

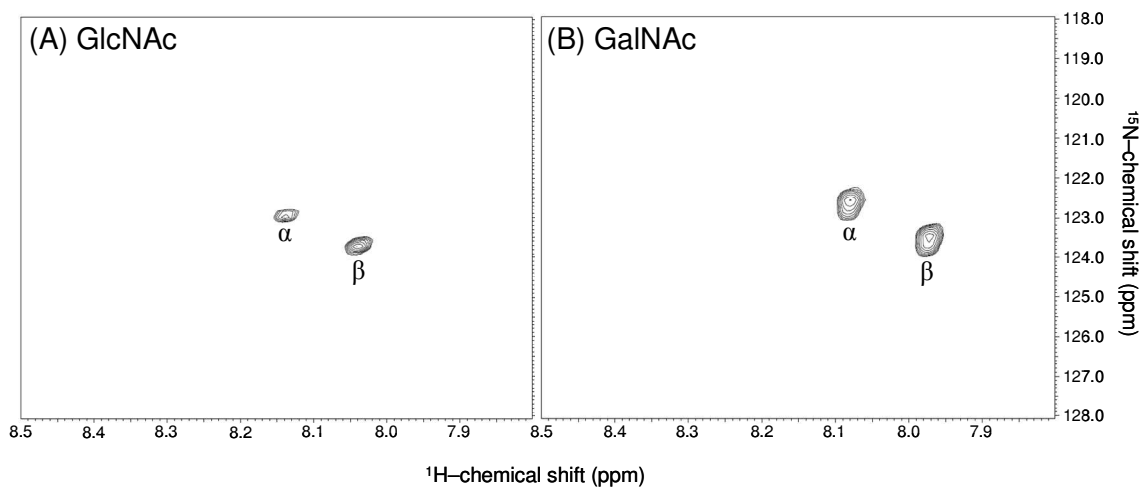
# SUPPORTING INFORMATION

## Characterization of Glycosaminoglycans by $^{15}\text{N}$ -NMR Spectroscopy and *in vivo* Isotopic Labeling

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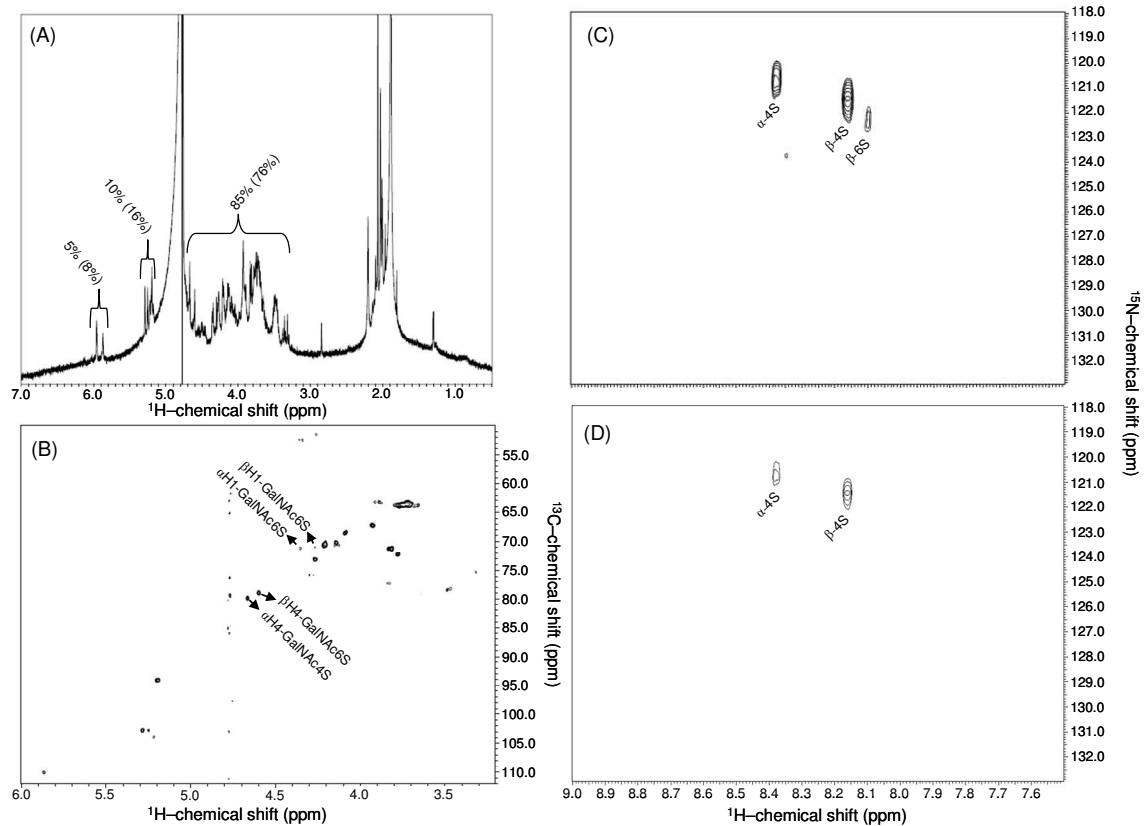
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### Figures:

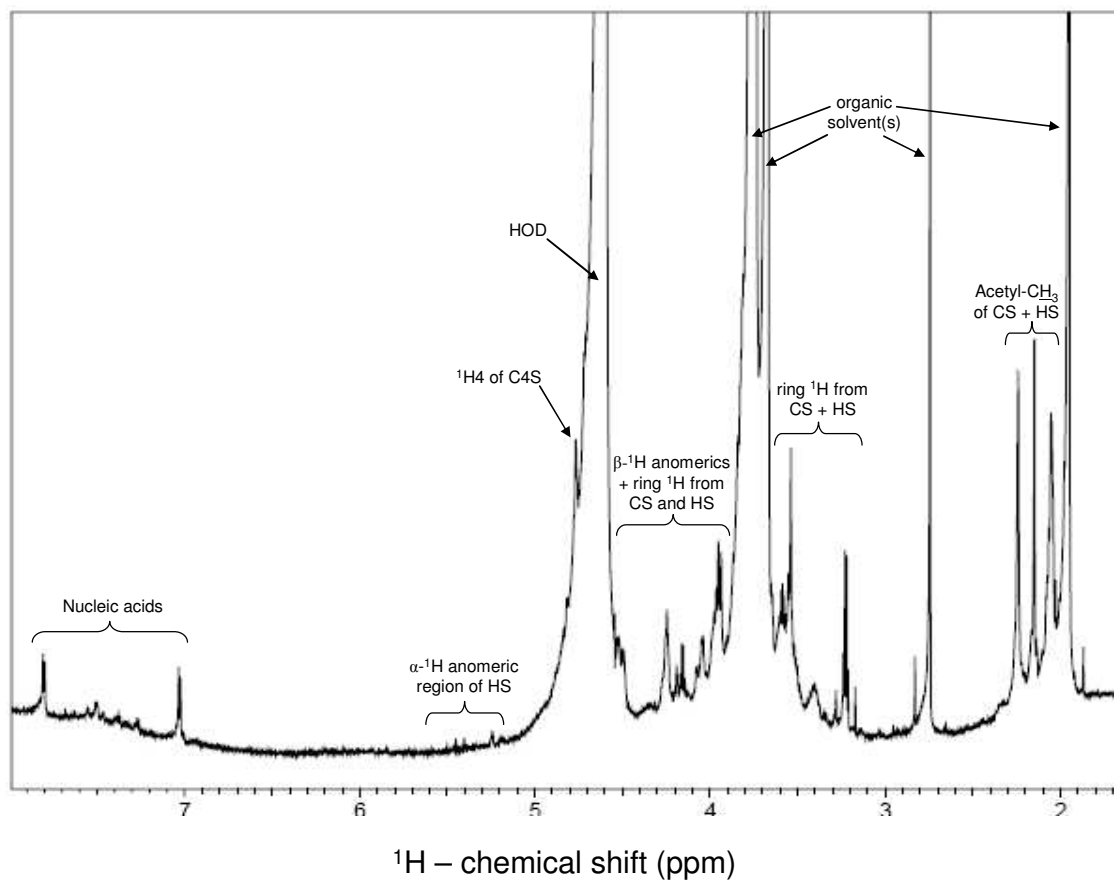


**Figure S1.**  $^{15}\text{N}$ -gHSQC spectra of the standard monomeric (A) GlcNAc, and (B) GalNAc.

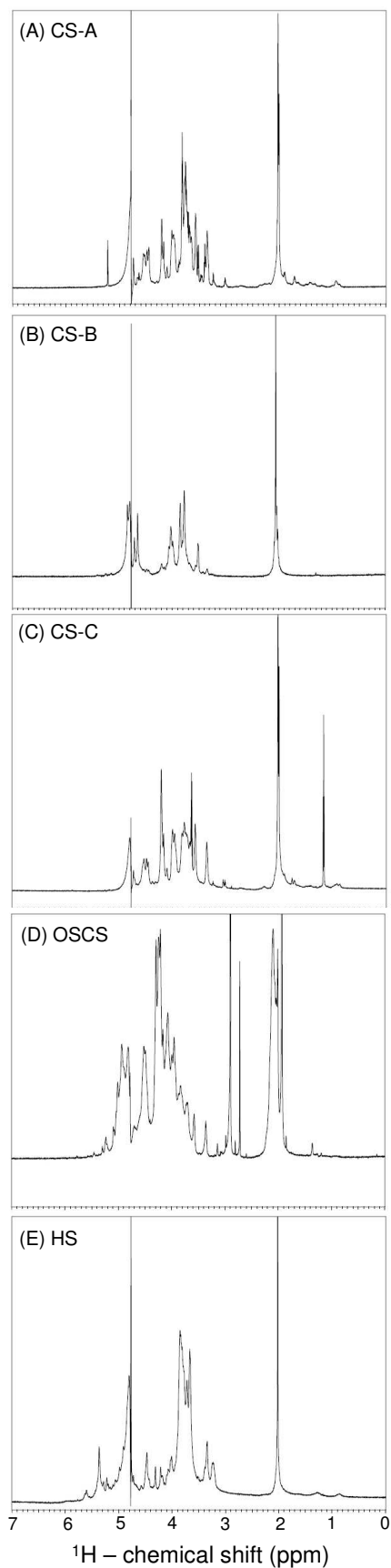




**Figure S3.** NMR analysis of the endothelial nuclease/ABC lyase-treated sample (unsaturated CS low-molecular weight products) (A-C), and the purified  $\Delta$ C4S dimer (D) obtained through SAX-HPLC chromatography. (A) 1D  $^1\text{H}$ -spectrum, the percentage without parentheses represents the real integral values of the peaks, whereas the percentages with parentheses belong to theoretical values assuming a pure sample of dimers. (B)  $^{13}\text{C}$ -gHSQC and (C, D)  $^{15}\text{N}$ -gHSQC spectra.



**Figure S4.** 1D  $^1\text{H}$ -NMR of the unprocessed endothelial GAGs (pool of negatively charged molecules). This spectrum was the only one recorded at 45 °C to induce an upfield shift of the HOD signal in order to prove the presence of the near  $^1\text{H}_4$  resonance from C4S. The endothelial HS:CS ratio was measured as 1:9.



**Figure S5.** 1D  $^1\text{H}$ -NMR analysis of the polymeric standard GAGs: (A) CS-A; (B) CS-B; (C) CS-C; (D) OSCS; (E) HS. These samples were also used to record the  $^{15}\text{N}$ -gHSQC spectra at Figure 3 of the main paper.