

## 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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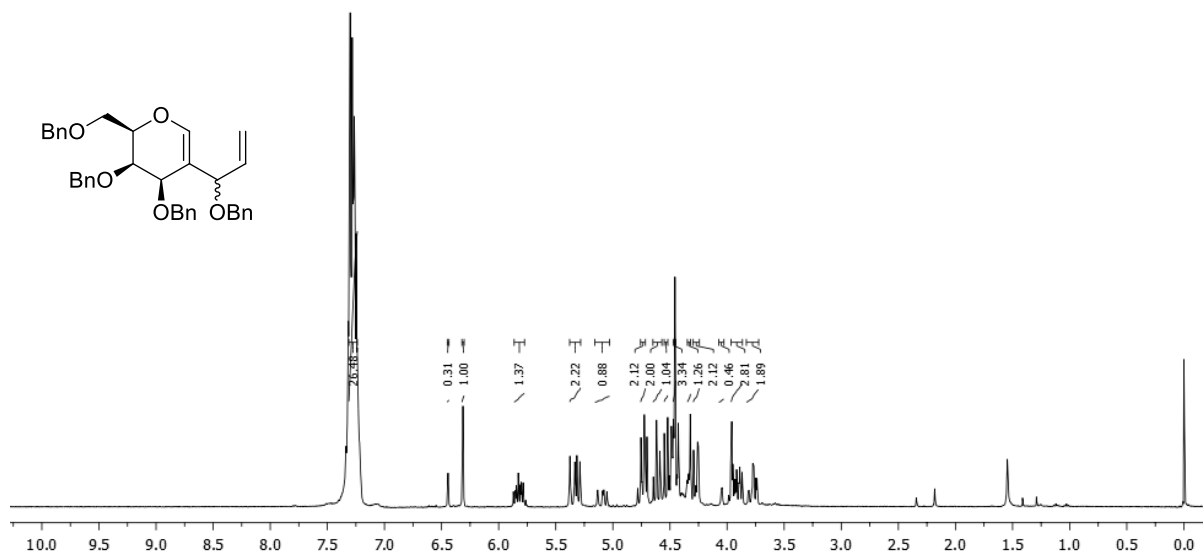
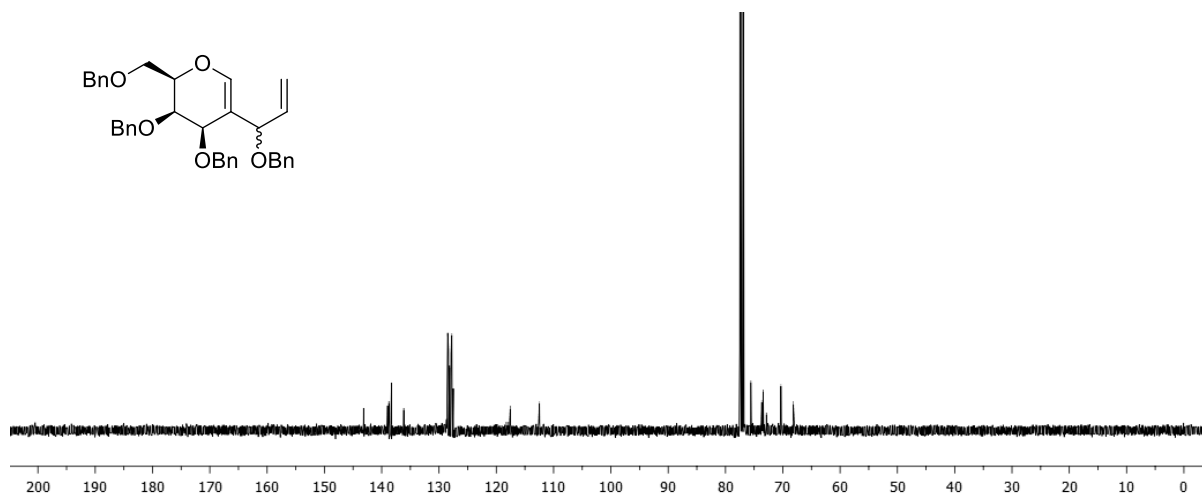
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Figures S 1. <sup>1</sup>H NMR spectrum of compound 17.Figures S 2. <sup>13</sup>C NMR spectrum of compound 17.



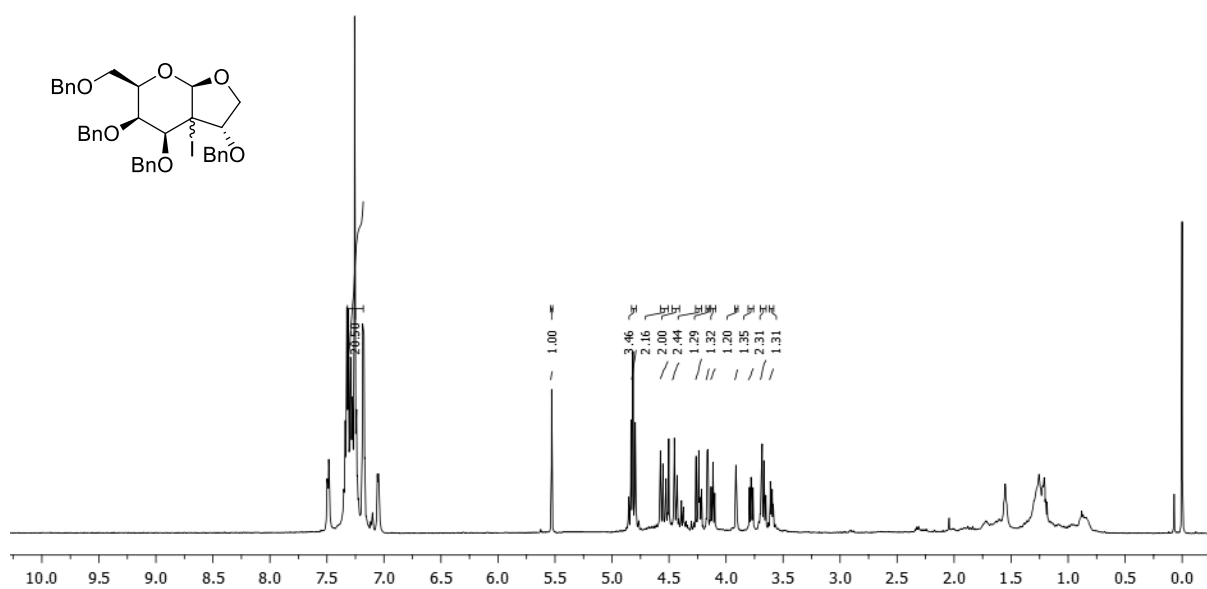


Figure S 5. <sup>1</sup>H NMR spectrum of compound 20a.

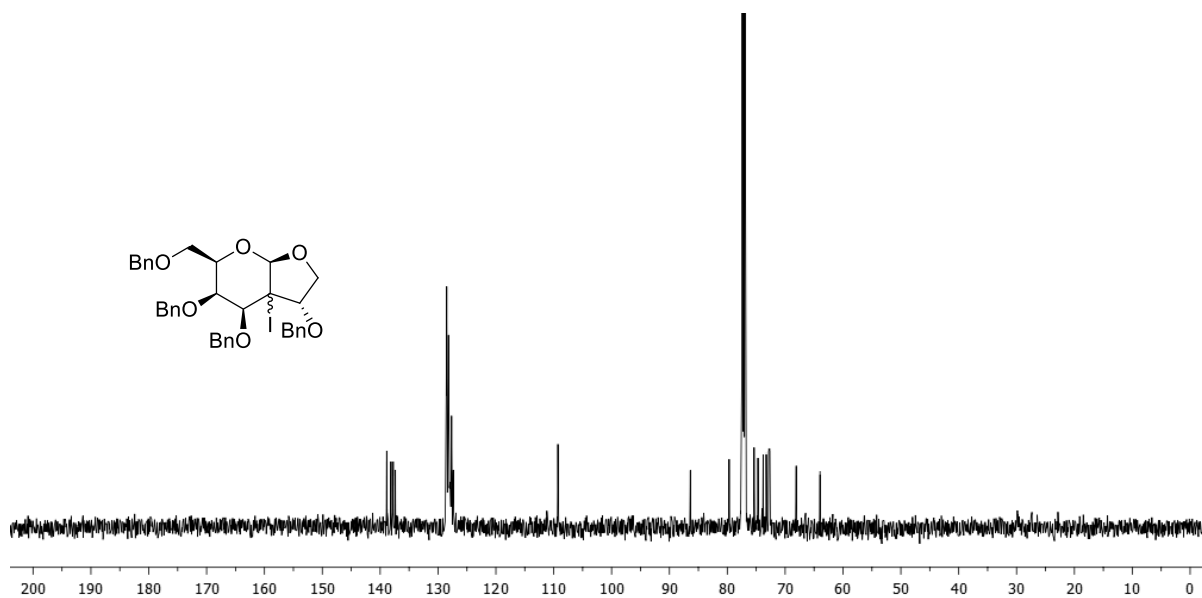
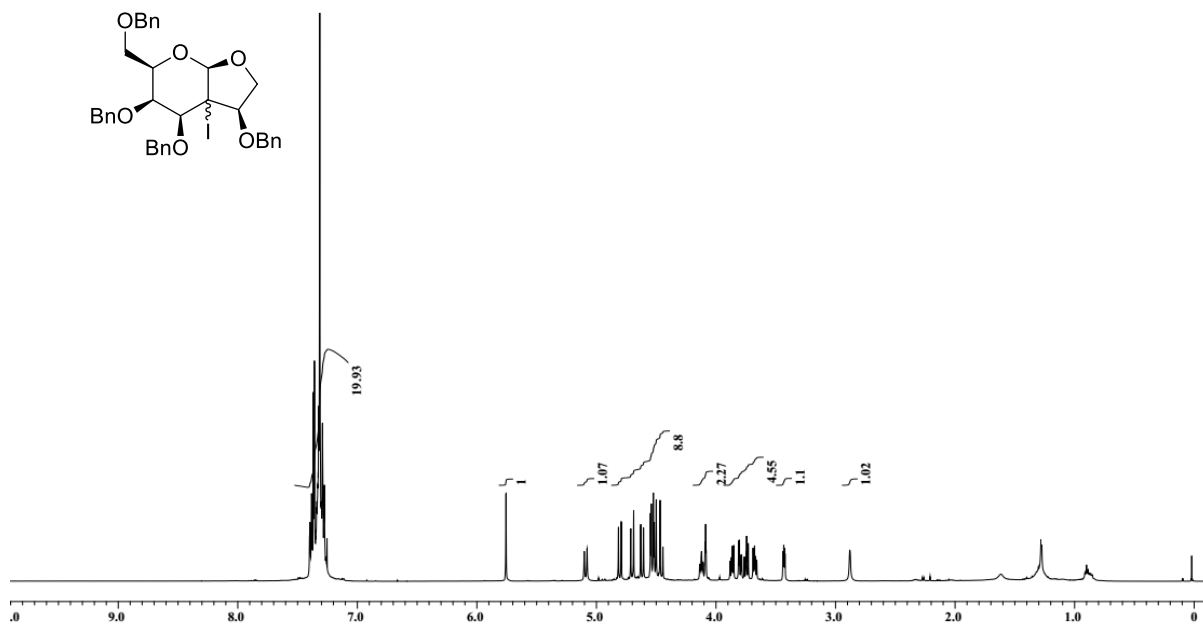
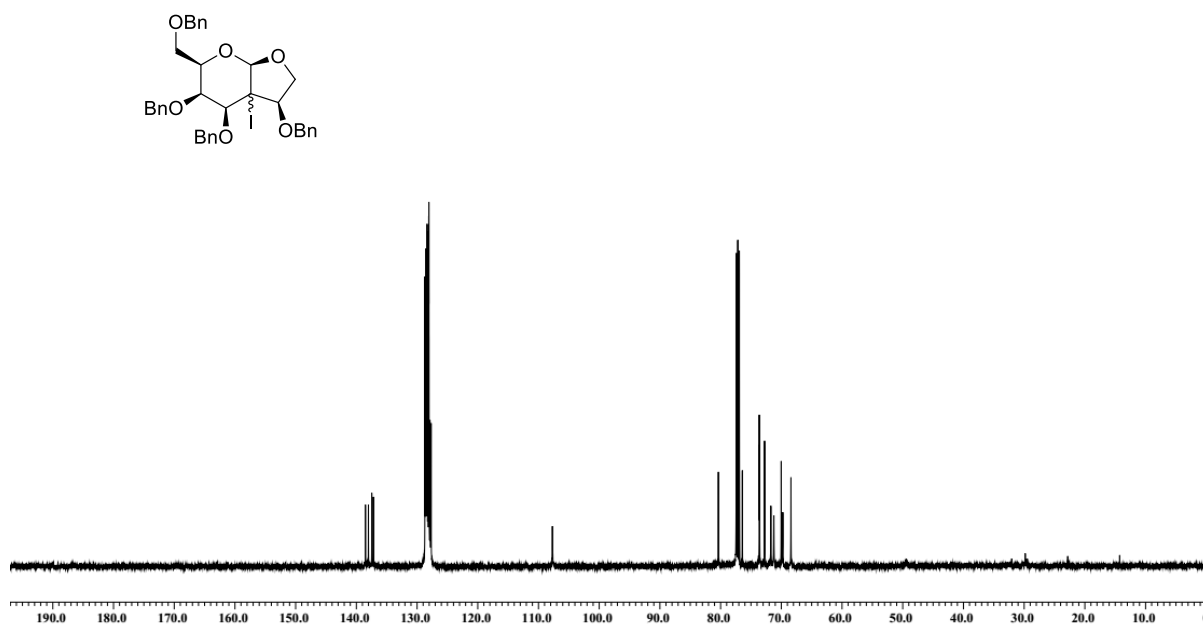
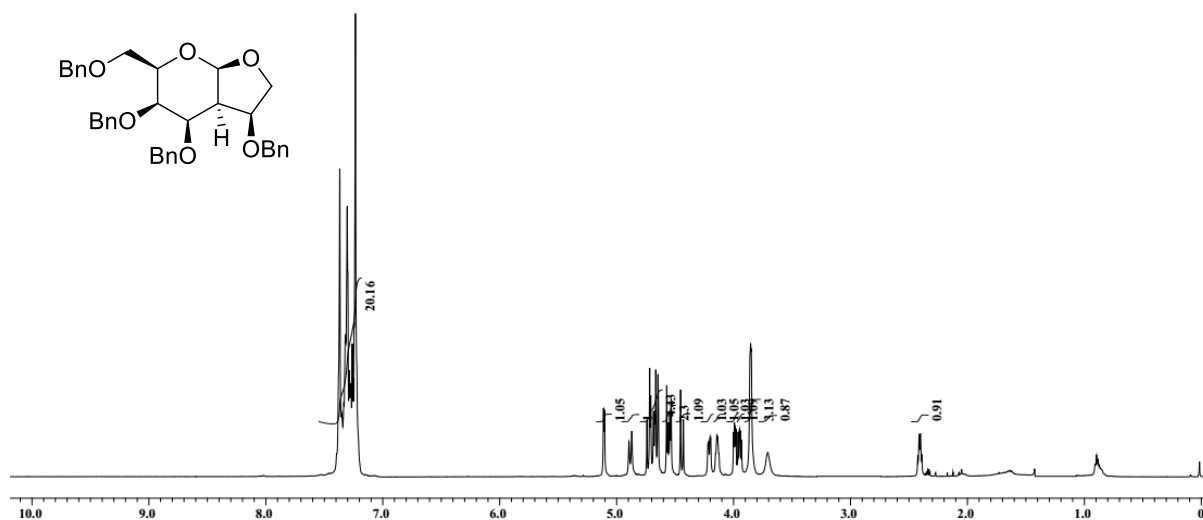
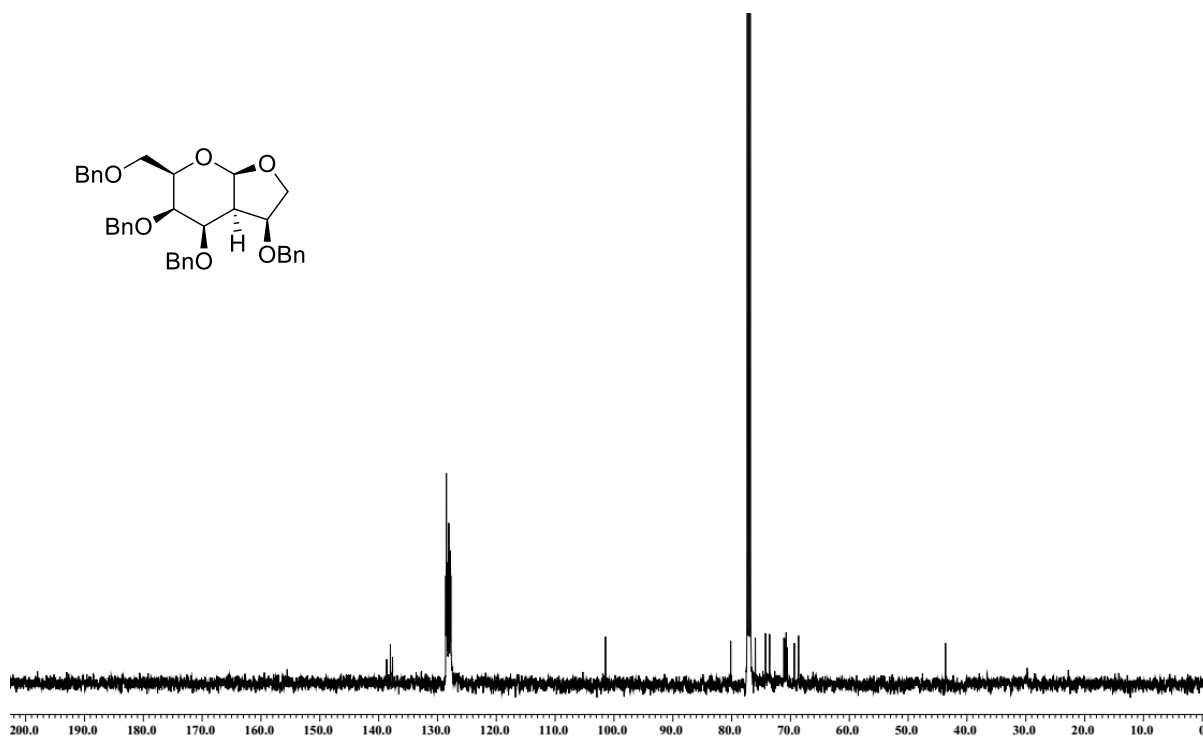


Figure S 6. <sup>13</sup>C NMR spectrum of compound 20a.

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.

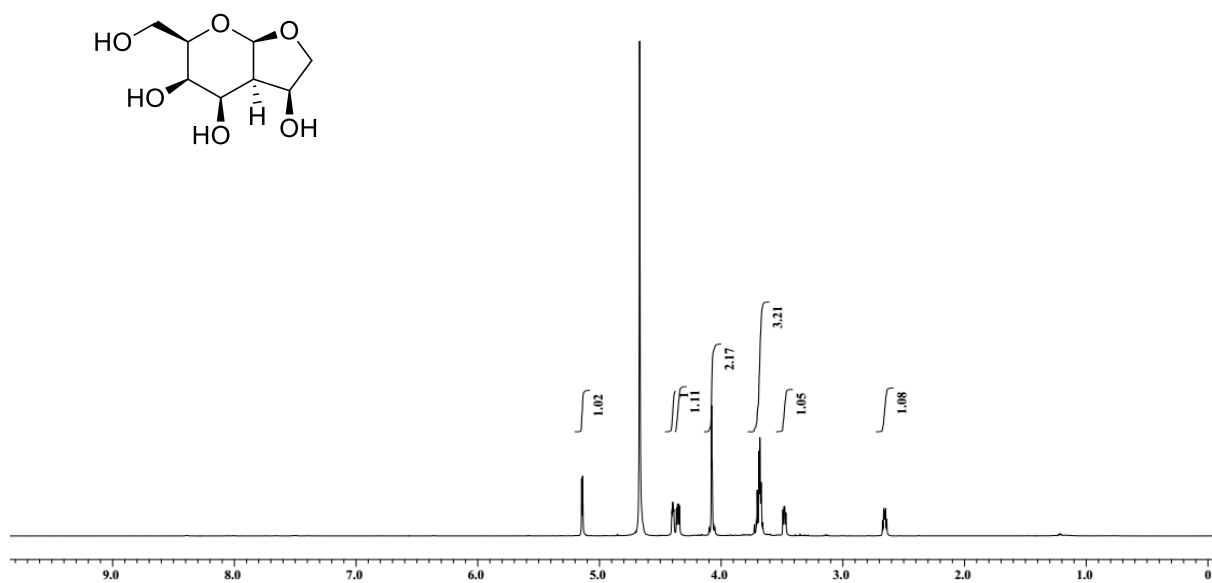


Figure S 11. <sup>1</sup>H NMR spectrum of compound 22.

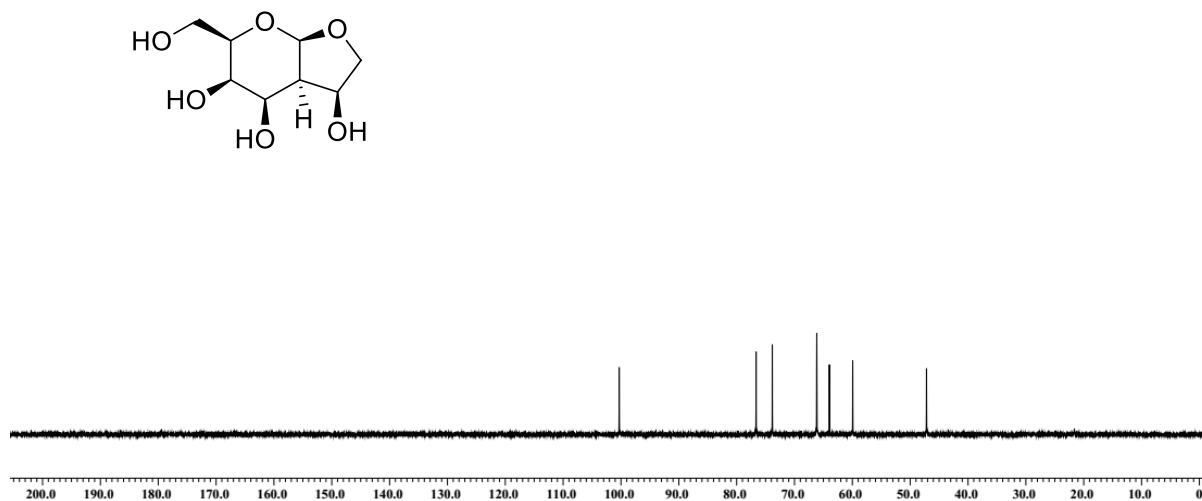


Figure S 12. <sup>13</sup>C NMR spectrum of compound 22.



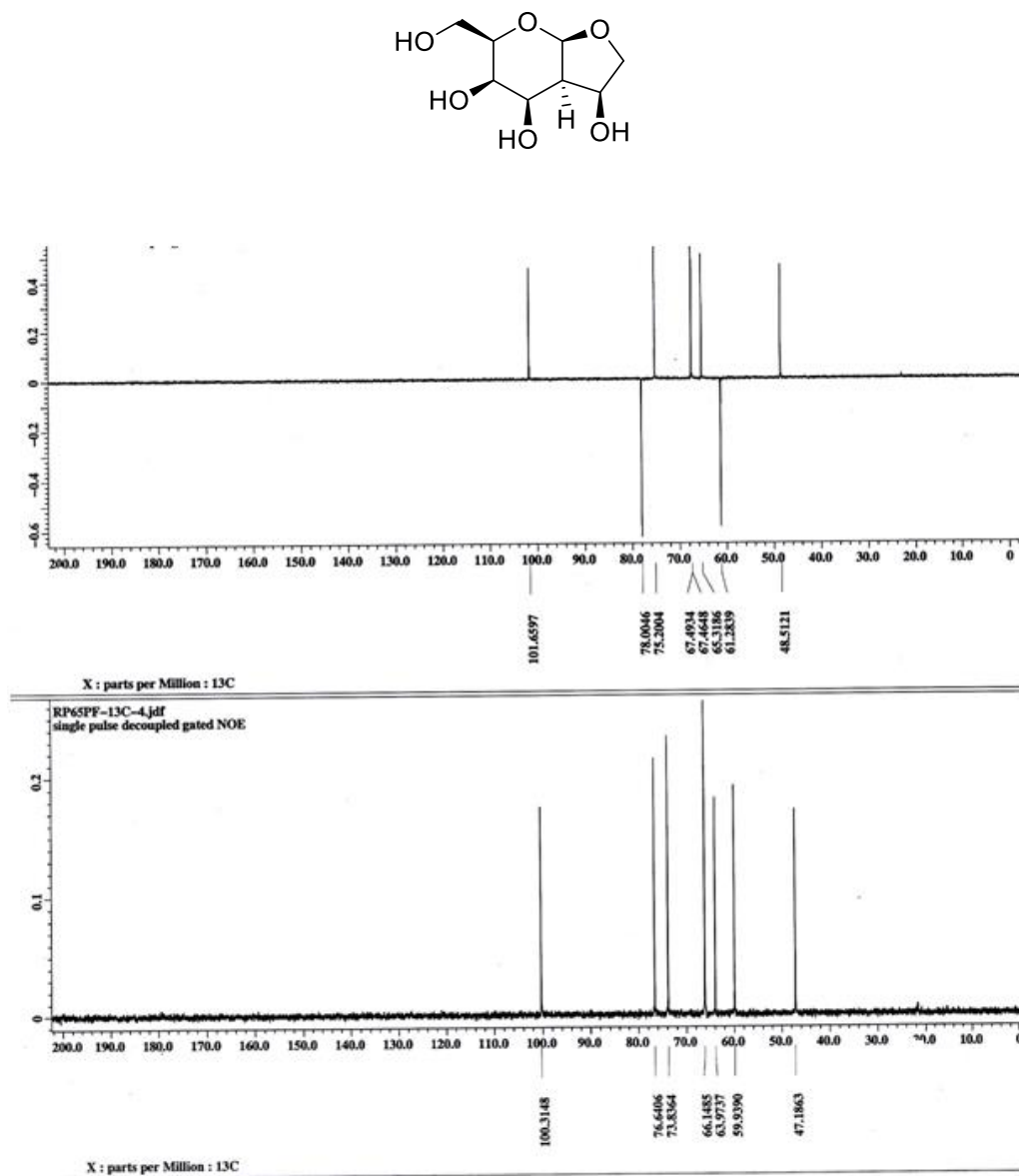


Figure S 13. Dept-135 spectrum of compound 22.

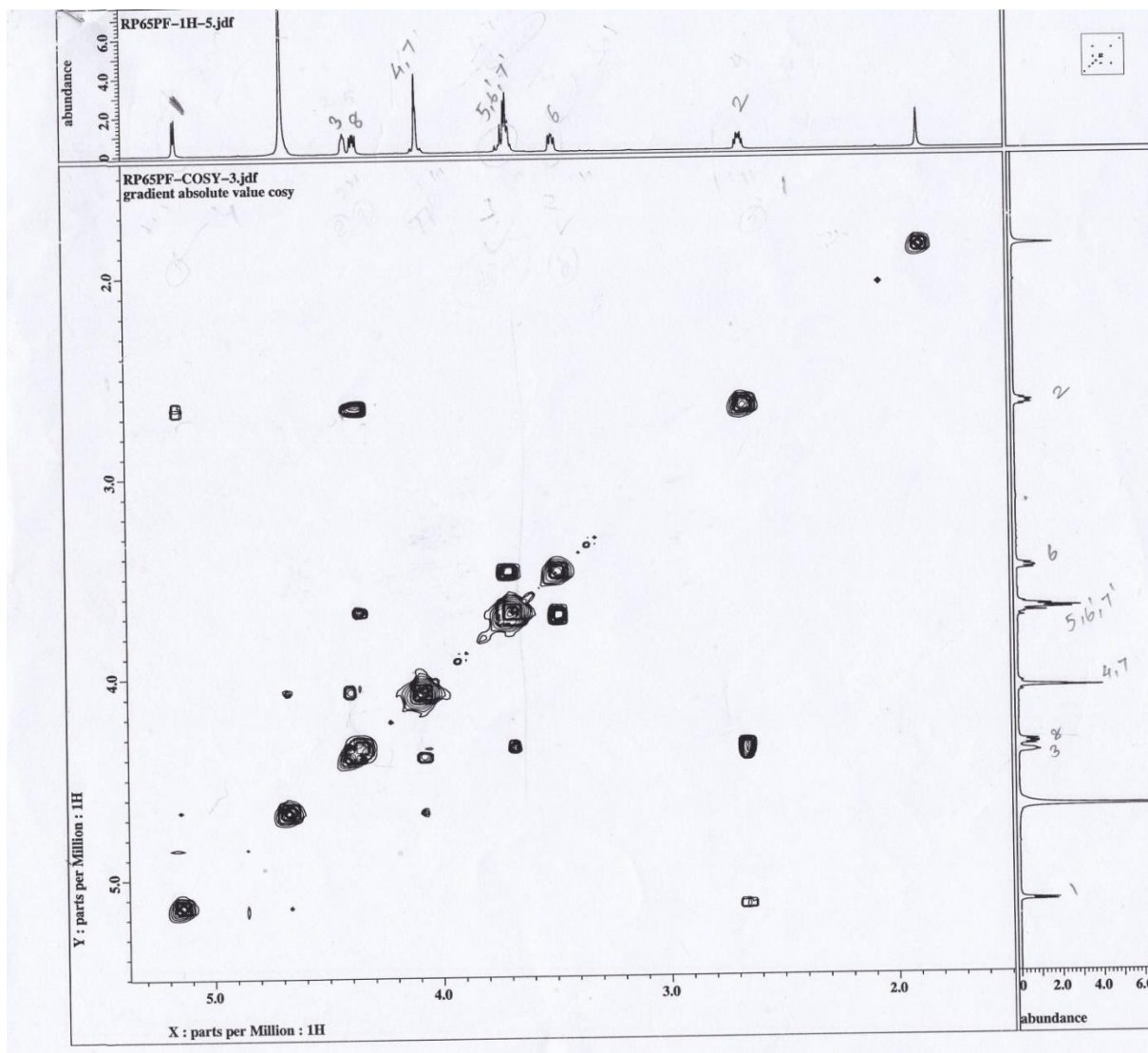
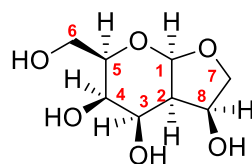


Figure S 14.  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of compound 22.



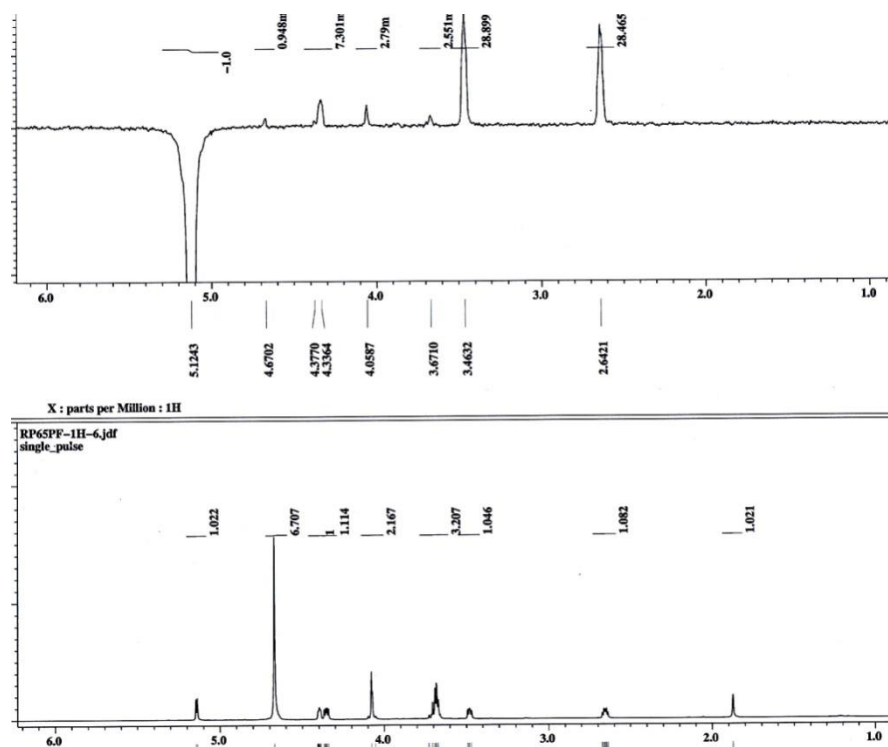


Figure S 15. nOe spectrum of compound 22 for irradiation of H-1.

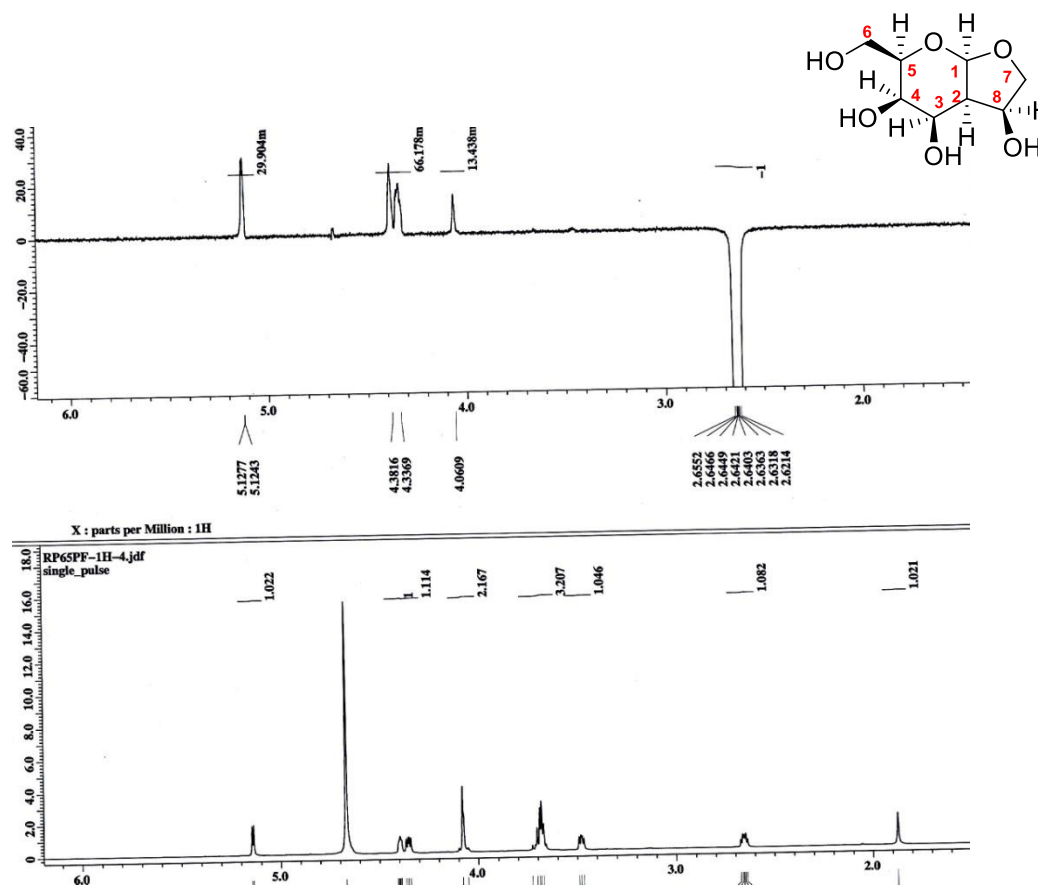


Figure S 16. nOe spectrum of compound 22 for irradiation of H-2.

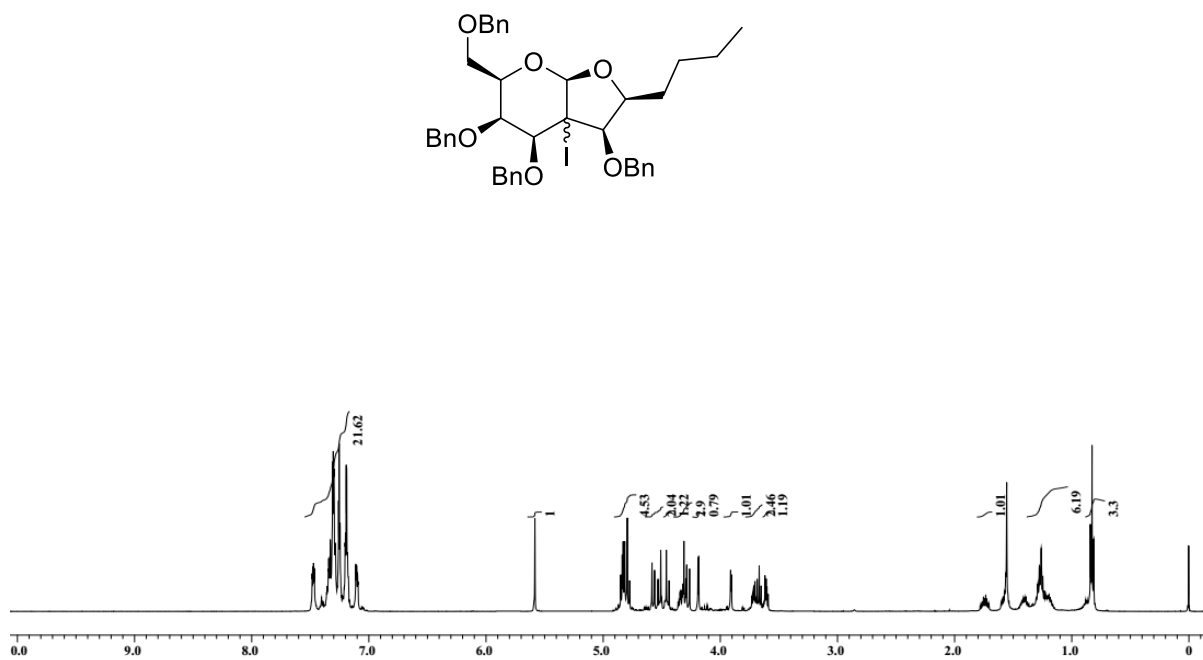


Figure S 17.  $^1\text{H}$  NMR spectrum of compound 23.

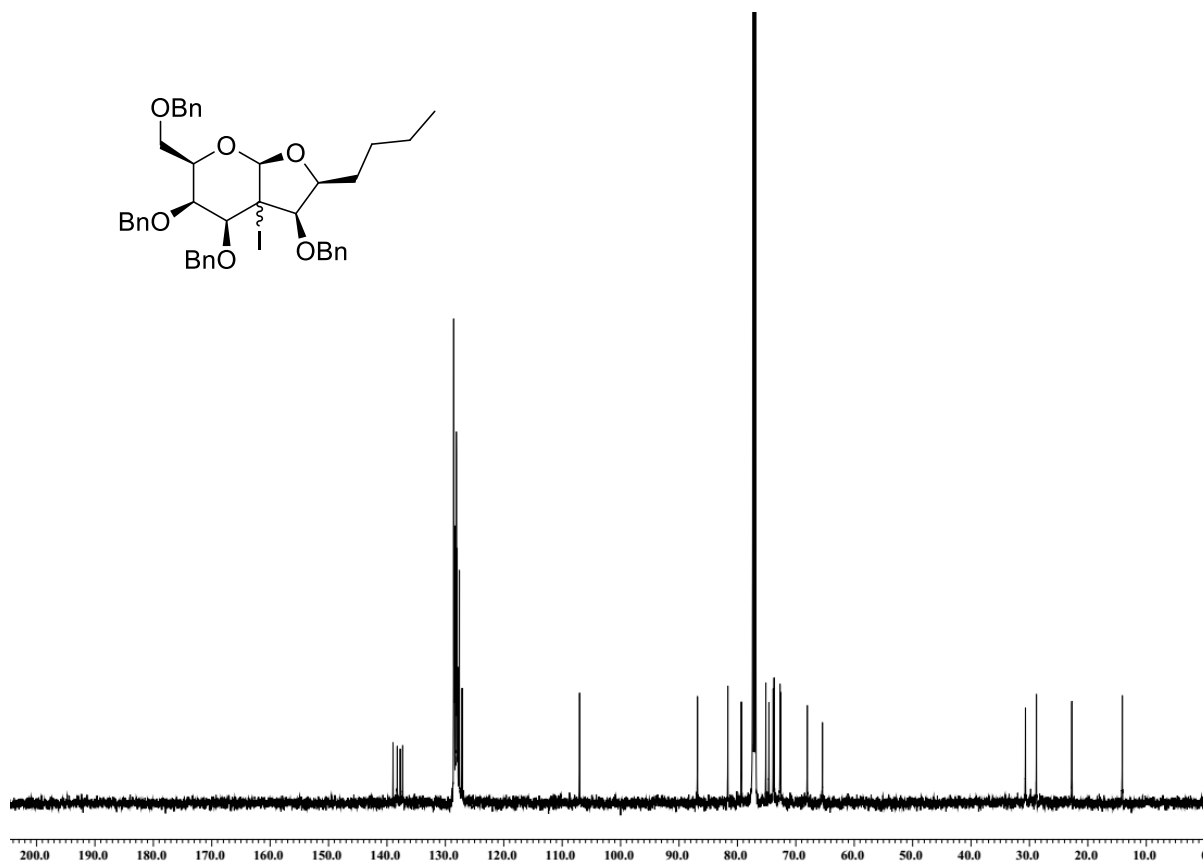
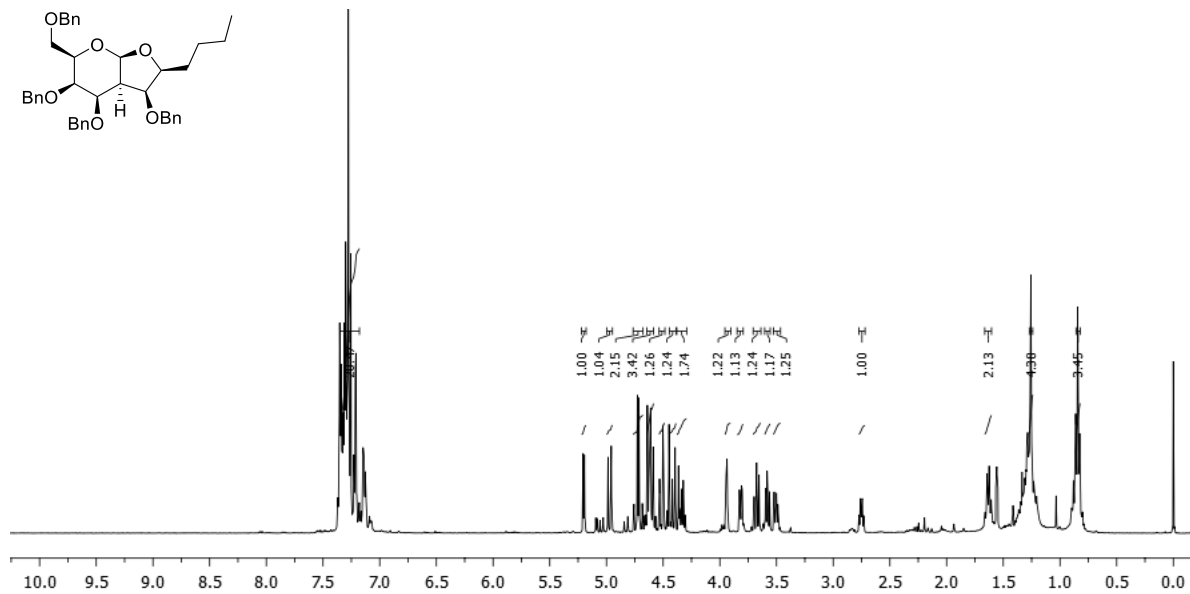
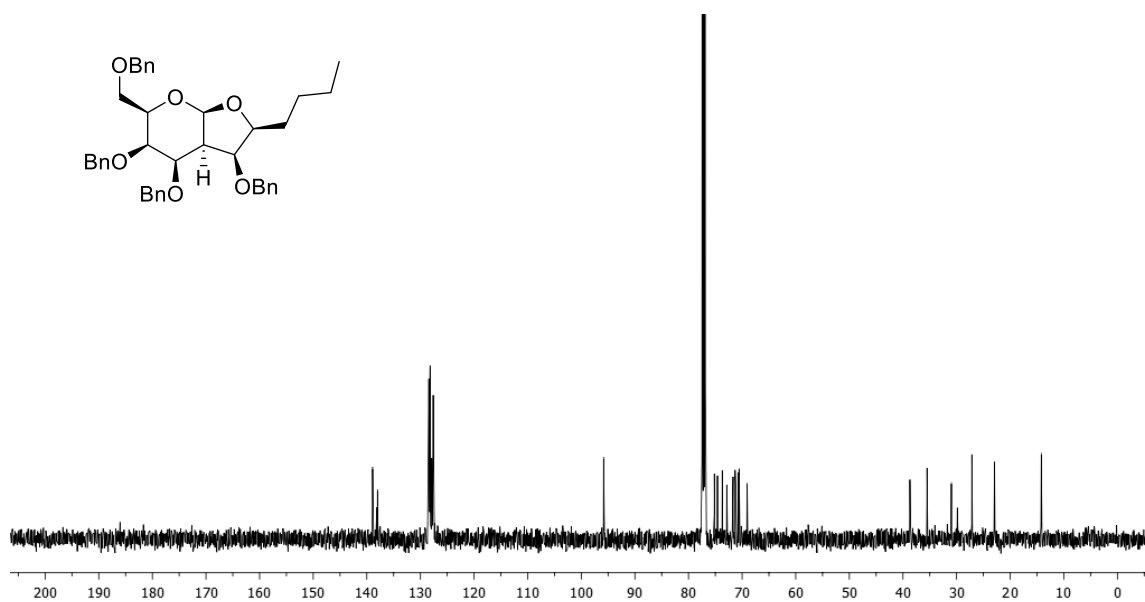
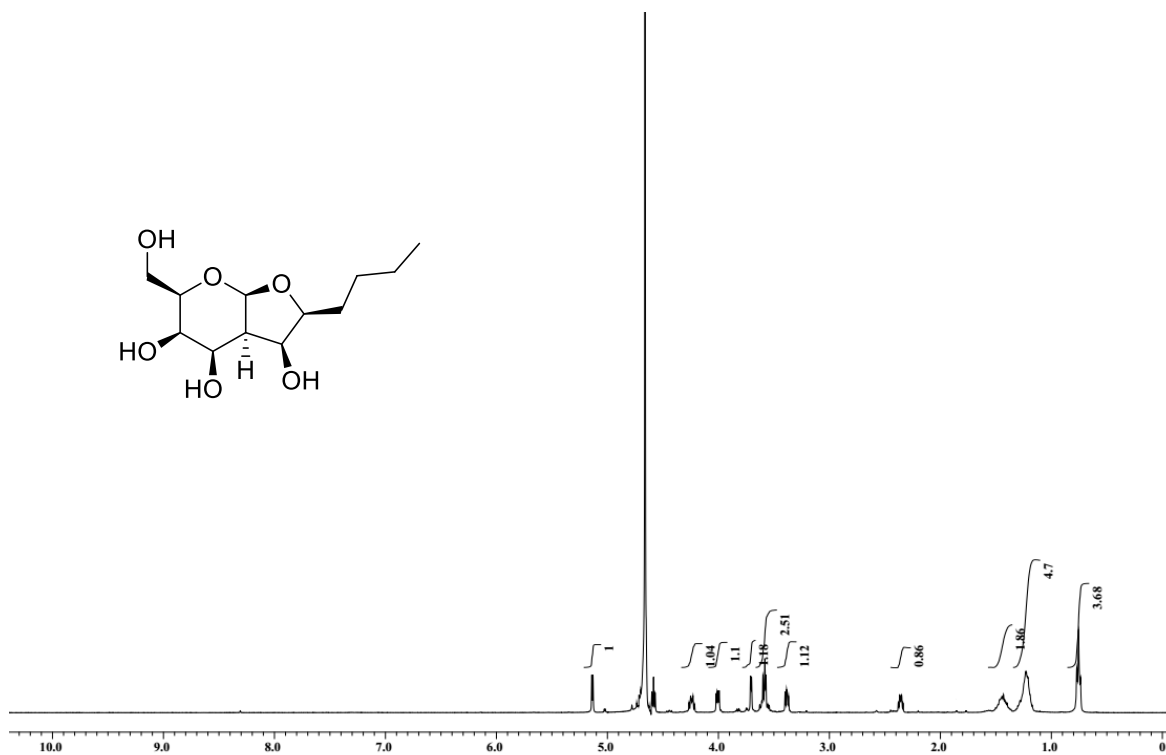
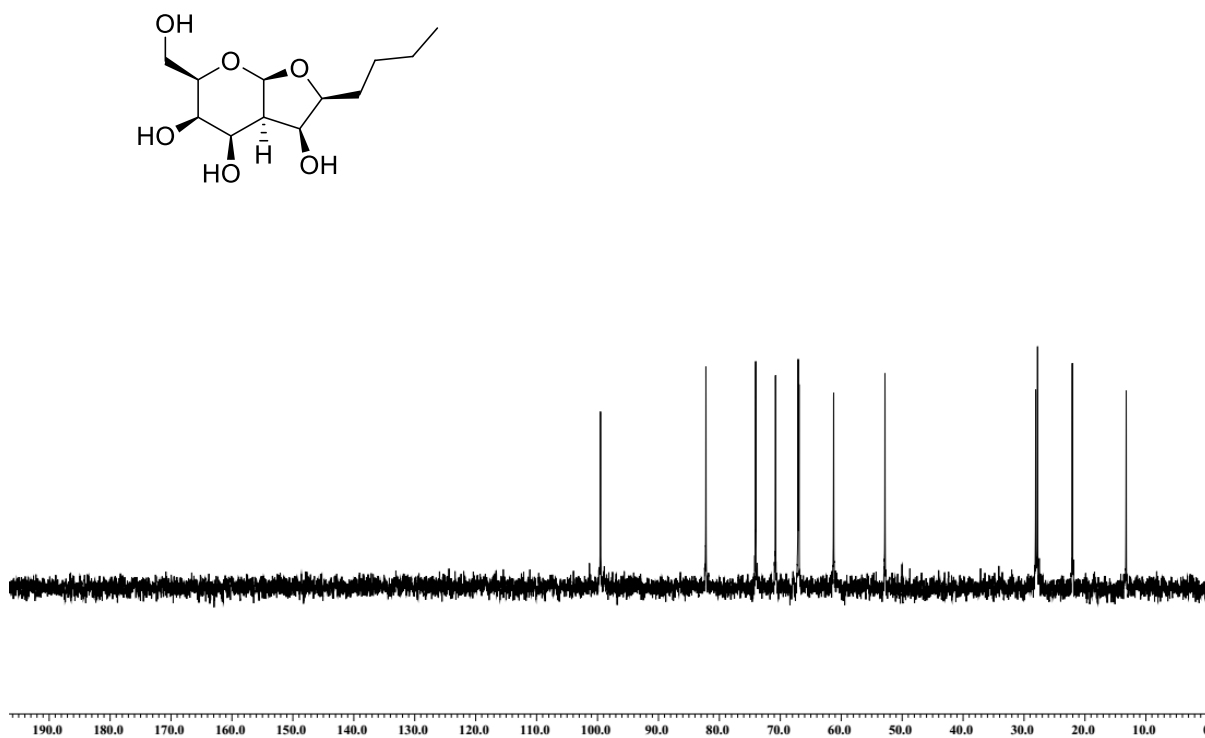
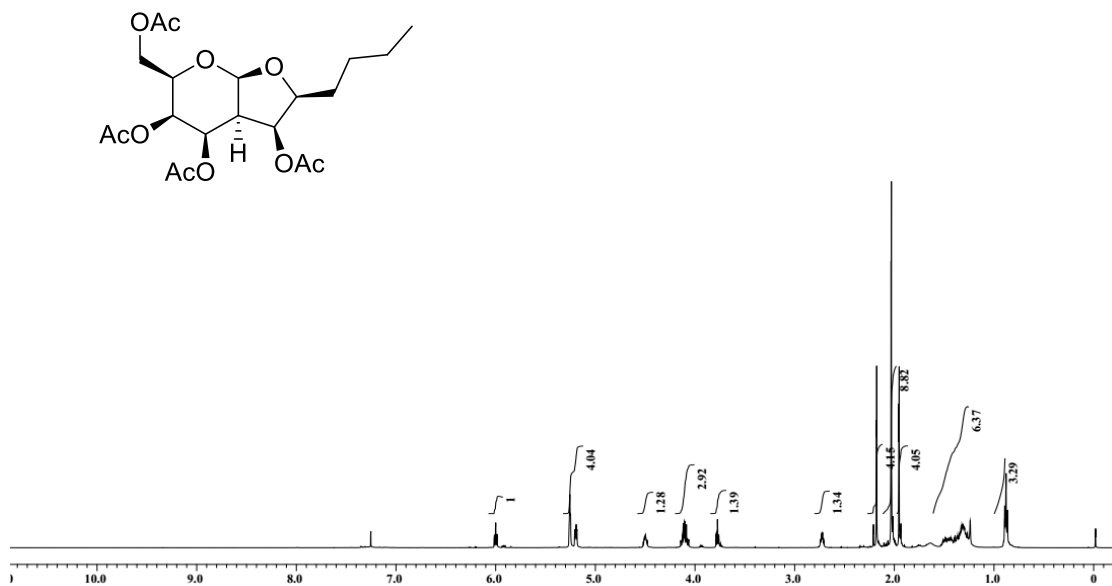
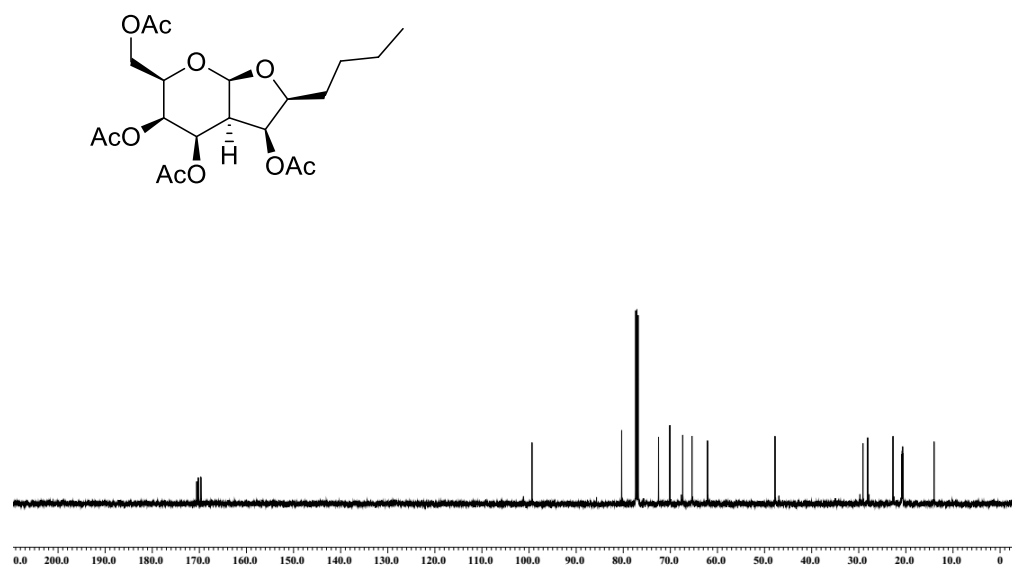
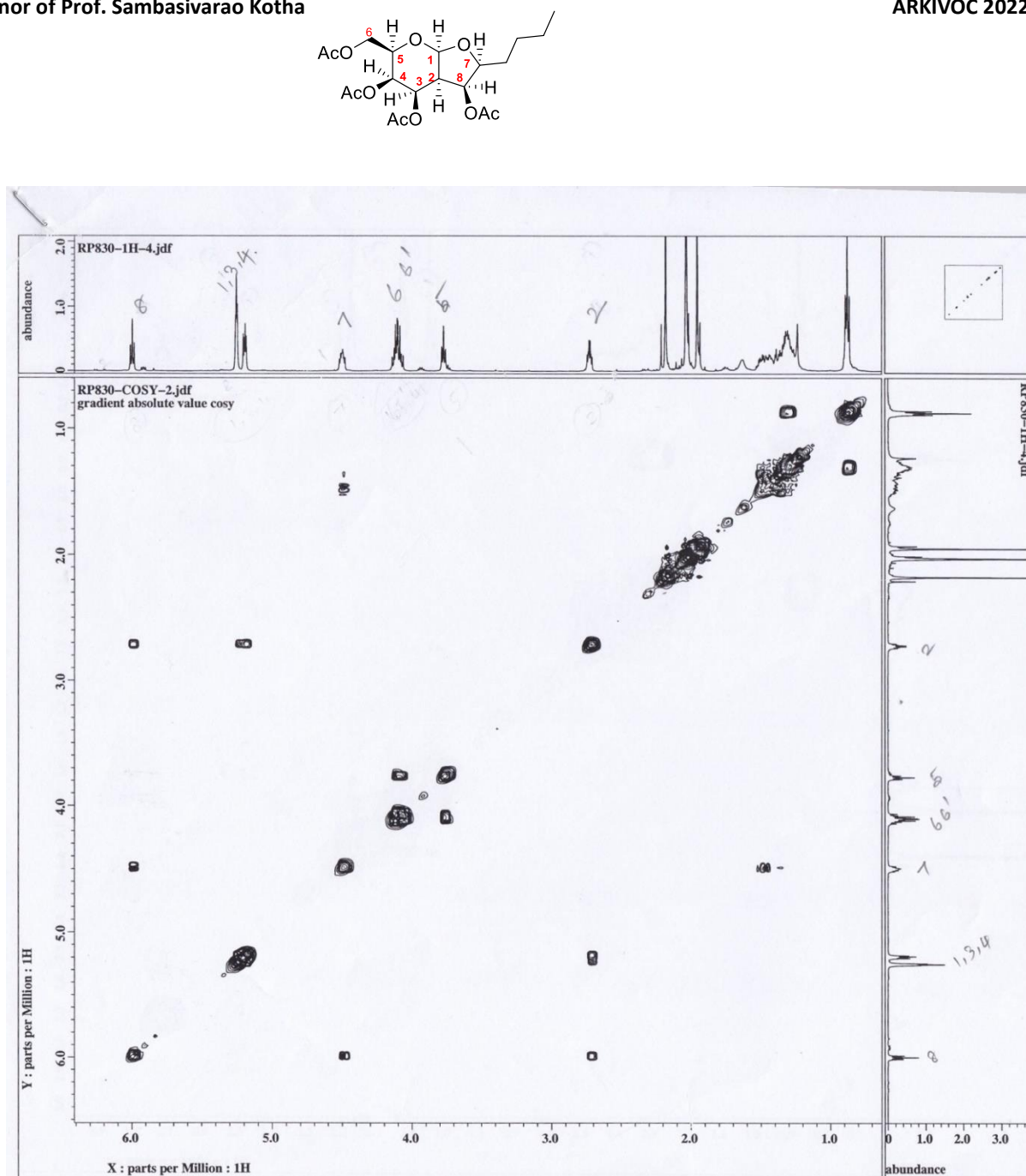


Figure S 18.  $^{13}\text{C}$  NMR spectrum of compound 23.

Figure S 19. <sup>1</sup>H NMR spectrum of compound 24.Figure S 20. <sup>13</sup>C NMR spectrum of compound 24.

Figure S 21. <sup>1</sup>H NMR spectrum of compound 25.Figure S 22. <sup>13</sup>C NMR spectrum of compound 25.

Figure S 23. <sup>1</sup>H NMR spectrum of compound 26.Figure S 24. <sup>13</sup>C NMR spectrum of compound 26.

Figure S 25.  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of compound 26.



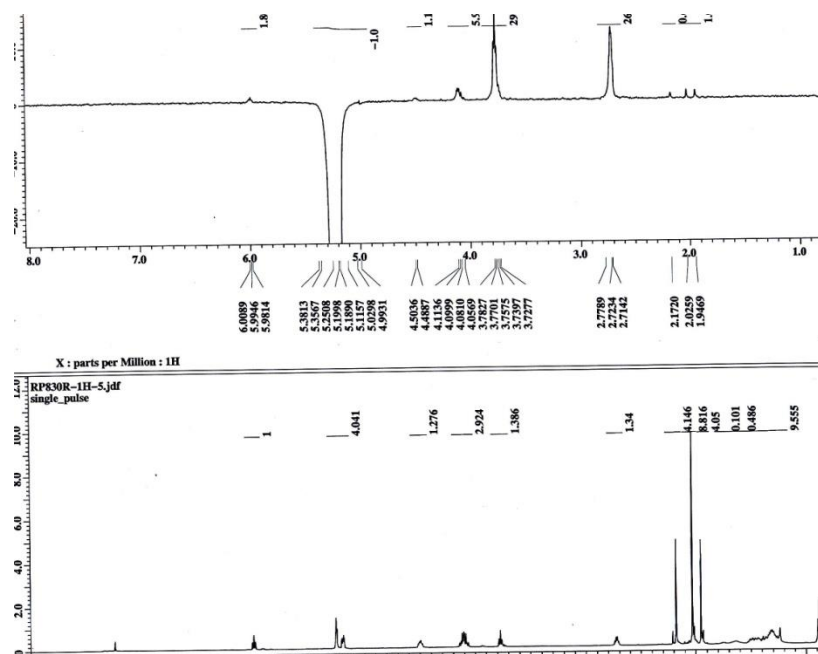


Figure S 26. nOe spectrum of compound 26 for irradiation of H-1.



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

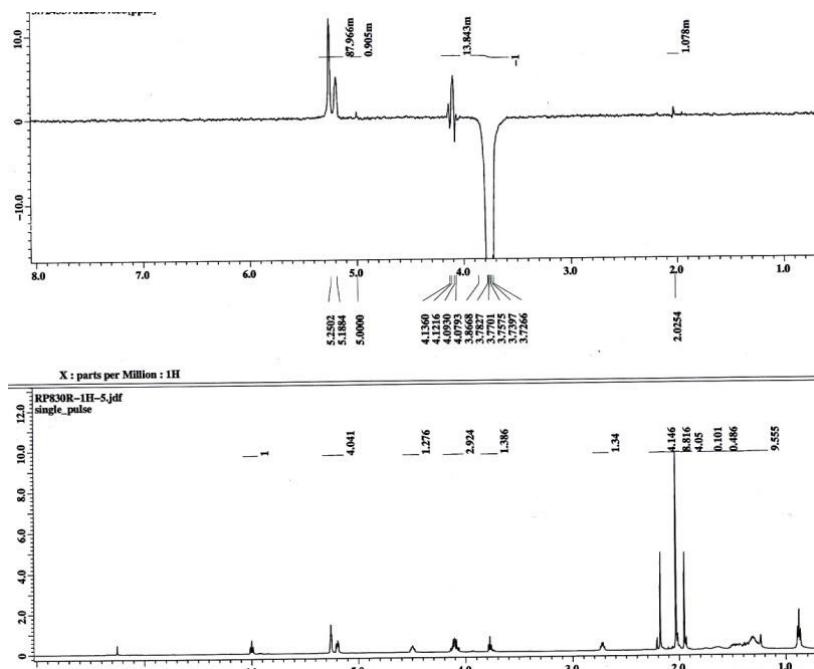


Figure S 28. nOe spectrum of compound 26 for irradiation of H-5.

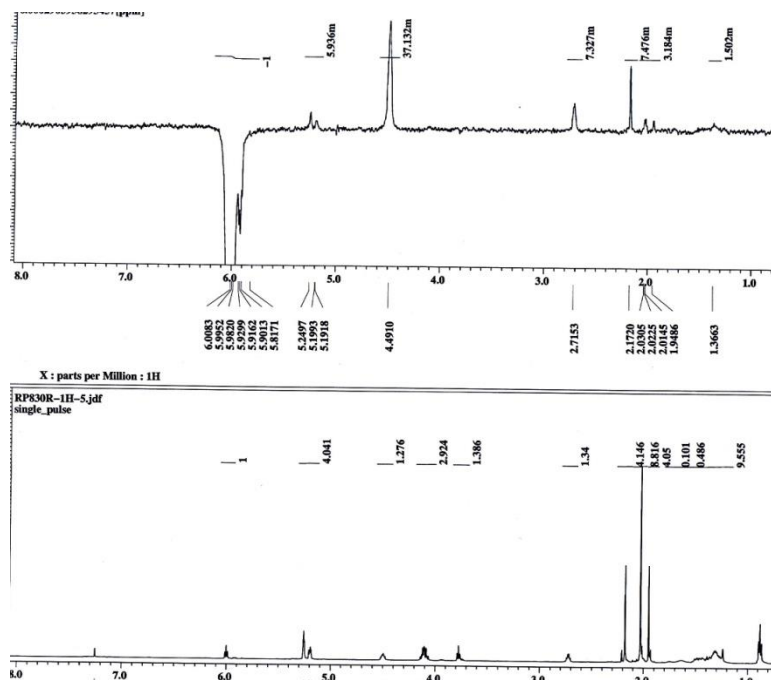
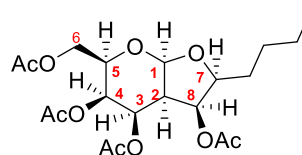
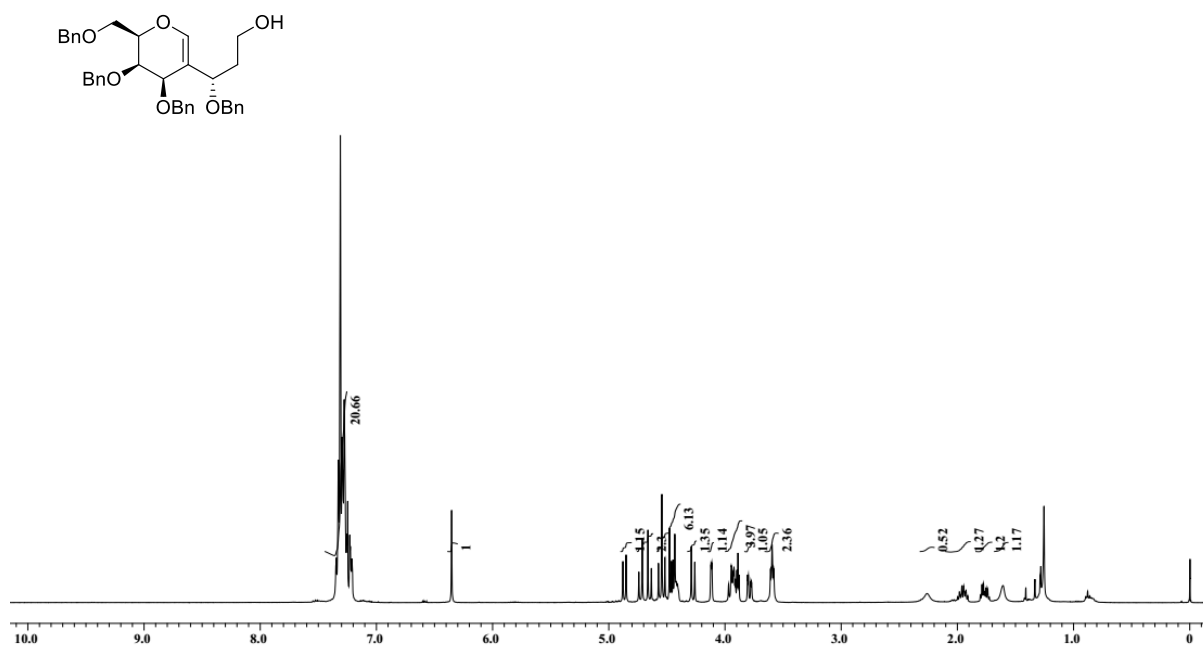
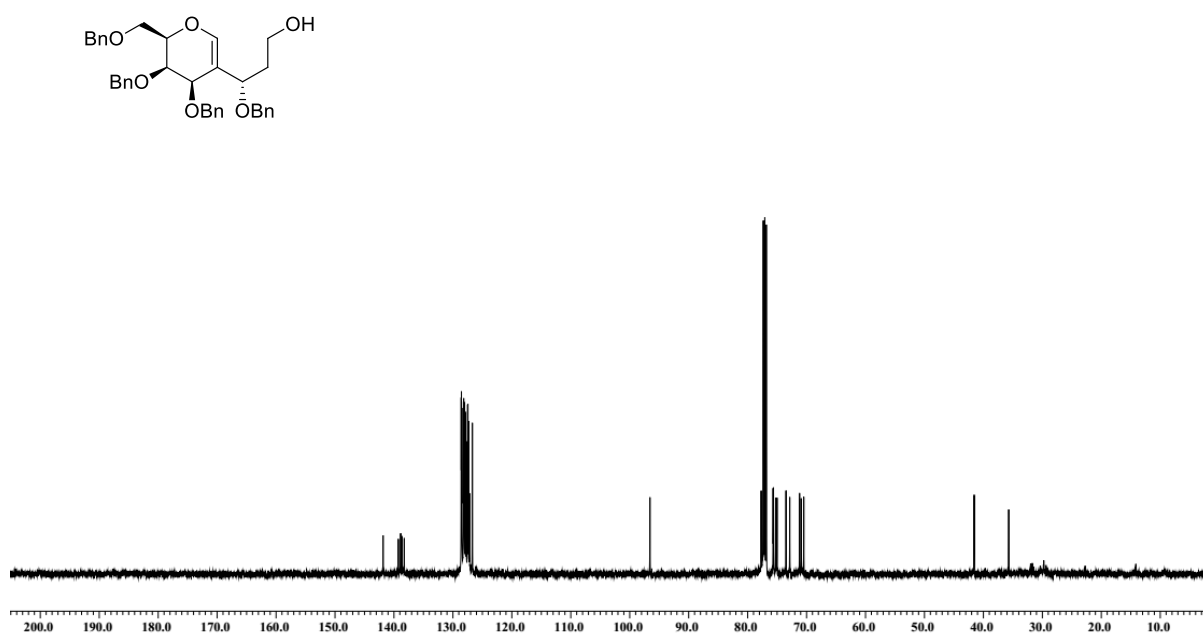
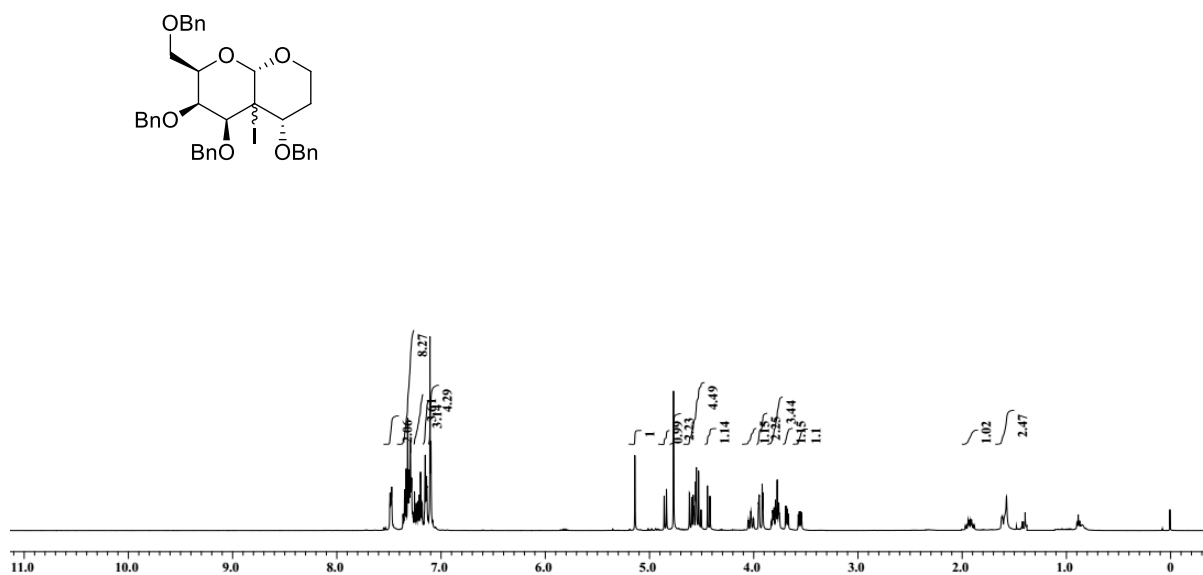
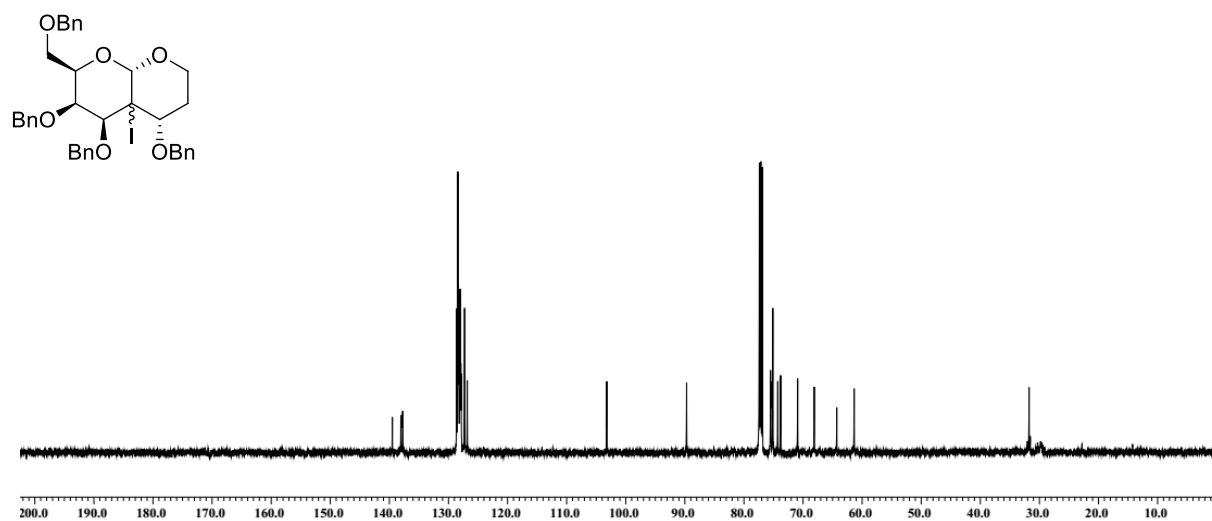
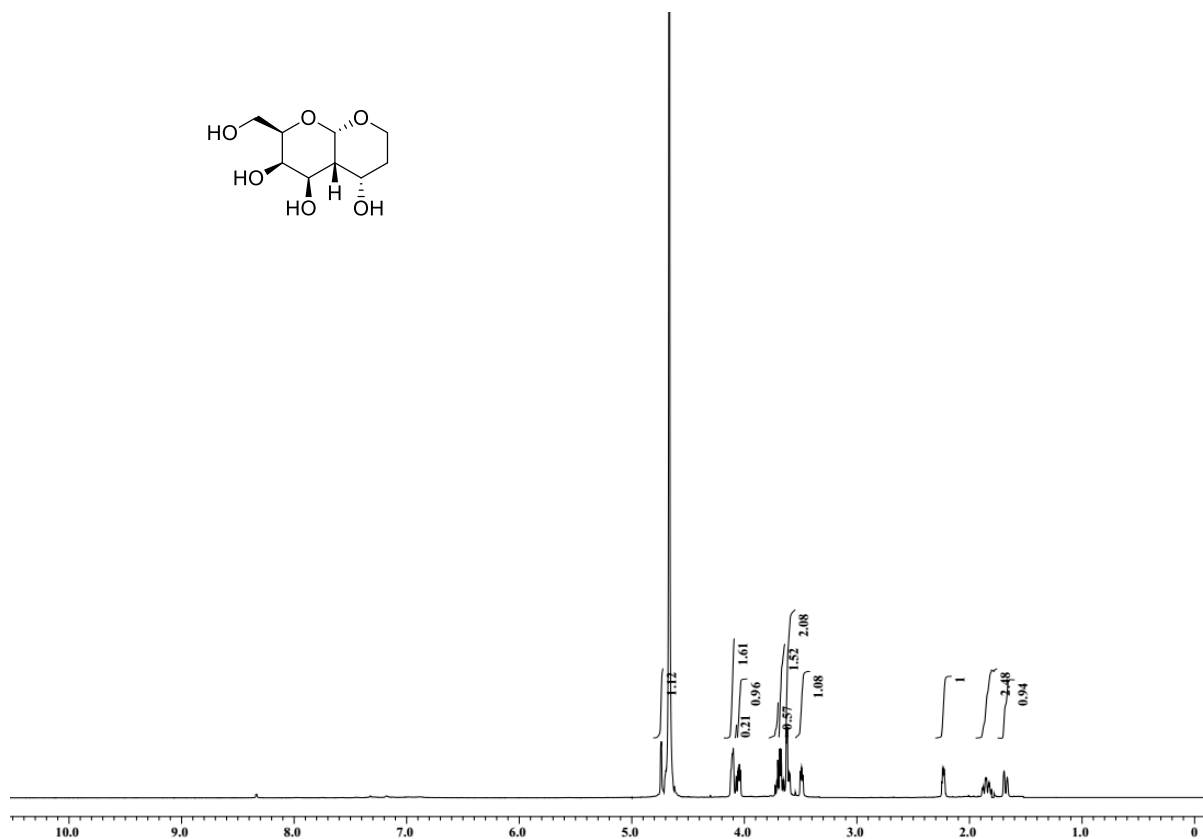
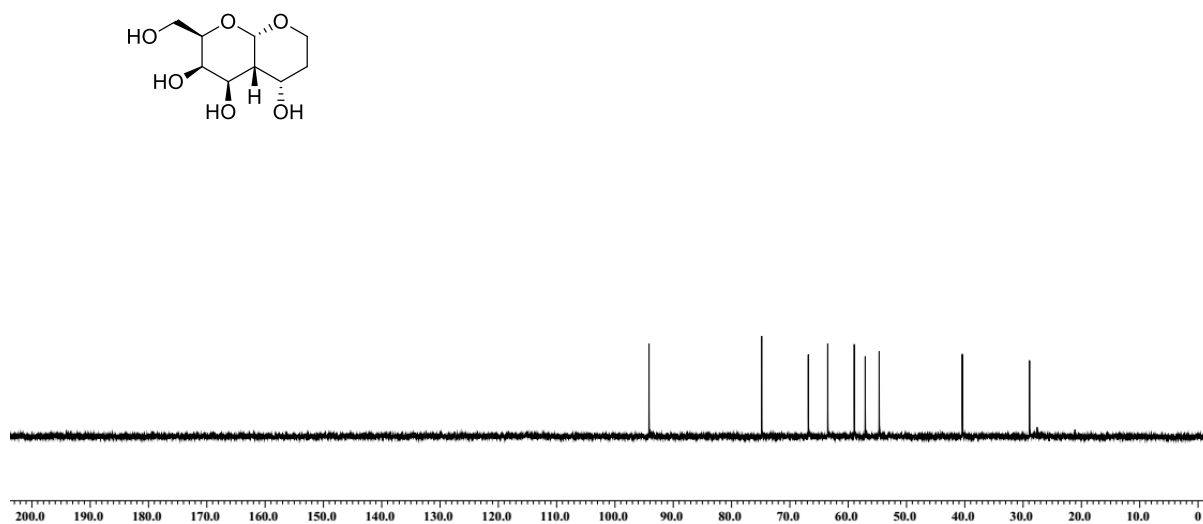


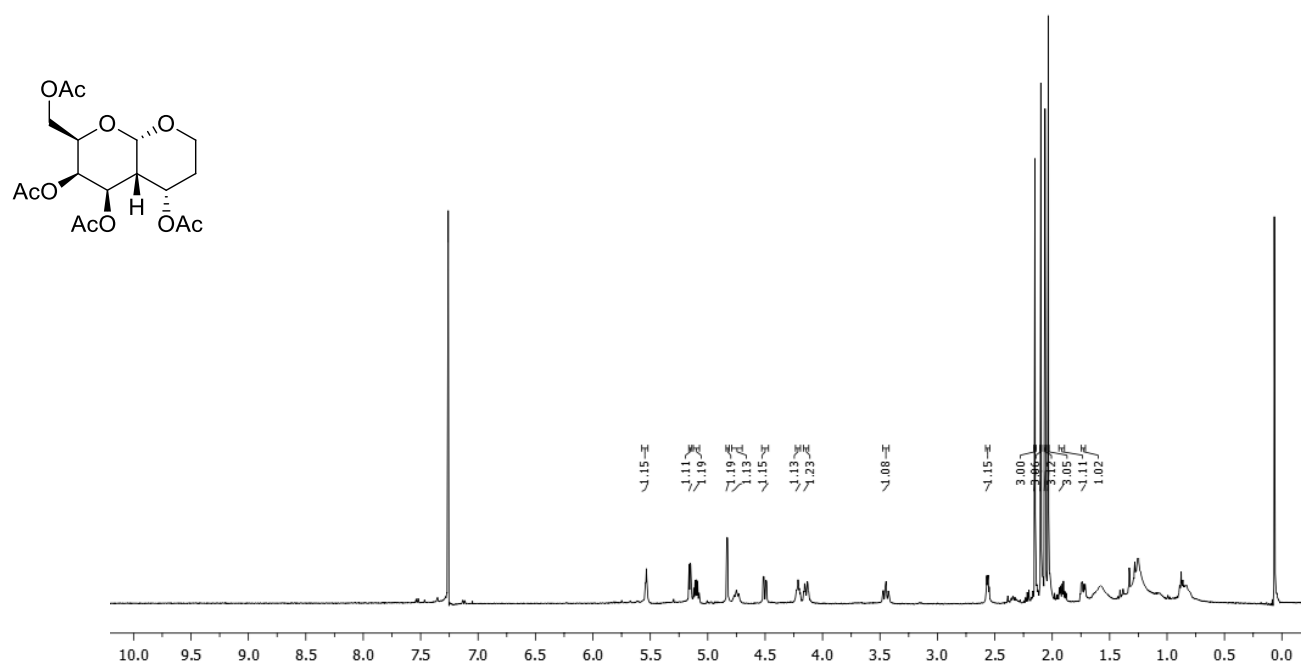
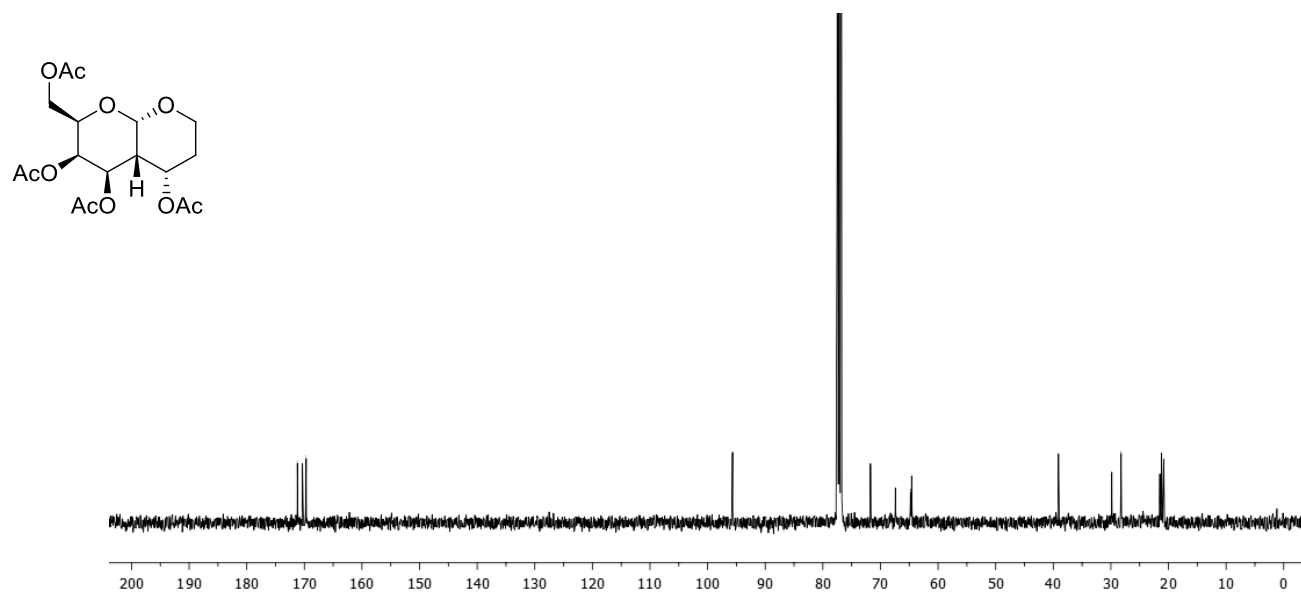
Figure S 29. nOe spectrum of compound 26 for irradiation of H-8.



Figure S 32. <sup>1</sup>H NMR spectrum of compound 28a.Figure S 33. <sup>13</sup>C NMR spectrum of compound 28a.

Figure S 34. <sup>1</sup>H NMR spectrum of compound 29.Figure S 35. <sup>13</sup>C NMR spectrum of compound 29.

Figure S 36. <sup>1</sup>H NMR spectrum of compound 31.Figure S 37. <sup>13</sup>C NMR spectrum of compound 31.

Figure S 38. <sup>1</sup>H NMR spectrum of compound 32.Figure S 39. <sup>13</sup>C NMR spectrum of compound 32.

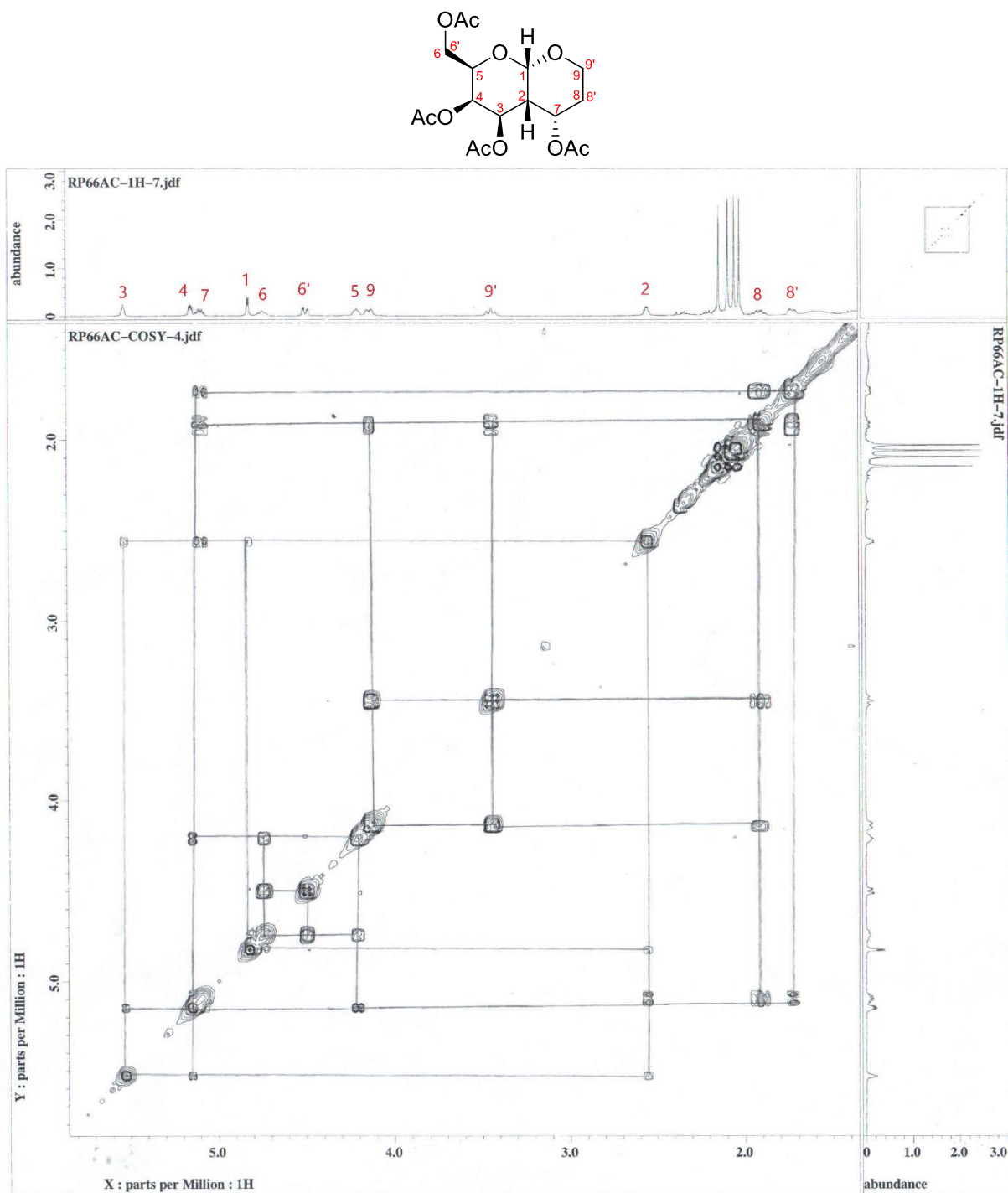


Figure S 40.  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of compound 32.



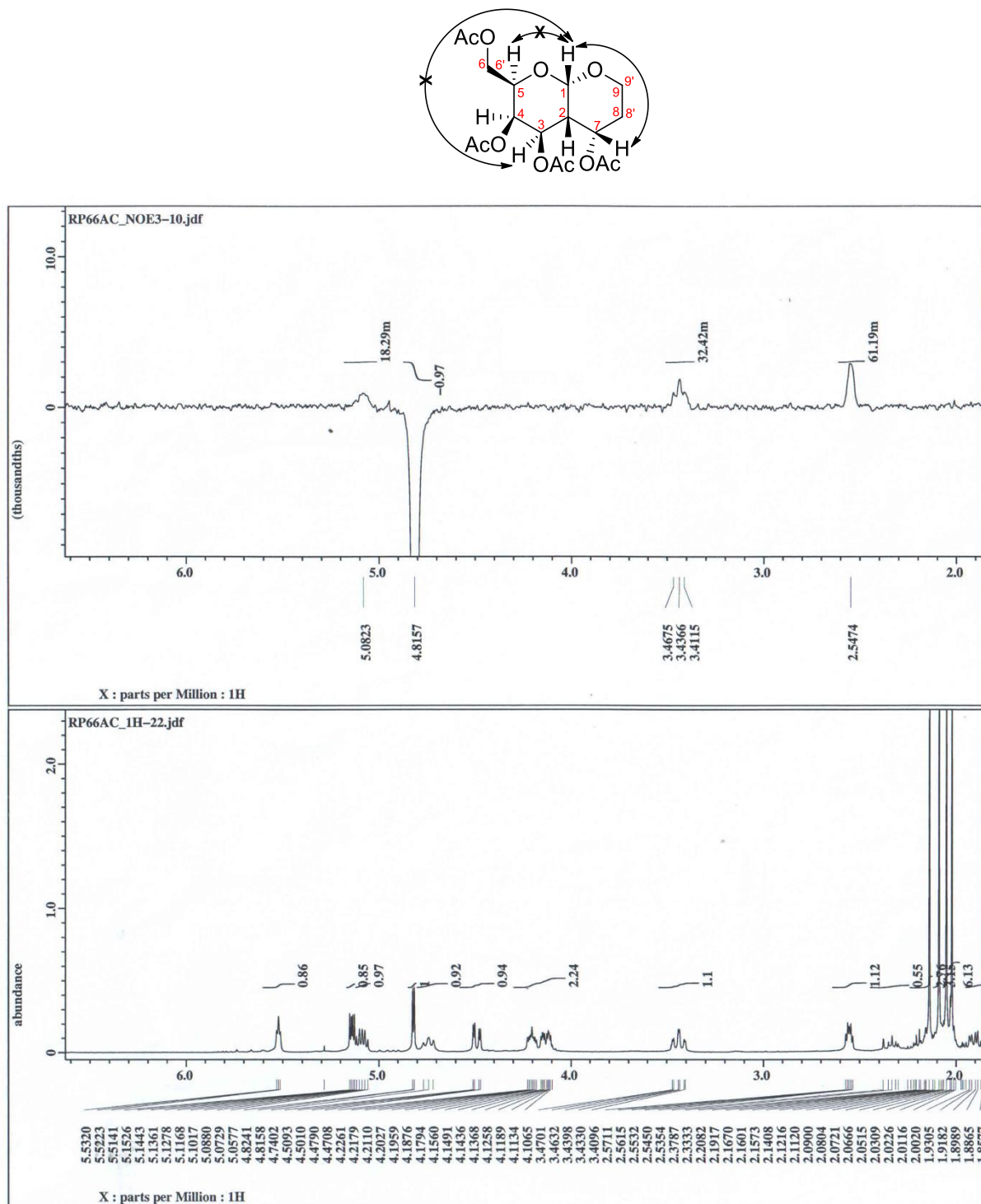


Figure S 41. nOe spectrum of compound 32 for irradiation of H-1.

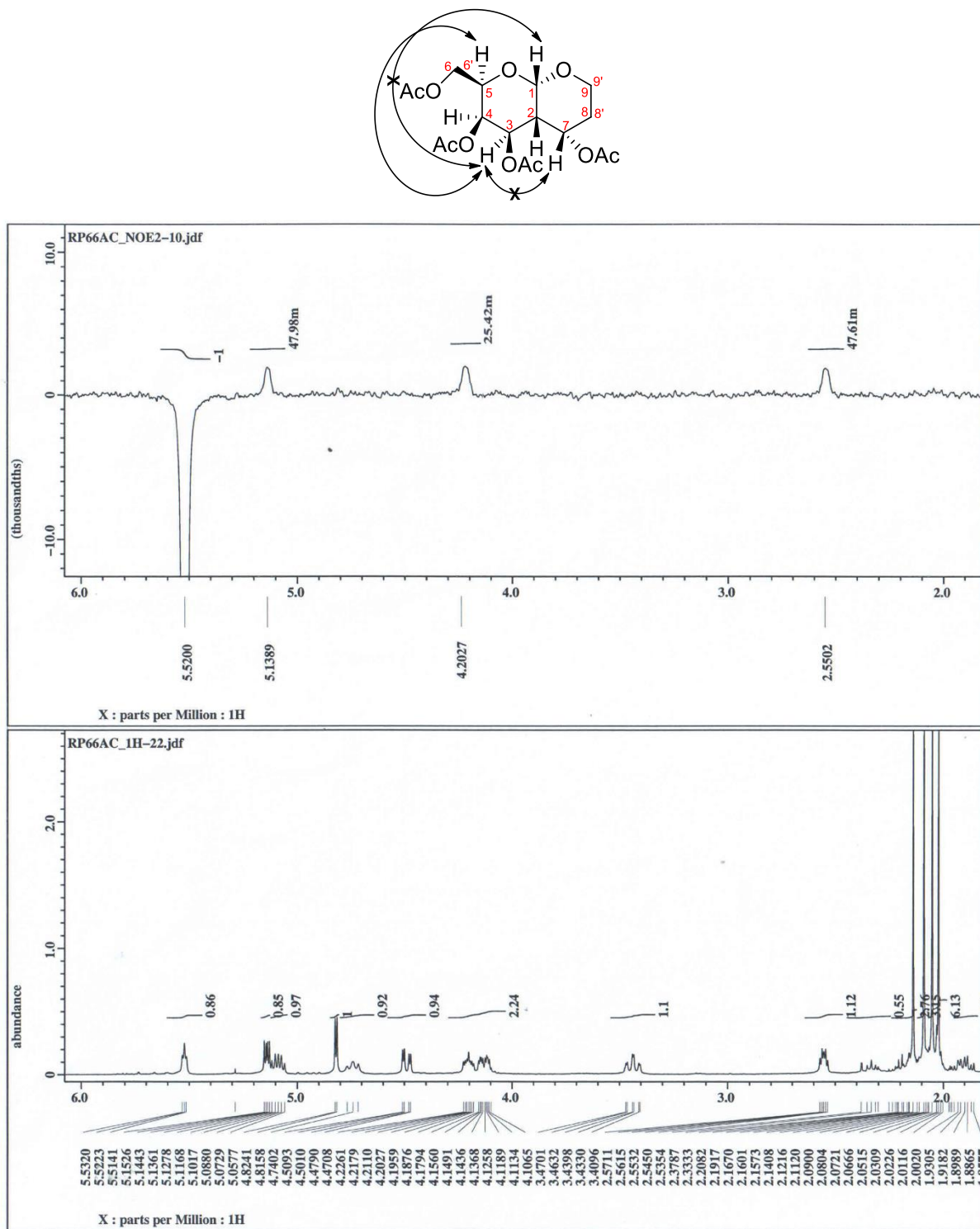


Figure S 42. nOe spectrum of compound 32 for irradiation of H-3.

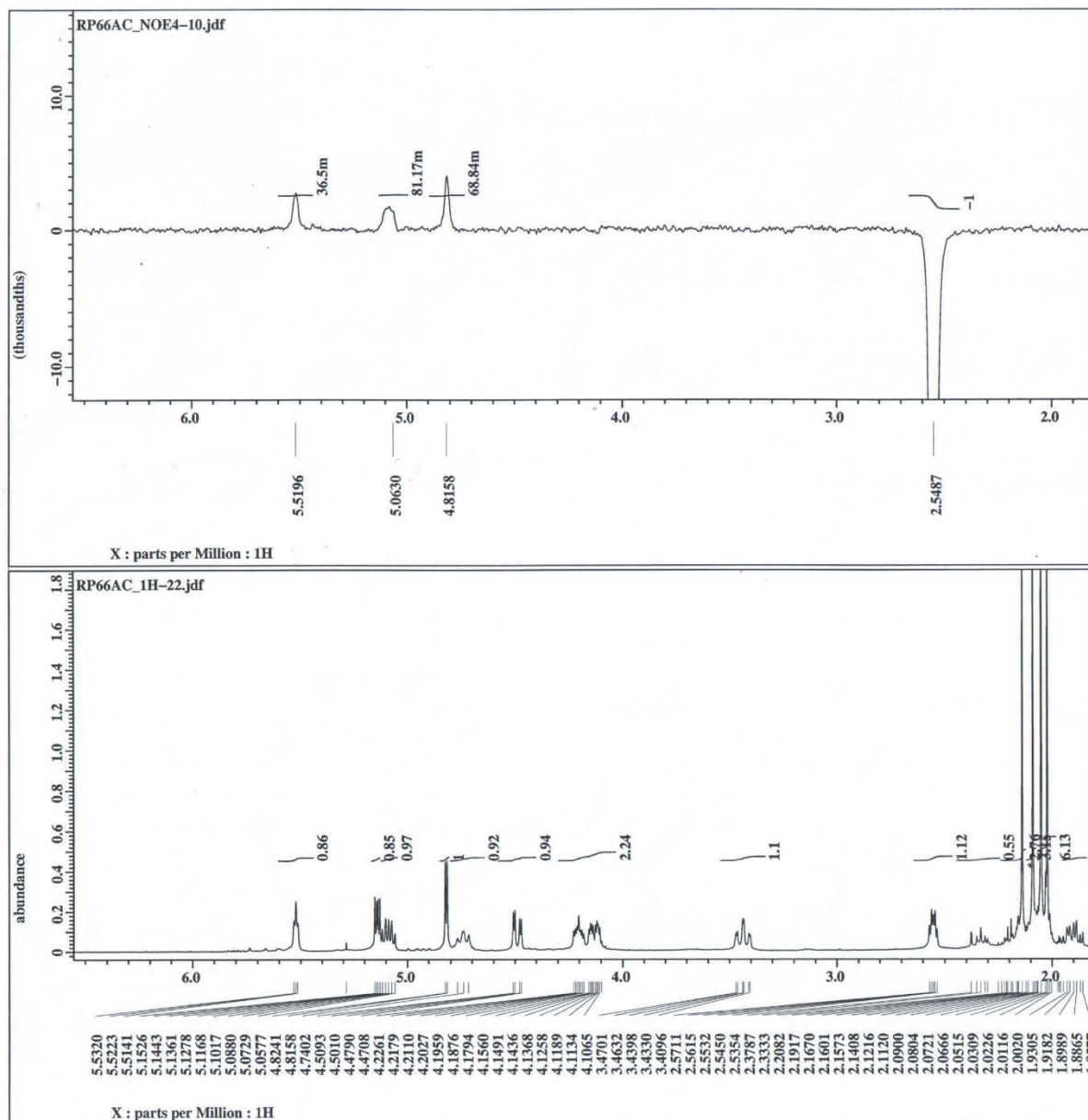
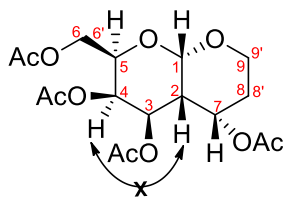


Figure S 43. nOe spectrum of compound 32 for irradiation of H-2.