## **Supplementary Material**

## Synthesis of (5,6 & 6,6)-oxa-oxa annulated sugars as glycosidase inhibitors from 2-formyl galactal using iodocyclization as a key step

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-



Figures S 1. <sup>1</sup>H NMR spectrum of compound 17.



Figures S 2. <sup>13</sup>C NMR spectrum of compound 17.



Figures S 3. <sup>1</sup>H NMR spectrum of compound 19.



Figures S 4. <sup>13</sup>C NMR spectrum of compound 19.



Figure S 5. <sup>1</sup>H NMR spectrum of compound 20a.



Figure S 6. <sup>13</sup>C NMR spectrum of compound 20a.



Figure S 7. <sup>1</sup>H NMR spectrum of compound 20b.



Figure S 8. <sup>13</sup>C NMR spectrum of compound 20b.



Figure S 9. <sup>1</sup>H NMR spectrum of compound 21.



Figure S 10. <sup>13</sup>C NMR spectrum of compound 21.



Figure S 11. <sup>1</sup>H NMR spectrum of compound 22.



Figure S 12. <sup>13</sup>C NMR spectrum of compound 22.



Figure S 13. Dept-135 spectrum of compound 22.



Figure S 14. <sup>1</sup>H-<sup>1</sup>H COSY spectrum of compound 22.





Figure S 15. nOe spectrum of compound 22 for irradiation of H-1.

![](_page_10_Figure_4.jpeg)

Figure S 16. nOe spectrum of compound 22 for irradiation of H-2.

![](_page_11_Figure_2.jpeg)

Figure S 17. <sup>1</sup>H NMR spectrum of compound 23.

![](_page_11_Figure_4.jpeg)

Figure S 18. <sup>13</sup>C NMR spectrum of compound 23.

![](_page_12_Figure_2.jpeg)

Figure S 19. <sup>1</sup>H NMR spectrum of compound 24.

![](_page_12_Figure_4.jpeg)

![](_page_13_Figure_2.jpeg)

Figure S 21. <sup>1</sup>H NMR spectrum of compound 25.

![](_page_13_Figure_4.jpeg)

Figure S 22. <sup>13</sup>C NMR spectrum of compound 25.

![](_page_14_Figure_2.jpeg)

Figure S 23. <sup>1</sup>H NMR spectrum of compound 26.

![](_page_14_Figure_4.jpeg)

Figure S 24. <sup>13</sup>C NMR spectrum of compound 26.

![](_page_15_Picture_2.jpeg)

![](_page_15_Figure_3.jpeg)

Figure S 25. <sup>1</sup>H-<sup>1</sup>H COSY spectrum of compound 26.

![](_page_16_Figure_2.jpeg)

Figure S 26. nOe spectrum of compound 26 for irradiation of H-1.

![](_page_16_Figure_4.jpeg)

Figure S 27. nOe spectrum of compound 26 for irradiation of H-2.

![](_page_17_Figure_2.jpeg)

Figure S 28. nOe spectrum of compound 26 for irradiation of H-5.

![](_page_17_Figure_4.jpeg)

Figure S 29. nOe spectrum of compound 26 for irradiation of H-8.

![](_page_18_Figure_2.jpeg)

Figure S 30. <sup>1</sup>H NMR spectrum of compound 27.

![](_page_18_Figure_4.jpeg)

Figure S 31. <sup>13</sup>C NMR spectrum of compound 27.

![](_page_19_Figure_2.jpeg)

Figure S 32. <sup>1</sup>H NMR spectrum of compound 28a.

![](_page_19_Figure_4.jpeg)

Figure S 33. <sup>13</sup>C NMR spectrum of compound 28a.

![](_page_20_Figure_2.jpeg)

Figure S 34. <sup>1</sup>H NMR spectrum of compound 29.

![](_page_20_Figure_4.jpeg)

Figure S 35. <sup>13</sup>C NMR spectrum of compound 29.

![](_page_21_Figure_2.jpeg)

Figure S 36. <sup>1</sup>H NMR spectrum of compound 31.

![](_page_21_Figure_4.jpeg)

Figure S 37. <sup>13</sup>C NMR spectrum of compound 31.

![](_page_22_Figure_2.jpeg)

Figure S 38. <sup>1</sup>H NMR spectrum of compound 32.

![](_page_22_Figure_4.jpeg)

Figure S 39. <sup>13</sup>C NMR spectrum of compound 32.

![](_page_23_Figure_2.jpeg)

Figure S 40. <sup>1</sup>H-<sup>1</sup>H COSY spectrum of compound 32.

![](_page_24_Figure_2.jpeg)

![](_page_24_Figure_3.jpeg)

Figure S 41. nOe spectrum of compound 32 for irradiation of H-1.

![](_page_25_Figure_2.jpeg)

![](_page_25_Figure_3.jpeg)

Figure S 42. nOe spectrum of compound 32 for irradiation of H-3.

![](_page_26_Figure_2.jpeg)

![](_page_26_Figure_3.jpeg)

Figure S 43. nOe spectrum of compound 32 for irradiation of H-2.