

## Supplementary Material

### Studies in glycopeptide synthesis

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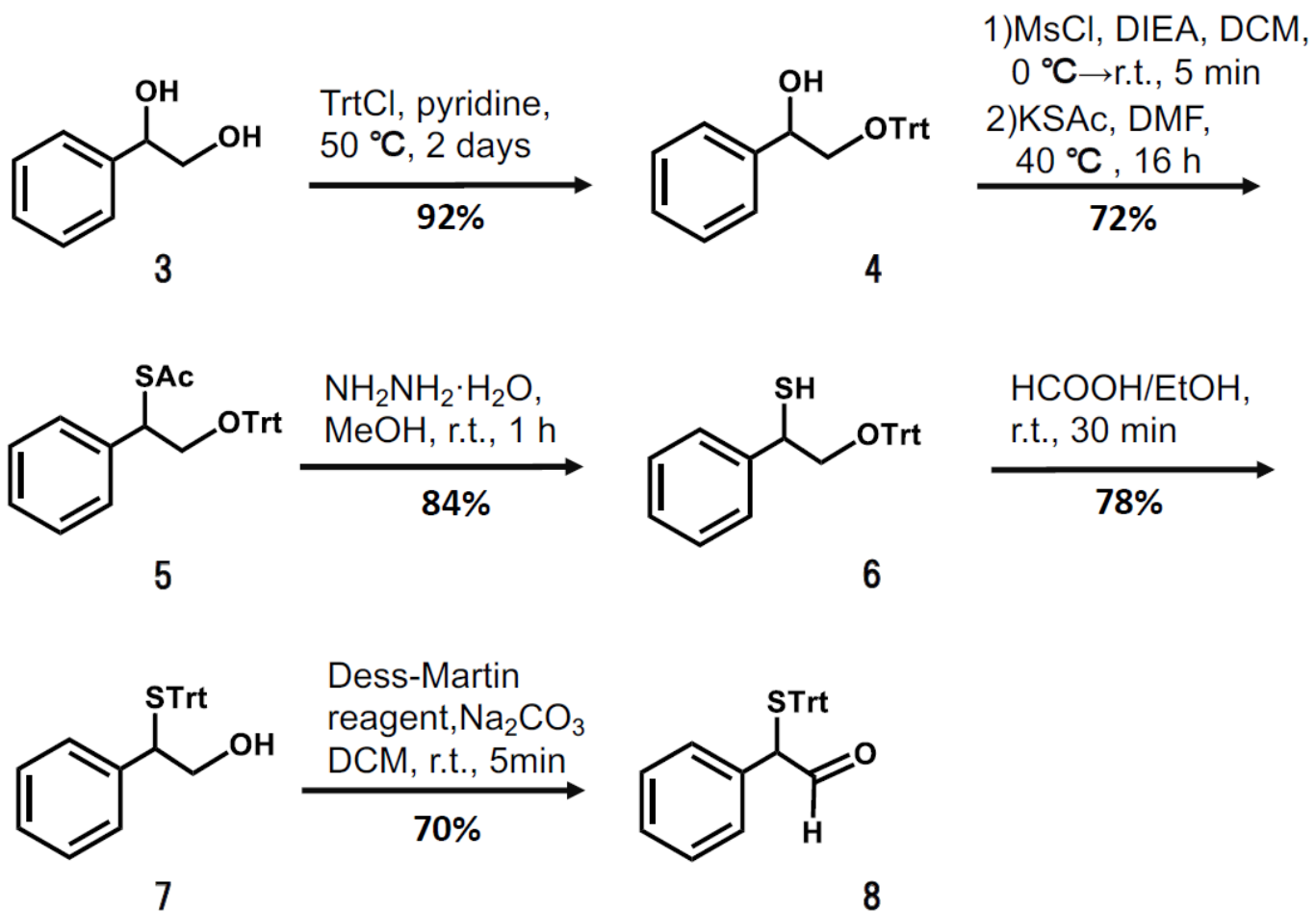
<sup>b</sup> Graduate School of Science, Osaka University, 1-1, Machikaneyama, Toyonaka, 560-0043 Japan

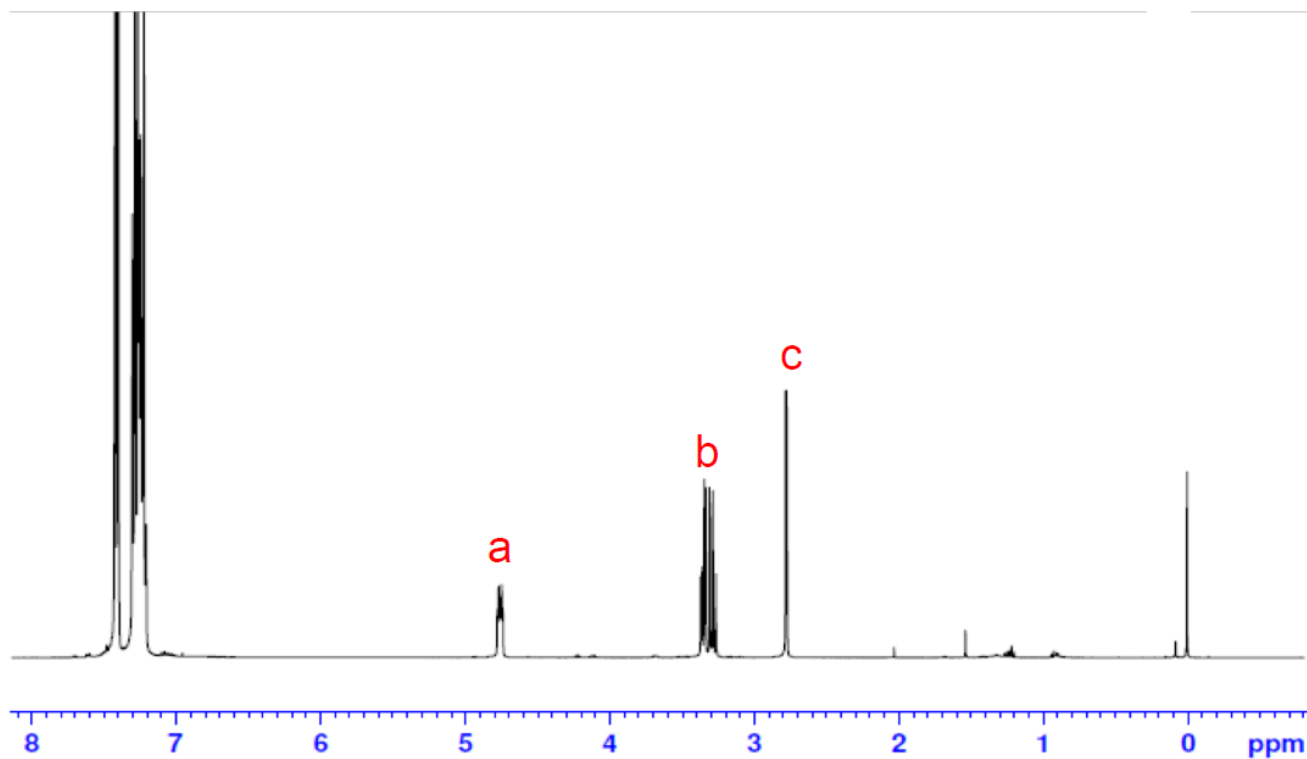
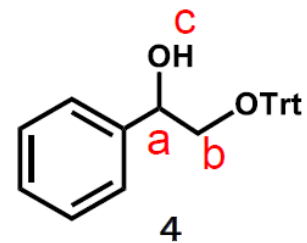
E-mail: [kajihara@chem.sci.osaka-u.ac.jp](mailto:kajihara@chem.sci.osaka-u.ac.jp)

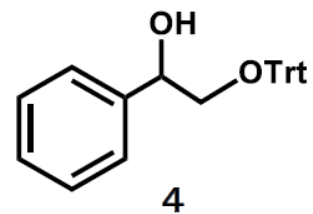
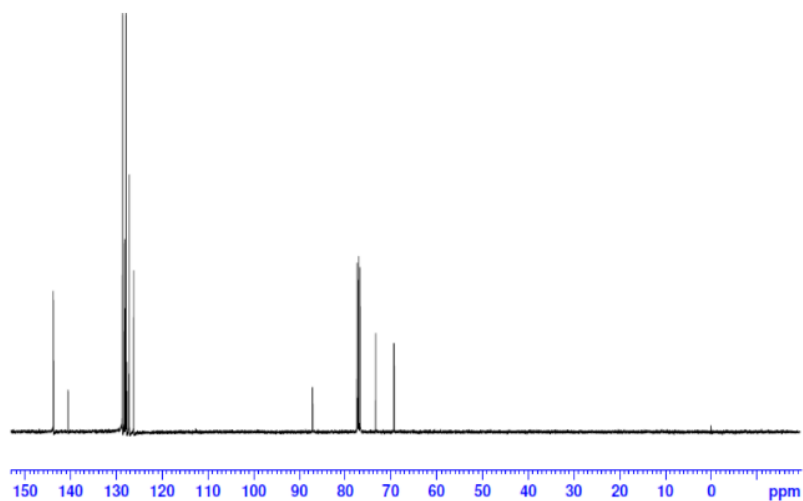
#### Table of Contents

<sup>1</sup> H, <sup>13</sup> C NMR spectra, Mass data, and HPLC profiles of compounds.....	S2
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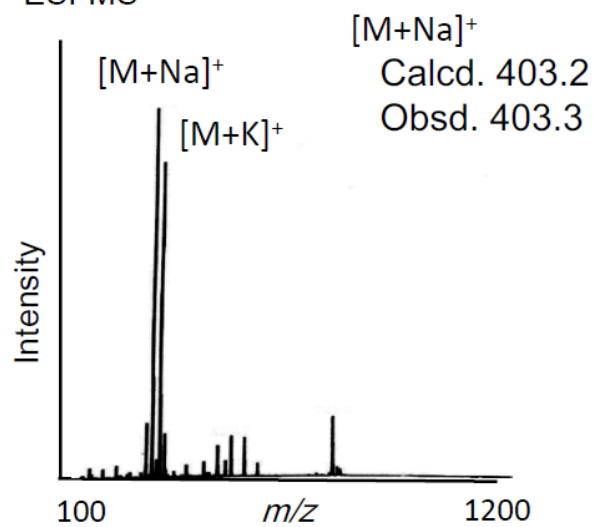
## Synthesis of Auxiliary group

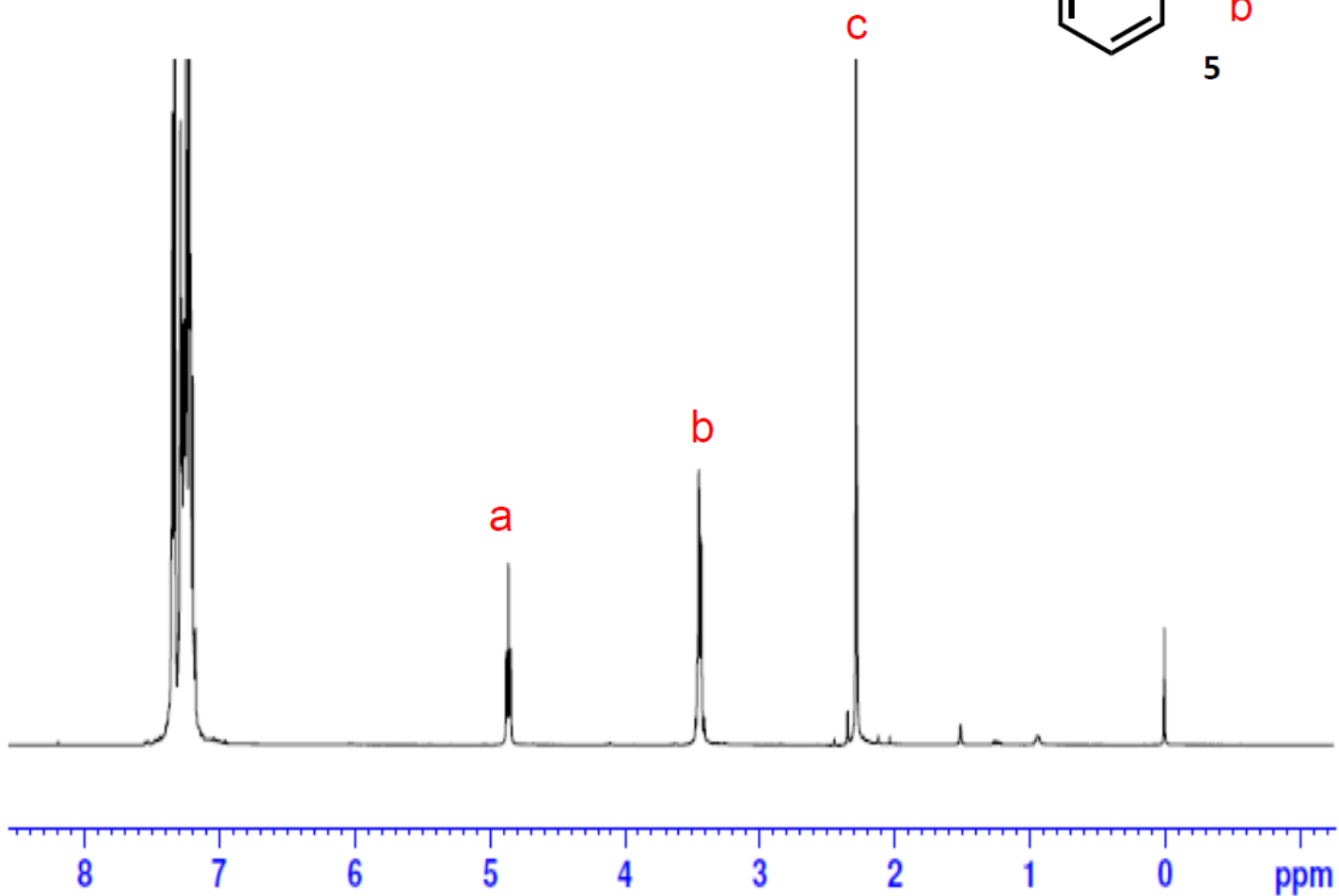
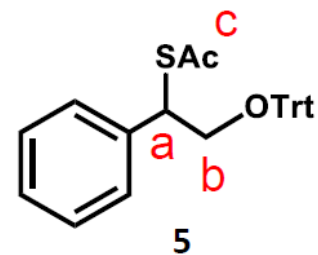


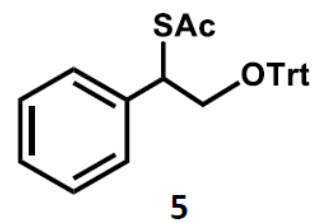
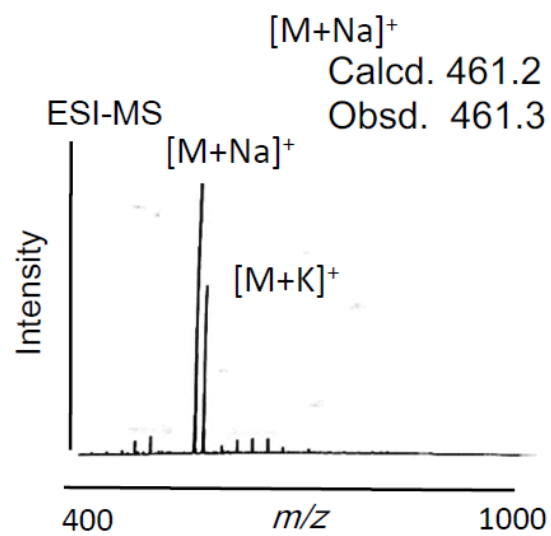
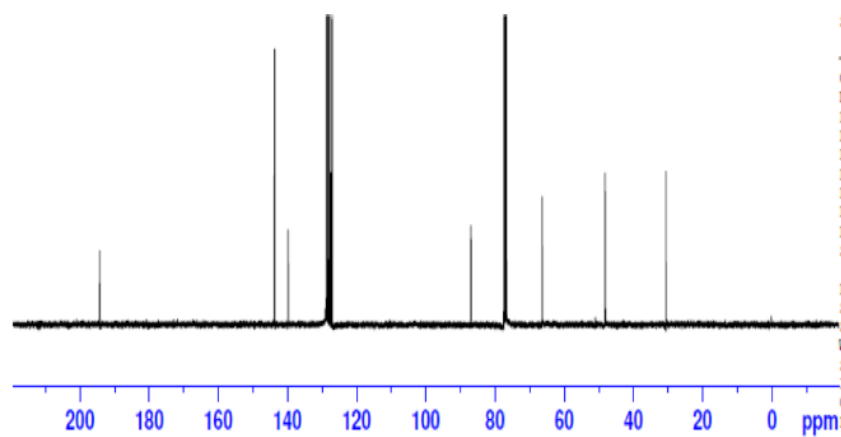
$^1\text{H}$ NMR

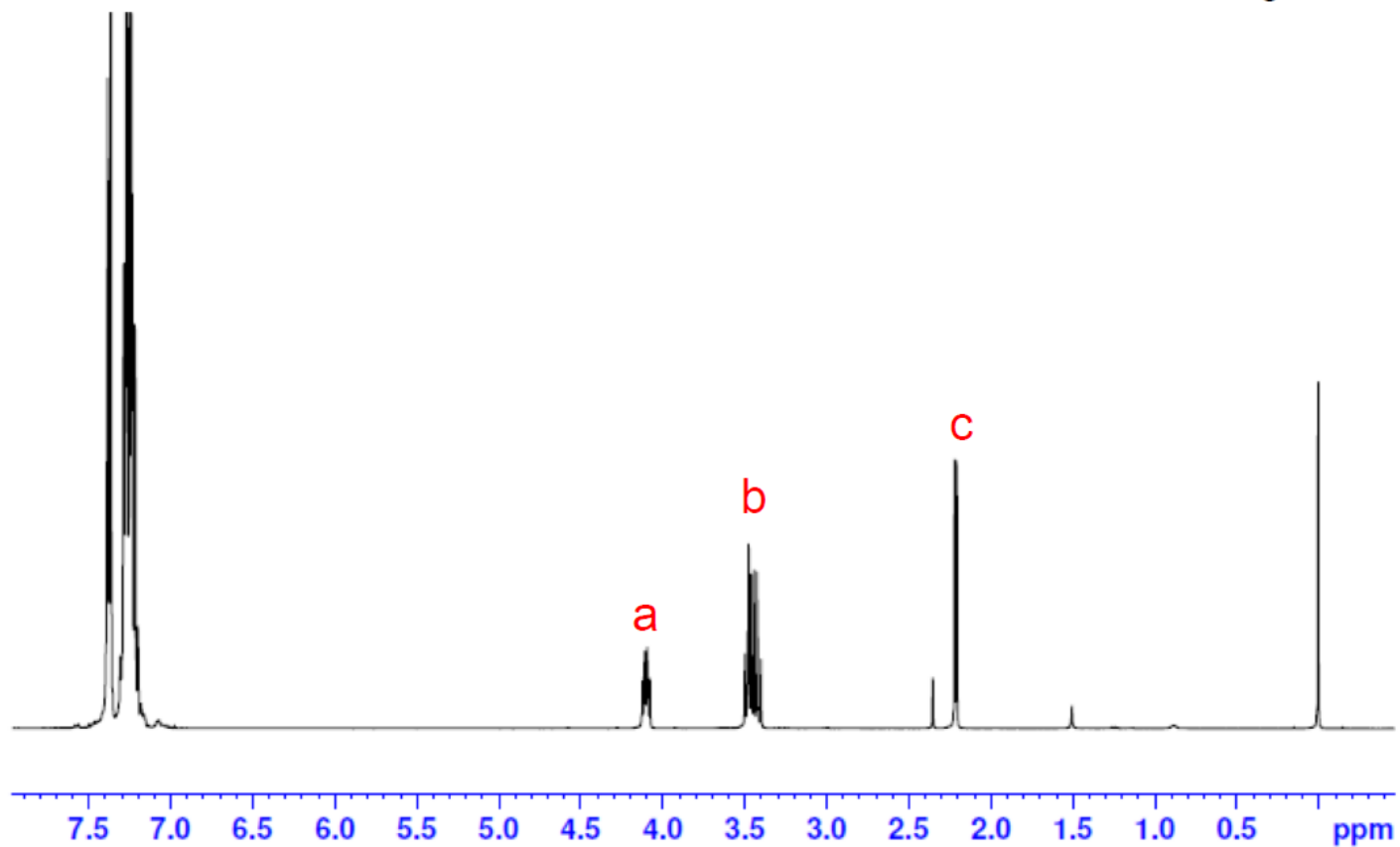
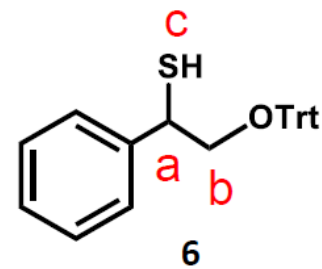
 $^{13}\text{C}$ NMR

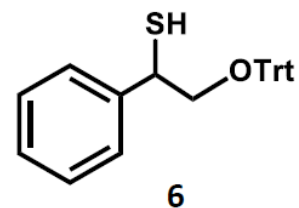
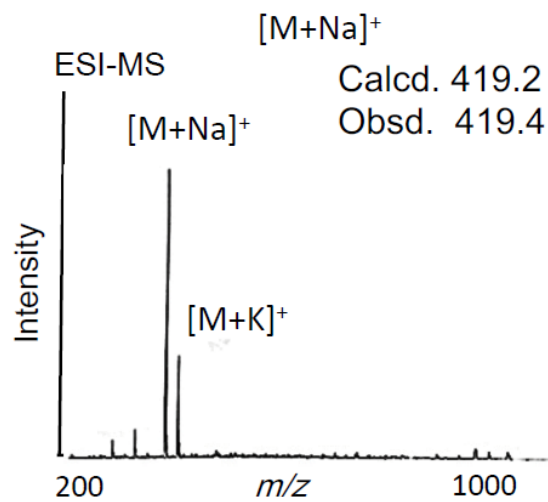
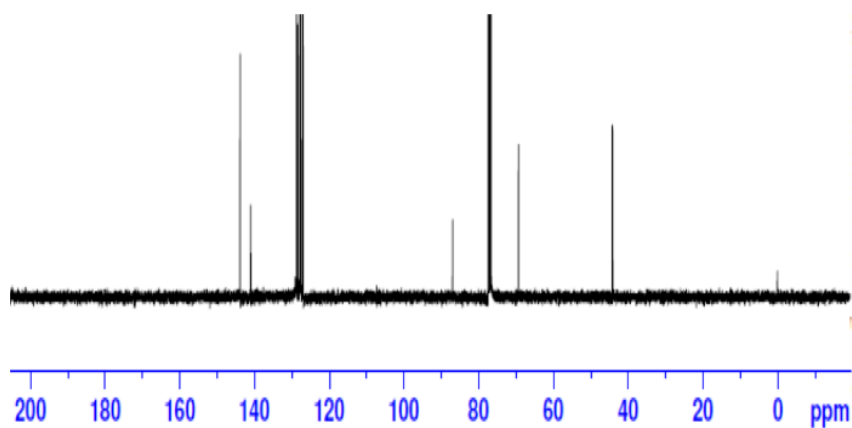
## ESI-MS



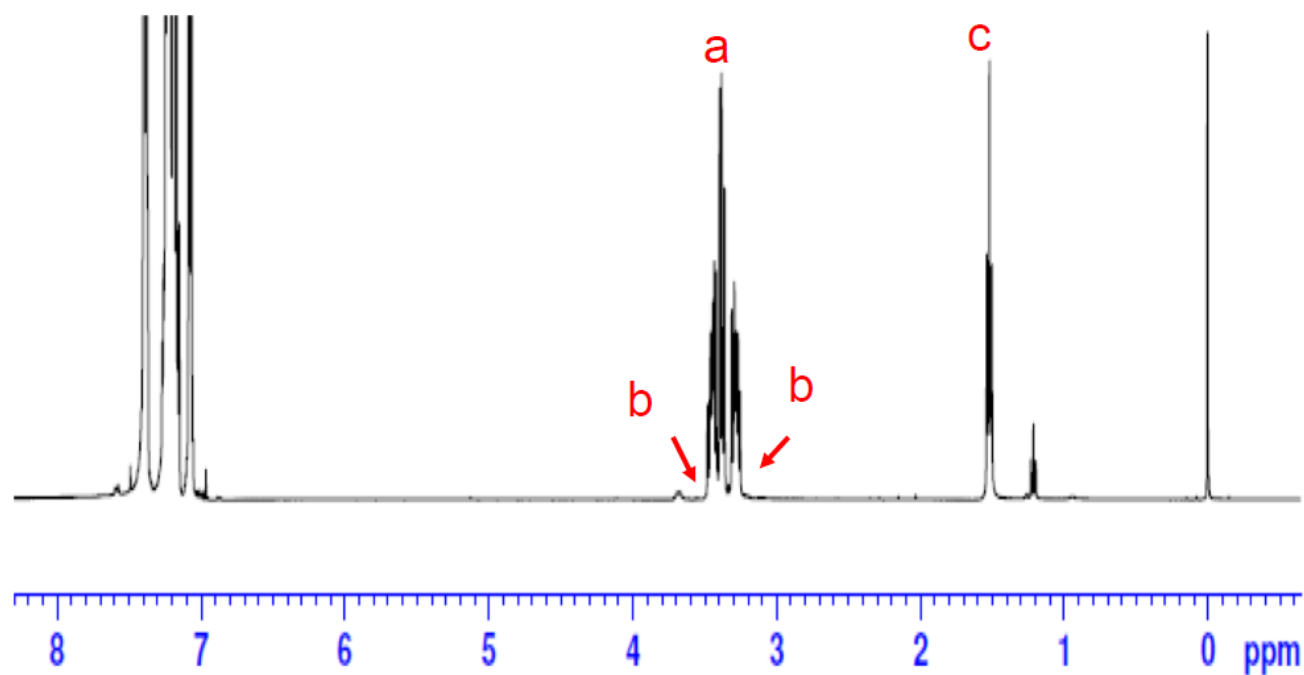
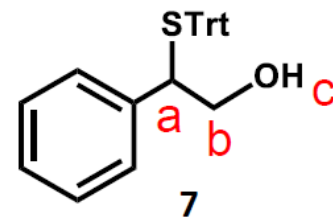
$^1\text{H}$ NMR

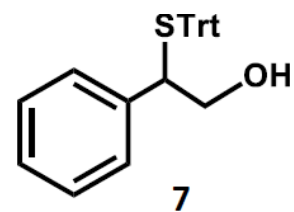
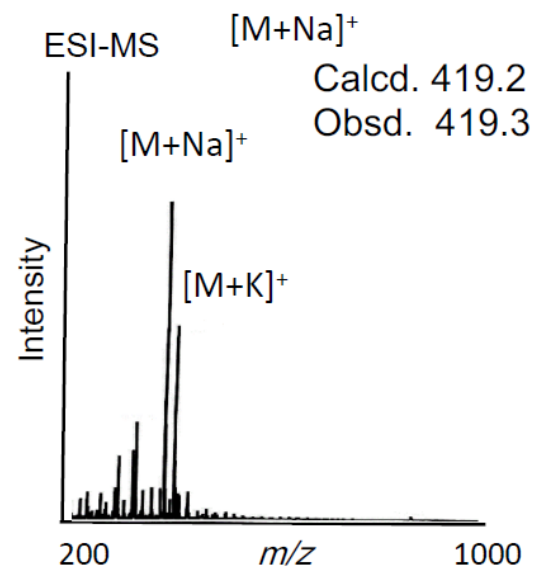
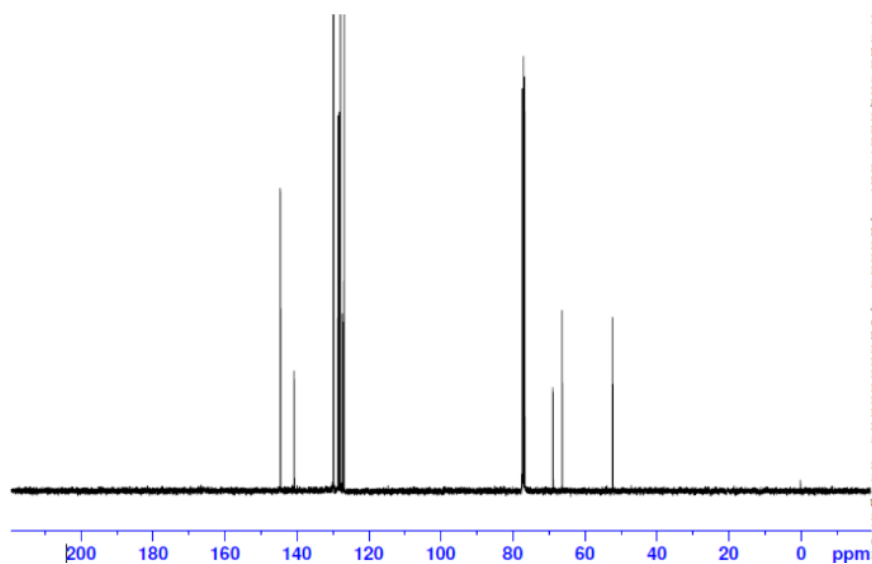
 $^{13}\text{C}$ NMR

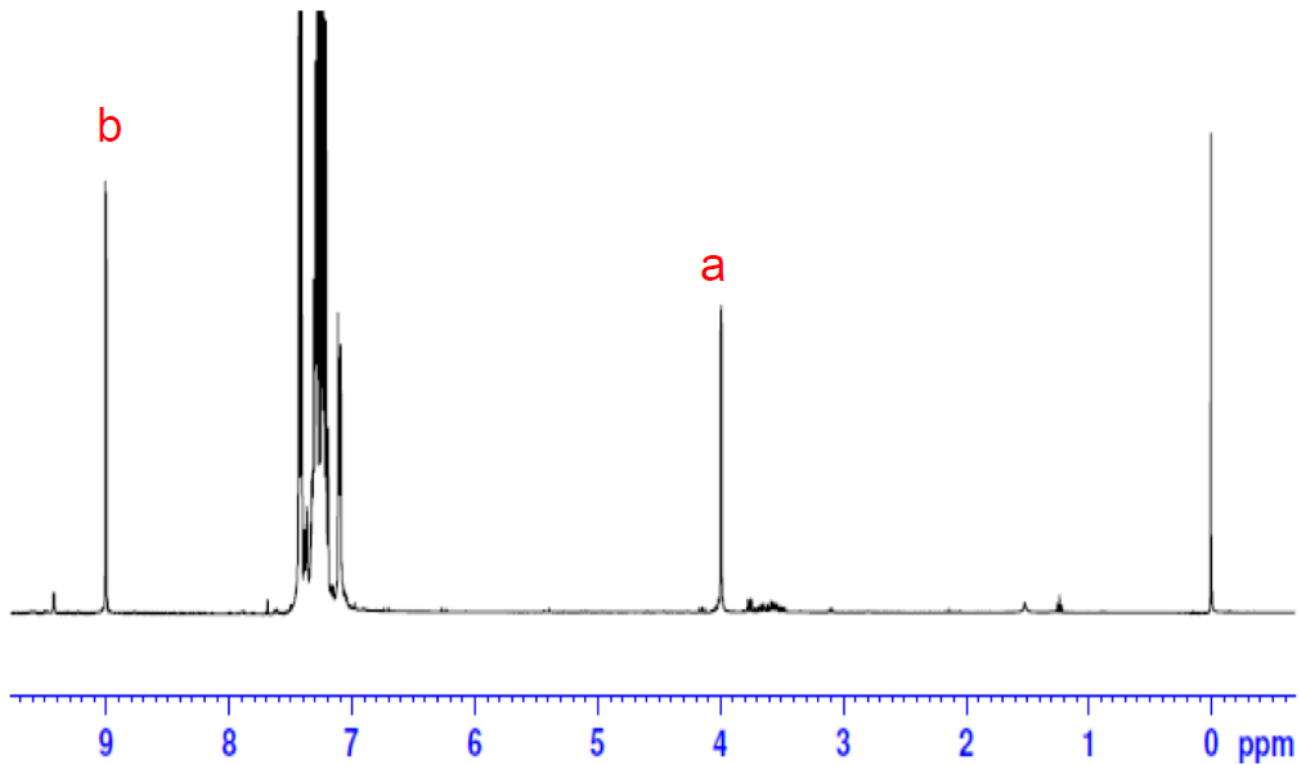
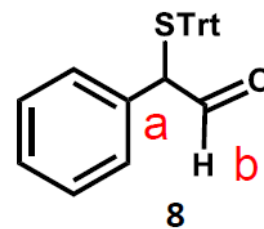
$^1\text{H}$ NMR

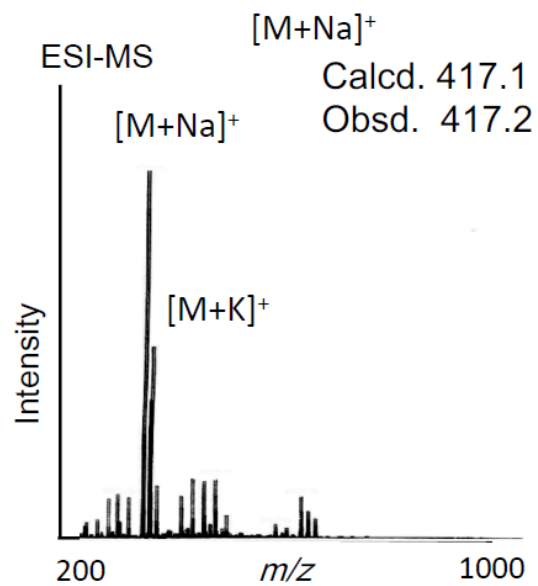
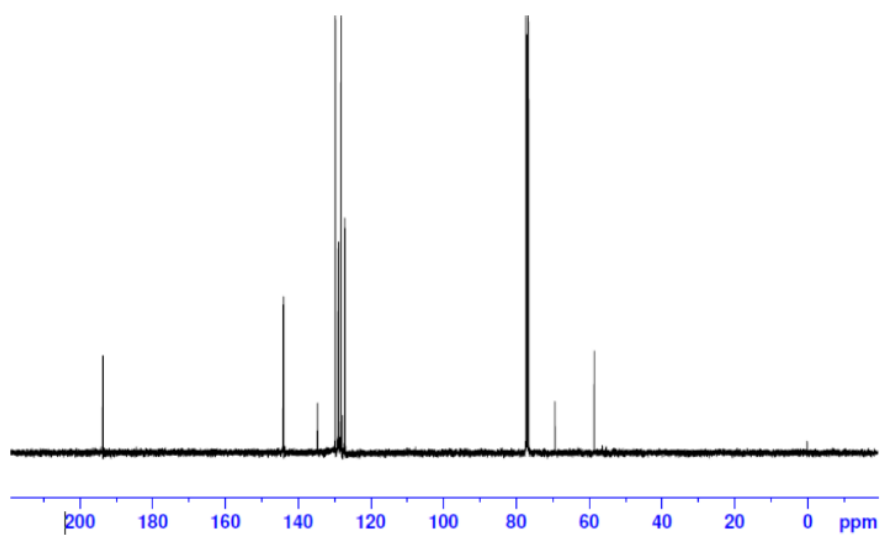
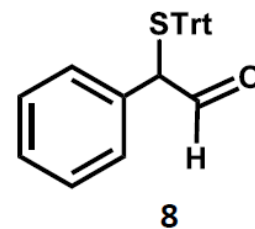
 $^{13}\text{C}$ NMR



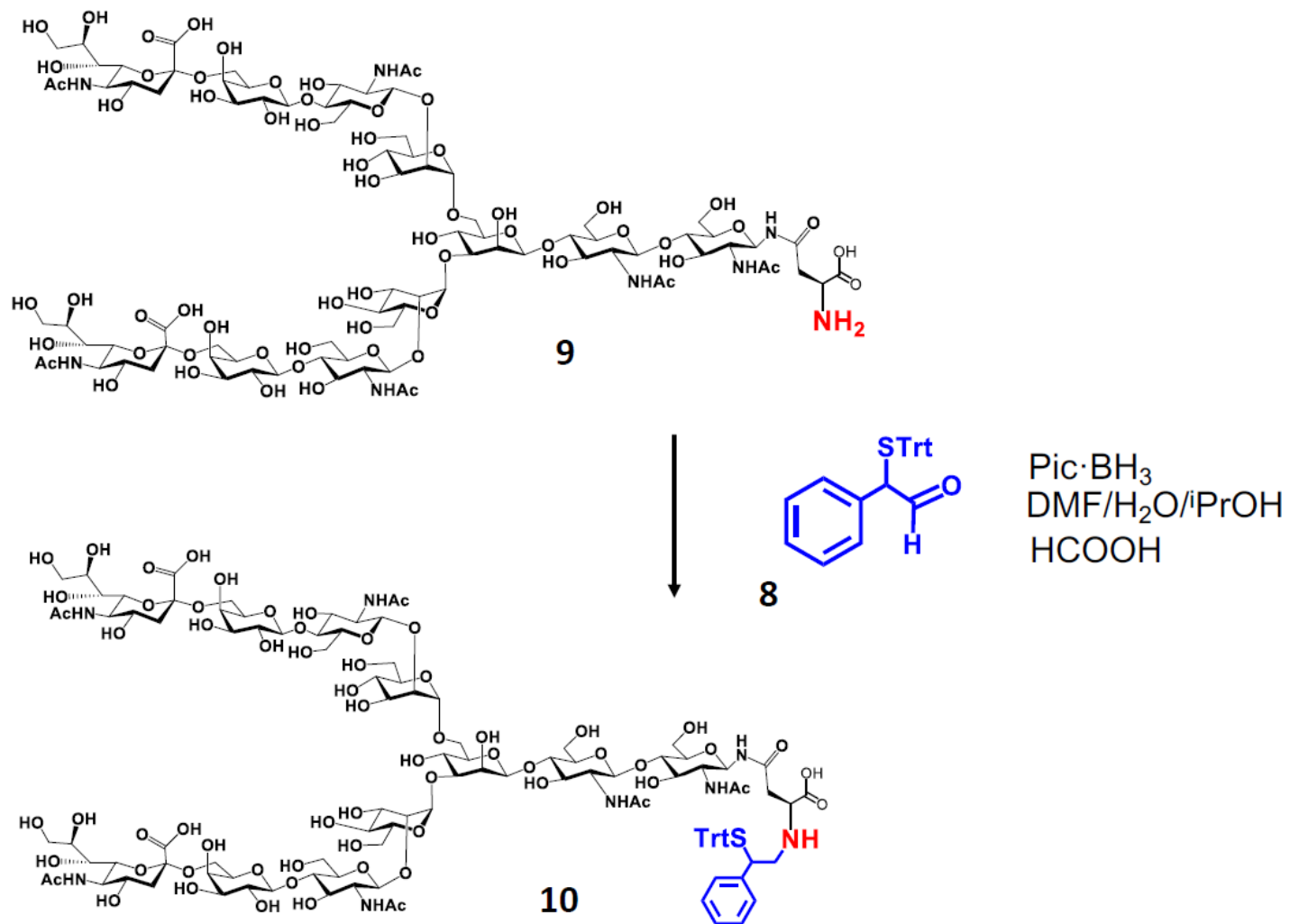
$^1\text{H}$ NMR

 $^{13}\text{C}$ NMR

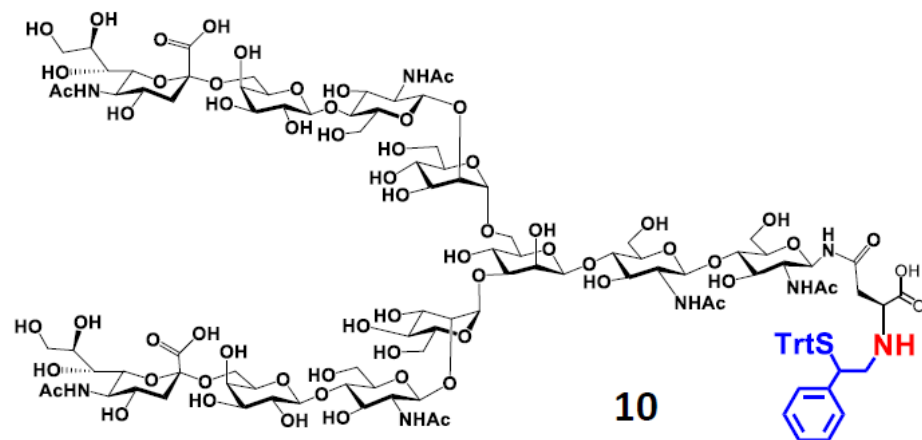
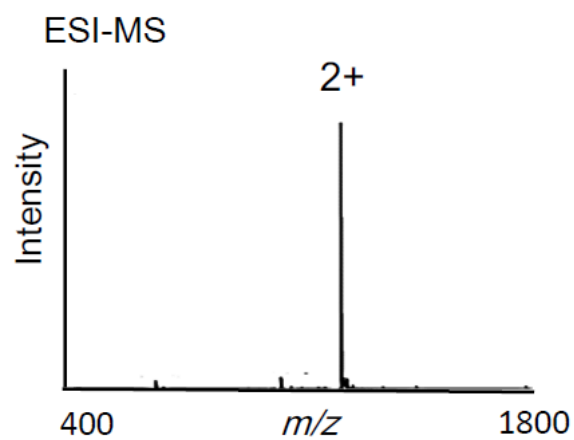
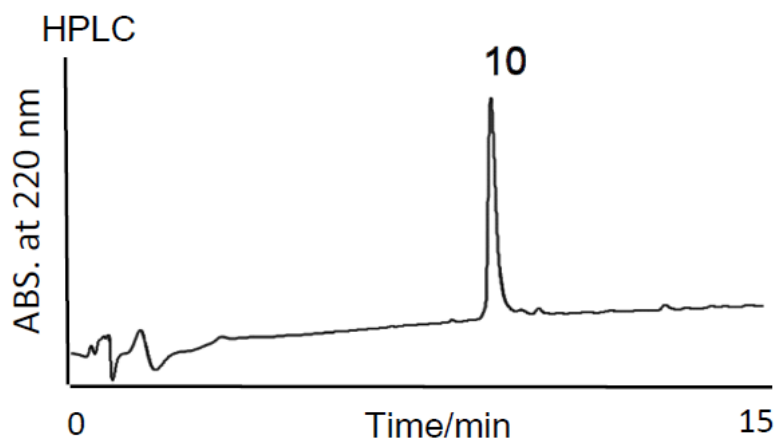
$^1\text{H}$ NMR

$^{13}\text{C}$ NMR

## Incorporation of Auxiliary Group to N-glycan

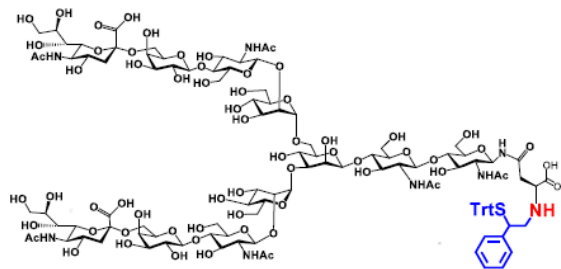
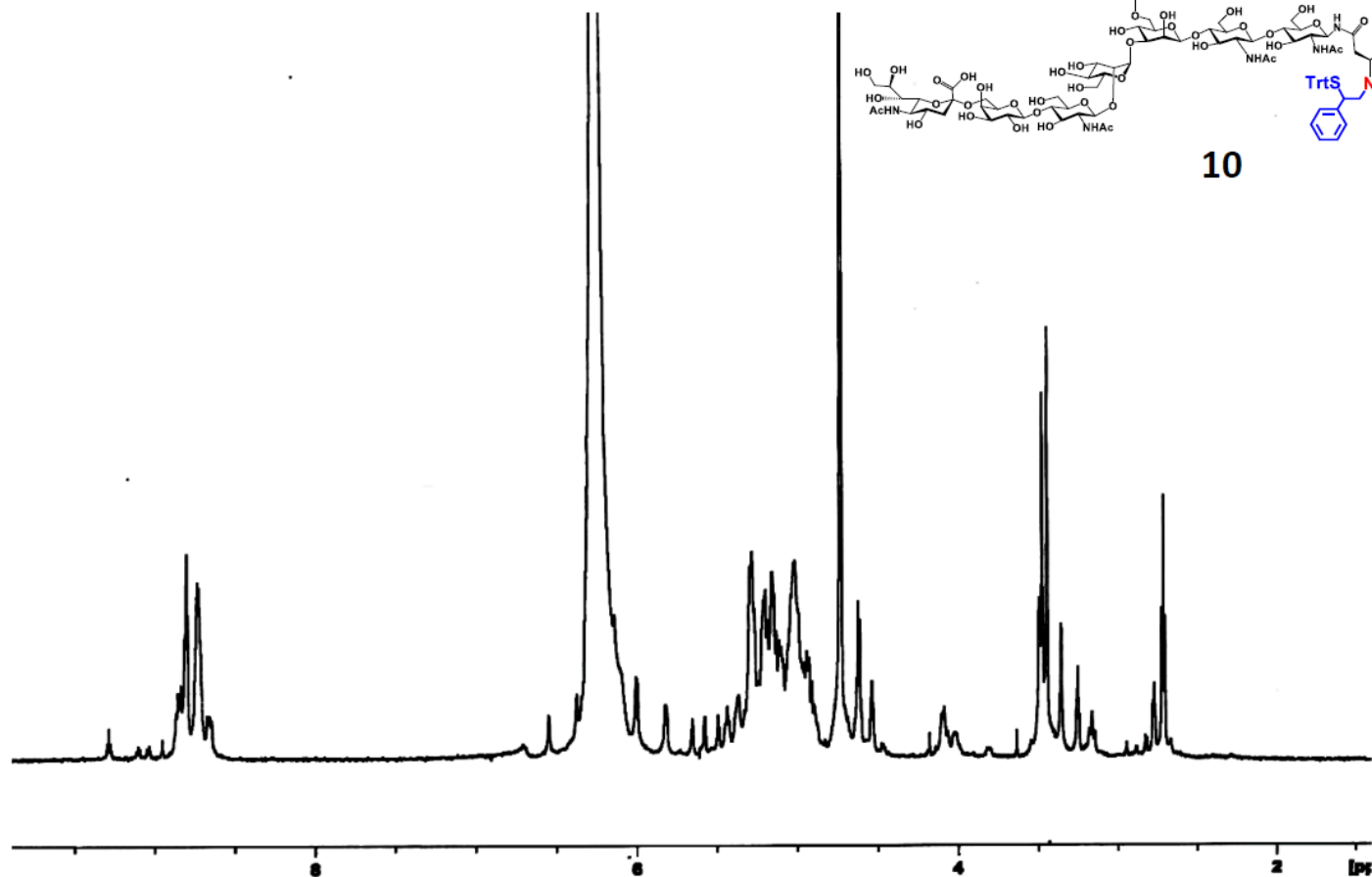


## Incorporation of Auxiliary Group to N-glycan

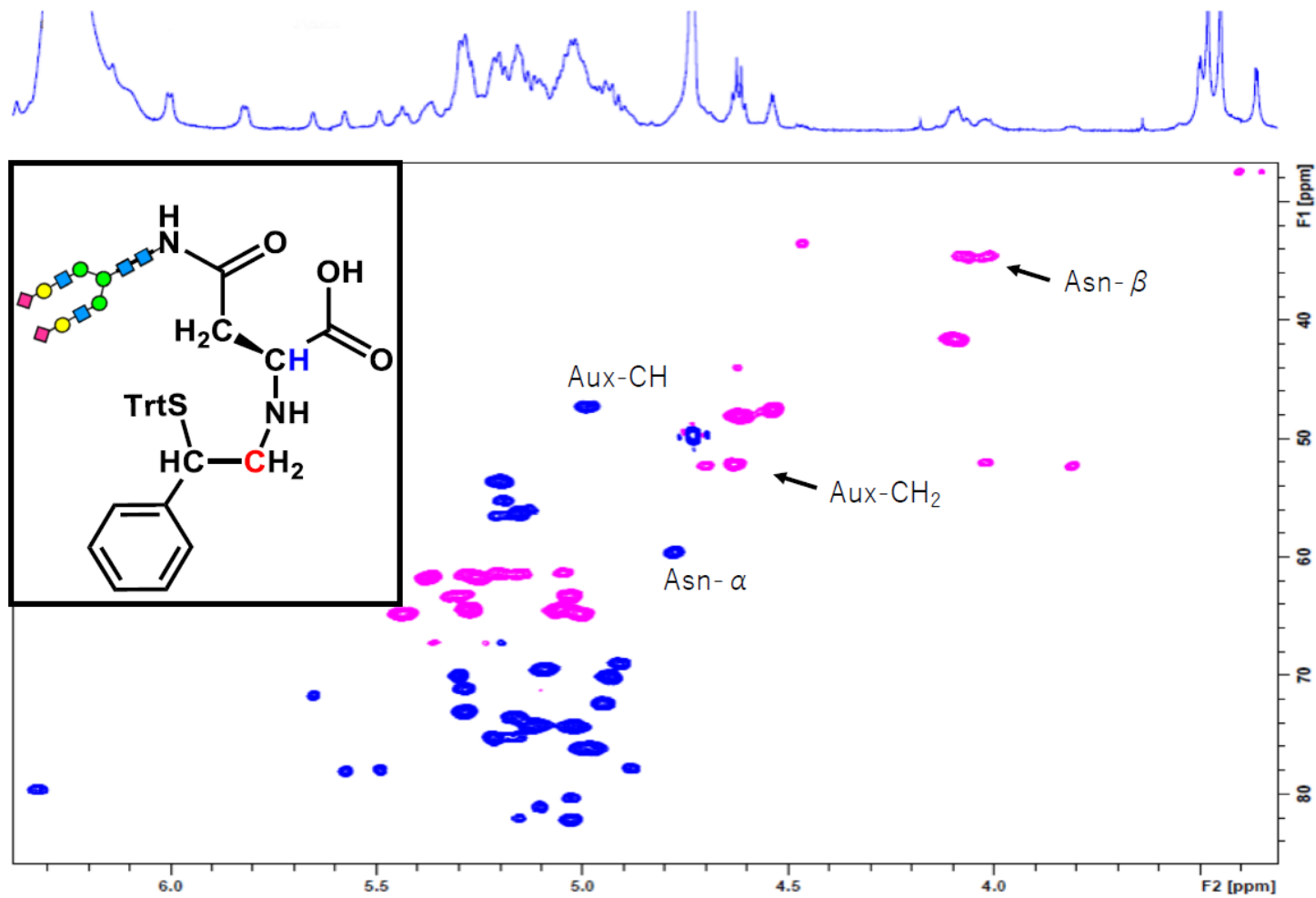


Calcd. 2716.0  
Obsd. 2715.8

yield 55%

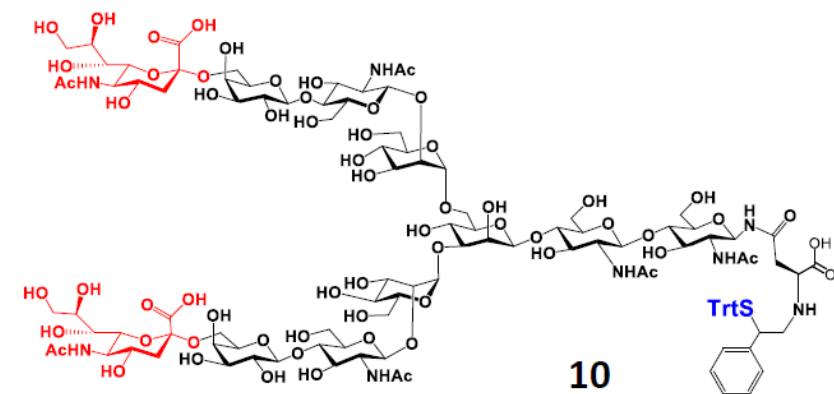
$^1\text{H}$ NMR (700MHz)**10**

## HSQC NMR (700MHz)

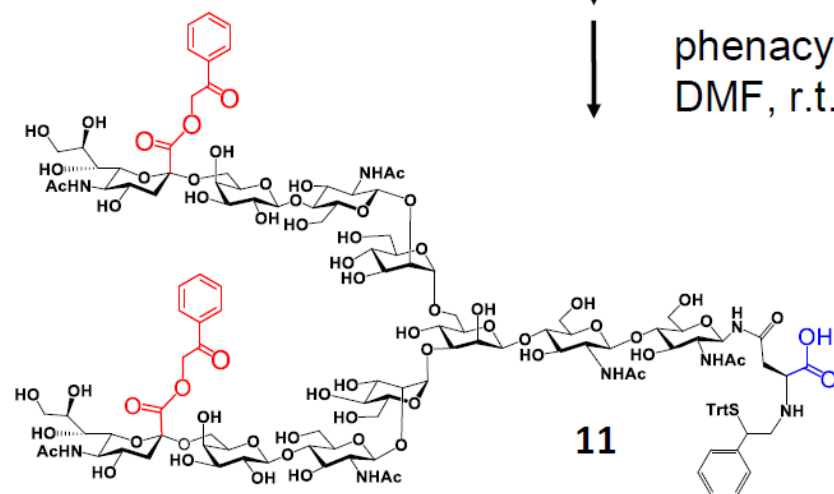




## Protection of sialic acids

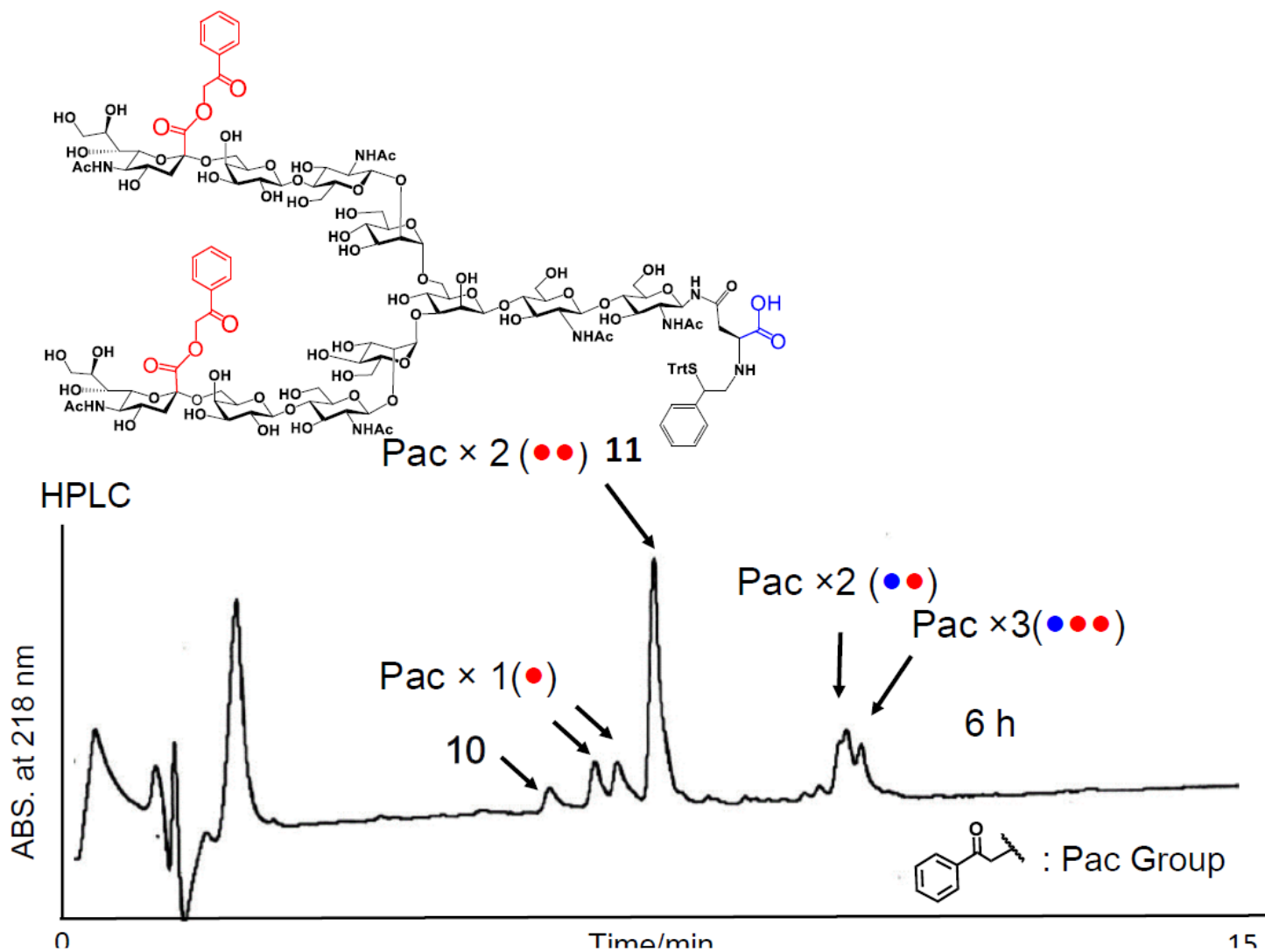


↓ adjusted to pH 4.0 by  $\text{Cs}_2\text{CO}_3$   
↓ phenacyl bromide (6 eq)  
↓ DMF, r.t., 6 h

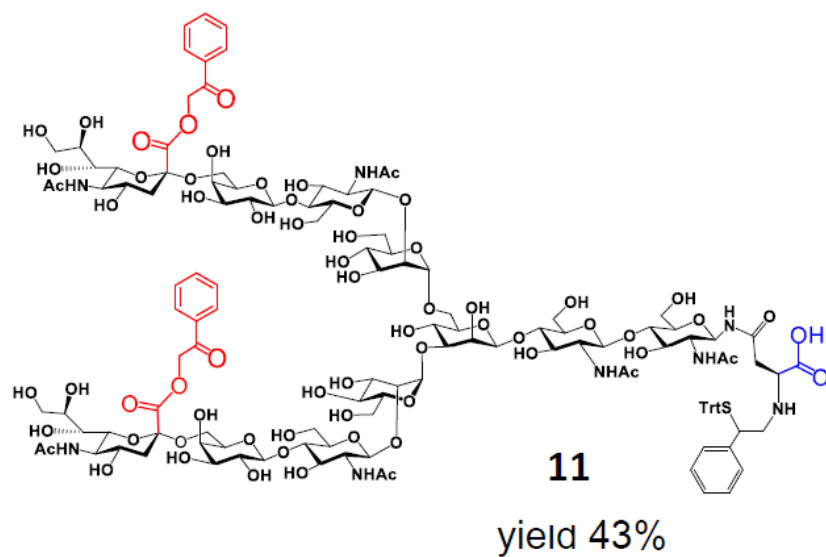
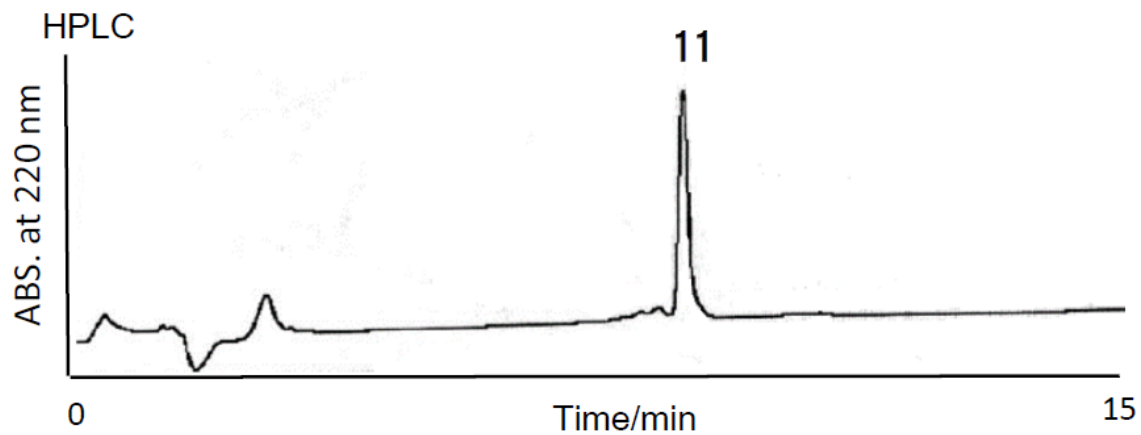


M. Murakami, *et.al.*, *Angew.Chem. Int.Ed.* **2012**, *51*, 3567-3572

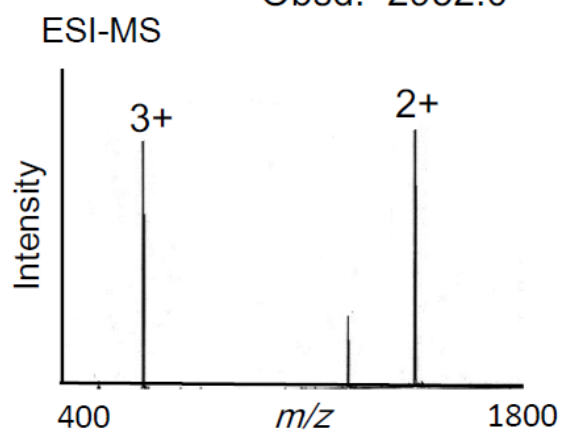
## Protection of sialic acids



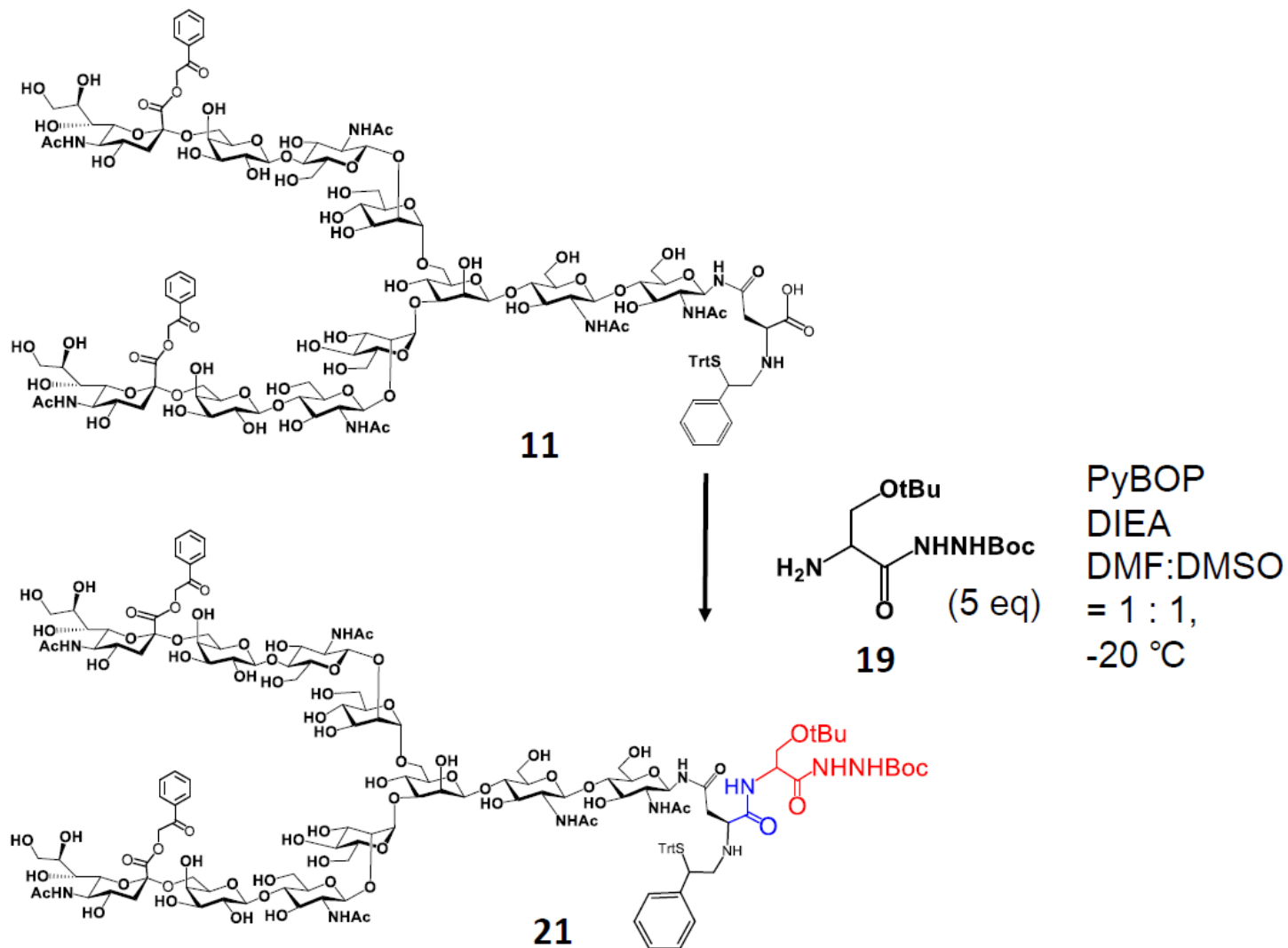
## Protection of sialic acids



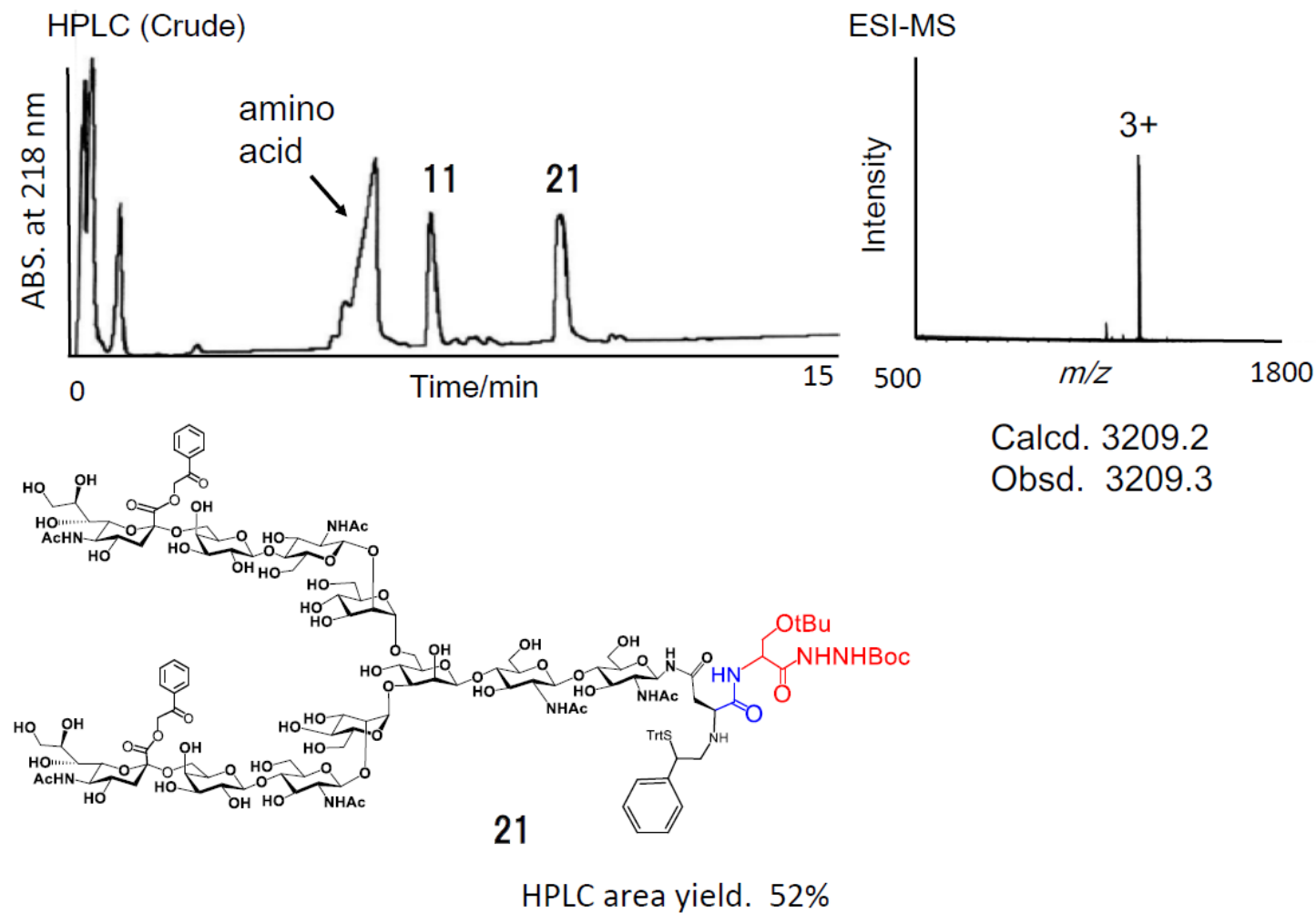
Calcd. 2951.1  
Obsd. 2952.0



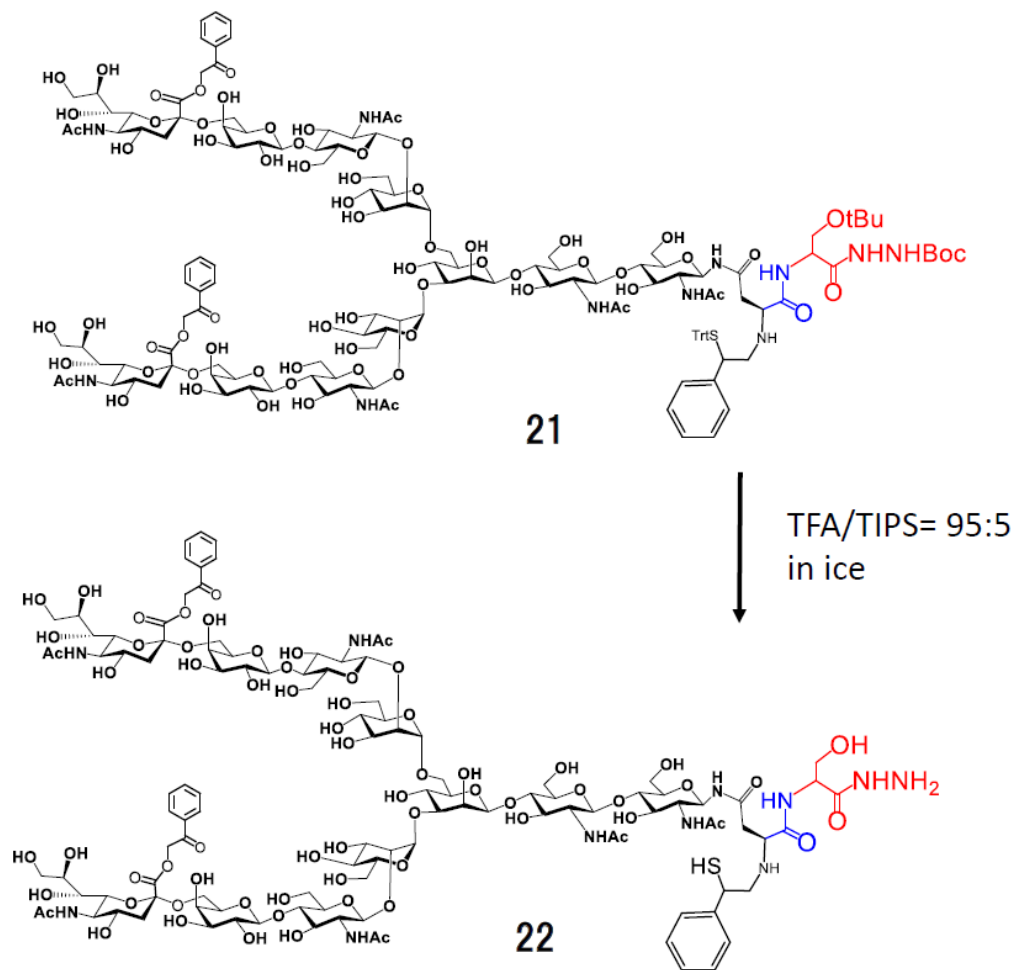
## Coupling Reaction



## Coupling Reaction



## Deprotection



Calcd. 2811.0  
Obsd. 2810.9

