

Microwave-assisted heating reactions of *N*-acetylglucosamine (GlcNAc) in sulfolane as a method generating 1,6-anhydrosugars consisting of amino monosaccharide backbones

Harumi Kaga ^{1,*}, Masaru Enomoto ², Hiroki Shimizu ³, Izuru Nagashima ³, Keigo Matsuda ⁴, Seigou Kawaguchi ⁵ and Atsushi Narumi ^{5,*}

¹ National Institute of Advanced Industrial Science and Technology (AIST); Sapporo 062-8517, Japan; h.kaga@aist.go.jp

² Laboratory of Applied Bioorganic Chemistry, Graduate School of Agricultural Science, Tohoku University; 468-1 Aramaki Aza-Aoba, Aoba-ku, Sendai 980-8572, Japan; masaru.enomoto.a2@tohoku.ac.jp

³ Bioproduction Research Institute, Department of Life Science and Biotechnology, National Institute of Advanced Industrial Science and Technology (AIST); 1-1-1 Higashi, Tsukuba, Ibaraki, 305-8568 Japan; hiroki.shimizu@aist.go.jp, izu.nagashima@aist.go.jp

⁴ Department of Chemistry and Chemical Engineering, Graduate School of Science and Engineering, Yamagata University; Jonan 4-3-16, Yonezawa 992-8510, Japan; matsuda@yz.yamagata-u.ac.jp

⁵ Department of Organic Materials Science, Graduate School of Organic Materials Science, Yamagata University; Jonan 4-3-16, Yonezawa 992-8510, Japan; skawagu@yz.yamagata-u.ac.jp, narumi@yz.yamagata-u.ac.jp

* Correspondence: h.kaga@aist.go.jp; Tel.: +81-11-857-8921 (H.K.) and narumi@yz.yamagata-u.ac.jp; Tel.: +81-238-26-3829 (A.N.)

Contents

1. Supporting data for AGPNAc.....	2
Figure S1.1: ¹ H NMR spectrum of AGPNAc (400 MHz, D ₂ O).	2
Figure S1.2: ¹³ C NMR spectrum of AGPNAc (100 MHz, D ₂ O).	3
Figure S1.3: ESI-HRMS of AGPNAc along with analytical data.....	4
2. Supporting data for AGFNAc	5
Figure S2.1: ¹ H NMR spectrum of AGFNAc (400 MHz, D ₂ O).	5
Figure S2.2: ¹³ C NMR spectrum of AGFNAc (100 MHz, D ₂ O).....	6
Figure S2.3: ESI-HRMS of AGFNAc along with analytical data.....	7
3. Supporting data for AGPNAcDA	8
Figure S3.1: ¹ H NMR spectrum of AGPNAcDA (400 MHz, CDCl ₃).	8
Figure S3.2: ¹³ C NMR spectrum of AGPNAcDA (100 MHz, CDCl ₃).....	9
Figure S3.3: ESI-HRMS of AGPNAcDA along with analytical data.	10
4. Supporting data for AGFNAcDA.....	11
Figure S4.1: ¹ H NMR spectrum of AGFNAcDA (400 MHz, CDCl ₃).....	11
Figure S4.2: ¹³ C NMR spectrum of AGFNAcDA (100 MHz, CDCl ₃).....	12
Figure S4.3: ESI-HRMS of AGFNAcDA along with analytical data.	13

1. Supporting data for AGPNAc

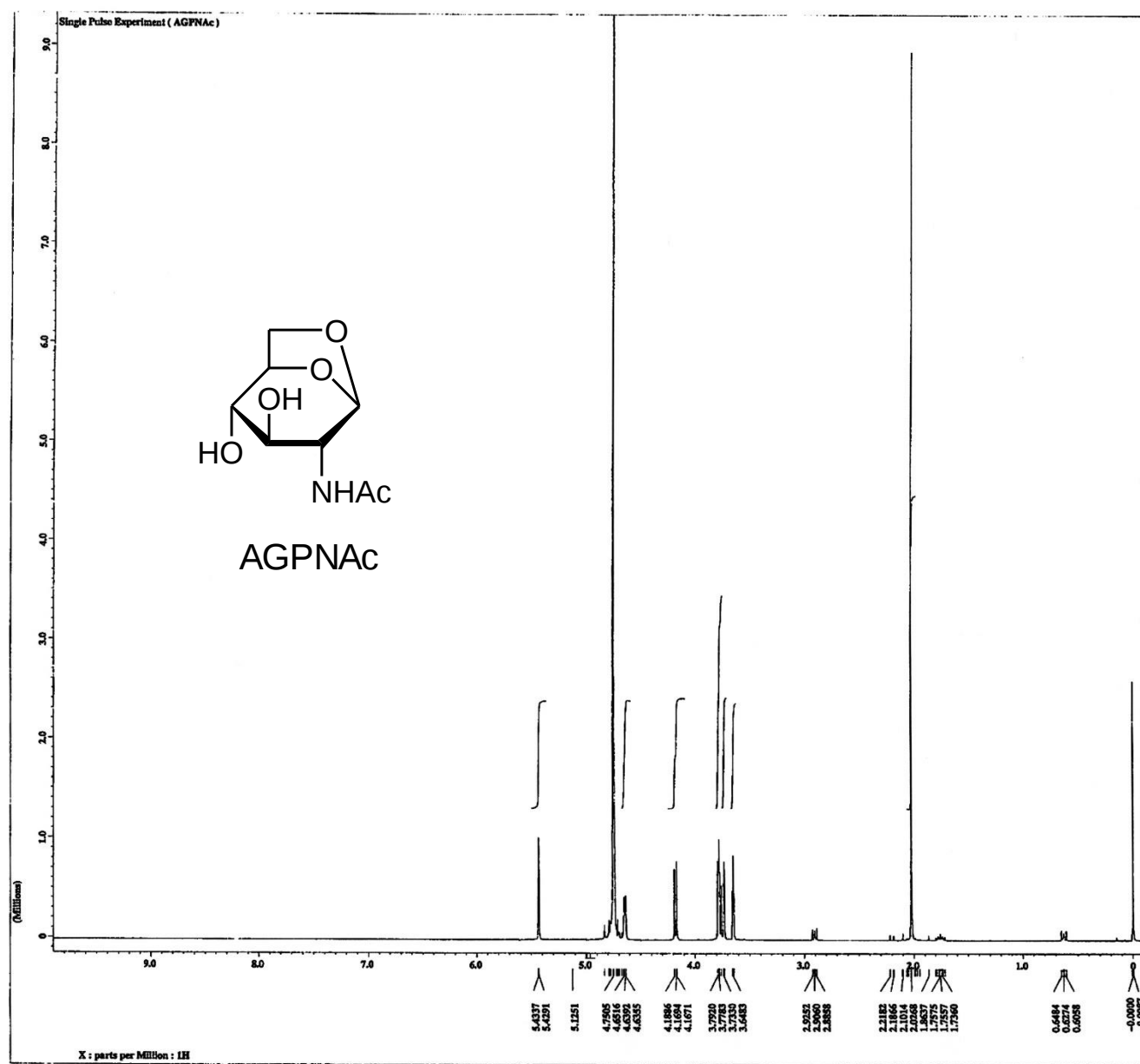


Figure S1.1: ^1H NMR spectrum of AGPNAc (400 MHz, D_2O).

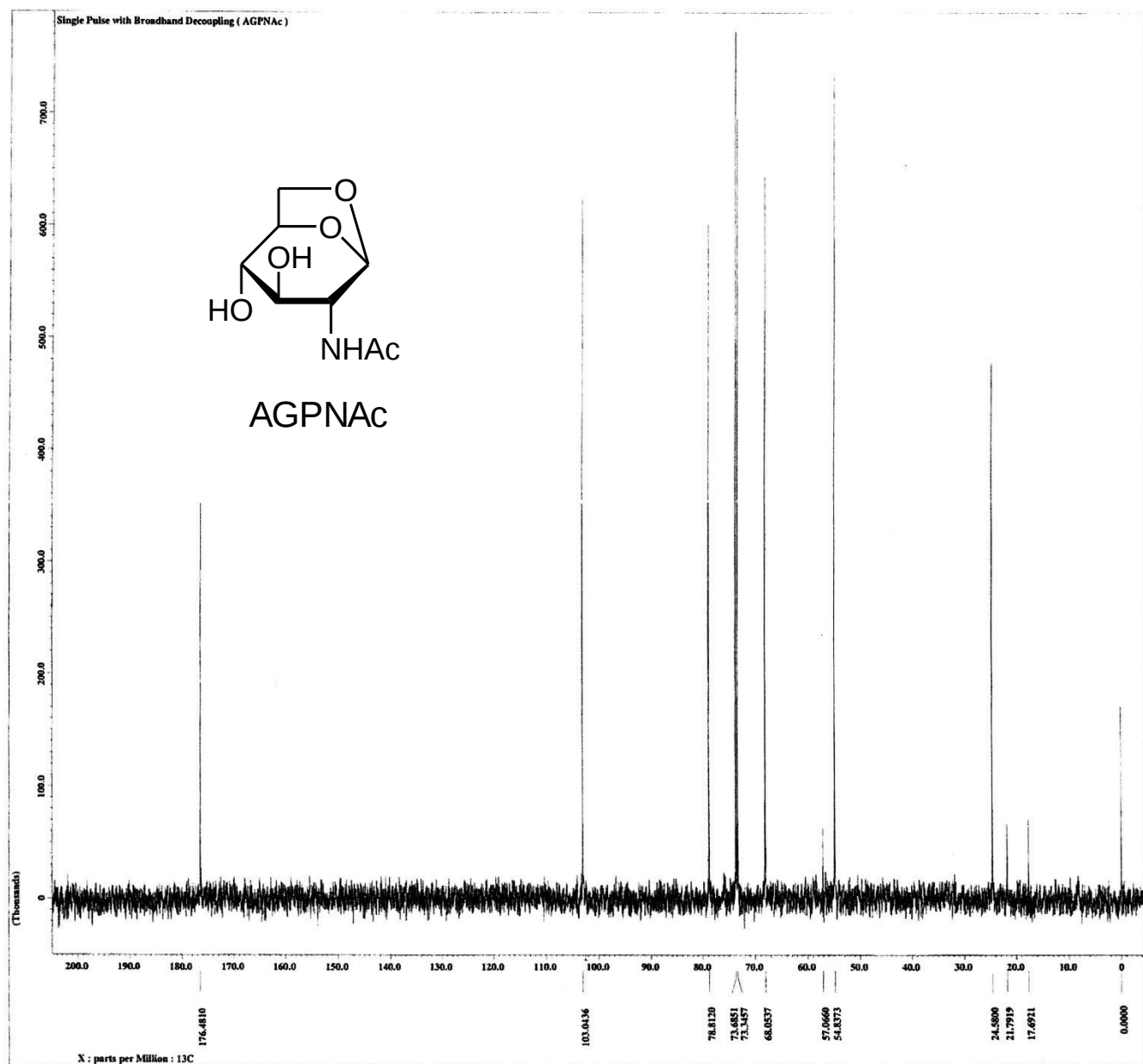
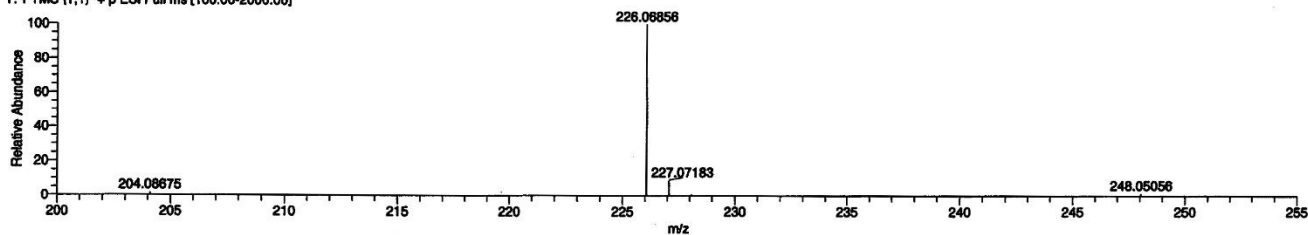


Figure S1.2: ^{13}C NMR spectrum of AGPNac (100 MHz, D_2O).

140478_AGPNAc.pn #21-24 RT: 0.29-0.32 AV: 2 NL: 1.26E8
T: FTMS (1,1) + p ESI Full ms (100.00-2000.00)



Elemental composition search on mass 226.06856

m/z= 221.06856-231.06856

Isotope Min Max

O-16 0 6

C-12 0 30

H-1 0 60

Na-23 0 1

N-14 0 1

Charge 1

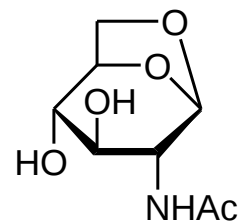
Mass tolerance 5.00 ppm

Nitrogen rule not used

RDB equiv -1.00-100.00

max results 100

m/z	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
226.06856	226.06859	-0.15	2.5	C ₈ H ₁₃ O ₅ N Na



AGPNAc

Figure S1.3: ESI-HRMS of AGPNAc along with analytical data.

2. Supporting data for AGFNAC

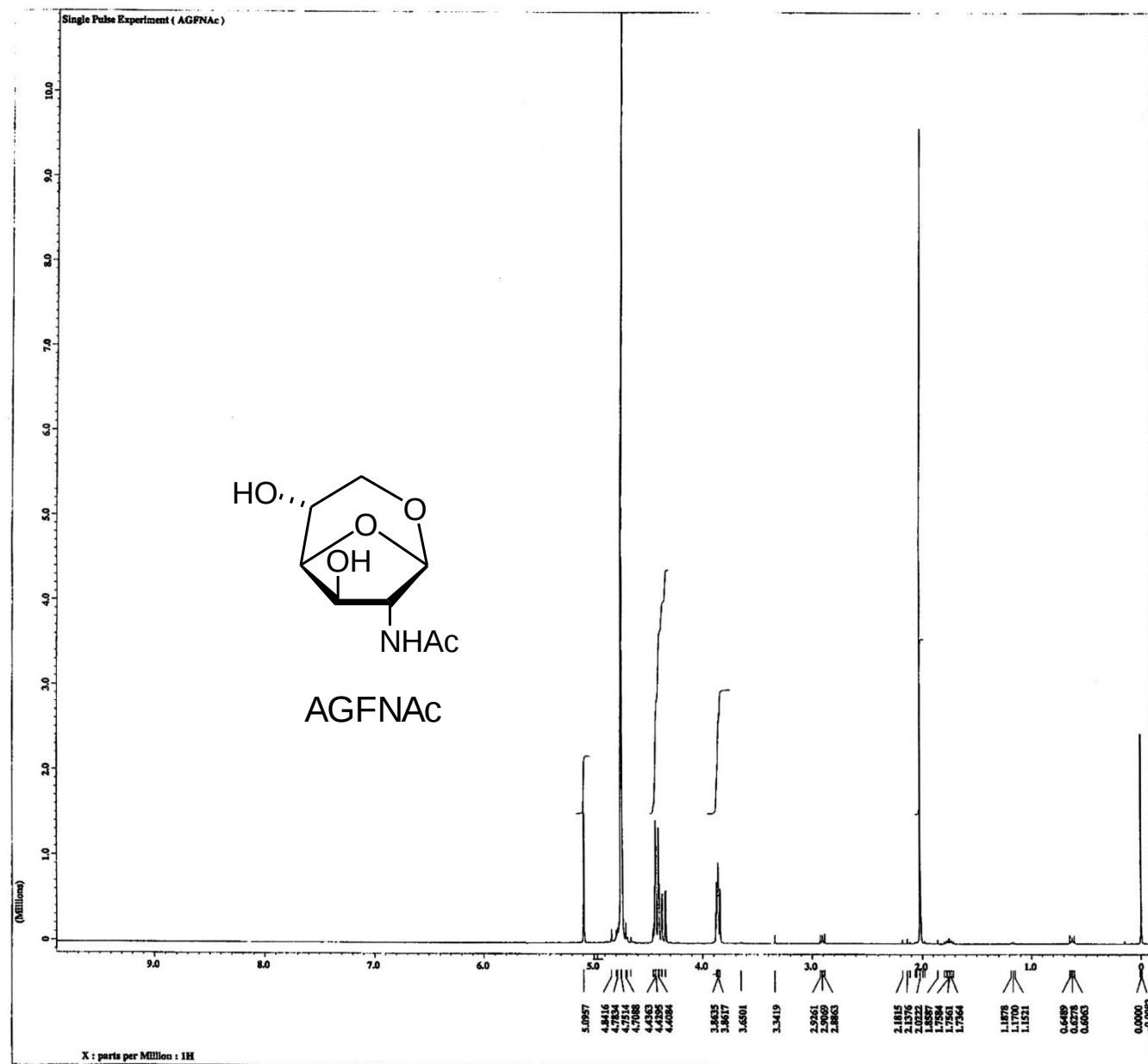


Figure S2.1: ^1H NMR spectrum of AGFNAC (400 MHz, D_2O).

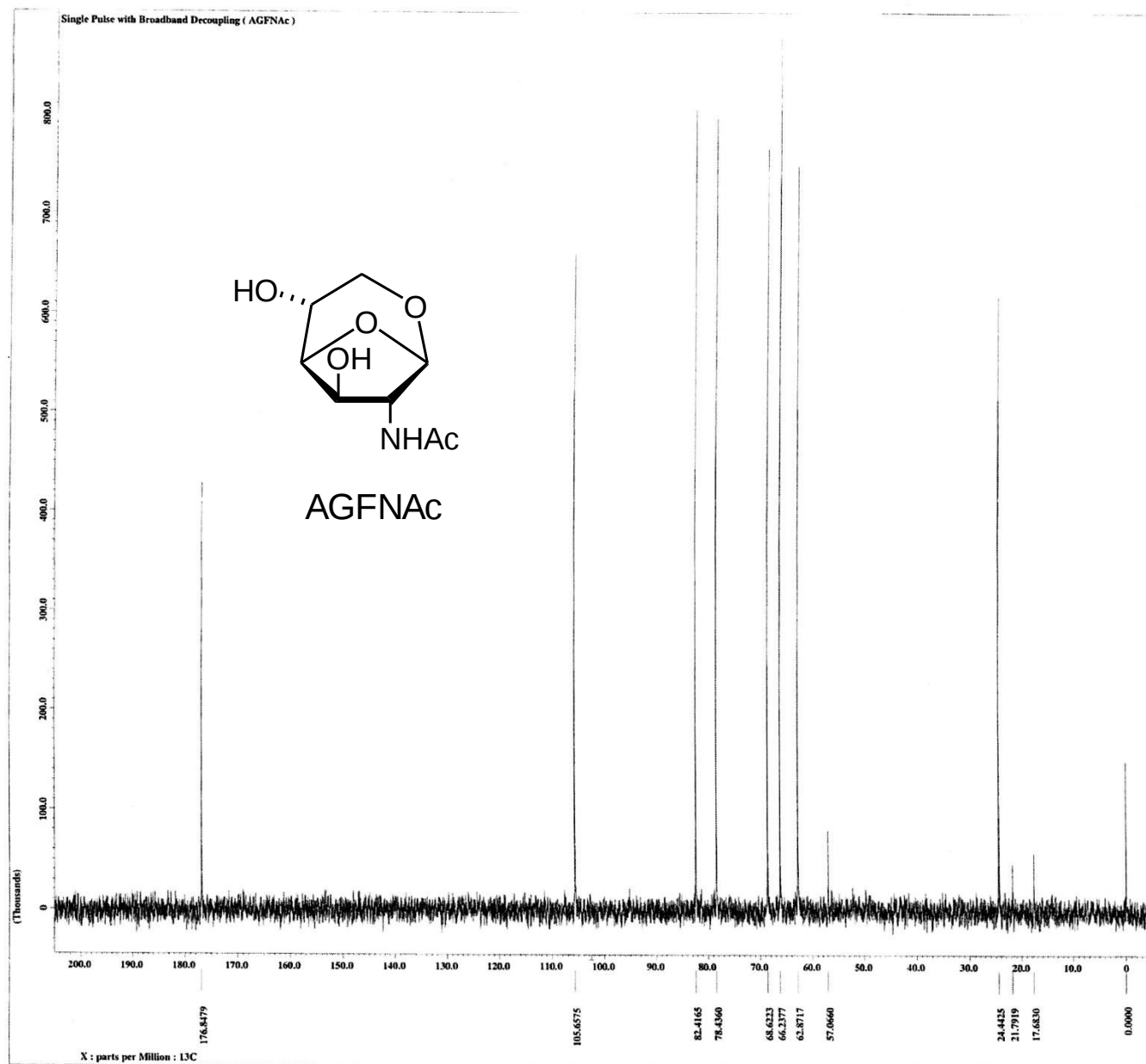
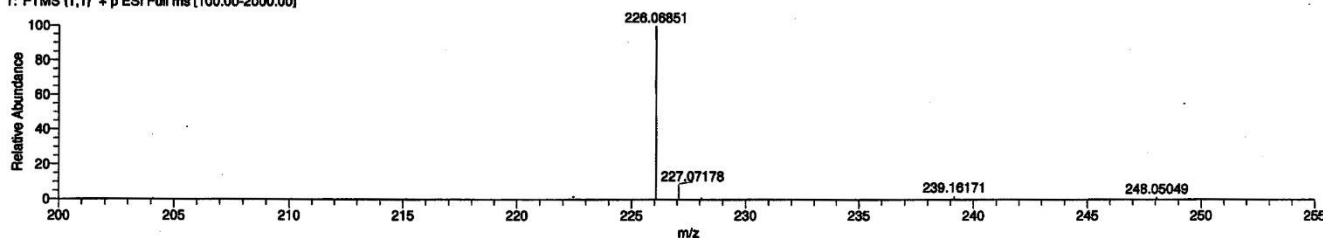


Figure S2.2: ^{13}C NMR spectrum of AGFNac (100 MHz, D_2O).



Elemental composition search on mass 226.06851

m/z= 221.06851-231.06851

Isotope Min Max

O-16 0 6

C-12 0 30

H-1 0 60

Na-23 0 1

N-14 0 1

Charge 1

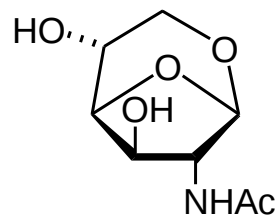
Mass tolerance 5.00 ppm

Nitrogen rule not used

RDB equiv -1.00-100.00

max results 100

m/z	Theo Mass	Delta (ppm)	RDB equiv.	Composition
226.06851	226.06859	-0.37	2.5	C ₈ H ₁₃ O ₅ N Na



AGFNAC

Figure S2.3: ESI-HRMS of AGFNAC along with analytical data.

3. Supporting data for AGPNAcDA

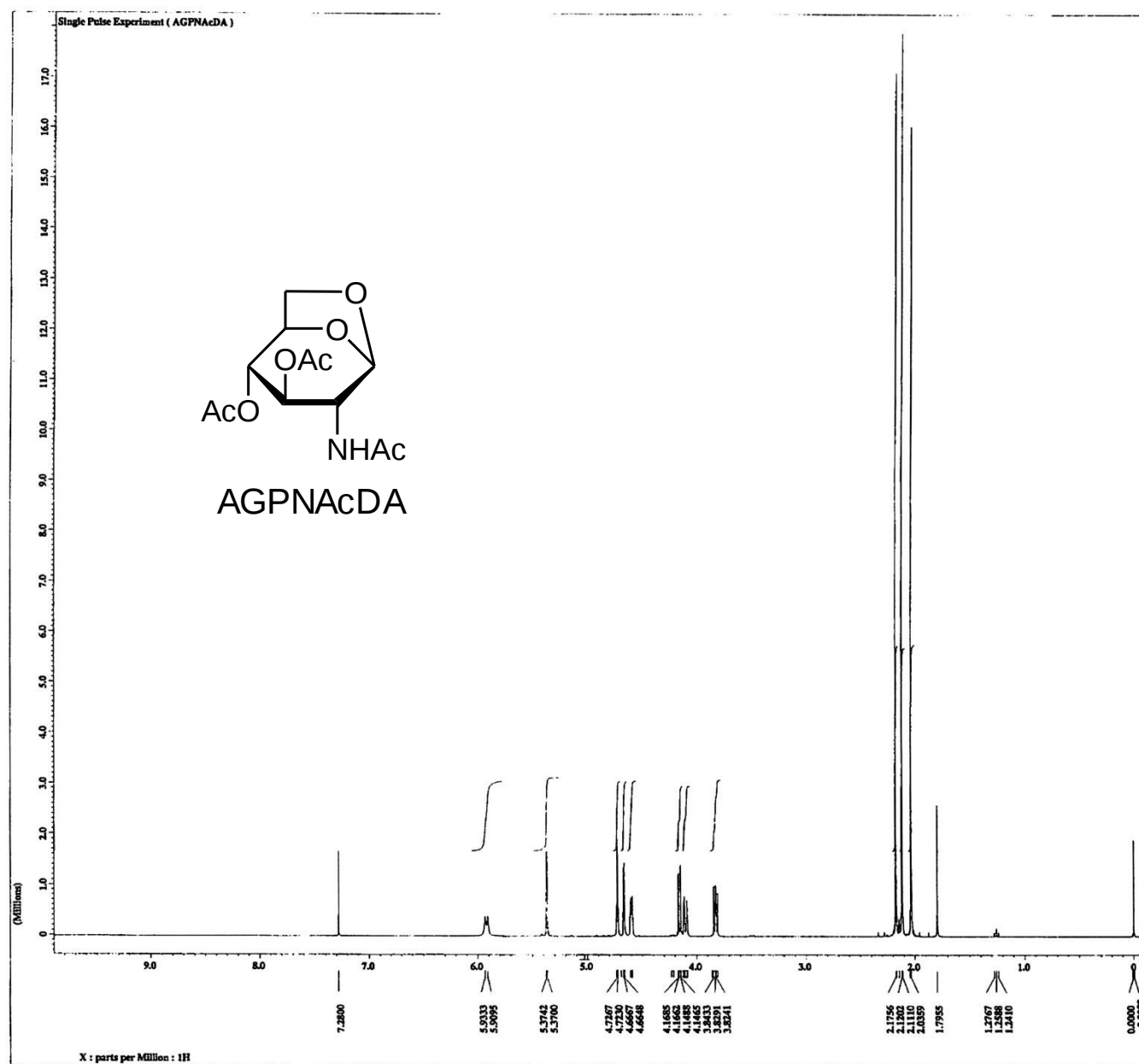


Figure S3.1: ^1H NMR spectrum of AGPNAcDA (400 MHz, CDCl_3).

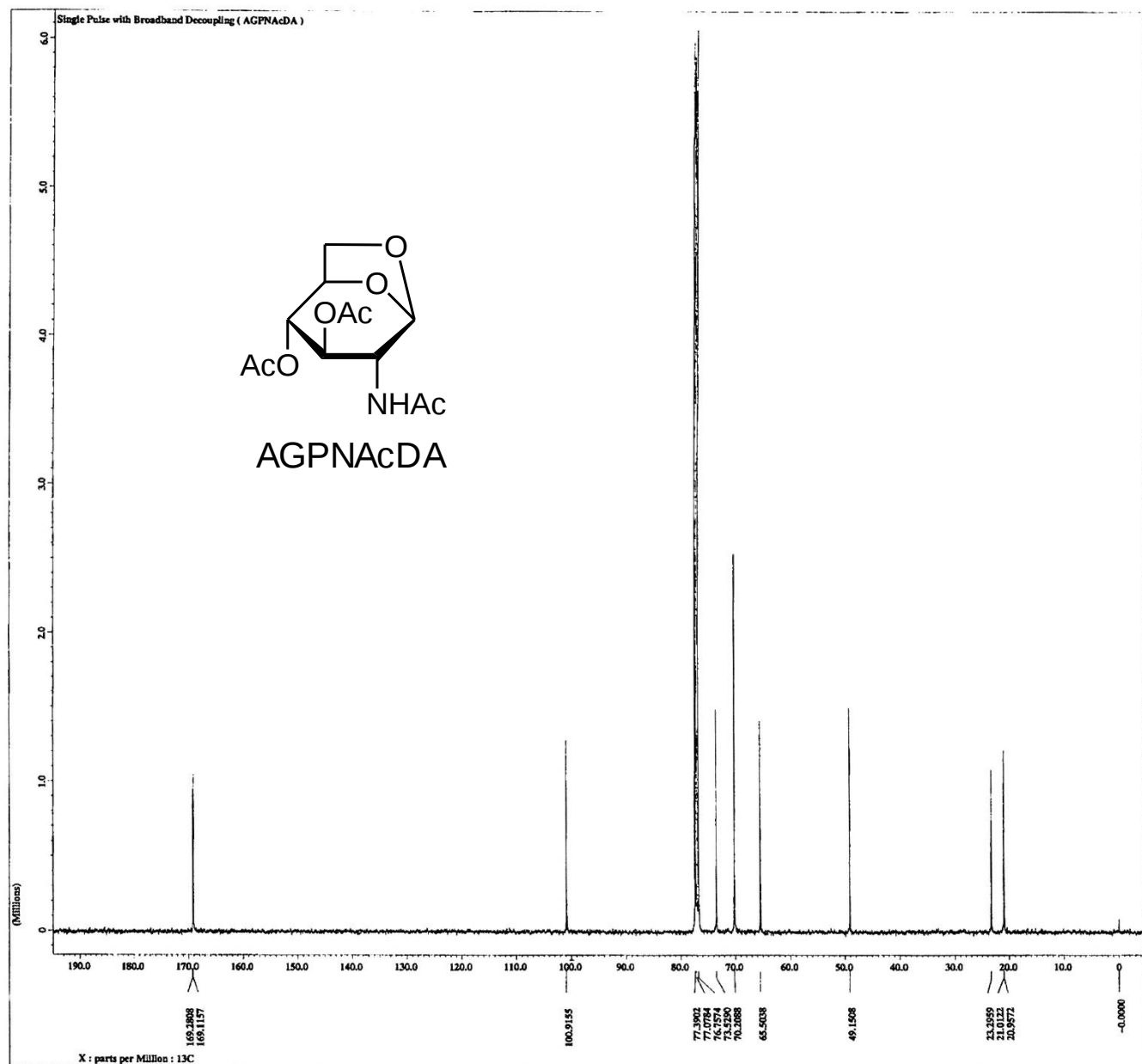
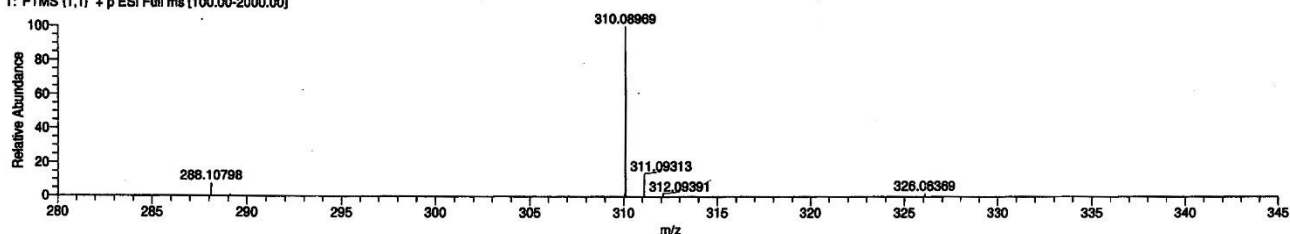


Figure S3.2: ^{13}C NMR spectrum of AGPNAcDA (100 MHz, CDCl_3).



Elemental composition search on mass 310.08969

m/z= 305.08969-315.08969

Isotope Min Max

O-16	0	10
C-12	0	30
H-1	0	60
Na-23	0	1
N-14	0	1
B-10	0	1
S-32	0	1

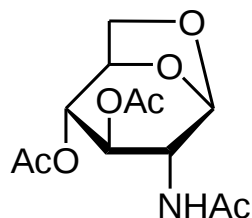
Charge 1

Mass tolerance 5.00 ppm

Nitrogen rule not used

RDB equiv -1.00-100.00

max results 100



AGPNAcDA

m/z	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
310.08969	310.08972	-0.11	4.5	C ₁₂ H ₁₇ O ₇ N Na
	310.08963	0.21	11.5	C ₁₈ H ₁₆ O ₂ NS
	310.08945	0.78	3.0	C ₁₁ H ₁₈ O ₁₀
	310.08863	3.43	12.0	C ₁₈ H ₁₃ O ₃ ¹⁰ B Na
	310.09103	-4.33	15.0	C ₂₀ H ₁₂ O ₃ ¹⁰ B

Figure S3.3: ESI-HRMS of AGPNAcDA along with analytical data.

4. Supporting data for AGFNAcDA

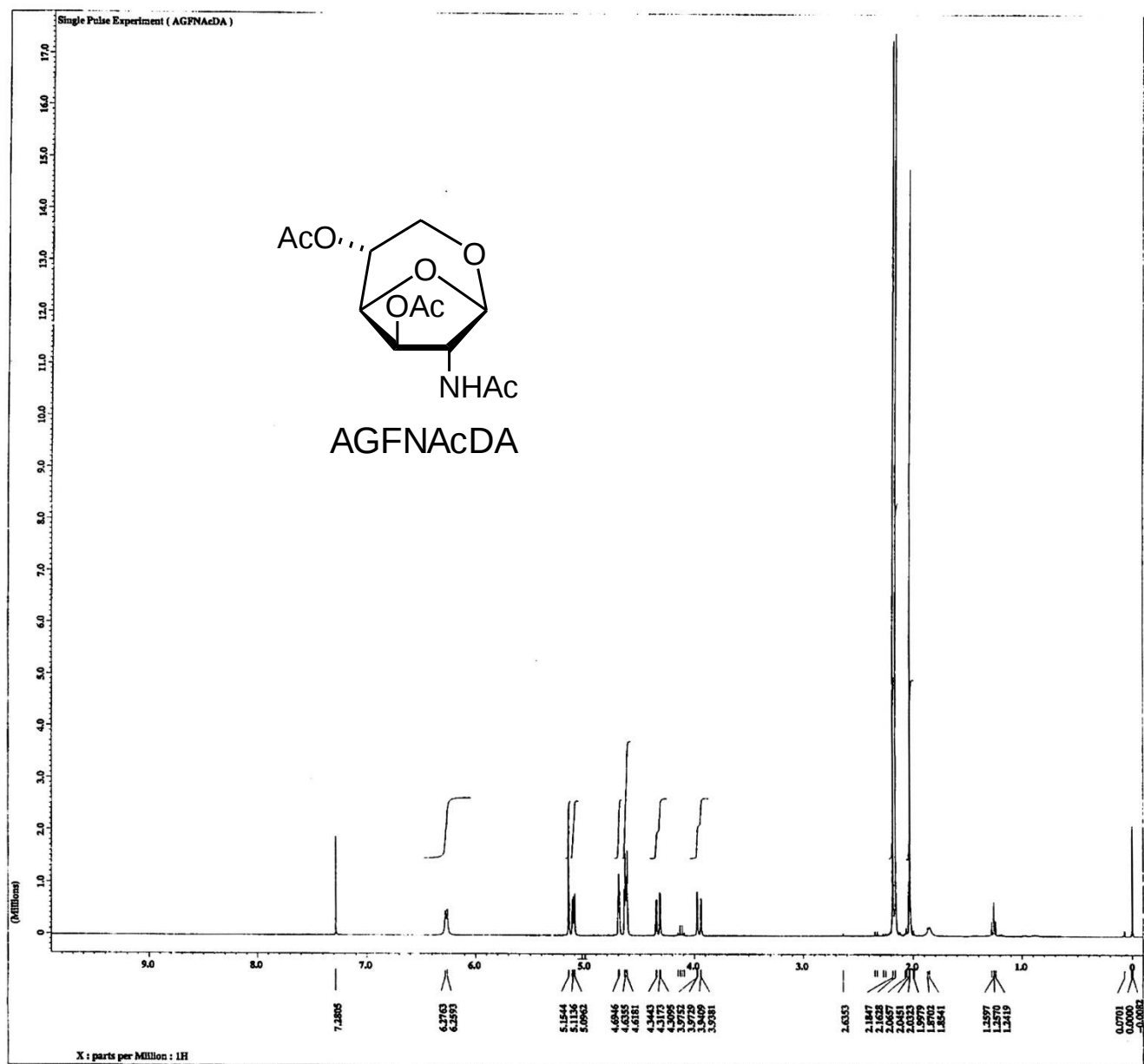


Figure S4.1: ^1H NMR spectrum of AGFNAcDA (400 MHz, CDCl_3).

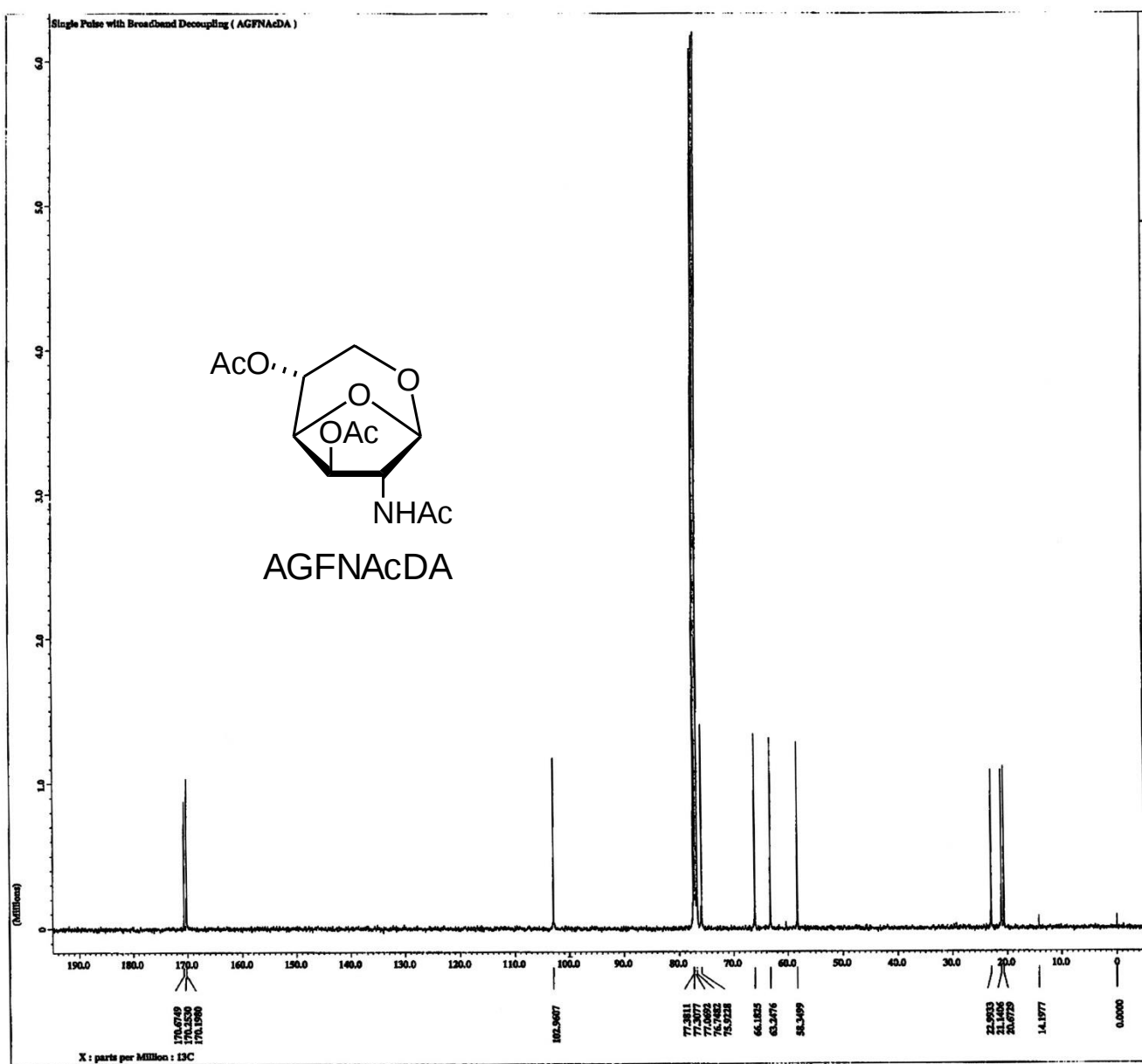
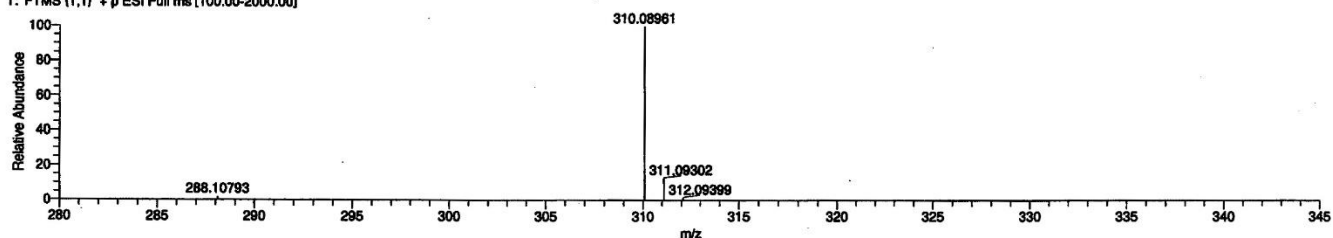


Figure S4.2: ^{13}C NMR spectrum of AGFNAcDA (100 MHz, CDCl_3).



Elemental composition search on mass 310.08965

m/z= 305.08965-315.08965

Isotope Min Max

O-16 0 10

C-12 0 30

H-1 0 60

Na-23 0 1

N-14 0 1

Charge 1

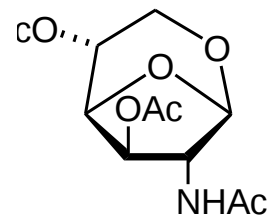
Mass tolerance 5.00 ppm

Nitrogen rule not used

RDB equiv -1.00-100.00

max results 100

m/z	Theo. Mass	Delta (ppm)	RDB equiv.	Composition
310.08965	310.08972	-0.24	4.5	C ₁₂ H ₁₇ O ₇ NNa
	310.08945	0.65	3.0	C ₁₁ H ₁₈ O ₁₀



AGFNACDA

Figure S4.3: ESI-HRMS of AGFNACDA along with analytical data.