

Supplementary Information

Non-Catalytic Dehydration of *N,N'*-diacetylchitobiose in High-temperature Water

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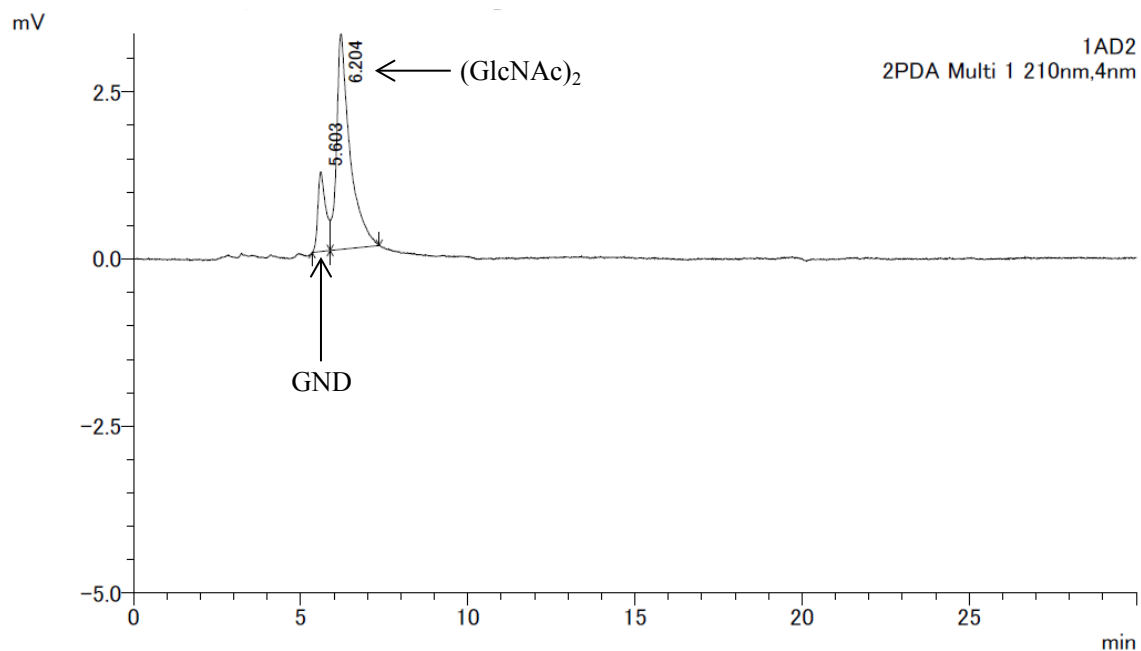
List of Supplementary Information:

S1. HPLC chromatograph examples

S2. Characterization of GND and GNL

S1. HPLC chromatograph examples

(a) 190°C, 25 MPa, reaction time 10 sec



(b) 200°C, 25 MPa, reaction time 18 sec

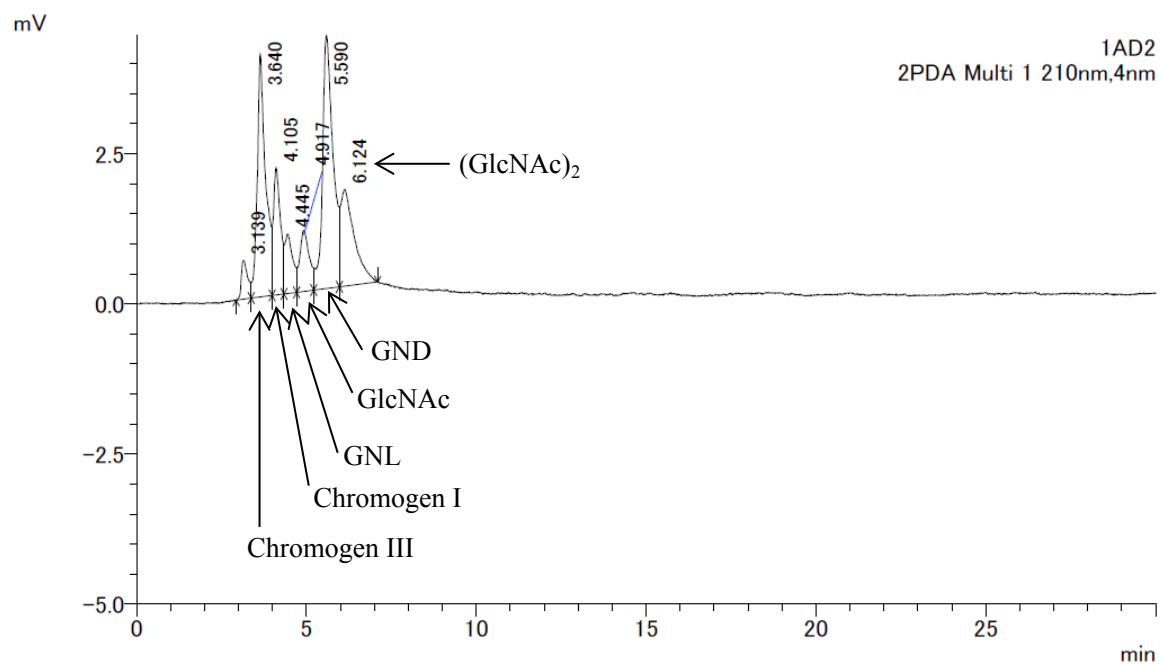


Figure S1. HPLC chromatograph examples.

S2. Characterization of GND and GNL

(a) GND ; HRESIMS m/z 429.14822 $[M + Na]^+$ (calcd. for $C_{16}H_{26}N_2NaO_{10}$, 429.14851).

1H -NMR (D_2O , 500 MHz) δ : 6.48 (d, 0.25H, $J_{3,4}$ 2.0 Hz, H-3 β), 6.43 (d, 0.75H, $J_{3,4}$ 2.0 Hz, H-3 α), 5.47 (s, 0.25H, H-1 β), 5.43 (s, 0.75H, H-1 α), 4.67 (d, 0.75H, $J_{1',2'}$ 8.5 Hz, H-1' α), 4.66 (d, 0.25H, $J_{1',2'}$ 8.5 Hz, H-1' β), 4.37 (dd, 1H, $J_{3,4}$ 2.0, $J_{4,5}$ 9.5 Hz, H-4), 3.94 (1H, H-6'b), 3.89 (m, 1H, $J_{4,5}$ 9.5, $J_{5,6a}$ 6.0, $J_{5,6b}$ 2.0 Hz, H-5), 3.83 (dd, 1H, $J_{5,6b}$ 2.0, $J_{6a,6b}$ 12.0 Hz, H-6b), 3.79 (1H, H-6a), 3.73 (dd, 1H, $J_{1',2'}$ 8.5, $J_{2',3'}$ 10 Hz, H-2'), 3.68 (dd, 1H, $J_{5,6a}$ 6.0, $J_{6a,6b}$ 12 Hz, H-6a), 3.58 (t, 1H, $J_{2',3'}$ 10, $J_{3',4'}$ 10 Hz, H-3'), 3.51-3.46 (2H, H-4', H-5'), 2.11 and 2.08 (s, 1.5H, $\underline{CH}_3CONH$ - β , $\underline{CH}_3CONH$ -' β), 2.10 and 2.09 (s, 4.5H, $\underline{CH}_3CONH$ - α , $\underline{CH}_3CONH$ -' α). ^{13}C -NMR (D_2O , 125 MHz) α -anomer δ : 177.3 ($\underline{CH}_3CONH$ -'), 176.3 ($\underline{CH}_3CONH$ -), 136.2 (C-2), 118.4 (C-3), 104.7 (C-1'), 90.3 (C-1), 78.60 (C-5'), 76.4 (C-3'), 75.5 (C-4), 72.7 (C-5), 72.5 (C-4'), 63.5 (C-6'), 63.3 (C-6), 24.9 ($\underline{CH}_3CONH$ -'). ^{13}C -NMR (D_2O , 125 MHz) β -anomer δ : 177.3 ($\underline{CH}_3CONH$ -'), 176.3 ($\underline{CH}_3CONH$ -), 137.3 (C-2), 117.7 (C-3), 104.3 (C-1'), 92.4 (C-1), 78.9 (C-5), 78.6 (C-5'), 76.4 (C-3'), 75.2 (C-4), 72.5 (C-4'), 63.7 (C-6), 63.5 (C-6'), 58.5 (C-2'), 25.8 ($\underline{CH}_3CONH$ -), 24.9 ($\underline{CH}_3CONH$ -').

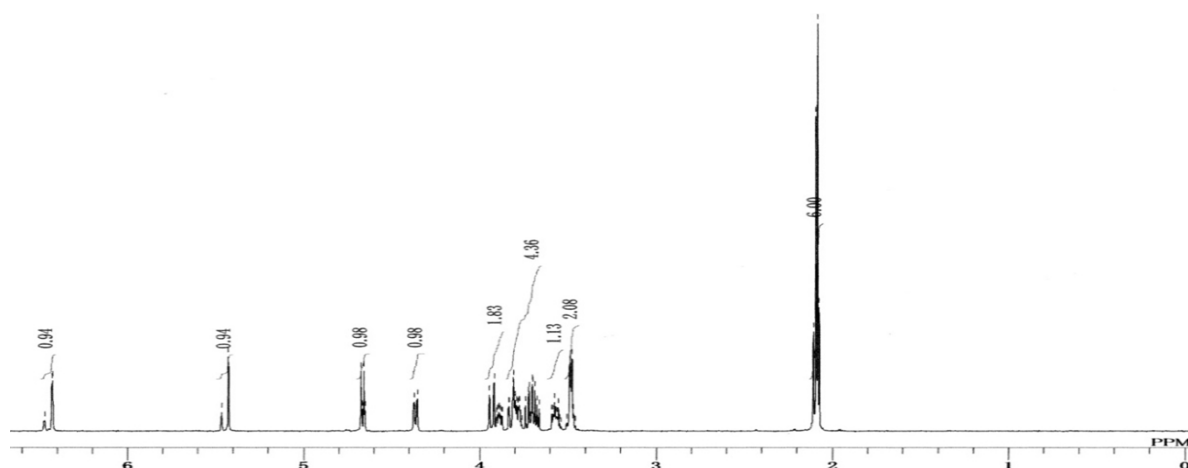


Figure S2. ^1H NMR Spectra of GND in D_2O (500 MHz). Chemical shifts are shown in ppm downfield from external TPS.

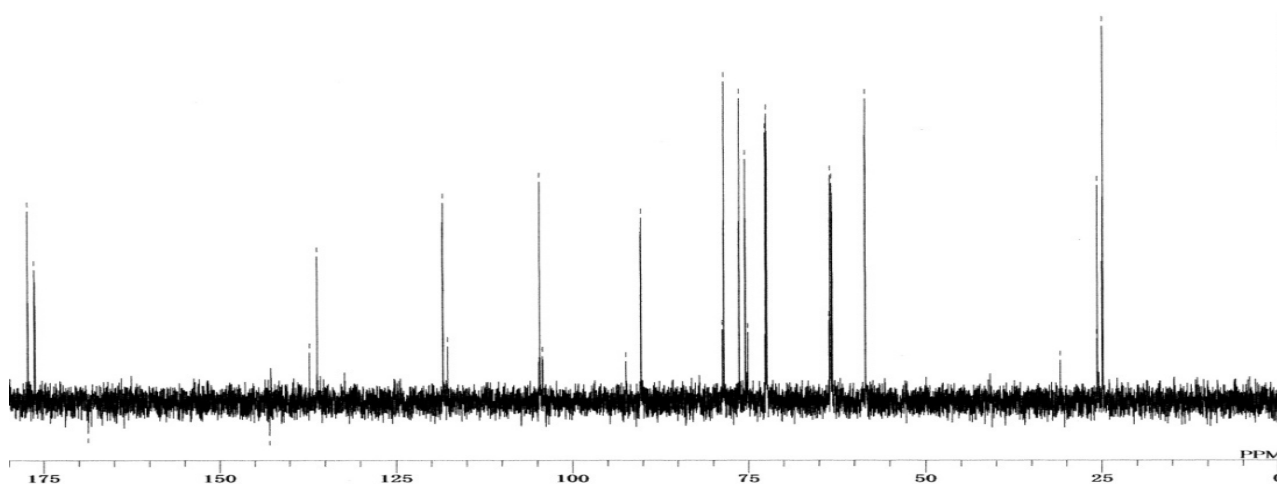


Figure S3. ^{13}C NMR Spectra of GND in D_2O (500 MHz). Chemical shifts are shown in ppm downfield from external TPS.

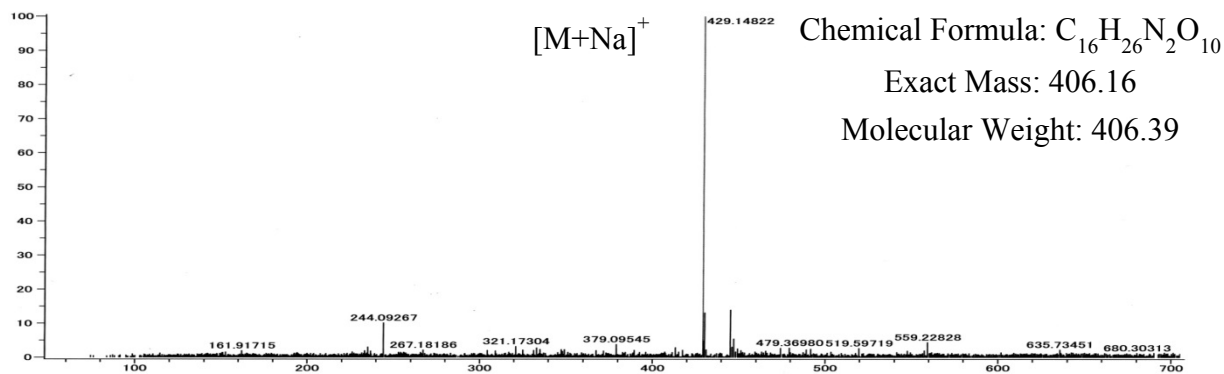


Figure S4. Positive ion mode HRESI-MS Spectrum of GND.

(b) GNL ; HRESIMS m/z 427.13260 $[M + Na]^+$ (calcd. for $C_{16}H_{24}N_2NaO_{10}$, 427.13286).

1H -NMR (D_2O , 500 MHz) δ : 7.44 (d, 1H, $J_{3,4}$ 4.0 Hz, H-3), 4.81 (dd, 1H, $J_{3,4}$ 4.0, $J_{4,5}$ 7.0 Hz, H-4), 4.74 (d, 1H, $J_{1',2'}$ 8.5 Hz, H-1'), 4.61 (1H, H-5), 3.94 (1H, H-6'b), 3.88 (dd, 1H, $J_{5,6b}$ 3.0, $J_{6a,6b}$ 12.5 Hz, H-6b), 3.80 (1H, H-6a), 3.79 (dd, 1H, $J_{5',6'a}$ 5.0, $J_{6'a,6'b}$ 12.5 Hz, H-6'a), 3.74 (t, 1H, $J_{1',2'}$ 8.5, $J_{2',3'}$ 8.5 Hz, H-2'), 3.59 (t, 1H, $J_{2',3'}$ 8.5, $J_{3',4'}$ 8.5 Hz, H-3'), 3.52-3.49 (2H, H-4', H-5'), 2.17 (s, 3H, $\underline{CH}_3CONH-$), 2.08 (s, 3H, $\underline{CH}_3CONH-$). ^{13}C -NMR (D_2O , 125 MHz) δ : 177.4 ($\underline{CH}_3CONH-$), 176.4 ($\underline{CH}_3CONH-$), 164.7 (C-1), 129.8 (C-3), 128.0 (C-2), 104.2 (C-1'), 83.9 (C-5), 78.7 (C-5'), 76.3 (C-3'), 73.1 (C-4), 72.5 (C-4'), 63.4 (C-6'), 62.9 (C-6), 58.4 (C-2'), 25.9 ($\underline{CH}_3CONH-$), 24.9 ($\underline{CH}_3CONH-$).

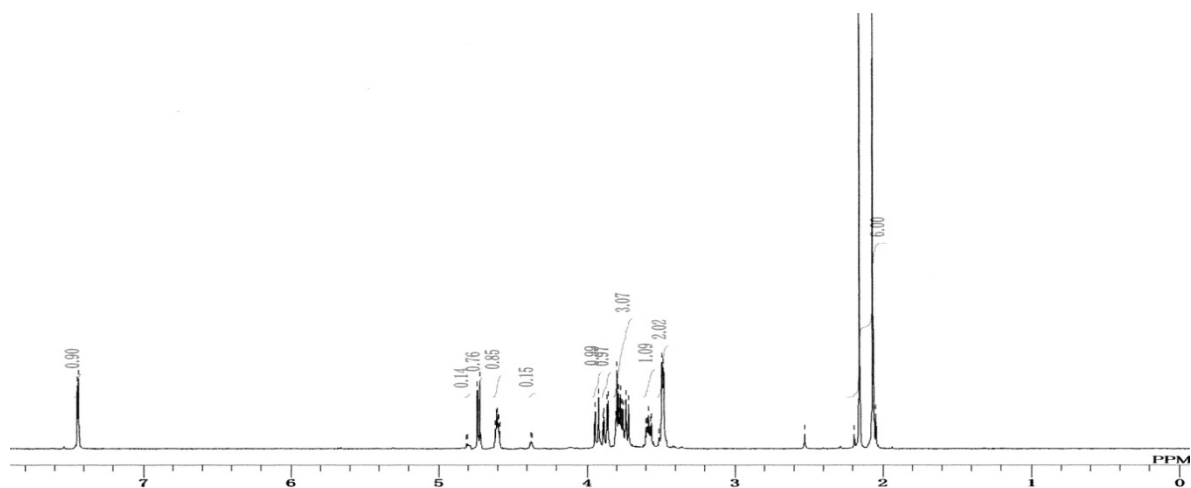


Figure S5. ^1H NMR Spectra of GNL in D_2O (500 MHz). Chemical shifts are shown in ppm downfield from external TPS.

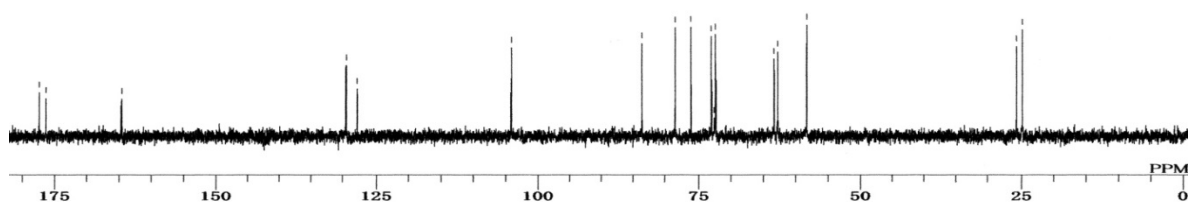


Figure S6. ^{13}C NMR Spectra of GNL in D_2O (500 MHz). Chemical shifts are shown in ppm downfield from external TPS.

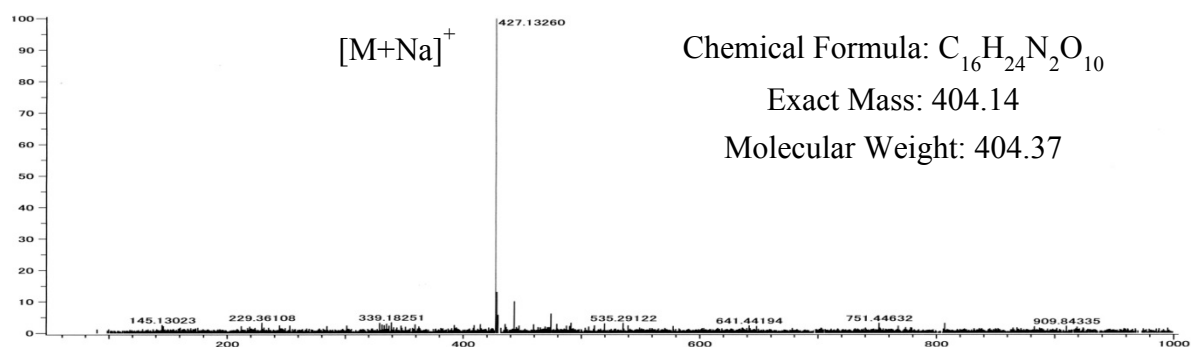


Figure S7. Positive ion mode HRESI-MS Spectrum of GNL .