

Figure S1. Negative-ion ESIMS/MS of the ion  $[\text{Fuc}(\text{SO}_3\text{Na}) - \text{Na}]^-$  at  $m/z$  245.00, labeled with  $^{18}\text{O}$

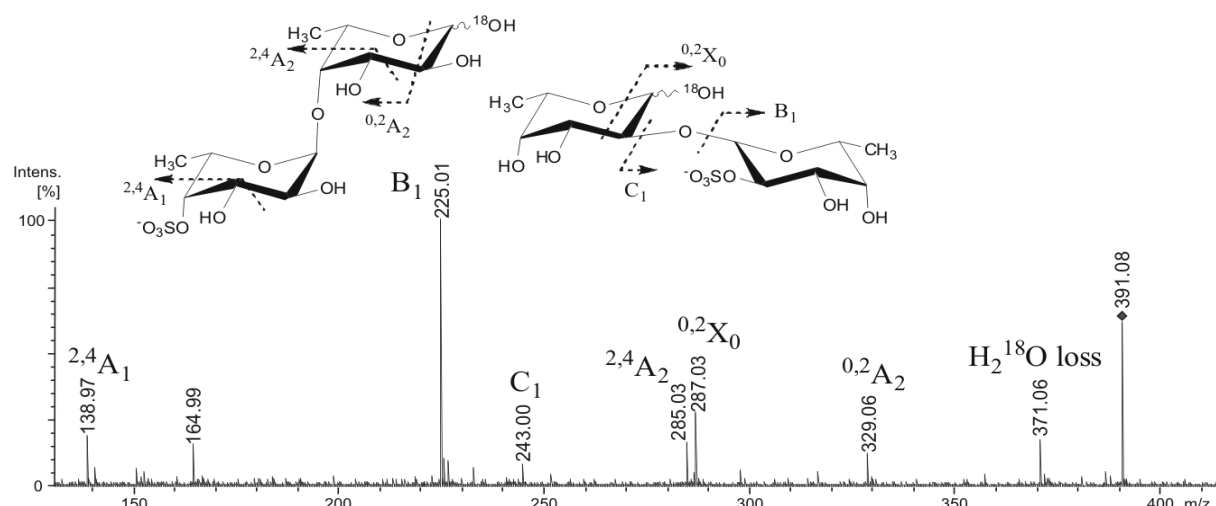


Figure S2. Negative-ion ESIMS/MS of the ion  $[\text{Fuc}_2(\text{SO}_3\text{Na}) - \text{Na}]^-$  at  $m/z$  391.08, labeled with  $^{18}\text{O}$

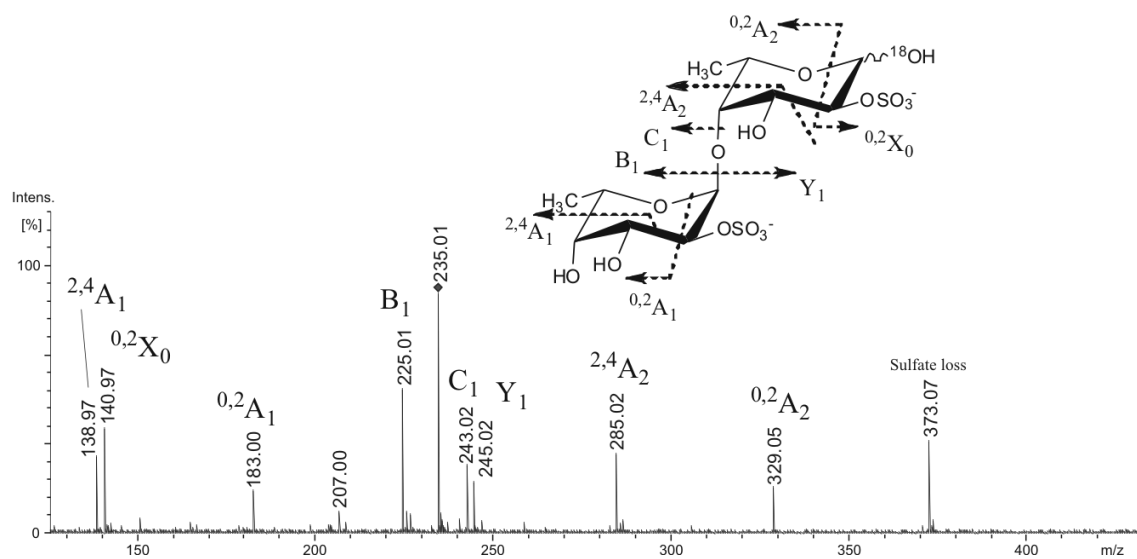


Figure S3. Negative-ion ESIMS/MS of the ion  $[\text{Fuc}_2(\text{SO}_3\text{Na})_2 - 2\text{Na}]^{2-}$  at  $m/z$  235.01, labeled with  $^{18}\text{O}$

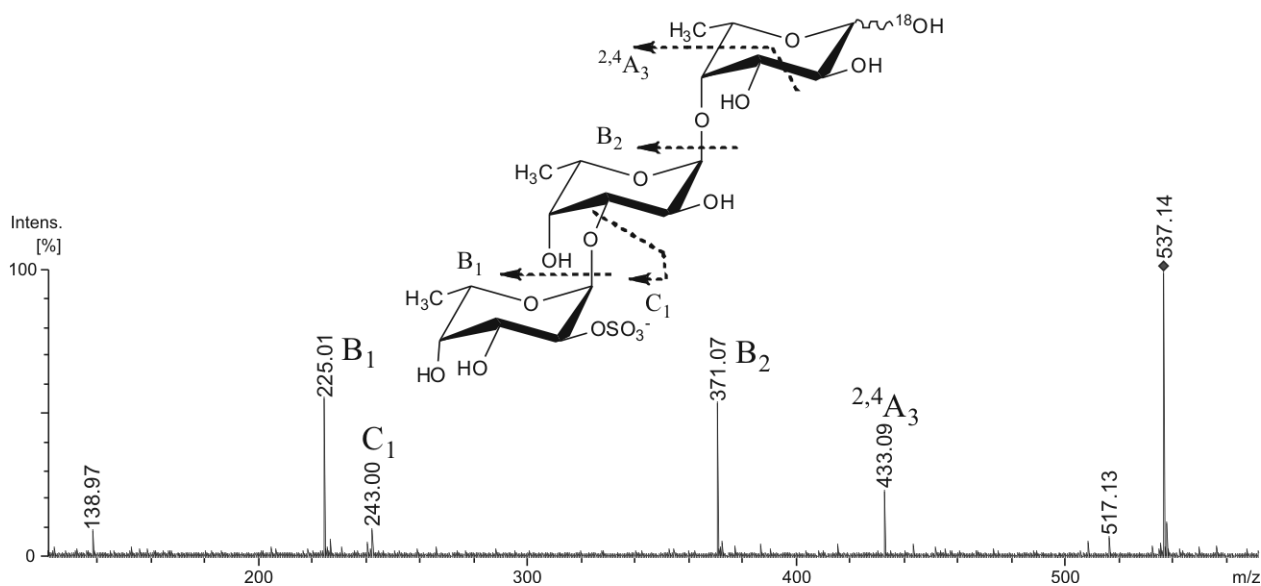


Figure S4. Negative-ion ESIMS/MS of the ion  $[\text{Fuc}_3\text{SO}_3\text{Na} - \text{Na}]^-$  at  $m/z$  537.14, labeled with  $^{18}\text{O}$

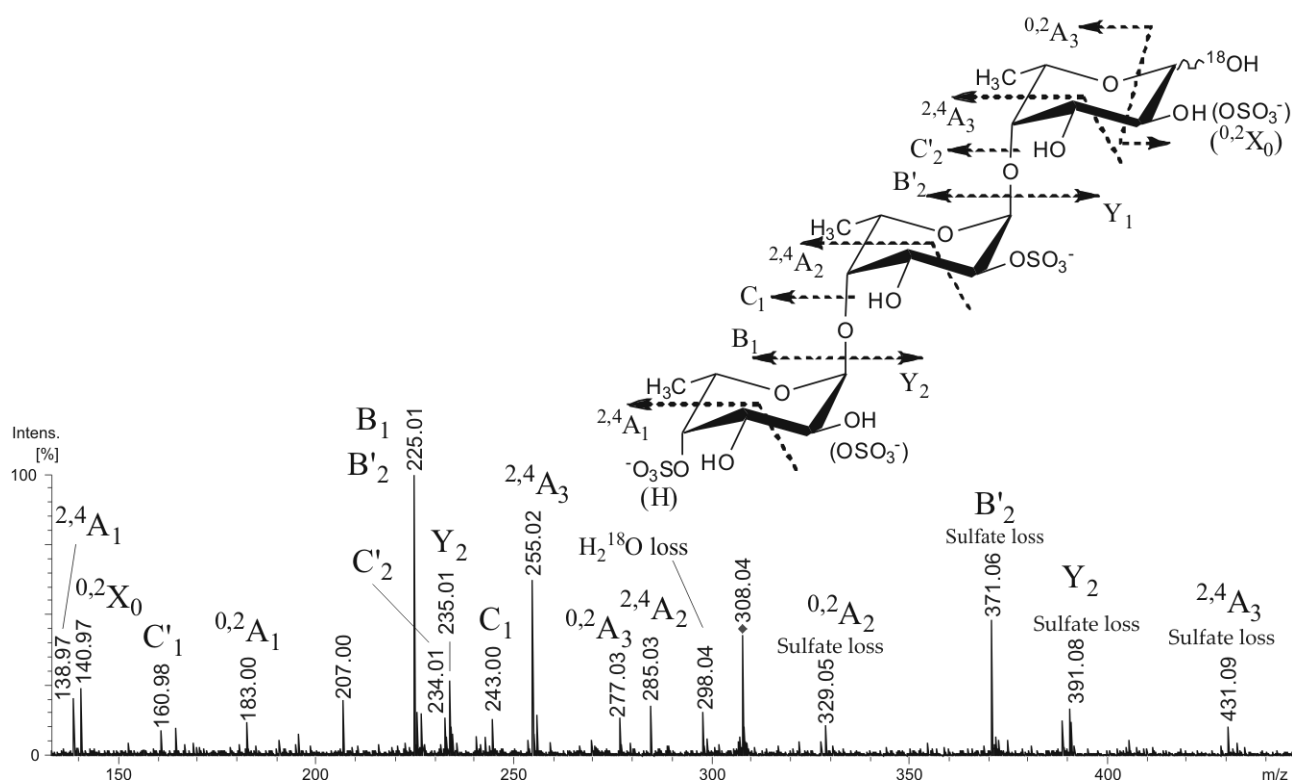


Figure S5. Negative-ion ESIMS/MS of the ion  $[\text{Fuc}_3(\text{SO}_3\text{Na})_2 - 2\text{Na}]^{2-}$  at  $m/z$  308.04, labeled with  $^{18}\text{O}$

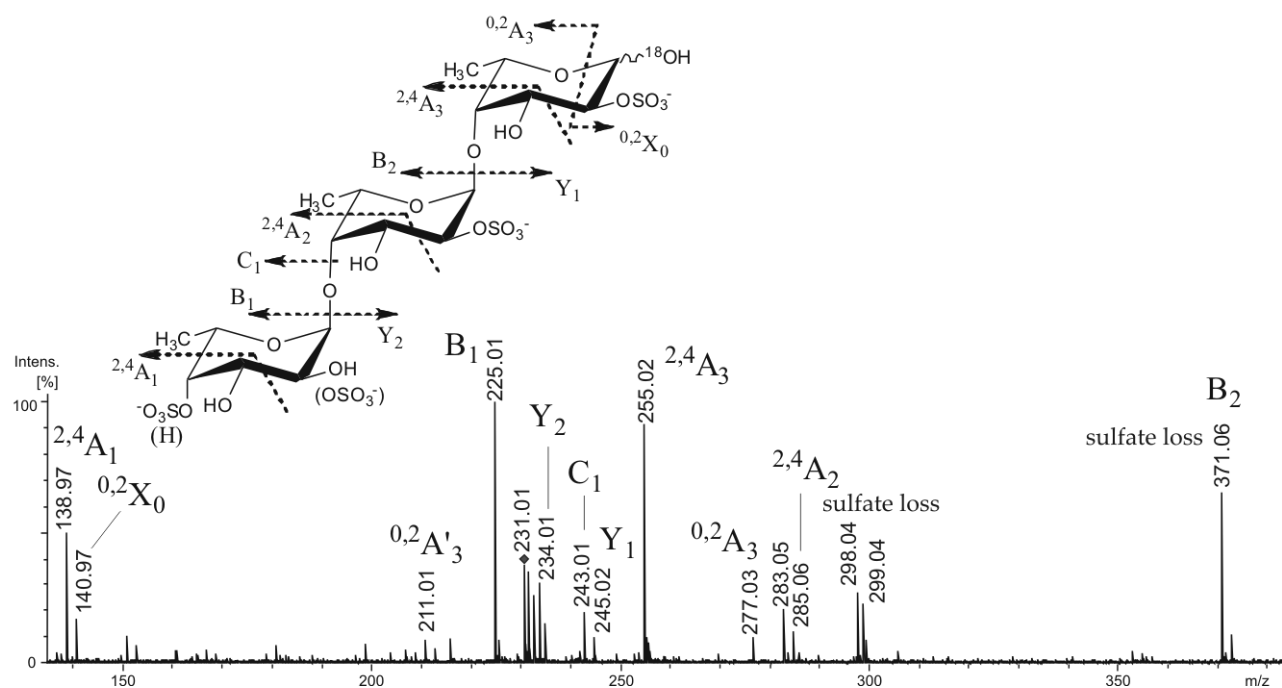


Figure S6. Negative-ion ESIMS/MS of the ion  $[\text{Fuc}_3(\text{SO}_3\text{Na})_3 - 3\text{Na}]^{3-}$  at  $m/z$  231.68 (with  $^{18}\text{O}$  label)

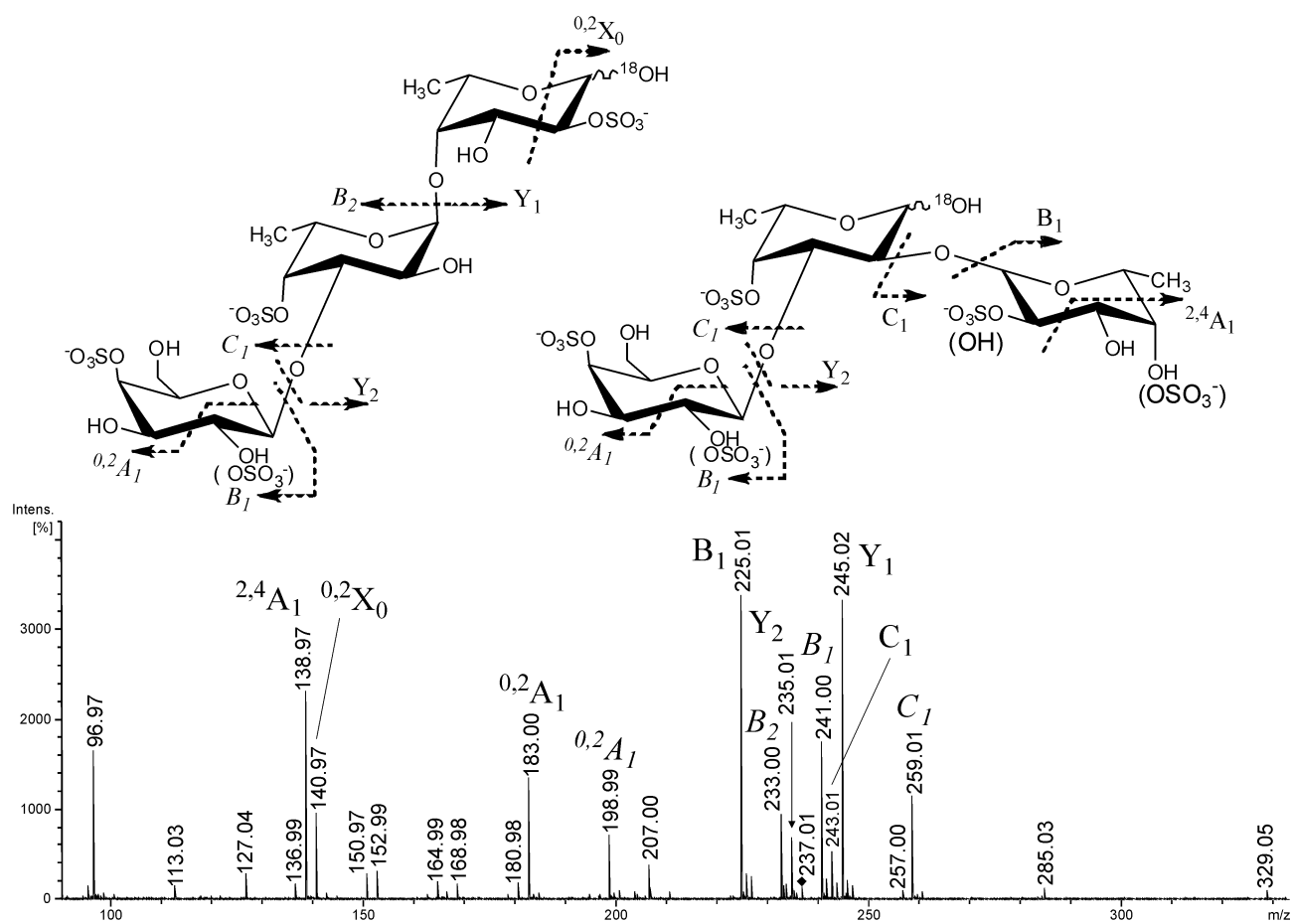
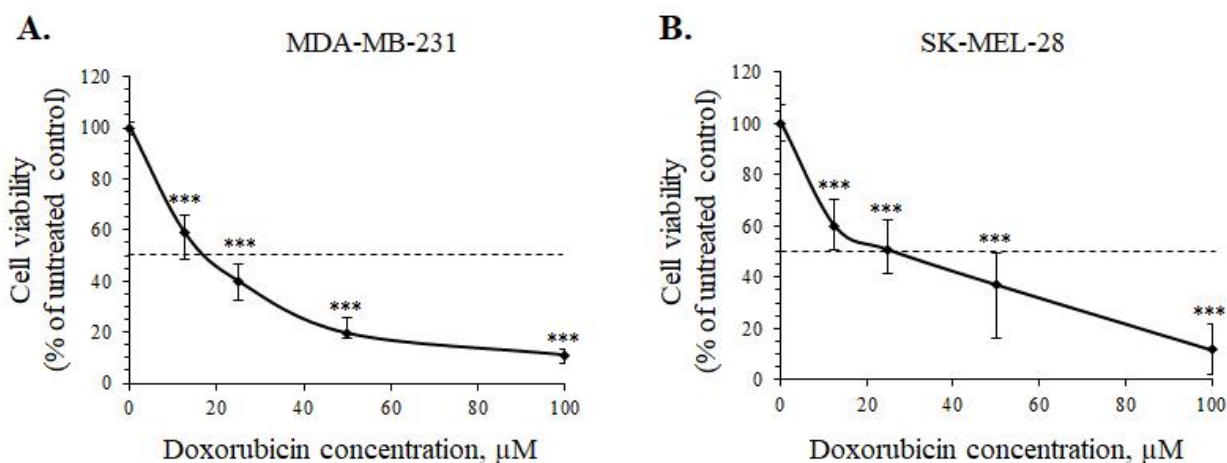


Figure S7. Negative-ion ESIMS/MS of the ion  $[\text{Fuc}_2\text{Gal}(\text{SO}_3\text{Na})_3 - 3\text{Na}]^{3-}$  at  $m/z$  237.01, labeled with  $^{18}\text{O}$



**Figure S8.** The cytotoxic effects of doxorubicin against breast cancer MDA-MB-231(A) and melanoma SK-MEL-28 (B) cells

MDA-MB-231 and SK-MEL-28 cells were treated by doxorubicin (12.5, 25, 50, 100  $\mu\text{M}$ ) and incubated for 24 h. The cell viability was estimated by MTS assay. The data are presented as mean  $\pm$  SD for triplicate experiments. A one-way ANOVA indicated the statistical significance compared to control (\*\* $p < 0.001$ ).