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Supporting Information

Novel strategies for the synthesis of unsymmetrical glycosyl disulfides

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General methods - NMR spectra for compounds 6b and 6c were generated on a JEOL ECA-60. NMR spectra for compounds 6a, 7a-7f, 9a, 9e-9g were generated on a Bruker AMX 400. Compounds 9b-9d have been reported previously (*Tetrahedron Lett.* 2007, 48, 7637-7641).





Figure 1. ¹H and ¹³C NMR spectra for compound **6a**.





Figure 2. ¹H and ¹³C NMR spectra for compound **6b**.



Figure 3. ¹H and ¹³C NMR spectra for compound 6c.



Figure 4. ¹H and ¹³C NMR spectra for compound **7a**.





Figure 5. ¹H and ¹³C NMR spectra for compound **7b**.



Figure 6. 1 H and 13 C NMR spectra for compound 7c.



Figure 7. ¹H and ¹³C NMR spectra for compound 7d.





Figure 8. ¹H and ¹³C NMR spectra for compound 7e.



Figure 9. ¹H and ¹³C NMR spectra for compound **7f.**





Figure 10. ¹H and ¹³C NMR spectra for compound 9a.



Figure 11. ¹H and ¹³C NMR spectra for compound **9e**. In the ¹H NMR spectrum the strong singlet peaks at 1.56 and 2.17 ppm correspond to water and acetone, respectively. In the ¹³C NMR spectrum the peak at 30.32 ppm corresponds to the methyl carbon of acetone.



Figure 12. ¹H and ¹³C NMR spectra for compound 9f.





Figure 13. ¹H and ¹³C NMR spectra for compound 9g.