Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Supporting Information

Synthesis of Glucosamine-NSAID Bioconjugates

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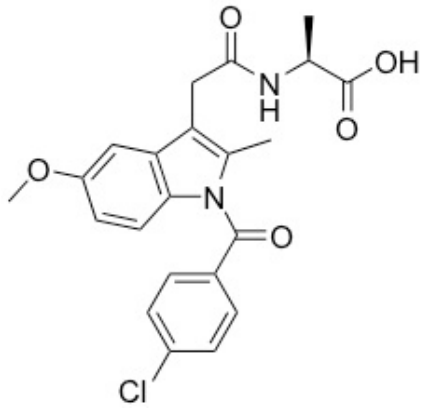
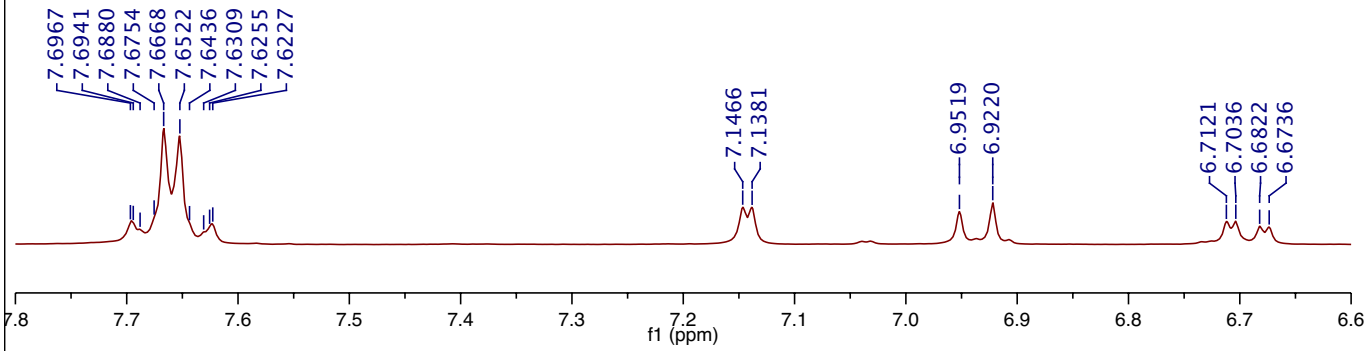
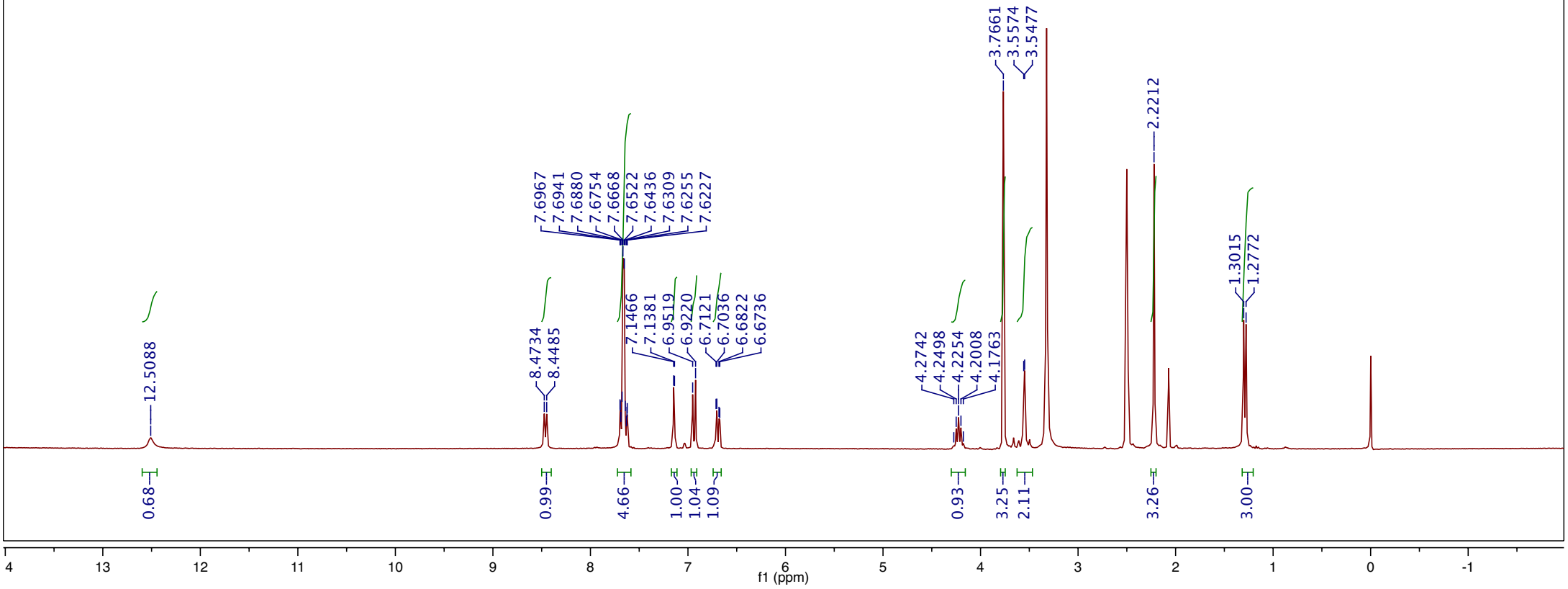
^{1}H and ^{13}C NMR spectra and CHN or ESI data for compounds 3a – d, 3g – h, 7a – h, 6a, 6b, 6b', 6c', 6d', 6e, 6f', 6g and 6h.

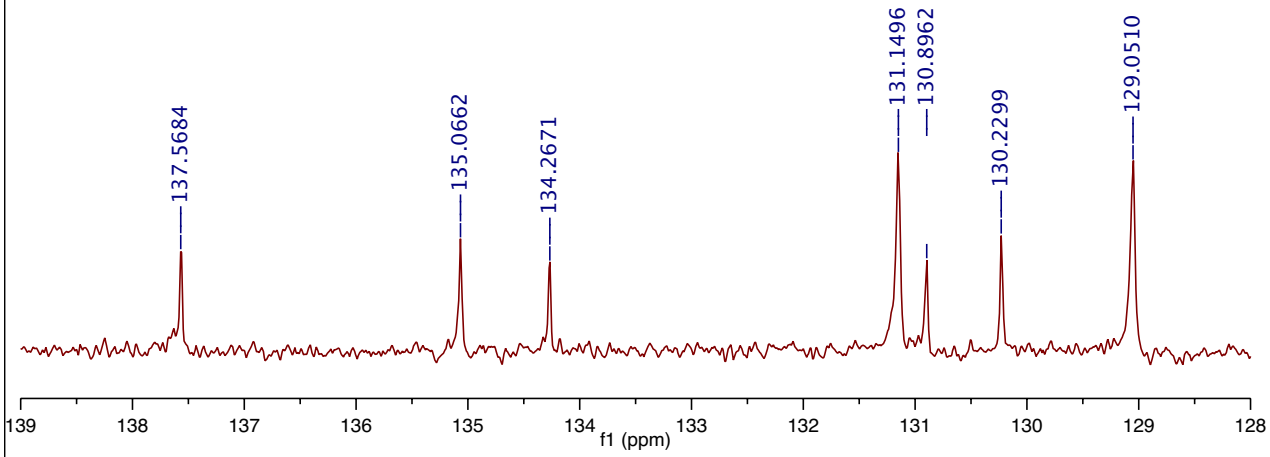
(2-(1-(4-Chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetyl)-L-alanine, 3a

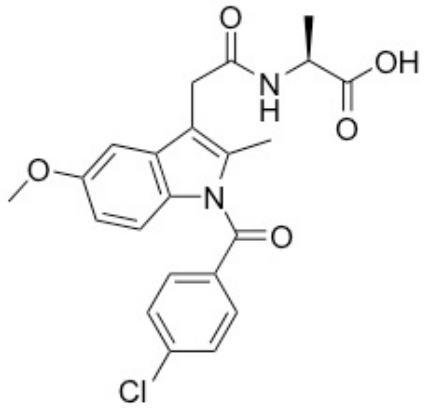


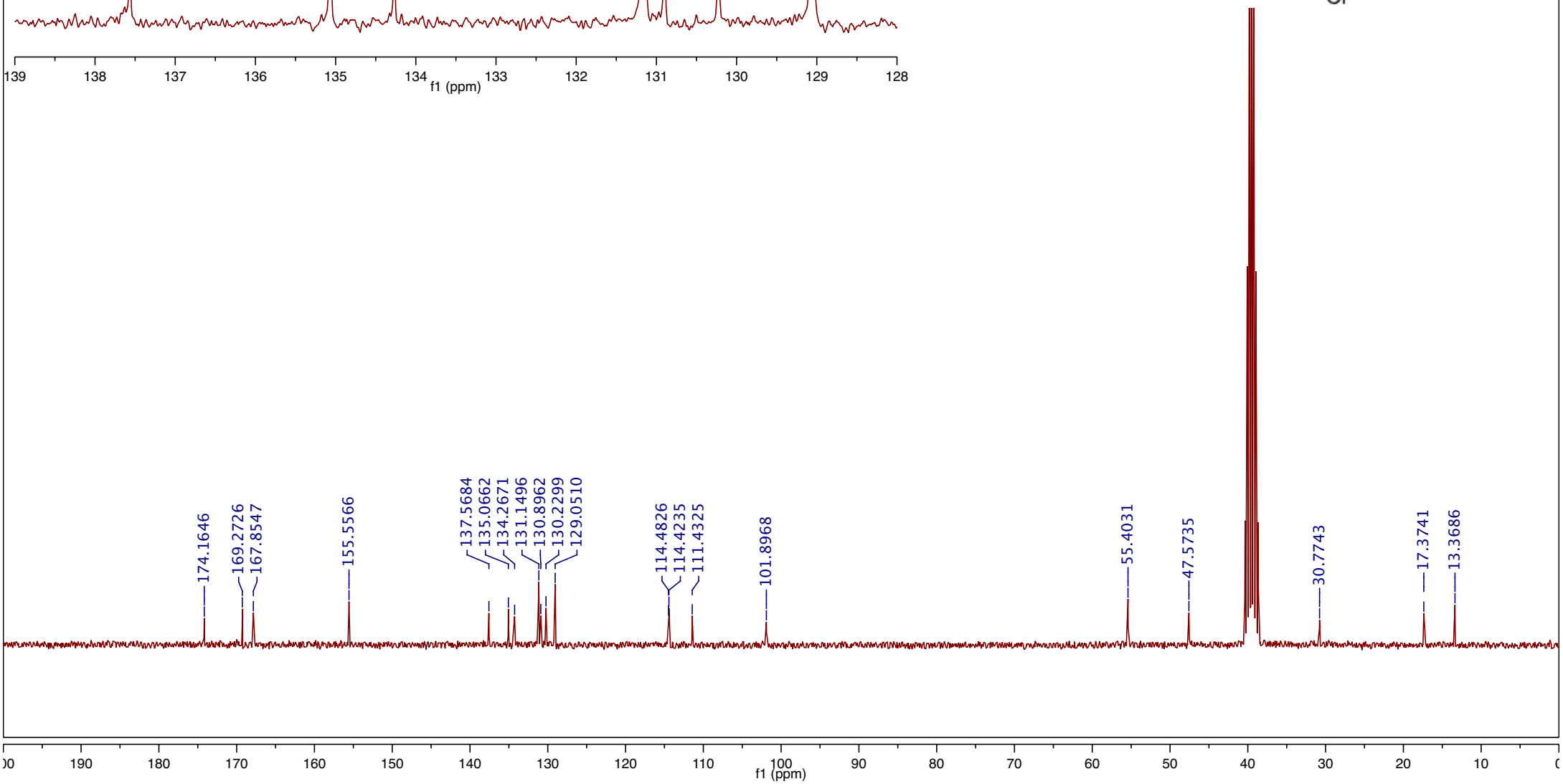




(2-(1-(4-Chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetyl)-L-alanine, 3a





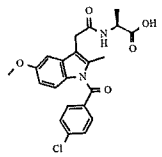


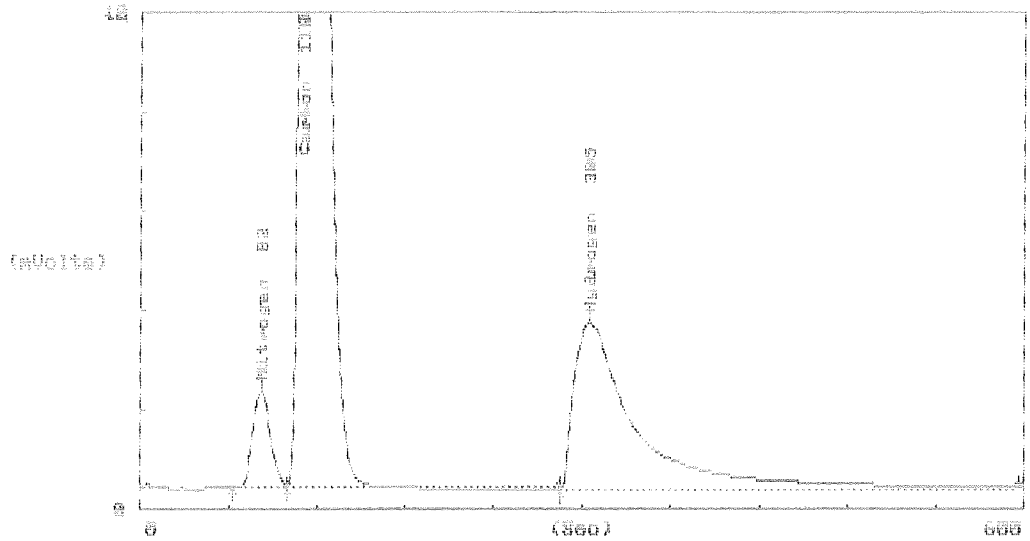
2-(1-(4-Chlorobenzoyl)-5-methoxy-2-methyl-1H-indol-3-yl)acetyl)-L-alanine, 3a

EAGER 200 Stripchart

Sample Ident. : 14 RaJ2-053-1a Filename :280514
Analysed : 07-30-13 11:19:14 Printed :07-30-2013 11:29:16

341-168





EAGER 200 Peak Integration Report

Instrument name : Instrument #1 Bline drift (fV):-2.3
Company Name : U of Florida Operator Ident. : KOU
Analysed : 07-30-13 11:19:14 Printed : 07-30-2013 11:29:17
Sample Ident. : 14 RaJ2-053-1a Filename : 280514
Sample Weight : 2.123 Calc.method: using 'K. Factors'

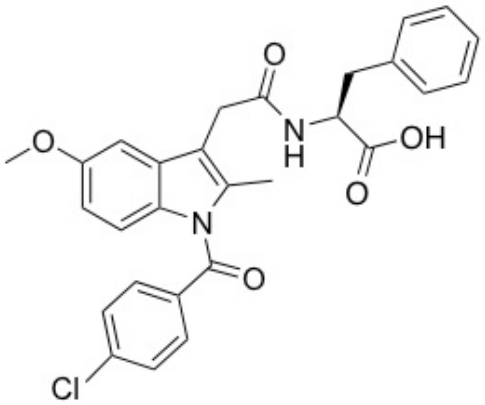
No. (#)	Type (#)	Start (Sec)	End (Sec)	Ret Time (Sec)	Height (fV)	Area (fV*Sec)	Area % (%)	Name
1	FU	63	100	82	1935.2	25840	2.78	Nitrogen
2	FU	100	285	110	50386.0	743785	79.92	Carbon
3	RS	285	597	305	3306.6	161014	17.30	Hydrogen
						930639	100.00	

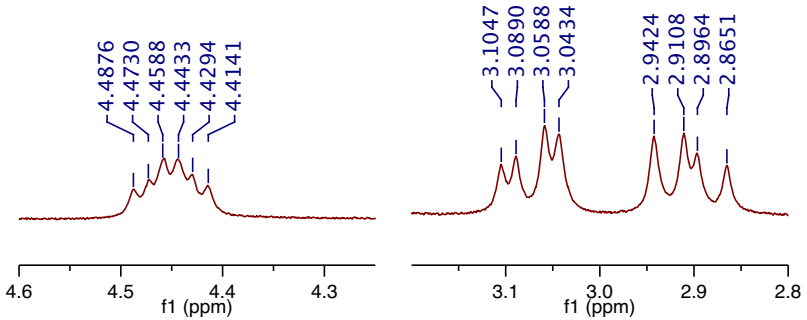
EAGER 200 Unk Report

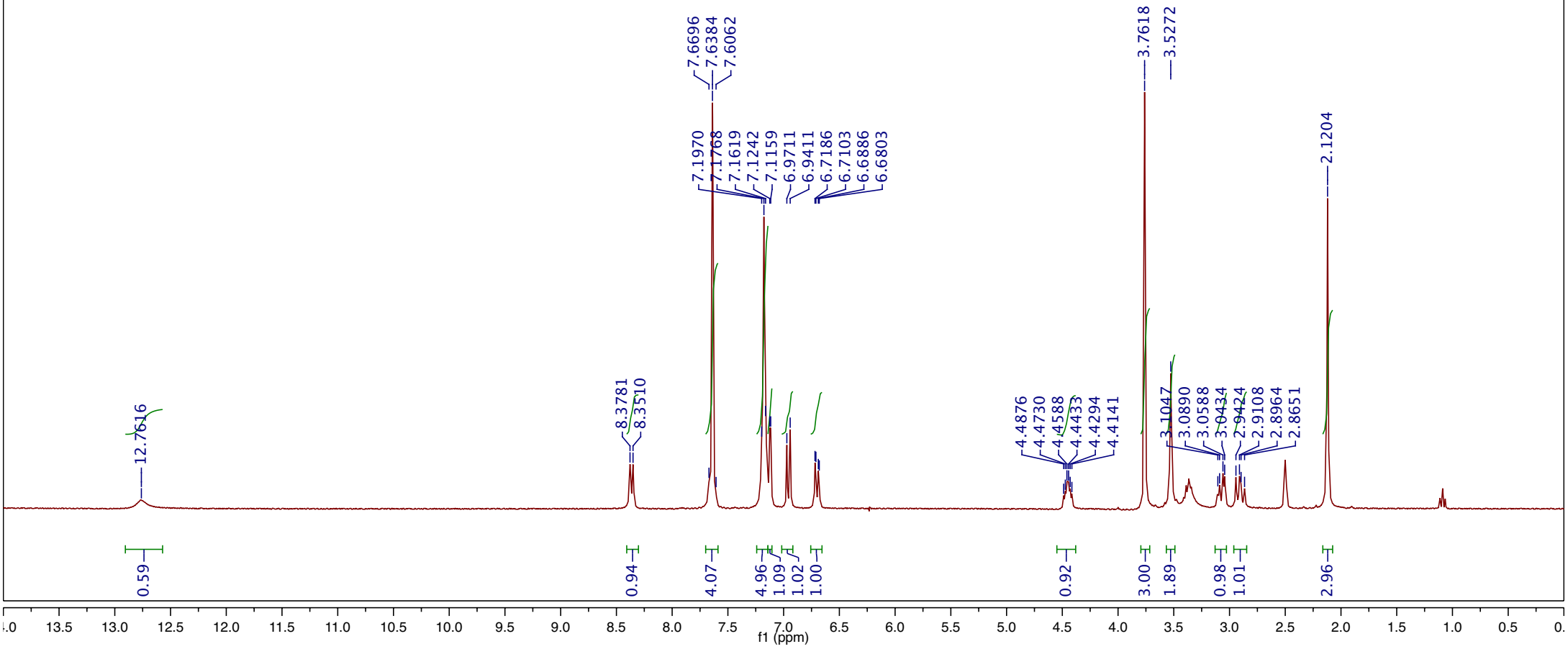
Instrument name : Instrument #1 Bline drift (fV):-2.3
Company Name : U of Florida Operator Ident. : KOU
Analysed : 07-30-13 11:19:14 Printed : 07-30-2013 11:29:17
Sample Ident. : 14 RaJ2-053-1a Filename : 280514
Sample Weight : 2.123 Calc.method: using 'K. Factors'

Pk. (#)	Ret Time (Sec)	Area (fV*Sec)	Element % (%)	Area Ratio	Name
1	82	25840	6.129	.287842E+02	Nitrogen
2	110	743785	61.316	.100000E+01	Carbon
3	305	161014	4.763	.461938E+01	Hydrogen

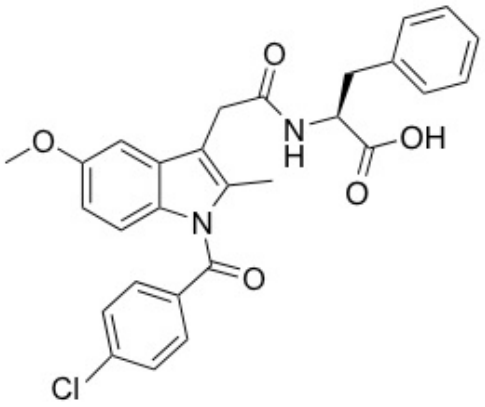
(2-(1-(4-Chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetyl)-L-phenylalanine, 3b

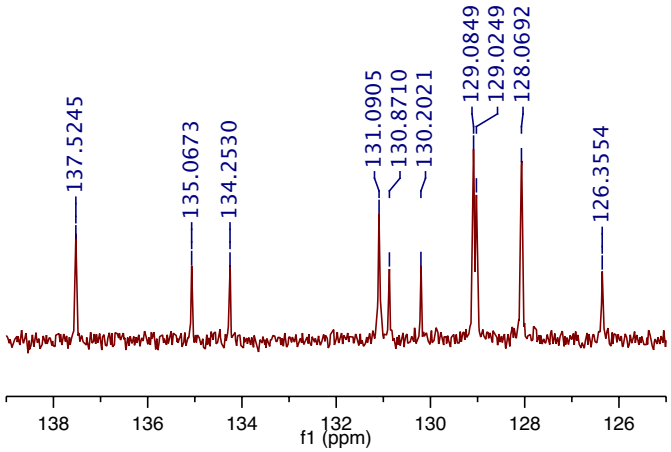


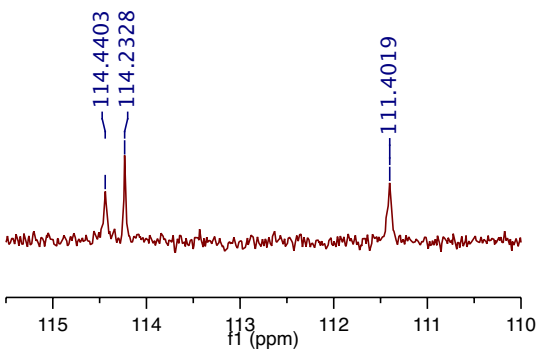


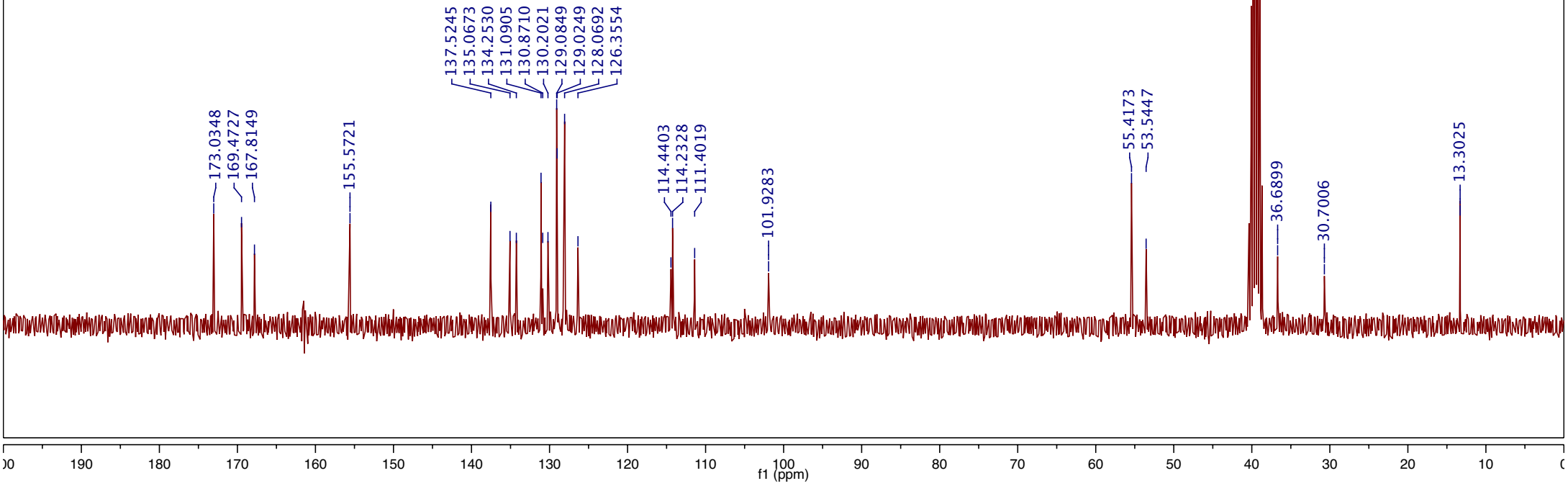


(2-(1-(4-Chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetyl)-L-phenylalanine, 3b







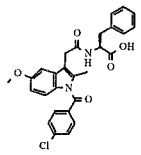


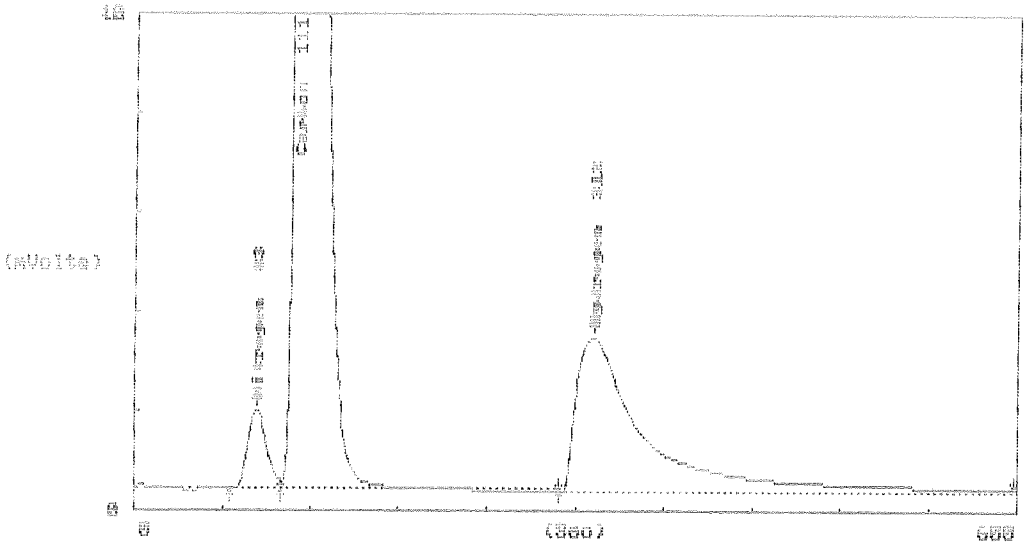
(2-(1-(4-Chlorobenzoyl)-5-methoxy-2-methyl-1H-indol-3-yl)acetyl)-L-phenylalanine, 3b

EAGER 200 Strip chart

Sample Ident. : 12 RaJ2-053-b2 Filename :279712
Analysed : 07-12-13 11:06:25 Printed :07-12-2013 12:55:05

341-164





EAGER 200 Peak Integration Report

Instrument name : Instrument #1 Bline drift (fV): .4
Company Name : U of Florida Operator Ident. : KOU
Analysed : 07-12-13 11:06:25 Printed : 07-12-2013 12:55:05
Sample Ident. : 12 RaJ2-053-b2 Filename : 279712
Sample Weight : 2.095 Calc.method: using 'K. Factors'

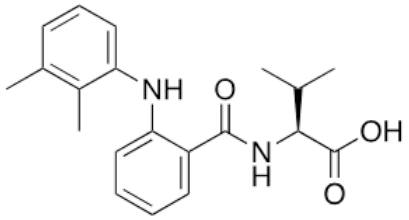
No. (#)	Type (#)	Start (Sec)	End (Sec)	Ret Time (Sec)	Height (fV)	Area (fV*Sec)	Area % (%)	Name
1	FU	65	100	83	1575.9	22295	2.24	Nitrogen
2	FU	100	289	111	51785.5	813969	81.69	Carbon
3	RS	289	598	312	3053.0	160095	16.07	Hydrogen
						996359	100.00	

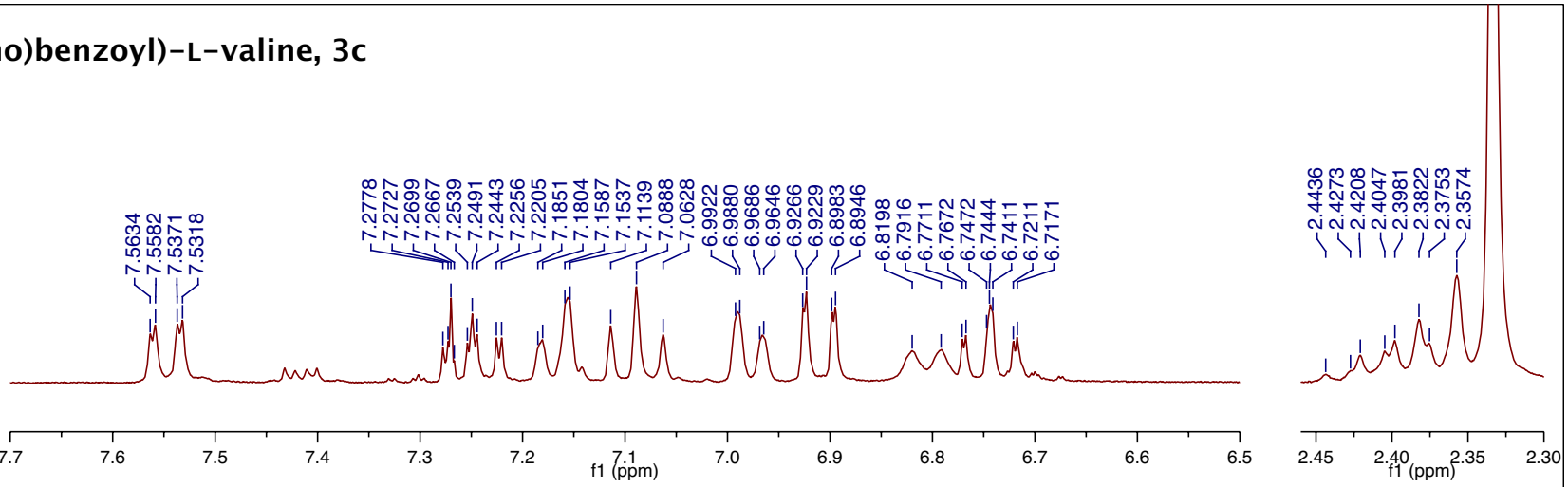
EAGER 200 Unk Report

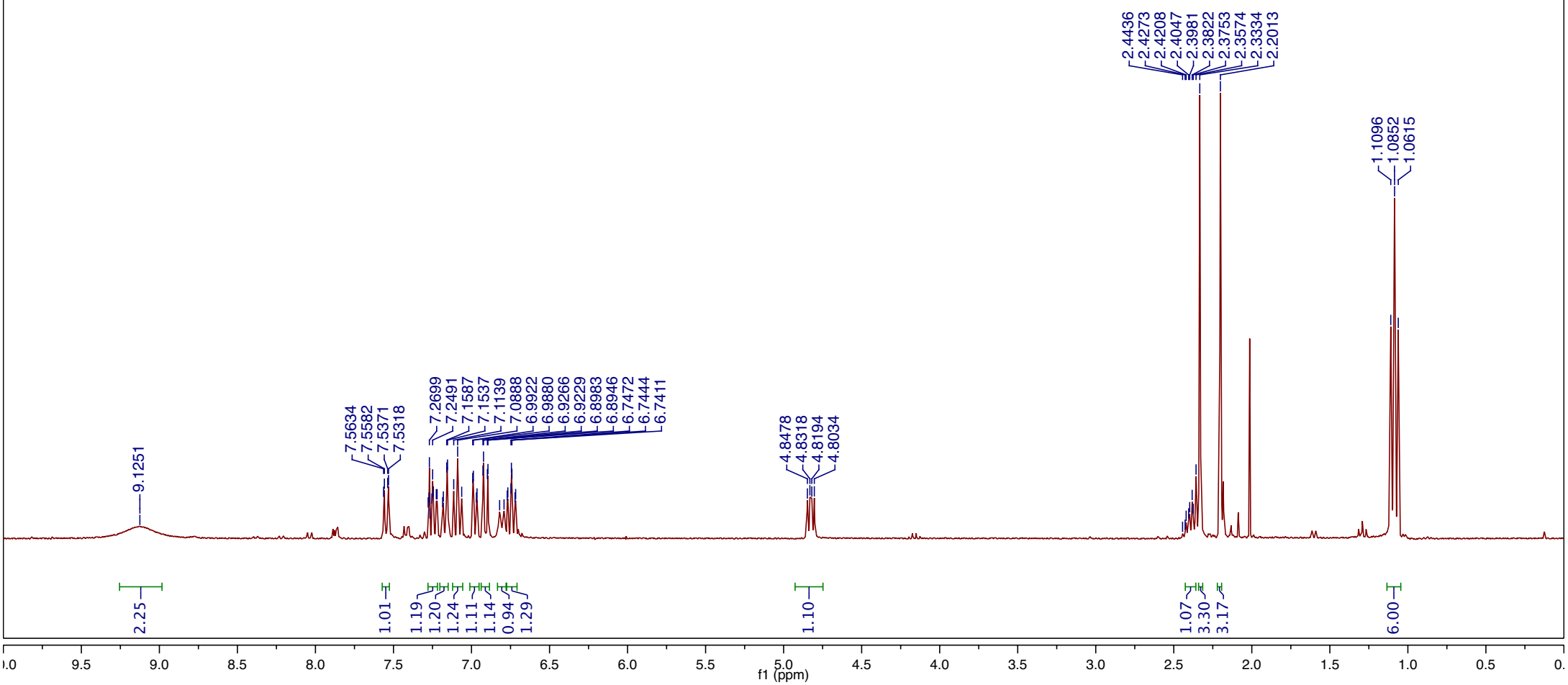
Instrument name : Instrument #1 Bline drift (fV): .4
Company Name : U of Florida Operator Ident. : KOU
Analysed : 07-12-13 11:06:25 Printed : 07-12-2013 12:55:05
Sample Ident. : 12 RaJ2-053-b2 Filename : 279712
Sample Weight : 2.095 Calc.method: using 'K. Factors'

Pk. (#)	Ret Time (Sec)	Area (fV*Sec)	Element % (%)	Area Ratio	Name
1	83	22295	5.212	.365097E+02	Nitrogen
2	111	813969	66.298	.100000E+01	Carbon
3	312	160095	4.926	.508427E+01	Hydrogen

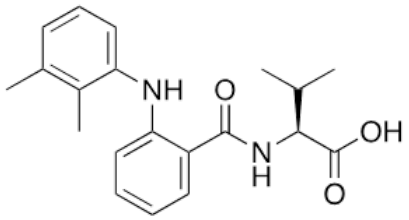
(2-((2,3-Dimethylphenyl)amino)benzoyl)-L-valine, 3c

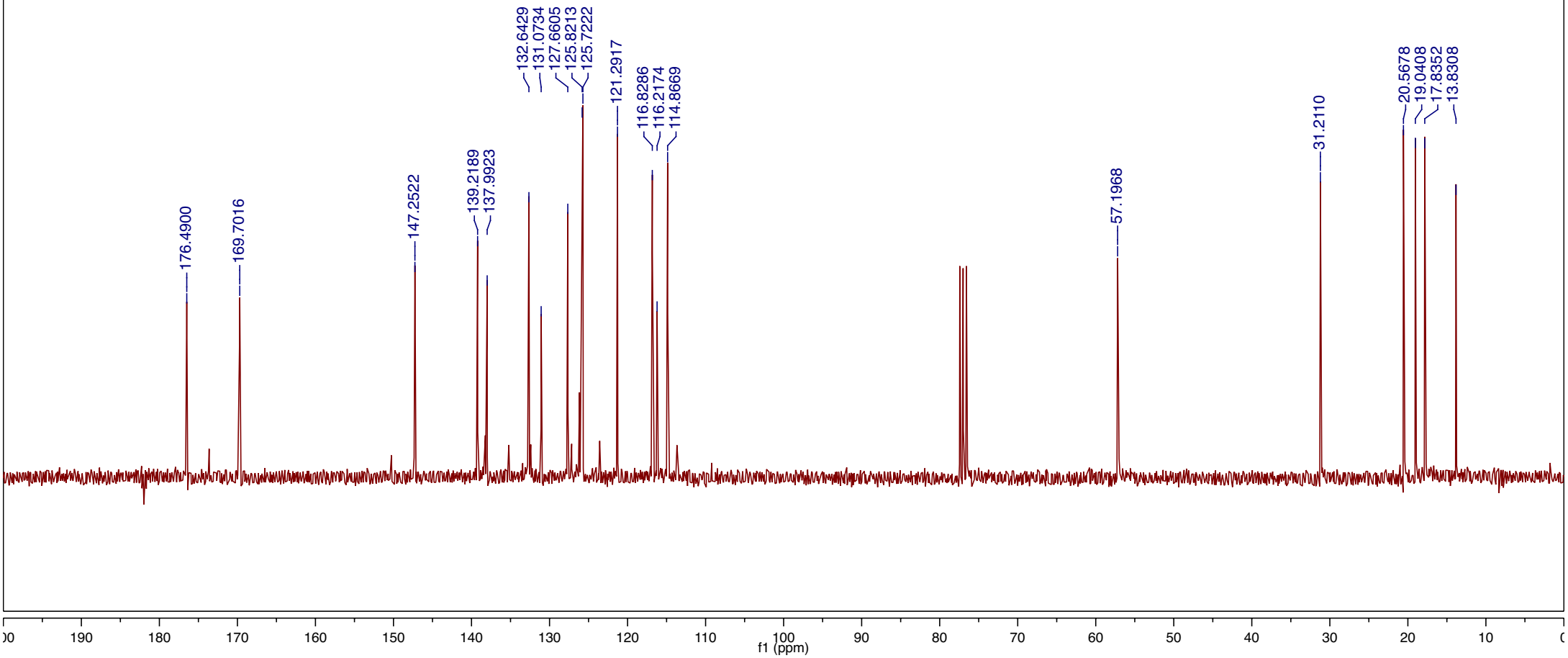




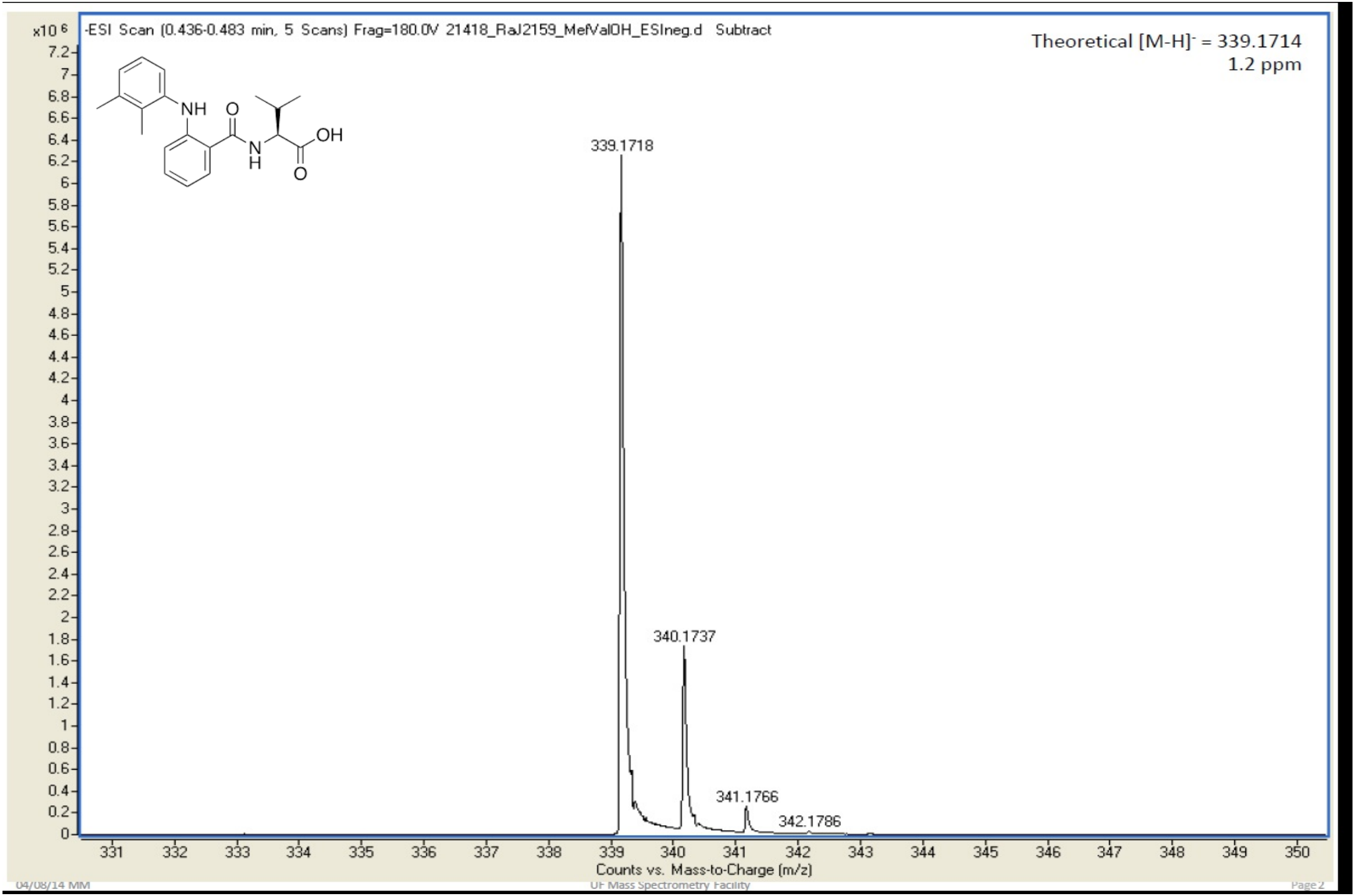


(2-((2,3-Dimethylphenyl)amino)benzoyl)-L-valine, 3c

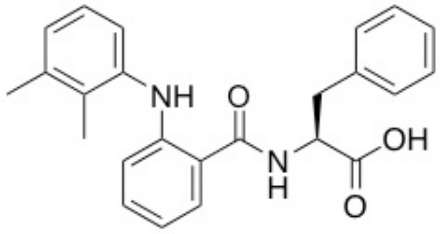


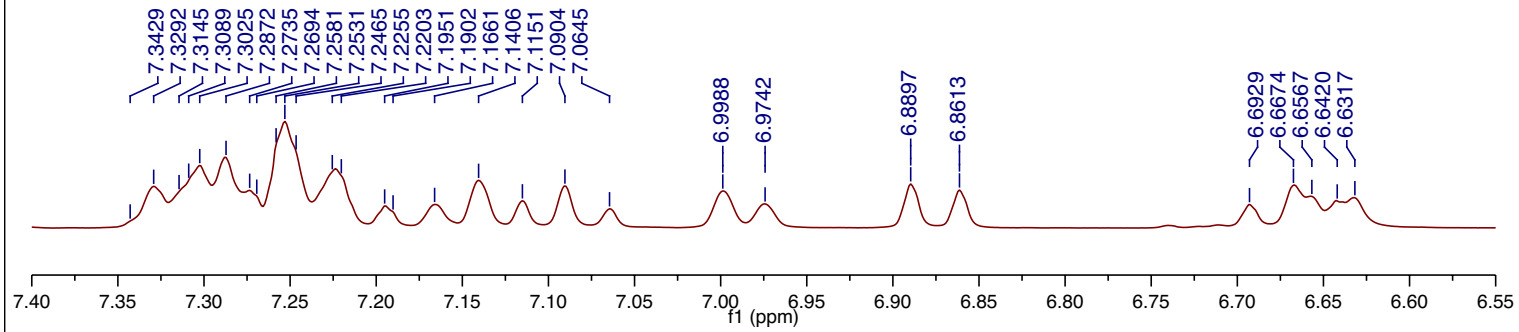


(2-((2,3-Dimethylphenyl)amino)benzoyl)-L-valine, 3c

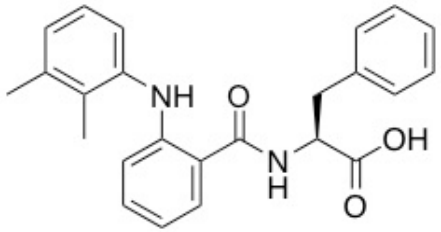


(2-((2,3-Dimethylphenyl)amino)benzoyl)-L-phenylalanine, 3d

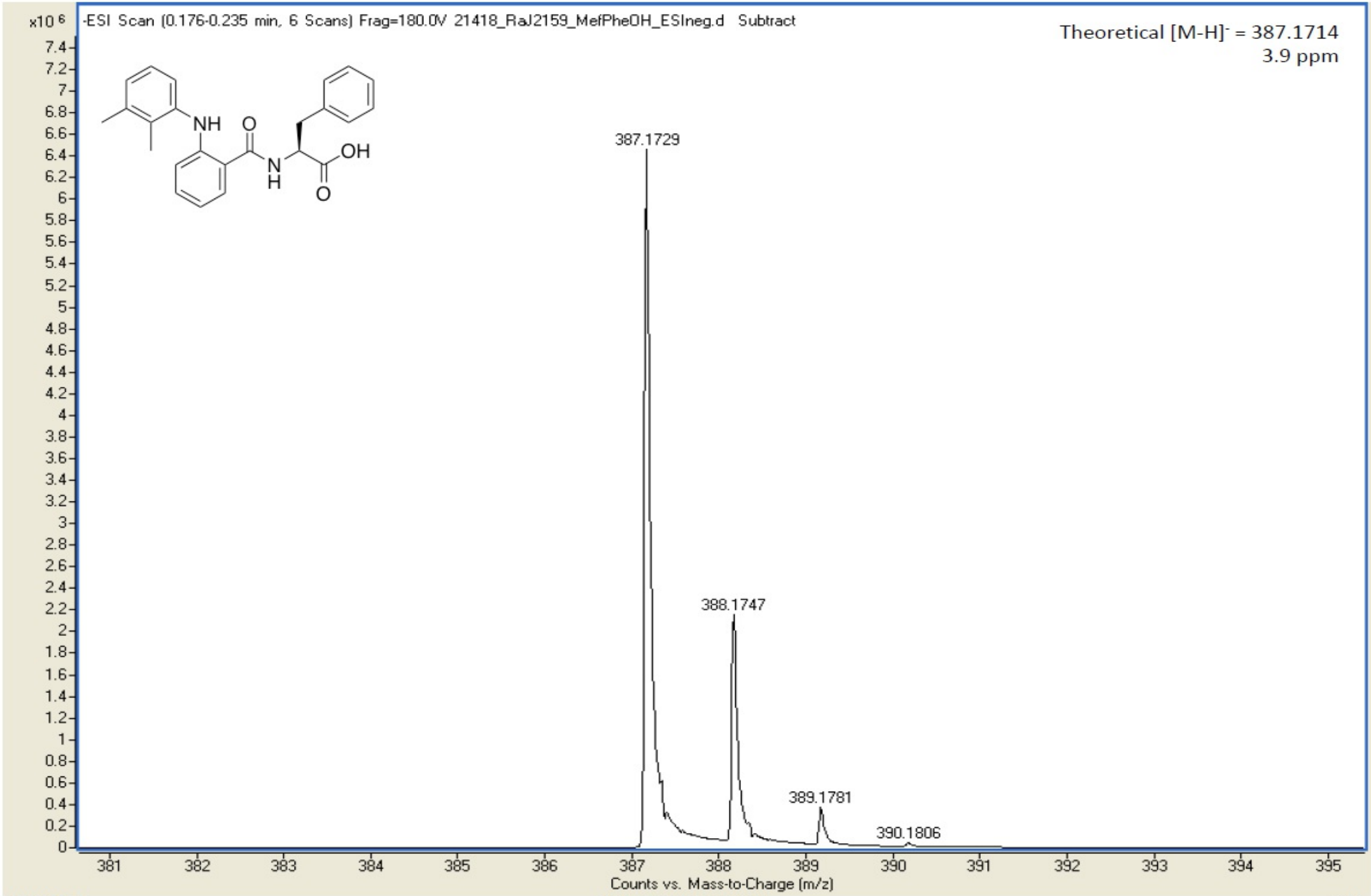




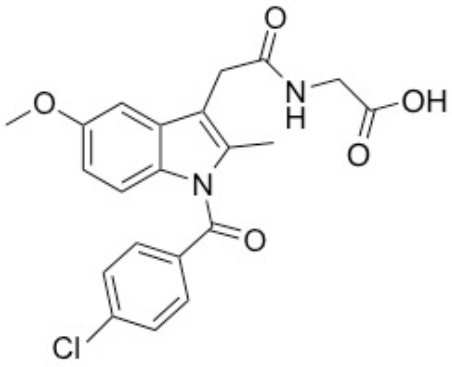
(2-((2,3-Dimethylphenyl)amino)benzoyl)-L-phenylalanine, 3d

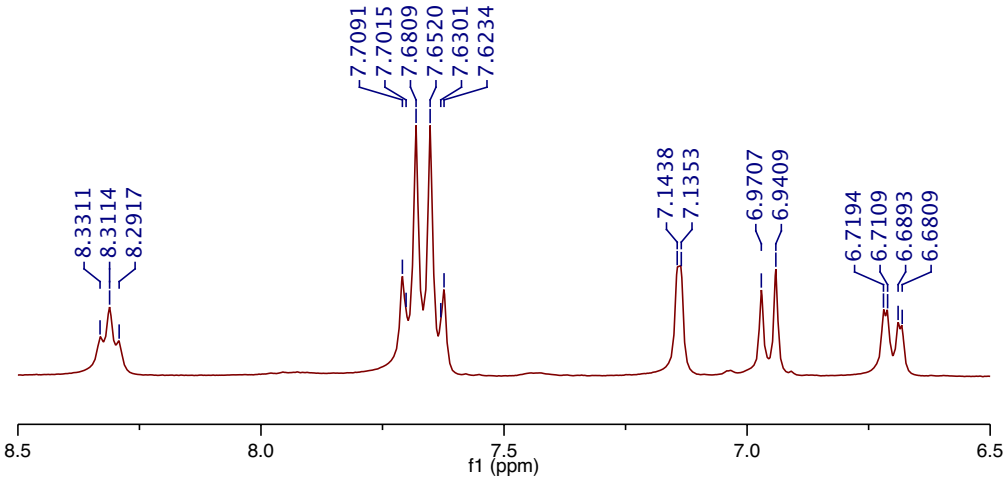


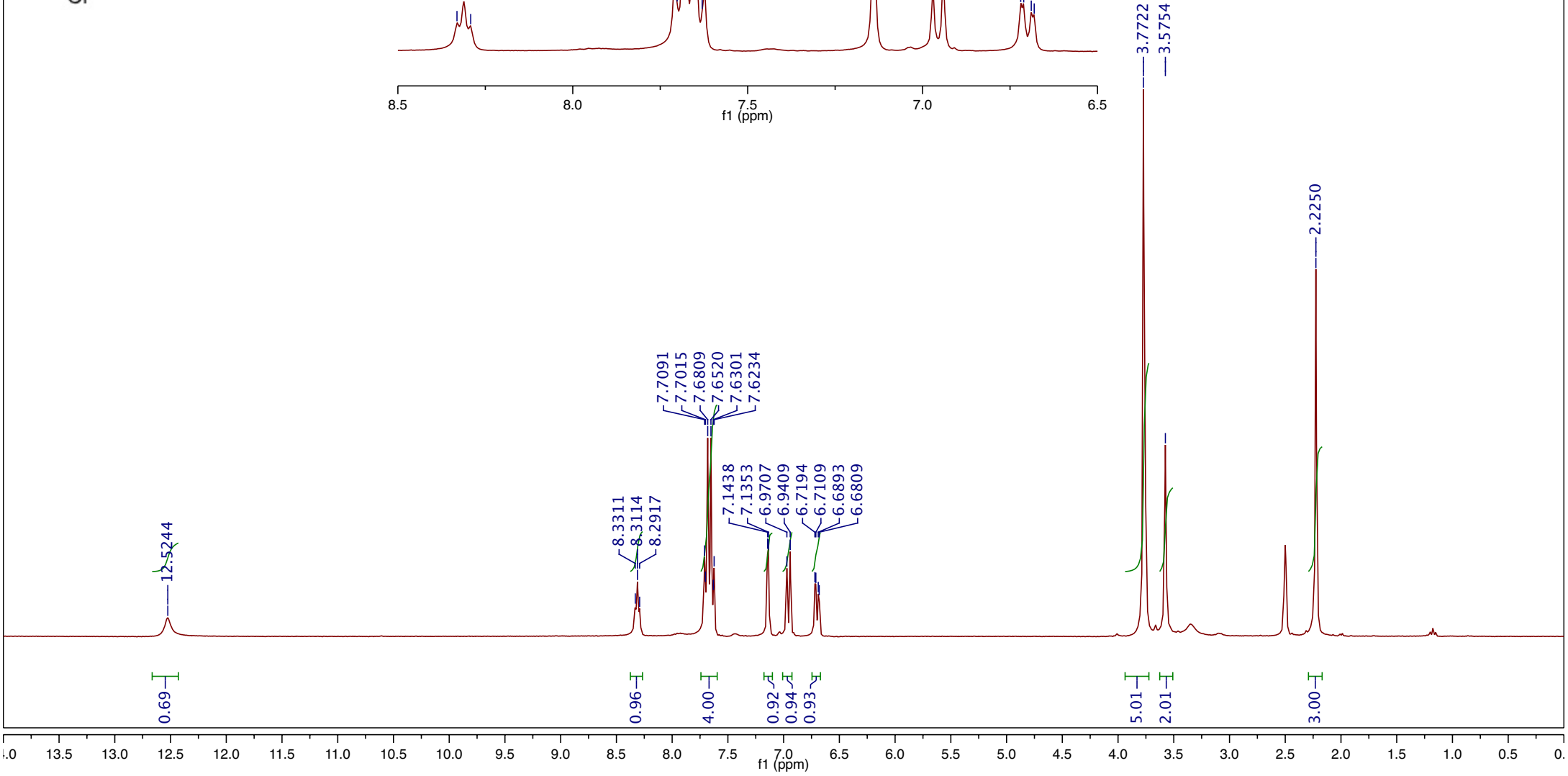
(2-((2,3-Dimethylphenyl)amino)benzoyl)-L-phenylalanine, 3d



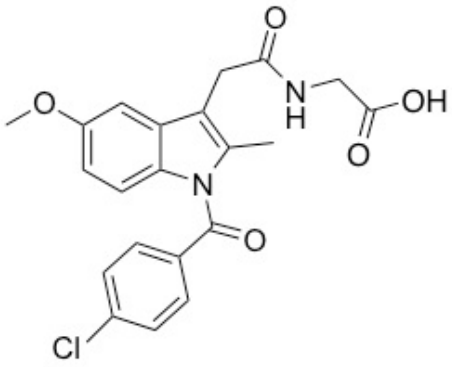
(2-(1-(4-Chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetyl)-glycine, 3g

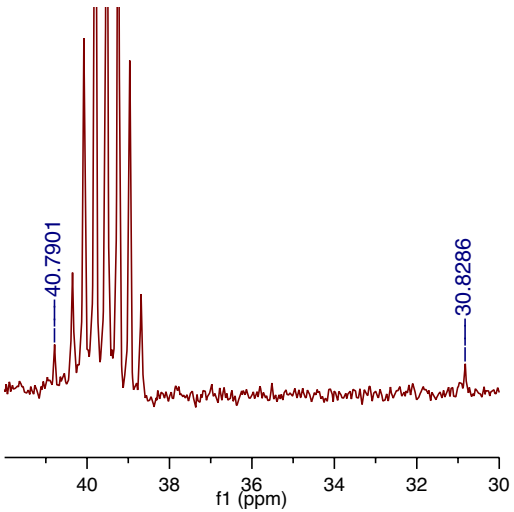






(2-(1-(4-Chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetyl)-glycine, 3g





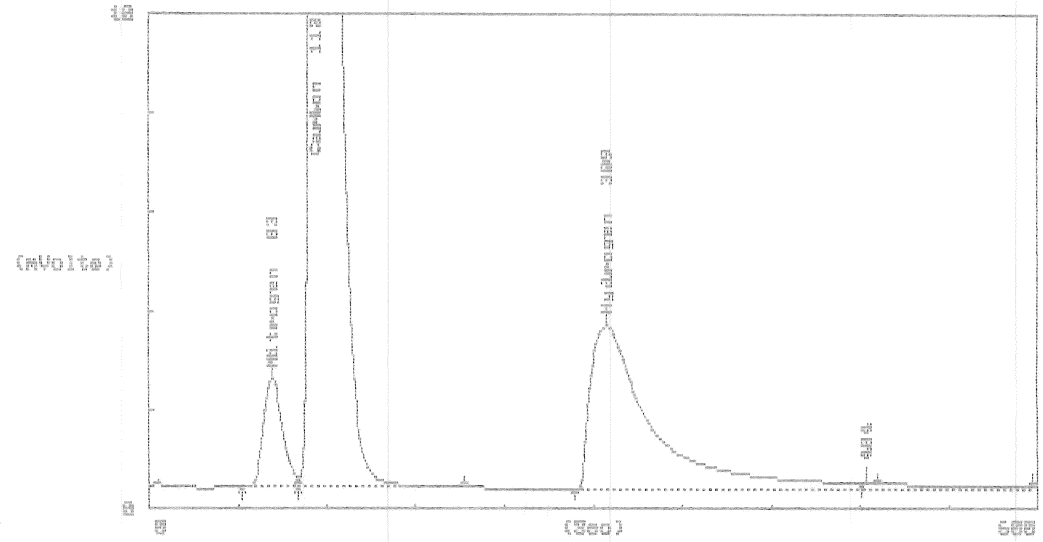
171.2931
169.8561
167.8310
155.5422
137.5458
135.1929
134.2330
131.1593
130.8259
130.2480
129.0134
114.4705
114.0776
111.4188
101.8639
55.3782
40.7901
30.8286
13.3677

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

(2-(1-(4-Chlorobenzoyl)-5-methoxy-2-methyl-1H-indol-3-yl)acetyl)-glycine, 3g

EAGER 200 Stripchart

Sample Ident. : 13 Indo-Gly-OH Filename :288313
Analysed : 07-14-14 09:53:05 Printed :07-14-2014 10:03:07



EAGER 200 Peak Integration Report

Instrument name : Instrument #1 Bline drift (fV):-3.4
Company Name : U of Florida Operator Ident. : KOU
Analysed : 07-14-14 09:53:05 Printed : 07-14-2014 10:03:08
Sample Ident. : 13 Indo-Gly-OH Filename : 288313
Sample Weight : 2.153 Calc.method: using 'K. Factors'

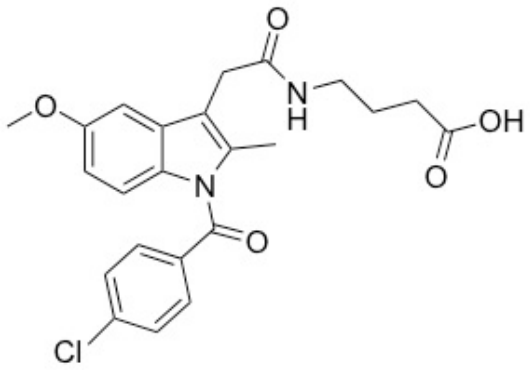
No. (#)	Type (#)	Start (Sec)	End (Sec)	Ret Time (Sec)	Height (fV)	Area (fV*Sec)	Area % (%)	Name
1	FU	63	101	83	2205.5	31401	3.26	Nitrogen
2	FU	101	213	112	49823.9	772008	80.04	Carbon
3	TL	288	597	309	3278.6	161074	16.70	Hydrogen
4	CR	481	492	484	1.2	12	0.00	
						964496	100.00	

EAGER 200 Unk Report

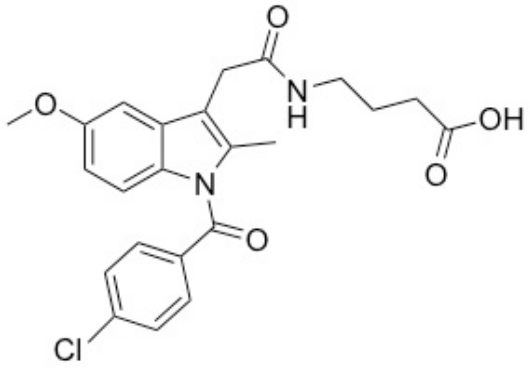
Instrument name : Instrument #1 Bline drift (fV):-3.4
Company Name : U of Florida Operator Ident. : KOU
Analysed : 07-14-14 09:53:05 Printed : 07-14-2014 10:03:08
Sample Ident. : 13 Indo-Gly-OH Filename : 288313
Sample Weight : 2.153 Calc.method: using 'K. Factors'

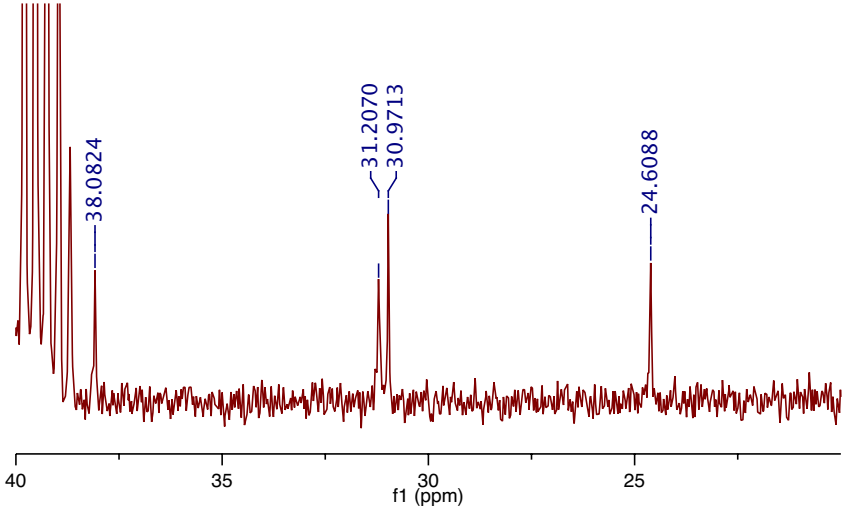
Pk. (#)	Ret Time (Sec)	Area (fV*Sec)	Element % (%)	Area Ratio	Name
1	83	31401	6.954	.245853E+02	Nitrogen
2	112	772008	60.594	.100000E+01	Carbon
3	309	161074	4.510	.479287E+01	Hydrogen

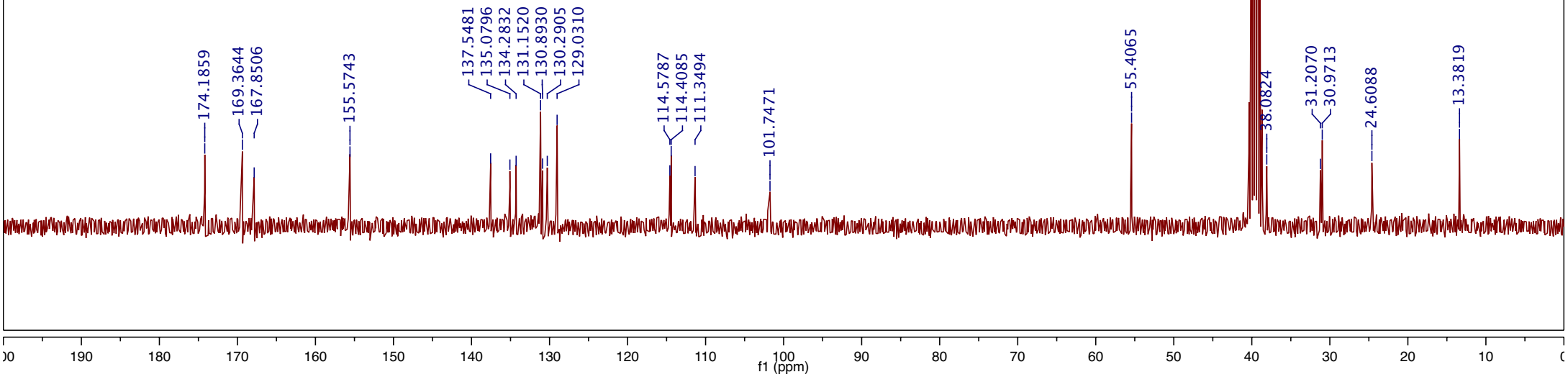
(2-(1-(4-Chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetyl)-γ-aminobutyric acid, 3h



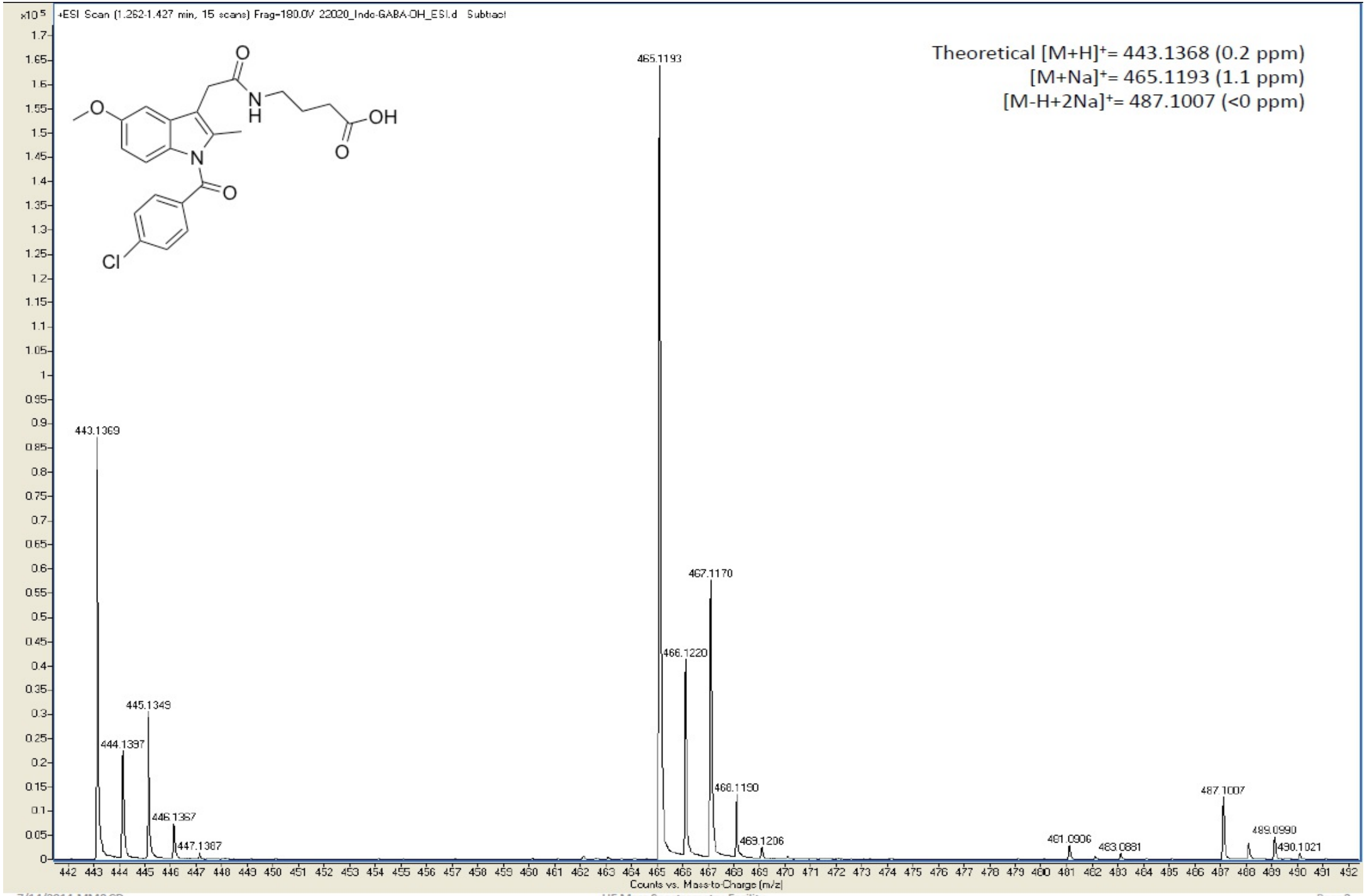
(2-(1-(4-Chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetyl)-γ-aminobutyric acid, 3h



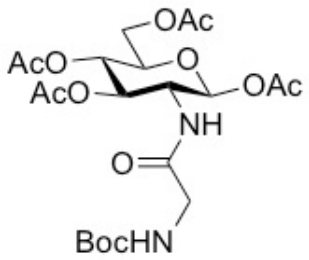




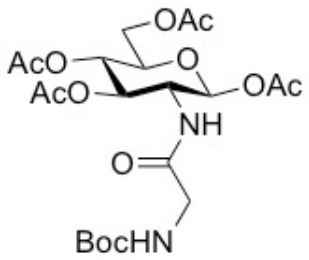
(2-(1-(4-Chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetyl)-γ-aminobutyric acid, 3h

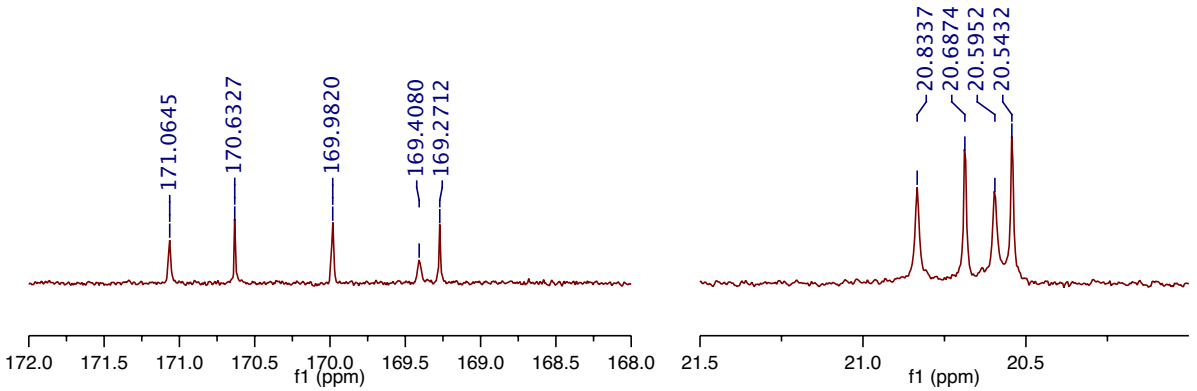


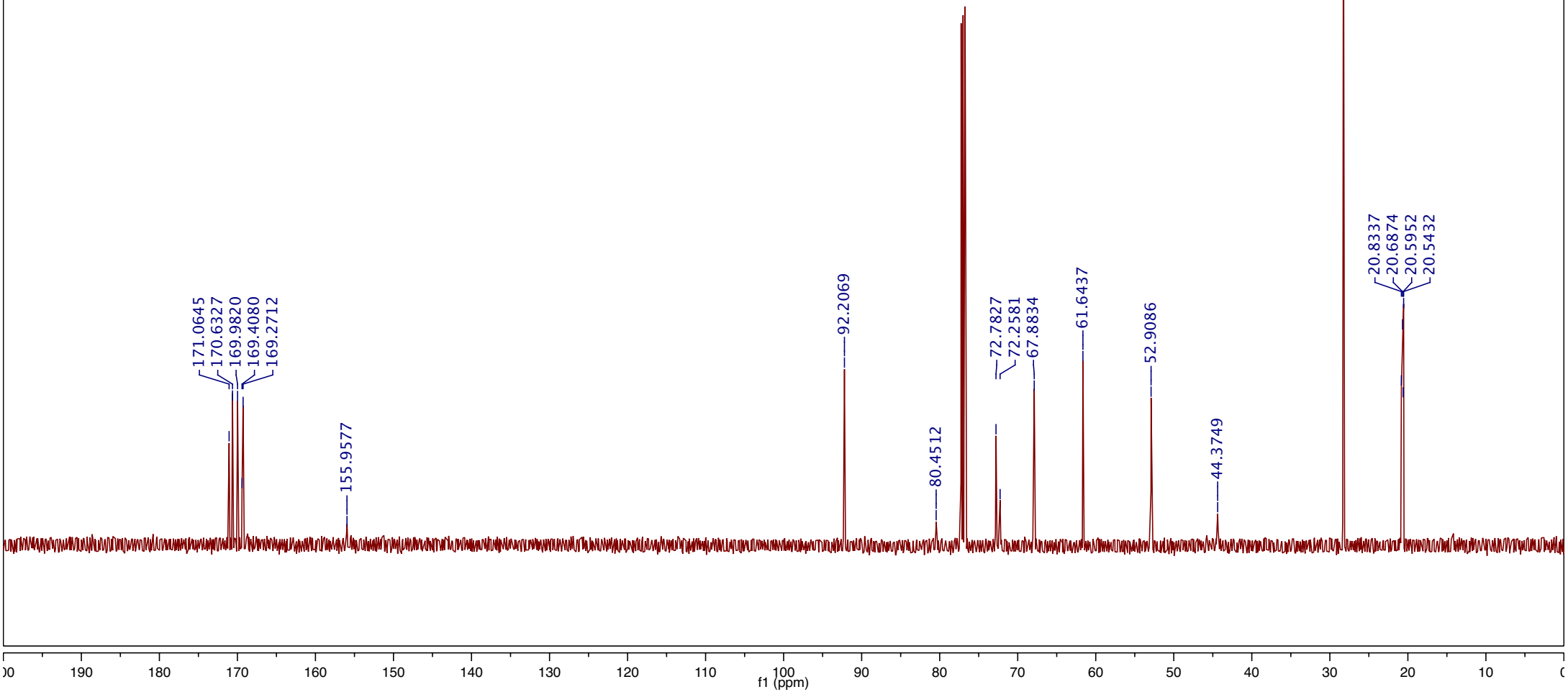
1,3,4,6-Tetra-*O*-acetyl-2-[(*N*-(*tert*-butoxycarbonyl)glycyl)-amino]-2-deoxy-β-D-glucopyranose, 7a



1,3,4,6-Tetra-*O*-acetyl-2-[(*N*-(*tert*-butoxycarbonyl)glycyl)-amino]-2-deoxy-β-D-glucopyranose, 7a



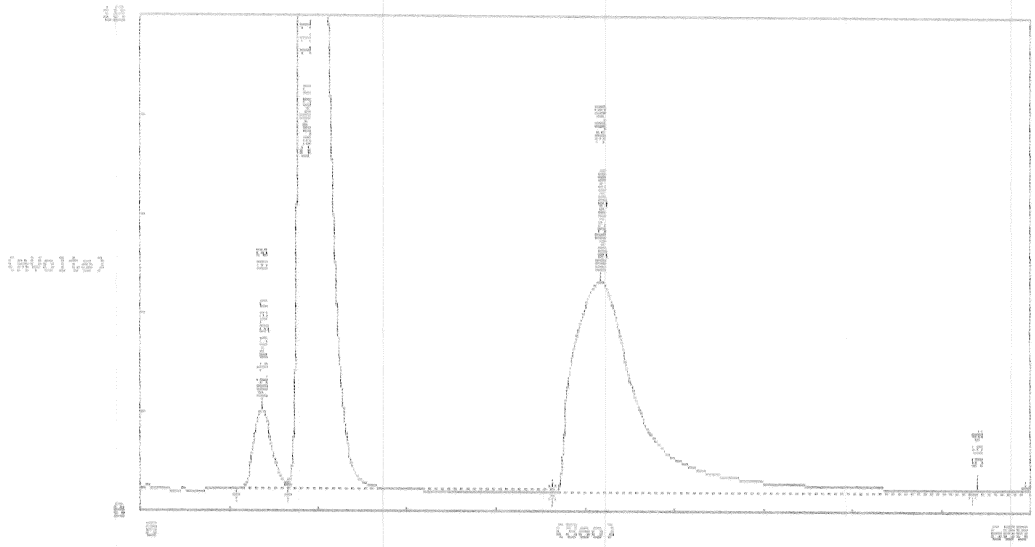




1,3,4,6-Tetra-O-acetyl-2-[(N-(tert-butoxycarbonyl)glycyl)-amino]-2-deoxy-β-D-glucopyranose, 7a 342 – 081

EAGER 200 Stripchart

Sample Ident. : 14 RaJ2159-20 Filename :285114
Analysed : 02-06-14 11:31:04 Printed :02-06-2014 11:41:07



EAGER 200 Peak Integration Report

Instrument name : Instrument #1 Bline drift (fV): 3.8
Company Name : U of Florida Operator Ident. : KOU
Analysed : 02-06-14 11:31:04 Printed : 02-06-2014 11:41:07
Sample Ident. : 14 RaJ2159-20 Filename : 285114
Sample Weight : 2.142 Calc.method: using 'K. Factors'

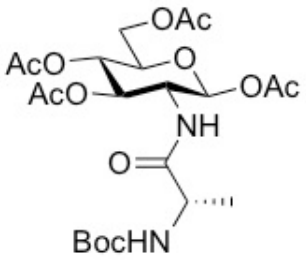
No. (#)	Type (#)	Start (Sec)	End (Sec)	Ret Time (Sec)	Height (fV)	Area (fV*Sec)	Area % (%)	Name
1	FU	65	99	82	1624.6	22296	2.58	Nitrogen
2	FU	99	278	111	41268.1	618263	71.64	Carbon
3	TL	278	597	310	4218.9	222466	25.78	Hydrogen
4	CR	561	597	564	1.6	23	0.00	
						863048	100.00	

EAGER 200 Unk Report

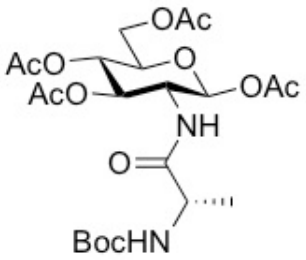
Instrument name : Instrument #1 Bline drift (fV): 3.8
Company Name : U of Florida Operator Ident. : KOU
Analysed : 02-06-14 11:31:04 Printed : 02-06-2014 11:41:07
Sample Ident. : 14 RaJ2159-20 Filename : 285114
Sample Weight : 2.142 Calc.method: using 'K. Factors'

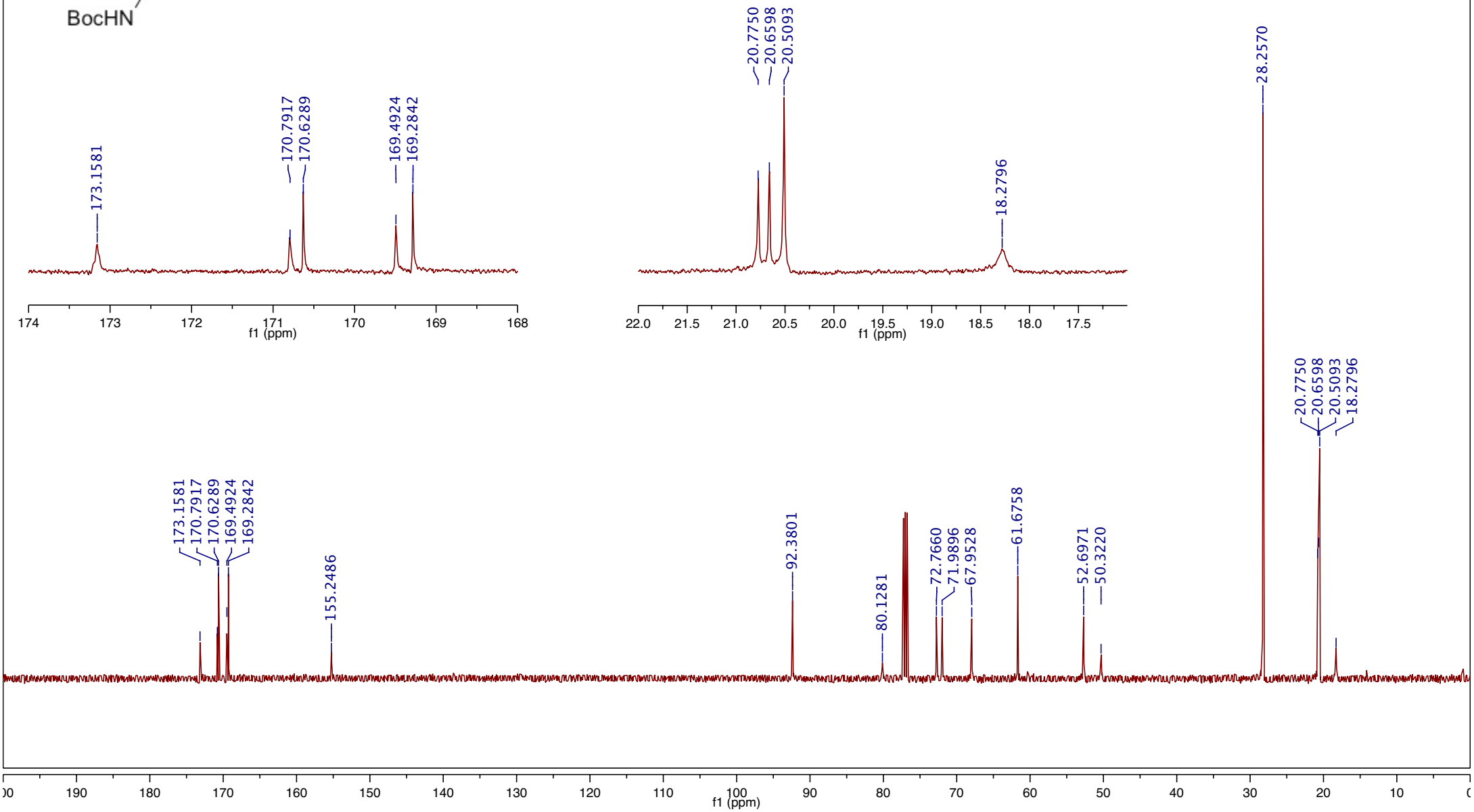
Pk. (#)	Ret Time (Sec)	Area (fV*Sec)	Element % (%)	Area Ratio	Name
1	82	22296	5.320	.277292E+02	Nitrogen
2	111	618263	49.997	.100000E+01	Carbon
3	310	222466	6.649	.277914E+01	Hydrogen

1,3,4,6-Tetra-*O*-acetyl-2-[(*N*-(*tert*-butoxycarbonyl)-L-alanyl)-amino]-2-deoxy-β-D-glucopyranose, 7b



1,3,4,6-Tetra-*O*-acetyl-2-[(*N*-(*tert*-butoxycarbonyl)-L-alanyl)-amino]-2-deoxy-β-D-glucopyranose, 7b



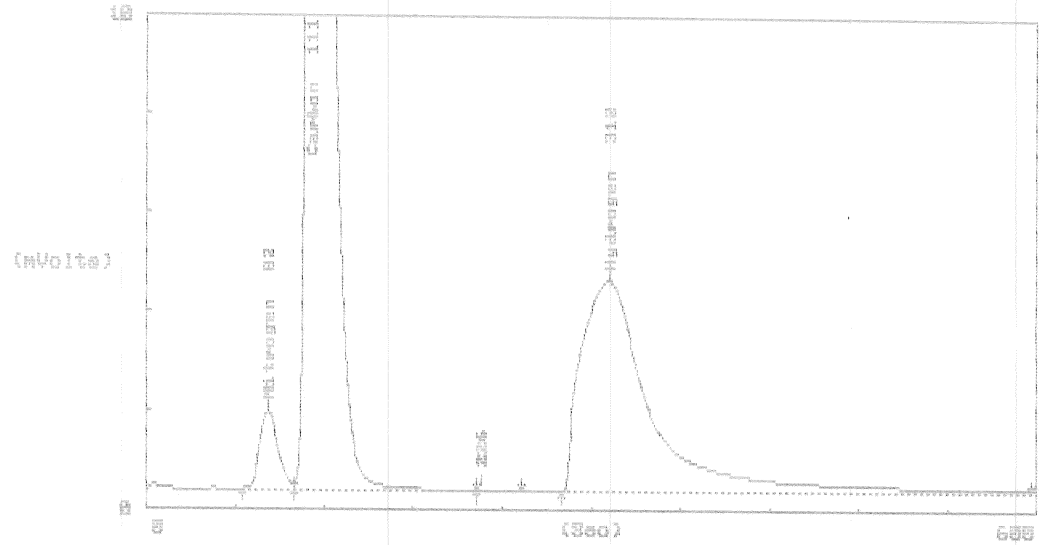


1,3,4,6-Tetra-O-acetyl-2-[(N-(tert-butoxycarbonyl)-L-alanyl)-amino]-2-deoxy-β-D-glucopyranose, 7b

342-082

EAGER 200 Strip chart

Sample Ident. : 10 RaJ2159-22 Filename :285110
Analysed : 02-06-14 10:50:53 Printed :02-06-2014 11:00:56



EAGER 200 Peak Integration Report

Instrument name : Instrument #1 Bline drift (fV): 19.4
Company Name : U of Florida Operator Ident. : KOU
Analysed : 02-06-14 10:50:53 Printed : 02-06-2014 11:00:56
Sample Ident. : 10 RaJ2159-22 Filename : 285110
Sample Weight : 2.139 Calc.method: using 'K. Factors'

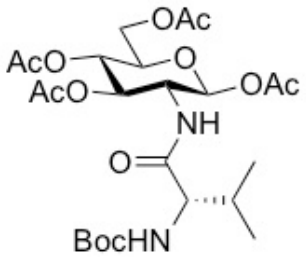
No. (#)	Type (#)	Start (Sec)	End (Sec)	Ret Time (Sec)	Height (fV)	Area (fV*Sec)	Area % (%)	Name
1	FU	65	99	82	1568.8	21271	2.43	Nitrogen
2	FU	99	223	111	41984.5	627630	71.61	Carbon
3	RS	223	253	226	2.4	30	0.00	
4	RS	280	597	312	4207.0	227505	25.96	Hydrogen
						876437	100.00	

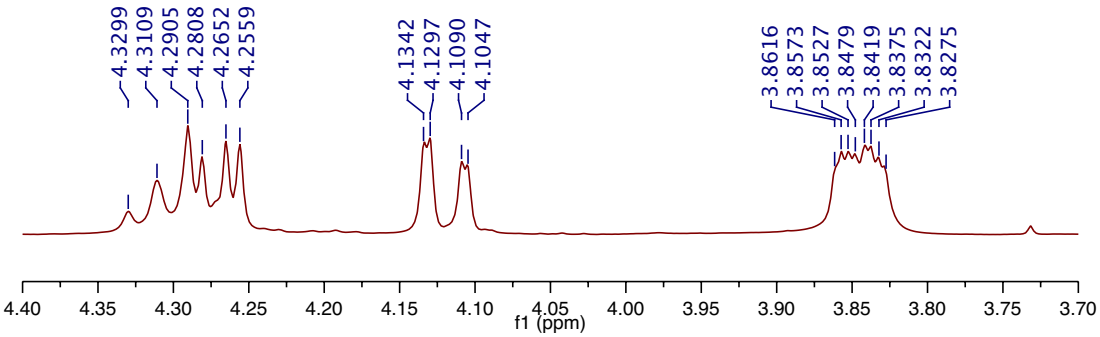
EAGER 200 Unk Report

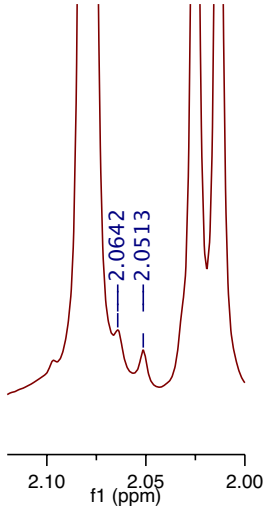
Instrument name : Instrument #1 Bline drift (fV): 19.4
Company Name : U of Florida Operator Ident. : KOU
Analysed : 02-06-14 10:50:53 Printed : 02-06-2014 11:00:56
Sample Ident. : 10 RaJ2159-22 Filename : 285110
Sample Weight : 2.139 Calc.method: using 'K. Factors'

Pk. (#)	Ret Time (Sec)	Area (fV*Sec)	Element % (%)	Area Ratio	Name
1	82	21271	5.082	.295059E+02	Nitrogen
2	111	627630	50.826	.100000E+01	Carbon
4	312	227505	6.809	.275875E+01	Hydrogen

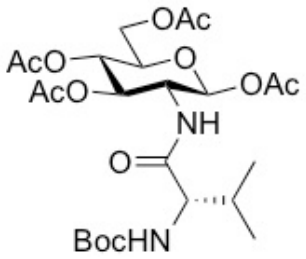
1,3,4,6-Tetra-*O*-acetyl-2-[(*N*-(*tert*-butoxycarbonyl)-L-valyl)-amino]-2-deoxy-β-D-glucopyranose, 7c

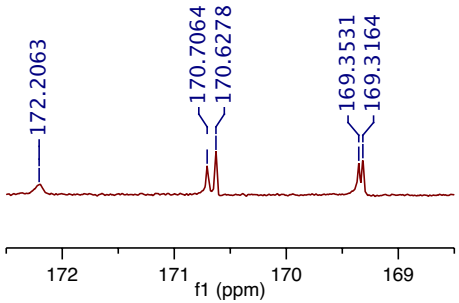


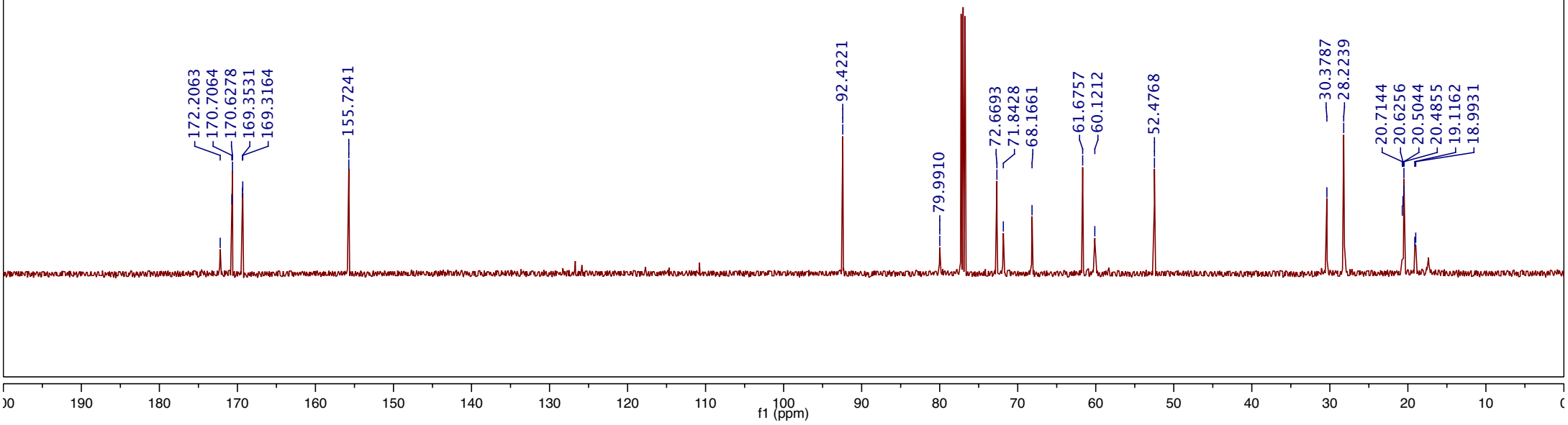




1,3,4,6-Tetra-*O*-acetyl-2-[(*N*-(*tert*-butoxycarbonyl)-L-valyl)-amino]-2-deoxy-β-D-glucopyranose, 7c



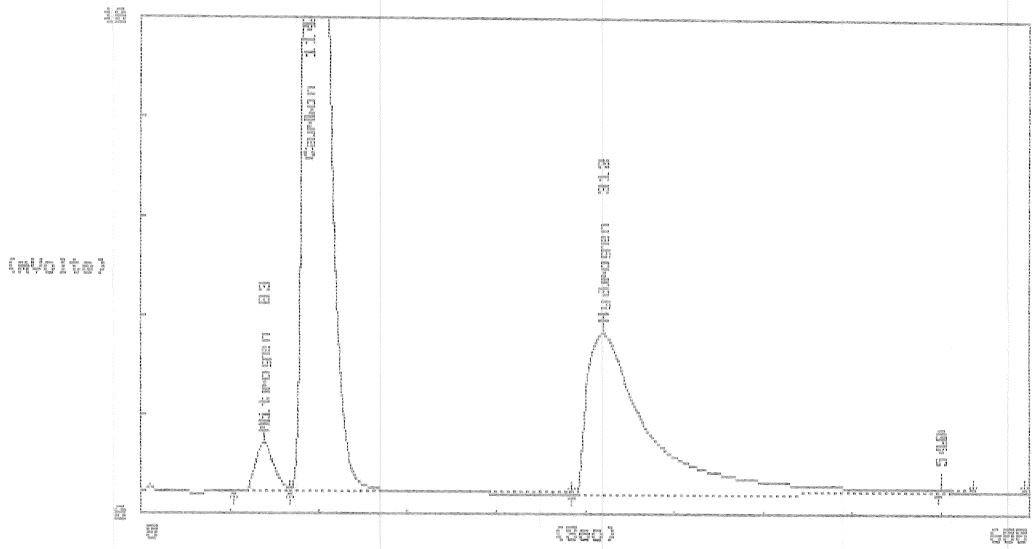




1,3,4,6-Tetra-O-acetyl-2-[(N-(tert-butoxycarbonyl)-L-valyl)-amino]-2-deoxy-β-D-glucopyranose, 7c

EAGER 200 Stripchart

Sample Ident. : 10 Boc-Val-GlcN Filename :288810
Analysed : 08-01-14 10:23:10 Printed :08-01-2014 12:19:34



EAGER 200 Peak Integration Report

Instrument name : Instrument #1 Bline drift (fV): 19.3
Company Name : U of Florida Operator Ident. : KOU
Analysed : 08-01-14 10:23:10 Printed : 08-01-2014 12:19:35
Sample Ident. : 10 Boc-Val-GlcN Filename : 288810
Sample Weight : 1.421 Calc.method: using 'K. Factors'

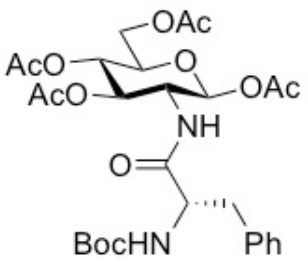
No. (#)	Type (#)	Start (Sec)	End (Sec)	Ret Time (Sec)	Height (fV)	Area (fV*Sec)	Area % (%)	Name
1	FU	63	101	83	981.6	13927	2.22	Nitrogen
2	FU	101	291	114	30749.2	452300	71.95	Carbon
3	TL	291	597	312	3201.6	162403	25.83	Hydrogen
4	CR	538	562	540	2.5	31	0.00	
						628660	100.00	

EAGER 200 Unk Report

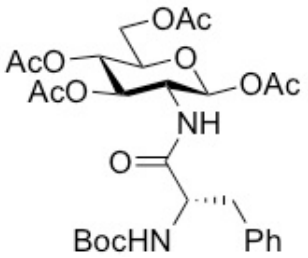
Instrument name : Instrument #1 Bline drift (fV): 19.3
Company Name : U of Florida Operator Ident. : KOU
Analysed : 08-01-14 10:23:10 Printed : 08-01-2014 12:19:35
Sample Ident. : 10 Boc-Val-GlcN Filename : 288810
Sample Weight : 1.421 Calc.method: using 'K. Factors'

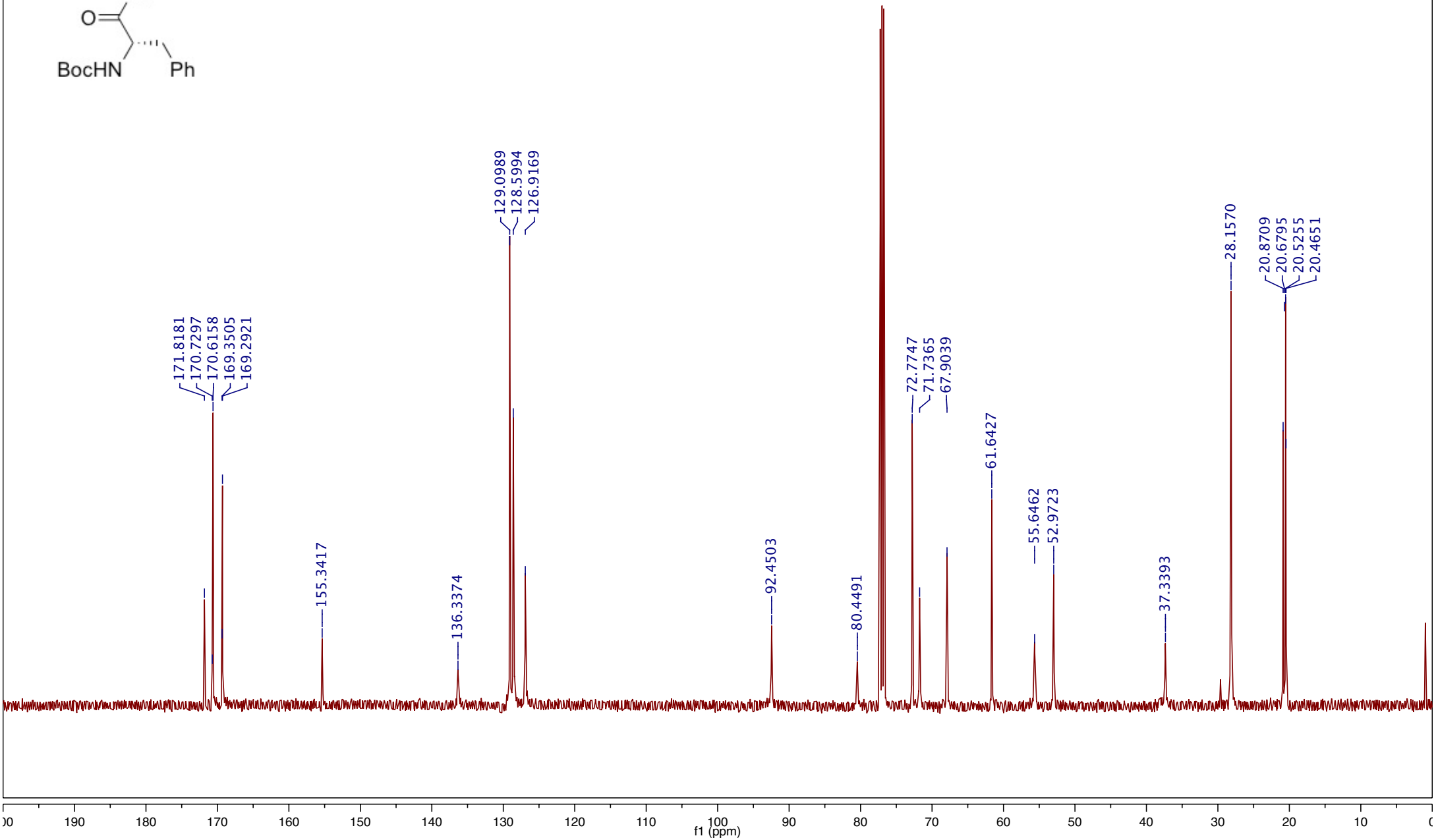
Pk. (#)	Ret Time (Sec)	Area (fV*Sec)	Element % (%)	Area Ratio	Name
1	83	13927	4.649	.324768E+02	Nitrogen
2	114	452300	53.031	.100000E+01	Carbon
3	312	162403	6.798	.278505E+01	Hydrogen

1,3,4,6-Tetra-*O*-acetyl-2-[(*N*-(*tert*-butoxycarbonyl)-L-phenylalanyl)-amino]-2-deoxy-β-D-glucopyranose, 7d



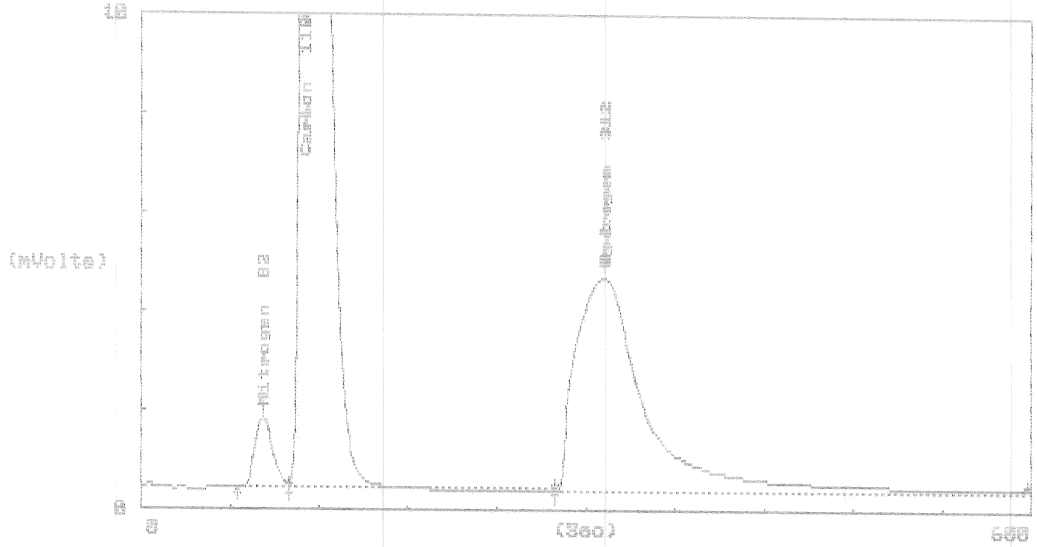
1,3,4,6-Tetra-*O*-acetyl-2-[(*N*-(*tert*-butoxycarbonyl)-L-phenylalanyl)-amino]-2-deoxy-β-D-glucopyranose, 7d





EAGER 200 Stripchart

Sample Ident. : 15 RaJ2159-25 Filename :285115
Analysed : 02-06-14 11:41:07 Printed :02-06-2014 11:51:10



EAGER 200 Peak Integration Report

Instrument name : Instrument #1 Bline drift (fV): 2.5
Company Name : U of Florida Operator Ident. : KOU
Analysed : 02-06-14 11:41:07 Printed : 02-06-2014 11:51:10
Sample Ident. : 15 RaJ2159-25 Filename : 285115
Sample Weight : 2.181 Calc.method: using 'K. Factors'

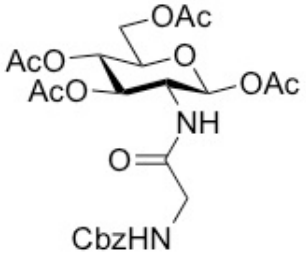
No. (#)	Type (#)	Start (Sec)	End (Sec)	Ret Time (Sec)	Height (fV)	Area (fV*Sec)	Area % (%)	Name
1	FU	65	99	82	1430.7	19348	2.01	Nitrogen
2	FU	99	279	110	47432.4	711309	73.91	Carbon
3	RS	279	598	312	4296.9	231798	24.08	Hydrogen
						962455	100.00	

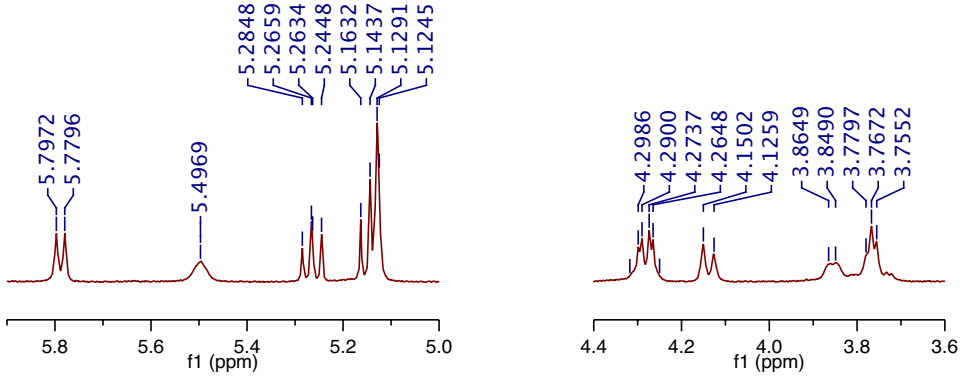
EAGER 200 Unk Report

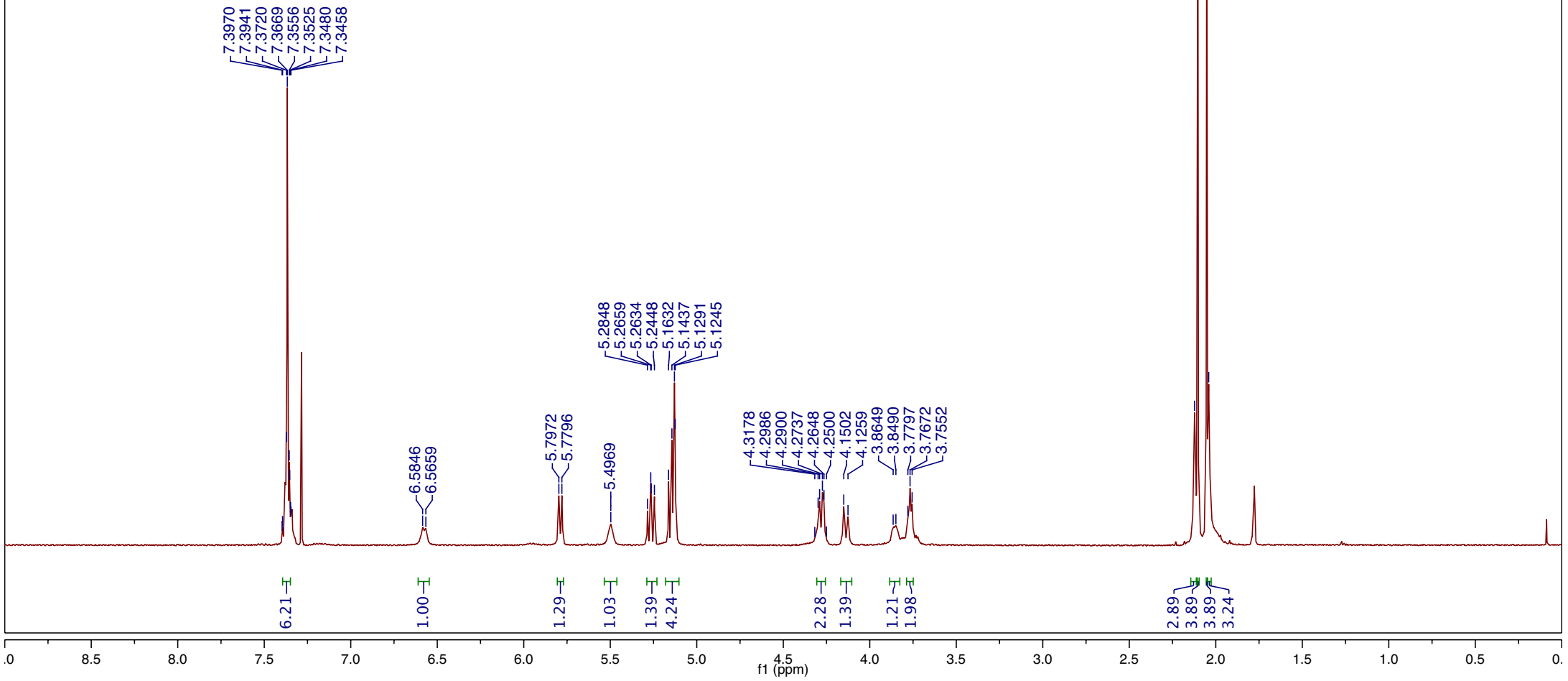
Instrument name : Instrument #1 Bline drift (fV): 2.5
Company Name : U of Florida Operator Ident. : KOU
Analysed : 02-06-14 11:41:07 Printed : 02-06-2014 11:51:10
Sample Ident. : 15 RaJ2159-25 Filename : 285115
Sample Weight : 2.181 Calc.method: using 'K. Factors'

Pk. (#)	Ret Time (Sec)	Area (fV*Sec)	Element % (%)	Area Ratio	Name
1	82	19348	4.533	.367633E+02	Nitrogen
2	110	711309	56.493	.100000E+01	Carbon
3	312	231798	6.804	.306866E+01	Hydrogen

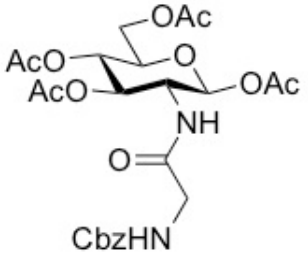
1,3,4,6-Tetra-*O*-acetyl-2-[(*N*-(carbobenzyloxy)glycyl)-amino]-2-deoxy-β-D-glucopyranose, 7e

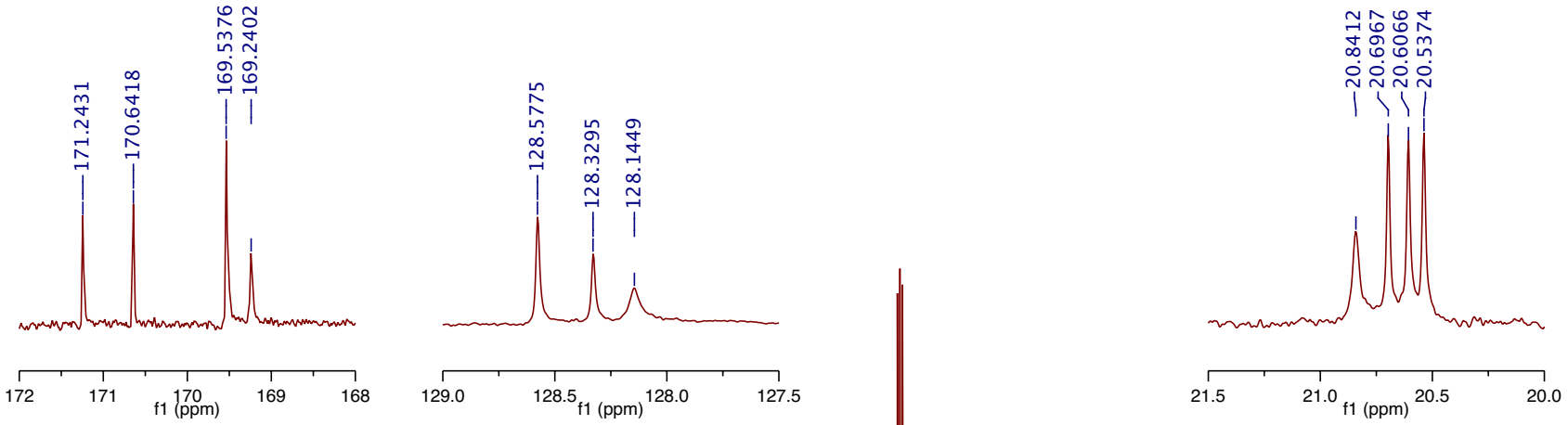


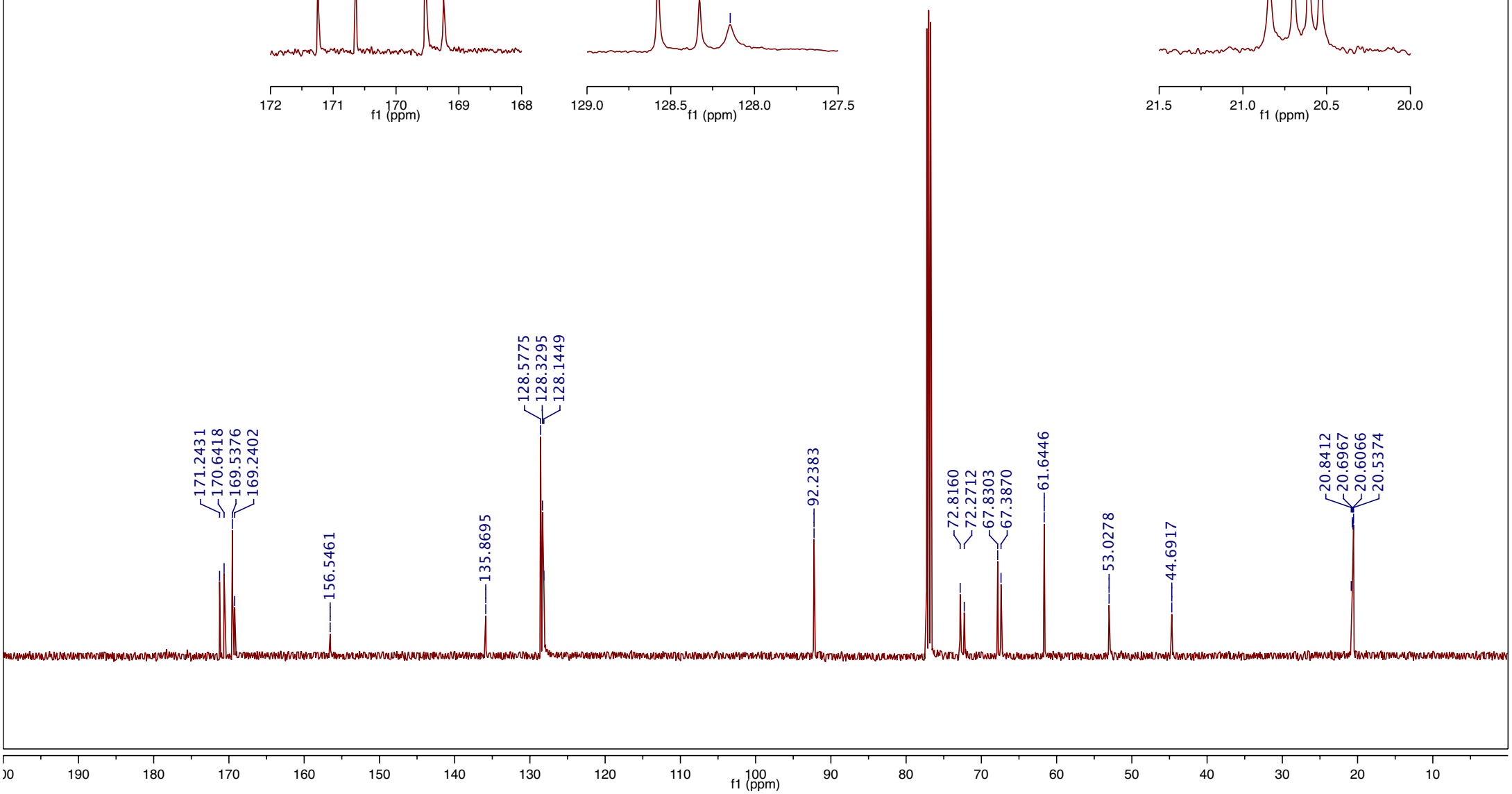




1,3,4,6-Tetra-*O*-acetyl-2-[(*N*-(carbobenzyloxy)glycyl)-amino]-2-deoxy-β-D-glucopyranose, 7e





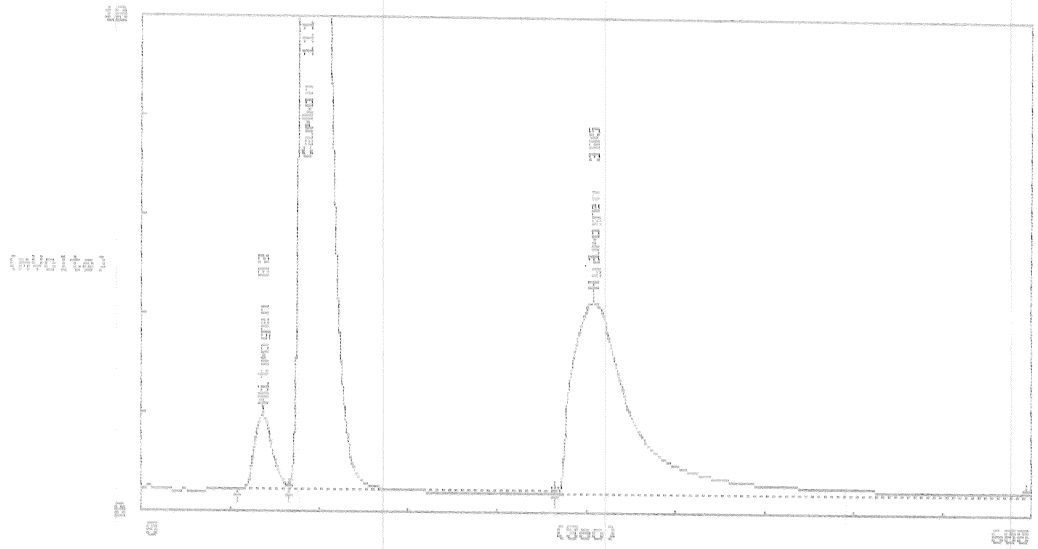


1,3,4,6-Tetra-O-acetyl-2-[(N-(carbobenzyloxy)glycyl)-amino]-2-deoxy-β-D-glucopyranose, 7e

342-083

EAGER 200 Stripchart

Sample Ident. : 13 RaJ2159-30 Filename :285113
Analysed : 02-06-14 11:21:01 Printed :02-06-2014 11:31:04



EAGER 200 Peak Integration Report

Instrument name : Instrument #1 Bline drift (fV):-.7
Company Name : U of Florida Operator Ident. : KOU
Analysed : 02-06-14 11:21:01 Printed : 02-06-2014 11:31:04
Sample Ident. : 13 RaJ2159-30 Filename : 285113
Sample Weight : 2.103 Calc.method: using 'K. Factors'

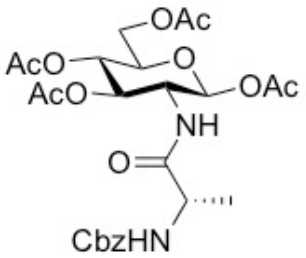
No. (#)	Type (#)	Start (Sec)	End (Sec)	Ret Time (Sec)	Height (fV)	Area (fV*Sec)	Area % (%)	Name
1	FU	65	99	82	1503.9	20518	2.39	Nitrogen
2	FU	99	279	111	43137.8	649341	75.56	Carbon
3	RS	279	597	305	3863.0	189560	22.06	Hydrogen
						859419	100.00	

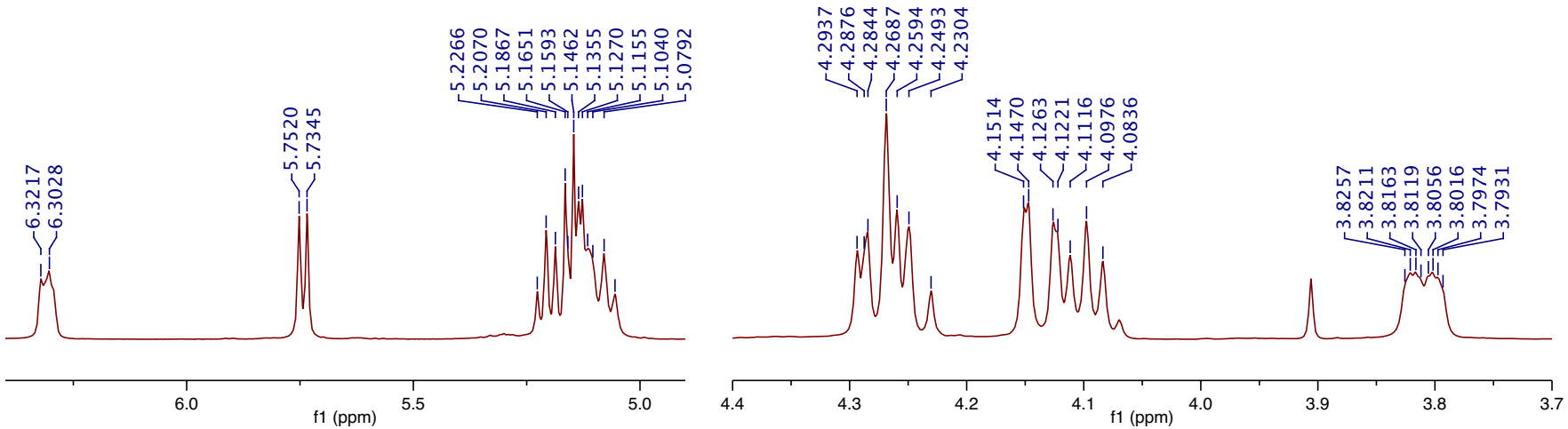
EAGER 200 Unk Report

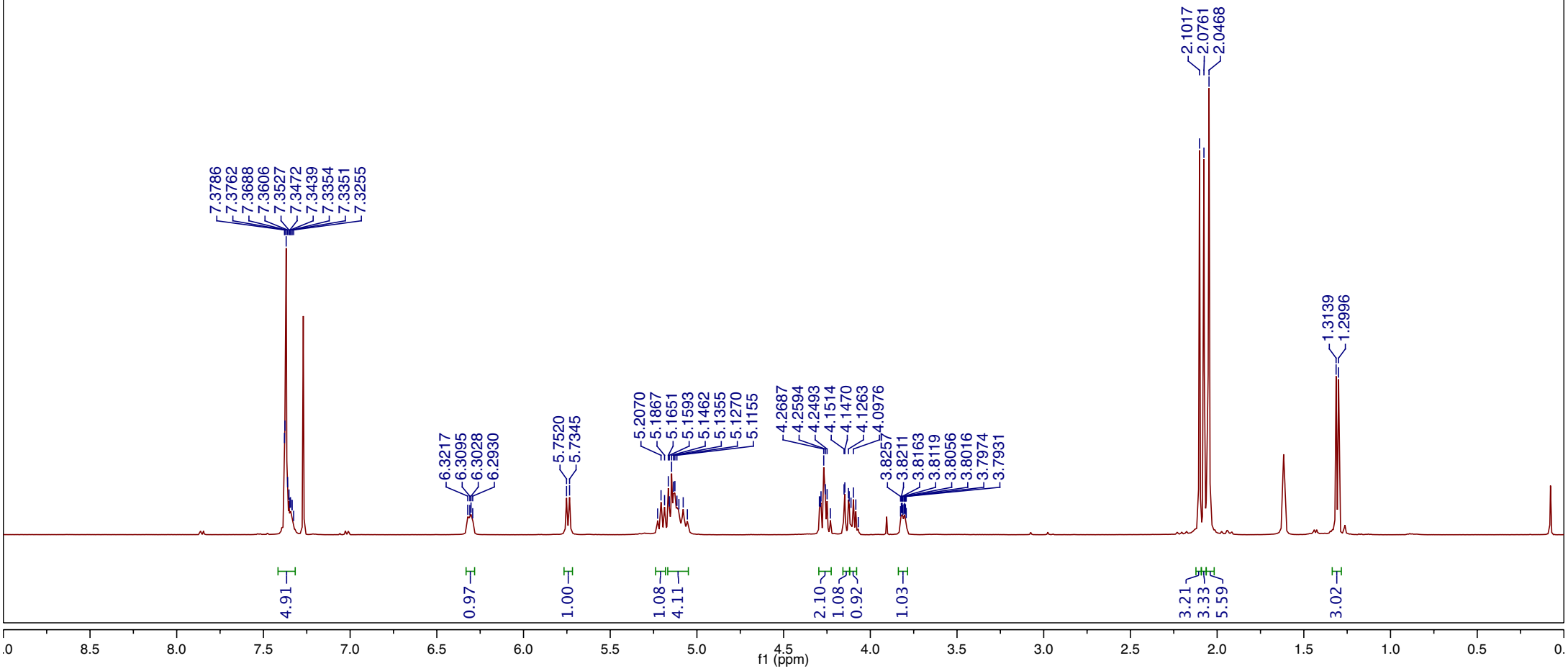
Instrument name : Instrument #1 Bline drift (fV):-.7
Company Name : U of Florida Operator Ident. : KOU
Analysed : 02-06-14 11:21:01 Printed : 02-06-2014 11:31:04
Sample Ident. : 13 RaJ2159-30 Filename : 285113
Sample Weight : 2.103 Calc.method: using 'K. Factors'

Pk. (#)	Ret Time (Sec)	Area (fV*Sec)	Element % (%)	Area Ratio	Name
1	82	20518	4.986	.316470E+02	Nitrogen
2	111	649341	53.484	.100000E+01	Carbon
3	305	189560	5.770	.342552E+01	Hydrogen

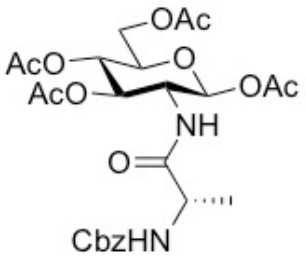
1,3,4,6-Tetra-*O*-acetyl-2-[(*N*-(carbobenzyloxy)-L-alanyl)-amino]-2-deoxy-β-D-glucopyranose, 7f

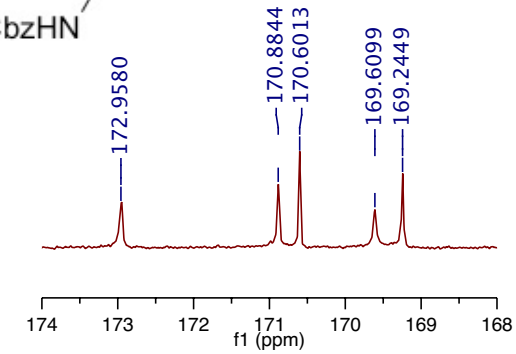


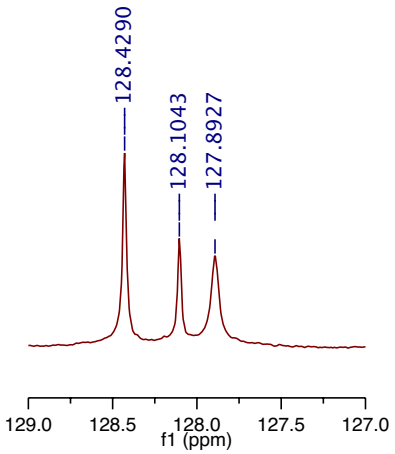


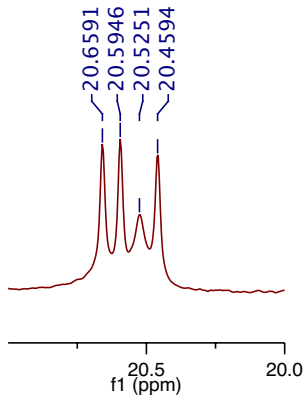


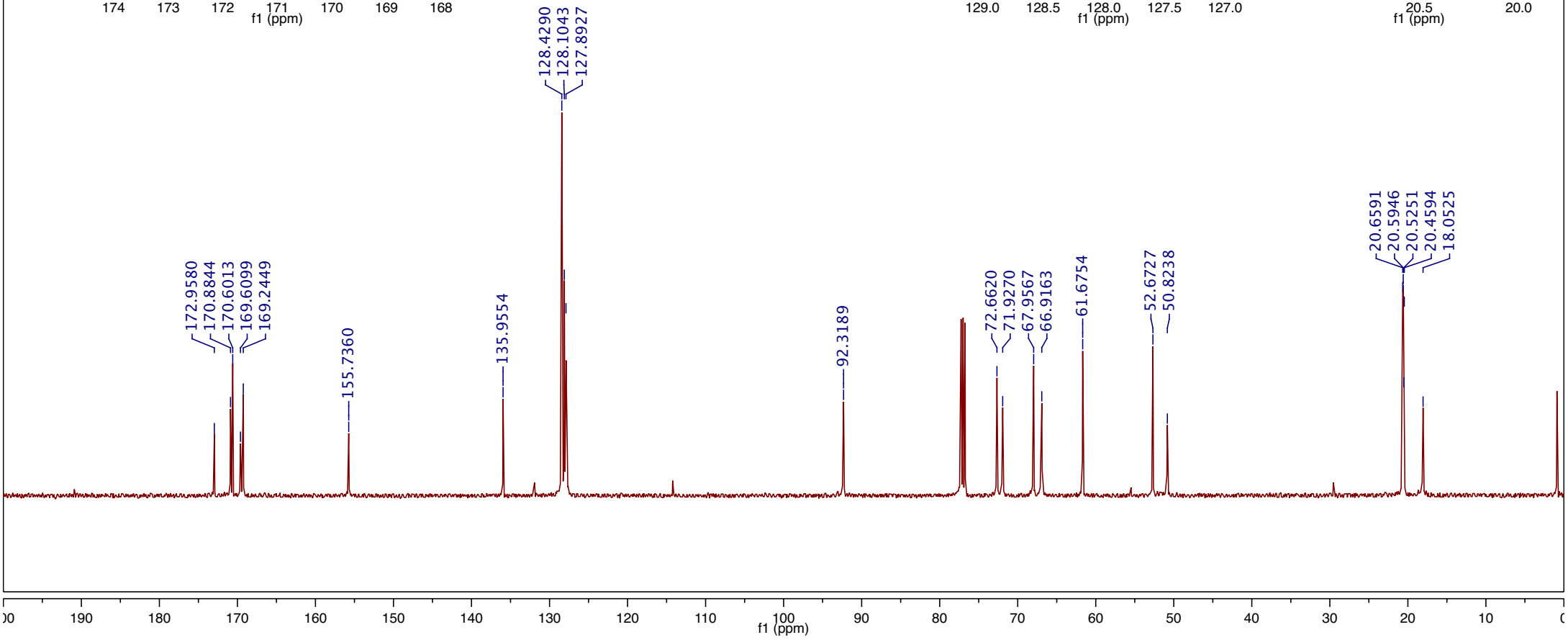
1,3,4,6-Tetra-*O*-acetyl-2-[(*N*-(carbobenzyloxy)-L-alanyl)-amino]-2-deoxy-β-D-glucopyranose, 7f









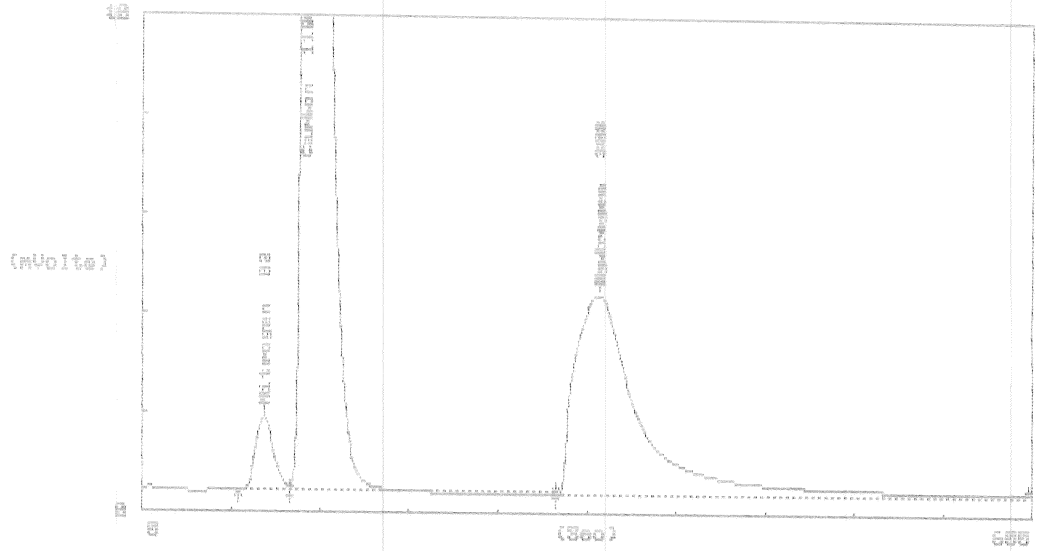


1,3,4,6-Tetra-O-acetyl-2-[(N-(carbobenzyloxy)-L-alanyl)-amino]-2-deoxy-β-D-glucopyranose, 7f

342-064

EAGER 200 stripchart

Sample Ident. : 12 RaJ2159-32 Filename :285112
Analysed : 02-06-14 11:10:58 Printed :02-06-2014 11:21:01



EAGER 200 Peak Integration Report

Instrument name : Instrument #1 Bline drift (fV):-.8
Company Name : U of Florida Operator Ident. : KOU
Analysed : 02-06-14 11:10:58 Printed : 02-06-2014 11:21:01
Sample Ident. : 12 RaJ2159-32 Filename : 285112
Sample Weight : 2.116 Calc.method: using 'K. Factors'

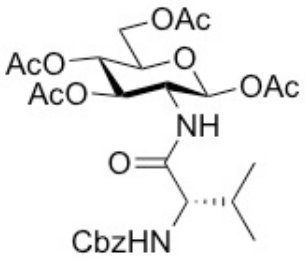
No. (#)	Type (#)	Start (Sec)	End (Sec)	Ret Time (Sec)	Height (fV)	Area (fV*Sec)	Area % (%)	Name
1	FU	65	99	82	1478.1	20016	2.25	Nitrogen
2	FU	99	279	110	44612.7	665319	74.92	Carbon
3	RS	279	598	308	3984.3	202724	22.83	Hydrogen
						888059	100.00	

EAGER 200 Unk Report

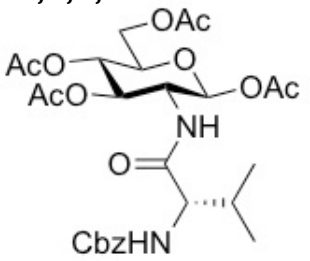
Instrument name : Instrument #1 Bline drift (fV):-.8
Company Name : U of Florida Operator Ident. : KOU
Analysed : 02-06-14 11:10:58 Printed : 02-06-2014 11:21:01
Sample Ident. : 12 RaJ2159-32 Filename : 285112
Sample Weight : 2.116 Calc.method: using 'K. Factors'

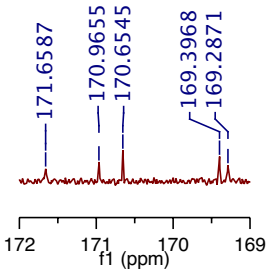
Pk. (#)	Ret Time (Sec)	Area (fV*Sec)	Element % (%)	Area Ratio	Name
1	82	20016	4.834	.332391E+02	Nitrogen
2	110	665319	54.464	.100000E+01	Carbon
3	308	202724	6.133	.328190E+01	Hydrogen

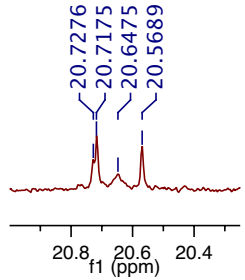
1,3,4,6-Tetra-*O*-acetyl-2-[(*N*-(carbobenzyloxy)-L-valyl)-amino]-2-deoxy-β-D-glucopyranose, 7g

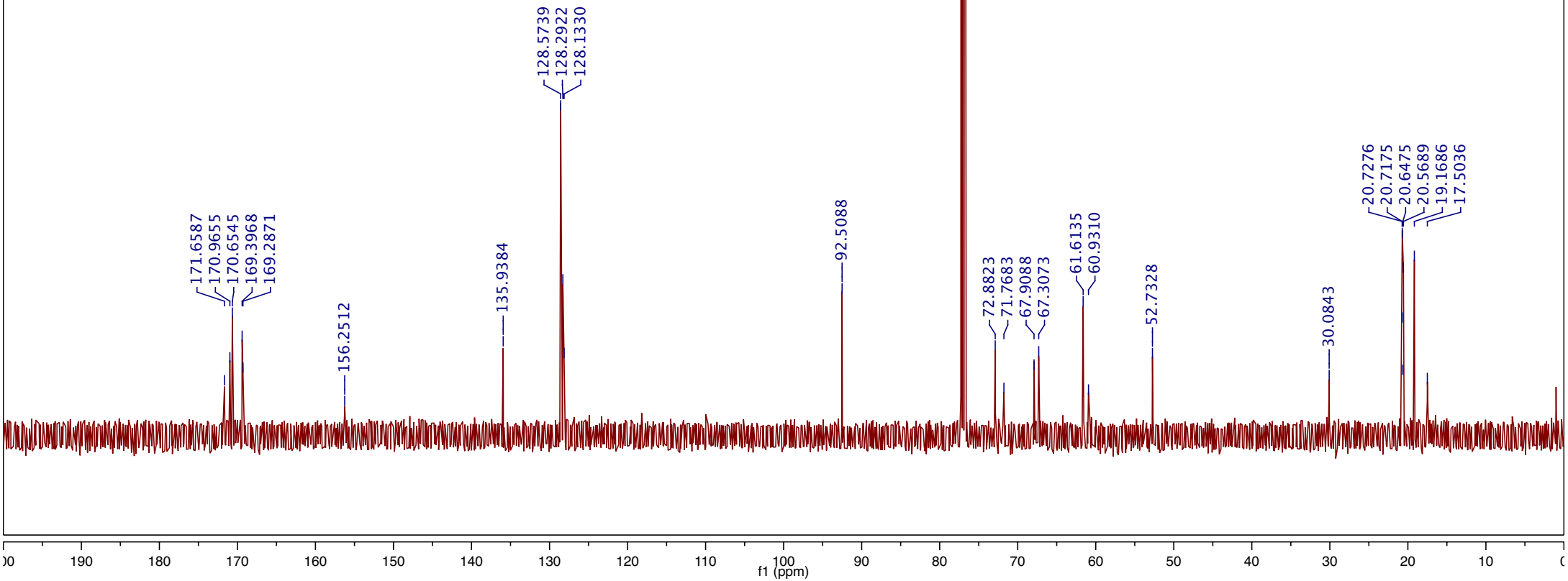


1,3,4,6-Tetra-*O*-acetyl-2-[(*N*-(carbobenzyloxy)-L-valyl)-amino]-2-deoxy-β-D-glucopyranose, 7g





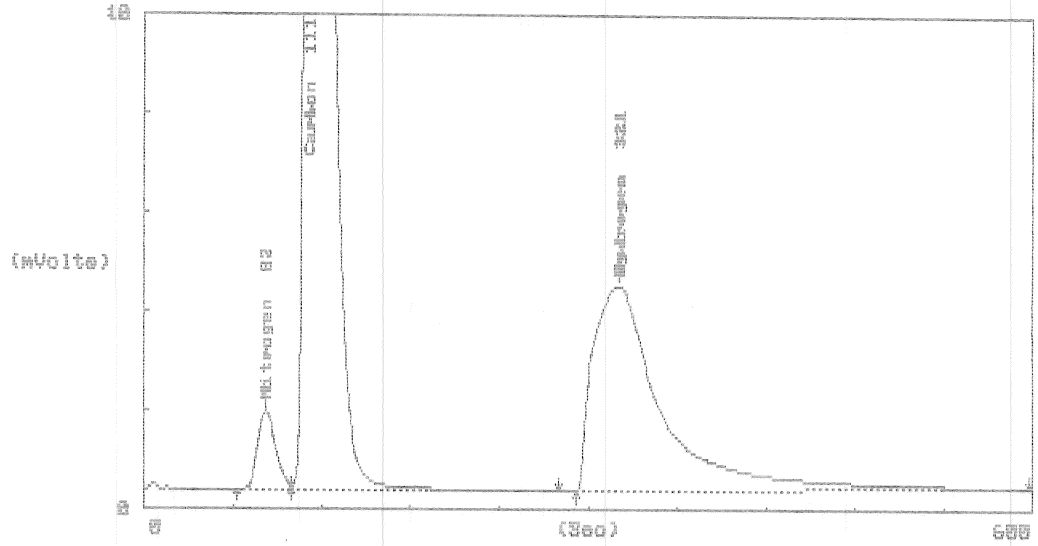




1,3,4,6-Tetra-O-acetyl-2-[(N-(carbobenzyloxy)-L-valyl)-amino]-2-deoxy-β-D-glucopyranose, 7g

EAGER 200 Stripchart

Sample Ident. : 10 CRg-Val-Glc Filename :288510
Analysed : 07-29-14 09:59:17 Printed :07-29-2014 14:42:41



EAGER 200 Peak Integration Report

Instrument name : Instrument #1 Bline drift (fV): 57
Company Name : U of Florida Operator Ident. : KOU
Analysed : 07-29-14 09:59:17 Printed : 07-29-2014 14:42:41
Sample Ident. : 10 CRg-Val-Glc Filename : 288510
Sample Weight : 2.221 Calc.method: using 'K. Factors'

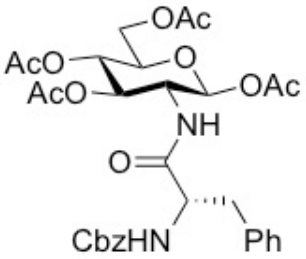
No. (#)	Type (#)	Start (Sec)	End (Sec)	Ret Time (Sec)	Height (fV)	Area (fV*Sec)	Area % (%)	Name
1	FU	63	100	82	1567.8	21165	2.16	Nitrogen
2	FU	100	280	111	49212.0	735802	74.96	Carbon
3	RS	292	598	321	4083.1	224678	22.89	Hydrogen
						981645	100.00	

EAGER 200 Unk Report

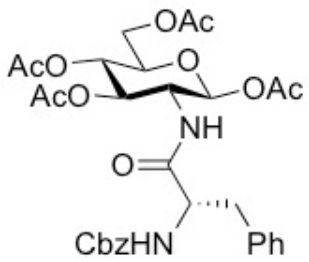
Instrument name : Instrument #1 Bline drift (fV): 57
Company Name : U of Florida Operator Ident. : KOU
Analysed : 07-29-14 09:59:17 Printed : 07-29-2014 14:42:42
Sample Ident. : 10 CRg-Val-Glc Filename : 288510
Sample Weight : 2.221 Calc.method: using 'K. Factors'

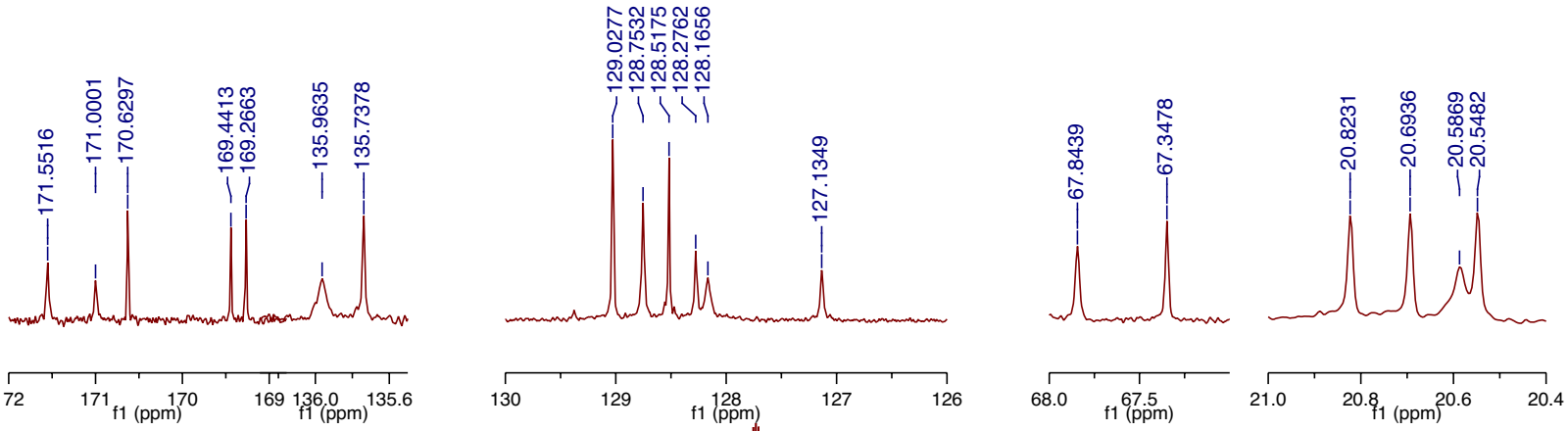
Pk. (#)	Ret Time (Sec)	Area (fV*Sec)	Element % (%)	Area Ratio	Name
1	82	21165	4.491	.347651E+02	Nitrogen
2	111	735802	55.613	.100000E+01	Carbon
3	321	224678	6.043	.327491E+01	Hydrogen

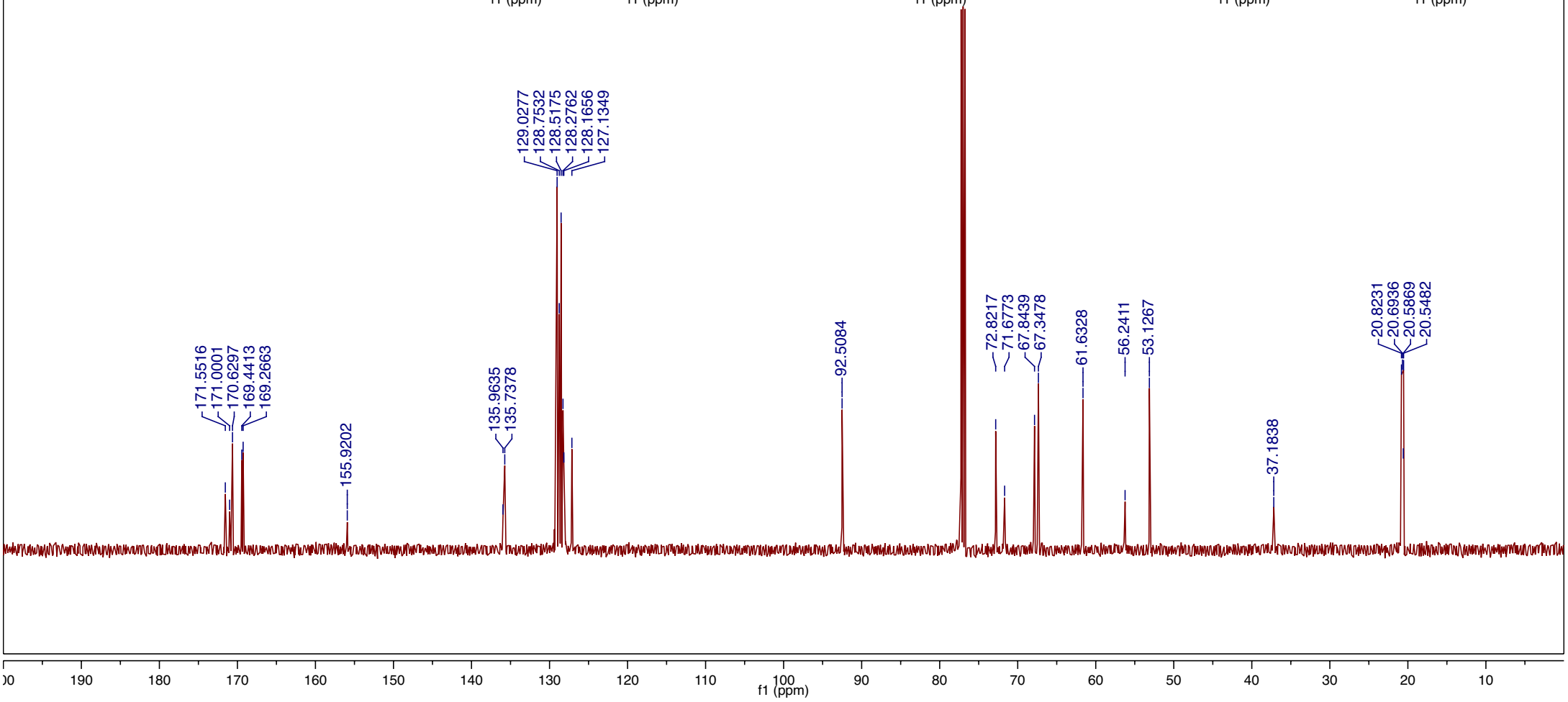
1,3,4,6-Tetra-*O*-acetyl-2-[(*N*-(carbobenzyloxy)-L-phenylalanyl)-amino]-2-deoxy-β-D-glucopyranose, 7h



1,3,4,6-Tetra-*O*-acetyl-2-[(*N*-(carbobenzyloxy)-L-phenylalanyl)-amino]-2-deoxy-β-D-glucopyranose, 7h



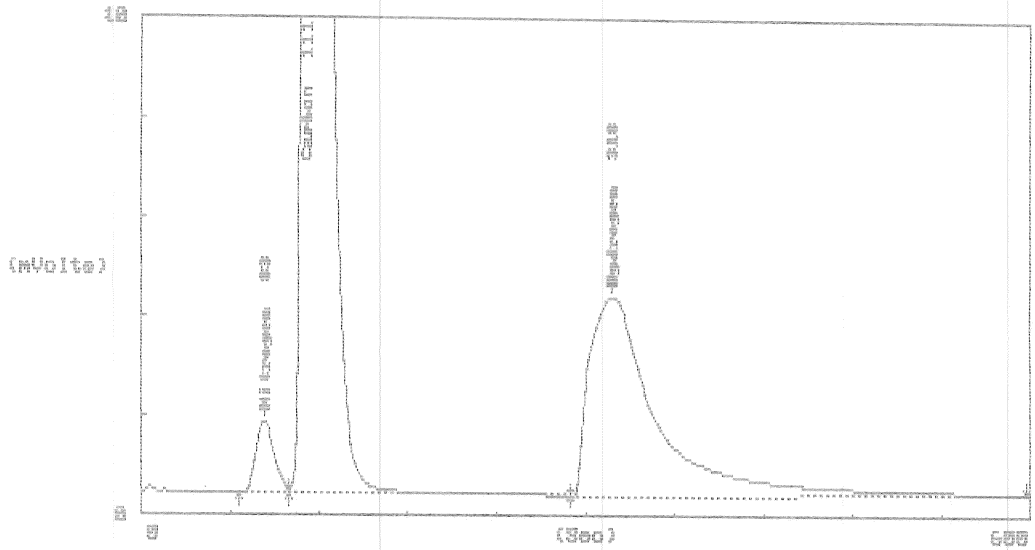




1,3,4,6-Tetra-O-acetyl-2-[(N-(carbobenzyloxy)-L-phenylalanyl)-amino]-2-deoxy-β-D-glucopyranose, 7h

EAGER 200 Stripchart

Sample Ident. : 13 T34-77 Filename :288713
Analysed : 07-31-14 09:36:11 Printed :07-31-2014 09:46:13



EAGER 200 Peak Integration Report

Instrument name : Instrument #1 Bline drift (fV):-3.7
Company Name : U of Florida Operator Ident. : KOU
Analysed : 07-31-14 09:36:11 Printed : 07-31-2014 09:46:14
Sample Ident. : 13 T34-77 Filename : 288713
Sample Weight : 2.17 Calc.method: using 'K. Factors'

No. (#)	Type (#)	Start (Sec)	End (Sec)	Ret Time (Sec)	Height (fV)	Area (fV*Sec)	Area % (%)	Name
1	FU	66	100	83	1442.2	19650	1.97	Nitrogen
2	FU	100	290	111	51255.5	769455	76.97	Carbon
3	RS	290	598	318	3956.6	210604	21.07	Hydrogen
						999709	100.00	

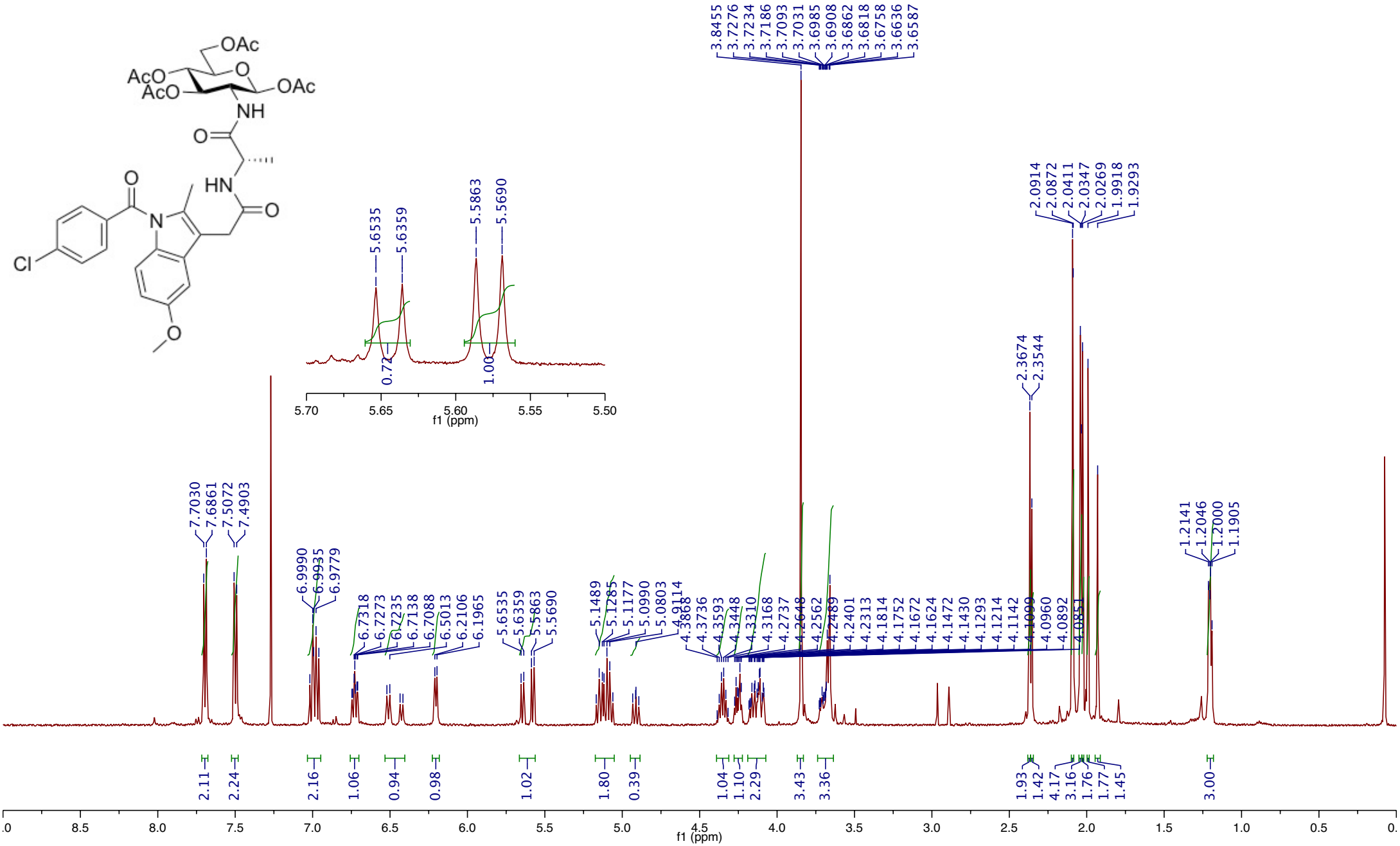
EAGER 200 Unk Report

Instrument name : Instrument #1 Bline drift (fV):-3.7
Company Name : U of Florida Operator Ident. : KOU
Analysed : 07-31-14 09:36:11 Printed : 07-31-2014 09:46:14
Sample Ident. : 13 T34-77 Filename : 288713
Sample Weight : 2.17 Calc.method: using 'K. Factors'

Pk. (#)	Ret Time (Sec)	Area (fV*Sec)	Element % (%)	Area Ratio	Name
1	83	19650	4.299	.391588E+02	Nitrogen
2	111	769455	59.469	.100000E+01	Carbon
3	318	210604	5.803	.365356E+01	Hydrogen

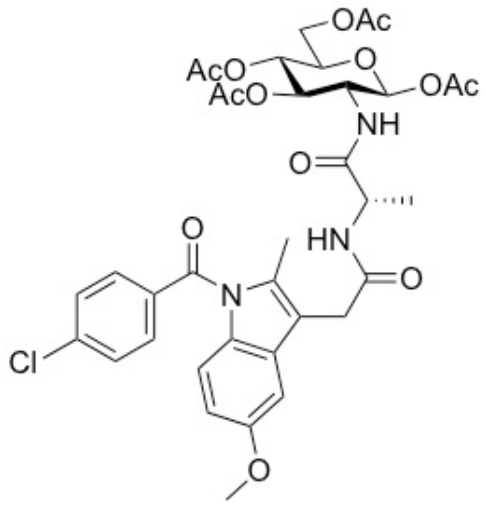
1,3,4,6-Tetra-*O*-acetyl-2-[(*N*-((2-(1-4-chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetyl)-L-alanyl)-amino]-2-deoxy-β-D-glucopyranose, 6a, ($CDCl_3$)

Rotamers – Scheme 1



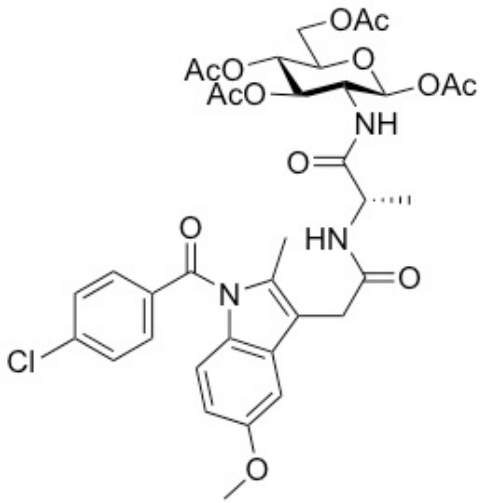
1,3,4,6-Tetra-*O*-acetyl-2-[(*N*-((2-(1-4-chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetyl)-L-alanyl)-amino]-2-deoxy-β-D-glucopyranose, 6a, (DMSO-d_6)

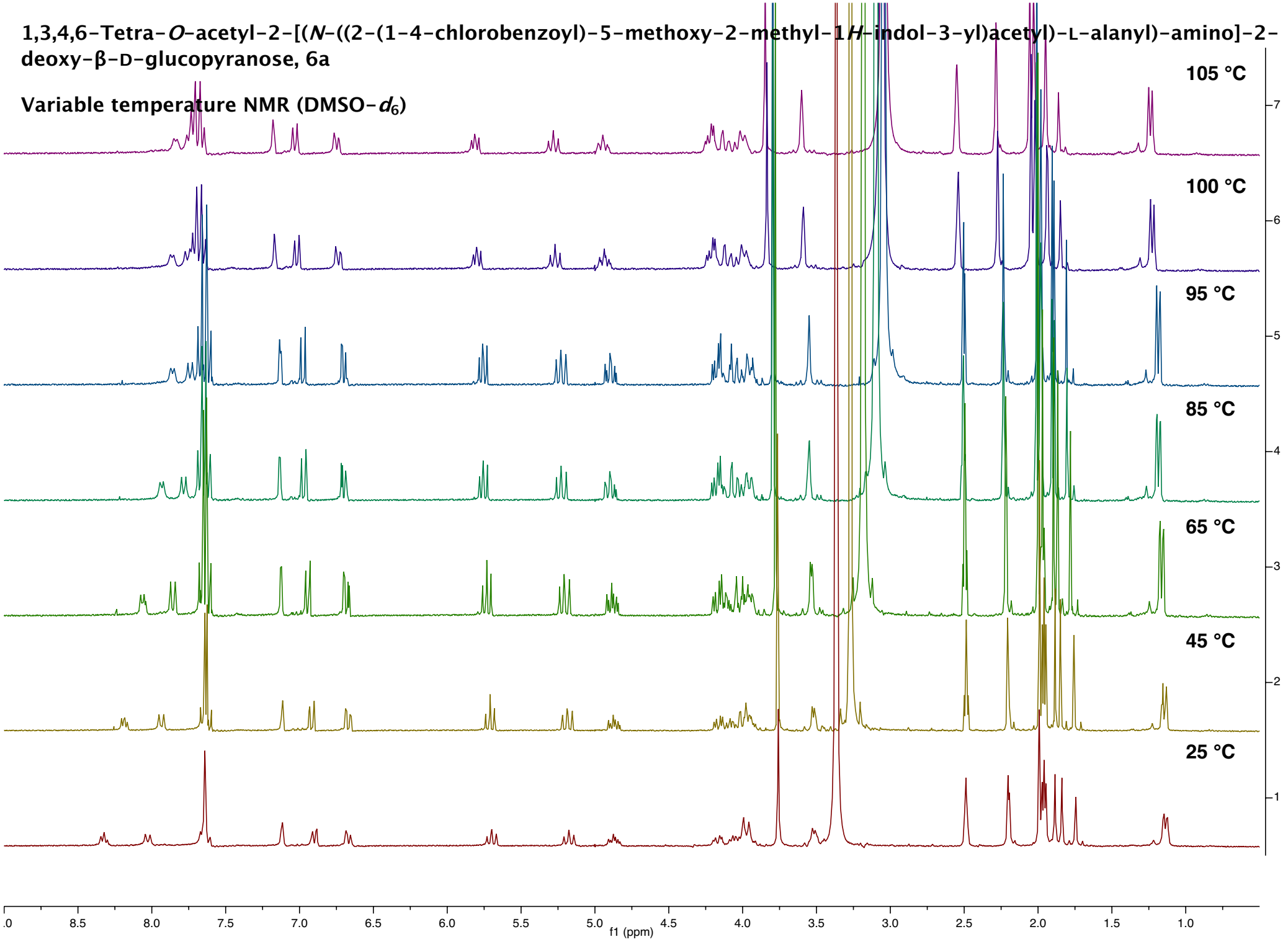
Rotamers – Scheme 1



1,3,4,6-Tetra-*O*-acetyl-2-[(*N*-((2-(1-4-chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetyl)-L-alanyl)-amino]-2-deoxy-β-D-glucopyranose, 6a

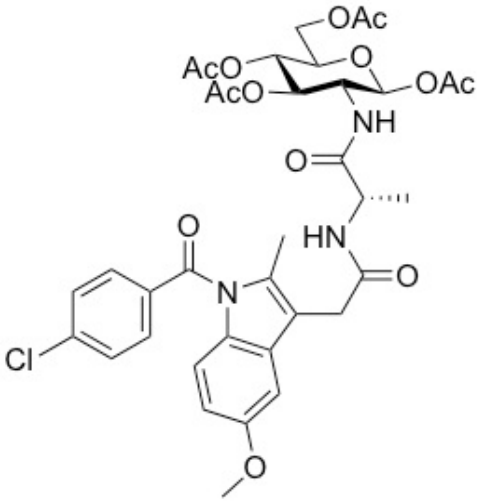
Rotamers - Scheme 1

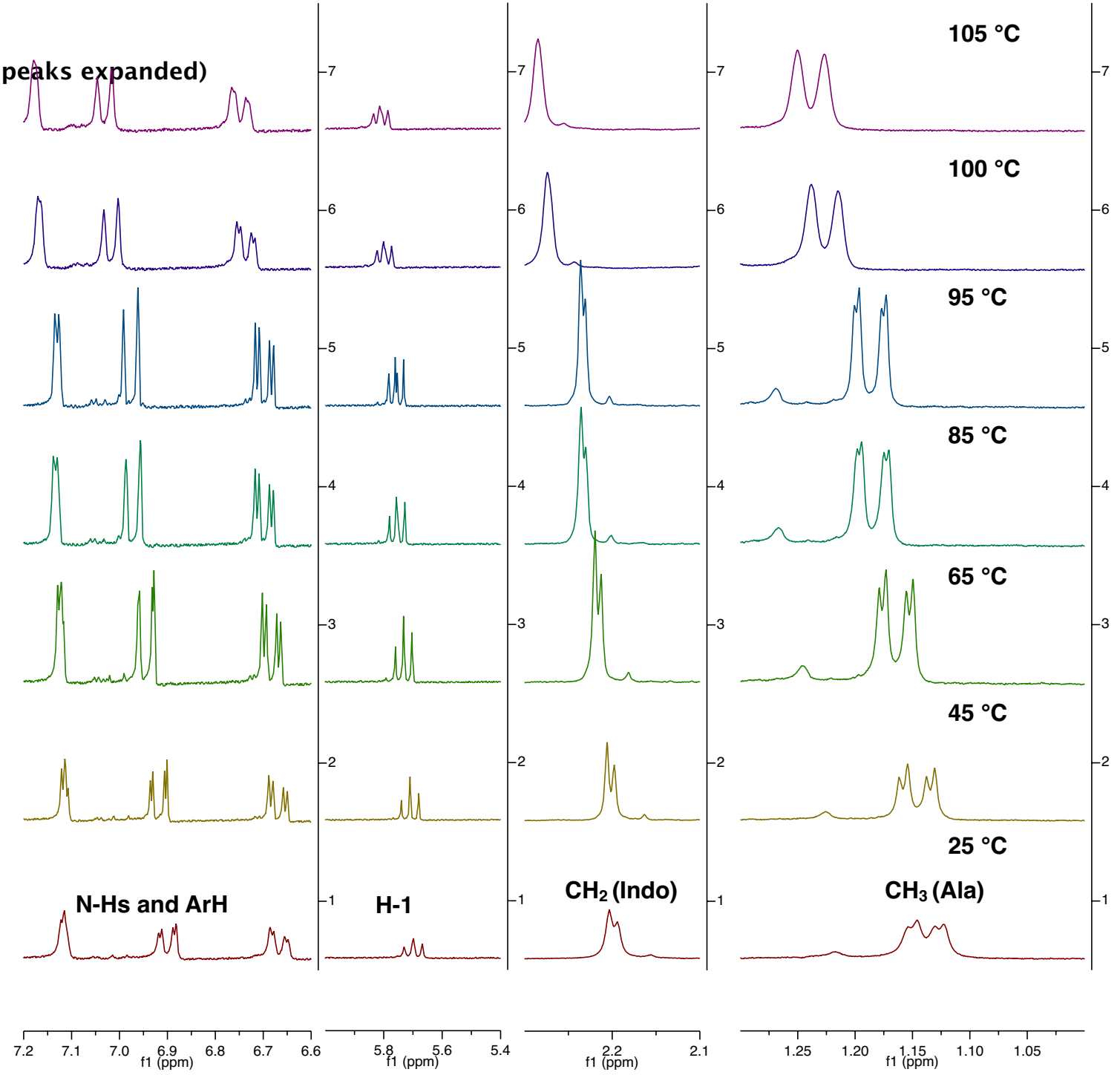



1,3,4,6-Tetra-O-acetyl-2-[(N-((2-(1-4-chlorobenzoyl)-5-methoxy-2-methyl-1H-indol-3-yl)acetyl)-L-alanyl)-amino]-2-deoxy-β-D-glucopyranose, 6a
Variable temperature NMR (DMSO-d6)
105 °C
100 °C
95 °C
85 °C
65 °C
45 °C
25 °C
f1 (ppm)

1,3,4,6-Tetra-*O*-acetyl-2-[(*N*-((2-(1-4-chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetyl)-L-alanyl)-amino]-2-deoxy-β-D-glucopyranose, 6a

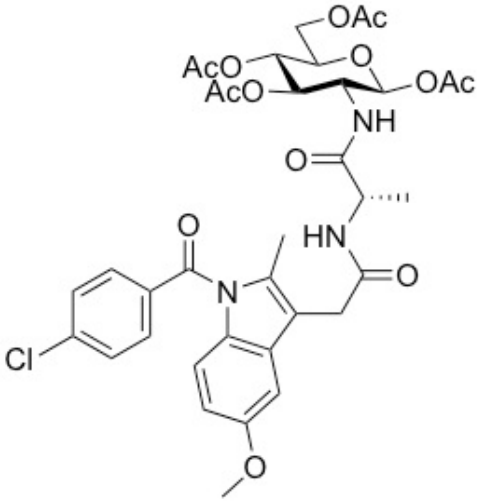
Variable temperature NMR (Selected peaks expanded)





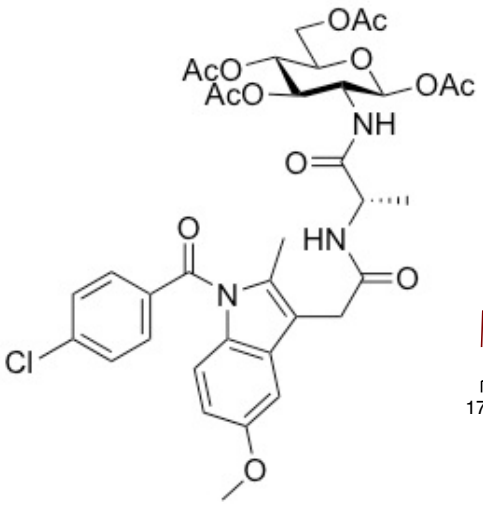
1,3,4,6-Tetra-*O*-acetyl-2-[(*N*-((2-(1-4-chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetyl)-L-alanyl)-amino]-2-deoxy-β-D-glucopyranose, 6a

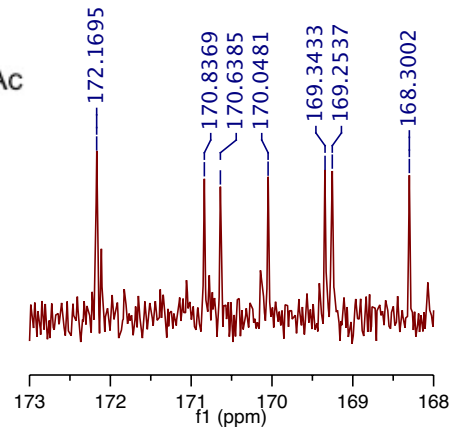
Single Rotamer – Scheme 2

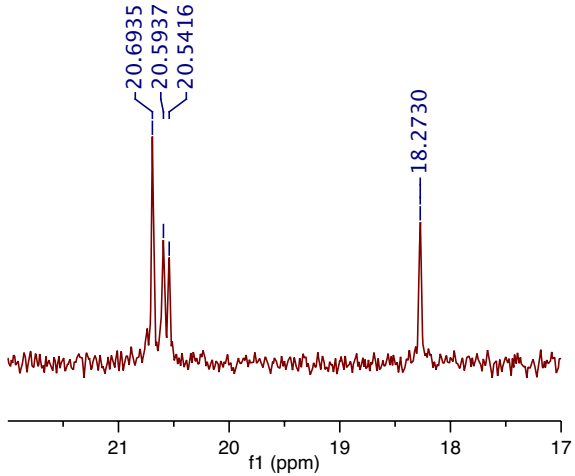


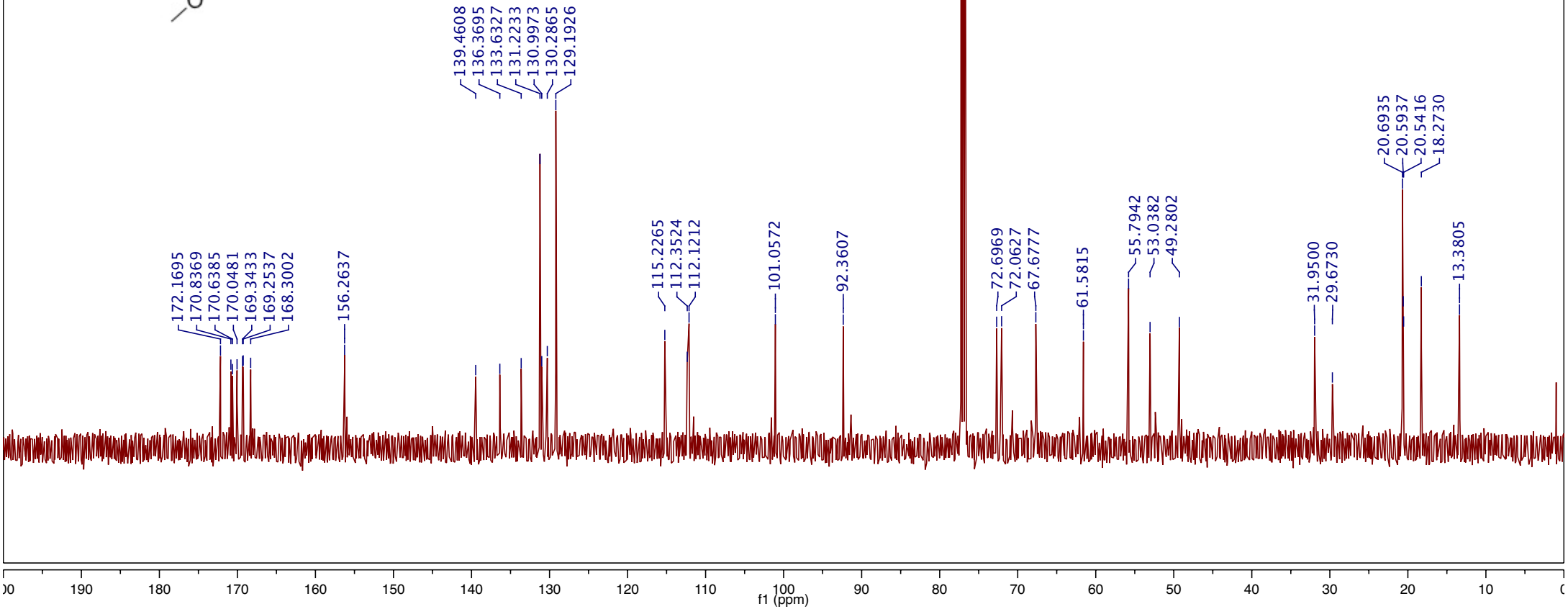
1,3,4,6-Tetra-*O*-acetyl-2-[(*N*-((2-(1-4-chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetyl)-L-alanyl)-amino]-2-deoxy-β-D-glucopyranose, 6a

Single Rotamer – Scheme 2

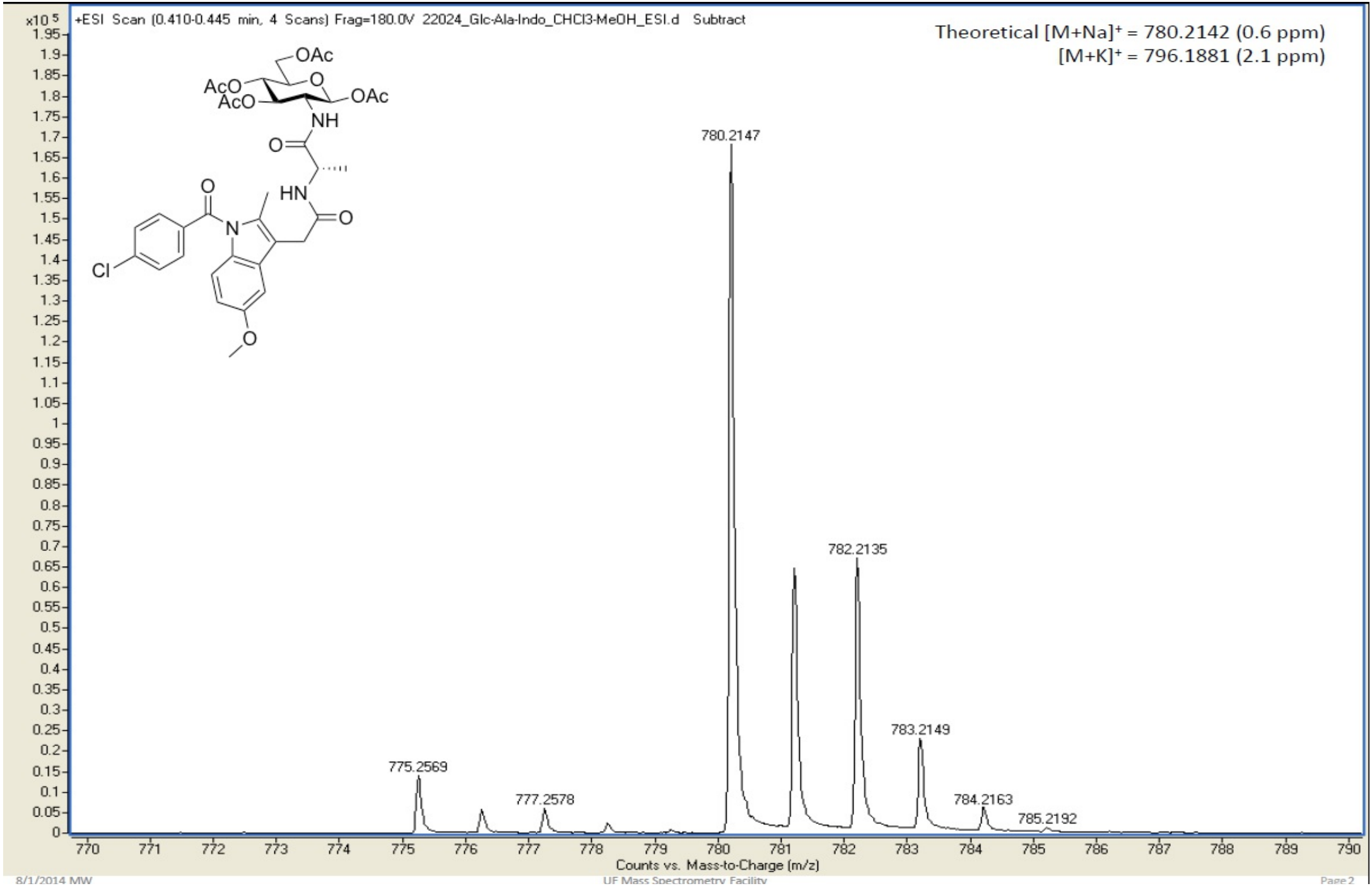




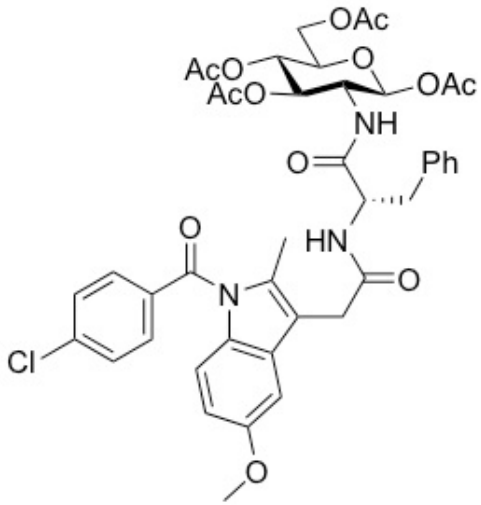


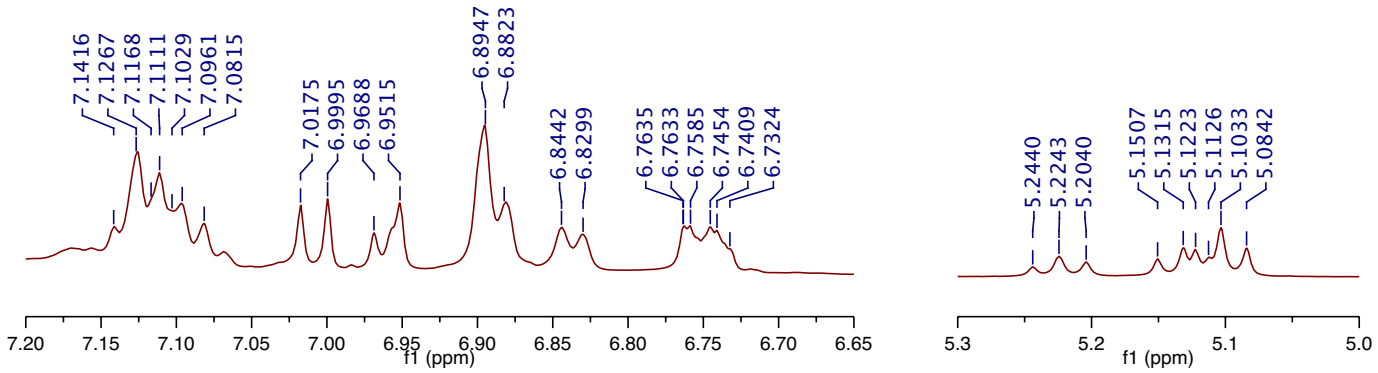


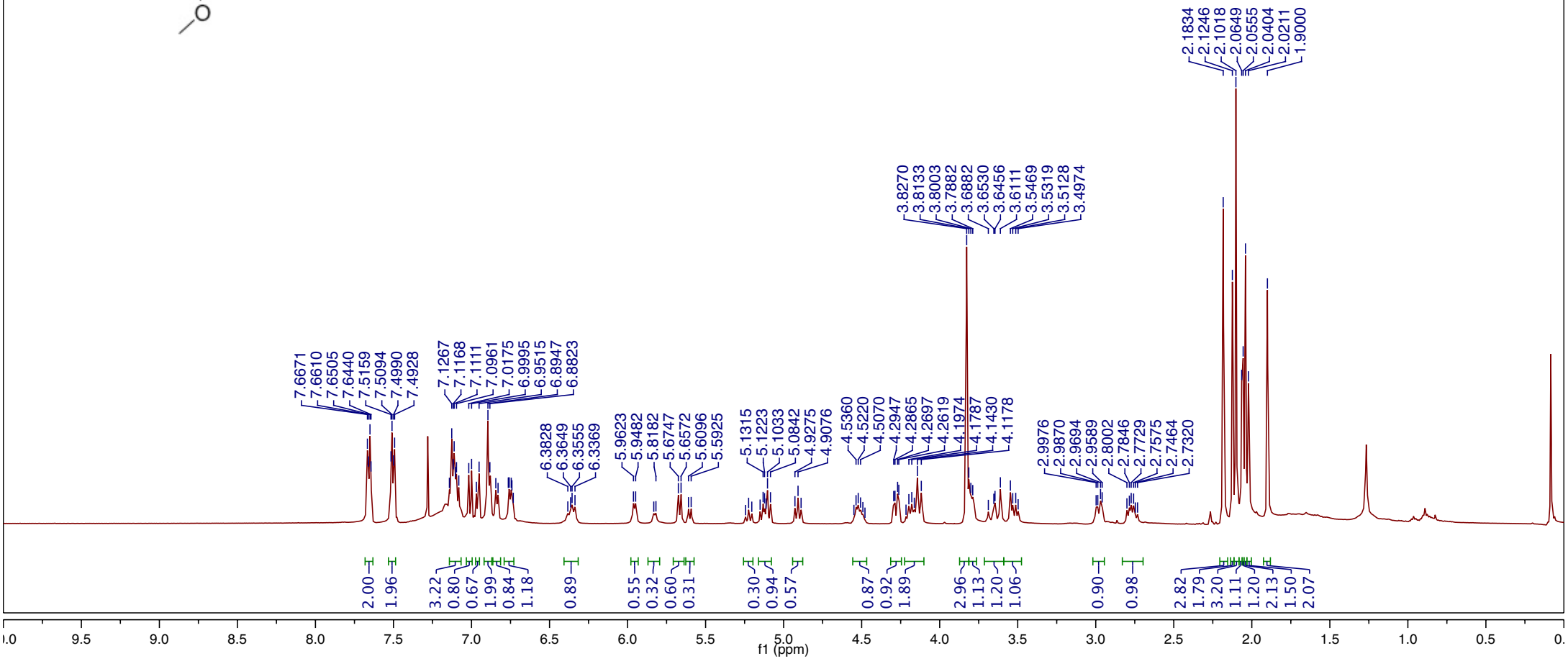
1,3,4,6-Tetra-*O*-acetyl-2-[(*N*-((2-(1-4-chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetyl)-L-alanyl)-amino]-2-deoxy-β-D-glucopyranose, 6a



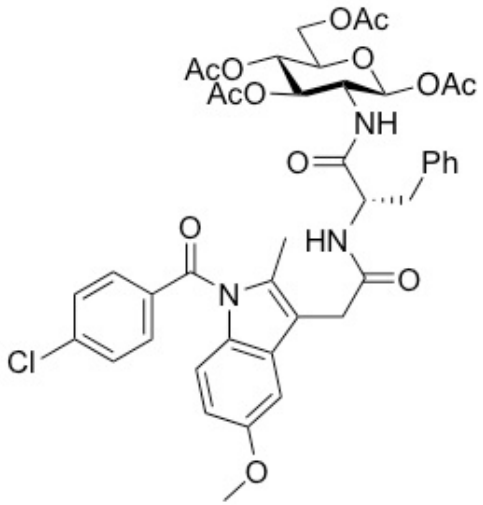
1,3,4,6-Tetra-*O*-acetyl-2-[(*N*-((2-(1-4-chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetyl)-L-phenylalanyl)-amino]-2-deoxy-β-D-glucopyranose, 6b

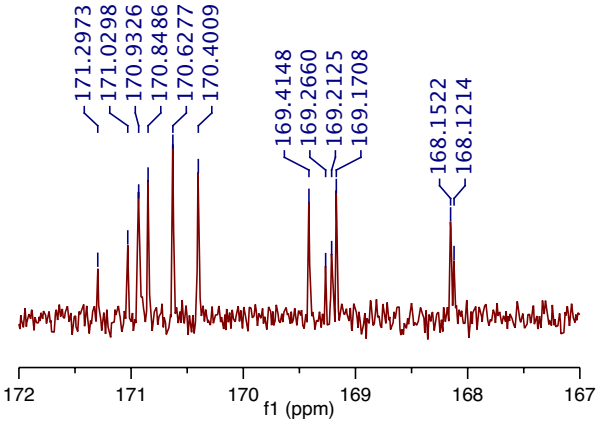


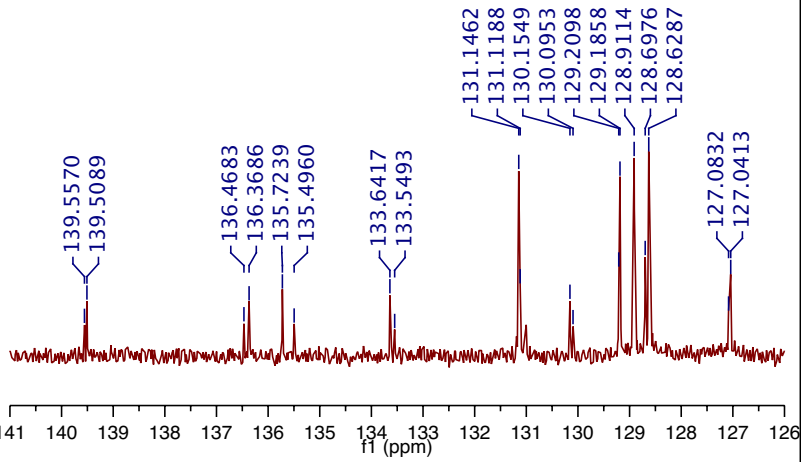


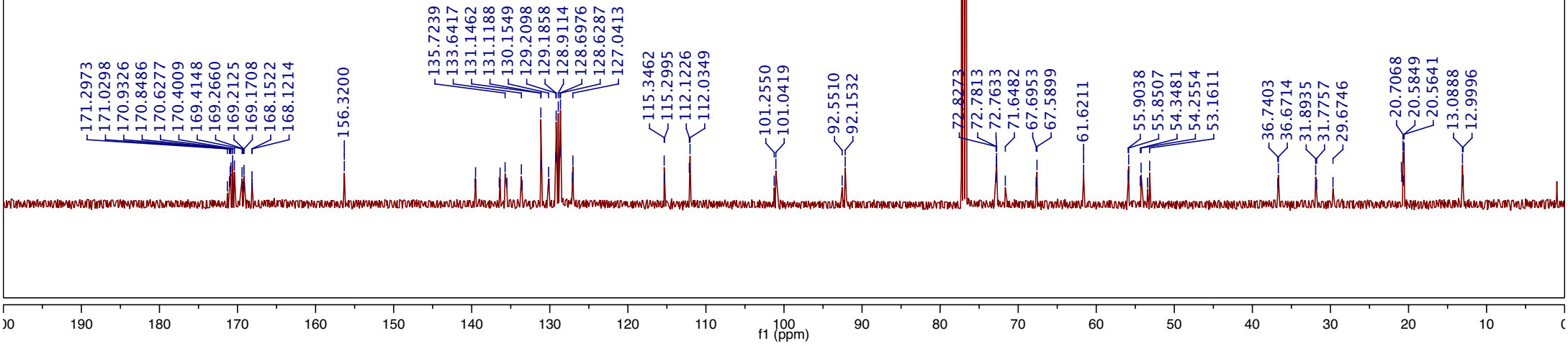


1,3,4,6-Tetra-*O*-acetyl-2-[(*N*-((2-(1-4-chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetyl)-L-phenylalanyl)-amino]-2-deoxy-β-D-glucopyranose, 6b

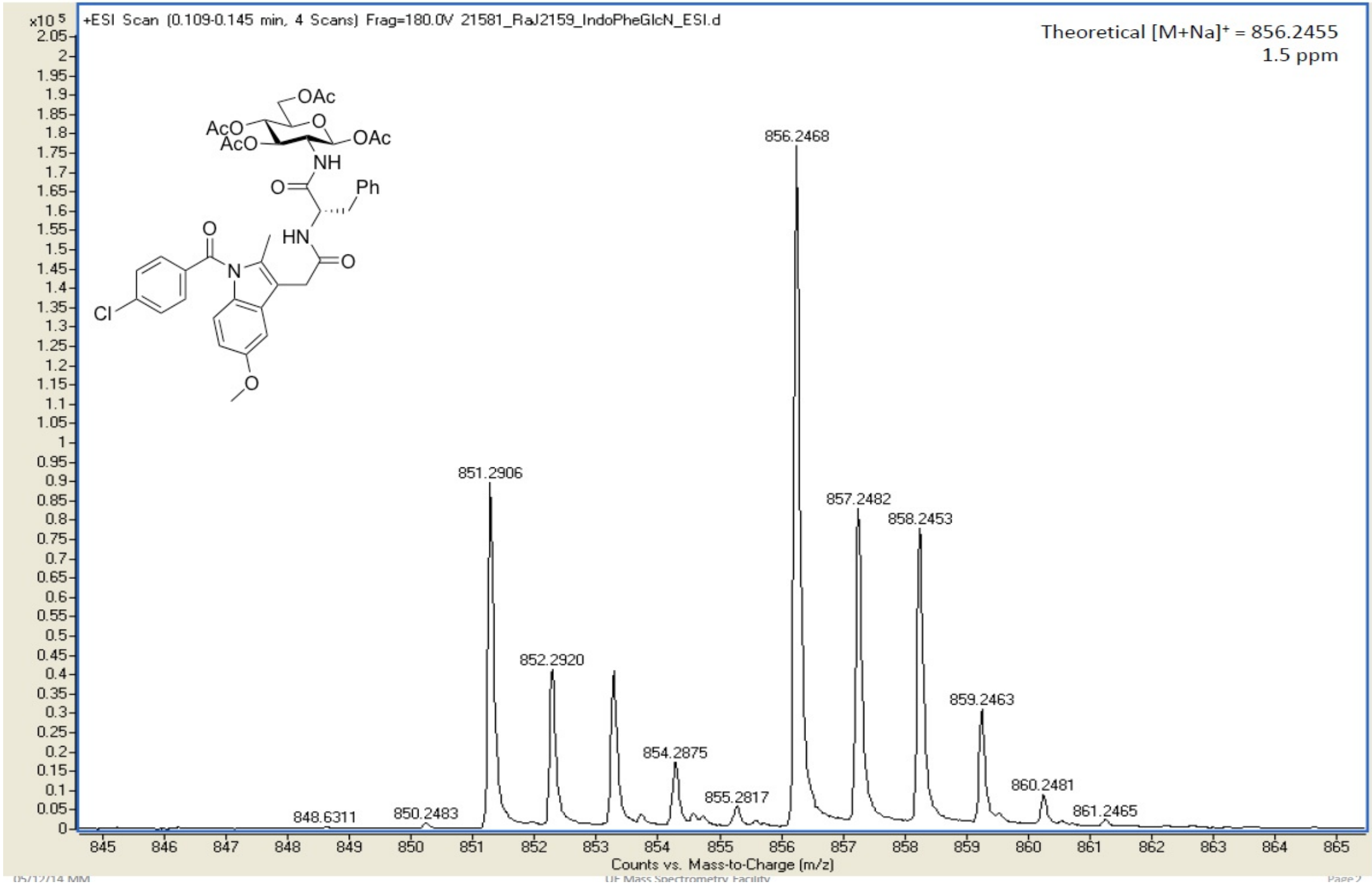




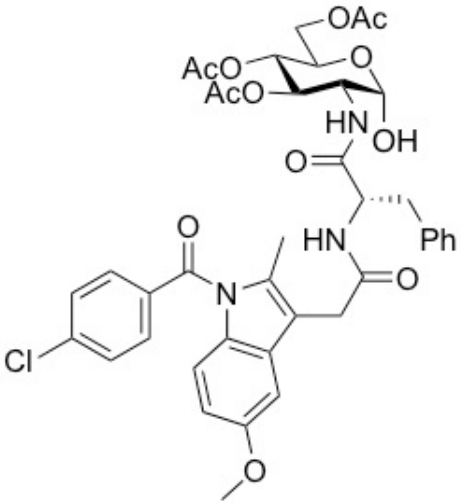


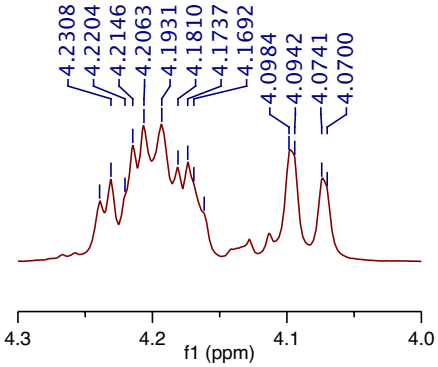


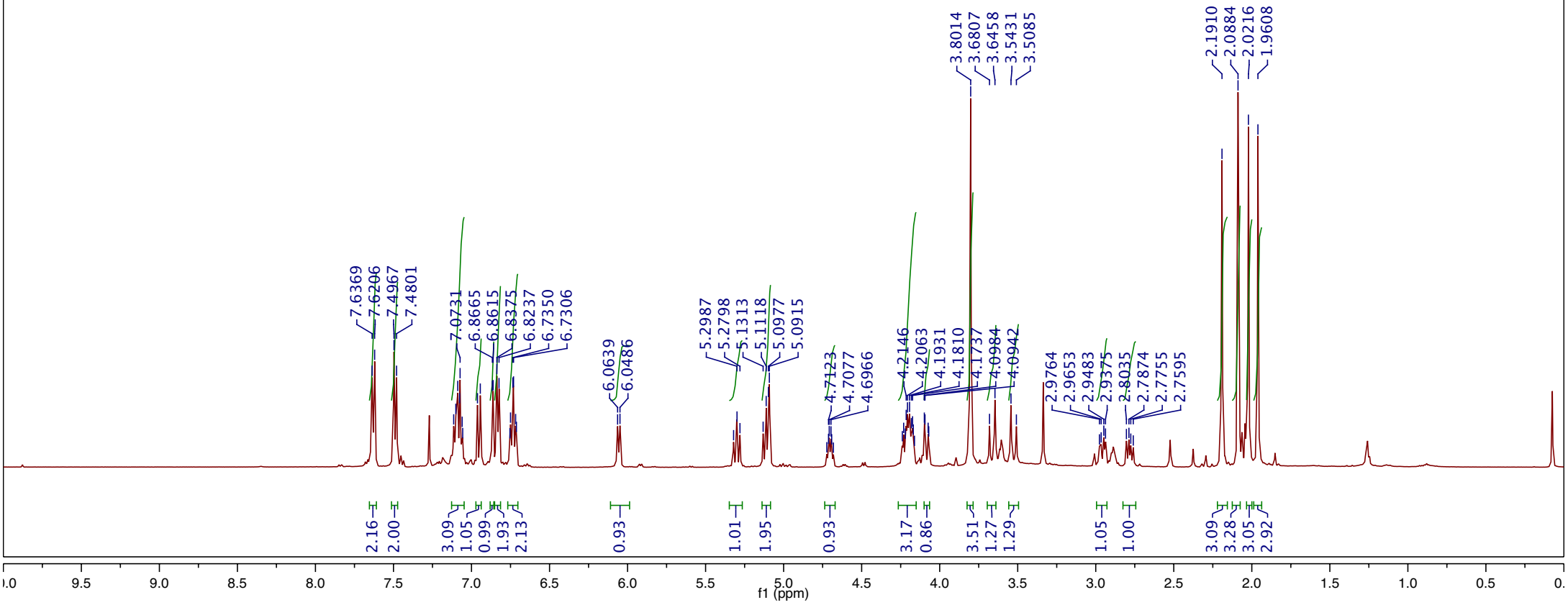
1,3,4,6-Tetra-*O*-acetyl-2-[(*N*-((2-(1-4-chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetyl)-L-phenylalanyl)-amino]-2-deoxy-β-D-glucopyranose, 6b



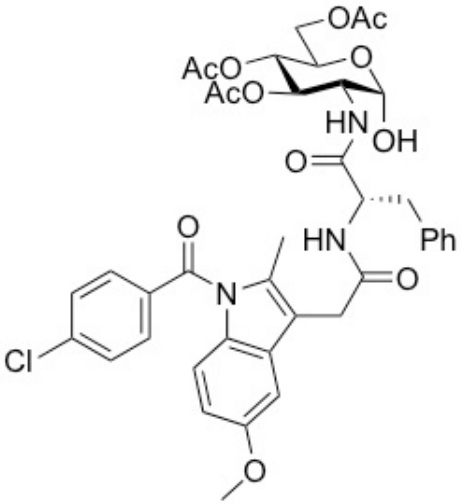
3,4,6-Tri-*O*-acetyl-2-[(*N*-((2-(1-4-chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetyl)-L-phenylalanyl)-amino]-2-deoxy-β-D-glucopyranose, 6b'

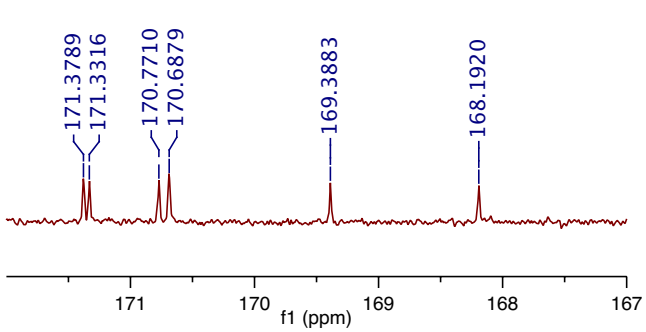


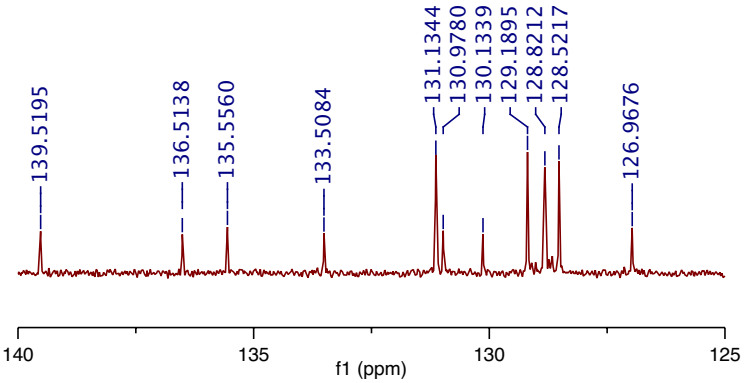


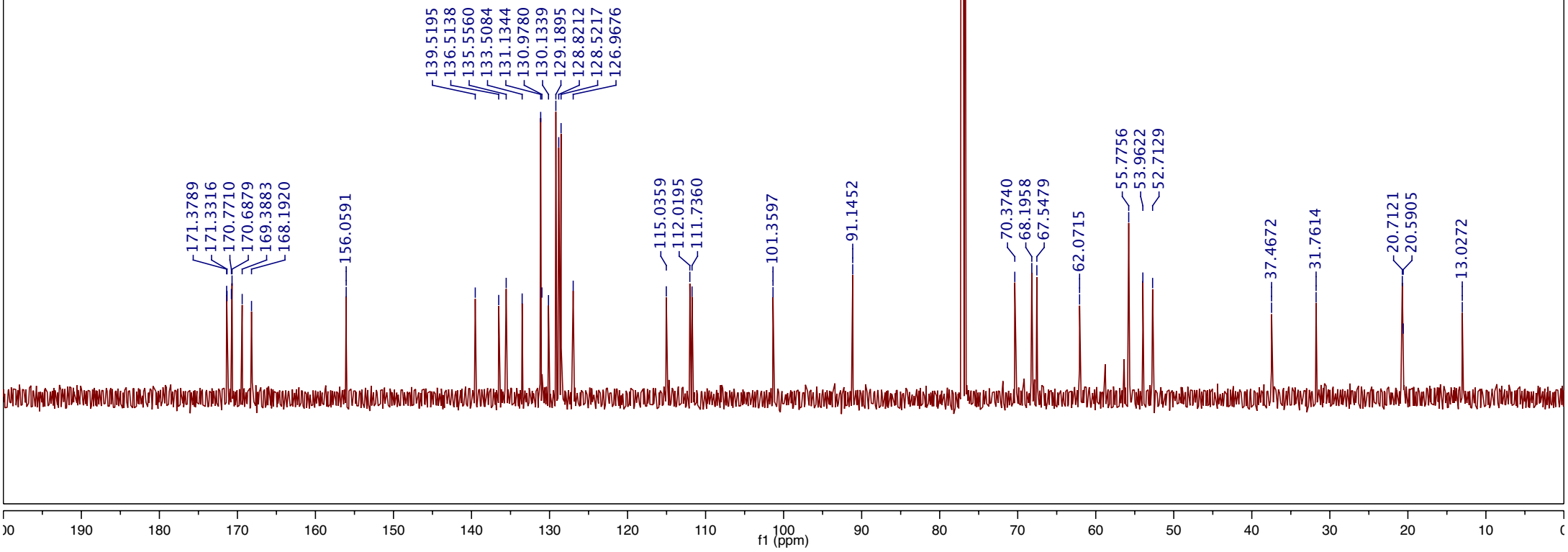


3,4,6-Tri-*O*-acetyl-2-[(*N*-((2-(1-4-chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetyl)-L-phenylalanyl)-amino]-2-deoxy-β-D-glucopyranose, 6b'

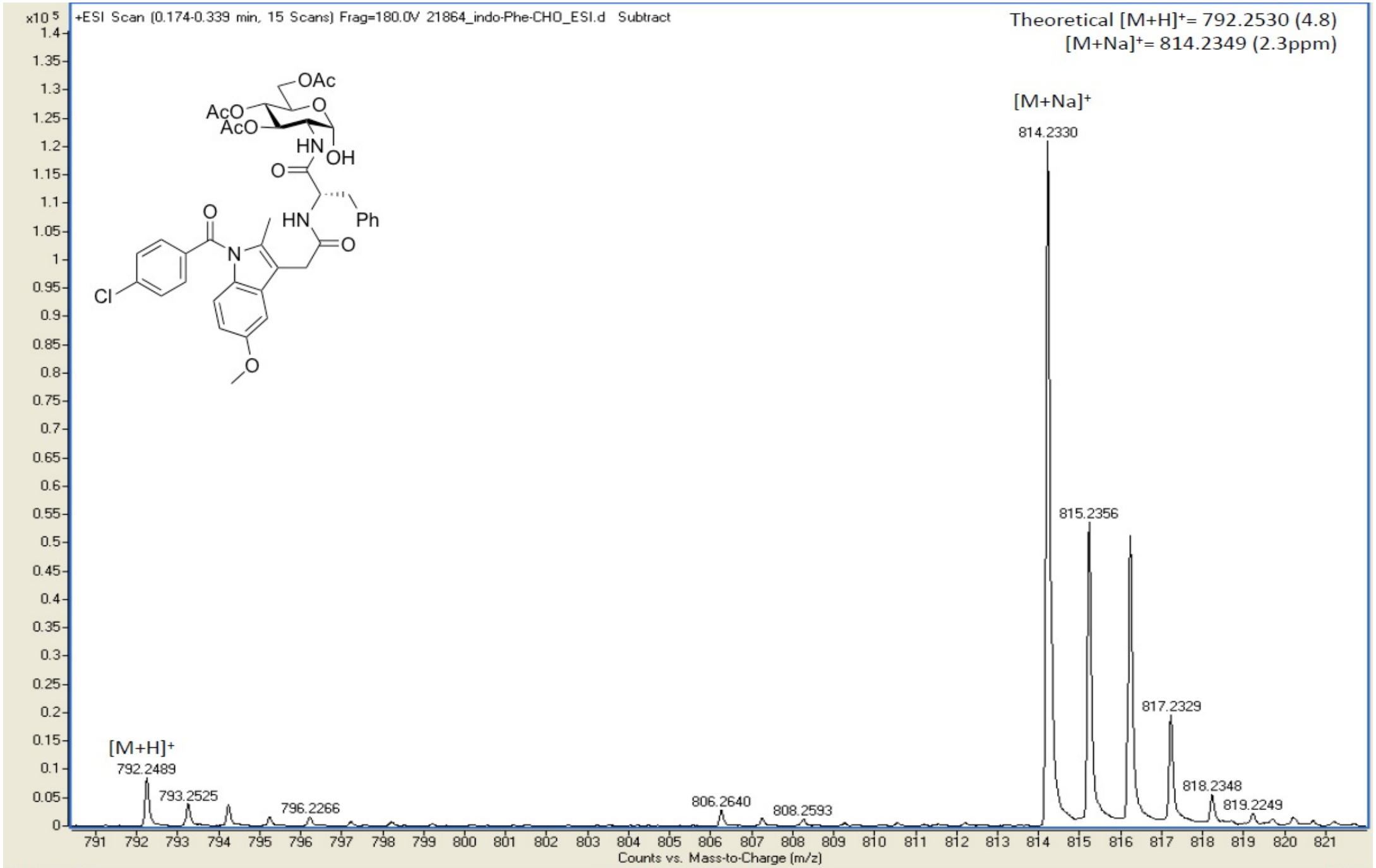




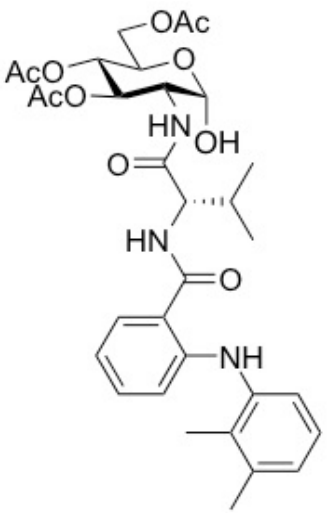




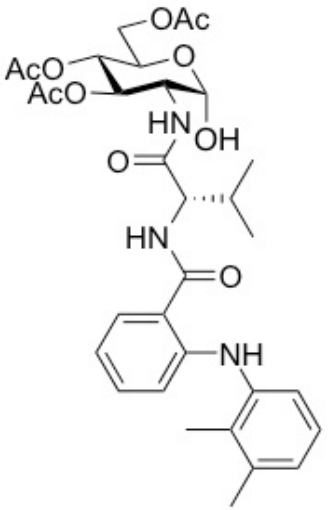
3,4,6-Tri-*O*-acetyl-2-[(*N*-((2-(1-4-chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetyl)-L-phenylalanyl)-amino]-2-deoxy-β-D-glucopyranose, 6b'



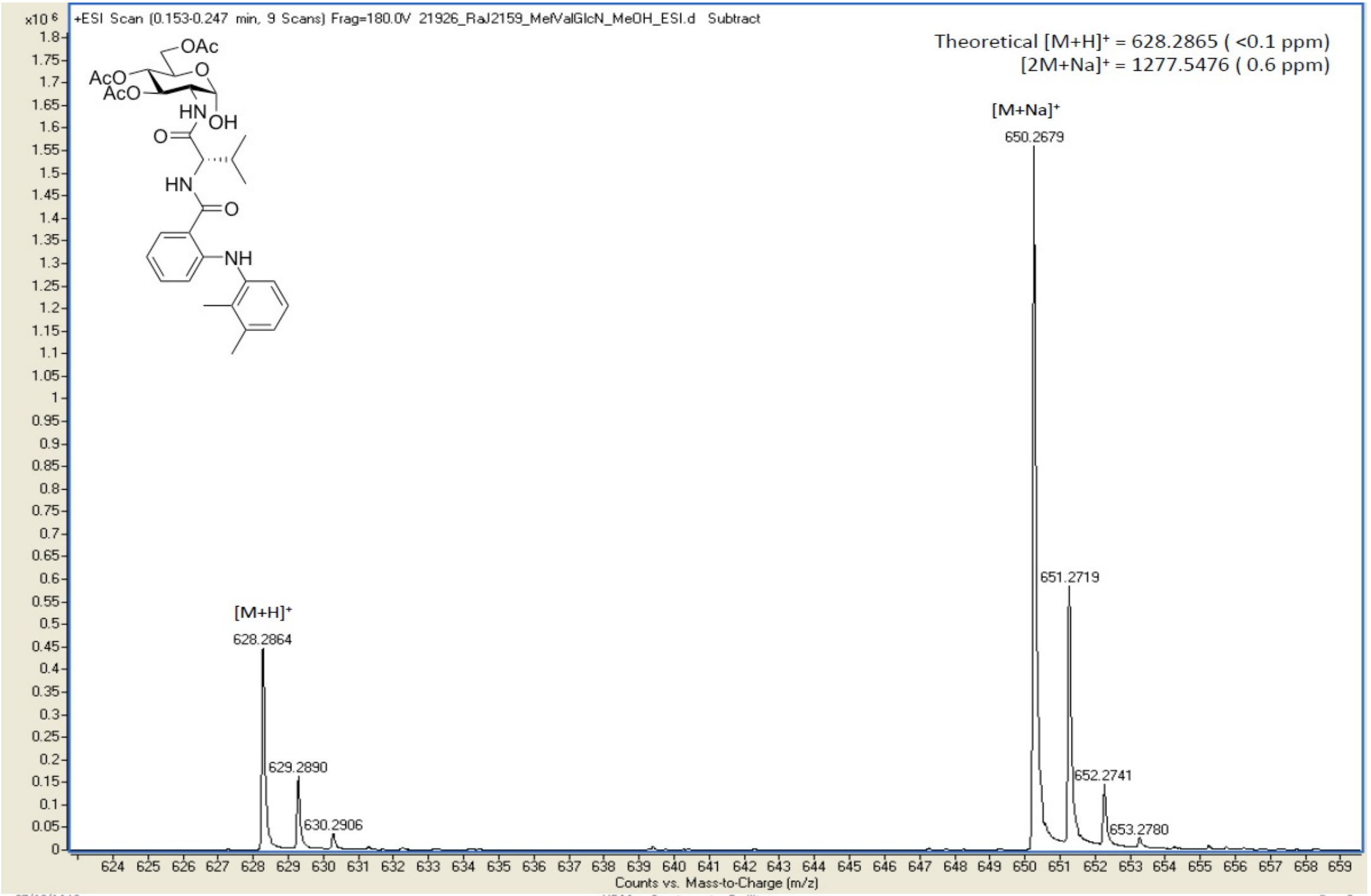
3,4,6-Tri-*O*-acetyl-2-[(*N*-(2-((2,3-(dimethylphenyl)amino)benzoyl)-L-valyl)-amino]-2-deoxy-β-D-glucopyranose, 6c'



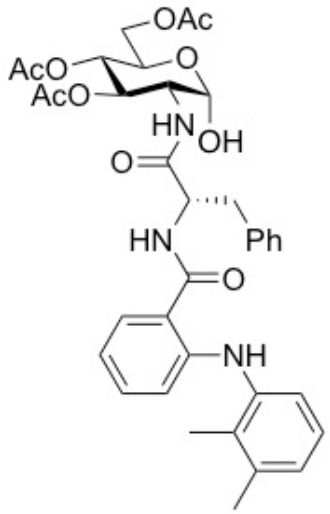
3,4,6-Tri-*O*-acetyl-2-[(*N*-(2-((2,3-(dimethylphenyl)amino)benzoyl)-L-valyl)-amino]-2-deoxy-β-D-glucopyranose, 6c'



3,4,6-Tri-*O*-acetyl-2-[(*N*-(2-((2,3-(dimethylphenyl)amino)benzoyl)-L-valyl)-amino]-2-deoxy-β-D-glucopyranose, 6c'



3,4,6-Tri-*O*-acetyl-2-[(*N*-(2-((2,3-(dimethylphenyl)amino)benzoyl)-L-phenylalanyl)-amino]-2-deoxy-β-D-glucopyranose, 6d'



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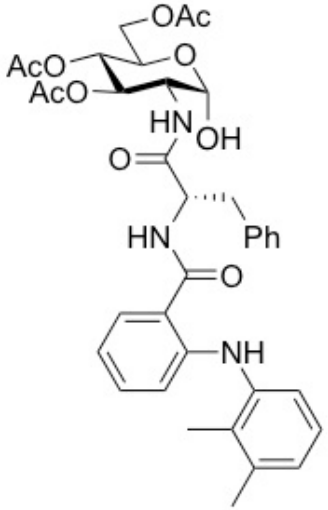
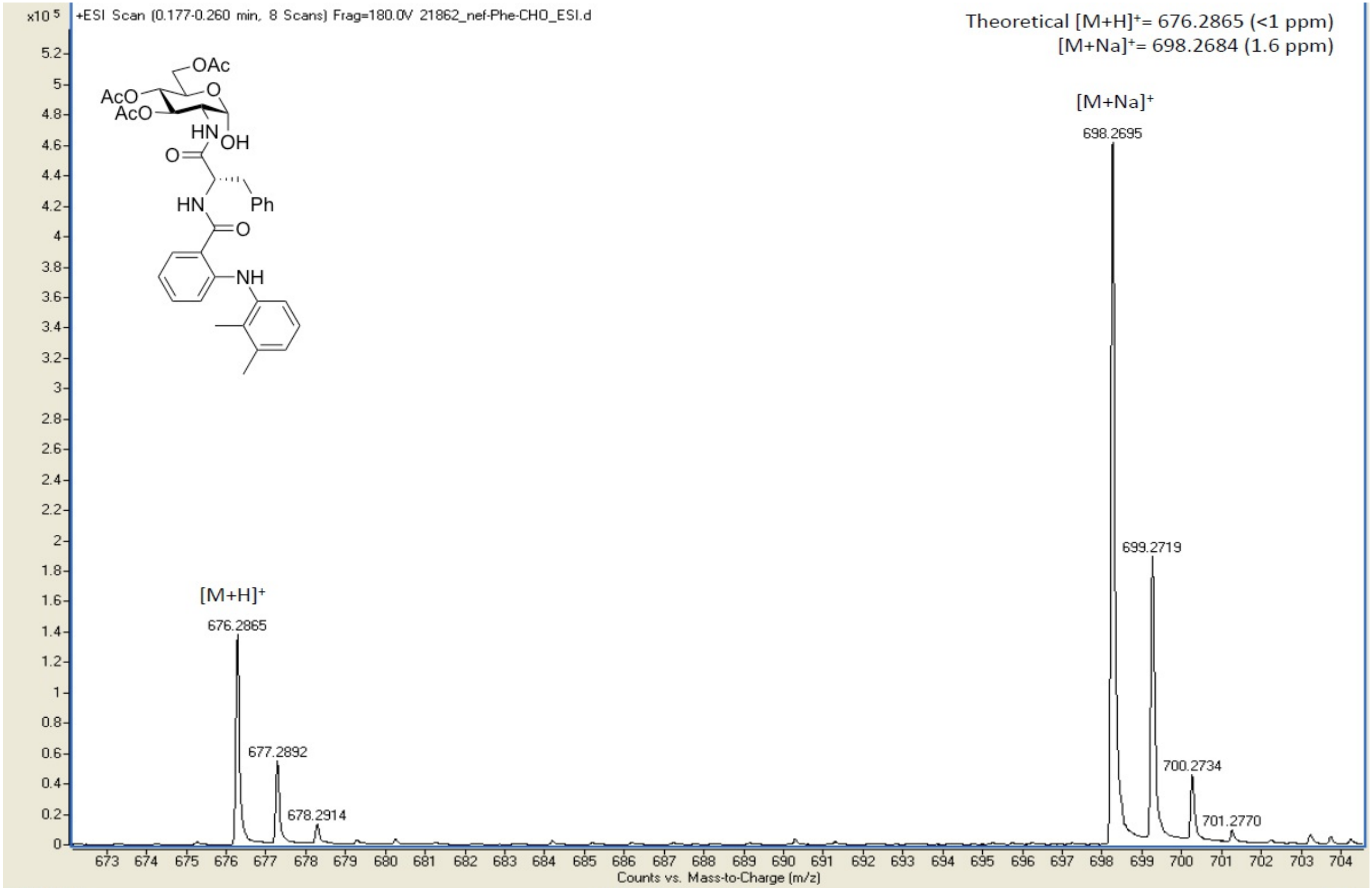
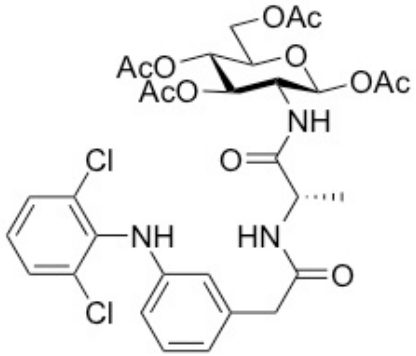


3,4,6-Tri-*O*-acetyl-2-[(*N*-(2-((2,3-(dimethylphenyl)amino)benzoyl)-L-phenylalanyl)-amino]-2-deoxy-β-D-glucopyranose, 6d'



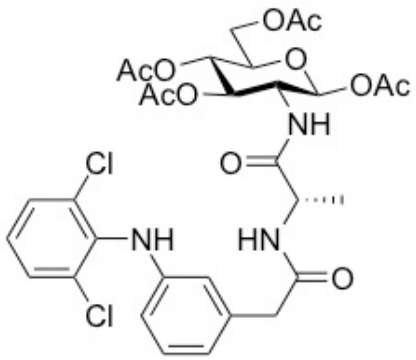
1,3,4,6-Tetra-*O*-acetyl-2-[(*N*-(2-((2,3-(dimethylphenyl)amino)benzoyl)-L-alanyl)-amino]-2-deoxy-β-D-glucopyranose, 6e

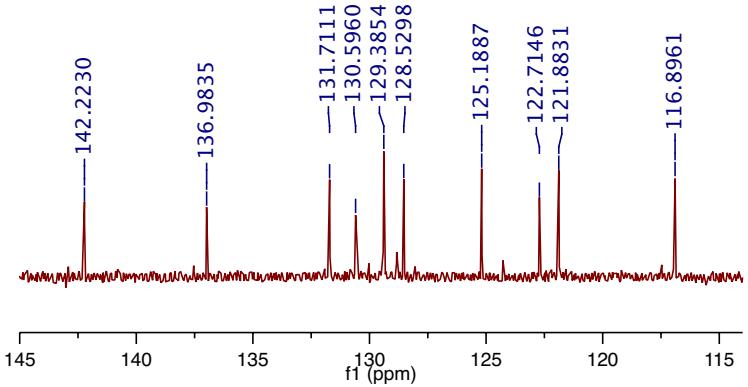
Rotamer I

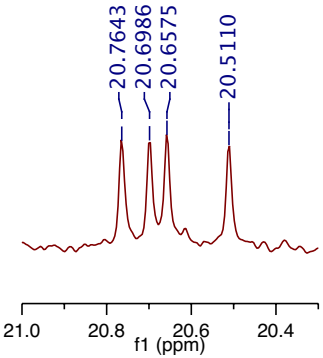


1,3,4,6-Tetra-*O*-acetyl-2-[(*N*-(2-((2,3-(dimethylphenyl)amino)benzoyl)-L-alanyl)-amino]-2-deoxy-β-D-glucopyranose, 6e

Rotamer I

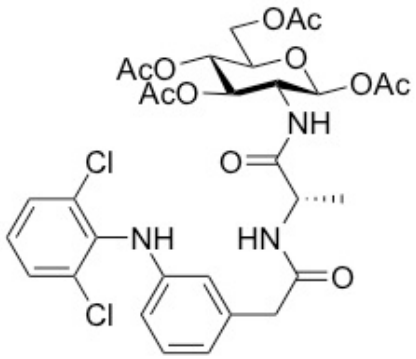


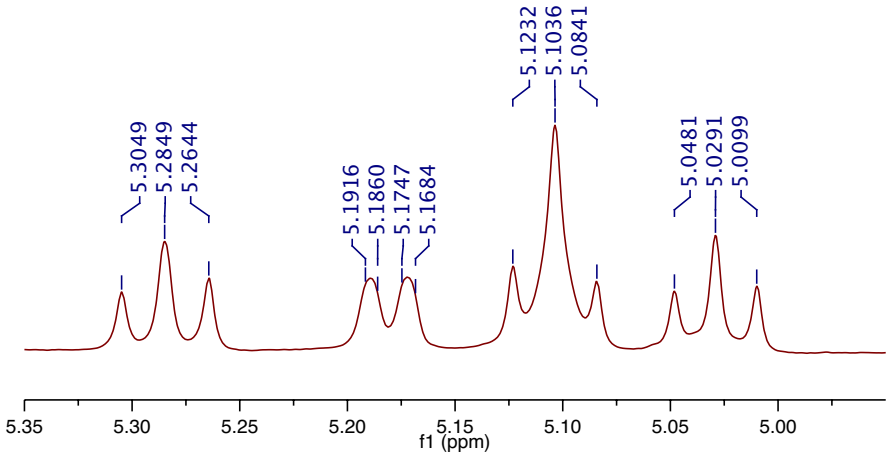




1,3,4,6-Tetra-*O*-acetyl-2-[(*N*-(2-((2,3-(dimethylphenyl)amino)benzoyl)-L-alanyl)-amino]-2-deoxy-β-D-glucopyranose, 6e

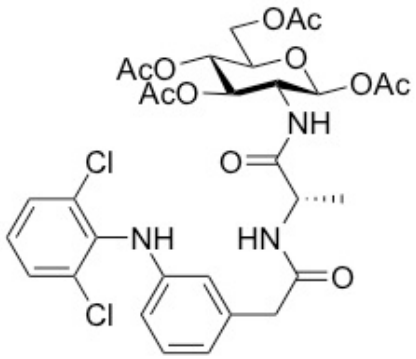
Rotamer I and Rotamer II

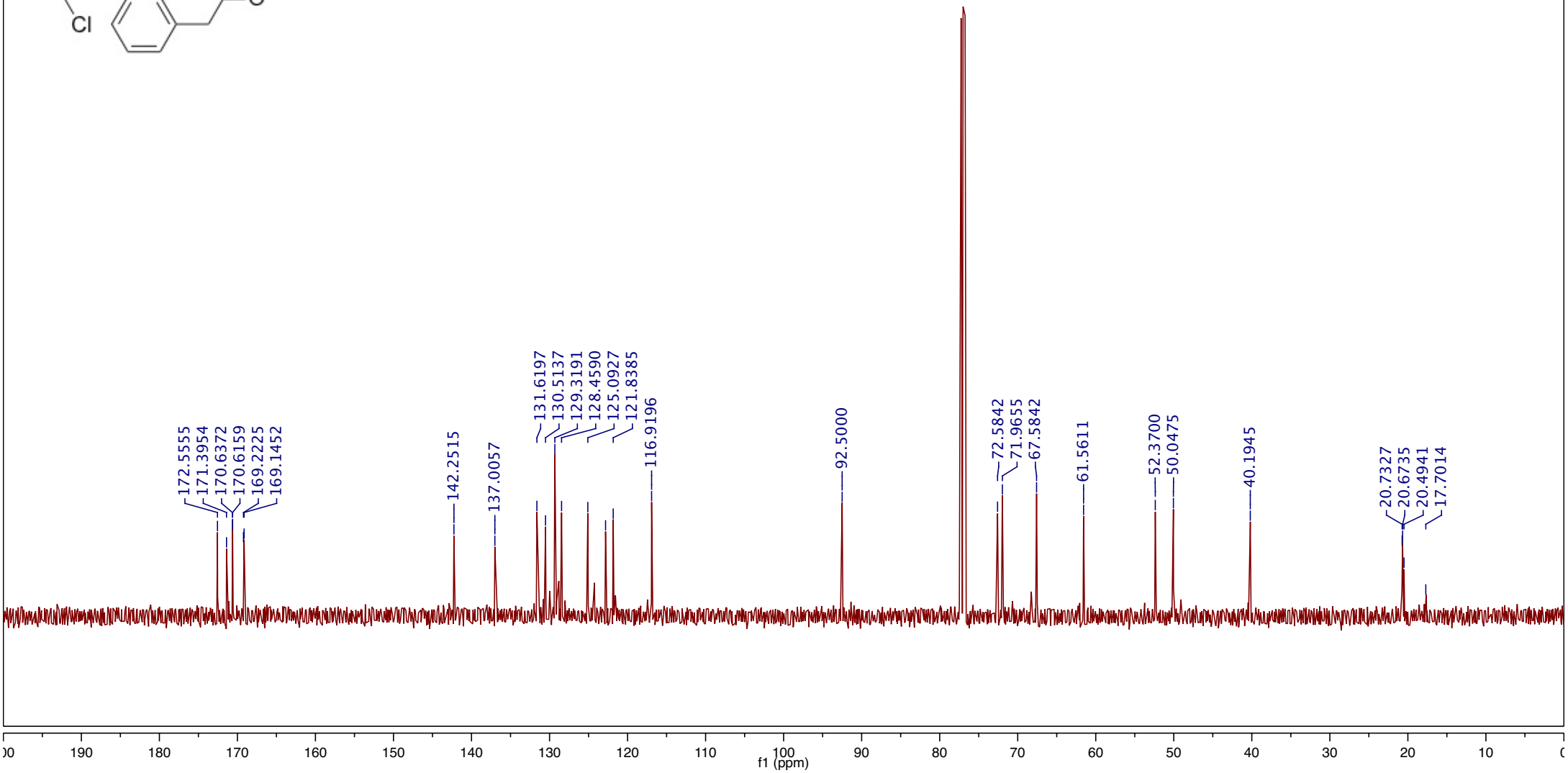




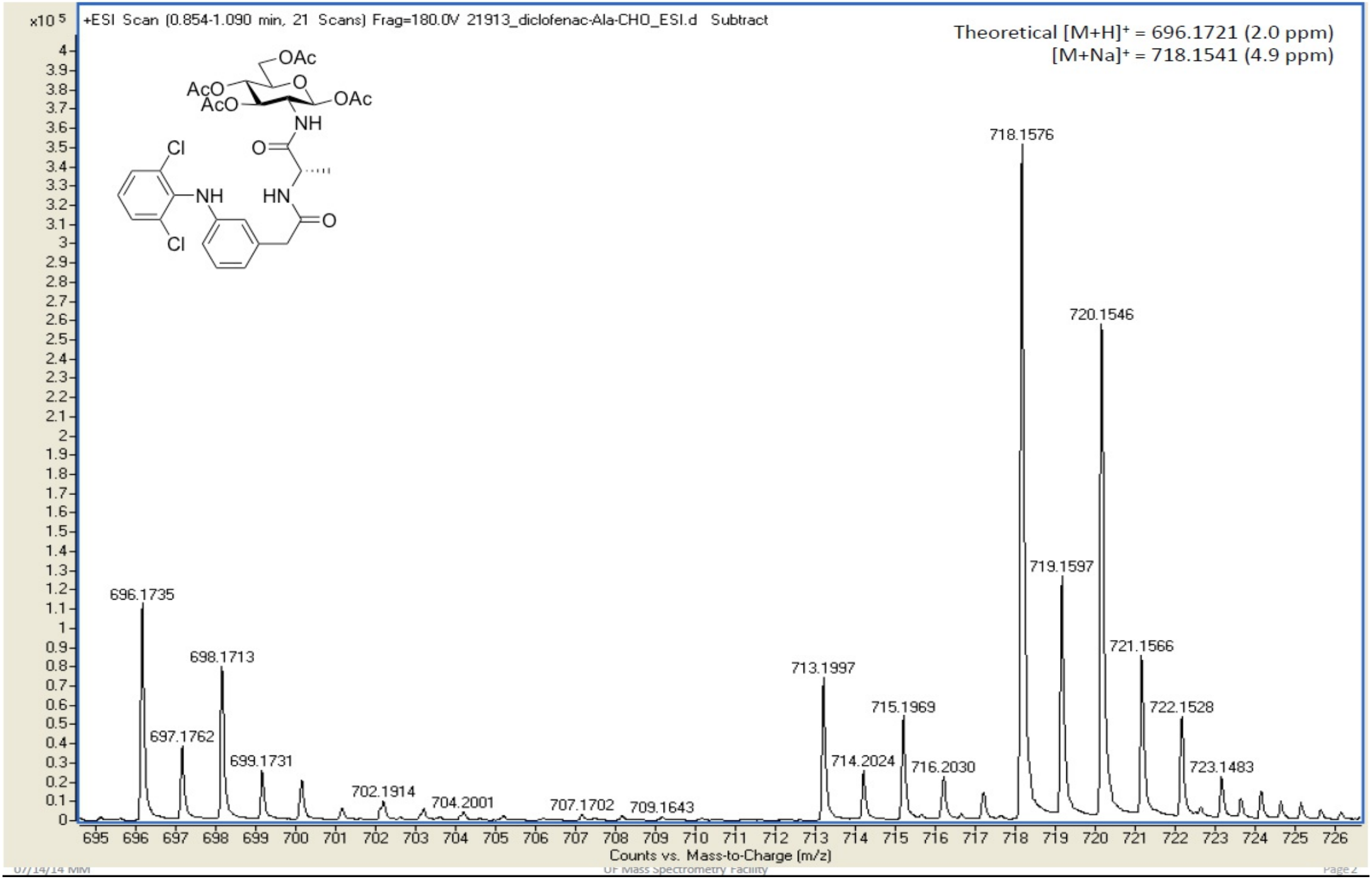
1,3,4,6-Tetra-*O*-acetyl-2-[(*N*-(2-((2,3-(dimethylphenyl)amino)benzoyl)-L-alanyl)-amino]-2-deoxy-β-D-glucopyranose, 6e

Rotamer I and Rotamer II

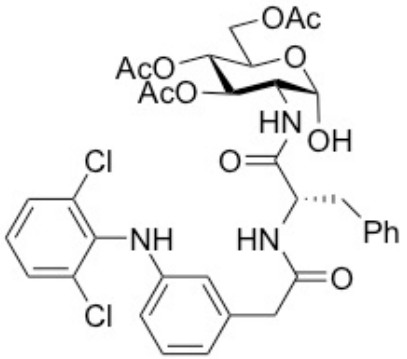


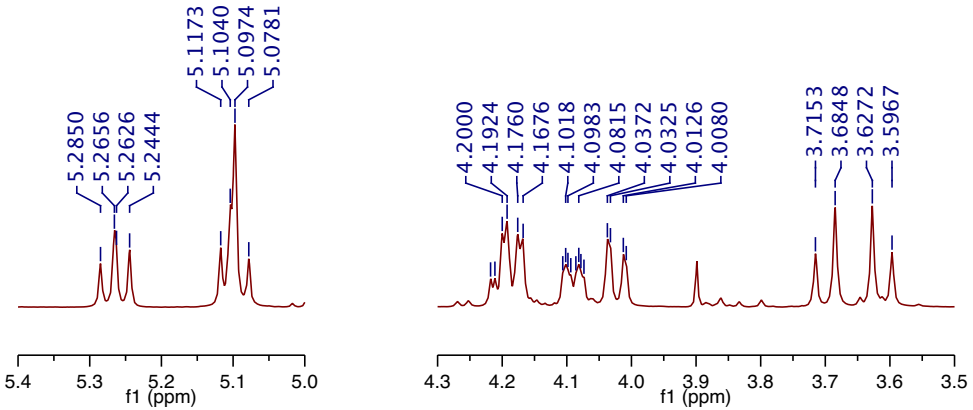


1,3,4,6-Tetra-*O*-acetyl-2-[(*N*-(2-((2,3-(dimethylphenyl)amino)benzoyl)-L-alanyl)-amino]-2-deoxy-β-D-glucopyranose, 6e

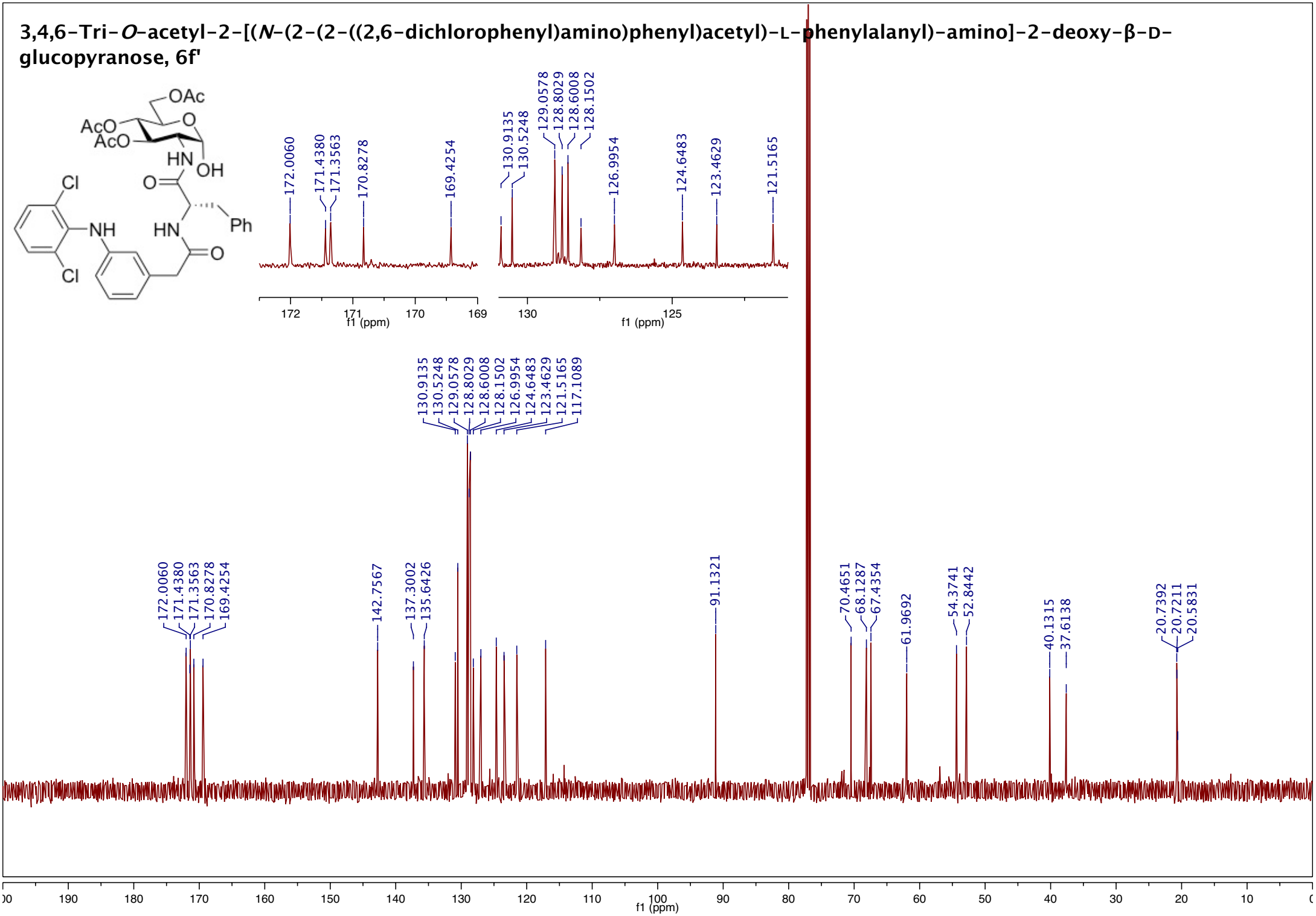


3,4,6-Tri-*O*-acetyl-2-[(*N*-(2-(2-((2,6-dichlorophenyl)amino)phenyl)acetyl)-L-phenylalanyl)-amino]-2-deoxy-β-D-glucopyranose, 6f'

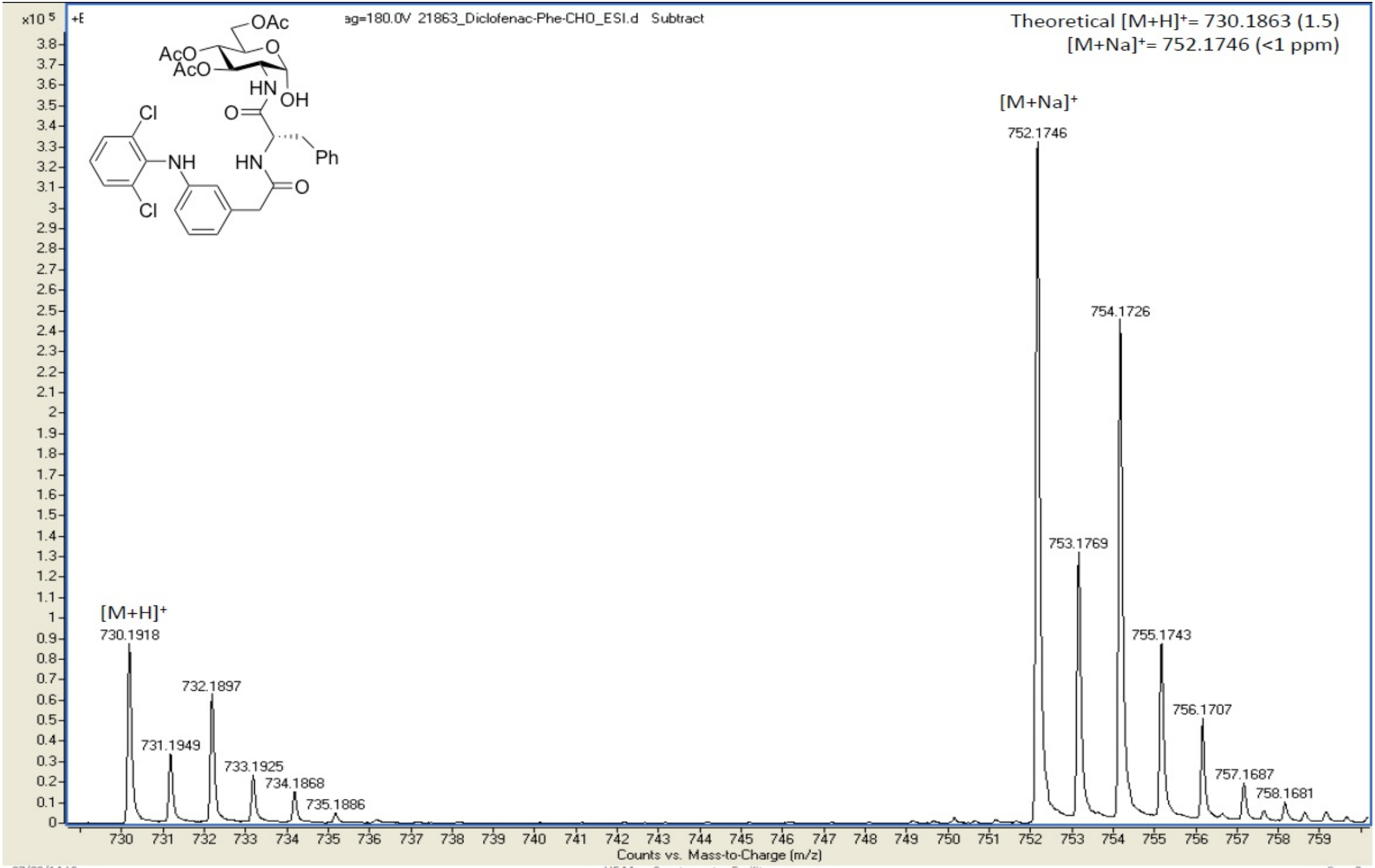




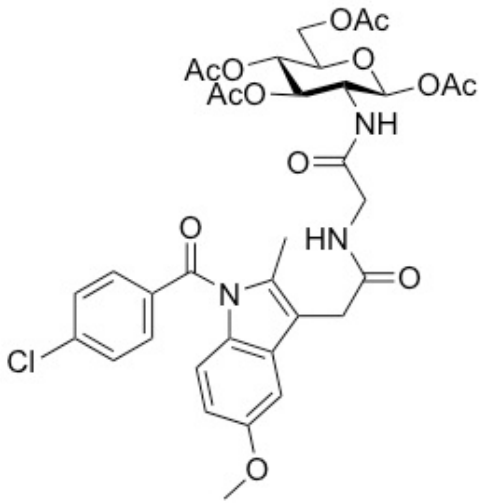
3,4,6-Tri-*O*-acetyl-2-[(*N*-(2-(2-((2,6-dichlorophenyl)amino)phenyl)acetyl)-L-phenylalanyl)-amino]-2-deoxy-β-D-glucopyranose, 6f'

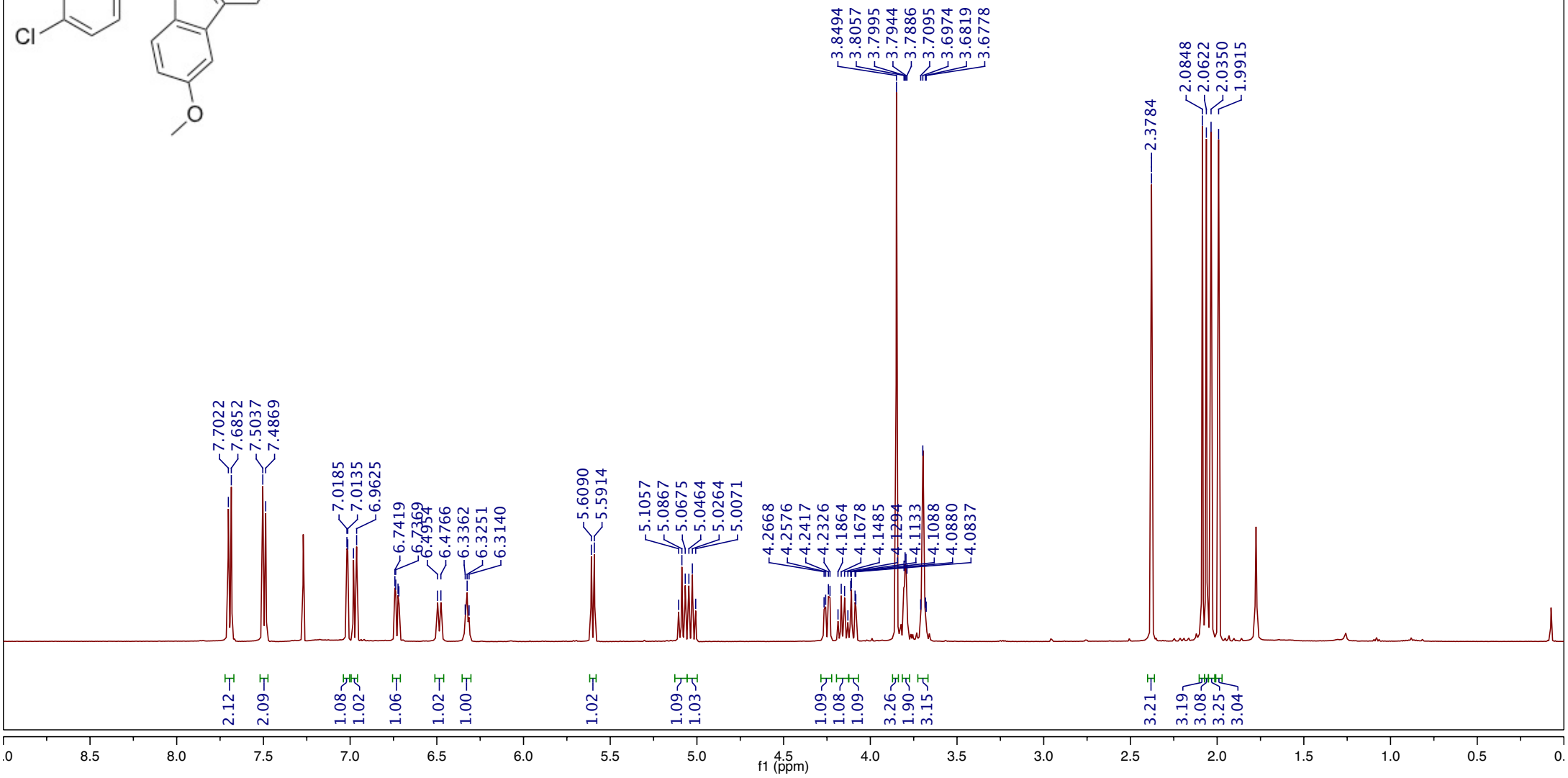


3,4,6-Tri-*O*-acetyl-2-[(*N*-(2-(2-((2,6-dichlorophenyl)amino)phenyl)acetyl)-L-phenylalanyl)-amino]-2-deoxy-β-D-glucopyranose, 6f'

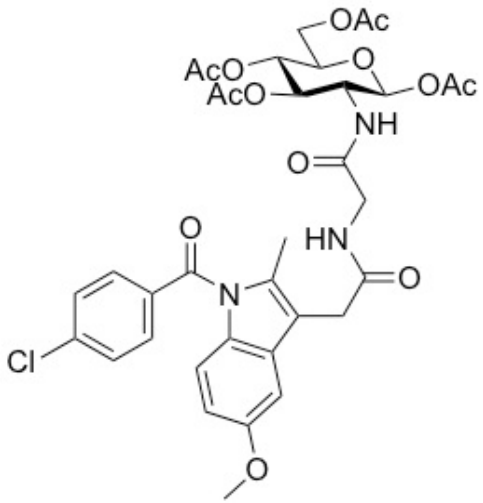


1,3,4,6-Tetra-*O*-acetyl-2-[(*N*-((2-(1-4-chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetyl)-glycyl)-amino]-2-deoxy-β-D-glucopyranose, 6g

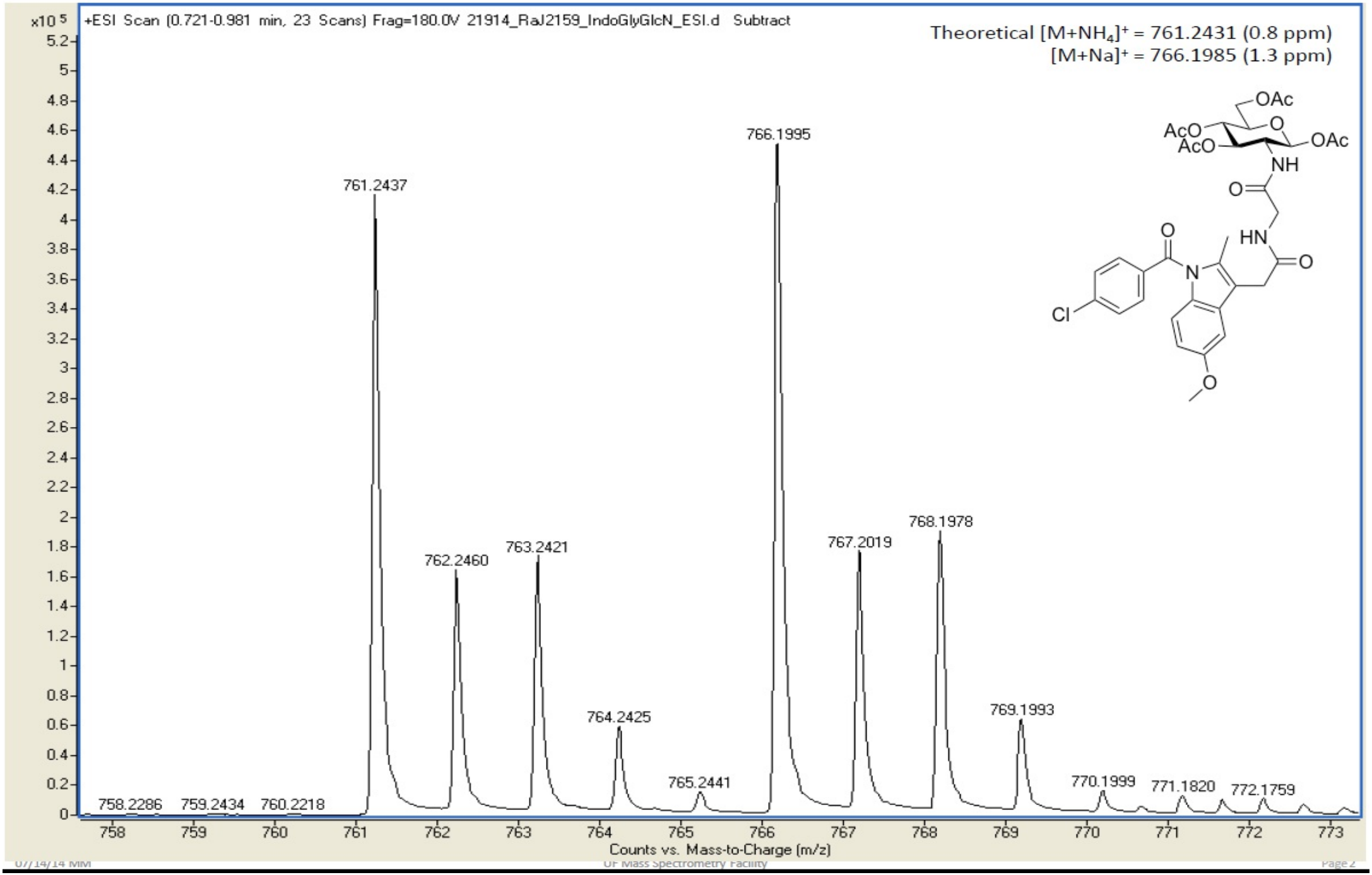




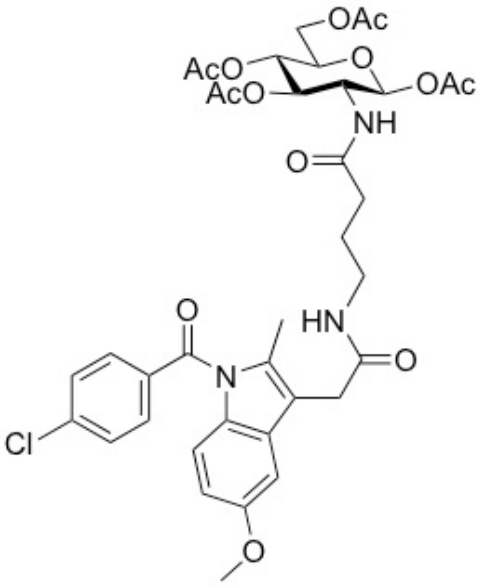
1,3,4,6-Tetra-*O*-acetyl-2-[(*N*-((2-(1-4-chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetyl)-glycyl)-amino]-2-deoxy-β-D-glucopyranose, 6g



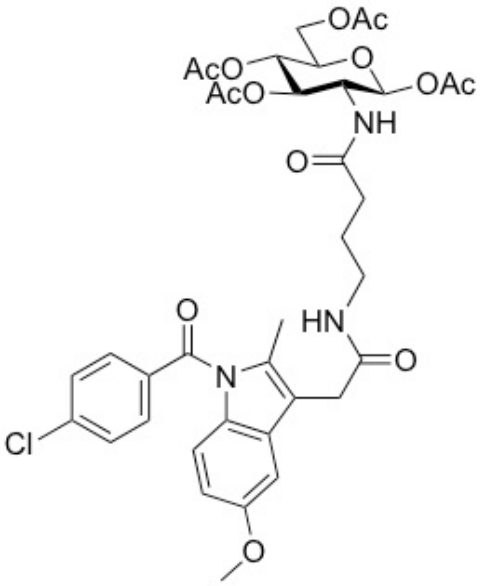
1,3,4,6-Tetra-*O*-acetyl-2-[(*N*-((2-(1-4-chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetyl)-glycyl)-amino]-2-deoxy-β-D-glucopyranose, 6g



1,3,4,6-Tetra-*O*-acetyl-2-[(*N*-((2-(1-4-chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetyl)-4-aminobutanoyl)-amino]-2-deoxy-β-D-glucopyranose, 6h



1,3,4,6-Tetra-*O*-acetyl-2-[(*N*-((2-(1-4-chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetyl)-4-aminobutanoyl)-amino]-2-deoxy-β-D-glucopyranose, 6h



1,3,4,6-Tetra-*O*-acetyl-2-[(*N*-((2-(1-4-chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetyl)-4-aminobutanoyl)-amino]-2-deoxy-β-D-glucopyranose, 6h