## **Supplementary Material**

# Scalable synthesis of 3-O functionalized deoxyribonolactones from biorenewable levoglucosenone

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5

0





Figure S1. <sup>1</sup>H NMR spectrum of 2a



Figure S2. <sup>13</sup>C NMR spectrum of 2a

MeO

500

### (1R,2S,5R)-2-methoxy-6,8-dioxabicyclo[3.2.1]octan-4-one (2b)



## Figure S3. <sup>1</sup>H NMR spectrum of 2b



Figure S4. <sup>13</sup>C NMR spectrum of 2b

BnO`

C

,0.

H<sub>3</sub> H

(1R,2S,5R)-2-(benzyloxy)-6,8-dioxabicyclo[3.2.1]octan-4-one (2c)



Figure S5. <sup>1</sup>H NMR spectrum of 2c



Figure S6. <sup>13</sup>C NMR spectrum of 2c

5 0.





Figure S7. <sup>1</sup>H NMR spectrum of 2d



Figure S8. <sup>13</sup>C NMR spectrum of 2d

HC

## (4S,5R)-4-hydroxy-5-(hydroxymethyl)dihydrofuran-2(3H)-one (3a)







Figure S10. <sup>13</sup>C NMR spectrum of 3a

#### (4S,5R)-5-(hydroxymethyl)-4-methoxydihydrofuran-2(3H)-one (3b)





Figure S11. <sup>1</sup>H NMR spectrum of 3b



Figure S12. <sup>13</sup>C NMR spectrum of 3b

₅ HO





Figure S13. <sup>1</sup>H NMR spectrum of 3c



Figure S14. <sup>13</sup>C NMR spectrum of 3c

#### (4S,5R)-4-(allyloxy)-5-(hydroxymethyl)dihydrofuran-2(3H)-one (3d)





Figure S15. <sup>1</sup>H NMR spectrum of 3d



Figure S16. <sup>13</sup>C NMR spectrum of 3d

(1S,1'S,2R,5R,5'R)-6,6',8,8'-tetraoxa[2,3'-bi(bicyclo[3.2.1]octan)]-2'-ene-4,4'-dione 5



Figure S17. <sup>1</sup>H NMR spectrum of 5



Figure S18. <sup>13</sup>C NMR spectrum of 5

#### ((2R,3S)-3-(benzoyloxy)-5-oxotetrahydrofuran-2-yl)methyl benzoate 6





Figure S19. <sup>1</sup>H NMR spectrum of 6



Figure S20. <sup>13</sup>C NMR spectrum of 6

## ((2R,3)-3-(benzyloxy)-5-oxotetrahydrofuran-2-yl)methyl benzoate 7





Figure S21. <sup>1</sup>H NMR spectrum of 7



Figure S22. <sup>13</sup>C NMR spectrum of 7