

**Electronic Supplementary Information for:**

**Rapid Cyclic Ion Mobility Separations of Monosaccharide Building Blocks as a First Step  
toward a High-Throughput Reaction Screening Platform for Carbohydrate Syntheses**

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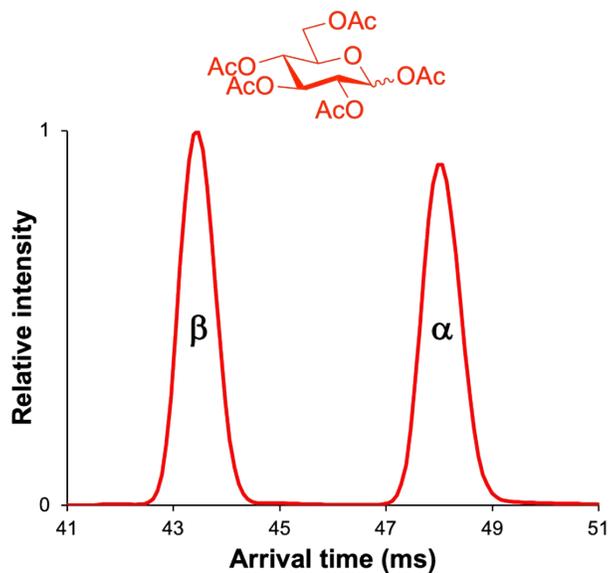
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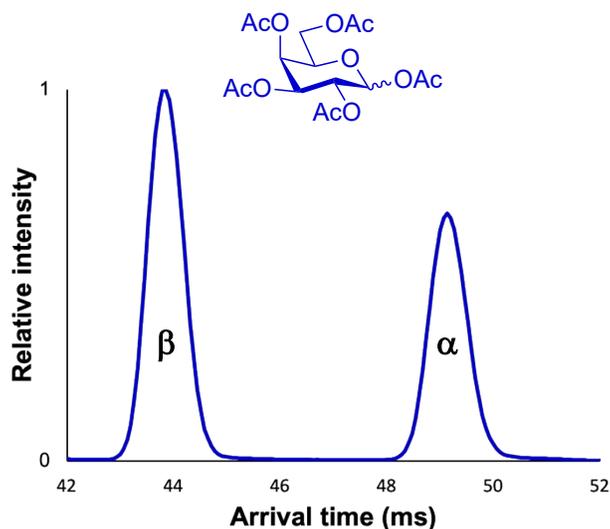
Additional Separation Results

S2–S7

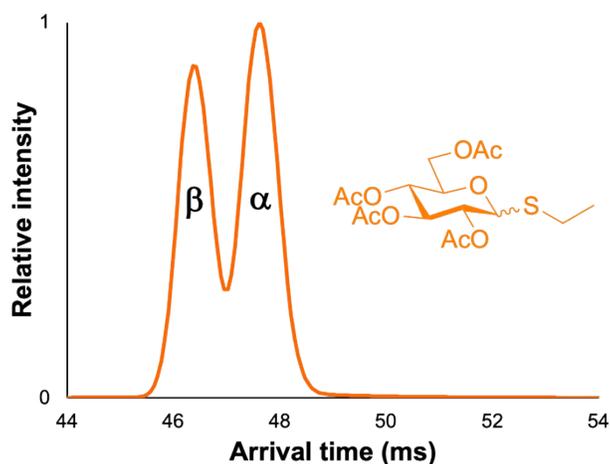
**Additional separation results:**



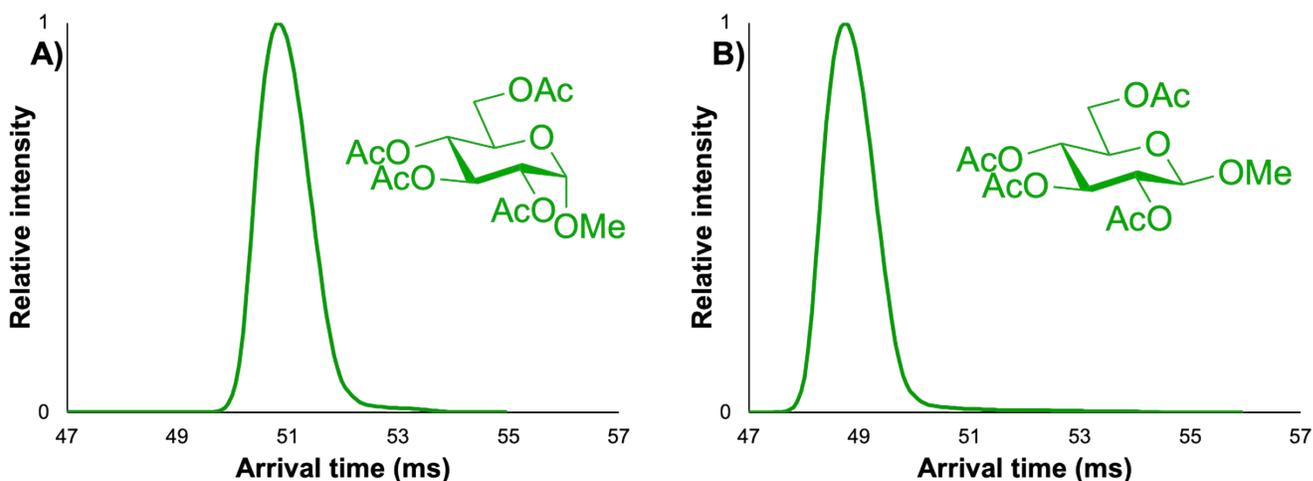
**Figure S1.** 5 m cIMS-MS separation of an equimolar mixture of  $\alpha/\beta$ -D-glucose pentaacetate as their  $[M + Na]^+$  adducts at traveling wave conditions of 450 m/s and 22 V. Each anomer was run individually (see manuscript) to assign the arrival time order.



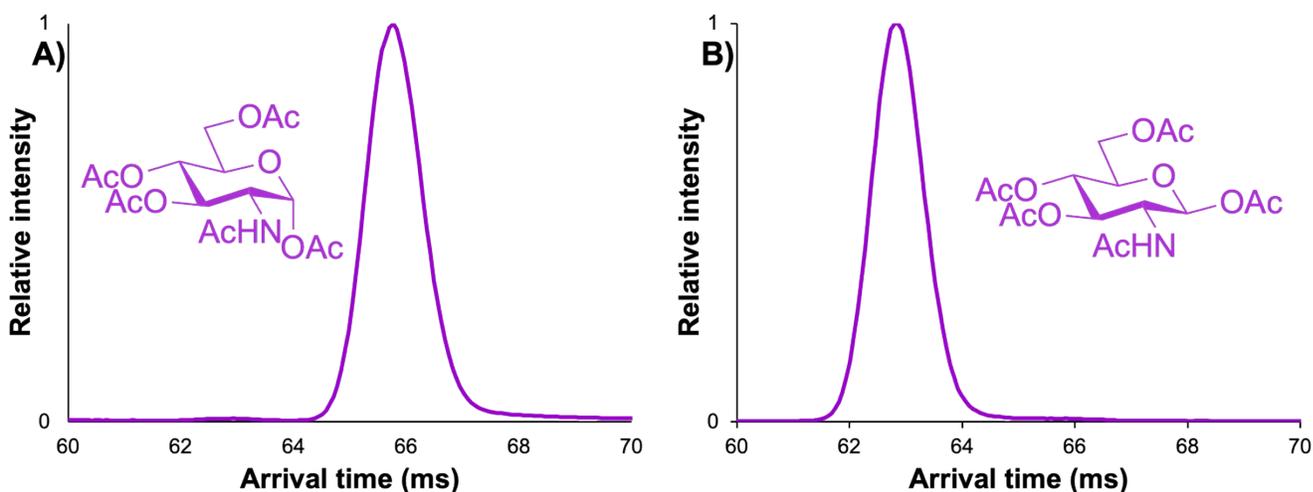
**Figure S2.** 5 m cIMS-MS separation of an equimolar mixture of  $\alpha/\beta$ -D-galactose pentaacetate as their  $[M + Na]^+$  adducts at traveling wave conditions of 450 m/s and 22 V. Each anomer was run individually (see manuscript) to assign the arrival time order.



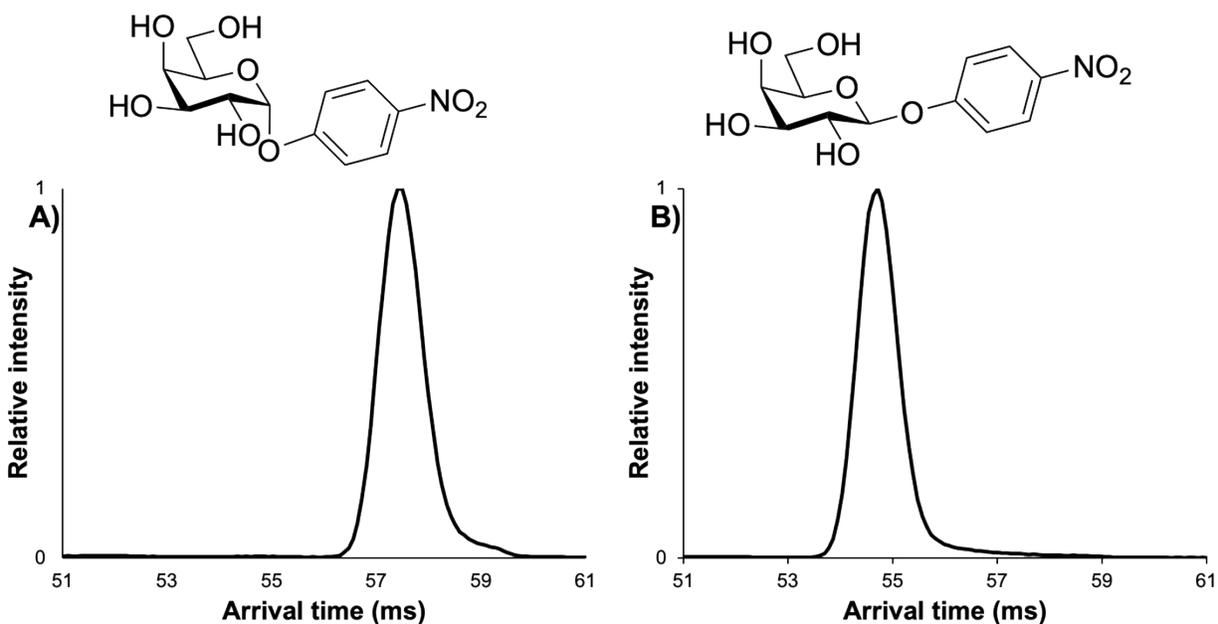
**Figure S3.** 5 m cIMS-MS separation of an equimolar mixture of ethyl 2,3,4,6-tetra-O-acetyl- $\alpha/\beta$ -D-thioglucopyranoside as their  $[M + Na]^+$  adducts at traveling wave conditions of 450 m/s and 25 V. Each anomer was run individually (see manuscript) to assign the arrival time order.



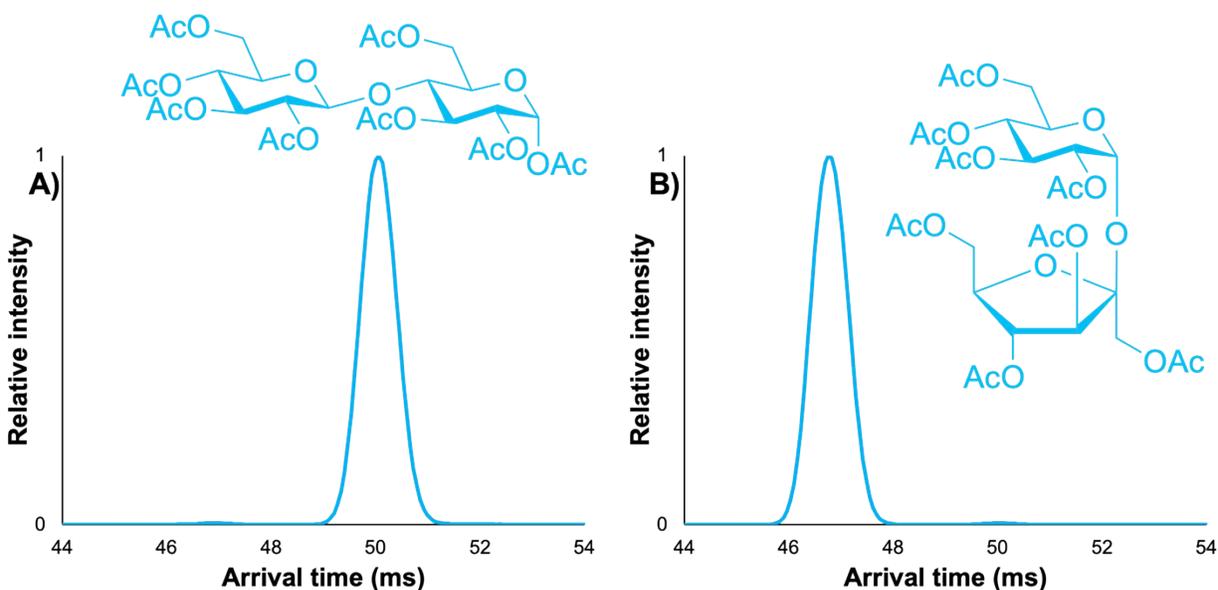
**Figure S4.** 5 m cIMS-MS separations of the  $[M + Na]^+$  adducts for methyl 2,3,4,6-tetra-O-acetyl- $\alpha$ -D-glucopyranoside (A) and methyl 2,3,4,6-tetra-O-acetyl- $\beta$ -D-glucopyranoside (B) at traveling wave conditions of 450 m/s and 20 V. We note no unwanted anomeric impurity was observed for each individual standard.



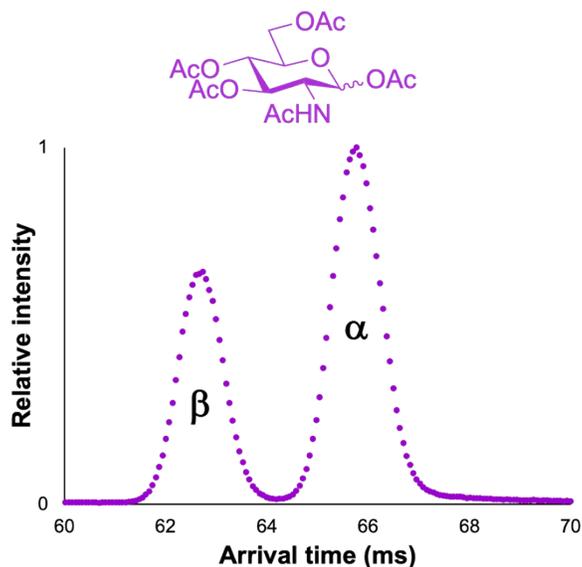
**Figure S5.** 5 m cIMS-MS separations of the  $[M + Na]^+$  adducts for  $\alpha$ -D-glucosamine-pentaacetate (A) and  $\beta$ -D-glucosamine-pentaacetate (B) at traveling wave conditions of 375 m/s and 17 V. We note no unwanted anomeric impurity was observed for each individual standard.



**Figure S6.** 5 m cIMS-MS separations of the  $[M + Na]^+$  adducts for 4-Nitrophenyl- $\alpha$ -D-galactopyranoside (A) and 4-Nitrophenyl- $\beta$ -D-galactopyranoside (B) at traveling wave conditions of 500 m/s and 20 V. We note no unwanted anomeric impurity was observed for each individual standard.

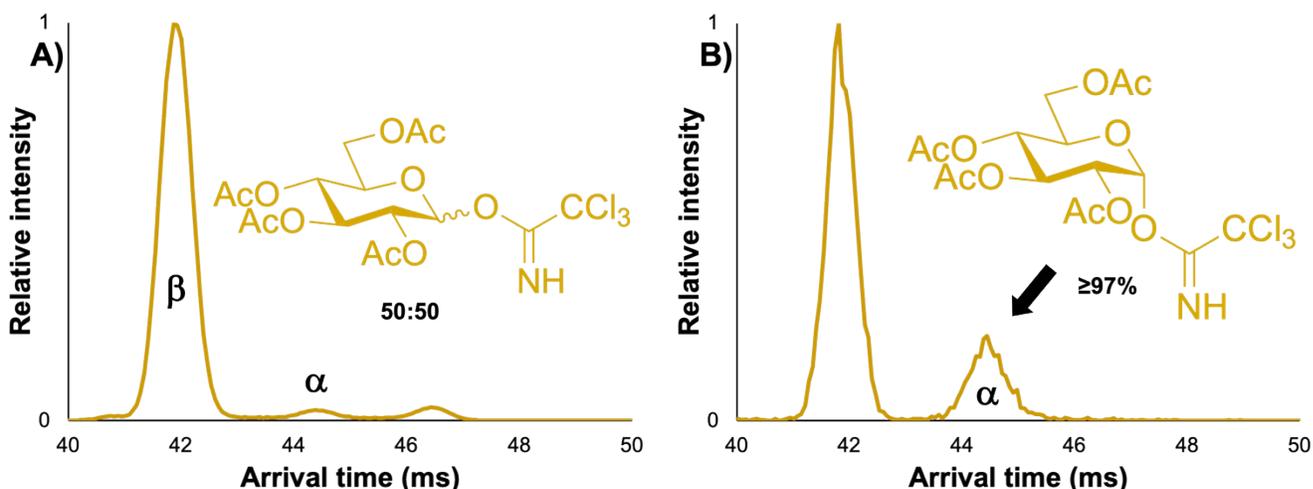


**Figure S7.** 5 m cIMS-MS separations of the  $[M + Na]^+$  adducts for Sucrose octaacetate (A) and  $\alpha$ -D-Cellobiose octaacetate (B) at traveling wave conditions of 350 m/s and 25 V. We note no unwanted impurity was observed for each individual standard.

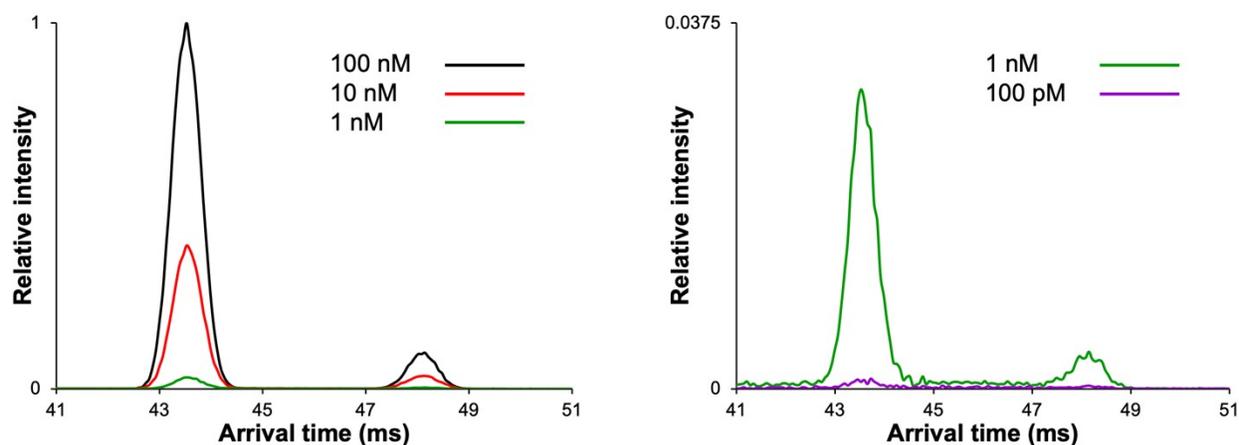
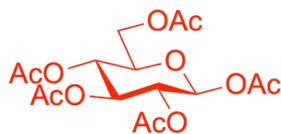


**Figure S8.** 5 m cIMS-MS separation of an equimolar mixture of  $\alpha/\beta$ -D-glucosamine-pentaacetate as its  $[M + Na]^+$  adducts at traveling wave conditions of 375 m/s and 17 V. Displayed are the 200 TOF pushes (purple dots).

We also analyzed the  $\alpha/\beta$  anomers for 2,3,4,6-tetra-O-acetyl-D-glucopyranosyl trichloroacetimidate. Interestingly, the  $\alpha$  anomer form showed signs of extremely poor ionization efficiency (as evidenced by  $\sim 100$  times lower signal when run individually at the same concentration as its  $\beta$  anomer counterpart). This was especially evident in our results from the equimolar (50/50) mixture in Figure S9A, where the anomers are indeed resolved after 5 m, but in drastically different intensities (contrary to our other results). Additionally, the  $\beta$  anomer was observed to ionize with much greater efficiency than the  $\alpha$  one as shown in Figure S9B, where the individually run  $\alpha$  species still contained the  $\beta$  anomeric impurity at much higher intensity levels.



**Figure S9.** 5 m cIMS-MS separation of an equimolar mixture of 2,3,4,6-tetra-O-acetyl- $\alpha/\beta$ -D-glucopyranosyl trichloroacetimidate as their  $[M + Na]^+$  adducts at traveling wave conditions of 450 m/s and 25 V (A) and 2,3,4,6-tetra-O-acetyl- $\alpha$ -D-glucopyranosyl trichloroacetimidate as its  $[M + Na]^+$  adduct at traveling wave conditions of 450 m/s and 25 V (B).



**Figure S10.** Arrival time distributions for the  $\beta$ -D-glucose pentaacetate anomer run at various concentrations as its  $[M + Na]^+$  adduct after 5 m of cIMS-MS separation at the same TW conditions as presented in the manuscript (Figure 2). We note the lower mobility peak is the minor  $\alpha$ -anomer impurity. We observe an LOD of  $\sim 1$  nM, where S/N ratios fell below 3 at concentrations of  $< 1$  nM.