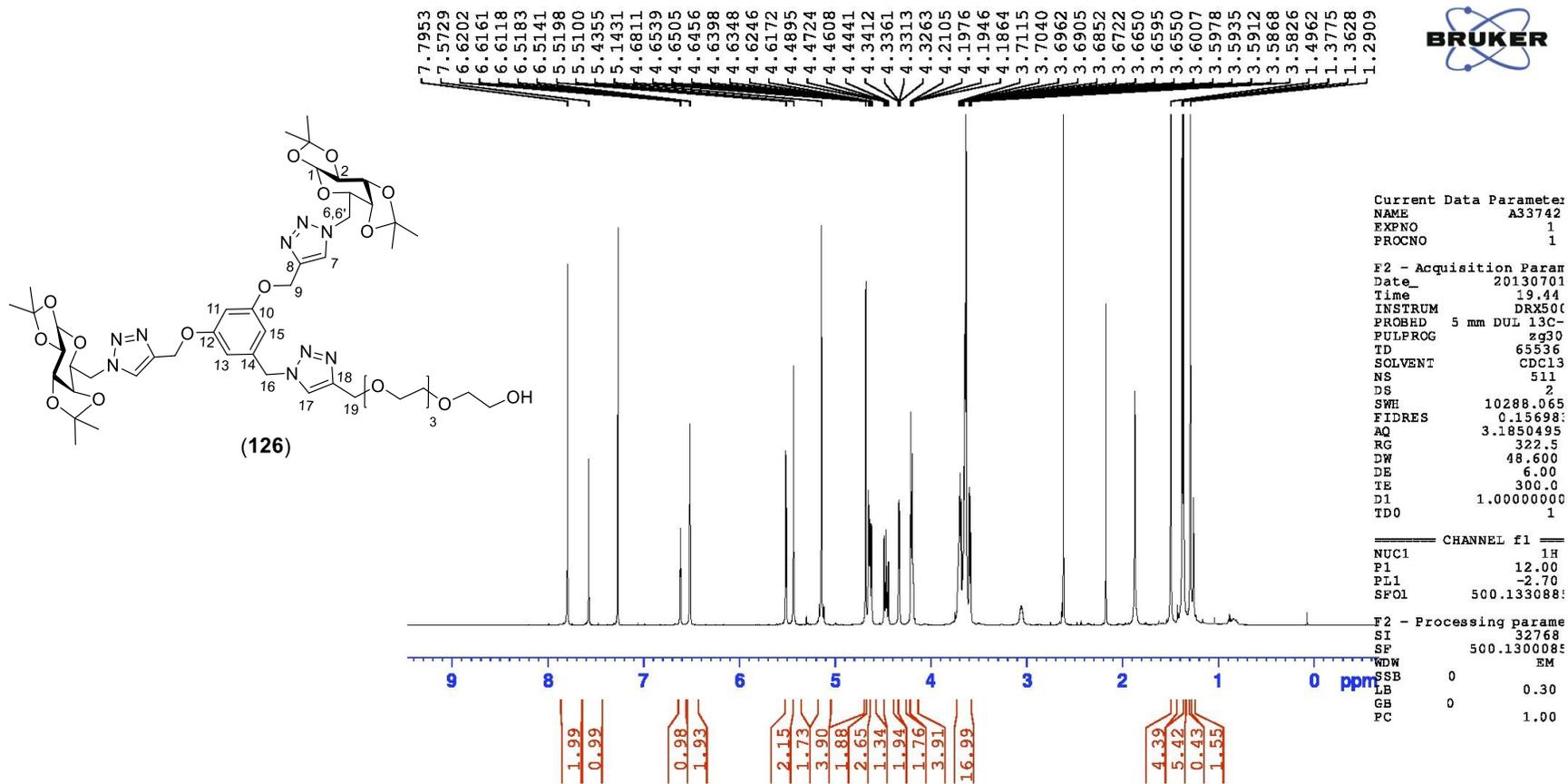
-1.5

Maximum:

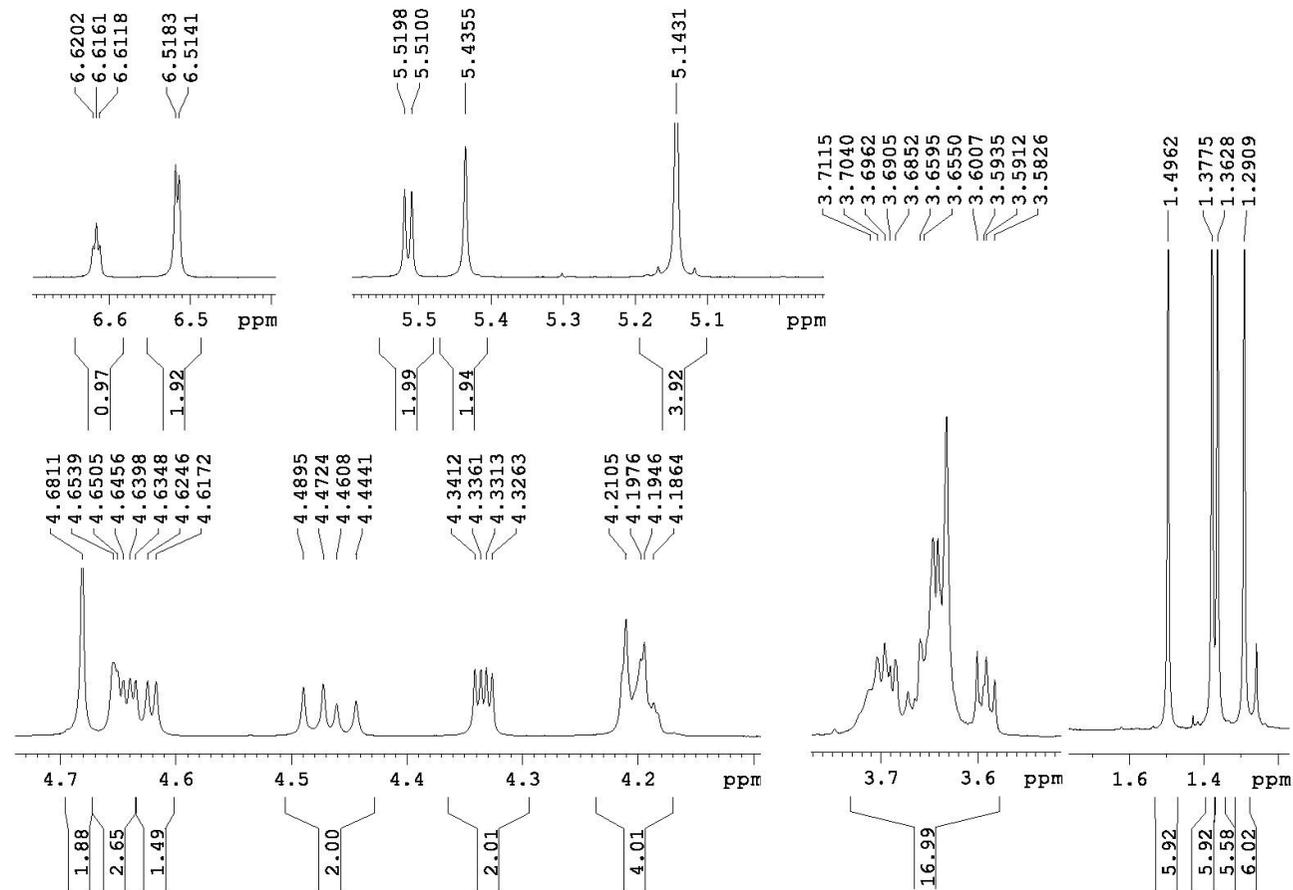
10.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
1066.4741	1066.4709	3.2	3.0	18.5	96.6	0.0	C ₄₈ H ₆₉ N ₉ O ₁₇ Na

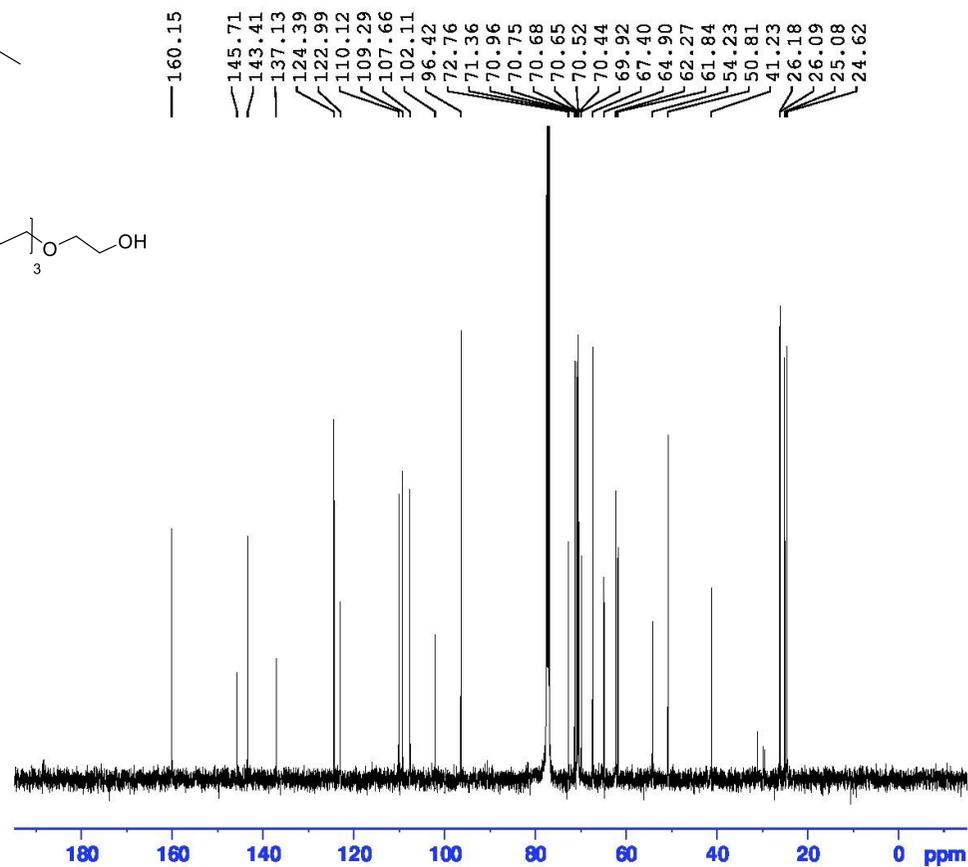
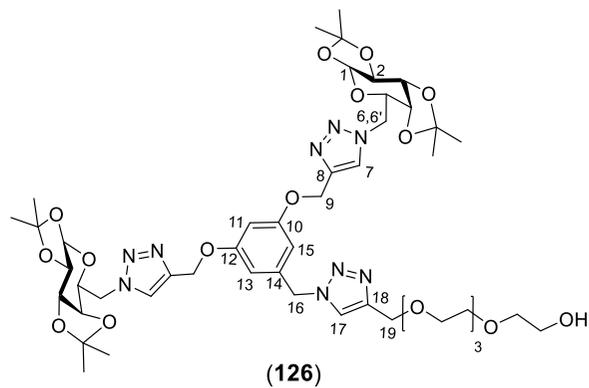
Appendix B89: HRMS (ESI) spectra of compound (126).



Appendix B90: ¹H NMR spectrum of compound (126).



Appendix B91: Selected areas ^1H NMR of compound (126).



Current Data Parameters
NAME A33742
EXPNO 2
PROCNO 1

F2 - Acquisition Parameter
Date_ 20130701
Time 22.05
INSTRUM DRX500
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 16384
SOLVENT CDCl3
NS 8192
DS 4
SWH 30120.482 Hz
FIDRES 1.838408 Hz
AQ 0.2719744 sec
RG 11585.2
DW 16.600 usec
DE 6.00 usec
TE 300.0 K
D1 0.69999999 sec
d11 0.03000000 sec
DELTA 0.59999996 sec
TD0 1

CHANNEL f1
NUC1 13C
P1 8.00 usec
PL1 -2.30 dB
SFO1 125.7703643 MHz

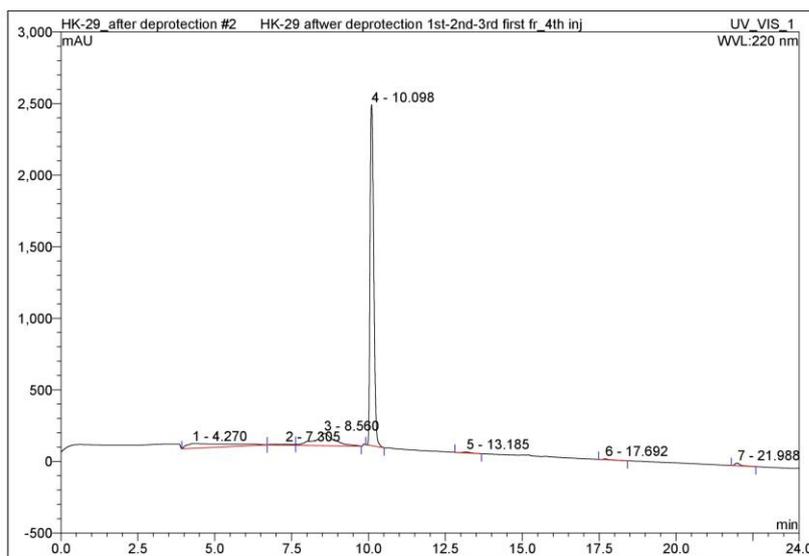
CHANNEL f2
CPDPRG[2] waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.30 dB
PL12 12.84 dB
PL13 120.00 dB
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577637 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

Appendix B92: ^{13}C NMR spectrum of compound (126).

2 HK-29 aftwer deprotection 1st-2nd-3rd first fr_4th inj

Sample Name:	HK-29 aftwer deprotection 1st-2nd-3rd first fr_4th inj	Injection Volume:	20.0
Vial Number:	RA2	Channel:	UV_VIS_1
Sample Type:	unknown	Wavelength:	220
Control Program:	poly-p4 28min +230nm	Bandwidth:	10
Quantif. Method:	dna method	Dilution Factor:	1.0000
Recording Time:	3/7/2013 17:32	Sample Weight:	1.0000
Run Time (min):	24.00	Sample Amount:	1.0000



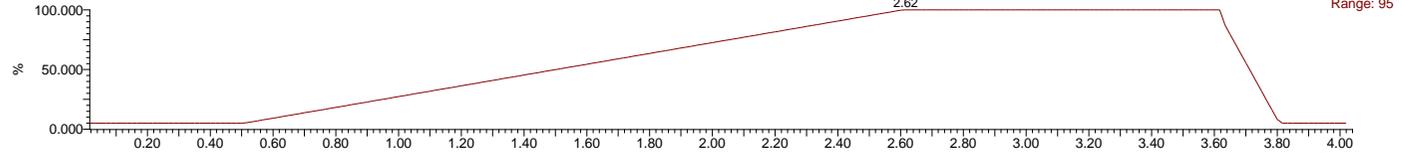
No.	Ret. Time min	Peak Name	Height mAU	Area mAU*min	Rel. Area %	Amount	Type
1	4.27	n.a.	32.958	49.937	10.92	n.a.	BMB
2	7.31	n.a.	6.678	5.158	1.13	n.a.	bM
3	8.56	n.a.	85.808	63.372	13.86	n.a.	MB
4	10.10	n.a.	2382.633	332.268	72.66	n.a.	BMB
5	13.19	n.a.	6.096	1.637	0.36	n.a.	BMB
6	17.69	n.a.	6.034	1.129	0.25	n.a.	BMB
7	21.99	n.a.	18.242	3.809	0.83	n.a.	BMB
Total:			2538.449	457.310	100.00	0.000	

default/Integration

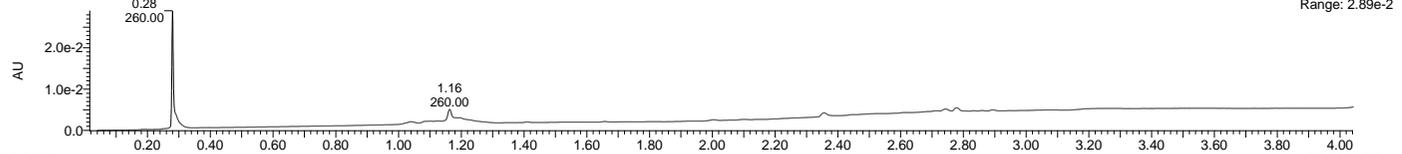
Chromeleon (c) Dionex 1996-2006
Version 6.80 SP4 Build 2361 (130805)*Appendix B93: HPL chromatogram of compound (26).*

AJF_HK_29_13 after deprotection

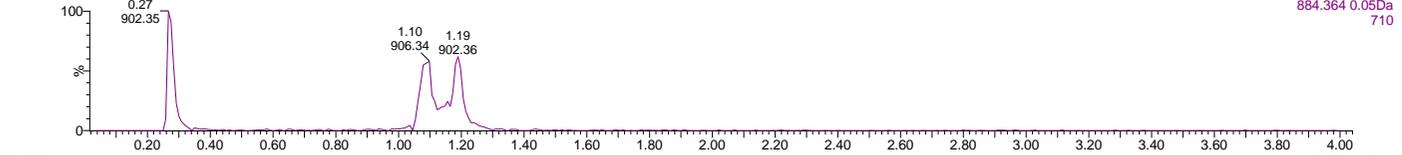
ML2338



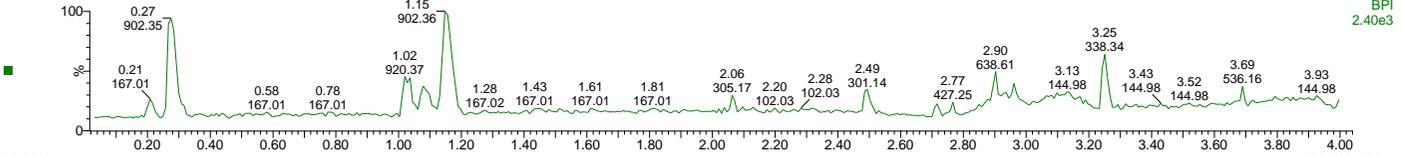
ML2338



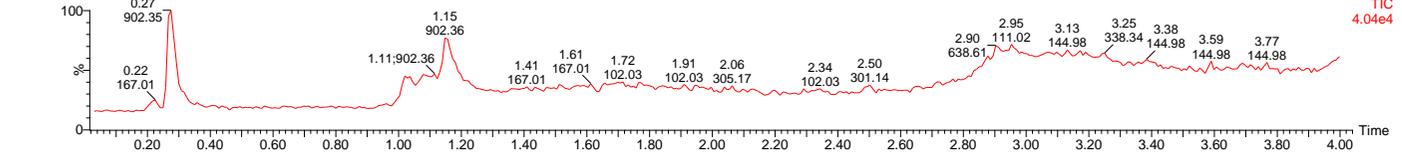
ML2338



ML2338



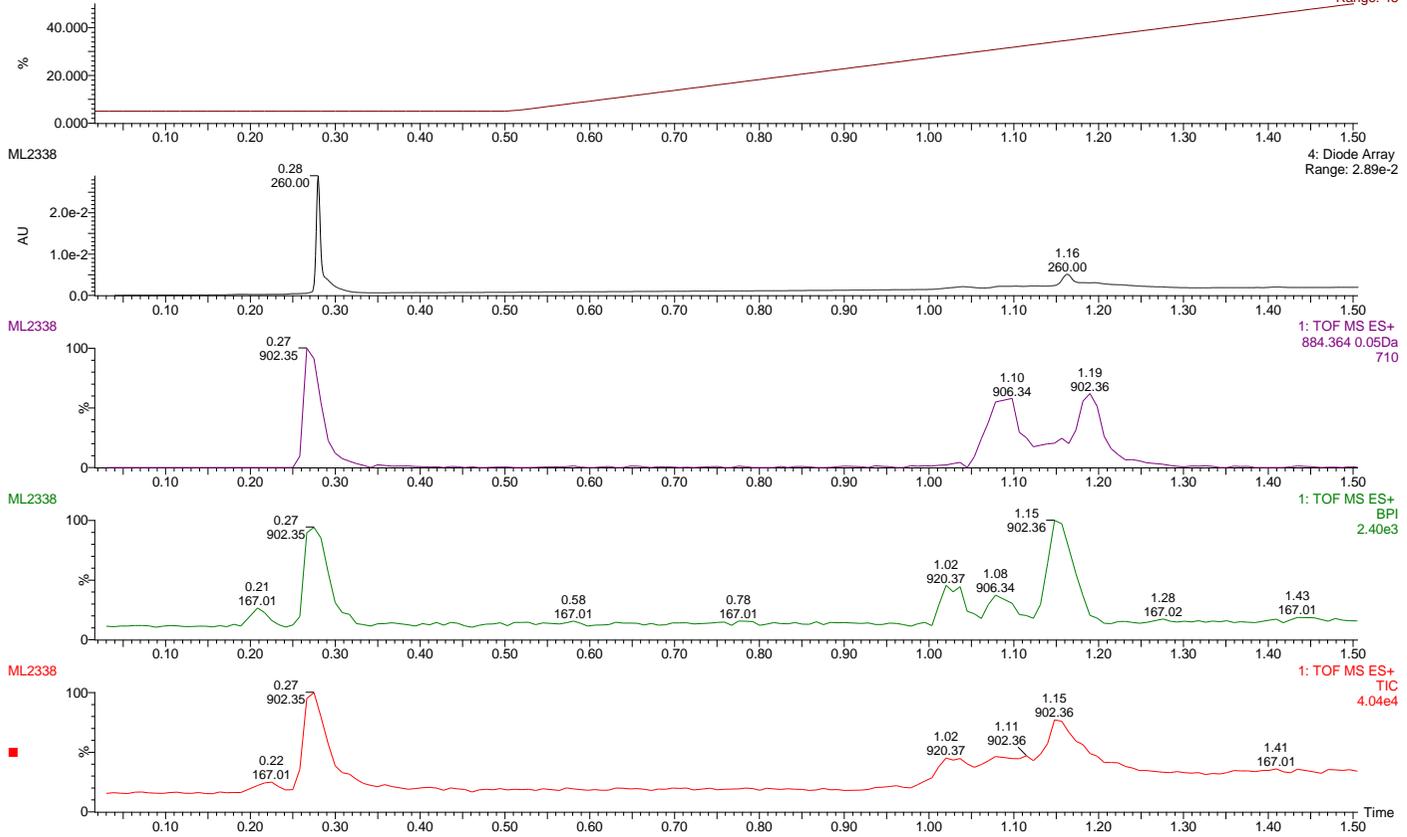
ML2338



AJF_HK_29_13 after deprotection

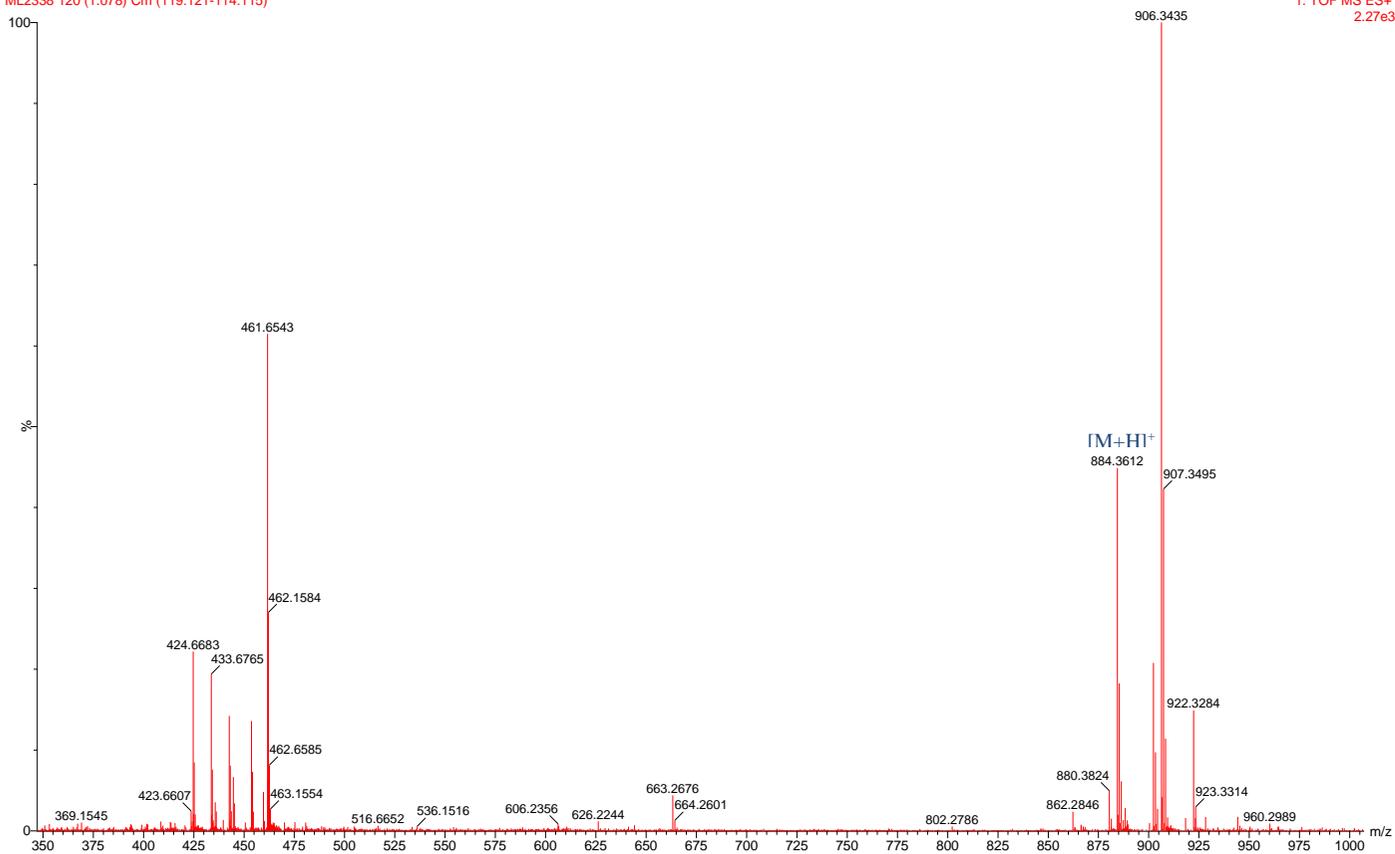
ML2338

BSM Composition B
Range: 45



AJF_HK_29_13 after deprotection
ML2338 120 (1.078) Cm (119:121-114:115)

1: TOF MS ES+
2.27e3



Elemental Composition Report: **AJF-HK-29-13 after deprotection**

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

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

Elements Used:

C: 36-36 H: 0-150 N: 0-30 O: 0-30

Minimum:

-1.5

Maximum:

10.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
884.3612	884.3638	-2.6	-2.9	14.5	38.7	0.0	C36 H54 N9 O17

Elemental Composition Report: **AJF-HK-29-13 after deprotection**

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

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

Elements Used:

C: 36-36 H: 0-150 N: 0-30 O: 0-30 Na: 1-1

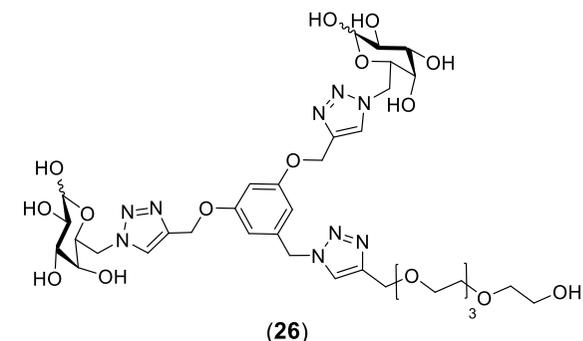
Minimum:

-1.5

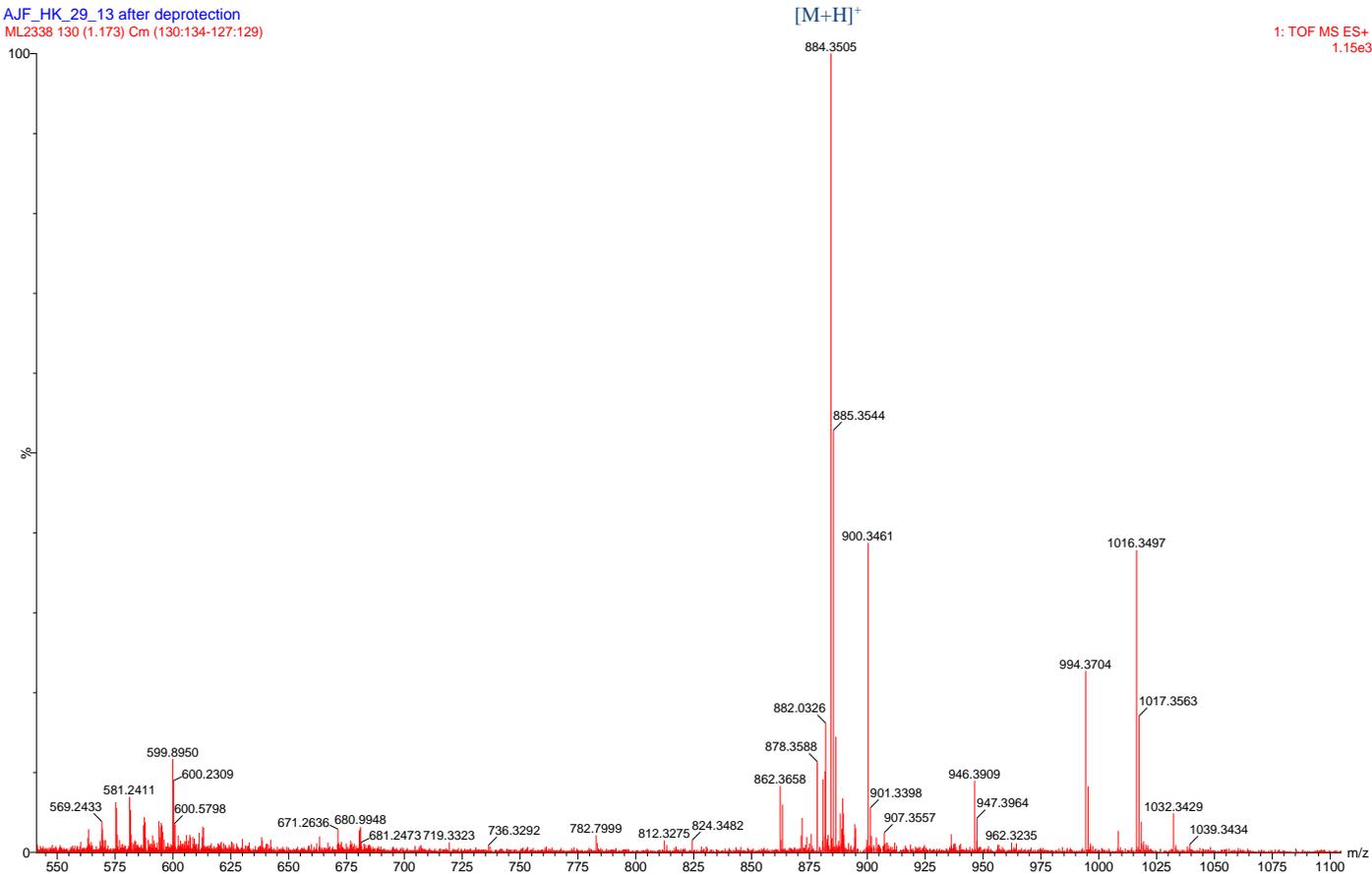
Maximum:

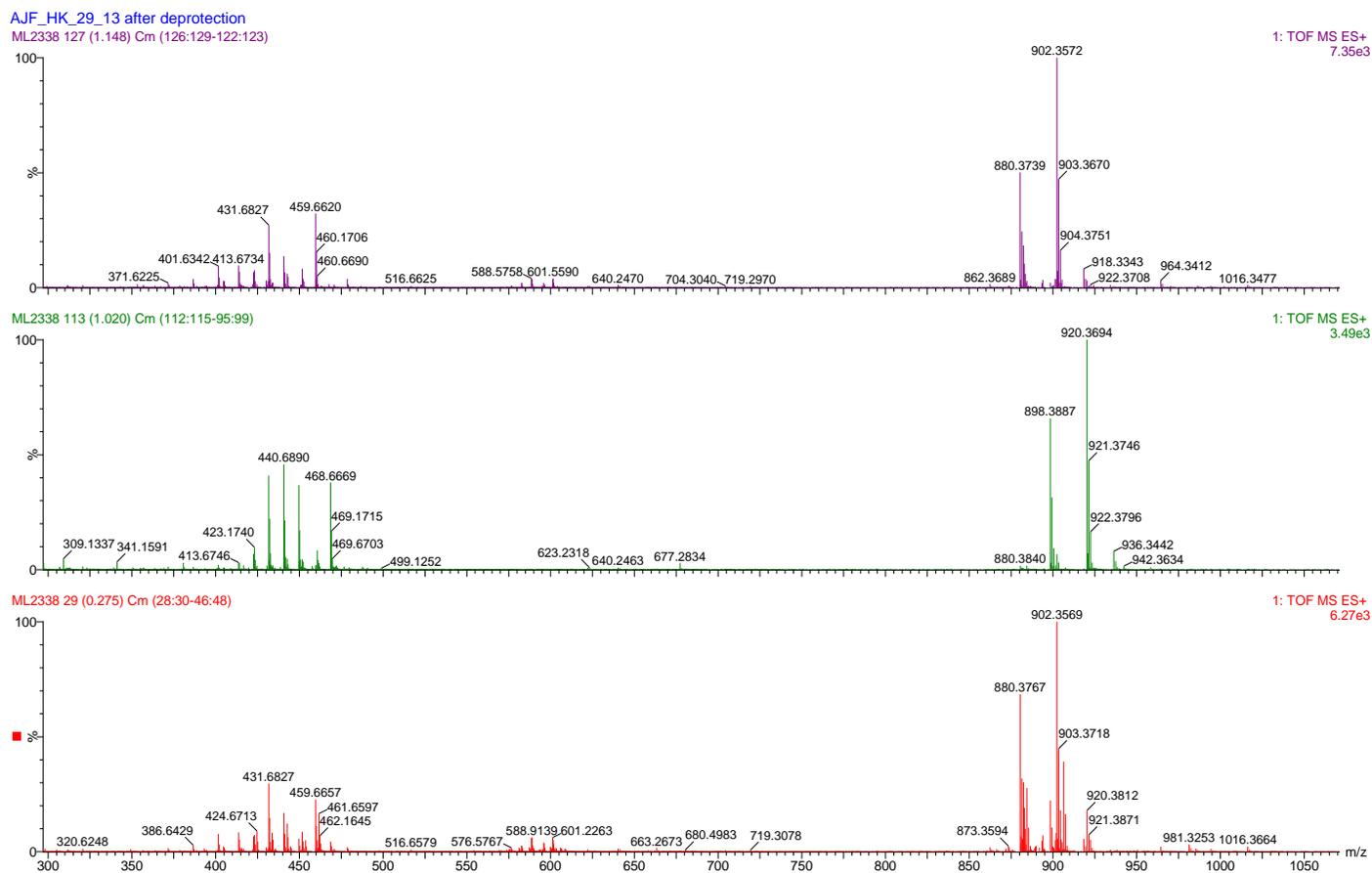
10.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
906.3435	906.3457	-2.2	-2.4	14.5	36.6	0.0	C36 H53 N9 O17 Na

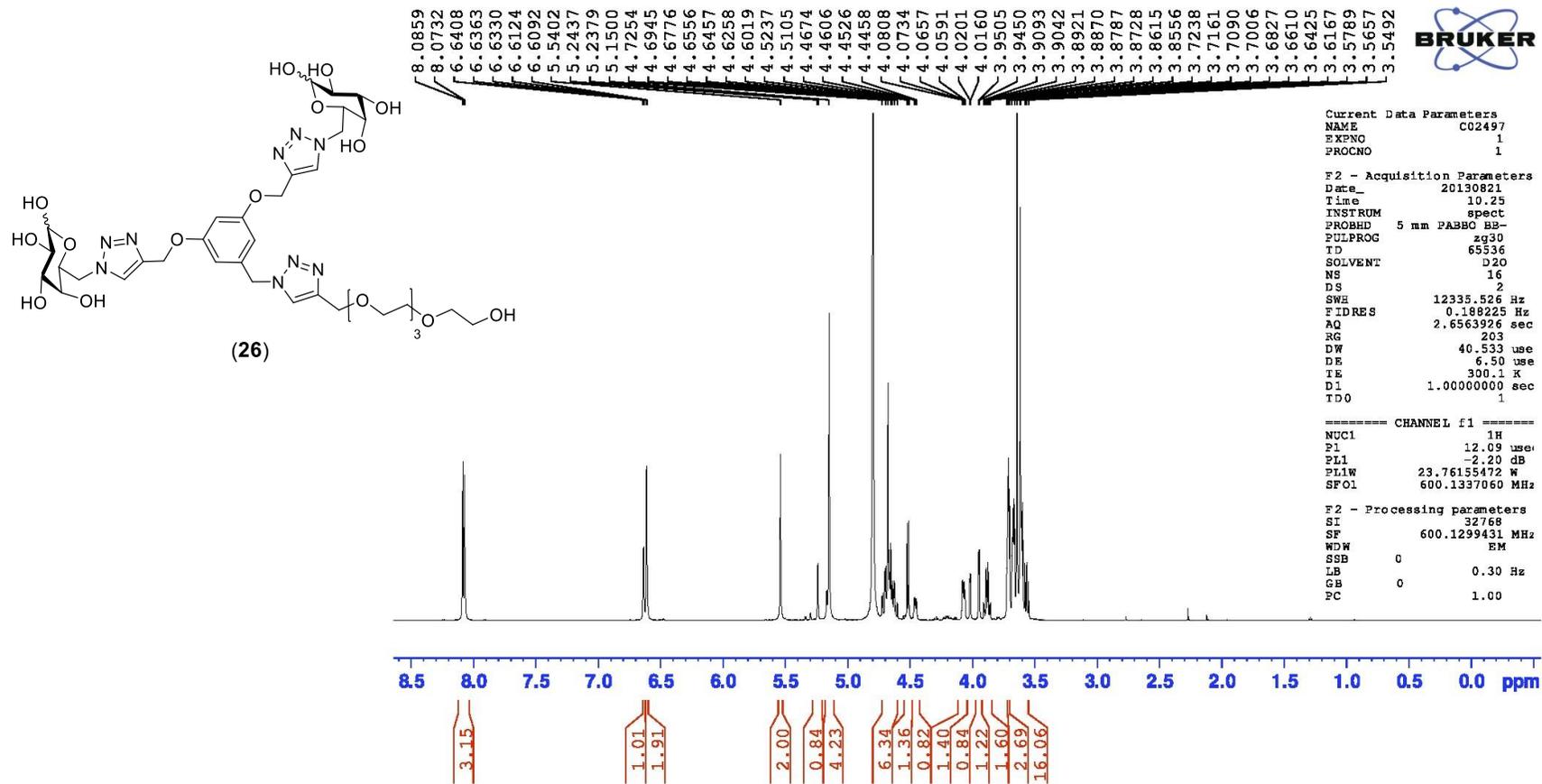


AJF_HK_29_13 after deprotection
ML2338 130 (1.173) Cm (130:134-127:129)

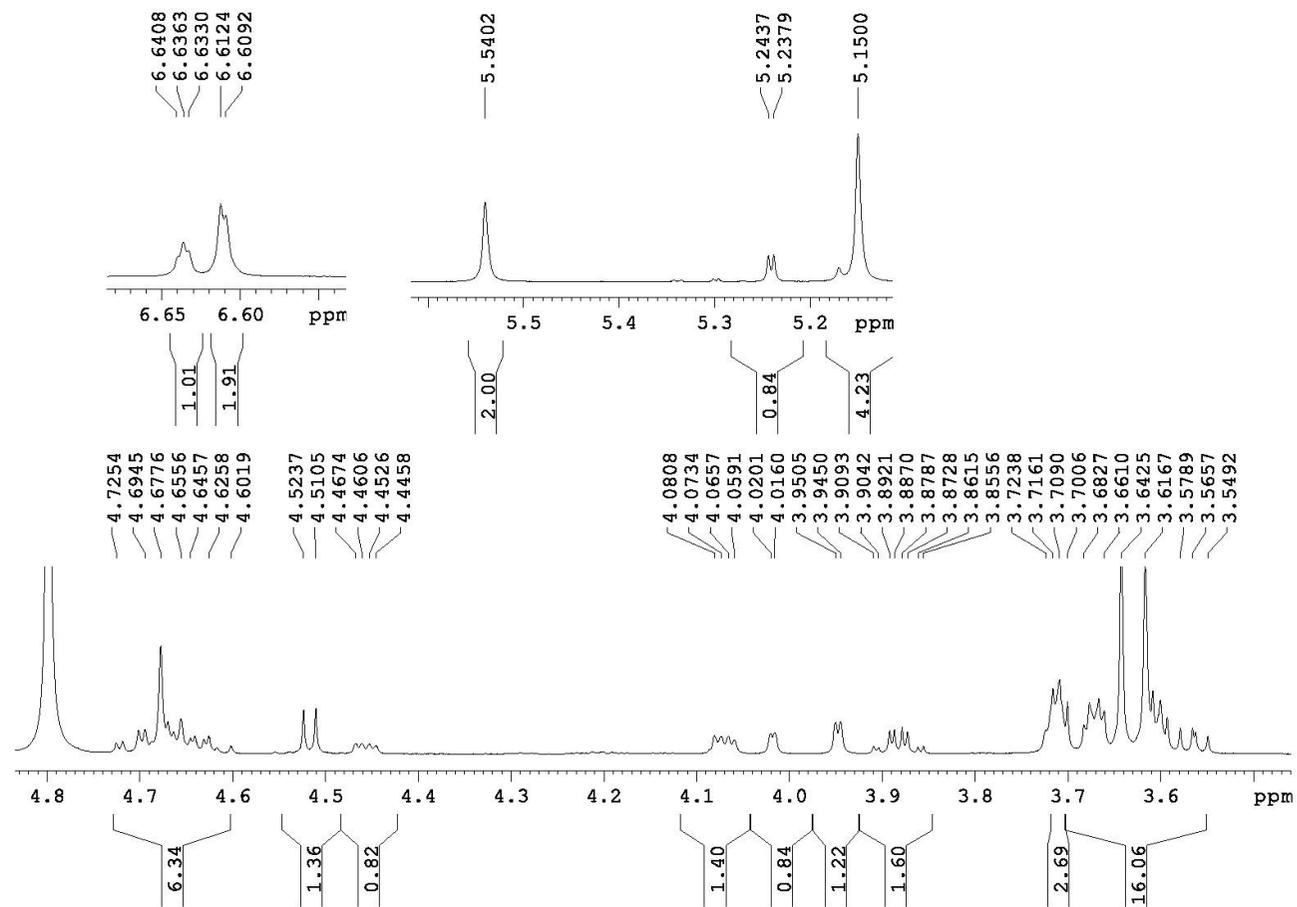




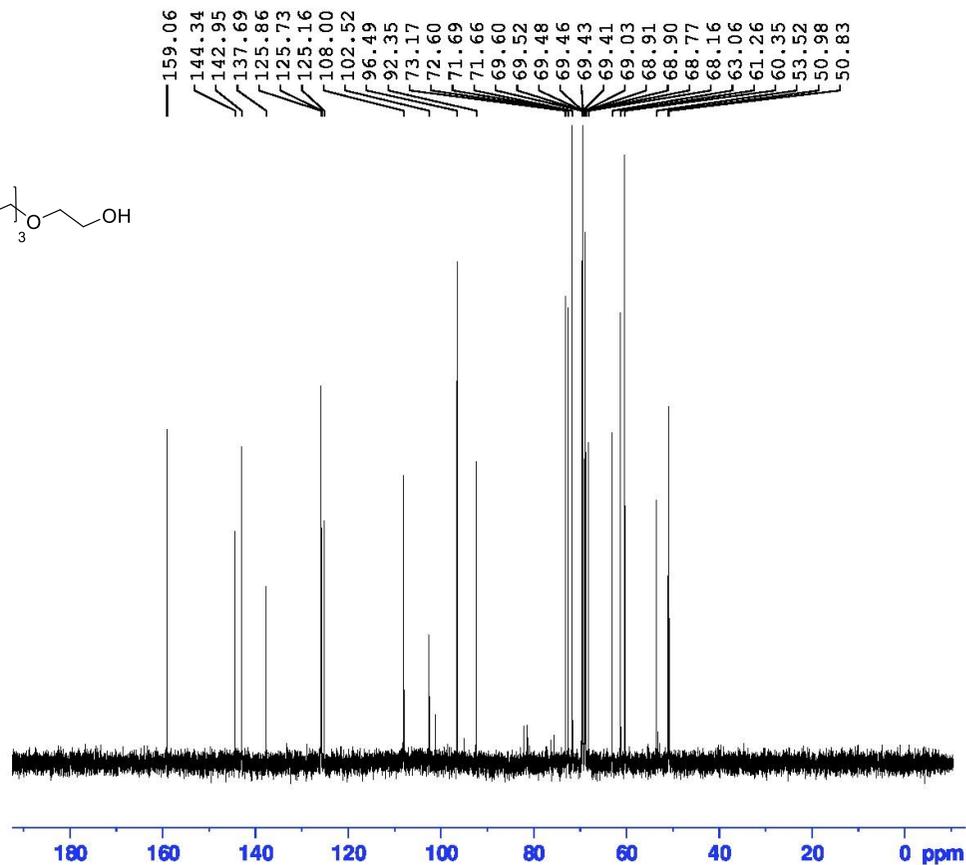
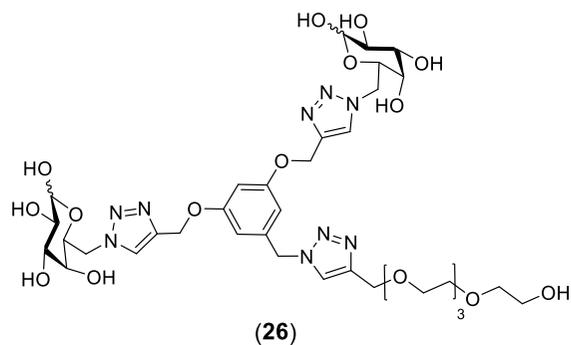
Appendix B94: HRMS (ESI) spectra of compound (26).



Appendix B95: ¹H NMR spectrum of compound (26).



Appendix B96: Selected areas ^1H NMR of compound (26).



```

Current Data Parameters
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EXPNO         3
PROCNO        1

F2 - Acquisition Parameters
Date_         20130821
Time          10.39
INSTRUM       spect
PROSHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            32768
SOLVENT       D2O
NS            8192
DS            4
SWH           33333.332 Hz
FIDRES        1.017253 Hz
AQ            0.4915200 sec
RG            2050
DW            15.000 usec
DE            8.25 usec
TE            300.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TDC           1

----- CHANNEL f1 -----
NUC1          13C
P1            8.00 usec
PL1           0 dB
PL1W          90.17671967 W
SFO1          150.9178993 MHz

----- CHANNEL f2 -----
CPDPRG [2]   waltz16
NUC2          1H
PCPD2         69.05 usec
PL2           -2.20 dB
PL12          13.00 dB
PL13          14.00 dB
PL2W          23.76155472 W
PL12W         0.71758747 W
PL13W         0.56999999 W
SFO2          600.1324005 MHz

F2 - Processing parameters
SI            32768
SF            150.9028090 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40

```

Appendix B97: ^{13}C NMR spectrum of compound (26).

H 14

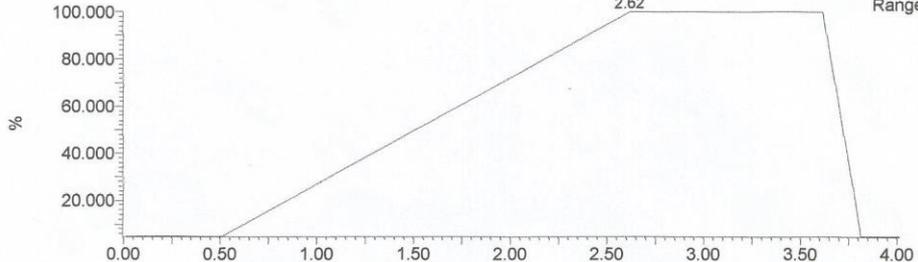
6.

10-Dec-2010

11:53:35

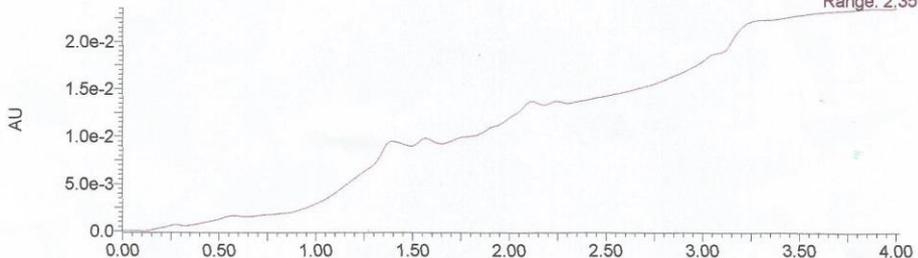
GE HK 9

BSM Composition B
Range: 95



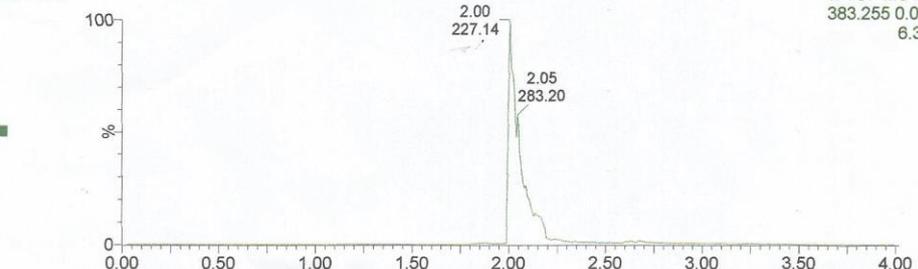
GE HK 9

4: Diode Array
Range: 2.359e-2



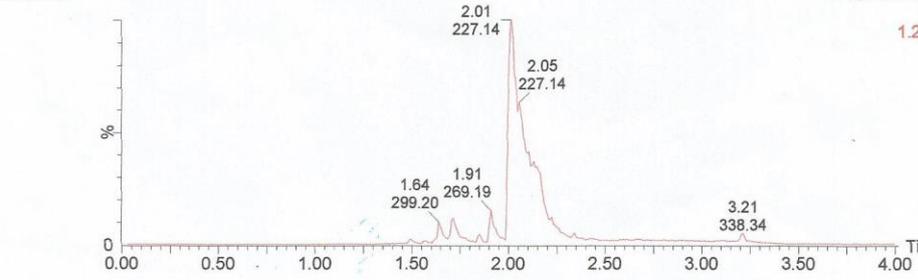
GE HK 9

1: TOF MS ES+
383.255 0.05Da
6.35e4



GE HK 9

1: TOF MS ES+
BPI
1.25e5



Elemental Composition Report

Page 1

Single Mass Analysis

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

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

1117 formula(e) evaluated with 2 results within limits (all results (up to 1000) for each mass)

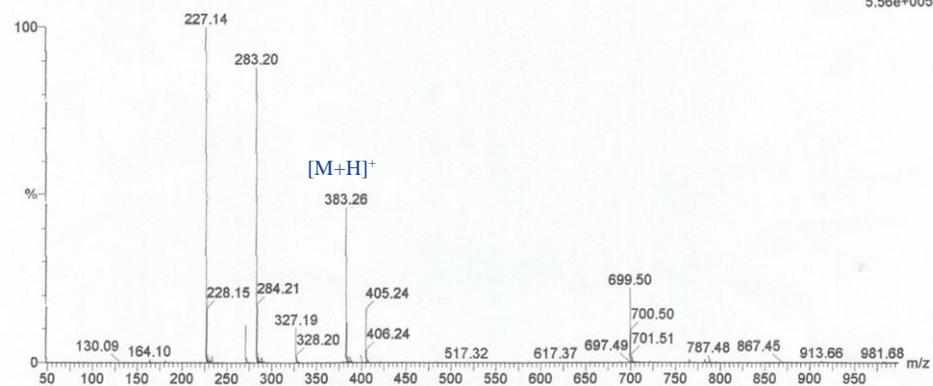
Elements Used:

C: 0-200 H: 0-200 N: 0-30 O: 0-20

H 14

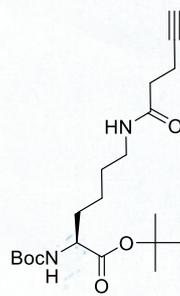
GE HK 9 224 (2.013) Cm (222:227-(198:212+261:280))

1: TOF MS ES+
5.56e+005



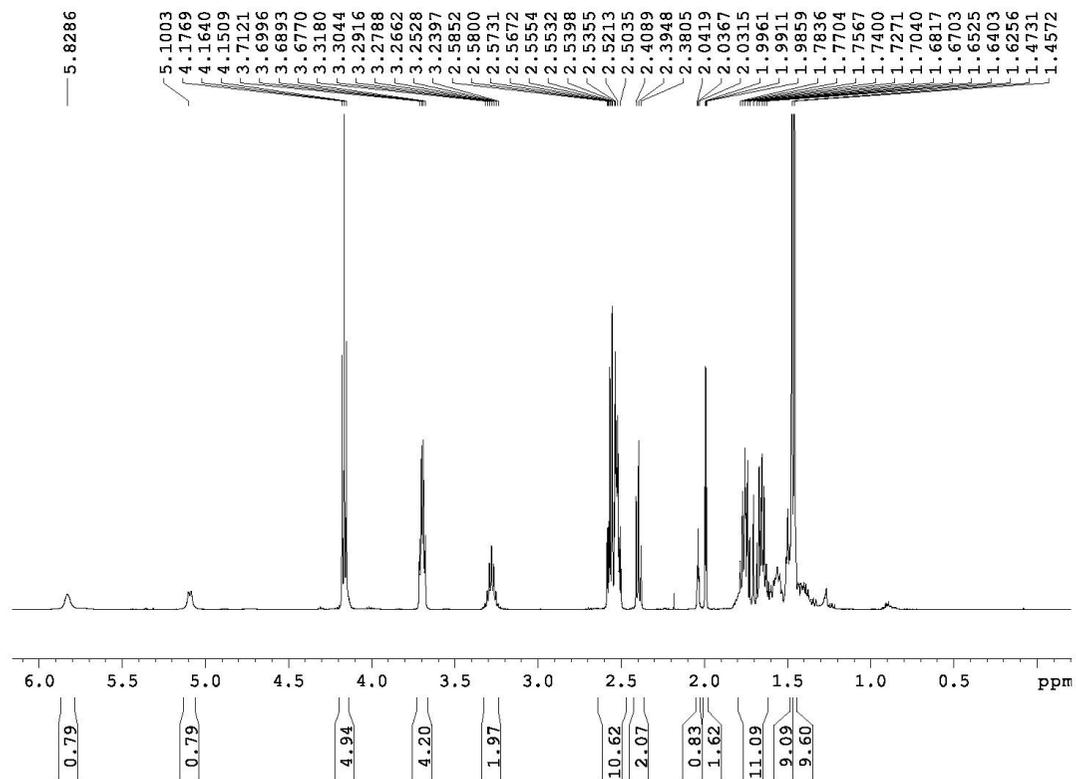
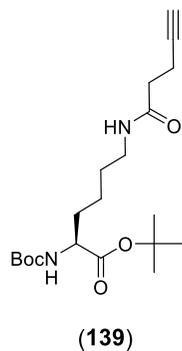
Minimum: -1.5
Maximum: 1000.0 3.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
383.2552	383.2559	-0.7	-1.8	9.5	340.0	0.3	C21 H31 N6 O
	383.2546	0.6	1.6	4.5	341.2	1.4	C20 H35 N2 O5



(139)

Appendix B98: HRMS (ESI) spectra of compound (139).



```

Current Data Parameters
NAME      hak14
EXPNO     1
PROCNO    1

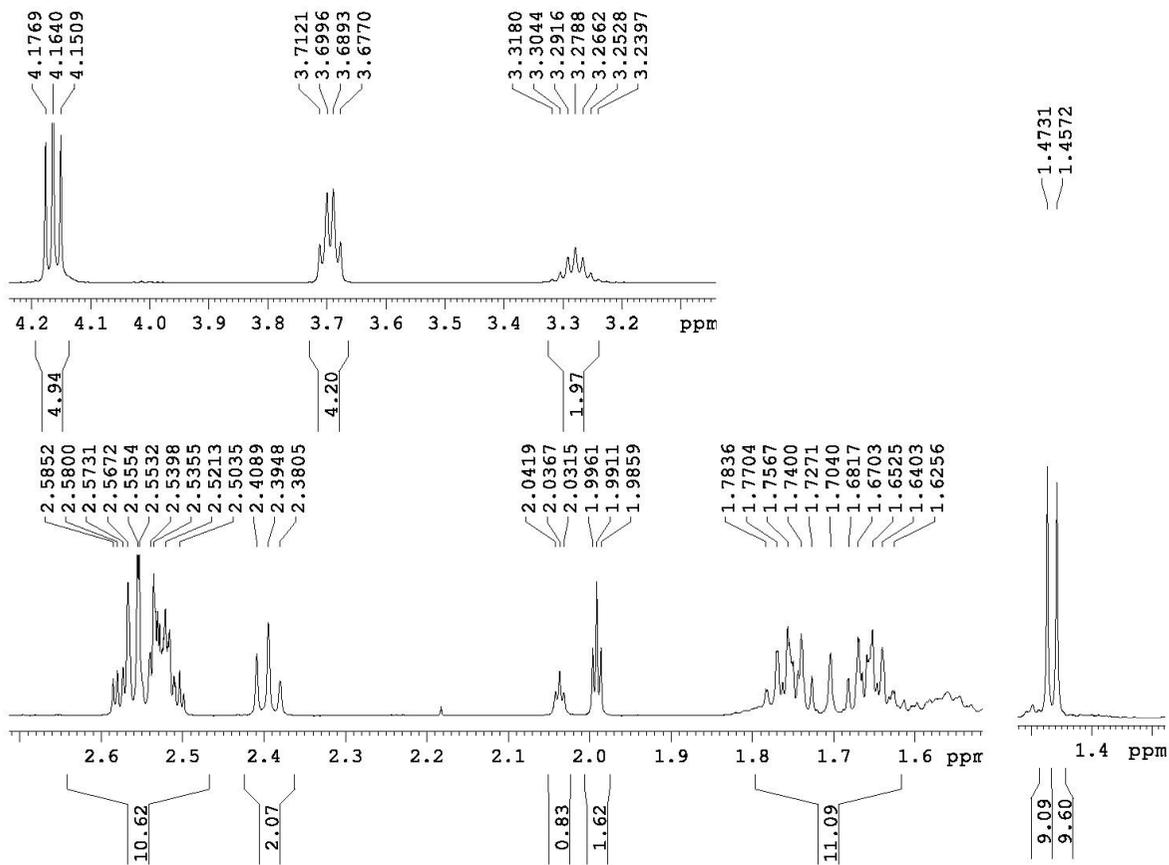
F2 - Acquisition Parameter
Date_     20110310
Time      11.25
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg
TD         32768
SOLVENT   CDCl3
NS         16
DS         2
SWH       4251.701 Hz
FIDRES    0.129752 Hz
AQ         3.8535168 sec
RG         114
DW         117.600 usec
DE         6.50 usec
TE         298.0 K
D1         8.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1      1H
P1         10.25 usec
PL1        0 dB
PL1W       22.85816574 W
SFO1       500.1318755 MHz

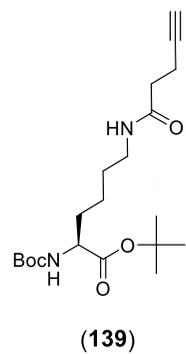
F2 - Processing parameters
SI         32768
SF         500.1300031 MHz
WDW        EM
SSB        0
LB         0.20 Hz
GB         0
PC         1.00

```

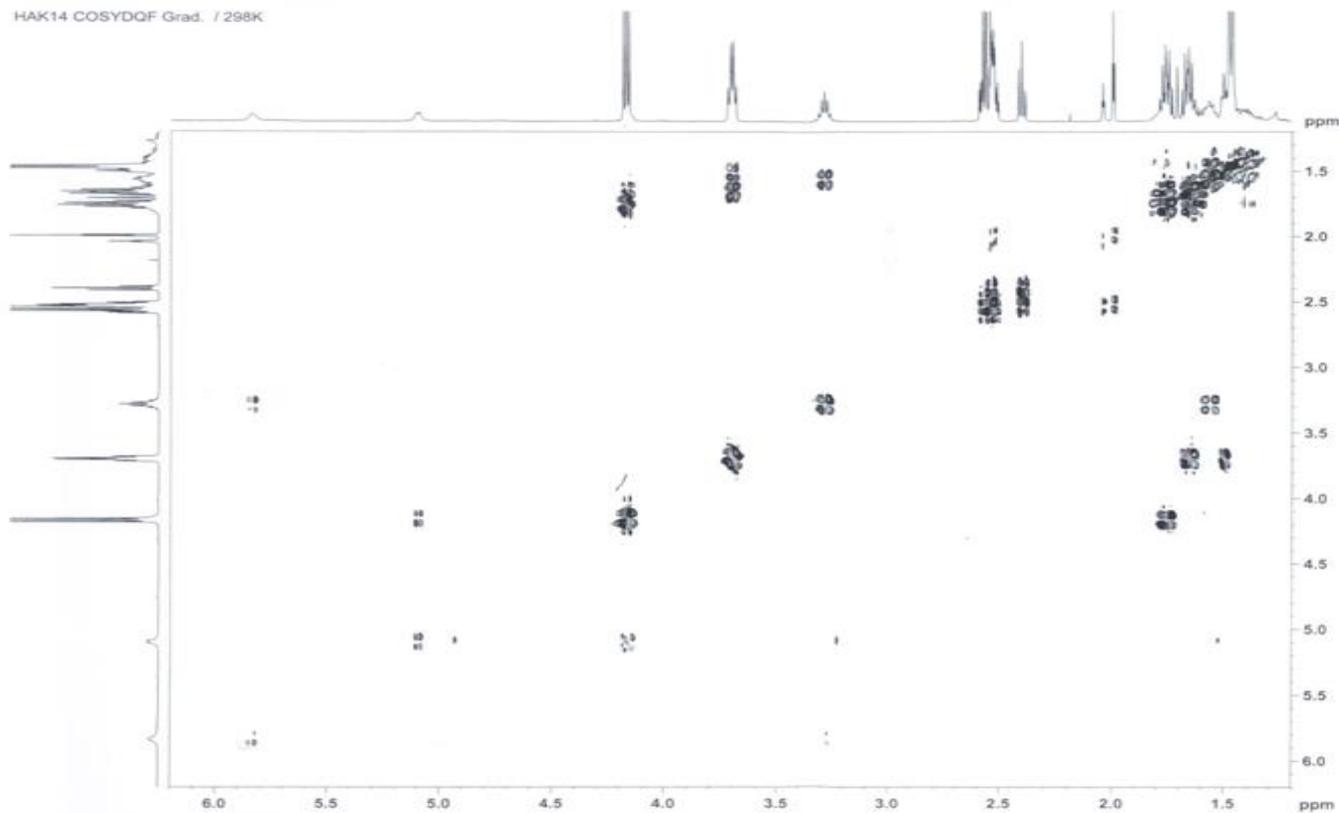
Appendix B99: ¹H NMR spectrum of compound (139).



Appendix B100: Selected areas ¹H NMR of compound (139).



HAK14 COSYDQF Grad. / 298K

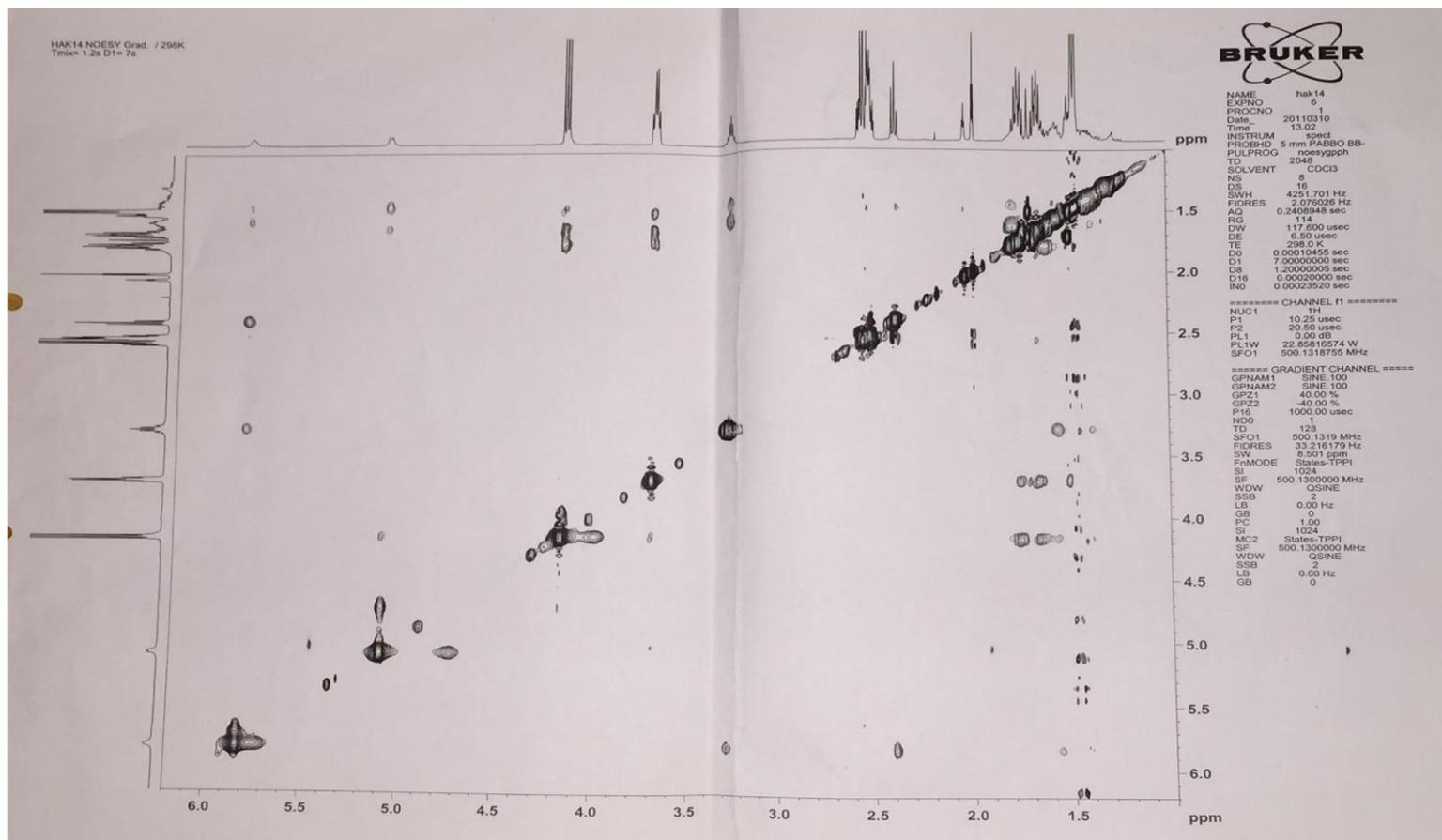
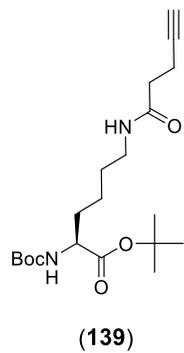


```

NAME          hsk14
EXPNO         4
PROCNO       1
Date_         20110310
Time         12:29
INSTRUM      spect
PROBHD       5 mm PABBO BB-
PULPROG      zgpg30
TD           65536
SOLVENT      CDCl3
NS           16
DS           16
SWH          4251.701 Hz
FIDRES       2.076028 Hz
AQ           0.2400000 sec
RG           114
DQ           117.000 usec
DE           6.50 usec
TE           298.2 K
TD1          0.00010000 sec
D1           7.00000000 sec
D11          0.00000000 sec
D15          0.00020000 sec
RG           0.00020000 sec
===== CHANNEL f1 =====
NUC1         1H
P1           12.00 usec
P2           20.00 usec
PL1          0.00 dB
PL2          0.00 dB
PLTW        22.85010374 W
SFO1         500.1318755 MHz
===== GRADIENT CHANNEL =====
GRNAM1       SINE 100
GRNAM2       SINE 100
GPR1         10.00 %
GPR2         20.00 %
GHS          1000.00 usec
NDG          1
TD           128
SFO1         500.1318755 MHz
FIDRES       32.218170 Hz
SW           8.501 ppm
HSAOCDE      zgpg30
SI           1024
SF           500.1300000 MHz
WDW          COSYME
SSB          0
LB           0.00 Hz
GB           0
PC           1.00
SI           1024
MC2          Sine-TFPH
SF           500.1300000 MHz
WDW          COSYME
SSB          2
LB           0.00 Hz
GB           0

```

Appendix B102: COSYDQF NMR spectrum of compound (139).



Appendix B 103: NOESY NMR spectrum of compound (139).

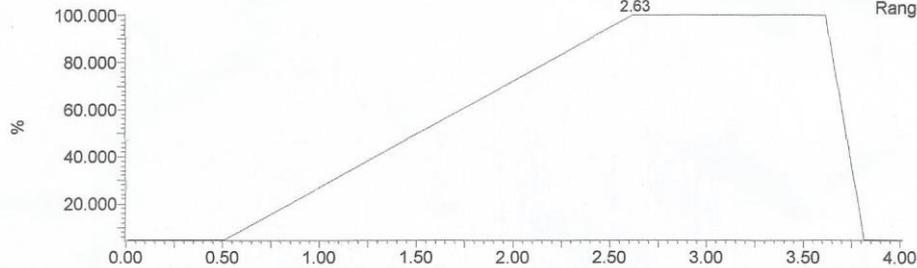
12 after dry

10-Dec-2010

11:40:56

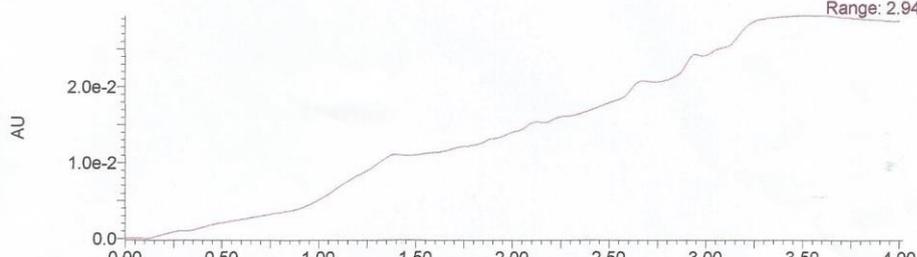
GE HK 8

BSM Composition B
Range: 95



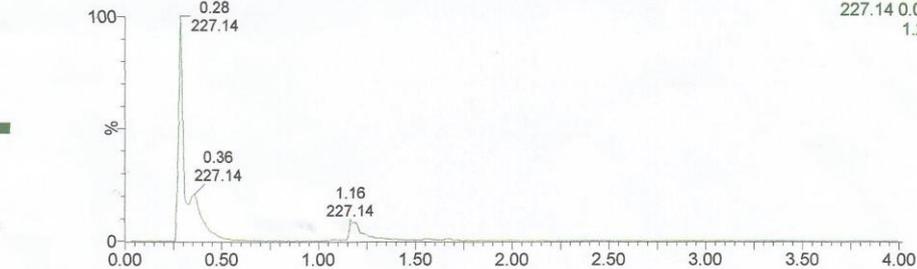
GE HK 8

4: Diode Array
Range: 2.947e-2



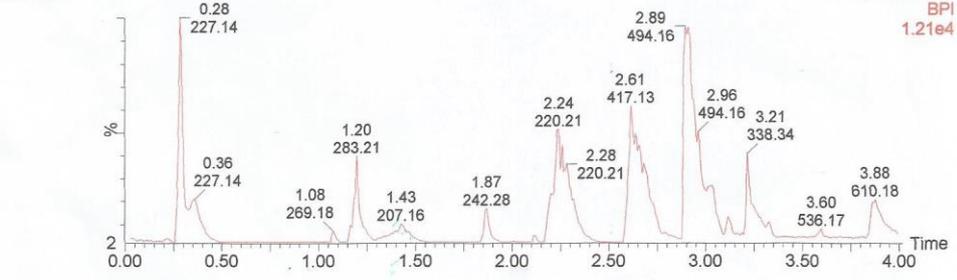
GE HK 8

1: TOF MS ES+
227.14 0.05Da
1.21e4



GE HK 8

1: TOF MS ES+
BPI
1.21e4



Elemental Composition Report

Page 1

Single Mass Analysis

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

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

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

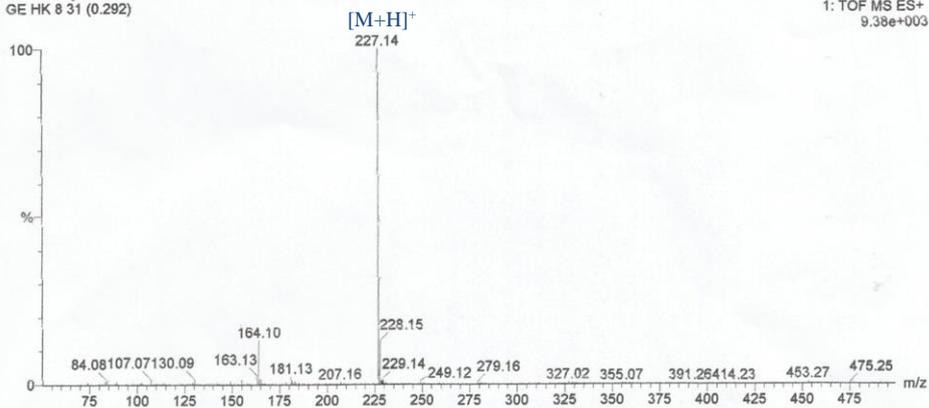
Elements Used:

C: 0-200 H: 0-200 N: 0-30 O: 0-20

12 after dry

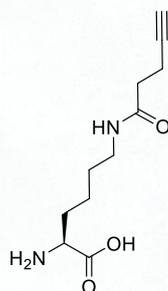
GE HK 8 31 (0.292)

1: TOF MS ES+
9.38e+003



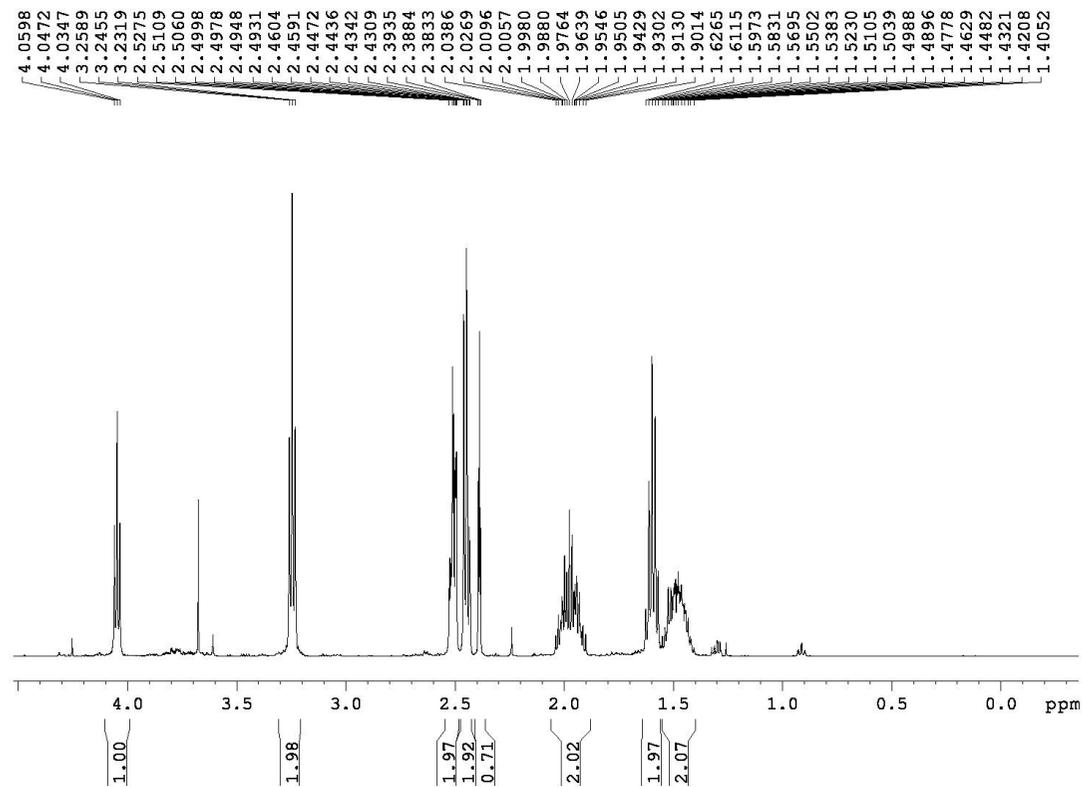
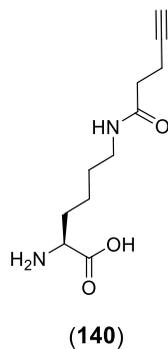
Minimum: -1.5
Maximum: 1000.0 3.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
227.1397	227.1396	0.1	0.4	3.5	126.9	0.0	C11 H19 N2 O3



(140)

Appendix B105: HRMS (ESI) spectra of compound (140).



Current Data Parameters
NAME ha_12
EXPNO 1
PROCNO 1

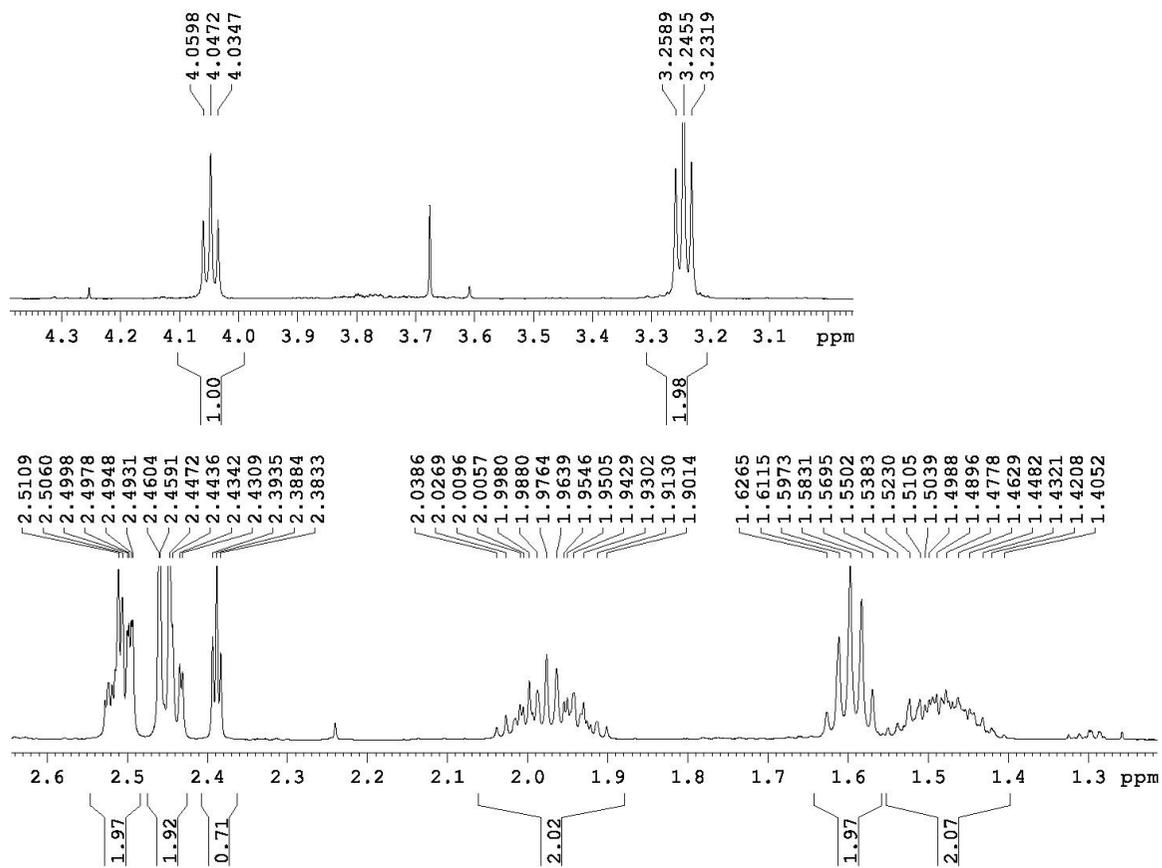
F2 - Acquisition Parameter
Date_ 20110106
Time 10.09
INSTRUM spect
PROBED 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT D2O
NS 16
DS 2
SWH 3501.401 Hz
FIDRES 0.106854 Hz
AQ 4.6792703 sec
RG 128
DW 142.800 usec
DE 6.50 usec
TE 298.0 K
D1 6.00000000 sec
TD0 1

CHANNEL f1

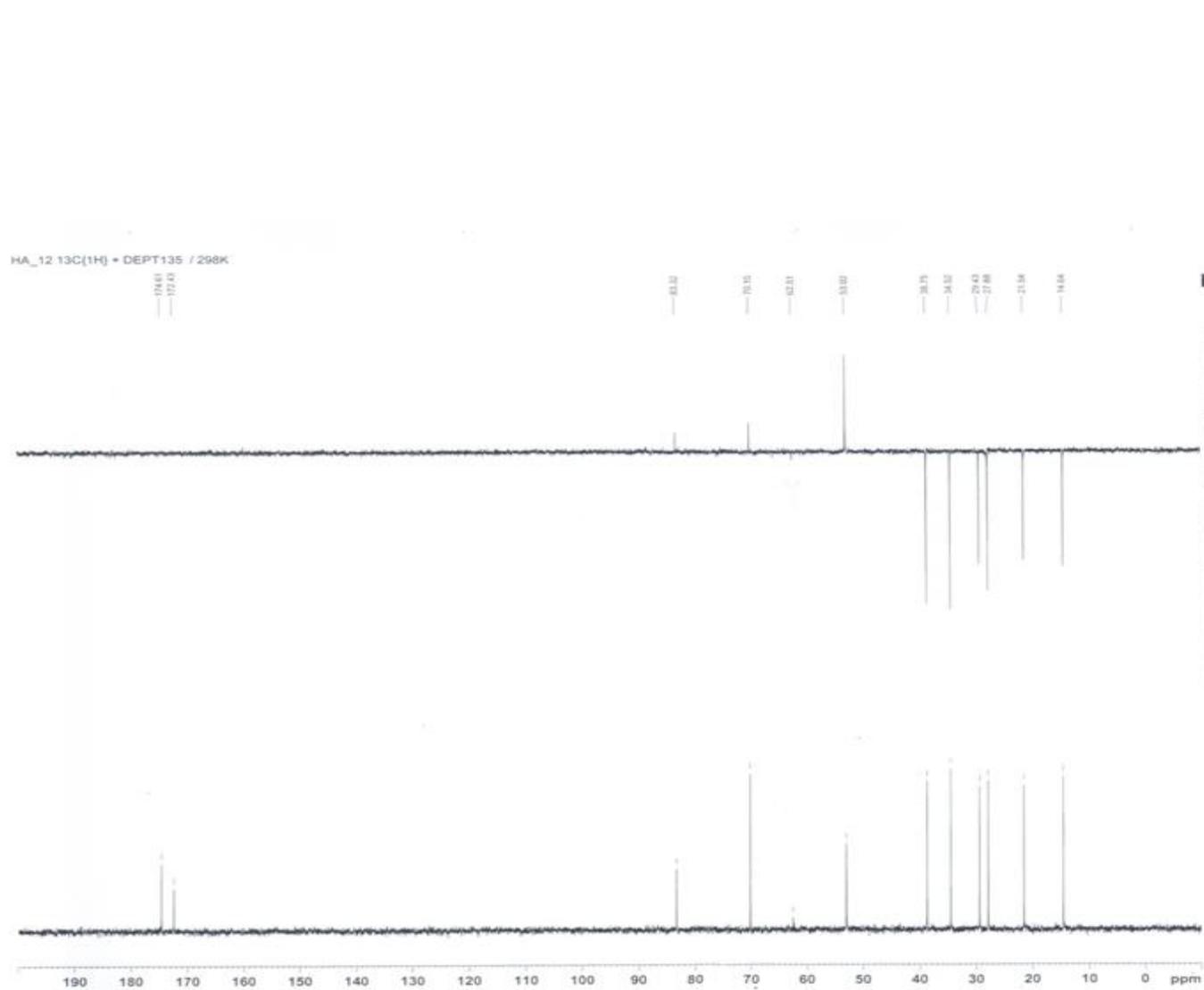
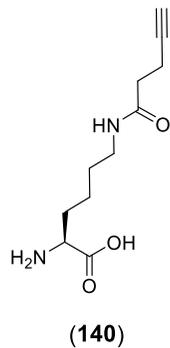
NUC1 1H
P1 10.25 usec
PL1 0 dB
PL1W 22.85816574 W
SFO1 500.1315004 MHz

F2 - Processing parameters
SI 32768
SF 500.1299529 MHz
WDW EM
SSB 0
LB 0.20 Hz
GB 0
PC 1.00

Appendix B106: ¹H NMR spectrum of compound (140).



Appendix B107: Selected areas ^1H NMR of compound (140).



```

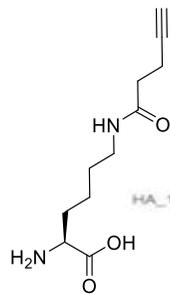
NAME HA_12
EXPNO 2
PROCNO 1
Date_ 20110108
Time 10.14
INSTRUM spect
PROBHD 5 mm PABBO BB
PULPROG zgpg30
TD 32768
SOLVENT DMS
NS 1000
DS 2
SWH 32064.738 Hz
FIDRES 1.003668 Hz
AQ 0.498728 sec
RG 2050
CW 15.250 umsec
DE 6.50 umsec
TE 300.2 K
DT 1.90000000 sec
D15 0.03000000 sec
TD0 1

***** CHANNEL f1 *****
NUC1 13C
PT 8.50 umsec
PL1 1.50 dB
PL1W 58.36315375 W
SFO1 125.7718224 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 umsec
PL2 0.00 dB
PL2W 17.00 dB
PL3 17.00 dB
PL3W 17.00 dB
PL3W 22.85816074 W
PL3W 0.45606038 W
PL3W 0.45606038 W
SFO2 500.1370650 MHz
SI 32768
SF 125.7677590 MHz
WOW 5M
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

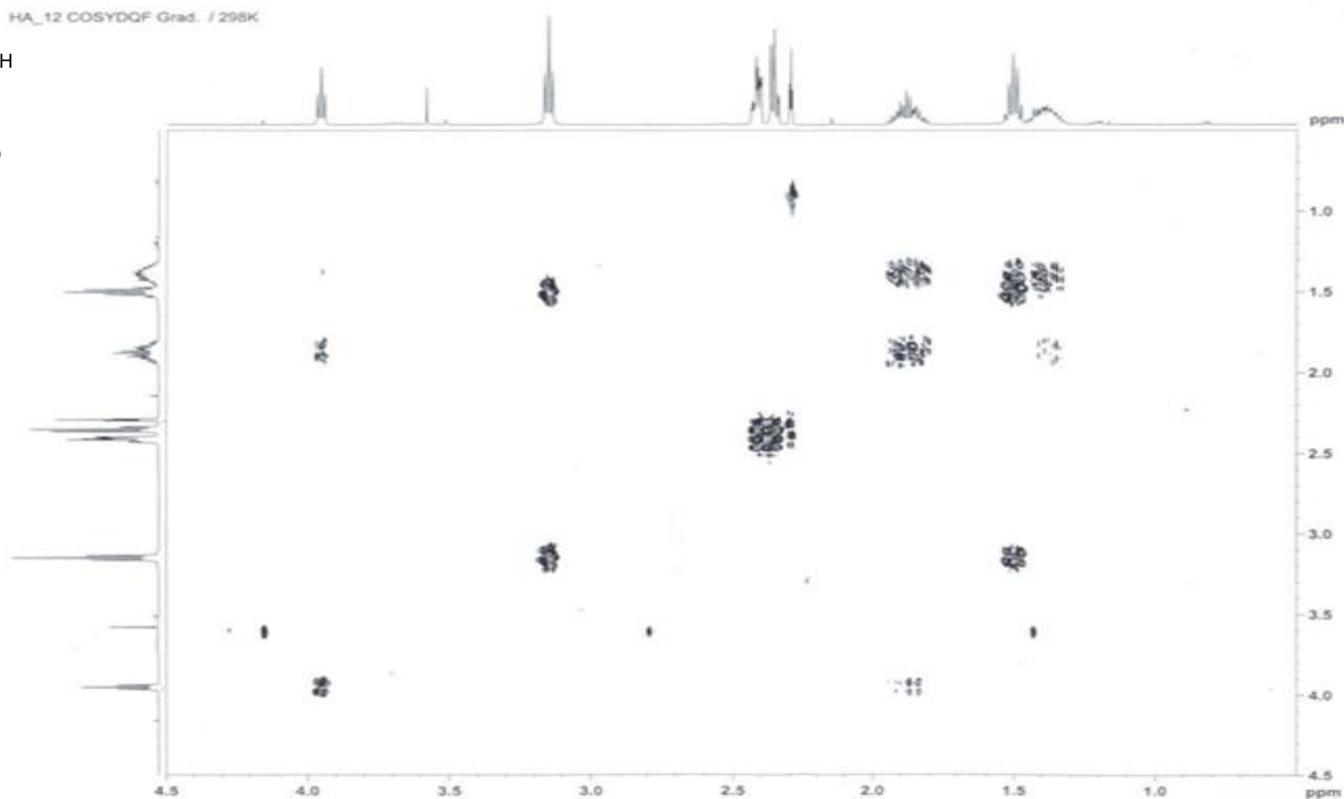
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Appendix B108: ¹³C NMR spectrum of compound (140).



(140)

HA_12 COSYDQF Grad. / 298K

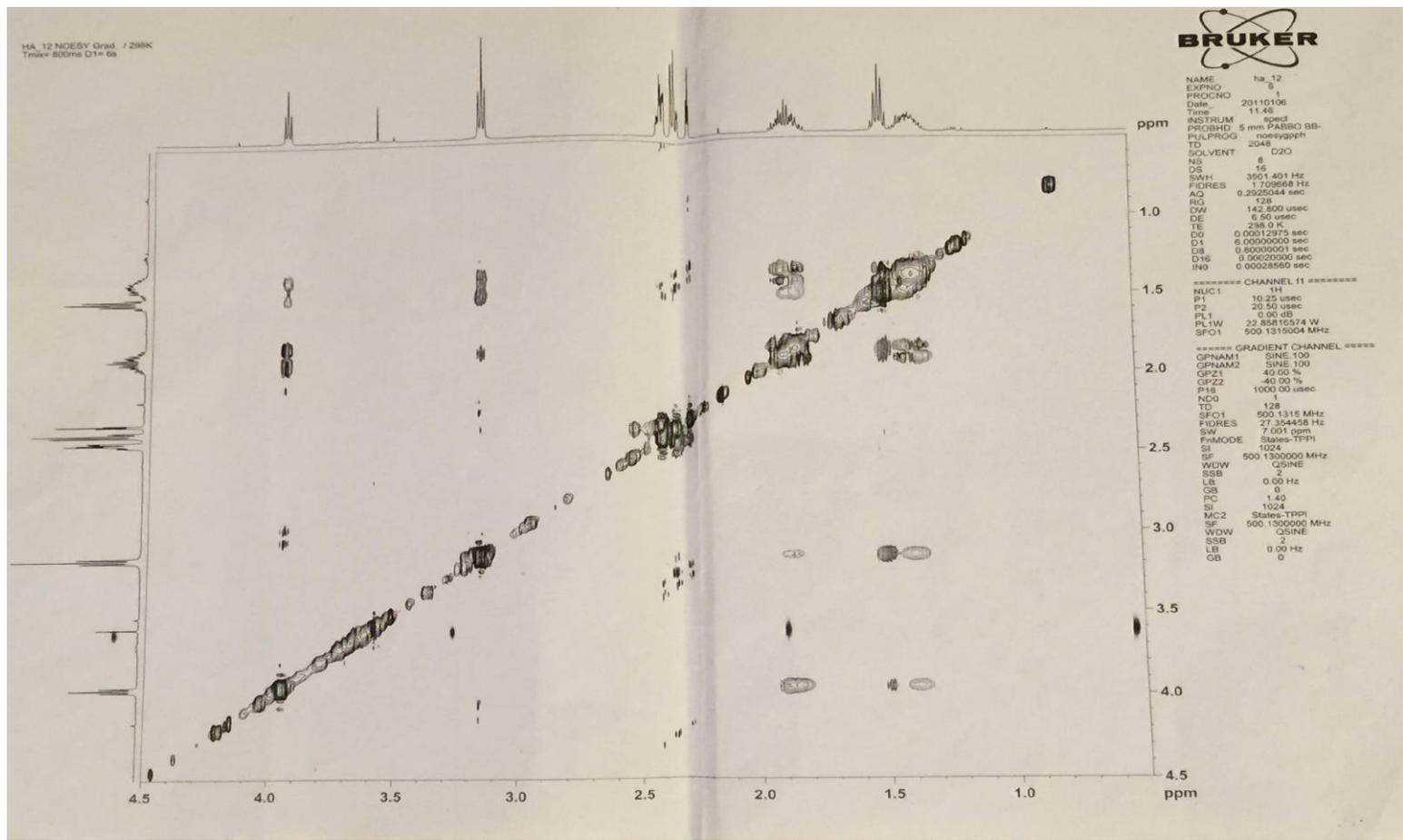
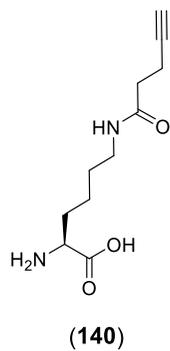


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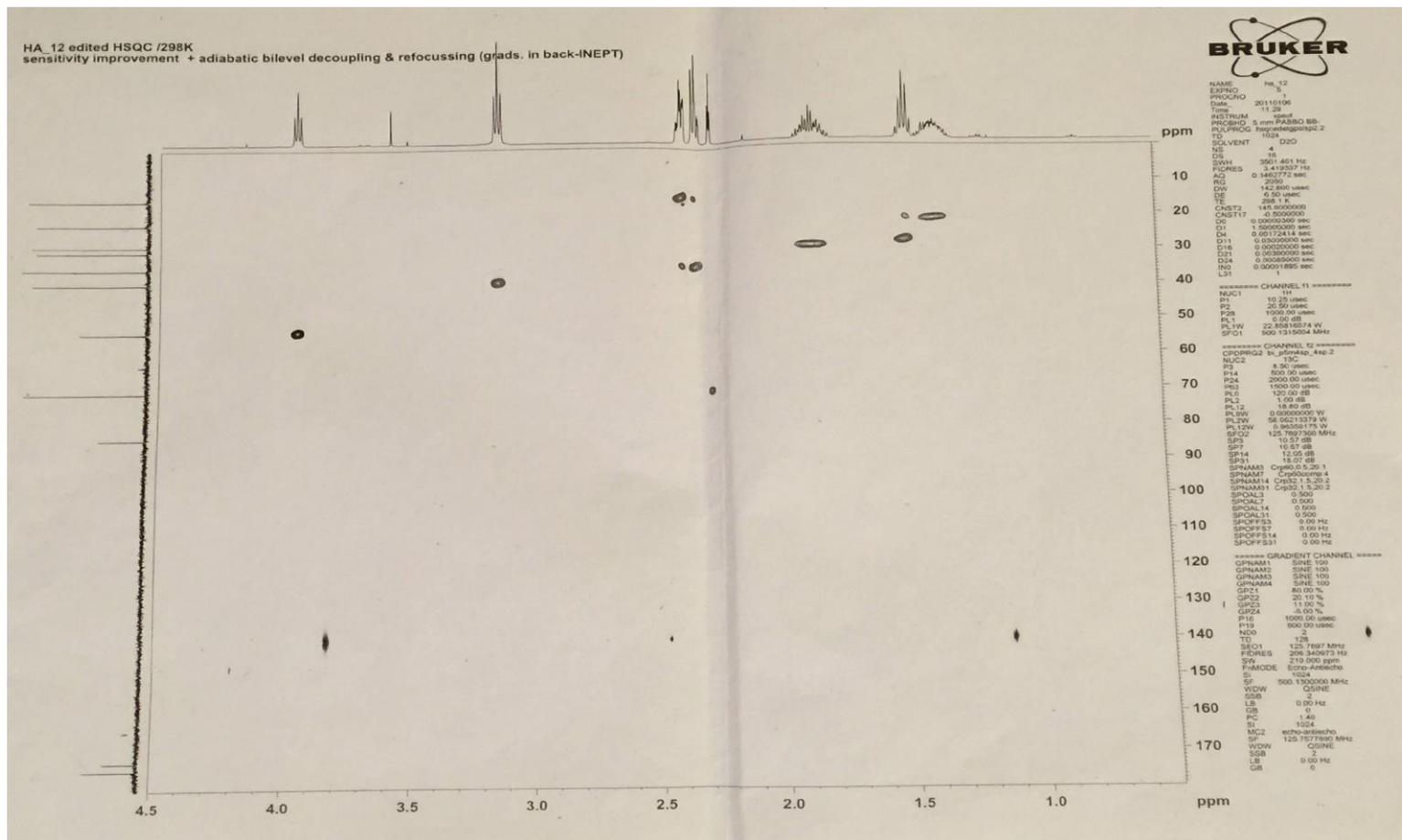
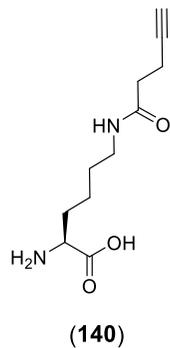
NAME          Ha_12
EXPNO         1
PROCNO        1
Date_         20110108
Time          11:15
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       D2O
AQ            0.2925044 sec
RG            128
ORF           1
AQ            0.2925044 sec
DE            6.50 uhm
TE            298.2 K
DS            0.0012375 sec
D1            0.05000000 sec
D12           0.00000000 sec
D18           0.00020000 sec
RG            0.00025000 sec
===== CHANNEL f1 =====
NUC1          1H
P1            12.00 uhm
PC            20.00 uhm
PL1           0.00 dB
PL12          22.00 dB
PL18          22.00 dB
PL1218       0.00 dB
SFO1          500.1315004 MHz
===== GRADIENT CHANNEL =====
GPRAM1       SINE 100
GPRAM2       SINE 100
GPT1         10.00 %
GPT2         20.00 %
P1S          1000.00 uhm
MDE          1
TD           128
SFO1         500.1315004 MHz
F0RES        27.564458 Hz
SFO2         500.1315004 MHz
SFO3         500.1315004 MHz
SFO4         500.1315004 MHz
SFO5         500.1315004 MHz
SFO6         500.1315004 MHz
SFO7         500.1315004 MHz
SFO8         500.1315004 MHz
SFO9         500.1315004 MHz
SFO10        500.1315004 MHz
SFO11        500.1315004 MHz
SFO12        500.1315004 MHz
SFO13        500.1315004 MHz
SFO14        500.1315004 MHz
SFO15        500.1315004 MHz
SFO16        500.1315004 MHz
SFO17        500.1315004 MHz
SFO18        500.1315004 MHz
SFO19        500.1315004 MHz
SFO20        500.1315004 MHz
SFO21        500.1315004 MHz
SFO22        500.1315004 MHz
SFO23        500.1315004 MHz
SFO24        500.1315004 MHz
SFO25        500.1315004 MHz
SFO26        500.1315004 MHz
SFO27        500.1315004 MHz
SFO28        500.1315004 MHz
SFO29        500.1315004 MHz
SFO30        500.1315004 MHz
SFO31        500.1315004 MHz
SFO32        500.1315004 MHz
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SFO42        500.1315004 MHz
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SFO63        500.1315004 MHz
SFO64        500.1315004 MHz
SFO65        500.1315004 MHz
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SFO70        500.1315004 MHz
SFO71        500.1315004 MHz
SFO72        500.1315004 MHz
SFO73        500.1315004 MHz
SFO74        500.1315004 MHz
SFO75        500.1315004 MHz
SFO76        500.1315004 MHz
SFO77        500.1315004 MHz
SFO78        500.1315004 MHz
SFO79        500.1315004 MHz
SFO80        500.1315004 MHz
SFO81        500.1315004 MHz
SFO82        500.1315004 MHz
SFO83        500.1315004 MHz
SFO84        500.1315004 MHz
SFO85        500.1315004 MHz
SFO86        500.1315004 MHz
SFO87        500.1315004 MHz
SFO88        500.1315004 MHz
SFO89        500.1315004 MHz
SFO90        500.1315004 MHz
SFO91        500.1315004 MHz
SFO92        500.1315004 MHz
SFO93        500.1315004 MHz
SFO94        500.1315004 MHz
SFO95        500.1315004 MHz
SFO96        500.1315004 MHz
SFO97        500.1315004 MHz
SFO98        500.1315004 MHz
SFO99        500.1315004 MHz
SFO100       500.1315004 MHz

```

Appendix B109: COSYDQF NMR spectrum of compound (140).



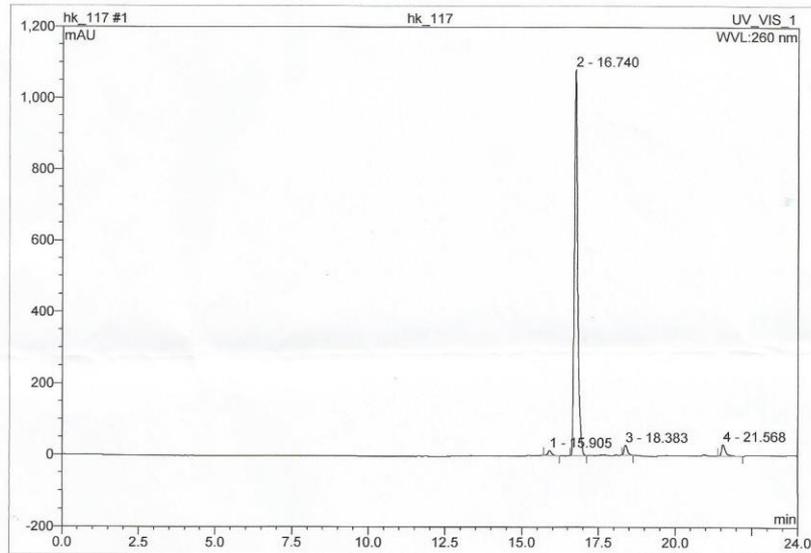
Appendix B 110: NOESY NMR spectrum of compound (140).



Appendix B 111: HSQC NMR spectrum of compound (140).

1 hk_117

Sample Name:	hk_117	Injection Volume:	20.0
Vial Number:	RA1	Channel:	UV_VIS_1
Sample Type:	unknown	Wavelength:	260
Control Program:	poly-p4 28min +230nm	Bandwidth:	10
Quantif. Method:	dna method	Dilution Factor:	1.0000
Recording Time:	11/7/2012 15:58	Sample Weight:	1.0000
Run Time (min):	24.00	Sample Amount:	1.0000



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	15.91	n.a.	14.186	1.976	1.34	n.a.	BMB
2	16.74	n.a.	1082.482	137.744	93.17	n.a.	BMB
3	18.38	n.a.	27.857	3.456	2.34	n.a.	BMB
4	21.57	n.a.	32.130	4.669	3.16	n.a.	BMB
Total:			1156.655	147.846	100.00	0.000	

DEFAULT/Integration

Chromeleon (c) Dionex 1996-2006
Version 6.80 SR10 Build 2818 (166959)**Appendix B112: HPL chromatogram of compound (132).**

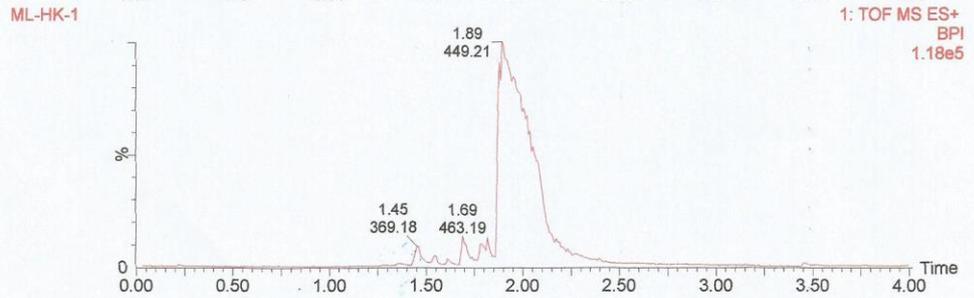
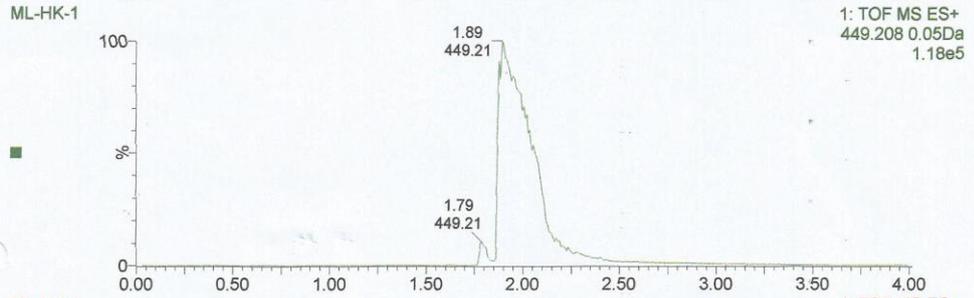
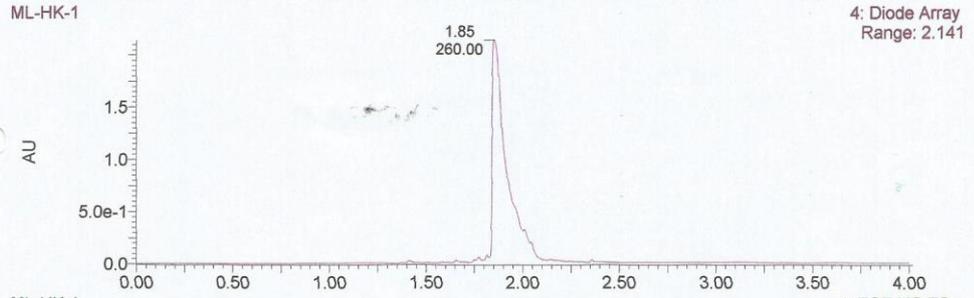
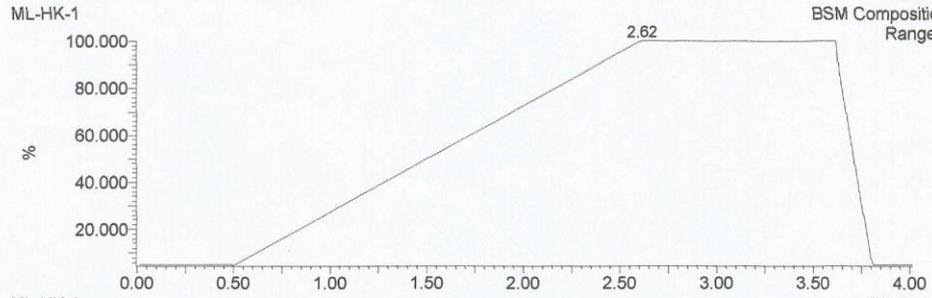
26 AFTER PURIFICATION

30-Mar-2011

15:30:49

BSM Composition B

Range: 95



Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 2.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

1710 formula(e) evaluated with 4 results within limits (all results (up to 1000) for each mass)

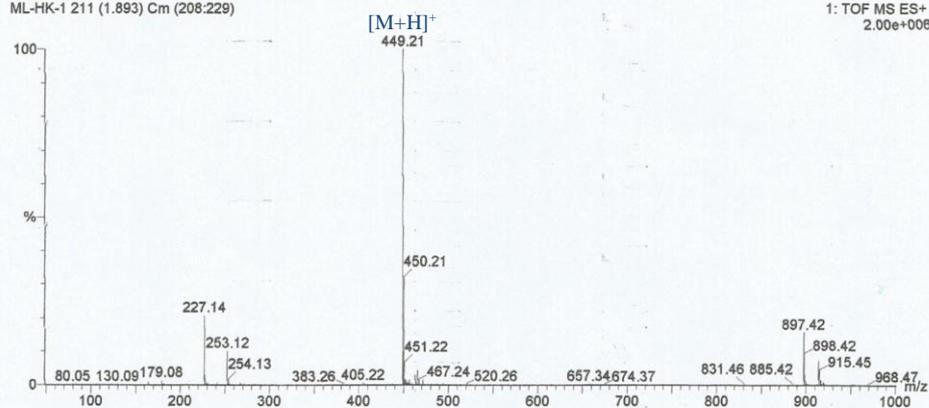
Elements Used:

C: 0-200 H: 0-200 N: 0-100 O: 0-100

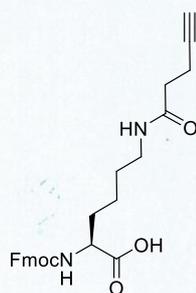
26 AFTER PURIFICATION

ML-HK-1 211 (1.893) Cm (208:229)

1: TOF MS ES+
2.00e+006

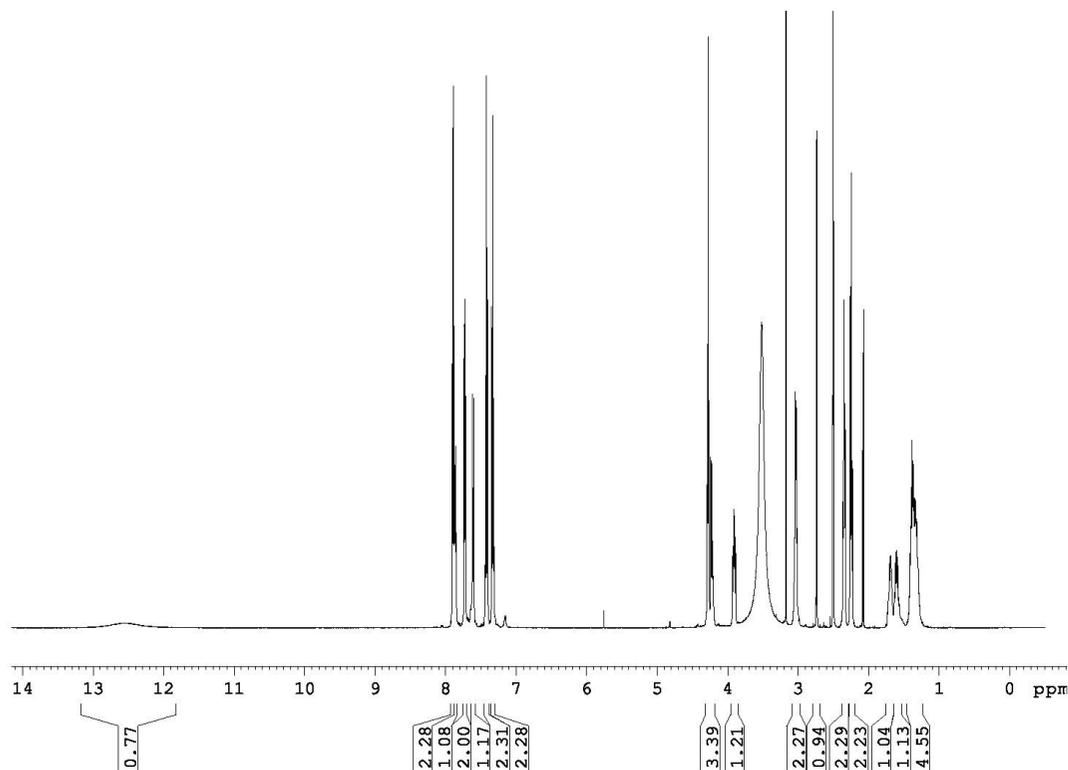
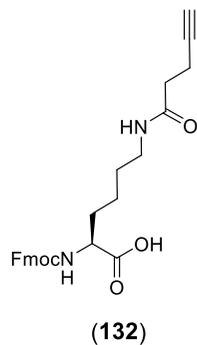


Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
449.2074	449.2076	-0.2	-0.4	13.5	552.1	0.0	C26 H29 N2 O5
	449.2081	-0.7	-1.6	6.5	559.7	7.6	C11 H25 N14 O6
	449.2068	0.6	1.3	1.5	560.2	8.1	C10 H29 N10 O10
	449.2068	0.6	1.3	12.5	561.1	9.0	C8 H17 N24



(132)

Appendix B113: HRMS (ESI) spectra of compound (132).



```

Current Data Parameters
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PROCNO        1

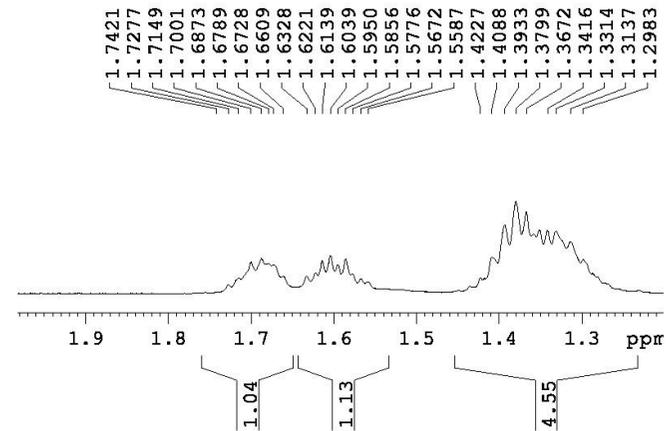
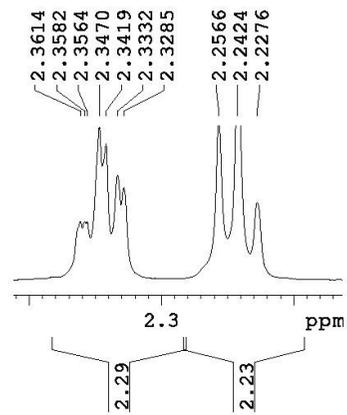
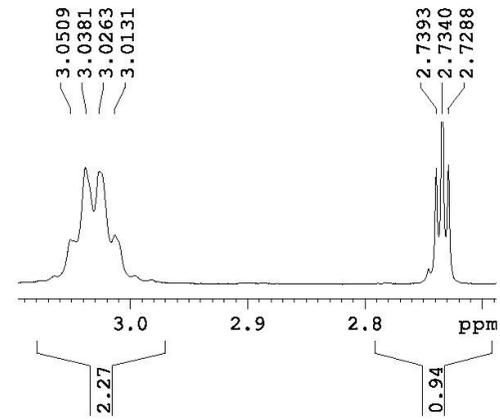
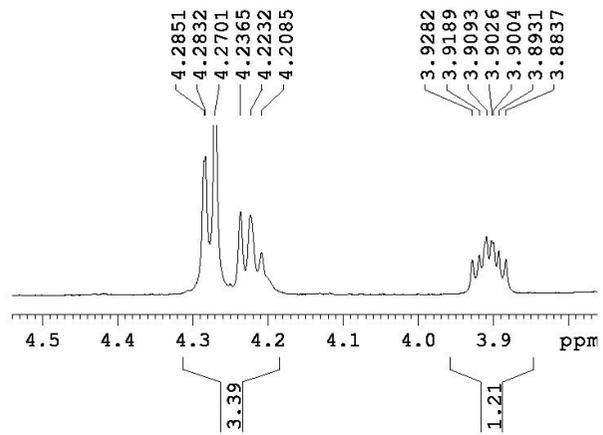
F2 - Acquisition Parameter
Date_         20110331
Time          15.37
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg
TD            65536
SOLVENT       DMSO
NS            16
DS            2
SWH           7500.000 Hz
FIDRES        0.114441 Hz
AQ            4.3690667 sec
RC            101
DW            66.667 usec
DE            6.50 usec
TE            298.0 K
D1            6.0000000 sec
TD0           1

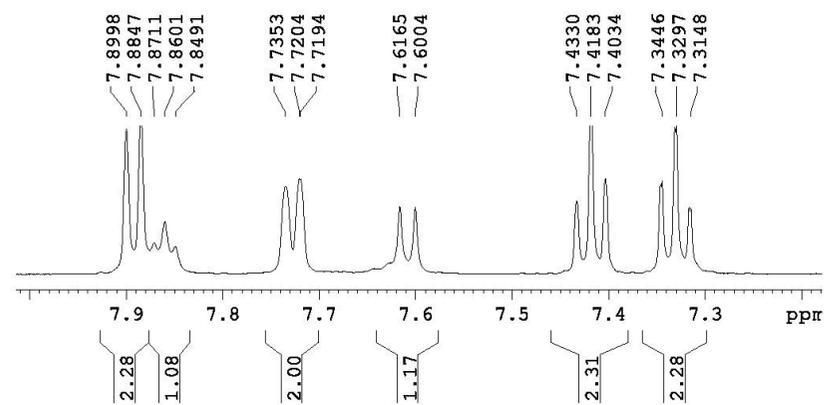
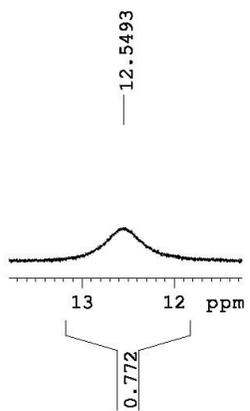
===== CHANNEL f1 =====
NUC1          1H
P1            10.25 usec
PL1           0 dB
PL1W          22.85816574 W
SFO1          500.1335009 MHz

F2 - Processing parameters
SI            32768
SF            500.1300051 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00

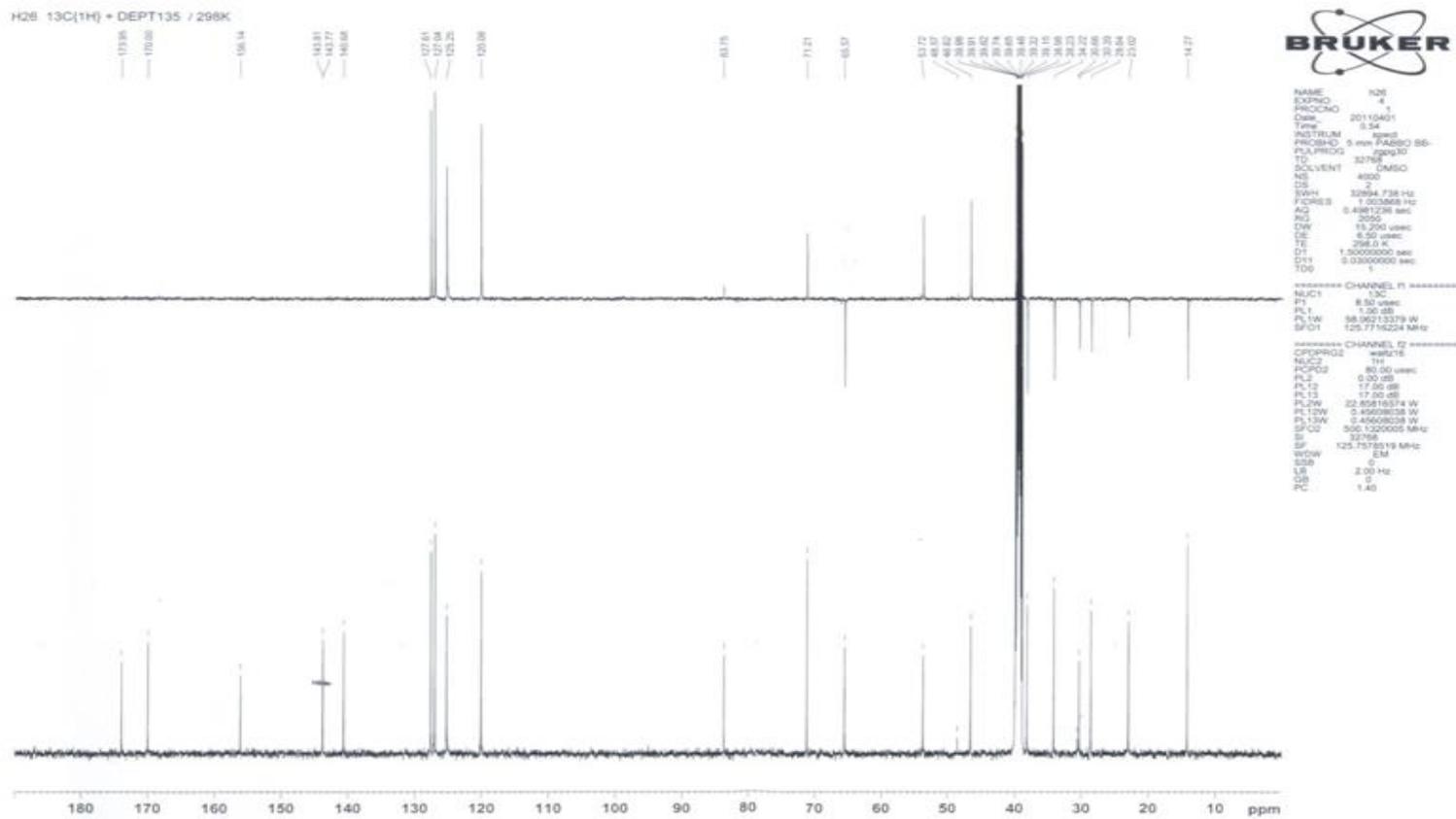
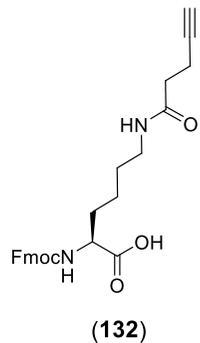
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Appendix B114: ¹H NMR spectrum of compound (132).

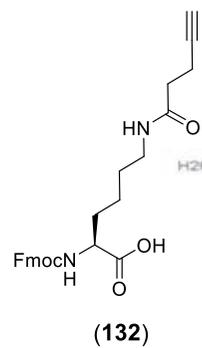




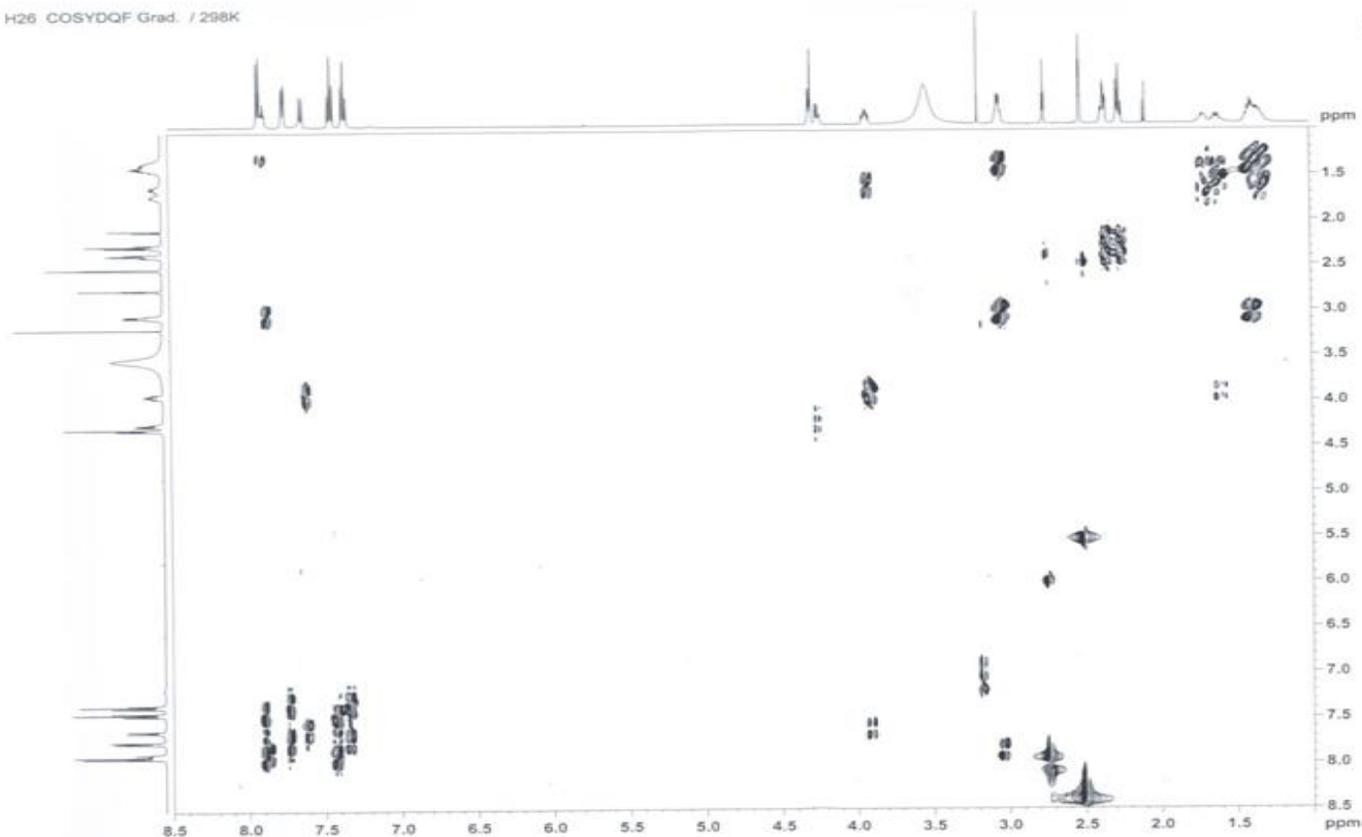
Appendix B115: Selected areas ¹H NMR of compound (132).



Appendix B116: ^{13}C NMR spectrum of compound (132).

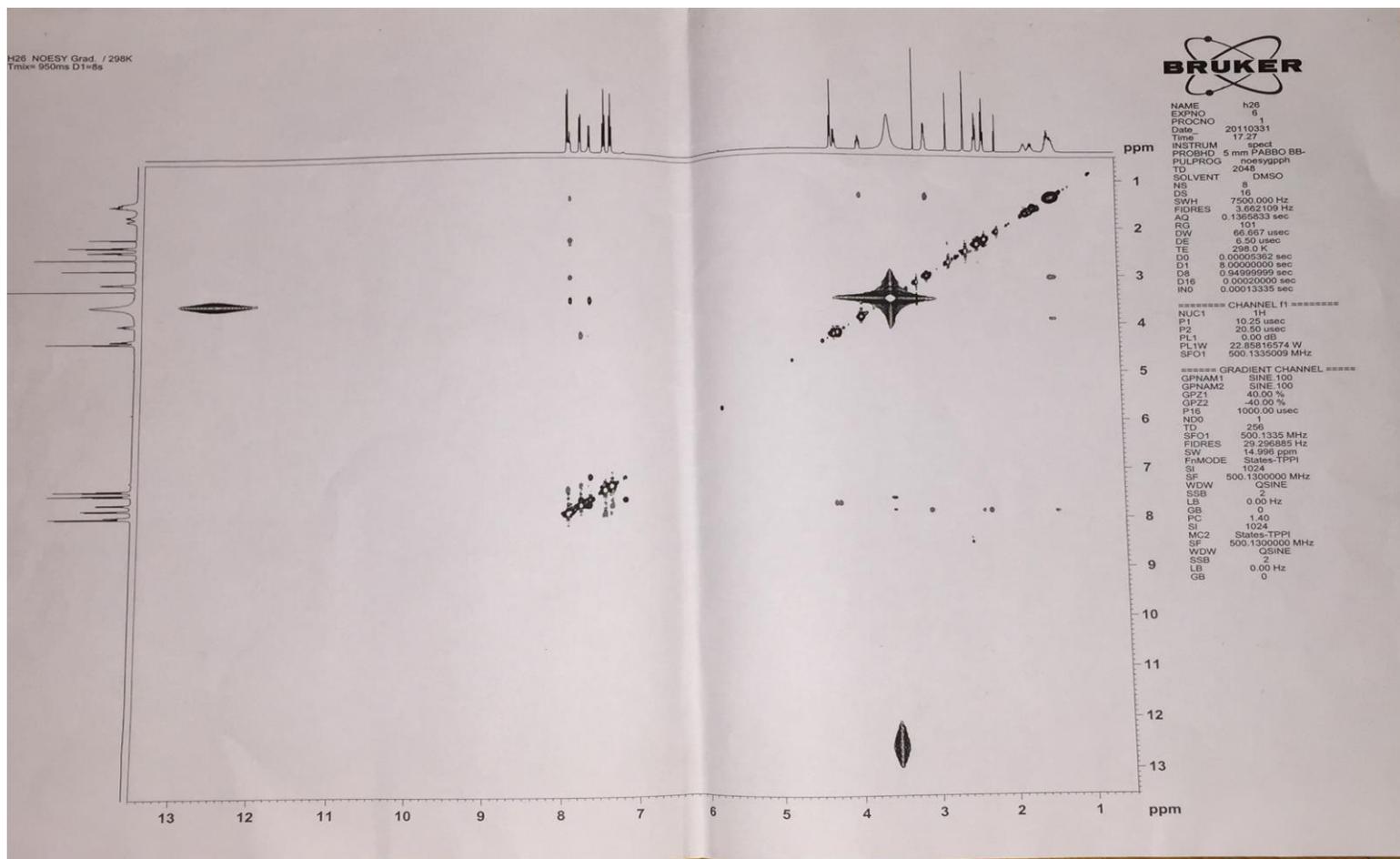
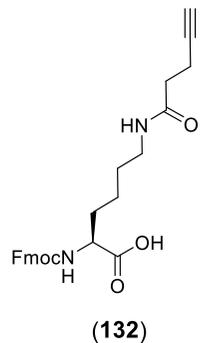


H26 COSYDQF Grad. / 298K

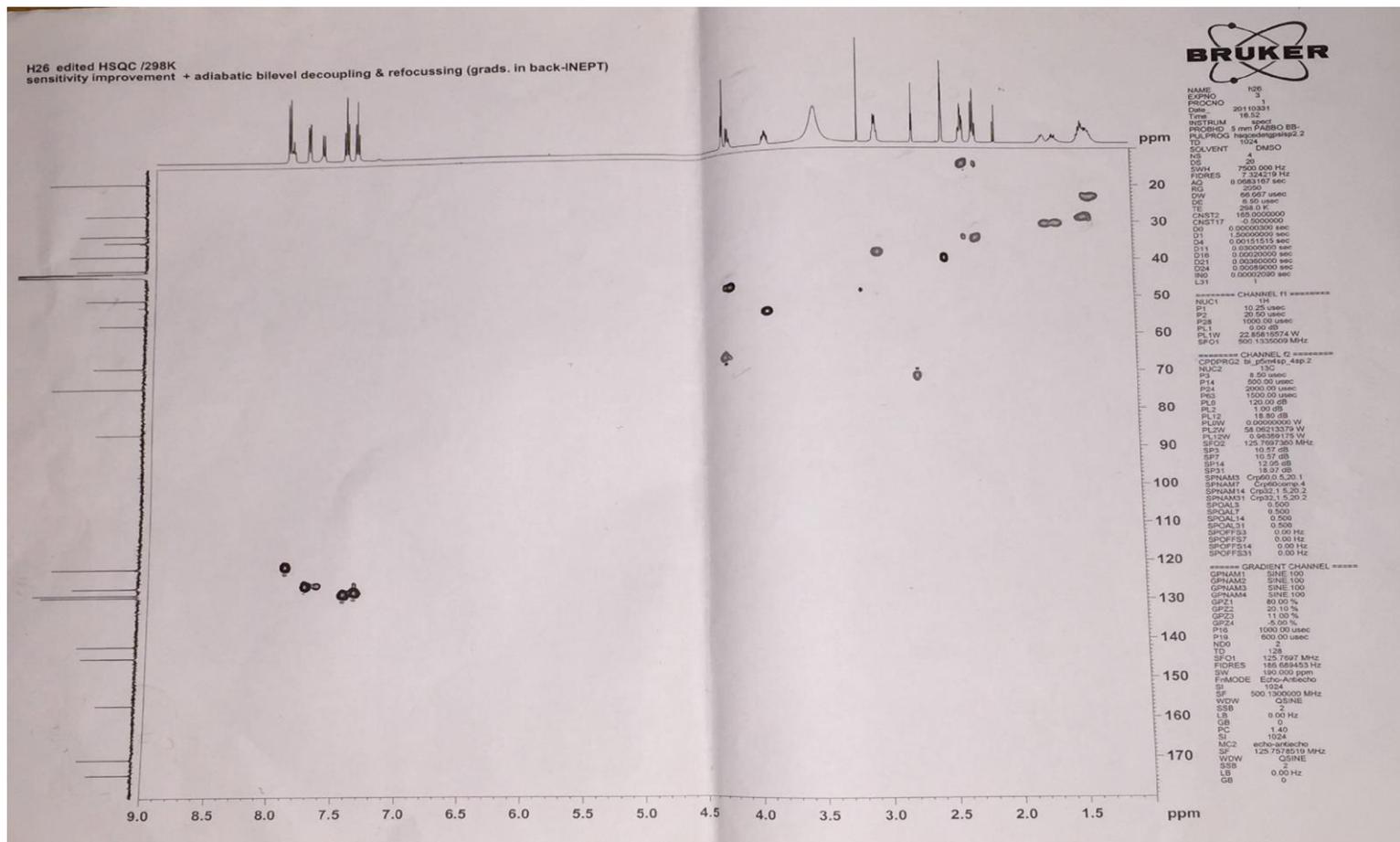
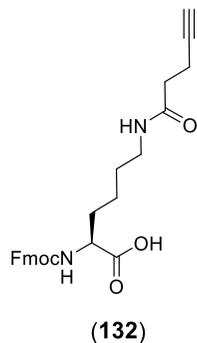


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 EXPNO: 2
 PROCNO: 1
 Date_ Time: 20110331 15:39
 INSTRUM: spect
 PROBHD: 5 mm PABBO BB-
 PULPROG: zgpg30
 TD: 3248
 SOLVENT: DMSO
 NS: 2
 DS: 16
 SWH: 7500.000 Hz
 FIDRES: 1.662109 Hz
 AQ: 0.1300633 sec
 RG: 501
 CW: 64.607 usec
 DE: 6.50 usec
 TE: 298.2 K
 D0: 0.0000302 sec
 D1: 8.0000000 sec
 D13: 0.0000400 sec
 D16: 0.0002000 sec
 R0: 0.0011335 sec
 ***** CHANNEL f1 *****
 NUC1: 13C
 P1: 10.25 usec
 P2: 20.50 usec
 PL1: 0.00 dB
 PL12: 22.45811574 W
 SFO1: 500.1350000 MHz
 ***** GRADIENT CHANNEL *****
 GPNAM1: SINE 100
 GPNAM2: SINE 100
 GP1: 10.00 %
 GP2: 20.00 %
 PH: 1000.00 usec
 NDO: 1
 TD: 128
 SFO2: 500.1326 MHz
 FIDRES: 58.892769 Hz
 SW: 14.596 ppm
 FWHM: 1024
 SF: 500.1300000 MHz
 WIDW: GSINE
 SSB: 2
 LB: 0.00 Hz
 GB: 0
 PC: 1.40
 SI: 1624
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 SF: 500.1300000 MHz
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 LB: 0.00 Hz
 GB: 0

Appendix B117: COSYDQF NMR spectrum of compound (132).



Appendix B 118: NOESY NMR spectrum of compound (132).



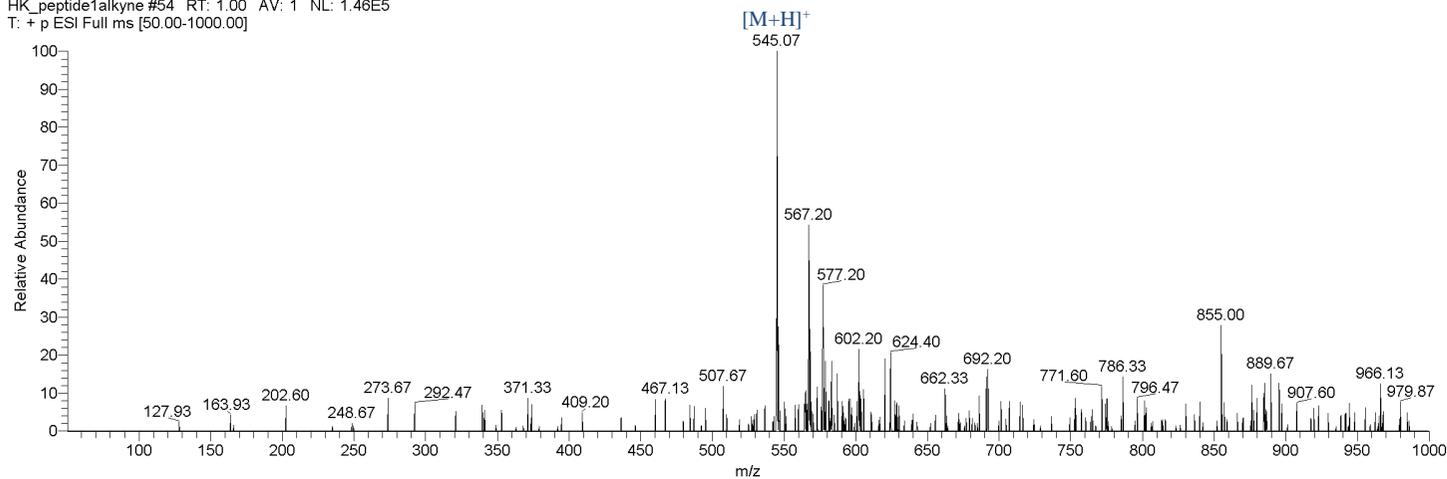
Appendix B 119: HSQC NMR spectrum of compound (132).

G:\data\...Burley\HK_peptide1alkyne
MeOH

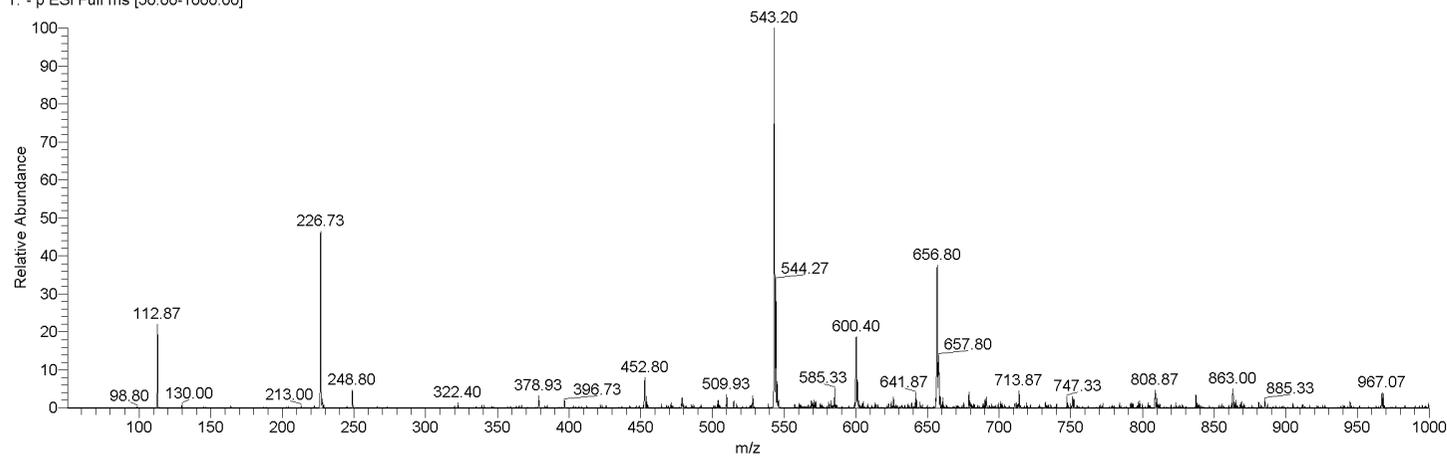
08/04/2014 15:47:20

HK_peptide1alkyne

HK_peptide1alkyne #54 RT: 1.00 AV: 1 NL: 1.46E5
T: + p ESI Full ms [50.00-1000.00]



HK_peptide1alkyne #56-98 RT: 1.06-1.85 AV: 43 NL: 2.25E4
T: - p ESI Full ms [50.00-1000.00]



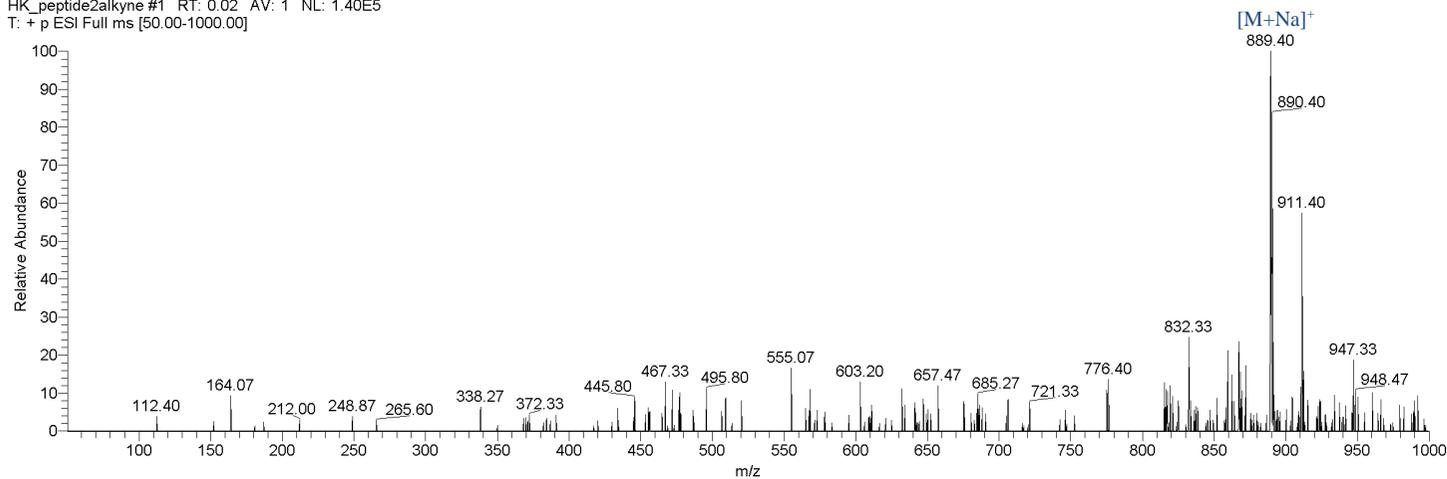
Appendix B120: LRMS (ESI) spectrum of compound (133).

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MeOH

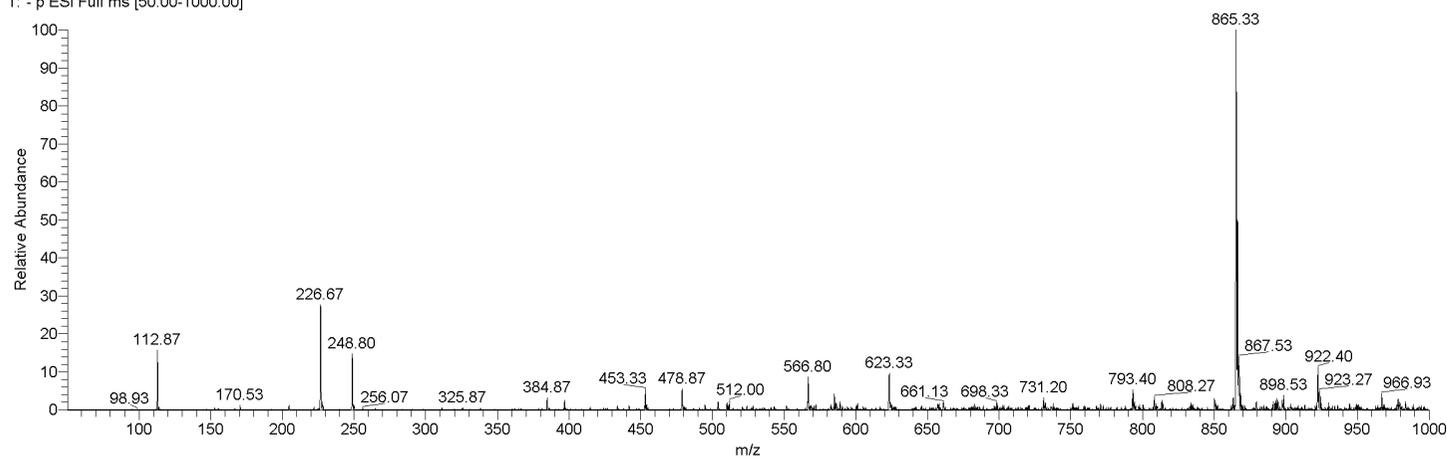
08/04/2014 15:56:11

HK_peptide1alkyne

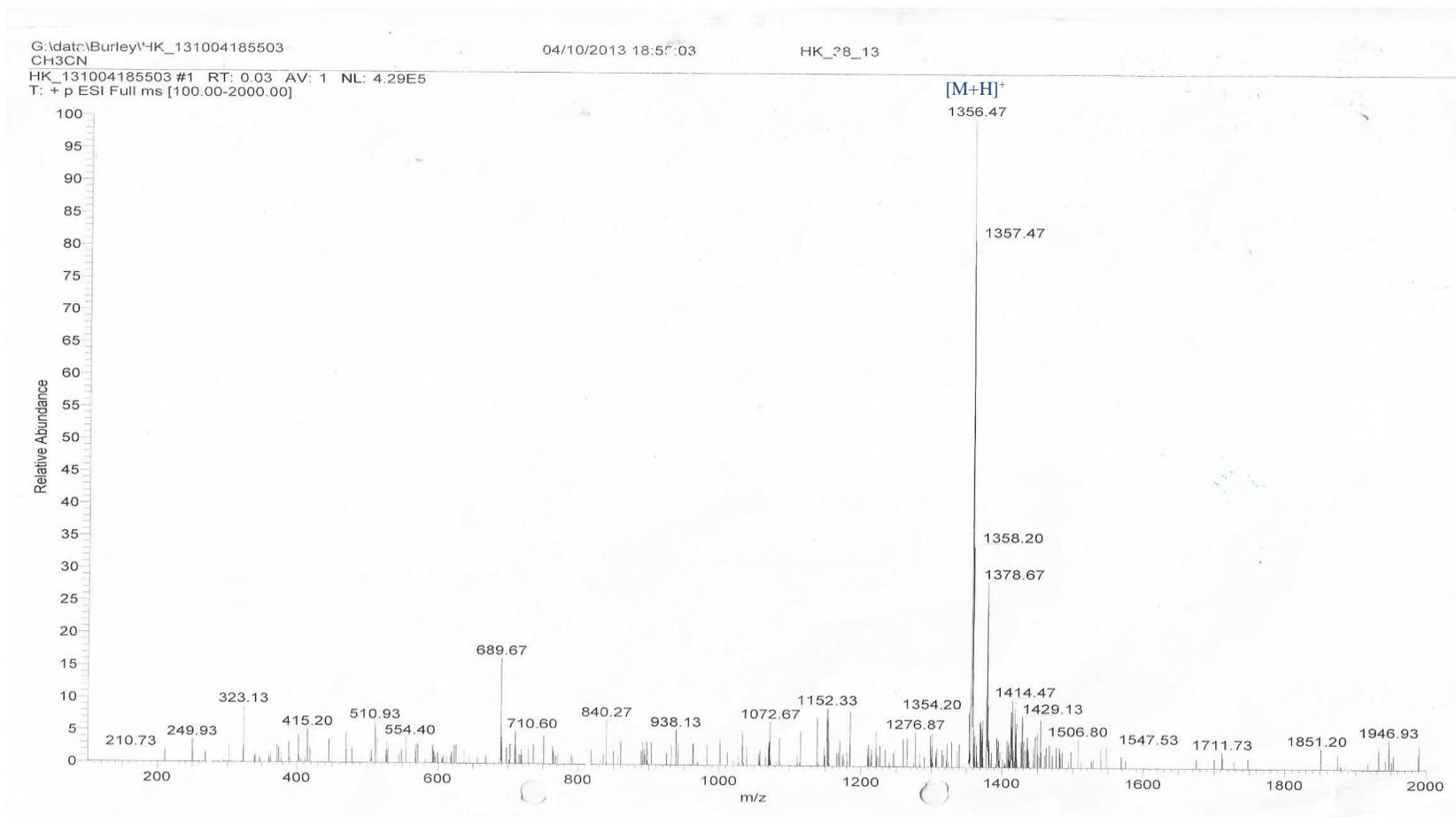
HK_peptide2alkyne #1 RT: 0.02 AV: 1 NL: 1.40E5
T: + p ESI Full ms [50.00-1000.00]



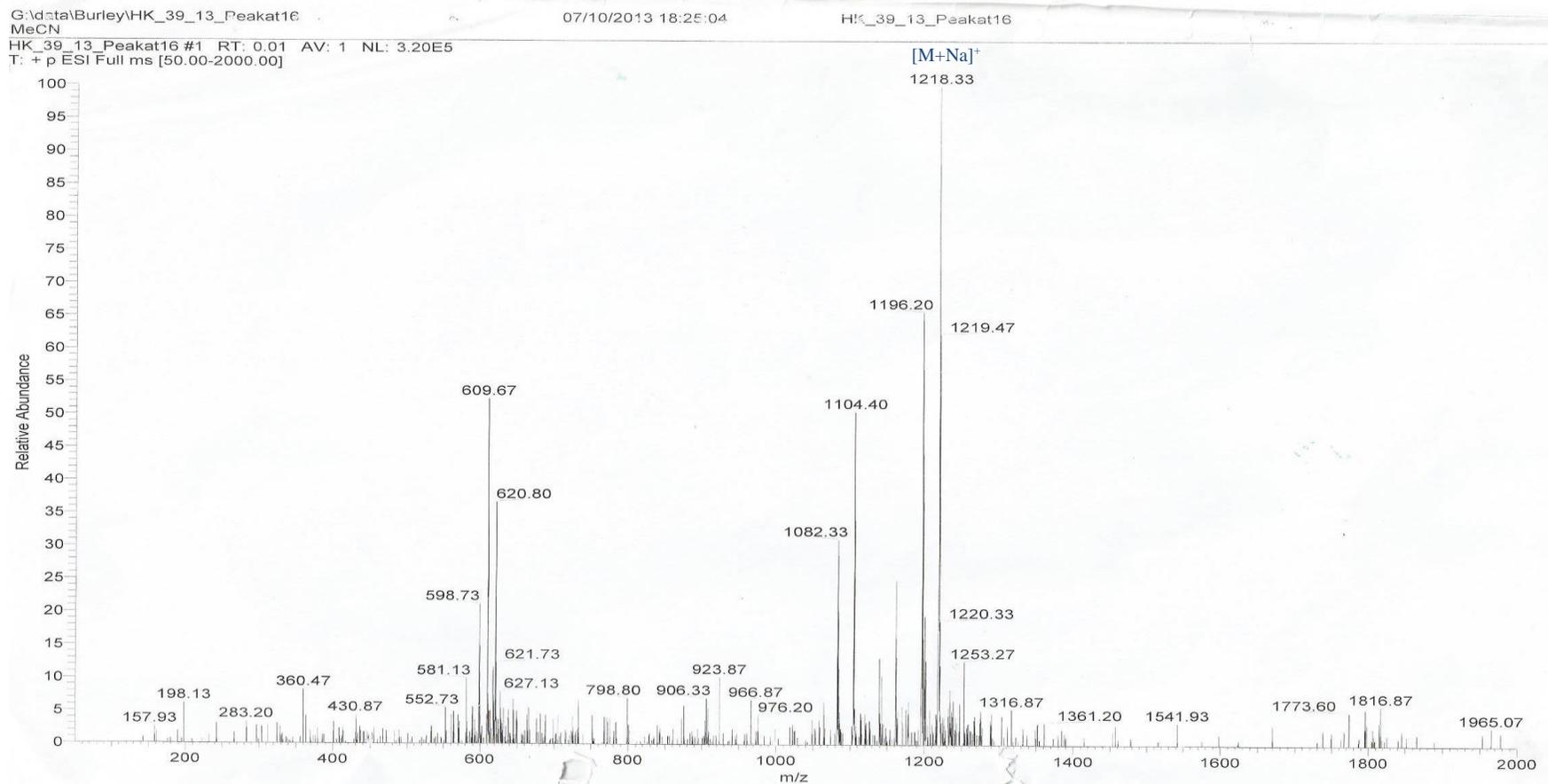
HK_peptide2alkyne #44-80 RT: 0.85-1.43 AV: 32 NL: 3.90E4
T: - p ESI Full ms [50.00-1000.00]



Appendix B121: LRMS (ESI) spectrum of compound (134).



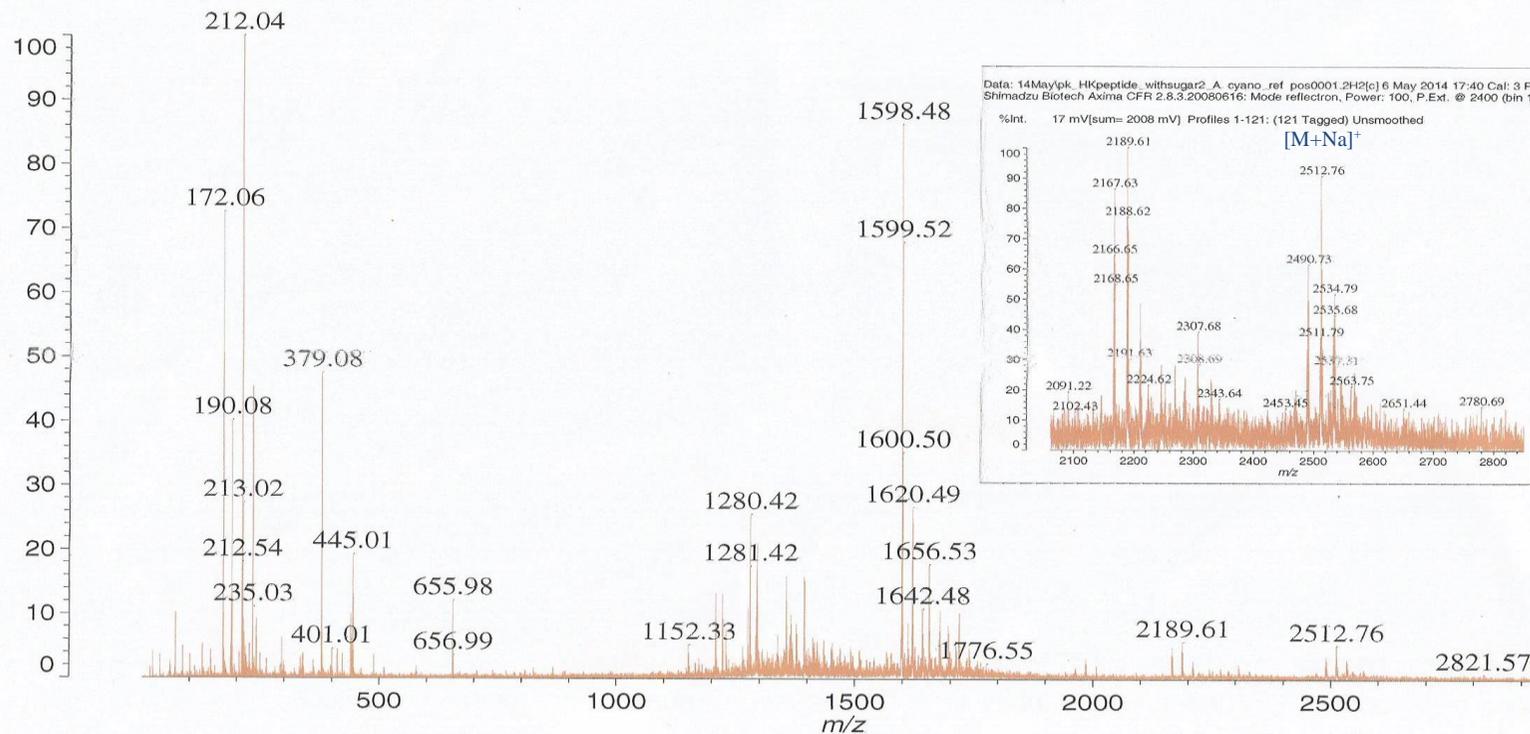
Appendix B122: LRMS (ESI) spectrum of compound (151).

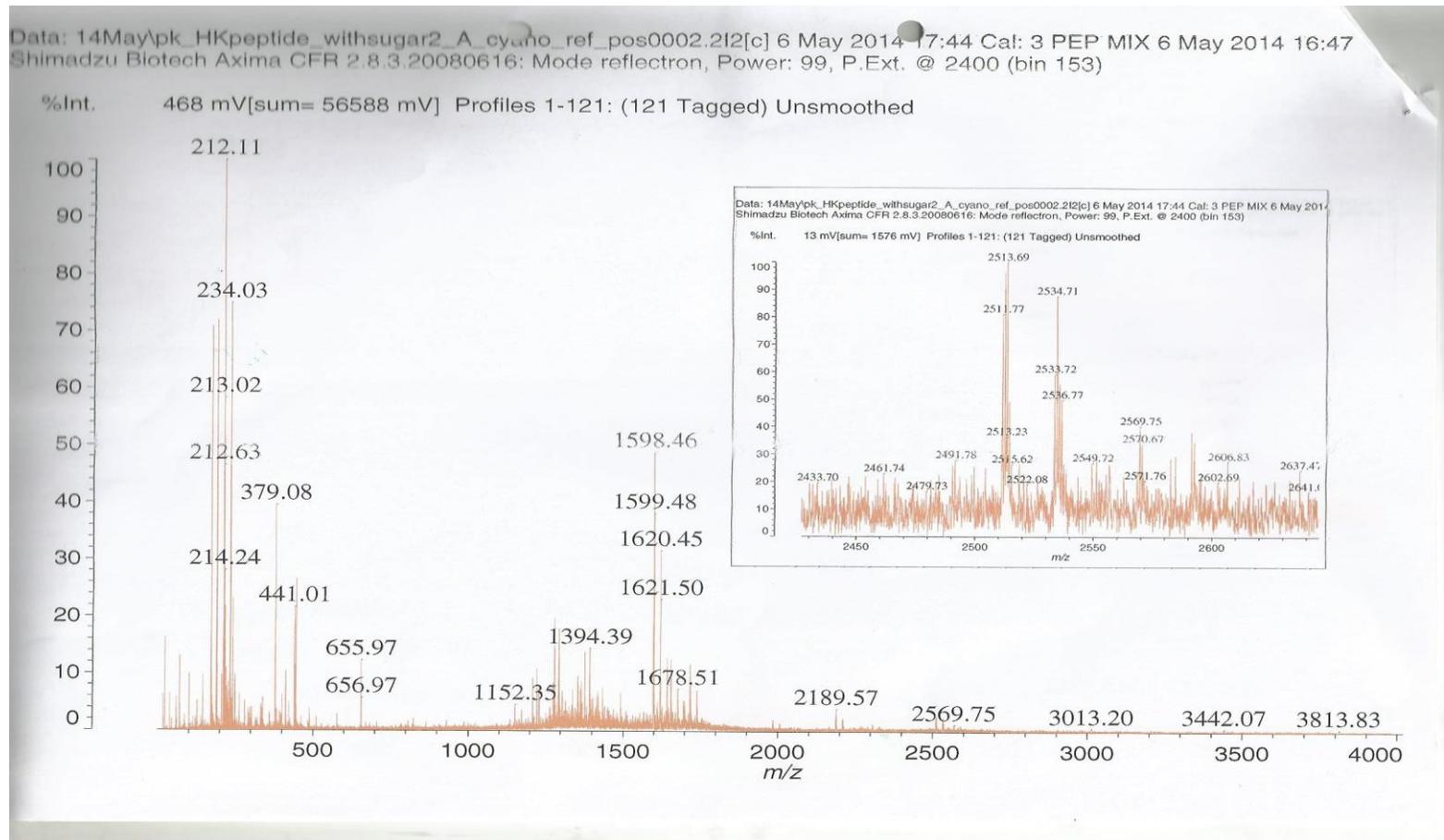


Appendix B123: LRMS (ESI) spectrum of compound (27).

Data: 14May\pk_HKpeptide_withsugar2_A_cyano_ref_pos0001.2H2[c] 6 May 2014 17:40 Cal: 3 PEP MIX 6 May 2014 16:47
Shimadzu Biotech Axima CFR 2.8.3.20080616: Mode reflectron, Power: 100, P.Ext. @ 2400 (bin 153)

%Int. 288 mV[sum= 34863 mV] Profiles 1-121: (121 Tagged) Unsmoothed

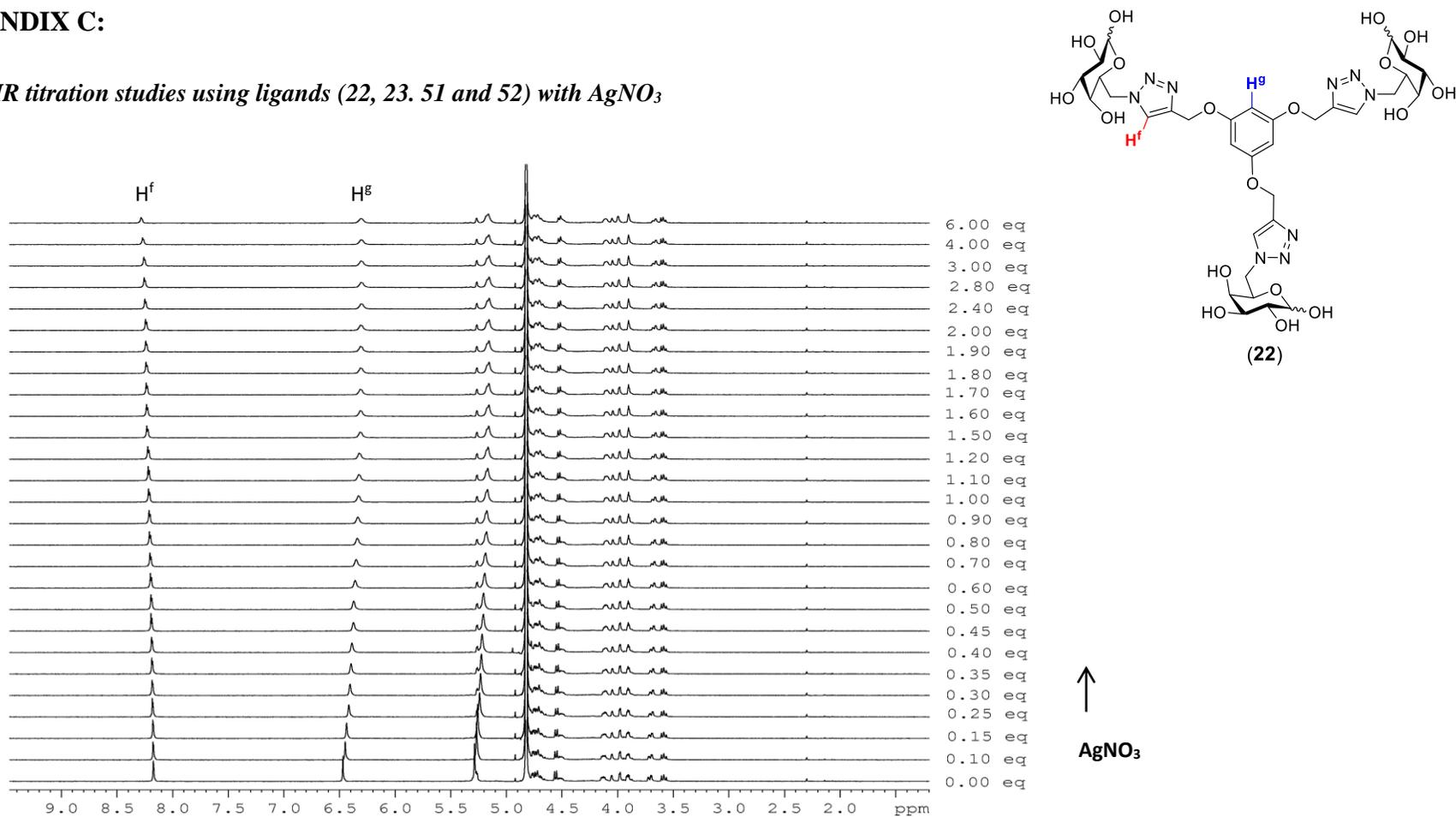




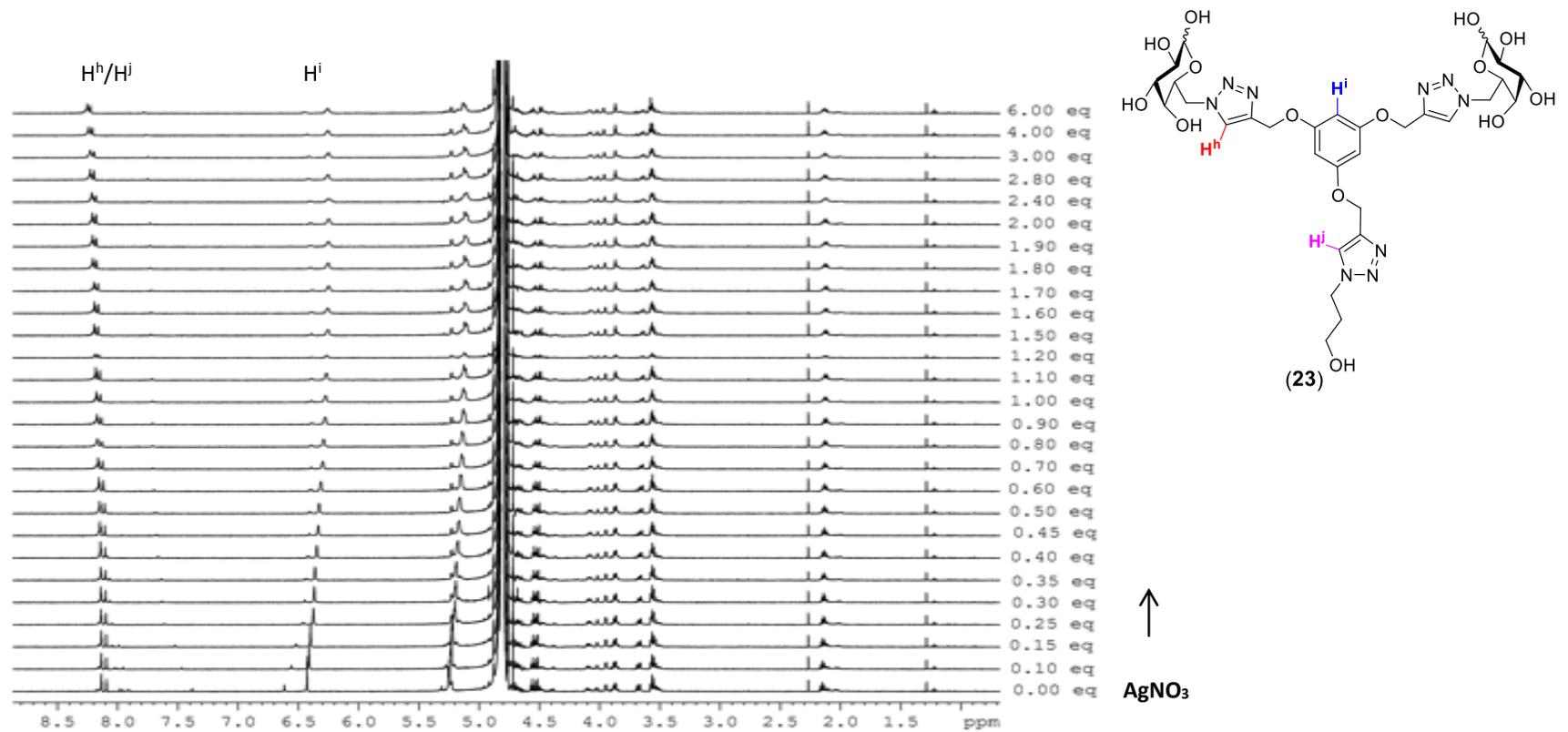
Appendix B124: MALDI spectrum of compound (152).

APPENDIX C:

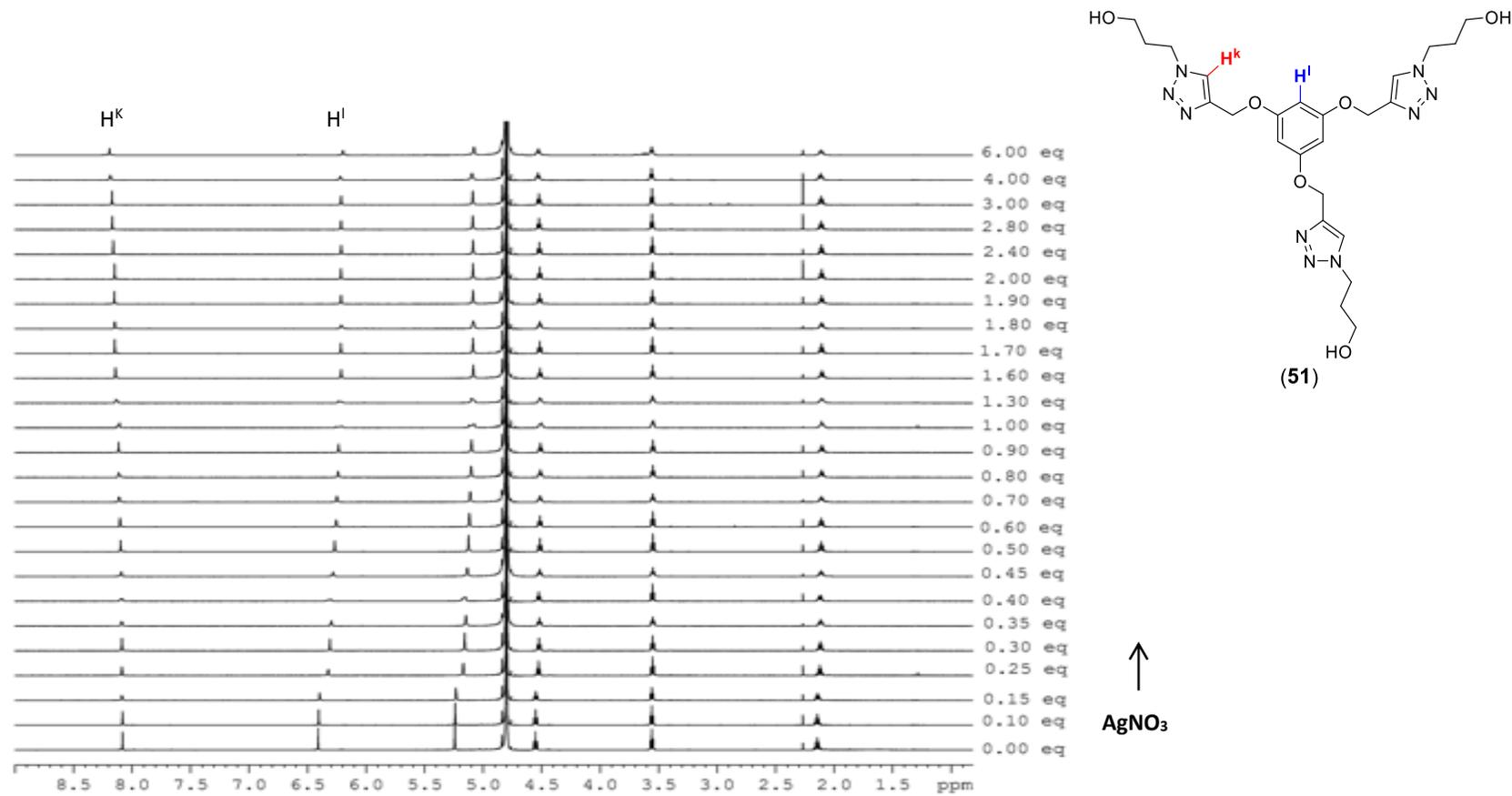
¹H NMR titration studies using ligands (22, 23, 51 and 52) with AgNO₃



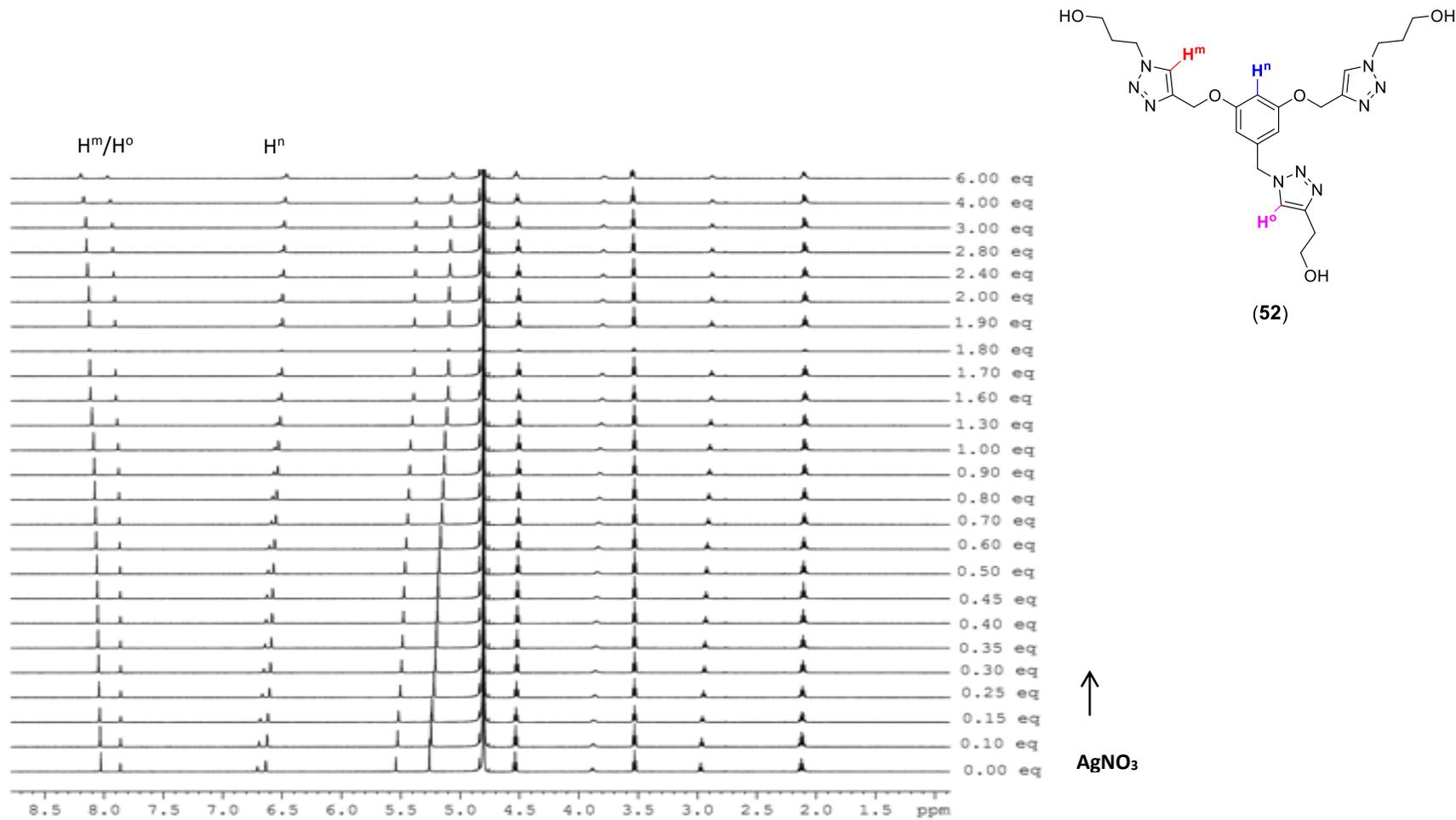
Appendix C1:(a) Stack plot of ¹H-NMR (500 MHz, D₂O) of (22) (stock concentration 2 mM) with an increasing amount of AgNO₃.



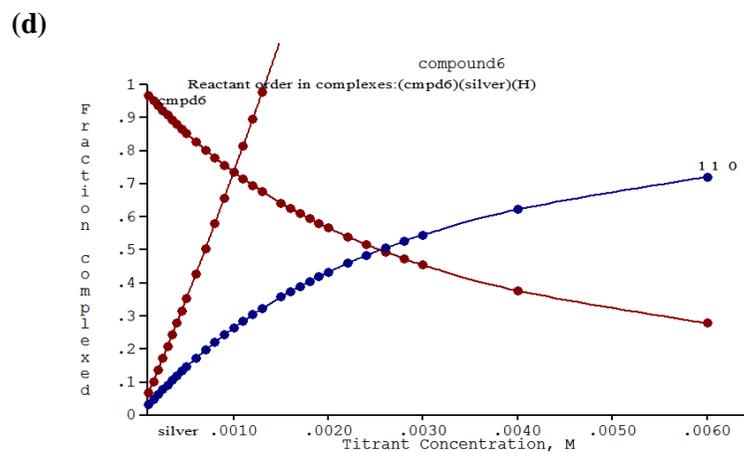
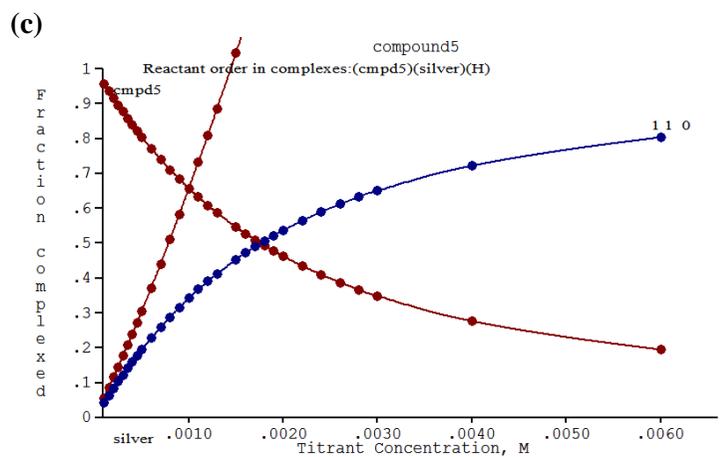
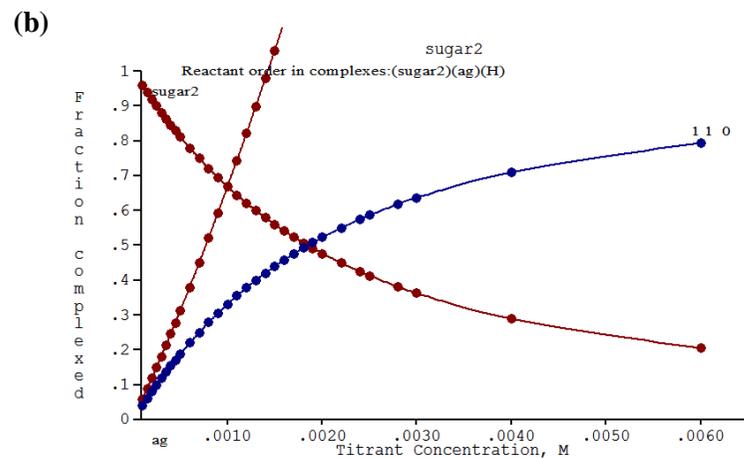
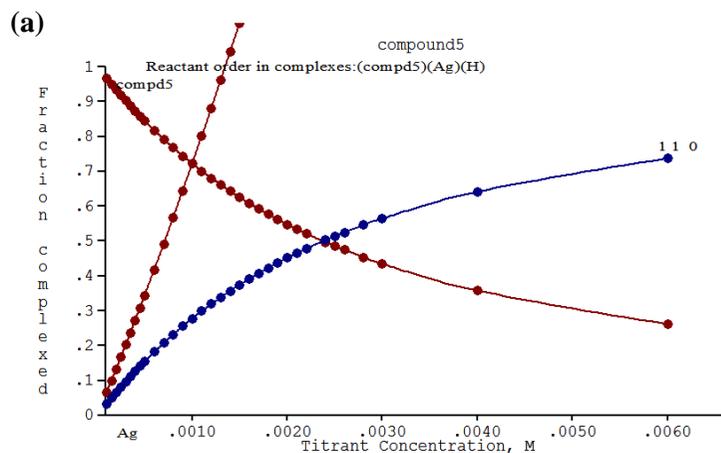
Appendix C2: Stack plot of ¹H-NMR (500 MHz, D₂O) of (23) (stock concentration 2 mM) with an increasing amount of AgNO₃.



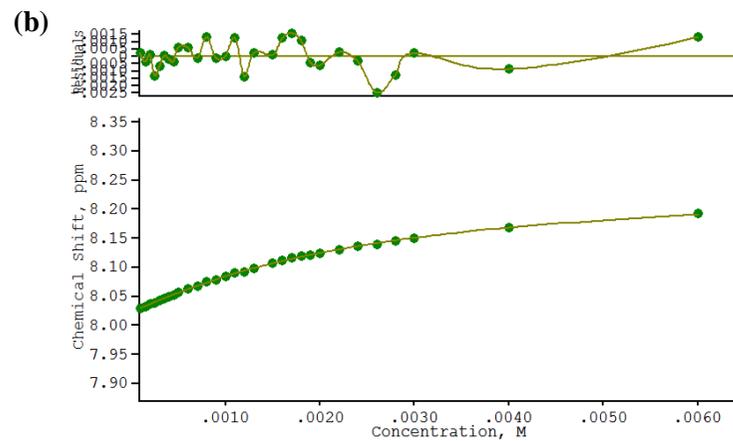
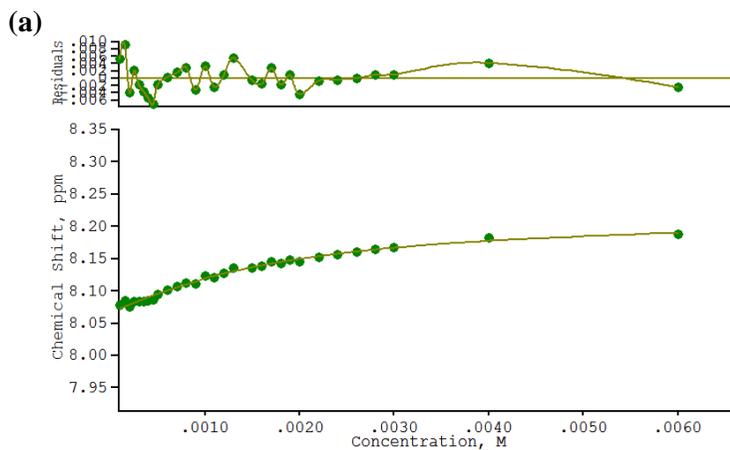
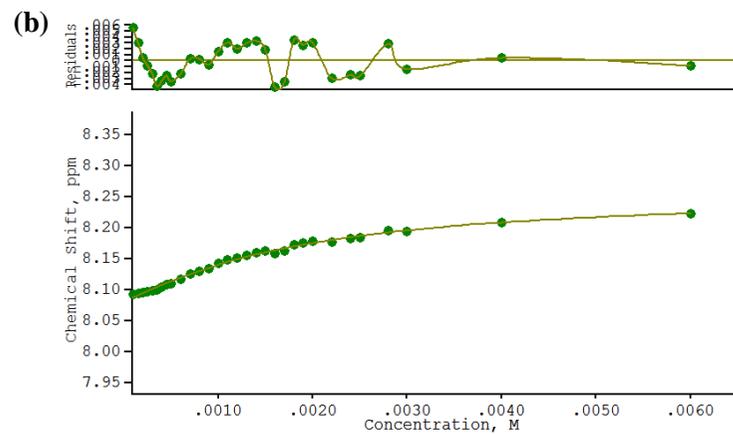
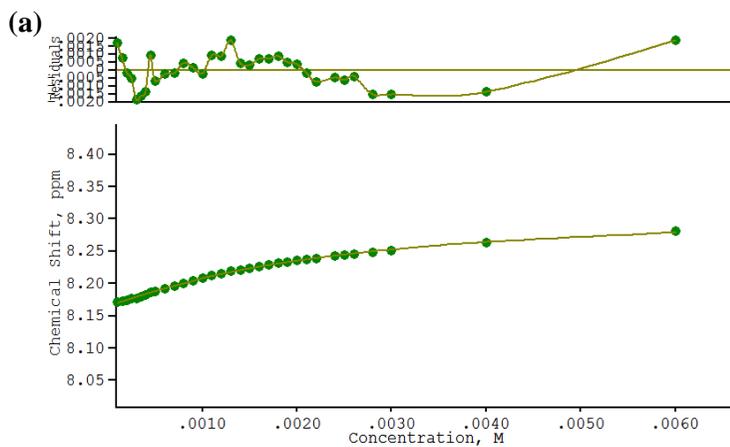
Appendix C3: Stack plot of $^1\text{H-NMR}$ (500 MHz, D_2O) of (51) (stock concentration 2 mM) with an increasing amount of AgNO_3 .



Appendix C4: Stack plot of ¹H-NMR (500 MHz, D₂O) of (52) (stock concentration 2 mM) with an increasing amount of AgNO₃.

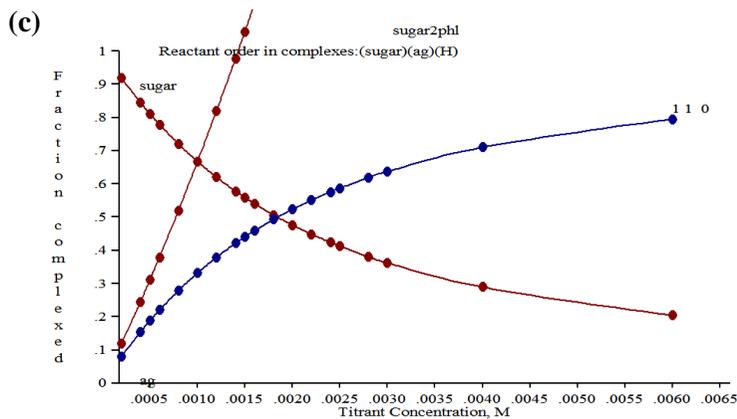
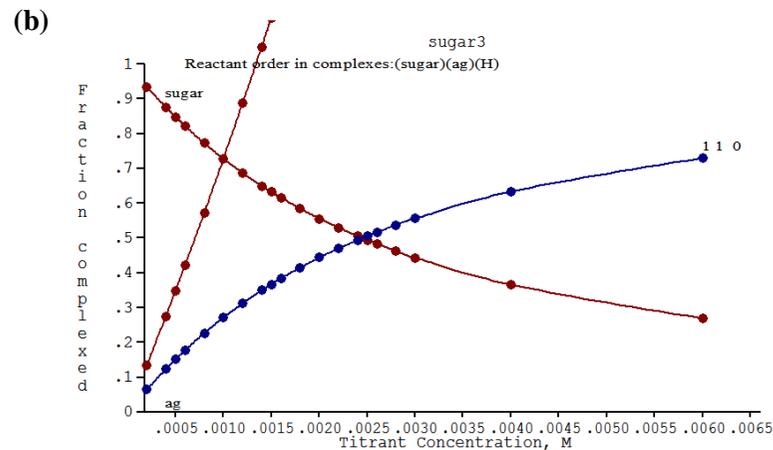
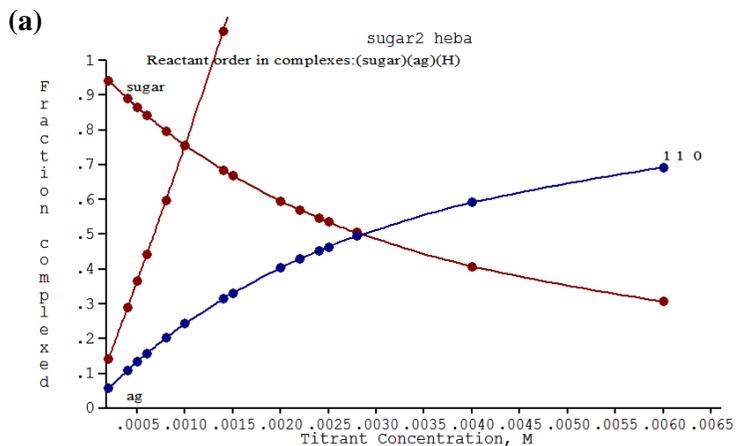


Appendix C5: Plots of the concentrations of free species and complex species expressed as molar fractions against the concentration of the titrant Ag using (a) (22), (b) (23), (c) (51) and (d) (52).

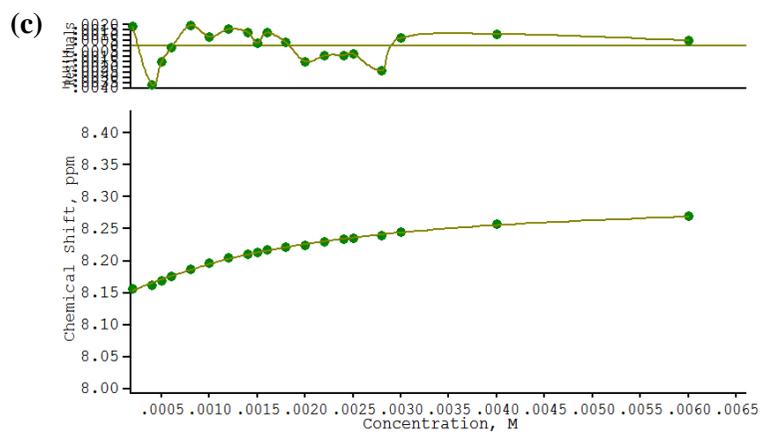
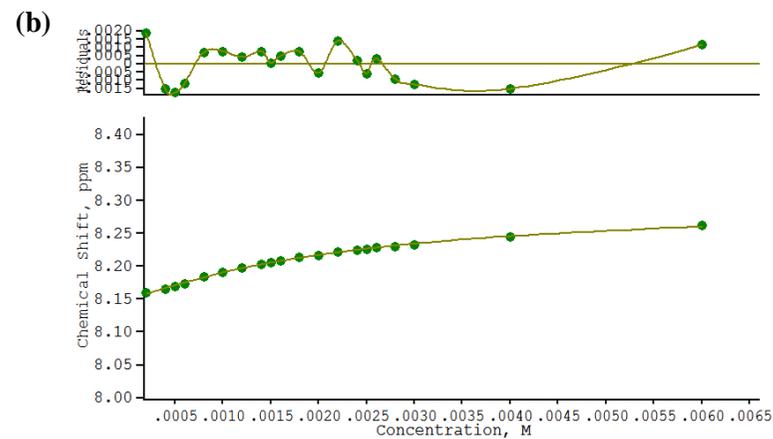
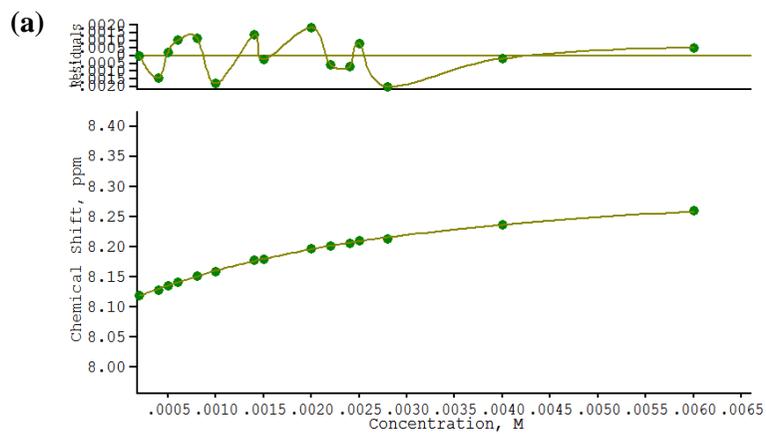


Appendix C6: Plots of the experimental points and the calculated best fit line against concentration of titrant Ag using (a) (22), (b) (23), (c) (51) and (d) (52).

¹H NMR titration studies using ligands (18, 22 and 23) with CF₃COOAg

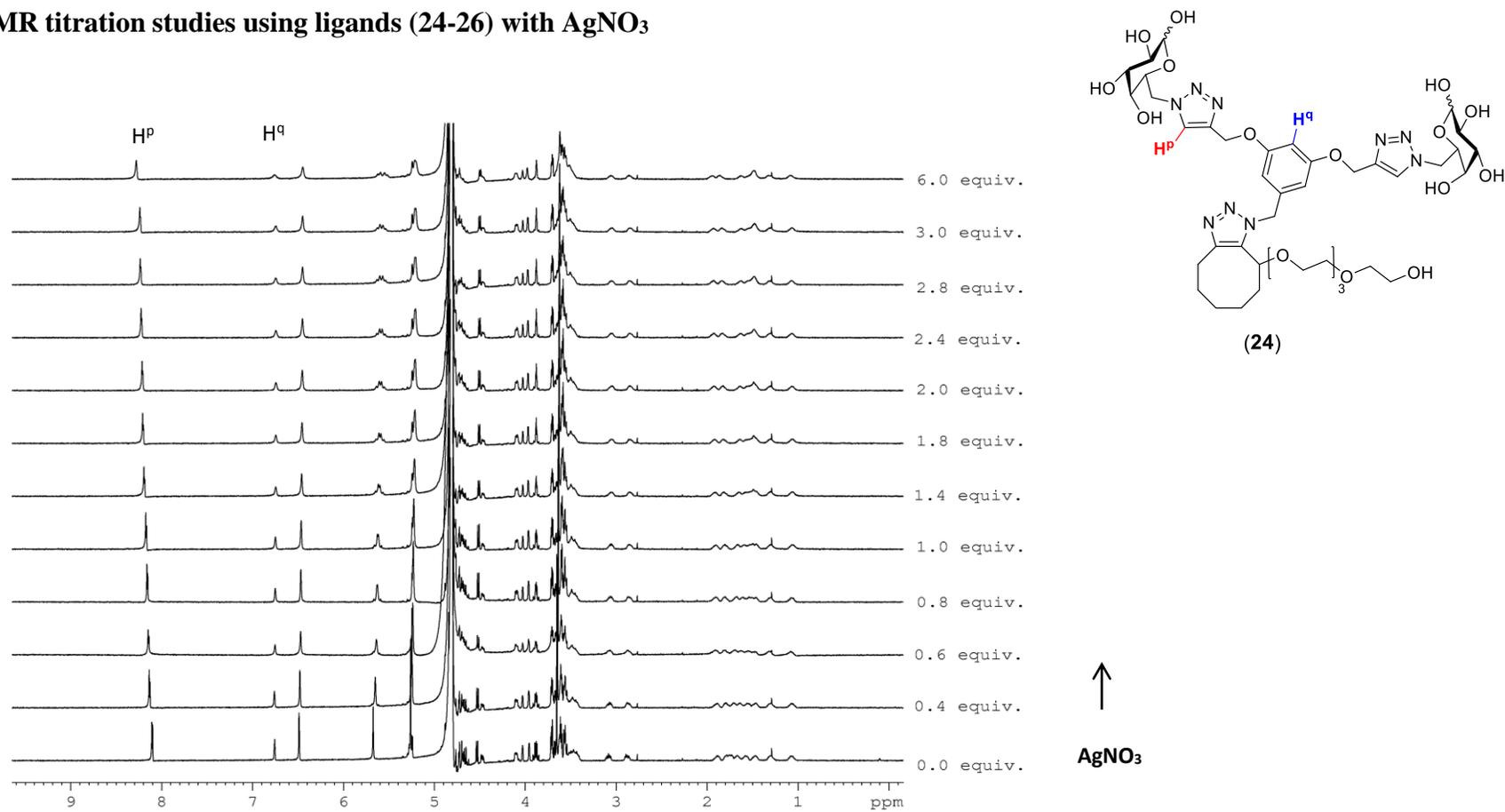


Appendix C7: Plots of the concentrations of free species and complex species expressed as molar fractions against the concentration of the titrant Ag using (a) (18), (b) (22) and (c) (23).

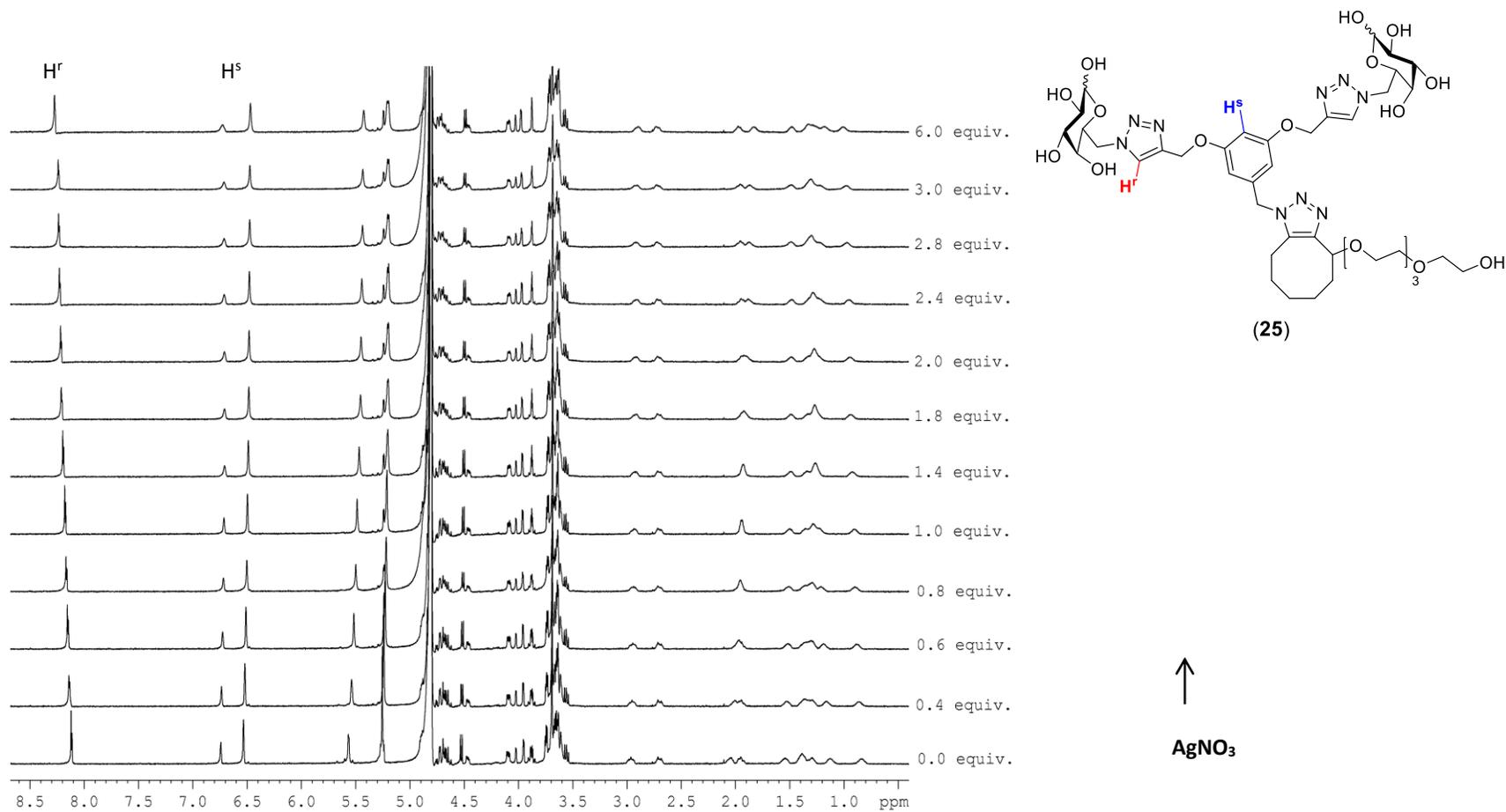


Appendix C8: Plots of the experimental points and the calculated best fit line against concentration of titrant Ag using (a) (18), (b) (22) and (c) (23).

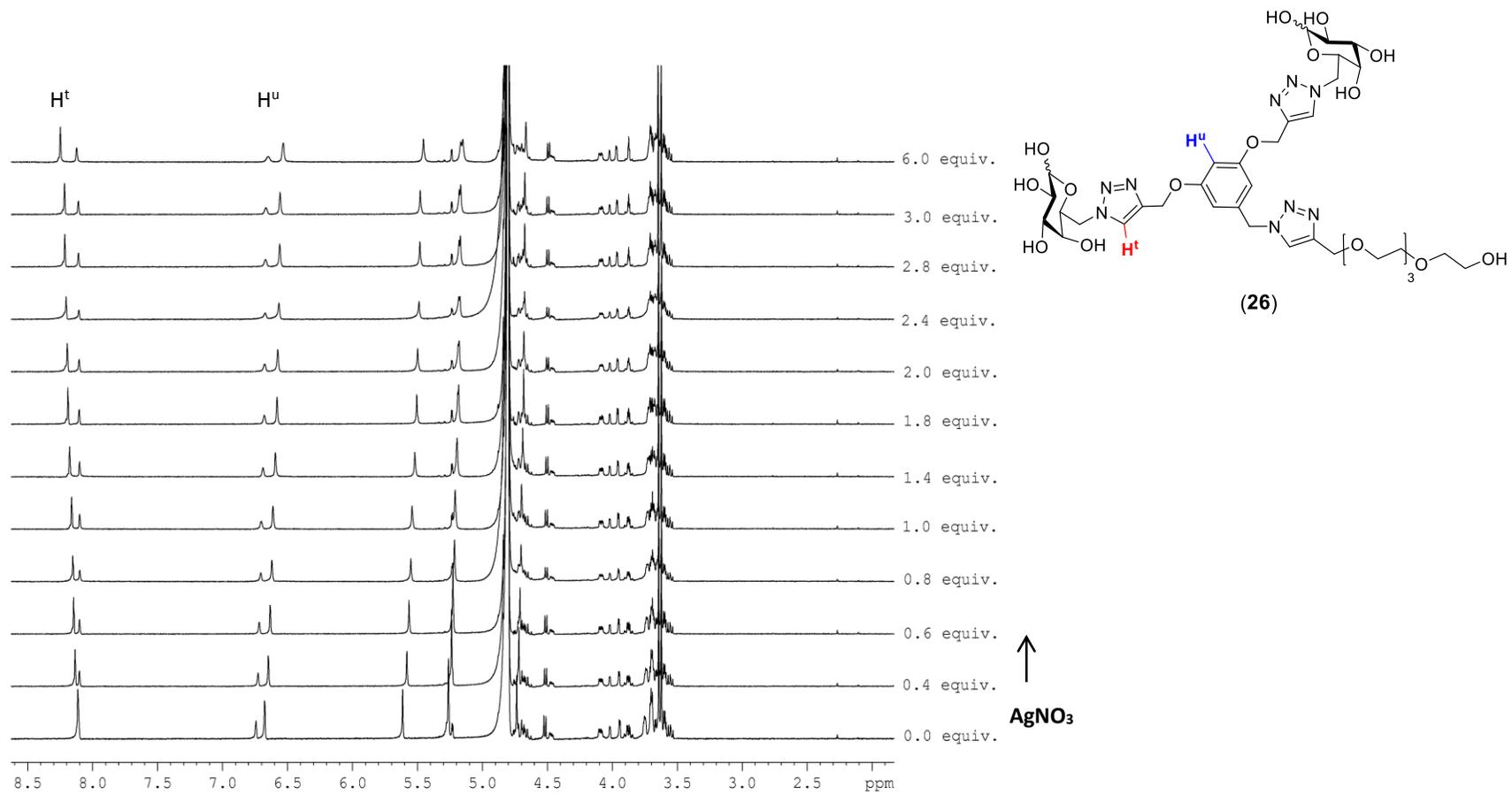
^1H NMR titration studies using ligands (24-26) with AgNO_3



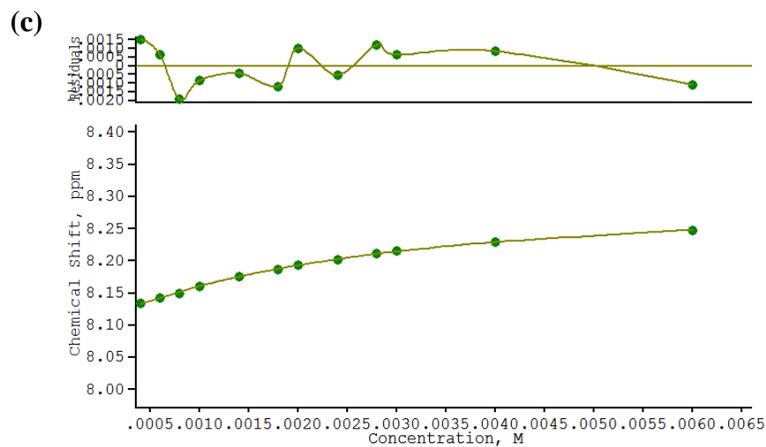
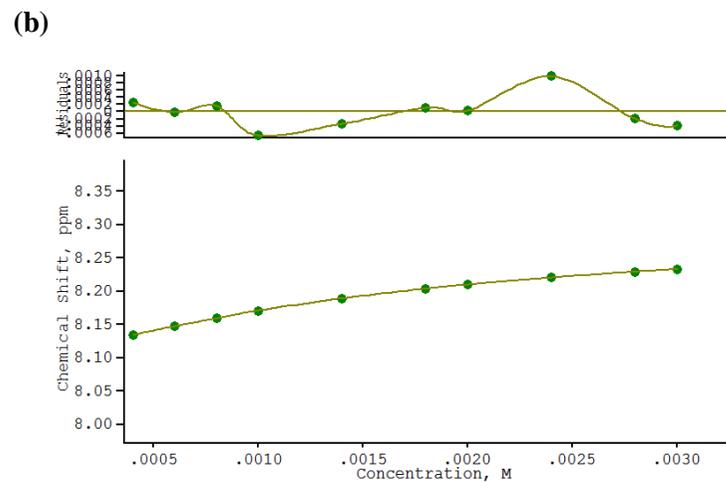
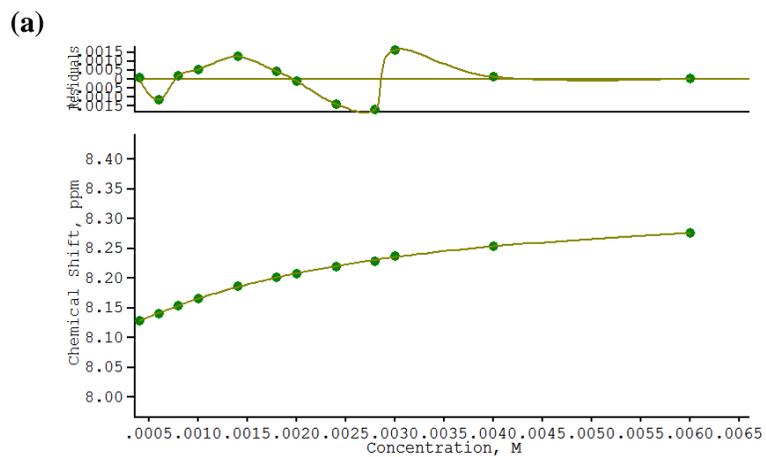
Appendix C9: Stack plot of ^1H -NMR (500 MHz, D_2O) of (24) (stock concentration 2 mM) with an increasing amount of AgNO_3 .



Appendix C10: Stack plot of ^1H -NMR (500 MHz, D_2O) of (25) (stock concentration 2 mM) with an increasing amount of AgNO_3 .



Appendix C11: Stack plot of ^1H -NMR (500 MHz, D_2O) of **(26)** (stock concentration 2 mM) with an increasing amount of AgNO_3 .



Appendix C12: Plots of the experimental points and the calculated best fit line against concentration of titrant Ag using (a) (24), (b) (25) and (c) (26).