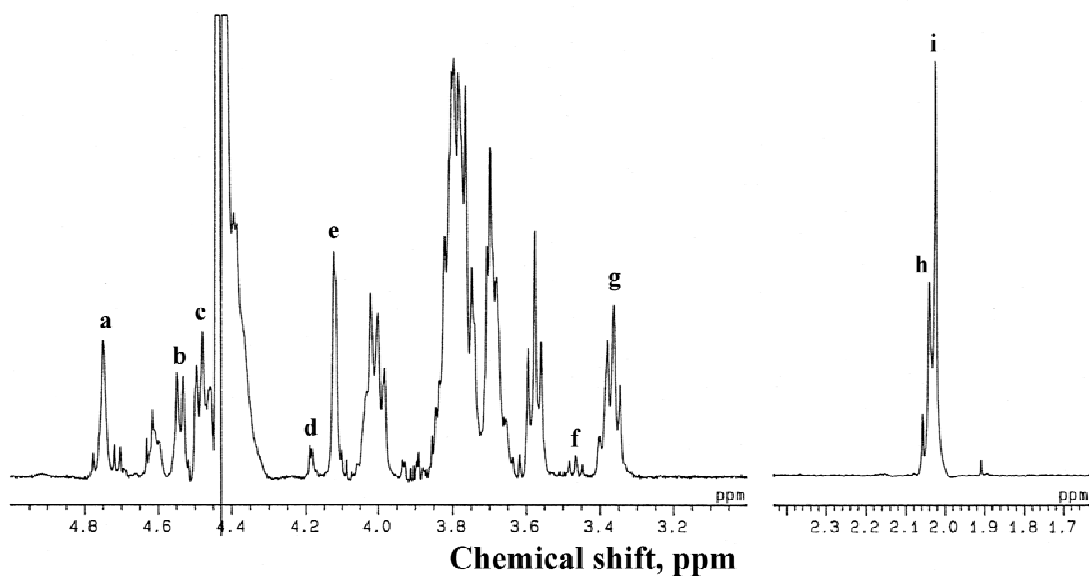


Figure S1. ^1H NMR spectrum of bikunin GAG



- a, GalNAc4S H-4;
- b, GalNAc0S H-1;
- c, GlcA H-1;
- d, Gal H-4 of the linkage region;
- e, GalNAc0S H-4;
- f, GlcA H-1 of the linkage region;
- g, GlcA H-2;
- h, GalNAc4S *N*-acetyl methyl protons;
- i, GalNAc0S *N*-acetyl methyl protons.

The data were collected by 500 MHz JEOL GSX500 α at 333 K.

Figure S2. PAGE analysis of bikunin GAG mixture (15% total acrylamide) showing a wide distribution of chain sizes observed at the higher sample concentration

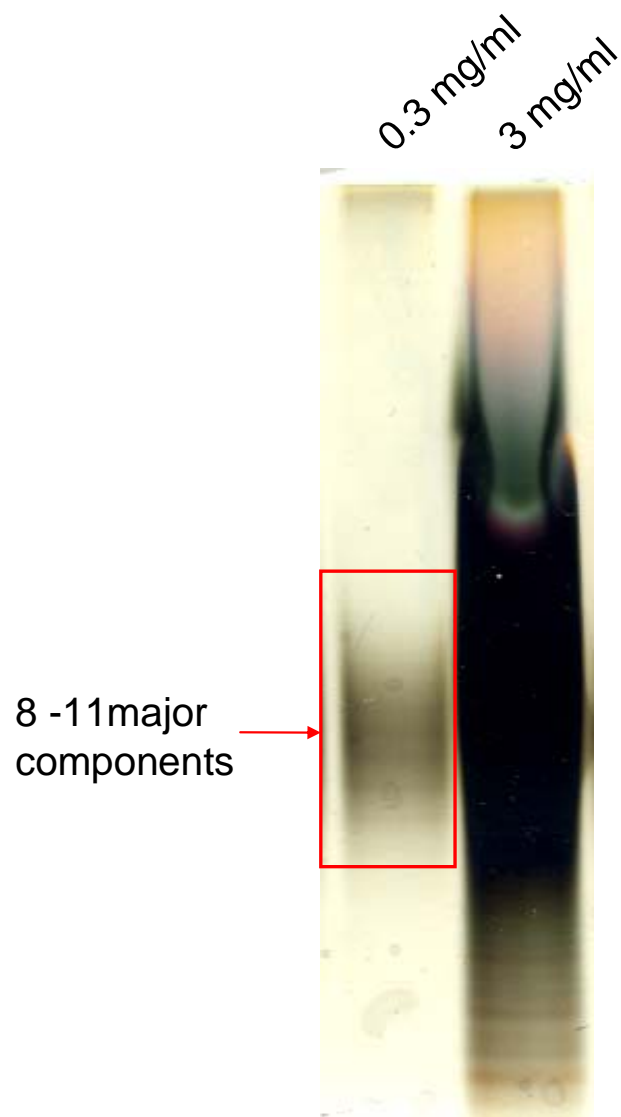


Figure S3. Deconvoluted FTICR mass spectrum of band 6 (lane 7, Figure 6A of the printed version). The number of disaccharide repeating units between the linkage region tetrasaccharide and NRE trisaccharide is denoted by m , the number of sulfo groups on these disaccharide repeating units is denoted by n .

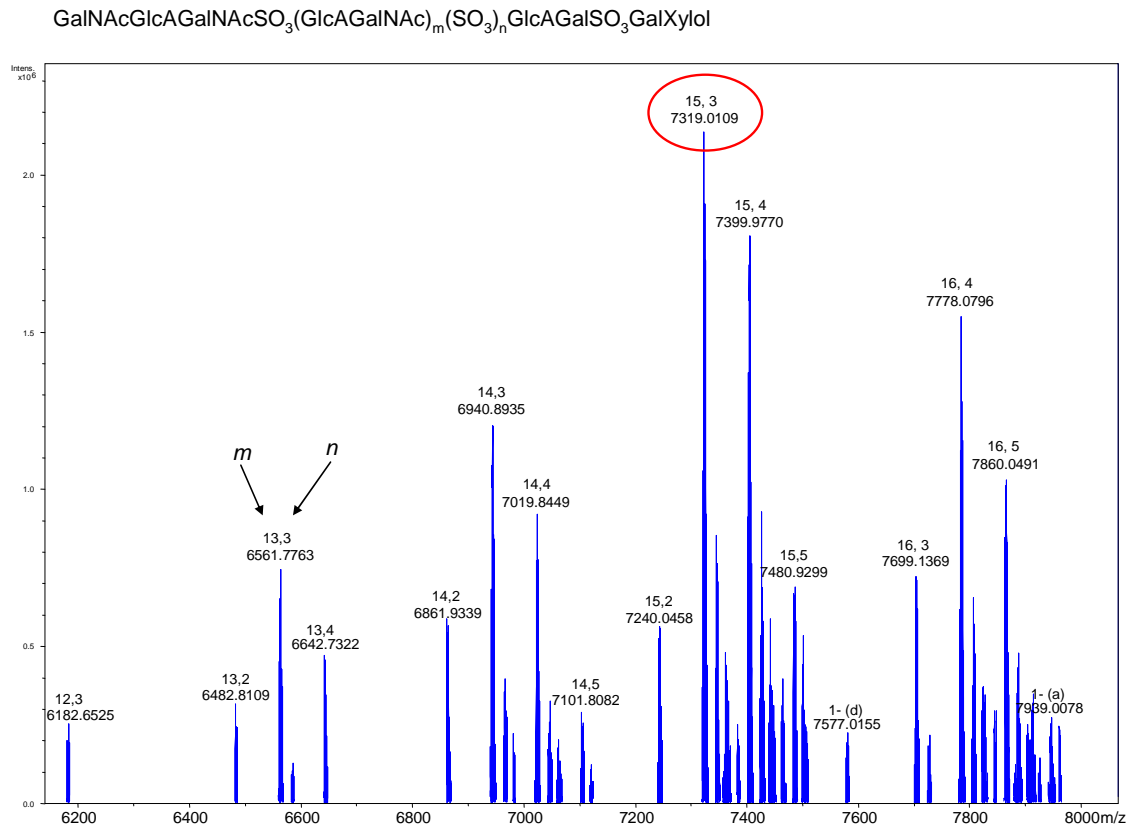


Figure S4. Experimental (top) and simulated (bottom) FTICR mass spectra of GalNAcGlcAGalNAcSO₃(GlcAGalNAc)₁₅(SO₃)₃GlcAGalSO₃GalXyloI (peak at *m/z* 7319 in Figure S3).

